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AD-P004 217	Oxide Dispersion Strengthened Alloys.
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AD-P004 219	Potential of Metal-Matrix Composites as Superalloy Substitutes.
AD-P004 220	Manufacturing Developments to Reduce Strategic Materials Usage.
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POTENTIAL OF METAL-MATRIX COMPOSITES AS SUPERALLOY SUBSTITUTES

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ABSTRACT

The potential of metal-matrix composites as gas-turbine blade materials has largely been judged on the increased temperature capabilities they offer over the strongest current superalloys. However, several composites that have been rejected on this basis are either independent of or have low concentrations of critical alloying elements. Consequently they should be reassessed as reserve materials matching the performance of existing widely used superalloys that depend on strategically vulnerable constituents. A review of metal-matrix composites for high temperature applications will be made in order to identify materials that are insensitive to the supply of critical elements.

An alternative approach might be to consider the potential of regenerating the original creep performance of certian monocarbide reinforced nickel based composites - such as Cotac 744 developed at ONERA - after service exposure. In this context the combination of perfectly elastic fibres in a creeping matrix would imply that high temperature deformation of these materials is almost completely recoverable. Hence, one should judge the potential of these materials in the light of total life expectancy through both the processing and regeneration conditions rather than from their simple creep behaviour alone.

INTRODUCTION

The principal motivation for the development of high temperature composite materials for gas turbine applications appears to have been to exceed the temperature capabilities of the very strongest superalloys. The aim has been to produce materials with an additional strengthening mechanism that remains operative after the usual hardening agent of nickel-base superalloys, viz γ' precipitate, has dissolved in the γ-matrix. Alloys have indeed been described that offer a clear technical advantage (1,2,3) over existing superalloys; however, processing difficulties affecting the economics of production have inhibited the exploitation of these materials. VerSnyder (4) has expressed the optimistic view that cost-considerations will only delay acceptance of these materials until the 1990's; however, there must be a danger that the advantages of these high strength composites will be superseded by developments in other materials (eg single crystals, oxide-dispersion strengthened alloys, ceramics).

The aim of this paper is to reassess the potential of high temperature composites in the context of the themes of the present meeting. Firstly, they will be considered as direct substitutes which match but do not necessarily exceed the performance of existing materials but which may offer significant advantages of reduced cost or reduced dependence on certain strategically vulnerable elements. Secondly, the response of composite materials to post-service rejuvenation procedures will be considered in order to assess the full potential service lives of components.

The emphasis will be on in-situ, rather than synthetic, composites since they are broadly compatible with existing investment casting and directional solidification technology and, consequently, are more likely to be accepted as direct substitutes for existing materials. It may be necessary to modify the processing conditions significantly (temperature gradient and solidification rate) and use different moulding materials in order to prevent mould-(or core-)melt interactions. However, the basic equipment and experience are available to allow commercial development of in-situ composites if an economic or strategic benefit warrants their exploitation.

CRITICAL METALS CONTENT

Analyses of import dependence of the US on critical metals have recently been published by Bradley (5) and by Metal Bulletin Monthly (6). These show that of the usual constituents of superalloys more than 90% of cobalt, chromium, tantalum, manganese and niobium originate from predominately Third World, South African and Eastern Bloc sources; consequently their supply is vulnerable to political and/or economic pressure. The situation is even more severe for the European industry which is dependent on virtually 100% of its raw materials so that nickel, titanium, tungsten, molybdenum and vanadium are also of strategic importance; the supplies of these elements, from ally nations, are perhaps politically assured but they are still subject to economic pressures. It would clearly be politic to have alternative materials less dependent on certain critical metals, available for use in important components in the event of disruption of supplies. It is unreasonable to consider the total elimination of all strategic elements in a metallic component; rather we shall describe the structure and properties of composites that have no or low concentrations of specific critical metals in relation to current materials. Their compositions are listed in Table 1.

CO-FREE ALLOYS

The mechanisms by which cobalt strengthens nickel-base superalloys have not been clearly established (7). Tien et al (7) have surveyed current evidence showing that the presence of cobalt can influence the volume fraction and solvus of the γ' precipitate, the stacking fault energy and the carbide chemistry which cumulatively lead to a large effect that makes cobalt an indispensible constituent of most advanced nickel-base superalloys.

