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AGARD CONFERENCE PROCEEDINGS No. 200

on

Advanced Fabrication Techniques in Powder Metallurgy and Their Economic Implications

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ADVISORY GROUP FOR AEROSPACE RESEARCH AND DEVELOPMENT

ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD

(11) Nov 76 (12) 251p.

(9) AGARD Conference Proceedings No.200

(6) ADVANCED FABRICATION TECHNIQUES IN POWDER METALLURGY AND THEIR ECONOMIC IMPLICATIONS

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Papers and summaries presented at the 42nd Meeting of the AGARD Structures and Materials Panel held in Ottawa, Canada, 4–9 April 1976.

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Published November 1976

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ISBN 92-835-0171-3

Printed by Technical Editing and Reproduction Ltd
Harford House, 7–9 Charlotte St, London, W1P 1HD

PREFACE

The Specialists' Meeting on "Advanced Fabrication Techniques in Powder Metallurgy and their Economic Implications" in Ottawa in Spring 1976 was planned by a Working Group of the Structures and Materials Panel under the same title. Planning started in 1974 by preparing pilot papers and a review of the situation in European NATO countries (AGARD Reports R-627 and R-641).

It was the intention to focus not only on the technological but also on the economic achievements of powder metallurgy techniques. The development of improved materials qualities was of equal priority to the reduction of cost, especially for complicated aerospace parts made from titanium or superalloys. Reduction of the cost of materials processing is an important issue for NATO countries. For the first time, organisation of a meeting of specialists mainly from the materials processing industry was attempted. It was hoped to combine a more economical approach to materials processing, close to the net shape of aircraft components, with improvements in materials quality, especially with regard to homogeneity of structure. Advanced powder metallurgy manufacturing techniques are attractive from both aspects but especially where machining to net shape dimensions dominates the costs. Parts used in jet engines are of main interest but structural applications are also included.

The first part of the Specialists' Meeting, which was attended by about 100 participants from nearly all NATO countries, was concerned with the production of powders. Four different processes of centrifugal atomization as the predominant powder metallurgy manufacturing technique for titanium alloys were presented. Argon and vacuum atomization of processes for nickel base superalloys were described as alternative techniques already proven in volume production.

The second part of the Meeting consisted of reporting the state-of-the-art of techniques for consolidation of titanium and superalloy powders to near net shapes. All stages of the production sequence were discussed; powder handling, canning techniques, consolidation, secondary metal-working and thermal treatments.

The specialists felt that a significant cost saving potential of more than 50% exists, using powder metallurgy processing compared with conventional manufacturing. Hot isostatic processing technology appears to have the most promising potential for production of high quality, lower cost aircraft components in the near future.

Areas recommended for further R & D work included powder contamination, canning technology, new non-destructive inspection approaches and a better understanding of deformation mechanisms during compaction. These problems must be overcome before detailed cost calculations for large scale production can be made.

On behalf of the Structures and Materials Panel I would like to express my thanks to all authors, recorders, discussors and especially to both coordinators, Dr P.W.Sutcliffe and Mr L.P.Clark, for their outstanding contributions and cooperation which led to the success of the Meeting.

Wolfgang BUNK
Chairman, Working Group on
Advanced Fabrication Techniques in Powder
Metallurgy and their Economic Implications

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TRENDS IN THE APPLICATION OF ADVANCED POWDER METALLURGY IN THE AEROSPACE INDUSTRY

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When considering new systems, the cost factor has recently entered in a way that we have not seen for many years. In the last five years, the dominance of cost has outweighed performance in many cases. This trend has created a fact - - a way of life - - that we all have to face as we develop our military and aerospace systems.

About five years ago, we entered into a substantial analysis of a variety of jet propulsion and airframe components - - typified by the superalloy turbine disk shown in Figure 1. We tried to break out the cost to find where the dollars were. By identifying the high cost centers, we could then address technological approaches that would help us reduce those costs. We literally looked at thousands of components throughout the industry in the United States. We followed them all the way from the raw material suppliers, through the parts producers, to the prime contractors. The dominant factor was metal removal. We found this factor prevalent in most of the aeropropulsion, and many of the airframe applications. This drove us immediately to look at why metal removal costs are so high. Typically, we buy ten to twenty times the material which we need and machine away up to 95 percent of it. With raw material costs escalating plus extensive machining, we, therefore, ended up with parts that were extremely expensive. Currently, disks in our propulsion systems run on the order of $20,000 to $35,000 each in both titanium and superalloys. We are looking at very high cost components primarily, we believe, because of the very poor "buy-to-fly" ratios.

I noted that materials costs are rising. Figure 2 shows the dramatic increases in the last two years. Moreover, it is my personal forecast that these prices will go up still more by 1980. Thus, the raw materials which used to account for about 11 percent of the part cost will become a more dominant cost factor. This means that we must seek ways to buy as little material as possible and efficiently translate it into the end product. In addition to reducing the amount of material utilized, we can also pursue reduced machining costs by getting closer and closer to net shapes.

The basic approaches to net shapes include: isothermal forging which is already being used for several parts, more extensive use of casting, a net-shape process, and powder metallurgy. I truly believe that powder metallurgy, at this juncture, looks like our brightest hope for achieving net shapes.

If we look at the way we currently make a component, such as a turbine disk, Figure 3 shows that you start with a 200 pound ingot and go through a number of processing activities to an ultrasonic outline at 70 pounds and then to a final part weight of 11 pounds. A large part of the cost is machining. Again, this says that we need a way to go from the raw material step to the final product step with as close as possible to net shape - - ideally the net shape itself. There is a tremendous amount of money to be saved in labor and processing utilizing a minimum number of steps. Therefore, this accounts for the excitement over powder metallurgy.

In Figure 4, you can see for conventional practice a materials cost of about 20 percent. Five to seven years ago this would have been in the region of 10%. Although material costs have risen dramatically, machining, at 55%, is the dominant cost.

We have current activities underway which we believe in the short term (that is, over the next 3 to 4 years) can result in a savings of about 12 percent as we get closer to net shape and reduce the machining cost. It is the reduction of this machining cost which, at this juncture, is being emphasized by us. I believe that the ultimate potential of the technology and the reason for all of the enthusiasm rests in a potential cost savings of about 50 percent. We believe these savings will be real and that this kind of cost savings potential is available. The process which happens to be spelled out here is Hot Isostatic Pressing (HIP). I'll talk more on that in a moment, but we believe there are a variety of ways to get to these kinds of cost savings, with HIP being one of the more promising. There are, however, certainly other approaches available in terms of powder consolidation.

There are two major thrusts to our current program activities. One involves the preparation of the powder. You will hear about this aspect in much greater detail in some of the other papers which are included in the proceedings. I merely want to point out from a management standpoint that the state-of-the-art in titanium powder is not where it should be. While Figure 5 denotes that the Rotating Electrode Process (REP) quality as "good", I consider this as "spotty good", with much improvement to be made. At this time, REP is not a production process for high quality powder. There is potential within that process, as well as several others, for obtaining improvements in quality. I certainly do not want to leave you with the impression that the titanium powder situation is one in which we can rest comfortably. There is much to be done in the area in terms of cost, as well as quality. You can see in Figure 5 that REP runs $25 to $35 per pound; that is an unacceptable price to pay for powder. Therefore, you will see great activity, both domestic and international, in terms of processes which will improve both the quality and cost of titanium.

In the REP technique, which currently represents one of our only sources for prealloyed titanium powder, a consumable titanium electrode is rotated against a non-rotating tungsten electrode. This is shown schematically in Figure 6. One of the problems is contamination. Also, we are not satisfied with the economics of the process. Currently, developmental activity to solve both these problems is underway. We are removing the contamination as well as enhancing the economics of this process.

I believe that we cannot afford a singular approach - - for example the REP. This area is wide open for innovative thinking and new concepts and I urge you all to consider efforts in this area. It could be very profitable and important to the development of titanium powder metallurgy.

Other potential powder sources are noted in Figure 7. Activity is not restricted to the United States. We see that Leybold Heraeus from Germany and AERE from the United Kingdom are involved. This is a cross-section; there may still be others. As I said, the area is open for contributions. There is a potential for substantial payoff for those who can devise innovative processes for high quality, low-cost titanium powder.

The superalloy powder situation, from our perspective, is a little bit different. We believe the quality of superalloy powder is good. In Figure 8 are shown some of the key techniques for making the powder and the capacities. The major drive we would like to see is a further reduction in cost. This is a dominating factor and we would like to see these prices come down. As I noted, it is our assessment that quality is good enough today to put it into production parts. In fact, as many of you know, IN-100 powder parts are going into the F-100 engine. In the superalloy situation, therefore, activity directed towards cost reduction is appropriate.

Let us now move from the powder itself to the consolidation processes. It is this area in which, in the United States, most activity will center in the near term. As shown in Figure 9, there are a number of techniques available. In addition to HIP (which was mentioned earlier), the list includes Press and Sinter as well as Extrusion. Some of the size and shape limitations are also noted.

Dwelling for a moment on HIP, Figure 10 shows a process schematic. We see the use of molds, filling the powder, the HIP itself, and end with a finished part. Pressures involved are fairly high - - about 15,000 psi. I believe we all have to think in terms of processes in which the capital equipment costs are minimized. HIP is expensive. I want to state unequivocally that the United States has not finalized on HIP as the only way to go. Other processes and techniques will have to be considered. Admittedly, however, at this juncture, much of our activities are geared around HIP.

The largest HIP facility currently in operation is at Battelle Memorial Laboratories, Columbus, Ohio. This unit is capable of temperatures up to 2400°F and pressures to 15,000 psi. The working chamber is 4 feet in diameter and 10 feet long. As we start to think about production applications, we recognize that this kind of equipment is extremely expensive. This is one of the drawbacks to HIP, although I should note that any alternative process may also have drawbacks in one sense or another. Nevertheless, these high costs will certainly stimulate us to look at other consolidation methods. Here, again, imagination and innovation are required. We are at an early stage in the technology and I urge you all to look at alternative ways to consolidate the powders. This will be a key factor, maybe THE key, to the future of this technology.

Some other approaches are noted in Figure 11. Let's look for a moment at vacuum hot pressing - - to give you an example of some of the differences. As noted in Figure 12, we are now talking about pressures on the order of 1000 psi and, therefore, there are dramatic differences in the type of equipment required. I believe there are going to be a number of different approaches, each of which may have certain advantages. At this stage of the process selection, we are still looking for the best approaches. Again, I reiterate, there will be much activity in the United States geared to the development of new consolidation processes. They are critical to the future of this new technology in terms of both quality and economics.

As we look at powder metallurgy, the reason for the great interest in the United States, and throughout the NATO member nations, is the cost reduction potential. That is the main driving force. There are other advantages, however, and I feel that it is important to note some of these. In the long run, they may turn out to be very large driving forces in themselves. As shown in Figure 13, these include things like improved properties. We believe that we can achieve properties better than wrought properties, in some cases. We believe there are advantages in terms of uniformity, machinability and our ability to inspect. I'll touch more on that in a moment. The powder process also affords us the ability to produce alloys which can not be made by any other process. Thus, it can open new materials horizons to us. These are significant advantages and, in many cases, may be a dominant advantage. And so, as we look to the future, I don't - - and I do not think any of us should - - think of powder metallurgy as simply a cost-reduction approach. It certainly has that potential and it would be foolish to overlook it. My point is that it is bigger than that, and these other factors may be dominant as we go into the future.

Looking briefly at properties, both titanium and PA-101 superalloy, are compared with specification values in Figure 14. We find ourselves with very satisfactory properties. While there is much more data to be generated, we are close to a statement that would say powder metallurgy is going to offer us at least equivalence and, in many cases, advantages over wrought properties. Again, a lot more data has to be generated, but at this juncture, it looks extremely promising.

I believe that ultimately powder metallurgy will afford us improved inspectability. However, in this drive towards net shape, we find non-destructive inspection (NDI) playing a very important role. Our ability to accept net shapes requires the matching development of an NDI capability to assure that you can legitimately inspect these parts and put a stamp of approval on them. This is critical to us. And so we find, and you should be aware, that net shape technology may be thwarted by an inability of NDI to accurately and reliably determine the quality of the parts. In the United States, we are extending a two-pronged effort to match NDI to the powder consolidation activities. This is critical if you are going to use net shapes in actual applications.

There are some additional considerations noted in Figure 15. Again, all of these are positive factors for powder metallurgy. In the environment we are now in, and I know you are in also, we find energy conservation a major issue. We believe that powder metallurgy will offer substantial savings of energy, particularly in terms of a reduction in machining required up and down the line. Ecologically, there is absolutely no comparison between the way powder parts are manufactured and the way we conventionally go from ingot to billet and on down to the final hardware. We are now forced to live with these considerations in production and powder metallurgy can offer major technological advantages.

We must also consider strategic materials conservation. We do not have all of the raw materials that we need; they are going to run out. Therefore, we must become more efficient in their utilization. We can no longer afford to take 20 pounds, machine it down to one pound, and throw 19 pounds away. This reduction of input weight is a major advantage which will have a growing importance to all countries.

As we look to the future, I've summarized our requirements in Figure 16. We will be looking at improved powder production techniques for high quality. This will be directed primarily to titanium but we certainly will look for improvements in superalloys as well. I look for the major thrust to involve titanium quality and the cost of both metals.

In terms of consolidation methods, the powder handling and canning for net shapes are arenas for innovation. We want to obtain a product which is economical and does not involve a massive capital investment. We also want to produce reliable, consistent hardware. Finally, we've got to match non-destructive inspection with our product form.

Looking at the spinoff potential in Figure 17, with success in this area, we see expanded usage of titanium and superalloys in military applications. I believe that powder metallurgy can open opportunities for titanium in areas where we would like to use it, but, because of the cost, we currently use steel or aluminum instead. A 50 percent reduction in part cost would certainly result in substantially expanded use. I'm not as optimistic in terms of expanding the applications of superalloys, although, I believe that we can make the current applications much more efficient.

We believe that spinoffs to commercial aviation will be fairly direct and immediate. Thus, we're not restricted to military applications. There are already powder parts, although not of superalloy and titanium, in the commercial marketplace. A spinoff of these products could result in a significant market in the non-aerospace arena. That is difficult for me to forecast, since it's not my background. However, I do believe that low cost titanium can open up significant consumer markets.

The last items in Figure 17 concern translation of this technology to other materials. I talked primarily about titanium and superalloy, but feel very strongly that the technology will apply to steel, aluminum, and other materials as well. For example, I believe it can open up applications in aluminum parts with much improved fatigue life compared to today's parts. This is one limitation with current aluminum structures. Another possibility is aluminum alloys with 100 to 150 degrees higher temperature capability. And so, we do not (nor should you) draw the line on powder metallurgy around titanium and superalloys alone.

I'd like to close with one thought. There are those who say that the way of the future is with composite materials. Therefore, why do we bother with improvements in metals? I can tell you that, from my perspective in the Air Force Materials Laboratory - - and I have been an exponent of composite materials, a strong one - - I do not believe airplanes of the future, civil or military, will be made 100 percent of any one kind of materials. I believe that in the future we have got to offer the designer as many options as we can for different vehicles with different missions and requirements. Speaking to you as a composites man, powder metallurgy is probably the brightest hope that we have in metals that can have an impact on the airplanes, the weapons systems and the commercial systems of the future.

I leave you with the message that, although we have not reached the top in this technology, the promise is bright. We have no option but to pursue it vigorously. I urge us all to work together cooperatively wherever and whenever we can. The area is big enough and important enough for international cooperation. It's certainly something we should strive for and the benefits would accrue to us all.

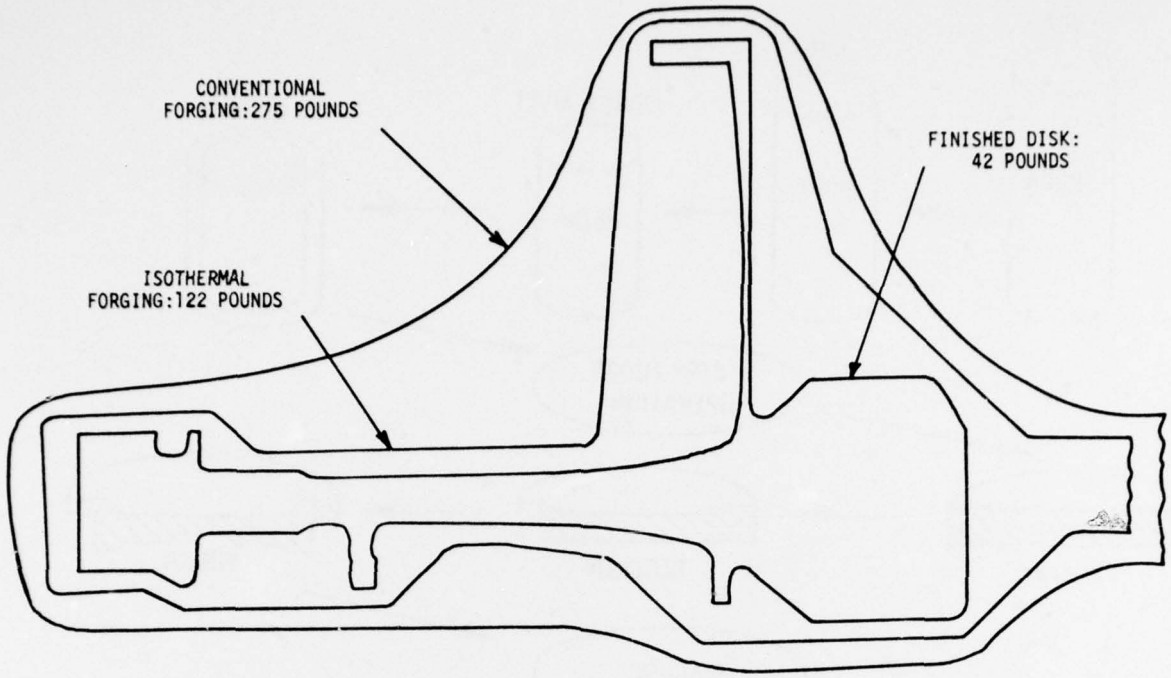


Figure 1. State-of-the-Art Turbine Disk Forging

MATERIAL	% INCREASE
● CARBON BAR AND BILLET	41
● VAR STRUCTURAL ALLOYS (300M)	45
● HIGH TEMPERATURE ALLOYS (Ni)	47
● STAINLESS STEEL (304)	38
● TITANIUM (Ti 6-4 BILLET)	46
● DIE BLOCKS AND DIE STEELS	20

Figure 2. Raw Material Price Increases, January 1974 to January 1976

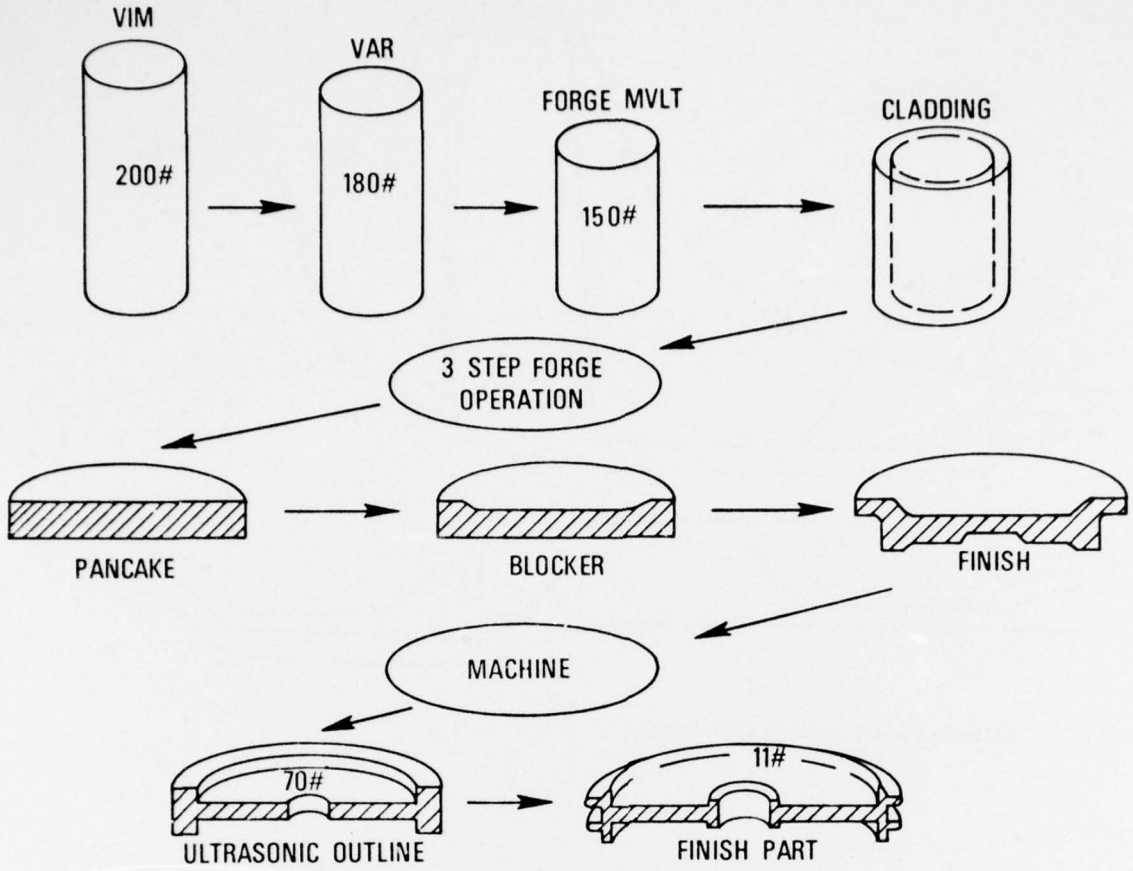


Figure 3. Conventional Processing for Superalloy Turbine Disk

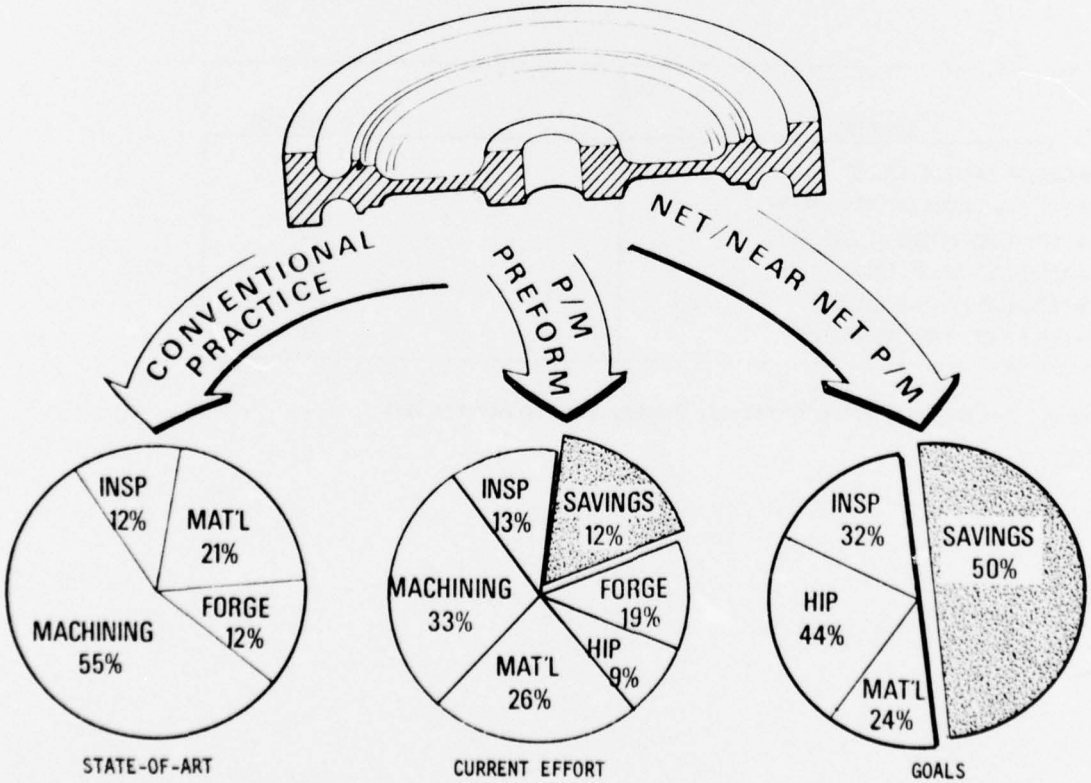


Figure 4. Powder Metallurgy Potential Cost Savings for Typical Superalloy Turbine Disk

POWDER PROCESS	QUALITY	COST ($/LB)	INDUSTRY CAPACITY (LB/YR)
● ROTATING ELECTRODE	GOOD	25-35	150,000
● HYDRIDE - DEHYDRIDE	MARGINAL	45	26,000
● BLENDED ELEMENTALS	MARGINAL	4-6	SUFFICIENT

Figure 5. Titanium Powder Metallurgy State-of-the-Art Powder Production.

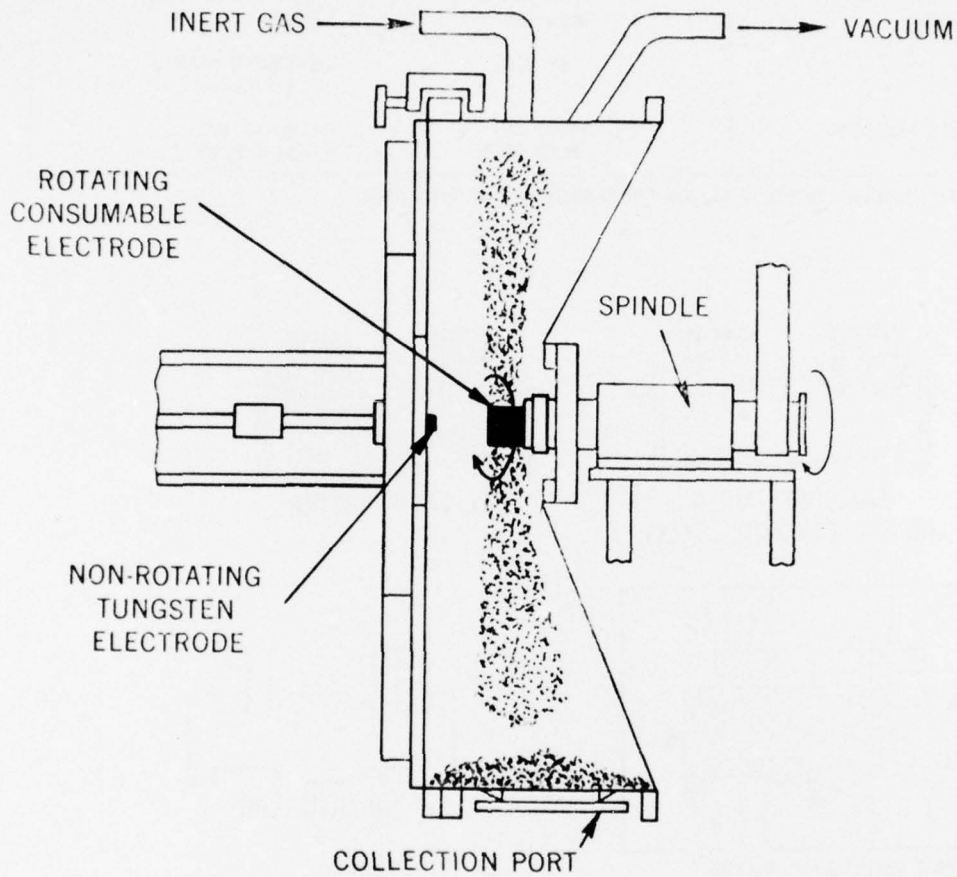


Figure 6. Rotating Electrode Process

● EBRD	LEYBOLD HERAEUS/GERMANY
● CSC	AERE/UNITED KINGDOM
● CMRC	CRUCIBLE STEEL/UNITED STATES
● ROTOTRODE	SCHLIENGER INDUSTRIES/UNITED STATES
● MELT EXTRACTION	BATTELLE MEMORIAL INSTITUTE/UNITED STATES

Figure 7. Potential Sources for Advanced Titanium Powder Production

POWDER PROCESS	QUALITY	COST ($/LB)	INDUSTRY CAPACITY (LB/YR)
● ARGON ATOMIZE	GOOD	7-10	$>5X10^6$
● VACUUM ATOMIZE	GOOD	8-12	310,000
● ROTATING ELECTRODE	GOOD	18-30	200,000

Figure 8. Superalloy Powder Metallurgy State-of-the-Art Powder Production

PROCESS	LIMITATIONS	
	SIZE	SHAPE
● PRESS & SINTER	<50 IN^2	PRESSED SHAPES
● EXTRUSION	6" DIA.	EXTRUDED SHAPES & BILLETS
● HOT ISOSTATIC PRESSING (HIP)	>4000 IN^2 PLAN AREA	COMPLEX DIE CAST SHAPES

Figure 9. Powder Metallurgy State-of-the-Art Consolidation Processes

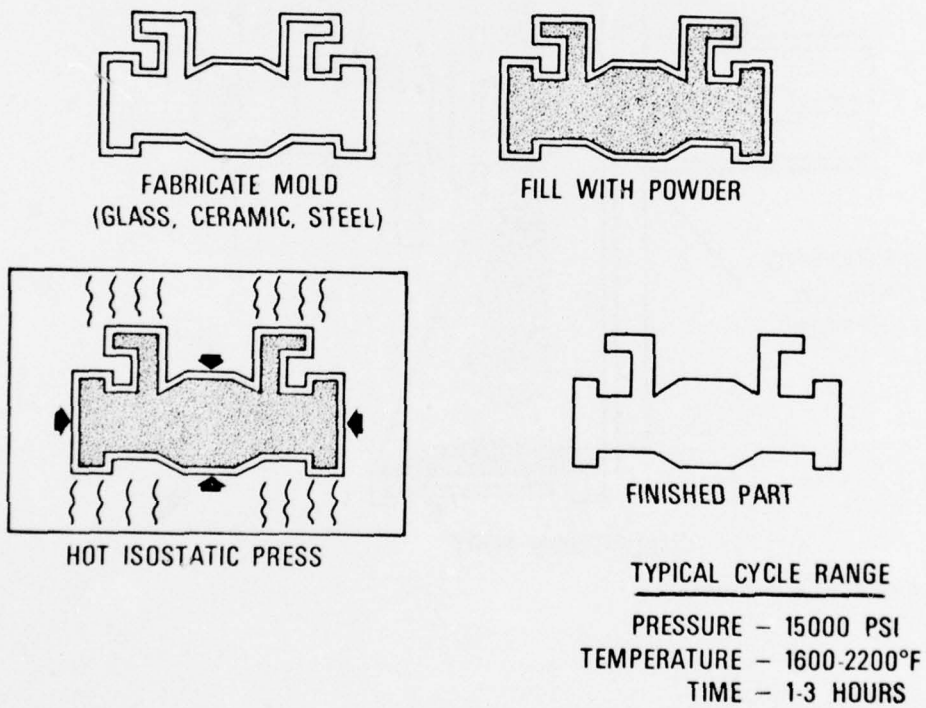
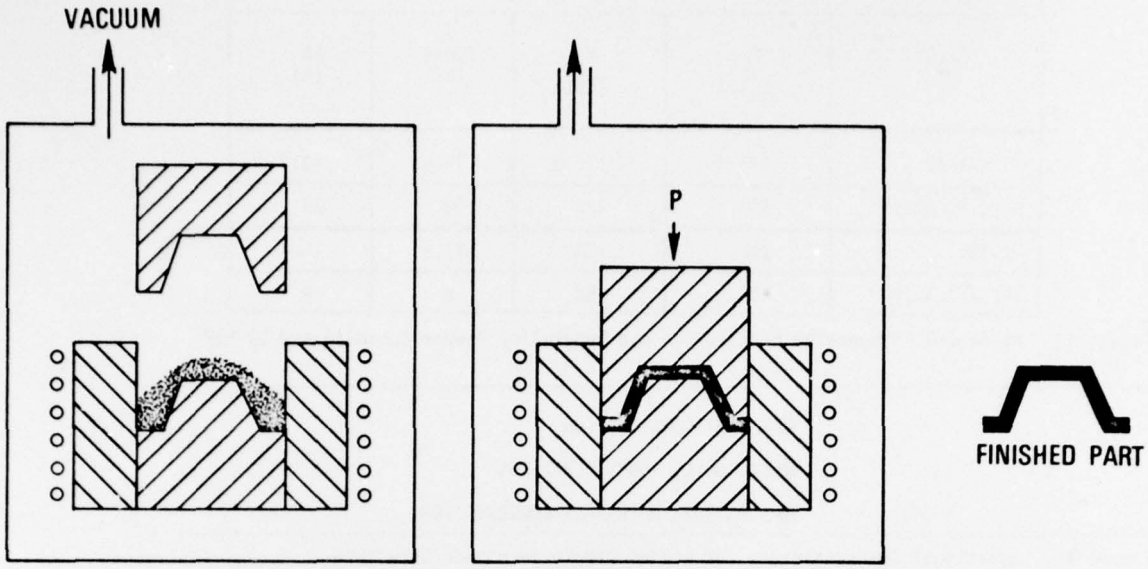


Figure 10. HIP Process for the Production of Near Net Shapes from Powder

- VACUUM HOT PRESS (TITANIUM)
- SOFT CAN FORGING (SUPERALLOYS)
- VACUUM SINTERING (SUPERALLOYS)

Figure 11. Additional Consolidation Methods for Advanced P/M Products



TYPICAL CYCLE

PRESSURE – 1000 PSI
TEMPERATURE – 1750°F
TIME – 1 HOUR

Figure 12. Vacuum Hot Press (VHP) for the Production of Net P/M Shapes in Titanium

COST REDUCTION

IMPROVED PROPERTIES

- HOMOGENEOUS MICROSTRUCTURES
- UNIFORM MECHANICAL PROPERTIES
  - THICK SECTIONS
  - DIRECTIONALITY
- IMPROVED MACHINABILITY
- GREATER ULTRASONIC TRANSPARENCY
- POTENTIAL FOR NEW, IMPROVED ALLOYS
  - NON-CASTABLE
  - NON-WORKABLE

Figure 13. Advantages of P/M Products

ALLOY	F_{tu} (KSI)	F_{ty} (KSI)	ELONG. (%)	RA (%)
Ti-6Al-4V	137.4	125.9	18.8	43.2
SPECIFICATION	130	120	10	25
PA-101	227	153	11.5	15.4
SPECIFICATION	180	150	8	8

Figure 14. Mechanical Properties of Titanium and Superalloy Powder Consolidated by HIP

- ENERGY CONSERVATION
- ECOLOGICAL CONSIDERATIONS
- STRATEGIC MATERIALS CONSERVATION

Figure 15. Additional Considerations for Use of P/M in Aerospace Structure

- IMPROVED POWDER PRODUCTION TECHNIQUES
  - HIGH QUALITY
  - LOW COST
- CONSOLIDATION METHODS
  - POWDER HANDLING
  - CANNING METHODS FOR NET SHAPES
- NONDESTRUCTIVE INSPECTION

Figure 16. Key Developmental Requirements for P/M

- BROADER USE OF TITANIUM/SUPERALLOYS IN MILITARY APPLICATIONS
- DIRECT APPLICATIONS TO COMMERCIAL AVIATION
- GENERIC APPLICATIONS
  - INCREASED USE OF TITANIUM IN CONSUMER MARKET
  - TRANSITION OF TECHNOLOGY TO OTHER MATERIALS

Figure 17. Spinoff of Advanced P/M Technology

SESSION I

PRODUCTION DE POUDRES D'ALLIAGES DE TITANE PAR FUSION-CENTRIFUGATION SOUS-VIDE

par

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RESUME. Nous présentons dans cette étude un procédé de fabrication de poudres d'alliages TA6V et TA6Z5D élaborées par fusion centrifugation sous bombardement électronique. Un dispositif de capacité industrielle pour la production de poudres métalliques est décrit et les caractéristiques des poudres ainsi élaborées sont exposées. Des pièces massives ont été mises en forme par frittage et filage à chaud entre 850°C et 1100°C. Les structures et les propriétés mécaniques à froid des produits densifiés sont comparées avant et après traitement thermique.

Le titane et ses alliages présentent un intérêt considérable dans le monde actuel, particulièrement dans le domaine aéronautique où tous les efforts tendent à obtenir des propriétés mécaniques supérieures pour des poids de matériaux utilisés toujours plus faibles. Cette notion, liée à l'accroissement des performances est aujourd'hui tempérée par des notions d'économie : économie énergétique quant au choix des alliages utilisés et quant au choix des procédés de transformation. En effet, le poids d'alliage de titane utilisé pour réaliser une pièce est souvent 10 à 20 fois supérieur à celui de la pièce finie. La métallurgie des poudres permet, dans le cas des alliages de titane, d'approcher, au cours de la mise en forme, les cotes définitives, tout en conférant à l'alliage une plus grande homogénéité de structure en éliminant les ségrégations dues à la solidification de lingots de grande dimension, ségrégations qui ne disparaissent pas complètement après forgeage.

Les alliages à base titane étant sensibles à l'oxydation, surtout à l'état divisé, nous allons exposé une méthode industrielle de fabrication de poudres d'alliages de titane élaborées sous vide et comparer rapidement les propriétés mécaniques obtenues après densification par filage à chaud et par frittage sous charge.

I - ELABORATION DES POUDRES

I.1. Description du procédé. Le procédé mis en oeuvre fait appel à la centrifugation sous vide d'un alliage liquide fondu sous l'impact d'un faisceau d'électrons. Une lingotière à axe vertical refroidie à l'eau, reçoit grâce à un dispositif de chargement automatique des électrodes de diamètre 50 mm et de longueur 200 mm (figure 1). L'extrémité de l'électrode, maintenue hors de la lingotière est soumise à l'impact d'un faisceau d'électrons issu d'un canon à accélération interne. On obtient la fusion locale de l'extrémité de l'électrode, et par centrifugation, expulsion de la zone fondue qui éclate en gouttelettes. Ces dernières se solidifient sur leur trajectoire après avoir heurté un écran refroidi à l'eau qui permet de limiter les dimensions de l'enceinte à vide, constituant le four de fusion, à des dimensions raisonnables. Les sphérules ainsi formées sont recueillies dans la partie inférieure du four. La difficulté, représentée par l'écrasement des particules sur l'écran refroidi, a été résolue en modifiant la tension interfaciale entre la gouttelette liquide et le support jouant le rôle d'obstacle. La capacité de production du dispositif dans sa géométrie actuelle en utilisant des électrodes d'alliages de titane de 50 mm de diamètre est de 7 tonnes/an.

I.2. Composition des alliages étudiés. Deux alliages de titane ont été étudiés. Les compositions nominales sont les suivantes :

Electrodes	V %	Aℓ %	Zr %	Mo %	Si %	C %	O_2 %	N_2 %	H_2 %	Ti
TA 6 V	4,2	6,56	-	-	-	0,045	0,160	0,006	0,004	Bal.
TA 6 Z 5 D	-	6,3	5	0,56	0,15	0,023	0,140	-	0,007	Bal.

I.3. Paramètres technologiques de la centrifugation. Les électrodes de diamètre 50 mm de ces deux alliages ont été centrifugées dans une enceinte à la pression de 10^{-4} torrs avec une vitesse de centrifugation de 4000 tours/minute et une avance de 5 cm/minute soit une vitesse d'ablation de l'alliage de 30 kg/heure. La puissance électrique délivrée par le canon en cours de fonctionnement est de 31 KW sous une tension de 24 KV.

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1.4. Composition des poudres élaborées. Après centrifugation, l'analyse des sphérules est la suivante.

Sphérules	V %	Aℓ %	Zr %	Mo %	Si %	C %	O_2 %	N_2 %	H_2 %	Ti %
TA 6 V	4,35	6,55	-	-	-	0,010	0,120	0,003	0,0012	Bal.
TA 6 Z 5 D	-	6,3	5	0,56	0,16	0,030	0,150	-	0,0006	Bal.

La diminution de la teneur en éléments interstitiels est plus sensible dans le TA 6 V que dans le TA 6 Z 5 D qui semble subir une légère carburation par l'atmosphère carburante du four et qui paraît plus sensible à l'oxydation, probablement du fait de la présence du zirconium dans la composition de l'alliage.

1.5. Répartition granulométrique et bilan matière. La répartition granulométrique est représentée sur le tableau suivant.

Dimensions des sphérules en microns	TA 6 V	TA 6 Z 5 D
$0 < \phi < 100$	3,51	1,20
$100 < \phi < 315$	33,21	32,98
$315 < \phi < 630$	36,34	45,57
$630 < \phi < 1000$	26,94	17,87
"Splat.cooling"	-	2,38
	100	100
Rendement de centrifugation	85,35 %	82 %

Le bilan matière s'établit comme suit :

	TA 6 V	TA 6 Z 5 D
Poids des électrodes	100	100
Poids de sphérules récupérées	85,35	81,94
Poids de sphérules applaties ou déformées	-	2
Poids des électrodes à recycler	9,20	8,60
Poids des sphérules agglomérées à recycler	1,45	2,88
Pertes	4	4,58

1.6. Examens micrographiques. La micrographie des particules (figure 2) montre l'aspect dendritique habituel dû au refroidissement rapide des sphérules dans le four à partir des conditions initiales de mise en solution. On remarque la distribution aciculaire $\alpha + \beta$ avec la précipitation primaire de β dans les joints de grains. Les distances interdendritiques de 10 à 20 µ traduisent un refroidissement rapide, mais sans effets de trempe, dû à l'absence de convection dans l'enceinte du four.

Un examen à la microscopie électronique ne montre pas de variation sensible dans la répartition des éléments entre le centre et la périphérie des sphérules. Le zirconium, en particulier, est bien réparti.

2 - CONSOLIDATION DES POUDRES.

Deux procédés de mise en forme ont été étudiés en fonction de différentes utilisations potentielles. Dans ce qui suit, on va comparer - en utilisant la fraction granulométrique comprise entre 315 et 630 µ - les alliages de titane densifiés en utilisant soit un procédé de filage à chaud dans le domaine $\alpha + \beta$ et dans le domaine β, soit un procédé de frittage sous charge en matrice non rigide dans le domaine β essentiellement. Le premier procédé est intéressant pour la mise en forme des produits longs ou de profilés, le second convenant mieux à l'élaboration de produits plats, mais de grand diamètre comme les disques.

2.1. Consolidation par filage à chaud.

2.1.1. Description du procédé. La consolidation des poudres a été effectuée en mettant en oeuvre un procédé de filage hydrostatique à haute température, grâce à un dispositif qui s'adapte sur une presse à filer conventionnelle. Ce dispositif est un grain de poussée mobile, situé à l'arrière du conteneur de la presse et assurant l'étanchéité à l'aide d'un joint en cuivre (figure 3). Cet ensemble, appelé tête d'étanchéité, est placé à l'arrière du conteneur de la presse dans laquelle on a préalablement introduit, à la température choisie, la billette à filer et le transmetteur de pression en quantité convenable.

Le fouloir de la presse agit directement sur ce grain de poussée qui réalise, par déformation du joint de cuivre, une étanchéité dynamique jusqu'à une pression de 13 Kb (pression admissible sur le fouloir) et pour des températures atteignant 1300°C. On peut utiliser un tel dispositif, soit en filage hydrostatique et/ou en compression isostatique. En effet, il suffit d'arrêter la course du fouloir avant la fin du filage de façon à laisser un "culot de filage" dans le conteneur. L'examen de ce culot permet d'analyser la consolidation de la poudre après une compression à la contrainte hydrostatique utilisée pour le filage.

2.1.2. Préparation de la billette de filage. La poudre est disposée dans une enveloppe cylindrique en acier doux qui est fermée sous vide par bombardement électronique. La billette est réchauffée dans un four électrique et transformée avec un taux de réduction connue par une opération de filage hydrostatique. Dans un premier temps, on a une compression isostatique de la billette pendant la montée en pression ; ensuite, la pression nécessaire pour filer est atteinte et l'alliage est éjecté hors de la filière.

2.1.3. Considérations techniques sur l'opération de filage. Les essais de filage qui ont été effectués dans le domaine $\alpha + \beta$ et β entre 850°C et 1050°C permettent de déterminer le coefficient de résistance à la déformation K, en fonction de la température et d'en déduire les rapports de réduction possible pour une contrainte hydrostatique donnée. La valeur de ce coefficient de résistance à la déformation est donnée par la formule suivante :

$$K = \frac{F}{S} \times \frac{1}{L_n R_a}$$

où F est l'effort
S la section du conteneur
R_a le rapport de réduction.

La figure 3 décrit l'évolution du coefficient maxima de résistance à la déformation pour les deux alliages TA 6 V et TA 6 Z 5 D en fonction de la température de filage. On trouve également sur la figure 3 la comparaison des résistances maxima à la déformation d'un alliage TA 6 V, l'un filé à partir de poudre, l'autre filé à l'état massif avec une gaine en acier doux.

On peut effectuer les observations suivantes :

- La densité théorique est atteinte quelle que soit la température de filage dans le domaine $\alpha + \beta$ comme dans le domaine β.
- La résistance à la déformation du produit pulvérulent est plus faible que celle du produit massif sous gaine, toutes conditions opératoires étant égales. Il sera donc possible d'obtenir des rapports de réduction plus importants à partir de billettes préparées par la métallurgie des poudres.
- L'état de surface après dégainage chimique est légèrement ligneux (figures 4 et 5). La profondeur des défauts reste faible (inférieure au 1/10è mm), mais elle est plus importante lorsque le filage est effectué dans le domaine $\alpha + \beta$. On observe une situation inverse pour le filage des produits massifs en alliages de titane où la profondeur des défauts est maxima pour le filage dans le domaine β.

2.1.4. Examens micrographiques. Les examens micrographiques ont permis de déterminer les domaines de phases dans lesquels on a effectué les filages comme le montre le tableau suivant.

	Domaine $\alpha + \beta$	Domaine β
TA 6 V	850. 900. 950°C	1000. 1050°C
TA 6 Z 5 D	850. 900. 950. 1000°C	1050°C

La taille des grains β après filage ne dépasse pas 100 microns, même pour les essais effectués à 1050°C. La figure 6 montre quelques aspects micrographiques après filage.

2.1.5. Propriétés mécaniques. Les caractéristiques mécaniques de traction ont été déterminées à l'ambiante

- sur le produit brut de filage,
- sur le produit filé ayant subi un traitement thermique de revenu de 700°C - 1 heure - refroidissement à l'air pour le TA 6 V ; et un traitement de trempe à l'huile de 1 heure à 1050°C suivi d'un revenu de 24 heures à 550°C, refroidissement à l'air pour le TA 6 Z 5 D.

Les figures 7 et 8 montrent l'évolution des caractéristiques en fonction de la température de filage. On peut faire les observations suivantes :

- il y a peu d'évolution dans les propriétés mécaniques après filage et après revenu pour l'alliage TA 6 V.
- la ductilité de l'alliage TA 6 V reste élevée (15% d'allongement à rupture) et quasi constante dans le domaine $\alpha + \beta$ comme dans le domaine β où l'on observe une diminution importante de la limite élastique.
- la forte augmentation de la charge à la rupture du TA6Z5D filé à 950°C est obtenue à ductilité constante.

2.2. Consolidation par frittage sous charge.

2.2.1. Description du procédé. La consolidation des poudres de TA 6 V et TA6Z5D a été réalisée dans un four de frittage sous charge en matrice flottante déformable. Les pressions mises en jeu dans un dispositif de frittage sous charge étant limitées au maximum à 1000 bars, il est nécessaire, pour éliminer toute porosité résiduelle, d'opérer à haute température et en tolérant un écoulement latéral contrôlé de l'alliage en cours de densification. Il est donc pratiquement impossible d'exécuter une consolidation de poudres d'alliages de titane par frittage sous charge dans le domaine $\alpha + \beta$.

2.2.2. Préparation du container de frittage. La poudre d'alliages de titane est disposée dans un container cylindrique en acier donc l'épaisseur des parois a été choisie en fonction du diamètre du disque à densifier. Deux pistons en graphite à haute résistance permettent la compression de la poudre en disposant entre cette dernière et le graphite des feuillards en molybdène pour éviter toute diffusion. L'ensemble est porté sous vide à la température de frittage.

2.2.3. Opération de frittage. Du fait des faibles contraintes mises en jeu, le frittage est réalisé dans le domaine β entre 1100 et 1150°C après un palier d'homogénéisation de 30 minutes à la même température. La compression est effectuée très rapidement en 60 secondes entre 500 et 750 bars. Le refroidissement de la pièce après compression est contrôlé.

2.2.4. Examens micrographiques. Les examens micrographiques (figure 9) révèlent une décomposition de la phase β initiale avec la formation d'un liseré de phase α primaire, principalement aux joints des grains. On observe également la nucléation et la croissance d'aiguilles de la phase α. La taille des grains de l'alliage TA6Z5D (280 μ en moyenne) est légèrement plus petite que celle des grains de TA6V (350 μ) toutes conditions de frittage étant égales. Après frittage, l'alliage TA6Z5D présente une dureté de 54 R_C alors que la dureté du TA6V est de 52 R_e. Nous n'avons pas observé de porosité, ni de traces de sphérules constituant la poudre dans les coupes effectuées.

Un examen à la microsonde électronique montre pour les deux alliages, un enrichissement des aiguilles en aluminium, alors que le liseré entourant les aiguilles du TA6V révèle un enrichissement en vanadium et le liseré du TA6Z5D un enrichissement en molybdène et en zirconium. Dans cet alliage le silicium est réparti de façon uniforme.

2.2.5. Caractéristiques mécaniques. Les caractéristiques mécaniques de traction ont été déterminées à l'ambiante sur le produit brut de frittage. Les résultats sur produits recuits à différentes températures seront publiés ultérieurement. Les résultats sont résumés dans le tableau ci-dessous.

	Pression de frittage bars	$R_{0,2\%}$ daN/mm²	R daN/mm²	A_R %	A_T %	Z %
TA 6 V	400	85	92,7	6,1	10,5	19
	500	86	93	6	12,2	30
TA 6 Z 5 D	400	87	94,1	5	12	25
	500	87	94	5	10,2	20

On peut faire les remarques suivantes :

- les caractéristiques mécaniques de l'alliage TA6V fritté sont supérieures à celles observées pour le même alliage fondu-forgé, ayant subi un traitement d'homogénéisation de 2 heures en β, un recuit sous vide entre 600° et 900°C. La limite d'élasticité est supérieure à celle d'un alliage fondu-forgé laminé à froid et recuit à 800°C. La ductilité (A %) est importante avec 12% d'allongement à la rupture, valeur supérieure ou égale à celle des alliages laminés à chaud et à froid et recuits à 800°C.

- les caractéristiques mécaniques de l'alliage TA6V fritté sont sensiblement égales à celles du TA6V filé avec un rapport de réduction de 14 à une température supérieure à 1000°C, c'est-à-dire dans le domaine β avec une structure aciculaire.

- les facies de rupture en microfractographie révèlent pour les deux alliages une rupture du type ductile à cupules.

CONCLUSIONS.

La réalisation de pièces frittées ou filées, présentant des caractéristiques mécaniques élevées, a pu être démontrée en mettant en oeuvre la métallurgie de poudres des alliages de titane avec pulvérisation sous vide.

Le frittage sous charge dans le domaine β, en utilisant une technologie d'écoulement dirigé de l'alliage en cours de compression et une vitesse de refroidissement contrôlé, a permis la réalisation de disques sans porosité résiduelle, qui permet d'entrevoir des solutions pour l'obtention directe de pièces sans transformations ultérieures.

REMERCIEMENTS.

Nous tenons à remercier la Direction des Recherches et Moyens-Essais pour l'aide qu'elle nous apportée et plus particulièrement Monsieur BESSONNAT qui a encouragé cette étude et en a suivi le développement.

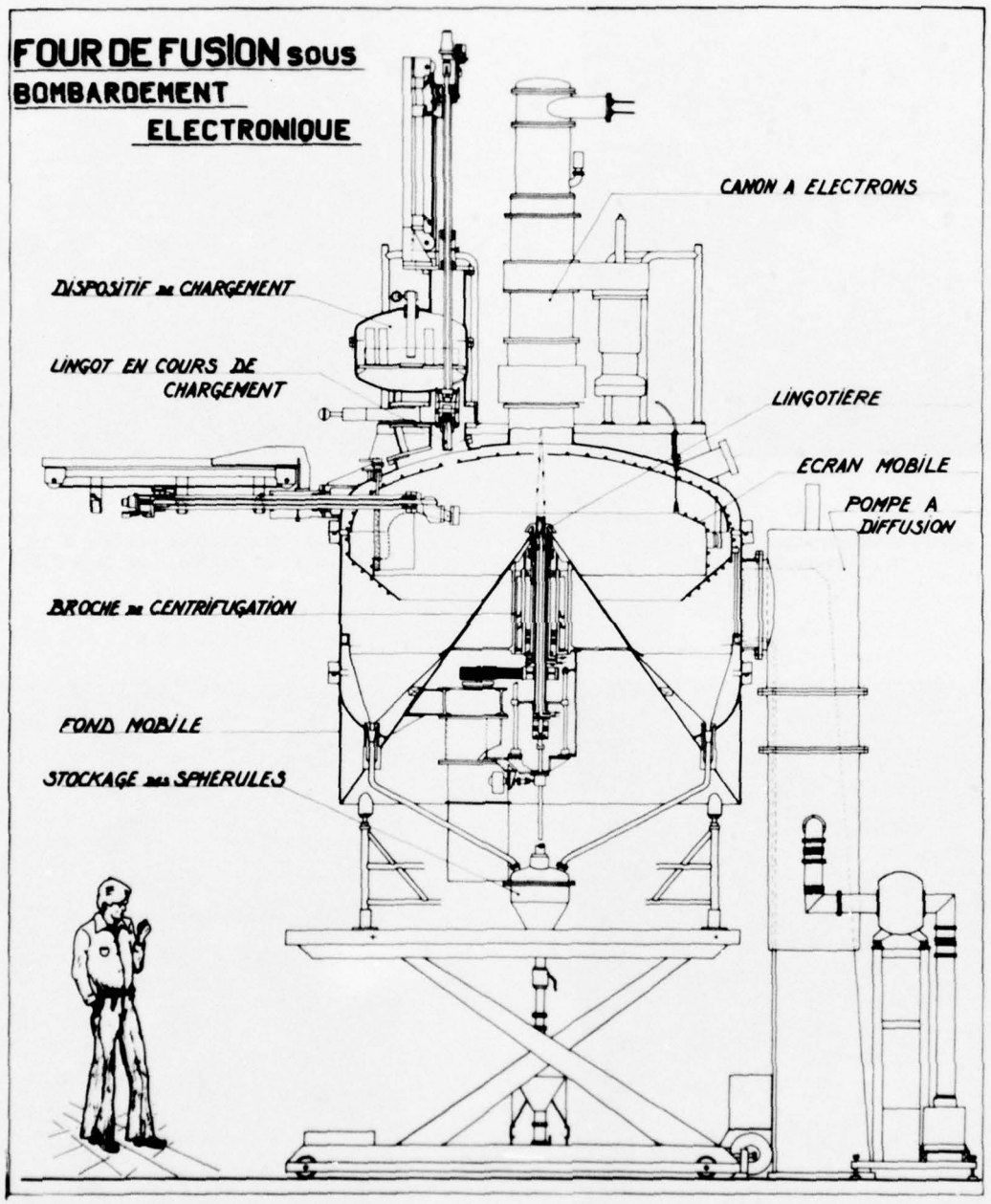
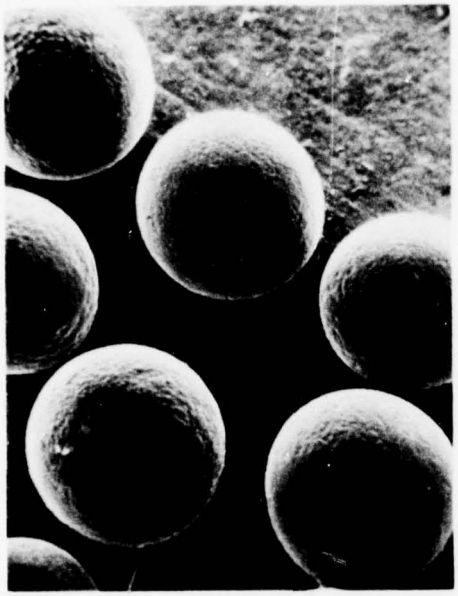

FOUR DE FUSION sous
BOMBARDEMENT
ELECTRONIQUE
CANON A ELECTRONS
DISPOSITIF DE CHARGEMENT
LINGOT EN COURS DE
CHARGEMENT
LINGOTIERE
ECRAN MOBILE
POMPE A
DIFFUSION
BROCHE DE CENTRIFUGATION
FOND MOBILE
STOCKAGE DES SPHERULES

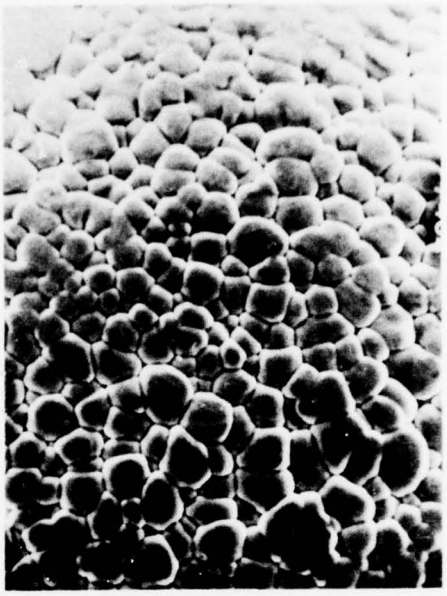
FIGURE 1

STRUCTURES METALLOGRAPHIQUES DE SPHERULES D'ALLIAGES DE TITANE



160µ

Aspect macroscopique des sphérules d'alliages de titane



20 µ

Structure dendritique visible à la surface d'une sphérule de TA 6 Z 5D

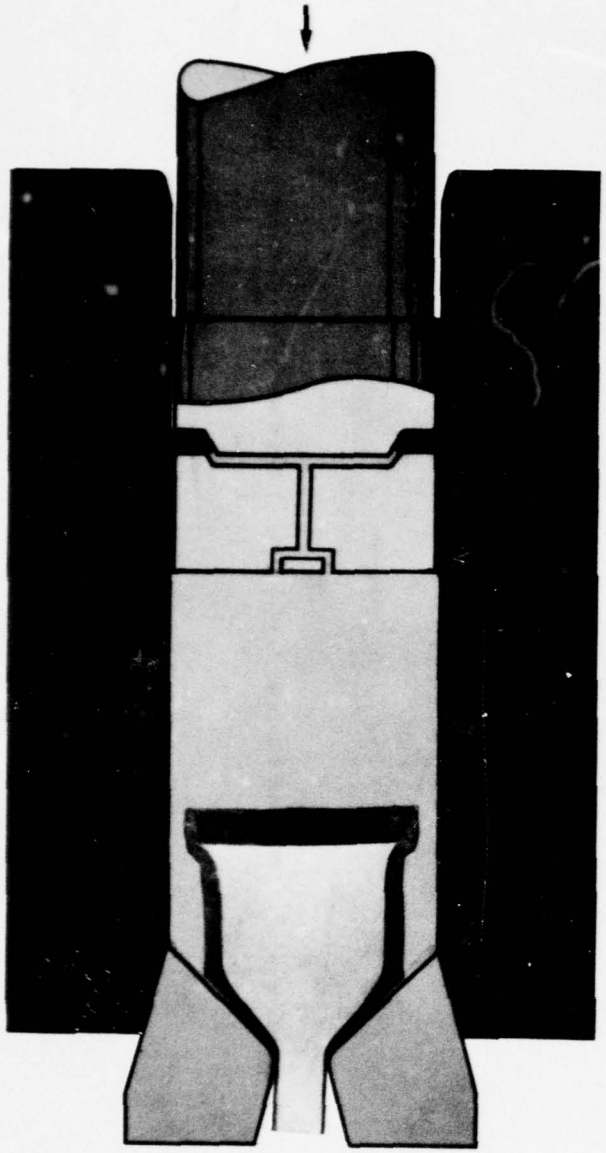
TA 6 V

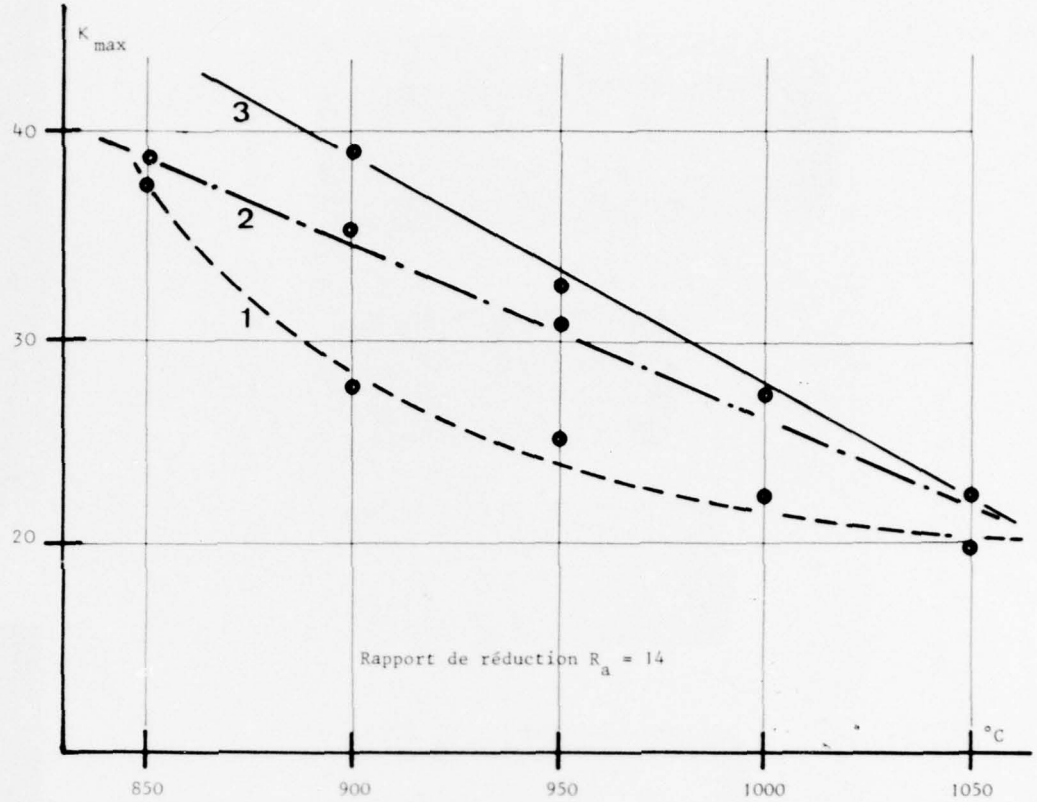
20 µ

TA 6 Z 5 D

La fusion centrifugation des alliages de titane, suivie d'un refroidissement très rapide entraîne une structure martensitique du type α' avec des aiguilles très fines et très peu de phase α dans les ex-joints de grain β (liseré blanc)

FIGURE 2





DISPOSITIF DE FILAGE HYDROSTATIQUE DE POUDRE SOUS GAINAGE ACIER

Evolution du coefficient maxima de résistance à la déformation des alliages de titane filés à l'état massif ou à partir de poudres.

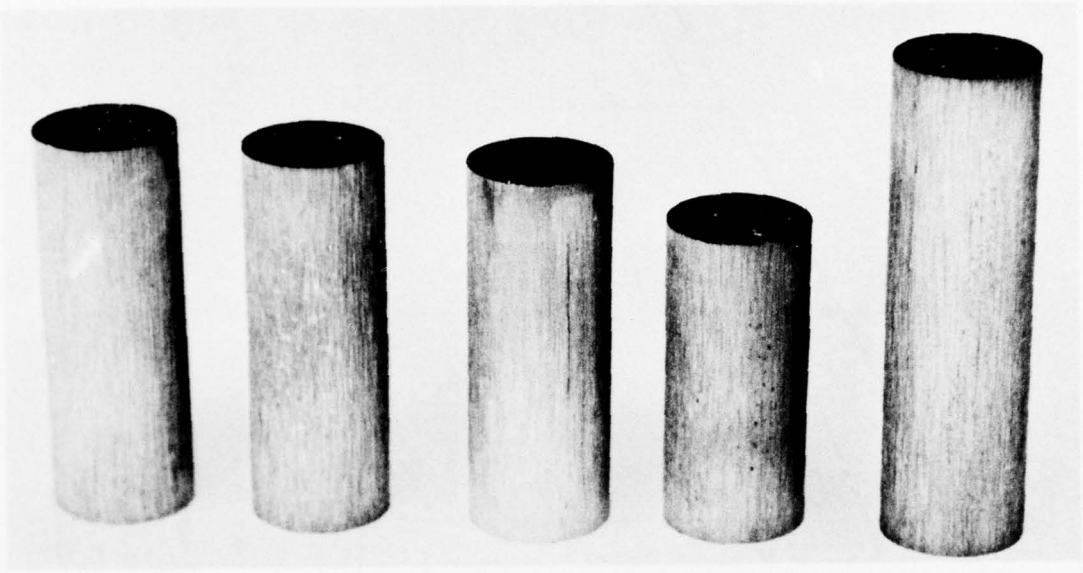
1 - Filage du TA 6 V en poudre

2 - Filage du TA 6 V massif

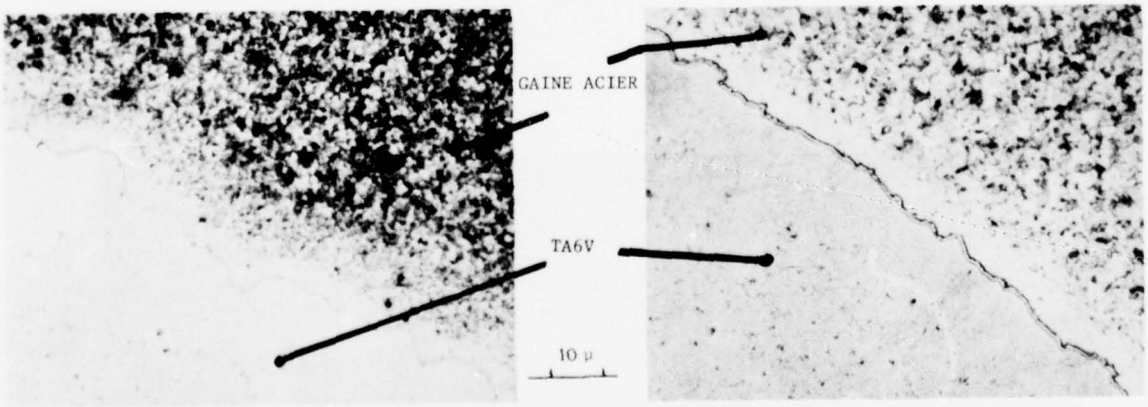
3 - Filage du TA 6 Z 5 D en poudre

FIGURE 3

ETAT DES SURFACES DU TA6V FILE A DIFFERENTES TEMPERATURES AVEC UN RAPPORT DE REDUCTION DE 14



850°C 900°C 950°C 1000°C 1050°C

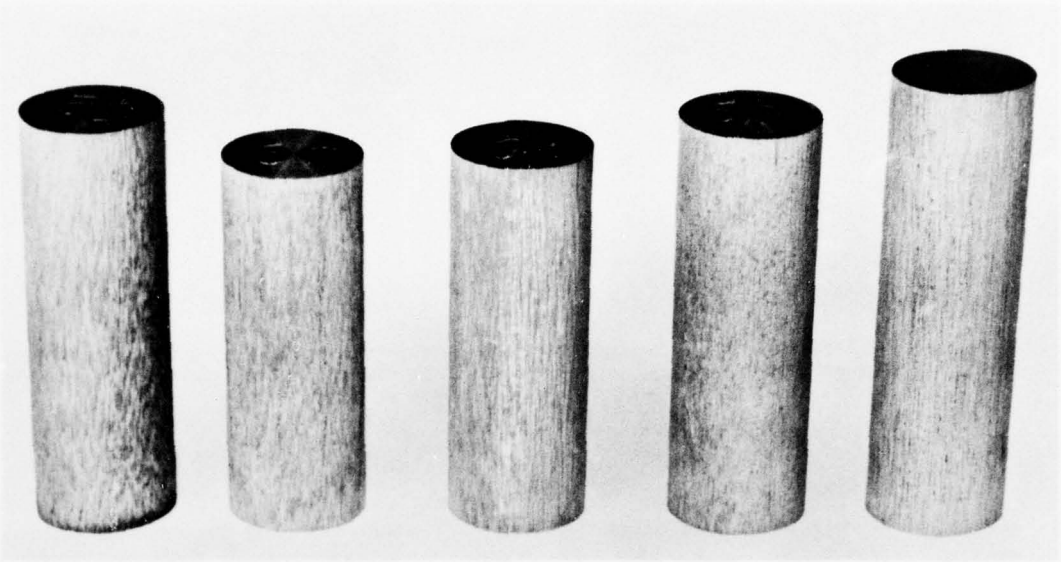


Interface gaine - TA6V après filage à 850°C

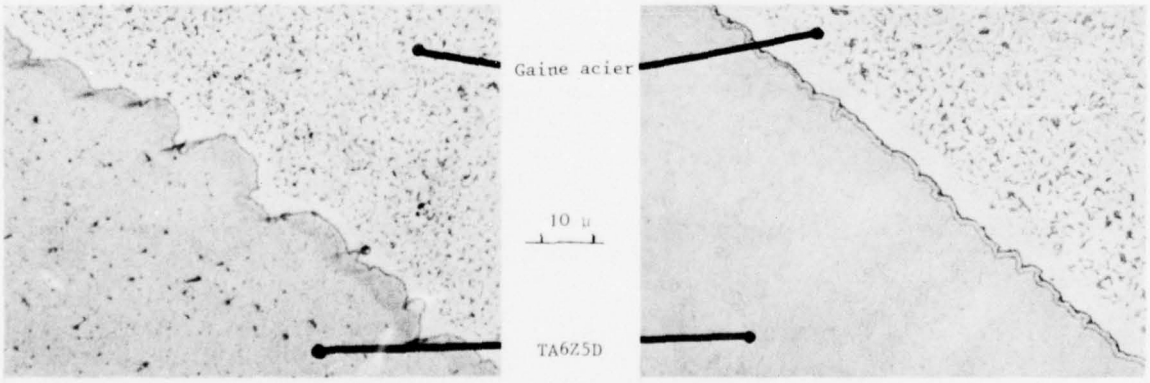
Interface gaine - TA6V après filage à 1050°C

FIGURE 4

ETAT DES SURFACES DU TA6Z5D FILE A DIFFERENTES TEMPERATURES AVEC UN RAPPORT DE REDUCTION DE 14



850°C 900°C 950°C 1000°C 1050°C



Interface gaine - TA6Z5D après filage à 850°C

Interface gaine - TA6Z5D après filage à 1050°C

FIGURE 5

MICROGRAPHIES D'ALLIAGES DE TITANE FILES A DIFFERENTES TEMPERATURES
AVEC UN RAPPORT DE REDUCTION DE 14

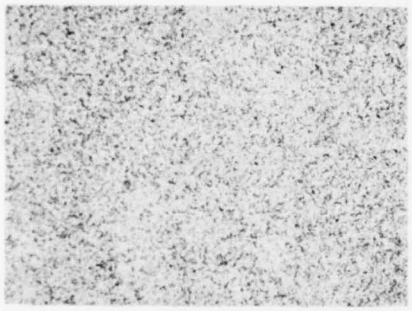
TA6V

TA6Z5D

850°C

20µ

Le filage est effectué dans le domaine α.β . La structure reste inchangée après refroidissement : matrice α avec précipités de phase β (points noirs). La vitesse de recristallisation est très lente



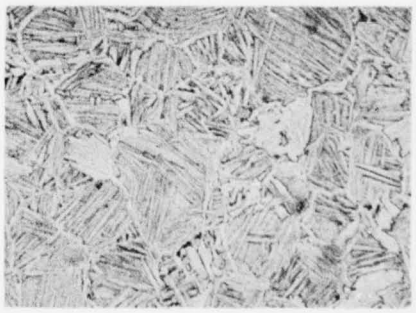
900°C

20µ

Le filage est effectué dans la partie haute du domaine α.β . Une partie de la phase β présente au cours du filage se transforme en phase α au cours du refroidissement. On distingue une phase α primaire (claire et bien dessinée dans le TA6Z5D) et une phase α dite β transformée, aciculaire.

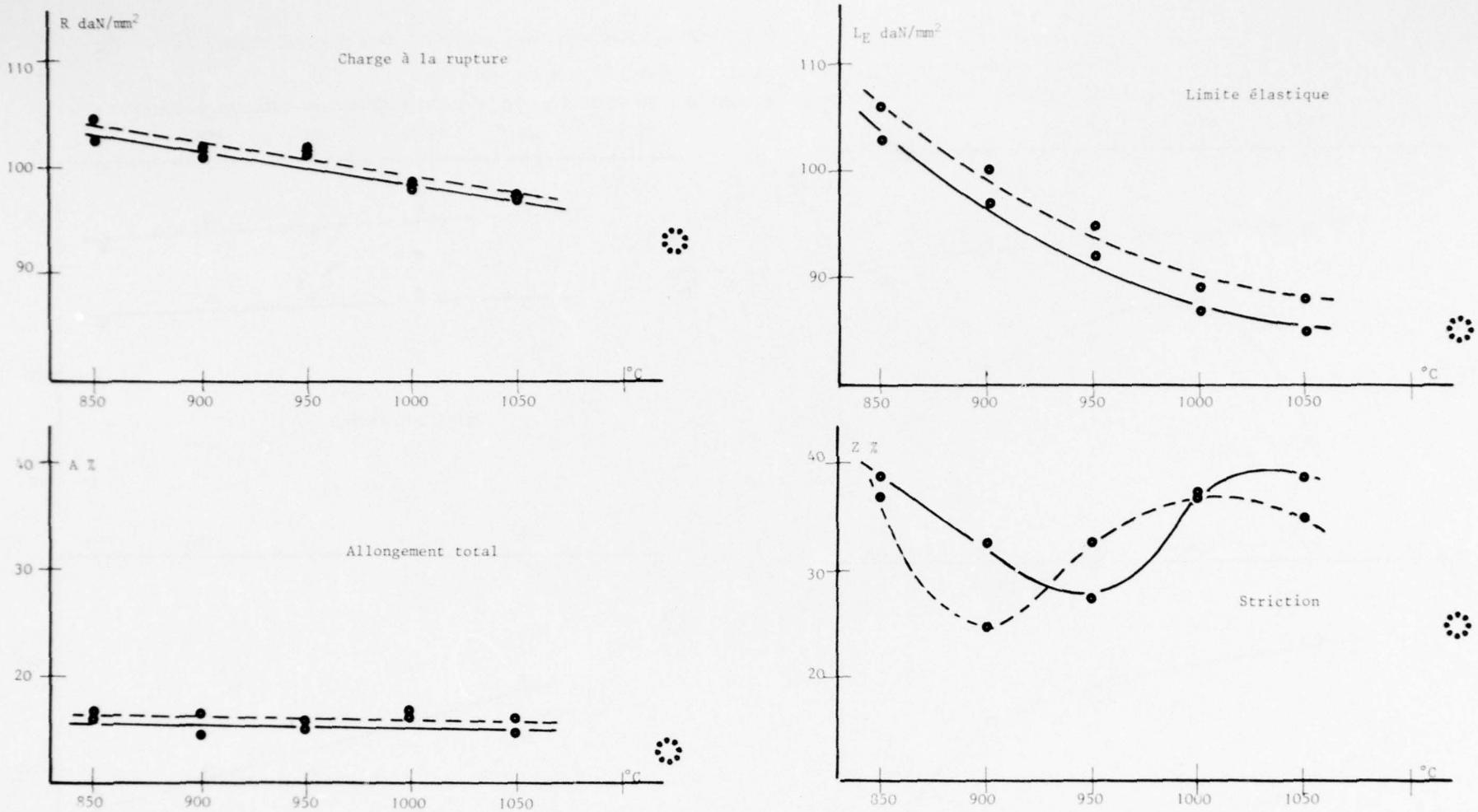
1000°C

20µ



Le filage est effectué dans le domaine β. Les éléments α-gènes (Aℓ et O_2) sont rejetés aux joints de grains β. Au cours du refroidissement lent, la phase α précipite d'abord aux ex-joints de grains β (liseré blanc), puis ensuite en plaquettes plus ou moins épaisses à l'intérieur des ex-grains β, soulignées sur la micrographie par des liserés noirs riches en éléments β-gènes (V pour le TA6V et Zr-Mo pour le TA6Z5D. C'est une structure β transformée. La recristallisation est très rapide dans le domaine β.

FIGURE 6

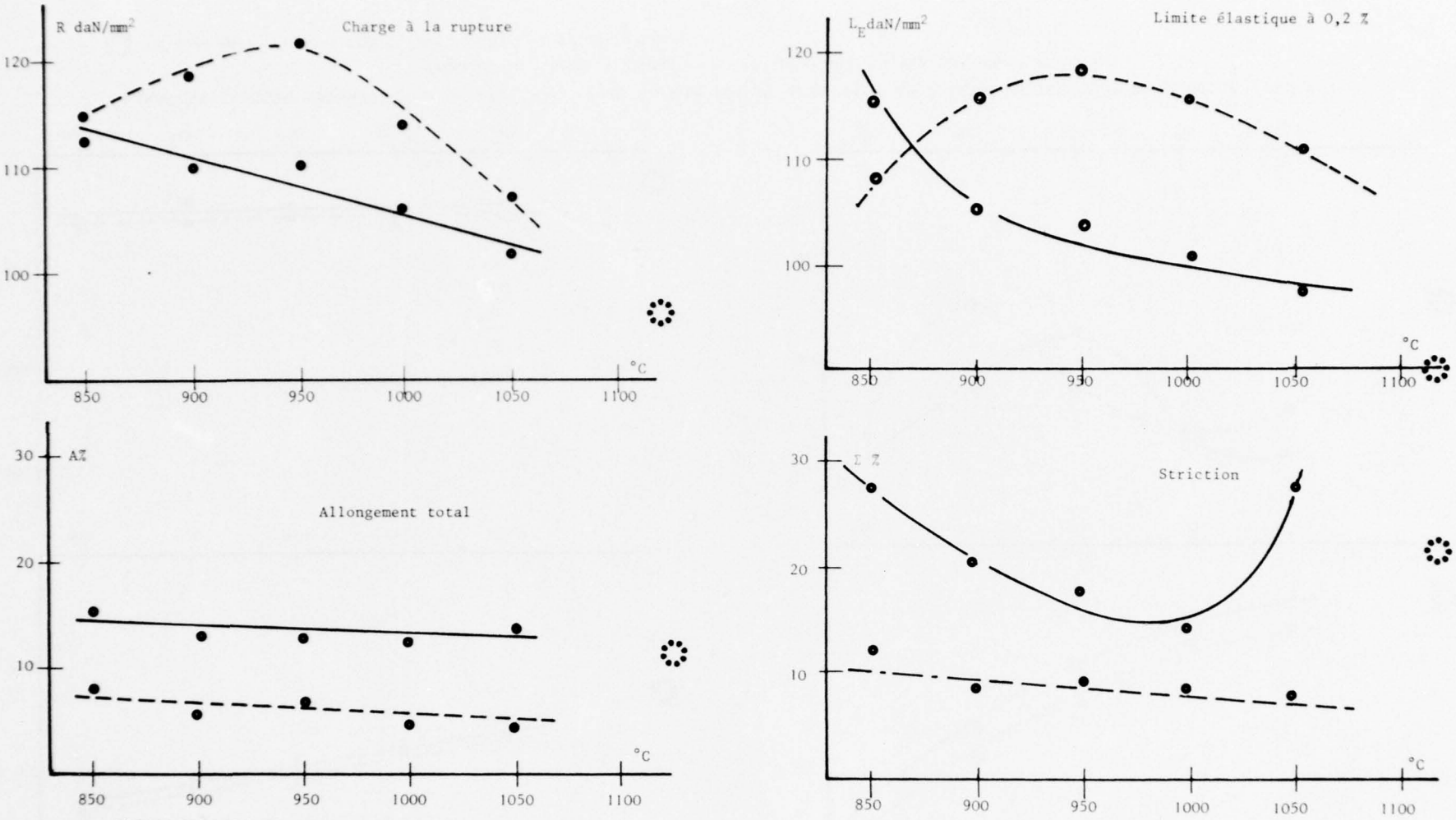


CARACTERISTIQUES MECANIQUES DU TA6V FILE AVEC UN RAPPORT DE REDUCTION 14 A DIFFERENTES TEMPERATURES DE FILAGE

Trait plein : brut de filage - Trait pointillé : après traitement thermique

Alliage TA6V fritté à 1140°C sous 500 bars, brut de frittage.

FIGURE 7



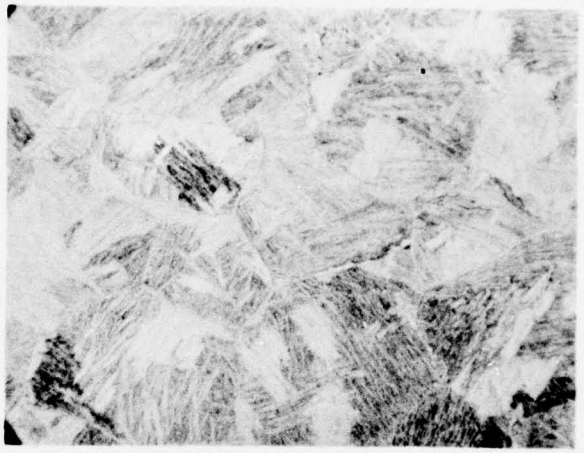
CARACTERISTIQUES MECANIQUES DU TA6Z5D FILE AVEC UN RAPPORT DE REDUCTION DE 14 A DIFFERENTES TEMPERATURES DE FILAGE

Trait plein : brut de filage - Trait pointillé : après traitement thermique

Alliage TA6Z5D fritté à 1140°C sous 500 bars, brut de frittage.

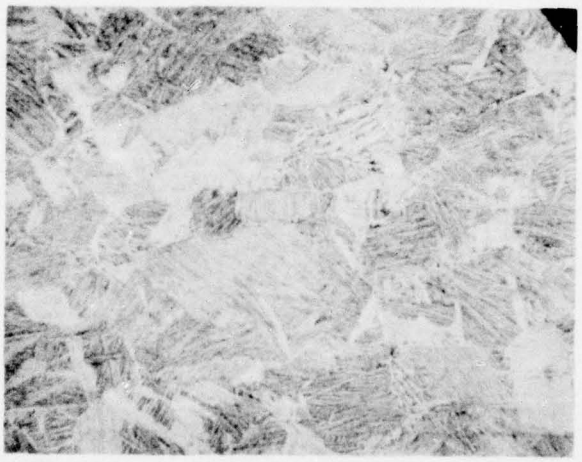
FIGURE 8

MICROGRAPHIES D'ALLIAGES DE TITANE FRITTES SOUS CHARGE A 1140°C



140μ

TA6V fritté à 1140°C sous 500 bars



140μ

TA6Z5D fritté à 1140°C sous 500 bars

La structure est très nettement β transformée. Les grains et les plaquettes α sont de dimensions plus grandes que dans les structures filées. Ce grossissement du grain est dû au refroidissement plus lent des pièces frittées sous vide.

FIGURE 9

PRODUCTION OF TITANIUM POWDER BY THE ROTATING ELECTRODE PROCESS

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Titanium is an important metal in aircraft gas turbine engines and for aerospace structural applications. It is also among the most costly engineering metals employed in modern aircraft. The high cost of titanium billet is subsequently multiplied manyfold - as much as 10 and 15 times - by "material utilization ratios", which relate the weight of billet to the weight of a finish-machined part. One of the most significant contributions expected from titanium powder metallurgy (P/M) technology is the ability of P/M processing to reduce the quantity of excess metal required for titanium parts so that the ratio of input to final part weight will be less than 2:1. For critical applications, such process gains must be realized without any compromise in properties, and therefore the traditional P/M technique of press-and-sinter is inadequate, since the residual porosity inherent with such processing is responsible for greatly reduced levels of ductility, toughness and fatigue strength. In fact, complete densification obtained through one or more hot consolidation operations is considered to be a minimum requirement for critical titanium P/M parts. The powders best suited to hot consolidation are those combining high bulk density and low surface area with high purity. It is therefore not at all surprising that much of the work performed on titanium P/M for aircraft applications is based on the titanium alloy powders made by the Rotating Electrode Process.

In the Rotating Electrode Process, one end of a rapidly rotating bar (the consumable electrode) is melted by an electric arc issuing from a non-rotating, non-consumable electrode. The melting takes place within a large helium-filled chamber. As the consumable electrode rotates, centrifugal force causes the molten metal at its end to fly off in the form of fine droplets which coalesce into spherical "microcastings" and fall to the chamber floor.

There are two variants of the Rotating Electrode Process, represented by the short-bar and the long-bar REP machines. The short-bar machine, Figure 1, accepts consumable electrodes (anodes) up to 3-1/2 inch (9mm) diameter by 10-inch (250mm) long. The entire consumable electrode and the collet or chuck in which it is held are contained within the machine chamber. After most of the anode has been converted to powder, the operator utilizes a glove port to remove the 1- to 2-inch (25-50mm) anode "stub" and replace it with a new anode. Standard 2-1/2 inch (6.35mm) diameter electrodes are introduced into the chamber through an O-ring-sealed bar feeder. Anodes of diameters that cannot be accommodated by the bar feeder are stored in an electrode box within the chamber prior to being introduced into the machine collet.

The short-bar machine is very useful for short runs and for experimental (e.g., cast) alloys which may be available only in short lengths of various diameters. The productivity of this machine, however, is not as high as that of the long-bar machines which can produce much more powder per hour.

The REP long-bar machines consume 2-1/2 inch diameter anodes that are up to 60 inches (1500mm) long. The anodes are held in a collet outside of and behind the machine, Figure 2. The rotating electrode enters the chamber through a gas-tight dynamic seal, and is advanced automatically into the arc. When most of the anode is consumed, the collet is opened and retracted and a fresh bar is loaded and advanced behind the first. These machines are thus far more efficient than the short-bar device, since very little time is lost in the loading of fresh electrodes, and they suffer only small stub losses. A typical long-bar machine can produce titanium powder at the rate of approximately 4000 pounds (1900 kg) per week.

As described above, the rotating consumable electrode in this powder-making process is the anode. The cathode for most REP powders is regarded as non-consumable and is made of thoriated tungsten because of its unique electrical and physical properties; e.g., low resistivity, high melting point. Since arc stability increases and the heat balance in the arc shifts to the anode in proportion to the cathode melting point, tungsten is the preferred material for non-consumable arc melting electrodes. In the operation of the arc, however, not even tungsten is absolutely non-consumable, for there must be a small pool of molten metal at the cathode tip to provide a source of ions, and some of this liquid gets carried off by the arc plasma flame. In addition, occasional powder particles fall upon and adhere to the tungsten cathode just back of the arc region. As more and more titanium fuses to the tungsten, the arc acquires the characteristics of the coating, rather than the tungsten substrate, and its stability degrades, adding additional droplets (particles) of tungsten to the powder.

Although the size and frequency of these tungsten particles is not great and their influence on the mechanical properties of the tougher alloys, such as Ti-6Al-4V and Ti-6Al-6V-2Sn, is minimal we recognize that the tungsten particles would not be tolerable in the more crack-sensitive alloys and that it would be best if no REP titanium powders contained any tungsten inclusions. It was therefore proposed to the U. S. Air Force that Nuclear Metals perform a program in which techniques would be developed by which REP

titanium powder could be made without the use of a tungsten cathode. A contract was subsequently awarded to Nuclear Metals whereby this objective would be pursued, and the REP modification program has begun.

We concluded, and the Air Force concurred, that the most acceptable cathode material for a titanium-producing REP machine must be titanium, of the same nominal composition as the anode. Since such a cathode would be less "ideal" than one made of tungsten, its natural tendency would be to melt or erode. To capitalize on this phenomenon, one part of our investigation is directed towards a double-REP concept, in which both the anode and the cathode are rotating consumable electrodes, both being held in external collets which advance the electrodes towards each other in a "double long-bar" mode. We are also examining the use of a massive non-consumable titanium cathode which rotates (at a relatively slow speed) for the purpose of constantly changing the arc-heated surface. For the immediate future, it appears most practical to optimize this non-consumable cathode approach, since it presents fewer engineering problems than the double long-bar. In the long run, however, it will obviously be most efficient to make powder by the double long-bar technique, in which the maximum amount of arc energy is utilized for electrode conversion to powder, rather than losing some of it as heat to be transferred to the non-consumable electrode's cooling system.

It is expected that the titanium powder made by the modified Rotating Electrode Process will possess the same desirable physical characteristics as that made up to the present.

As described above, the titanium electrodes are melted in the absence of any crucible in an atmosphere of high purity inert gas, and so the powder composition is virtually identical to that of the electrode from which it is made. Particle size is a function of metal density, electrode diameter, and electrode speed. Typical attributes of an REP titanium alloy powder are shown in Table I; scanning electron micrographs, Figure 3, attest to the sphericity and uniformity of the powder.

In conclusion, the titanium alloy powder made by the Rotating Electrode Process consists of closely-sized, high purity spherical particles within the range of 50-500μm. Although earlier REP powders had been produced by a technique employing a tungsten cathode, this source of contamination has been eliminated as a result of process modifications which make use of titanium cathodes, in either a consumable or non-consumable mode.

TABLE I

CHARACTERISTIC ATTRIBUTES OF REP Ti-6Al-4V POWDER

1. COMPOSITION

Aluminum	Vanadium	Oxygen Standard	Oxygen ELI	Iron
5.50-6.75	3.50-4.50	0.12-0.20	0.05-0.12	0.3, Max.
Carbon	**Nitrogen**	**Hydrogen**	**Other, Total**	**Ti**
0.10, Max.	0.05, Max.	0.0125, Max.	0.4, Max.	Balance

2. SIZE

a) Size Range: 50-500μm

b) Size Distribution:

Screen Size, μm:	500	354	250	177	125	88	63	44	<44
Fraction Retained, %:	0	8.3	23.6	38.8	19.8	6.0	2.5	1.0	1.0

c) Average Particle Size: 225μm

3. DENSITY

a) Bulk: 2.69 g/cm^3 (60% of theoretical)

b) Tap : 2.90 g/cm^3 (64% of theoretical)

4. FLOW RATE: 24 sec/50g

5. SURFACE AREA: 0.009 m^2/g

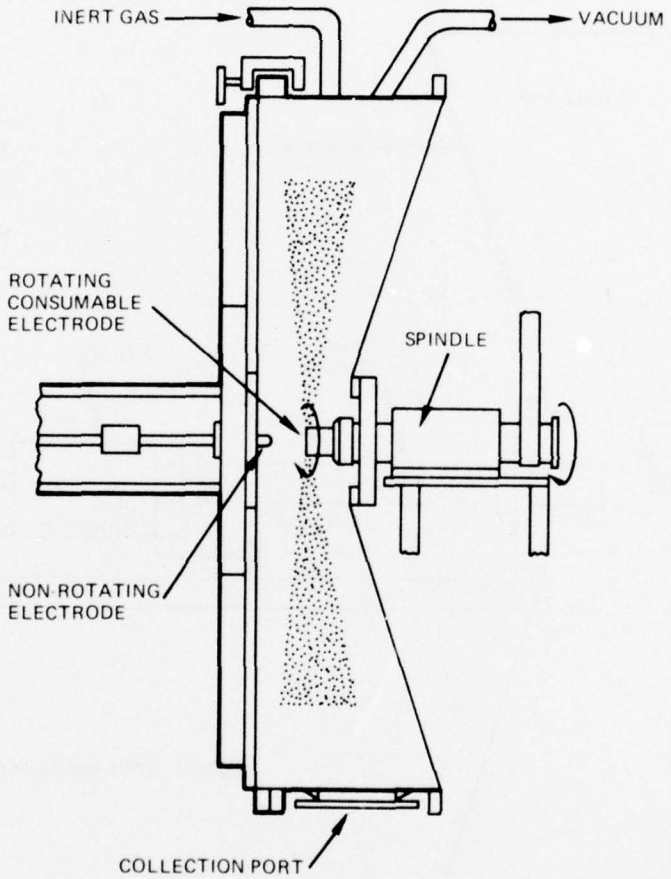


Figure 1. REP Short-Bar Apparatus

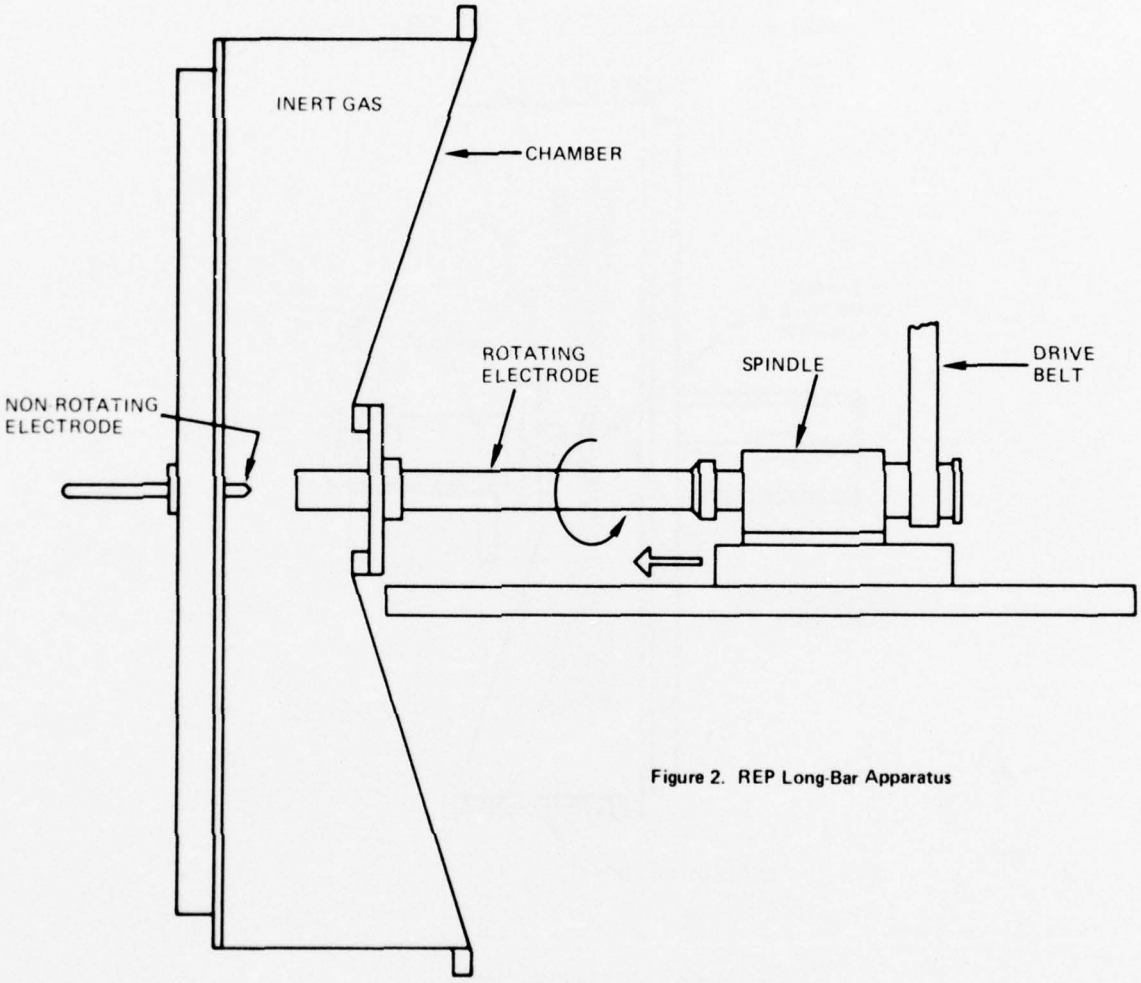
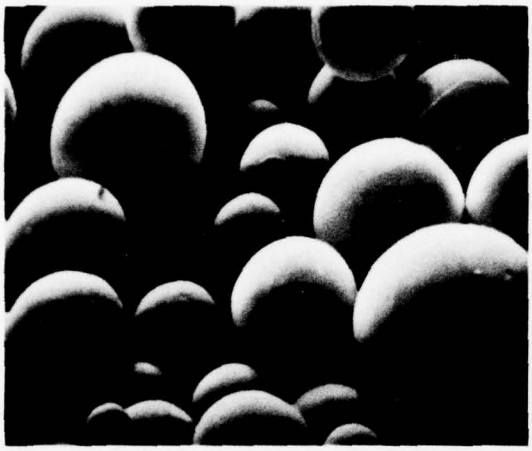


Figure 2. REP Long-Bar Apparatus

100X



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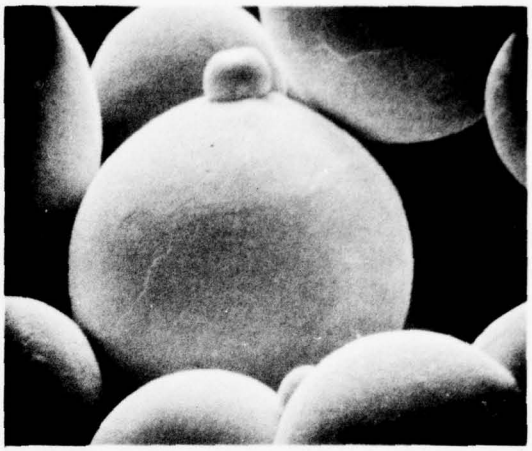

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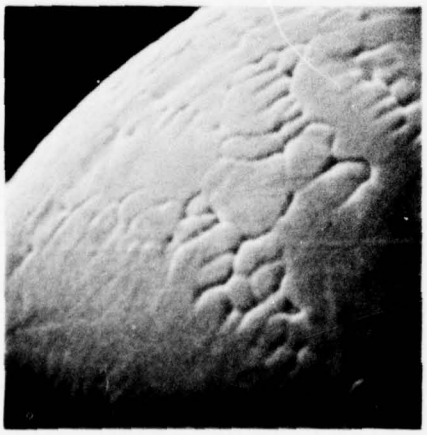

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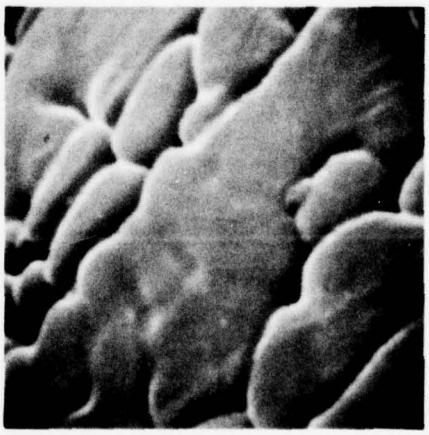

Figure 3. REP Ti-6Al-4V, SEM

PRODUCTION OF HIGH PURITY METAL POWDER BY ELECTRON BEAM TECHNIQUE

by
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SUMMARY

Scope of the new process is manufacturing of metal powders, especially of titanium and nickel-super-alloys of highest purity in the most economical way. The powder can be of 100 % spherical shape and of 50 - 600 micron diameter and of flake size of 20 - 400 mesh or a mixture of both.

High purity is achieved now by processing in high vacuum, melting with the programmed electron beam and atomizing with a water-cooled rotating disk. This allows reduction of hydrogen from Ti-alloys and reduction C, O_2, H_2 and N_2 from Ni-super-alloys and avoids contamination of the metal powder from the environment, the heat source and the atomizing system.

Highest economy will be achieved by: largest quantities per load, cheap starting material by direct application of cast or melted ingots, high atomizing speed of 300 - 250 kg/h continuous production over several days, low energy costs (0.6 - 0.8 kWh/kg), low labour costs, high efficiency of nearly 95 % and constant quality by full automatization of the process.

Filling the powder in cans and vacuum-tight sealing of the same by e.b. welding will maintain the high purity of the powder during transportation and storage. The powder qualities and processing data show that we can follow our program achieving our realistic, promising goal.

1. SCOPE

When we were confronted with the economical production of metal powder of highest purity we determined that our main interest should be laid on reactive metals and such ones where a low oxygen content is essential to meet the high demands of PM parts.

Before carefully studying the processes and furnaces available and being under development we wrote down the scope of such a process. Bearing in mind that the highly developed aircraft industry was going to be the most critical user of the powder to be produced we have listed up the demands for quality, which means in detail:-

a) No contamination of the metal to be melted and the powder to be produced by the heat source, the atomizing system and the environment.

b) The gas content of the powder should be, if necessary - e.g. Ni-super-alloy - as low as possible, which means that the melting process should be a refining process.

c) The particle size should be between 50 and 600 microns, for special application particle sizes of 5 - 50 should also be producible.

d) The process should allow to produce either spherical particles of a 100 % density, smooth surface and high tap density or flakes of 20 to 400 mesh with a high green strength and of very small grain size.

e) The losses of volatile alloy elements should be neglectable.

f) The process should be automatable to achieve a constant quality of the product.

Since a product of highest quality will only find a very small field of application if the process cannot be significantly reduced, the second feature of our aims was the economical production of high quality powder. In order to bring the powder price very close to the ingot price high economy can be achieved, if:-

a) Large and consequently cheap loads can be melted and atomized etc. by direct usage of small and medium VAR and VIM ingots.

b) A high productivity can be achieved by having a high melting and atomizing speed, a continuous production over several days and weeks and a high material efficiency.

c) The costs for material preparation, powder packing and transportation and quality control can be kept down.

Trying to meet all these points of our scope the result must be a sophisticated expensive furnace. In order to keep the capital costs at a reasonable level it is necessary to extent the application of the furnace to all metals and alloys, which means that all metals with a melting point higher than 1400° C should be powderizable in an economical way.

2. PROBLEMS IN THE PRODUCTION OF METAL POWDER

In order to meet all the above-mentioned aims of the specification the following main groups of powder production should be carefully considered:-

Melting - Atomizing - Solidification and Cooling and at last the most important one - Collection of the Powder.