While the benefits of Co have been incorporated in the development of the most advanced in-situ composites, such as the NITAC (2) and COTAC (1) series, some of the earlier versions were cobalt-free. However, the processing difficulties, low transverse ductilities and poor thermal fatigue resistance militate against any commercial exploitation of these materials. The γ-γ'-Cr_3C_2 alloy, developed at NPL (8), although not matching the high temperature strengths of the strongest in-situ composites is both cobalt-free and compares favourably with many current alloys.

a) Processing. Unlike many in-situ composites which must be directionally solidified very slowly (< 10 mmh^{-1}) in very high temperature gradients (~ 20 Kmm^{-1}), γ-γ'-Cr_3C_2 can be successfully prepared at high rates (up to 1000 mmh^{-1}) and in relatively low temperature gradients. This is similar to the conditions used in the directional solidification of superalloys. The alloy is relatively insensitive to the precise compositions acceptable microstructures being obtained from a range of casts made to commercial specifications (Figure 1). It would be necessary, however, to develop new mould and core materials since there are significant interactions between the melt and the conventional silica-base shell moulds (Figure 2). However, such a development may also be required to allow effective recycling of directionally solidified superalloys since silicon leached from the moulds can apparently weaken the longitudinal grain boundaries in directionally solidified components prepared from revert superalloys.

b) Physical properties. The density of 7.5 Mgm^{-3} is lower than that of most current superalloys (similar to IN100) and the melting temperature of 1320°C is as high as present superalloys.

c) Oxidation/corrosion resistance. The alloy has excellent cyclic oxidation resistance assessed by laboratory thermogravimetric tests and metallographic examination; this has been confirmed in burner rig tests performed by Rolls-Royce Ltd at 1323 K in an atmosphere containing 4 ppm NaCl. The results, shown as depth of attack as a function of time in Figure 3, compare favourably with previous data for typical stator and rotor blade alloys (C 1023 and MarMoo2 respectively). Indeed the uncoated γ-γ'-Cr_3C_2 has similar oxidation resistance to aluminised superalloys.

In the more severe corrosion rig test for 300 h at 850°C with fuel impurities of 0.15 sulphur and 50 ppm NaCl injected in the atmosphere there is no detectable loss of section although IN713 is severely affected in these conditions (Figure 4).

d) Mechanical Properties. The stress rupture data for γ-γ'-Cr_3C_2 are compared with those for currently available materials in Figure 5. The absolute comparison of 1000 h rupture stresses, relevant to stator blade applications, shows a substantial benefit over current stator alloys. A density compensated comparison, of interest in relation to rotating components, shows that γ-γ'-Cr_3C_2 matches most blading alloys in the appropriate stress range. In addition γ-γ'-Cr_3C_2 has adequate impact, fatigue, thermal fatigue, notch sensitivity and transverse creep behaviour.

FERROUS-ALLOYS

Although the NATO nations have a reasonably assured supply of nickel, there would be both cost and technical advantages if iron-base alloys could replace nickel-base superalloys for certain applications. As with γ-γ'-Cr_3C_2, the processing conditions for in-situ composites with M_7C_3 reinforcement in Ni, Co or Fe matrices are broadly compatible with current commercial directional solidification practice, economic solidification rates of ~ 30 mmh^{-1} being possible (9). A typical microstructure of an (Fe, Cr)-Cr_7C_3 in-situ composite solidified at 30 mmh^{-1} is shown in Figure 6. Several studies of such iron-base alloys have been reported (10,11,12) and the current status of these materials is summarised below.

a) Physical Properties. The alloys have low densities (~ 7.3 Mgm^{-2}) and melting temperatures of ~ 1200°C.

b) Oxidation/corrosion resistance. Iron based alloys containing aluminium and chromium together with minor additions of active elements (such as yttrium, cerium or lanthanum) have outstanding oxidation and corrosion resistance but are relatively weak. There has been considerable success in increasing the creep strengths of such materials, by incorporating carbide fibres, while retaining the excellent surface stability. The alloys with ferritic matrices tend to have better oxidation resistance than those with austenitic matrices although the latter have improved creep resistance. Figure 7 indicates the critical role that yttrium plays in improving the oxidation resistance.

c) Mechanical Properties. The stress rupture data are summarised in Figure 8 as plots of the stress to cause rupture in 100 h as a function of temperature. Information on current iron- and nickel-base superalloys are included for comparison. The ferritic in-situ composite (NPL A) shows a very substantial strength advantage over the single-phase FeCrAlY alloy which is finding increasing commercial application. However, the combination of strong fibre and weak matrix would be expected to lead to poor thermal fatigue resistance. Conversion of the matrix to the austenitic form has the dual effects of substantially increasing the rupture strength (Figure 8) and of giving better thermal fatigue performance. Two approaches have been taken to stabilizing the fcc form of the matrix by substituting nickel (11) (NPL B) or manganese (12) (UTRC) for iron. Both lead to similar stress rupture properties with about three times the rupture strength of the ferritic alloy. More importantly, they exceed the performances of advanced austentic ferrous alloys (CRM-6D) and of some nickel-base superalloys (Nimonic 80A, 90). Indeed with suitable density compensation these alloys are comparable to the strongest wrought superalloys (Nimonic 115).

CHROMIUM-FREE ALLOYS

Chromium is, perhaps, the most vital constituent of superalloys imparting the required oxidation/corrosion resistance; however, it is also among the most vulnerable to political or economic pressure. Rapid developments in coating technology, the subject of other sessions at this meeting, give the hope of using chromium-free or low-chromium alloys as the main structure of a component which can then be protected by a corrosion resistant coating. Several studies have been made of in-situ composites in the Ni-Al-Mo (3,13,14) system which show some promise. These range from alloys having superalloy-type γ-γ' matrices reinforced by α-Mo fibres (3,13) to γ'-α (14) eutectics.

a) Processing. These alloys can usually be directionally solidified at relatively high rates (up to 30 or 40 mmh^{-1}) but this requires very high temperature gradients. Thus exploitation of these materials would require substantial modifications to the current commercial directional solidification equipment and procedures. Also the dependence on coatings would introduce a substantial cost penalty.

b) Physical properties. This family of alloys has melting temperatures in excess of 1350°C and densities of about 8.5 Mgm^{-3}.

c) Oxidation/corrosion resistance. The total absence of chromium means that these materials have very poor intrinsic oxidation resistance. Some preliminary work has been carried out to determine their compatibility with coatings, but a great deal of further work would be required before such composite/coating systems could be exploited.

d) Mechanical properties. γ-γ'-α shows a substantial temperature advantage in stress rupture testing over all available directionally solidified and single crystal materials (Figure 9). Harf (13) has demonstrated that suitable heat treatments can give the material adequate shear creep performance and, like most in-situ composites the fatigue properties are very good.

ECONOMIC CONSIDERATIONS

The basic raw material costs of the various alloys described above, at 1980 prices, are listed in Table 2 together with similar information for current and projected rival materials. There is a clear potential cost advantage in all of these materials if they can be shown to be technically acceptable. The most advanced in-situ composites, such as COTAC 744 and NITAC 3-116A are presently much more expensive. However, one must consider the future usefulness of these materials both in terms of specific applications and the potential for savings through multiple service/rejuvenation cycles. Perhaps the most promising application of monocarbide reinforced eutectics is in small aeronautical engines (≤ 1500 kW). Here the use of advanced cooling technology is extremely difficult due to the small dimensions and thin sections of high pressure (HP) blades. It is not surprising that even the most modern small engines generally use solid uncooled blades. The turbine inlet temperatures (TIT) can therefore not be increased substantially as in the case of large gas turbine engines which benefit from the latest cooling technology. However, the present trend towards higher TIT for achieving a higher specific thrust will either require cooling of these already very thin airfoils or use of materials capable of sustaining higher temperatures. Belaygue (15) has discussed and analysed in detail the various complications arising from an increase in TIT from 1050° to 1200°C which will necessitate the introduction of cooled blades in these small engines if conventional materials are used.

Since some advanced eutectic composites give a temperature gain of about 75°C over presently available DS superalloys and about 50°C over current single crystal alloys, the use of uncooled eutectic blades for such specialised applications may be viable. Examples of such components are shown in Figure 11.

COMPONENT RECYCLING

Another interesting property of certain monocarbide reinforced eutectics allows the original creep performance to be regenerated after service exposure. Therefore the overall economics of these basically expensive materials must take into account the life that can be achieved by multiple service/regeneration cycles.