In melting we are convinced that the usage of the electron beam as source of heat, using the end of an electrode and vacuum would be the best combination to avoid contamination of the material to be melted and atomized. For atomization we have decided for a rotating disc, because only this system allows the best control of the particle size and its size distribution. Solidification and cooling down should be made slowly to obtain gas free shrinking holes even in large particles. Only energy transmission by radiation should be allowed.

Collection of particles is the most important and most difficult problem in high quality metal powder production. A collection of 100 % without touching the powder by any tool must be a necessity, in order to

a) maintain the cleanliness of the metal powder. Any handling would bear the danger of spoiling the powder;

b) keep the tank clean and easily inspectable so that, if changing the metal or metal alloy, crosswise contamination can be avoided.

Melting, atomizing and cooling down in vacuum is especially very important in the production of Ti-powder. Evaporated metal can condensate in a massive layer on the melting tank wall. Doing the same step in a reduced nobel gas pressure the evaporating metal condensates in a loose sponge-like layer, which can be eroded by the powder particles and spoil the particle surface. Arousing of explosions when opening the tank must be considered and avoided by well-proven steps of tank venting.

3. PROCESS

Figure 1 shows that the EBRD (Electron Beam Rotating Disc) Process is a combination between a vertical drip-melting process and an atomizing process with a rotating disc.

The vertical fed, slowly rotating electrode will be drip-melted as in all drip-melting furnaces. The well programmed e.b. takes care for a pencil-shape of the electrode tip, that all material will run down the conical electrode tip and that the material will drop into the centre of the rotary disc.

The programmed beam provides that the outspreading metal film at the disc surface will be atomized at the corner of the disc in small particles. The size of these particles is given by the surface tension of the material to be atomized and the centrifugal force.

The particles start to solidify on their way to the chamber wall.

Depending on the process parameter it is possible to produce

100 % spherical powder or Figure 2
100 % flakes or a convenient
mixture of both shapes.

Also depending on the beam programme the shape of the flakes can be changed between microspear-heads and micro potato pancakes.

In the first stage of process development the electrodes to be atomized are of 4 - 6" diameter and 16 - 32" length. The atomization speed should be in the range of 2 - 4 lbs/min and the process is of the batch type.

In the second stage the electrodes could be of 8, probably 10" diameter and 200" length. The atomization speed will be 5 - 10 lbs/min. The process will be continuous.

In both steps the powder will be collected in vacuum-tight stainless steel cans. The powder can be stored under vacuum. If the storing time will last long the cans can be e.b. welded.

As starting material several types and qualities can be used. For first-grade powder double melted VAR-Ti-alloy ingots with sand blasted surface or single melted VIM-Ni-base super alloys can be used. For second grade powder large solid scrap pieces, scrap mosaic electrodes or e.b. consolidated small scrap or virgin material can be used.

The application of cheap but high value scrap material is one of the main points to reduce the powder costs and one of the main advantages of the EBRD process.

4. PRELIMINARY RESULTS FROM A SMALL LABORATORY FURNACE

In a small laboratory furnace - Figure 3 - we have studied the function of atomization with e.b. We have seen that our ideas could be realized and powder could be produced. In the first step we have atomized just by bombarding the metal disc.

The chemical analyses of Ti- and Ni-alloys are given in Figure 4. For Ti the atomization speed has been around 1 lb/min and for Ni-super-alloys around 2 lbs/min with an e.b. power of only 15 kW.

The spherical particles have led to a 100 % density up to a particle diameter of 0.7 mm. The grain size of the Ti-sphericals is about 20 microns for 0.380 mm balls and about 5 - 10 microns for Ti micro potato cake flakes.

The top density of shperical Ti-powder is approx. 60 %, of flakes approx. 28 %.

In the second step we have drip-melted on to the rotating disc and seen that this process works also to our satisfaction if the e.b. is programmed according to the sophistication of the process.

So far so good. In the course of development we have been confronted with housekeeping problems. We have learnt how many impurities are contained in a visible clean high vacuum furnace and in the environment of the furnace.

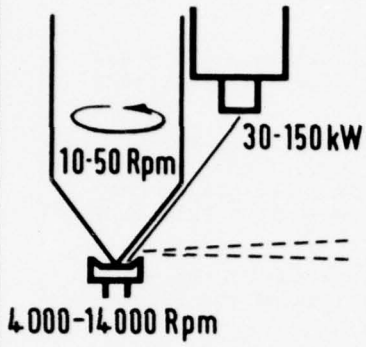
The consequence of that long development period has led to the decision to build a production furnace which is now ready for the first test runs. This furnace - Figure 5 - is able to atomize all metals with a melting point higher than 1400° C. It allows the atomization of electrodes of 6" diameter and 32" length and can be extended to atomize electrodes of 10" diameter and 200" length.

5. FIRST RESULTS FROM THE PRODUCTION FURNACE

The results will be shown during the AGARD-Meeting in Ottawa.

6. CONCLUSION AND ASPECTS

It has been shown that the electron beam rotating disc process offers the possibility to produce economically very pure Ti- and Ni-base super-alloys in large quantities. The existing furnace is applicable to a growing powder market. If the market demands for large quantities of second or third grade powder the process can be changed so that raw material or scrap can be atomized in a direct way and e. g. Ti-powder of high purity can be delivered at a lower price than double melted ingots and forged bars.



	Steps of development 1	2
Loaded mat.	Consumable	electrodes
Max. Ti-charge	60	850 kg
Max. Ni-charge	120	1 600 kg
Max. atom. speed	60	150 kg/h
Production	Batchtype	Continuous

Fig. 1 EBRD-Process and its Main Data

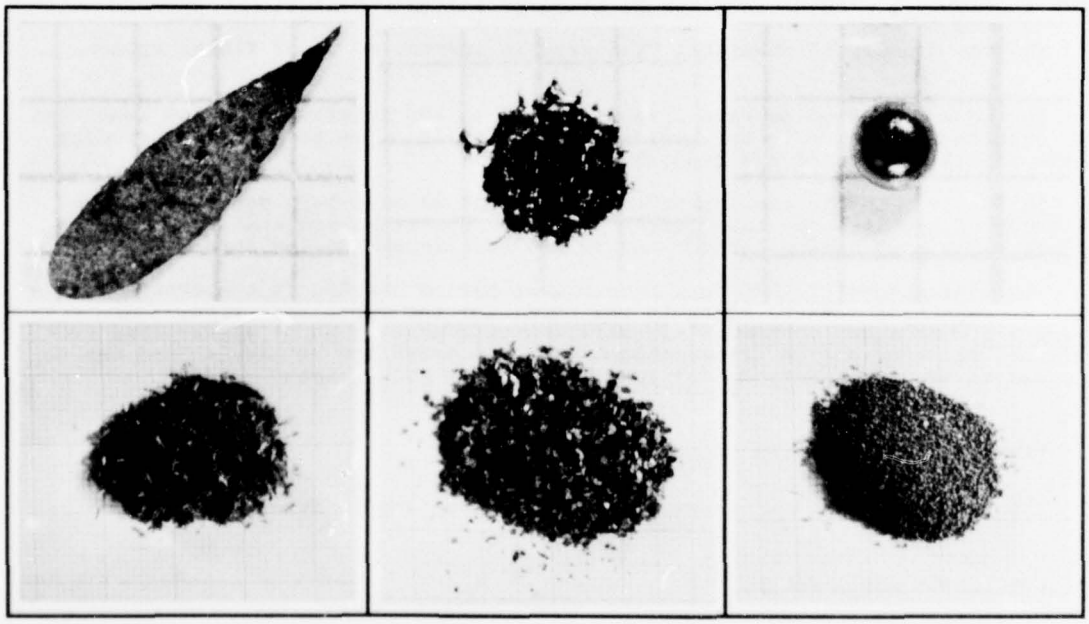


Figure 2: Shapes of metal powder particles
Microspear-head flakes
Micro potato pancake flakes
Spherical

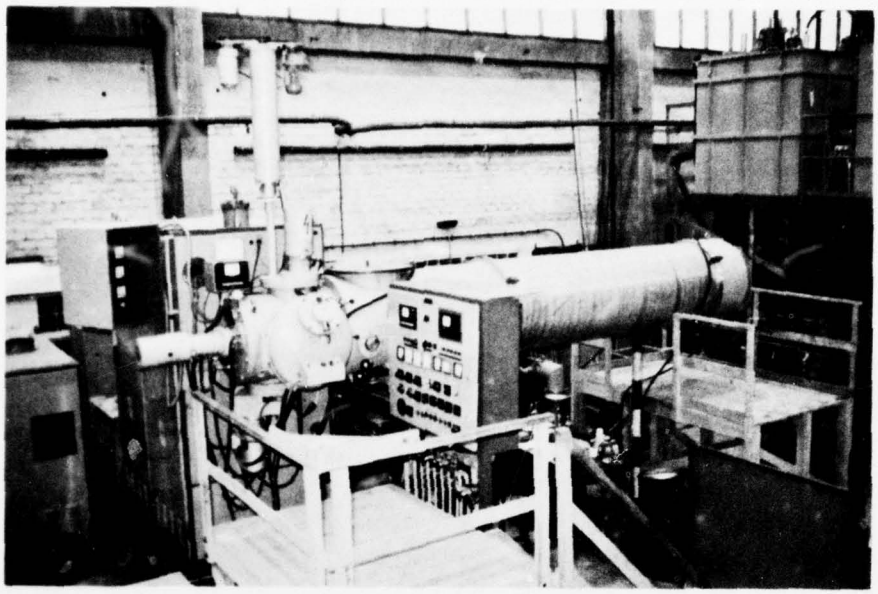


Figure 3: Small laboratory EBRD atomization plant

Ti - alloys

	Al	V	Fe	O_2	H_2
Starting material	6,6	4,2	0,11	0,20	0,007
Spherical powder	6,4	4,1	0,11	0,21	0,005

	Al	Zr	Mo	Si	Fe	O_2
Starting material	6,2	5,0	0,5	0,24	0,023	0,14
Spherical powder	6,0	5,1	0,5	0,24	0,021	0,13

	Al	V	Fe	O_2	H_2
Starting material	6,0	4,1	0,11	0,11	0,007
Splashed powder	5,9	4,1	0,12	0,12	0,005

Ni - super-alloys

	Al	Cr	O_2	N_2	H_2
Starting material	5,0	9,0	60	8	6
Spherical powder	4,95	8,95	64	10	9

Figure 4: Chemical Analysis Data of Ti- and Ni-alloys

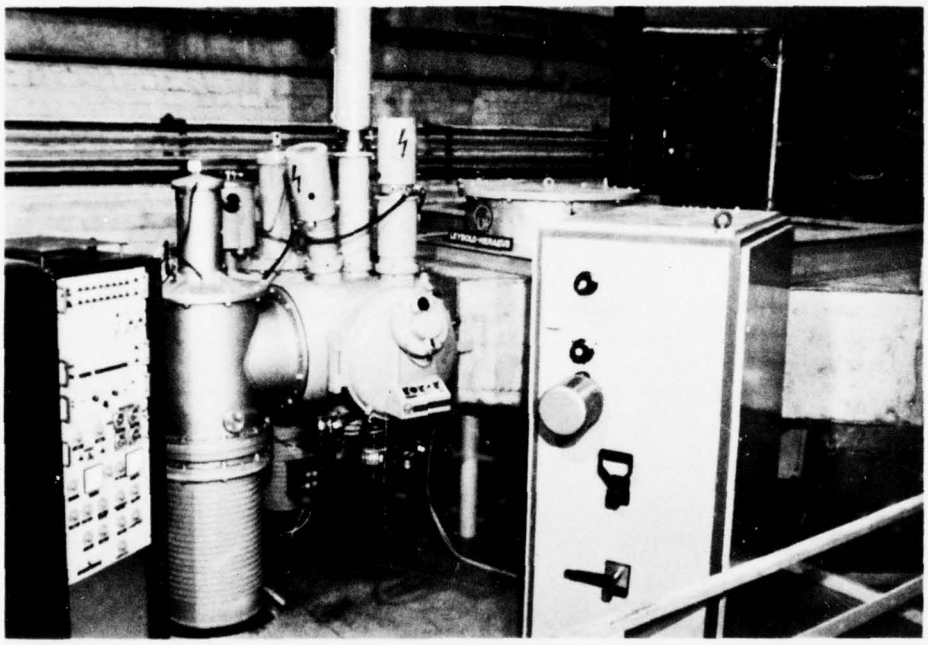


Figure 5: EBRD atomization plant for production of metal powder

TITANIUM POWDER PRODUCTION BY THE HARWELL CENTRIFUGAL SHOT CASTING PROCESS

by
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SUMMARY

The Harwell Centrifugal Shot Casting (CSC) process, which has particular relevance to the production of titanium alloy powders is briefly described. The process is one of several centrifugal atomisation techniques being developed throughout the world, in all of which the mechanism of disintegration of molten titanium into discrete droplets has many similarities. Part of the presentation to the Specialists Meeting is in the form of a cinefilm of the melting of a titanium alloy electrode and the subsequent atomisation as it takes place in the CSC process. In addition, calculations of the time taken for pure titanium droplets of 50-500μm diameter to solidify in flight are presented, as well as the distances traversed during solidification. The dependence of these parameters upon the nature and pressure of the inert gas environment is considered.

1. INTRODUCTION

The highly reactive nature of titanium and its alloys towards interstitial elements, such as O, N, H and C, at elevated temperatures severely limits possible melting routes for these materials. In general the source material is required to be in the form of an electrode, and metal crucibles into which it is cast under high purity environments must be efficiently water- or liquid metal-cooled. The Centrifugal Shot Casting process, which operates in the manner of a consumable arc melting furnace, with a rotating crucible in place of a stationary one, would therefore appear ideally suited as a process for the conversion of titanium electrodes into relatively coarse, high purity powders for further fabrication by hot consolidation methods. Indeed, centrifugal atomisation in one form or another is currently the predominant powder manufacturing process for P/M fabrication in high-intregrity, highly stressed applications.

The observation of the mechanism in the CSC process for the disintegration of molten titanium into droplets, which subsequently solidify to form the discrete powder particles, has relevance to the several centrifugal atomisation processes presently being developed.

An important factor in the design and capital cost of a production unit for the manufacture of titanium alloy powders by such a process is the size of atomisation chamber required for the solidification in flight of the powder particles; this requirement ensures that the particles preserve their purity and spheroidal shape (and hence good flow and packing characteristics). Calculations of the trajectory and cooling of such particles and their dependence upon operational variables is an obvious first step towards defining the required chamber dimensions.

2. THE CENTRIFUGAL SHOT CASTING PROCESS

The process(1) involves a stationary electrode of the material to be converted into powder and a rotating water-cooled crucible as shown in Fig.1. Heating is accomplished by an electric arc struck between the electrode and the crucible, which causes the end of the electrode to melt and fall as molten drops into the crucible. Under the action of centrifugal force the melt moves up the side wall to the lip of the crucible where it breaks up and is ejected as droplets. The whole process of melting, atomisation and solidification takes place within a leak tight enclosure under a high purity inert atmosphere, typically at a pressure in the range 0.3-1.0 atm.

Under conditions of adequate collection chamber geometry, the product is essentially spheroidal, of high packing density and with a particle size distribution approximately logarithmic Gaussian; the mean diameter can be controlled in the range 150-1000μm and the geometric standard deviation is typically 0.3-0.4. Purity can be maintained to that of the starting electrode.

3. CINE-PHOTOGRAPHY OF THE CSC PROCESS

The cine-film, to be shown as part of this presentation, examines two aspects of the CSC process:

(1) The melting and dropwise consumption of a titanium alloy electrode into the rotating crucible.

(2) The disintegration of such drops of molten metal by the action of centrifugal forces in the region of the lip of the crucible.

All the films, from which sequences have been selected, were taken during the melting of ~60mm diameter Ti-6Al-4V alloy electrodes. The arc acted as both the power source for melting and as the sole source of illumination. The electrode consumption was filmed in a horizontal direction using a high speed camera at 500 frames/sec, the electrode being withdrawn out of the crucible as far as possible to enable its tip to be visible. The disintegration into droplets at the lip of the crucible was filmed in a near-vertical direction at 5000 frames/sec whilst at the same time the rotation speed of the crucible was reduced from the usual 3000-4000rpm to 1000-1500rpm. These conditions were necessary to give sufficient slowing down of events taking place at the periphery of the crucible.

4. PARTICLE TRAJECTORY AND COOLING CALCULATIONS

Calculations of the trajectory of an initially molten droplet from the point of detachment at the lip of a rotating crucible, in a direction tangential to that crucible, have previously been reported for the case of pure iron moving through inert atmospheres at atmospheric pressure(1). The solution of two-dimensional equations of motion of a particle moving under the action of frictional and gravitational forces is required. Since for the majority of situations met in the CSC process, the Reynolds number lies in an intermediate region between streamline and turbulent flow ($0.5<Re<10^3$) where the drag coefficient has a complex dependence upon particle velocity, a method of incremental approximation has been followed, based on the earlier work of Lapple and Shepherd(2).

The cooling of a molten droplet can be calculated by consideration of the heat transfer from the droplet by radiation and forced convection, as it traverses its trajectory. The forced convection contribution uses an empirical correlation(3) for the dependence of Nusselt number on Re in the case of fluid flow past a sphere. Using a similar incremental approximation method to that employed for the trajectory calculations, heat losses during small time increments can be converted into incremental temperature changes; this presumes a knowledge of the change of enthalpy with temperature for the material of the sphere. Incorporation of the latent heat of solidification in the enthalpy/temperature variation allows the cooling of the sphere to be followed from an initial temperature above the melting point, through solidification to any temperature below the solidus.

Some arbitrary assumptions have to be made; for instance the initial temperature of the droplet, the emissivity of the molten and solidifying sphere, the temperature uniformity within the sphere, the temperature to which the sphere must cool to avoid gross deformation on impact with the walls or base of the atomisation chamber and the temperature and state of motion of the inert gas within the chamber.

Values assumed for these and other parameters in extending the calculations to the case of pure titanium are shown in Table 1. For the purpose of optimising the chamber

TABLE 1

PARAMETERS ASSUMED FOR MODEL OF TITANIUM SPHEROIDISATION

Crucible diameter	75mm
Crucible rotation speed	4000 r.p.m.
Sphere diameter	50-500μm
Initial sphere temperature	1990K (1717°C)
Titanium melting point, T_m	1940K (1667°C)
Sphere temperature for no deformation on impact	1890K (1617°C)
Inert gas temperature	323K (50°C)
Chamber wall temperature	293K (20°C)
Sphere emissivity	0.5
Inert gas pressure	7.6-760mm Hg (0.01-1atm.)

size so that droplets of the required size range can solidify prior to impact with the chamber walls, the time taken (and hence the horizontal and vertical distances travelled in that time) to cool to a temperature just below the freezing point is of major interest. Fig.2 illustrates the dependence upon sphere diameter (d) of the times (t_c) for titanium spheres to cool from T_m + 50K (where the melting point, T_m, = 1940K) to T_m - 50K for helium and argon atmospheres at 760mm Hg pressure. The marked decrease in cooling time with decreasing sphere diameter is clearly shown. In the range of sphere diameters of 100-500μm, an approximate linear relationship between log d and log t_c is suggested with an exponent (n) in the relationship $d \propto t_c^n$ of 0.6 for both helium and argon. For a particular sphere diameter, the cooling times for the higher thermal conductivity gas, namely helium, are shorter by a factor of between 3.5 and 5 than in the case of argon.

Cooling time calculations have also been made for reduced pressures of inert gas, down to 7.6mm of Hg (0.01atm.); the limiting pressure is determined by the requirement that for the empirical correlations for drag coefficient and Nusselt number to be valid, the mean free path of the gas molecules must remain small compared to the sphere

dimensions. The extreme case of very low pressures is however amenable to calculation since both drag forces and convection losses are reduced to zero. Reduced argon pressure cooling times, as well as the ultimate case of radiation only, are also illustrated in Fig.2. The displacement to considerably longer times shown by the 'radiation only' line demonstrates the dominance of convective heat losses even at a few mm. Hg pressure.

In the perspective of chamber dimensions, the more relevant parameter to consider is the distance travelled by a given sphere in the cooling time (t_c) for the temperature interval described above. Fig.3 shows the horizontal component of this distance for selected sphere diameters as a function of pressure for helium and argon atmospheres, as well as the 'radiation only' case. An approximate doubling of the horizontal distance travelled in time t_c can be seen for a change of argon pressure from 760 to 7.6mm Hg pressure, with a further increase by a factor of at least two for the very low pressure case. Helium again demonstrates its superiority over argon in either reducing the required chamber dimensions for a given maximum sphere size or alternatively allowing a larger maximum sphere diameter to be collected in a chamber of given dimensions.

5. CONCLUSIONS

(1) The CSC process provides a method for the production of high purity, spheroidal titanium alloy powders in the size range of 100-1000μm, with considerable potential for scale up and development into a production process.

(2) Observation in detail of the processes of melting and atomisation has been achieved by means of high speed cine-photography.

(3) Calculations of the trajectories and cooling rates of particles have demonstrated the clear advantages of near-atmospheric pressure operation in helium atmospheres as a means of reducing the minimum chamber dimension.

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ACKNOWLEDGEMENTS

The authors wish to thank the Procurement Executive of the Ministry of Defence for their financial support of the cine-photography part of this paper and D. J. Hodkin and the Photographic Section at Harwell for the execution of the photography.

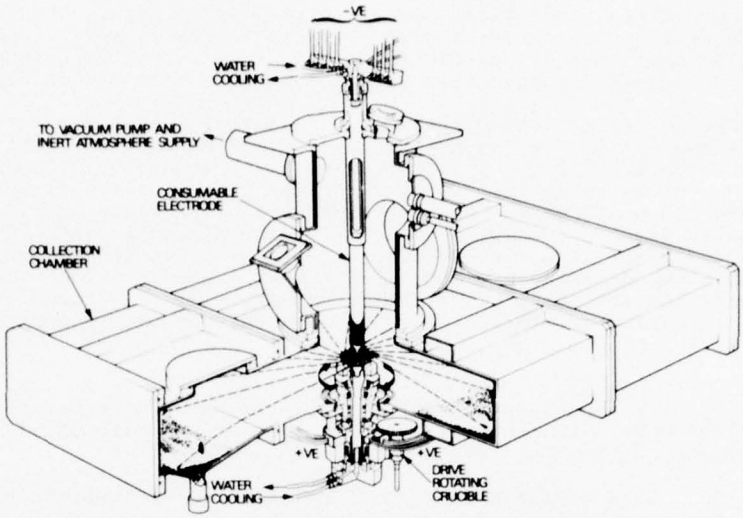

Figure 1 Schematical layout of the Centrifugal Shot Casting Process

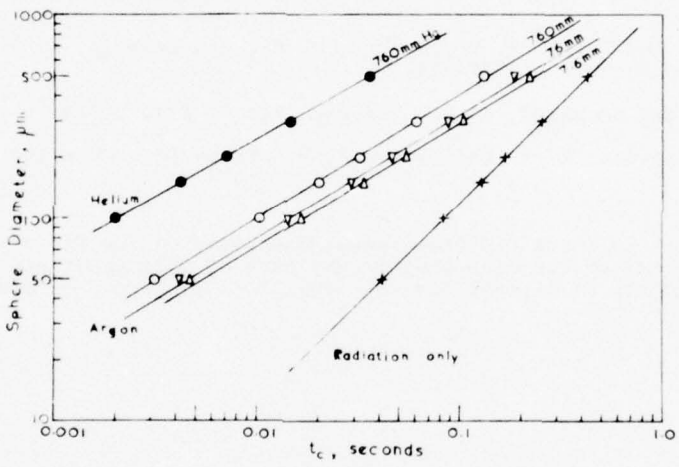

Figure 2 Time taken to cool titanium spheres

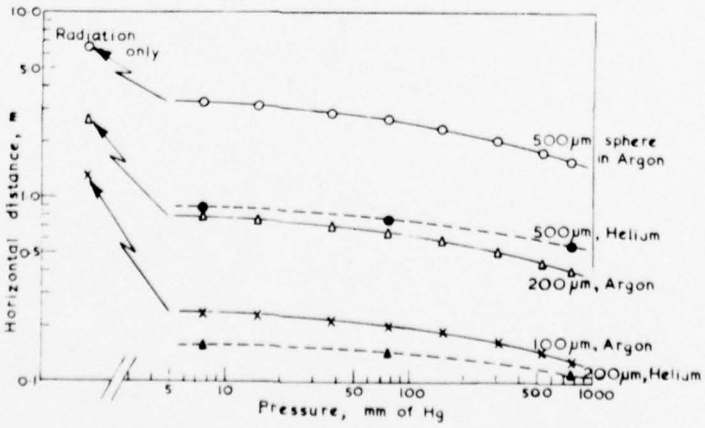

Figure 3 Horizontal distance travelled in time t_c

PERFORMANCE AND ECONOMICS OF HIP EQUIPMENT IN INDUSTRIAL USE

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SUMMARY

Hot isostatic pressing (HIP) is now an established process within some segments of industry and ample experience has been gained from the use of ASEA QUINTUS(R) HIP equipment in production, both cold loaded for cemented carbide products and hot loaded for high speed tool steel. A design for the insulation system of HIP furnaces invented about ten years ago and then further developed has particularly for medium size and large equipment proven to give high reliability and low maintenance cost. Examples of HIP processing costs for a cold loaded unit and a hot loaded pressing line are given. The calculated costs ranging from some tens of cents to about a dollar per kg material being treated should enable a rapidly increasing use of the HIP process.

INTRODUCTION

Hot isostatic compaction was first carried out in the mid fifties at Battelle but industrial application on a broader scale came only this decade and there are now good reasons to expect a more rapid growth of this versatile process.

ASEAs interest in the high pressure field dates more than 30 years back. The aim initially was to make diamonds and the first diamonds were synthesized in Febr 1953 in an ASEA laboratory. Part of the equipment originally developed for that task, the QUINTUS(R) presses with wire wound press frames and wire wound cylinders were later further developed for other advanced applications. A high pressure laboratory was 1965 set up by ASEA at Robertsfors for the development of high pressure techniques for industrial applications. One of the main fields of activity from the beginning was hot isostatic pressing and this is even more pronounced today.

BASIC DEVELOPMENT

Development of equipment for hot isostatic pressing was initiated already during the planning for the laboratory. A hot isostatic press was one of the main equipments. In order to use easily available argon gas as pressure medium and achieve as big useable volume as possible with even and stable temperature in a given pressure vessel a radically new design for the furnace insulation was developed. This basic concept which was developed during 1965 and patented in many industrial countries from 1966 is based on the use of an insulation built up of permanent, gas tight cells. These cells communicate with other spaces inside the pressure vessel only through pressure equalization openings with small height in relation to the total height of each cell. The openings are so located that the dense gas under steady-state conditions will not be exchanged between different cells or spaces inside the vessel but remain stable there. Fibrous insulation material is usually

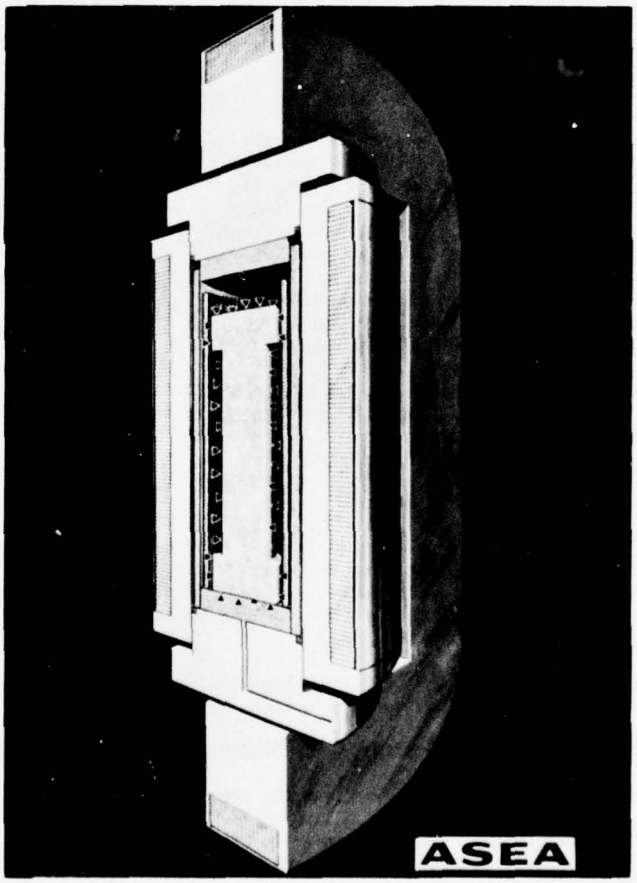


FIG 1

Diagrammatic section of a QUINTUS(R) Hot Isostatic Press. The pressure vessel consists of a wire-wound cylinder with end closures kept in position by a wire wound frame. The heater assembly (cross-hatched) and the thermal insulation (gray) of the furnace are separated in order to minimize thermal stresses.

used to cut down internal convection and radiation within each cell.

This design has provided the basis for further development of furnaces characterized by the reliability and long term stability that is a necessity for the economical use of hot isostatic presses in industrial production. From the beginning it was intended to be applicable even for large furnaces and it has later proven its value in such applications. Even the first furnace utilizing this design at the high pressure laboratory was big at that time with a workspace height with flat temperature of over one meter at 1350°C and 200 MPa argon gas pressure.

HIP EQUIPMENT FOR PRODUCTION PURPOSES

The ASEA QUINTUS(R) presses (Fig 1) for HIP use a pressure vessel built up of a wire wound cylinder and non-threaded end closures kept in position when the vessel is pressurized by a wire wound frame. This design gives a very high degree of safety against major failure and is particularly advantageous for large units. The pressure vessel is opened by sliding the frame to one side whereafter the lower or as in Fig 2 the upper end closure can be pulled out of the cylinder and moved to one side to give free access to the work space. The ducts for heating and permanent thermocouples are always led in through the bottom end closure. In the case of bottom loading these ducts pass through an outer ring-shaped closure permanently attached to the cylinder while an inner closure frees the charging opening. Gas is fed in either through the bottom or top closure.

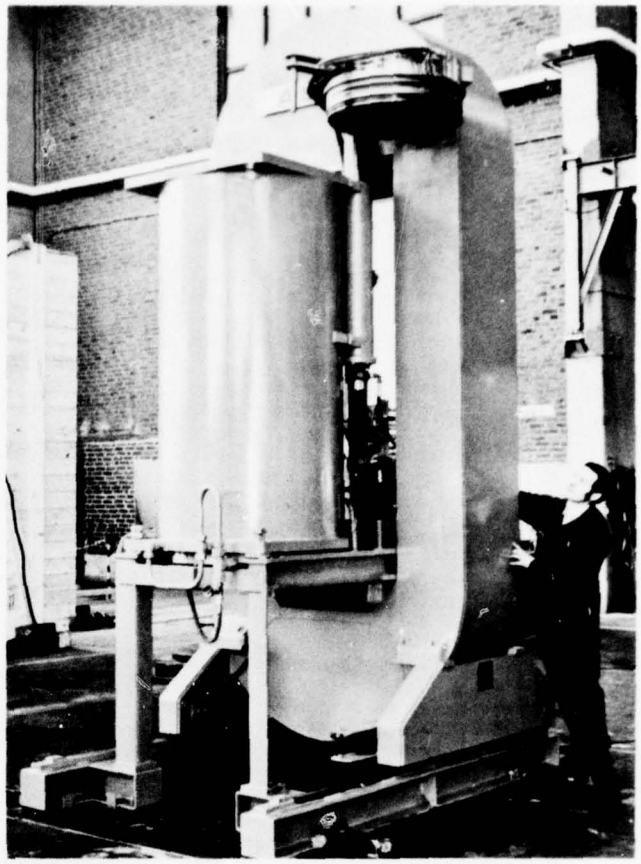


FIG 2

Top loaded medium size QUINTUS HIP unit in an ASEA workshop before delivery.

The furnace insert (Fig 1) is of a modular design. The cylindrical and top insulation, the bottom insulation and the heater assembly can easily be disassembled because no insulation material is packed between insulation mantle and pressure vessel wall or heater assembly. This arrangement both gives a better service life of the furnace because it minimizes the thermal stresses and it facilitates necessary maintenance. Depending on application and parameters, e g max temperature and cold or hot loading, standard designs of heater assembly and insulation are used.

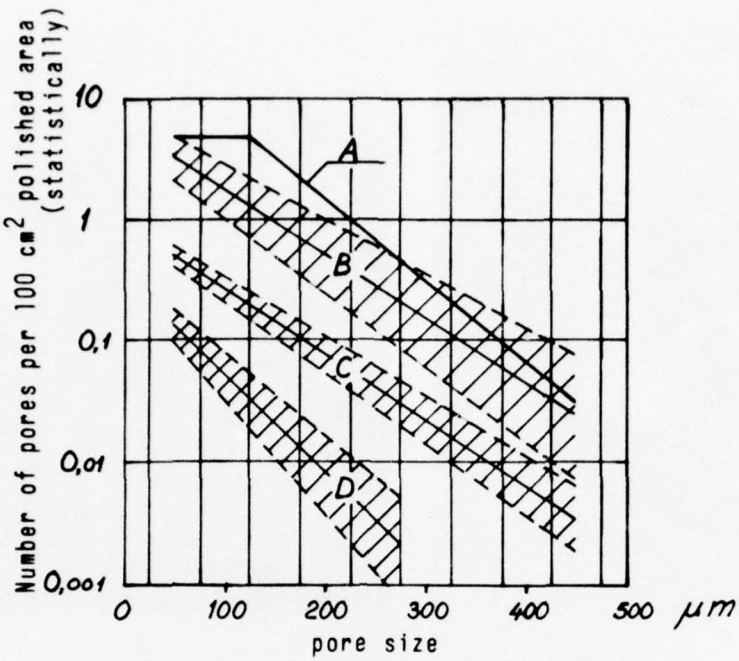
The current production applications are covered by two types of furnaces. One type used for tool steel production and nickel alloy treatment is designed for hot loading and a maximum temperature of 1260°C. Another type used for treatment of cemented carbide is designed for cold loading and max 1400°C. The temperature capability of this furnace can be extended to 1750°C with an additional insulation module inside the standard insulation.

The furnaces operate equally well at 100 or 320 MPa; this is a consequence of our advanced insulation system, but the pressure vessels and gas supply system must of course be designed accordingly.

PERFORMANCE AND ECONOMICS OF 1400°C HIP UNITS

The commercial breakthrough for HIP came about five years ago with a process for pore elimination (defect healing) in cemented carbide materials. It was announced in 1971 by Sandvik Coromant in Sweden and Kennametal Inc in USA, the two largest manufacturers of cemented carbide in the world. The process had been independently developed by Sandvik in cooperation with ASEA and by Kennametal. The very significant effect of hot isostatic pressing on porosity is visualized from Fig 3.

ASEA has delivered most of the hot isostatic presses for treatment of cemented carbide materials (Fig 4). The heater assembly is of molybdenum and has a minimum of ceramic components to allow rapid heating up and cooling down (Fig 5) as the equipment is loaded and unloaded cold. Top loading is most common but bottom loading is sometimes used. The load is usually placed in graphite structures which can allow thousands of components per cycle to be treated (Fig 6).



Porosity distribution in cemented carbide samples from production

A) Small parts (~10 g each)
B) Large parts, conventionally sintered
C) Large parts, hot pressed in graphite dies
D) Large parts, hot isostatically pressed
(courtesy S Amberg, Sandvik Coromant)

FIG 3

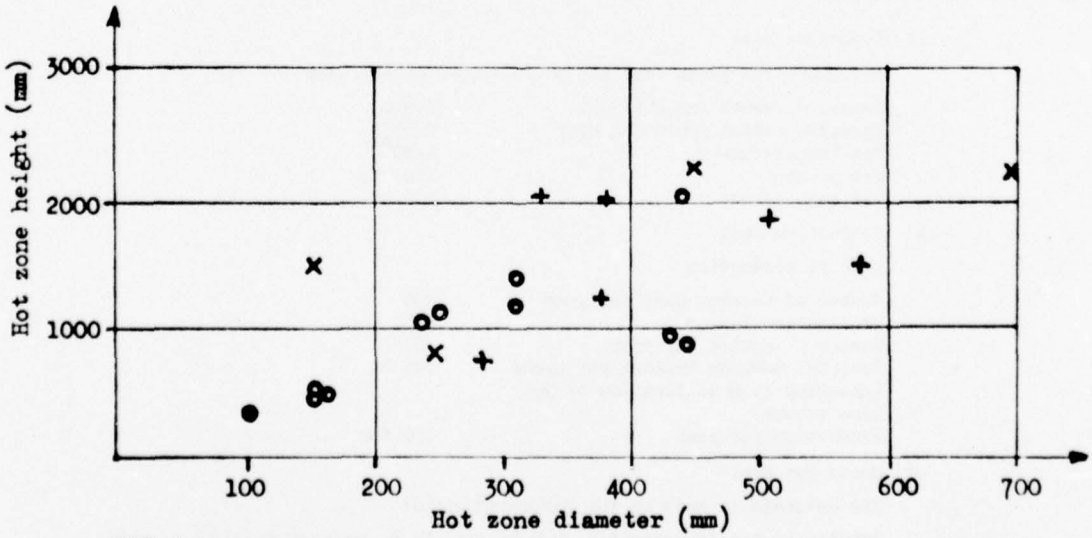


FIG 4

Data for ASEA QUINTUS Hot Isostatic Presses for treatment of cemented carbide materials delivered 1970-76.

Symbols

x Unit for max pressure 100 MPa
+ Unit for max pressure 160 MPa
o Unit for max pressure 200 MPa
● Unit for max pressure 320 MPa

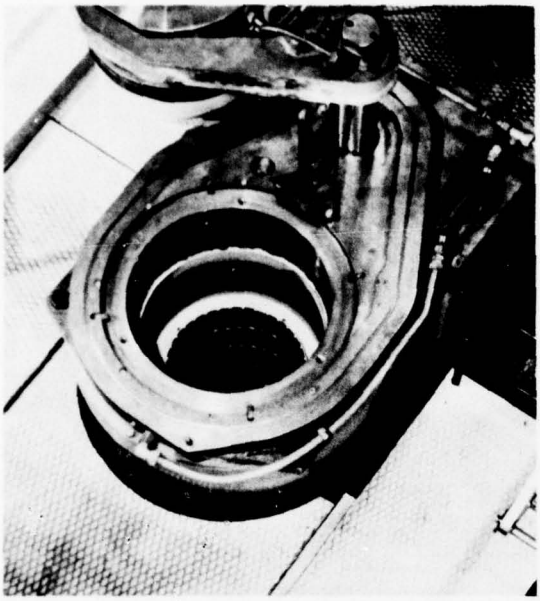


Fig 5

View of the interiour of a 1400°C HIP unit.

Fig 6

Multi-component cemented carbide charge on graphite charging fixtures being lowered into a hot isostatic press.

Example of HIP processing costs

The following example is for a medium size unit for HIP treatment of cemented carbide parts. In 1-shift production and cold loading one cycle per shift can be made because part of the cooling down of the charge can be made unattended. The experience gained from customers running QUINTUS(R) HIP equipment for this product in production has helped us to asses realistic figures for e g utilization factor and maintenance cost.

The installation in the example consists of a hot isostatic press complete with gas system, electrical system and cooling system.

The investment cost (Europe, Jan 1976) used in the calculation below includes installation cost but costs for the building are paid as rent.

a) Technical Data

Hot isostatic press type QIH 50-160/2,0-0,63 UCT 1400

Pressure vessel inner height	2,0 m
Pressure vessel inner diameter	0,63 m
Max temperature	1400°C
Max pressure	160 MPa
Hot zone volume	0,2 m^3

b) Production Data

1-shift production

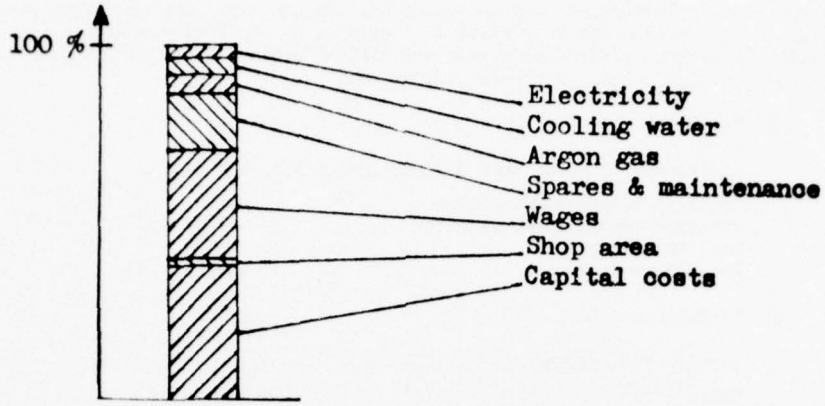
Number of working days per year	230
Utilization factor	0,9
Number of cycles per year	207
Cemented carbide treated per cycle (assuming 25 % utilization of hot zone volume)	725 kg
Production per year	150 ton

c) Costs per cycle

The estimate is made on the following basis:

Annuity on the investment 21,9 % (7 yrs	12 %, rest value 20 %)
Shop area	$ 57 per m^2 and year
Wages	$ 11,4 per hour
Argon gas	$ 2,3 per Nm^3
Cooling water	$ 0,23 per m^3
Electricity	$ 0,034 per kWh

The cost per cycle will then be appr $ 700 built up as follows:



The cost for HIP treatment of cemented carbide would then be $ 0,97 per kg. The actual costs vary of course with the average utilization of the hot zone volume.

ECONOMICS OF 1260°C HIP UNITS FOR HOT LOADING

For hot-loaded HIP equipment operating up to 1260°C are oxidation resistant materials used throughout the furnace and Kanthal bands are used as heating elements. We have after a careful study come to the conclusion that hot bottom loading is clearly to prefer before hot top loading. Bottom loading permits the use of fully ceramic charging fixtures which makes multi-component treatment possible. The charge is placed on one of several charging plates which follows the charge from loading station through preheating and HIP furnaces to the unloading station. Workpiece thermocouples can be attached to the charge while cold and no personnel needs to be close to the charge while hot. Furthermore the service life of the furnace is improved because cold air does not flow into a hot bottom loaded (bell type) furnace as it does with a top loaded one. Inert handling manipulator for the charge can be supplied.

A high speed tool steel billet (weight 1700 kg) consolidated from powder in a hot bottom loaded HIP line according to the ASEA-STORA process is shown in Fig 7.

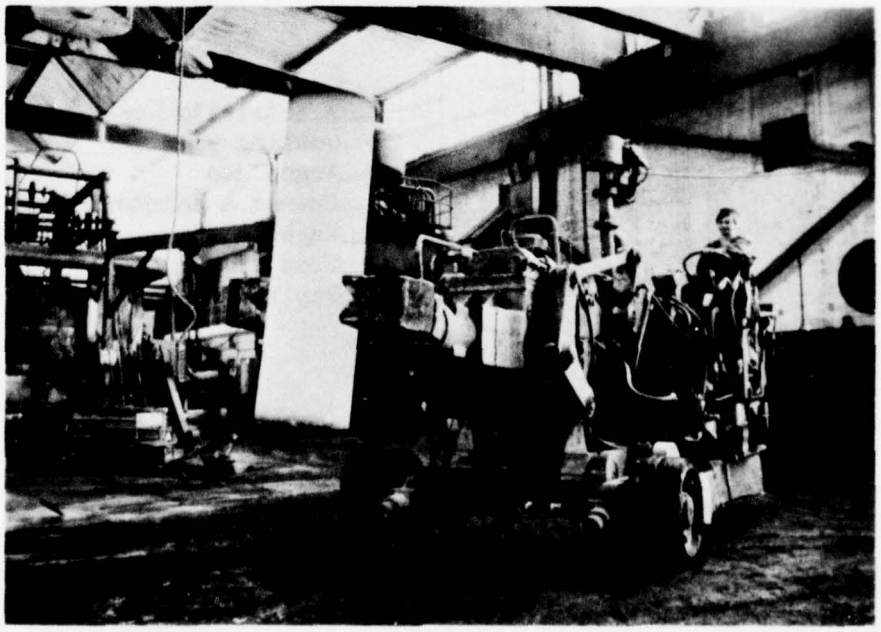


FIG 7

Big high speed tool steel billet (weight 1700 kg) in a forging manipulator. The billet has been made in a hot bottom loaded HIP line.

Example of costs in a HIP plant for high production

The plant used in the following cost calculation is a complete HIP line for high production of e g superalloy parts from powder or defect healing of castings. It consists of a bottom loaded hot isostatic press, four preheating furnaces, one loading and one unloading station and six charging plates with work piece thermocouple ducts. The charge is moved from station to station by an inert gas manipulator. The installation is complete with gas system, electrical system and cooling system. Installation costs are included in the investment but not building costs, which are paid as rent.

a) Technical data

Hot isostatic press type QIH 80-100/2,5-1,0, UHB 1260

Pressure vessel inner height	2,5 m
Pressure vessel inner diameter	1,0 m
Max temperature	1260°C
Hot zone volume	0,63 m^3

b) Production data

3-shift production

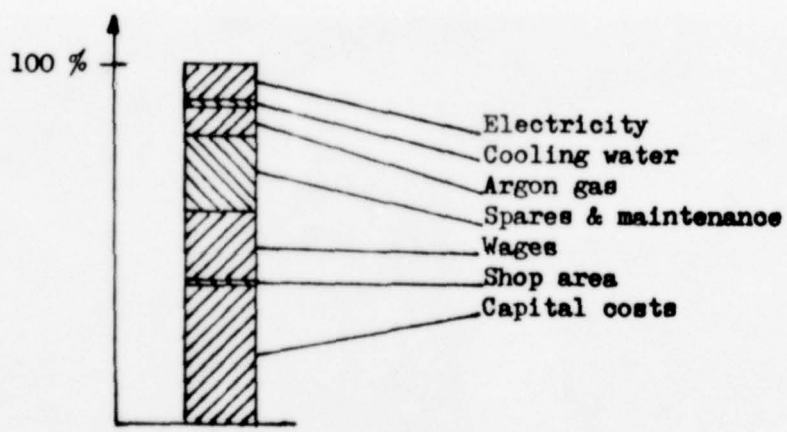
Cycle time	4 h
Number of working days per year	230
Utilization factor	0,9
Number of cycles per year	1242
Weight material processed per cycle (assuming 30 % utilization of hot zone volume starting from superalloy powder or 20 % utilization starting with solid superalloy parts)	appr. 1000 kg

c) Costs per cycle

The estimate is made on the following basis:

Annuity on the investment 21 9 % (7 yrs, 12 %, rest value 20 %)	
Shop area	\$ 57 per m^2 and year
Wages	\$ 11 4 per hour
Argon gas	\$ 1,35 per Nm^3
Cooling water	\$ 0,23 per m^3
Electricity	\$ 0,034 per kWh

The cost per cycle will then be appr. \$ 650 built up as follows:



The costs for HIP treatment of superalloy products would with these assumptions be \$ 0,65 per kg. With a higher utilization of the hot zone volume the costs per kg for consolidation of superalloy powder could be as low as \$ 0,20.

CONCLUSIONS

HIP is now a well-proven industrial process for some applications. Efficient equipment with the reliability and low maintenance cost needed for a wider industrial use is now available to the market. The level of the processing costs ranging from a few tens of cents to about a dollar per kg processed material should make the process of interest for a rapidly increasing number of applications.

PROCESS AND ECONOMIC CONSIDERATIONS FOR PRODUCTION SCALE HOT ISOSTATIC PRESSING EQUIPMENT

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Equipment which makes the Hot Isostatic Pressing Process an economical and viable industrial process is now well developed.

Hot and Cold Loading Process Systems are compared at a production rate of one cycle per eight hour day.

Other comparisons for the two systems include equipment types, equipment costs, and the effect of material processed.

In the past two years a number of systems have been designed and built which process powder compacts during a continuous duty eight hour cycle. These systems are capable of three production cycles in a twenty-four hour day.

The experience of the users and the equipment suppliers has resulted in a much more definitive equipment and usage specification.

Two basic system types are currently considered for production applications. These systems are generally defined as:

A. Cold to Cold

B. Hot to Hot

The selection of the best system from the users standpoint depends on the processing requirements of the material and the economics of the system.

A Cold to Cold System is defined as a process in which a cold material load is placed in a cold furnace. Subsequently, the furnace and the load are heated, pressurized, and allowed to cool together to a safe unloading temperature.

A Hot to Hot System is defined as a process in which a part is loaded into a hot furnace. The part is heated and pressurized simultaneously. The part is then removed hot from the furnace.

In order to achieve the most economical production costs, it is necessary to minimize the furnace/pressure vessel cycle time. Pressure vessel cycle time for the Hot to Hot System is reduced by preheating the work load. Cold to Cold cycle time is reduced by using a furnace capable of rapid heating and cooling rates. This type of furnace is usually characterized by low thermal inertia and a high power input.

In evaluating the Hot to Hot and Cold to Cold System types, one must give consideration to processing features which affect metallurgical results and to the degree of flexibility required to process various material types. In general, it should be noted that most parts can be processed in either a Cold to Cold or Hot to Hot System. Two notable exceptions are: (1) Titanium, which requires a high purity atmosphere, and (2) Carbides, which requires temperatures in excess of 1250° C. for processing.

Table #1 lists advantages of the Cold to Cold System.

TABLE #1

COLD TO COLD

1. Pure environment.
2. Fast thermal response.
3. High temperatures.
4. Flexible thermal programs.
5. Ease of specimen thermal monitoring.

ADVANTAGES HOT TO HOT

1. More production orientated of economical parts run to run.
2. Repeated process parameters.
3. Less cost per pound of material processed.
4. Lower operating cost and maintenance.

EQUIPMENT

When production equipment is considered, the concept of multiple work stations must be employed. The following equipment descriptions make use of this concept.

A typical Cold to Cold System is described in Figure #1. This approach consists of one loading station, three movable furnaces used both for preheat and isostatic pressing, one pressure vessel, and a combination load furnace transfer mechanism. This equipment represents the major equipment items; support equipment is discussed later.

The process functions as follows:

A. The workpiece is placed at the loading station where thermocouples are connected as required.

B. One of the three preheat/HIP furnaces is placed over the workpiece and connected to electrical and vacuum utilities located on a base assembly.

C. A typical Cold to Cold process temperature cycle is shown in Figure #2. This cycle consists of eight hours preheat, eight hours at pressure, and eight hours cooling.

D. It is noted again that these furnaces are evacuated, purged, and preheated outside the pressure vessel and transferred hot to the pressure vessel. During the transfer, the furnace is so designed that the outside remains cool.

Process control of the furnace on the preheat stand and inside the pressure vessel is accomplished using computer or conventional control equipment.

Gas, power, and control utilities are connected using an automatically operated plug-in arrangement located on each furnace base and in the pressure vessel.

The Hot to Hot concept is described in Figure #3. This system consists of one loading station, three stationary hot preheat furnaces, one pressure vessel containing a hot HIP furnace, and a work load transfer mechanism.

The process as shown by Figure #3 is as follows:

A. A workpiece is placed in transfer container at the loading station.

B. The cold workpiece is transferred to the hot preheat furnace.

C. After preheating, the hot load is transferred to the pressure vessel furnace combination.

D. Following heating and pressurization, the hot billet is extracted and returned to the loading station.

E. A typical Hot to Hot process pressure temperature cycle is shown in Figure #4.

SYSTEM COST CONSIDERATIONS

Table #2 compares equipment required for each system. Major differences show up in Items #1 through #5. Although there are minor differences in other areas, these are not considered to have a major effect on the total cost.

For competitive reasons, actual costs or sizes for the systems are not revealed. However, it should be noted that the systems described are based on workpiece sizes in excess of 24" diameter x 50" long. For this evaluation, units of equal production volume have been considered and are based on turnkey type installations complete with building costs.

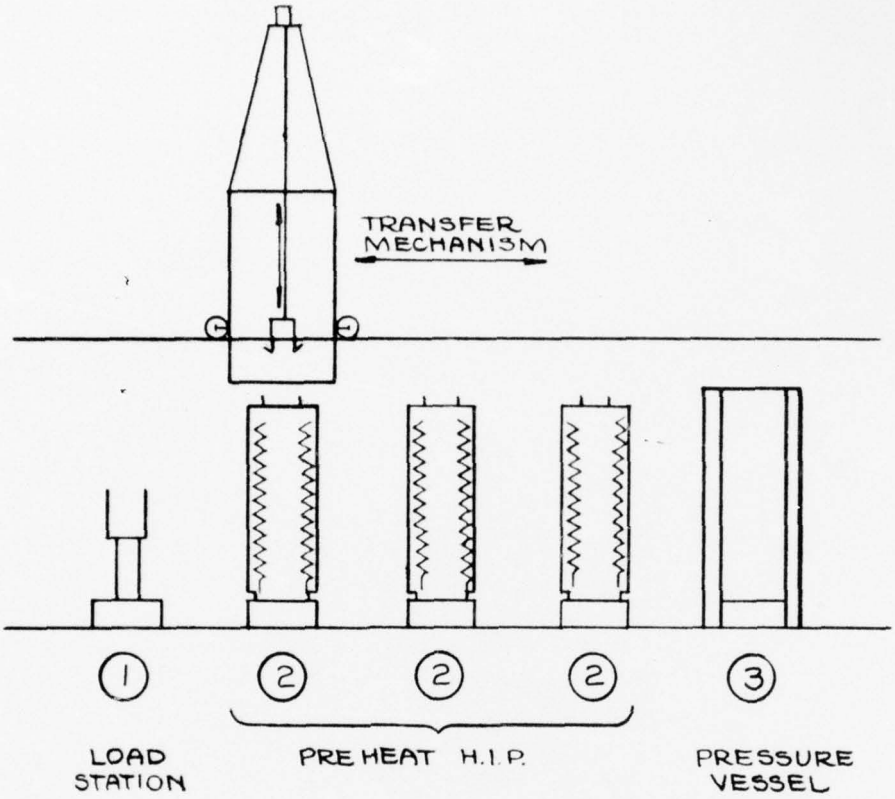
TABLE #2

	COLD TO COLD	HOT TO HOT
1. Pressure Vessels	1	1
2. HIP Furnace	3	1
3. Preheat Furnace	---	3
4. Load Stations	3	1
5. Transfer Mechanism	1	1
6. Power Systems	4	4
7. Control System	4	4
8. Vacuum System	4	4
9. Purge System	3	3
10. Cooling System	1	1
11. Compressor System	1	1
12. Gas Storage	1	1

SUMMARY

Our current data suggests that a Hot Loading System, complete as noted, can be installed for approximately 75% to 80% of the cost of an equivalent Cold to Cold System.

Once a basic system style has been selected, it is of great interest to compare the effect of diameter and length on the cost to process an equivalent volume of parts. This consideration is more difficult to evaluate since it depends primarily on customer product mix and maximum workpiece diameter. It is not considered at this time.



COLD TO COLD SYSTEM
WITH FURNACE TRANSFER SYSTEM

FIG. 1

AUTOCLAVE ENGINEERS, INC.

COLD TO COLD SYSTEM

1. LOAD WORK & CONNECT THERMOCOUPLES
2. PLACE FURNACE OVER WORK PIECE
3. EVACUATE & PURGE
4. START HEATING
5. TRANSFER LOADED FURNACE AT POSITIVE PRESSURE
6. PRESSURIZE & COMPLETE THERMAL CYCLE
7. RETURN TO LOAD STATION AT POSITIVE PRESSURE

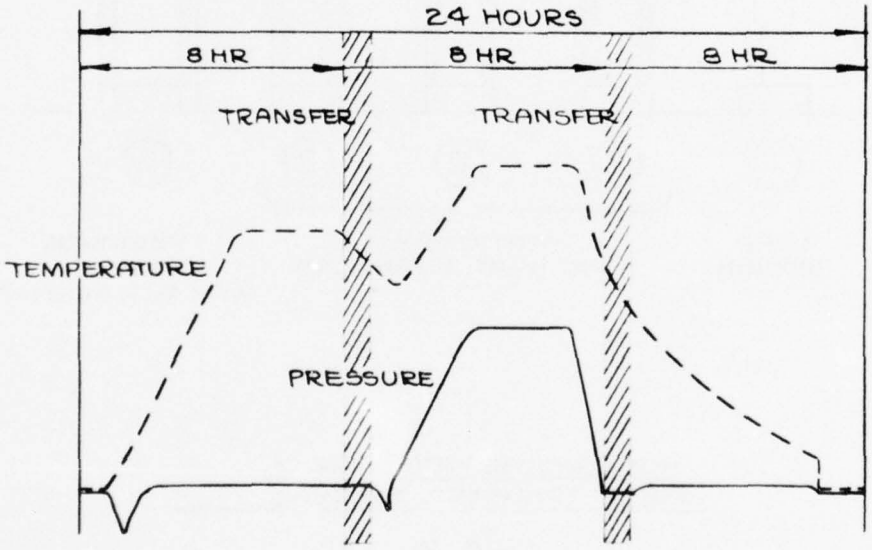
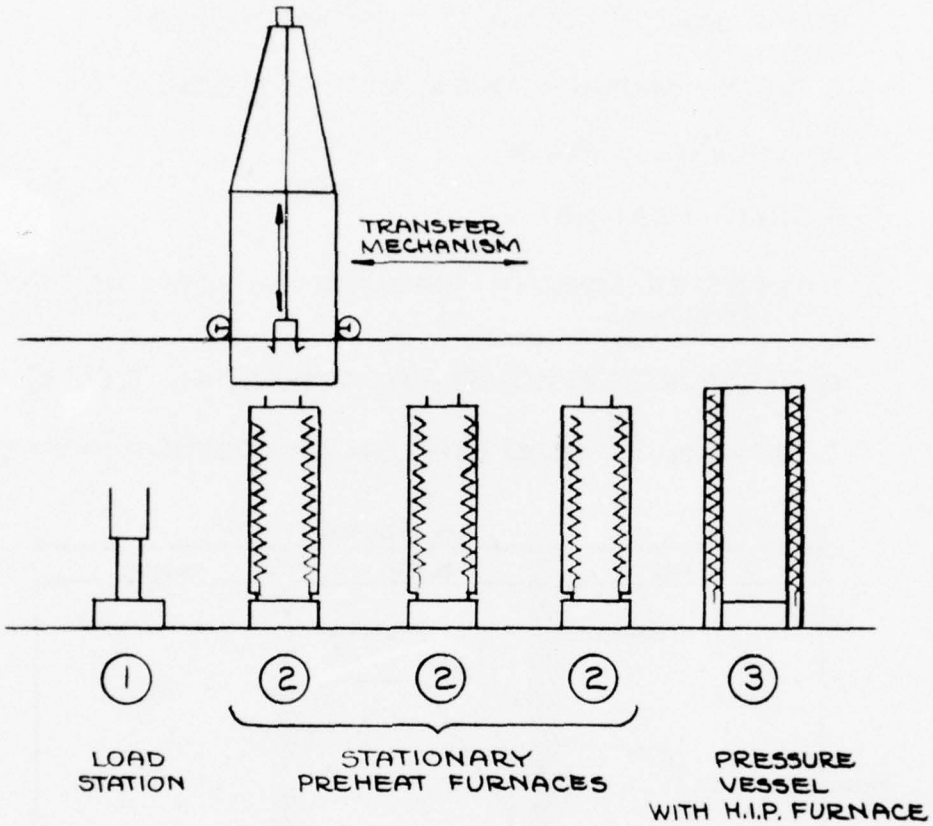


FIG. 2

AUTOCLAVE ENGINEERS, INC.



HOT LOADING WITH WORK-PIECE TRANSFER SYSTEM

FIG. 3

AUTOCLAVE ENGINEERS, INC.

HOT LOADING SYSTEM

1. LOAD WORK IN TRANSFER FIXTURE
2. PLACE IN PREHEAT FURNACE
3. PURGE
4. TRANSFER LOAD TO H.I.P.
5. PRESSURIZE & COMPLETE THERMAL CYCLE
6. RETURN TO LOAD STATION (HOT)

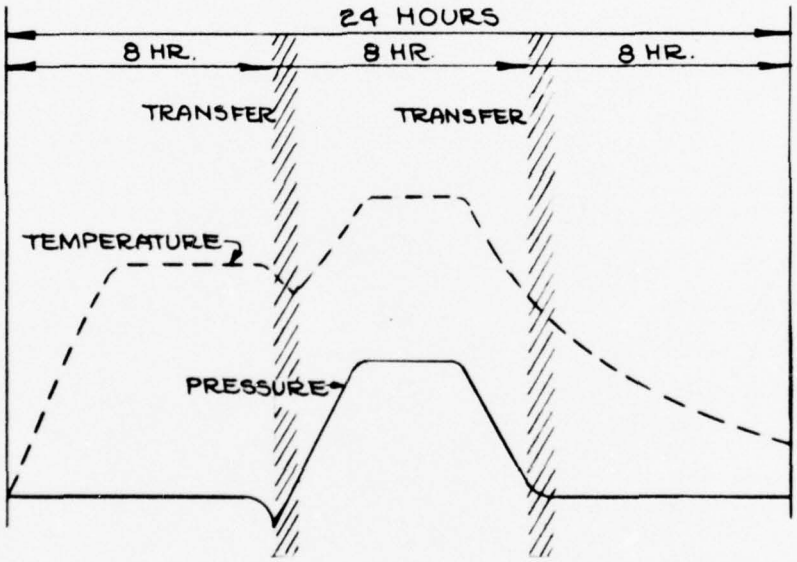


FIG. 4

AUTOCLAVE ENGINEERS, INC.

NOTES ON SOME ECONOMIC ASPECTS OF HIP

by

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Summary

Two areas of interest involving Hot Isostatic Processing are addressed. Some of the considerations involved in the concept of operation with respect to preheat are presented. Additionally, some of the results of a study intended to give indications of floor-to-floor processing costs are briefly presented.

Considerable activity has been directed in the configuring of HIP manufacturing systems where the utilization of preheat is viewed as a necessity. Clearly, there is a class of products where the utilization of preheat is indicated. However, this concept of operation should be chosen on the basis of a consideration of the trade-offs involved.

The primary reason for preheat is usually for the reduction of vessel residence time with an attendant increase in throughput. The alternative to preheat is vessel resident heating. A contrast of these two methods will show the kinds of considerations that should be kept in mind when selecting a concept of operation.

First, I would like to dispose of the matter of partial preheat. Figure 1 illustrates the centerline temperature of a constant property material when heated with the most rapid method through the surface. It is obvious that the minimum internal temperature change rate, while fairly rapid initially, has a slow roll-in when the temperature defect is small.

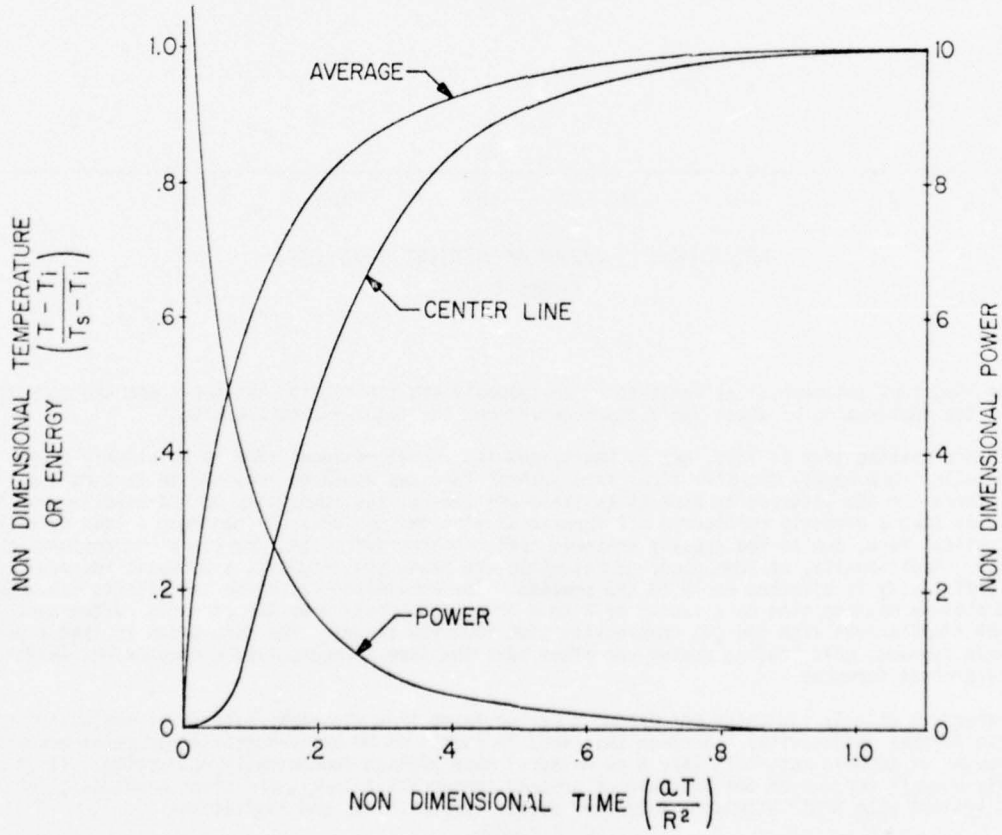

FIGURE 1

Figure 2 is an illustration of this effect by comparing a uniform preheat of a certain amount (expressed as a percent of the desired temperature), to the reduction in subsequent heating time required at various allowable temperature defects (expressed as a percentage of nondimensional temperature). Clearly, preheat of at least 95% is necessary to significantly reduce subsequent heating time.

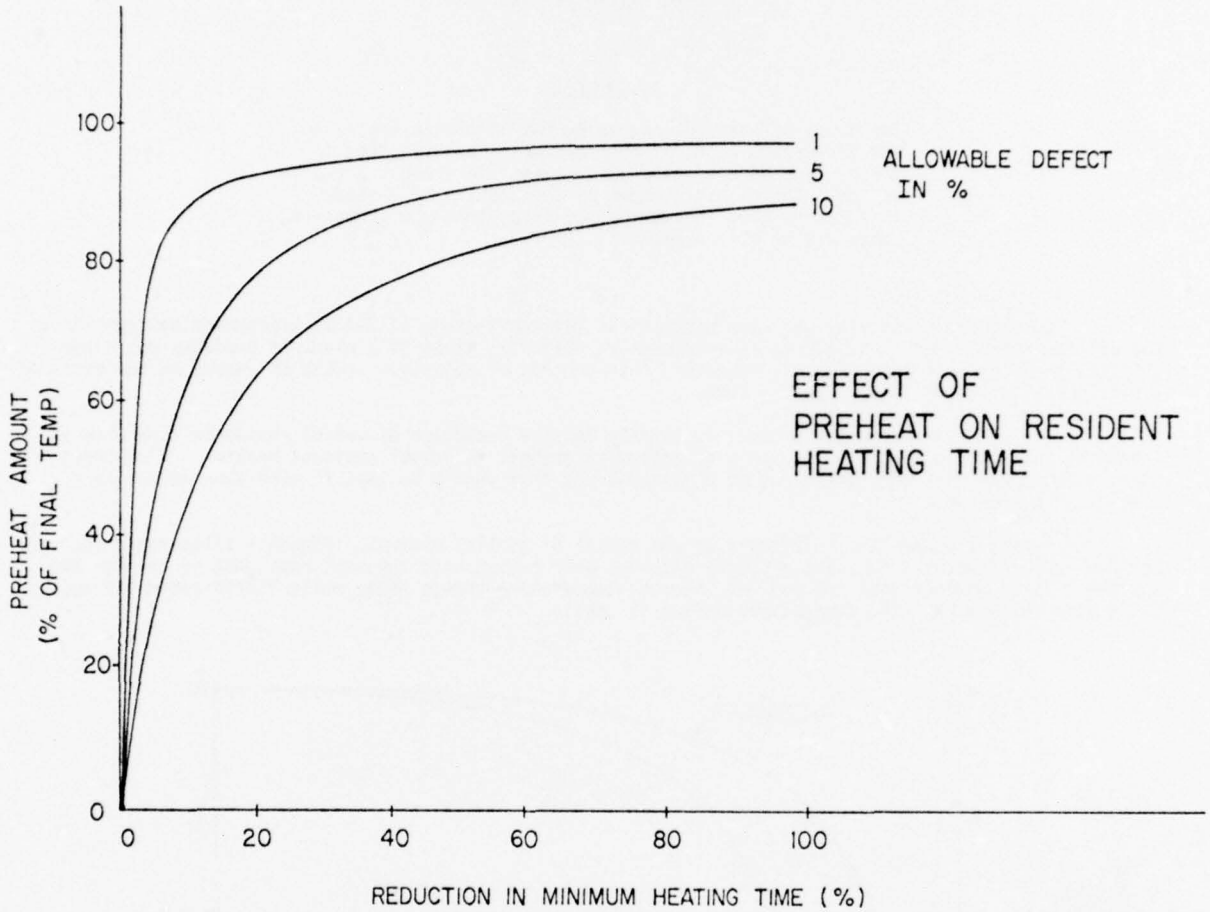

FIGURE 2

The important thermophysical features of an assembly are the section thickness and the mean thermal diffusivity. The combination of these two factors determines the required heating time.

If this heating time is long, say 20 hours, and the vessel resident time is relatively short, say 5 hours, even allowing a modest transfer time, five preheat furnaces would be required to support the throughput. There are two interesting aspects to trade-off against the complexity of hot handling and hot loading. One is that a properly configured HIP furnace at elevated gas pressure can heat a load near the maximum theoretical rate, due to the greatly enhanced heat transfer mechanism, whereas a conventional preheat furnace cannot. Additionally, as some powdered materials are being consolidated, a dramatic increase in the thermal diffusivity is effected early in the process. The combination of these two effects can reduce the required minimum heating time by a factor of 2 to 4 or more, depending on the product. Often this heating can be simultaneous with the gas compression time required anyway. The conclusion is that a properly designed single furnace, cold loading system can often have the same throughput as a complex hot loader with multiple preheat furnaces.

Preheat is clearly indicated for products having large time constants (square of characteristic dimension/mean thermal diffusivity). Section thickness in excess of 50 cm in most alloyed metal powders or large thicknesses of ceramic materials (say 8 cm or more) make preheat economically attractive. It is my view that only a small portion of the products of present commercial interest are clear candidates for preheat type systems with their attendant increased costs, complexities, and limitations.

Obviously each producer contemplating HIP should conduct appropriate analysis of the expected product mix prior to committing to any particular concept of operation.

Cost Indication Study

CPSI conducted a cost study that was intended to give rough indications of "floor-to-floor" HIP processing costs. "Floor to floor" is defined to be that portion of the manufacturing process from the time the assembly is ready for loading into the HIP equipment (including preheat where appropriate) until the assembly is consolidated and ready for subsequent processing. The analysis was broadened as much as practical to allow for variations in concept of operation and type of processed material. The following cost items are included:

(1) Capital equipment amortization, including all items added to the installed cost

(2) Labor for operation, maintenance, supervision, and administration

(3) Electrical power required

(4) Materials consumed

(5) Factory space, including other utilities, taxes, and general upkeep.

The rates utilized for the various items were high average and were based on equipment manufacturers quotes and actual operating experience. An equipment availability factor of 65% was utilized.

The most convenient correlation between cost items and throughput was found in the concept of gross volumetric displacement of the HIP processing chamber. This is the space available for load within a HIP unit. This allows for any packing and tooling considerations to be taken up separately. Although correlations exist for each cost item, I will present only the overall results. Figure 3 presents total cost-per-gross volume of product as a function of throughput in gross volume per year at various vessel residence times (VRT).

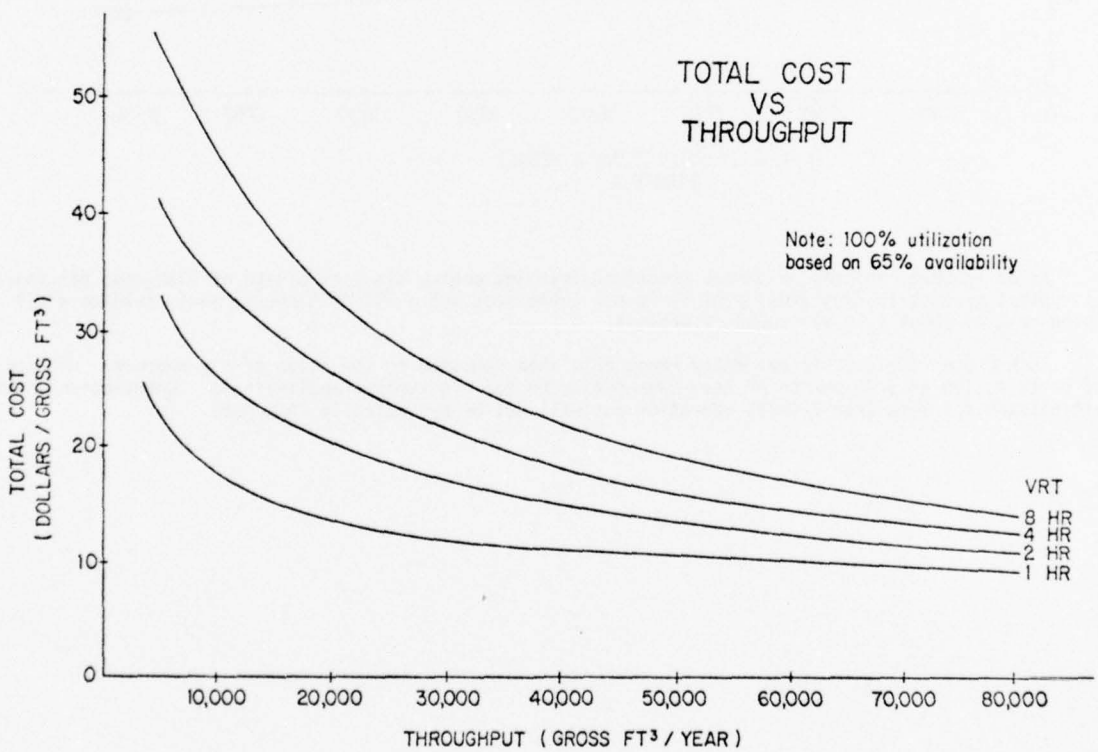

FIGURE 3

Obviously, it's up to each producer to make an estimate on the amount of product he can get into the gross volume unit and how many such units will be processed per unit time.

Figure 4 illustrates a further breakdown for only one VRT (8 hours), giving cost-per-unit mass as a function of unit mass throughput at various product packing densities, a more immediately meaningful correlation.

I wish to emphasize that a number of simplifying assumptions were necessary, and these figures should be used only as an indication of expected costs. For a given product mix and factory location, far more accurate results can be obtained.

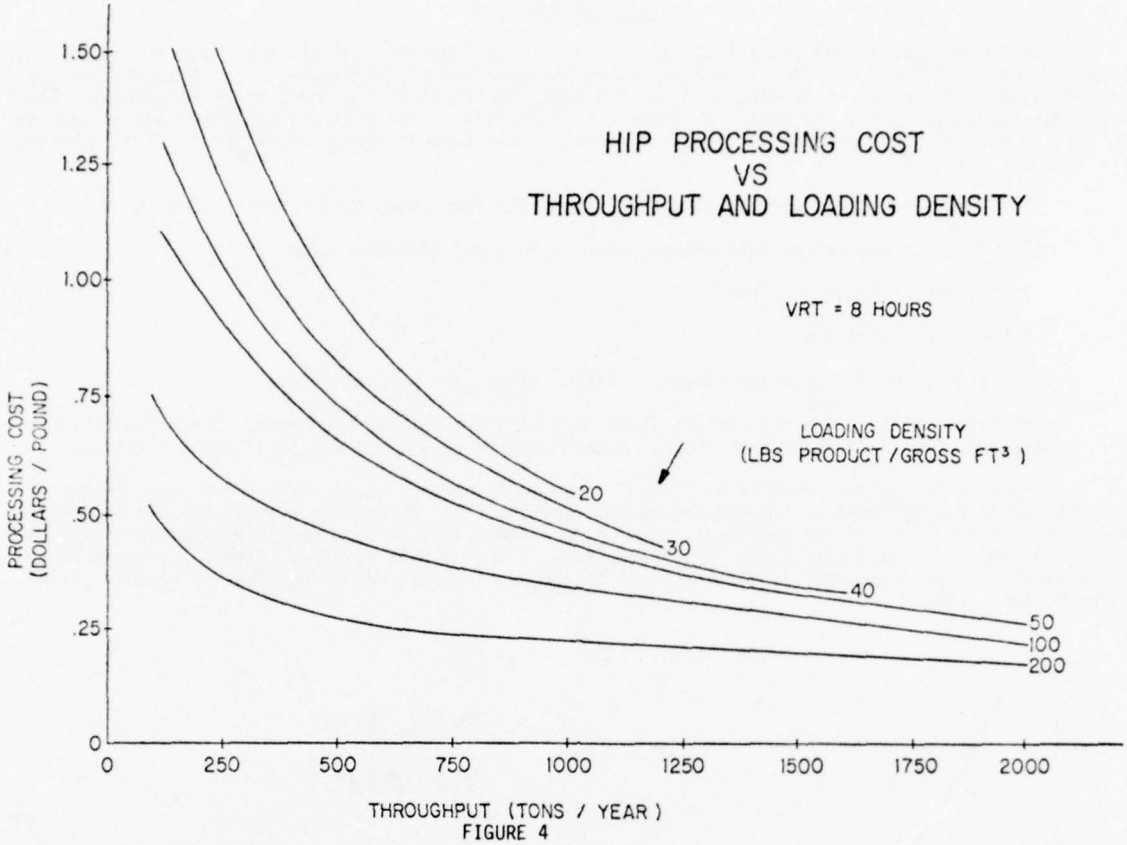

FIGURE 4

As an example, superalloy powder processed into jet engine discs at a rate of 1000 tons per year, using a typical product loading density of 50 lb per cubic foot and a VRT of 8 hours, would involve a HIP processing cost of about $.50 per pound of product.

Such processing cost is eminently reasonable when compared to the value of the product. HIP processing costs as low as $.03 per pound have been projected for high-volume applications. Correlations have been established for less than 3-shift operation but will not be presented in this note.

DISCUSSION SUMMARY OF SESSION I

by

J.N.Fleck

Because of a late start, the discussion periods in this session were somewhat abbreviated.

Most of the discussion regarding powder manufacture centred about the problem of inclusions. It was pointed out that inclusions are especially detrimental to fatigue performance. Thus, fatigue testing should be included in any powder evaluation activity.

Concern was also expressed over use of potential sources of contamination such as crucibles, electrode holders, or even chamber linings in powdermaking equipment. Potential solutions offered include special consumable coatings which are not detrimental to the powder.

The cost situation was also the subject of some discussion. Regarding the Rotating Electrode Process, it was noted that efforts are underway to scale the electrode size from the present 2.5 inch (6.4 cm) to 5 inch (12.7 cm) in diameter. In response to a query regarding energy efficiency of the electron beam process, Dr Stephan noted a that the current power consumption is 2.5 to 3.0 kilowatt-hour per kilogram (kWhr/kg). In production, this should be reduced to about 0.8 kWhr/kg for nickel and 1.0 kWhr/kg for titanium alloys.

In a brief discussion of the hot-to-hot HIP loading concept, the amount of preheat was questioned. It was agreed that either a partial or full preheat could be technically feasible. It was also noted that Autoclave Engineers favors top loading equipment whereas ASEA advocates bottom loading. The ASEA representative suggested that, for hot loading, the bottom load approach is advantageous.

SESSION II

NICKEL SUPERALLOY POWDER PRODUCTION AND FABRICATION TO TURBINE DISCS

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&

F.A. Thompson

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SUMMARY

The ever-increasing demand for better alloys to enable gas turbine engines to operate more efficiently has led to the development of powder techniques for the production of certain critical nickel-base alloy components. One area of application in particular has received more attention than any other, this being turbine discs where the requirement is not for the ultimate in high temperature resistance but for optimised mechanical properties at intermediate temperatures. These components lend themselves ideally to manufacture by the powder route since nearly all the advantages of powders over conventional routes can be utilised. At Henry Wiggin and Company Limited an integrated powder production and compaction plant has been installed aimed at the commercialisation of this type of component. Work within the Company on the forging of discs from Hot Isostatically Pressed (HIPed) billet is reviewed along with indications of the potential for other techniques applicable to the production of powder discs, i.e. preforms plus forging, direct HIP to shape, isothermal forging, thermoplastic processing.

INTRODUCTION

The technical advantages of the atomised, prealloyed powder route for the manufacture of nickel-base superalloys and certainly for those of advanced design have been apparent to those engaged in the aerospace industry for a number of years. To maintain its role as a leading supplier of such materials and to promote this new technology within Europe, the author's Company has installed a powder production and consolidation plant at its Hereford works. Recent economic trends have caused engine builders to become increasingly cost-conscious and they are now encouraging the cost saving aspects of the powder metallurgical (P/M) approach rather than merely its technical advantages. Thus depending on the applications, properties required and costs of production, a whole range of possibilities and component production routes need to be considered and evaluated.

The areas in which the major effort on superalloys is currently being concentrated is in the production of turbine discs. For this component the advantages of the P/M approach can be summarised as:

a) Technical - P/M allows large components of advanced alloys to be produced and processed enabling improved engines to be designed and built.

b) Economic - by consolidating powder directly to the required disc shape, considerable cost savings are possible. On this basis it should be more cost effective in some instances to replace existing discs produced by conventional cast/wrought procedures with powder components.

Bearing in mind these major advantages a number of possible processing routes become evident and it is proposed to highlight some of them by outlining the work being performed at Henry Wiggin and Company Limited.

POWDER PRODUCTION AND CONSOLIDATION

1. INERT GAS ATOMISATION

A number of atomisation techniques for nickel-base superalloys are at present operational on a production basis. It is not intended to review these techniques in this paper but to outline inert gas atomisation and in particular the practices followed at Henry Wiggin and Company Limited. A schematic diagram of the atomiser is shown in Fig. 1 consisting primarily of a vacuum melting chamber set on top of an atomisation tower. The furnace is a 500 kg induction melting unit which is capable of maintaining temperature with the varying level of melt experienced during an atomisation run. The melting chamber, Fig. 2 is equipped with a bulk loader, small additions hopper, sampling device and temperature probes, all of which are needed for the making of complex superalloys. Although most heats of superalloy powder made to date have been from selected raw materials, the unit is capable of melting master alloy, but this would no doubt increase costs due to the double melting operation. Practices have in part been established also which allow for the recycling of certain percentages of scrap.

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Melting is performed under a vacuum of 10^{-3} mm Hg after which the melt chamber and atomisation tower are back-filled with argon. The molten metal is poured steadily into a preheated tundish incorporating a slag trap before being fed through a small diameter refractory nozzle. Immediately below the nozzle is the gas jet assembly which atomises the molten stream, the powder so produced falling to the bottom of the atomisation tower for collection in water cooled skips. The argon is then directed through cyclones to remove any fine powder after which it is vented to atmosphere. The technique of inert gas atomisation has been under study at International Nickel Inc. for a number of years and the basics of their technology have been applied to the Henry Wiggin production atomiser.

Powder particle size and overall yields are controlled by:

a) Degree of superheat
b) Refractory nozzle diameter
c) Hydrostatic head
d) Atomising jet gas pressure
e) Gas jet diameter and configuration

Although a great deal of fine detail is still to be learnt about these parameters, a large quantity of superalloy powder has been produced. Typical appearance, physical properties and gas levels are shown in Fig. 3 and Table 1. The powder is essentially spherical with the occasional satellite particle but the design of the atomiser and jet configuration is such that misshapen particles, due to impingement on the atomiser wall, do not occur.

Argon levels determined analytically to a precision of σ = 0.17 ppm at the 2 ppm level, are 0.5 to 3.0 ppm, with the higher figures occurring in the coarser fractions. This is confirmed by occasional gas porosity seen inside the coarse powder which at this stage is always removed by sieving to -80 or -100 mesh before further processing. Oxygen and nitrogen contents are well below the limits of 100 and 50 ppm respectively which are specified for atomised superalloy powder. Each heat of powder is subjected not only to routine gas analysis as indicated above but also to analysis for basic composition including tramp element content. (Table 2). An additional feature of the quality assurance procedure is to monitor routinely the powder in the context of contaminant particles by examining small representative samples of each heat and blend.

2. POWDER HANDLING AND CONSOLIDATION

Once the powder has been produced and passed quality control checks, it is sieved and, depending on the quantity required, a number of heats are blended together. Blending is not used to correct the analysis of the material. Up to this stage all powder handling has been entirely in an argon atmosphere which must be removed prior to canning. Degassing is performed in an evacuated inclined tube furnace operating in the range 150-500°C through which the powder is vibratory fed, essentially as a mono-layer, thus ensuring that all adsorbed gas is removed. The powder, on leaving the furnace, is fed directly into evacuated metal cans which, when full, are crimped off ready for compaction.

The most suitable consolidation technique for material destined for turbine discs, is hot isostatic pressing (HIP) since large diameter or complex shaped material can be produced. The HIP unit is installed, along with preheating furnaces, Fig. 4, adjacent to the atomiser and powder handling equipment in a purpose built fully integrated plant. The HIP is capable of operating at 1250°C and 1000 atmospheres of argon although slightly higher temperatures can be achieved with reduced pressures. A hot load/unload press with an envisaged total cycle time of 8 hours was specified so that throughput could be maximised and it is also for this purpose that three preheat furnaces are installed along with all the necessary manipulating equipment. The dimensions of the working zone in the press are 19-in diameter by 65-in long.

An aspect that has received considerable study is that of the integrity of HIPed compacts, which although in a few instances can look successfully consolidated may not indeed be so. It has been found that argon determinations on as-HIPed material are a relatively simple, quick and cheap way of determining whether a compact has been fully consolidated. Knowing the argon level in the powder to be 1-2 ppm, similar figures should be

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obtained on consolidated material. It has been found occasionally that compacts with up to 30 ppm argon can be obtained which to all intents and purposes appear satisfactorily consolidated. The presence of higher than normal argon levels gives rise to a fall-off in forgeability and porosity after heat treatment. Argon determinations are now considered to be an essential part of material quality control on all HIPed components, whether for billet, preforms or close to size components.

DISC PRODUCTION ROUTES VIA P/M

The P/M route offers a number of possible alternatives for disc manufacture (Table 3) depending on whether the requirement is for the ultimate in properties or cost saving or indeed a measure of both.

At the present stage of alloy development it is true to say that for the ultimate in properties to be achieved for disc applications, current alloys of the types exemplified by IN 100, RENE* 95 and low carbon ASTROLOY* need a thermo-mechanical forging step in their production cycle. The requirement establishes one extreme for P/M i.e. the route where the powder approach simply provides a workable billet in a more advanced alloy suitable for conventional or isothermal forging. From the economic point of view this route may be more costly than the cast/wrought route for a less advanced alloy since billet production costs can be greater. However some of this cost may be recouped due to greater overall yields from a more homogeneous starting billet.

The other extreme is to consolidate powder to the final disc shape. A number of problems still need to be solved, one of which is the non-destructive testing of a complex shape. This is normally performed by ultrasonic testing of rectilinear machined components which are then machined to the finished shape. At present, therefore, as-HIPed discs are being produced to the rectilinear outline but even this represents a potential cost saving. The mechanical properties achieved on as-HIPed and heat treated material are very encouraging being above those of conventionally produced current disc alloys, but at present they do not attain the levels available on forged powder materials.

In between these two extremes is the possibility of HIPing shaped forging preforms, so offering some of the material cost saving along with forging to give the optimum properties.

A number of these approaches are at present being pursued with work concentrating on one alloy, a low carbon version of a nickel-chromium-cobalt-molybdenum-titanium-aluminium superalloy known internally as APK 1. The following is a summary of this work at the present time:

1. HIP AND CONVENTIONAL FORGE

The major aim of this route was to utilise present day UK forging technology to produce an alloy with a respectable uplift in mechanical properties over current disc alloys. Initial problems with prior particle boundary phases were solved by modifying the carbon content of the alloy and at the same time encouraging carbides to precipitate intragranularly rather than at prior particle boundaries by attention to HIP cycle parameters, i.e. forming $M_{23}C_6$ type carbides in preference to MC types.

The majority of this work has been performed on 8-in diameter discs Fig. 5., although an exercise is at present underway producing larger discs of approximately 18-in diameter. Billets were HIPed in mild steel cans and forged in-house by a two stage, press and screw press route, strain rates of the order of 0.1 sec^{-1} and 6 sec^{-1} being used. During all operations cladding in mild steel or nickel-base alloy was found to be necessary to obtain optimum forgeability.

Forging temperatures and reductions were investigated and ultimately chosen such that a partially warm-worked "necklace" structure was obtained, Fig. 6, which was found to be necessary in order to maximise mechanical properties, particularly low cycle fatigue. Typical properties obtained are shown in Table 4. The material is notch strengthened in both tensile and creep-rupture with high levels of ductility. However, the low cycle fatigue results are the most outstanding property since it has been found that fully recrystallised APK1 tested under similar conditions fails after a maximum of 9,000 cycles.

* TRADE MARK

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2. HIP AND ISOTHERMALLY FORGE

Although there is a paper in this conference specifically on the subject of isothermal forging of superalloys it is worthwhile outlining some of the work being performed in the author's Company. To date only small-scale forgings have been produced with the work concentrating again on APK 1. Material in the as-HIP condition has been forged and although in this condition it is not superplastic the trials performed up to the present using a modified 1000 tonne hydraulic press have confirmed the following advantages:-

a) At the lower forging speeds the flow stress of the material is reduced. It has been found that the forging load on APK 1 rises by 400% when going from a strain rate of 0.018 min^{-1} to a conventional press forge at 0.25 sec^{-1}.

b) Greater control and uniformity of warm-worked structures can be obtained by being able to select the exact forging temperatures and strain rates required. Mechanical properties slightly in excess of those obtained by conventional forging have been achieved on 4-in diameter forgings in APK 1. The attainment of low cycle fatigue properties in particular would appear to be easier.

c) Shorter processing routes for discs are envisaged since a one step forging operation from billet to finished forged component can be conceived. This would also maximise the utilisation of the HIP unit since full loads of cylindrical billet can be used.

d) Although not fully investigated it is known that there is the probability of being able to forge closer to sonic shape and the International Nickel Co. Inc. developed technology of 'thermoplasticity' will be of value in this context. This latter aspect is discussed in more detail later in the paper.

e) Nickel-base die materials are being assessed since they are readily available in-house and are considerably cheaper than molybdenum base alloy dies.

3. HIP A PREFORM AND FORGE

The cost saving potential of this route is readily apparent in terms of reducing the number of forging operations. Savings have been placed in the range of 10% to 40% over the HIP billet plus forge route [1,2] which is clearly a desirable feature and particularly so when viewed from the ultimate customers point of view. In terms of manufacturing preform shapes, the cost of making a preform will clearly be in excess of that for billet manufacture since individual shaped cans have to be made, tested, filled, HIPed and individually assessed to ascertain their integrity. Additionally, the design of preform shapes will be a combination of the forgers needs and those of the metallurgist to ensure uniformity of structure and properties throughout the component. The technology exists for producing shaped cans and activity has recently commenced at the author's Company to examine the preform/forge route in detail.

4. HIP TO SHAPE

The requirement for non-destructively testing an as-HIP component is at present limiting our current activity to the sonic outline but even so potential cost savings in the range 50-65% over the forged billet route have been estimated. [1,2]

Perhaps the most critical aspect of this approach is the technique used to manufacture the shaped powder container. Kelsey-Hayes have developed their "glass-bag" technology [3] and Crucible Inc. a "ceramic container" [4] but other workers in the field are concentrating on metal cans. Within the Company our effort is being concentrated on two approaches:-

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a) For moderately complex sonic outlines metal spinnings in ferritic or austenitic steel sheet are produced which are then welded together and filled with powder. The production of spinnings is a relatively simple technique and quite cheap starting materials are utilised. A typical spun can and resultant sonic shape disc are shown in Figs. 7 and 8.

b) For more complex shapes a "superplastic bagging" technique developed by International Nickel Inc. is used. Two sheets of superplastic alloy are welded together and blown into moulds of the required shape. The degree of complexity achievable with this technique has yet to be established but initial trials are promising.

In both cases after HIPing the can is removed by machining or pickling followed by a light machining skim to the sonic outline. Mechanical property data has been established for as-HIPed and heat-treated APK 1 and some of this information is shown in Table 5. The attractiveness of these properties is evident, with adequate ductility being obtained and the material being notch strengthened in tensile and creep rupture. By varying the HIP cycle and heat-treatment parameters slight variations in the balance of these properties can be achieved. Although the strength levels developed are not as high as those obtained on the alloy in the warm forged condition they are higher than those achieved on many present day conventionally produced disc alloys, e.g. WASPALOY*, NIMONIC* alloy 901. Fig. 9 shows comparative 650°C tensile properties for APK1 and some of these conventional alloys. This data highlights the fact that if the cost savings envisaged can be realised, as-HIPed components in APK1 may be more cost effective than forgings in these conventional disc alloys.

NEW DEVELOPMENTS

All the above techniques are now becoming well established in terms of development activity and discs made by a number of these routes by various organisations are at various stages of assessment or indeed, in some cases, in production usage. In future there appears to be one additional technique which can be made to any of these processing routes to give added beneficial effects. This new technique is termed Thermoplastic Processing [5] (T/P) the basis of which is to heavily cold-work the atomised powder, by attrition or rolling, prior to compaction such that on heating to compaction temperatures fine recrystallised grain sizes of the order of 1 μm are produced. This results in a considerable reduction in the flow stress of the material at hot working temperatures.

For disc production using T/P powder in place of atomised powder the following advantages can be seen:-

a) A superplastic as-HIPed billet can be produced to a size limited only by the HIP unit size capability. In the equation:-

$\sigma = K\dot{\varepsilon}^m$ where σ = flow stress
K = constant
$\dot{\varepsilon}$ = strain rate
m = strain rate sensitivity factor

it is generally accepted that values for 'm' greater than 0.3 denote superplastic behaviour. Values determined by tensile testing T/P APK1 powder in the as-HIPed condition are in the range 0.45 to 0.7.

Thus, it is anticipated that the number of forgings stages from billet can be considerably reduced and closer to size forgings should be obtainable.

b) Larger powder sizes than is the current practice can be utilised since the resulting grain size in the powder is very small. The economics of powder atomisation are thus improved.

c) Since the powder has greatly reduced flow stresses, lower HIP temperatures and pressures can be used. This will allow even more scope for varying the structure and properties of as-HIPed material and improve HIPing economics.

d) Conventional forging of T/P APK1 has been performed with a 30-40% reduction in forging loads over ordinary atomised powder.

* TRADE MARK

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e) Even though larger powder size fractions are used the tap density remains above 60% of the theoretical density.

T/P processing is at present being investigated on a production scale at Henry Wiggin and Company with 8-in diameter discs being forged and shaped components being directly HIPed. At present it is difficult to determine just what will be the level of cost saving offered by including T/P processing in the disc production sequence, but the benefits are thought to be considerable.

CONCLUSIONS

The P/M approach for manufacturing superalloy discs will no doubt, in the next few years, become an accepted production route. Variations in the route allow for a choice between maximum properties and maximum cost savings. It is therefore apparent that there could be the situation where one alloy could be used in a wide range of engine designs the properties and cost being controlled by the details of the production process used.

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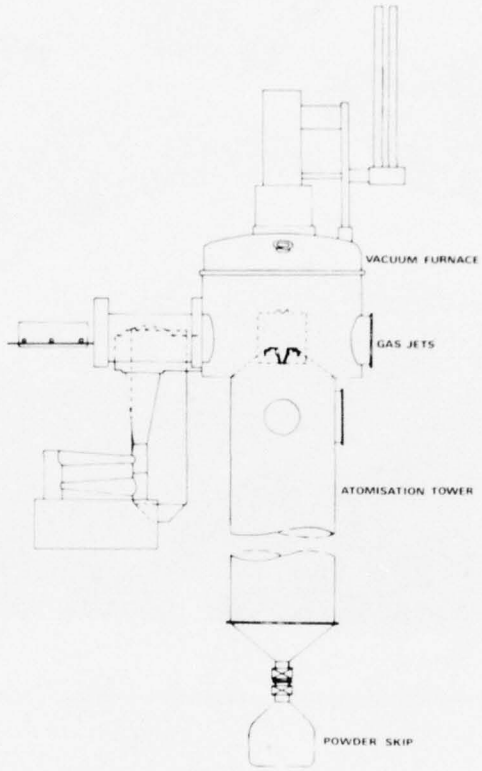

FIG. 1 SCHEMATIC DIAGRAM OF THE ARGON ATOMISER

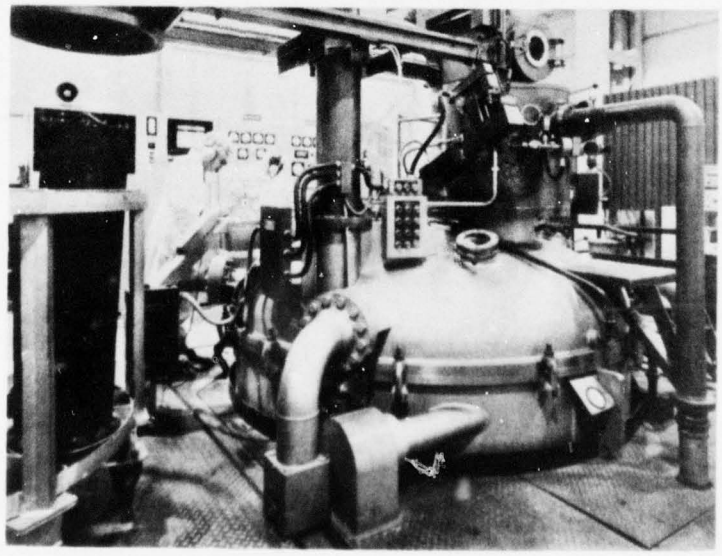

FIG. 2 MELTING CHAMBER OF ATOMISER

FIG. 3 SCANNING ELECTRON MICROGRAPH OF ATOMISED POWDER

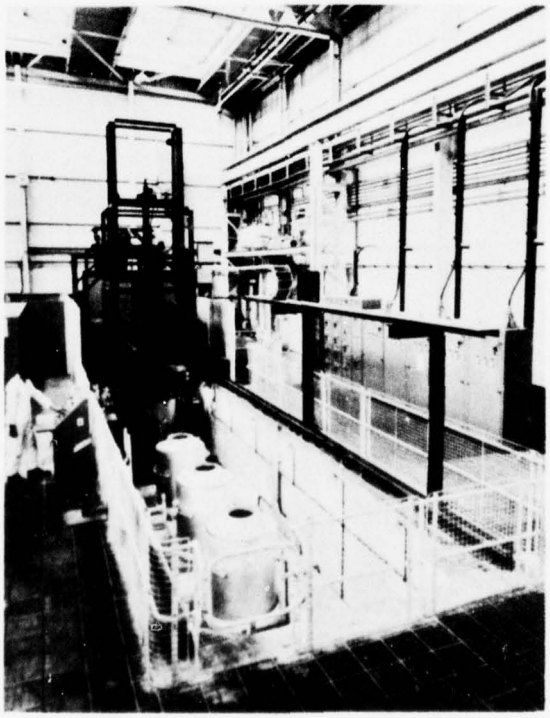

FIG. 4 HOT ISOSTATIC PRESS AND PREHEAT FURNACES

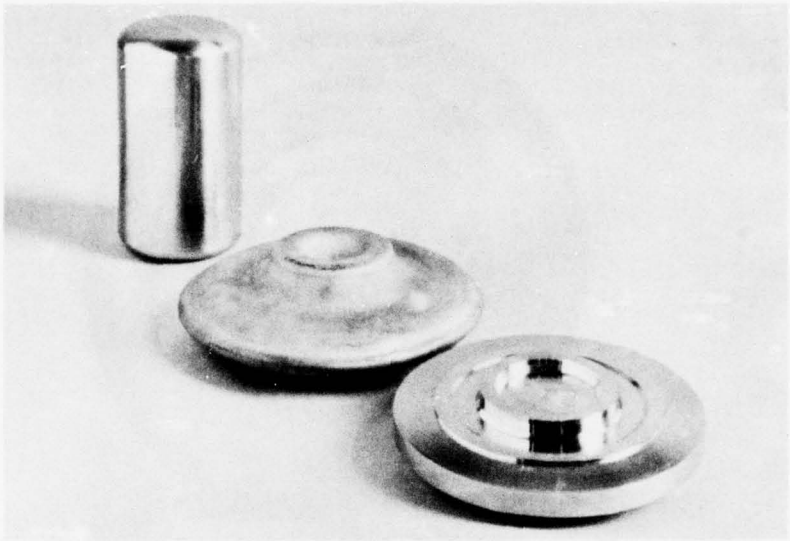

FIG. 5 HIPed BILLET, FORGED OUTLINE AND SONIC MACHINED 8-in DISC IN APK 1

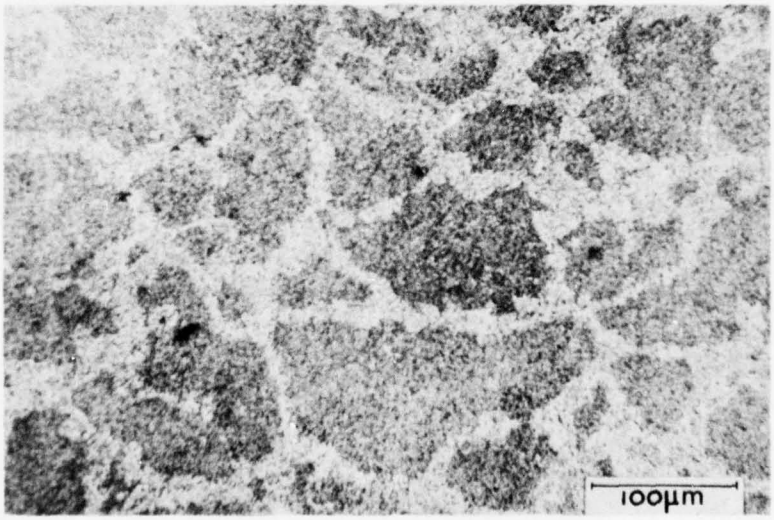

FIG. 6 NECKLACE STRUCTURE OF FORGED AND HEAT TREATED APK 1

FIG. 7 A SPUN METAL CAN FOR A 15⅛" DIAMETER DESIGN DISC

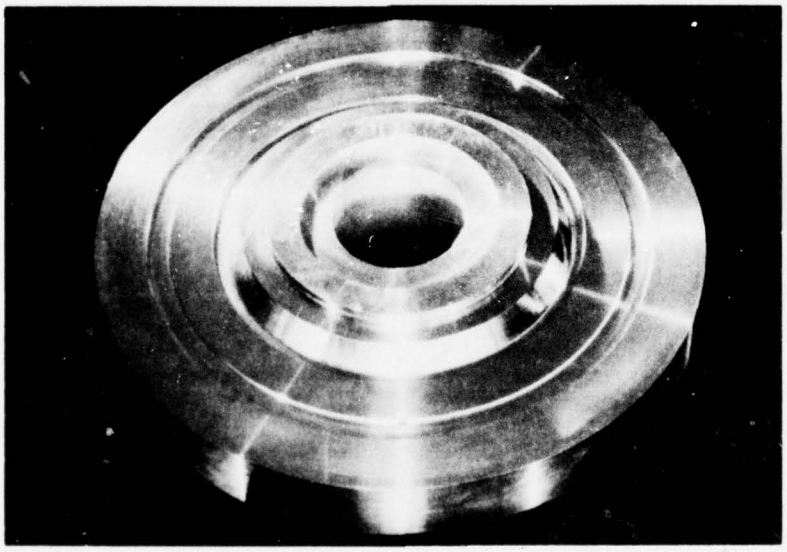

FIG. 8 SONIC DISC IN APK 1 PRODUCED FROM METAL CAN SHOWN IN FIG. 7

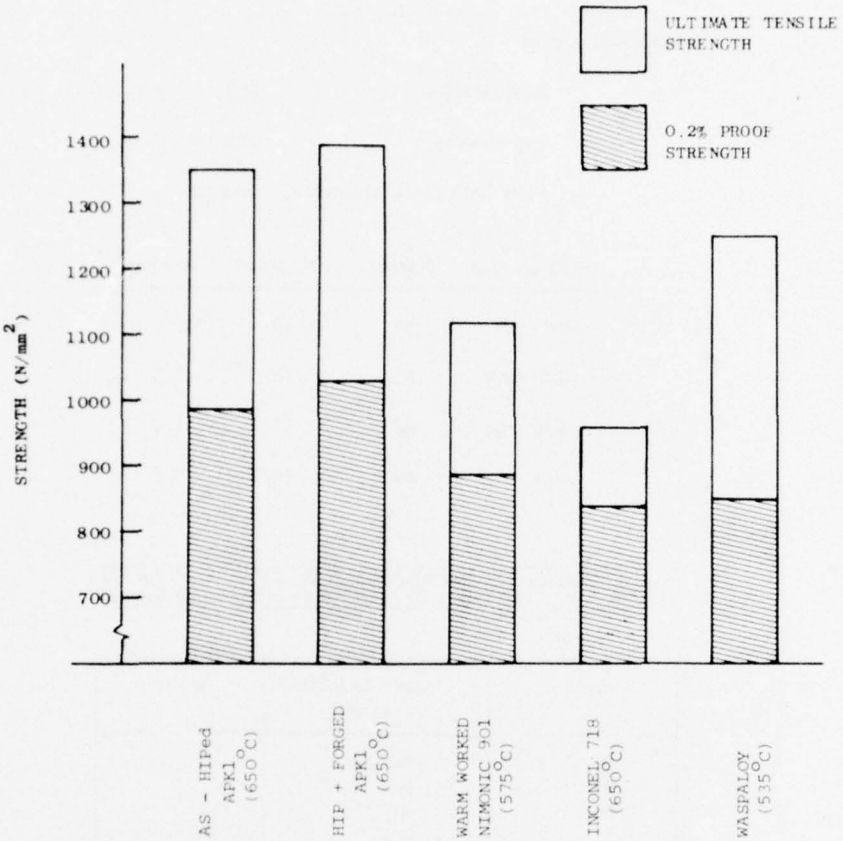

FIG. 9. COMPARISON OF APK1 TENSILE PROPERTIES WITH THOSE ACHIEVABLE ON PRESENT DAY CAST/WROUGHT DISC ALLOYS (TEST TEMPERATURE SHOWN IN BRACKETS).

TABLE 1.

PHYSICAL PROPERTIES AND GAS CONTENTS OF ARGON ATOMISED APK1

-100 mesh powder

Bulk Density	50%
Tap Density	64%
Flow Rate (Hall flowmeter)	16 secs.

BS Mesh Size	O_2(ppm)	N_2(ppm)	A(ppm)
-36 +100	25	17	2.6
-100 +200	32	16	1.5
-200 +300	42	17	1.0
-300	68	16	0.6

TABLE 2.

ANALYTICAL CAPABILITY FOR TRAMP ELEMENTS IN APK 1 BY THE HOLLOW CATHODE[6] TECHNIQUE

Element	Lower Reporting Limit	Accuracy
Pb	0.1 ppm	All elements to an accuracy of ± 5 to 15% of the content
Bi	0.1 "	
Sn	2.0 "	
Sb	0.5 "	
Tl	0.2 "	
Ag	0.1 "	
As	10.0 "	
Te	1.0 "	
Cd	0.1 "	
Zn	0.5 "	

TABLE 3.

POSSIBLE DISC PRODUCTION ROUTES UTILISING POWDER SUPERALLOYS

(A)	(B)	(C)	(D)	(E)
HIPBillet	HIP Billet	HIP Preform	HIP to sonic	HIP to final shape
↓	↓	↓	↓	↓
Multistage Forging	Isothermally Forge	Single Stage Forge		
↓	↓	↓		
Machine to Sonic	Machine to Sonic	Machine to Sonic	Light Machine	
↓	↓	↓	↓	
UST	UST	UST	UST	
↓	↓	↓	↓	
Machine to Final Shape	Machine to Final Shape	Machine to Final Shape	Machine to Final Shape	Light Machine

TABLE 4.

TYPICAL MECHANICAL PROPERTIES ACHIEVED ON A HEAT-TREATED 8 INCH DISC OF APK1 FORGED ON CONVENTIONAL EQUIPMENT TO GIVE A NECKLACE STRUCTURE

Property	Position			
	Radial Mid-Radius		Tangential Periphery	
650°C tensile 0.2% PS, N/mm²	1019	1053	1045	1045
TS, N/mm²	1362	1385	1403	1395
El, %	22.8	26.0	27.1	25.0
R of A, %	27.9	26.0	26.0	28.3
NTS, N/mm²	1809	1859	1811	1824
N/P Ratio	1.31-1.36		1.29-1.31	
20°C tensile 0.2 PS, N/mm²	1155			
TS, N/mm²	1538			
El, %	26.0			
R of A, %	35.9			
NTS N/mm²	2013			
N/P Ratio	1.31			
Creep Rupture Plain Life, h	85	71	90	89
760N/mm², 705°C El, %	16.8	11.0	12.3	12.6
Notch Life, h	269	276	397	417
TPS in 100h at 770N/mm², 650°C %			0.10	
500N/mm², 700°C %			0.06	
LCF at 1080N/mm², 600°C cycles	148000d		105000d	
	123000d		145000d	

d - discontinued

Heat treatment
4h 1080°C OQ + 24h 650°C AC + 8h 760°C

TABLE 5.

TYPICAL PROPERTIES OF AS-HIP PLUS HEAT TREATED APK1

650°C tensile 0.2% PS, N/mm^2	970	975
TS, N/mm^2	1338	1348
El, %	25.0	19.6
R of A, %	23.8	17.4
NTS, N/mm^2	1717	1722
N/P Ratio	1.27 -	1.29
20°C tensile 0.2 PS, N/mm^2	1036	1042
TS, N/mm^2	1412	1388
El, %	20.7	17.4
R of A, %	24.3	19.8
NTS N/mm^2	1871	1858
N/P Ratio	1.32 -	1.35
Creep Rupture Plain Life, h	124	126
760N/mm^2, 705°C El, %	6.0	5.6
Notch Life, h	439	216
LCF at 1080N/mm^2, 600°C, cycles	3731	8330

MANUFACTURE OF LOW COST P/M ASTROLOY TURBINE DISKS

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The performance requirements of advanced military gas turbine engines have fostered the development of many high strength alloys for high temperature disks. Typical of these high strength, high temperature capability disk alloys is Astroloy.

The use of powder metallurgy to produce components from difficult to forge alloys has been demonstrated. However, as the costs of raw materials, labor and processing increase rapidly, the stimulus for continued powder processing development shifts from performance to that of raw material conservation and cost reduction.

It was the object of this program to demonstrate the reproducibility of the product obtained from the forging of annular preforms using a carbon modified Astroloy powder and to establish production processes and specifications relevant to this product.

The technical approach taken was to procure hot isostatically pressed low carbon Astroloy forging preforms from two powder sources. One source utilized high pressure consolidation, the other low pressure consolidation. These as-HIP'ed preforms were hammer forged at Ladish Company. Subsequent mechanical property evaluation verified the quality of these components and a disk for engine qualification was made available for testing.

Three as-HIP'ed annular contour preforms were purchased from Kelsey Hayes Company, Brighton, Michigan, for evaluation as a source for powder metal preforms consolidated under low pressure (750 psi) and high temperature (~2250F). The preforms were fabricated as shown schematically in Figure 1.

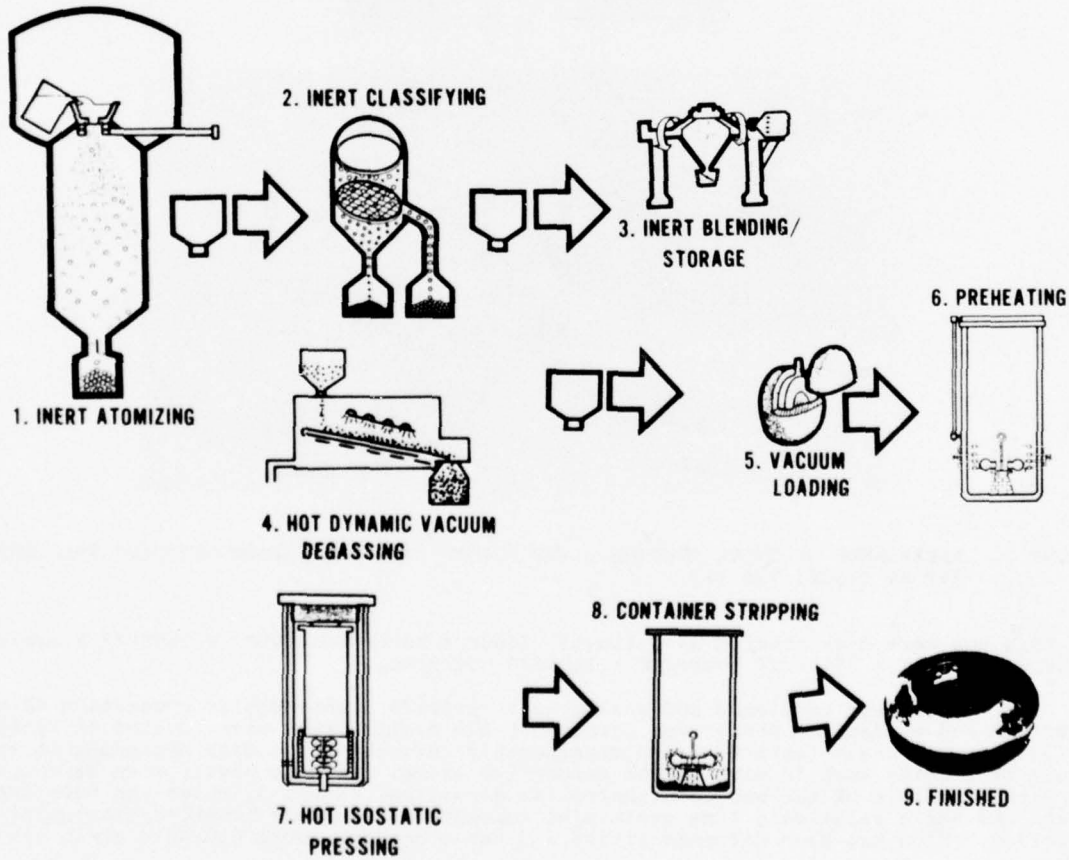

FIGURE 1. SCHEMATIC OF PROCESS USED TO PRODUCE HIP PREFORMS AT KELSEY HAYES.

Vacuum induction melted ingot of the desired chemistry was obtained from Special Metals Inc., then vacuum melted and argon atomized into powder by Kelsey Hayes. The all inert

handled -80 mesh powder was hot dynamically vacuum outgassed by vibrating the loose powder down an inclined plane in vacuum at approximately 900F into pre-evacuated fused silica containers. These filled containers were sealed vacuum tight and preheated at 2200F for approximately three hours, loaded hot into the Kelsey Hayes internally heated autoclave and consolidated at 2250F for two hours at 750 psi.

Impact forging of the preforms was successfully accomplished at Ladish. Intermediate reheats were used between strikes when necessary to maintain the proper forging temperature. Forging to completion (die fill) was not possible in the first series of strikes as the oversize nature of the preforms created excess flash in the rim preventing backfilling the bore. The forgings were trimmed of flash and successfully forged to completion. Appearance of the as-forged disks is shown in Figure 2. An average reduction in thickness of 25% was imparted during the forging.

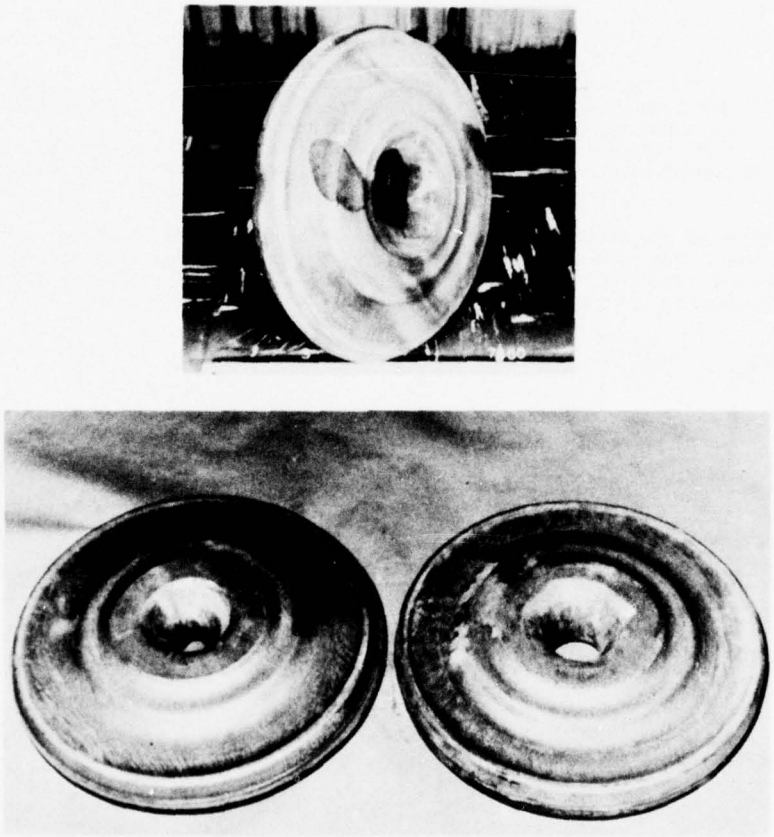

FIGURE 2. APPEARANCE OF THREE TURBINE DISKS FORGED FROM LOW CARBON ASTROLOY PREFORMS HIP AT 2250F, 750 PSI.

All forgings were heat treated as follows: 2060F/4 hours/SQ (600F) + 1600F/8 hours/AC + 1800F/8 hours/AC + 1200F/24 hours/AC + 1400F/8 hours/AC.

The forging was sectioned and evaluated to provide a thorough documentation of microstructure and mechanical properties throughout the disk. Grain size, degree of recrystallization and grain texture varied considerably throughout the disk depending on the extent of forging work in each of the respective areas. This is easily seen by comparing the microstructure of the web location/radial direction, Figure 3, which has been heavily worked and has a relatively fine grain size compared to the bore location/tangential direction, which has been deformed little and has a coarser, more equiaxed grain size.

Mechanical properties of this disk were determined using tensile, stress rupture, creep, low cycle fatigue and fatigue crack growth rate tests.

Tensile testing at room temperature and 1400F was conducted at six representative locations. Figure 4 shows room temperature strength and ductility to be satisfactory. The 1400F strength was also satisfactory, however, the bore ductilities were generally lower than in other test locations. Low bore ductility is attributed to insufficient work penetration during forging.

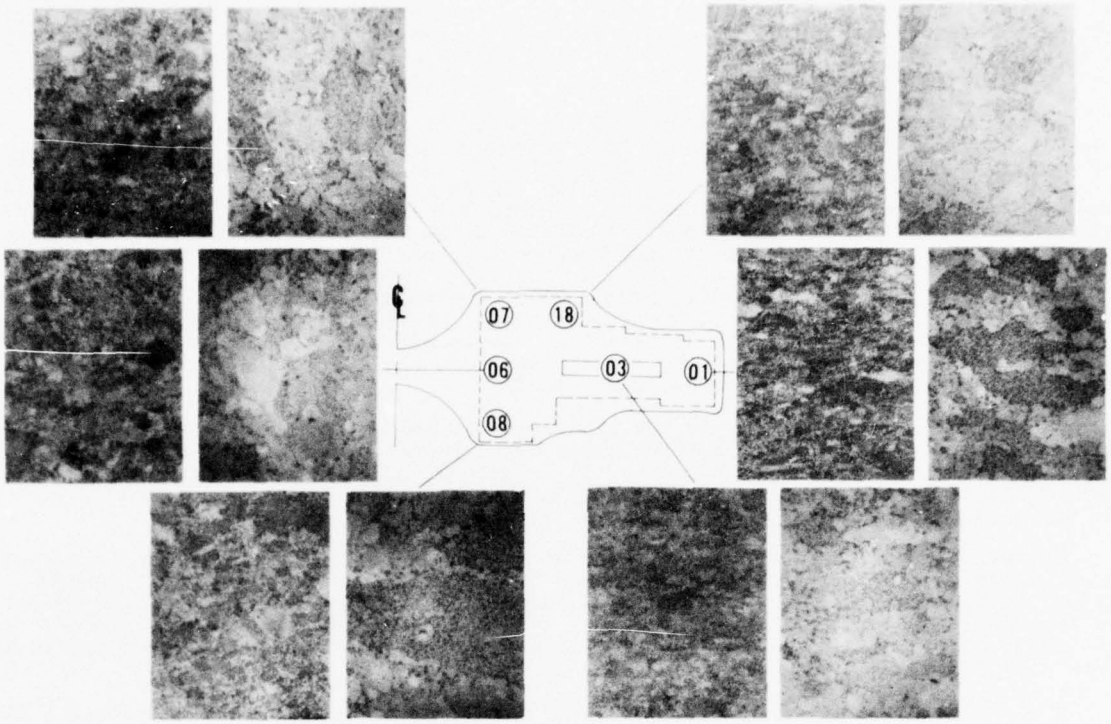

FIGURE 3. MICROSTRUCTURE IN VARIOUS LOCATIONS THROUGHOUT A FULLY HEAT TREATED LOW CARBON ASTROLOY HIP PLUS FORGED TURBINE DISK.

TENSILE

TEMPERATURE	2% YS	UTS	EL	RA
70 °F	150-159 KSI	205-219 KSI	15-22%	19-34%
TARGET	140	195	15	18
1400°F	125-132	150-165	15-23	18-42
TARGET	125	150	15	20

STRESS - RUPTURE

1400°F/85 KSI	19-29 HOURS	12-15% EL
TARGET	15	12

CREEP

1300°F/74 KSI	210-220 HOURS	0.1%
TARGET	110	

FIGURE 4. MECHANICAL PROPERTIES OF HIP PLUS FORGED LOW CARBON ASTROLOY TURBINE DISKS.

Stress rupture testing was performed using combination smooth-notched (Kt = 3.5) specimens taken from each of the above-mentioned six areas. Testing was done at 1400F at a stress of 85,000 psi. Again, ductilities in the bore area were lower than more heavily worked regions, with adequate lives in all areas.

Creep testing at 1300F/74 ksi proved the material to be adequate, with time to 0.1% elongation exceeding 200 hours in all specimens tested. A 40 specimen low cycle fatigue program was conducted at frequency of 60 cps to provide a basis for comparing the fatigue lives of powder processed low carbon Astroloy to conventional cast plus wrought Astroloy. Stress controlled testing was performed on smooth and notched (Kt = 2, 3) specimens at 1175F at various stress levels. One set of Kt = 3 specimens was tested at 1000F, with the life under all conditions exceeding component requirements.

A second disk forged by Ladish from a Kelsey Hayes HIP preform was machined to the sonic configuration and inspected by production NDI procedures to conventional Astroloy requirements and was shown to be acceptable. Subsequently, the disk was finish machined prior to experimental engine testing. The disk began engine testing in a static experimental engine test initiating in December, 1975.

To evaluate an alternative to low pressure HIP preform consolidation, Pratt & Whitney Aircraft ordered from Ladish Company turbine disks forged from preforms HIP'ed at 15,000 psi. One set of three forging preforms was ordered by Ladish and produced in much the same way as those produced by Kelsey Hayes, with the primary exception of the use of 15,000 psi HIP pressure instead of the 750 psi HIP used by Kelsey Hayes.

Subsequent to argon atomization, much of the handling of the powder was performed in air. The removal of water vapor adsorbed during air handling was accomplished by baking the powder in air at 250F for approximately one hour. After drying, the powder was transferred (hot) in air into a chamber which was then evacuated to 1-5 microns vacuum. The powder was poured in vacuum at room temperature into pre-evacuated austenitic stainless steel containers.

The preform container was removed by machining and the preform inspected using fluorescent penetrant. No flaws were found. A macroetch of the surface did reveal striations caused by segregated layers of coarse and fine powder particles.

Microstructures of powder fill tube locations were documented using optical microscopy and were found to possess heavy prior particle boundary precipitates. Material in two of these evacuation tubes was analyzed for oxygen and nitrogen and found to contain more than 200 ppm of oxygen. This heavy concentration of oxygen of the prior particle boundaries indicate improper handling and/or insufficient outgassing of the powder.

Since one forging preform had an acceptable structural appearance in both the fill tube and on the surface replica, it was decided that this preform had the best chance of yielding a low oxygen, forgeable structure. A 1" diameter pin was removed from the mid-radius, macroetched and analyzed for oxygen and nitrogen at the top, middle and bottom of the preform. Striations were shown to exist throughout the preform and were not just associated with the surface. An oxygen gradient of approximately 70 ppm also existed from the outside to the center of the compact (52-77 ppm in the center and 121-145 ppm oxygen within 3/16" of the surface).

Apparently the combination of air handling and a subsequent lack of adequate hot dynamic outgassing permitted the retention of high quantities of oxygen on the surfaces of the powder encapsulated in the preform. A large portion of this oxygen diffused toward the outer walls of these preforms in the initial stages of HIP as the outer portions heated first gettering the available oxygen.

A second set of high pressure HIP preforms was ordered by Ladish from another powder supplier. These forging preforms were made using argon atomized low carbon Astroloy powder, canned in stainless steel containers and HIP'ed at 15,000 psi.

Austenitic stainless steel containers were loaded with powder in air, evacuated with a mechanical roughing pump to a low vacuum and hot bulk outgassed at a temperature of 1500F for approximately 48 hours. The containers were sealed and shipped to Battelle Memorial Institute where they were HIP'ed at 2050F for three hours at 15,000 psi in an internally heated autoclave.

Oxygen analysis was performed on material taken from the fill tubes of all three preforms to determine if outgassing procedures were adequate. These results showed two preforms to have over 200 ppm of oxygen with the third preform having approximately 100 ppm of oxygen.

In order to verify these levels of oxygen in the bulk of consolidations of the high oxygen preforms, a one-half inch thick ring was trepanned from the top of these preforms. Oxygen analysis was then made on four samples taken from the interior of this ring 90° apart. This verified the high oxygen contents found in the fill tube with between 100 and 216 ppm oxygen. The microstructure of these rings were examined, and found to still exhibit heavily outlined prior particle boundaries.

Forging was then attempted on the lowest oxygen preform using a preheat above 2200F, standard impact forging technique and Ladish die shape; i.e., a repeat of the previously successful procedure. After only two blows, in which less than 10% deformation was

achieved, a radial crack occurred in the outer diameter of the billet. The cracked area was removed intact and examined macroscopically and microscopically in an attempt to identify the cause for cracking.

No identifiable foreign material could be found. There was also no indication of heavy prior particle boundary precipitates. Oxygen analyses on specimens taken 1/8" below the surface of the crack face indicated levels between 120 and 190 ppm. To determine whether these values were typical of the entire preform, a 1/2" diameter axial core was removed from the thickest portion of the disk, with oxygen content measured at several locations. Analysis of this core showed the oxygen levels to be acceptable with a gradient of 47 ppm in the center to 93 ppm at the bottom of the core. Therefore, in view of the acceptable oxygen levels in the test core, it was decided to continue the forging. The test core hole was fitted with a steel plug and the forging attempted again. After five forging passes, the forging was halted as numerous cracks had occurred in the outer edge of the disk.

Prior to the selection of the third source for high pressure HIP'ed preforms, a review of the program with P&WA, AFML and Ladish personnel resulted in the following conclusions.

1. An improvement in powder handling and outgassing procedures must be obtained to insure a low level of oxygen and an acceptable prior particle boundary structure. It was, therefore, decided to have the powder handled completely in an inert atmosphere and to be hot dynamically outgassed prior to vacuum loading into pre-evacuated containers.

2. It was also apparent that subscale companion consolidations are not representative of much larger consolidations when air handling of powder and marginal outgassing is performed; i.e., it is much easier to effectively bulk outgas small compacts than large, 250 pound containers. Therefore, a bulk sample must be included in the preform shape to permit evaluation of the as-HIP'ed material prior to forging.

3. Finally, it was concluded that the effects of high HIP temperature (~ 2250F) were probably beneficial in improving the forgeability of low carbon Astroloy, therefore, a higher HIP temperature was specified. Federal Mogul, now the Udimet[R] Powder Division of Special Metals, was selected to supply the replacement preforms.

The powder used in this phase of the program was handled completely under inert atmosphere. Outgassing of the powder was accomplished by exposing the loose powder to a dynamic vacuum during the loading of the pre-evacuated mild steel containers. After filling, these containers were heated to 500F and further outgassed. The containers were crimped vacuum tight and shipped to Kawecki Berylco Industries, Inc. (KBI), Hazelton, Pennsylvania, where they were hot isostatic pressed at 2215F (the highest autoclave temperature permitted at KBI), 15,000 psi for three hours.

Oxygen and nitrogen analyses were taken on material from test rings located at the bore of the HIP preforms. Analyses were made in three different locations to determine the degree of outgassing. This data show the oxygen level to be at or below 70 ppm oxygen and 30 ppm nitrogen in all tested areas.

The Federal Mogul preforms were forged using conventional impact forging techniques. A preheat temperature of over 2000F was used during the forging. Both disks were successfully forged to completion; however, the cycle was stopped to permit blending of several small edge cracks that occurred in the final stages of forging. The forged disks are shown in Figure 5.

One disk was sectioned into quarters to permit heat treat studies to be performed in order to optimize the balance of tensile, creep and stress rupture properties. The treatment selected was 2040F/4 hours/RAC + 1600F/8 hours/AC + 1800F/8 hours/AC + 1200F/24 hours/AC + 1400F/8 hours/AC. The second disk was heat treated by Ladish, sectioned with approximately 40% being retained by Ladish for tensile, stress rupture and creep testing. The remainder was shipped to P&WA for low cycle fatigue and low cycle fatigue crack growth rate testing.

Tensile test data generated by Ladish indicated marginal properties, particularly at elevated temperature. A review of the microstructure revealed that an unacceptably high temperature, near or slightly above the gamma-prime solvus, had accidentally been reached. The result was to relieve much of the forged-in strength.

In order to determine if an improvement in tensile and stress rupture properties could be obtained by developing a duplex microstructure of coarse unrecrystallized grains surrounded by fine recrystallized grains, the remaining as-forged portion of the first disk was heat treated at 2030F for four hours and given a rapid air cool. The four-step carbide stabilization and age followed. The results of the testing showed improvements in 1400F yield strengths and ductilities, as well as increased stress rupture ductility and notched-specimens life. Room temperature strengths were 136-143 ksi yield, 199-209 ksi ultimate, 19-20% elongation and 24-37% R.A. At 1400F, strengths were 120-126 ksi yield, 148-158 ksi ultimate, 15-29% elongation and 18-41% R.A.

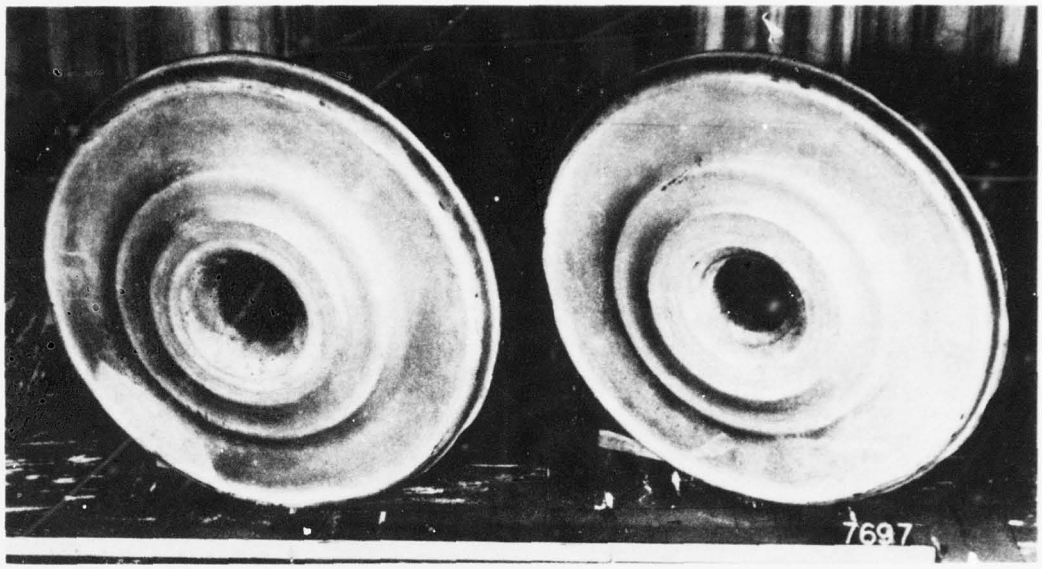

FIGURE 5. APPEARANCE OF TWO TURBINE DISKS FORGED FROM LOW CARBON ASTROLOY PREFORMS HIP AT 2215F, 15,000 PSI.

In order to determine the effectiveness of the Federal Mogul vacuum outgassing and loading process, material was taken from five locations in the second disk and analyzed for oxygen and nitrogen. Oxygen and nitrogen contents were very low with a gradient of 40 to 70 ppm oxygen detected.

As a result of this work, it was concluded that conventional forging of hot isostatically pressed Astroloy preforms yields acceptable properties for application to turbine disks.

ADVANCEMENTS IN SUPERALLOY POWDER PRODUCTION AND CONSOLIDATION

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SUMMARY

A program was initiated to reduce the cost of fabricating superalloy turbine engine components through the utilization and improvement of powder metallurgical techniques. To date, investigations have been conducted on both powder production and powder consolidation. Specifically, it was demonstrated that the cost of powder production could be significantly reduced at the expense of small property changes through the use of virgin materials and powder revert during melting, minimizing inert handling, and the use of coarser mesh fractions. Relative to consolidation, it was also shown that ceramic molds can be used to produce near net shaped parts by direct HIP or by sinter plus HIP techniques. The verification of these processes is currently in progress through the fabrication and evaluation of a full scale turbine disc with an integral stub shaft.

INTRODUCTION

Initial superalloy powder developments resulted from efforts to improve the quality and homogeniety of wrought billet material. Subsequently, activities were directed at achieving very high strength levels using compositions normally considered suitable only for casting. This has resulted in a unique class of PM turbine disc materials; however, they can generally only be cost effective where high levels of performance are required.

The basic purpose of this program was to reduce the cost of superalloy powder components so as to broaden their base of application and make them cost competitive with a wider range of conventional superalloys. Specifically, the program was to establish improved and more economical processes for both superalloy powder production and powder consolidation. Argon atomization for powder production and two different hot isostatic pressing (HIP) techniques for powder consolidation were selected at the outset as basic processes. Variables that were investigated in this program included melting and screening practices, and consolidation times and temperatures.

The program involved two major subcontractors, the Crucible Materials Research Center (CMRC) and the Udimet Powder Division of Special Metals Corporation (SMC). CMRC used a direct HIP process which employed an outer pressure tight can with an inner ceramic mold, while the SMC process consisted of a pre-sinter cycle in a reusable ceramic mold followed by a HIP cycle. The density obtained during sintering was high enough to produce closed-cell porosity and thereby eliminate the need of a can during HIP. In each case, the program goal was to produce a near net shape and thereby minimize material input.

The superalloy composition used in the program is PA101, a hafnium modified IN792 material. It has demonstrated exceptionally high properties when extruded billet was forged and direct aged; room temperature yield strengths were typically 230 ksi (1585 MPa). Using such a material in the HIP condition followed only by heat treating would obviously result in some loss of strength. However, prior data indicated that this level of strength would be adequate to meet or exceed the properties of other conventional wrought superalloys, such as Inconel 718, Waspaloy, and D979.

PROGRAM PLAN

The overall program was divided into two phases. The objective of the first phase was to investigate various process variables and then define an optimum powder atomization and consolidation process based on property and cost trade-offs. The selected processes were then demonstrated by the fabrication and evaluation of a simulated turbine disc. Phase II involves verifying the selected processing procedure by the fabrication of a full-scale turbine disc and then conducting a complete material characterization. An organization diagram for the major program tasks is shown in Figure 1.

The Phase I billet consolidation trials for each of the major subcontractors were based on

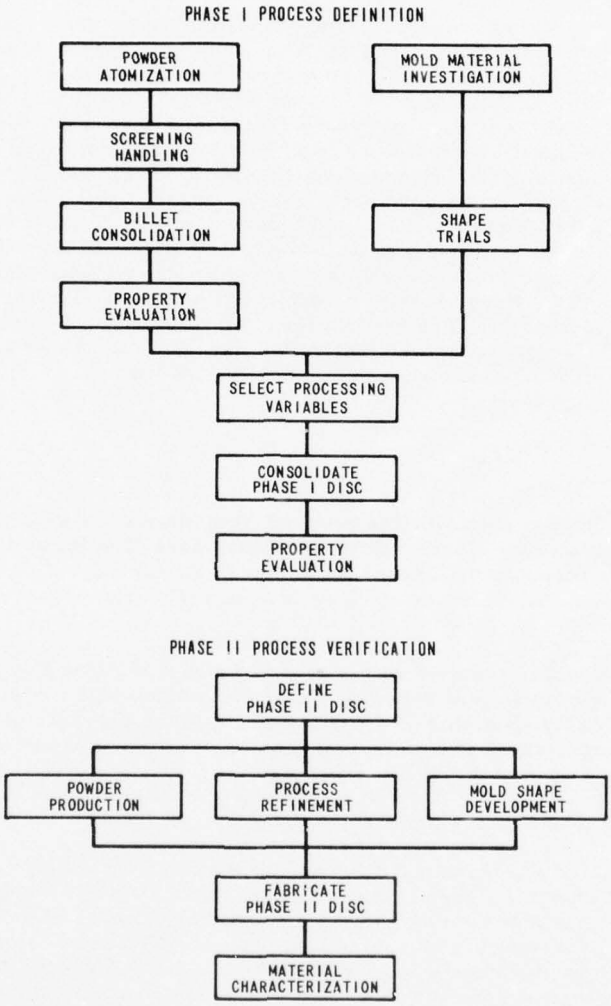

Figure 1. Program Task Organization Diagram

separate partial factorial experiments. Processing variables that were investigated in each case were related to atomization, powder handling, and consolidation parameters as noted in Table I. Evaluation of test material resulting from these experiments included ultrasonic inspection, metallography, gas analysis, room temperature tensile and 1200° F (649° C) stress rupture testing. In addition, ceramic mold material studies were conducted to determine the compatibility of the superalloy powder with the mold material during consolidation. Following these activities, the optimum processing variables and techniques were selected and demonstration trials conducted using the simulated turbine disc shown in Figure 2 as a shape goal.

The second phase of the program was directed at verifying a PM process by the production of full scale turbine discs and then characterizing pertinent material properties. The Phase II disc has a stub shaft, as shown in Figure 3, that is inertia welded to a forged disc when conventionally fabricated. The material input for the conventional D979 disc is 102 lbs. (46.27 Kg), whereas the goal for the PA101 disc was initially set at 35 lbs. (15.88 Kg). Phase I of this program has been completed and fabrication of Phase II discs is currently in progress. Only Phase I results will be discussed in this paper.

TABLE I - PROGRAM VARIABLES

	DIRECT HIP	SINTER + HIP
Melting Practice	VIM Virgin Revert	VIM Virgin + Revert
Powder Particle Size	-28 Mesh (595 μ) -60 Mesh (250 μ) -100 Mesh (149 μ)	-28 Mesh (595 μ) -60 Mesh (250 μ) -325 Mesh (44 μ)
Powder Handling	Air Inert	Air Inert
HIP Temperature	2000° F (1093° C) 2100° F (1149° C) 2175° F (1191° C)	2100° F (1149° C) 2175° F (1191° C) 2250° F (1232° C)
Sintering Time	---	2 Hours
HIP Heating Method	Internal External	Internal

DISCUSSION AND RESULTS

The billets consolidated for the Phase I partial factorial experiments were simple cylinders nominally 4 inches (10.2 cm) diameter by 10 inches (25.4 cm) high. Slices 1 inch (2.5 cm) thick were removed from each end of the billet and heat treated following ultrasonic inspection. The heat treat cycle consisted of solutioning for two hours at 2050° F (1121° C) and double aging for 16 hours each at 1400° F (760° C) and 1250° F (677° C). Room temperature tensile tests and stress rupture tests at 1200° F/160 ksi (648.9° C/1103.2 MPa) were conducted on specimens machined from each slice.

Sound billets were obtained for most of the processing combinations except for two of the sintered and HIP billets which contained some porosity throughout; several others had minor porosity at one end. The incidence or porosity appeared unrelated to processing variables being investigated. Only data from sound material was used in the analysis of the matrix experiments.

Average properties representing the 15 direct HIP billets and 22 sinter plus HIP billets are listed in Table II. Properties for direct HIP material were slightly higher at room temperature but its stress rupture properties were lower compared to the sintered and HIP material. This was attributed to a difference in grain size; the direct HIP material showed a nominal grain size of ASTM No. 7 to 9 (32 to 16 microns) while the sintered and HIP material had a larger grain size that ranged from ASTM No. 4 to 7 (90 to 32 microns). The grain boundaries of the direct HIP material were typically very clean and frequently not distinguishable as shown in Figure 4. The sinter plus HIP material showed more distinct outlining of the grain boundaries by discrete particles as shown in Figure 5. These particles were identified as $M_{23}C_6$ carbides and apparently form as a result of some slight solutioning of the primary MC carbides during the high temperature sinter cycle. Otherwise the structures are similar, with large agglomerated particles of primary gamma prime and small particles of aging gamma prime in a gamma matrix.

The analyses of the partial factorial experiments to determine the relative influence of processing

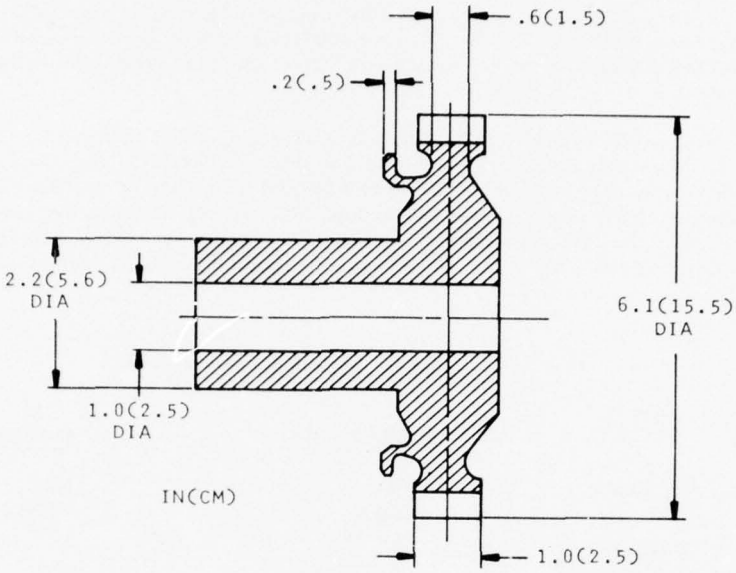

Figure 2. Shape Goal for Phase I Turbine Disc

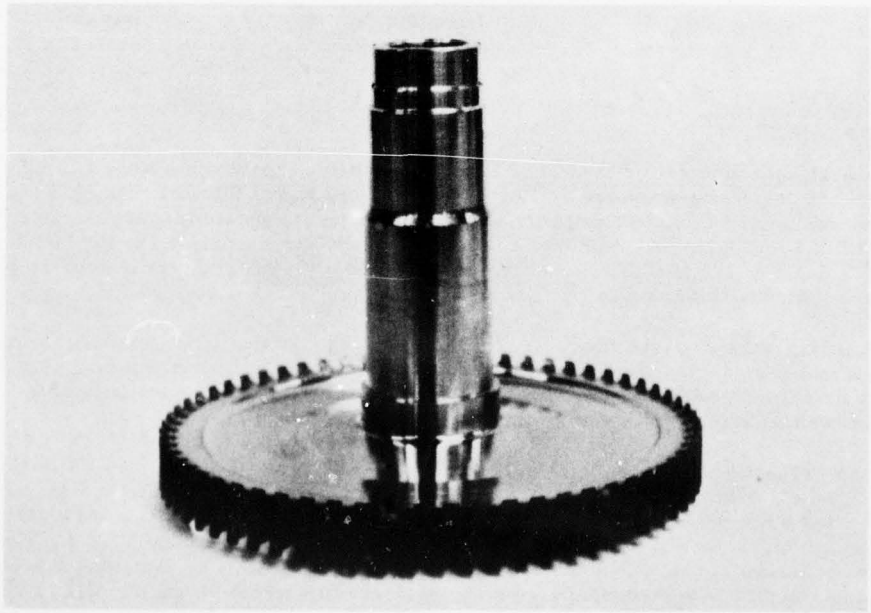

Figure 3. Full Scale Phase II Turbine Disc

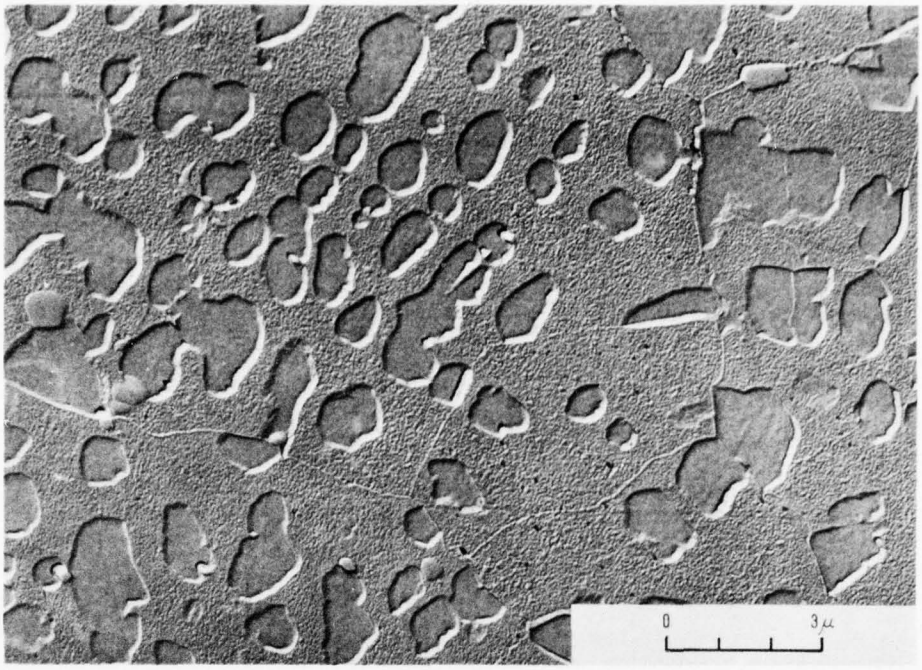

Figure 4. Microstructure of Direct HIP PA101

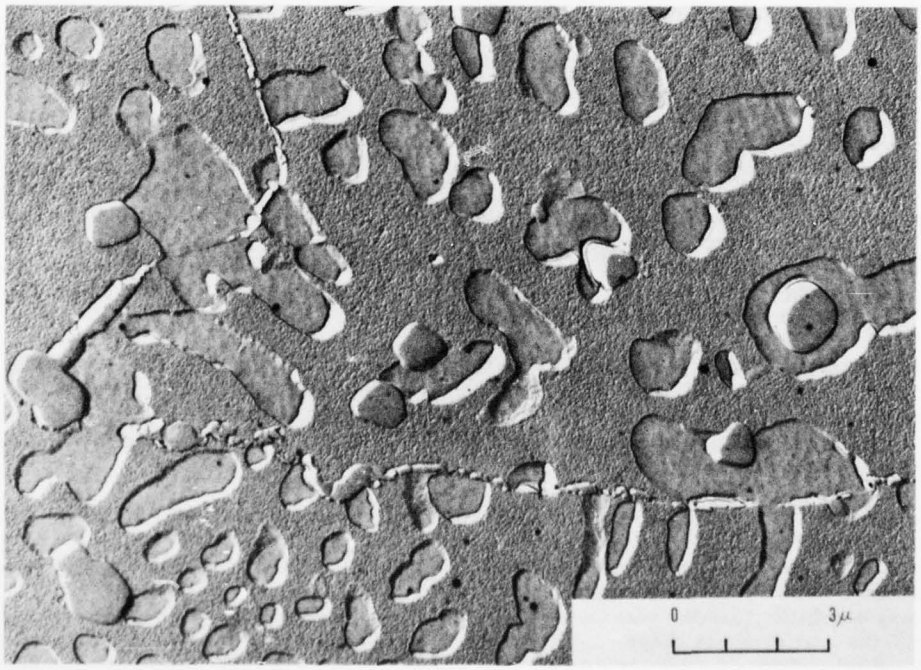

Figure 5. Microstructure of Sinter Plus HIP PA101

TABLE III - ANALYSIS OF VARIABLES RELATED TO POWDER PRODUCTION

A. INFLUENCE OF MELT PRACTICE

		DIRECT HIP (CMRC)			SINTER + HIP (SMC)	
		VIM	VIRGIN	REVERT	VIM	VIRGIN + REVERT
Tensile						
UTS	△%	+0.4	-1.1	+0.7	0.0	0.0
YS	△%	+0.7	-0.7	0.0	+1.2	-0.6
Elong	△%	+2.9	-3.8	0.0	-1.9	+1.9
RA	△%	+11.2	-6.5	-4.7	-4.5	+3.6
Stress Rupture						
Log Life	△%	+5.5	+5.2	-16.4	+4.7	-6.5
Elong	△%	+13.6	-13.6	0.0	+6.5	-6.5
RA	△%	-4.5	-18.2	+25.0	+1.6	-3.1

B. INFLUENCE OF POWDER PARTICLE SIZE

		DIRECT HIP			SINTER + HIP		
		-28 Mesh (595 μ)	-60 Mesh (250 μ)	-100 Mesh (149 μ)	-28 Mesh (595 μ)	-60 Mesh (250 μ)	-325 Mesh (44 μ)
Tensile							
UTS	△%	-2.0	+1.3	+0.8	-1.3	-0.4	+1.8
YS	△%	-0.6	+0.2	+0.4	-0.6	+1.2	0.0
Elong	△%	-6.7	+2.9	+3.8	-3.8	-3.8	+7.0
RA	△%	-6.5	+2.8	+4.8	-3.6	-3.6	+7.3
Stress Rupture							
Log Life	△%	-16.5	-3.9	+14.2	-3.1	+0.5	+1.8
Elong	△%	-13.6	-4.5	+18.2	+3.2	-6.5	+6.5
RA	△%	0.0	-4.5	+4.5	+1.6	-3.1	0.0

C. INFLUENCE OF POWDER HANDLING

		DIRECT HIP		SINTER + HIP	
		Air	Inert	Air	Inert
Tensile					
UTS	△%	+1.2	-1.2	+0.6	-0.9
YS	△%	+0.1	-0.1	+0.6	-1.2
Elong	△%	+4.9	-5.9	+2.0	-2.0
RA	△%	+9.2	-9.2	-3.6	+1.8
Stress Rupture					
Log Life	△%	+1.3	-1.3	+3.4	-3.9
Elong	△%	+8.7	-8.7	0.0	+3.4
RA	△%	0.0	0.0	+1.6	-1.6

TABLE IV - ANALYSIS OF POWDER CONSOLIDATION VARIABLES

A. INFLUENCE OF HIP TEMPERATURE

		DIRECT HIP (CMRC)			SINTER + HIP (SMC)		
Tensile		2000°F (1093°C)	2100°F (1149°C)	2175°F (1191°C)	2100°F (1149°C)	2175°F (1191°C)	2250°F (1232°C)
UTS	△%	-2.1	+2.1	0.0	+0.4	0.0	0.0
YS	△%	+2.9	+0.8	-3.7	-0.6	+0.6	+1.9
Elong	△%	-17.3	+6.7	+9.6	+3.8	-2.8	-0.9
RA	△%	-12.2	+5.6	+6.5	0.0	-0.9	+0.9
Stress Rupture							
Log Life	△%	+3.0	+2.2	-5.1	-1.9	+0.1	+3.2
Elong	△%	-22.7	+18.2	+4.5	0.0	-12.9	+6.5
RA	△%	-2.3	+2.3	+2.3	-4.7	-3.1	+6.3

B. INFLUENCE OF SINTERING TIME (SINTER + HIP ONLY)

Tensile		2 Hours	5 Hours
UTS	△%	+0.4	-0.4
YS	△%	0.0	+0.6
Elong	△%	+5.7	-3.8
RA	△%	+3.6	-3.6
Stress Rupture			
Log Life	△%	-2.5	+0.9
Elong	△%	+6.5	-3.2
RA	△%	+6.4	-1.6

C. INFLUENCE OF HIP HEATING METHOD (DIRECT HIP ONLY)

Tensile		Internal	External
UTS	△%	+1.8	-1.8
YS	△%	+2.6	-2.6
Elong	△%	+5.7	-5.7
RA	△%	+4.6	-4.6
Stress Rupture			
Log Life	△%	+5.9	-5.9
Elong	△%	+8.3	-8.3
RA	△%	-2.2	+2.2

ACKNOWLEDGEMENT

This work has been supported by the United States Air Force Materials Laboratory under Contract AF33-615-73-C-5040. Special acknowledgement is given to L. P. Clark, program monitor at AFML, to J. N. Fleck and F. J. Rizzo at Crucible Materials Research Center, and to S. H. Reichman at Udimet Powder Division for their direct contributions to this effort.

Figure 6. A Consolidated Phase I Turbine Disc

ISO-FORGING OF POWDER METALLURGY SUPERALLOYS FOR ADVANCED TURBINE ENGINE APPLICATIONS

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SUMMARY

The GATORIZING™ forging process is a hot die isothermal technique used to produce complex configurations. This process utilizes the superplastic behavior imparted to advanced superalloys through prior processing and/or controlled forging parameters. This technique is currently being used in the production of all of the turbine disks and many of the compressor disks used in the F100 engine program. The GATORIZING technique has allowed P&WA to produce a diverse array of precision forged net and near net shape superalloy components. Furthermore, because the process uses hot dies and relatively low forming rates, the response of the as-GATORIZED workpiece to subsequent heat treatment is remarkably uniform. Finally the GATORIZING process offers strong economic advantages over conventional forming techniques in applications which use expensive raw materials, require maximum material properties, or require complex component configuration.

I. INTRODUCTION

The F100 engine which powers the Air Force's F-15 Eagle air superiority fighter and the F-16 air combat fighter is, perhaps, the most advanced augmented turbofan jet engine in existance in the world. The design of this engine was predicated on the philosophy of maximum thrust to weight ratio. This design philosophy implied the need for turbine and compressor materials which would exhibit extremely high strength to density ratios. Early in the F100 development program, it became obvious that the most advanced operating turbine disk material at that time, Astroloy, possessed an insufficient strength to weight ratio to satisfy the goals of the program. Consequently, a new breed of high strength, light weight turbine disk alloys was required. P&WA met this challange with the development of powder metallurgy processed IN100, a high gamma prime content superalloy characterized by superior strength and excellent reproducibility of properties in the temperature range RT to 1400°F.

However, the major attribute of this alloy system, namely excellent high temperature strength, became its major detriment when the alloy system proved to be essentially unforgeable by conventional techniques for both economic and metallurgical reasons. First, the price of the powder processed billet was high, and the need to protect critical dimensions by oversize forgings led to terrific waste of the expensive raw materials. Secondly, the effects of die chill with this high strength system often led to gross edge cracking during the forging operation.

P&WA, at the Government Products Division, solved these problems with the invention and development of the GATORIZING™ forging process. As will be shown below, this technique offers the most economical method for forming high strength nickel and titanium based alloys.

II. THE GATORIZING PROCESS - WHAT IS IT?

The details of the GATORIZING process are presented in U.S. Patent No. 3-519-503. However, the process is basically a hot die forming technique. Most of the work to date has been performed as a hot isothermal operation where both forging dies and work piece are heated to the established forging temperature and maintained at that temperature throughout the forming operation.

The basic elements required for the process are shown schematically in Figure 1. The dies, themselves, are normally fabricated from TZM grade Molybdenum. Molybdenum is used because of its relatively high strength at elevated temperatures and its excellent thermal conductivity which ensures isothermal conditions. However, since Molybdenum oxidizes rapidly at elevated temperatures, some form of protective environment must be maintained. Alternatively, it is possible to utilize high strength cast nickel base dies where the flow stress of the workpiece is low and long soak cycles are applied to equilibrate temperatures prior to forming, thus facilitating a large reduction in die costs.

The workpiece is normally processed to produce a temporary condition of low strength and high ductility (superplasticity) at the forging temperature prior to charging to the press. For superalloys, this condition implies an extremely fine grain size (often ASTM 10 or smaller) which is developed during a preliminary compressive working operation such as extrusion. Alpha/Beta titanium alloys such as Ti 6-4 and Ti 6-2-4-6 are inherently superductile at the forming temperatures. Consequently, these alloys are normally GATORIZED directly after the conversion operation. Figures 2, 3 and 4 illustrate how IN100, Astroloy, and titanium 8-1-1 respond to tensile loading in this condition of high ductility and low strength as a function of temperature. The forging temperature is usually chosen to correspond with that temperature which exhibits the best combination of high ductility and low flow stress.

Physically, the GATORIZING process relies on the establishment of a dynamic balance between the mechanical damage induced in the workpiece by the forging operation and the thermally activated process of recovery, recrystallization and grain growth. When properly established, this balance leads to the maintenance of a uniformly fine, essentially strain free matrix which stays highly superplastic throughout the forging operation.

III. CHARACTERISTICS OF THE GATORIZING OPERATION

Since the workpiece remains essentially strain free and fine grained throughout the forging operation, very high reductions can be taken in each forging pass and most components are normally formed in a single step operation. In fact, the limitation of the reduction is generally established by either the thinning and consequent breakdown of the forging lubricant or the limitations of the press, as the forging expands and the area reaches the point where the flow stress requires a prohibitative press load. At GPD this second limitation is normally reached when a ten (10) inch high, six (6) inch dia. forging mult is reduced much below approximately one inch in height in a single pass, a single reduction of greater than 90%.

The need to establish the previously mentioned dynamic balance between thermal and mechanical effects implies a relatively low strain rate. This requirement is fortuitous in that relatively low loads are required and, consequently, small forging equipment may be utilized. Most of the production GATORIZING work has been accomplished on forging presses of 5000 tons capacity or less. This work includes the forging of all of the F100 turbine disks. The GATORIZING process does not require the acquisition of specially designed forging equipment, although some modification of conventional equipment is normally necessary to allow for heating and protecting the dies.

The workpiece, after forging, is extremely fine grained in nature, often finer grained than the input stock. An added advantage of the process is the uniformity of strain applied throughout the forging. This uniformity in grain size and strain yield a remarkably uniform response to subsequent heat treatment.

However, the greatest attribute of the GATORIZING process is its shape making capability. Because of the low flow stresses required to activate the process and the freedom from die chill, extremely complex die configurations can be filled with relative ease. More will be said about this fact later, but the ability to forge to net or near net shape leads to large savings in input weight, metal removal time, and scrap. Unfortunately complex dies of TZM Molybdenum are not inexpensive. Consequently, the savings associated with the GATORIZING process become most pronounced when expensive and difficult to machine raw materials are to be fabricated to intricate configurations.

IV. CURRENT APPLICATIONS OF THE PROCESS

The only present production application of the GATORIZING process is in the fabrication of rotating components for the F100 engine. These nine as listed in Figure 5 are produced from IN100 powder metallurgy billet stock.

Shown in Figure 6 is the as-forged configuration of the 9-11-13 composite disk forging along with the forging multiple utilized in fabricating this shape. Early in the F100 program, before IN100 powder billet became available, this component was produced from Astroloy by conventional forging as shown in Figure 7 and weighed 250 lbs. This generous configuration was necessary to protect the actual part geometry from edge cracking and tearing normally associated with conventional forging techniques. Also, shown in Figure 7 is the schematic section of the as-GATORIZED component which was shown in Figure 6. Note that the GATORIZED component in the as-forged configuration weighs 124 lbs less than the conventional forged component.

Figure 8 shows the cross sections of the as-forged IN100 forgings used in the F100 engine. These parts are all being forged to near net sonic shape. All of these shapes are the product of a one-step GATORIZING operation from a cylindrical forging multiple. Due to changes in the design of the parts, several of these configurations are now obsolete. FRDC has produced over 1500 full scale forgings in over 25 configurations for use in the F100 engine since the program was initiated.

V. FUTURE APPLICATIONS

In order to realize the full potential of the GATORIZING process for reducing costs, it is necessary to forge to net, or near net, shape. For aircraft applications, however, this has not been possible because of the requirement of a square cut outline with a minimum envelope of material to facilitate ultrasonic inspection. However, recent improvements in ultrasonic techniques made inspection of close tolerance forgings feasible. As a result, through the use of multiple forging operations, near net shape forgings of IN100 for the nine (9) F100 engine parts could be produced with a total billet input weight of 500 lbs instead of the current 990 pounds. Consequently, a major development effort is being expended to achieve this reduction in component costs for the F100 engine program.

The full extension of the near net shape forging approach is illustrated in Figures 9 and 10. Figure 9 exhibits the same component illustrated previously in Figure 7. Also shown, is the proposed near net shape GATORIZED forging outline. Note that this outline includes some negative draft angles in the integral arm sections. Utilizing a two-step GATORIZING sequence, this configuration has been produced in subscale form as is illustrated in Figure 10. One of the major advantages of the GATORIZING process is the ability to directly upgrade subscale configurations to full scale size. Work is currently being conducted to qualify this configuration for engine use. When qualified, this configuration will save approximately 200 lbs per stage, or about 600 lbs per engine in input weight over an equivalent conventionally forged Astroloy component.

This approach is also applicable to titanium components as is illustrated in Figures 11 and 12. Figure 11 shows the two-step GATORIZING process utilized in producing this configuration. The top and bottom view of this component are shown in Figure 12. This component is fabricated from Ti-6Al-2Sn-4Zr-6Mo, a deep hardenable titanium alloy because it was initially designed as a conventional forging with the out-

line shown on the left side of Figure 13. The gross cross section of this conventional forging made it impossible to harden less sophisticated alloys such as Ti 6-4 to meet the requirements of the F100 engine. The cross sectional outline of the equivalent GATORIZED component is shown on the right side of Figure 13 and represents a net reduction in input weight under the conventional forging of more than 80 lbs. Note, also, that the cross sections of the GATORIZED component are significantly thinner than those of the conventional component, thus allowing the potential use of Ti 6-4 instead of Ti 6-2-4-6 with the potential achievement of equivalent mechanical properties.

In addition to the on-going effort on the forming of components for the F100 engine, development work is currently being conducted to demonstrate the applicability of the GATORIZING process for the production of rotor components for small turbine engines primarily for automotive and industrial use. The primary emphasis of this work has been toward low manufacturing cost. Consequently, our objective has been to GATORIZE as close as possible to actual net component size and shape, thereby minimizing finish machining.

Under a contract funded by the Environmental Protection Agency, a program is being conducted to demonstrate low cost manufacturing techniques for producing integrally bladed automotive turbine rotors and estimating the manufacturing costs for production rates of 1,000,000 rotors per year. Phase I of this effort, which has been successfully completed, dealt with demonstrating the feasibility of producing such a rotor.

Utilizing a 1 3/4" dia. x 3 3/8" long cylindrical forging multiple produced from conventionally processed IN100 ingot, the preform configuration shown in Figure 14 was produced in a one-step GATORIZING operation. In the as-forged condition, the preform exhibited the uniform, fine grained, and recrystallized microstructure also illustrated in Figure 14. After relubricating the preform, it was recharged into the forging press and GATORIZED to the configuration shown in Figure 15. Figure 16 shows the same forging after tip grinding and vapor blast. Cross sectional schematic views of the preform and final tooling are shown in Figures 17 and 18, and the actual TZM grade Molybdenum tooling is shown in Figures 19 and 20. The cavities for the 53 blades are formed by simple split inserts illustrated in the 5X model shown in Figure 21. With this two step approach, the preliminary cost estimates indicate that this component can be fabricated at rates of one million units per year for approximately $52 per unit.

A similar program, conducted under Air Force funding, has been completed with the aim of producing an 18 inch diameter rotor. This component, shown in the as-forged condition in Figure 22 and in finish machined condition in Figure 23, was actually engine tested and exhibited a stage efficiency of over 90%. The sequence used to generate this configuration is illustrated in Figure 24.

VI. SUMMARY

The GATORIZING forging process is an extremely flexible technique for forming complex configurations of high strength, sophisticated alloy compositions. It utilizes relatively small forging equipment and becomes progressively more economical as raw material and machining costs increase, and when extremely complex configurations are to be formed. The product of the GATORIZING operation is a fine grained, uniformly worked structure which exhibits excellent and reproducible response to heat treatment. The process has been fully production demonstrated and has proven its ability to form some of the most advanced aerospace materials to directly usable configurations.

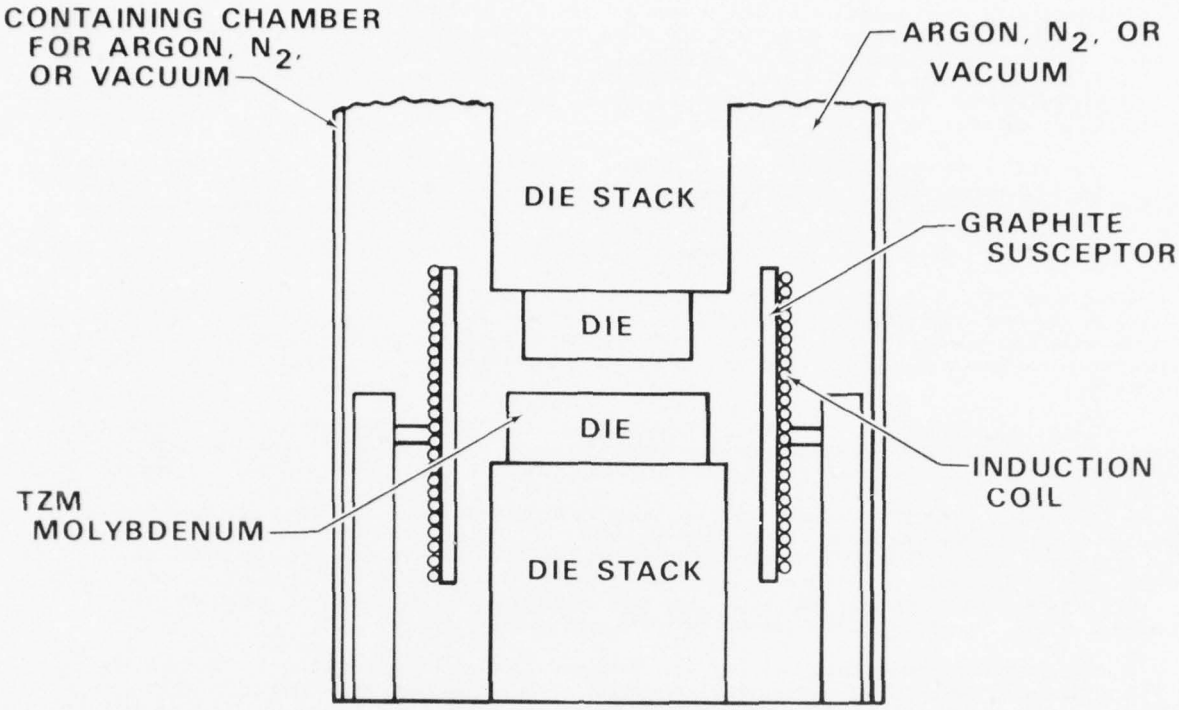
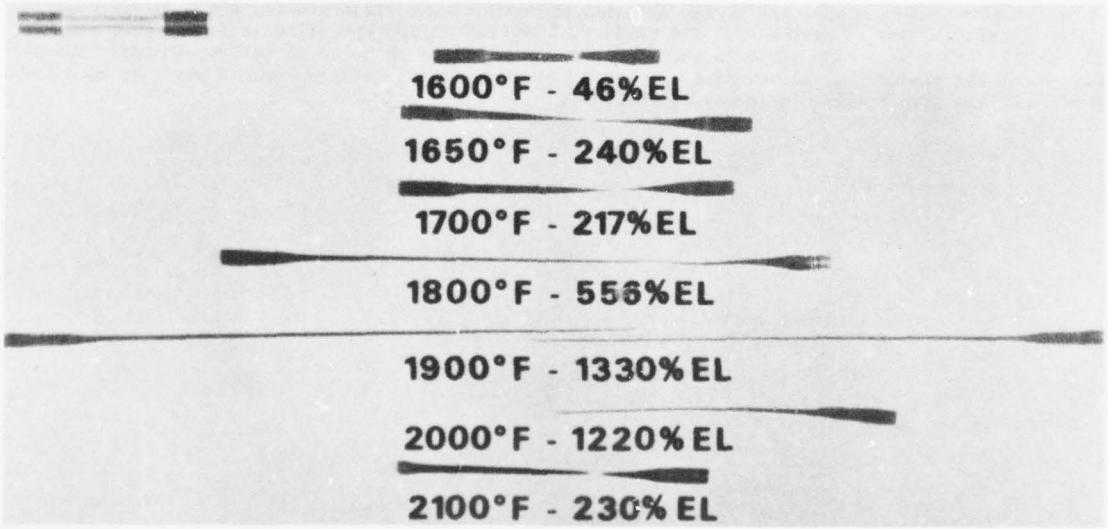


Fig.1 GATORIZING™ forging apparatus



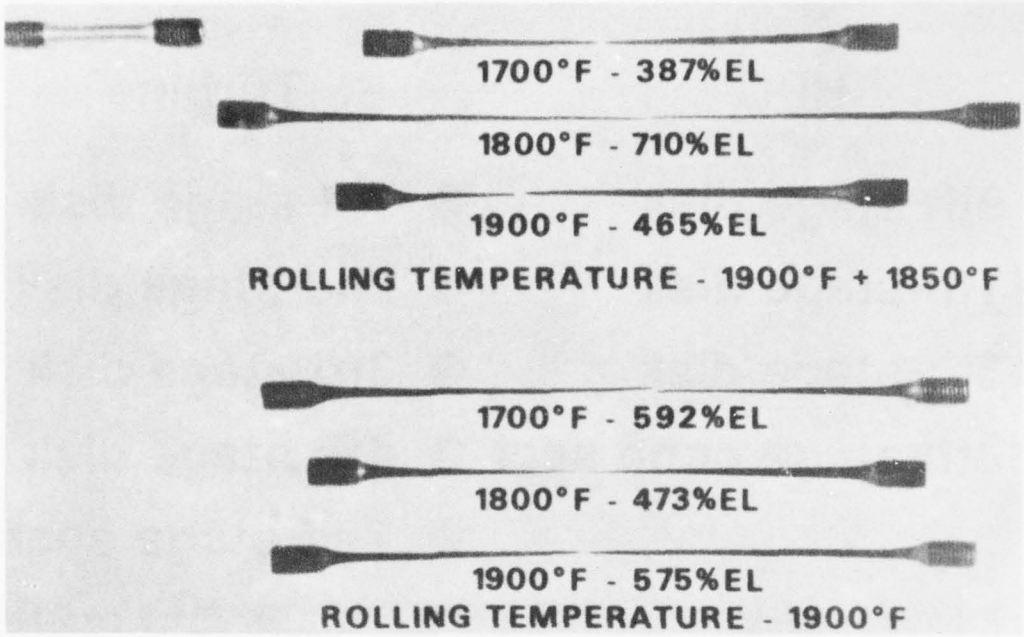
EXTRUSION RATIO → 16 TO 1 ← EXTRUSION TEMPERATURE 2100°F

PLUS

16 TO 1 ← EXTRUSION TEMPERATURE 2000°F

NOTE: TEMPERATURES SHOWN UNDER EACH SPECIMEN ARE TEST TEMPERATURES

Fig.2 IN100 specimens after stress rupture testing



ROLLING REDUCTION FOR THE TWO CONDITIONS ABOVE 7.3 TO 1

NOTE: TEMPERATURES SHOWN UNDER EACH SPECIMEN ARE TEST TEMPERATURES

Fig.3 Astroloy specimens after stress rupture testing

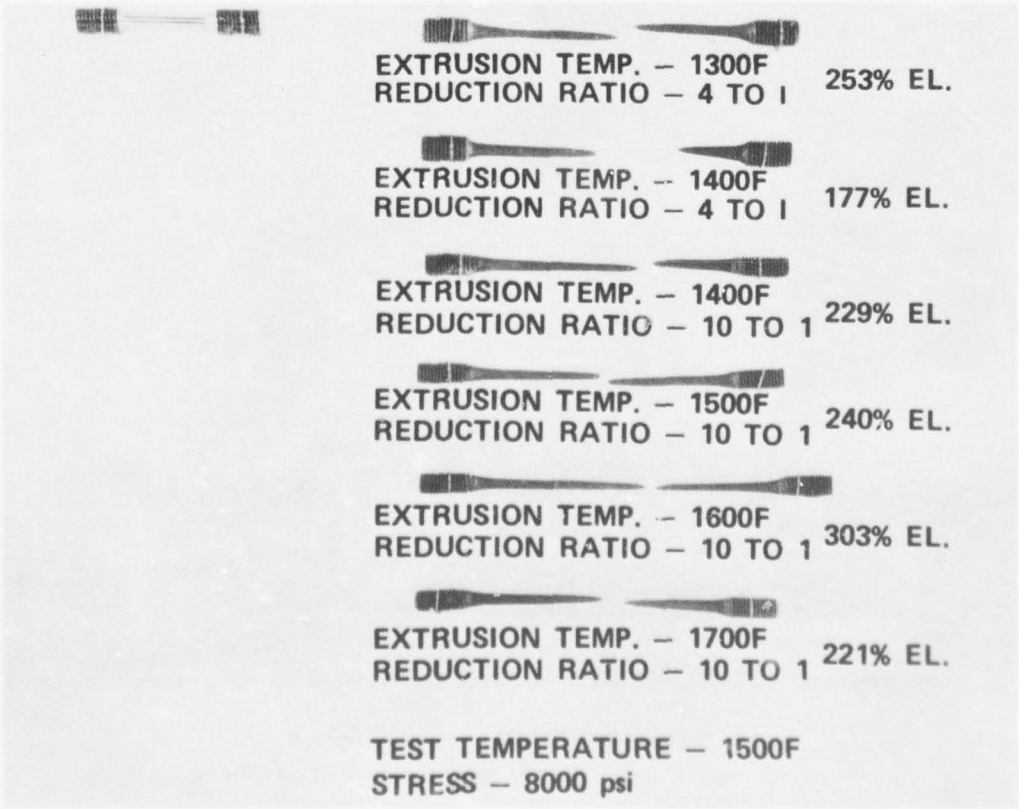


Fig.4 Titanium 8-1-1 specimens after stress rupture testing

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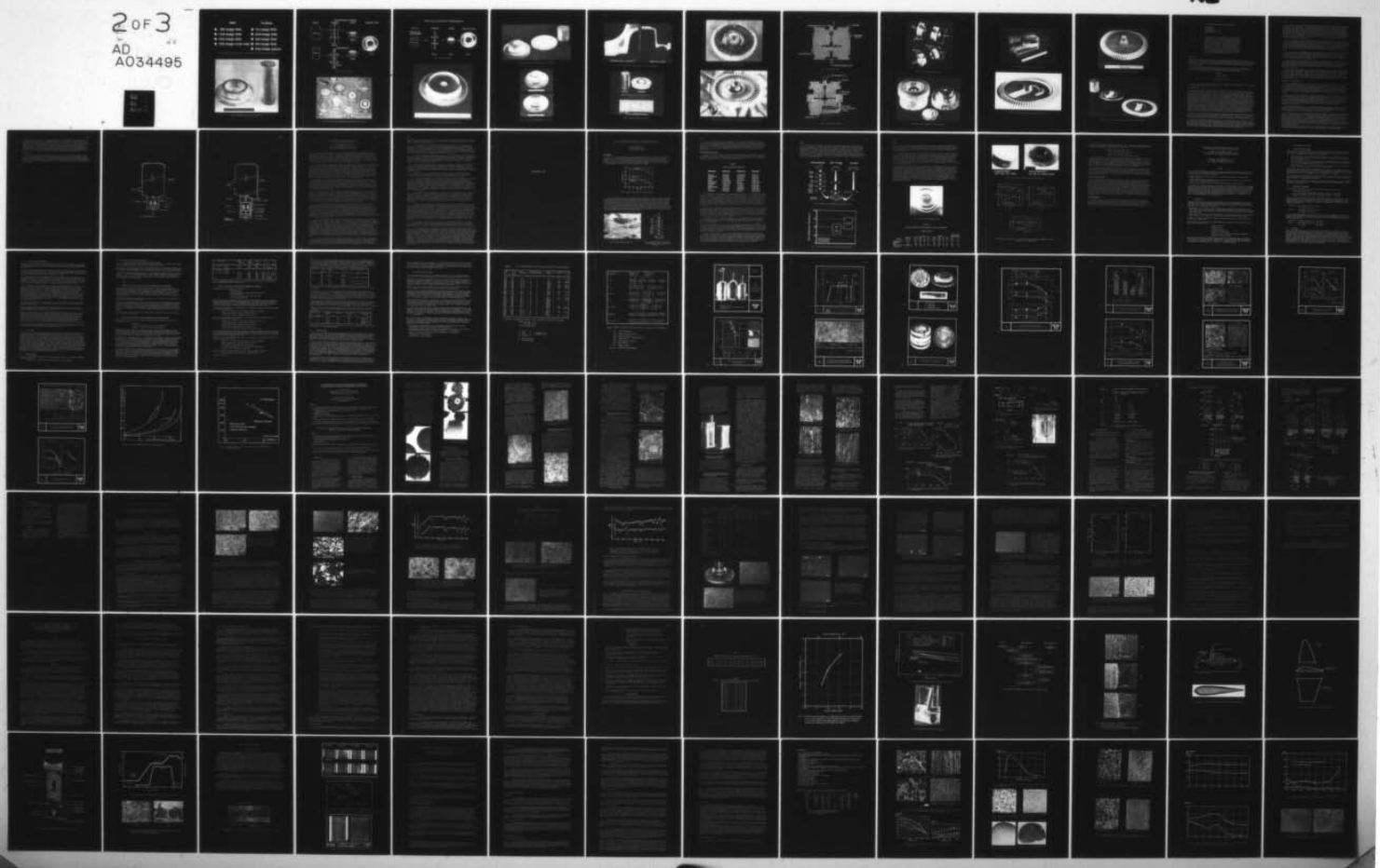

HPC	Turbine
● 9th-stage disk	● 1st-stage disk
● 11th-stage disk	● 2nd-stage disk
● 13th-stage disk	● 3rd-stage disk
● 13th-stage cone seal	● 4th-stage disk
	● 2nd-stage spacer

Fig.5 F100 engine components produced by GATORIZING™ from IN100 powder metallurgy billet

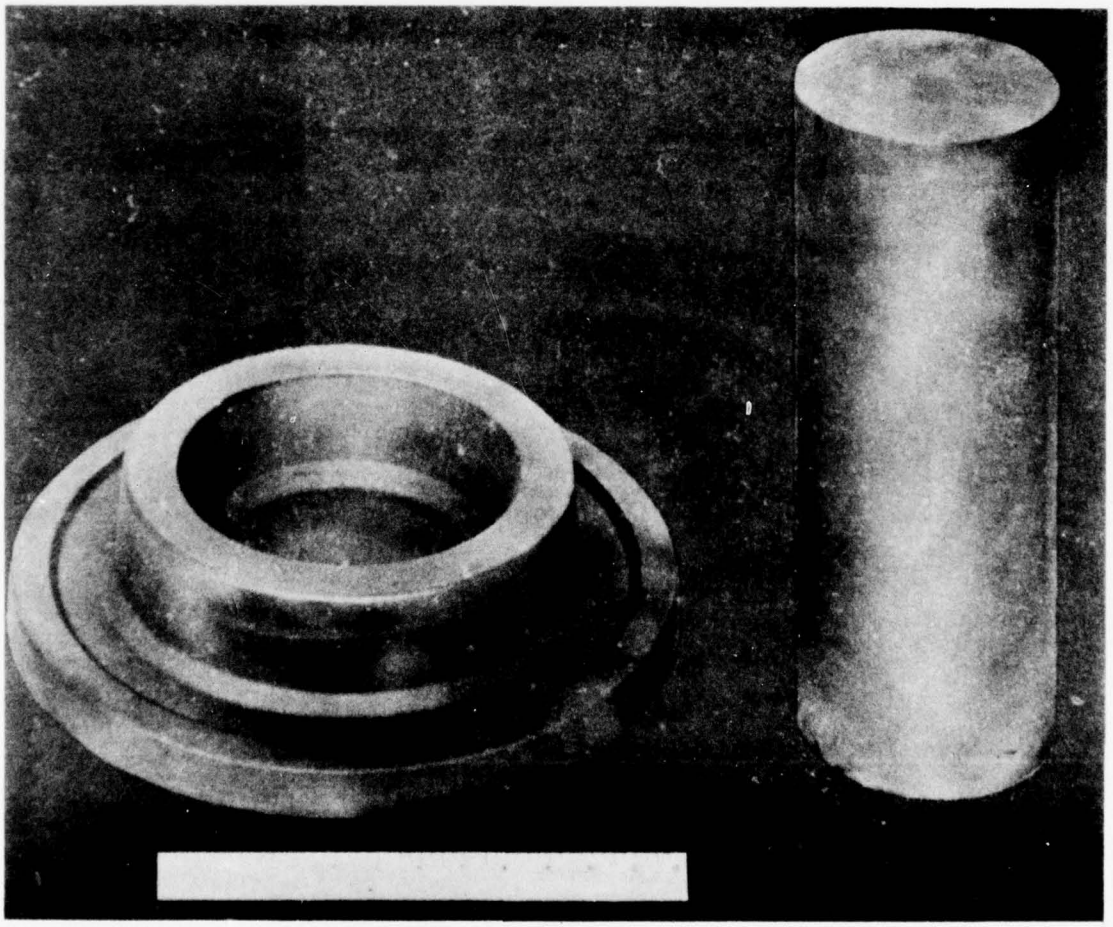

Fig.6 GATORIZED™ IN100 advanced engine disk

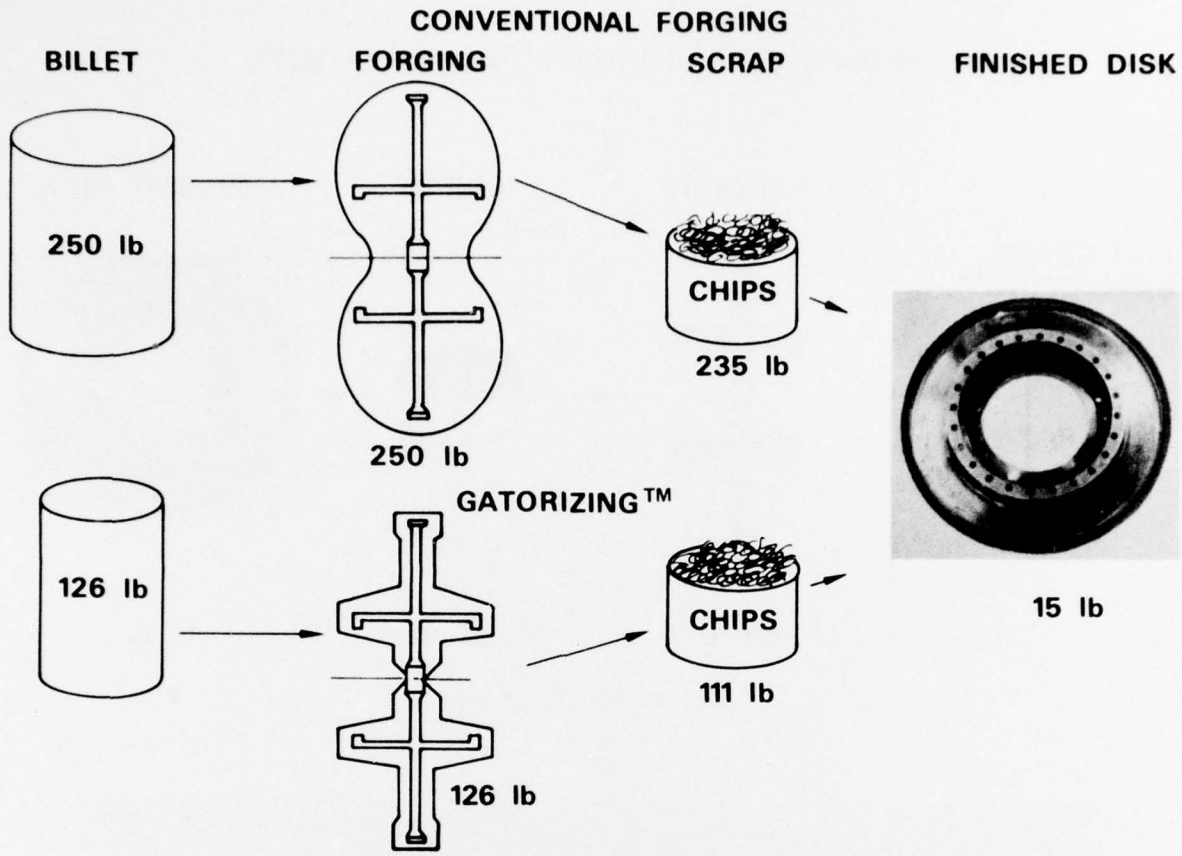

Fig.7 Process cost reduction

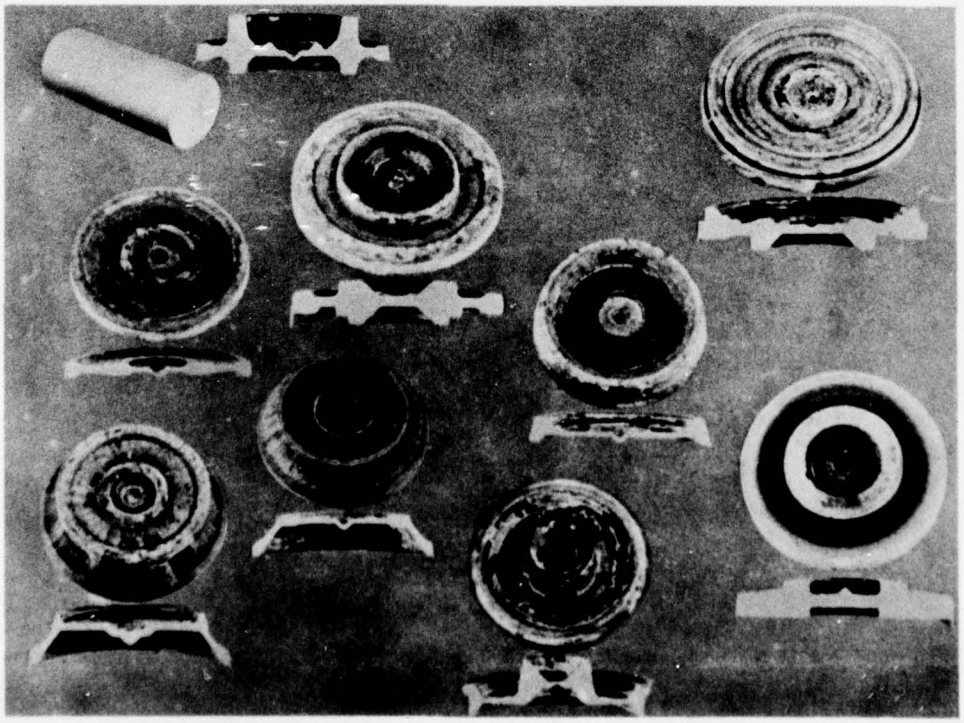

Fig.8 IN100 billet and GATORIZED™ forged disks with cross sections

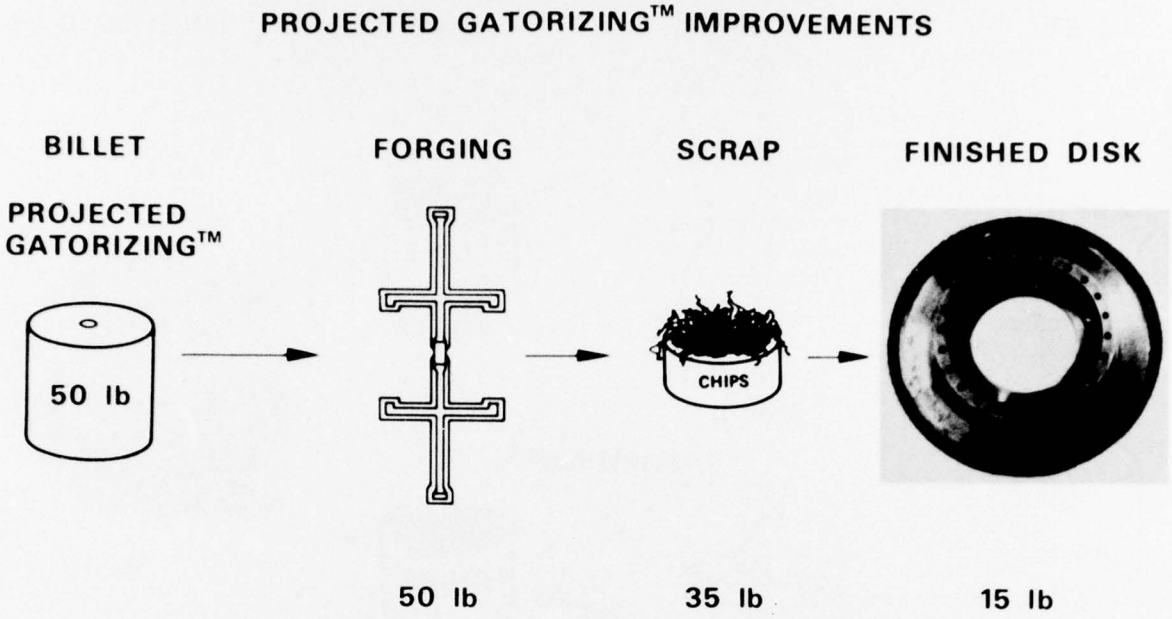

Fig.9 Process cost reduction

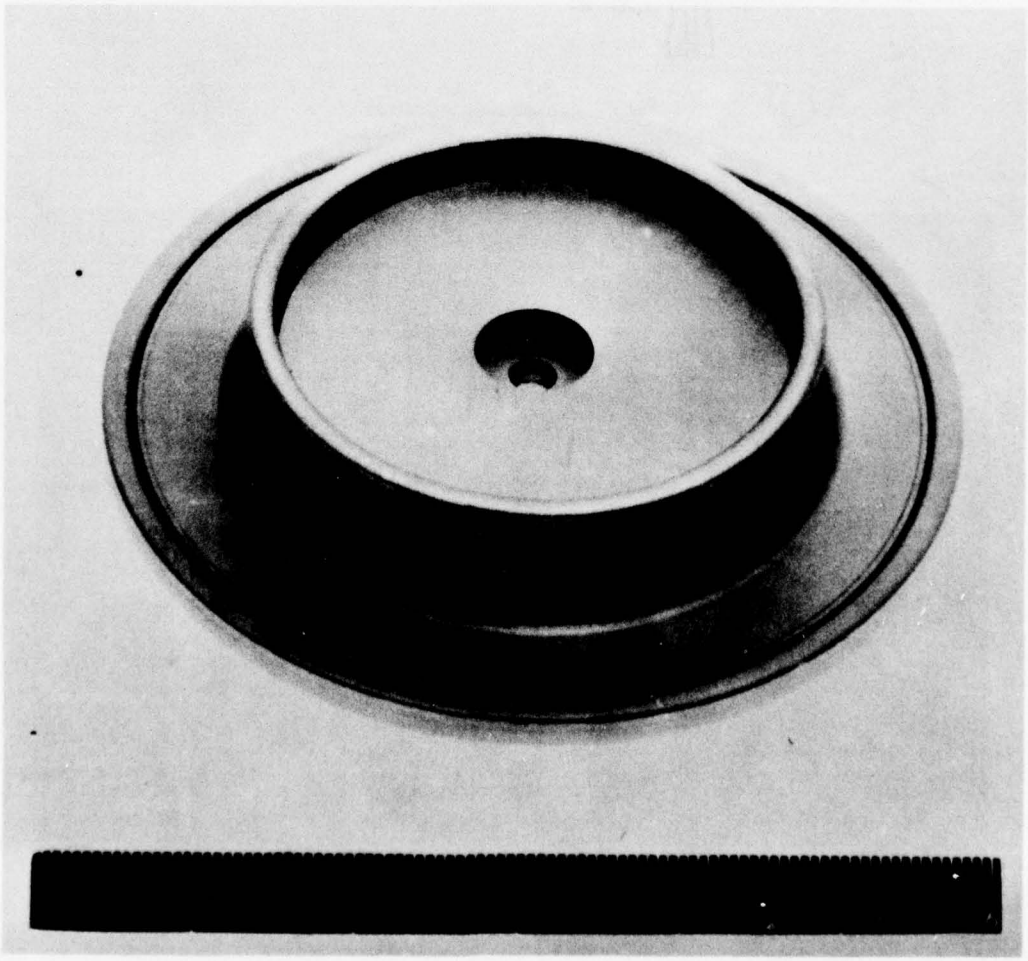

Fig.10 F100 compressor disk subscale near net shape forging

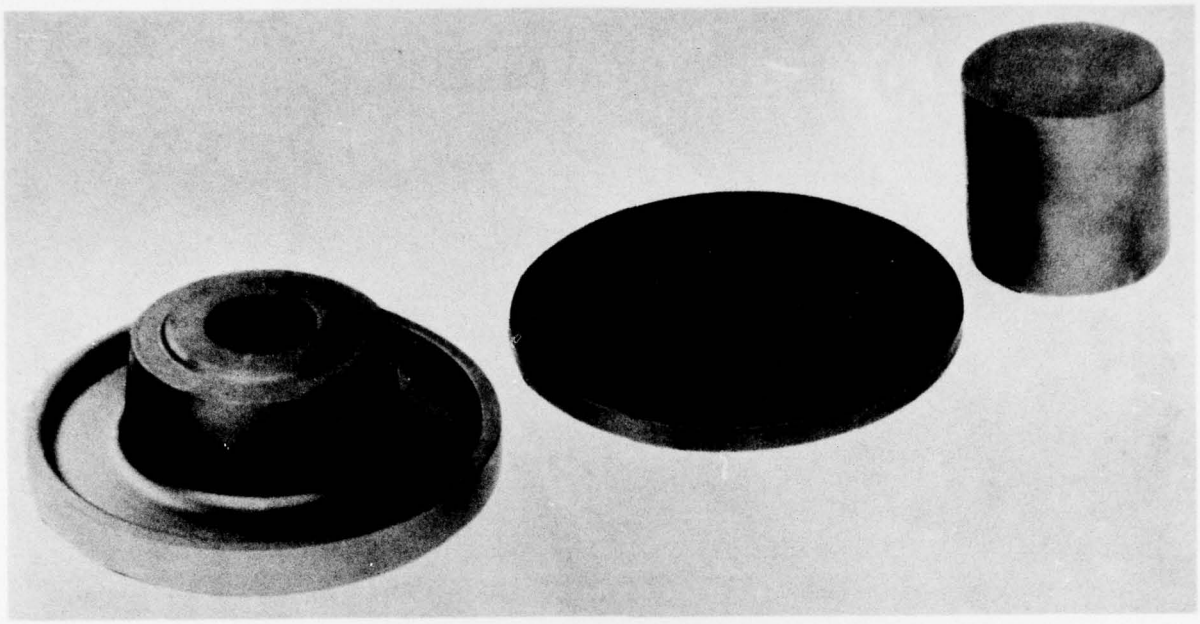

Fig.11 F100 fan disk. Sequence of steps to near net shape

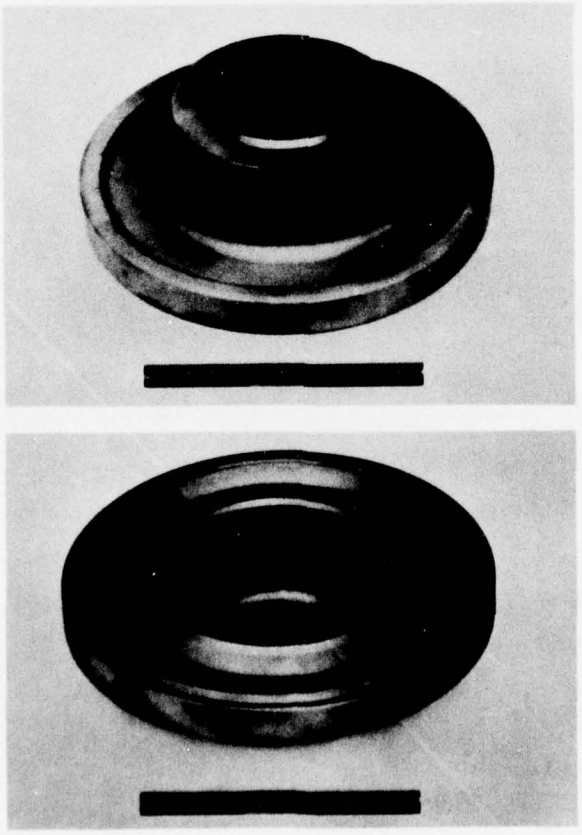

Fig.12 F100 fan disk GATORIZED to near net shape

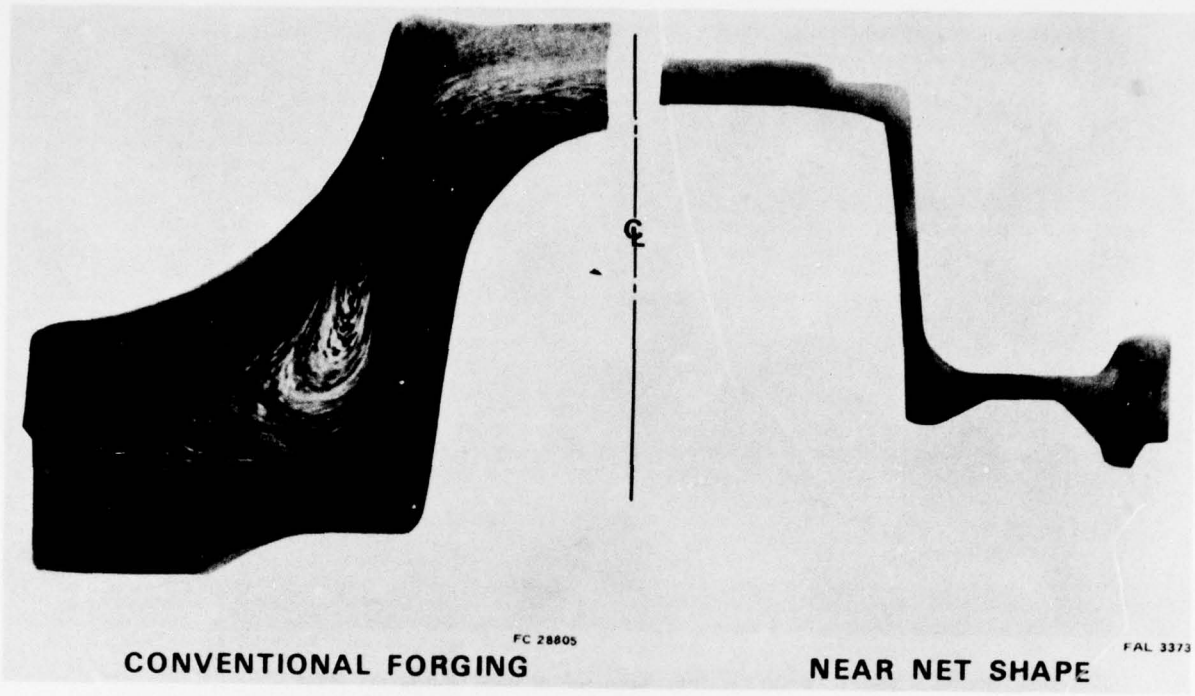

Fig.13 F100 fan disk. Conventional vs near net shape cross-sections

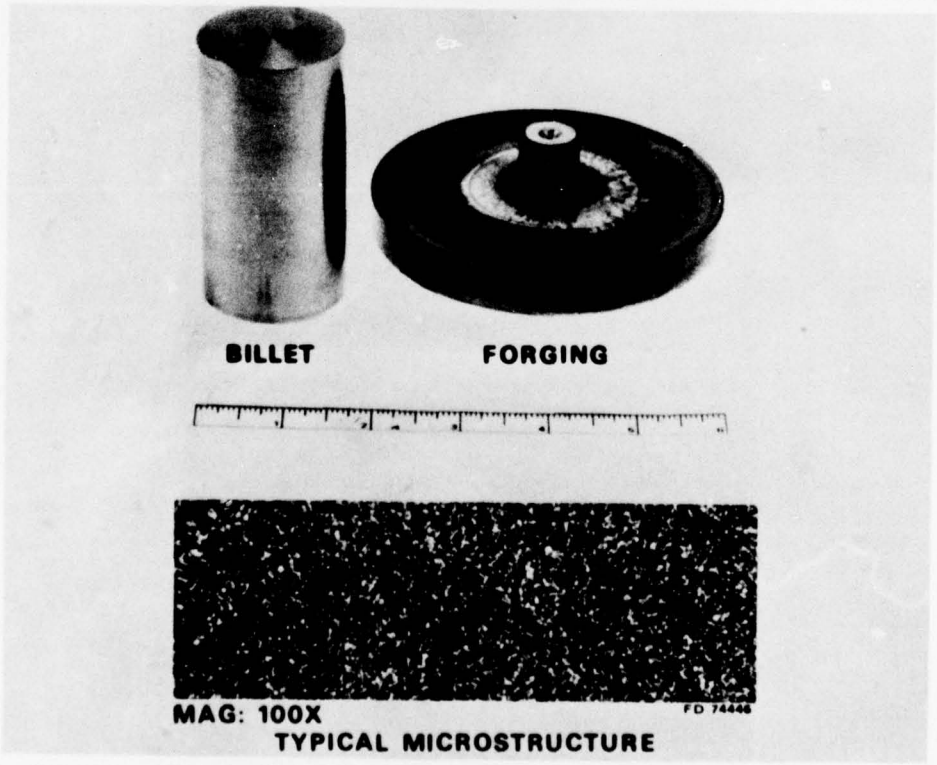

Fig.14 Automotive turbine rotor preform

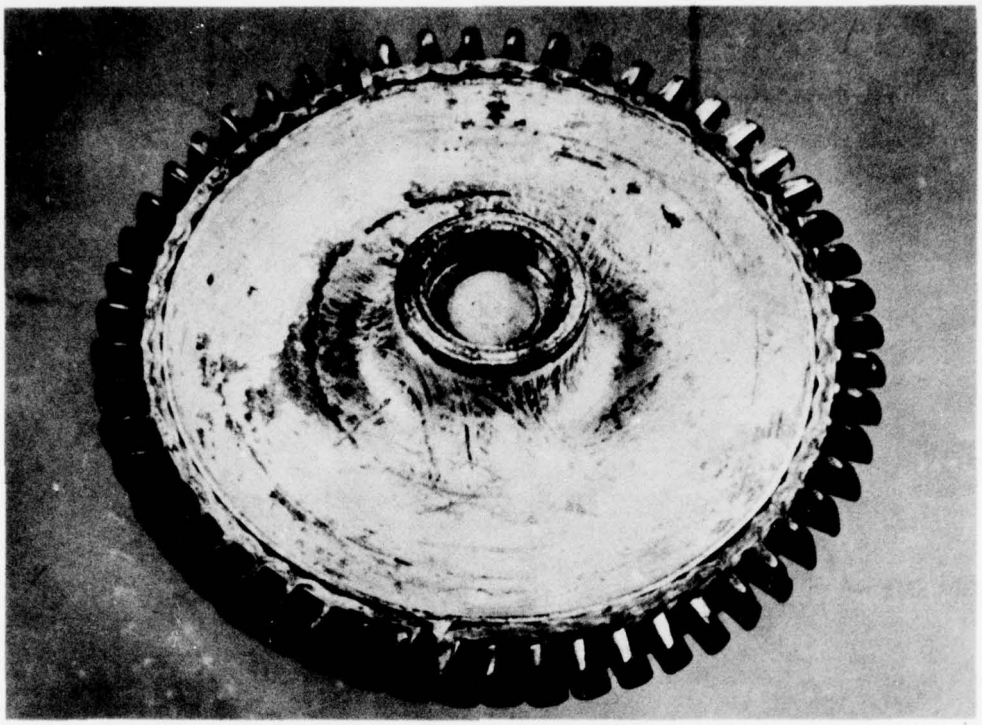

Fig.15 Fully bladed rotor forging

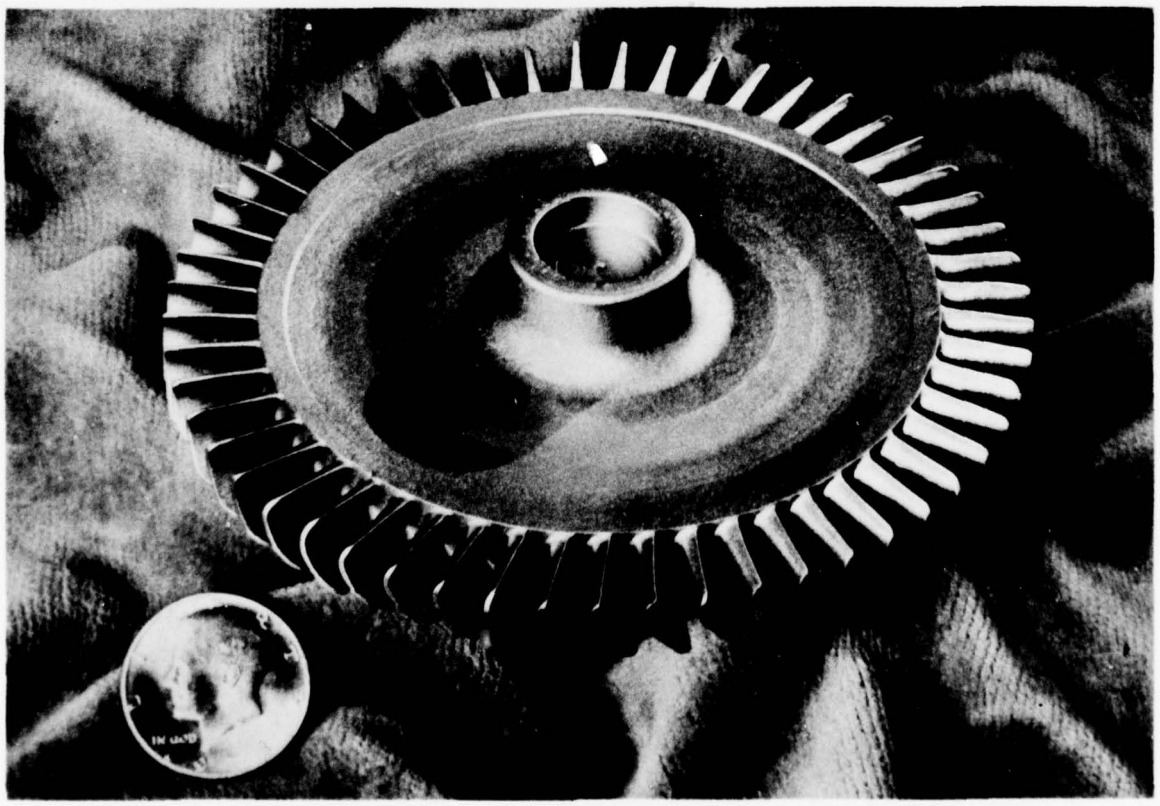

Fig.16 GATORIZED™ automotive turbine rotor (as-forged plus tip grind and vapour blast)