The creep deformation of composites consisting of refractory fibres entrained in a relatively weak matrix is quite different from that in superalloys. When the fibres and matrix have widely different melting temperatures as, for example, NbC or TaC in nickel (eg in COTAC and NITAC alloys) it is unlikely that both phases will creep. Indeed transmission electron microscopy shows no carbide dislocations after creep at temperatures up to 1100°C. Rather, in service conditions the fibres, operating at very low homologous temperatures, will deform elastically gradually off-loading stress from the matrix to the fibres. The elastic deformation of the fibres is recoverable by heat treatment. This effect was first proposed and demonstrated by Khan et al (1) who designated it the "length memory effect".

Specimens of COTAC 744, whose microstructure is shown in Figure 12, were creep tested at various temperatures and stresses to about 1.2% strain in 200 to 300 hours. Previous work had shown that fibre fracture did not occur until about 1.3 to 1.6% strain. The specimens were re-heat treated without load (20 mins at 1200°C, AC + 16 hours at 850°C/AC) and observed to contract to virtually their original lengths as documented in Table 3. On re-testing, the material had similar properties to the virgin material. Consequently, the life expectancy of such materials can, in principle, be substantially extended by repeated rejuvenating heat treatments and an effective evaluation of the potential of advanced *in-situ* composites should take this into account. Clearly, the number of cycles that can be used in practice will depend on the dominance of other factors such as corrosion or fatigue crack growth.

Not all *in-situ* composites fully exhibit the length memory effect. For example, heat treatment of creep tested γ-γ'-Cr_3C_2 results in recovery of only part of the creep strain, the extent of recovery being greatest at low temperatures (Figure 10). This is probably due to the relatively high homologous temperature of Cr_3C_2, particularly at 825°C, resulting in creep of the fibres.

An important consequence of the stress carrying capacity of elastic fibres is that established extrapolation procedures, such as Larson-Millar plots, that have evolved for conventional engineering alloys, will always underestimate the long term properties of *in-situ* composites. In a recent theoretical treatment, McLean (16) has shown that for elastic fibres in a creeping matrix there is:

a) no steady state deformation

b) a decreasing creep rate that approaches zero when all of the stress is carried by the fibres

c) a break-down in the linear relationship between log (stress) and log (creep rate at 1% extension) or log (time to 1% extension) at low stresses where the creep performance is much better than indicated by data extrapolation.

CONCLUSIONS

Although *in-situ* composites have so far failed to meet the combined economic and technical targets for premier components such as HP1 turbine blades, at least some alloys appear to offer less direct benefits that could be of importance in the event of disruptions of supplies of critical metals

a) γ-γ'-Cr_3C_2 is a cobalt free alloy with good oxidation resistance and comparable strength to most existing superalloys.

b) Several iron-base composites with M_7C_3 reinforcement having no or little nickel, combine good oxidation resistance with the creep performance of many nickel-base superalloys.

c) γ-γ'-α is a high strength chromium-free alloy that may have uses in combination with protective coatings.

In addition, the potential of the most advanced composites, such as NITAC3-116A and

COTAC 744 (or their derivatives), may not be fully appreciated for the following reasons:

1) Time-dependent deformation in alloys with highly refractory fibres is totally recoverable so that the original properties can be re-established several times by heat treatment.

2) Extrapolation of short-term data always provides a pessimistic estimate of the long term performance of in-situ composites.

3) Introduction of cooled HP blades in small engines (< 1500 kW) may not be a practical way of increasing the overall engine efficiency and consequently the higher temperature capabilities of some eutectic composites may offer an attractive solution.

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Table 1. Compositions of in-situ composites (wt%)

Alloy	Ni	Co	Cr	Al	C	Fe	Mo	W	Ta	Nb	Mn	Y	V	Re
a) $\gamma-\gamma'-Cr_3C_2$	bal	-	12.3	6.9	1.8	-	-	-	-	-	-	0.5	-	-
b) Fe-base														
NPL A (ferritic)	-	-	33.2	3.2	2.6	bal	-	-	-	-	-	0.8	-	-
NPL B (austenitic)	15.0	-	18.2	3.2	2.6	bal	-	-	-	-	-	0.8	-	-
UTRC (austenitic)	-	-	20.0	-	3.2	bal	-	-	-	-	10.0	-	-	-
c) $\gamma-\gamma'-\alpha$	bal	-	-	6.0	-	-	32.0	-	-	-	-	-	-	-
$\gamma'-\alpha$	bal	-	-	2.2	-	-	21.0	-	35.9	-	-	-	-	-
d) COTAC 744	bal	10	4	6	0.47	-	2	10	-	3.8	-	-	-	-
NITAC 14B	bal	3.7	1.9	6.5	0.24	-	-	-	8.2	-	-	-	4.2	6.3

Table 2. Raw material costs of in-situ composites and conventional high temperature alloys. (1980) prices.