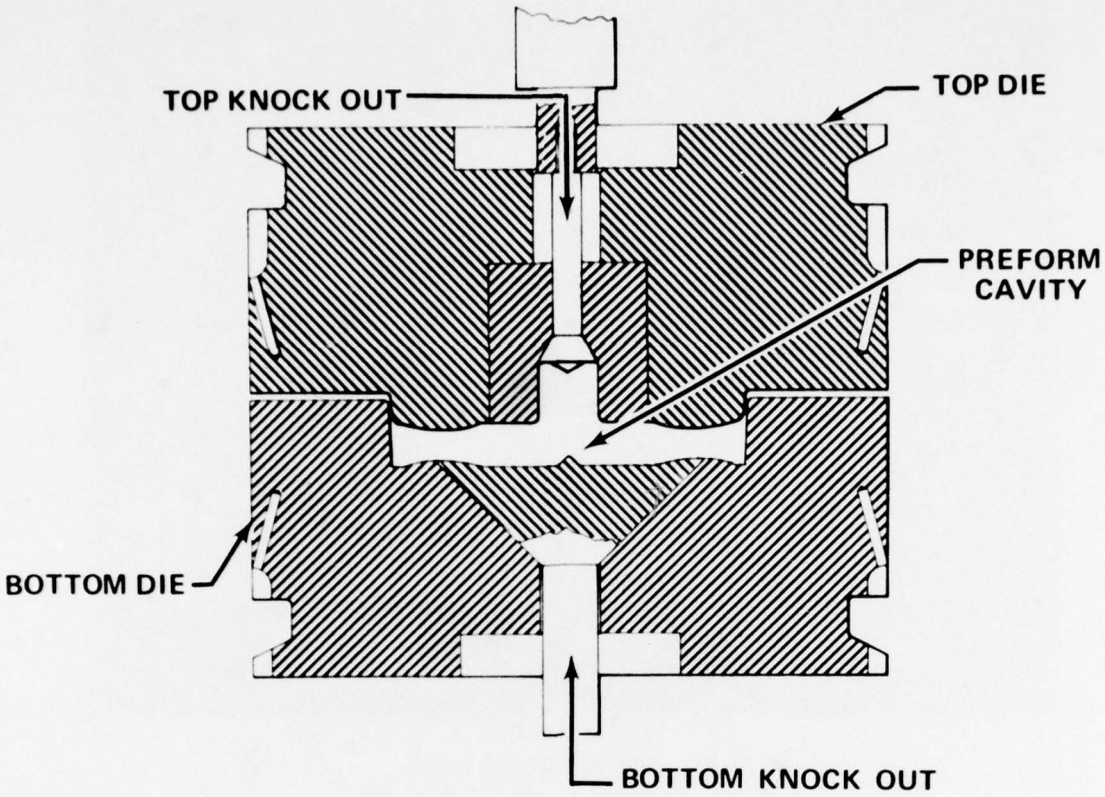

Fig.17 Rotor preform tooling

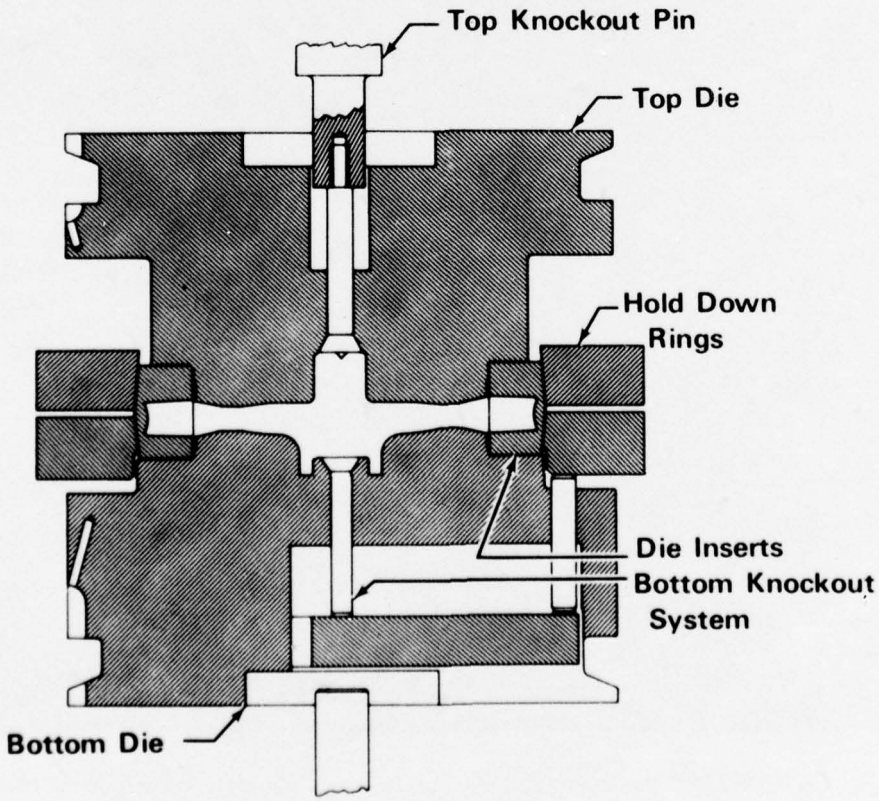

Fig.18 Final bladed rotor tooling

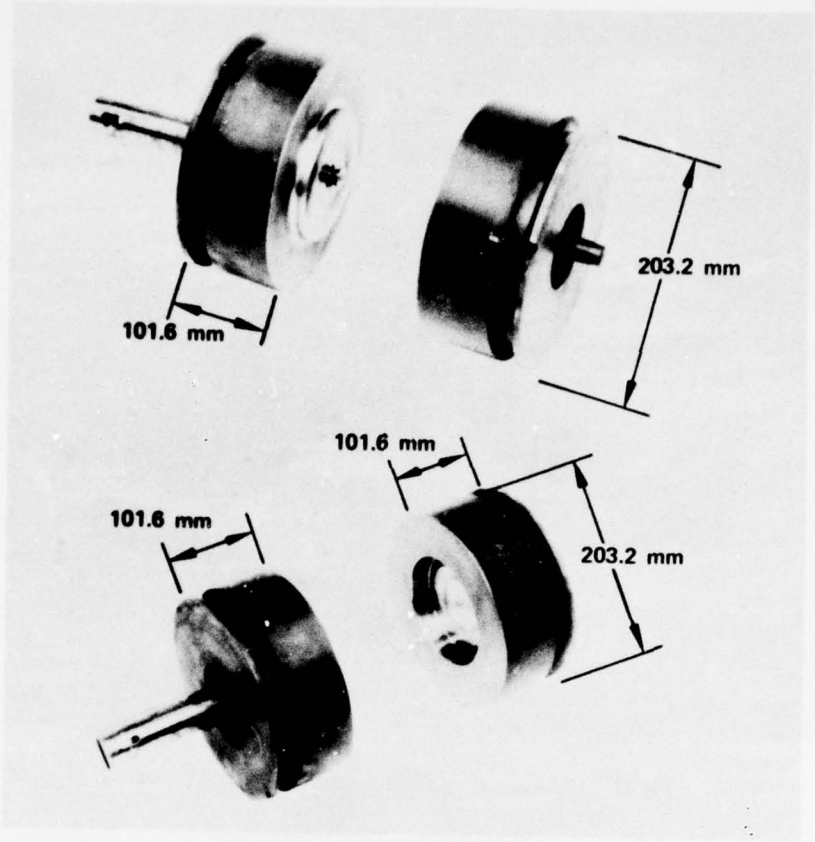

Fig.19 Finish machined preform tooling

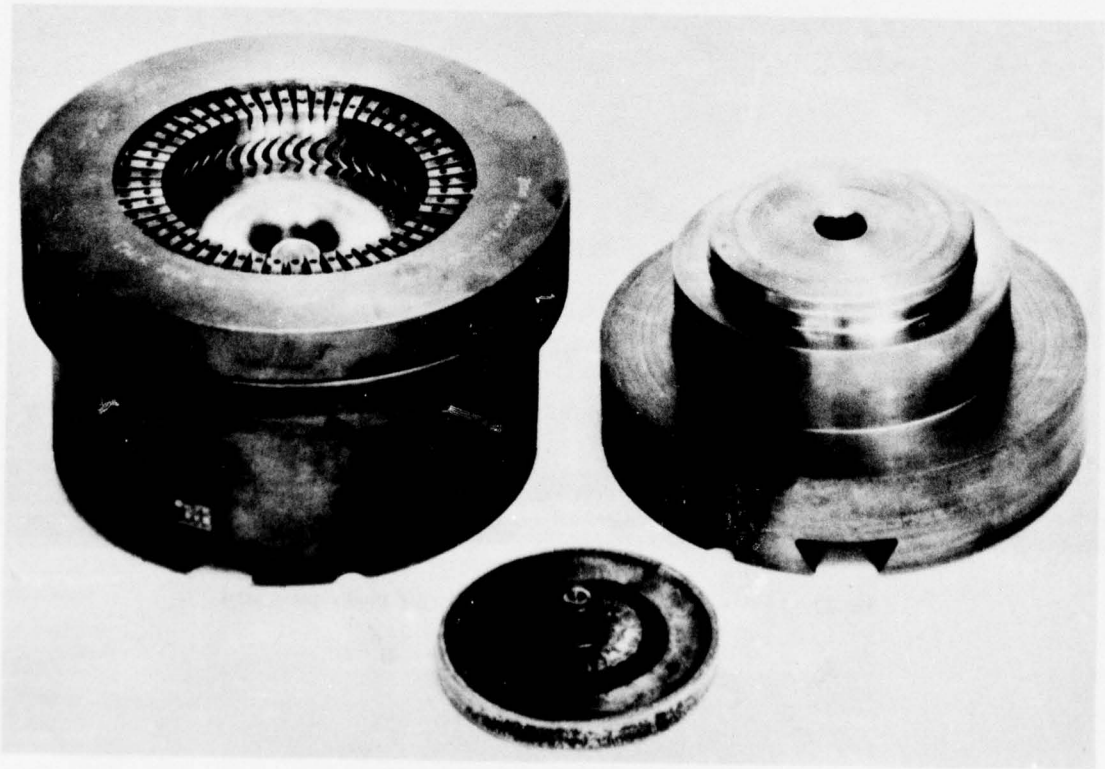

Fig.20 Finish machined bladed rotor tooling and preform

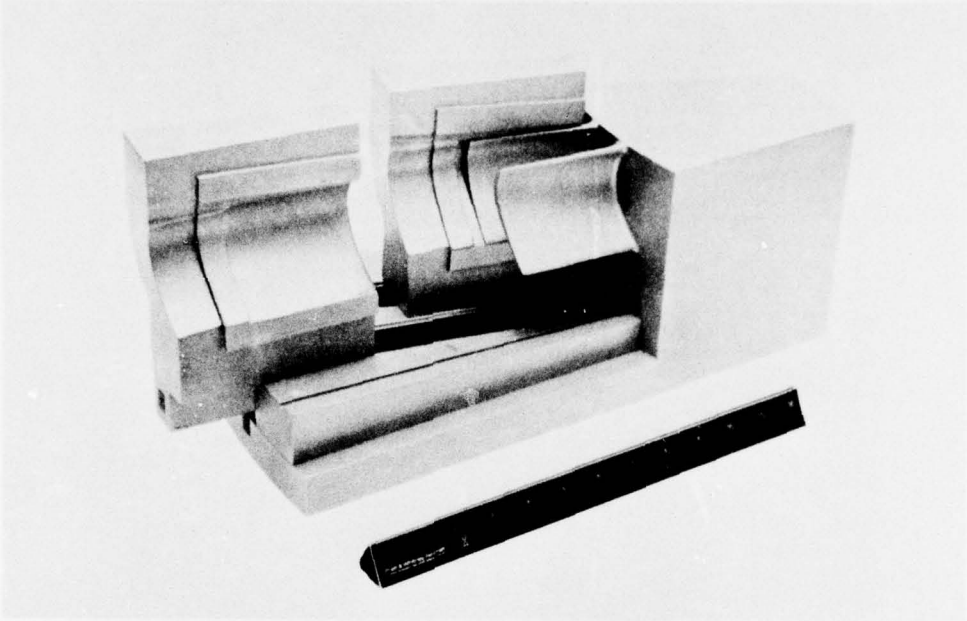

Fig.21 Blade insert concept used for finish die design

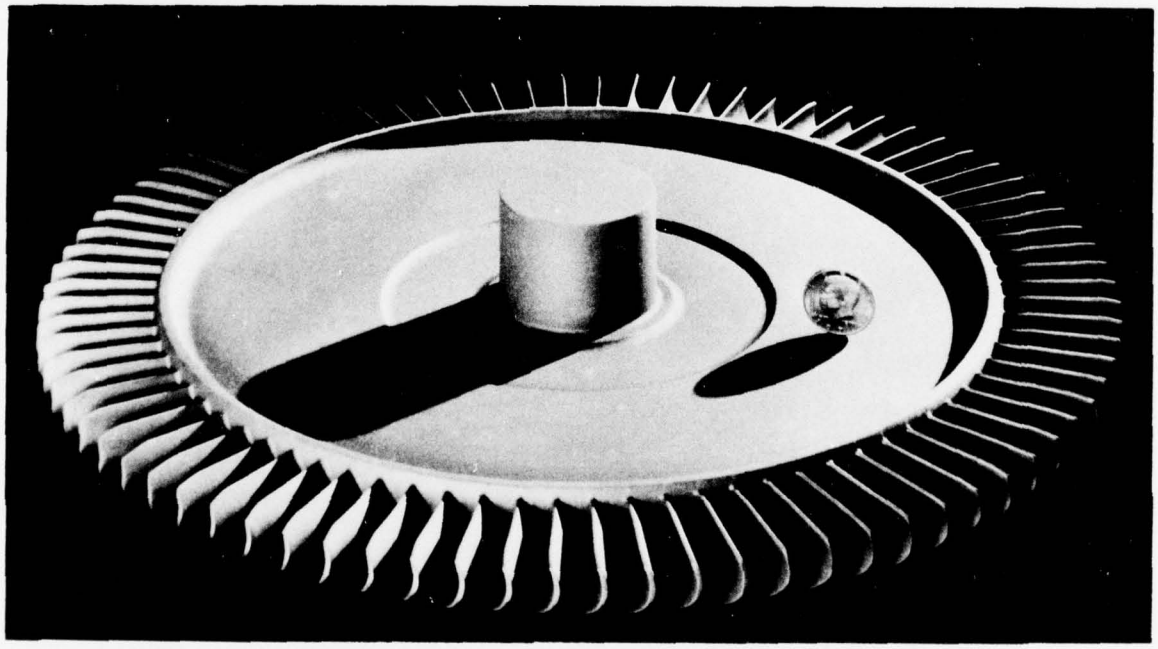

Fig.22 Low cost small turbine disk. As-forged plus vapour blast

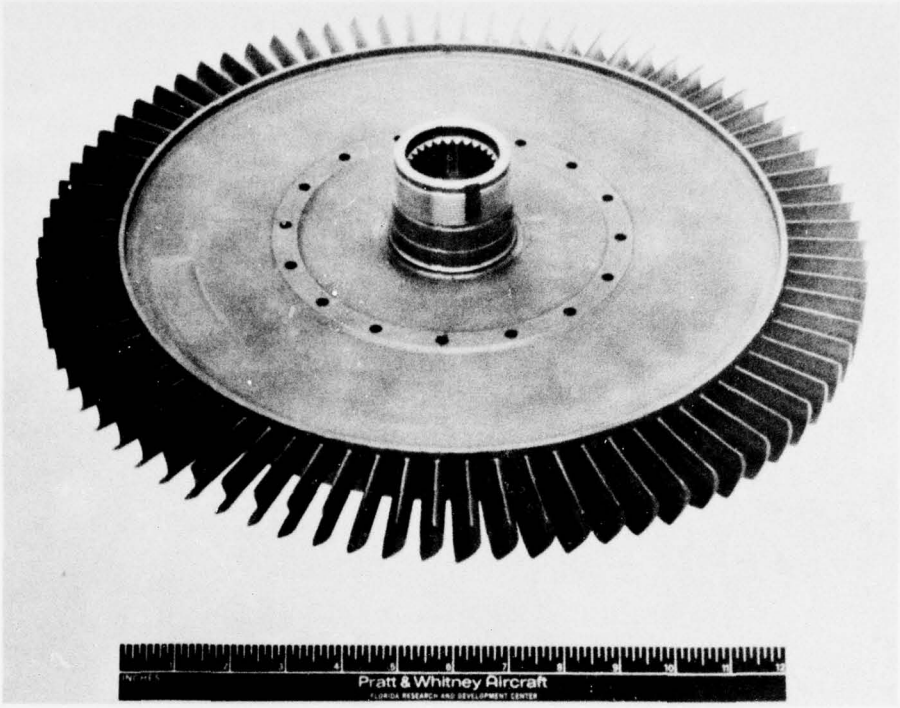

Fig. 23 Low cost turbine disk (finish machined)

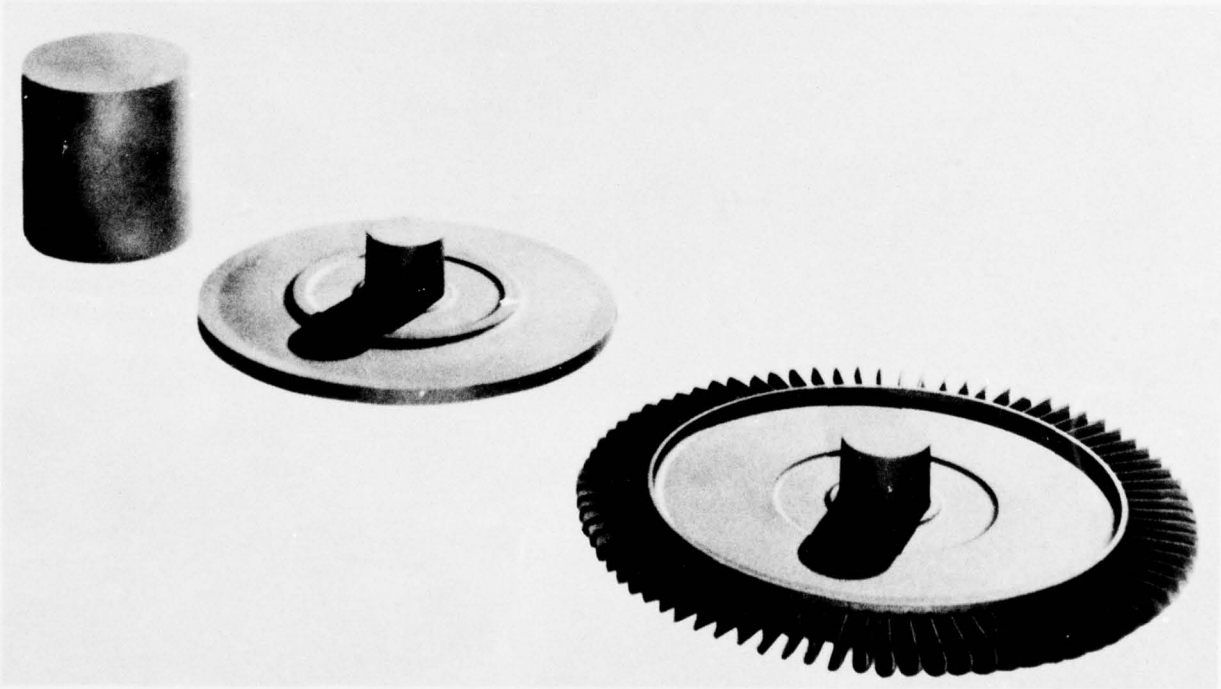

Fig. 24 Low cost small turbine disk. Sequence of steps to final part shape

METAL POWDER PRODUCTION BY VACUUM ATOMIZATION

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The problems inherent in metal atomization have spurred the development of vacuum atomization, a process showing much promise. In this process, the potential energy for atomization can be stored within the molten metal, which increases the efficiency. Also, the higher tap densities of vacuum-atomized powders, as compared with argon-atomized powders, is a distinct advantage. Alloy powders based on Ni, Co, Fe, Cu, Al, and misch metal have all been made successfully by this process. Powder metallurgy will play a significant role in this era of conservation and cost reduction, and vacuum atomization has shown that the unconventional approach may offer the most practical solutions to some of our current problems.

INTRODUCTION

Powder metallurgy became a feasible way to eliminate the segregation problems in conventionally cast, complex alloys over a decade ago. The highly reactive elements in superalloys ruled out the possibility of making powders of these alloys by conventional atomizing techniques. Substituting inert gas for the water or steam in conventional atomizers and developing a "dry" system was the natural step toward getting a higher quality product.

Henry and Sievert developed the very useful gas laws which state that the higher the pressure of a gas over a solution, the higher is the concentration of that gas in the solution. In the case of a diatomic gas such as hydrogen, the law is developed from the equilibrium equation

$$H_2 \rightleftharpoons 2H+$$

$$K=\frac{(aH+)^2}{p} \qquad K=\frac{(aH+)^2}{p}$$

$$aH+=K(P_{H_2})^{1/2} \qquad aH+=K(P_{H_2})^{1/2}$$

Hence, in the case of hydrogen, the amount of gas in solution is directly proportional to the square root of the pressure of the gas over the solution.

METAL ATOMIZATION

Considering the energy required to disperse a high-mass material with a low-mass gas whose velocity was limited by the speed of sound, metal atomization appeared to have inherent restrictions. The expense of large quantities of gas, purification trains, and pumping and storage equipment seemed to dictate development of an alternative method. It was realized that there could be a way to atomize metal using energy stored in the system while maintaining a very pure atmosphere. The molten metal could be atomized by using the heat energy supplied by the bath. As hydrogen goes into solution as H+, the H-H bond is broken, taking just over 100 cal/mole. Pressurizing a 100-kg heat of material, in the order of 5 moles of H_2 could be held in solution at pressures of about 7 atm. If it is assumed that all of the heat released during the degassing was used to heat the H_2, its temperature would rise from the melting temperature of about 2000°K to over 10 000°K instantaneously. The amount of energy available for atomization due to the recombination of the H-H to H_2 is almost 200 times that needed to obtain an average particle size of 25 μ diam. Part of the energy is used to superheat the hydrogen-metal interface thus lowering the surface tension making metal separation easier; much of the excess energy is radiated to the receiver walls. The effluent gas would be pure H_2. Further estimates, using the surface tension and available energy, indicated that the average droplet size would be in the proper size range for a powder-metallurgy product. Building a receiver to contain the predicted explosion became of concern. The force would not be great; however, receiver dimensions could not be calculated and this necessitated a few experiments.

First, a 50-lb. furnace was constructed which was capable of maintaining vacuum and 150-psi pressure. It was built in such a manner that the molten metal could be blown into the atmosphere. The initial melt was cast iron, and N_2 was used as the soluble gas at a pressure of 100 psi. The resulting metal-gas explosion could not be controlled; molten metal literally filled the room and gas vented at sonic velocity.

It was then estimated that a tank approximately 30 ft. high would contain most of the spray. Further experiments in the completed pilot-plant unit depicted in Fig. 1 proved that a superior product could be made. During the initial work, a resistance-heated alumina tube was used to transfer the molten metal from the melt chamber to the vacuum receiver. Unheated graphite pipe was also used, but some carbon pickup was experienced when using graphite. Various tube sizes were tried, ranging in diameter from 1/2 to 1/4 in. The smaller tube sizes had a tendency to freeze. Nozzles were also tried with some success.

The production unit incorporated the same principles as used in the pilot plant plus many additional features to allow fast turnaround. The production unit is depicted in Fig. 2. Because it is desireable not to expose the powder to air, the unit was designed with a sloping floor so the powder could drain from the tank under vacuum. The lower chamber was designed so the melt could be made remote from the unit and moved under vacuum to the blasting position. Hence, two lower units could be in operation at the same time, almost doubling production. Locks were designed into the vacuum receiver, so the furnace could be moved into position, sealed, teemed, and removed, the tube changed, and the powder removed while maintaining the receiver under vacuum. The unit has been operated in excess of 2-month periods without exposing the receiver to atmospheric pressure. The receiver need only be brought to the atmosphere when a change of alloy requires cleaning. The receiver is 10 ft. in diameter and 50 ft. high. An elevator is assembled in the receiver for cleaning.

In going from the pilot plant to the production unit, unforeseen but predictable problems were encountered; the production unit would produce only very coarse powder and flake. The nucleation and growth of the gas in the molten metal occurs at the speed of sound. During design of the large unit, the transfer tube was lengthened to two and one-half times that used in the pilot plant. The six atmosphere-pressure-differential distributed over the longer tube resulted in degassing in the tube rather than at the orifice. Once the problem was recognized, a solution was rapidly worked out. The powder became finer as Δ pressure/ Δ time increased. By simply increasing the pressure drop per unit time, the unit became productive.

Any atomizing process is dependent on the surface tension of the liquid being atomized. The surface tension increases with decreasing temperature, and is also affected by the addition of elements which lower the molecular attraction. The "fluidity" of steel can be increased with very small additions (3 to 5 ppm) of calcium. Therefore, we have two important factors causing variations in the surface tension and consequently the powder particle size. We also have the pressure-time relationship and the mass-velocity relationship of the gas being utilized.

It was discovered several years ago that argon, which is the common gas used in conventional atomization, became entrapped in the powder particles.[1] Being a large and inert molecule, it was impossible to exclude the entrapped argon by diffusion. Upon reheating a consolidated powder bar to near the incipient melting point, the argon was found to expand, causing porosity in what previously appeared to be a theoretically dense bar. It was further found that the vacuum-atomized product was not subject to thermally induced porosity.

One superalloy powder producer tried to atomize with hydrogen by blasting the molten stream of metal with the gas, knowing hydrogen could be readily diffused. Unfortunately, the velocity of a gas leaving a nozzle is limited by the speed of sound in the gas, and although the speed of sound in hydrogen is four times that in argon, the density of hydrogen is only about one-twentieth that of argon, so the energy availability in hydrogen is about 80% that of argon. In addition, since hydrogen does not cool adiabatically when expanded, the metal would not be cooled as when atomizing in argon. This one attempt was not followed by a successful process, indicating that hydrogen is not a satisfactory gas for conventional atomization

ADVANTAGES OF VACUUM ATOMIZATION

In the vacuum atomization process, the mass-velocity relationship exists. We have blown steel with both Co and N_2 successfully. Not enough experiments were run nor were the solubilities measured to confirm whether the mass effect was responsible for the variation in particle distribution. Argon has been percolated into the transfer tube to stimulate the conditions existing during hydrogen degasification. [The ratio of initial temperature to teeming temperature in the case of argon is about equivalent to that caused by the theoretical bond energy of hydrogen on recombination, and the change in particle distribution is about what could be expected from the mass-velocity relationship in comparing the two gasses.]

Vacuum atomization is a rapid process. The melt itself contains the potential energy for the atomization and the more rapidly that energy is released, the finer the powder. In the pilot plant, 50 lb. of metal is blown in less than 10 sec.; in the production unit, we take as long as 2 min. to blow 400 lb. The unit was constructed in what would be considered an "upside down" design. It was done to allow the particles time in suspension to radiate most of their thermal energy. It would have been impractical to construct a receiver high enough to allow the particles, particularly the larger ones, to cool in a free fall before collecting. The collected powder in the bottom of the receiver is about 600°F immediately following the blow.

Alloy powders based on Ni, Co, Fe, Cu, Al and misch metal have all been made successfully by vacuum atomization. The most exotic alloy was a 60% misch metal-40% aluminum composition. We have avoided making alloys containing highly volatile elements, such as zinc, and also alloys containing Pb, Bi, Sb and other elements which could lead to deleterious contamination of superalloys.

The tap density of the vacuum-atomized powders is about 5%-7% higher than that of the argon-atomized powders. The difference has been attributed to the particle-distribution difference rather than to the difference noted in the powders. The vacuum-atomized powders have very few particles that are stuck together and are termed "satellites," as compared with the large numbers found in argon-atomized particles. The vacuum-atomized powder has a uniform distribution with a notable absence of a normal curve. It is the sort of distribution one would expect from an explosion. Other known processes result in classic normal curves. The higher tap densities have very definite advantages in consolidation, particularly when consolidating to near net shapes, and when consolidating using the HMI "Soft Can" technique. Disadvantages in the random distribution occur when a narrow distribution of particle sizes is desired, such as is required for plasma spraying.

[1]NASA TN D-6072 Evaluation of a Cobalt Base Alloy, HS-31 made by extrusion of prealloyed powders: John C. Freche, Richard L. Ashbrook & William J. Waters; Lewis Research Center, Cleveland, OH 44135

From the time the melt stock is placed in the furnace until the powder is a consolidated product, the alloy is never exposed to air. The powder is handled in vacuum, hydrogen, nitrogen, or argon. Considerable controversy concerning the deleterious effect of air exposure is still going on but we know of no data that confirm that inert handling is absolutely necessary to achieve a satisfactory product. However, we have considerable data to show that inert processing does yield an excellent product. Many of the same parameters that are subject to control in argon atomization must also be controlled in vacuum atomization. Being able to store the energy for atomization within the molten metal has proved to be very efficient in the atomization process. Now, after several years of intensive development work, the production unit is operating properly and producing a product with excellent properties.

CONCLUSION

We are entering a new era in metallurgical processing, one of shortages and conservation. In the aerospace market we are seeing an ever increasing variety of very expensive materials and relatively low volumes, and continuing high interest in cost reduction without a deterioration in technology. Powder metallurgy will play an important role in this era of conservation and cost reduction, and vacuum atomization has shown that the unconventional approach may well offer the most practical solution to some of our current problems.

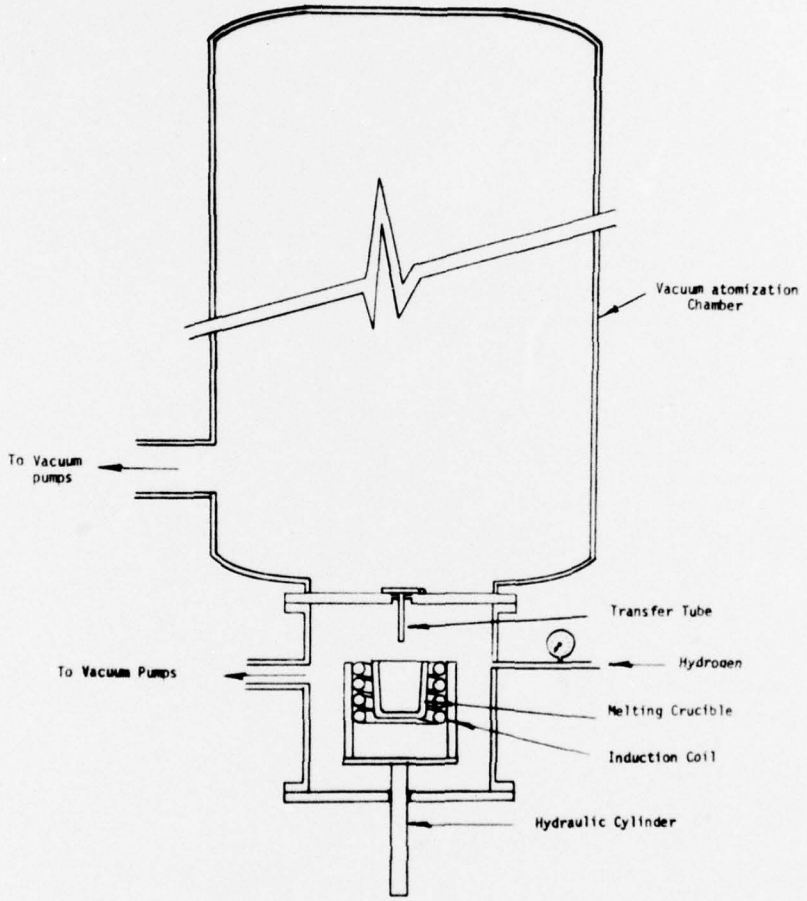

Figure I Vacuum Atomization Pilot Plant

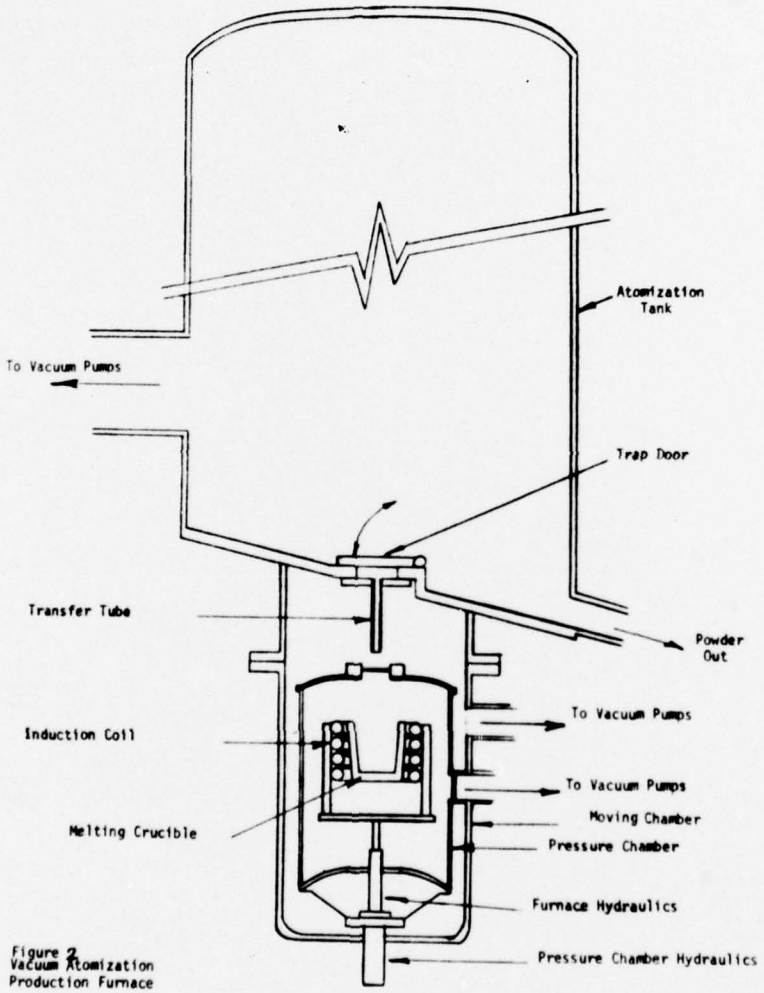

Figure 2
Vacuum Atomization
Production Furnace

TX - 67 Advanced Fabrication Techniques
and Their Economic Implications.

DISCUSSION SUMMARY OF SESSION II

by W. Wallace,
National Aeronautical Establishment,
National Research Council of Canada.

The session consisted of three papers and four short contributions, two of which were included as additional items to the program and presented during an extended discussion at the end of the day. Session II emphasized the consolidation and thermo-mechanical processing of superalloy powders to produce "wrought type" products such as discs: one contribution discussed powder production, one considered the economics of HIP processing as it is influenced by work load characteristics and the use of preheat, and a further contribution described explosive consolidation of powders. To close the session a film was shown describing the ASEA Process for powder processing.

It was apparent in most papers that economic factors are important in justifying the interest in, and use of powders. Lower costs were often associated with lower material input to produce a given part (papers P3 and P5, and contribution P4A). For example at Pratt and Whitney (P4A), P/M Astroloy disc forgings were produced using 120 lb less material than would be needed to produce the same part from ingot material. Advantages were also associated with the elimination of forging operations (P5, P4A), with reduced machining after forging (P5, P4A), and with improved machinability (P3 and P4A). In certain cases, such as in the Pratt and Whitney forging of Astroloy discs, these benefits were considered sufficient to justify the use of powder without necessarily expecting a gain in mechanical performance. In this case therefore, target properties for the P/M products were those used for the equivalent cast plus wrought products.

Studies reported by Messerschmitt-Bölkow-Blohm have indicated that for their titanium products, cost savings through HIP could only be realized if the part otherwise involved high machining costs, that is, costs exceeding about 60% of the total. HIP was considered viable only if it allowed substantial reduction in these machining costs. In practice the cost savings are not directly proportional to the reduction in machining, since finish machining represents the most expensive operation. HIP was also considered to be limited because of size restrictions. Consequently only about 15-20 parts had been identified as candidates for HIP.

The consensus was that the greatest savings through powder would be achieved by net-shape HIP processing of parts, without subsequent forging, and indications of progress towards this objective were given. Data presented by Henry Wiggin and Co. (P3) showed that while improvements could be achieved by forging, mechanical properties of as-pressed parts were often equivalent to, and sometimes better than those of wrought ingot material of the same composition. The contribution from AVCO Lycoming (P4B) showed that complex, close tolerance parts could be produced by a vacuum sinter plus HIP route. The success of this latter work was largely dependant on the development of ceramic mold technology, with its ability to provide excellent shape definition in the products.

While economic considerations were of prime concern, most authors were able to demonstrate technical advantages through powder processing. Much of the work was concerned with powder processing of high strength "casting type" alloys such as IN-100 at Pratt and Whitney (P5), or modified IN-792 at AVCO Lycoming (P4B); or borderline forging alloys such as Astroloy and its modification APKI at PWA-East Hartford (P4A) and Henry Wiggin and Co. (P3) respectively. The superior homogeneity of powder products was considered important in improving forgeability during conventional forging (P4A) and during superplastic forging (P5). Similarly homogeneity was considered important in achieving uniformly high mechanical properties in P/M products. These were said to compare favourably with the better properties in wrought ingot material where properties show much greater directionality (P3).

While developments were reported in net-shape HIP technology, the indications are that forging will remain an important part of superalloy and titanium powder metallurgy processing for some time to come. Papers P3, P5 and P4A discussed many aspects of forging, but emphasis was on superplastic forging. The T/P Process of Henry Wiggin and Co. emerged as an alternative to the Gatorizing Process of Pratt and Whitney. "Thermoplastic Processing" involves cold working prior to compaction, so that the powder recrystallizes during compaction to develop the fine grain size necessary for superplastic forming. The cold rolling does not appear to interfere with tap density or subsequent compaction, although some stratification of powder was noted. The T/P Process avoids the extrusion treatment inherent in gatorizing, and therefore may offer greater flexibility in preform configuration. Preliminary work reported by Pratt and Whitney on as-hipped compacts indicates that this material responds well to isothermal forging. As-hipped billet offers cost savings over extruded billet and therefore this may become the preferred material at PWA.

The economic benefits of isothermal forging relate to lower material usage and to reduced machining as a result of dimensional accuracy. Some of the potential savings are offset against the high costs of precision dies, however overall, a net saving is achieved. In the case of the nine IN-100 production parts for the U.S. Air Force F 100 engine (P5), a net saving of $16000 was reported by Pratt and Whitney. Die life was

reported to be good and usually sufficient to produce the required quantity of a given part. PWA report quantities of 300-400 parts without replacing dies, however the upper limits have not been determined.

According to Pratt and Whitney another attractive feature of superplastic forging is the ability to re-strike a part that shows defects such as incomplete die filling. In conventional forging these small finishing strains would lead to abnormal, and unacceptable, grain growth. In order to retain this re-strike capability, forging design has to be carefully considered. Pratt and Whitney also report that grain size in gatorized wheels can be controlled to some degree, to achieve high temperature properties in blade sections and lower temperature properties in the disc.

Discussion revealed much interest in non-destructive inspection of P/M products. According to Pratt and Whitney (P4A) sonic inspection was not a problem with their Astroloy disc forgings, and no changes in sonic standards or requirements are being considered. Similarly the gatorized parts of PWA-Florida (P5) could be inspected, with resolution to within 0.05" of the surface without attenuation. Their aim is inspect on as-forged material without using a surface cut.

In the case of the net-shape, sinter plus HIP parts of AVCO (P4B) the ability of the ceramic molds to produce intricate shapes was limited by ultrasonic inspection capability. AVCO explained that simpler shapes must sometimes be produced to allow inspection. AVCO reported that thermally induced micro-porosity (residual gas) cannot be detected non-destructively and therefore metallography on heat treated samples is the most satisfactory test.

Extensive discussion centered on the three priority items identified by Dr. Peterson in the Keynote Address, these were: a) the quality and cost of powders, b) improved methods of inspection, and c) alternative methods of compaction.

Concern was expressed over the trend to lower material consumption. An estimate of 2½ million pounds of powder for the 1980-81 market was considered insufficient to sustain two producers. Alternative markets for aerospace powders must therefore be developed to maintain business viability. Powder producers emphasized the relationship between cost and market volume, and confirmed that low cost powders will be available to bulk buyers. Superalloy powders can be produced by the Vacuum Atomization Process for less than $9/lb for volumes of about 50,000 lb/year, or less than $8/lb if scrap can be included in the input charge. Similarly a projection of less than $10/lb was given for either superalloy or titanium powder by the Rotating Electrode Process in quantities of 200,000-250,000 lb/year.

Recycling scrap was considered a viable route to lower powder costs. Problems were related to the cross contamination of alloys and to the presence of high oxygen contents in titanium turnings. AVCO reported the use of large volumes of scrap (up to 50% of the charge) by certain powder producers with no deterimental effects, however, internally generated scrap was recommended from the point of view of cleanliness. In general, scrap could be used for superalloy powders without major problems, since alloy chemistries are generically similar and adjustments can be made in the bath before atomization.

In the case of titanium, cross contamination of alloys, foreign object contamination and oxygen contamination were considered more serious problems. However, work by Alvac Teledyne Titanium, under U.S. Air Force sponsorship, was reported to have shown that titanium scrap can be recycled. In the Alvac process which uses a cold hearth skull melting furnace, heavy metal contamination from tool bits, and possibly titanium nitride particles, are trapped in the skull. Single melted or consumably melted bar from this process was considered suitable input material for the Rotating Electrode Process. At Pratt and Whitney Florida, 6-2-4-6 Ti alloy from this process, produced from 100% scrap, was confirmed to have oxygen within specification (0.15% Max), and this had been used to produce third stage discs. These discs are currently being machined and if qualified this will qualify the Teledyne process for reclaiming Ti scrap for rotating part quality. The scrap used in this case was probably premium material, retired parts rather than turnings.

According to other users, experience with P/M titanium material has shown it to be more tolerant to oxygen than ingot material, and therefore the arbitrary limits, typically 2000 ppm in current specifications, may eventually be raised. A great deal more information on fracture toughness and crack growth rates are needed to establish this.

In conclusion, it was suggested that many of the alternative powder production processes were very similar, and probably all capable of meeting volume requirements. Some processes may offer advantages in providing refining atmospheres, rather than simply maintaining the inherent purity of the charge. Others were felt to offer advantages in their ability to produce extra fine powder. This was considered important in providing ultra-high solidification rates (greater than 10^5°F/sec) required to hold hardening elements in solution. Future trends in powders may see precipitation hardening by phases other than gamma-prime, phases such as carbides or other intermetallic compounds. The controlled precipitation of these phases will require very fine, fully austenitic starting powders. Elsewhere, interest was expressed in the development of cold compactable powders, that would allow volume production of precision parts or performs by a press plus sinter route. This might circumvent the problems of low volume, cost, and limited dimensional accuracy in HIP parts.

SESSION III

RENE' 95 POWDER METALLURGY OPPORTUNITIES FOR GAS TURBINE APPLICATIONS

by

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INTRODUCTION

Rene' 95 is a wrought nickel-base alloy developed by General Electric under United States Air Force sponsorship. It is considered the strongest of the commercially available alloys for turbine and compressor disks, shafts, rotating seals, and related parts, operating at temperatures up to 1200F (Figure 1). To date, over 700,000 pounds of double vacuum melted Rene' 95 have been produced and used by General Electric for applications in various development and demonstrator engines. The alloy has been made commercially available to any source within the free world.

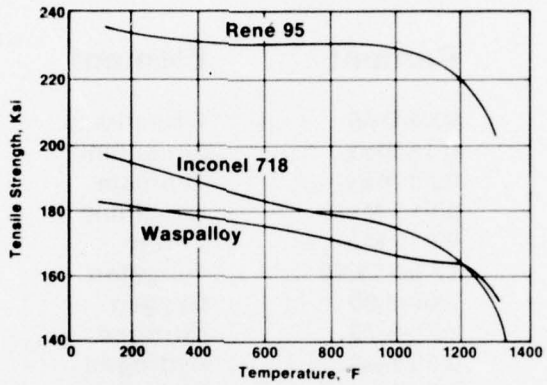


Fig.1 Tensile strength: René 95 vs other disk alloys

As would be expected, the very high strength of Rene' 95 poses challenges to conventional methods of producing gas turbine hardware parts. Optimum properties in forged Rene' 95 parts are associated with a duplex, necklaced structure, consisting of large, warm-worked grains surrounded by a necklace of fine, recrystallized grains (Figure 2). Achieving this structure in intricate gas turbine hardware requires stringent control of melting and forging practices. As with other wrought alloys, the total hardware cost is further escalated by less efficient material utilization and excessive machining labor which characterize the processing and manufacturing sequences required to produce engine parts from cast ingots. Figure 3, for example, illustrates a typical example of the multiple forging steps, machining operations and the rectilinear sonic envelopes required for the making of a compressor disk. Note that the finish-part weight amounts to only about 5% of the input weight.

Fig.2 Necklace structure in René 95

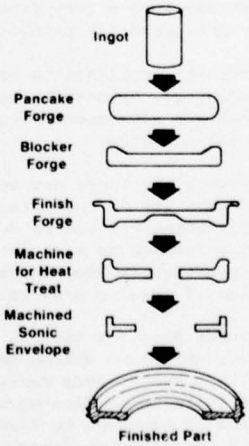


Fig.3 Typical processing sequence for cast and wrought René 95 disk – input/output weight ratio = 19:1

In order to take full advantage of high-strength alloys, fundamental changes capable of substantially lower cost of hardware production are desirable. To that end, General Electric initiated an intensive effort in powder metallurgy of Rene' 95 several years ago when the powder metallurgy approach emerged as a viable technology through the combined development of powder production and hot isostatic pressing (HIP). It is the purpose of this paper to briefly review the status of this effort and to indicate major directions in which powder metallurgy Rene' 95 is likely to make the most significant contribution.

POWDER METALLURGY PROCESSING

The Rene' 95 composition (Table 1) is uniquely suited to powder metallurgy processing. The balance between carbon content, M_6C carbide formers (W and Mo), and MC carbide formers (Cb and Ti) suppresses unwanted carbide precipitation at prior powder particle boundaries. Suppression of this phenomenon is considered a prerequisite for an acceptable powder metallurgy alloy.

TABLE 1

Chemical Composition of PM René 95

Element	Percent	Element	Percent
Carbon	0.04-0.09	Columbium	3.30-3.70
Manganese	0.15 Max.	Zirconium	0.03-0.07
Silicon	0.20 Max.	Titanium	2.30-2.70
Sulfur	0.015 Max.	Aluminum	3.30-3.70
Phosphorus	0.015 Max.	Boron	0.006-0.015
Chromium	12.00-14.00	Tungsten	3.30-3.70
Cobalt	7.00-9.00	Oxygen	0.010 Max.
Molybdenum	3.30-3.70	Nitrogen	0.005 Max.
Iron	0.50 Max.	Hydrogen	0.001 Max.
Tantalum	0.20 Max.	Nickel	Remainder

Numerous methods of powder making have been developed and used for years in the powder metallurgy industry. Unfortunately, the nature of nickel base superalloys makes none of the traditional techniques acceptable. The chemical complexity of superalloys virtually precludes the use of blended elemental powders. Water or nitrogen atomization used for other prealloyed powders is not feasible because of the possibility of contamination by such interstitial elements as oxygen, nitrogen, and hydrogen. Minimization of interstitial contamination is critical not only to maintain the very high chemical purity required in rotating part alloys for gas turbines but also to achieve full density and intimate interparticle bonding during HIP consolidation. Finally, due to the high cost of the starting materials for superalloys, powder manufacturing methods not capable of producing a high yield of usable powder are unacceptable.

In recent years, significant advances have been made in developing technically acceptable superalloy powder manufacturing techniques and installing production-sized facilities to produce superalloy powders. The argon atomization process can produce high purity prealloyed superalloy powders. In this process, elemental materials or prealloyed vacuum melted ingots are vacuum induction melted and a stream of the molten alloy is poured into a large chamber filled with argon gas. High pressure argon jets explode the molten stream into a very finely dispersed spray of powder particles. The argon atomization process is currently preferred for producing Rene' 95 powder.

The processes available to consolidate powders cover a wide spectrum, but one of these, HIP (Hot Isostatic Pressing), appears to be the most promising. HIP consolidation has been used to produce dense preforms for subsequent forging or rolling and offers the long range potential of producing near net, complex shapes.

High temperature inert gas autoclaves which are capable of consolidating Rene' 95 powders to full density fall into one of two categories; namely, high (> 5000 psi) and low pressure (< 5000 psi) units. High pressure autoclaves can be further subdivided into those using an integral furnace and those which use a separate furnace to heat the container of powder to the consolidation temperature. In all cases hydrostatic pressure is applied with an inert gas. Both high and low pressure autoclaves are suitable for production of forging preforms.

Significant progress made in recent years in the processing technologies of powder production, consolidation, and shape making has motivated large scale exploitation in reducing costs of expensive rotating parts. Two process variations which are prime candidates for accomplishing this exploitation are: (1) HIP followed by forging and heat treatment and (2) As-HIP followed by heat treatment alone. HIP plus forge is expected to find broader applications initially. With time, however, opportunities exist to eliminate forging completely for some parts. A key to the success of either approach is the development of technology to reproducibly manufacture precision shape contoured preforms.

PAYOFF

The potential for cost reduction has to date been the major driving force in powder metallurgy Rene' 95. Diagrammatically shown in Figure 4 are the reductions in material input weight and manufacturing operations when the typical disk shown in Figure 3 is produced by HIP + forge and As-HIP powder metallurgy processes, in comparison with conventional processing. The relative costs in production quantities associated with the three approaches in making rotating Rene' 95 hardware are illustrated in Figure 5. Although the cost benefits will vary with the particular part size and geometry, it is clear that the overall potential for cost reduction associated with the powder metallurgy approaches are very substantial, especially with the As-HIP process. In addition to the projected cost reductions, significant decreases in input raw material will be realized. These potentially amount to over 3,000 lbs/engine.

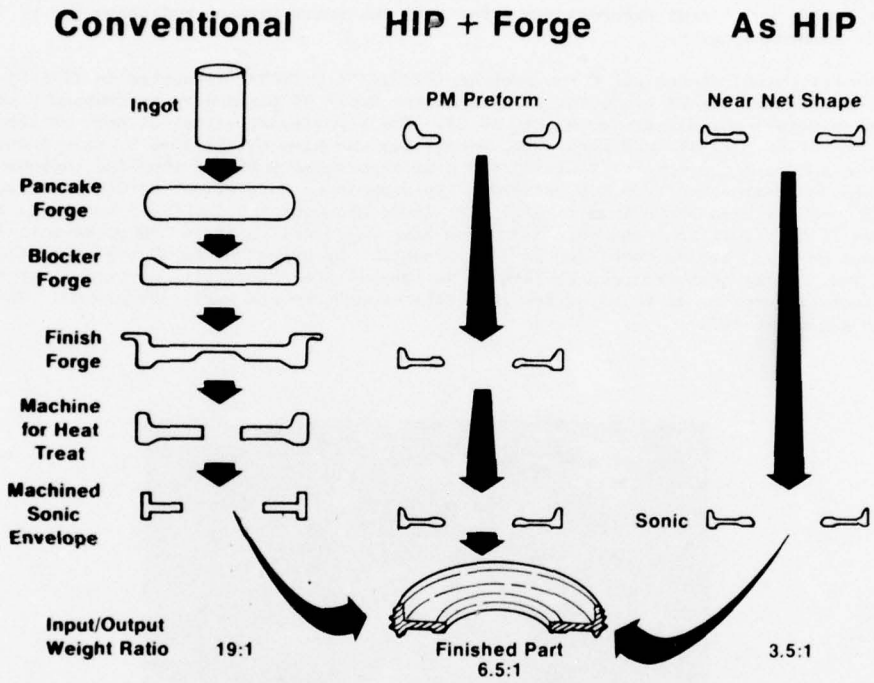


Fig.4 Comparison of manufacturing methods of René 95 disk

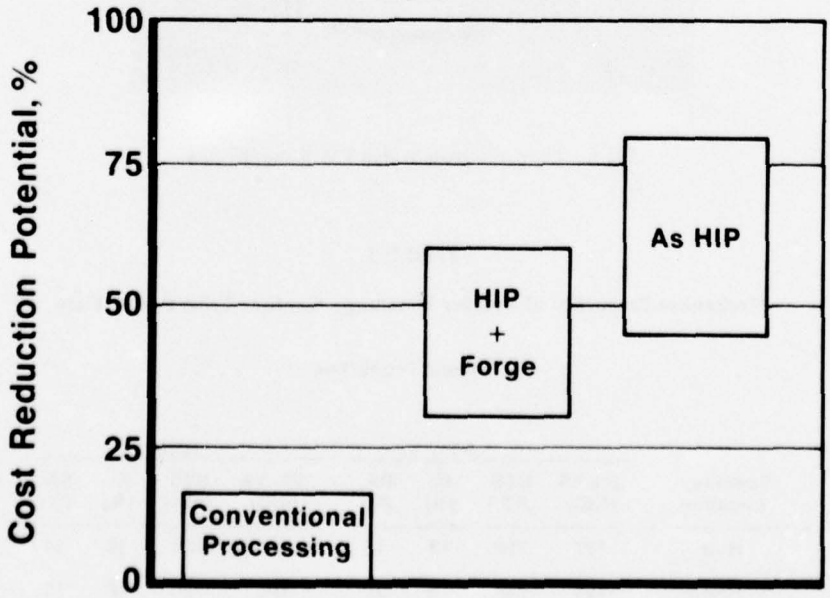


Fig.5 Cost reduction potential for a selected group of René 95 parts

STATUS

Initial work on powder metallurgy of Rene' 95 at General Electric focused on the processing of HIP Rene' 95 preforms using conventional metalworking techniques. This was soon followed by a United States Air Force contract (F33615-69-C-1825) to determine the purity of powder required for superalloy PM forgings. Two primary conclusions were derived from this study which had a major impact on PM Rene' 95 processing.

First, low carbon (0.08%) Rene' 95 has a significantly greater tolerance for oxygen contamination that the standard chemistry (0.15% C). This conclusion resulted in a reduction of the carbon level specified for the powder metallurgy version of Rene' 95 to a composition range of 0.04 to 0.09 weight percent. Second, large reductions were not required to achieve acceptable mechanical properties in powder metallurgy Rene' 95 forgings. This finding was in direct contrast to prior experience on cast + wrought material, where total reductions of 80 to 95% are required to produce the desired properties. The economic implications of this conclusion were significant, since the manufacture of close contour powder metallurgy forgings requiring only one small forging reduction (30 to 50%) appeared to be a viable production goal.

An additional United States Air Force program (F33615-71-C-1428), conducted in 1971-73, first demonstrated the feasibility of producing large diameter Rene' 95 components by contour cross rolling of shaped powder metallurgy HIP preforms (Figure 6). The significant effect of heat treatment and cooling rate on hot workability and mechanical properties was also established by this investigation. The excellent mechanical properties (Table 2) and microstructures achieved provided increased impetus toward producing PM components from HIP preforms. An example of this effort at General Electric is illustrated by the HPT disk shown in Figure 7. The large diameter (20 in. O.D.) and 4-in. bore thickness make it difficult to forge this disk from cast ingot due to increased propensity toward segregation and massive carbide formation in large ingots. By using PM-shaped preforms (Figure 7), disks of the same design were produced by forging in conventional dies, with mechanical properties fully equivalent or superior to those of the cast plus wrought counterparts (Figure 8). Two of these PM disks were engine tested.

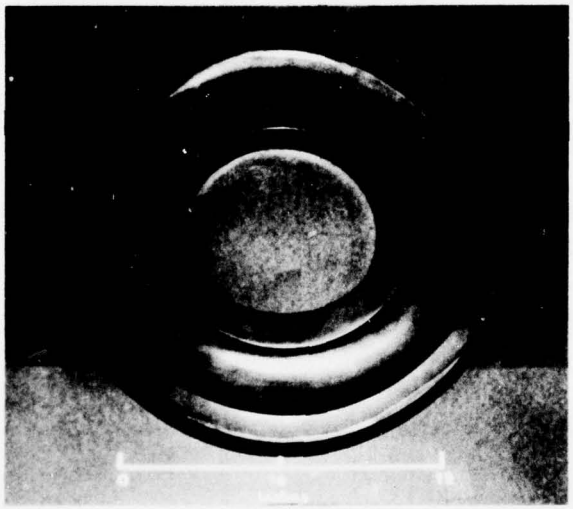


Fig.6 Contour cross rolled PM René 95 disk

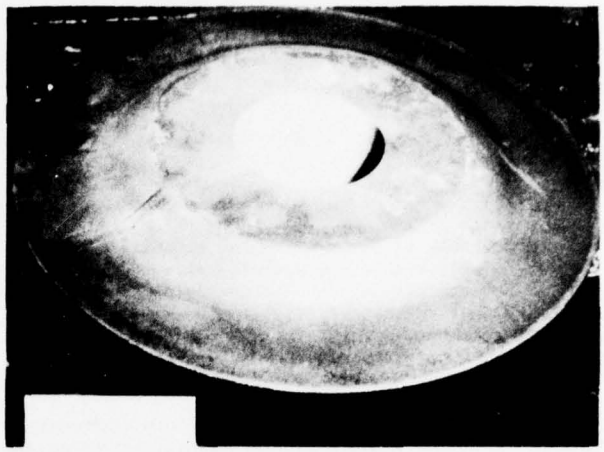
TABLE 2

Mechanical Properties of Powder Metallurgy Contour Cross-Rolled Plate

Tensile Properties

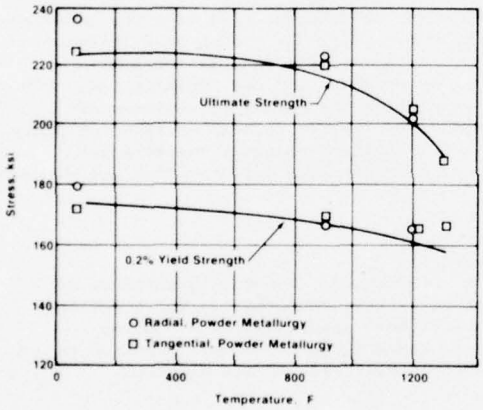
		RT				1200F				1200F/150 KSI Stress-Rupture	
Material	Specimen Location	.2% YS (KSI)	UTS (KSI)	El. (%)	RA. (%)	.2% YS (KSI)	UTS (KSI)	El. (%)	RA. (%)	Life (Hrs)	El. (%)
Contour Cross Rolled PM Disks	Hub	197	250	13	16	187	225	10	14	251	4.5
	Rim	199	254	15	22	189	226	10	15	126	5.5
Cast + Wrought Forgings	Specification	180	230	10	12	167	207	8	10	56	3.0

Forging Preform:
(15½" OD × 8½" thick)

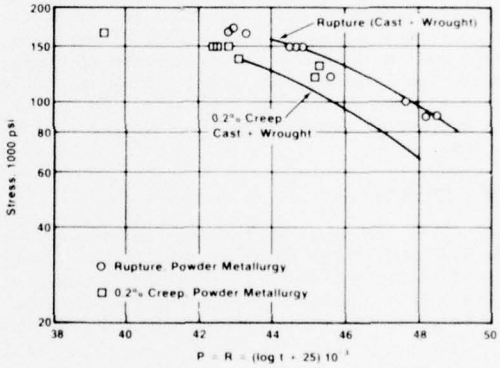


Finish Forged Disk:
(20" OD × 4" thick at bore)

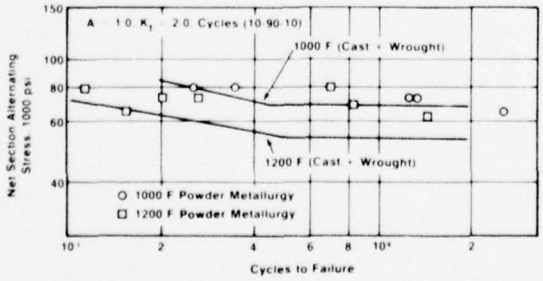
Fig.7 Full-scale René 95 forging preform and HPT disk forging



(a)



(b)



(c)

Fig.8 Tensile (a), creep/rupture (b), and systained peak LCF (c) properties of **PM** René 95 HPT disk, compared to cast + wrought forging

A series of outstanding accomplishments produced by HIP + conventionally formed PM Rene' 95 programs, along with rapidly rising raw material costs, logically led to additional cost reduction activities. The primary objectives of these activities are:

1) Reduce raw material input weight
2) Reduce number of metalworking operations
3) Reduce number of machining operations
4) Improve near net ultasonic inspection techniques

Development of hot die forging capabilities is a key requirement in this endeavor. The hot die systems greatly reduce the severity of surface chilling inherent in the conventional "cold" die system, thus permitting a reduction in protective material envelope around the part. In addition, shaped PM preform technology will eliminate large quantities of unnecessary input material and also reduce the number of metalworking operations required to produce the desired microstructure and mechanical properties. An integral part of the approach will be the development of an ultrasonic inspection technology capable of inspecting curved, near net shape components.

Development of the PM forging preform technology, including the ability to fabricate shaped components, is paving the way for even greater cost benefits from powder metallurgy processing. Initial studies of PM preforms indicated that mechanical properties competitive with those achieved in forgings could be attained in HIP and heat treated Rene' 95 compacts. Additional cost reduction by eliminating the forging operation from the manufacturing sequence of some PM parts is thus well within the realm of reality.

CHALLENGES & FUTURE EFFORTS

While great strides have been made in the past several years, further efforts are required to develop powder metallurgy processing of superalloys into a mature technology. Among the areas of interest are powder handling, shape-making reproducibility, productionized HIP facilities, and nondestructive inspection. Argon atomization is a well-established process of powder production. However, handling of loose powders up to and including the point of consolidation requires industry-wide, stringent quality control measures to avoid contamination. While the making of relatively simple shapes by the As-HIP process is now at hand, the capability of reproducibly fabricating large and complex shapes with the desired precision has yet to be developed and demonstrated. HIP facilities must be improved for production thruputs. In most instances today, cycle times are excessively long, circa eight hours. Finally, the elimination of harmful defects in rotating parts of turbine engines is a prime requisite. Development of NDE techniques uniquely suitable for inspecting complex, near-net shapes requires continuation of the intensive efforts which are already in progress.

CONCLUDING REMARKS

The utility of Rene' 95 is enhanced considerably by PM processing. The dual approaches of HIP + forging and As-HIP offer opportunities of very substantial cost reduction in the production of critical rotating components in aircraft engines. Some of these opportunities are being materialized today, while others require advances in several areas reviewed above. Based on recent progress there is every indication that the industries involved will rise to the occasion.

INVESTIGATIONS FOR MANUFACTURING TURBINE DISCS OF NI-BASE SUPERALLOYS BY POWDERMETALLURGY METHODS

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SUMMARY

In this paper the potential advantages arising from powder metallurgical production of turbine discs using different processing methods are discussed. Our research work included the following production methods:

- powder compressed by hot extrusion, disc shaping by forging in a conventional forging press;
- Argon-atomised powder compressed by hot isostatic pressing (HIP), disc shaping by forging in a conventional forging press;
- Argon-atomised powder, compression and simultaneous disc shaping by HIP

The influence of different steps is discussed, e.g. HIP-parameters, forging parameters and heat treatments on microstructure and results of tensile tests (from RT up to 800°C) creep rupture test (650°C and 730°C) and low cycle fatigue tests (test bars at RT and 600°C and spinned discs at RT). Parameters have been found for HIP in combination with thermomechanical post treatment which produce a microstructure in which the previous particle grain boundaries are not densely covered by carbides. This microstructure leads to mechanical and technological properties which appear to be adequate for the use of these PM-materials for turbine discs.

1. INTRODUCTION

Powder-metallurgy processes have been developed so far that the manufacture of critical components for aircraft engines by these processes can be considered. If those processes are used to produce turbine discs,such parts are expected to offer the following advantages over conventional forged ones:

- Improvement of the minimum values of the mechanical properties of components, by either raising the mean values using higher-alloyed materials, and/or by restricting the scatter-band of the mechanical properties in comparison to conventionally manufactured components.
- Reduction of the total costs of the components by saving material and machining work using shapes of the rough parts closely near the net shapes of finished parts.

The goal of the work reviewed here was to investigate the extent to which these expectations can be fulfilled.

On principle to produce discs by powder-metallurgy processes the following steps are needed (Cf. scheme in Fig. 1):

Melting of alloy
Powderization
Canning of powder
Consolidation of powder
Testing of blank properties after heat treatment
Machining to finished part

Various possibilities and alternatives are known for carrying out each of the basic steps shown in the scheme. Various combinations of these separate steps are possible to produce the finished component. The choice will depend on which of the objectives - improvement of properties or cost savings - is made predominant.

2. EXPERIMENTAL PROCEDURES

2.1. Selection of the process

The selection of process steps for the investigations described here was determined partly by the aimed-at goal and partly by the availability of test and production facilities. Three methods of production were adopted:

A. Argon atomization of the alloy and consolidation to 150 mm dia stock by hot extrusion pressing (procured from Federal Mogul, USA), forging to "pan cakes" in a drop-forging press.

B. Argon atomization of the alloy (at TEW: Thyssen Edelstahlwerke AG, Krefeld, BRD resp. KH: Kelsey Hayes, Detroid, USA) and hot isostatically pressing (hipping) in steel cans (at TEW) without any forging.

C. Argon atomization of the alloy (at TEW resp. KH), hipping in steel cans and forging in cannings into disc blanks in a forging press.

It was possible to adopt method A without delays, this serving to gather experience on the parameters for hot-forming powder-metallurgy prematerial. The handicap of this method is the restriction imposed on the disc diameter by the diameter of the available extrusion presses and by the height/cross section ratio in upsetting.

Cost savings can most likely be expected from method B, particular attention having to be given to ensuring the aimed-at properties. A hot isostatic press with an adequate useful diameter is a prerequisite, however.

Method C, like A, offers good prospects of achieving the mechanical properties aimed-at without close restrictions on the diameter but entails the disadvantage of higher production costs than B.

2.2. Selection of materials

Investigations were carried out with Udimet 700 and IN 100 as materials.

Mean compositions are:

	C	Al	Ti	Mo	Cr	Co	Fe	V	Ni
U 700	0,15	4,25	3,50	5,25	15,0	18,5	1,0 max.	-	Rem
IN 100	0,16	5,49	4,82	3,04	9,86	15,0	0,38	1,02	Rem

Discs of U 700 are already produced by conventional forging, so that data for comparison are available. This is not the case for IN 100, originally developed for cast turbine blades. The alloy composition, however, promises some advantages for high temperature applications. Relative to the nominal composition of IN 100 the carbon content was lowered.

- Udimet 700: TEW powder to 0.030 - 0.050 % C
  KH powder to 0.020 % C
- IN 100 : TEW powder to 0.030 - 0.050 % C
  KH powder to 0.020 % C

For canning, powder particles less than 80 mesh (particle diameter less than 180 microns) were screened out under vacuum or inert gas, the average tap density of all powders being about 65 %. The oxygen content of the powders, determined with samples prior to canning, was

- Udimet 700: TEW powder between 80 - 140 ppm O_2
- IN 100 : TEW powder between 80 - 200 ppm O_2
  KH powder 130 ppm O_2

2.3. Canning

After atomization, the powder was collected in containers which could be evacuated; particles with diameters less than 180 microns were separated under vacuum or inert gas, transferred from the container to the cans in a glove box respectively vacuum screener. After this, the filled cans were heated up during evacuation, and welded tightly. The can consisted of two deep-drawn pot sections of 18/8 steel welded together. Figure 2 shows how a can looked prior to (a) and after hipping (c). The slightest leaks, particularly at the weld, lead to puffing-up of the can during the dropping of the pressure after the hipping operation (b). This means in the case of large diameters, that the furnace of the press can be destroied. Checks for hermetic sealing and special pre-heating cycles virtually excluded such mishapes.

2.4. Selection of HIP parameters

Of particular importance is the determination of the right HIP parameters for processing methods B and C. Hot isostatically pressed specimen with micropores, particularly if these must be attributed to gas inclusions, cannot be healed out by subsequent hot working.

The best hipping temperatures for René 95, Udimet 700 and IN 100 powder materials were to be identified from a sequence of pressing temperatures between 1050 and 1300°C using the same pressing time of two hours (Figure 3). No connection was found between micropores and hipping temperature at the temperatures selected.

All indications are, that if there are a lot of micropores, they are caused by faulty canning or even by the use of powders with gas inclusions. If the HIP parameters are assessed on the basis of tensile tests at 535°C and 730°C, and of time-to-rupture tests at 650°C on smooth and notched specimens taken from hip-consolidated material up-set-forged into discs and heat treated, it is evident that high pressures produce fairly good results at all temperatures for a pressing time of 2 hours (Figure 3).

For the assessment of HIP parameters, posthipping microstructures must be taken as a guide: in the case of the alloy IN 100 with the lowered carbon content, no heavy carbide deposits do form on the previous powder particle boundaries at pressing temperatures lower than the γ-solution temperature (Fig. 3). The previous powder particle boundaries are still visible in the consolidated material using press-temperatures up to about 1050°C. In this temperature region one can also find isolated micropores neighbouring coarser particles. Using press temperatures above the γ'-solution temperature (up to about 1220°C) complete recristallisation occures during the consolidation process. In some cases, however, the previous particle boundaries are limiting the grain growth. Pressing at still higher temperatures result in a microstructure showing features of eutectic γ/γ' -formation similar to incipient melting in cast specimen.

In the tests thereafter, HIP temperatures, which might lead to incipient melting were avoided since the effect of such structural changes was not known, especially with regard to fatigue life.

The otherwise untreated, hipped Udimet 700 specimens of the first test series, at hipping temperatures above 1160°C, all showed heavy deposits of carbides on what once were the boundaries of the powder particles.

Various hipping cycles (Fig. 4) were then employed in an attempt to achieve a microstructure which after hipping would be free from heavy precipitations on the former powder particles. Hipping cycle IV gave this result, the former powder particle boundaries however remained in the microstructure in the form of grain boundaries. Cycle V again produced carbide precipitations on the boundaries of the powder particles, but these did not form so densely closed layers as obtained in previous tests. Cycles I, II and III employed additional pre-heattreatments achieving a structure which would be less susceptible to MC precipitation during the HIP process.

Fig. 5 illustrates the results on microstructure obtained by this measure. Also from the aspect of attainable mechanical characteristics, double pressing (process III) is to be preferred to single pressing (Processes I and II).

2.5. Hot Forming

Disc-forming tests on extruded PM prematerial (method A) showed that hot-forming of such highly alloyed materials requires protective cannings. This cans have to keep the temperature nearly constant and roughly consistent over the entire billet and its surfaces. For method C, the protective cannings normally include the canning materials from HIP consolidation and additionally a canning of structural steel (Fig. 6). Using starting temperatures of 1170°, 1160°, 1150° and 1120° for PM Udimet 700, and 1200°, 1180°, 1160° and 1150° for PM IN 100; the upset operation on a forging press was done successfully in several steps or else in one up to a total deformation of about 70 %. From the HIP consolidated prematerial of about 130 mm in diameter and 180 mm in height, flat discs were made approximately 270 mm in diameter and 45 to 50 mm high. Photographs of some discs after hot forming are shown in Fig. 7. Of the 20 discs made of this size, no more than two exhibited tears, which will have to be viewed in connection with leakages after hot isostatic consolidation, however. Hot forming does only deform the material; it will not remedy microflaws coming as a result of remaining gas between the powder particles enclosed during consolidation.

2.6. Heat Treatment

For Udimet 700 material produced by the normal melting process, the commonly recommended heat treatment is:

1180°C 4hr/Ac + 1080°C 4hr/Ac + 850°C 24hr/Ac + 760°C 16hr/Ac.

For PM-prematerial produced per method A,

1120°C 4 hr/Ac + 850°C 24 hr/Ac + 760°C 16 hr/Ac

has given greater benefit in terms of the 0.2-yield strength up to 800°C proof temperature creep rupture strength at 630° and 730°C under high load.

For HIP consolidated material without any forging (method B), we feel that heat treatment depending on HIP-parameters still needs perfecting. Discs according method C (HIP+ hot forming), when heat treated by the second method, have already shown good results.

No established heat treatment processes for conventionally produced materials of IN 100 is known. For PM-IN 100 hot formed material, the following treatment has shown good results: 1220°C 2 hr/Ac + 890°C 24 hr/Ac + 760°C 4 hr/Ac. Before a selection of a process useful for the turbine disc application, can be made the heat treatment will still need some optimisation work.

3. RESULTS

3.1. Results from Discs from hot extruded prematerial U 700 (Method A)

Specimens, taken from discs produced by upsetting hot extruded prematerial and conventionally heat treated (HT I: 1180°C 4 hr/Ac + 1080°C 4 hr/Ac + 850°C 24 hr/Ac + 760°C 16 hr/Ac), showed at elevated temperature tensile test results (Fig. 8) at or just below the mean values for conventionally produced material. Using a modified heat treatment (HT II: 1120°C 4 hr/Ac + 850°C 24 hr/Ac + 760°C 16 hr/Ac), the characteristics for the 0.2 limit were improved considerably especially for temperatures below 760°C.

No relationship between properties and position resp. orientation of the specimen in the disc would seem to be indicated.

The stress rupture performance at 630°C surpasses the data for conventional material expected by extrapolation of Larson-Miller (cf. Table 1). At 730°C the expected times at rupture are not fully achieved. For both temperatures, heat treatment HT II has afforded an advantage. Times to rupture of notched specimens are normally longer than those of plain specimens. Considering the usual scatter band for stress rupture test results, the orientation of the specimen in the disc has no effect on stress rupture strength.

Method A produces turbine discs in PM-Udimet 700 with moderately improved properties over conventionally made discs.

3.2. Results from unforged, HIP consolidated Disc Blanks (Method B)

Assessment of the material got by the hot pressing method employed here goes by the following criteria:

- micropores
- mechanical properties in only hipped condition
- mechanical properties after heat treatment

A reproducible relationship of micropores to the hot pressing parameters would not seem to be indicated. While certain HIP parameters would normally produce material free from micropores, some of the pressings showed considerable microporosity at the same HIP parameters. The survival of micropores in the structure, then, will have to be attributed to canning methods and the selection of powder.

The mechanical properties (Fig. 9) in the only hipped condition (without heat treatment) vary with the γ'-distribution and -size achieved by the thermal treatment during pressing and by the "bonding" achieved between the former powder particles. The 0.2 yield strength (Fig. 9) at test temperatures up to about 700°C greatly reflects the γ'-size achieved by the thermal exposure. At elevated temperatures the "bonding" of the particles is of greater importance with regard to the 0.2 yield strength and the stress rupture performance. The examples shown in Fig. 9, indicate that the two stage HIP-process will give the best bonding.

This holds true also for heat treated HIP specimens (Fig. 10), where at test temperatures above 700°C the double-pressed specimens prove to be superior, after both heat treatments, to the singlepressed specimens. Difficulties are encountered at temperatures above 700°C in the matter of stress rupture performance. The material is very sensitive to abrupt transitionsin cross section such as they occur between the grip end and the gauge length. Notch sensitivity becomes apparent also from the time to rupture of notched specimens:

HIP pattern			Heat treatment	Test temp. °C	Load N/mm²	t_r h	δ %	ψ %
IV	1000°C	500 bar 5 hr	HT II	650	1030	8.3	3.6	9.7
			HT II	650	1030	6.1	notched	
V	1050°C	1900 bar 2 hr						
	+1170°C	1900 bar 2 hr	HT II	650	981	43.7	2.0	8.5
			HT II	650	981	16.7	2.0	7.8
			HT II	650	981	27.4	3.2	11.2
			HT II	650	981	48.2	4.4	11.5
			HT II	650	981	28.6	notched	
			HT II	650	981	30.4	notched	

In our investigation Method B dit not yet give discs which would be sure to provide all desired characteristics reproducibly.

3.3. Results from Discs from forged HIP-prematerial (Method C)

Assessment of the re-formed discs went by

- microstructure
- variation of tensile test characteristics with test temperature
- fatigue performance (LCF at RT and 600°C)
- creep rupture strength

The microstructure is assessed from the aspects of microporosity and grain structure. Microporosity comes with the hipped material and is not healed out by post HIP hot forming, especially not if it is attributed to gas inclusions. The grain structure is subject to change by hot forming, especially when after HIP consolidation particle boundaries are not densily covered by carbide precipitations. (Fig. 11).

The production parameters for the material to investigate the not-hot-formed condition (U 11) were the following:

980°C 15 hr + 980°C 3 hr 1780 bar + 1120°C 3 hr 1920 bar

powder manufacturer: TEW, O_2 content: 105 ppm,

size after hipping: 130 mm dia and 180 high

Disc U 15 was hot formed from prematerial with nearly identical HIP-parameters as pressing U 11 (pressing cycle III)

980°C 15 hr + 980°C 3 hr 1930 bar + 1120°C 3 hr 1950 bar

powder manufacturer: TEW, O_2 content: 140 ppm

hot formed on a forging press in bicanning at

1150°C in two upset steps of 50 % and 20 %.

Final size of the pancake: 275 mm dia and 50 mm high.

Fig. 11 shows, that the microstructure resulting from heat treatment strongly depends on the microstructure entering the heat treatment. In tensile tests at 600°C and 700°C on heat treated specimen pressing U 11 shows higher 0.2 yield strength but poorer rupture strength and ductility compared with U 15.

One disc (U 3) was investigated in a total cut; the production parameters were nearly the same as with disc U 15.

980°C 15 hr + 980°C 3 hr 1870 bar + 1120°C 3 hr 1960 bar

powder manufacturer: KH, O_2-content: 150 ppm

hot formed on a forging press in bicanning

at 1160°C with steps of 30 %, 20 % and 25 % deformation.

Final size: 270 mm dia and 45 mm high.

The microstructure achieved after hot forming and after heat treatment

1120°C 4 hr/Ac + 850°C 24 hr/Ac + 760°C 16 hr/Ac

is given in Fig. 12.

On this disc, 0.2 yield strength and rupture strength up to proof temperatures of 700°C (Fig. 13) range above the mean values of conventionally manufactured material. Results obtained from this material in fatigue tests indicate a notable independence of specimen orientation in the disc. The obtained LCF test results were slightly superior to the characteristics obtained for discs in Waspaloy. It is merely the time to rupture of 730°C of radial specimens which does not meet expectations.

Test temp. °C	Load N/mm²	Time to rupture h	Elongation %	Reduction of area %
650	932	17.7	6.0	10.5
	932	26.5	notched	
	882	31.8	4.5	rupture at transition
730	882	35.5	notched	
	588	23.4	3.5	rupture at transition
	588	4.5	notched	
	539	22.0	3.5	rupture at transition
	539	1.5	notched	

It appears that discs of hipped material exhibit notch sensitivity even if they are hot formed.

By method C also discs of IN 100 were produced. The microstructure of two of these discs is given in Fig. 14. The difference in their manufacturing parameters is, one, in different temperatures at the second step of hot isostatic pressing in the course of double pressing and, two, in different forging steps: three steps for disc IN 19 and two steps for disc IN 30. These differences produced different grain sizes at the same heat treatment (Fig. 14). On disc IN 30, remainders of former powder particle boundaries with precipitation were still noted.

The characteristics for these two discs (Fig. 15) obtained from tensile testing up to proftemperatures of 700°C and from LCF testing at 600°C differ little one from the other. It is only at 800°C that disc IN 30 gives superior 0.2 yield strength and rupture performance. No differences were noted between radial and tangential specimens.

Present results from stress rupture testing at 730°C on disc IN 30 still give no indication of the effect the orientation of the test specimen may have on the data at rupture:

Temp. °C	Load kp/mm²	Time at rupture h	Elongation %	Reduction in area %	Orientation of specimen
730	60	162.6	6.0	9.6	tangential
		31.9	7.2	12.3	radial
	55	370.3	1.2	8.3	tangential
		416.9	3.6	8.2	radial

The comparatively low deformation at rupture, which also is obtained in tensile tests above 730°C, is a notable indication.

3.4. Spinning test

In addition to the common testing methods using test bars for tensile test, creep test and fatigue test, discs produced from PM-material have been spun. Two types of tests have been used for getting a better knowledge of the service performance of these materials utilizing the uneven stress condition of a rotating disc. The discs show almost the some strength along the radial direction and have got a bore in the centre, which acts as a notch. The diameter of the test disc is about 120 mm.

At test type A at room temperature the revolutions per minute of the disc are increased to certain steps. The disc is stopped then and the gained enlargement of diameter is recorded. In Fig. 16 this parameter is drawn against the revolutions per minute for two discs from only hipped PM-U 700 (U 11) and for one disc from hipped and additionally forged PM-U 700 (U 15). By this the results of tensile test are confirmed qualitatively: the disc, produced from the only-hipped material U 11 which shows the higher yield strength compared with U 15, is less deformed than the disc from post forged material at the same revolutions per minute. The spinning was not continued until burst of the discs, since these were needed for further LCF-tests. The number of r.p.m. at burst would show whether the higher ultimate strength of postforged U 15-material is confirmed also.

In test type B also at room temperature the revolutions per minute of the disc are increased within about three minutes to a fixed maximum value and are then reduced to zero again. This cycle is repeated until the first crack appears respectively the disc bursts. Fig. 17 shows this number of cycles drawn against the maximum stress in the

central notch of the disc at maximum r.p.m. For comparison some results are given for discs of Waspaloy and IN 718 materials which are quite similar to U 700. With 8400 cycles to fracture the only hipped U 11-PM-material nearly reaches the values of the well established conventional materials. At 5000 cycles the last crack inspection was done and no cracks have been found in the disc.

4. DISCUSSION AND CONCLUSIONS

In this investigation the goal of improoved strength could be reached for tensile properties in the temperature range up to 700°C for both alloys U 700 and IN 100.

Above this temperature the tensile strengths of PM-U-700 just meet the properties of conventionally produced material or even drop to lower values. There is almost no difference between the properties obtained according to the different production methods A, B and C for PM-U-700 in the temperature region up to 700°C, but above this temperature method A gives slightly better values then method B and C.

Similar results are found for PM-IN 100: at temperatures above 700°C to 750°C the tensile properties drop considerably below that of the cast material.

For both alloys this behaviour might be explained by insufficient strength at high temperature of the boundaries between former powder particles. The better properties of extruded PM-material (method A) show that some improvement could be achieved for HIP-material also. Another hint for attainable improvements with regard to powder particle boundaries is given by the good performance of double pressing in method B and C, which aims strictly at influencing the structure of these boundaries.

The notch sensivity, found in testing of bars produced according to method B, did not show a negative influence on the performance of the disc in spinning test. In this LCF-test the number of cycles to fracture is well comparable to values gained from conventionally produced disc material like Waspaloy and IN 718. Further tests will show how reproducible the results are.

In creep test the longest times to rupture were achieved with method-A-material. The reason for that seems to be found in choise and handling of the powder, since method C-material was forged also and the times to rupture are not satisfactory in this case. Comparing the not extruded materials, IN 100 shows the best times to rupture under similar test conditions. In general the creep performance of PM-material still needs a lot of improvement.

As a result of this investigation it shall be pointed out, that it seems to be possible to achieve by powdermetallurgical methods, the material properties demanded for critical engine parts like turbine discs. Further investigations aiming at performing the demanded material properties at full scale and reliably, should be guided by the following points:

- development of modified alloys for PM-purposes, which do not tend to form precipitation layers on the surface of powder particles.
- Pretreatment of the powder for getting an optimum structure within the particles before pressing with regard to stronger boundaries in the pressing.
- reproducible good degasing and tight encapsulation of the powder.
- optimisation of HIP-parameters.
- optimisation of heat treatment.

Table 1 Stress rupture properties of PM U 700 according to production method A

Temp. °C	stress N/mm²	heat treatment	orientation and kind of specimen		time to rupture h	elongation %	R.A. %
650	981	HT I	R	s	17,3	23,2	10,0
650	981	HT II	R	s	30,6	22,0	29,3
650	981	HT II	R	s	111,0	21,0	26,6
650	981	HT II	R	n	570,4	-	-
650	981	HT II	T	s	29,8	22,4	29,6
650	981	HT II	A	n	26,3	13,2	26,3
650	981	HT II	A	s	64,4	15,2	22,5
650	981	HT II	A	n	45,7	-	-
650	932	HT I	R	s	69,0	10,5	15,5
650	932	HT I	R	n	97,4	-	-
650	932	HT I	A	s	38,8	12,4	21,9
650	932	HT I	A	n	87,7	-	-
730	540	HT I	R	s	314,9	8,4	14,0
730	540	HT I	R	s	245,1	5,6	12,0
730	540	HT I	R	n	374,4	-	-
730	540	HT I	T	n	514,4	-	-
730	540	HT I	A	n	338,8	-	-
730	540	HT I	A	n	589,2	-	-
730	540	HT I	A	n	417,8	-	-
730	540	HT I	R	s	198,7	8,0	18,3
730	540	HT I	T	s	341,5	9,6	10,3
730	540	HT II	R	n	791,8	-	-
730	540	HT II	A	s	495,7	8,8	18,4
730	540	HT II	R	s	331	-	-
730	540	HT II	R	n	1244,3	-	-
730	540	HT II	A	s	217,2	7,2	13,0

Properties of conventional forged U 700 are according to Larson-Miller-diagram:

650°C 981 N/mm²: 0,3 hr
932 N/mm²: 1,8 hr
730°C 540 N/mm²: 844,5

A: axial
R: radial
T: tangential
} orientation of specimen in disc

s: smooth specimen
n: notched specimen

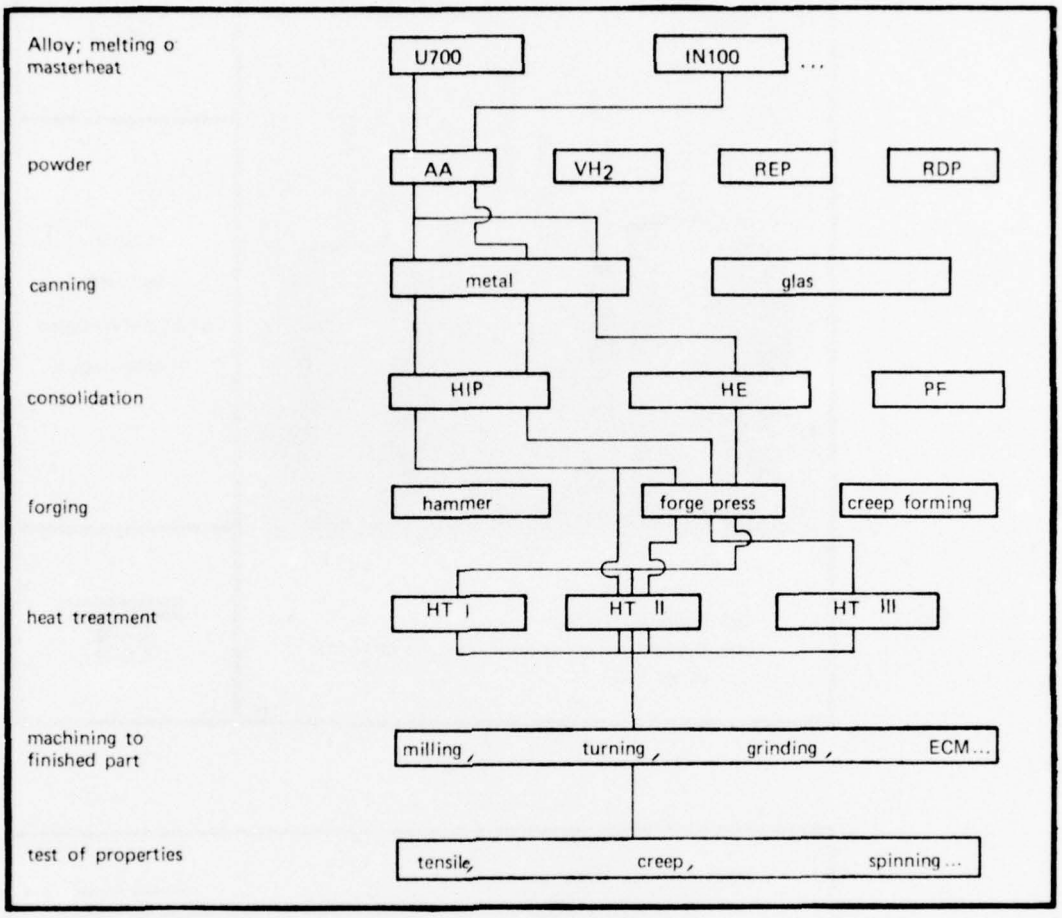
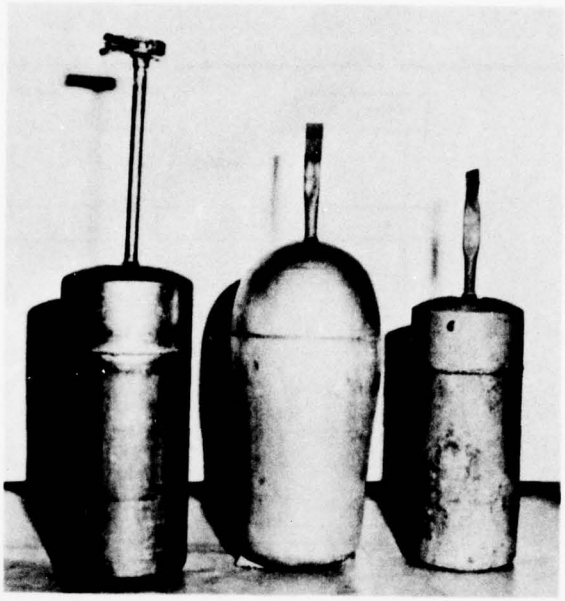


Fig. 1. Scheme of possible production steps for a turbine disc of PM-material.

AA	argon atomisation
VH_2	vacuum atomisation by H_2
REP	rotating electrode } powderisation
RDP	rotating disc } powderisation
HIP	hot isostatic pressing
HE	hot extrusion
PF	powder forging
HTI..III	different heat treatments



a) b) c)

a) before filling
b) puffed up after HIP due to defects
c) after HIP

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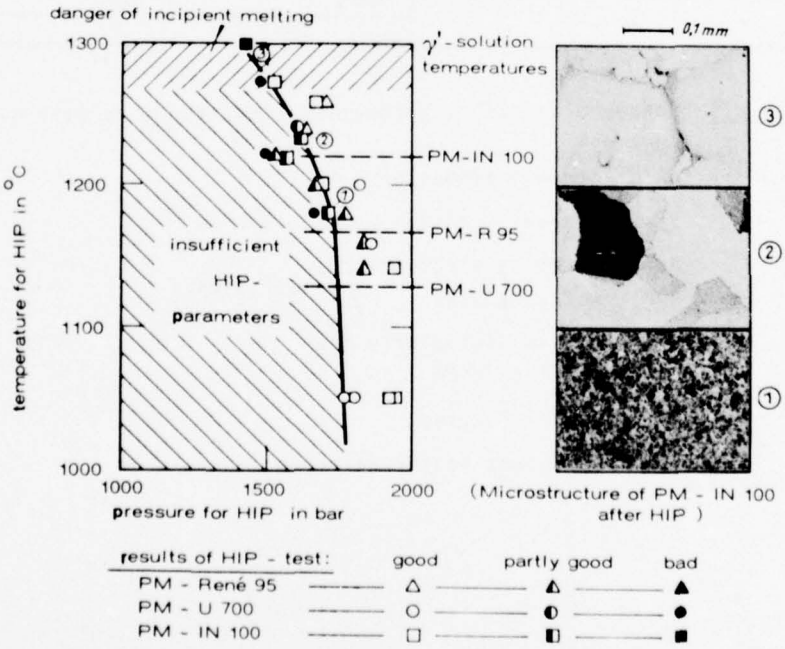
FI
Krefeld

Cans
for HIP
of PM-Ni-base
superalloys

Fig: 2



FI
Krefeld

Test- results for optimizing
HIP- parameters

Fig: 3

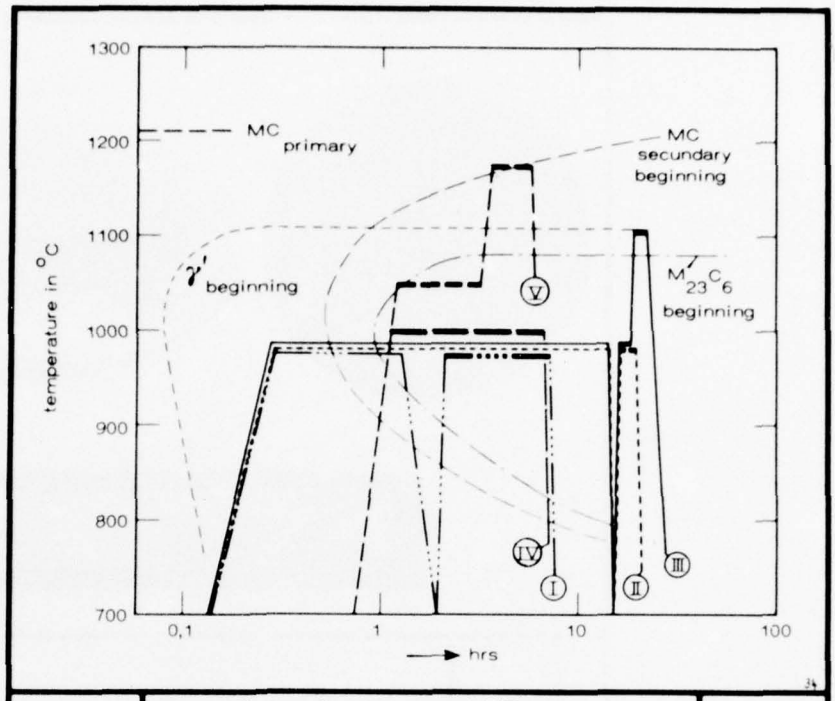
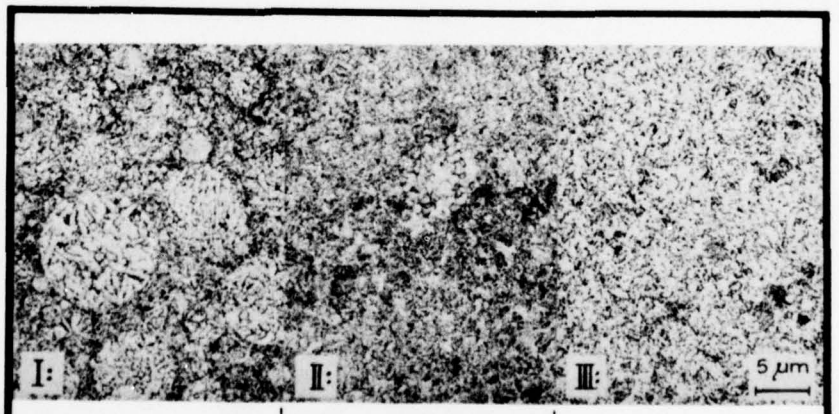


Fig: 4

FI Krefeld	Temperature exposure at HIP-process compared with TTT-diagramm for Udimet 700 (scheme) — with pressure / without pressure	

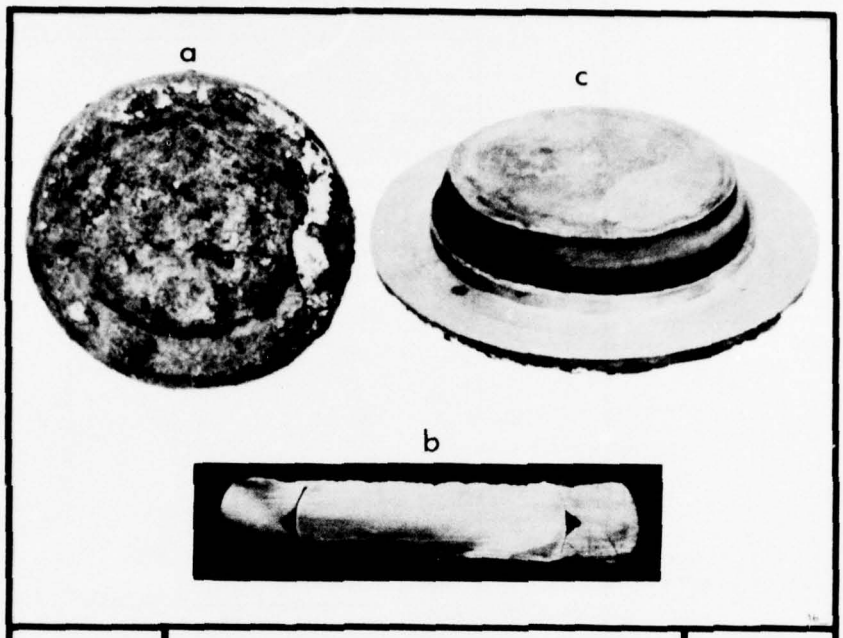


I:	II:	III:
980°C 1h + 980°C 5hrs 1919 bar	980°C 15hrs + 980°C 5hrs 1880 bar	980°C 15hrs + 980°C 3hrs 1945 bar + 1120°C 3hrs 1950 bar

34

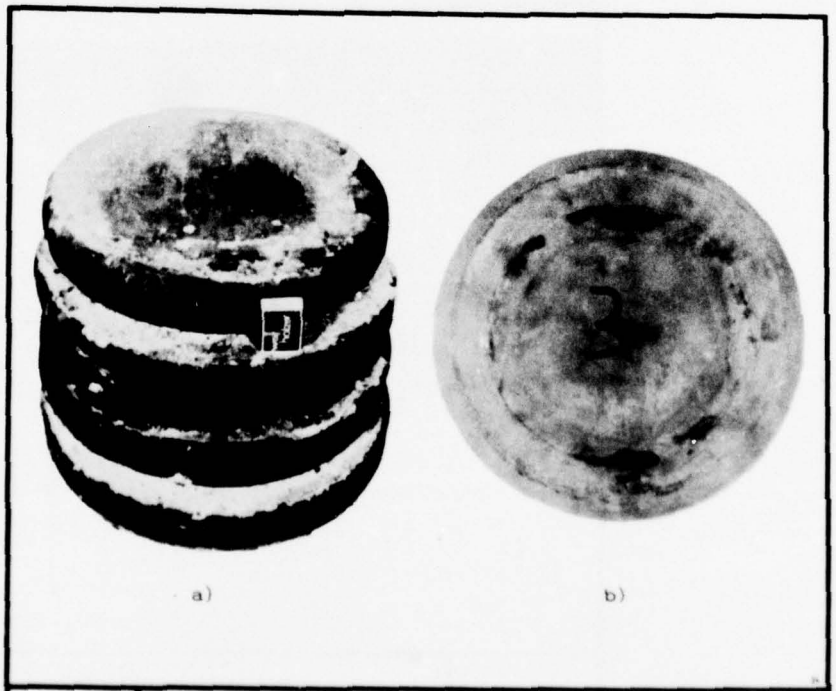
FI Krefeld	Influence of HIP-parameters on microstructure of PM-Udimet 700	

Fig: 5



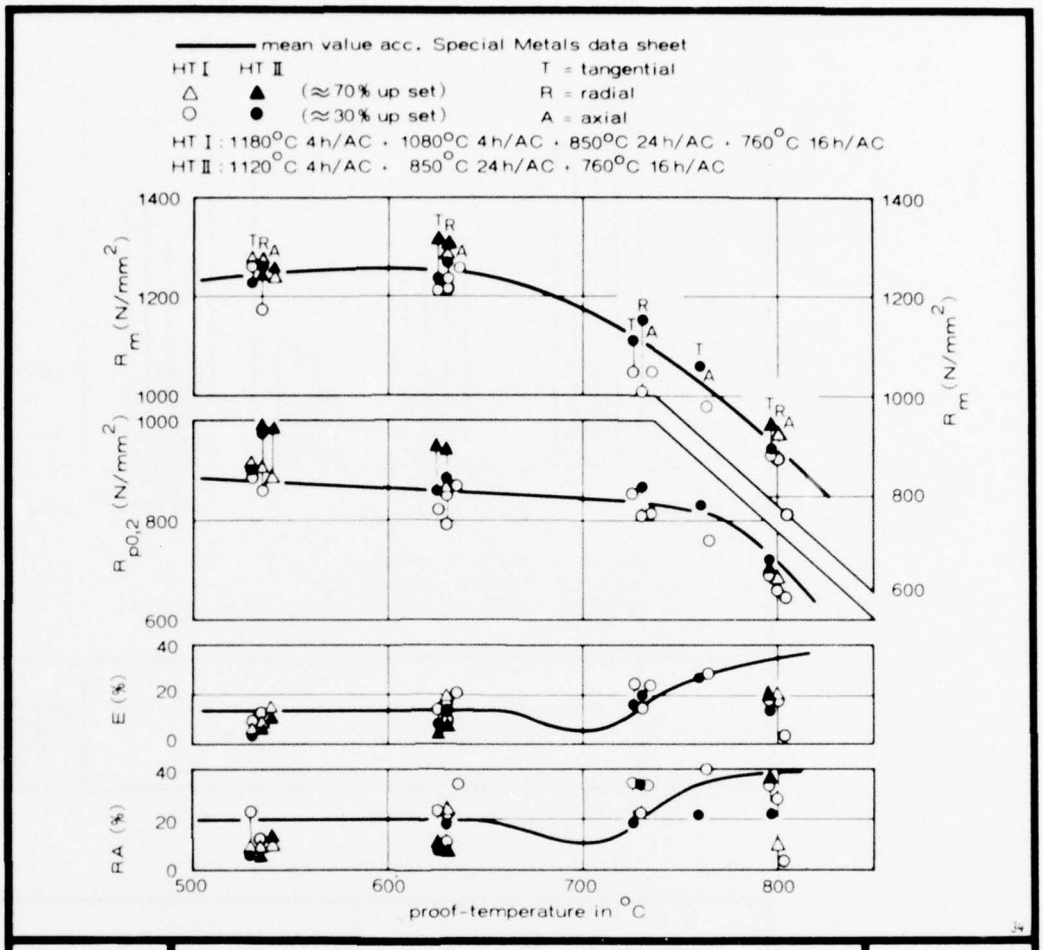
FI Krefeld	Upset forged discs a) in full canning b) cross-section c) canning partly removed	

Fig:6



FI Krefeld	Discs of PM Udimet 700 after forging a) in canning b) de-canned	

Fig:7



FI Krefeld	Result of tensile test on specimen of PM-Udimet 700 disc (hot extruded and forged)	

Fig: 8

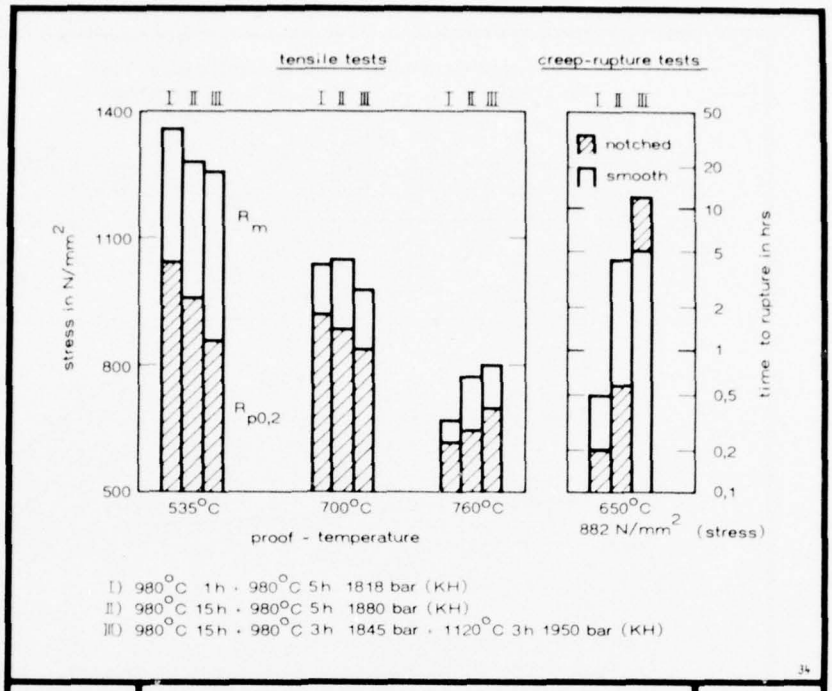


Fig: 9

FI Krefeld	Mechanical properties of hipped PM Udimet 700 without any post heat treatment	

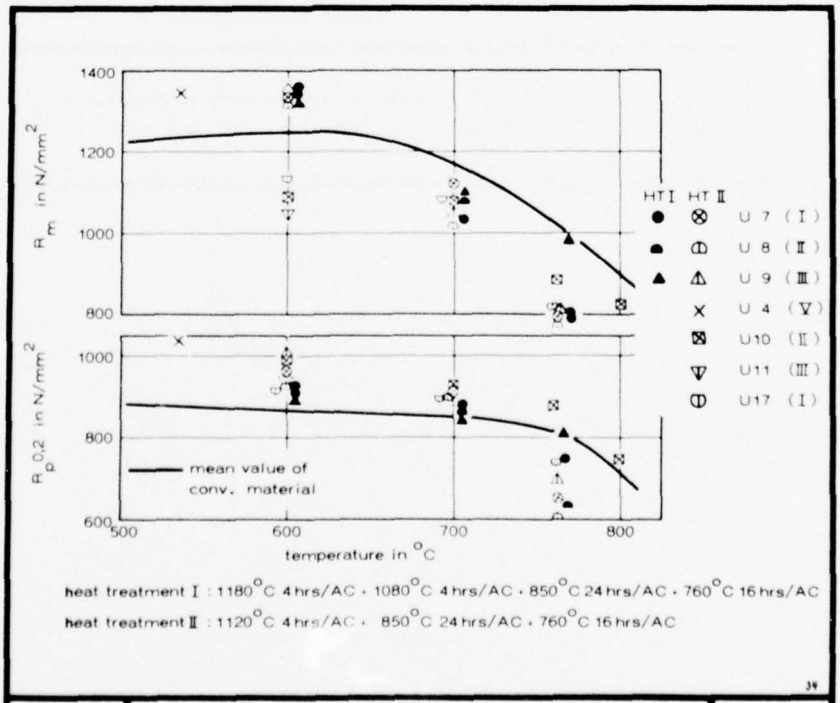


Fig: 10

FI Krefeld	Results of tensile tests on hipped and heat treated PM Udimet 700	

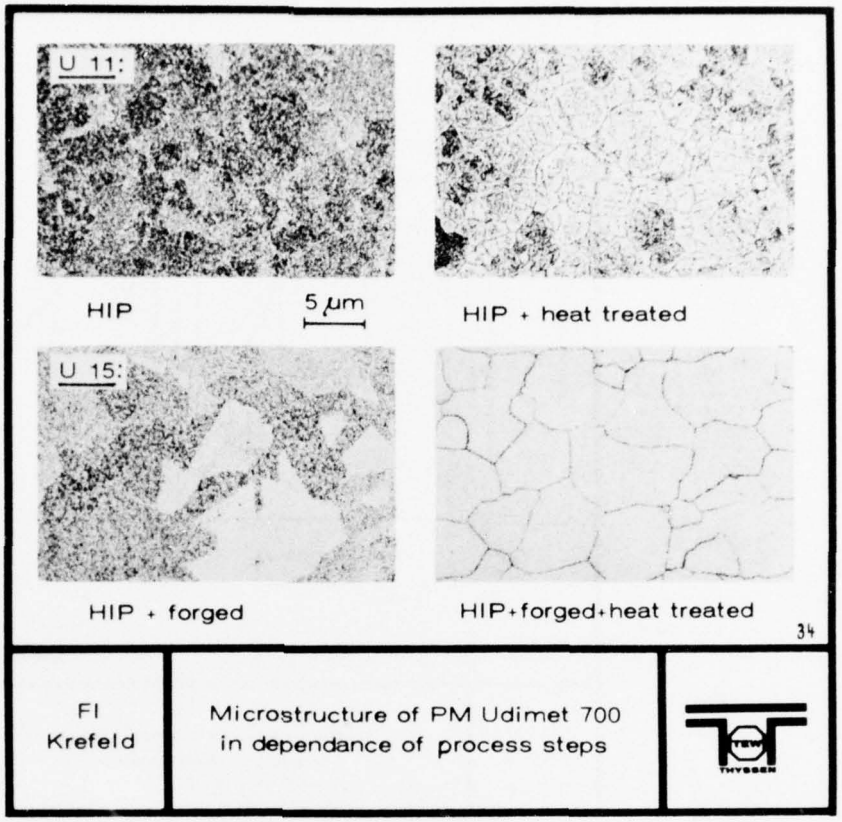

U 11:
HIP
5 µm
HIP + heat treated
U 15:
HIP + forged
HIP+forged+heat treated
34
FI
Krefeld
Microstructure of PM Udimet 700
in dependance of process steps

Fig: 11

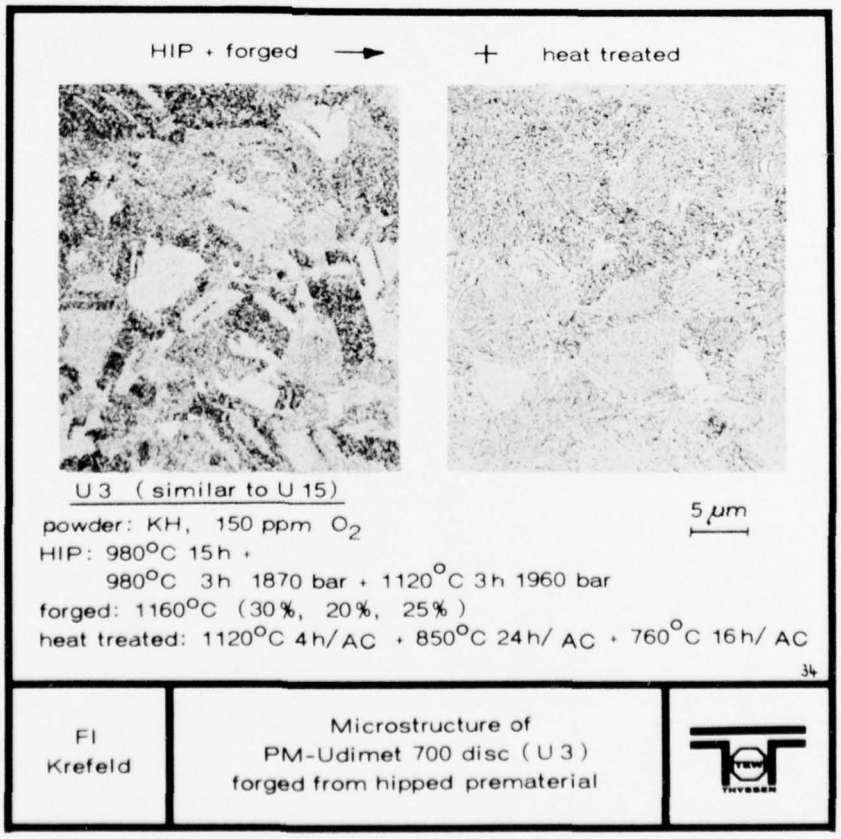

HIP + forged
+ heat treated
U 3 (similar to U 15)
5 µm
powder: KH, 150 ppm O_2
HIP: 980°C 15h +
980°C 3h 1870 bar + 1120°C 3h 1960 bar
forged: 1160°C (30%, 20%, 25%)
heat treated: 1120°C 4h/AC + 850°C 24h/AC + 760°C 16h/AC
34
FI
Krefeld
Microstructure of
PM-Udimet 700 disc (U 3)
forged from hipped prematerial

Fig: 12

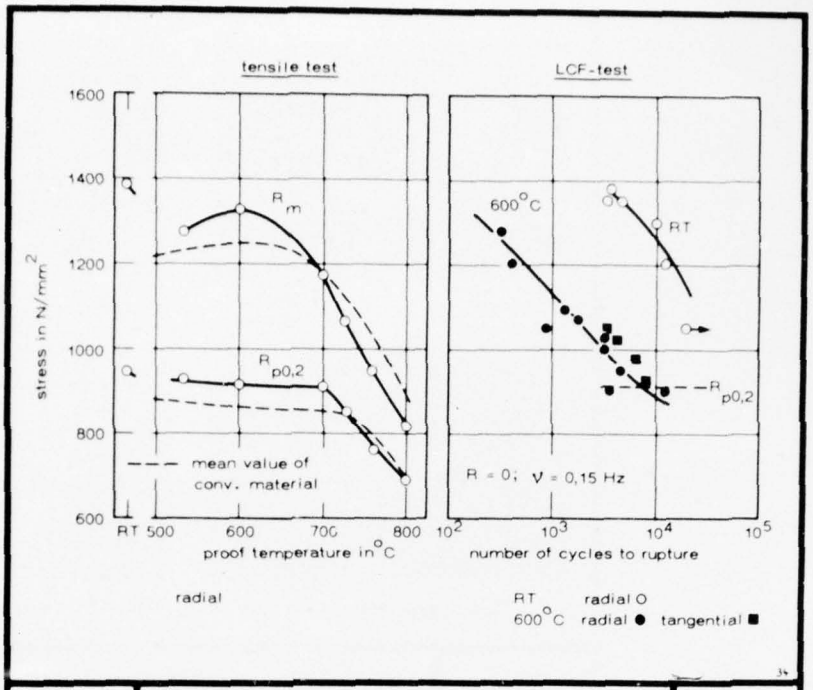
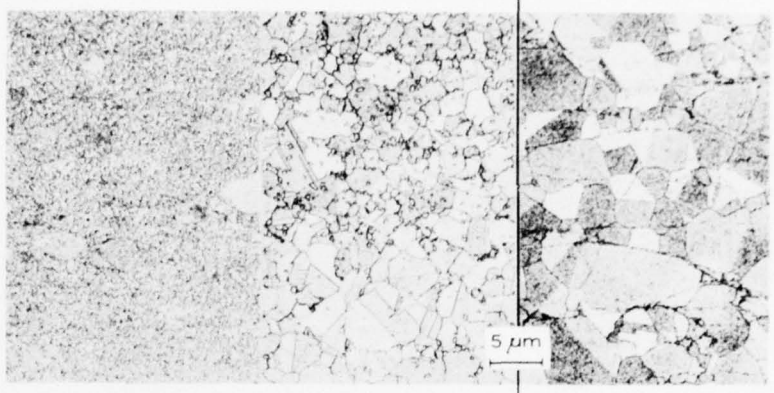


Fig: 13

FI Krefeld	Results of a PM Udimet 700 disc(U3) forged of HIP-prematerial	



	IN 19	IN 30
powder:	TEW 100 ppm O_2	TEW 125 ppm O_2
HIP :	980°C 15 hrs	980°C 15 hrs
	+ 980°C 3 hrs 1870 bar	+ 980°C 3 hrs 1860 bar
	+ 1100°C 3 hrs 1950 bar	+ 1160°C 3 hrs 1950 bar
forged:	1160°C (30%, 20%, 25%)	1160°C (50%, 20%)

heat treated: 1220°C 2 hrs/AC + 890°C 24 hrs/AC + 760°C 24 hrs/AC

34

Fig: 14

FI Krefeld

Microstructure of PM-IN 100 disc forged from HIP-prematerial

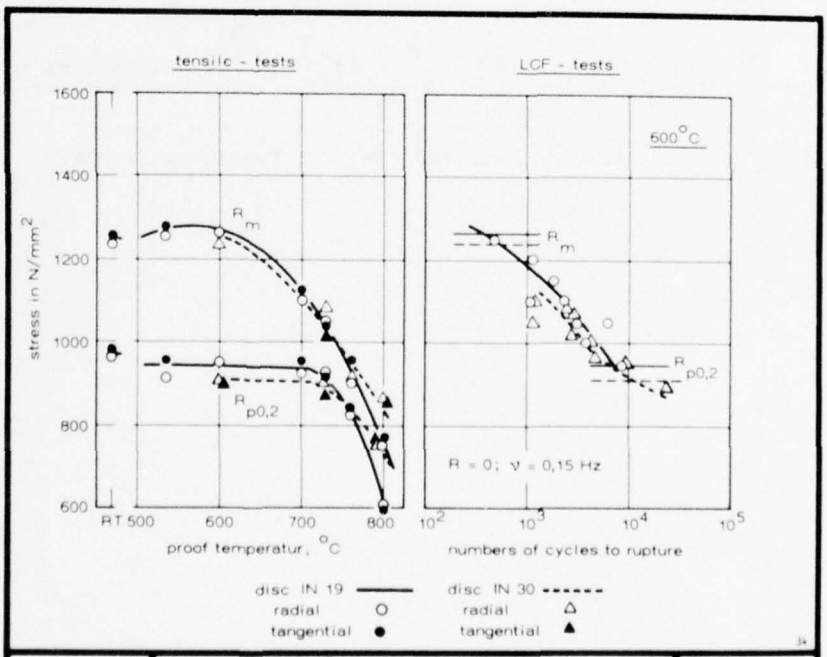


Fig: 15

FI Krefeld

Results of two PM-IN 100 discs forged of HIP -prematerial

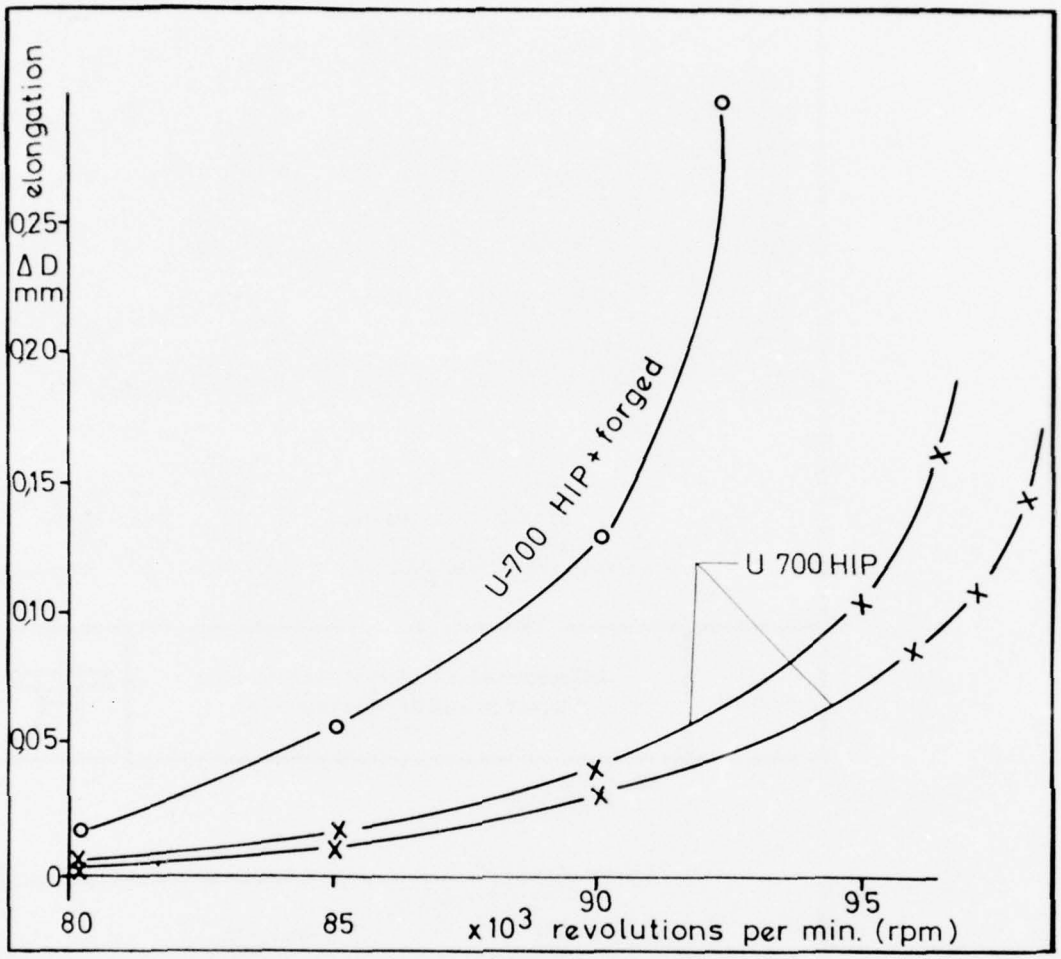


Fig. 16 Spin test on PM-U-700 discs at room temperature.

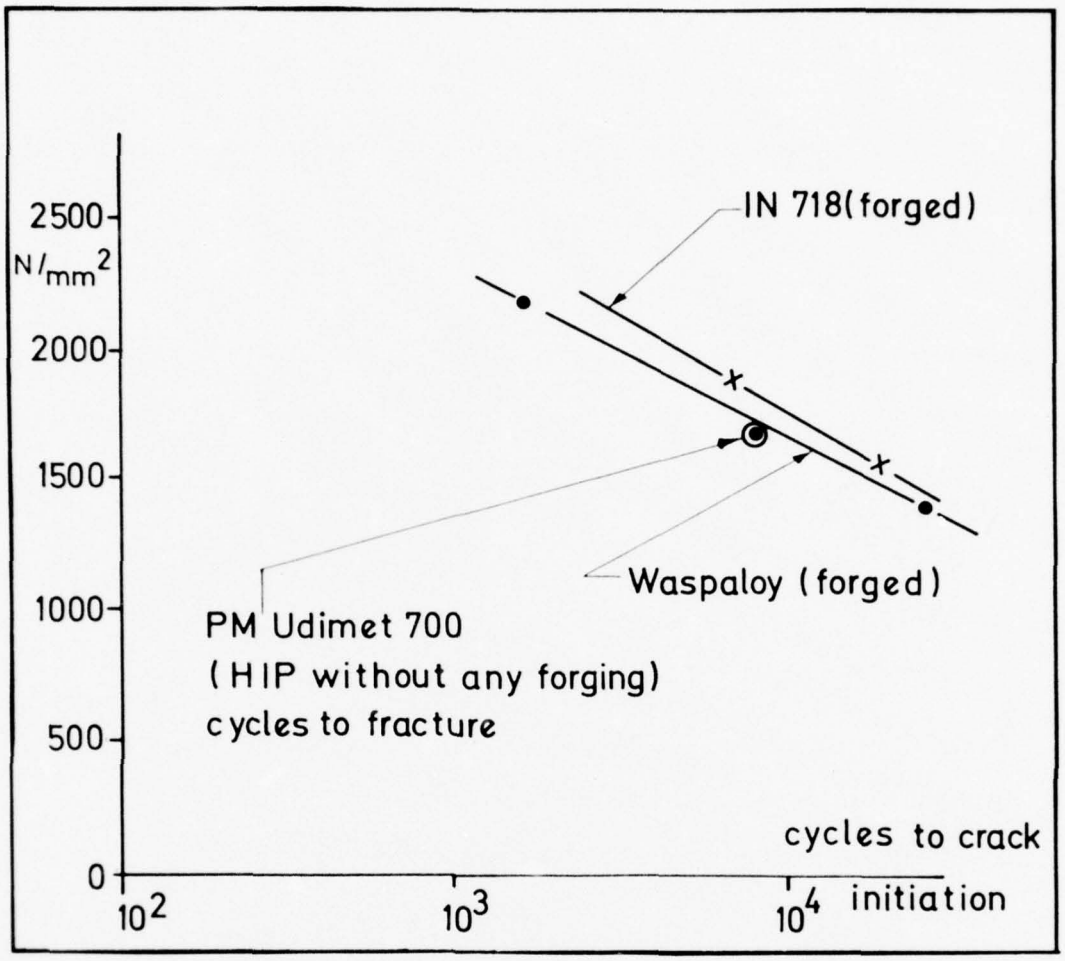


Fig. 17 Low cycle fatigue (LCF) spin test of some disc materials at room temperature.

INFLUENCE SUR LES CARACTÉRISTIQUES MÉCANIQUES DES CONDITIONS DE MISE EN OEUVRE DE POUDRES DE SUPERALLIAGES A BASE DE NICKEL

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Résumé

Les caractéristiques des alliages à base nickel, élaborés à partir de poudres préalliées, sont influencées par chacune des opérations successives de mise en œuvre. Le but de ce travail est de montrer l'effet des principaux paramètres tels que :

- le procédé d'atomisation de la poudre (électrode tournante, atomisation à l'argon),
- la granulométrie de la poudre,
- la teneur en carbone de l'alliage,
- le mode de densification (filage, pressage isostatique conventionnel, pressage isostatique rapide, compression uni-axiale pseudo-isostatique),
- les traitements thermiques et les conditions de forgeage selon le domaine de température envisagé pour l'application.

Les structures et les propriétés mécaniques (traction, fluage, fatigue oligocyclique) sont examinées sur des alliages élaborés à l'échelle du laboratoire (IN 100, Astroloy) et sur des produits industriels de diverses provenances (René 95, Astroloy bas carbone).

INFLUENCE ON THE MECHANICAL PROPERTIES OF VARIOUS PROCESSING PARAMETERS APPLIED TO NICKEL BASE SUPERALLOYS POWDERS

Summary

The characteristics of nickel-base alloys, fabricated from pre-alloyed powders, are influenced by each of the successive operations. This paper aims at showing the effect of the main parameters, such as :

- powder atomisation process (rotative electrode, argon atomisation),
- powder granulometry,
- carbon content of the alloy,
- densification mode (extrusion, conventional isostatic compacting, fast isostatic compacting, pseudo-isostatic uniaxial compression),
- thermal treatments and forging conditions according to the temperature range considered for the application.

The structures and the mechanical properties (tension, creep, low cycle fatigue) are examined on alloys fabricated at laboratory scale (IN 100, Astroloy) and on industrial products of various origins (René 95, Astroloy low carbon).

I - INTRODUCTION -

Les caractéristiques mécaniques des superalliages à base de nickel, élaborés à partir de poudres préalliées, sont influencées par chacune des opérations successives de mise en oeuvre, tant au niveau de la préparation de la poudre et de la densification qu'à celui des traitements thermiques ou thermo-mécaniques.

Dans le but de mieux saisir l'influence des différents paramètres d'élaboration, des travaux ont été menés, parallèlement, à l'échelle du laboratoire sur l'IN 100 et l'Astroloy, et dans une optique plus industrielle sur le René 95 et l'Astroloy bas carbone.

La méthode d'atomisation de la poudre intervient en particulier dans la répartition et la finesse des carbures. Sa granulométrie, quant à elle, semble influencer la taille finale du grain de l'alliage densifié. La teneur en carbone de ce dernier peut en modifier totalement la structure et même interdire l'utilisation de certains procédés de compaction.

Les différents modes de densification qui ont été utilisés (filage, pressage isostatique conventionnel à chaud, pressage isostatique rapide à chaud, compression uni-axiale pseudo-isostatique) ont permis de juger de leur intérêt respectif et de mesurer leur influence sur les propriétés mécaniques des alliages densifiés après traitements thermiques. Ces derniers dont le choix dépend du domaine de température de l'application envisagée, ainsi que les conditions de forgeage, constituent en fait la partie essentielle de la mise au point des alliages puisqu'ils déterminent les propriétés des matériaux.

II - EXAMEN DES POUDRES -

Le procédé d'élaboration de la poudre et la teneur en carbone sont deux paramètres importants qui agissent sur la qualité et la structure des alliages densifiés. Nos observations ont exclusivement porté sur des poudres préparées selon deux procédés parmi les plus courants, c'est-à-dire à l'électrode tournante où l'on fait jaillir un arc dans une atmosphère

neutre entre une électrode fixe en tungstène et une électrode tournante consommable (IN 100 0,18 % C) et par atomisation à l'argon où l'alliage liquide est dispersé en fines gouttelettes par des jets de gaz (IN 100 0,006 % C, IN 100 0,18 % C, Astroloy 0,03 et 0,06 % C).

Le procédé à l'électrode tournante conduit à des particules sphériques à structure dendritique de 200 µm environ de diamètre dont la composition dépend de la plus ou moins grande homogénéité du lingot utilisé comme électrode consommable. En effet, la fusion très localisée, à la surface du lingot, donne des particules dont la composition varie au cours du temps comme la composition ponctuelle du lingot coulé. La taille des précipités de carbures, essentiellement TiC, est assez grossière car la fusion brève de l'alliage n'en permet vraisemblablement pas la dissolution complète (fig. 1). Les teneurs en oxygène et azote sont respectivement de 60 et 30 ppm.

Le procédé d'atomisation à l'argon conduit pour sa part à des particules également à structure dendritique, beaucoup plus fines (environ 100 µm), de formes très tourmentées et dont la surface développée est très grande. La teneur en oxygène est d'ailleurs plus élevée que dans le cas précédent et se situe autour de 100 ppm. Certaines particules présentent des cavités de taille assez importante (40 à 90 µm pour les plus grosses) probablement remplies d'argon. La précipitation de carbures est fine et bien répartie (fig. 2).

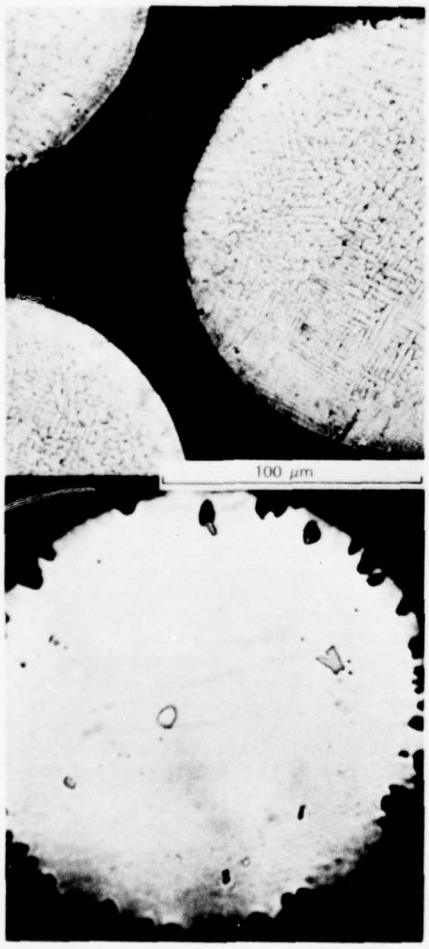

Fig. 1 – Poudre d'IN 100 à teneur en carbone normale (0,18 %).

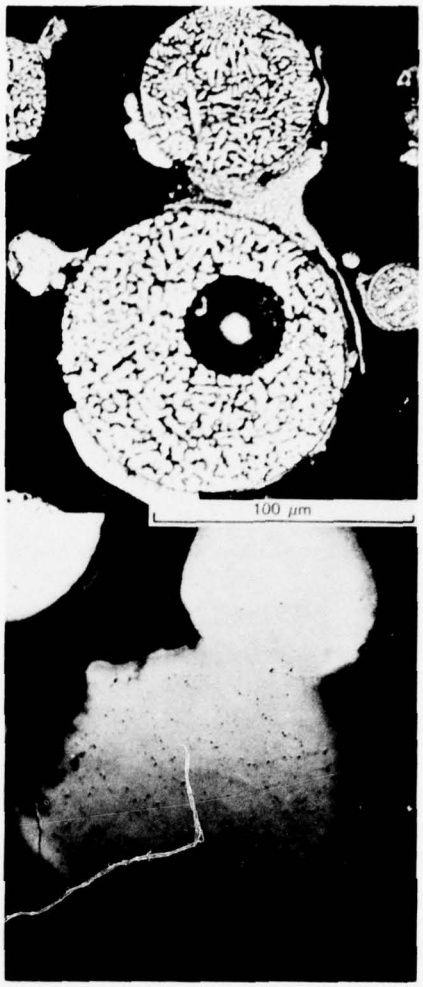

Fig. 2 – Poudre d'IN 100 à bas carbone (0,006 %).

III - MODES DE DENSIFICATION -

Quatre méthodes ont été utilisées pour la densification des poudres d'alliages réfractaires : le filage à chaud sous gaine, le pressage isostatique conventionnel à chaud, le pressage isostatique rapide et la compression uni-axiale pseudo-isostatique.

1°) Densification par filage à chaud -

La densification par filage à chaud impose de grandes déformations qui paraissent favorables à la qualité du matériau. Ce procédé est par contre limité par la puissance des presses de filage disponibles et la réalisation de pièces de grand diamètre nécessiterait un matériel de très grande dimension. Il reste par contre le seul procédé permettant la réalisation de profilés de formes complexes par réduction homothétique d'empreintes remplies de poudre. Aucune méthode d'usinage ou même de filage de matériau massif ne permet d'aboutir au même résultat surtout dans le cas de formes présentant des évidements sur de grandes longueurs. La qualification des matériaux densifiés par filage reste par conséquent très utile.

L'étude a été menée sur une presse de 250 tonnes équipée pour le procédé de filage à chaud UGINE-SEJOURNET à lubrification par le verre. Le filage de la poudre s'effectue sous

gaine protectrice d'acier doux ou d'acier inoxydable, obturée sous vide secondaire par bombardement électronique. Le domaine de température pour lequel le filage conduit à un matériau sain dépend de la nature de l'alliage et semble lié à sa teneur en phase γ'. Pour l'IN 100, il est très étroit (une vingtaine de degrés environ) tandis que pour l'Astroloy, il peut s'étendre sur une centaine de degrés. L'opération de filage constituant un système pratiquement adiabatique, une certaine quantité de chaleur due à la déformation est introduite. Il s'ensuit que la température à laquelle l'alliage doit être porté avant extrusion est liée au rapport de filage et varie en sens contraire.

La vitesse et le rapport de filage ont été respectivement fixés à 1 m/s et à 15:1 pour les deux alliages. Les températures pour lesquelles les matériaux se sont alors révélés sains sont 1 150°C pour l'IN 100 et 1 000°C pour l'Astroloy. Les analyses de gaz ont montré que l'accroissement de la teneur en oxygène dans les matériaux filés par rapport à la poudre ne dépasse pas 10 ppm.

Avec les conditions de filage adoptées, la structure des alliages densifiés est presque entièrement recristallisée avec une taille de grains d'environ 10 µm. On retrouve parfois la forme de certaines particules et quelques zones dendritiques (fig. 3). Dans le cas de l'IN 100, les précipités de phase γ' ont une taille assez uniforme d'environ 2 000 Å ; toutefois, sur les nuances densifiées à partir de poudre élaborée suivant le procédé d'atomisation sous argon, on observe à côté de la fine précipitation de γ', une précipitation plus grossière atteignant 2 à 4 µm (fig. 4). Ceci n'apparaît pas dans le cas de l'Astroloy.

Fig. 3 – IN 100 à 0,18 % C (poudre atomisée à l'électrode tournante) brut de filage 1 150°C.

Au cours de la période d'échauffement qui précède le filage et par un mécanisme qui n'est pas totalement élucidé, la répartition des carbures évolue et l'on constate que ces derniers se concentrent à la périphérie des particules de poudre. Après filage, les précipités sont alors alignés dans le sens de la déformation, décorant ainsi les limites des anciennes particules. Ce phénomène s'observe surtout sur l'IN 100, à teneur en carbone normale (0,18 %) mais est également visible sur l'Astroloy.

Par traction à haute température (au-delà de 1 000°C), les alliages filés ont un comportement superplastique avec des allongements à rupture supérieurs à 600 %. Ceci est à relier à la grande finesse du grain métallurgique qui est obtenue par ce procédé de compaction.

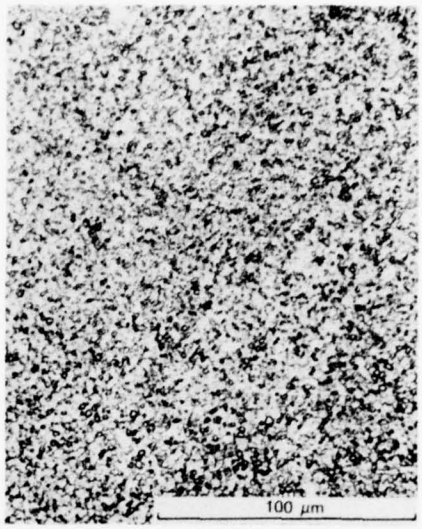

Fig. 4 – IN 100 à 0,006 % C (poudre atomisée à l'argon) brut de filage 1 150°C.

2°) Densification par pressage isostatique conventionnel à chaud - (PICC) -

Cette méthode qui est surtout envisagée pour des pièces de grand diamètre permet l'utilisation de préformes limitant autant que possible l'usinage. La poudre d'alliage placée dans un récipient facilement déformable à chaud (métal ou verre) est compactée à haute température dans une enceinte de type autoclave, par l'intermédiaire d'un gaz sous pression. Généralement dans le cas des superalliages, pour des temps de maintien de quelques heures, les pressions vont jusqu'à 200 MPa, les températures utilisées atteignant 1 200°C.

Seul l'IN 100 à bas carbone (0,006 %) a été compacté par ce procédé par la firme DEW (RFA), à deux températures 1 100 et 1 200°C.

Fig. 5 – IN 100 à 0,006 % C densifié par PICC (1 200°C).

La pression était, dans les deux cas, d'environ 190 MPa et le temps de compaction de 2 heures. L'alliage densifié par cette méthode se différencie des alliages filés par une structure équiaxe entièrement recristallisée. Mais comme dans le cas de l'alliage filé de même composition, atomisé à l'argon, on constate la présence de précipités grossiers (plusieurs microns)de phase γ' (fig. 5).

Certains semblent décorer des joints de grains, d'autres plus fins, répartis linéairement sont vraisemblablement localisés sur des lignes de glissement. Les microfractographies d'alliage compacté à 1 100°C sont caractéristiques d'un manque de cohésion entre les particules ; seule la température de 1 200°C conduit à un matériau sain et parfaitement densifié.

Dans les mêmes conditions d'essais de traction que le matériau filé, le matériau densifié par PICC n'est pas superplastique mais possède malgré tout un allongement à rupture d'au moins 12 %.

3°) Densification par pressage isostatique rapide à chaud - (PIRC) -

Le procédé consiste à comprimer le matériau dans un pot de presse, pendant un temps très court, en utilisant un fluide transmetteur de pression de façon à réaliser des conditions de compaction isostatique.

Le procédé de densification par pressage isostatique conventionnel à chaud présente quelques inconvénients, en particulier un investissement coûteux pour des installations spécifiques et un temps de traitement relativement long, pour assurer une densification totale du matériau. Des raisons métallurgiques limitant l'augmentation de la température, la diminution du temps de compaction n'est possible que si l'on accroît notablement la pression.

Lors d'une opération de filage, la pression développée à l'intérieur du conteneur peut atteindre 1 500 MPa, par contre la température ne peut être maintenue que quelques dizaines de secondes, le matériau se refroidissant rapidement. Des essais ont été préalablement menés à l'aide de la presse utilisée pour le filage, dont la filière a été remplacée par une pièce pleine. Ils ont conduit à des résultats encourageants mais pas entièrement satisfaisants. En effet, dès les premiers instants de l'opération, le matériau est écrasé et occupe tout le volume disponible. Il vient alors en contact avec la paroi du conteneur, se refroidit fortement et bloque la déformation. La solution réside dans l'interposition entre matériau et conteneur d'un film de fluide transmetteur de pression (verre fondu ou matière organique).

En utilisant une presse de filage hydrostatique de 575 tonnes *, il a été possible de compacter sous gaine de la poudre d'IN 100 à teneur en carbone normale (0,18 %) et à très bas carbone (0,006 %) à la température de 1 200°C. La pression développée atteignait 1 300 MPa et le temps de maintien une vingtaine de secondes. La pression s'exerçant en milieu hydrostatique (visqueux), tout contact est évité avec le conteneur et les conditions de déformation sont voisines de celles obtenues en pressage isostatique conventionnel. Les alliages sont parfaitement densifiés et sans porosités.

Toutefois, l'IN 100 à taux de carbone normal (0,18 %) présente des lisérés de carbures segrégés à la limite des anciennes particules de poudre (fig. 6). Cette structure fragilisante est très défavorable et se traduit lors des essais mécaniques par des ruptures interparticulaires sans allongement notable. Par contre, dans le cas de l'IN 100 à bas carbone, ce phénomène n'apparaît pas et l'on retrouve une structure très voisine de celle obtenue sur le même matériau compacté par pressage isostatique conventionnel (fig. 7). De même, il ne présente pas de comportement superplastique.

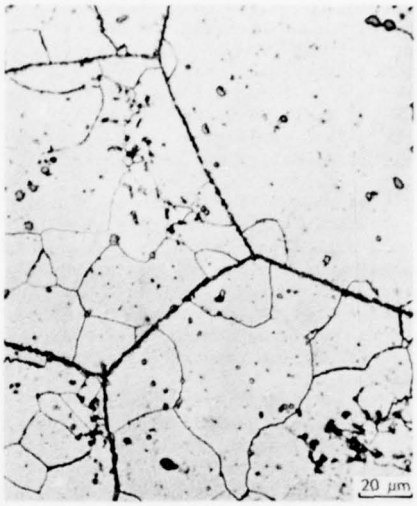

Fig. 6 – IN 100 à 0,18 % C densifié par PIRC (1 200°C).

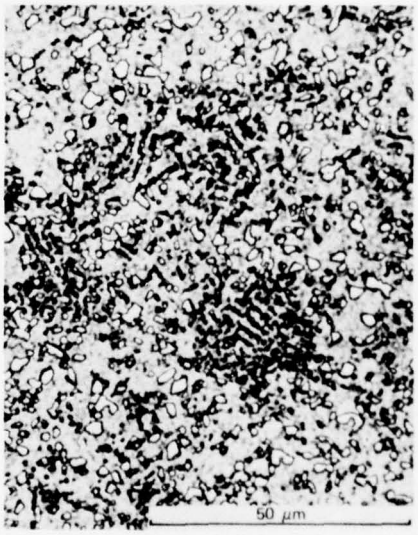

Fig. 7 – IN 100 à 0,006 % C densifié par PIRC (1 200°C).

Il semble donc que le choix d'une poudre d'alliage, pour une application donnée, doit être faite en fonction de la méthode de densification utilisée, particulièrement dans le cas où apparaît une segrégation des carbures à la périphérie des particules. En effet, si le filage en augmentant la surface de chaque grain de poudre par étirement dans une direction, détruit ce film presque continu de carbures, les isole et supprime leur action fragilisante, il n'en est pas de même du pressage isostatique.

Il apparaît évident que l'IN 100 à teneur en carbone normale n'est absolument pas adapté à une méthode de compaction ne provoquant que des déformations limitées.

* CEA Saclay : Service de Recherches Métallurgiques Appliquées.

En ce qui concerne la méthode de pressage isostatique rapide par elle-même, le diamètre des pièces compactées dépend essentiellement de la force disponible sur les presses de filage. A titre d'exemple, pour une pression de 1 300 MPa, une force de 4 000 tonnes sera nécessaire pour un diamètre de 200 mm tandis que pour 700 mm il faudra atteindre 65 000 tonnes. L'utilisation des presses de filage est néanmoins très utile à des fins expérimentales ou pour des pièces de petit diamètre. On peut penser malgré tout à adapter un outillage permettant la réalisation de pièces de grandes dimensions sur des presses de forgeages industriel. La réalisation de préformes limitant l'usinage serait parfaitement envisageable. Un essai dans ce sens a été effectué sur de L'IN 100 à bas carbone à la température de 1 150°C et sous une pression maximum de 1 300 MPa. La figure 8 montre l'échantillon avant et après compaction.

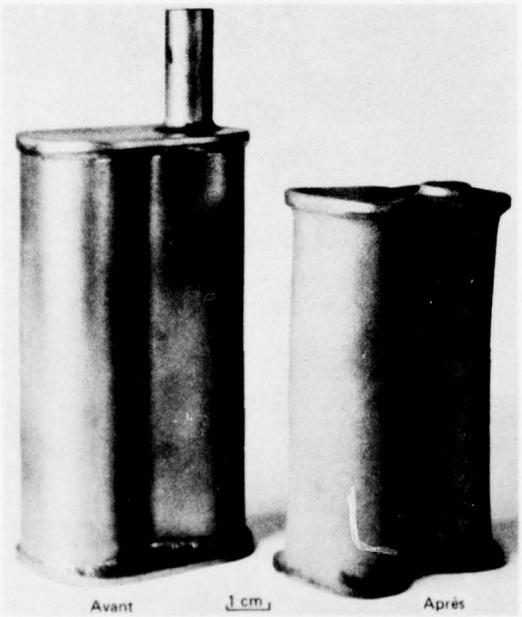

Fig. 8 – Exemple de compactage par PIRC sur de l'IN 100 bas carbone (1 150°C, 1 mn, 1300 MPa).

4°) Densification par compression uni-axiale pseudo-isostatique

Ce procédé se rapproche des méthodes classiques utilisées pour la compaction à chaud de poudres métalliques. La poudre d'alliage est comprimée sous vide entre une matrice et un piston en graphite protégé. La température est d'environ 1 200°C ; la pression qui est limitée par la résistance du graphite est d'environ 20 MPa. La durée de l'opération est de plusieurs heures.

IV - INFLUENCE DES TRAITEMENTS THERMIQUES SUR LA STRUCTURE ET LES PROPRIETES MECANIQUES DES ALLIAGES DENSIFIES -

Les alliages bruts de densification possèdent des propriétés mécaniques qui ne représentent pas la valeur intrinsèque du matériau. Ce dernier est, la plupart du temps, dans un état hors d'équilibre, tant sur le plan de la cristallisation qu'en ce qui concerne les phases durcissantes (γ' et carbures). Le maintien prolongé à une température parfois élevée peut le faire évoluer et les résultats, en particulier dans le cas d'essais de longue durée comme le fluage n'en expriment pas réellement les propriétés. Mais ils permettent de le situer et en particulier de juger rapidement de sa qualité sur le plan de la compaction. De plus, ils témoignent d'un état du matériau et peuvent servir de référence pour l'étude de traitements thermiques ou thermo-mécaniques. Néanmoins, nous n'en ferons pas état ici et les résultats qui seront présentés porteront toujours sur des matériaux traités après densification.

L'utilisation de poudres préalliées conduisant à des matériaux plus homogènes avait fait espérer obtenir des alliages plus fiables dont les applications s'étendraient dans tout le domaine des températures d'emploi, y compris les hautes températures (cas de l'IN 100 par exemple). A la lumière de l'expérience acquise, les applications à haute température ne semblent pouvoir être envisagées qu'à plus long terme, principalement à cause des difficultés rencontrées dans l'obtention d'une taille de grain compatible avec une bonne résistance au fluage. Néanmoins, il est intéressant de connaître les propriétés dans ce domaine de température et en particulier de voir l'influence de certains traitements thermiques sur la structure de l'alliage en fonction de certains paramètres (origine des poudres, modes de compaction) et leur incidence sur les propriétés recherchées. Il reste que l'application la plus immédiate des superalliages densifiés à partir de poudre concerne l'élaboration de disques de turbine ou de compresseur présentant une résistance améliorée à la fatigue oligocyclique à des températures comprises entre 550 et 650 °C.

Les traitements thermiques appliqués à l'alliage densifié sont conditionnés par le domaine de température de l'application envisagée. Si l'on vise des températures élevées, le critère essentiel est une bonne résistance au fluage et l'alliage devra posséder un grain métallurgique assez gros. Par contre, pour une application à température modérée (disques), c'est la résistance à la fatigue oligocyclique dont on tiendra compte. L'alliage devra alors présenter un grain fin et une limite élastique aussi élevée que possible. D'une façon générale, les traitements thermiques qui ont été appliqués en premier correspondaient à ceux utilisés sur l'alliage coulé et visaient souvent à une application haute température. Les propriétés mécaniques mesurées, y compris la résistance à la fatigue oligocyclique peuvent alors servir de référence pour des essais d'amélioration de ces propriétés par des traitements thermiques adaptés aux conditions d'utilisation (disques à température modérée par exemple). Les résultats présentés ici, qui ont surtout porté sur l'IN 100 et l'Astroloy, ne représentent que l'état actuel des études dans ce domaine.

1°) Cas de l'IN 100 -

Dans le cas de l'IN 100, la température retenue pour le traitement de grossissement du grain dans l'optique d'une bonne résistance au fluage est de 1 220°C (température maximale autorisée par le matériau, c'est-à-dire n'entraînant pas d'apparition locale de phase liquide). Des observations effectuées sur les matériaux traités, on peut déduire que :

- ces alliages étudiés ont une structure entièrement recristallisée avec une taille de grain de 50 microns environ, indépendante de la teneur en carbone de l'alliage.

- la teneur en carbone exerce une influence sur la structure en particulier dans le cas du matériau filé. Alors que l'alliage à bas carbone possède une structure isotrope à grains équiaxes (fig. 9), le matériau à teneur en carbone

normale conserve le souvenir du mode de densification employé. Dans le sens du filage, les joints de grains sont linéaires, décorés de carbures et correspondent souvent aux surfaces des anciennes particules de poudre (fig. 10). Le franchissement de ces limites par les joints est assez rare.

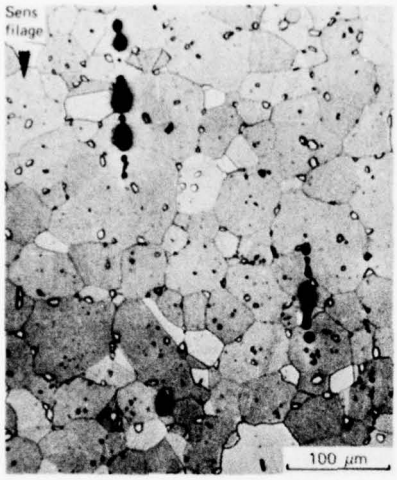

Fig. 9 – IN 100 à 0,006 % C, filé puis traité à 1220°C 24 h.

Fig. 10 – IN 100 à 0,18 % C, filé puis traité à 1220°C 24 h.

- la structure des alliages à bas carbone est pratiquement indépendante du mode de densification utilisé. Des précipités de phase γ' de plusieurs microns se retrouvent comme dans l'état brut de densification.

- les alliages élaborés à partir de poudre atomisée à l'argon présentent après traitement des porosités de plusieurs dizaines de microns qui n'existent pas sur le matériau brut de densification (fig. 9). Ce phénomène est à relier à la présence de cavités observées sur la poudre et vraisemblablement remplies d'argon. La température à partir de laquelle elles apparaissent se situe autour de 1 100°C. Cette dernière constituerait la température limite à laquelle pourraient être effectués des traitements thermiques visant une application à moyenne température et une amélioration de la résistance à la fatigue oligocyclique, car la présence de cavités serait défavorable par création locale de zones à forte concentration de contraintes.

- la taille du grain est reliée à la granulométrie de la poudre utilisée pour la densification. Les figures 11 et 12 relatives à de l'IN 100 à teneur en carbone normale, filé puis traité, témoignent de la différence de taille du grain métallurgique pour des fractions d'un même lot de poudre ayant respectivement comme granulométrie : inférieur à 100 µm et supérieur à 250 µm.

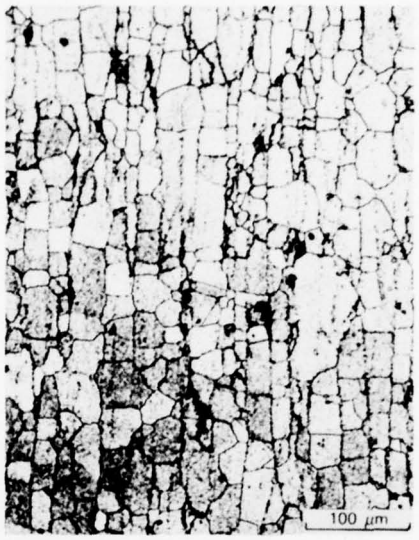

Fig. 11 – IN 100 à 0,18 % C, filé puis traité à 1220°C 24 h (ϕ particules < 100 µm).

Fig. 12 – IN 100 à 0,18 % C, filé puis traité à 1220°C 24 h (ϕ particules > 250 µm).

- le traitement thermique fait disparaître le comportement superplastique à haute température (cas des alliages filés).

Le traitement thermique qui a été adopté en vue d'améliorer les propriétés mécaniques à haute température et essentiellement la résistance au fluage est le suivant :

1 220°C 24h + 1 080°C 8h + 850°C 24h.

La comparaison des propriétés mécaniques d'un alliage IN 100 filé à teneur en carbone normale avec celle d'un IN 100 à bas carbone conduit aux remarques suivantes :

- la teneur en carbone ne semble pas exercer

d'influence très sensible sur la résistance à rupture et la limite élastique par traction (fig. 13).

- à basse et moyenne température, l'allongement à la rupture par traction est notablement supérieur pour l'alliage peu chargé en carbone. Au contraire, il est plus faible pour les températures au-delà de 750°C mais toujours supérieur à celui de l'alliage coulé.

- la résistance au fluage de l'alliage à bas carbone reste inférieure à toute température à celle de l'alliage plus chargé. La différence est surtout sensible pour des températures inférieures à 650°C et supérieures à 850°C (fig. 14). Les allongements à rupture restent très comparables. L'alliage coulé a des propriétés supérieures en fluage au-delà de 700°C environ.

La comparaison des propriétés mécaniques d'alliages de même composition mais densifiés par des moyens différents (filage, PICC, PIRC) puis traités n'a été effectuée que pour l'IN 100 à bas carbone (fig. 15). La limite élastique est sensiblement la même quel que soit le mode de densification utilisé. Tout se passe comme si le traitement thermique effaçait l'histoire antérieure de l'alliage. Ceci est à rapprocher du fait que la structure a été trouvée identique pour les alliages à bas carbone. L'allongement à rupture qui reste toujours supérieur à 20 % pour les températures inférieures à 700°C, permet de classer les alliages en fonction de leur ductilité décroissante dans l'ordre suivant : alliage filé, PICC, PIRC.

Des essais de fatigue oligocyclique ont été effectués à 550°C sur l'IN 100 bas carbone densifié-traité. Le schéma des éprouvettes utilisées ainsi que la forme du cycle sont présentés sur la figure 16. La taille réduite des éprouvettes conduit à un effet d'échelle mais permet néanmoins des mesures comparatives, les niveaux de contraintes ne devant pas être pris en valeur absolue. La figure 17 présente les résultats pour les séquences du traitement utilisé précédemment. La figure 18 compare les résultats obtenus pour deux modes de densification : filage et pressage isostatique conventionnel à chaud. On voit que le matériau densifié par filage présente une résistance supérieure à celui compacté par PICC.

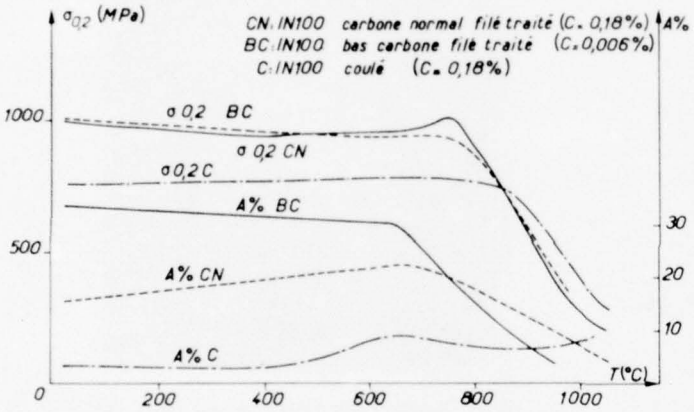

Fig. 13 — Influence de la teneur en carbone sur la limite élastique et l'allongement à rupture de l'IN 100 filé-traité.

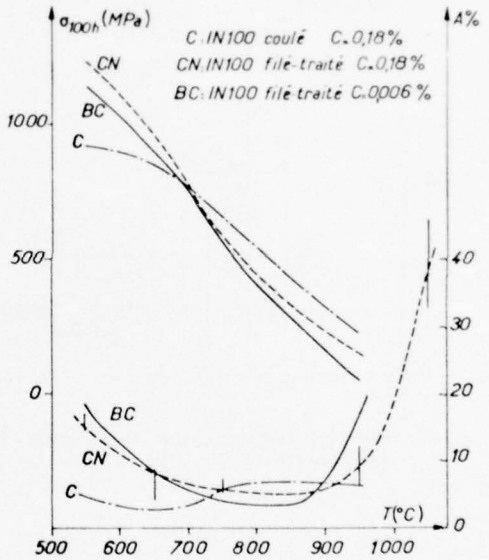

Fig. 14 — Influence de la teneur en carbone sur la résistance et l'allongement à la rupture par fluage pour l'IN 100 filé-traité.

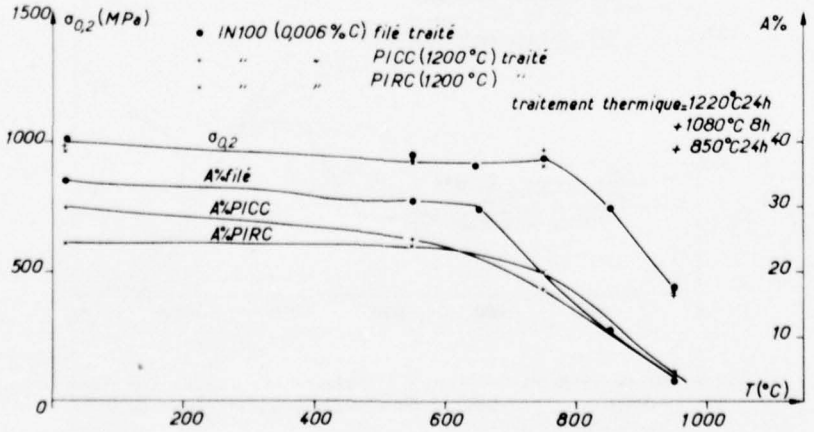

Fig. 15 — Influence du mode de densification sur la limite élastique et l'allongement à la rupture pour l'IN 100 traité.

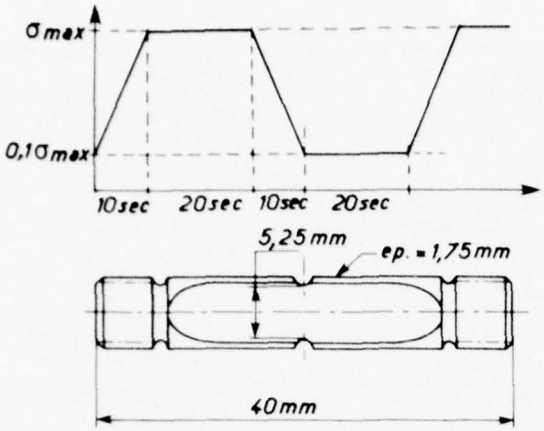

Fig. 16 — Eprouvette et cycle utilisés pour les essais de fatigue oligo-cyclique.

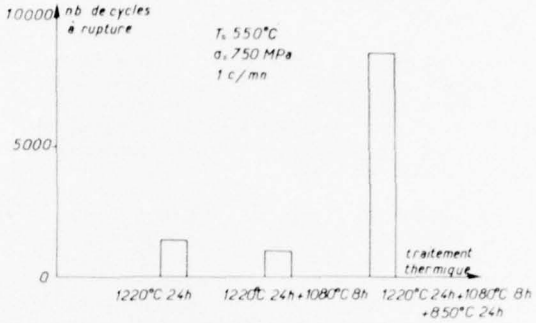

Fig. 17 — Influence des séquences des traitements thermiques sur la résistance à la fatigue oligocyclique de l'IN 100 à 0,006 % C filé.

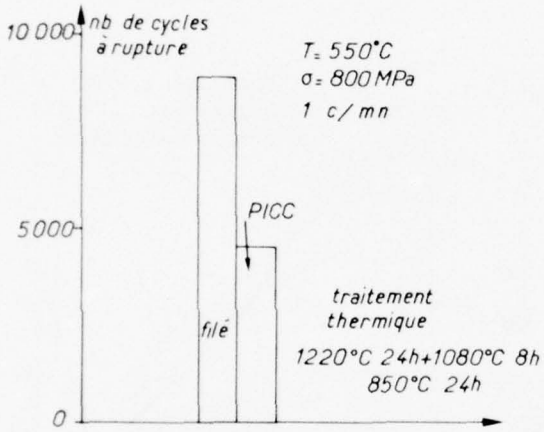

Fig. 18 — Influence du mode de compaction sur la résistance à la fatigue oligo-cyclique de l'IN 100 à 0,006 % C traité.

Fig. 19 — Astroloy à 0,06 % C filé puis traité 1 180° C 24 h.

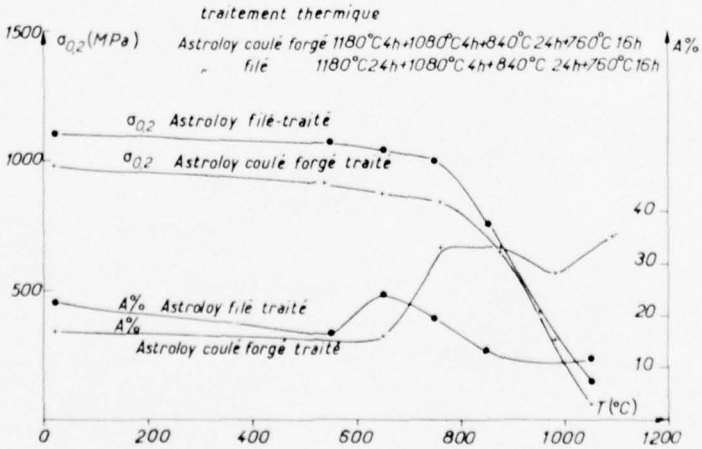

Fig. 20 — Comparaison de la limite élastique et de l'allongement à rupture pour deux alliages Astroloy traités : l'un obtenu par métallurgie classique, l'autre par métallurgie des poudres (filage).

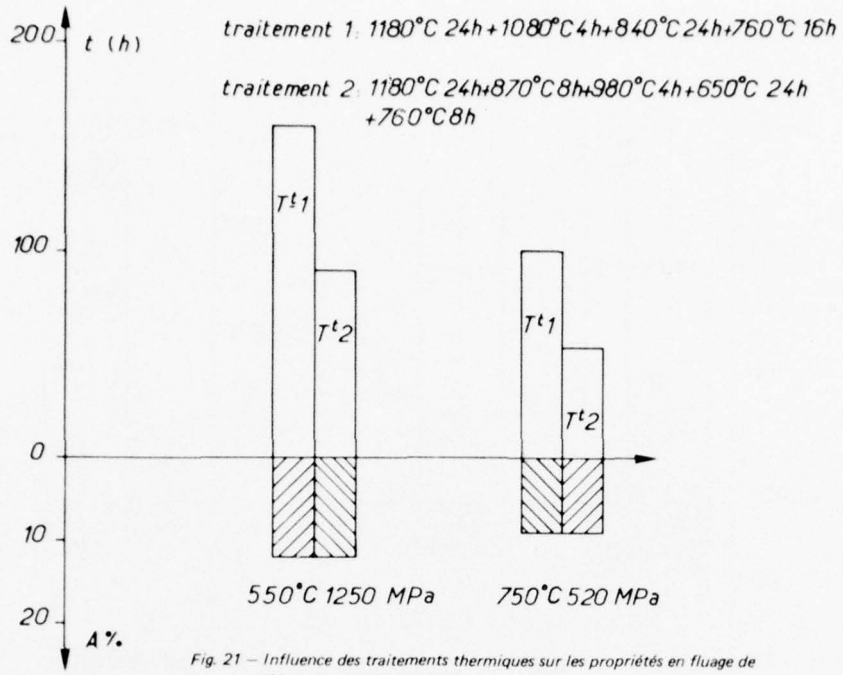

Fig. 21 — Influence des traitements thermiques sur les propriétés en fluage de l'Astroloy à 0,06 % C filé.

2°) Cas de l'Astroloy -

L'étude a été menée sur de l'Astroloy de composition normale (0,06 %) qui a été compacté par filage à chaud et sur de l'Astroloy à bas carbone (0,03 %) densifié par PICC.

a) Astroloy de composition normale

Le traitement de grossissement du grain est effectué à 1 180°C. L'alliage est entièrement recristallisé avec une taille de grain d'environ 50 microns, comme celle obtenue sur l'IN 100. Par contre, les joints de grains franchissent aisément les limites des anciennes particules de poudre. La structure est à grains sensiblement équiaxes et les précipités de carbures passent en solution (fig. 19). On note l'absence de précipités grossiers de γ' .

Comme pour l'IN 100 élaboré à partir de poudre atomisée à l'argon, on remarque la présence de cavités d'une taille atteignant plusieurs dizaines de microns. Deux traitements thermiques complets ont été essayés :

Traitement n° 1 :

1180°C 24h + 1080°C 4h + 840°C 24h + 760°C 16h.

Traitement n° 2 :

1180°C24h + 870°C8h + 980°C4h + 650°C24h + 760°C8h.

et conduisent à des propriétés mécaniques en traction très voisines. La figure 20 est relative à un alliage Astroloy filé et à un alliage coulé forgé ayant subi le même traitement thermique n° 1. Le matériau filé-traité présente une limite élastique supérieure à celle de l'alliage coulé dans tout le domaine de température, bien qu'au-delà de 800°C les deux matériaux se situent pratiquement sur un plan d'égalité. Les allongements à rupture sont sensiblement identiques jusqu'à 700°C. Pour les températures plus élevées, l'alliage coulé devient nettement plus ductile que l'alliage filé.

Les essais de fluage effectués à 550°C sous 1 250 MPa et à 750°C sous 520 MPa (fig.21) montrent que le traitement n° 1 conduit à des temps de rupture doubles de ceux obtenus avec le traitement n° 2.

b) Astroloy à bas carbone

La figure 22 montre pour l'Astroloy bas carbone (0,03 %), brut de compaction par PICC, l'influence de trois traitements thermiques sur les caractéristiques de traction à 20 et 650°C, le temps de rupture en fluage à 650°C et la propagation des criques (éprouvette ASTM).

- Traitement n° 1

La température de mise en solution est inférieure à celle de la mise en solution complète de γ' (1 135°C).

Les traitements de revenu sont du type dit "YO-YO" parce que les deux revenus usuels à température décroissante sont à chaque fois suivis d'un nouveau maintien à une température supérieure.

Le but de ce traitement compliqué est de précipiter et de coalescer les carbures et également de régler la morphologie de la phase γ' .

- Traitement n° 2

Même température de mise en solution que précédemment, mais revenu classique à deux températures décroissantes.

- Traitement n° 3

La température de mise en solution est supérieure à celle de la mise en solution de γ' .

Les traitements de revenu sont les mêmes qu'en 1.

Le traitement n° 2 qui donne de fins précipités confère les meilleures caractéristiques de limite élastique, de fluage et de résistance à la propagation des criques. La ductilité en fluage n'a pas pu être appréciée, puisque l'éprouvette n'a pas rompu.

Les revenus du traitement n° 1, dont on ne perçoit pas ici l'avantage, ont été conçus initialement pour une température de fonctionnement plus élevée que 650°C.

Le traitement n° 3 qui est le traitement n° 1 avec une mise en solution complète de la phase γ' donne une limite élastique et une résistance à la propagation des criques, plus faibles, mais le fluage est augmenté ainsi que la ductilité à rupture.

En conclusion, les caractéristiques des produits compactés sont très dépendants des

traitements thermiques qui doivent être sélectionnés en fonction des conditions d'emploi.

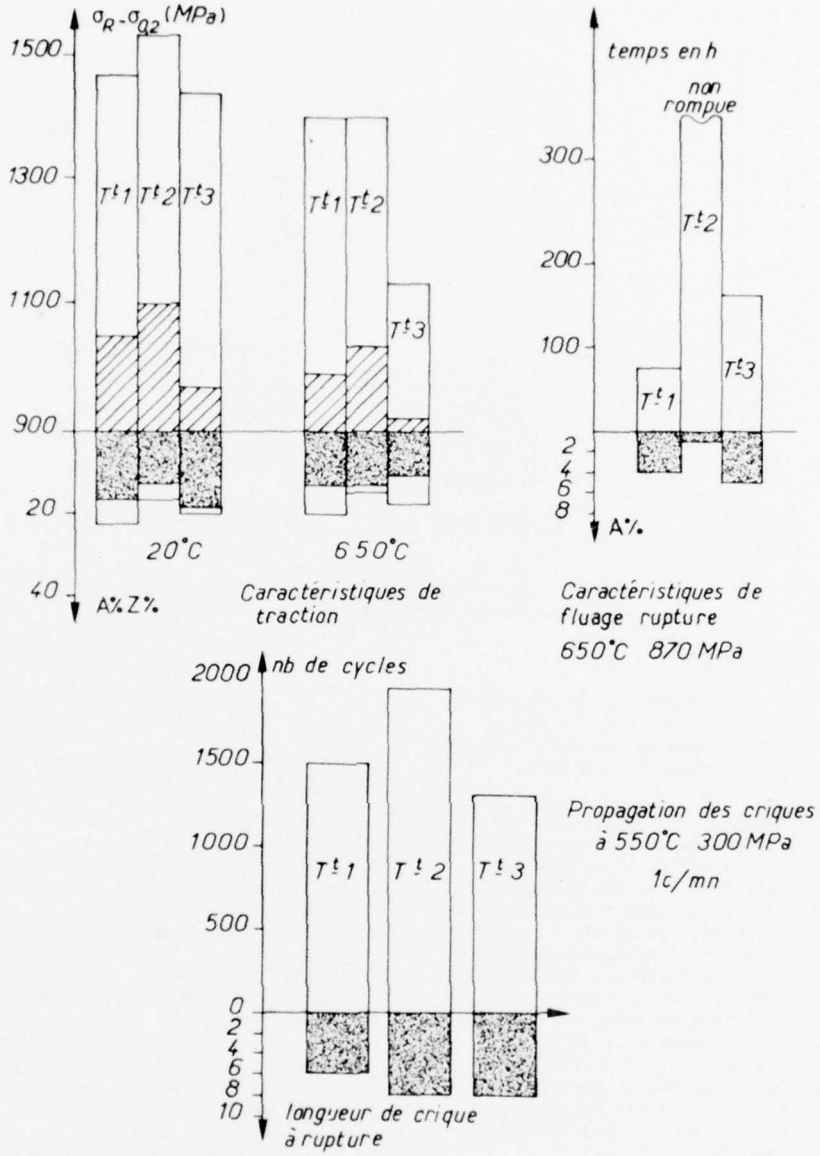

Fig. 22 — Influence des traitements thermiques sur les propriétés mécaniques de l'Astroloy bas carbone densifié par PICC.

Traitement 1 : «YO-YO»		Traitement 2		Traitement 3	
T	1100°C 4 h huile	T	1100°C 4 h huile	T	1160°C 4 h air
R1	850°C 8 h air	R1	850°C 24 h air	R1	850°C 8 h air
R2	980°C 4 h air	R2	760°C 16 h air	R2	980°C 4 h air
R3	650°C 24 h air			R3	650°C 24 h air
R4	760°C 8 h air			R4	760°C 8 h air

V - COMPARAISON DES CARACTERISTIQUES MECANIQUES DE DIFFERENTS PRODUITS INDUSTRIELS -

Les figures 23 et 24 montrent les caractéristiques de résistance à la traction à l'ambiante et à 650°C, respectivement pour le René 95 et l'Astroloy bas carbone.

Les produits examinés sont disponibles sur le marché commercial et réalisés d'après trois techniques de compaction :

1°) Compaction P.I.C.C. à basse pression - 50 bars - à l'autoclave. Lopins de 160 mm de diamètre.

2°) Extrusion
Diamètre 110 mm pour le René 95
Diamètre 160 mm pour l'Astroloy.

3°) Compaction uniaxiale
Galette de 60 mm.

Malgré la diversité des méthodes de compaction et d'origine des poudres, on constate en premier examen que les caractéristiques sont peu différentes entre elles et satisfont au mirima des normes des matériaux forgés.

Un examen plus détaillé montre dans le cas du René 95 que le produit extrudé qui a une structure à grain très fin - 13 ASTM - possède les meilleures limite élastique et

résistance à la traction ; le produit compacté uniaxialement vient en second.

Pour l'Astroloy, le produit extrudé aurait encore à l'ambiante un léger avantage, mais à 650°C il est inférieur au produit brut de compaction.

La détermination des caractéristiques de fatigue lente et de propagation des criques serait nécessaire pour différencier éventuellement la qualité de ces trois types de produits.

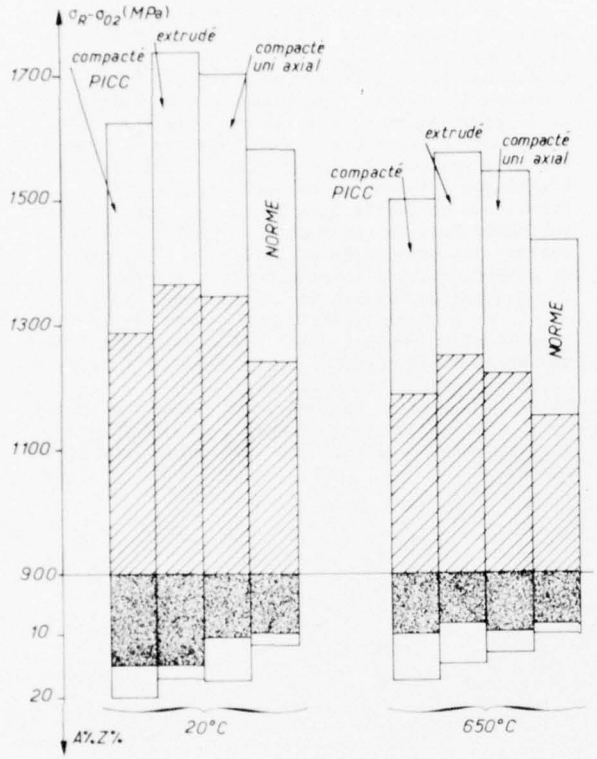

Fig. 23 — Caractéristiques en traction pour différents modes de densification pour du René 95.
Traitement thermique : Préchauffage 900°C 4 h
1095°C 1 h huile
760°C 16 h air

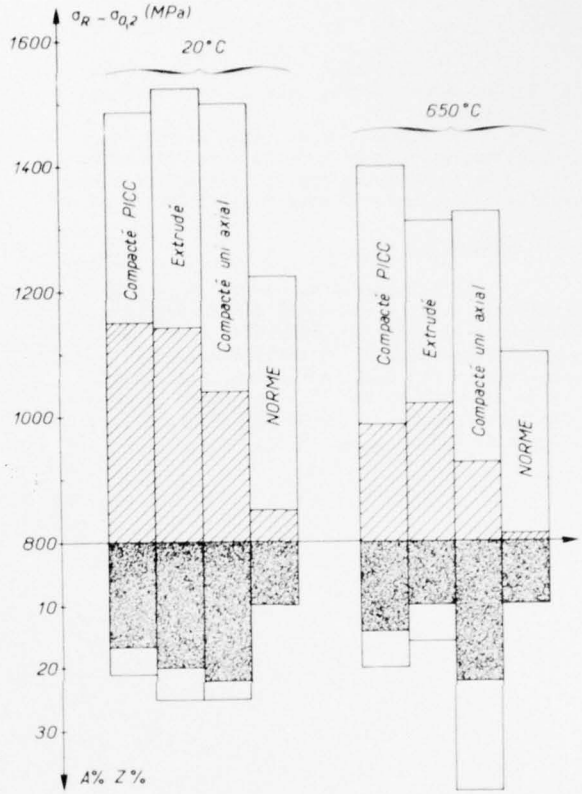

Fig. 24 — Caractéristiques en traction pour différents modes de densification pour de l'Astroloy bas carbone.
Traitement thermique : 1100°C 4 h huile
850°C 8 h air
980°C 4 h air
650°C 24 h air
760°C 8 h air

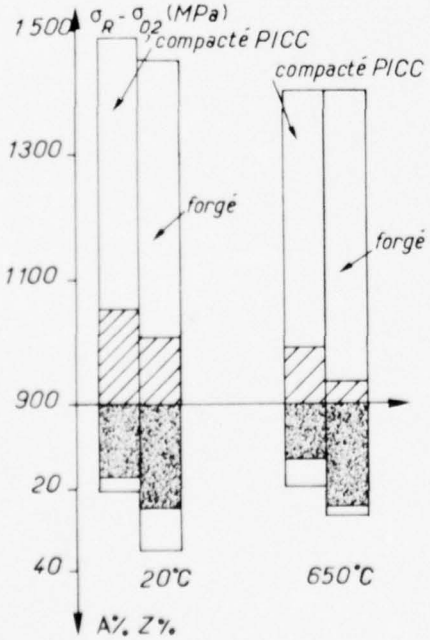

Fig. 25 — Influence d'une opération de forgeage sur les propriétés mécaniques d'alliage Astroloy bas carbone densifié par PICC.
Traitement thermique : 1100°C 4 h huile
850°C 8 h air
980°C 4 h air
650°C 24 h air
760°C 8 h air

VI - INFLUENCE DU FORGEAGE -

Le forgeage modifie les structures et améliore en général les caractéristiques mécaniques.

La figure 25 montre les caractéristiques de traction pour de l'Astroloy bas carbone, comparativement entre l'état brut de compaction et le même produit forgé avec des grains équiaxes, de façon à ne pas superposer un effet typique de la structure.

A l'état forgé, la limite élastique est légèrement abaissée, mais la ductilité est améliorée.

Cet essai démontre dans le cas de l'Astroloy, et pour un type de structure comparable, que le matériau brut de compaction a une qualité équivalente à celle du matériau forgé.

VII - CONCLUSION -

L'influence d'un certain nombre de paramètres de mise en oeuvre déterminant les structures et les propriétés mécaniques des superalliages élaborés à partir de poudres préalliées a été évaluée tant au laboratoire sur de l'IN 100 et de l'Astroloy que sur un plan plus industriel sur du René 95 et de l'Astroloy bas carbone.

Au niveau de l'élaboration des poudres, le procédé utilisé (électrode tournante ou atomisation à l'argon) en intervenant sur la granulométrie et la distribution des carbures peut influencer les structures et par conséquent les propriétés mécaniques des alliages densifiés.

La méthode de densification en provoquant, soit une forte déformation unidirectionnelle des grains de poudre (cas du filage), soit au contraire une déformation limitée (cas du pressage isostatique conventionnel ou rapide et de la compression uniaxiale) peut modifier complètement la structure des alliages après traitement thermique. En particulier, l'utilisation d'alliages à teneur en carbone relativement élevée est proscrite dans le cas où apparaissent des phénomènes de segrégation de carbures à la surface des particules de poudre, si le procédé de densification n'introduit pas une forte déformation détruisant ces films fragilisants.

Les caractéristiques du matériau brut de compaction peuvent être profondément modifiées par traitement thermique et celui-ci doit être adapté à l'application envisagée.

Quant au forgeage, qui constitue l'opération ultime de mise en oeuvre, il modifie les structures et peut améliorer les caractéristiques mécaniques.

CONTROL OF GRAIN STRUCTURE DURING SUPERALLOY POWDER PROCESSING

by

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SUMMARY

Factors which influence the control of grain structure during hot isostatic pressing of nickel-base superalloy powders are examined. The γ' precipitate can be used to control grain structure below the γ' solvus, while carbide precipitation on grain and particle boundaries controls the structure at higher temperatures. Carbon and sulphur are the main interstitials on particle boundaries. Flow properties, fracture behaviour and recrystallization during hot forging depend on temperature, strain, strain rate and initial grain structure of the compact. The effects of these forging variables on final microstructure are examined.

INTRODUCTION

Powder metallurgy processing of nickel-base superalloys has been actively pursued for at least 10 years, encouraged by promise of both technical and economic benefits. Experience has shown that powder metallurgy superalloys are a generically discrete class of materials that not only present special opportunities but also unique problems. It has been necessary to establish effective and economical ways of consolidating superalloy powders and to determine ways of controlling their microstructures and mechanical properties.

This paper examines means by which grain sizes and grain structures can be controlled in superalloy compacts and the influence of these on mechanical properties. The first section examines grain control during hot isostatic pressing, as it is influenced by powder type, powder chemistry, pressing conditions and subsequent heat treatment. Consolidation by hot isostatic pressing was chosen because of its potential in press to shape applications for parts or preforms. The second section examines factors that influence the choice of hot working conditions for superalloy preforms and the way that flow characteristics, workability and final microstructure can be influenced by the initial grain structure of the preform.

MICROSTRUCTURAL CONTROL DURING PRESSING

The conditions of temperature, time and pressure selected for hot isostatic pressing should be such that full densification and interparticle bonding are achieved. These require that the applied pressure is in excess of the flow stress of the powder at the pressing temperature, and that the temperature is sufficient to allow diffusion bonding to occur between particles. These conditions define broad temperature ranges within which considerable variations in microstructure can be achieved.

Pressing below the γ' solvus

The γ' $Ni_3(Al, Ti)$ precipitate can be used to impede recrystallization and grain growth so that the grain structure of the compact is essentially that of the original atomized powder. Thus the grain size of the compact can be controlled by the appropriate choice of powder size and type. Figure 1a shows a typical microstructure from a - 44 mesh AA† 713LC compact pressed below the γ' solvus. The structure is uniform and shows overaged γ'. The grain structure can be revealed using a partial γ' solution treatment plus age to precipitate carbides, Fig. 1b. The grain size is in the range 7-20 μm, and a prior powder particle boundary can be seen. By comparison, Fig. 1c shows the structure of a low carbon Mar M200 compact also pressed below the γ' solvus. The particle size was 90% less than 120 mesh and predominantly (75%) less than 170 mesh. The inherent grain size of the powder is now much finer and is retained during pressing to give grain sizes in the range 2-8 μm in the compact. Prior particle boundaries are less evident in this low carbon, high tungsten alloy, although these boundaries can be located.

Fine grained compacts of this type usually produce high yield and tensile strengths, and high tensile and stress rupture ductilities at low and intermediate temperatures.(1)

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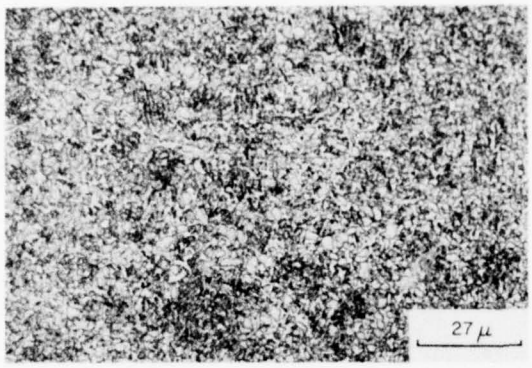
†AA, RE and VA refer to argon atomized, rotating electrode and vacuum (hydrogen) atomized powder respectively.

(a)



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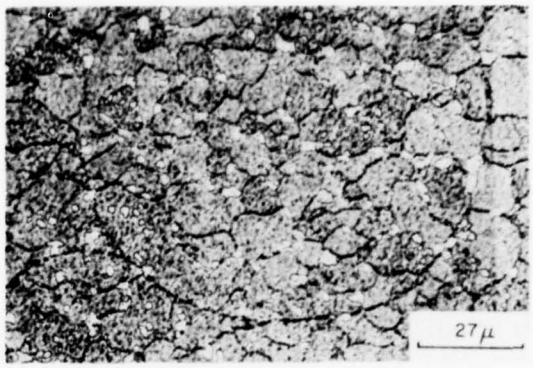


(c)

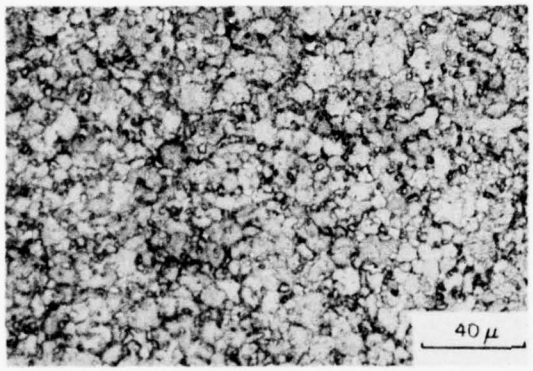


Fig. 1 Microstructures of compacts pressed below γ' solvus, (a) -44 mesh AA 713LC pressed 2 hrs./1190°C 103 MN/m^2, (b) as (a) plus a partial γ' solution treatment and age, and (c) -120 mesh AA, low carbon Mar M200 pressed 2 hrs./1050°C, 69 MN/m^2.

Pressing or heat treatment above the γ' solvus

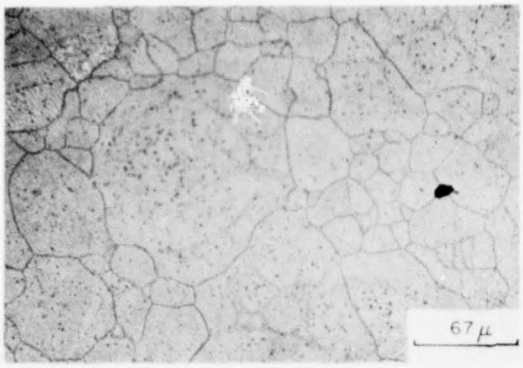
Grain structures other than those of the as-atomized powders can be obtained either by pressing or heat treating above the γ' solvus. The extent of recrystallization and grain growth depends on time and temperature of pressing and on the extent of carbide precipitation on grain and powder particle boundaries. These latter reactions are particularly dependent on the carbon and refractory metal content of the alloy, as discussed later.

Figure 2a shows the microstructure of a -60 mesh VA, 713LC compact, similar to that of Fig. 1a, after a complete γ' solution treatment. The material is fully recrystallized to a coarse grained (12 - 150 μm) structure and segments of prior particle boundary can be seen within the new grains. When this material is fractured by bending at room temperature the fracture is macroscopically brittle and the fracture surface contains many particle boundary facets, Fig. 2b. The fine details on these facets show micro-plasticity with ductile dimples nucleated at small second phase particles, Fig. 2c. The original as-pressed material (e.g. Fig. 1a) produces a predominantly ductile transgranular fracture. This demonstrates that interparticle bond strength is good, and better than the transgranular fracture strength when the γ' is in an overaged condition. When γ' is present as a more effective matrix strengthener the particle boundaries are the weak links in the structure. It also demonstrates that recrystallization alone is not sufficient to completely eliminate prior particle boundaries. We therefore need to determine the factors that influence the stability of these boundaries, and ways by which they can be eliminated or controlled.

Studies of as-atomized powders (AA, RE and VA) have revealed the presence of fine films on powder surfaces. These can be extracted chemically or electrolytically, and typical surface skins are shown in Fig. 3. Auger spectra, Fig. 4a, obtained from the interparticle fracture facets of Fig. 2b show that carbon and sulphur are the main interstitials segregating to surface boundaries. In contrast Auger spectra obtained from neighbouring areas show carbon but no sulphur, Fig. 4b. Also these spectra show little or no evidence of surface contamination by atmospheric impurities such as oxygen or nitrogen, and therefore the vacuum canning procedures used with these powders appear to be acceptably good. The particle surface films of Fig. 3, the fine precipitates of Fig. 2c, and any powder boundary pinning associated with them appear to be related primarily to the interstitials C and S, rather than to atmospheric impurities.

In alloys such as IN-100, very heavy carbide films are observed on powder boundaries after pressing,[2,3] and it is clear that the majority of this

(a)



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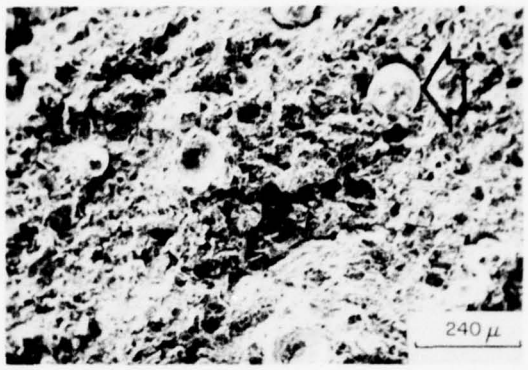

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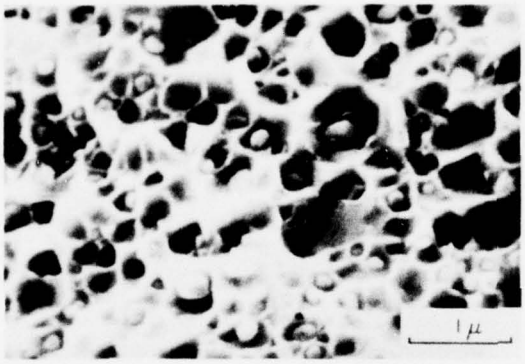


Fig. 2 Microstructures of -60 mesh AA, 713LC powder pressed 4 hrs./980°C plus 2 hrs./1180°C. The structure is similar to that of Fig. 1a after pressing. Recrystallization and grain growth is achieved by a full γ' solution heat treatment. This sample was also aged to emphasize the grain boundaries. (b) Interparticle fracture facets in material of Fig. 2a (solution treated only), and (c) fine details on the particle fracture facet marked in (b).

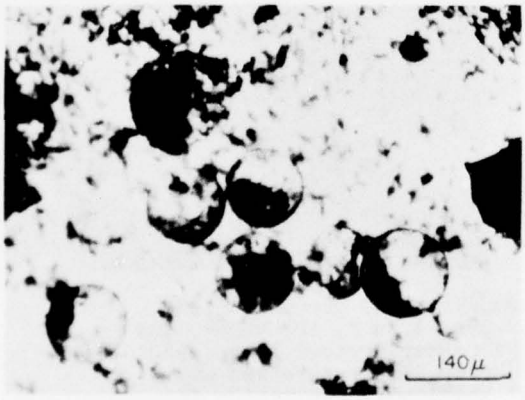


Fig. 3 Powder particle surface films extracted chemically from a -35 mesh RE 713LC powder. The films are shown suspended in methanol.

precipitation occurs during pressing, Fig. 5a. These heavy carbide networks impede growth of recrystallized grains and promote brittle interparticle fracture. Extraction and analysis of the carbides from as-atomized powders and their compacts, Table 1, shows that considerable amounts of carbon are retained in metastable solid solution in the powders due to their rapid rates of solidification and cooling. It is this carbon that migrates to and precipitates on grain and powder boundaries during pressing.[4,5] The reasons for the preferential precipitation on powder boundaries are not clear.

The decoration of powder boundaries by carbide can be minimized either by lowering the carbon content[6] or by increasing the refractory metal content of the alloy.[4,7] Figure 5b shows a low carbon IN-100 compact in which recrystallization occurred during pressing to produce an equiaxed grain structure with grain sizes in the range 20-30μm. In alloys such as Mar M200 or Mar M246 with high tungsten or tantalum contents, carbon migration to powder boundaries is minimal and carbides precipitate more uniformly in grain boundaries and grain interiors. Figure 6a shows a grain size of 20-100 μm obtained by long time, high temperature pressing (20 hours at 1304°C, 187 MN/m^2) with

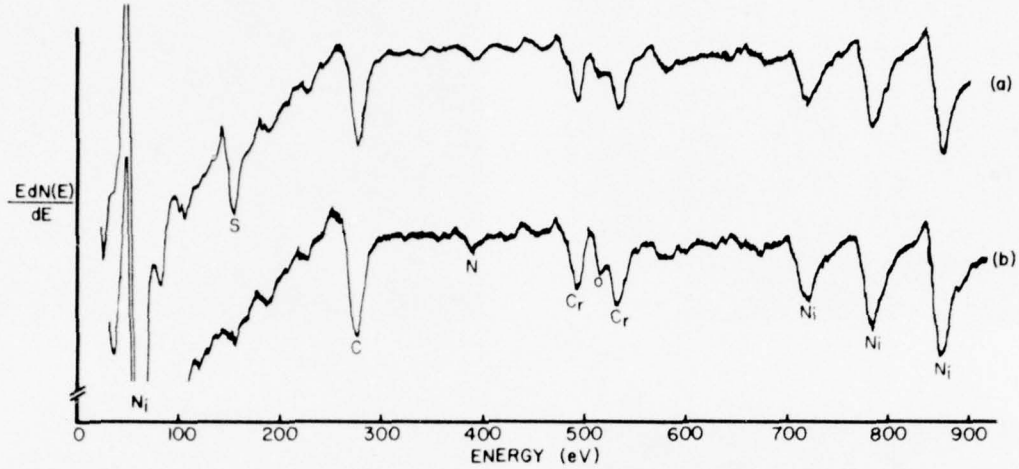


Fig. 4 Auger spectra obtained from (a) the particle fracture facet shown in Fig. 2b and 2c, and (b) from a neighbouring non-particle area, note absence of sulphur. Samples fractured under vacuum, incident beam diameter <20μm.

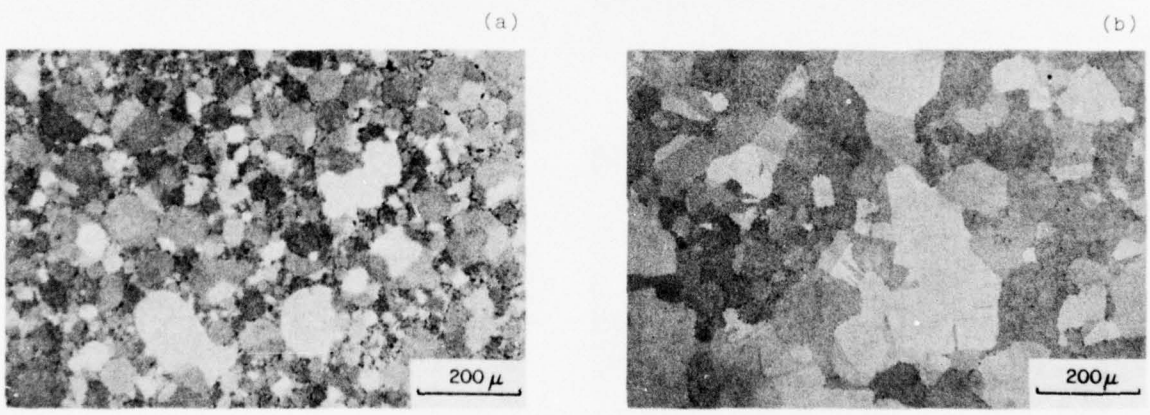


Fig. 5 Microstructures of IN-100 compacts, (a) carbon content 0.17%, -60 mesh AA powder, pressed 2 hrs./1270°C, 103 MN/m^2, and (b) carbon content 0.03%, -60 mesh VA powder pressed 4 hrs./980°C plus 2 hrs./1180°C, 138 MN/m^2. The upper temperature was above the γ' solvus in both cases. In (a) the powder boundaries are pinned by secondary TiC carbide particles.

-20 mesh, VA Mar M246 powder having a carbon content of 0.14%, Fig. 6b shows a similar grain size obtained under less severe conditions with a low carbon Mar M200 powder. This was the same powder used to produce the fine grained material of Fig. 1c. The recrystallization and considerable grain growth achieved here is due to the absence of γ' and limited grain boundary pinning by carbides.

Pressing above the solidus temperature

Extremely coarse grained compacts (200-800 μm grain diameter) can be obtained using heat treatments above the solidus of the alloy.[8] Under these conditions, and in the presence of a liquid phase, carbides and other obstacles are taken into solution and rapid grain growth occurs. Unless these treatments are carried out under pressure as part of the HIP cycle considerable porosity will occur, due both to the condensation of entrapped or absorbed gases and to the irreversible plastic expansion of solid metal

TABLE 1

Minor phase contents expressed as weight % of the total alloy digested in as-cast stock, as-atomized powder and HIP compacts.

	Weight % minor phase content			
	AA 713LC	RE 713LC	VA IN-100	Low carbon VA-IN-100
As-cast	0.55		0.92	0.18
Powder	0.29	0.32	0.71	0.03
Compact	0.83		1.54	0.23

(a) (b)

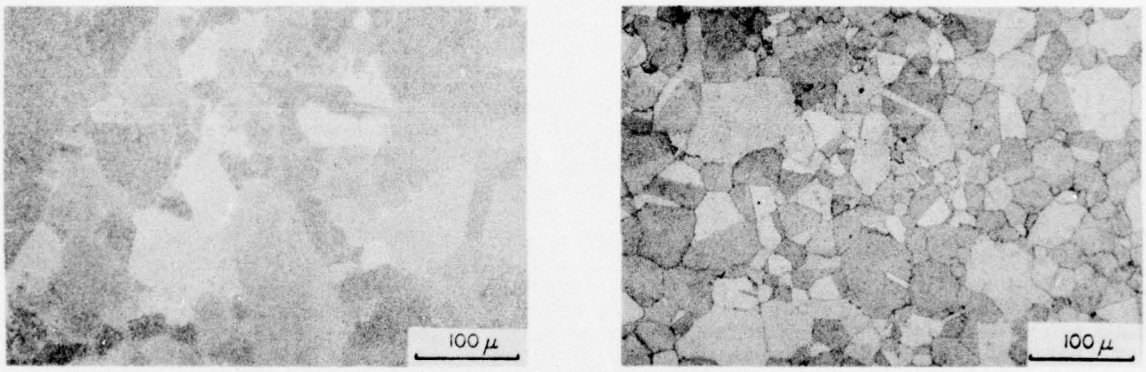


Fig. 6 Microstructures of (a) -120 mesh, VA Mar M246 with 0.14% carbon, pressed 20 hrs./1304°C, 187 MN/m^2 and (b) low carbon -120 mesh AA Mar M200 pressed 2 hrs./1250°C, 103 MN/m^2.

around liquid pools. A typical grain structure produced in this way from -35 mesh, RE 713LC powder is shown in Fig. 7.

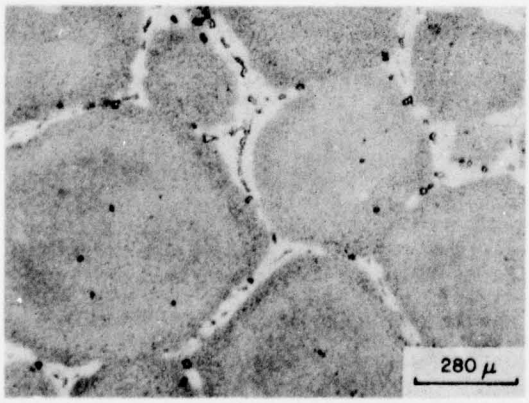


Fig. 7 Coarse grained material obtained from -35 mesh RE 713LC powder, pressed 20 hrs./1304°C, 187 MN/m^2. The grain boundary constituents are (Nb,Ti)C and $(Cr,Mo)_{23}C_6$ carbides and $(Ti,Zr)_2SC$ sulphocarbides. Refs. 8 and 9.

While improved high temperature stress-rupture lifetimes can be obtained in these compacts, due to their coarse grain sizes, ductilities at all temperatures are poor and fractures are invariably intergranular.(8) Grain boundary embrittlement is associated with the segregation of carbon and sulphur to liquid grain boundary pools. During solidification and cooling these elements form MC and $M_{23}C_6$ carbides and hexagonal plates

of M_2SC sulphocarbide.(8,9) Auger spectra, Fig. 8, from intergranular fracture facets again show carbon, or carbon and sulphur together as the main interstitials, and little evidence of atmospheric contaminants.

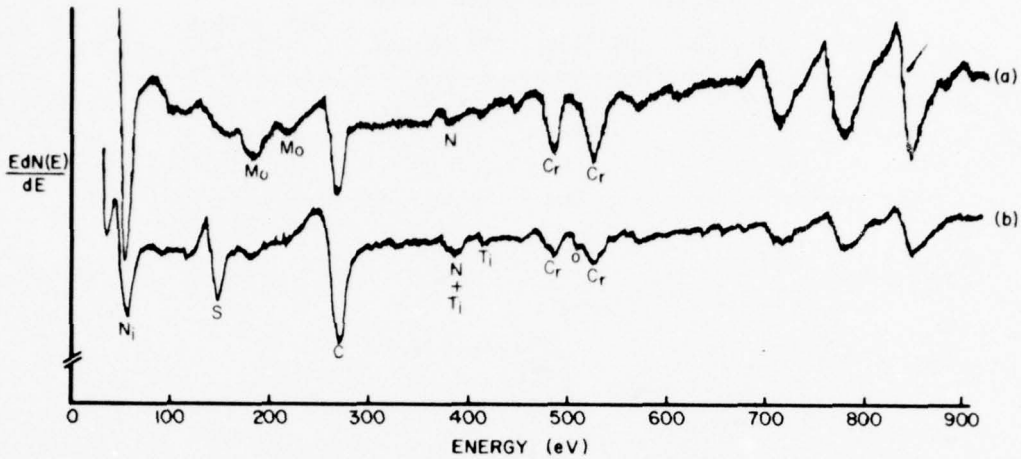


Fig. 8 Auger spectra obtained from intergranular fracture faces from a sample of the type shown in Fig. 7, (a) from a grain face containing mainly $(Cr,Mo)_{23}C_6$ carbides, and (b) from a grain face containing a $(Ti,Zr)_2SC$ sulphocarbide plate. Sample fractured under vacuum, incident beam diameter <20μm.

Mechanical properties of as-pressed compacts

A selection of mechanical properties from some of the compacts examined above is given in Table 2, together with data for cast 713LC and wrought Udimet 700.(10) These data are presented as typical blade and disc alloy properties respectively, but they do not represent the best available materials for these applications. The data for the compacts are from as-pressed, or from pressed and heat treated materials, the heat treatments being used either to adjust grain size or to develop particular precipitate structures.

These data show that several of the fine grained compacts examined produce room temperature tensile properties better than those of cast 713LC and similar to those of wrought Udimet 700. Similarly at intermediate temperatures, stress rupture properties of HIP compacts can be better than those of cast 713 LC. However, they usually fail to produce the combination of long rupture life and high ductility of good wrought material. At 980°C, fine grained compacts yield dramatically short rupture lives, and the longest rupture life obtained in a coarse grained compact of the type shown in Fig. 7 is only about half that of cast 713LC.

The above results suggest that as-pressed HIP compacts are unlikely to compete mechanically with conventional materials. The best promise for HIP compacts appears to be as preforms for subsequent thermo-mechanical processing where further plastic deformation may help to eliminate surface boundary effects, to improve grain size control and to develop substructure strengthening.

MICROSTRUCTURAL CONTROL DURING FORGING

Forging after pressing can be used to further the shaping process and also to develop a preferred microstructure. The potential of the two stage, press plus forge approach is indicated by Fig. 9a, which shows a disc in the form of the high compressor turbine disc of the PWA-JT15D engine. The disc was forged in a single deformation from a HIP Astroloy preform and finish machined, thus saving on intermediate forging steps and allowing the shaping of a hitherto difficult-to-work alloy. The development and testing of this disc have been described elsewhere,(5) suffice it to say that evidence of poor fracture toughness was found with sudden brittle fracture occurring during spin-rig testing. Examinations revealed zones of non-recrystallized material in the highly stressed areas adjacent to the bore, Fig. 9c, and brittle cracks were found to have propagated radially away from the bore along powder surface boundaries in these regions.

This example demonstrates that attention must be paid to the forging process to ensure that the preferred microstructure is obtained. Data are therefore required on the

TABLE 2

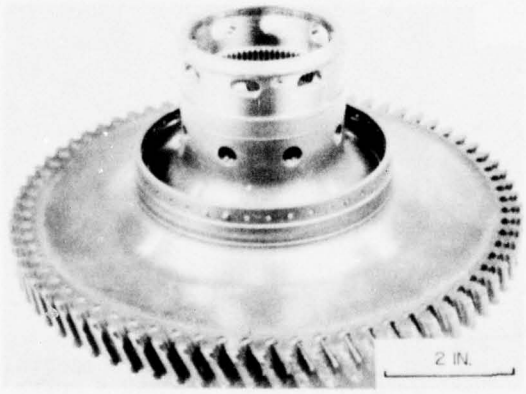
Mechanical properties from some as-pressed, or pressed and heat treated superalloy compacts

Material and Condition	RT. Tensile			Stress Rupture Properties				For grain structure see Fig. or Ref.	
	Yield MN/m²	UTS MN/m²	Elong %	760°C/586 hrs.	MN/m² %El	980°C/152 hrs.	MN/m² %El		
As cast 713LC machined from bar	726	880	5.7	36.8	2.6	63.1	5.0		
Wrought Udimet 700	896	1307	12.5	55.5	18.8	----	----		10
713LC HIP @ 1200°C	786	1317	19.0	45.7*	6.3	0.1	10.1	1a, 1b	1
As above plus 3 stage heat treat	931	1296	10.1	59.5*	4.1	2.9	3.6		1
IN-100 HIP @ 1200°C	910	1089	7.1	0.1	1.8	<0.1†	3.0		
IN-100 HIP @ 1270°C	868	1262				0.7†	4.4	5a	
As above plus 2 stage heat treat	1000	1289				2.2†	1.0		
Mar M246 HIP @ 1304°C	745	1069	6.8			5.0	2.6	6a	
713LC HIP @ 1304°C	728	818	4.8	23.6	1.7	9.3	2.0	7	8
As above plus 4 stage heat treat	915	935	4.2	38.7	2.0	33.1	1.0		8

*Tested at 530.9 MN/m²

†Tested at 200 MN/m²

(a)



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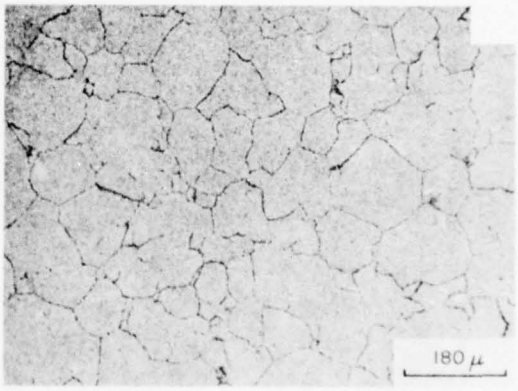


(c)

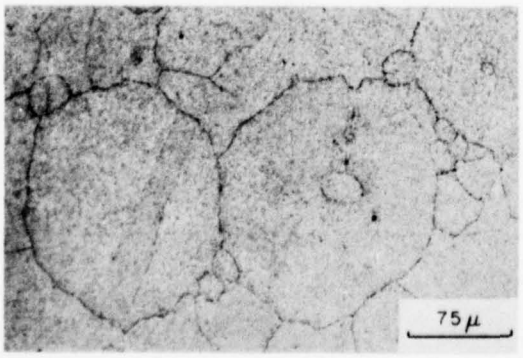


Fig. 9 (a) A powder fabricated Astroloy disc in the form of the high compressor turbine disc of the PWA JT-15D engine, (b) showing the equiaxed recrystallized grain structure observed in the rim of the disc, and (c) non-recrystallized particulate microstructure observed in the bore region. Ref. 5, reproduced by courtesy of the Canadian Metallurgical Quarterly.

influence of the important forging variables of temperature, strain, and strain rate on the flow properties, fracture, and recrystallization behaviour of compacts. Also since the grain structure of the initial preform can be controlled to some degree, the influence of this on the forging process should be determined.

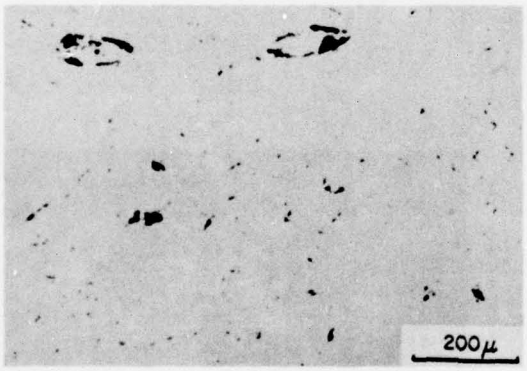
Current work at the National Aeronautical Establishment is concerned with these problems: a 10,000 kg MTS hydraulic testing machine has been modified for high temperature axisymmetric compression of cylindrical specimens. Subscale forging blanks (10 mm high) can be processed at temperatures up to 1200°C and at constant true strain rates in the range from 10^{-5} to 10 s^{-1}. In order to retain the hot work structures for metallographic examination, the strained specimen can be quenched within 1 s of the end of deformation. In this way, the effects of the processing variables on the flow and fracture characteristics of the superalloy compacts can be examined and some of the early results are described below.

So far, the high temperature flow characteristics of two HIP P/M superalloys have been investigated: a VA IN-100 of standard chemistry pressed below the γ' solvus and two batches of low carbon MAR M200 pressed above and below the γ' solvus.* The as-compacted IN-100 exhibits a microstructure of overaged γ' in the austenitic γ matrix similar to that shown in Fig. 5a. The powder particle boundaries are decorated by a continuous thin film of TiC. The microstructures of the MAR M200 pressed below and above the γ' solvus are shown in Fig. 1c and 6b respectively, the major difference being their grain sizes. As emphasized previously, the powder particle boundaries in these pressings are not noticeably decorated by carbides. Compression tests have been carried out at intervals of 50°C in the range from 1050 to 1200°C and at five strain rates between 3 × 10^{-4} and 1 s^{-1}.

Effects of Processing Variables on Ductility

Cracking occurs in powder particle boundaries during compression of the IN-100 compacts as evidenced by the photographs of Figs. 10 and 11. The severity of the cracking varies with the processing conditions and this has been used to obtain a qualitative estimate of the relative ductilities from one test condition to another.

(a)



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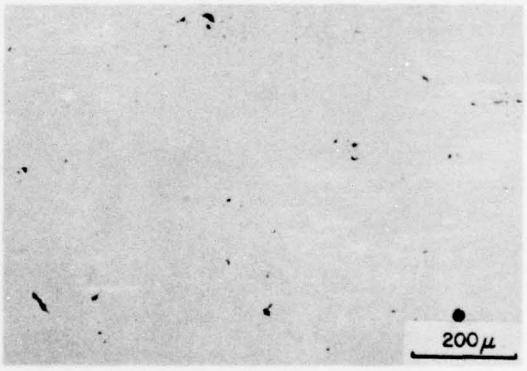

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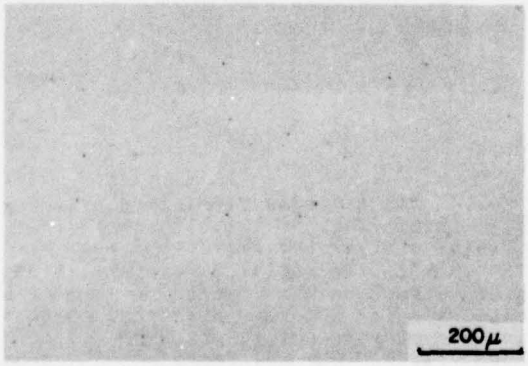

Fig. 10 Effects of temperature on the hot ductility of IN-100 compacts, (-60 mesh VA powder, pressed 4 hours at 1010°C/138 MN/m² followed by 2 hours at 1180°C/138 MN/m²); compressed at a constant true strain rate at 10^{-2} s^{-1} to a strain of 0.8 at a) 1050°C, b) 1150°C, c) 1200°C.

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*The respective HIP cycles involved:
IN-100: 4 hours at 1010°C/138 MN/m² followed by
2 hours at 1180°C/138 MN/m²

MAR M200
1) 2 hours at 1050°C/69 MN/m²
2) 2 hours at 1250°C/103 MN/m²

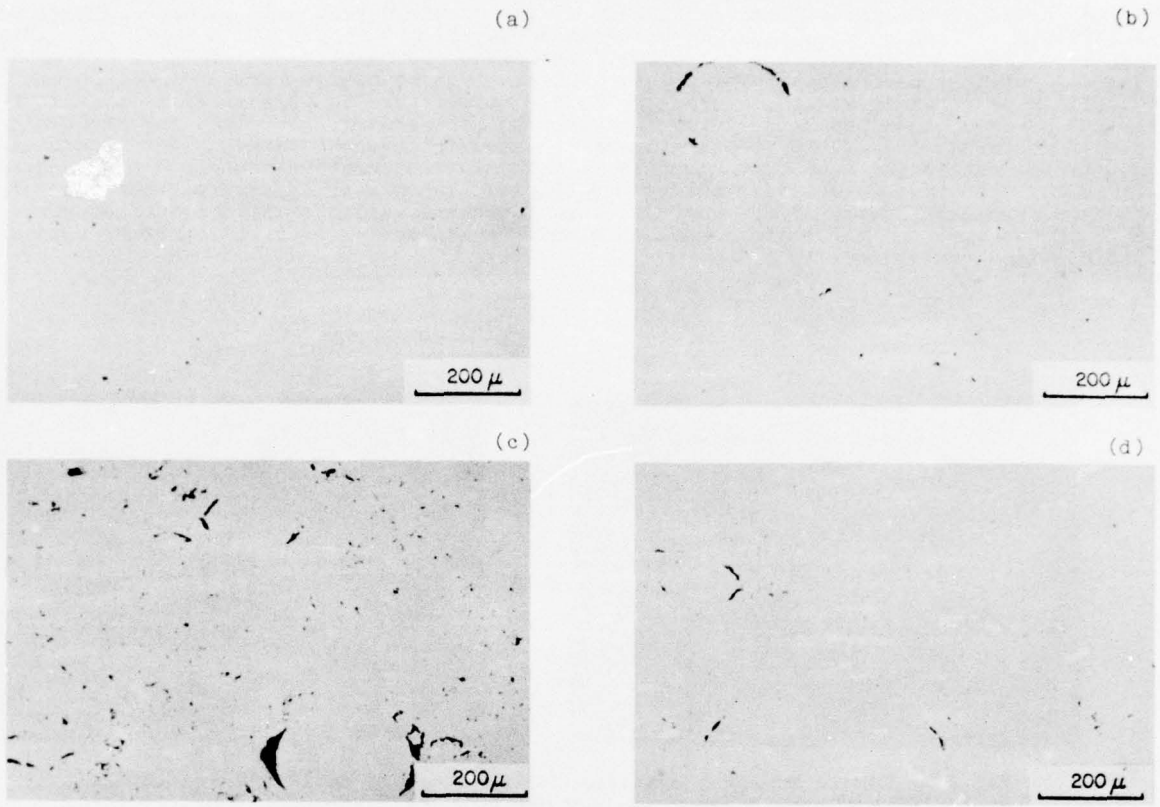

Fig. 11 Effects of strain rate on the hot ductility of IN-100 compacts (-60 mesh VA powder, pressed 4 hours at 1010°C/138 MN/m^2 followed by 2 hours at 1180°C/138 MN/m^2); compressed to a strain of 0.6 at 1050°C and at a) 3.0×10^{-4} s^{-1}, b) 1.7×10^{-3} s^{-1}, c) 10^{-1} s^{-1}, d) 0.98 s^{-1}.

The ductility of the powder compact increases with temperature as illustrated in Fig. 10. The soundness of the compact forged at 1200°C is reasonable considering that some of the apparent porosity is due to carbides being pulled out of the matrix during polishing. This increase in ductility with temperature is characteristic of the hot working of superalloys and is well documented.(11) However, over the temperature range examined no ductility minimum or maximum was observed as reported for solution strengthened(12) and precipitation strengthened(13) austenitic matrices.

It is generally accepted that the major factor governing the hot ductility of metals and alloys is the nature of the softening process concurrent with deformation.(14) The increase in ductility with temperature can be easily rationalized: the compacts recrystallize dynamically during forging and the gradual dissolution of the second phase particles, with increasing temperatures, increases grain boundary mobility thereby isolating initial cracks and preventing further propagation.

Over the ranges of strain rate examined, and at a fixed temperature, the apparent ductility increases with decreasing strain rate. A minimum in ductility as a function of strain rate was observed at the lowest test temperature. This effect is illustrated in Fig. 11. It can be seen that the severity of cracking is a maximum at a strain rate of 10^{-1} s^{-1} and improves with either increase or decrease in forming rate. This ductility minimum was not detected at the higher temperatures, possibly due to the limited range of strain rates investigated.

There is evidence that ductility depends critically on strain rate in both single phase(12,15) and duplex structures.(13,16) In an austenitic stainless steel,(12) for example increasing the strain rate improves the ductility in the temperature range from 950 to 1150°C. Above 1200°C, however, the effect is reversed. Torsional ductility data on Udimet 700(13) also show evidence of such a reversal in the vicinity of 1000°C. Adiabatic heating, at the higher strain rates has been previously suggested to lead to higher ductilities.(17) It is more likely, however, that the reversal observed at 1050°C in the IN-100 compacts is the result of interactions between the dynamic softening mechanisms and the fracture mechanisms. Crack propagation processes can be modified by the structural changes taking place during forging, and since these changes are a function of the strain rate it can be expected that crack propagation processes and hence ductility will vary with forming rate. Work is presently in progress which should help clarify the nature of the mechanisms of ductility improvement with strain rate variations as well as the nature of the embrittlement at intermediate strain rate.

The available evidence suggests that powder boundary cracking is due primarily to the presence of embrittling carbides.(2) The morphology of this carbide film also appears to influence ductility. It has been shown, Fig. 5a, that if an IN-100 of similar chemistry is pressed above the γ' solvus,* the powder boundaries contain networks of a coarser but more discrete carbide phase. This appears to have a marked effect on the ductility as shown in Fig. 12: cracking is far more severe in this material than in the IN-100 pressed below the γ' solvus. However, the HIP canning procedures for the two alloys differed, the 1270°C pressing being produced from vacuum canned powder whereas an additional outgassing treatment was used with the low temperature pressing. The ductility may also be influenced by the γ' morphology. These results suggest that the thermomechanical history of the compact is an important variable influencing hot workability and that the HIP + forge processes must be considered as a whole rather than as two distinct operations.

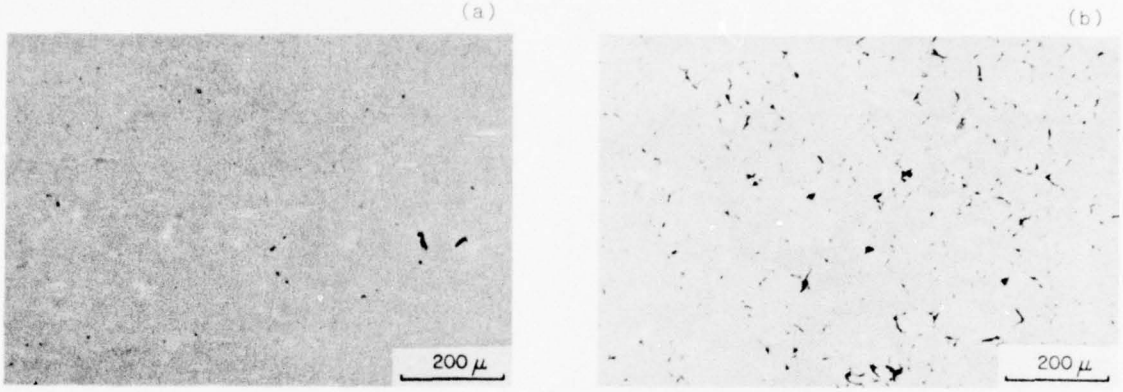

Fig. 12 Powder boundary cracking during forging, at 1150°C and $10^{-2}s^{-1}$, of IN-100 compacts pressed a) from -60 mesh VA powder for 4 hrs./1010°C/138 MN/m² followed by 2 hrs./1180°C/138 MN/m²; and b) from -60 mesh AA powder for 2 hrs./1270°C/103 MN/m². The compacts were forged to the same strain of 0.4.

Effects of Processing Variables on Strength

The effect of strain rate and temperature on the high temperature flow stress of the IN-100 compact, pressed below the γ' solvus, is shown in Fig. 13. The flow behaviour is typical of metals and alloys which undergo dynamic recrystallization during hot working.(18) At constant strain rate and temperature the flow curve is characterized by a high rate of work hardening at low strains and by a subsequent rapid flow softening until, at high strains, a steady state regime is established. The peak flow stress and the peak strain increase with strain rate and decrease with temperature. Similar trends have been reported for Inconel 600(19) and Udimet 700(13) deformed in torsion. Such behaviour would be expected during hot forming of nickel-base superalloys.(20)

The hot strength of a powder compact is also markedly affected by its grain size, and therefore by the choice of powder size and hot isostatic pressing conditions. To illustrate this point, two compacts of MAR M200, with different grain sizes have been forged under the same conditions of strain rate and temperature. The microstructures of the two HIP compacts have been described previously: pressing below the γ' solvus results in a fine grain material, Fig. 1c, whereas pressing above the γ' solvus leads to recrystallization and considerable grain growth, Fig. 6b. During subsequent forging, at 1050°C and and 3×10^{-4} s^{-1}, the compacts exhibit different flow behaviours, as illustrated in Fig. 14. The coarse grain material shows a six fold increase in peak flow stress over the fine grain compact and undergoes more strain softening. The steady state flow stress, is also higher in the coarse grain material.

The advantages of lower working loads during forging are self evident and, on these grounds, pressing below the γ' solvus appears preferable for HIP + forge thermomechanical treatments. Moreover, the hot ductility is improved by the presence of a fine grain structure as illustrated in Fig. 15. The two micrographs are for the compacts whose compression flow curves are shown in Fig. 14. The microstructures show evidence of recrystallization particularly when they are compared to those of the as-pressed materials. The fine grain compact appears to have recrystallized during forging to an even finer grain size. Deformation occurs homogenously throughout this fine grain material and little or no cracking occurs. In the coarse grain compact, the recrystallization is confined to the prior grain boundary regions and deformation appears to be localized to these soft recrystallized bands. Although a true strain of 1.0 was achieved in this test the initial coarse grains show little evidence of deformation. This is

*HIP cycle: 2 hrs. at 1270°C/103 MN/m²

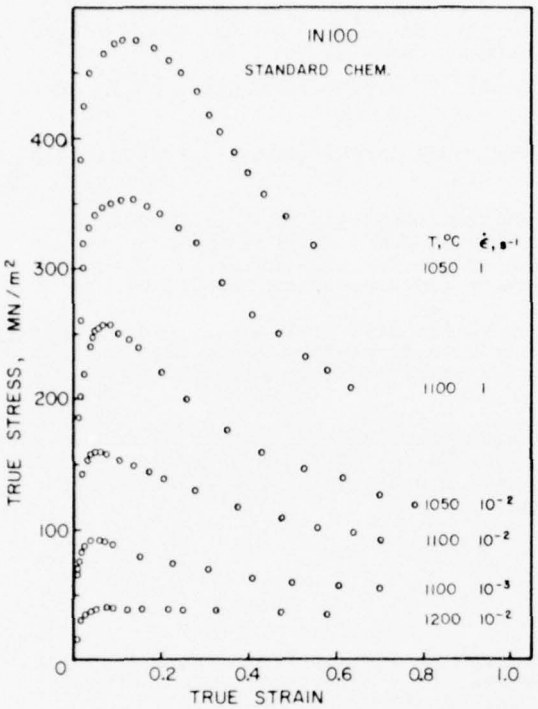

Fig. 13 Effects of strain rate and temperature on the high temperature flow curve in IN-100 compacts, -60 mesh VA powder pressed 4 hrs./1010°C/ 138 MN/m^2 followed by 2 hrs./1180°C/ 138 MN/m^2; deformed in compression at constant true strain rate.

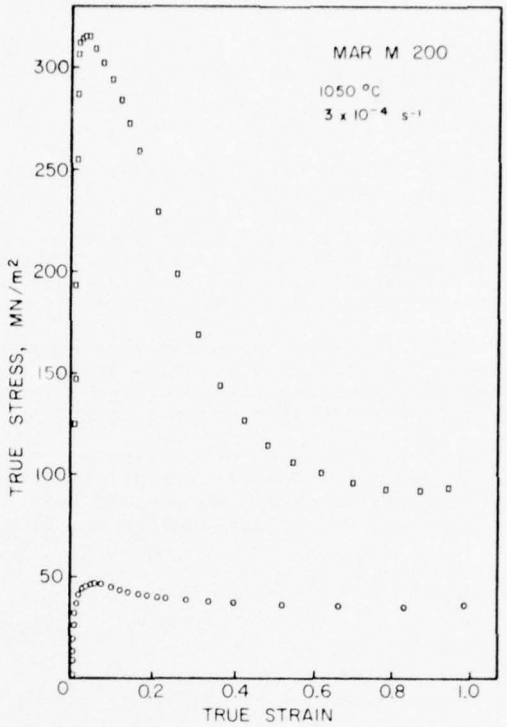

Fig. 14 Effects of thermomechanical history on the high temperature flow curve in Mar M200 compacts pressed
a) 2 hrs./1050°C/69 MN/m^2 ○
b) 2 hrs./1250°C/103 MN/m^2 □

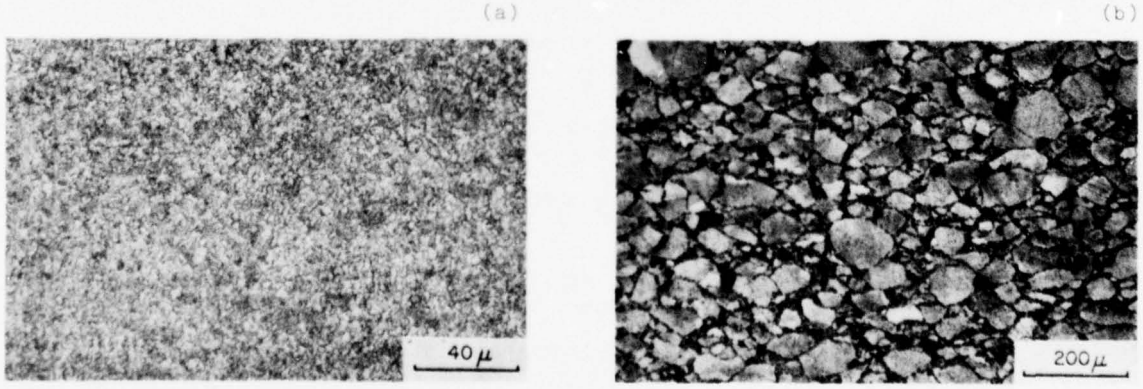

Fig. 15 Effects of thermomechanical history on the microstructure of -120 mesh AA low carbon Mar M200 compacts upset forged at 1050°C and at a constant true strain rate of $3\times10^{-4}s^{-1}$, a) pressed 2 hrs./1050°C/ 69 MN/m^2, and b) pressed 2 hrs./1250°C/103 MN/m^2.

further evidence that deformation is confined to the original boundary regions. This concentration of strain in the recrystallized grain boundary regions leads to the early accumulation of the critical fracture strain and appreciable cracking is observed in these compacts after forging. Thus both strength and ductility are markedly affected by prior thermal history, and improved forging properties are obtained with fine grained compacts pressed below the γ' solvus.

CONCLUSIONS

1. The grain sizes and mechanical properties of HIP superalloy compacts can be controlled by appropriate choice of powder type and pressing conditions.

2. Fine grained compacts can be obtained by pressing below the γ' solvus while progressively coarser grain sizes are obtained above the γ' solvus.

3. Recrystallization and grain growth are influenced by carbide precipitation on grain and powder surface boundaries.

4. Carbide precipitation on powder surface boundaries is influenced by HIP conditions and alloy chemistry.

5. The ductility of superalloy compacts at hot working temperatures is influenced by prior thermal history through its influence on grain size, grain boundary structure and γ' morphology. Ductility is also affected by the forging variables of temperature and strain rate, increasing with temperature and decreasing with strain rate.

6. The flow behaviour of superalloy compacts also varies with grain size, temperature, strain and strain rate. Flow stresses decrease with decreasing grain size and strain rate, and increase with decrease in temperature. Variations in flow stress with strain decrease with decreasing grain size.

7. Superior hot working properties are obtained with fine grained compacts pressed below the γ' solvus. However further studies are required to fully characterize the hot working behaviour and subsequent heat treatment and mechanical properties of superalloy compacts; these studies are in progress.

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Acknowledgments

Several of the studies reported above have been supported by Pratt and Whitney of Canada Limited and by Westinghouse Canada Limited. The support of these organizations, and also the many discussions and considerable assistance given by their engineering personnel are gratefully acknowledged. One of the authors (BDP) would like to thank the Science Research Council (UK) for support while carrying out the Auger Spectroscopy examinations.

POWDER FABRICATION OF FIBRE-REINFORCED SUPERALLOY TURBINE BLADES

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SUMMARY

Westinghouse Canada is conducting a program to develop a high temperature composite material and the fabrication processes to produce composite turbine vanes and blades for advanced industrial gas turbine engines. Blade and vane designs suited to the composite material are part of the overall program. The composite system includ the starting matrix, Mar M-200 nickel base superalloy powder, and the fibre, tungsten -2 thoria wire (0.5 mm diameter). The main objective is to develop a composite materia for blades and vanes to withstand temperatures of 1100°C with potential to 1200°C.

Processes under development include: (1) a chemical vapour deposition process to deposit continuously hafnium carbide or hafnium nitride diffusion barrier coatings on the tungsten wire, (2) composite monolayer stacking techniques, and (3) hot isostati pressing to form the consolidated net or near-net composite shapes.

From a materials viewpoint, the program is investigating the limitations impose by: (a) matrix-fibre interaction during fabrication and service exposure; (b) dimensio changes as a result of thermal cycling; (c) oxidation and hot corrosion requirements. Fabrication limitations center around consistently producing the desired fibre placemen and volume fractions in blade and vane shapes.

INTRODUCTION

All aircraft and industrial gas turbine manufacturers have increased their engine cycle temperatures in order to improve their overall thermal efficiency or specific power output for a given machine size. These temperature increments have been achieved largely by material substitution or by improved cooling techniques on blading materials. Further improvements in efficiency are essential if a manufacturer is to participate in future markets.

Figure 1 illustrates the performance improvements resulting from changes to first row vanes and blades in a regenerative cycle mechanical drive gas turbine engine. At 1975 natural gas prices, an increase of 2.8 percent in thermal effieiency, resulting from the changes shown, would result in a fuel saving of approximately $150,000 per yea of operation.

Reviews of potential materials systems which can meet advanced airfoil temperat goals were made. They indicated that significant temperature increases of up to severa hundred Celsius degrees could be obtained by research and development efforts in artificial metal matrix composites, directionally solidified (DS) eutectic composites, and ceramics. Figure 2 is a log-log plot of specific stress rupture strength against rupture time showing extrapolated 10^5 hours strength for some of the above materials (1, 2, 3, 4). The composite material curves were calculated on the basis of the matrix alloy making no strength contribution. The plot shows that the high strength W-2ThO_2 and the experimental W-Re-Hf-C composite materials can potentially meet our 1100 C specific strength objective of 1700 m for more than 10,000 hours. Besides mechanical property considerations, comparisons were made for factors such as chemical stability, oxidation resistance, fabricability, economics, and system flexibility. The latter refers to the ability to alter the matrix and reinforcement components independently.

Superalloy matrix-tungsten alloy composites ranked highest in absolute mechanical properties, fabricability, economics and system flexibility, but lowest in chemical stability. Directionally solidified eutectics rank high in chemical stability but lowest in system flexibility, strength and oxidation resistance. Ceramics such as silicon nitride and silicon carbide may have more long range potential than artificial or DS eutectic composites but the latter two are superior, in that they can have ductil properties similar to the superalloys now in use and hence fit in with the present design technology and experience. Ceramic materials are brittle and their utilization in a gas turbine is dependent on the development of the design technology to use brittl materials in the gas path. Composite airfoils also require modified design approaches but to a lesser extent than ceramics.

In 1973, a composite airfoil development program was initiated by the Gas Turbi Systems Division of Westinghouse Electric Corporation. The total program involved studies into (a) the physical metallurgy of superalloy matrix and tungsten alloy compos systems, (b) the determination and optimization of mechanical properties, (c) the desig and structural analysis of composite airfoils and (d) the development of manufacturing processes and quality control techniques. It is the latter task that Westinghouse Canada is primarily concerned with, although participation in the other tasks is required.

The fabrication phase involves development in high technology areas such as continuous wire coating, monolayer composite ply production techniques, powder metallurgy compaction by hot isostatic pressing, and thermomechanical processing (TMP). Nondestructive testing techniques, that need development to ensure the quali of the composite material throughout its processing, include ultrasonic and radiogra inspection.

A representative first row turbine blade that the composite material would re is shown in Figure 3. Presently this blade is produced by investment casting and we approximately 5 kilograms. It is characterized by a short, slightly twisted, tapere airfoil section, and a bulky root section.

COMPOSITE BLADE FABRICATION PROCESSES

Approach

This section describes the fabrication approach that is under development at Westinghouse Canada and the individual processes that together result in composite airfoil shapes.

The selected composite fabrication techniques must result in composite proper that meet those required for application of the composite. The processes must be ca of first producing vane and blade shapes to required dimensions, second incorporating both uniaxial and off-axis fibre positioning, third providing uniform matrix cladding to prevent fibre oxidation, and fourth providing if necessary, for cooling or airfoil weight reduction passages. The combined fabrication techniques must of course be cos effective and reproducible. The processing route under development at Westinghouse Canada is shown in Figure 4.

Commercial and development grades of thoriated tungsten wire (0.5 mm diameter are procured from the Lamp Divison of Westinghouse Electric Corporation. It was confirmed that a diffusion barrier coating was required on the fibre to prevent a strength loss as a result of nickel-induced recrystallization above 1000°C. Studies indicated that 4 micron thick coatings of hafnium carbide or hafnium nitride of suita stoichiometry were effective diffusion barriers. Chemical vapour deposition (CVD) wa the process selected to deposit continuously both of these coatings.

The need for uniaxial and off-axis positioning of thousands of fibres in a twisted, variable thickness airfoil shape ruled out molten metal infiltration as a technique for combining the fibres and matrix alloy. It was decided that stacking of monolayer composite plies was potentially a cost effective method of fabricating the desired shapes. The plies would consist of aligned coated fibres sandwiched between layers of matrix alloy. The matrix alloys considered were generally cast nickel base superalloy compositions. Matrix tape can be produced commercially from alloy powders combined with suitable organic binders and plasticisers.

Stacked plies could be consolidated by pressure and heat between hot dies or hot isostatic pressing (HIP). The latter process was selected since it was considere to be more adaptable to producing combined root-airfoil shapes, and had applicability other areas.

The primary objective of the fabrication process is to produce vane and blade shapes which are close enough to final dimensions such that root machining and touch- grinding of the airfoil is all that is required to attain the desired final shape. T is defined as a net shape. It is recognized that this net shape objective may not be attained since distortion during HIP of the envelope containing the composite airfoil preform stack and matrix alloy powder will be difficult to eliminate completely. For this reason, the develo ment includes a study of thermomechanical processing (TMP), s as isothermal forging or creep forming, as a means of attaining final airfoil dimensi from near-net shapes.

The need for a protective surface coating is considered to be a requirement f thousands of hours of operation with metal surface temperatures of 1100°C and above. Commercial modified aluminide pack cementation coatings, physical vapor deposited MCrAlY overlays, or a combination of the two, have shown potential for 1100°C use for one to two thousand hours of operation in pressurized burner rig tests before recoati is required. For industrial gas turbines, it is desired that this service life be extended to at least 10,000 hours before recoating. The requirement may impose a limitation on the use of the composite material.

Machining is required to produce blade root serrations, and vane shrouds. Conventional techniques such as broaching, grinding and milling can be used for these requirements. Attaining final airfoil dimensions from a net shape may require techni and cost evaluations of techniques such as contour grinding, electrical discharge grinding or machining, and electrochemical machining.

Optimum matrix alloy properties are obtained by heat treatment. Consideratio include fibre-matrix interaction, and matrix alloy grain size and phase morphologies after solution heat treatment and aging.

Fibre Coatings by Chemical Vapour Deposition

The need for diffusion barrier coatings on thoriated tungsten fibres was ascertained by performing compatibility tests at elevated temperatures. Uncoated fibres were combined with low carbon Mar M-200 powder, HIP-consolidated at 1200°C and 70 MPa, and exposed in vacuum for periods of time up to 1000 hours at 1100°C - 1200°C. Figure 5 (a) illustrates the recrystallization occurring in the tungsten fibres after 100 hours at 1200°C.

Hafnium carbide and hafnium nitride were selected from several potential diffusion barrier coating materials by a comparison of predicted thermodynamic behaviour and experimental compatibility tests with Mar M-200 alloy at temperatures as high as 1200°C. Other materials evaluated were: Al_2O_3, HfO_2, TaC, ZrC, TiC and TiN. Figures 5 (b) and (c) show the effectiveness of 4 μm thick HfC and HfN layers in preventing the recrystallization of tungsten after 100 hours at 1200°C.

The processes evaluated for the deposition of HfC and HfN coatings were chemical vapour deposition, sputtering, ion-plating and electron beam evaporation. The choice of CVD was based on a comparison of factors such as attainable deposit chemistry and microstructure, adhesion, thickness control, process throwing power, efficiency, adaptability to continuously coat long lengths of fibre, and scale-up costs.

Deposition of HfC and HfN has been performed previously (5, 6, 7, 8). The temperatures used for the deposition of these materials were, however, higher than thought suitable for the retention of optimum mechanical properties of the thoriated tungsten fibres. Conditions for the deposition of HfC and HfN at lower temperatures were sought where reasonable deposition rates could be retained to take advantage of CVD as a coating method. A description of the method for establishing suitable conditions for the deposition of HfC and HfN has been given elsewhere (9) and should be consulted for details.

Prior to coating, the thoriated tungsten fibres are subjected to surface polishing and cleaning. Two methods were evaluated for surface polishing. Electrolytic polishing results in smoother surfaces than a fused salt treatment and the former was adopted as standard procedure. Prior to deposition, the thoriated tungsten fibres are continuously straightened in high purity argon at 1200°C by application of an axial stress of about 550 MPa.

Figure 6 shows a diagram of the system used for the continuous deposition of HfC and HfN on thoriated tungsten fibres. It consists of two parts. The double walled chlorination chamber is used for the in situ chlorination by HCl gas of hafnium metal chips at 700°C. The reaction gases, H_2 and CH_4 for HfC deposition, or H_2 and N_2 for HfN deposition, mix in the outer tube before entering the deposition chamber. The deposition chamber is also of double wall construction and has water-cooled mercury seals at both ends to isolate the chamber from the outside atmosphere and also to serve as electrical contacts for resistively heating the moving fibre.

The walls of the deposition chamber are maintained at about 400°C by heating with thermal tape or by ceramic wool insulation to retain the hafnium chlorides in the gas phase. The wire is moved through the inner tube of the deposition chamber at a uniform rate using a variable speed motor in combination with 400 mm diameter take-up and feed spools. The spools are controlled by clutches to maintain constant tension on the wire.

The effect of changes in the deposition temperatures in the range 900°C - 1300°C, gas compositions and gas flow rates on the rates of deposition, deposit morphologies and stoichiometries of the HfC and HfN deposits are being investigated. Conventional microscopy, scanning electron microscopy and x-ray diffraction are used to characterize the deposits. Hafnium carbide deposits having widely differing morphologies and stoichiometries, deviating $\pm$ 2 a/o in carbon from the stoichiometric composition can be obtained by introducing changes in the deposition parameters. Control of stoichiometry and deposit morphologies for HfN deposits is more complex than for HfC (9). In general, however, HfN deposits close to stoichiometric can be deposited at H_2 to N_2 ratios of 1:1 at temperatures in the range 1200°C - 1300°C.

In summary, the CVD system developed has been shown to be capable of coating long lengths of thoriated tungsten fibres in a continuous manner with HfC and HfN diffusion barrier coatings. It is considered to be a laboratory scale process at this stage but it can be scaled up for production with only minor changes being necessary.

Composite Ply Process

Composite plies of varying shape and size are required to produce the prototype reinforced airfoil shape shown in Figure 7. The plies are fabricated from the coated reinforcing fibre and matrix alloy tape. The tape is produced from -100 mesh matrix alloy powder. The chemical composition and particle size distribution of the argon atomized Mar M-200 are presented in Tables I and II respectively.

Presently, quantities of the powder are sent to a commercial supplier who combines them with proprietary mixtures of organic binders, optimized for our application, to form a slurry. The slurry is dispensed through an orifice from a mixing container and onto a moving sheet of polyethylene. The required tape thickness is achieved by an adjustable plate which removes excess slurry as the slurry passes under the plate. The sized tape

is oven dried and wound onto dispensing rolls with protective waxed paper interleaving. For our purposes, a tape width of 200 mm and a thickness of 0.25 mm are provided.

The tape and coated wire are combined to form an airfoil shape by the following steps.

1. A silicone rubber mold is formed with a cavity shape corresponding to the desired airfoil, with an allowance for consolidation shrinkage.

2. A low melting point bismuth alloy is cast into the mold to form an airfoil replicate that can be deformed and easily machined.

3. The casting is flattened, without distortion, to produce a simplified shape.

4. The flattened casting is mounted on a suitable base and successive milling machine cuts are made. The thickness of material removed is dependent on the desired fibre volume fraction, the known matrix tape shrinkage factor, and the measured thickness of the pressed composite ply, as described later.

5. The machined surface is photographed after each cut to provide a trace for each ply pattern. Suitable indexing marks are provided so that each ply can be placed relative to another in the proper position as they are stacked. Figure 8 shows the variation of ply shapes between two selected sections of the flattened airfoil pattern.

6. Duplicate shapes of matrix alloy tape are cut for each ply.

7. The required fibres for each ply are positioned in a heated die which is grooved to provide the desired fibre spacing.

8. Matrix alloy tape is positioned on one side of the aligned fibres and pressed so that the fibres adhere to the tape. The tape and fibres are then reversed and another tape layer is placed over the fibres to form the composite ply.

9. Both surfaces of each ply are covered with a layer of polyethylene sheet and pressed to allow the matrix tape to flow between each fibre.

10. After removal of the polyethylene sheets, the plies are ready for stacking, and forming back to the original airfoil shape. The stacking is performed on a lacquer-coated, split ceramic mold having the desired airfoil shape as a cavity. It is at this point that cross-plies could be incorporated for off-axis strengthening. Sufficient layers of unreinforced tape are wrapped around The composite bundle to ensure at least 0.50 mm of uniform matrix alloy cladding so that no fibres are exposed after consolidation.

11. The shaped ply bundle is inserted into the container that is used for HIP-consolidation. The container is airfoil shaped and is sealed at only one end to make possible the insertion of the bundle and to maximize gas exit area for binder outgassing.

12. The container and its contents are placed in a retort and outgassed in hydrogen to remove all traces of the binders present in the matrix alloy tape. A typical outgassing cycle consists of initially evacuating the retort and backfilling with hydrogen before heating commences. A maximum heat-up rate of 100°C per hour is employed to 300 °C. At this temperature, the retort is evacuated and back filled with hydrogen again. The bundle is subsequently heated to 650°C, after which the furnace is cooled to room temperature to complete the cycle. Stress rupture tests were conducted to evaluate the effectiveness of the hydrogen outgassing treatment. Specimens were prepared by HIP-consolidation dry Mar M-200 powder and Mar M-200 powder produced from hydrogen-outgassed tape. Tests conducted at 980°C and at a stress level of 48.3 MPa yielded Larson-Miller parameters of approximately 50 for both types of specimens.

13. Following removal from the retort, the root section shell is welded to the container. This part of the container also includes the evacuation tube that is required for pre-HIP processing. Dry matrix powder is added to the container through the evacuation tube to form the root section.

14. At this point, the container is ready for evacuation and sealing.

The described process contains several steps and is considered to be a batch process, suitable only for prototype composite airfoil fabrication. Automation potential exists for several of these steps in a pilot plant or pre-production scale operation. For example, fibre collimation, tape placement, and composite ply pressing would be incorporated into a single continuous operation.

HIP Consolidation

Nickel base superalloy powders can be consolidated by either hot die pressing or HIP (Hot Isostatic Press). HIP was chosen for two primary reasons:

(a) die pressing is subject to frictional effects which can lead to incomplete consolidation.

(b) the HIP process has other potential applications in our industry, such as diffusion bonding and the healing of casting defects.

Powder consolidation, by HIP or gas pressure bonding, involves the simultaneous application of isostatic pressure and temperature to metal powders contained in an evacuated, pressure tight, deformable envelope. The pressure forces the powder particles into intimate contact by creep deformation so that diffusion bonding occurs at temperatures considerably lower than those normally required for conventional sintering. HIP-consolidated powders normally exhibit greater than 99% of the theoretical alloy density.

Several avenues are open in the choice of HIP container materials for net or near-net shape production. The two main requirements are that the material be plastic at the process temperature and capable of being sealed into a pressure-tight container. Other factors such as cost, formability, and ease of removal also enter into the ultimate materials choice. Our experience to date has been mainly with stainless and low carbon steel sheet in thicknesses ranging from 0.7 to 2.5 mm. These materials are relatively easy to form and weld. However, both stainless and carbon steel would have to be removed after HIP-consolidation.

We are presently exploring the possibility of using an oxidation resistant sheet alloy such as Nichrome, Nimonic C-263 or one of the MCrAlY overlay coating type alloys (where M can be Fe, Co or Ni). These containers would not be removed after processing but would function as an oxidation resistant coating for the airfoils. The possibility of using ceramic or glass containers is also being evaluated, since they offer cost effectiveness for complex shapes and are easy to remove. Oxidation and ceramic infiltration may result in surface contamination of the consolidated powder. This is potentially a limiting factor to the use of ceramic containers for the production of net shapes.

When the container is filled with the superalloy powder, it is checked for low pressure leaks and evacuated to between 10^{-5} and 10^{-6} mm of Hg. The evacuation tube is heated, pinched flat, and is then burned off by TIG welding to achieve the final vacuum seal. The part is now ready for consolidation.

The HIP system at Westinghouse Canada was obtained from Autoclave Engineers. It is shown schematically in Figure 9. The system consists of a five zone furnace, 300 mm I.D. by 900 mm long, located in a steel pressure vessel which can be pressurized to 140 MPa with argon. The furnace is capable of temperatures up to 1230°C. For maximum life of the Kanthal elements, the temperature is maintained at approximately 800°C between cycles. The vessel is pressurized by two diaphragm compressors operated in series. The argon is normally reclaimed at the conclusion of each cycle. The various steps in conducting a typical composite consolidation run are shown in Figure 10.

The parameters shown in Figure 10 were developed as a compromise between two conflicting goals. One should use a low HIP temperature to minimize matrix-fibre interaction whereas a high HIP temperature will yield improved matrix properties. It was found that complete densification could be obtained between 1000°C and 1100°C for the Mar M-200 alloy. However, this temperature range lies well below the γ' solvus temperature and results in a very fine-grained, weak, superplastic matrix (10), as shown in Figure 11 (a). The high temperature strength of the airfoils is largely supplied by the reinforcing fibres. In this part of the blade, a superplastic matrix might be acceptable. However, the blade root and the airfoil tip will not be fibre-reinforced; these areas must therefore possess adequate strength in the unreinforced condition. Such strength levels can only be obtained by processing at temperatures above the γ' solvus temperature, followed by aging to optimize γ' and grain boundary strengthening. In the HIP cycle illustrated in Figure 10, the first hour of the soak period takes place at 1100°C and most of the consolidation will occur at this temperature. During the second hour, the temperature is increased to 1230°C, which is above the γ' solvus temperature, and yields the equiaxed, fully solutioned microstructure desired for the unreinforced parts of the blade. Such a structure is illustrated in Figure 11 (b). If in the future it should prove necesary to subject the parts to an isothermal forging operation for final shaping, we should certainly take advantage of the superplastic properties by consolidation at 1100°C. This also minimizes the thermal exposure of the fibres. The microstructure desired for the unreinforced regions can be obtained by subsequent heat treatment.

Post-HIP Processing

Several processing steps remain to be performed after consolidation. These can be broadly divided into the areas of envelope removal, machining and forming, quality control and heat treatment.

It was previously suggested that one might be able to use an inherently oxidation resistant material for the HIP envelope. If this proved feasible, envelope removal would not be necessary. If the envelope material were stainless or carbon steel, they could be removed by either conventional or electrolytic pickling. Ceramic envelope materials tend to spall off during cooling from the HIP temperature. One of the advantages of ceramic molding materials is that only a grit blasting operation is required

to complete envelope removal.

If net shapes cannot be produced consistently by HIP-consolidation, isothermal forging will be required to refine the vane and blade airfoil shapes. In this case, the consolidation parameters would be chosen to take advantage of the superplastic properties of the matrix alloy, thereby minimizing press loads and die wear. The existence of a superplastic matrix is also expected to minimize the possibility of fibre damage during a forging operation.

Conventional machining techniques such as grinding and milling can be used on blade roots and vane shrouds since these will consist solely of matrix alloy. If any metal removal is required from the composite airfoils, more care than usual will probably be required. A characteristic of composite materials consisting of a matrix with a thermal expansion coefficient larger than that of the reinforcing fibres is the presence of residual stresses. At room temperature, the matrix is subject to a residual tensile stress whereas the fibres are in compression. Local heating from abusive grinding or machining could result in localized yielding or even cracking of the matrix alloy.

Quality control encompasses the areas of dimensional control, powder consolidation, and reinforcement fibre integrity and location.

Dimensional control of a composite article at the time of manufacture should not differ markedly from that for conventional cast or forged turbine parts. There is, however, one potential problem area that must be kept in mind when dealing with composites. The thermal expansion coefficient difference between the matrix and the reinforcement fibres results in the possibility of dimensional change or distortion as a result of thermal cycling during manufacture or during use. This distortion or "thermal ratchetting", as it has been called, arises when residual stresses exceed the yield stress of the reinforcing fibres. The amount of distortion is strongly dependent on the average volume fraction of the reinforcing component (11). Unpublished data suggests that, at the volume fractions required to meet the strength goals of the present program (50% or more), thermal distortion should not result. An exception to this conclusion might be in localized areas such as the trailing edges where fibre volume fractions as low as 25% may result because of the need to maintain matrix protection completely around the fibres. Testing of prototype airfoils will be required to determine if thermal distortion will indeed be a problem with the particular matrix and reinforcement under consideration.

The defects leading to poor quality in HIP-consolidated powder can generally be traced to different types of container leaks. One type is the loss of vacuum in the powder container just prior to or during the vacuum sealing operation. The result is still a sealed container but with air between the powder particles. The reactive alloying elements combine with the air to form nitrides and oxides on the surface of the powder particles during HIP processing and will still yield an apparently consolidate shape. However, if one examines the microstructure of the matrix, one observes prior particle boundaries (PPB). The effect is usually most apparent in thin sections, as these heat up faster than the more massive sections. The nearly continuous film which forms, inhibits subsequent recrystallization and can result in poor ductility. Metallography appears to be the only way by which the presence of air in the HIP container is detectable. It appears likely that a metallographic specimen will have to be prepared from each composite article. This specimen could be obtained from the evacuation tube. It is also possible for leaks to develop during the HIP process by the tearing of welds or splitting at thin spots in the envelope. These defects will result in incomplete consolidation.

Quality control for fibre location is necessary since the poor oxidation resistance of tungsten dictates that all reinforcing fibres must be surrounded by matrix alloy. When the minimum matrix thickness required to protect the fibres has been determined, nondestructive testing will be required to measure the matrix cladding thickness. It is expected that existing techniques such as x-ray radiography and ultrasonic testing can be adapted to this problem area. The existence of broken fibres should also be detectable by such techniques.

The final processing step(s) will consists of the application of an oxidation resistant coating (if oxidation resistant HIP envelopes are not used), followed by heat treatment. Heat treatment would largely be aimed at optimizing blade root properties since the operating temperature of the vane and blade airfoils will be too high to expect appreciable long term benefits from heat treatment.

CONCLUDING REMARKS

A powder metallurgy process has been developed on a laboratory scale to produce superalloy matrix-thoriated tungsten composite shapes. The continuous deposition of hafnium carbide and hafnium nitride diffusion barriers on tungsten wire by chemical vapour deposition has been demonstrated. Coated fibres have been combined with matrix alloy tape to produce composite plies. The plies can be stacked, outgassed and consolidated by hot isostatic pressing into near-net or possibly net composite shapes.

Several factors require serious consideration before the proposed process could be adapted to a pilot plant scale for producing a set of prototype blades for simulated turbine testing. Some of the most important of these factors are:-

(a) The need to demonstrate the effectiveness of the diffusion barriers in inhibiting nickel-induced recrystallization for at least 10,000 hours.

(b) Creep and stress rupture properties of composites produced by the proposed process must meet design requirements.

(c) Distortion resulting from thermal stresses on the composite airfoils must be controlled within acceptable design limits.

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ACKNOWLEDGEMENTS

This program is supported by the National Research Council of Canada through the Industrial Research Assistance Program and by Westinghouse Canada Limited.

We wish to acknowledge assistance provided to the program by the staff of the Product Assurance Laboratory of the Westinghouse Canada Turbine and Generator Division, and Dr. J.A. Cornie of the Westinghouse Electric Corporation Research Center.

TABLE I

CHEMICAL ANALYSIS OF Mar M-200 MATRIX ALLOY POWDER (w/o)

Ni	C	Co	Cr	Al	Ti	Nb	W	Zr	B	N_2	O_2	S
Bal.	0.022	11.08	9.80	5.23	2.10	1.02	12.82	.047	.018	.0014	.0060	.006

TABLE II

PARTICLE SIZE DISTRIBUTION OF Mar M-200 MATRIX ALLOY POWDER

Mesh	w/o
+ 80	0.3
+ 100	4.3
+ 120	5.5
+ 140	6.6
+ 170	9.3
+ 230	15.6
+ 270	7.8
+ 325	8.8
+ 500	23.2
- 500	18.6

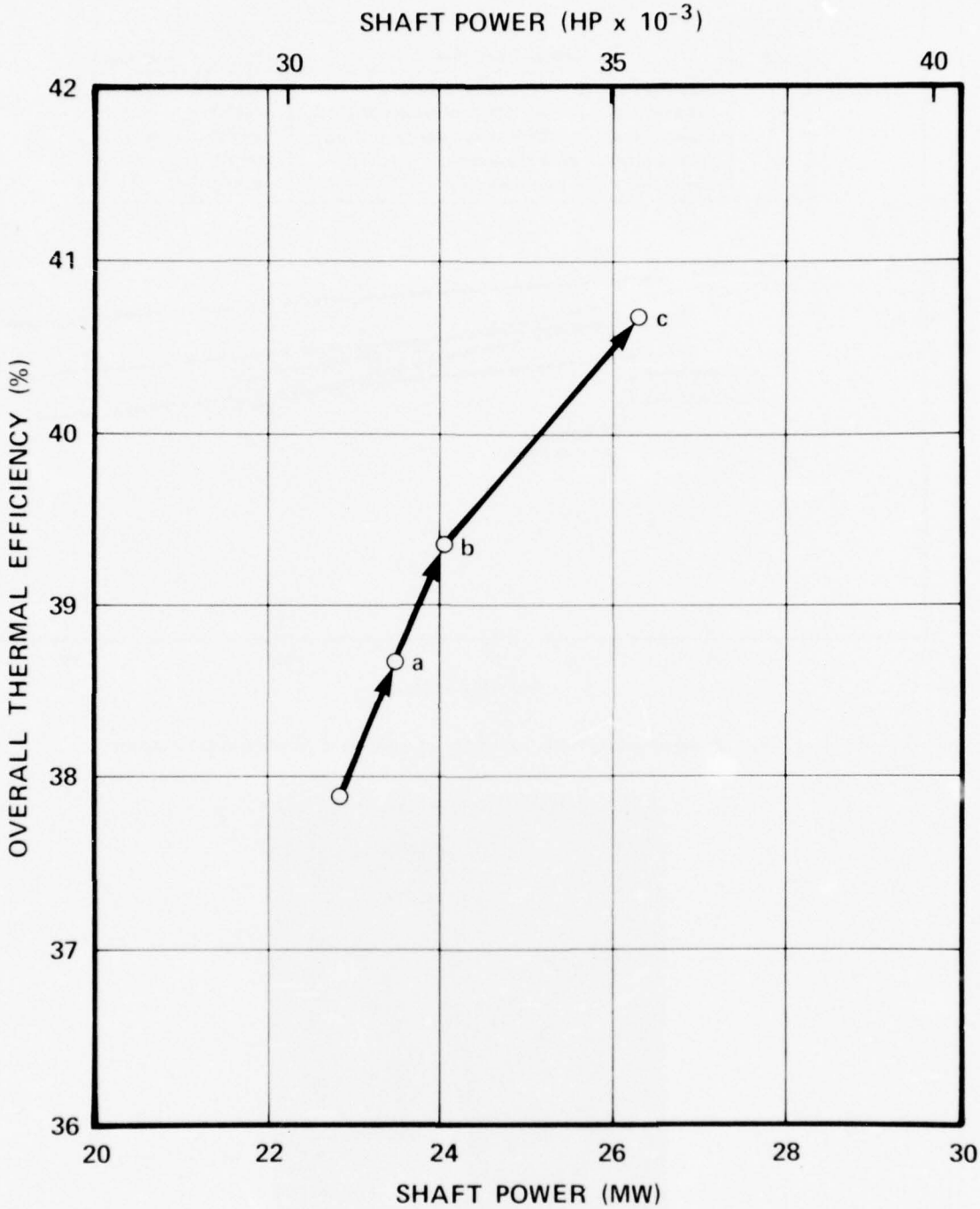
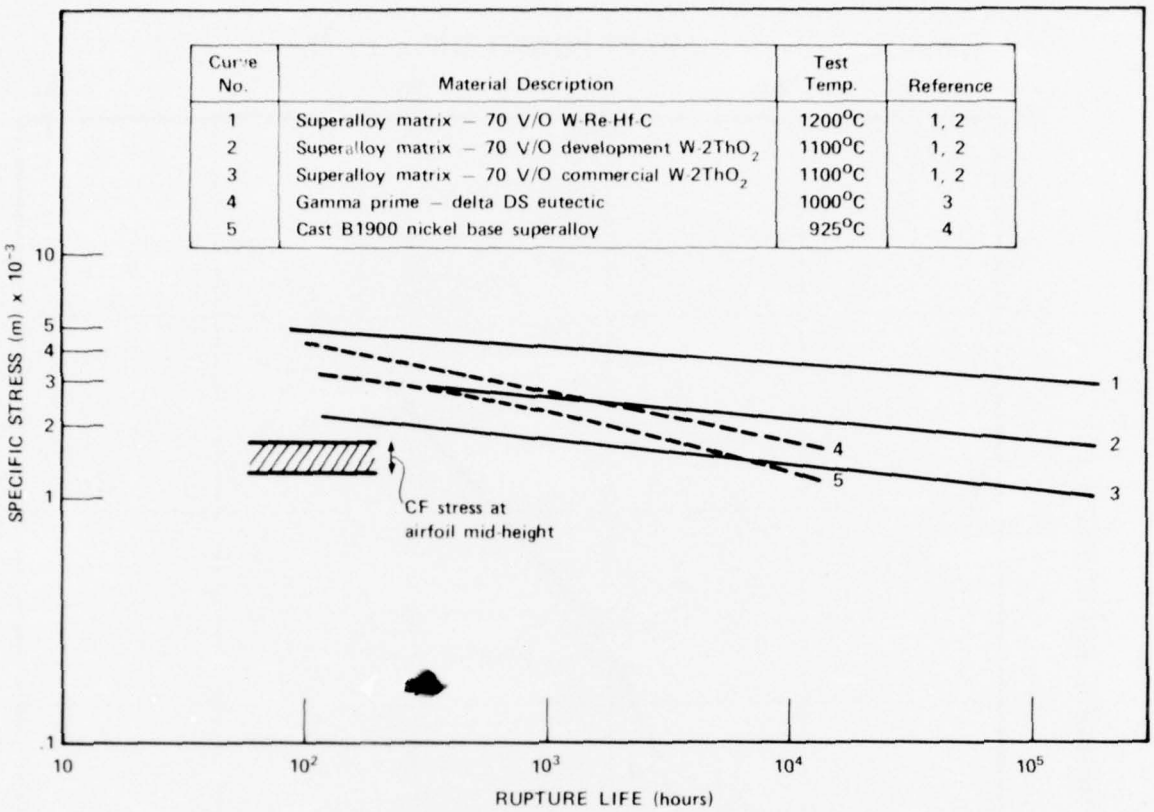


FIG. 1 Performance improvements in a regenerative industrial gas turbine engine by (a) cooling removal on first row vanes and blades; (b) halving first row vane and blade trailing edge thickness; (c) increasing compressor turbine inlet temperature from 1010°C to 1060°C.



Curve No.	Material Description	Test Temp.	Reference
1	Superalloy matrix – 70 V/O W-Re-Hf-C	1200°C	1, 2
2	Superalloy matrix – 70 V/O development W-2ThO_2	1100°C	1, 2
3	Superalloy matrix – 70 V/O commercial W-2ThO_2	1100°C	1, 2
4	Gamma prime – delta DS eutectic	1000°C	3
5	Cast B1900 nickel base superalloy	925°C	4

FIG. 2 Specific stress rupture properties of current and advanced airfoil materials.

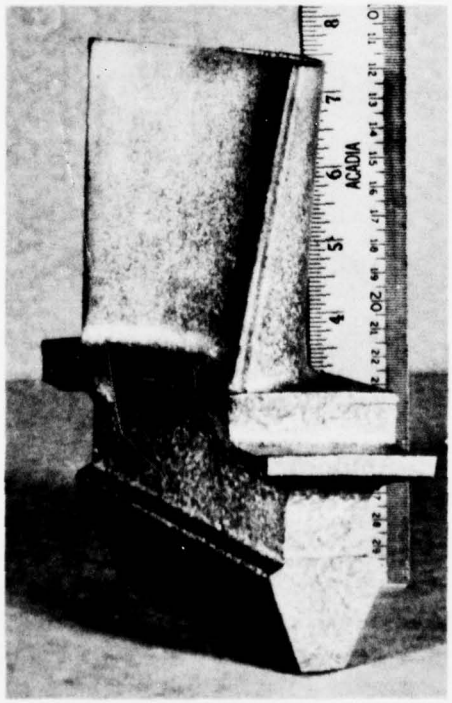


FIG. 3 Cast superalloy first row industrial gas turbine blade

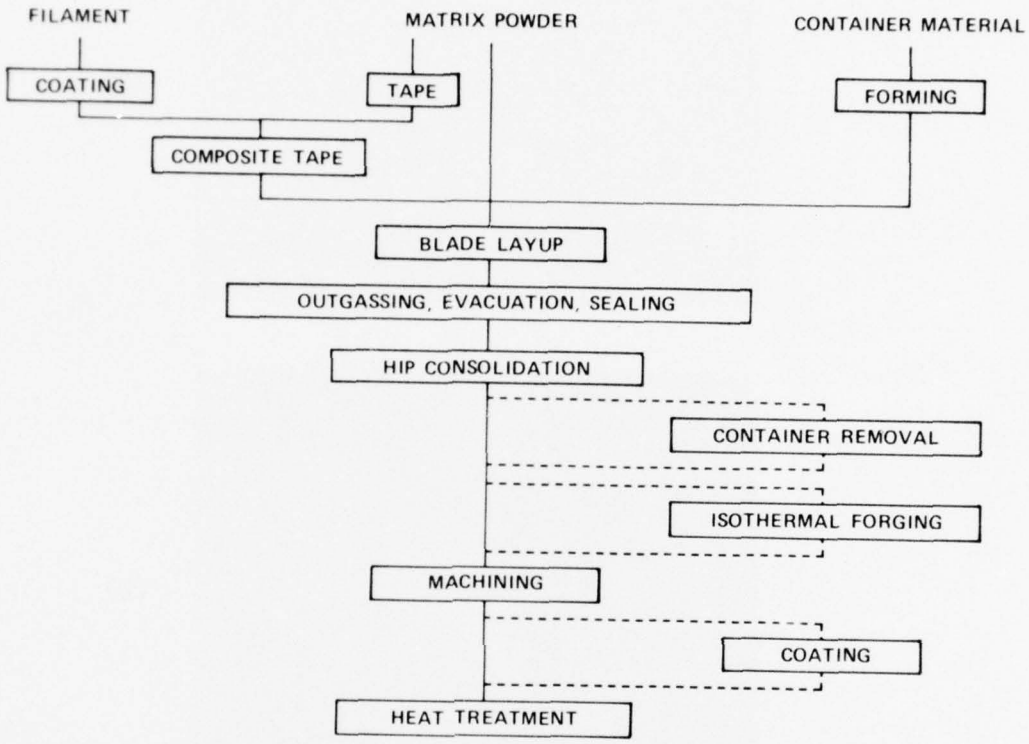


FIG. 4 Flowchart of the Westinghouse Canada composite airfoil manufacturing process

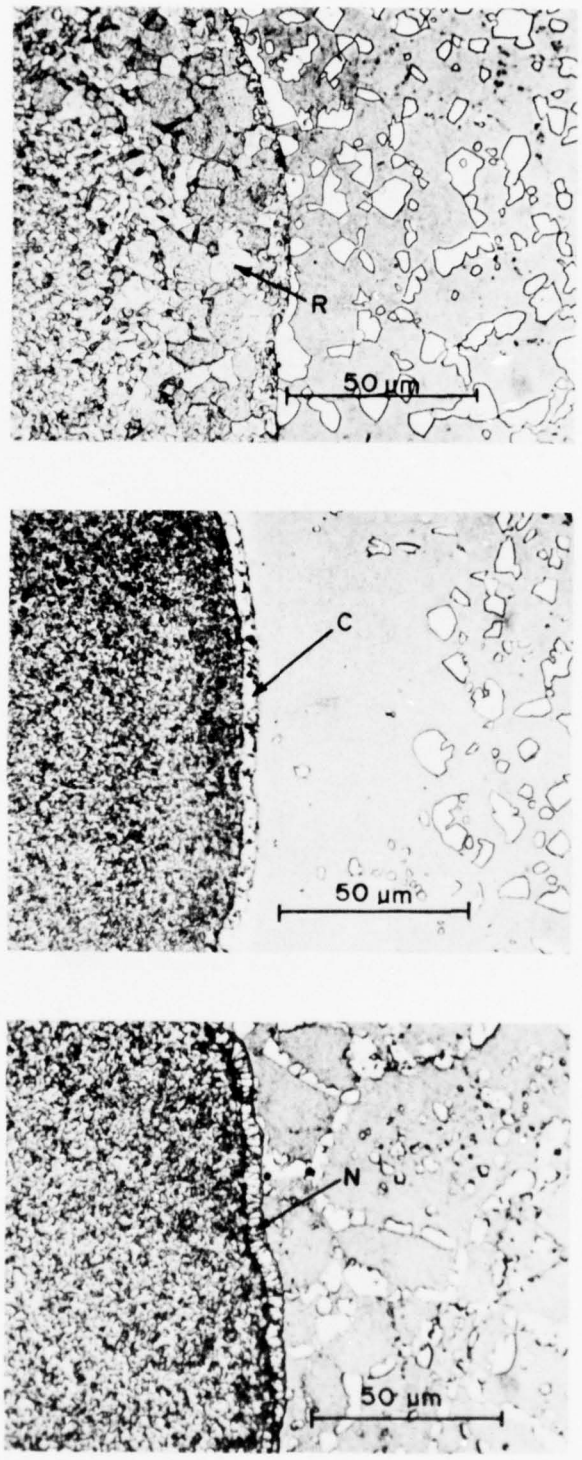


FIG. 5 The results of thermal exposure at 1200°C for 100 hours on uncoated and coated $W\text{-}2ThO_2$ fibres embedded in a Mar M-200 matrix.
(a) uncoated fibre showing recrystallization (R).
(b) a fibre coated with 4 microns of HfC (C) showing no recrystallization.
(c) a fibre coated with 4 microns of HfN (N) showing no recrystallization.

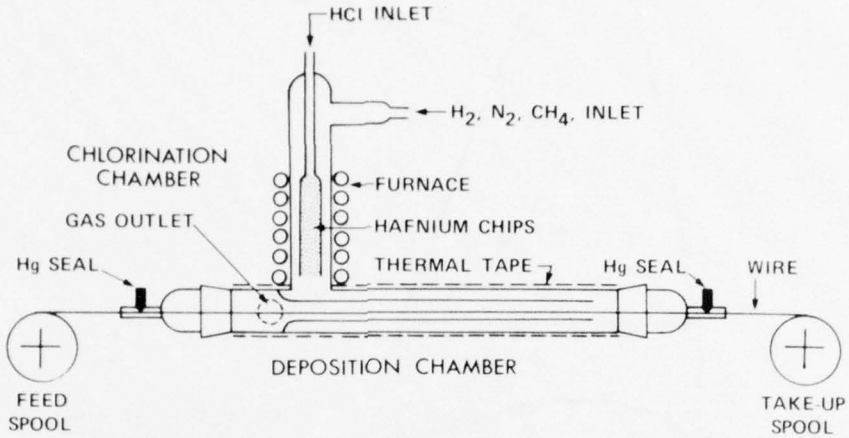


FIG. 6 Schematic diagram of the CVD apparatus for depositing HfC and HfN coatings on thoriated tungsten wire.

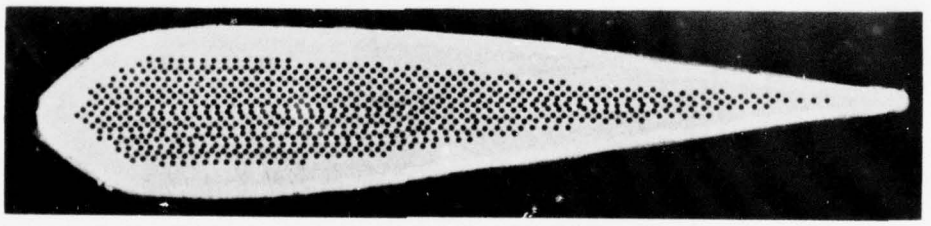


FIG. 7 A cross-section of a consolidated composite prototype airfoil. The HIP envelope has not been removed.

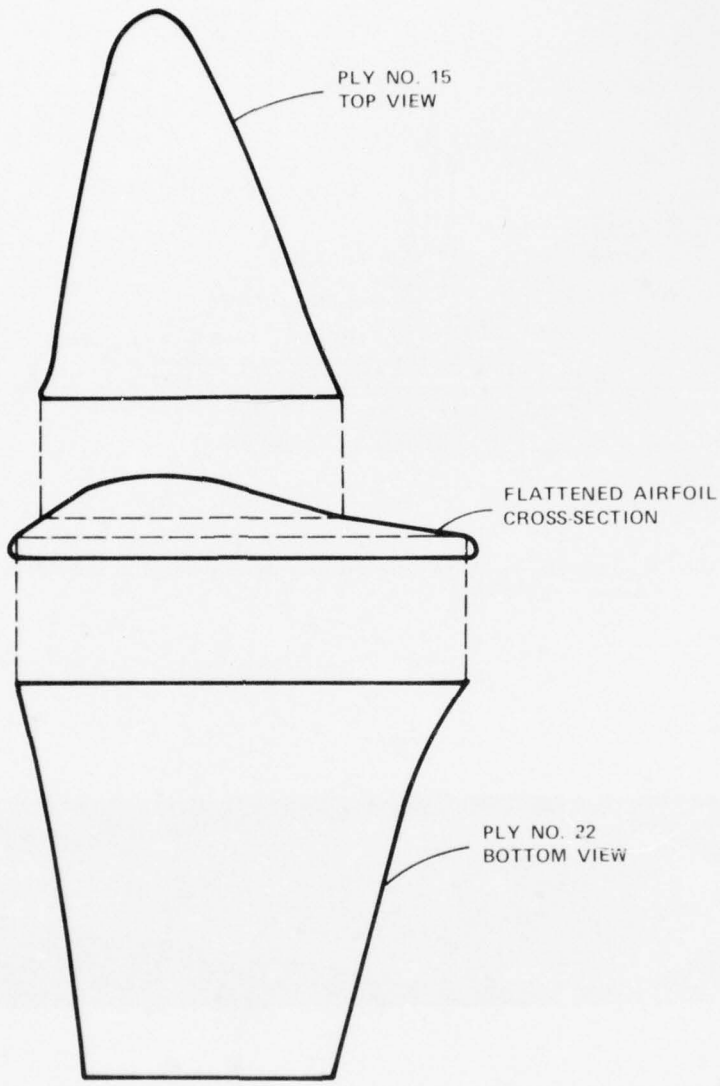


FIG. 8 Typical ply patterns in a flattened prototype airfoil shape.

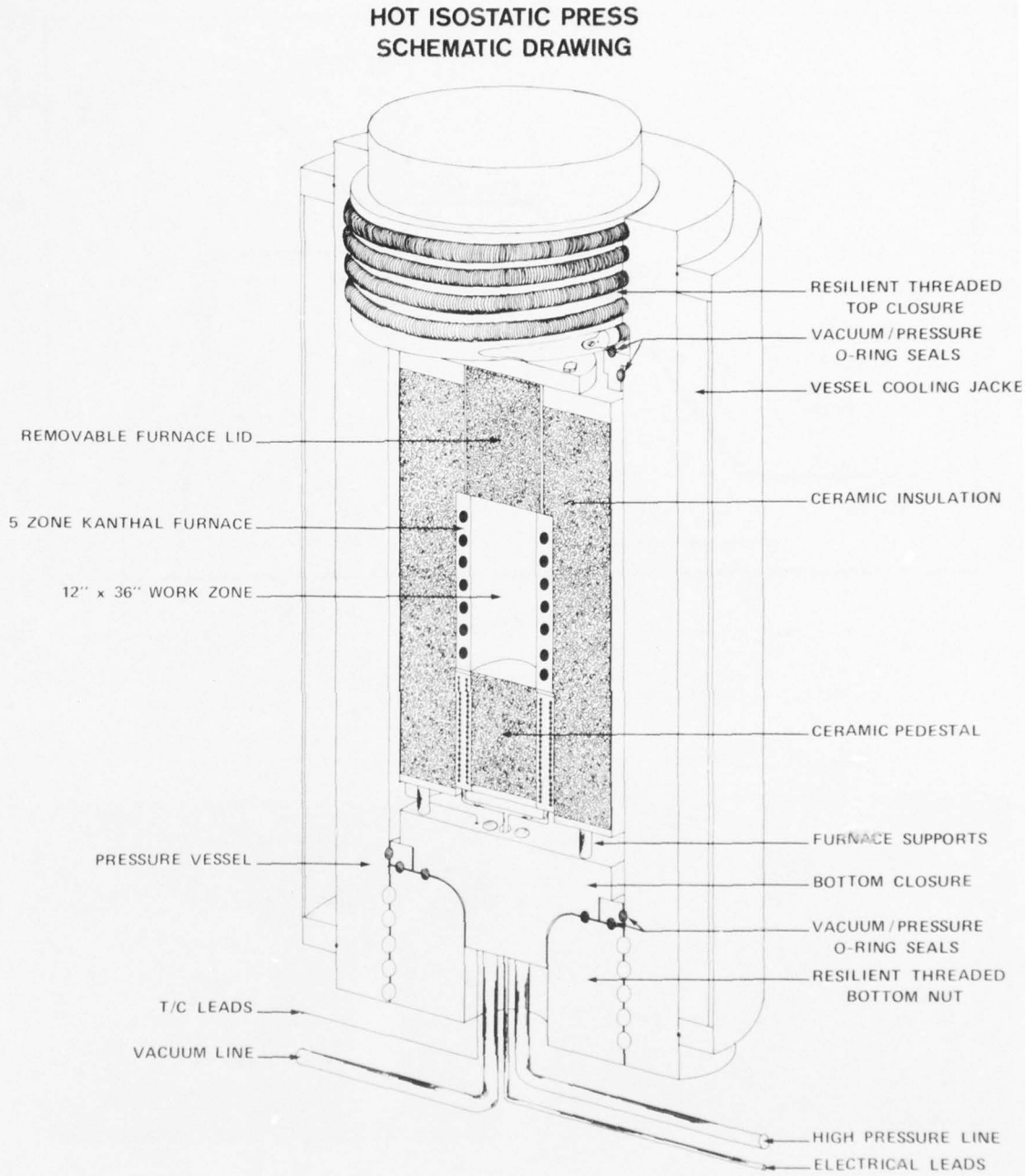


FIG. 9 Schematic of the Westinghouse Canada HIP vessel and furnace

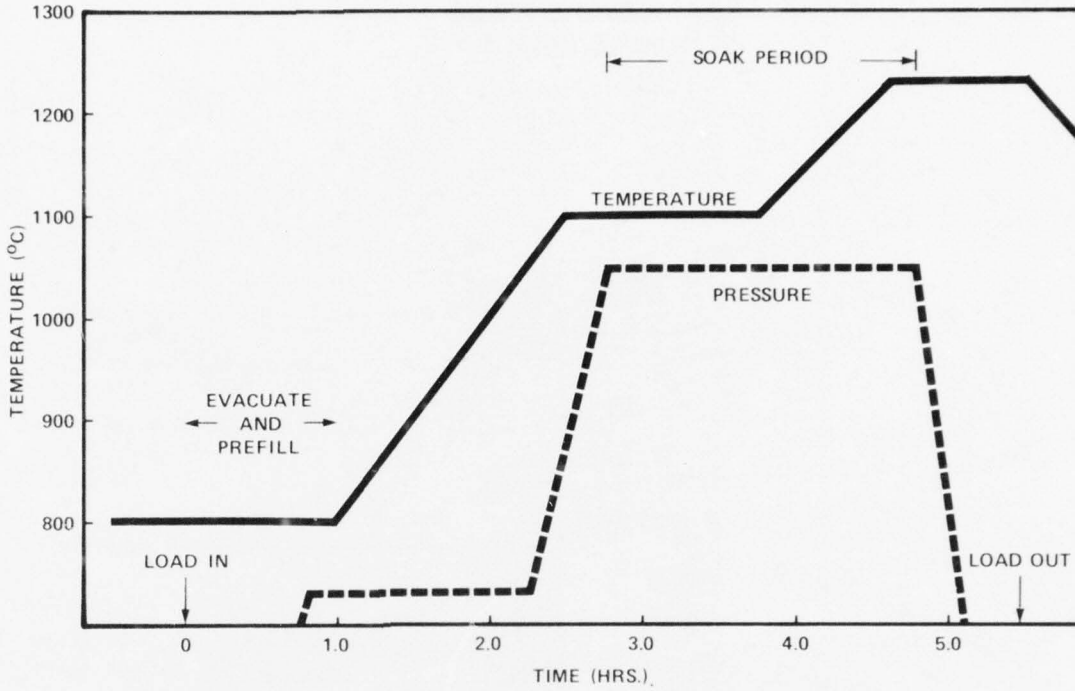
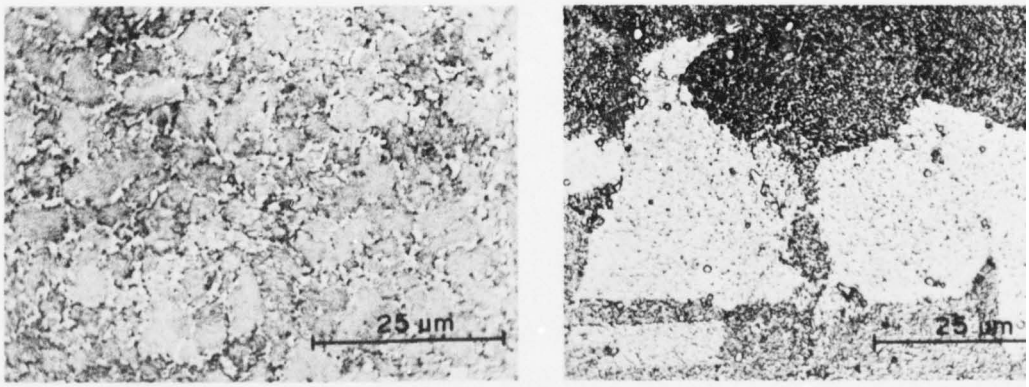


FIG. 10 Typical HIP cycle for P/M Mar M-200 matrix - $W/2ThO_2$ fibre composite material



(a) (b)

FIG. 11 The microstructure of HIP consolidated Mar M-200 powder. The consolidation parameters wer
(a) 1000°C / 2 hours / 70 MPa, 4 µm average grain size.
(b) 1200°C / 2 hours / 70 MPa, 23 µm average grain size.

COMMENT ON THE SHORT CONTRIBUTION NO. 7

by

Dr. G. Wirth, DFVLR, Germany

As has been shown by Mr. Mazzei in the previous contribution (SC 7), dispersion hardening of the tungsten fibres by thoria does not prevent the nickel induced recrystallization during exposure at 1200°C for 100 hours (see his Fig. 5a). Also, a precipitation hardening by hafnium carbide was not successful in this sense as can be seen from the work of Signorelli at NASA-Lewis. He claims also that his composites could be substantially improved by coating the tungsten fibres with a diffusion barrier. Unfortunately, diffusion barriers consist of brittle ceramic materials like oxides, carbides or nitrides which tend to crack due to thermal stresses under service conditions. This fact has been found by Kotov. Besides this, the additional coating treatment of the fibres complicates the production process of the composite, and in turn raises costs. In searching for a more economic way of protecting the fibres reliably I used another concept which allowed the nickel do diffuse into the tungsten fibres at service temperature without causing recrystallization to a detrimental large, equiaxed grain structure. The concept consists of a double fibre reinforcement (see Figure I). The tungsten fibres get an oxide short-fibre reinforcement, simultaneously produced in situ by HERF-extrusion, swaging and drawing of sintered tungsten-zirconia powder blends. These tungsten fibres may afterwards be used as reinforcement for a nickel-base matrix without a coating, the whole double fibre-reinforced composite being produced by the same methods as mentioned,i. e. in the previous contribution.

The microstructural stability at 1200°C can be seen in Figure 2 where the fibre-like microstructure of tungsten-10% ZrO_2 wire (0.8 mm dia.) is maintained after annealing different times up to 100 hours at 1200°C in the presence of nickel and without any coating. Co does not markedly diffuse but forms brittle intermetallic compounds on the interface.

Improvements of stress-rupture strength by the oxide short fibre reinforcement with and without nickel is shown in Figure 3. No detrimental effect of Ni on the stress-rupture strength can be detected in this case because the tungsten fibres maintain their fibrelike microstructure. Besides this stabilizing effect, the oxide short fibres strengthen the tungsten wire at these temperatures. If a recrystallization of the W-ZrO_2 wire occurs at higher temperatures or prolonged times of exposure, there will be a grain growth in the longitudinal direction because of the aligned oxide short fibres. This results in a large aspect ratio of the recrystallized tungsten grains which according to Wilcox and Clauer exhibits a greater high-temperature strength than a coarse equiaxed grain structure.

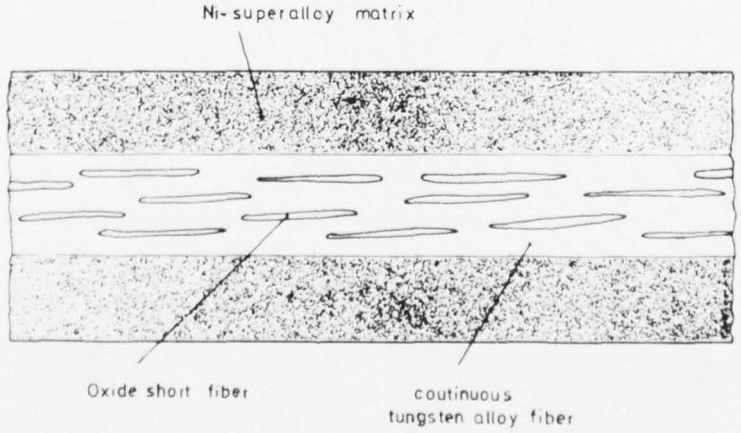


Fig. 1: Principle of double fibre reinforcement in high-temperature tungsten wire nickel-base alloy composites.

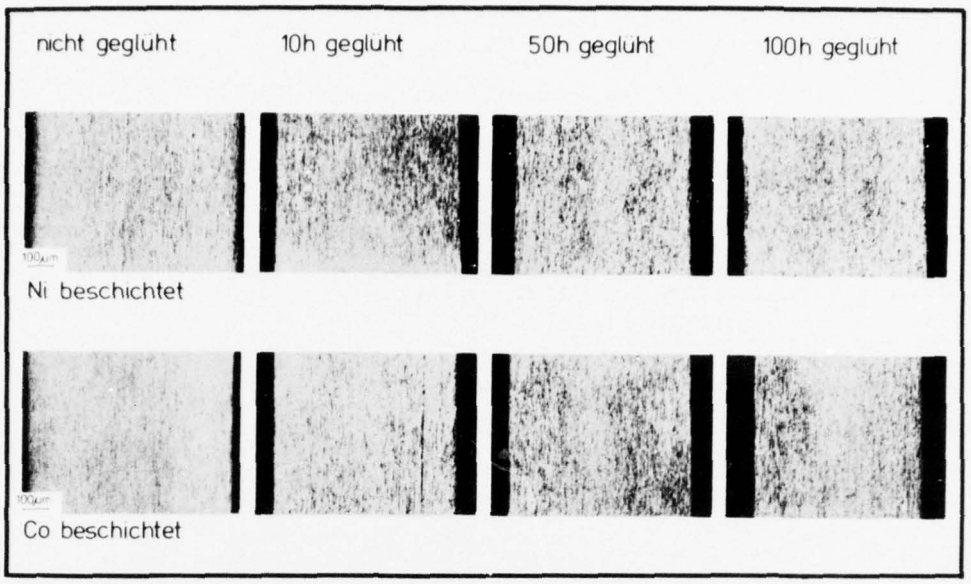


Fig. 2: Effect of Ni and Co on the recrystallization behaviour of W-10% ZrO_2 wire at 1200°C.

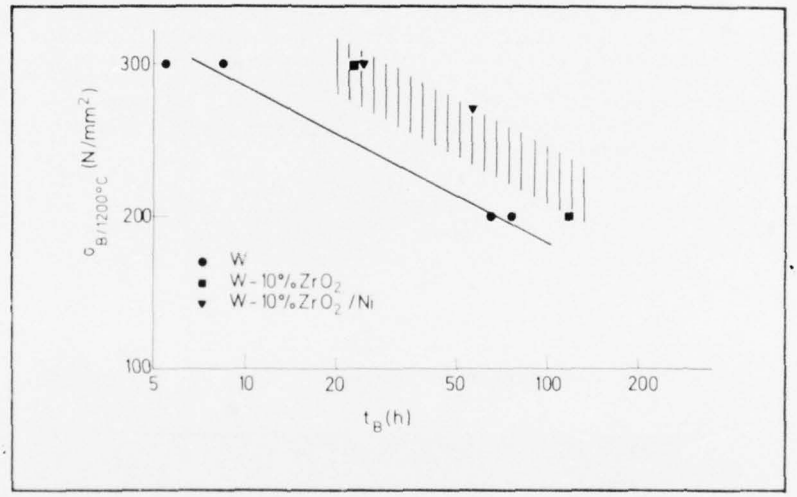


Fig. 3: Stress-rupture strength of W-10% ZrO_2 with and without Ni compared to pure tungsten at 1200°C.

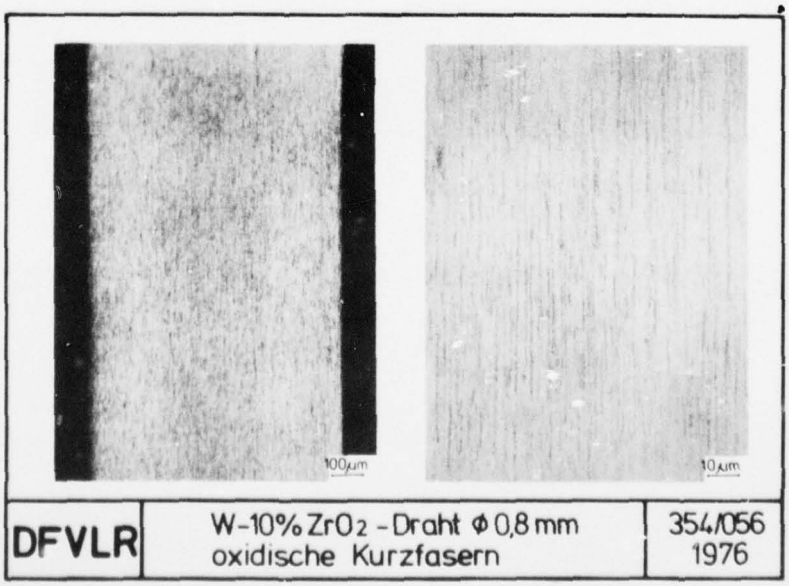


Fig. 4: W-10%ZrO_2 wire with a diameter of .8mm. Short fibres out of zirconia.

HIGH-STRENGTH POWDER-METALLURGY COBALT-BASE ALLOYS FOR USE UP TO 650°C(*)

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ABSTRACT

The aim of the present study was to ascertain the possibility of obtaining high strength levels at intermediate temperatures in experimental cobalt-base alloys prepared by powder metallurgy (P/M) techniques.

The first part of the work concerned P/M grades containing (in wt. %) 10 to 15%Ni, 20%Cr, 10%Mo and up to 1.8%C, strengthened mainly by solid-solution effects and precipitation of carbides. The second part dealt with P/M grades containing (in wt. %) 16%Cr, 3 to 5%Mo, 5%Ti and less than 0.1%C strengthened by solid-solution effects and precipitation of the ordered f.c.c. γ'-Co_3Ti intermetallic compound.

Prealloyed powders sizing less than 500 µm were prepared by N_2 atomization and, for some of the Ti-containing grades, by the rotating electrode process. After consolidation by hot extrusion of canned powders, the alloys were hot worked by rolling or swaging and subjected to a final aging treatment.

At the present stage of the work, ultimate tensile strengths up to 1850 MN/m^2 at room temperature and 1350 MN/m^2 at 650°C (1200°F) were obtained in the γ'-Co_3Ti strengthened alloys. Relationships between microstructures and mechanical properties are discussed in terms of the powder characteristics, and the extrusion and subsequent hot-working and aging conditions.

INTRODUCTION

Current developments in the field of gas turbines for aircraft and industrial applications calls for materials with improved strength properties at temperatures up to about 650°C. In particular, such materials are required for turbine discs operating at ever higher temperatures and, generally speaking, must exhibit the following properties :

- high and stable tensile strength ;
- high resistance to low cycle fatigue ;
- high resistance to crack propagation;
- good creep-rupture strength.

Numerous studies described in recent literature have shown that the application of powder metallurgy (P/M) techniques, viz. hot extrusion and hot isostatic pressing (HIP) of prealloyed powders followed by conventional or isothermal forging, markedly enhances the tensile and fatigue strength of nickel-base superalloys such as René 95(**), IN-100(**) and Astroloy(**) at intermediate temperatures (1-4). In particular, the René 95 P/M alloy seems to be one of the strongest materials of this type; after HIP and forging, its yield strength at R.T. is about 1250 MN/m^2 and remains stable up to 650°C (2). However, as shown on a modified version of Astroloy (4) the final properties are highly dependent on the forging conditions. Forging well above the γ' solvus of the alloy gives rise to a relatively coarse equiaxed grain structure with good stress-rupture lives; forging at progressively lower temperatures first produces a mixed coarse and fine-grained "necklace" structure, then a very fine-grained structure with increasing tensile properties.

An important feature of the work conducted up to now on nickel-base superalloys is that the general trend has been to apply P/M techniques to conventional alloys normally used in the cast or wrought conditions. At the very most, the alloy chemistries have been slightly modified in order to overcome problems due to the precipitation of MC-type carbides on the prior particle boundaries (PPB) when heated for compaction (5).

On the contrary, the aim of the present study was to evaluate the possibility of combining, in experimental cobalt-base P/M alloys, several strengthening mechanisms (solid solution strengthening, carbides and/or intermetallic compounds precipitation) with a view to achieving high strength levels at intermediate temperatures.

(*) Work performed under the joint sponsorship of the Cobalt Information Centre (C.I.C.) and the Institut pour l'Encouragement de la Recherche dans l'Industrie et l'Agriculture (I.R.S.I.A.), within the framework of a European Concerted Action on the Gas Turbine Materials (COST 50).

(**) René 95 and Astroloy are trade-marks of General Electric, USA
IN-100 is a trade-mark of International Nickel, USA

BACKGROUND

As compared to nickel-base superalloys, the information available on P/M cobalt-base alloys is scanty. Previous work conducted on conventionally cast and carbide-strengthened alloys has shown that the carbide network formed during solidification is completely destroyed by hot deformation or processing by P/M techniques and is replaced by a homogeneous dispersion of carbides

Fig. 1. illustrates the microstructural evolution of cast cobalt-base alloys, Mar-M-509 and Stellite 6, during hot working and processing by P/M techniques, respectively (6, 7).
The structural changes observed have a beneficial effect on the tensile strength at intermediate temperatures, as shown in Fig. 2 for the X-40 alloy (6, 8). It can be seen that, up to about 700°C, the strength and ductility of the forged or P/M alloy are superior to those of its cast counterpart. Simultaneously, however, the high-temperature stress-rupture strength is decreased. As an example, the 1093°C/ $63MN/m^2$ rupture life of hot-worked Mar-M-509 is only of the order of several minutes, as compared with more than 20 hours for the cast alloy (6). It is thought that this decrease stems from the destruction of the carbide network, which is normally the cause of significant local carbide reinforcement.

However, it is possible to improve considerably the stress-rupture properties by heat-treating the hot-worked or P/M materials at a temperature above the solidus temperature (6, 9). In particular, it has been shown for the X-40 alloy that by applying such a treatment under pressure in an autoclave, it is possible to obtain, on extruded P/M products, high-temperature stress-rupture lives considerably higher than those of the cast product, though at some expense to ductility (9). It has been reported that this improved stress-rupture life is related to the presence of a fairly large amount of "cast structure" at the grain boundaries, as well as to an increased grain size.

The aim of the present work was to obtain experimental cobalt-base P/M alloys, characterized by a fine-grained and stable microstructure, a uniform distribution of fine carbides and/or intermetallic compounds, and a stable dislocation substructure. In this respect, previous work showed that hardening of cobalt-base alloys by means of uniform precipitation of a f. c. c. ordered γ' type phase was of particular interest in obtaining high strength levels at intermediate temperatures (10). This work also showed that among the various possible γ'-Co_3X compounds, where X represents elements such as Ti, Ta, Nb, Mo or W, the γ'-Co_3Ti phase was the most stable at elevated temperatures.

EXPERIMENTAL PROCEDURE.

Materials.

Prealloyed powders of carbide-strengthened alloys containing 10 to 15% Ni, 20%Cr, 10%Mo and up to 1.8%C were obtained by nitrogen atomization. Powders of alloys containing 15%Cr, 3 to 5%Mo 0.005 to 0.1%C and 5%Ti were also prepared by either nitrogen atomization or the rotating electrode process (REP). The purpose of these powders was to study strengthening by precipitation of a γ'-Co_3Ti type phase, and their Ni and Mo contents were reduced in order to overcome γ' destabilization (10).

Table I gives the actual composition of the powders, including the O_2 and, in some cases, the N_2 contents. After nitrogen atomization, the O_2 content is relatively high, in particular for the Ti-containing powders V 6; in the carbide-strengthened V 0 to V 5 powders, this content decreases sharply as the carbon content is raised up to about 0.6%. On the other hand, the REP Ti-containing powders are characterized by a low O_2 content of less than 100 ppm.

The size of the powders lies below 400-500 μm : the size distribution is illustrated in Fig. 3 for the V 13 grade. Figs. 4 and 5 show that the powders are spherical and exhibit a dendritic structure. In addition, the REP powders contain pores located within the interdendritic areas (Fig. 5).

Compaction and hot-working.

The prealloyed powders were compacted by hot extrusion in mild steel cans 45 mm in diameter and 100 mm long at temperatures ranging from 1050 to 1200°C, with extrusion ratios comprised between about 4.5 and 6.2. The extruded compacts were then hot worked by swaging or rolling at 1100°C. For the carbide-strengthened V 0 to V 5 alloys, the amount of reduction per pass was limited to 20%, the total deformation ratio being of about 50%. For the Ti-containing grades, the amount of reduction per pass was dependent on the O_2 and C contents in the powders ; it was of the order of 5 and 30% for the V 6 and V 14 grades, respectively.

EXPERIMENTAL RESULTS.

Carbide-strengthened materials.

Figs. 6 and 7 illustrate the microstructure of the 0.03%C and 0.65%C alloys V 0 and V 2, respectively, after extrusion and further hot working and aging. The low-carbon V 0 grade exhibits a coarse-grained microstructure with $M_{23}C_6$ carbides precipitating on grain boundaries during heat-treatment. Both $M_{23}C_6$ and M_7C_3 carbides were identified in the microstructure of V 1 to V 5 grades, the amount of M_7C_3 carbides increasing with the carbon content.

Increasing the carbon content to above 0.3% gives rise to an important grain refinement in the as-extruded condition (Fig. 6); subsequent hot working improves the uniformity of the primary carbide distribution (Fig. 7).

Fig. 8 shows the relationship between the room-temperature and 650°C tensile properties and the carbon content, after hot working and a final 800°C/48h/AC aging treatment to enhance precipitation of carbides. The tensile properties of the low carbon V 0 alloy are to be attributed mainly to solid-solution and work hardenings. With increasing carbon contents, the R. T. ductility is seen to decrease continuously, whereas the R. T. ultimate tensile strength first increases up to 1450 MN/m^2 and then decreases slightly, for carbon contents above 0.65%, due to the formation of increasing amounts of coarse carbides at grain boundaries.

On the other hand, the 650°C tensile strength decreases continuously from about 1000 to 800MN/m^2 with increasing carbon contents. This variation is probably related to the removal of solid solution strengtheners through formation of $M_{23}C_6$ and M_7C_3 carbides.

Fig. 9 illustrates the effect of carbon content on the 650°C stress-rupture life after rolling at 1100°C and applying final 800°C/48h/AC aging treatment, with or without an intermediate 1200°C/1h solution treatment. The applied stress, 363 MN/m^2, is that leading to a rupture life of 100 hours in the as-cast X-40 alloy. As compared to the X-40 alloy, the low-carbon V 0 P/M alloy exhibits an improved stress-rupture life after both types of heat-treatment.

After rolling and direct aging, the stress-rupture life decreases down to the level of the as-cast X-40 alloy as the carbon content is increased up to about 1%. On the other hand, after solutioning and aging, the stress-rupture life increases with the carbon content to a maximum of 15 times the life of as-cast X-40 for the 1.8%C grade V 5. This relationship probably results from the fact that the carbides are solutioned during the 1200°C heat treatment so that the amount of grain-boundary carbides present after aging increases. As shown in Fig. 10 for the 1.8%C alloy V 5, the intermediate solution treatment gives rise after aging, to a "duplex" microstructure in which coarse carbides are present in both the grain boundaries and the matrix.

γ'-strengthened materials.

Fig. 11 illustrates the longitudinal microstructure of the V 6 and V 13 P/M alloys in the as-extruded condition. Both materials are characterized by internally recrystallized and elongated prior particles with numerous fine PPB precipitates. The elongated particles become finer after further swaging at 1100°C, as shown in Fig. 11 for the V 13 alloy.

Electron and ion microprobe analyses performed on longitudinal sections of the as-extruded V 6 alloy showed that the prior particle boundaries are enriched mainly in titanium, oxygen and carbon (Fig. 12). The corresponding PPB precipitates are probably Ti-rich oxides, in relation with the high oxygen content of the N_2 atomized powders (Table I), and MC carbides formed when heating the canned powders prior to compaction.

Electron microprobe examinations were also made on the low-oxygen REP powders (Table I), of the 5%Ti-0.1%C grade V 13. As shown in Fig. 13, the dendritic microstructure of the as-received powders is related to Ti segregation in the interdendritic regions. The dendritic structure tends to disappear after holding the powders under argon for 1 hour at 1100 to 1200°C (Fig. 13). There is a corresponding decrease of internal Ti segregation but a Ti-rich envelope forms at the same time on the particles. The latter observation is in agreement with the formation of Ti-rich MC carbides on the PPB's when the powder is heated for compaction (5) and with the observed microstructure of the extruded V 13 P/M alloy (Fig. 11).

The occurrence of a Ti-rich envelope around the V 13 powders after heat treating at 1100 to 1200°C seems to be related to the carbon content, as confirmed by similar experiments made on powders of two IN-100 grades containing 0.18 and 0.007%C and less than 100 ppm O_2. Fig. 14 shows that, after a 1150°C/1h treatment under argon, the Ti-rich envelope is observed only around the high-carbon IN-100 powders : it has been identified by X-ray diffraction analysis as a carbo-nitride phase.

On the other hand, transmission electron microscope observations on the Ti-containing compacts reveal a uniform precipitation of small and coherent particles of a f. c. c. ordered γ'-Co_3Ti type phase within the grains. This is illustrated in Fig. 15 for the V 6 alloy after solutioning at 1200°C for 1 hour and aging at 800°C for 8 hours. Fig. 15 also shows that the γ' phase does not remain stable during exposure at 800°C, but gives rise to the discontinuous precipitation of a hexagonal ordered η-Co_3Ti compound starting from the grain boundaries. This reaction has previously been studied in cast or wrought experimental cobalt-base alloys strengthened by the γ'-phase (10).

In order to overcome the problems associated with the PPB precipitation of oxides and MC particles, and with the $\gamma' \rightarrow \eta$ phase transformation during heat treatment, further tests were made on REP powders of the grade V 14, the C and Mo contents of which were reduced respectively to 0.005 and 3% (Table I). As shown in Fig. 16 for the as-extruded material, the prior particles are finely recrystallized and their boundaries seem to be relatively clean as compared to the as-extruded V 6 and V 13 alloys (Fig. 11). However, it has been shown for the V 14 alloy that the O_2 and N_2 contents increase to 280 and 150 ppm respectively after extrusion, as compared with the 100 ppm O_2 and 30 ppm N_2 determined on the as-received powders. This enrichment is probably due to the fact that heating before extrusion was carried out in air without prior degasing of the canned powders.

Compression tests performed at increasing temperatures on the as-extruded V 14 alloy have shown that the prior particle boundaries tend to disappear and that recrystallization of new grains starts to occur during hot working at temperatures of at least 1100°C. This observations is in agreement with the results of hardness measurements made after a 4-hour exposure at increasing temperatures which have shown that the intermetallic compound (γ' η or Laves phase) solvus temperature for the wrought Co-15%Cr-3%Mo-5%Ti-0.1%C alloy is about 1000-1050°C. As a matter of fact, Fig.16 shows that the microstructure of the extruded V 14 P/M alloy after rolling at 1100°C is recrystallized, with the grain size increasing, under the experimental conditions considered, with the total amount of deformation.

The variations in hardness as a function of aging time and temperature are given in Fig.17 for the fine-grained V 14 P/M alloy, i.e. rolled in one blow at 1100°C with 30% reduction in cross section. As was the case for previously studied γ'-strengthened cobalt-base alloys (10), the hardness first increases for short aging periods due to the uniform and coherent precipitation of γ' particles. Overaging occurs in relation with the γ' destabilization giving rise to the discontinuous precipitation of the η phase starting from the grain boundaries.

Fig.18 summarizes most of the tensile properties determined so far on the γ'-strengthened P/M grades. As compared to the V 13 alloy, the relatively low strength and ductility of the V 6 alloy at R.T. and 650°C probably stem from the formation of significant amounts of PPB oxides. The V 13 alloy exhibits a good compromise between tensile strength and ductility, better than those achieved with the carbide-strengthened grades (Fig.8), although the ultimate tensile strength decreases from about 1700 to 1200 MN/m^2 as the test temperature is raised from R.T. to 650°C.

As regards the V 14 alloy in the as-rolled condition, the highest tensile strength levels are found in the fine-grained materials, with ultimate tensile strengths of about 1850 and 1350 MN/m^2 at R.T. and 650°C, respectively. On the other hand, the coarse-grained materials exhibit better tensile ductilities.

The influence of a 100-hour exposure at 700°C on the tensile properties of the rolled material seems to depend of the grain size and the extent of recrystallization. Aging the fine-grained material reduces the tensile strength at R.T. and 650°C to about 1400 and 1300 MN/m^2 respectively, as well as the R.T. ductility. These variations can tentatively be attributed to grain-boundary embrittlement through transformation of γ' to η, probably enhanced by the work hardening of the microstructure. On the contrary, aging the coarse-grained material improves both tensile strength and ductility, at least at 650°C. Microstructural examinations are in progress in order to clarify the effect of the aging treatment on the tensile properties of the rolled V 14 P/M alloy.

CONCLUSIONS.

At the present stage of the work, the following conclusions can be drawn :

- carbide strengthening of Co-10 to 15%Ni-20%Cr-10%Mo P/M alloys gives rise to a moderate tensile strength. The most stable properties are obtained with the low-carbon 0.03%C grade, which exhibits an ultimate tensile strength of about 1300 at R.T. and 1000 MN/m^2 at 650°C.
- combined γ' and carbide strengthening in Ti-containing P/M alloys gives rise to problems related to the precipitation of Ti-rich carbides at prior particle boundaries (PPB) even with carbon additions as low as 0.05 to 0.1%.
- strengthening of Co-Cr-Mo-Ti P/M alloys by means of a uniform precipitation of a γ'-Co_3Ti type phase calls for the use of prealloyed powders with very low oxygen and carbon contents, in order to overcome the PPB problem. Furthermore, the contents of solid-solution hardeners must be limited to less than 3-5% in order to restrict the extent of the γ'- η transformation and the resultant grain-boundary embrittlement.
- the tensile properties of the γ'-strengthened P/M alloys can be controlled by acting on the hot-working conditions, which govern the obtention of a fine-or coarse-grained recrystallized microstructure, and on the final aging conditions.
  Ultimate tensile strengths of about 1850 at R.T. and 1350 MN/m^2 at 650°C have been achieved in the fine-grained Co-16%Cr-3%Mo-5%Ti P/M alloy with the corresponding ductilities limited to 4%. The ductility is markedly higher for the coarse-grained material; however, the corresponding strength is limited to 1400 at R.T. and 1000 MN/m^2 at 650°C but can be improved through a final aging treatment.
- Further improvements in the tensile properties should be possible by increasing γ'-strengthening. In this respect, work is in progress on a Co-16%Cr-3%Mo-7%Ti-0.005%C P/M alloy prepared by hot extrusion and controlled hot working.

AKNOWLEDGEMENTS.

The authors wish to thank Mr. P. Guillaume for his contribution to the study of certain alloys.

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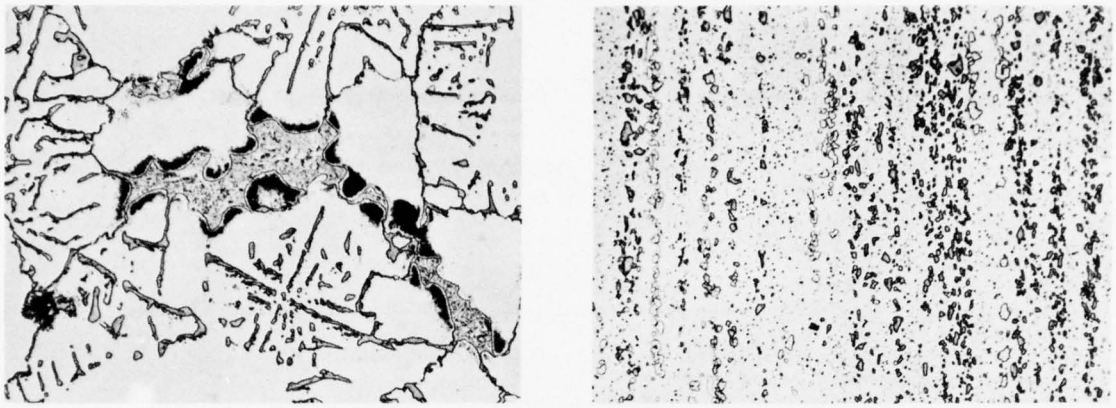
TABLE I - Actual composition (wt. %) of the prealloyed cobalt-base powders.

Grades(*)	Ni	Cr	Mo	Ti	C	O_2 (ppm)	N_2 (ppm)
V 0	14.9	20.1	10.0	-	0.03	703	nd**
V 1	11.0	21.1	10.5	-	0.29	522	"
V 2	14.7	20.1	10.2	-	0.65	226	"
V 3	14.5	20.1	10.4	-	0.95	296	"
V 4	14.7	20.0	10.0	-	1.36	280	"
V 5	14.8	20.0	10.0	-	1.8	273	"
V 6	-	15.7	5.0	5.1	0.05	1806	"
V 13	-	15.3	3.3	4.5	0.103	60	16
V 14	-	16.2	3.0	5.0	0.005	100	32

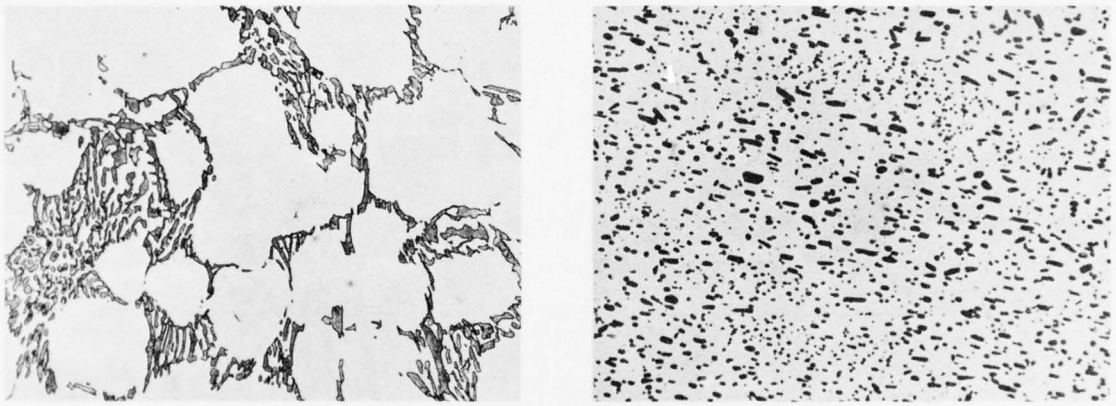
(*) V 0 to V 6 powders obtained by N_2 atomization
V 13 and V 14 powders obtained by REP
(**) n.d. : not determined



As-cast

After extrusion and hot forging

Mar-M-509 alloy (5)



As-cast

Sintered for 2 hours at 1260°C and forged at 1100°C (Red. in cross-section : 5.8).

Stellite 6 alloy (6)

Fig. 1. Microstructural evolution of cast cobalt base alloys during hot working (Mar-M-509) or processing by P/M techniques (Stellite 6). Magnification : x 500

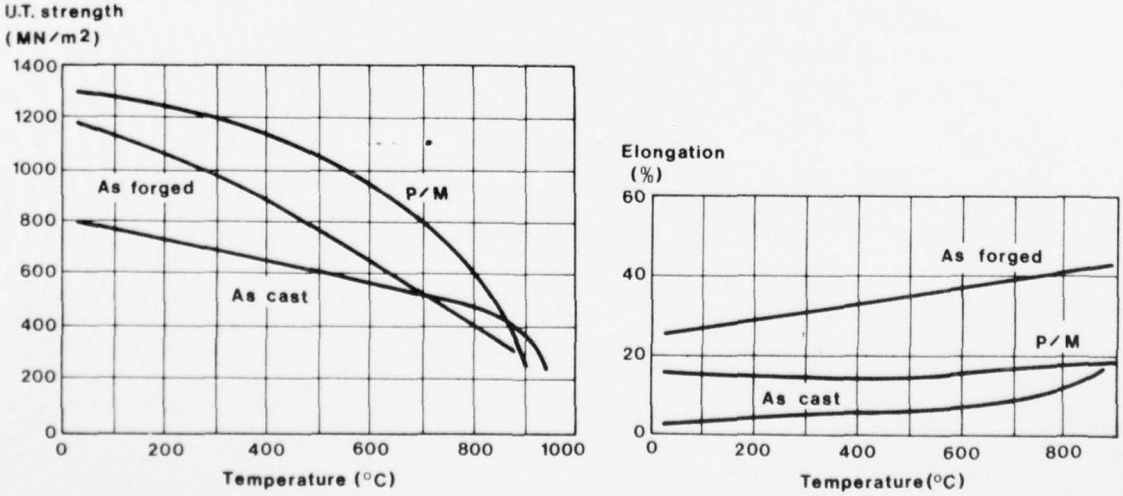


Fig. 2. Tensile properties versus temperature for as-cast, as-forged (5) and P/M (7) X-40 alloy.

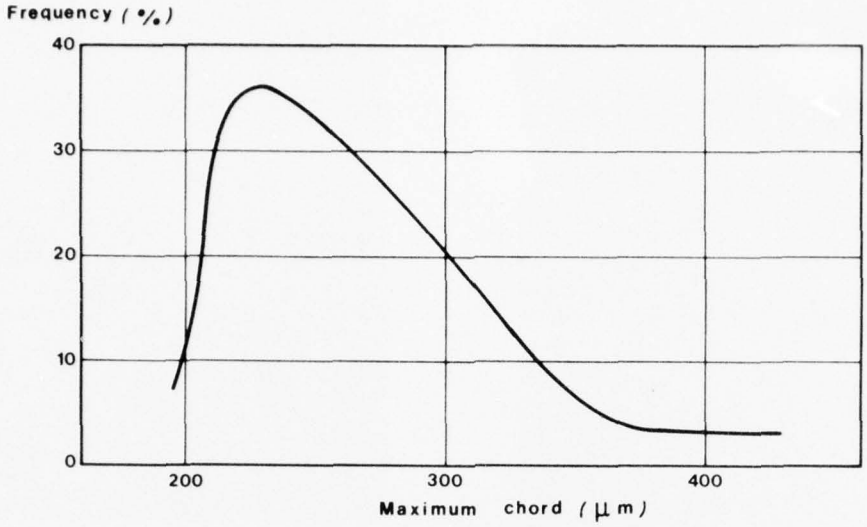
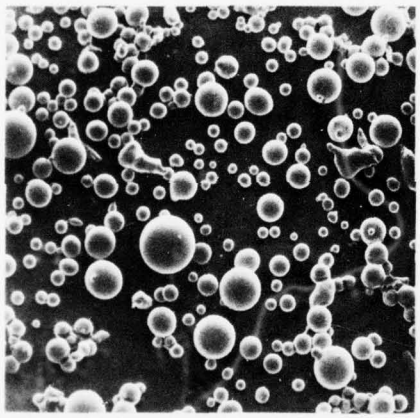
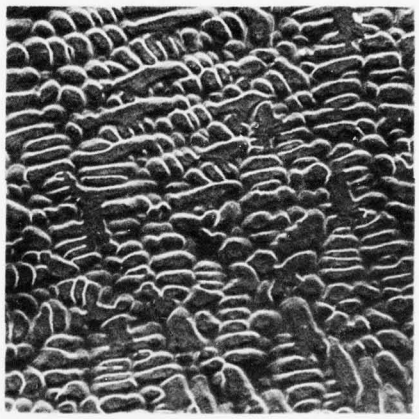


Fig. 3. Size distribution of the V 13 (Co-15Cr-3Mo-5Ti-0.1C) powders produced by the Rotating Electrode Process.

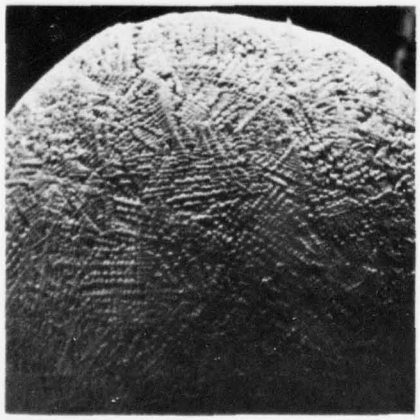


x 40



x 2000

Fig. 4. Scanning electron micrographs of the 1.36%C-V 4 powders.



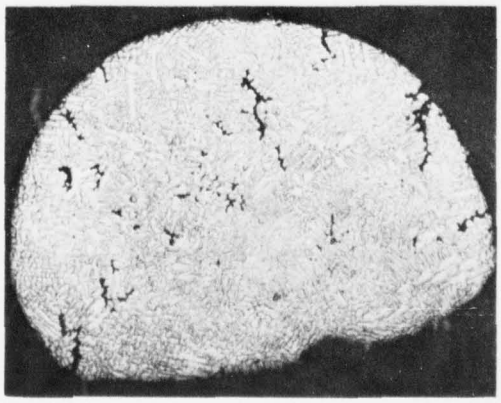
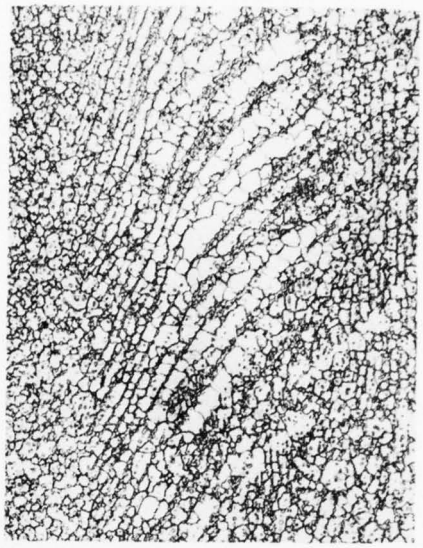


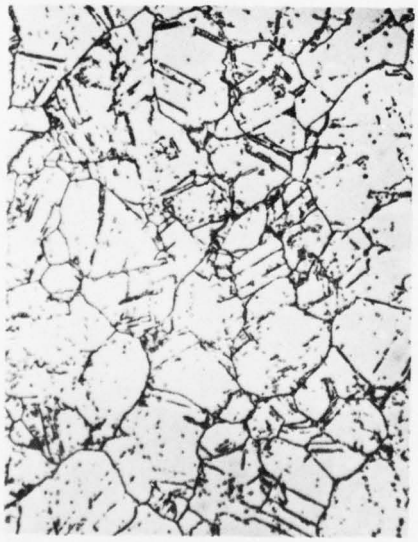
Fig. 5. Microstructure of the V 13 (Co-15Cr-3Mo-5Ti-0.1C) REP powders (x 200)

V 0 (0.03%C) alloy

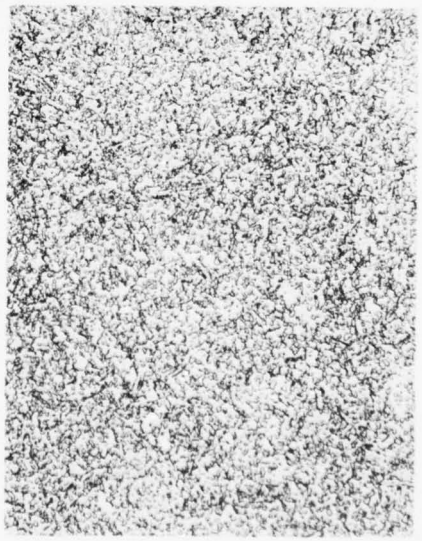


V 2 (0.65%C) alloy

Fig. 6. Optical microstructure of two P/**M** alloys in the as-extruded condition (x 500)

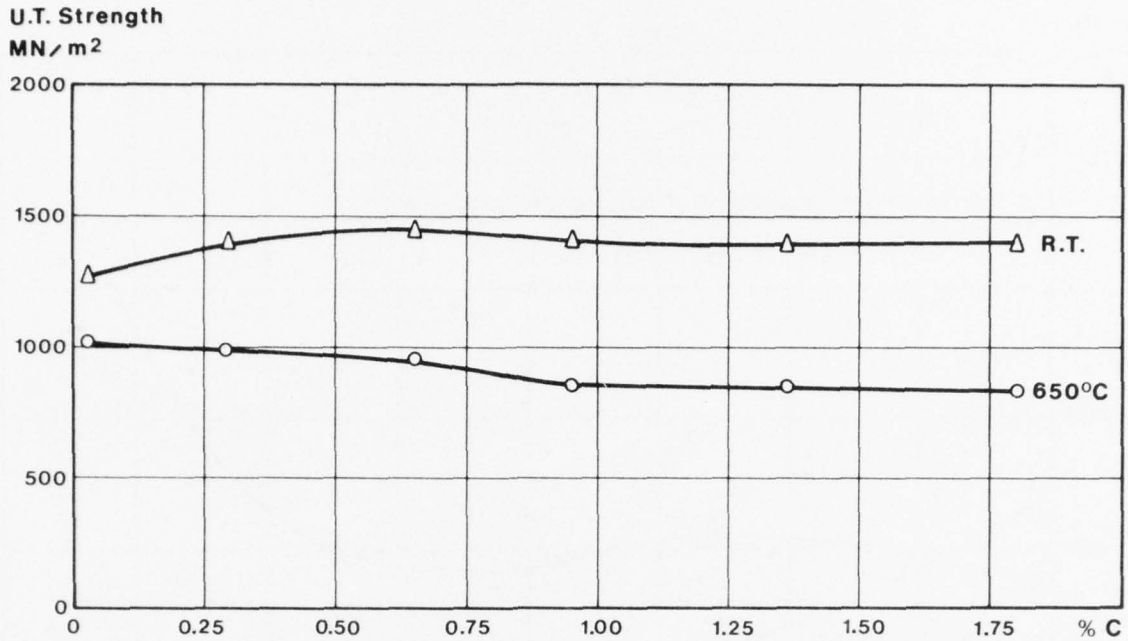


V 0 (0.03%C) alloy



V 2 (0.65%C) alloy

Fig. 7. Optical microstructure of P/M alloys extruded at 1200°C (Red. in area : 4.6), rolled at 1100°C (20% per pass) and aged at 800°C (48h, A.C.) (x 500)



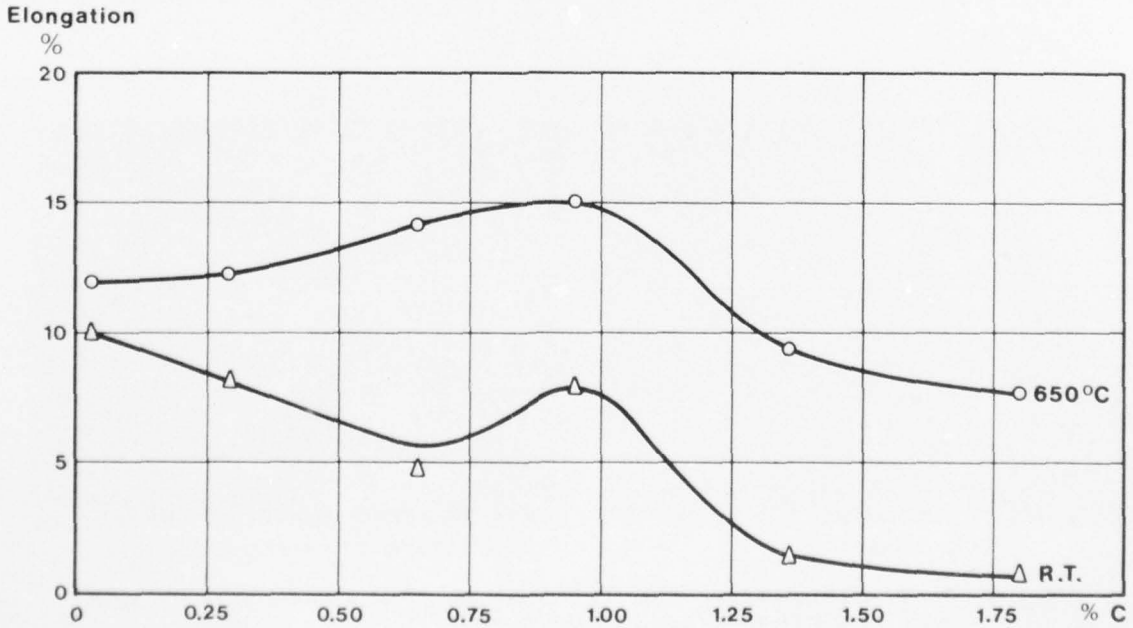


Fig. 8. Dependence of tensile properties on carbon content for Co-20Cr-10/15Ni-10Mo-C P/M alloys rolled at 1100°C and aged at 800°C (48h, A.C.)

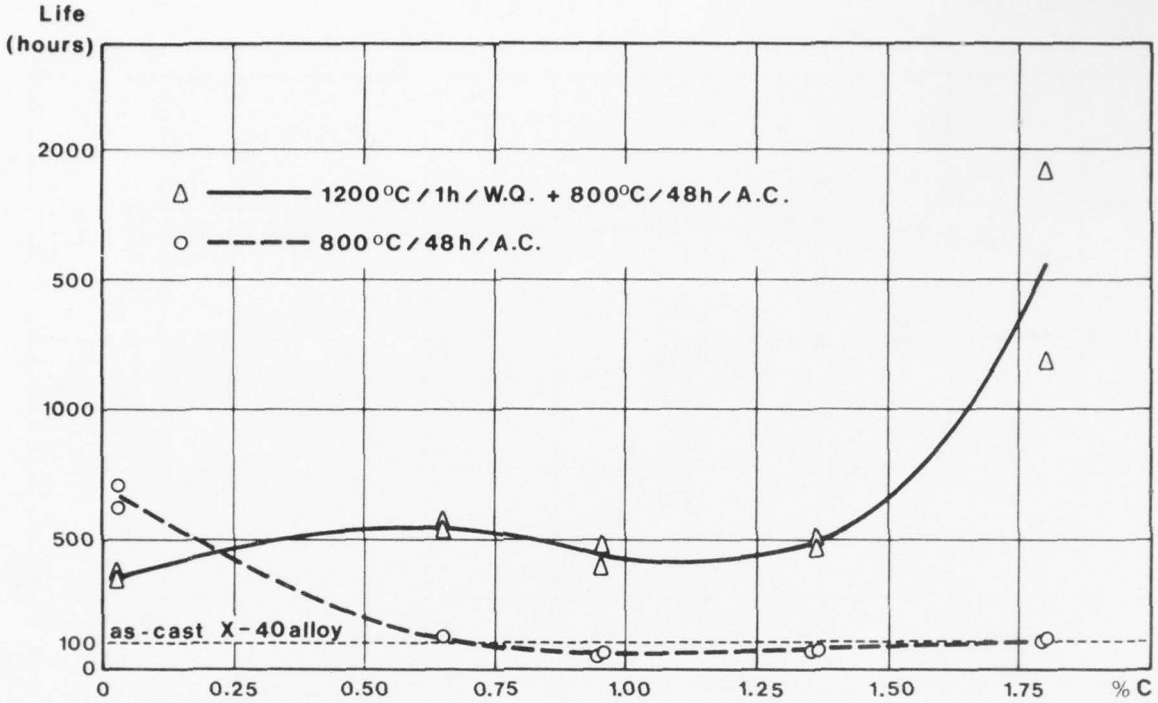
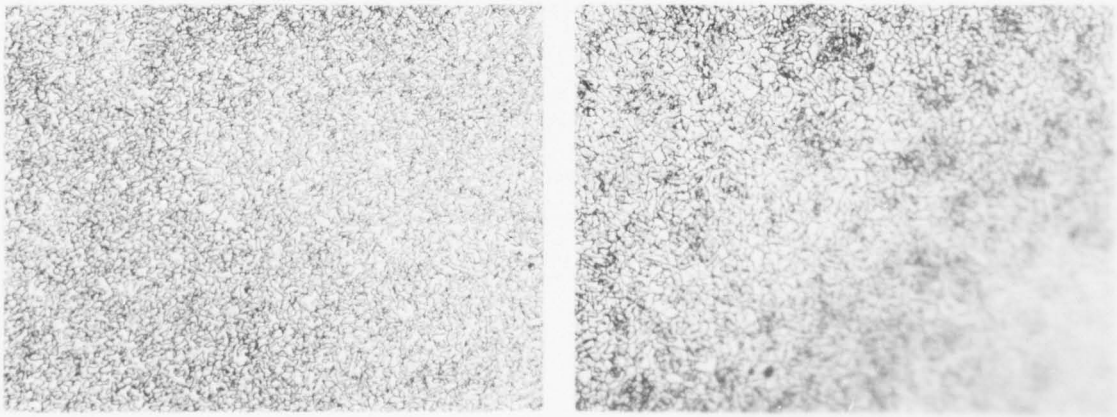


Fig. 9. Effect of carbon content on 650°C stress-rupture life(applied stress : 363 MN/m^2) of Co-20Cr-15Ni-10Mo-C P/M alloys rolled at 1100°C



800°C/48h/A.C. 1200°C/1h/W.Q. + 800°C/48h/A.C.

Fig. 10. Optical microstructure of the 1.8%C-containing P/M X-5 alloy after rolling at 1100°C and heat-treating as indicated (x 500)

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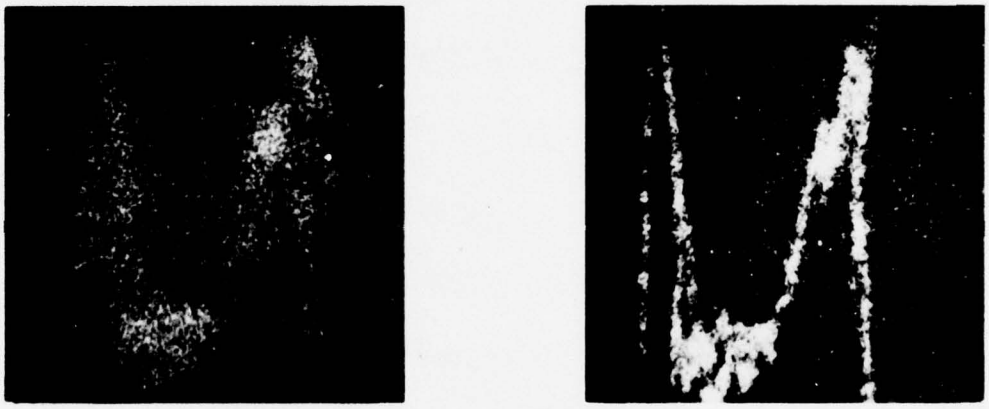
As-extruded V 6 (5Mo-0.05C) alloy obtained from N_2 atomized powders.

as-extruded

after swaging at 1100°C (15% red. in area per pass)

V 13 (3Mo-0.1C) obtained from REP powders

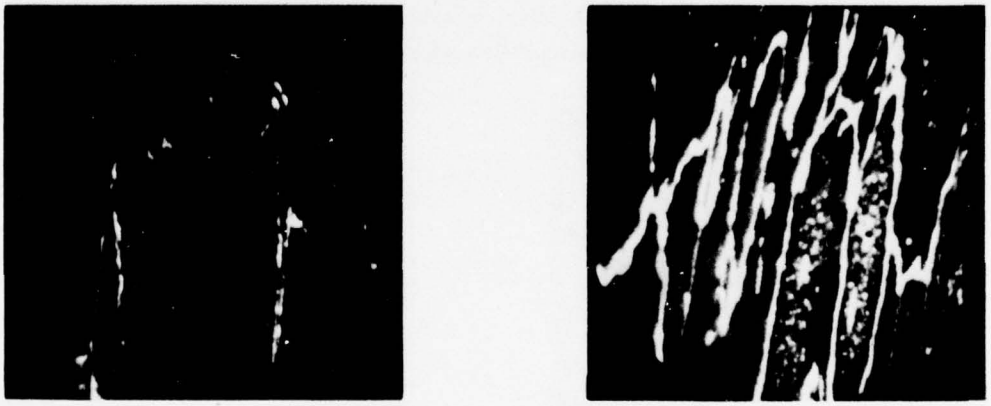
Fig. 11. Optical microstructure of Co-15Cr-5Ti-Mo-C P/M alloys (x 100)



Oxygen

Titanium

Electron-microprobe analysis (scanning 100 μm x 100μm)

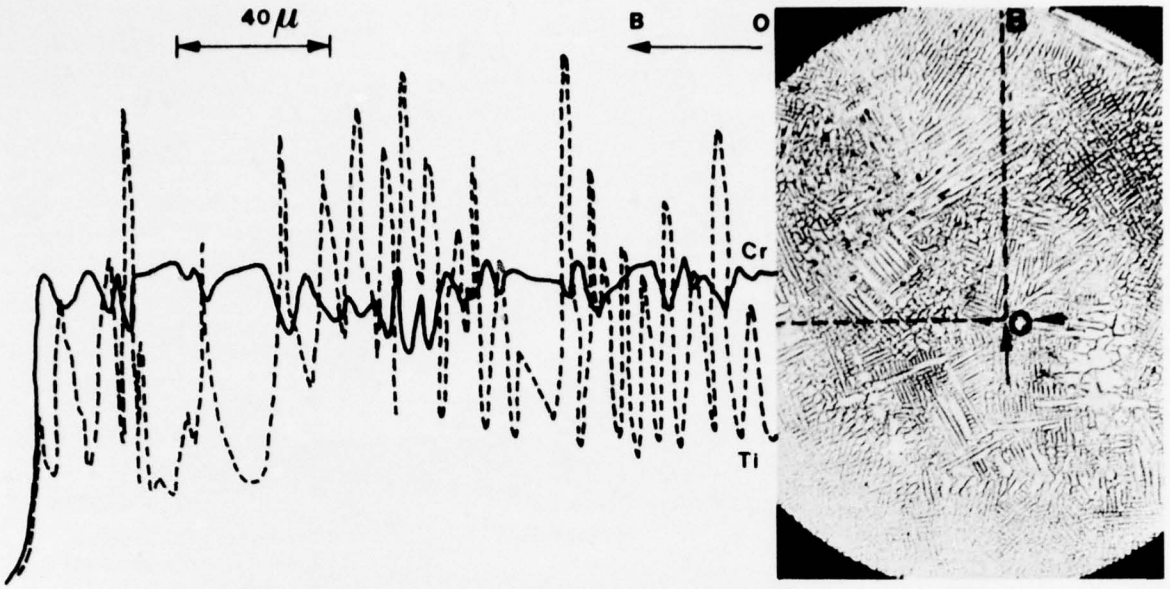


$24\ C^-_2$

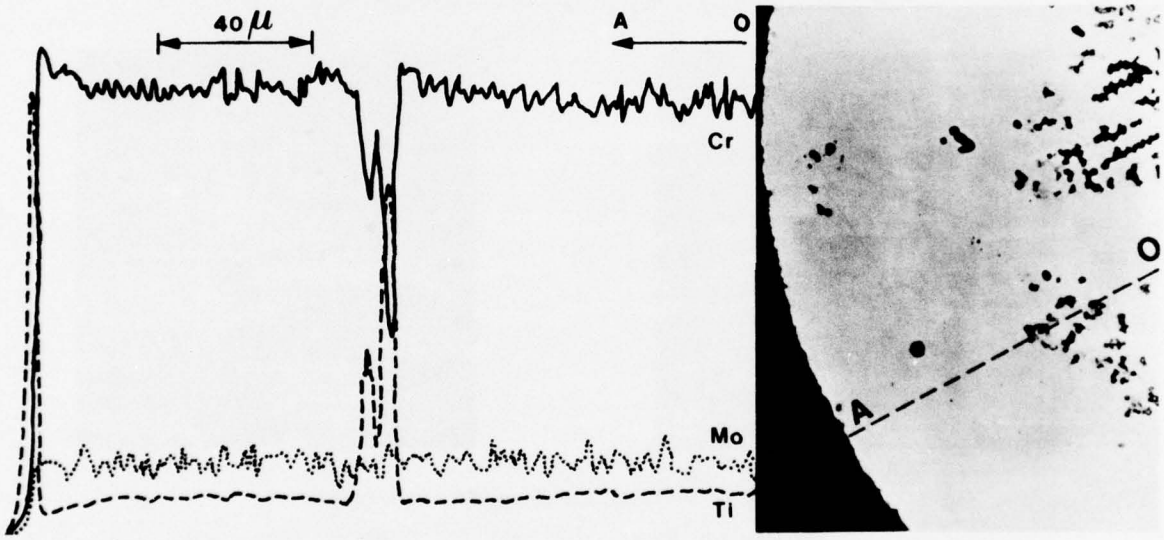
$48\ Ti^+$

Ion-microprobe analysis

Fig. 12. Distribution of elements in the PPB's particles of the as-extruded V 6 (Co-15Cr-5Mo-5Ti-0.05C) P/M alloy.

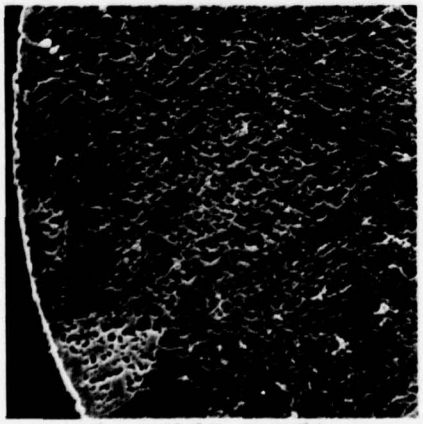


As-received

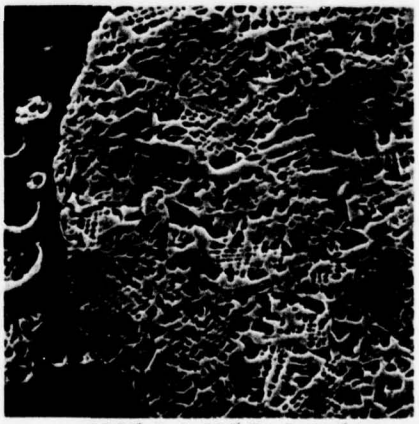


After a 1200°C/1h/A.C. heat treatment.

Fig. 13. Qualitative electron-microprobe analysis of the V 13 (Co-15Cr-3Mo-5Ti-0.1C) REP powders.

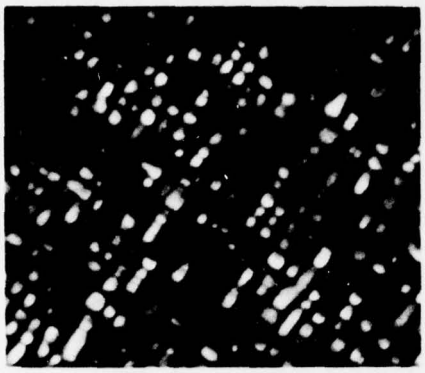


0.18%C-4.81%Ti-5.62%Al
(REP powders-44 ppm O_2)



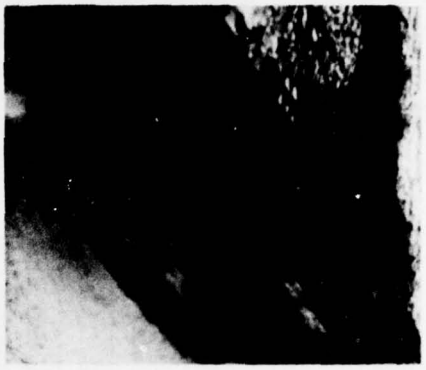
0.007%C-4.30%Ti-5.03%Al
(argon atomized powders 94 ppm O_2)

Fig. 14. Scanning electron micrographs of IN-100 powders with different carbon contents after a 1150°C/1h/A.C. heat-treatment (x 2000)



x 90.000

γ' -phase
(dark-field picture)



x 40.000

$\gamma' \longrightarrow \eta$ transformation

Fig. 15. Transmission electron micrographs of the V 6 (Co-15Cr-5Mo-5Ti-0.05C) P/M alloy extruded at 1200°C, solution treated at 1200°C (1h, W.Q.) and aged at 800°C (8h, A.C.)

As-extruded x 100 30% total red. in area (fine graine) x 200 70% total red. in area (coarse grains) x 200

After rolling at 1100°C

Fig. 16. Optical microstructure of the V 14 (Co-15Cr-3Mo-5Ti-0.005C) P/M alloy.

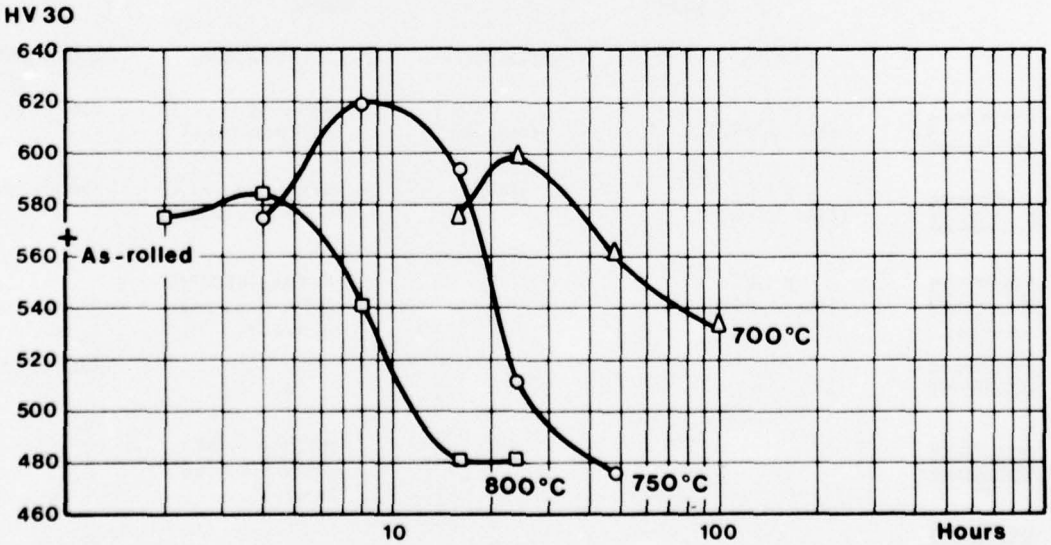
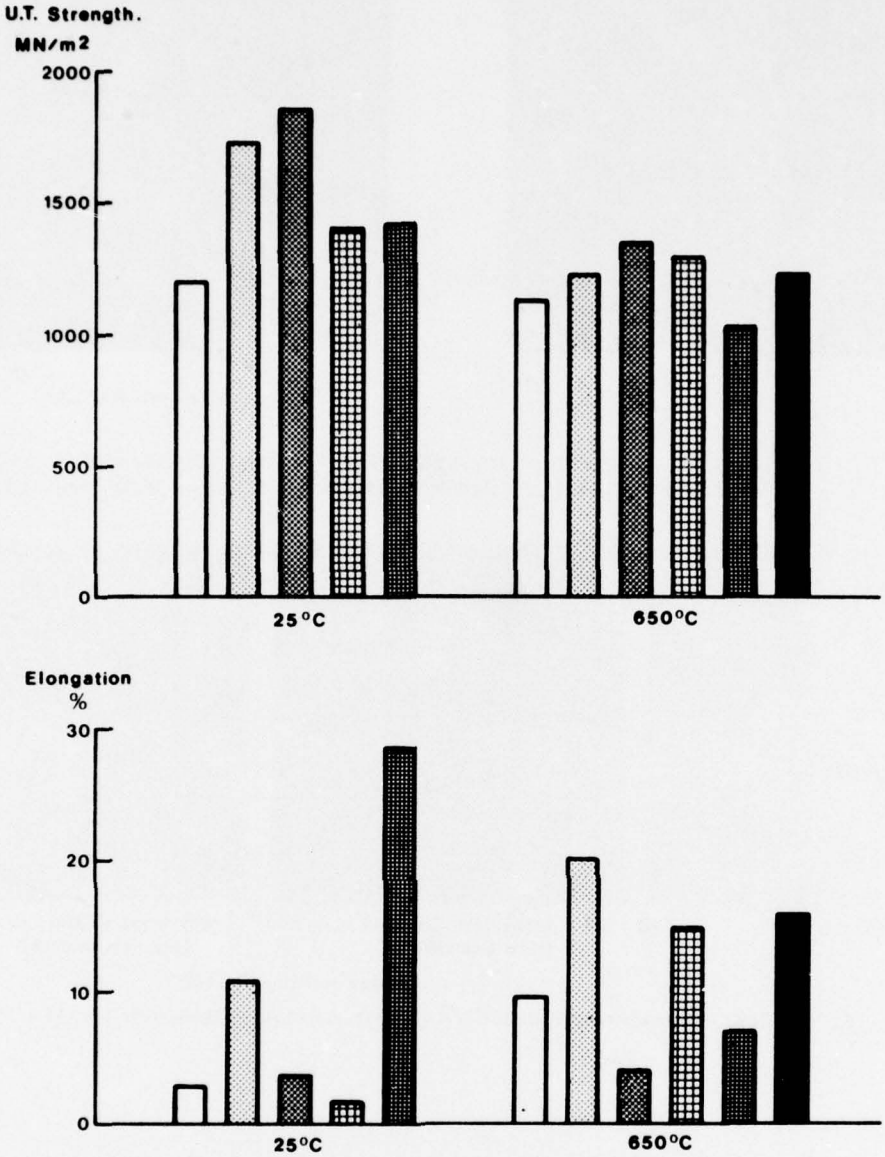


Fig. 17. Hardness evolution versus aging time and temperature for the V 14 P/M alloy rolled in one blow at 1100°C (30% red. in area)



	Alloy	Powders	Working	Heat-treatment
	V 6 (5Mo-0.05C)	N_2 atomized	Swaging 1100°C (5% per pass)	750°C/48h/AC
	V 13 (3Mo-0.1C)	REP	Swaging 1100°C (15% per pass)	700°C/100h/AC
fine grained	V 14 (3Mo-0.005C)	"	Rolling 1100°C (30% total red. in area)	-
	"	"	"	700°C/100h/AC
Coarse grained	"	"	Rolling 1100°C (70% total red. in area)	-
	"	"	"	700°C/100h/AC

Fig. 18. Temperature dependence of tensile properties of Co-15Cr-5Ti-Mo-C P/M alloys.

ADVANCED FABRICATION TECHNIQUES

DISCUSSION SUMMARY OF SESSION III

by

A. J. Williams
Dept. of Energy, Mines & Resources, Ottawa, Canada

The principal discussion of Dr. Arnold's paper (P. 6) addressed the question of whether an as-HIP Rene 95 product with its great cost advantage can be produced with sufficiently good properties to satisfy present Rene 95 applications. It appears that General Electric has done sufficient work to convince themselves that their as-HIP Rene 95 with its ASTM8 grain size can be used for most present applications in spite of the loss of the duplex necklace microstructure. Because of the very large cost saving available, they are also looking at the possibility of substituting as-HIP Rene 95 for lower strength superalloys (718 for example).

After the presentation of Dr. Betz and Dr. Schubert (P. 7) there was considerable discussion of the unusual scatter in the low cycle fatigue results on Udimet 700 and IN100 at 600°C. It was established that the scatter in LCF data was due to the presence of many Ti-rich and some Fe-rich inclusions, with the evidence pointing to the fact that premature failure occurred when inclusions were located near the specimen surface. The origin of the inclusions was not clear but it appeared that they did not originate during powder handling or pre-treatment of the pressings. It was speculated that the Ti-rich ones may have originated in the ingot used for powder production and the Fe-rich ones at the wall of the powder production vessel. Although no firm conclusion could be drawn as to the origin of the inclusions, the discussion drew attention to the necessity for extreme care in powder production and in all subsequent handling and processing steps.

Discussion of M. Walder's presentation (P. 8) centred on whether forging of the alloys discussed was really necessary after HIP consolidation, since the properties of the as-HIP materials were virtually the same as those of the forged materials. M. Lescop explained that it was true that tensile properties were virtually identical in both forged and unforged parts but that forging conferred superior LCF properties. Particularly in Astroloy, the duplex necklace structure with its concomitant superior fatigue properties could only be produced by a forging procedure.

Discussion of Dr. Wallace's paper (P. 9) dealt with the occurrence of sulpho-carbide flakes in superalloys consolidated by super-solidus sintering und pressure. Relatively few sulpho-carbide particles formed when the pressing was done at sub-solidus temperatures. However, when super-solidus sintering was performed to achieve the large grain size favourable to good creep properties, the structure was characterized by masses of sulpho-carbide flakes at the grain boundaries. These were seriously detrimental to ductility. Dr. Wallace postulated that the presence of the liquid phase during consolidation allows the rapid diffusion of C and S, and the gettering elements present in the alloy combine readily with these to form the coarse intergranular flakes. He doubted, for this reason, that super-solidus sintering had much potential as a powder consolidation method.

In answer to a number of questions following his paper (SC 7), Mr. Mazzei provided a number of further details of the proposed manufacturing process for W reinforced composite turbine blades for ground based turbine application. The model system being used is that of W fibres coated with an HfC diffusion barrier in a Mar M200 powder matrix. Experiments have been conducted with composites containing 25-65 vol % W wire but most testing has been done on a 50 vol % composite. A simulated root section is being formed as an integral part of the experimental blade and the W fibres are being terminated in a randomized fashion with an average 1/2-in. penetration into the root section. An attempt is being made to optimize the fibre termination method by finite element analysis. During lay-up the powder is handled incorporated in a polymethyl methacrylate tape. If careful vacuum degassing is employed, the tape can be completely removed and no undue carbide precipitation or porosity occurs if low carbon (.02% or less) powder is used. An isothermal forging step is envisaged as the final blade shaping procedure. Westinghouse is well aware of the potential thermal fatigue problem with this type of composite material. However, they believe that the combination of high volume percentage of fibre together with the final isothermal forging step will sufficiently reduce the tendency towards thermal fatigue damage. Thermal cycling under load up to 1100°C for two thousend hours has not yet produced any evidence of thermal fatigue tendencies or barrier coating damage.

During discussion of M. Drapier's paper (P. 10) several suggestions were made as to how to avoid producing carbides on particle boundaries during the processing of experimental cobalt-base superalloys, other than that suggested by him of keeping the C content low. Work described by Simms and Hagel on Rene 41 suggests that if consolidation is carried out at a low enough temperature (1000-1100°C in their case) subsequent heat treatment produces random carbide precipitation. Therefore, a duplex heating for consolidation would seem to be a useful approach to avoidance of boundary precipitation. The addition of niobium to complex the carbides might also be useful. Very rapid heating for consolidation would reduce the time for diffusion of Ti and C to the particle surfaces before compaction. In addition to these suggestions, Dr. Wenzel offered his view regarding the mechanism of this boundary carbide precipitation. At temperatures below the γ' solvus the free energy of formation of γ' is higher than that of titanium carbide. As the temperature is raised, the γ' tends to go into solution and the activity decreases. During this period, C migrates to the particle boundaries because of the high dislocation density there. As the Ti is released from the γ' as the latter dissolves, it also migrates to the particle boundaries where it reacts with the C already there since, at the higher temperature, the free energy of formation of the carbide is higher than that of the γ'.

SESSION IV

COMPARATIVE EVALUATION OF FORGED Ti-6Al-4V BAR MADE FROM SHOT PRODUCED BY THE REP AND CSC PROCESSES

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Imperial Metal Industries (Kynoch) Ltd
Witton, Birmingham B6 7BA

SUMMARY

Ti-6Al-4V shot was obtained from two sources, the Centrifugal Shot Casting Process (CSC) and the Rotating Electrode Process (REP). The chemistry and size distribution of the two types of shot were compared prior to an evaluation of the consolidated products. Alloy shot billets were produced by hot isostatic pressing (HIP) and the mechanical properties of as-HIP and as-HIP + forged/rolled material compared.

The two types of shot behaved in a similar manner and generally produced microstructures, tensile and fracture toughness properties similar to cast and wrought material. However, the low cycle fatigue behaviour of the consolidated material was inferior to that observed in the conventional product. Internal fatigue origins were found to be associated with defects in the shot and in general the degree of scatter was higher and fatigue strengths were lower than in conventional material of a similar section size.

INTRODUCTION

Like other studies of titanium alloy powder the present evaluation was targetted towards potential aerospace applications of consolidated material, in particular use in rotating aero-engine components.

The technical advantages, which are expected to accrue from an alloy powder route are summarised below:

(1) Billet can be produced which is free from contamination by large defects. The fine grain size should also improve the sensitivity of ultrasonic inspection for defects.

(2) Long range compositional variations caused by ingot segregation will be avoided.

(3) Consistent fine grain size and microstructure will be achieved throughout the billet. The fine structure is expented to be maintained in all parts of the forging itself.

(4) As a result of 2 and 3 even large complex forgings are expected to possess a higher and more consistent fatigue strength and improved crack propagation characteristics.

(5) Greater flexibility will be possible in the design of the forging route to the finished shape. Control of metal flow in different parts of the forging will be less important and this could result in cost reduction.

(6) In the long term new alloys will be developed which cannot be produced by conventional casting and working.

The initial approach to the question of titanium alloy powder has been similar to that of other organisations in the field. After first considering the alternative forms of powder, ie hydride-dehydride, elemental, gas atomised and shot, the conclusion was reached that the latter route offered the best prospects of success, particularly when interstitial and surface contamination and ease of handling were considered.

The data presented in this paper detail the preliminary evaluation of Ti-6Al-4V alloy shot obtained from two sources, the Rotating Electrode Process[1] (REP) and from the Centrifugal Shot Casting process[2] (CSC). An initial assessment of the shot was made prior to consolidation of two billet sizes by hot isostatic pressing (HIP). Comparative data was then produced on the mechanical properties (tensile, low cycle fatigue and fracture toughness) of the consolidated material both as-HIP and after rolling and forging.

ASSESSMENT OF SHOT

The Ti-6Al-4V alloy shot was obtained from AERE Harwell, UK (CSC) and Nuclear Metals Inc, US (REP). The details of the processes are given elsewhere[1,2].

The CSC shot was produced from a 50 mm Ø x 500 mm long cast and wrought bar. The shot was formed in an argon-helium atmosphere with a yield of about 60% useable material. (The yield of shot is limited by the size of the furnace chamber and not by any basic limitation of the process). The analyses of the starting electrode and the shot are given in Table 1 and the sieve analysis is detailed in Table 2.

The corresponding analytical and size information for the REP shot are given in Tables 1 and 2, respectively. No information was available on the composition of the starting electrode for REP shot.

TABLE 1

ANALYSIS OF SHOT AND ELECTRODE MATERIAL

Type	Al	V wt %	Fe	O_2 ppm
CSC Electrode	6.56	4.50	0.14	1600
CSC Shot	6.56	4.54	0.16	1650
REP Shot	5.8	4.4	0.08	1159

TABLE 2

SHOT SIEVE ANALYSIS

Sieve Size μm	1180	710	500	355	250	180	125	90	PAN
% Retained on Screen CSC	0.28	5.4	21.79	39.24	21.85	7.52	2.73	1.17	0.27
% Retained on Screen REP	-	-	-	7.20	26.79	39.42	18.13	5.83	2.63

CONSOLIDATION OF SHOT

Although a number of consolidation processes were considered for the alloy shot, it was felt that hot isostatic pressing offered the best and most reliable route, particularly as comparative data was being generated.

Both types of shot had a tap density of about 65%. All encapsulation was in mild steel cans. Two sizes of cans were used, one 150 mm x 25 mm ø and the other 216 mm x 100 mm ø. Both electron beam and TIG welding techniques were used in the construction and sealing of the evacuated cans. No significant difference was observed in the subsequent behaviour of the cans, irrespective of which welding process was used.

All cans were HIP pressed for 4 hours at a temperature of 950°C and a pressure of 1.03 kbar. The pressing reduced linear dimensions of the cans by about 10%. The mild steel cans were removed from the titanium alloy billets by machining prior to further evaluation and processing.

THERMO-MECHANICAL PROCESSING

(1) Small Billets

After removal of the cans the billets were approximately 20 mm ø. A section was first removed for assessment of the as-HIP + heat treated properties of the material. The remaining material was then rolled at 950°C to 12 mm square; a reduction of about 56%. Both types of consolidated material deformed in an identical manner, similar in fact to the normal cast and wrought product.

All samples were heat treated at 960°C air cooled + 2h at 700°C.

(2) Large Billets

The as-machined billets were approximately 175 mm x 85 mm ø. A slice was taken from each billet for evaluation of the as-HIP material, and the remaining material was forged.

The material was forged between flat plattens. A small amount of preliminary deformation was carried out at 1125°C to square the section, but the majority of the processing was performed at 950°C, to 50 mm square (54% deformation). Figure 1 shows the final shape of one of the forgings. Although some cracking occurred it was limited to the surface. Both forms of consolidated billet performed in a similar manner, and were not significantly different from conventional material.

The forged REP and CSC billets were heat treated at 960°C, water quenched and annealed for 2 hours at 700°C.

METALLURGICAL AND MECHANICAL PROPERTY EVALUATION

The microstructures of the as-HIP + heat treated material, in both the small and large billets were very similar. The only difference between the CSC and REP material (Figures 2 and 3) was in the fact that the former contained more alpha phase. This fact was due to the difference in β transus of the two materials, it being higher in the CSC case, due to the higher level of oxygen in the alloy.

Room temperature tensile values for the as-HIP + heat treated material (20 mm ø) are given in Table 3. The small quantity of rolled bar produced from the 20 mm ø billet was used entirely to generate low cycle fatigue data. All the testing was carried out at room temperature under constant load (zero minimum) conditions and a frequency of 10 cycles/minute. The results are presented in Figure 4.

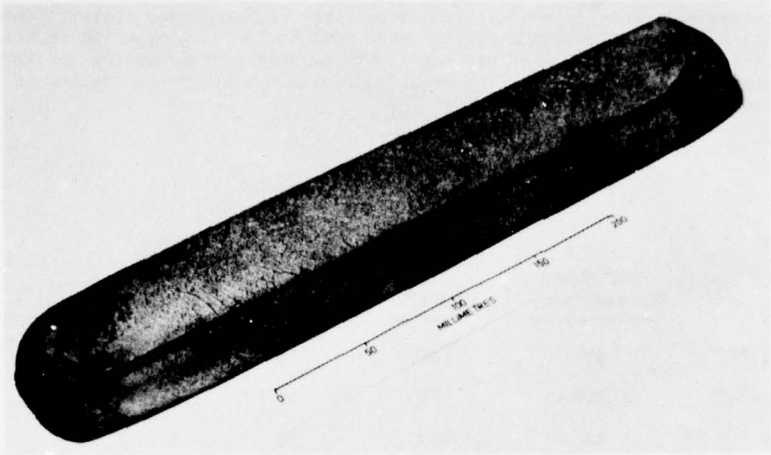

Figure 1. HIP + Forged CSC Shot Billet

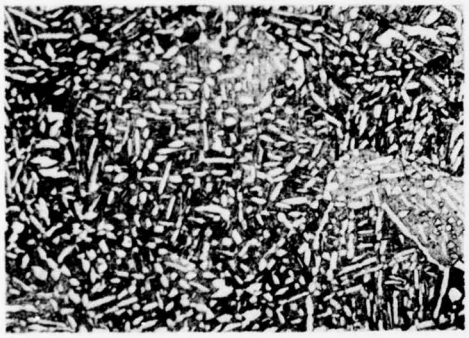

Figure 2. Microstructure of CSC, HIP + Heat Treated Billet X150

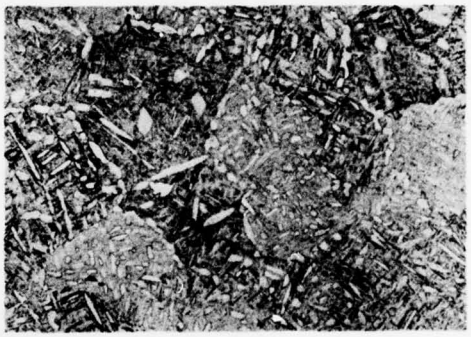

Figure 3. Microstructure of REP, HIP + Heat Treated Billet X150

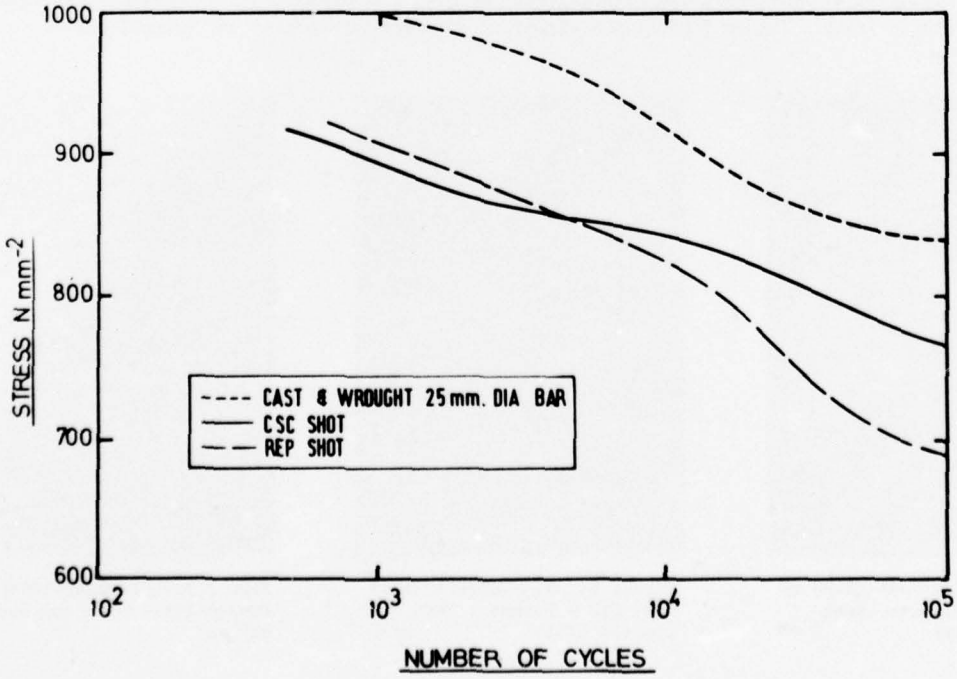

Figure 4. Low Cycle Fatigue Data for Alloy Shot Bar (12 mm square) and Conventional 25 mm Ø Bar

A more extensive mechanical property evaluation was possible on the forged billet. The tensile property levels in the as-HIP + heat treated condition were similar to those observed in the small billet material. Both transverse and longitudinal tensile tests were carried out on the forged bar. The tensile results are presented in Table 3. The results of fracture toughness testing in the transverse direction are shown in Table 4.

TABLE 3

TENSILE PROPERTIES

Material		0.2% Proof Stress $N.mm^{-2}$	UTS $N.mm^{-2}$	Elong $4\sqrt{So}$ %	R in A %
CSC HIP + Heat Treated*		957	1075	18	36
REP HIP + Heat Treated*		907	1030	21	40
CSC HIP + forged + heat treated	T **	922	1038	16	38
	L **	942	1053	15	39
REP HIP + forged + heat treated	T **	940	1048	20	40
	L **	903	1026	19	40
Conventional material (75 mm Ø)	T	951	1032	20	37

* 20 mm Ø billet

** 50 mm square forging

TABLE 4

FRACTURE TOUGHNESS PROPERTIES

	Material	K_Q $MNm^{-3/2}$
(50 mm square)	REP HIP + forged + heat treated	68
(50 mm square)	CSC HIP + forged + heat treated	67
	Conventional material	57

Microstructurally the materials appeared similar, Figures 5 and 6 show the structure in REP and the CSC forgings respectively. Figure 7 shows the structure of conventional cast and wrought billet for comparison.

Figure 5. Microstructure of REP, HIP + forged + heat treated Billet X150

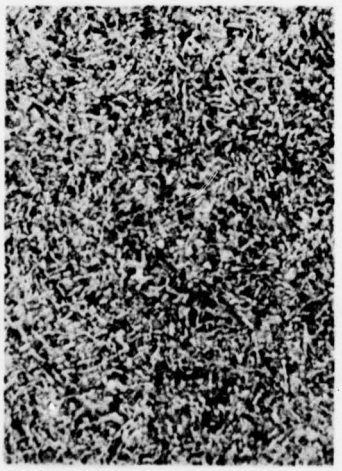

Figure 6. Microstructure of CSC, HIP + forged + heat treated Billet X150

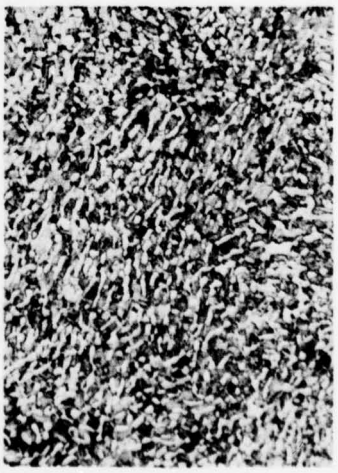

Figure 7. Microstructure of conventional cast and wrought Billet X150

Low cycle fatigue data was obtained on longitudinal specimens and is presented in Figure 8.

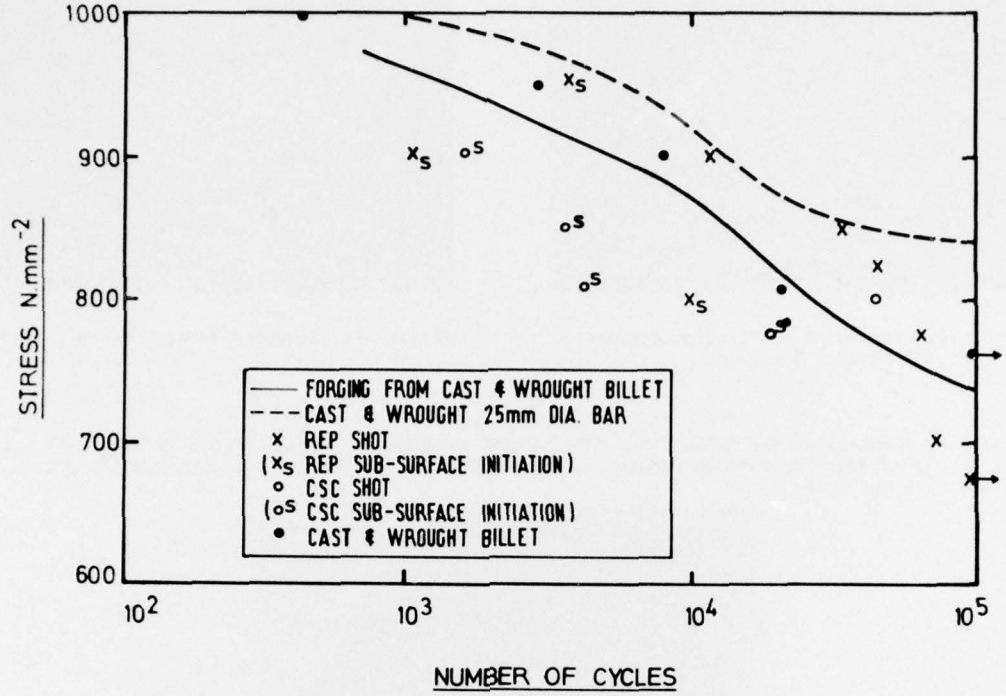

Figure 8. Low Cycle Fatigue Data for Alloy Shot Forgings (50 mm square) and Conventional Billet and Bar

A striking feature of all the low cycle fatigue data was the degree of scatter and the presence of internal initiation sites. Both optical and electron metallographic techniques were used to study the phenomenon of internal initiation. Metallographic sections through the REP material revealed the presence of a significant number of angular particles, Figure 9. The particles were up to 200 μm long and were identified by electron probe micro-analysis, as tungsten. Scanning electron microscopy was used to study the fracture surfaces of the failed fatigue samples. Figure 10 shows the origin in one of the REP specimens, while Figure 11, an x-ray distribution photograph, reveals the presence of tungsten at the origin. In fact all the internal origins were found to be associated with tungsten particles.

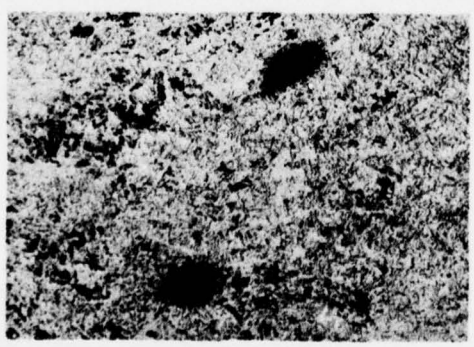

Figure 9. Tungsten Inclusions in REP Billet

X70

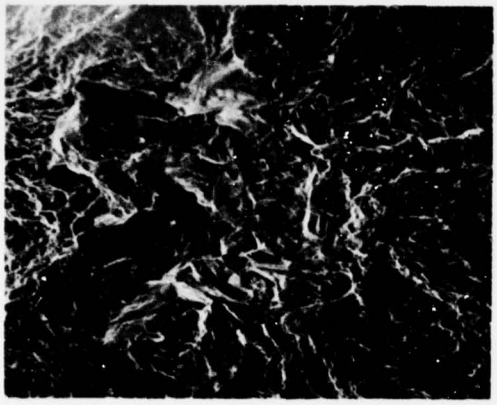

Figure 10. Inclusion in Fracture Surface of REP Fatigue Specimen X120

Figure 11. Tungsten X-ray Image of Figure 10 X120

A similar examination was made of the CSC fatigue specimens. Once again the internal origins were found to be associated with foreign particles, Figure 12. In this particular instance the origin was an alloy steel shot particle.

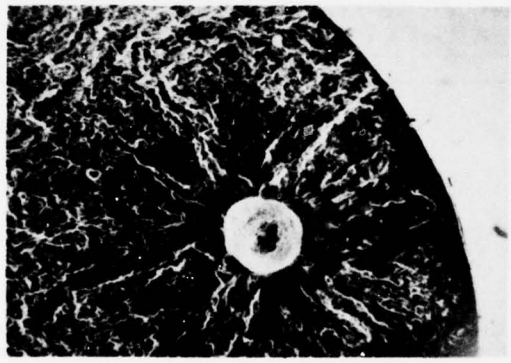

Figure 12. Inclusion in Fracture Surface of CSC Fatigue Specimen X150

DISCUSSION

One of the observations to come out of the results is that although there was a significant difference in the size range between REP and CSC shot the two materials handled in a similar way. No differences were observed in the way they consolidated or subsequently deformed. The chemistry of both the CSC and REP shot was controlled and the interstitial contamination in the CSC shot was negligible. The problem of yield in the CSC process appears to be a function only of the tank size and not a fundamental limitation of the method.

In general both consolidated materials behaved in a normal manner and the tensile and fracture toughness levels were at least comparable with conventional cast and wrought material. However, a major divergence from normal was detected in the low cycle fatigue behaviour of both materials, in particular the degree of scatter, the levels of results and the presence of internal initiation sites.

The internal initiation sites were readily explained by the presence of 'foreign' particles. However, the observation does underline the necessity of entirely eliminating defects, if an alloy shot route is ever to be successfully used for rotating components. Furthermore it is significant that only the fatigue test revealed the presence of the defects, unlike the tensile and fracture toughness tests which were apparently unaffected. The sources of the defects in the present batches of material are known. Tungsten particles have been seen before in REP shot[3], and come from the non-consumable electrode. They could clearly be eliminated by a change in the electrode material. The contamination of the CSC shot is not a basic problem of the process, but due to cross contamination from previous runs. The situation should be overcome by the exclusive use of a furnace for titanium alloy melting and redesign of the tank for ease of cleaning.

Although the presence of internal origins reduced the fatigue life significantly, the general trend in surface initiated specimens, in both the small and large billet material, was for lower fatigue strengths and a greater degree of scatter than in conventional cast and wrought material of a similar section size (Figures 4 and 8). It is perhaps relevant to compare the results on alloy shot with the fatigue data of conventional billet, which has had about 80% work from the ingot (solid circles Figure 8). Whereas no internal origins were seen in the conventional material the scatter in the fatigue results is similar to that of the consolidated materials, which have had about 54% work from the as-cast

state. The microstructure in the conventional billet (Figure 7) is similar, though slightly coarser, than the consolidated and deformed materials (Figures 5 and 6). An explanation of the fatigue behaviour of conventional billet has been sought in the degree of chemical segregation, texture and grain size. Work has shown that a minimum amount of reduction, of the order of 100:1, is required to maximise the fatigue properties. The fatigue results of the consolidated and forged materials would also suggest that further work is necessary to maximise the levels. It could be argued that this deformation is necessary to minimise the content of micro-segregation within the former shot particles and possibly to strengthen the inter-particle bonding. However, although the evidence is not conclusive, because of the presence of defects leading to internal fatigue origin and premature failure, it does support observations of other workers[3] on the high cycle fatigue properties of consolidated REP shot. The results presented do call into question some of the possible advantages of the alloy shot route. In particular the claim that, because of the fine grain size, components can either be pressed to shape or nearly to shape, requiring either none or little subsequent forging; such components may have significantly inferior fatigue performance compared with conventionally cast and wrought materials.

CONCLUSIONS

1. The behaviour and performance of CSC and REP consolidated shot are similar.

2. Microstructure and tensile and fracture toughness properties in HIP and HIP + deformed materials are at least as good as those in cast and heavily worked material.

3. Scatter and fatigue strengths in HIP + worked material are inferior to those in conventional material of an equivalent size.

4. The fatigue properties in HIP + worked materials are highly susceptible to the presence of foreign particles, which lead to internal initiation origins.

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ACKNOWLEDGEMENTS

The authors wish to thank the Ministry of Defence (Procurement Executive) for their financial support of the work and their permission to present this paper.

SOME COMMENTS ON THE MECHANICAL PROPERTIES OF HIP TITANIUM

by

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1. INTRODUCTION AND SCOPE

Titanium users are interested in HIP processing because of the potential cost savings in producing intricately shaped parts.

The feasibility of fabricating complicated shapes in a one-step process has already been proven. However, fabrication techniques have to be optimied in order to obtain high mechanical properties without sacrificing economy.

It is the purpose of this short contribution to discuss briefly the mechanical properties of HIP titanium TiAl6V4 produced under different conditions and to investigate the factors which influence the fatigue values.

2. EXPERIMENTAL PROCEDURES

2.1 POWDER

It is obvious that the quality of the starting material will greatly influence the final material properties. Several methods for producing titanium powder have been discussed in literature (1), (2), (3), (4). For the present investigation only commercially available powders have been used.

Fig. 1 shows the characteristics of three typical powders, produced by the Rotating Electrode method (REP) or by Electron Beam Melting (EBM). While the physical properties, e.g. shape and microstructure, are similar, there are important differences in the foreign element content. The EBM powder shows the highest purity, this may be influenced by the fact that it has been produced in a pilot plant set-up. It has to be noted that in the REP materials contamination was found to be present not only as solid solution in the titanium matrix but also as discrete inclusions of foreign particles, such as tungsten and the alkali metals. Powders A and B have been produced in the same plant but with modifications to the operational mode. The increased purity of batch B indicates that improvements in powder manufacturing are possible; the results of this study show that they are necessary.

Fig. 1 also indicates that there are considerable differences in the gas content and the mesh size of the three powders. Therefore, detailed specifications had to be developed to insure reproducibility of the final product.

2.2 POWDER HANDLING

Although titanium is less sensitive to oxygen pick-up than superalloy powders, adsorption of gases and moisture may be a problem. Therefore three different operating procedures are being investigated.

PROCEDURE A

The powder is stored in air and filled into the capsule without special precautions. The capsule is then evacuated, degassed at 300°C, for 8 hours and sealed. Several days may elapse before HIP processing.

This is a low-cost procedure.

PROCEDURE B

The powder is stored in a protective atmosphere (purified N_2). It is then transferred to a specially developed filling station (see figure 2) while still being in a nitrogen atmosphere. Subsequently, filling station and capsule are evacuated and the powder is filled into the capsule under vacuum conditions. After degassing at 450°C for 16 hours the capsule is sealed and stored in an evacuated container.

PROCEDURE C

In this procedure (not yet fully realized) the powder will be filled into evacuated containers right at the powder production plant. Any contamination during shipping and storage will be minimized.

In all cases low alloy carbon steel was used as the capsule material. Simulated production parts were run in steel, nickel and titanium capsules (Fig. 3).

2.3 HIP PROCESSING

All samples were hot pressed in ASEA QIH 16 presses. Al_2O_3 was used in some cases as filling between samples as a pressure carry over medium. Some problems were encountered in obtaining uniform temperature distribution along the specimens.

3. MECHANICAL PROPERTIES

To find use in airframe applications, HIP material must reach or exceed the strength values of conventionally produced TiAl6V4, aspecially forged and machined material. Comparison is not always easy because testing conditions vary widely between different laboratories.

3.1 STATIC VALUES

Figure 4 shows the static tensile properties of HIP TiAl6V4 obtained with different sets of HIP parameters. It is obvious that strength values comparable to forged material can be obtained by choosing suitable pressing temperatures. In the range of pressures investigated in the present study, there appears to be no dependence of tensile properties on HIP pressure.

If the ß transus of the alloy is exceeded during HIP processing, UTS and YS values decrease by 10 to 15 %. While the reduction of area decreases, there is some increase in total elongation. This is somewhat contrary to the results obtained in the heat treatment of conventionally processed material.

It is interesting to note that the static properties are relatively independent of the processing parameters and of the soundness of the material:

Mil-specification values can be obtained even with relatively porous structures and large quantities of inclusions. Therefore, it is concluded that HIP processed

material cannot reasonably be evaluated by static tensile testing.

3.2 FATIGUE PROPERTIES

Fatigue properties are much more dependent on internal defects than static properties. They are preferred for comparison of different powders and fabrication schemes.

3.2.1 NOTCHED SAMPLES

To evaluate the notch sensitivity of the material, two batches of samples were tested; one processed above, one processed below the beta transus. In both cases procedure A was used, the samples contained inclusions of foreign elements. During sample preparation it was discovered that, for reasons not yet determined, a large number of specimens processed in the α/β region contained some porosity.

However, they were included in the test program to obtain information on the influence or internal defects.

All specimens were tested under tension at a notch intensity factor $\alpha_k = 3.2$.

The results are shown in figure 5 together with mean values for forged material (5) (full line). The data permit the following conclusions to be drawn:

- scatter of data of the α/β material is surprisingly small, considering the influence of porosity in some samples
- β processing (no porosities) results in similar fatigue properties
- the fact that no influence of porosity is visible indicates that some other factor, too, is influential in limiting notched fatigue life.

3.2 SMOOTH SPECIMES

The influence of processing variables on the fatigue behavior of smooth round specimens has been investigated extensively. Some typical results are summaried in Fig. 6 for material processed under the following

conditions

Procedure A

curve	1	60 min	1000 bar	920/875°C
	2	30 min	2000 bar	1050°C

Procedure B

curve	3	60 min	1300 bar	920°C

For comparison purposes the data obtained under the same experimental conditions form a forged and rolled plate are also included in the graph. (Directionality was excluded by taking samples at 90°, 45° and 0° to the rolling direction.) The following conclusions can be drawn:

- Fatigue strength of the rolled material is approximately 220 N/mm^2. In all cases HIP-processed material is inferior to conventional titanium
- Fatigue strength of β-processed HIP titanium amounts to 160 N/mm^2. By α/β processing, this value increases to about 180 N/mm^2, Similar values have been

given in literature (6). The influence of pressing below or above the ß transus on the fatigue properties corresponds to what is known as the influence of forging temperature (7).
In the α/β range minor temperature variations (920 vs 875°C) do not appear to have a significant influence on properties

- Controlled powder handling markedly increases fatigue strength to 195 N/mm^2.

4. DISCUSSION

Results obtained to date show fatigue properties to be inferior to those of forged or rolled material. The main reason appears to be impurities in the starting powder. The fact that controlled handling in an linert atmosphere improves the mechanical properties, points to the influence of adsorbed gases and moisture. Obtaining optimum properties may well require an integrated powder cycle, where the powder may stay under vacuum or in an inert atmosphere from the powder production plant to the final filling and canning installation. Such a system would certainly influence the economic picture. However to date, this influence of adsorbed gases has been shown only indirectly by comparing mechanical properties of products handled in air or under vacuum. Chemical analysis has not indicated any clear-cut differences.

While the effect of gases is not yet fully understood, it is obvious that inclusions play a vital role in diminishing the fatigue life. In most cases the fracture origin is well discernible in broken samples, and micrprobe analysis can be made. It was found that fractures tend to start at inclusions, typically tungsten or alkali metals. Examples of inclusions are given in figures 7 and 8 both optically and by SEM. Fig. 9 correlates the fracture origin and the microprobe data. The fact that the majority of the broken samples investigated contains inclusions in the fracture surface suggests that these inclusions are the true reason for the relatively low dynamic properties.

Elimination of these impurities should not be impossible: Tungsten quite certainly stems from the W-electrodes frequently used in the REP processes. Efforts are being made to replace them by titanium electrodes. Careful analysis of the alkali inclusions corroborates the hypothesis that they may originate in the heat shields of the REP installation. Here, again, improvements should not be impossible. As to the use of EBM powders, results to date are somewhat conflicting and will have to be substantiated before final conclusions are possible.

5. OUTLOOK

To be truly competitive with conventional fabrication methods, the HIP technology of titanium will have to fullfill three essential conditions:

Possibility of manufacturing complicated components

cost advantages

satisfactory mechanical properties

- The first point appears to be solved. Even complicated parts can be fabricated without major problems and with good dimensional reproducibility (Figs. 10 - 12 show some titanium airframe parts produced by HIP). For intricate shapes the manufacture of appropriate canning may pose some problems but electroforming on wax or aluminium master forms has been found to be an elegant solution

- Cost still is a problem. Although we do not have truly detailed calculations, there appears to be a cost savings potential for parts requiring a large amount of machining, especially for hollow parts such as shafts. Should scrap conversion to powder become a reality, HIP titanium would appear even more advantageous.

  Total cost for HIP processing of titanium can only be determined after all technical problems are solved, especially:

  powder characteristics

  powder handling and storage

  control of HIP parameters, e.g. uniformity of temperature distribution

  microstructure

- mechanical properties of HIP TiAl6V4 still are to be improved. The challenge will be to find processing parameters yielding optimum fatigue data without unduly increasing cost.

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Powder	A	B	C
Process	REP	REP	Electron beam melting
form	globular	globular	globular
structure	martensite	martensite	martensite
grain boundanes	none	none	present
inclusions	W, Ca, K, N	W	none
gascontent			
H_2 (ppm)	100	35	3,5
N_2 (ppm)	50	148 - 157	74 - 81
O_2 (ppm)	650	2000	1100
grain size (µm) w. r. t. 50 weight-%	200	100	≥10
apparent density (%)	61	60	60
	5µm	5µm	5µm

Fig.1 Comparison of Ti Al6V4 powder properties

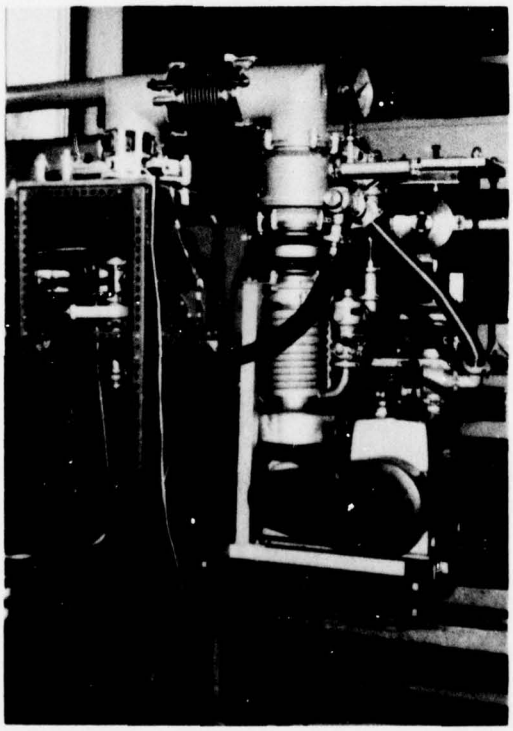
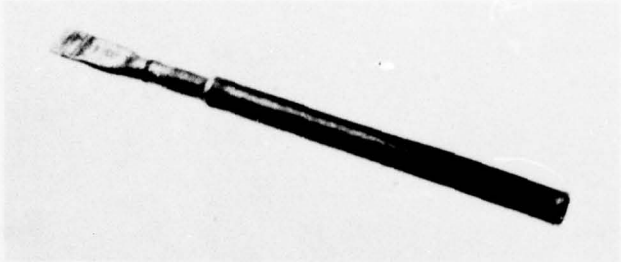
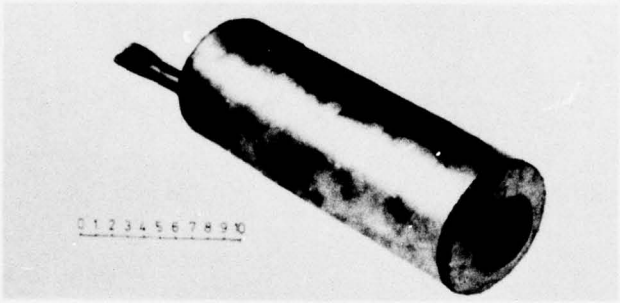


Fig.2 Vacuum filling station



Container for cylindrical specimens
low carbon steel 20ø × 230mm



Container for 20 cylindrical specimens

90ø×45ø×240mm

Fig.3 Container for fatigue specimens

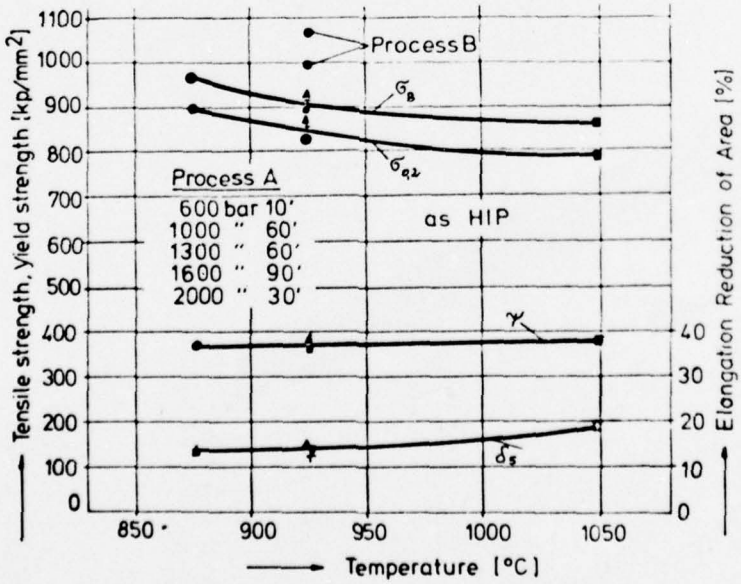


Fig.4 Variation of mechanical properties of HIP – TiAl6V4 with HIP – temperature

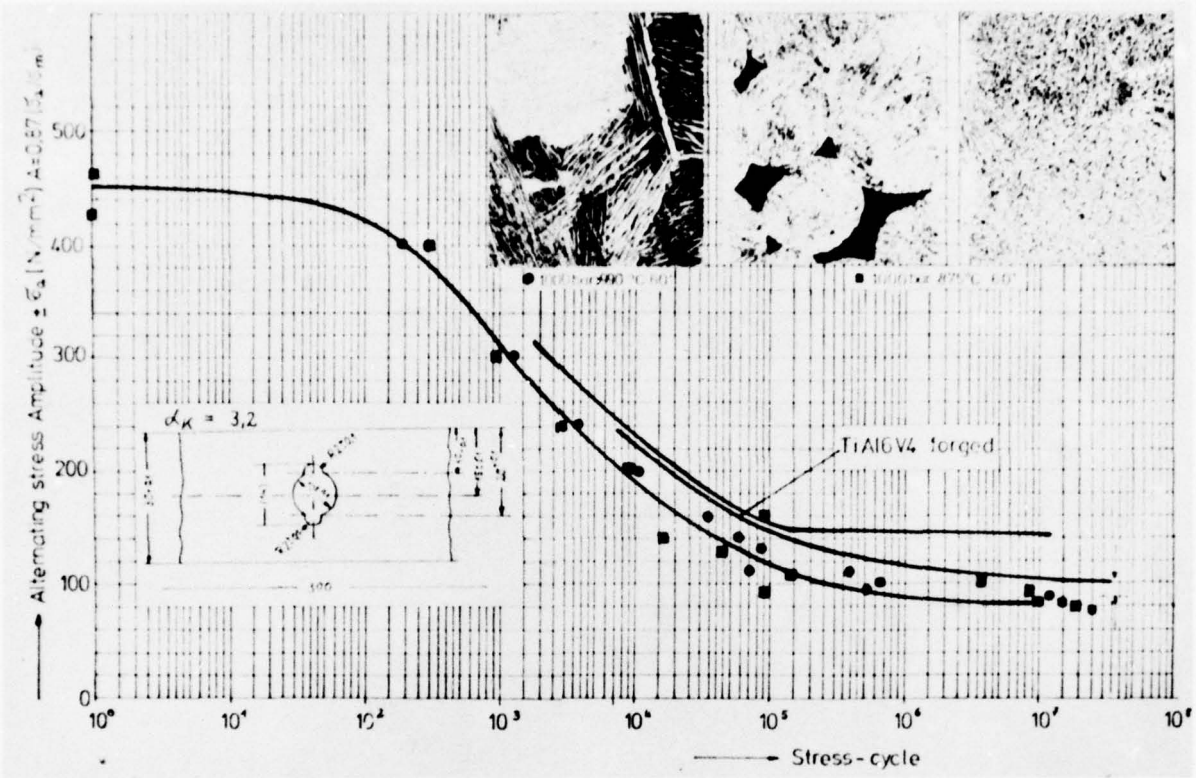


Fig.5 Comparison of notched fatigue strength HIP – TiAl6V4 and forged TiAl6V4 (Process A)
$\alpha_K = 3,2$

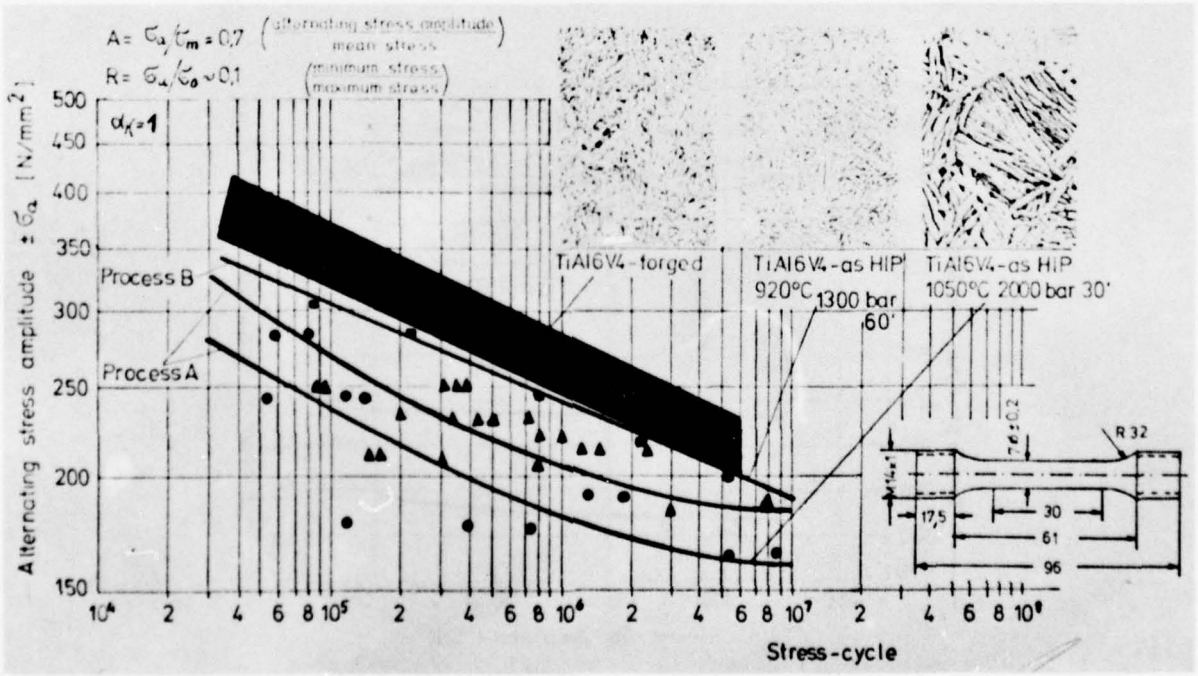


Fig.6 Comparison tensile stress HIP – TiAl6V4 and forged TiAl6V4

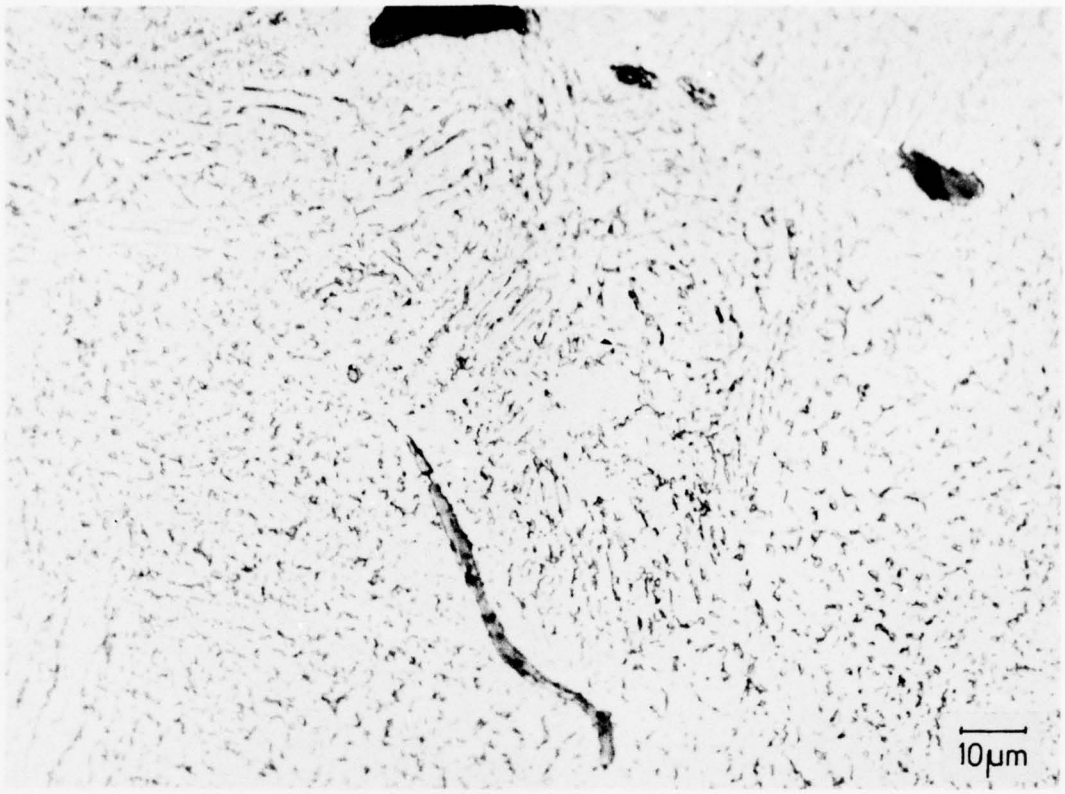


Fig.7 Inclusion in HIP titanium

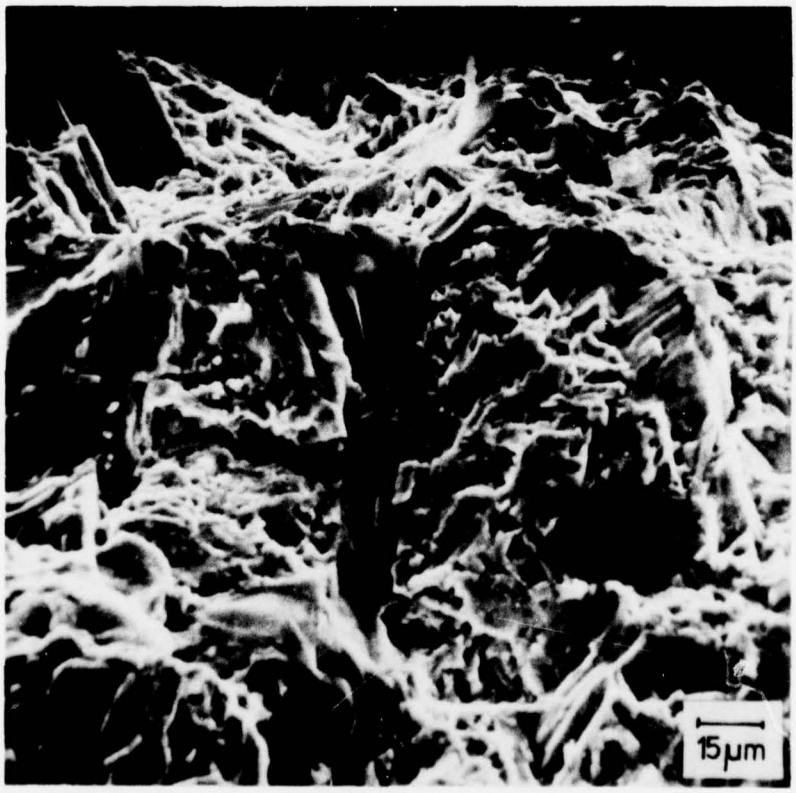


Fig.8 Inclusion in fracture surface

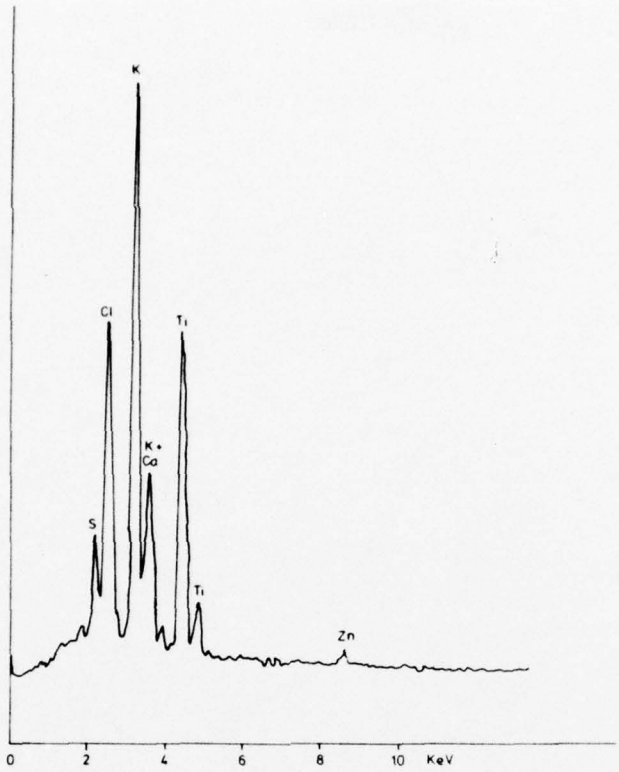


Fig.9(a) Microprobe analysis of inclusion shown in Figure 8

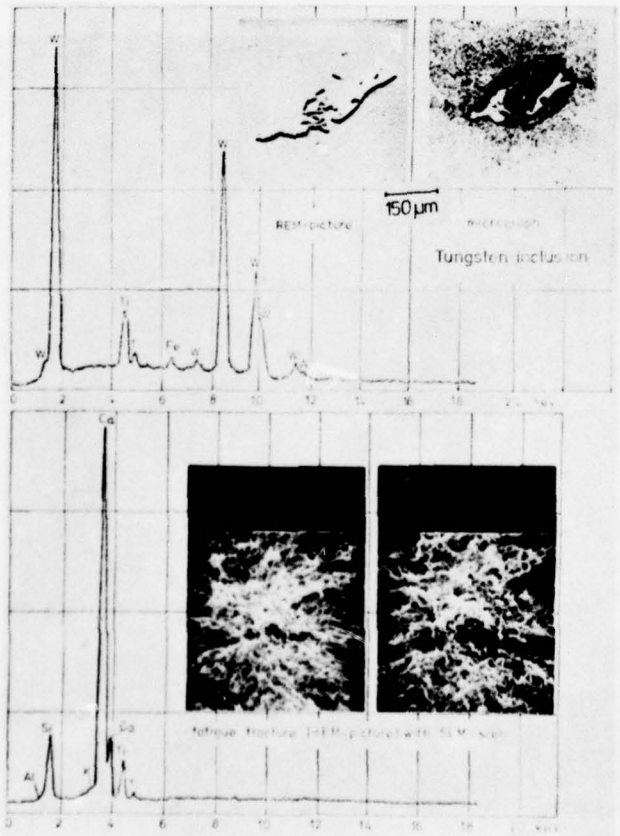


Fig.9(b) Tungsten and alkalimetal – inclusions in HIP – TiAl6V4 – structure

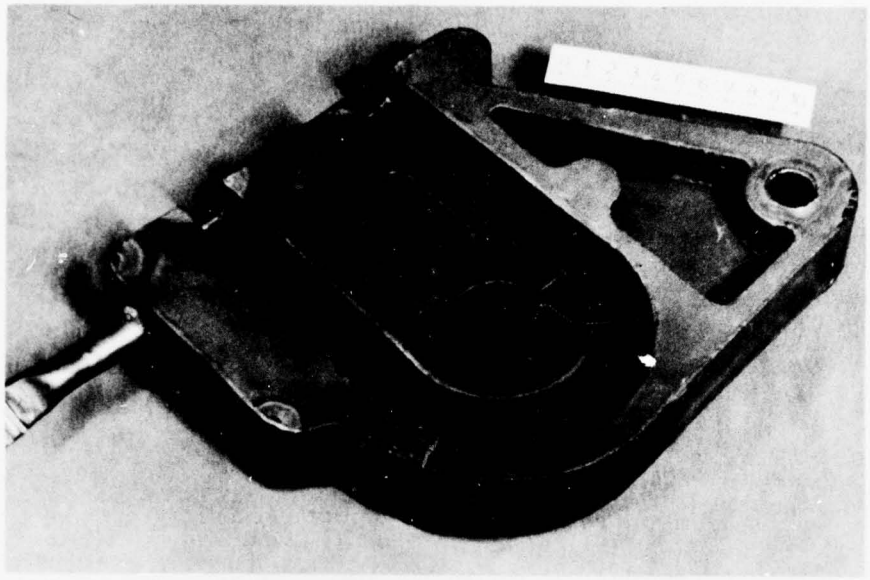


Fig.10 HIP-titanium airframe part

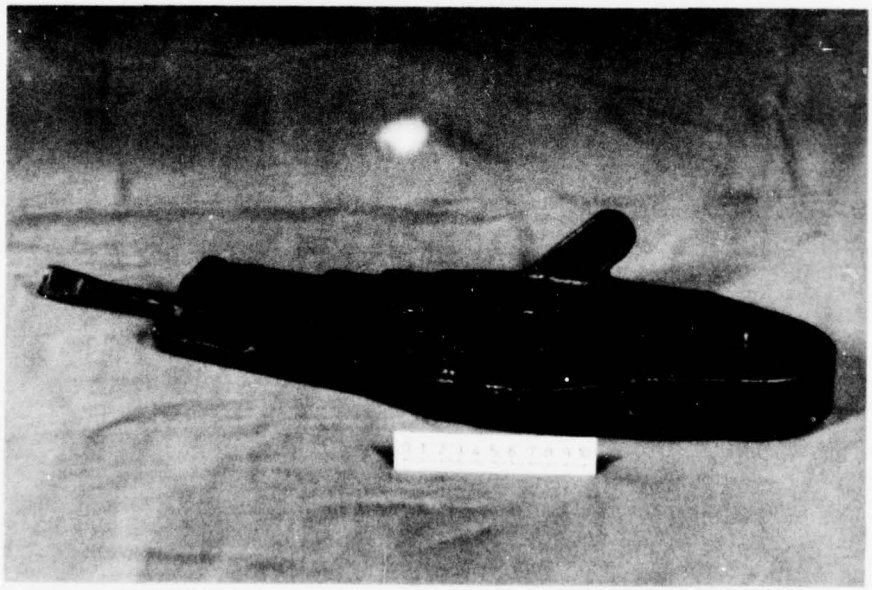


Fig.11 HIP-titanium airframe part

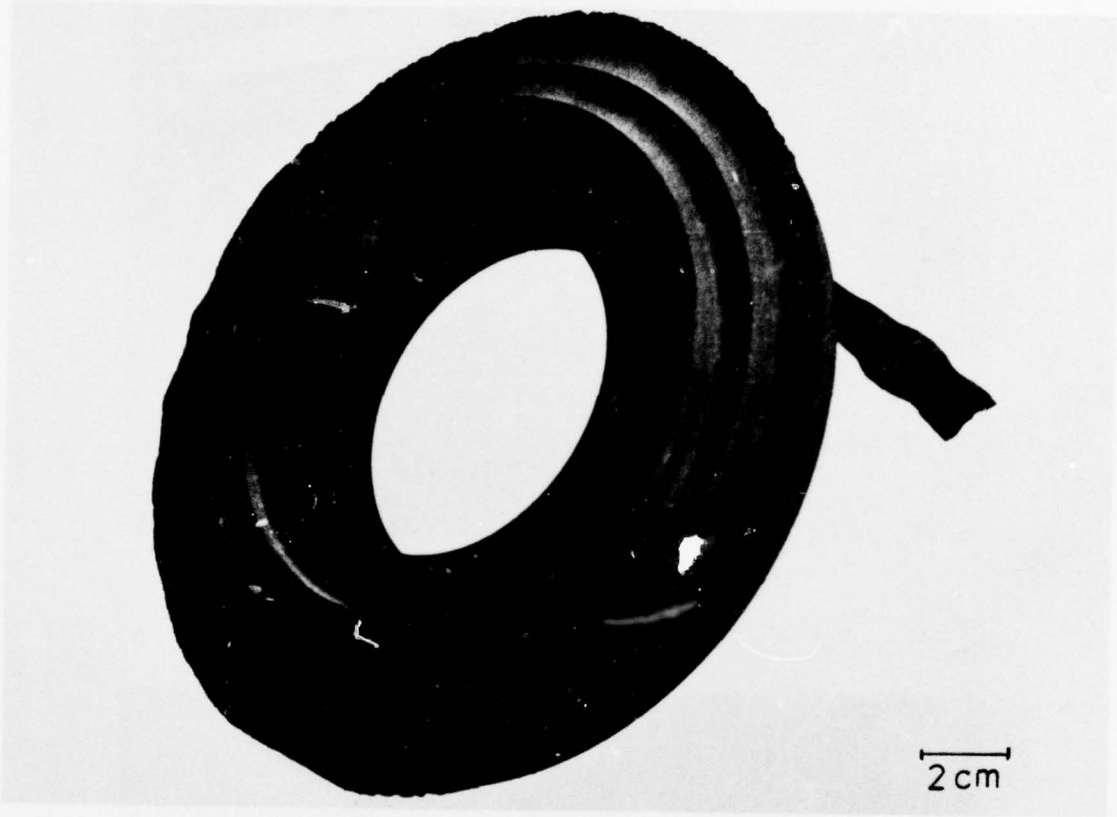


Fig.12 HIP-titanium part

HOT ISOSTATIC PRESSING OF Ti-6Al-4V POWDER FORGING PREFORMS

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45215

SUMMARY

A condensation of the results obtained from an Air Force Materials Laboratory sponsored contract, of the same title, (F33615-72-C-1449) are reported in this paper. Data are presented concerning powder characterization, hot isostatic pressing (HIP) parametric studies, producing and forging HIP preforms, and the determination of many mechanical properties from forgings as well as as-HIP material. The results of the mechanical property testing are compared to those of the conventional cast and wrought approach as well as to each other. The economics of forging powder preforms, and of using the alloy in the as-consolidated (by HIP) are discussed as well as current problems.

DISCUSSION

The objective of this effort was to establish a manufacturing method for producing aerospace quality titanium parts by forging hot isostatically pressed (HIP) prealloyed titanium powder preforms.

To accomplish this objective, an ingot was ordered to ASM 4928 (Ti 6Al-4V) except the oxygen content was specified at 800 ppm (max.). The ingot was converted to the appropriate size for conversion to powders via the rotating electrode process (REP) of Nuclear Metals, Inc., via the hydride-dehydride (HDH) process by Numec, and by Timet (Division of TMCA). Hydrided (HYD), and crushed powder, per se, produced by Timet, was also evaluated. The results of the powder characterization indicated the spherical REP powders contained a greater fraction of coarser particles than did the HDH produced by either Numec and Timet, but contained a larger fraction of the finer mesh sizes than did the HYD produced by Timet. The physically apparent differences among these powders is indicated in Fig. 1. The powder particle size and shape are believed to be the major determining factors which resulted in the differences in the vibrated theoretical densities (TD): REP - 65% to 66%, HDH - 51% to 52%, and HYD - about 54%. The results of previous efforts indicated a high (~60% TD) tap density is required to prevent the metal container, used in this effort, from wrinkling in the autoclave at temperature and pressure. A reproducible vibrated density is necessary since this characteristic determines the amount of powder required to fill the container for consolidation. Decreased vibrated theoretical densities are tolerable with other container systems, but it must be reproducible from lot to lot.

The chemistry of the billet and the powders (Table 1) indicated the REP did not appreciably alter the chemistry; the Numec HDH shows about a 200 ppm increase in oxygen content; and the Timet HYD indicated about an 800 ppm increase in oxygen as well as a minor amount of residual chlorine inherent in its process at that time.

TABLE 1

MAJOR CHEMISTRY - WEIGHT PERCENT

	Al	V	Cl	C	O	H	N
Billet (Supplied by GE)	6.4	4.2	-	0.022	0.06	0.01	0.01
Nuclear Metals, Inc. (REP)	6.3	4.2	Tr	0.014	0.06	0.007	0.004
Numec (HDH)	6.2	4.1	Tr	0.012	0.08	0.005	0.01
Timet (HYD)	6.1	4.0	0.018	0.014	0.14	ND	0.006

It was determined that the REP powder could be outgassed at room temperature with no detrimental effect, but all other types required hot outgassing to prevent contamination of the consolidated billet, presumably as a result of adsorbed water vapor on the high surface to volume angular powder particles. It was also determined the minor technical benefit obtained from using a selected screen fraction could not be economically justified. These conclusions are based on the results of density, microstructure, room temperature tensile properties, and K_Q, a measure of fracture toughness as determined by a slow-bend pre-cracked Charpy specimen fracture. (1)

A hot isostatic pressing (HIP) parametric study was performed, based on a statistical plan, to determine the effect of temperature - 1450F (790C), 1550F (845C) 1750F (955C) and 1860F (1015C); of pressure - 1000 psi (6.9 MPa), 4000 psi (27.6 MPa), 8000 psi (55.2 MPa), 10,000 psi (68.9 MPa), and 15,000 psi (103 MPa); and of time (0.5, 1,2, and 3 hours).

Fourteen different autoclave cycles were made in a unit similar to the one indicated in Figure 2. A preferred cycle of 1750F - 10,000 psi - 3 hours (955C-68.9 MPa - 3 hrs) was selected based on the results of theoretical density, room temperature tensile properties and K_Q. The REP powder was selected because of its superior properties, especially K_Q; its higher vibrated density; and its capability of being cold outgassed.

The preferred forging parameters were determined by the use of five (5) subscale flat forging preforms all consolidated in one autoclave cycle at the preferred HIP parameters (Figure 3). Included in the same autoclave cycle were two as-HIP parts. The forging of the five subscale preforms plus a section of the six inch (15.24 cm) billet, used as feed material to the various powder production processes, was accomplished on a highly instrumented press. Panel reductions of 20%, 35%, and 50% at 1750F (955C) in the alpha plus beta temperature field were imparted in one operation; whereas reductions of 35% and 50% were imparted via "conventional" beta processing in two operations, the first forging blow above the beta transus temperature, and the final 30% reduction being imparted in the alpha plus beta field in a separate operation. The wrought billet was forged 35% via the alpha plus beta forging procedure; i.e., one operation at a 1750F (955C) in the alpha plus beta temperature regime.

The room temperature tensile properties and fatigue strengths of the six forged conditions indicated the 50% panel reductions by both forging procedures was preferred. A preform was designed to be forged in the finish dies to produce an actual J79 first stage compressor disk. These preforms were machined for two reasons (1) the forgeability of an as-HIP surface was unknown; and (2) the capability of the preform source to produce preforms to tight dimensional tolerances was unknown (up to this time dimensional accuracy had not been a requirement). To obtain material in the as-HIP condition for a side by side comparison with the forged preforms, two parts of different configurations were consolidated in the same autoclave cycle as the forging preforms. The two configurations are shown in Figure 4.

The forging sub-contractor forged two powder preforms in one blow in the alpha plus beta temperature field, and two powder preforms via "conventional" beta forging practice, imparting a total of approximately 50% panel reduction in all cases. The forgeability was excellent, although probably influenced by the excellent surface finish and weight control (Figure 5). All six parts, four forged and two as-HIP, were non-destructively tested (NDT) via fluorescent penetrant inspection, X-Ray and ultrasonics. All passed the current GE standard, although the ultrasonics did indicate some marginal areas, which will be discussed later.

The three conditions were subjected to extensive laboratory mechanical property testing, consisting of room and 600F (315C) tensile properties, creep, stress rupture, notch-time-fracture, high cycle fatigue (smooth and notched, $K_t = 4.0$) low cycle fatigue, fracture toughness, crack growth rate, and Charpy impact. In all testing modes all properties met the AMS 4928 requirement, except the strength of the as-HIP material was 2-3 KSI (13.8 - 27.6 kPa) low, a result of the unusually low oxygen content of this material (600 ppm vs. the normal 1600 to 1800 ppm) and the lack of residual warm work from forging. It is anticipated that higher oxygen material would increase the strength, with only minor effects on the other mechanical properties tested.

The technical feasibility of using as-HIP material was proven in this effort. The one problem currently associated with this technology is the presence of inclusions, primarily tungsten, as shown in Figure 6a. The source of this type of inclusion is the non-consumable tungsten electrode used in the REP process. (A current AFML funded contract with NMI is directed toward exclusion of all material other than titanium from the REP process). Although these inclusions were present, as indicated by X-Ray evaluation of failed tensile bars, no failure of any test throughout the entire contract effort could be attributed to an inclusion of any kind. The marginal areas noted during the ultrasonic NDT were probably inclusions; however, this was not proven. The inclusion shown in 6b was identified as pure chromium, the source of which has not been identified. It is shown to indicate the potential problem of cross contamination in any powder metallurgy process regardless of alloy. This potential problem may only be alleviated by extreme care in all phases of powder making and handling, rigidly controlled by excellent quality control procedures.

A rather careful economic analysis indicated about a 5 to 15% cost reduction potential through the use of the forging preform technology. The larger portion of this cost savings comes from somewhat decreased material input (and consequently less final machining) and from the reduced number of forging operations. The fact that forging remains a necessity decreases the cost reduction potential.

A much greater cost reduction may be realized through the use of as-HIP to near-net-shape (NNS) technology. An in-depth study of three rotating components indicated cost reductions of 30 to 40% should be achievable. The hot isostatic pressing to NNS technology is being developed under an AFML sponsored contract scheduled for completion late in 1976

or early 1977. With the NNS technology coming to fruition in the same time frame as high quality Ti alloy powders the entire technology should be "ready to fly".

ACKNOWLEDGEMENTS

The success of this program is attributed to the excellent cooperation received from Battelle Columbus Laboratories, especially Mr. Hugh Hanes; from Wyman-Gordon, especially Mr. Oscar St. Thomas; and Mr. Larry Clark, AFML Program Manager, whose assistance throughout the conduct of the program was invaluable.

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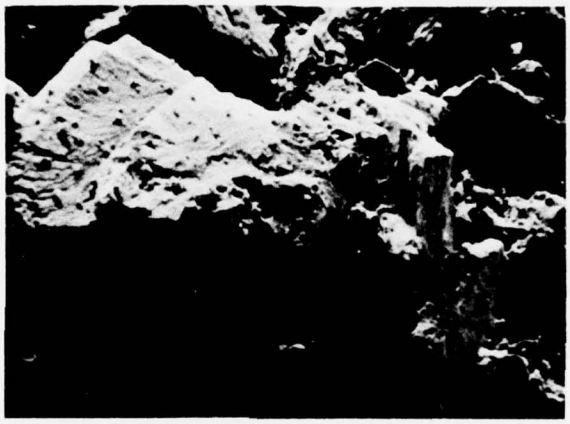


As-Received Timet Hydride (100X)

As-Received Numec Hydride-Dehydride (100X)

As-Received Nuclear Metals Inc. REP (100X)

FIGURE 1. SCANNING ELECTRON MICROSCOPE (SEM) PHOTOS OF THREE TYPES OF Ti-6Al-4V POWDER

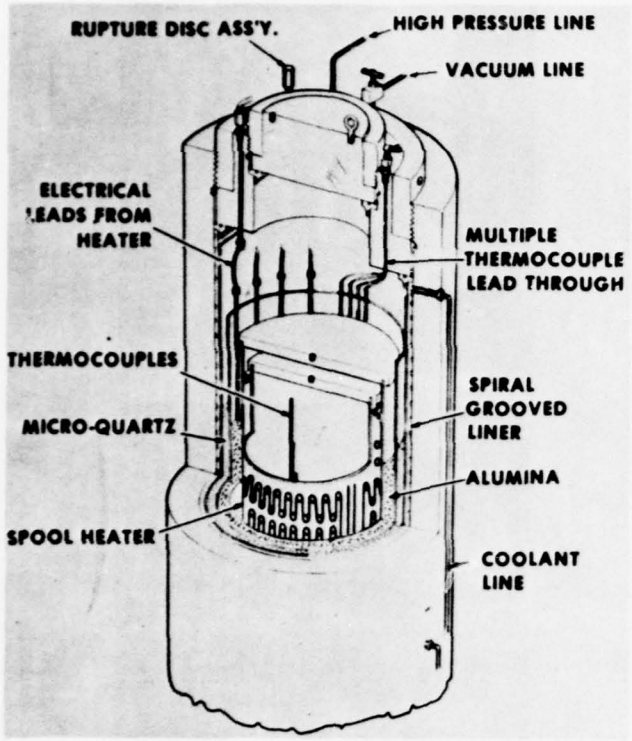
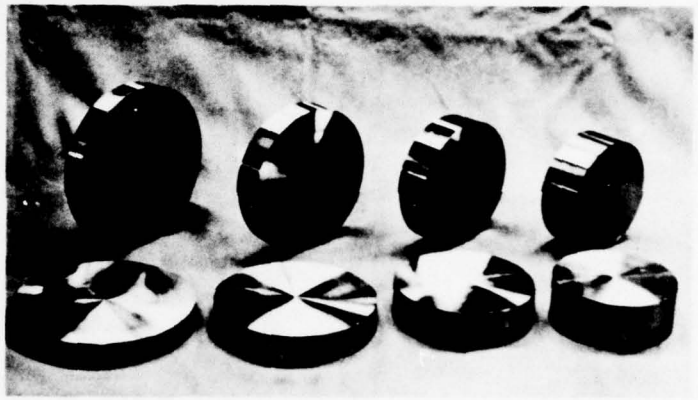


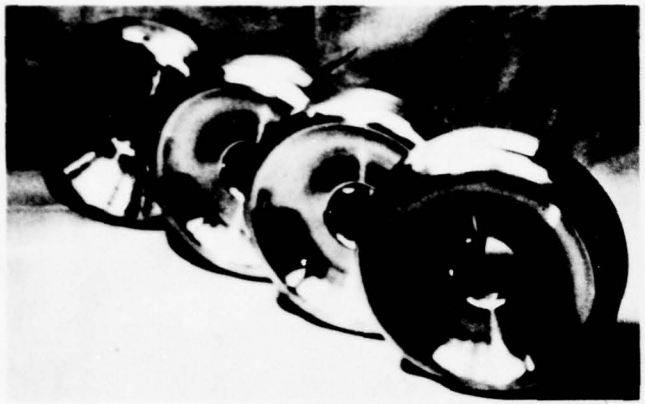
FIGURE 2. SCHEMATIC OF AUTOCLAVE

As-HIP - Prior to Decanning

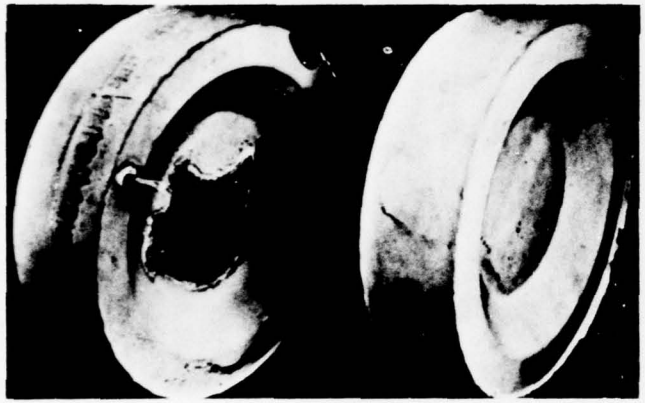


Decanned and Machined

FIGURE 3. SUBSCALE FORGING PREFORMS

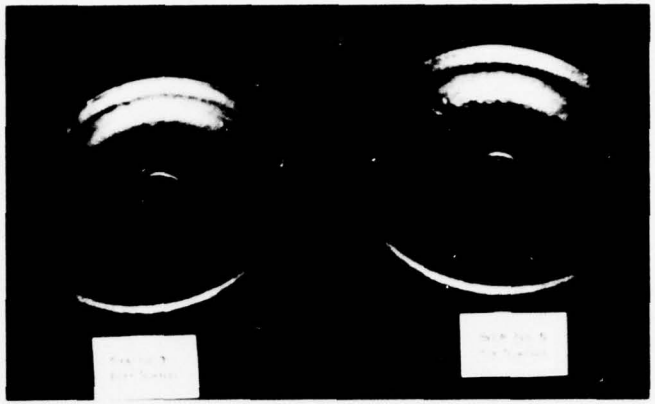


Forging Preforms - HIP Plus Machined

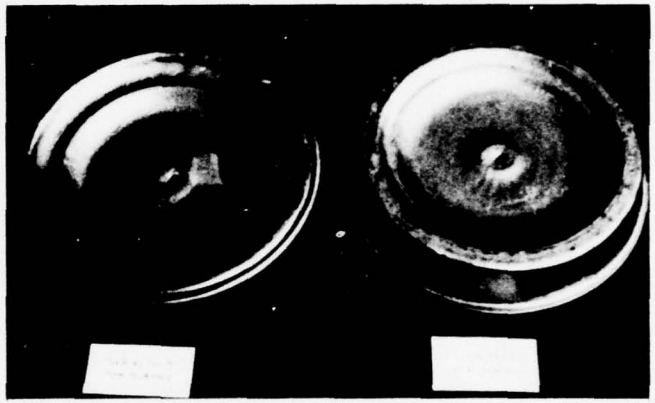


As-HIP Plus Chemically Decanned Disks

FIGURE 4. FORGING PREFORMS AND AS-HIP DISKS



Beta Forged - After First Operation

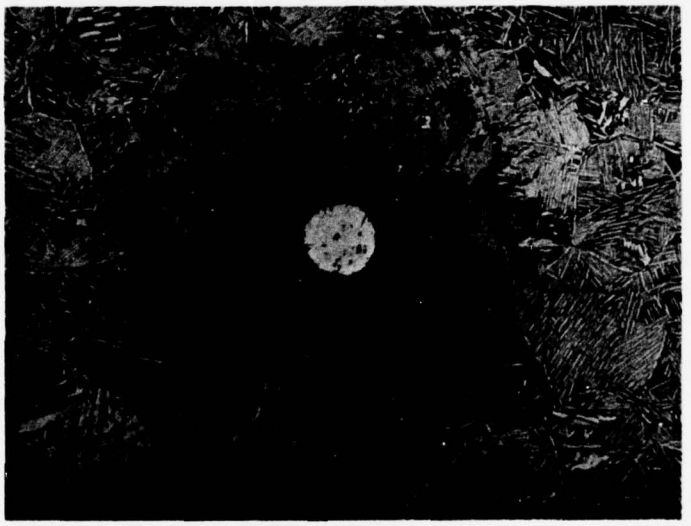


Beta Plus Alpha-Beta Forge

Alpha-Beta Forge

FIGURE 5. POWDER PREFORMS FORGED TO J79 DISK CONFIGURATION VIA TWO FORGING SCHEDULES

Tungsten Inclusion in As-HIP Disk



Pure Chromium Inclusion in Beta Processed Disk

FIGURE 6. INCLUSIONS LOCATED VIA RADIOGRAPHY ISOLATED BY METALLOGRAPHY AND IDENTIFIED BY MICROPROBE

WELDABILITY OF HOT ISOSTATICALLY PRESSED PREALLOYED TITANIUM 6Al-4V POWDERS

by

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I. INTRODUCTION

A recently completed study by Peebles,[1] summary results of which are presented in this report (Short Contribution Number 9, Session IV), showed that hot isostatic pressing (HIP) of rotating electrode (REP) prealloyed Titanium 6Al-4V powder produced fully dense forging preforms. A current study by Fleck[2] is attempting to demonstrate the capability of producing near net parts for use in the as-HIP condition without secondary working operations and with total cost reductions of up to 50%. It is therefore desirable to determine as many applications as possible for applying this developing technology. Since many airframe and engine parts require welding, the powder metallurgy product must be weldable if useage is to be maximized. Although some preliminary work has been done to determine the weldability of consolidated titanium powder, no systematic study has been carried out. Therefore, this study was undertaken to gain a better understanding of the weld characteristics of the as-HIP product in anticipation of future requirements.

Two prealloyed Ti 6Al-4V powder shapes were investigated: REP spherical powder (Nuclear Metals, Inc.) and H/DH irregular powder (Numec). Both types were evaluated after 3 different time-temperature-pressure HIP combinations. In addition, a fourth HIP cycle above the beta transus was conducted with spherical powder only. Welding was conducted on as-compacted material using the bead-on-plate gas tungsten arc technique with full penetration and constant weld parameters. Weldments were evaluated by bend, tensile, and toughness testing in conjunction with radiographic and metallographic techniques.

II. EXPERIMENTAL PROCEDURE

A. Material

All starting material was from the same heat of Ti 6Al-4V alloy purchased to AMS 4928 except that a low oxygen content (800 ppm maximum) was specified to allow for potential pickup during powder production and handling. The chemistries, mechanical properties and microstructure of the starting materials, full characterization of the starting powders, and canning, outgassing and compaction techniques used are given in the report by Peebles.[1] Seven compacts from Phase I of the Peebles study were selected for weldability evaluation to cover a range of compaction parameters including a cycle known to yield less than fully dense compacts. The HIP parameters used for both powder types are shown in Table I. In order to perform weldability tests, the as received compacts were decanned and machined into flats. Figure 1 shows a typical decanned compact and the final flats obtained.

B. Welding

Specimens were welded in the as-HIP condition. All specimens were pickled in a HNO_3-HF acid solution prior to welding to remove any surface contamination. Full penetration bead on plate gas tungsten arc welds were made using 10.5V dcsp, 190 amp., and 5.5 ipm travel speed (21.8 Kj/in. energy input). Argon was used for the shielding gas with the torch operated at 15 cfh, trailing 30 cfh and backing 15 cfh.

C. Testing

Tensile, face bend, and slow bend Charpy testing along with microhardness measurements were used to characterize the mechanical behavior of the welded HIP powder product. All tests were conducted in laboratory air at room temperature. The face bend specimens (approximately 7 in. long, 1.5 in. wide and 0.10 in. thick) were ground, polished, and pickled to remove any surface contamination occurring after the welding operation. These specimens were bent in the longitudinal direction with the face of the weld in tension. Guided bend test procedures were followed with the die radii between 2 inches and 3/4 inches. The radius of the die used prior to failure was recorded and the elongation (in one inch) measured for each specimen. The longitudinal weld guided bend test was used in order to determine the ductility of the welded samples. This test is preferred since all portions of the weld zone (FZ, HAZ, Base) are strained equally, therefore giving useful ductility data.

Room temperature tensile tests transverse to the weld bead were conducted with a standard sheet specimen geometry with a 1 inch gauge length. Slow bend fracture toughness determinations were made in the heat affected zone and fusion zone, using precracked Charpy V-notch specimens of .125 inch thickness, in conjunction with a Manlabs Model 5B-750 tester at a crosshead speed of 0.1 in./minute. The load-deflection curves recorded allowed the determination of K_Q values using the methods recommended in ASTM Standard T399 for 3-point bend specimens and rising load conditions.

III. RESULTS

The results of transverse tensile tests on as-welded REP and H/DH powder consolidated under different compaction parameters are presented in Table I. In all cases the specimens failed in the base metal with properties similar to typical wrought Ti-6Al-4V weldments. Longitudinal bend tests, Table II, showed that the fusion zone was the initiation site for fracture. As the bending strain was increased the cracks grew outward through the HAZ and base metal. The slow bend precracked fracture toughness results are also presented in Table I. It can be seen that the fusion zone and HAZ both exhibited superior toughness to the alpha-beta HIP base metal and inferior values to the beta HIP compacts. Although a direct quantitative

comparison of these data cannot be made since the base metal specimens were of a different thickness (standard Charpy size of 0.394 inches as opposed to 0.125 inches), the trend that was exhibited has been previously reported[3] for alpha-beta processed versus beta processed material.

In most cases the welding of HIP specimens presented no problems when compared to Ti-6Al-4V wrought product. Figures 2 and 3 are montages of the microstructures of weldments of REP and H/DH compacted powders, respectively. As would be expected the fusion and near heat affected zones of each are similar; however large differences did occur in the middle and far HAZ, as will be discussed later.

Although there was no difference in the fusion zone microstructures of the two powder types, there was considerable difference in the tendency for void formation. Porosity was encountered in all H/DH welds although compacts H/DH6 and H/DH7 were determined to be fully dense. The occurrence of this porosity was not the result of poor weld process control, since no voids were evident in the starting and stopping wrought Ti-6Al-4V tabs. Likewise, no porosity was encountered in fully dense REP compacts.

IV. DISCUSSION

It became apparent during this investigation that density determinations of compacted powders alone were insufficient to describe the material's weldability. Fully dense material, as determined volumetrically can still have porosity or lack of bonding at the individual particle interfaces in the base material. Although this type of defect was very fine (and not observable optically), the stresses and heat supplied during welding were sufficient for voids of large diameter to form, presumably by coalescence, in the fusion zone. In the instances where porosity was more extensive, as in the case of material measured volumetrically as 99.9% dense, it made the material unweldable due to outgassing in the fusion zone with the resultant lack of penetration. This is clearly shown on the welds of compact H/DH5, a montage of which is shown in Figure 4. A quantitative image analysis (using a Quantimet Model 720) of the base metal region of Figure 4 showed 2.7 $\pm$ .5% porosity. During welding, the arc became unstable and, as shown in Figure 5 erratic penetration resulted in a very poor underbead.

In order to rationalize these results, it is necessary to discuss the relative ease of compaction of these two different types of powders. Compacted under the same conditions R1 and H/DH5 achieved different densities as previously noted. The fact that REP can achieve vibrated packing densities of greater than 65% of the theoretical density and that H/DH powder with 51% vibrated density, required cold isostatic compaction prior to HIP to achieve a comparable density is indicative of the effect of shape and, to a lesser extent, particle size distribution on achievable packing.

The H/DH powder typically has a B.E.T. surface area of .060 m^2/g as opposed to a value of .009 m^2/g for the REP powder.[4] This six fold difference in surface area is particularly significant in titanium alloys where the tendency for contamination by gas adsorption/absorption readily occurs. These differences in particle shape and specific surface area affect compaction and later the tendency for void formation during welding. The base microstructures of both types of powder compacts from the 1700F/8000psi/3hr HIP cycle are shown in Figure 6. The hydride-dehydride compact, 6a, shows irregular boundaries at the particle interfaces with evidence of recrystallized equiaxed alpha. In addition, stored work is evident as seen by the bending of the acicular platelets showing that the powder has mechanically deformed as it joined together during cold compaction or early in the HIP cycle. This is less evident in the R3 compact, 6b, where an apparently strain free acicular structure with very few prior particle interfaces is seen.

Linear porosity in the fusion zone adjacent to the heat affected zone was always observed in compacts of H/DH powder. In order to determine that this porosity was not a result of residual hydrogen, a special blank was welded and then dehydrided in a vacuum furnace at 1500F for 6 hours. A second weld was made on the same plate and it can be seen in the radiograph (Figure 7) that the porosity was still evident. It is unlikely that hydrogen is the cause of this porosity. In fact, chemical analysis showed that the second pass was made when the total hydrogen content of the plate was only 27 parts per million. The origin of the linear porosity in the weld zone is believed due to adsorbed gases, other than hydrogen, on these very high specific surfaces. The linear porosity encountered in H/DH compacts did not appreciably affect the properties measured in this investigation; however, it is anticipated that property degradation would have occurred if fatigue tests had been performed.

The fracture toughness in the heat affected zone and fusion zones in all but the beta processed, R4, compact showed higher values than the base material, as mentioned earlier. This was not an effect of the powder but a result of the change in microstructure from that of equiaxed alpha particles to a more acicular structure. In the R4 compact, since the entire material was beta processed and exhibited an acicular structure, the fracture toughness of the heat affected and fusion zones in fact was slightly lower because the rapid cooling rates in these zones did not allow the platelets to grow to sufficient thickness for optimum toughness.[5]

V. CONCLUSIONS

This study has shown that rotating electrode process (REP) powder consolidated by hot isostatic pressing (HIP) is weldable; whereas, hydride-dehydride (H/DH) process powder compacts consistently exhibited linear porosity in the fusion zone and hence may not be acceptable for some applications. Of the two powder types evaluated, the REP powder appears less sensitive to the requirements for full density. The porosity encountered in the H/DH powder compacts, as exhibited before and after welding, is attributed to the high specific surface of the powder and therefore the greater amount of adsorbed or absorbed gases and/or other contaminants which persist through the HIP cycle. Both powder types have acceptable as-welded tensile, bend, and toughness properties after HIP.

This study suggests that a simple bead on plate type weldability test may be a useful technique for screening as consolidated powder compacts prior to further processing.

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5. Greenfield, M.A. and Margolin, H., "The Interrelationship of Fracture Toughness and Microstructure in a Titanium Alloy", Met. Trans., Vol. 2, March, 1971. pp 841-847.

TABLE I AS WELDED PROPERTIES OF HOT ISOSTATICALLY PRESSED Ti-6Al-4V

Compact* Number	HIP Parameters	Density	Yield Strength ksi	Ultimate Strength ksi	Reduction of Area %	Elongation %	Fracture Toughness Base ksi√in	HAZ ksi√in	Fusion Zone ksi√in
R1	1550°F/10ksi/2hrs	>99.9	124	133	33	10	42	54	54
H/DH5		99.9	NOT WELDABLE						
R2	1750°F/10ksi/1hr	100	113	123	36	11	51	56	55
H/DH6		100	119	132	27	8	48	54	55
R3	1750°F/8ksi/3hrs	100	111	124	38	11	58	53	55
H/DH7		100	120	131	26	11	46	55	58
R4	1750°F/10ksi/1hr plus 1860°F/10ksi/1/2hr	100	111	122	27	10	63	54	56
Wrought	---------	100	125	133	30	8	51	56	57

* R - Rotating Electrode Powder

H/DH - Hydride/Dehydride Powder

All specimens failed in the base material

TABLE II AS WELDED LONGITUDINAL BEND PROPERTIES

	Radius of Die at Onset of Cracking	%Elongation in 1 inch	Crack Initiation Site
R1	7.5T	6.7	FZ
H/DH5	NOT WELDABLE		
R2	7.5T	7.1	FZ
H/DH6	7.5T	8.6	FZ
R3	10T	5.1	FZ
H/DH7	10T	6.3	FZ
R4	7.5T	7.4	FZ
Wrought	7.5T	9.4	FZ

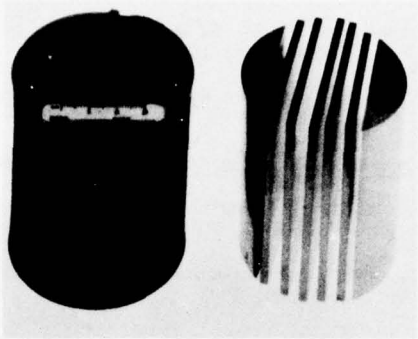


Figure 1. Typical Compact and Final Flats for Evaluation.

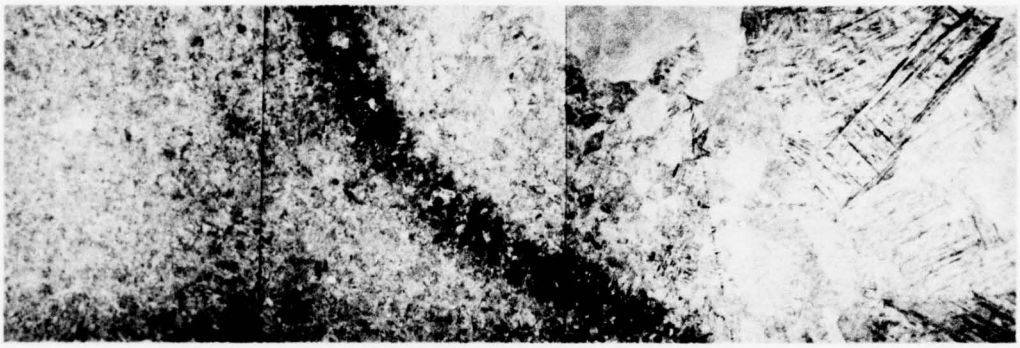


Figure 2. Montage of Weld in R3.

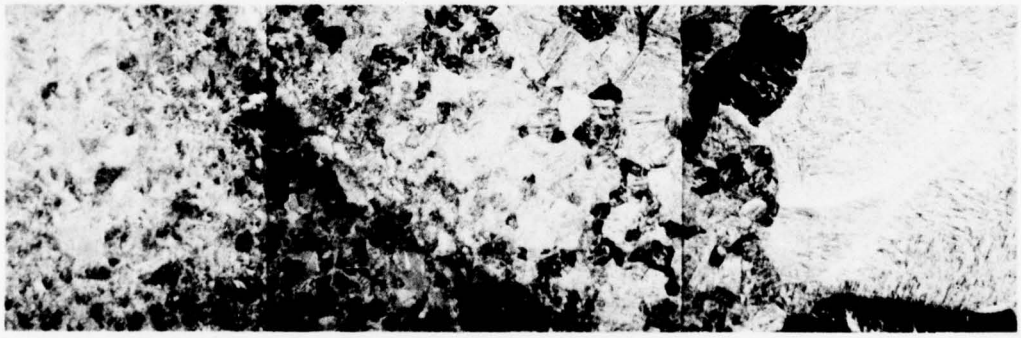


Figure 3. Montage of Weld in H/DH7.

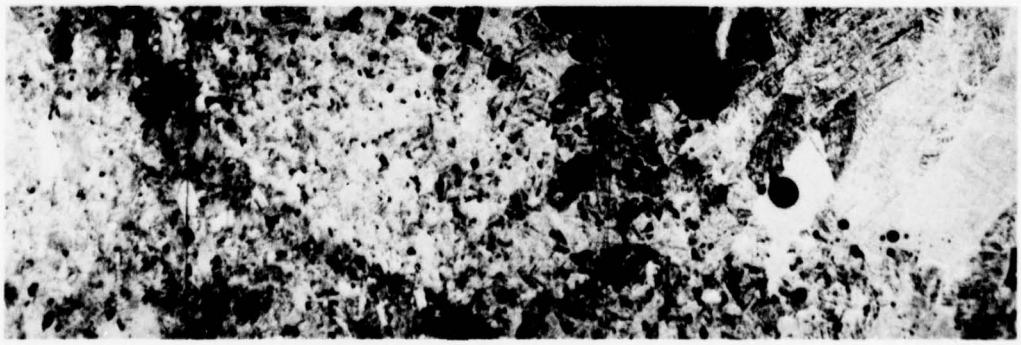


Figure 4. Montage of Weld in H/DH5.

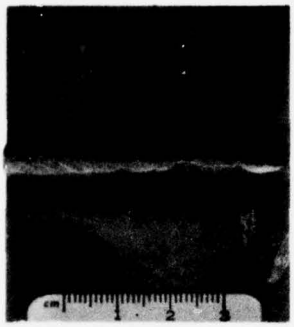
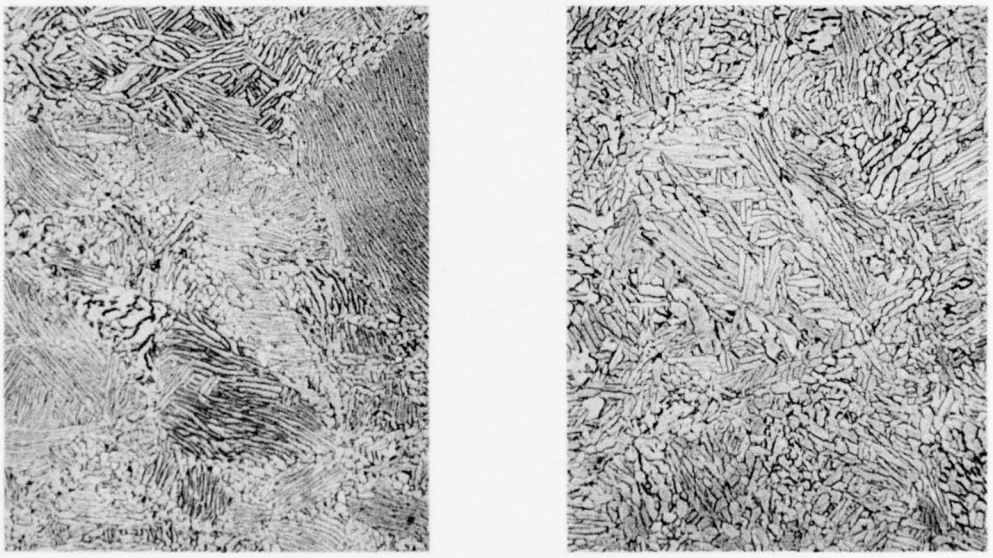


Figure 5. Erratic Weld Bead Penetration in H/DH5.



A B

Figure 6. Base Microstructures of Powder Compacts; A. H/DH and B. REP.

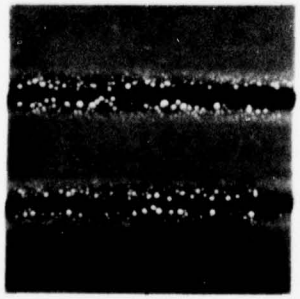


Figure 7. Radiography of 2 Pass Weld in an H/DH Compact (2nd pass lower).

NEAR-NET POWDER METALLURGY AIRFRAME STRUCTURES

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SUMMARY

Interest in Powder Metallurgy (P/M) for titanium airframe parts has been directed toward reduction or minimization of the acquisition cost of titanium alloy parts such as those produced from machined forgings or plate. As a result, efforts are being directed primarily toward the use of P/M techniques to produce near-net shapes. Most airframe parts with large potential cost savings have deep pockets and cannot be easily densified to close to final dimension using conventional powder metallurgy processes (e.g. cold press-sinter, hot pressing). This paper deals primarily with results of studies related to this challenge and the following approaches:

- Cold isostatic pressing (CIP) and sintering to produce high-density preforms for subsequent hot forging to full-density, near-net shapes
- Hot-pressing (HP) of shapes
- Hot isostatic pressing (HIP) to full-density, near-net shapes in a one-step operation.

The primary titanium alloys investigated in such work have been Ti-6Al-4V and Ti-6Al-6V-2Sn. Advantages, disadvantages, technological and economic considerations are summarized for each approach and potential future airframe applications are presented.

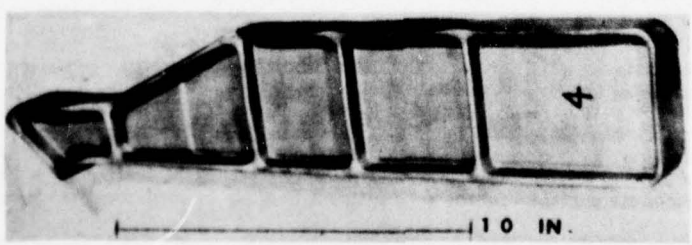
INTRODUCTION

The high cost of manufacturing titanium hardware using conventional techniques such as forging and machining has been a definite deterrent to the use of titanium alloys in the aerospace industry except where absolutely required. This high cost is due primarily to the limitations on producing close-to-final-shape parts. For example, it is often necessary to buy five to ten times the amount of titanium alloy that actually is used in the aircraft. Most of the alloy is machined into scrap. The ratio of the original forging weight to the final machined weight of the finished part is known as the buy-to-fly ratio (BFR). This problem has manifested itself in recent years on major aircraft such as the F-14, F-15 and B-1 programs. The materials utilization, therefore, is poor and in the present economy it becomes extremely important for aircraft manufacturers to seek methods of manufacture of components which can maintain economy with quality. For titanium to be competitive in many future applications, it is absolutely necessary that costs be cut effectively.

Since the late sixties and early seventies, attempts have been made to employ various powder metallurgy techniques to produce near-net shapes in titanium alloy to effect such potential savings. The following P/M techniques have been considered critically to attempt to produce typical, deep-pocketed airframe parts such as those shown in Figure 1:

- Cold pressing (CP) or cold isostatic pressing (CIP) and sintering
- CIP, sinter and forge or extrude
- CIP, sinter and hot isostatic pressing (HIP)
- Hot Pressing (HP)
- Direct HIP of billets, preforms or shapes

Listed below are the sequential steps involved in manufacturing typical forgings by conventional forging techniques and those involved in producing close-tolerance forgings utilizing elemental powder metallurgical preforms, as an example of an essentially one-step operation.



ULTIMATE STRENGTH (F_{tu}) – 149 KSI
YIELD STRENGTH (F_{ty}) – 144 KSI
ELONGATION – 11%

Fig. 1 Tensile Properties of Close-Tolerance, Low Draft-Angle Experimental Part Isothermally Forged From Ti-6Al-4V Powder Preforms

Conventional Forging Sequence	P.M. Forging Sequence
1. Cut stock	1. Isostatic Consolidation (PVC molds)
2. Upset (Die No. 1)	2. Vacuum Sinter
3. Block (Die No. 2)	3. Forging (Die No. 1)
4. Trim	4. Hand Finish
5. Semi-Finish (Die No. 3)	
6. Trim	
7. Finish Form (Die No. 4)	
8. Warm Trim	
9. Hand Finish	

Note that the P/M forging uses only one forging operation. The preform used could alternately be fabricated by HIP or in some cases by HP.

In the following sections, results are briefly summarized for CIP processes, hot pressing and direct HIP processes. A concluding section summarizes results and possible future trends. The prime factors considered throughout these evaluations were:

- As-compacted properties
- Near-net shape production capability
- Cost

CIP PROCESSES

In the sixties it was found that CIP plus sintering of Ti-6Al-4V titanium alloys could produce from 90 to 98% dense compacts having tensile strengths above 110 ksi with decent elongations at the higher densities. Since these properties were encouraging, but not adequate, it was then considered possible to reach specification minimum requirements by using powder compacts for preforms and then forging or extruding to obtain the necessary properties. In the following paragraphs, results obtained with forgings are discussed but extrusions are not included for one primary reason, namely, the investigation was concerned with processes that can produce net components at minimum cost.

Two approaches have been investigated in producing CIP plus sintered preforms for forgings mainly based on the type of powders employed:

- Pre-alloyed powders
- Soft blended elemental with master alloy powders

The former has the advantage that the alloy is homogeneous and generally the problem of segregation is eliminated. In both processes, it appeared that use of hydride-dehydride (HDH) powders might add to cost-effectiveness by using scrap as feed material, but to date, high-quality powders have not been produced from scrap feed material.

It is to be noted that in these studies only powders made from sponge or by the HDH process were utilized. Spherical powders such as those produced by the rotating electrode process (REP) cannot be cold-compacted effectively and, therefore, were considered only in later hot-pressing and HIP studies.

The CIP consolidation process utilizes preshaped flexible molds which are filled with powders, evacuated, and subjected to the required pressure in either liquid or gaseous medium. In the course of the consolidation operation, pressure is applied uniformly over the surface of the mold resulting in a uniform "green" density distribution. The isostatic consolidation process permits manufacturing of preforms close to the required configuration, thereby increasing freedom in design of complex forgings and minimizing material waste. Hydraulic isostatic presses equipped with compression chambers up to 16 inches in diameter and 6 feet in length are currently in commercial use. Installation of larger chambers is being contemplated by many industrial concerns.

Controlled vacuum sintering is required for parts that have been cold-compacted. Sintering takes place between 2250°F to 2450°F usually in two- to four-hour cycles.

In using pre-alloyed powders (Ref. 1), it was found that as-sintered densities normally ran between 85 to 90% of theoretical and it was necessary to develop multiple steps to fabricate a component. The initial operation for sealing surface porosity was critical to ultimately producing parts of acceptable quality. This initial forging step and subsequent reconditioning of the surface seemed to be difficult to apply to deep-pocketed parts.

Therefore, an investigation commenced (Ref. 2) which determined the feasibility of manufacturing close-tolerance, low-draft-angle Ti-6Al-4V forgings from preforms fabricated from elemental powders. In the course of this program, forging processes and powder selection studies were initially performed prior to producing the part shown in Figure 1 for subsequent mechanical property evaluation.

In the course of the work, five preform vendors were utilized. All employed powders produced from sponge

except one, which used HDH powders. Three forging processes were also evaluated, namely, conventional die forging (800°F), high-energy-rate forging (HERF-400°F) and isothermal forging (1650/1750°F).

All preforms in the forging process selection studies were manufactured by isostatic pressing of elemental powders at 60 ksi and sintering at 2250°F in vacuum at 10^{-4} torr. The characteristics and properties of the powders and preforms utilized are summarized in Tables I and II. This powder had a relatively high NaCl content (0.25 percent) but was used, since some data indicated that residual chloride could be removed by modification of the sintering process performed by the selected vendor. Electron-beam welding and chemical studies performed later did not confirm this information (Figure 2). Nevertheless, the work confirmed the fact that isothermal forging gave 100% dense parts. The other processes each indicated residual porosity even after reductions as high as 45%. Tensile evaluations of isothermally forged parts that had been stress relieved at 1300°F for two hours showed that tensile and yield strengths exceeded requirements of Mil-T-9047, but elongation values were low, around 5 percent. However, later work on isothermal forging HDH powders in powder selection studies showed that the specification minimum elongations of 10% could be exceeded consistently. The preform variables used in these studies are presented in Table III. As indicated, only the forgings produced from elemental HDH titanium blended with Al-V master alloy powders exceeded specification requirements. Complete densification could not be obtained in forgings prepared from powder blends which were prepared from Na- and Mg-reduced sponge, even though proprietary processes were employed to improve powder characteristics.

It is noted that complete densification was also obtained when pre-alloyed HDH powders were forged under identical conditions. However, properties were drastically reduced. It was found in metallographic evaluations that an intergranular network (probably oxide) was the cause of nil ductility and low strength. These preforms had a density of less than 90% (Table III) and contained interlocking porosity. This indicates on an exaggerated scale what can happen when pre-alloyed powders are used for CIP + sintered preforms and surface porosity is not properly sealed prior to forging.

Forgings prepared from blends containing titanium powders processed by vendors' proprietary purification techniques had elongation values in the range of 7 to 9%.

Forgings prepared from blends which contained titanium powder manufactured from the sponge exhibited low elongation properties and contained extensive regions of microstructural inhomogeneity. Constant-amplitude, axial tension-tension (R = 0.1) fatigue tests indicated the endurance limit (10^7 cycles) of forgings prepared from hydride/dehydride titanium and Al-V master alloy powders to be in the range of 55 to 75 ksi for unnotched specimens. It is believed that these results could be improved by additional thermal treatment. Notched fatigue specimens ($K_t = 4$) indicated a 10^7 cycle endurance limit of approximately 23 ksi, which compares favorably with conventional material.

Coated specimens subjected to 1750°F/4 hr treatments in an air furnace revealed no indications of thermally induced porosity for the elemental powder approach whereas the ones made from pre-alloyed powders showed porosity.

Table I Characteristics of Elemental Powders Utilized in the Course of Preliminary Studies

ELEMENT	COMPOSITION, WT %		
	ELEMENTAL Ti POWDER	Al-V MASTER ALLOY	SINTERED PREFORMS
C	0.01	N.D.	0.02
N	0.013	N.D.	0.039
O	0.10	0.07	0.18
H	0.004	N.D.	0.002
Na	0.099	N.D.	N.D.
Cl	0.15	N.D.	N.D.
Al	–	56.59	6.42
V	–	41.67	4.15

NOTE: N.D. = NOT DETERMINED.

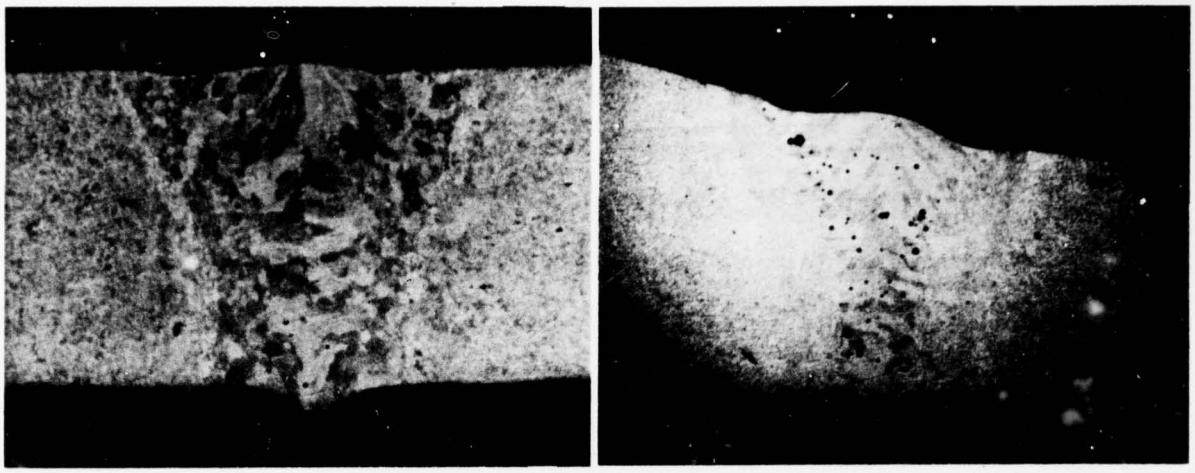
SCREEN ANALYSIS (TYPICAL)

MESH	PERCENT
–100 + 200	42
–200 + 325	32
–325	26

Table II Some Physical and Mechanical Properties of Sintered Preforms

PROPERTY	VALUE
DENSITY (% THEORETICAL)	94.6–95.1
TENSILE STRENGTH, F_{tu}, KSI	114–122
YIELD STRENGTH, F_{ty}, KSI	97–104
ELONGATION	3–7%
R.A. %	4.8–7.0

METALLOGRAPHY: NO EVIDENCE OF GROSS VOIDS, INCLUSIONS, OR INHOMOGENEITY.
RADIOGRAPHY: MEETS AMS 2635 FILM-DENSITY REQUIREMENTS.



a. Low Halide Content

b. Higher Halide Content

Fig. 2 Electron-Beam Welds in P/M Pieces

Table III Properties-Isothermal Forgeability Study

	VENDOR					
	A		B		C	D
POWDER TYPE	E + MA	PREA	E + MA	E + MA	E + MA	E + MA
Ti POWDER MFG.	H/D	H/D	PROP.	EXP. PROP.	CR + P (PROP.)	MG-RED
COMPACTION	ISO.	ISO.	ISO.	ISO.	ISO.	MECH.
COMPOSITION	STD.	STD.	ELI	STD.	ELI	STD.
NO. OF PREFORMS	4	2	6	1	6	6
DENSITY, % THEOR.	98 MIN	<90	95 MIN	98.5 MIN	94 MIN	N.D.
FORGED DENSITY % THEOR.	100	100 (1)	(3)	(3)	(3)	(3) (2)
F_{tu}, KSI	152.4	71.2		140.4	145.3	125.6
	150.2	82.3		140.0	144.8	115.0
F_{ty}, KSI	151.7	(4)		138.4	138.2	124
	148.0	(4)		139.0	138.8	(4)
ELONG. % (1" GAGE LENGTH)	14	NIL		9	7	1
	13	1		8	8	NIL

MIL-T-9047 REQUIREMENTS: F_{tu} = 130 KSI MINIMUM.
F_{ty} = 120 KSI MINIMUM.
ELONG. = 10% MINIMUM.

NOTES: (1) INTERGRANULAR PHASE DETECTED.
(2) STRUCTURE INHOMOGENEITY.
(3) POROSITY.
(4) PREMATURE FAILURE.

EXPLANATION OF SYMBOLS:

E + MA = ELEMENTAL TITANIUM AND AL-V MASTER ALLOY.
PREA = PREALLOYED Ti-6Al-4V POWDER.
H/D = HYDRIDE/DEHYDRIDE.
PROP. = PROPRIETARY.
EXP. = EXPERIMENTAL.
CR + P = CHEMICAL REDUCTION AND PURIFICATION.
ISO. = ISOPRESSED.
N.D. = NOT DETERMINED.

The final task of the program to produce prototype bulkhead parts employed HDH elemental titanium blended with Al-V master alloy powder preforms. Isothermal forging took place above 1650°F following preheating to 1750°F prior to forging. Fully dense forgings were obtained except at the top of the ribs where the compressive forces were not adequate. The lack of work was confirmed by coarser grain sizes in these locations. One other problem was experienced with die-ejection pressures which resulted in deformed webs in the part. This appears to be related to proper lubrication otherwise draft angles may have to be increased. As a result of these investigations, it was concluded that further studies are required to optimize preform configurations and temperature controls in the forging operation to provide improved microstructural control and adequate lateral metal flow under compressive stresses in all parts of a complex forging. Much work is also required on

lubrication techniques and ways to lower part ejection pressures after forging. It was also surmised that a process such as HIP might be capable of producing 100% dense preforms, which should improve forgeability. This is discussed further in a subsequent section.

HOT PRESSING (HP)

This process utilizes hot pressing in a direct vacuum or an encapsulating method employing vacuum techniques. Convair Aerospace has reported results of their approach (patent pending) which employs the latter technique (Ref. 3).

Advantages claimed for the process include the following:

- Potential cost savings by reducing machining and affording material reductions of 70 to 80%.
- Dense titanium alloy parts with fine and uniform grain structure
- Tensile properties equivalent to wrought alloys
- Essentially a one-step process (some surface machining may be required though).
- No draft angles required in tools
- High-temperature alloy tools are reusable
- No precompacting required
- Relatively low pressures are adequate
- Parts with deep pockets, differing radii and wall thicknesses, tapered walls, bosses, etc., are possible.
- Parts can be nondestructively tested for porosity or impurities (advantage claimed over diffusion bonded parts).

The properties of HP parts at 99.5% density levels in Ti-6Al-4V are listed in Table IV and compared with CIP + sintered and CIP, sinter, forged properties. Four complex fittings, each nine inches square with 1 1/2-inch-deep pockets and different wall thicknesses and radii, were hot pressed by Convair. The parts were fine-grained, hardness $30R_c$ and tensile properties were equivalent to those of wrought Ti-6Al-4V alloy. Fracture toughness was equivalent to castings or wrought metal. Flexure fatigue life at 40% of flexure ultimate load exceeded 10^7 cycles, but tension-tension fatigue showed an endurance limit of only 50 ksi. No doubt this was caused by residual porosity.

This process is capable of producing some complex shapes but it suffers from the following limitations of die compaction techniques:

- Cannot compact parts with re-entrant angles
- Confined to pressing relatively simple shapes with moderate L/D ratios
- Densities attainable are only 99.5% of theoretical fatigue properties

Mainly because of the latter limitation, this process can only be used for secondary structures in the airframe industry. To develop full fatigue properties, it is necessary to forge to final shape. No data have been reported on forged, hot-pressed parts, however.

HOT ISOSTATIC PRESSING (HIP)

The HIP process consists, in essence, of encapsulating metallic powders in suitably shaped (shells) molds, evacuating and sealing the mold assembly, and positioning it in a high-temperature/pressure autoclave designed to contain gaseous media (Fig. 3). In the course of the HIP cycle, built-in heaters increase the temperature of

Table IV Comparison of HP, CIP + Sinter and CIP, Sinter + Forged Properties

PROPERTY	HP	CIP+SINTER	CIP, SINTER, FORGE
• DENSITY, % THEORETICAL	99.5	95/98	100
• TENSILE			
F_{tu}, KSI	135.1	110/125	148
F_{ty}, KSI	122.2	99/110	143
% ELONGATION (1")	14.2	7/12	11
E (10^6 PSI)	16.5	–	17.0
• FRACTURE TOUGHNESS, KSI$\sqrt{IN}$	60*	–	65
• CHARPY V-NOTCH			
IMPACT STRENGTH (FT-LBS)	16.5	–	–
• FATIGUE LIMIT, KSI, (10^7 CYC)			
$K_t = 1$	50	–	55.75
$K_t = 4$	–	–	22.5

*KQ

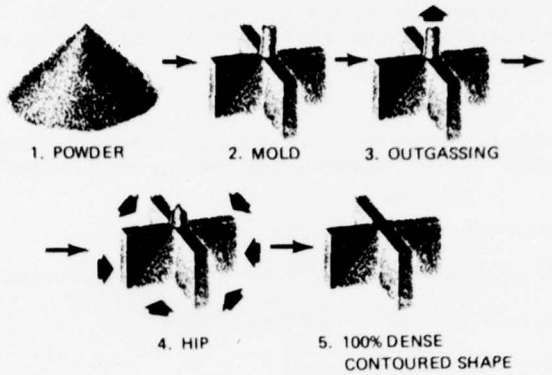


Fig. 3 Fundamentals of the HIP Process

the mold to its softening range and the applied pressure is transmitted through the walls of the mold to the enclosed charge. By the combined action of temperature and pressure the powder can be consolidated into 100% dense shapes of desired configuration in a one-step operation.

Compared with P/M processes discussed previously, HIP offers a number of unique advantages. Some of the following may ultimately become important for airframe applications:

- Process gives 100% dense configurations in one-step operation
- Highly alloyed powders can be used
- Fabrication of complex configurations with close tolerances is potentially feasible
- Original fine-grain microstructure can be retained
- Substantial cost savings can be realized through reductions in excess material and machining time
- Exploratory studies have shown that tensile properties of hot isostatically pressed parts meet forging specification requirements when proper parameters are used.

The above potential advantages of the process are based on preliminary investigations (Ref. 4) on pancake-type compacts and some initial work on making a 1.5-pound shape (Figure 4). Most of this work was accomplished using a process patented by Crucible Materials Research Center which utilizes ceramic molds. Another process patented by the Kelsey-Hayes Company which employs vitreous molds has been utilized to a limited extent.

One important aspect of this process is that the apparent density (tap density) of the powder is important and should be the maximum attainable for packing into molds. Spherical powders are preferred. For example, HDH powder does not have a high apparent density and was not used in these studies. Powders produced at Nuclear Metals Corp by the rotating electrode process (REP) are spherical and exhibit excellent packing characteristics. Most of the work we have done on HIP, therefore, has been with REP powders.

The component shown in Figure 4 was HIP-processed using the Crucible process and while feasibility is shown for near-net shapes further work is required to meet engineering drawing requirements. Some tradeoffs will most likely have to be effected before compliance is achieved.

In this work the importance of oxygen content of powder and processing parameters on tensile properties is noted from the results of various HIP cycles on Ti-6Al-4V powder as shown in Table V.

Figure 5 shows tension-tension fatigue results for material processed at HIP cycles significantly above and below the beta transus in comparison with annealed plate as a baseline. Fatigue limits above 70 ksi appear to be attainable, but further work is required on components to compare results directly with forged pieces.

Work on Ti-6Al-6V-2Sn in powders shows similar trends for tensile (Figure 6) and fatigue properties. The oxygen content in this case appears to have a more inportant bearing on the properties attainable for the processing times utilized.

The fracture toughness of HIP materials also appears to be acceptable (Table VI). These results generally are equivalent to recrystallized annealed titanium alloys.

In general, it can be concluded that the HIP process exhibits significant potential to continue work to determine its niche in producing net or near-net shapes. The most critical requirement found so far appears to be the fatigue properties attainable. Should future investigation show that forging is required, extensive work will have to be directed toward preform design studies coupled with optimization of the forging process itself for producing net shapes.

CONCLUSION

The major advantages and disadvantages of P/M techniques for producing airframe components have been presented throughout the foregoing discussion of various processes. The major incentives towards using P/M techniques for airframe components is predominantly economic for reasons explained in the introduction relative to cost of machined forgings and the effect of the BFR on usage of titanium. Some technological advantages may also be found but it is too early to define where these exist.

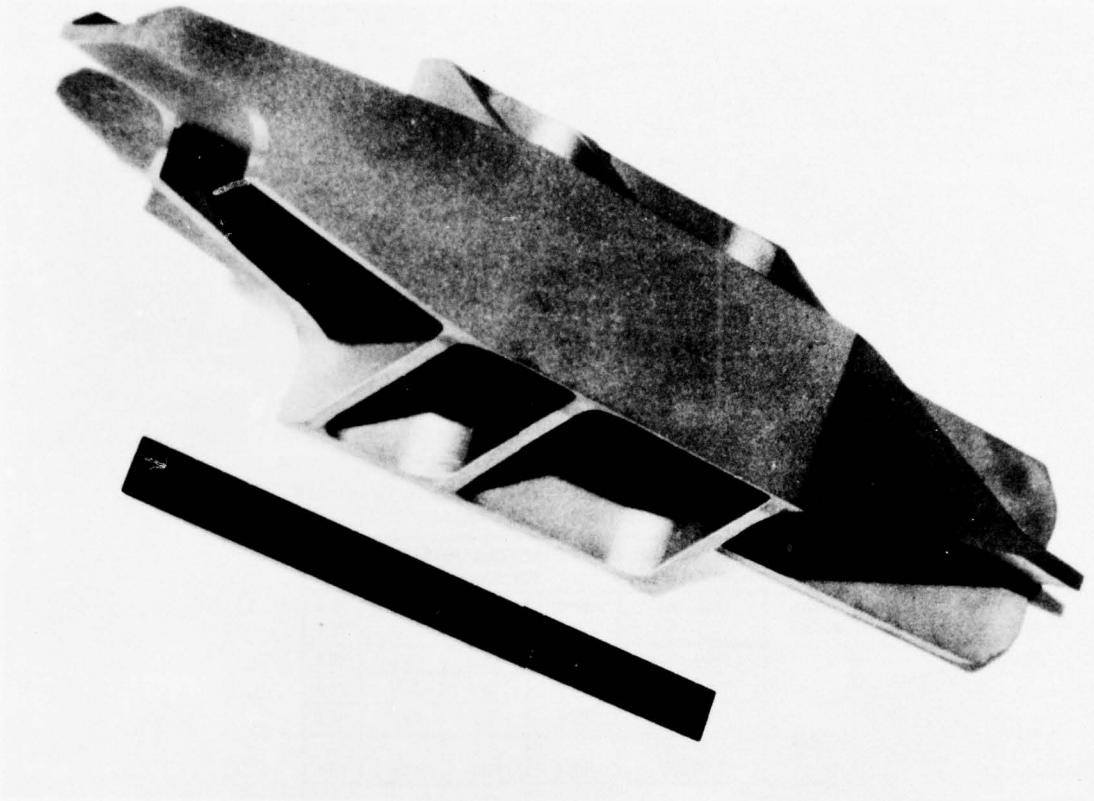


Fig. 4 Fuselage Brace Produced by HIP

Table V Effect of HIP Cycle and Oxygen Content on Tensile Properties (Ti-6Al-4V)

OXYGEN CONTENT, PPM	HIP CYCLE TEMP./PRESS./TIME °F/KSI/HR	F_{tu}, KSI	F_{ty}, KSI	ELONG, %	REDUC AREA, %
1000	1750/15/2	124.3	113.7	17.5	44.3
1140	1550/15/3	135.5	126.2	18.3	42.5
1140	1550/15/1	137.1	128.8	16.7	36.8
1140	2250/10/1	130.4	126.9	7.5	17.2

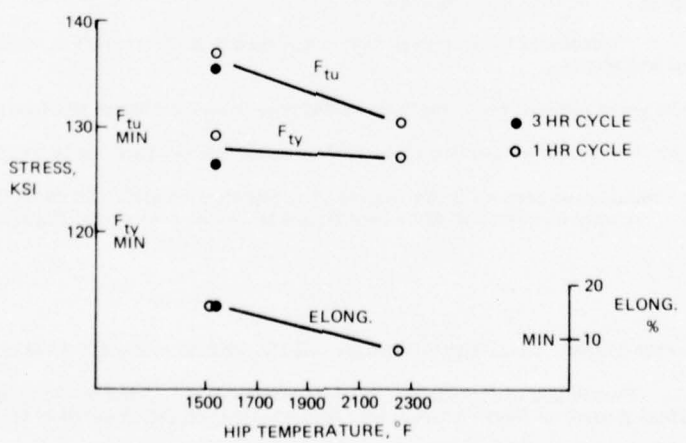


Fig. 5 Tensile Properties of HIP Ti-6Al-4V

It is believed at present that HIP of fully dense preforms and/or near-net shapes would have a great impact on production of deep-pocketed shapes that presently are produced from machined forgings. Such components are mainly used in fuselage, wing and nacelle or related structures. In these areas, titanium alloys may give aluminum and steels more competition, if the savings promised by utilization of P/M to produce net-shapes ultimately materialized.

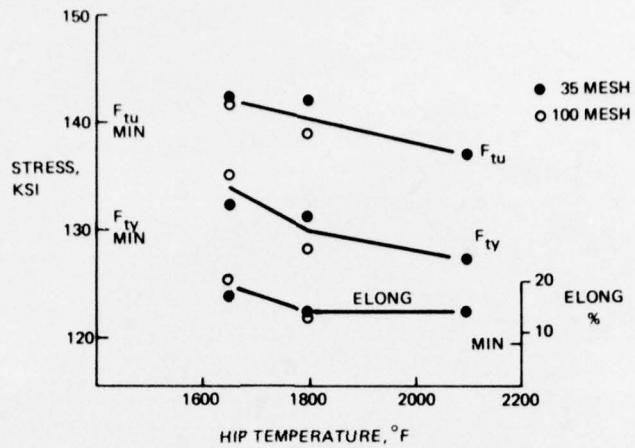


Fig. 6 Tensile Results of HIP Ti-6Al-6V-2Sn

Table VI HIP Ti-6-6-2 and Ti-6-4 Fracture Toughness Properties (1400 PPM Oxygen)

ALLOY	HIP TEMP, °F, POWDER MESH	FRACTURE TOUGHNESS (Kq), KSI INCH$^{1/2}$			AVG
Ti-6Al-6V-2Sn	1650, -35	67.5 70.5*	68.8 70.9*	69.5	68.6 70.7
	1650, -100	53.0	54.0	63.3	56.6
	1800, -35	71.8	73.2	76.9	74.0
	1800, -100	60.4	63.8	65.3	63.2
Ti-6Al-4V	1550, -35	62.3	66.1	68.5	65.6

*K_{ic}

On the basis of results obtained for HIP mechanical properties, there is a good possibility that an essentially one-step consolidation process from powder to component will be fully developed for some alloys. However, if the results are not repeatable on scale-up, or if forging quality cannot be attained, it will be necessary to provide mechanical deformation to assure meeting specification requirements.

The greatest limitations to advancement lie in the following areas:

- Sources of non-contaminated, reasonably priced powders with good apparent densities
- Limited number of processes for HIP of near-net shapes.
- Limited availability of HIP retorts of adequate size
- Need to run extensive dimensional runs on each new component to be fabricated to establish net dimensions and finalize tooling.
- If HIP parts must be given further work, methods of defining preform shapes must be perfected
- The need for data on mechanical properties of as-HIP or as-forged surfaces is lacking

Perhaps the greatest limitation at present is the degree of acceptance by airframe designers for P/M products. Such acceptance can only be advanced after experience is gained with actual flight hardware.

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DISCUSSION SUMMARY OF SESSION IV

by Robert F. Geisendorfer

The discussion of Session IV centered on attempts to explain the differences in test results shown between European and American studies of consolidated prealloyed REP titanium powders, principally tensile, toughness, and high cycle fatigue properties and the effect of inclusions on fracture behavior. Other topics discussed were canning techniques and the possible effect of the canning material on consolidated powder properties, powder purity and standards to control it, comparative tooling needs, and the prospects for increased powder usage in aerospace component fabrication.

Considerable discussion was prompted by the suggestion that the very low oxygen level (600 ppm) of the material evaluated in the American study (R.E. Peebles) contributed a level of forgiveness or tolerance to defects or inclusions that, although present in the material of each study, were found associated with specimen fracture initiation sites only in the European studies (P.A. Blenkinsop, W. Keinath). It was further suggested that perhaps the way to insure good toughness in a titanium structure is to lower the oxygen content. However, it was quickly pointed out that oxygen has historically been needed for strength and designers would be reluctant to accept lower strength for most applications. It was further suggested that perhaps the oxygen level should be shown along with the tensile properties. (It should be noted here that the low oxygen level of material in the Peebles study was specified to allow for pickup during powder production and subsequent handling experienced primarily with hydride/dehydride powders evaluated in Phase I. The result is that in the future for REP powders or others of equivalent quality, low oxygen will most likely not be specified.)

The intriguing question was further pursued of why no inclusions were found on the fracture surfaces of test specimens of the Peebles study; whereas, tungsten (and alkali metal) inclusions were often found at fracture initiation sites of specimens of the European studies reported here. Even though Keinath, et.al. examined perhaps several times the number of fracture surfaces (primarily fatigue specimens) than did Peebles, it is unlikely that none would be detected upon examination of some thirty specimens assuming, of course, that the quality of the powder was the same in each study. It was indicated during subsequent discussion that the disparity in high cycle fatigue is perhaps explained by specimen geometry. In the Peebles study, rotating beam specimens with a minimum cross-section at the center (hour glass shape) were used thereby forcing the failure at that location, the fracture surface of which is less likely to contain an inclusion in the associated small central volume assuming a uniform distribution of defects. The Peebles study used parallel-sided low cycle fatigue specimens however, and the results were comparable to the results of Keinath and coworkers.

With respect to canning techniques, discussion brought out that the Europeans have evaluated mild steel, titanium and some other materials but detailed analysis of diffusion zones and any other effect the can has on compact properties has not been done but perhaps should be looked at. It was pointed out that in studies at AFML using mild steel cans and similar HIP cycles as those used by Peebles, the reaction zone depth in the compacted powder is in the order of .003-.005 inches. Making the container in an economic way does not appear to be a problem and several proprietary processes exist with good shape making capability.

With regard to purity level, the comment was made that the powder producer is blamed primarily for contamination and not the user even though ample opportunity for contamination exists during subsequent handling and processing by the user and hence a specification is needed to check the quality of the powder before it leaves the producer. The response was that existing military specifications are close to what must be produced but perhaps not detailed enough to effectively cope with specific contamination problems that have been experienced; namely, tungsten in the case of REP powders.

Tooling needs for HIP were cited in the presentation by Witt as a problem; whereas, it was commented that others consider the less costly tooling requirements as an advantage of HIP powder metallurgy. The response was that indeed an advantage is shown in the case of HIP, but that at this relatively early stage of development, it is important to get at the tooling problems now to avoid lengthy developments as occurred in the analogous case of diffusion bonded component development.

Granted the undesirable effects of tungsten on properties, and further, if the tungsten is no longer a source of contamination (process modification is currently being attempted to eliminate the tungsten electrode in REP powder production), then the question was where was the response from users indicating that the PM process is a good one and now parts can be made from powders as well as wrought materials. The response was that even though a great deal of interest in PM exists with aircraft manufacturers, the designers and stress analysts must be convinced by substantial comparative data. It was further indicated that due mainly to the increasingly important role of economics in the production of aircraft, powder metallurgical components will be used in the future in place of their wrought counterparts where economically justified.

FINAL SUMMARIES

FINAL SUMMARY
PART I - POWDER PRODUCTION

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1. INTRODUCTION

A significant proportion of the papers and short contributions presented to the Specialists' Meeting were concerned with the necessary precursor of any powder metallurgical fabrication process, namely the production of the powder itself. Since in the planning of this meeting it had been anticipated that the production of titanium alloy powders, for fabrication to high-integrity, highly stressed aerospace parts, was a more critical area than their nickel based counterparts, rather greater emphasis in the selection of presentations was given to the processes relevant to titanium alloys.

Four presentations (P1, SC1, SC2 and SC3) described alternative forms of centrifugal atomisation which currently is the predominant powder manufacturing process for titanium alloys. One paper (P3), representative of a number of sources capable of meeting volume requirements of nickel-base alloy powders, described the more established technique of argon atomisation. One contribution (SC6) described the alternative technique for nickel alloys already proven in a production environment, that of vacuum atomisation.

The main features of the powder production processes described in the meeting, are summarised in Table I. Although not complete in all details, the table provides a rapid comparison of many of their similarities and differences. More specific comments on the status the various processes have currently achieved, their limitations and the quality of powder produced, are most conveniently made by consideration separately of those applicable to titanium alloys and to nickel alloys.

2. TITANIUM ALLOY POWDER PRODUCTION

2.1 Processes

The reactive nature of titanium towards both interstitial elements and refractory crucible materials has led to the development of processes closely related to conventional ingot melting procedures, but incorporating centrifugal action to break up the melt into discrete droplets. Hence all four of the titanium processes of Table I, require the start material to be in solid electrode form. Two of these processes (Rotating Electrode and Centrifugal Shot Casting) use arc melting in an inert atmosphere and so benefit from the reduced flight path required for solidification of droplets provided by the predominant convective heat loss, although capable only of maintaining the composition and purity of the starting electrode. Two processes (Electron Beam Rotating Disc and Electron Beam Rotating Electrode) involve electron beam melting within a high vacuum environment; are restricted to radiation losses for droplet cooling; offer potential for interstitial refinement, but suffer from significant material loss or composition variation by volatilisation.

Two of the processes (REP and EBRE) rotate the electrode whose tip is being melted and hence place more stringent requirements on the shape, straightness and integrity of the electrode. In the CSC and EBRD processes the water-cooled crucible and disc respectively, are rotated at high speeds not the electrode, and offer greater flexibility of electrode form.

The four centrifugal atomisation processes differ more significantly in the nature and extent of powder contamination influencing their quality and also in the present stage of development and progress towards volume production. The Rotating Electrode Process has clearly been used most widely in titanium alloy powder fabrication and evaluation programmes. It has the experience of volume production in other materials that provides the capability to operate at 100 tonne per annum per 'long-bar' machine if the demand for titanium powders required it. However all this production experience involves the use of 'non-consumable' tungsten cathodes which can give rise to the presence of tungsten inclusions within the alloy particles. Although apparently conflicting evidence was heard (P11, SC8, SC9) of the deleterious effect upon fatigue properties of such inclusions in titanium alloys, it may be possible to rationalise this conflict in terms of matrix strength sensitivity and fatigue specimen and test differences. It is unlikely however that REP powders containing tungsten contamination will gain acceptance for use in aerospace applications and possible ways of replacing tungsten as a cathode material with titanium alloy are currently under investigation. Until these studies are complete, the Rotating Electrode Process for high quality titanium alloy powder production must be regarded as being in a development phase.

The Centrifugal Shot Casting Process, although under investigation for a number of years, has made limited progress towards volume production. The present pilot plant operates on a single electrode batch cycle, and has restricted collection chamber dimensions, leading to low material throughput and utilisation for high tap density spherical product. Although potentially free of contamination from process components,

TABLE I Powder Production Processes

Meeting Ref.	Process	Company	Method	Start Material			Atomisation Atmosphere	Melting Rate Kg/min	Present Capacity te/y	Powder Properties			Material Utilisation %
				type	form	quantity Kg				size range µm	mean size µm	tap density %	
P1	Electron beam Rotating Electrode (EBRE)	C.E.N.G. France	Centrifugal atomisation of rotating electrode	Ti	Electrodes 50mm dia. 200mm long	2	Vacuum ~10^{-4} torr	0.5	7	50-1000	400	-	85 (spheres <1000µm)
SC1	Rotating Electrode (REP)	Nuclear Metals Inc. USA	Centrif.atomis. of rotating electrode	Ti	Electrodes 62mm dia. 1.5m long	35	He ~1atm	-	100/machine	50-500	225	65	95 (for 1.5m electrodes)
SC2	Electron beam Rotating disc (EBRD)	Leybold-Heraeus GmbH, Germany	Centrif.atomis. from rotating disc	Ti	Electrode up to 150mm dia. 800mm long	64	Vacuum	1-2	-	50-700	-	60	-
SC3	Centrifugal Shot Casting (CSC)	A.E.R.E. Harwell, U.K.	Centrif.atomis. from rotating crucible	Ti	Electrode up to 76mm dia. 600mm long	12	He/Ar ½-1atm.	1	1-3	50-1000	300-400	65	60 (spherical fraction)
P3	Argon atomisation	H.Wiggin & Co. U.K.	Atomis.of molten stream by argon jets	Ni	Vacuum induction melt	500	Argon >1atm	-	>500	5-500	-	64 (<150µm)	- (<150 or 180µm)
SC6	Vacuum atomisation	Homogeneous Metals Inc. USA	Exposure of supersaturated solution of H_2 in melt to vacuum	Ni	Vacuum induction melt	200	Vacuum/H_2	~100	>150	1-200	10-50	60-65	-

some instances of cross-contamination (see P11) of non-titanium alloys previously processed have occurred due to a non-optimised chamber design for ease of cleaning.

The present equipment of the Electron Beam Rotating Electrode process is clearly a purpose built production unit capable of operating at modest capacity, almost 10 te/annum for 50mm electrodes, with facilities for remote electrode changing and powder take-off without re-evacuation. The small size of each electrode is the greatest limitation to increased scale and material utilisation. In addition, a novel method of achieving solidification to spherical particles within reasonable collection chamber dimensions, using an ablative proprietary coating on a cooled deflector plate, requires exposure of the impinging droplets to fresh regions of coating at successive intervals. Such a consumable coating offers a potential source of carbon pick up although evidence to date suggests this is insignificant.

The most recent and least evaluated of the four centrifugal atomisation processes is the Electron Beam Rotating Disc process. One critical aspect of this process identified so far is the preferential loss of more volatile alloying additions such as aluminium. Losses of aluminium from Ti-6 Al-4V alloy, in excess of the specification range have been experienced during initial trials at the present scale of operation, presumably due to the larger area of molten surface, and the more extended time the alloy is molten in the drip melting/disc atomisation as compared to earlier laboratory experiments of actually consuming the rotating disc.

2.2 Conclusions and Recommendations

None of the titanium alloy powder production processes has yet clearly demonstrated its capability of producing powder of sufficient quality to give unequivocally acceptable static and dynamic property levels. Contamination of powders can be divided into categories. That which is basic to the process itself for which process modifications are required to eliminate the source of contamination. That of cross-contamination of powders of different composition, which in principle can be minimised by restriction of a particular atomiser to use for titanium alloys only. In practice however in a situation of insufficient demand for titanium powders to economically monopolise the use of one atomiser, its elimination is less straightforward from a powder producers viewpoint.

The non-destructive examination of powders, or destructive examination of consolidated powder samples, for quality control purposes to establish freedom from contamination to an acceptable level is one area requiring further investigation.

With such technical difficulties still outstanding, it is premature to consider powder cost projections for volume production or perhaps large capital investment in volume production facilities until their solution has been achieved and user confidence in acceptable property levels is established.

3. NICKEL ALLOY POWDER PRODUCTION

3.1 Processes

Although the processes, considered in the last section to be more relevant to titanium alloy powder production, are inherently capable of application to nickel-base alloys, the economic penalty of the provision of solid electrode start material, particularly in high temperature creep-resistant alloys, rules out their use in volume production. Processes such as argon atomisation and vacuum atomisation have in general reached a degree of development, as demonstrated by acceptable powder quality and property levels in production quantities, for them to be more established processes.

Argon atomisation, as illustrated by the new integrated powder production and consolidation facilities of Henry Wiggin and Co. (P3), is the more prevalant of the two processes with a number of production facilities, particularly in the USA. Earlier problems of restriction of grain growth by prior particle boundary phases, though not specific to argon atomised nickel alloy powders, were a consequence of their metastable state as produced, and have been overcome by modifications to the alloy chemistry and consolidation conditions. Difficulties of elevated temperature induced porosity, caused by argon filled pores within the larger particle sizes of the distribution of sizes normally produced by argon atomisation, were experienced. These have been eliminated by restricting the useful portion of the total size distribution to be less than 150 or 180μm particle size where argon levels of <2ppm are found to be acceptable (see P3). Clearly this places a limitation on material utilisation of the argon atomisation process. Inert gas handling facilities have been adopted between atomisation and encapsulation. Though not fully proven to be entirely necessary, they are probably an advisable precaution because of the increased proportion of finer particle sizes below ~50μm that are present in argon atomised as compared to centrifugally atomised powders. Tramp element contents, that could be influenced by crucible interactions and nozzle erosion, appear to be capable of being controlled within acceptable levels.

Vacuum atomisation, a process unique to Homogeneous Metals, Inc (SC6), has been shown to produce acceptable powder at comparable cost, that behaves on fabrication with almost indistinguishable similarity, to argon atomised powder. It is immune to argon filled porosity difficulties. The finer size distribution makes it even more vital to

handle under high purity inert gas cover. It was claimed that vacuum atomisation offers potential advantages over alternative processes in that it is capable of producing extremely fine particle sizes (~10μm mean diameter). This was considered important in future alloy development, in which retention in solution of increased contents of hardening elements may be achieved by higher solidification rates. The vacuum environment providing radiative cooling only, may partially nullify this potential advantage as compared to rather coarser sizes cooled in an inert atmosphere environment (see SC3), unless the residual hydrogen content of the cooling environment provides significant convective cooling.

3.2 Conclusions and Recommendations

Argon and Vacuum Atomisation processes are well established nickel-base superalloy powder production processes. No serious technical limitations have so far been identified that are likley to usurp their present position. Three sources of such powders are currently fully qualified to produce powders for flight quality powder billets (P5).

Their present price range of $7-12/lb. together with the significant materials and processing savings offered by powder fabrication routes, make them economically viable in high performance military aeroturbines. The potential for reducing powder costs with increased market volume and possibly scrap re-utilisation will dictate their penetration into civil aircraft applications.

Adequate capacity to meet projected early 1980's demand for nickel alloy powders is already available. One twist of fate that results from the major advantage of P/M processing, namely improved material utilisation, may lead to a situation of overcapacity unless alternative outlets for P/M nickel-super alloys can be found or alternatively use of P/M facilities in other compatible nickel containing alloy fields can be increased.

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FINAL SUMMARY

PART II - POWDER CONSOLIDATION

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I. INTRODUCTION

The rapid advancement in powder metallurgy (PM) for aerospace applications in recent years has been due primarily to the availability of acceptable quality powders at reasonable prices; and secondarily, to the development of consolidation techniques which lend themselves readily to scale-up for low-cost manufacturing. The state-of-the-art for both titanium and superalloy powder production techniques was discussed in the previous summary. The state-of-the-art for consolidation and associated processing techniques will be discussed in this summary.

The format of this meeting was established to follow a logical sequence, beginning with powder production methods and ending with consolidation methods. This was accomplished through specific papers and short contributions by the various authors on the results of their efforts in the consolidation of both titanium and superalloy powders.

This discussion is organized to follow the same general sequence from the handling of powder, through consolidation, and to evaluation of the final products where all of the contributions will be integrated into an assessment of the technology. Conclusions are drawn as to the state-of-the-art of powder consolidation into usable shapes and finally, recommendations are made for future activities.

II. DISCUSSION

For the purpose of this discussion, the technology of powder consolidation leading to finished compacts has been divided into five distinct stages. They are: (1) powder handling, (2) canning techniques/procedures, (3) consolidation techniques, (4) secondary processing, and (5) post compaction thermal treatments. Furthermore, the evaluation of the final consolidated product has been separated into three distinct areas of concern. Continuing then, these are: (6) nondestructive evaluation, (7) mechanical properties, and (8) process economics. Each of these areas will be discussed with reference being made to papers and short contributions presented at this meeting. These areas will be discussed in the order that they are listed above.

1. Powder Handling

Powder handling is vital to the production of consolidated products that have acceptable properties. The inclusion type of contamination in the consolidated product was noted by several contributors for both superalloy products (P7)(Note 1) and titanium products (P11, SC8, SC9). Most of the contamination could be attributed to the powder production process through cross-contamination (use of the same facility for the production of more than one type of powder) or through the inherent nature of the process - - tungsten contamination in the REP process for titanium (SC1). However, the distinct possibility exists that the powder could become contaminated after its manufacture. Particulate matter from the atmosphere (if air handled), foreign matter from screening, storage, degassing, canning or other non-dedicated facilities required for processing the powder could harbor additional sources of contamination.

Gaseous contamination poses another problem for PM technology in that powder has a relatively high surface to volume ratio which permits the absorption of significant quantities of surface gases. Experience has been varied with respect to atmospheric exposures. Fiedler (P4B) reported that air handling of PA101 posed no unusual problems in As-HIP-and-heat-treated products, whereas Evans (P4A) reported the lack of forgeability in LC Astroloy unless extreme measures were taken to either vacuum handle or hot dynamic vacuum outgas prior to consolidation. A point of agreement was that inertly (argon gas) handled powder must be outgassed to preclude the possibility of thermally induced porosity (TIP) in the microstructure during post-consolidation thermal treatments. Symonds (P3) reported that argon analysis of HIP compacts is a good technique for validating that compact full density has been achieved, thus precluding the possibility of TIP. Generally, the consensus was that inert handling of superalloy powder is the most reasonable approach. This, in turn, requires outgassing procedures to preclude TIP.

Powder handling requirements are less well defined for titanium. Both Keinath (SC8) and Peebles (SC9) examined powder handled in various ways; including vacuum, inert gas (argon) and air, with conflicting results. Peebles concluded that air handling REP titanium powder was an acceptable procedure. On the other hand, Keinath concluded that inert gas handling improved fatigue and possibly other mechanical properties. However, early fatigue failures in REP powder due to nonmetallic inclusions of unknown origin suggest that handling may be critical. It can be concluded, therefore, that there is a need for additional work in the area of titanium PM processing.

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Note 1: Refers to numbered Papers (P) and Short Contributions (SC) contained in this Report.

2. Canning Techniques

Suitable low-cost and reproducible canning techniques are essential to the consolidation of powder into fully-dense, usable products regardless of the consolidation technique used. Several canning techniques were presented at this meeting. Mild steel, and in some instances stainless steel, canning techniques were the most widely used for both titanium and superalloys. In most cases, the cans were simple cylindrical shapes; as described by Blenkinsop (P11), Allen (P5), Symonds (P3), Lescop (P8) and Betz/Huff (P7). Symonds (P3) also discussed spinning and superplastic forming of more complex (sonic outline) steel cans for producing disks up to 40 cm in diameter. Fiedler (P4B) discussed and showed a fully-dense and complex 28 cm diameter disk made by a proprietary ceramic mold technique developed in the United States. Fiedler indicated that the process was both low-cost and reproducible. Likewise, Keinath (SC8) showed photographs of several complex titanium airframe structural parts made by a proprietary European process which consists of electroforming nickel on master forms. Keinath indicated that the process was within the state-of-the-art, with complex shapes posing no major problems for them. Mazzei/van Drunnen (SC7) showed results of the technique that they used in which stainless and low-carbon steel sheet was formed into complex airfoil shapes for subsequent powder consolidation.

Other canning techniques, such as the "Glass Bag" technique of Kelsey-Hayes (US) and the "Quick HIP" technique of Snecma (France) were mentioned but no details were provided.

In large measure, the potential economics of PM are tied to the development of low-cost, reproducible canning techniques which will result in parts which are near to net shape (i.e. buy-to-fly ratios of less than 2:1). Therefore, considerable effort should be, and is being, expended to develop reproducible and economical canning techniques that will yield near to net shape parts with a minimum of surface contamination.

3. Consolidation

Although several consolidation methods were discussed, the primary emphasis at this meeting was on Hot Isostatic Pressing (HIP). HIP technology is being evaluated in almost every NATO country (in 11 papers) for potential application to the production of powder metallurgy parts for aerospace components. Other consolidation techniques discussed were extrusion (P5,P7), sinter-plus-HIP (P4B), press-and-sinter (P12) and explosive compaction. (Note 2).

One paper (P2) and two short contributions (SC4,SC5) were given which dealt with general HIP technology and equipment/economics, respectively. These contributions pointed out several reasons why HIP technology is receiving so much attention today. First, the technique is simple in concept and is relatively low cost. Both Larker (SC4) and Smith (SC5) indicated HIP costs in the order of $.20 to $1.00 (U.S.) per Kg, depending on the kind of HIP facility used. Thus, the process, when used in conjunction with a variety of canning techniques, has demonstrated the capability of producing complex parts at considerable cost savings when compared with conventional processing techniques.

The HIP process is versatile in that it can be used for producing any product configuration from simple billet stock for conventional processing to shaped preforms for subsequent forging and complex near-net shapes which do not require further metal working. The numerous approaches that can be taken in producing parts by the HIP process were discussed in detail by both Symonds (P3) and Arnold (P6). HIP is also versatile since it can be used for any alloy system or composite systems. An example is the work done by Mazzei/van Drunnen (SC7) to produce superalloy matrix/thoriated tungsten composite shapes. Also, HIP processing has demonstrated a capability to produce parts which, in many instances, have properties equivalent to their wrought counterparts; as will be discussed later.

HIP technology is currently used in production by Crucible in the U.S. and Stora/Kopparburg in Europe (SC5) for producing high grade tool steels. The technology application is, therefore, a proven production process and the equipment has been shown to be capable of sustained production usage. Several approaches to HIP systems were discussed, from cold load/cold unload to hot load/hot unload. It was generally concluded that the high cost of capitalization for either type of system and the supportive equipment warrants a close examination of the potential usage and throughput before selection of a given system is made.

The extrusion process is used to consolidate cylindrical billet stock which is subsequently isothermally forged to produce parts. This technology is currently being used by Pratt and Whitney Aircraft (P&WA) for the production of IN-100 turbine engine parts for the F100 engine. Allen (P5) reported that P&WA production and service experience with the extruded/isothermally forged approach was excellent. Work on extrusion of PM products was also reported by Betz/Huff (P7) for IN-100 and U-700 and Grapier, et. al. (P10) for cobalt base alloys. However, Drapier subsequently worked the alloys by swaging and rolling rather than extrusion.

The sinter-plus-HIP approach (P4B) appears to be a novel, low-cost method for the production of small superalloy parts such as turbine buckets. The process is rather new and hasn't been fully evaluated for part properties, shape making capability or economics, but looks promising for the future.

The press-and-sinter approach was discussed by Witt (P12) as applied to titanium. Several different kinds of titanium powder, such as the blended elemental and hydride-dehydride, were examined in this effort. The approach has found little support for the production of primary structure but may hold some promise in the future for non-structural or secondary structural parts due to the potential economic advantages inherent in the process - - such as high production rates and low input material costs.

Both Kellerer and Geisendorfer reported work done to compact titanium powders into billet stock by explosive techniques. The results suggest that some potential may exist for this method, although further work must be done to establish it as a viable process.

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Note 2: Comments from the floor by Kellerer (Germany) and Geisendorfer (US),

4. Secondary Metalworking

For superalloys, conventional and isothermal forging of HIP preforms and billet stock have been extensively evaluated. Likewise, the forging of extruded billet stock has also been extensively evaluated. Symonds (P3), Evans (P4A), Arnold (P6), Betz/Huff (P7), Lescop (P8) and Wallace (P9) all reported on work done with HIP-plus-forged products. Allen (P5), and Betz/Huff (P7) reported work done on extruded-plus-forged products, while Drapier (P10) worked with extruded-plus-swaged and extruded-plus-rolled material. Discussion brought out that unless a superalloy powder compact is fully dense to begin with, subsequent metalworking will not satisfactorily close nor metallurgically heal porosity. Apart from that consideration, it was shown that PM preforms or billets are easily worked by conventional methods and properties equivalent to wrought are attainable. Furthermore, the state-of-the-art has advanced to the point where some PM-plus-secondary-metalworked products are being used in major engine structural applications in the U.S. Isothermal forging of extruded IN-100 powder billet is a current production process for major rotating structural parts in the F100 engine, while conventional forging of HIP-consolidated Astroloy powder preforms has been qualified for use on the TF30 engine. In both instances, the use of the PM approach has resulted in considerable savings in both material and cost.

Peebles (SC9) and Blenkinsop (P11) both reported the results of conventional forging work done on HIP Ti-6Al-4V preforms and billet stock, respectively. Neither contributor noted any unusual problems encountered during secondary metalworking which would suggest any problems in working HIP titanium powder products.

In summary, a variety of deformation techniques were reported; none of which experienced any difficulty in working any of the various alloys and alloy systems. It is therefore, concluded that, for PM products consolidated to full density material, deformation behavior is predictable and similar to that expected from conventionally processed material with similar metallurgical structures.

5. Post-Compaction Thermal Treatments

Wallace (P9) indicated that partitioning of MC type carbides to the surface of superalloy powder particles occurs during solidification. This behavior requires subsequent processing of the powders during both consolidation and secondary metalworking, with close attention given to the specific thermal and thermo-mechanical cycles during processing to insure that desired microstructures are obtained in the final product. Betz/Huff (P7) reported on an effort directed at integration of the thermal cycles to produce compacts with acceptable properties. Although this work has been discussed by the other participants, little additional information was presented and not much discussion was directed at the subject area.

It is, therefore, presumed that for most individuals in this area, sufficient importance has been emphasized in the early stages of their development of the technology with little additional discussion at this meeting. It is apparent however, that as the consolidation technologies continue to evolve; such as hot load/unload vs cold load/unload HIP cycles, canning method variations, secondary metalworking improvements, powder production methods and alloy modifications; the consolidation and thermo-mechanical cycle will continue to require considerable effort in order to produce metallurgically stable products with desired mechanical properties.

6. Nondestructive Inspection Techniques

It is apparent from the complex As-HIP shapes shown by Fiedler (P4B), Keinath (SC8) and Witt (P12) that, as PM technology moves toward the production of near-net shapes which are inside the conventional "ultrasonic envelope" capability and nonrectilinear in shape, NDI will be the limiting factor for the future use of such complex parts in critical applications. Fiedler indicated that the shapes he presented were too complex to be adequately inspected; and in production, the shape would have to be modified to acommodate current capability. The complex spun cans shown by Symonds (P3) were made to the sonic shape for inspection purposes, and apparently could have been a more complex shape if it were not for the NDI shape restrictions. Symonds and Arnold (P6) both concur and place additional work on net shape NDI high on their priority list.

In Mr. Peterson's keynote address, he summarized the need for improved NDI techniques as a two-fold problem. First, there is a need to develop more sophisticated ultrasonic test methods which reduce or eliminate the dead zone at the surface of part and can inspect non-rectilinear parts including non-parallel surfaces, undercuts and sharp radii. Also, multi-axis tracking systems must be adapted to NDI equipment for automated inspection of complex shapes.Computer techniques also need to be established for analysis of the data generated during parts inspection. Secondly, NDI techniques are "seeing" defects which are much smaller than can be observed in conventional wrought products due to greater "transparency"; which is attributed to more uniform, fine-grained microstructures. Therefore, the need exists for establishing new accept/reject criteria for PM products which is not excessive and hence, overly restrictive.

In summary, the technology for making complex shapes by PM techniques has surpassed the ability to adequately inspect them for use in critical applications. Therefore, in order to derive full economic benefit of PM technology, NDI techniques must be advanced for the inspection of complex, near-net shapes. This will doubtless be the pacing item for the near future introduction of critical, net-shape PM components in the aerospace industry.

7. Mechanical Properties

A great deal of discussion was generated by the large amount of property data on PM products processed by the various methods described at the meeting. With regard to HIP, data was presented for As-HIP products as well as HIP-plus-secondary-metalworking processes.

For superalloys, it was generally reported that PM properties were somewhat less than their wrought counterparts. For this reason, Wallace (P9) suggested that the best near-term application for HIP superalloys is the production of forging preforms. Others indicated, that for many applications, high grade

superalloys (such as Astroloy, René 95 and PA101) could be As-HIP produced to near-net shapes and substituted for lower grade alloys (such as Waspaloy, IN-718 and D-979 respectively) at substantial cost savings.

Properties of HIP-plus-secondary-metalworking (forging) were reported by Evans (P4A), Symonds (P3), Arnold (P6) and Lescop (P8) to meet or exceed conventional product properties. Symonds further noted outstanding low-cycle fatigue properties in APK1 (modified Astroloy).

Betz/Huff (P7) evaluated both extrude-plus-forge and HIP-plus-forge properties for IN-100 and U-700. Their work led them to conclude that superalloys must be worked to obtain properties equivalent to wrought products. However, they believed that additional work was needed in alloy modification and process optimization to achieve these goals.

Allen (P5) indicated that extruded-plus-isothermal-forged properties of IN-100 were acceptable and that service experience with this alloy was excellent.

Properties reported for PM titanium alloys were equivalent to their wrought counterparts with the exception of smooth low-cycle and high-cycle fatigue properties as reported by Keinath (SC8) and Blenkinsop (P11). Both reported low values and scattered results which were attributed to inclusions (both tungsten and nonmetallic type) at the fracture origins.

Peebles (SC9), on the other hand, showed acceptable low-cycle and high-cycle fatigue results. However, his results were rationalized as due to the low oxygen content (600 PPM) of the selected powder and heat-treat condition of the compacts; as well as the hourglass specimen configuration in the case of high-cycle fatigue. Both Keinath and Blenkinsop tested material in the STA condition, while Peebles conducted his tests on fully annealed material. Peebles did indicate that he had also observed inclusions in the titanium compacts, but that no failures could be attributed to these inclusions. It was generally agreed that the primary difficulty remaining with titanium PM is powder cleanliness. With the anticipation of a near-term solution to this problem, near-net HIP titanium parts for primary structural applications will soon be a reality.

8. Economics

Much of the discussion centered around the potential economics of PM technology. It was generally agreed that significant cost savings potential exists through many of the possible PM processing methods. If the "consolidate to preform plus forge" method is considered, savings accrue from the requirement for less material and fewer forging steps in order to achieve an equivalent end product when compared to conventional processing. Symonds (P3) indicated a 10-40% cost reduction possible by this method. Arnold (P6) was somewhat more optimistic, indicating a 30-40% cost savings potential. The "consolidate to near-net shape" method is believed to have the greatest cost savings potential, since in addition to significant materials savings, secondary operations are completely eliminated and machining cost is reduced significantly, with little or no machining required. Symonds anticipated a 50-60% cost reduction potential via this method, whereas Arnold anticipated a 45-80% savings by this same method.

For titanium alloys, Peebles (SC9) reported that their cost analysis showed a savings of about 15% for the "HIP preform plus forge" method. Keinath (SC8) indicated that considerable cost savings are possible for titanium parts which presently require "large amounts of machining".

III CONCLUSIONS

It has been demonstrated that HIP technology is capable of producing titanium and superalloy powder compacts to full density. The process is generic and has significant potential for the production of critical parts for aerospace applications.

Powder handling techniques are critical to the production of acceptable compacts. The potential for cross-contamination or degradation of properties due to adsorbed gases dictates that either inert gas handling with advanced outgassing techniques be utilized or that powder be vacuum handled to preclude adsorbed gas problems.

Canning techniques are rapidly advancing, resulting in the manufacture of complex net shapes in both titanium and superalloys. However, additional work is required to extend the techniques into production practice, demonstrate reproducibility and validate potential economics.

Of the many PM processing methods, HIP technology appears to be the nearest-term process with production potential for aerospace applications. The process is versatile and can be used for the production of billet, forging preforms and net shapes. As-HIP-plus-heat-treated superalloy products show good properties but slightly less than wrought products. The highest grade superalloys (IN-100, Astroloy, René 95) have improved properties over lower grade alloys (Waspaloy, IN-718 and D-979) and could be directly substituted as near-net shapes with economic advantages. As-HIP-plus-heat-treated titanium alloys show equivalent properties compared to wrought products, with the exception of fatigue. Most PM product premature fatigue failures can be attributed to inclusions in the powder. With the advent of "clean" titanium powder, it is anticipated that PM titanium alloys can be used as direct substitutes for their wrought counterparts.

Non-destructive inspection of PM products looms as the greatest near-term barrier to the use of complex near-net or net shapes. The current NDI technology limits shapes to being rectilinear with envelopes of greater than .100 inch (.25 cm).

The potential cost reductions for superalloys were reported as 10-40% for forging preforms and 30-80% for net shapes. The cost savings potential for titanium alloys was somewhat less, but still significant, at 15% for forge preforms and greater than 35% for net shapes.

IV. RECOMMENDATIONS

As a result of the powder consolidation developments presented and discussed at this meeting, the following recommendations are made:

1. Efforts should continue to establish HIP technology as a viable PM part production technique for aerospace applications.

2. Developments in canning technology should continue, with the reproducibility and potential economics of the more sophisticated net shape canning technologies validated.

3. Outgassing techniques should be refined to assure that low cost, effective methods are available for removing adsorbed gases from powder.

4. Effort should be devoted to the NDI of complex net shapes in order to achieve the maximum economic benefits possible with PM products.

5. Efforts should continue for improving the fatigue performance of PM titanium alloys. Hopefully, this will result from improved, high quality powders.

6. PM parts must be produced and flight-tested to verify their usefulness and reliability. Sufficient data must be generated to provide designers with "allowables" properties. Specifications must be written for both powder material production and processing.

7. New or improved alloys should be developed and tailored to the PM process, since PM alloys would not be limited by constraints of being either "castable" or "workable".

8. The adaptation of advanced PM techniques to the production of metal matrix composite structures, or other "engineered" systems, should be pursued to take full advantage of the versatility of the PM process.

9. Research and development of a more fundamental nature should be pursued to increase our understanding of the deformation mechanisms of specific alloy systems occuring during compaction, as well as the metallurgical effects occuring during and after consolidation. This will assist in the selection of optimum processing cycles.

10. Workers in PM technology should stay abreast of other competing technologies currently being developed in order to best assess the competitive position that PM technology does (and will) have in the future. Competitive technologies being established include advanced structural castings, dual alloy concepts for disks and blades, and complex built-up structures utilizing diffusion bonding, superplastic forming or other approaches.

REPORT DOCUMENTATION PAGE

1. Recipient's Reference	2. Originator's Reference	3. Further Reference	4. Security Classification of Document
	AGARD-CP-200	ISBN 92-835-0171-3	UNCLASSIFIED

5. Originator Advisory Group for Aerospace Research and Development
North Atlantic Treaty Organization
7 rue Ancelle, 92200 Neuilly sur Seine, France

6. Title ADVANCED FABRICATION TECHNIQUES IN POWDER METALLURGY AND THEIR ECONOMIC IMPLICATIONS

7. Presented at the 42nd Meeting of the AGARD Structures and Materials Panel held in Ottawa, Canada, 4–9 April 1976.

8. Author(s)	9. Date
Various	November 1976

10. Author's Address	11. Pages
Various	266

12. Distribution Statement This document is distributed in accordance with AGARD policies and regulations, which are outlined on the Outside Back Covers of all AGARD publications.

13. Keywords/Descriptors

Powder metallurgy	Titanium alloys
Atomizing	Nickel alloys
Fabrication	Heat resistant alloys

14. Abstract

The purpose of this meeting was to consider the economic achievements of powder metallurgy techniques as well as the technological techniques. Cost reduction and improved materials qualities were given equal priority.

The first part of the proceedings was concerned with powder production. Four different processes of centrifugal atomization techniques for manufacture of titanium alloys were presented together with descriptions of Argon and vacuum atomization processes for nickel base superalloys.

The second part reported on the state-of-the-art of techniques for consolidation of titanium and superalloy powders to near net shapes, discussion also included all stages of the production sequence – powder handling, canning techniques, consolidation, secondary metal-working and thermal treatments.

Recommended areas for further R & D work were discussed.

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