Alloy	Cost £/Kg	
$\gamma-\gamma'-Cr_3C_2$	5.3	in-situ composites
Fe-base		
NPL A	1.5 - 4.0	
NPL B		
UTRC		
$\gamma-\gamma'-\alpha$	9.5	
$\gamma'-\alpha$	53.7	
COTAC 744*	12.5	
NITAC 14B*	>20	
IN 738LC	11.3	superalloys
IN939	14.0	
IN 100	11.4	
Mar M002	16.3	
X-40	25.2	

* The low yield of COTAC and NITAC alloy components due to slow processing rates and the small number of blades produced per cycle may lead to a substantial premium on the processing stage over the cost of DS and single crystal superalloy castings.

Table 3. Improvement in stress rupture life of COTAC 744 after repetitive heat treatments

Creep test conditions		Gauge length mm					Time to rupture, h	
Temp °C	Stress MPa	Initial	1st creep	Heat treat	2nd creep	Heat treat	cumulative	un-interrupted
850	400	30.02	30.40	30.06	30.42	30.10	1040	700
900	310	29.98	30.36	30.08	30.35	30.08	1303	700
1000	220	30.00	30.36	30.06			302	200

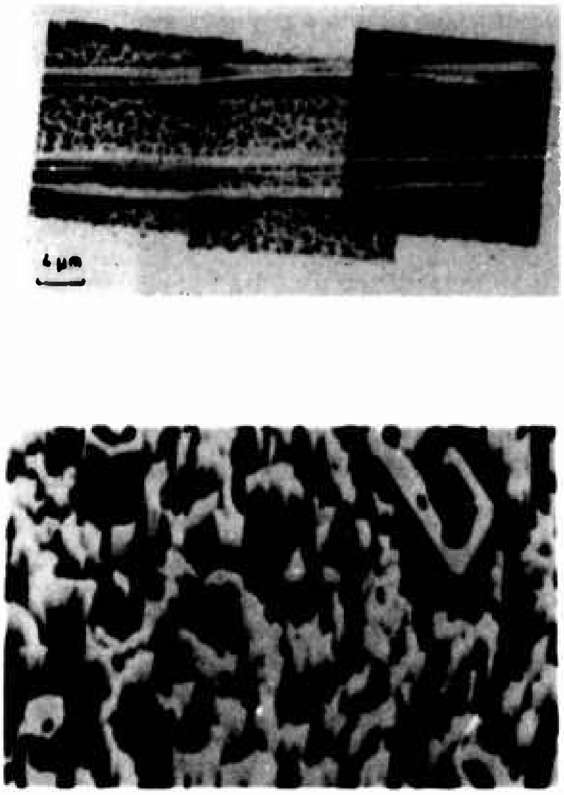

Figure 1. Micrographs of (a) longitudinal and (b) transverse sections of γ-γ'-Cr_3C_2 directionally solidified at 300 mm h^{-1} in a temperature gradient of ~ 13 Kmm^{-1}.

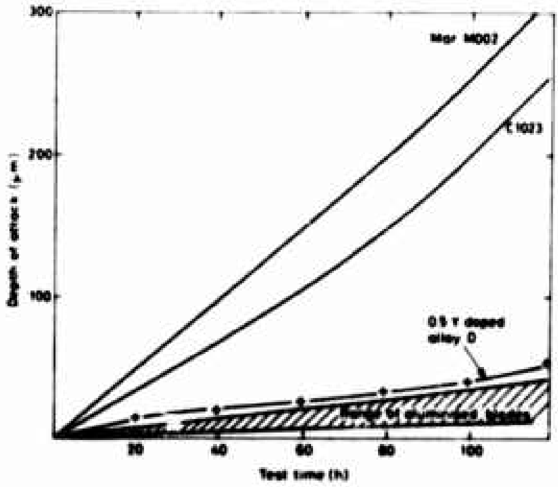

Figure 3. Depth of penetration of corrosion products during burner rig tests at 1323 K with 4 ppm salt.

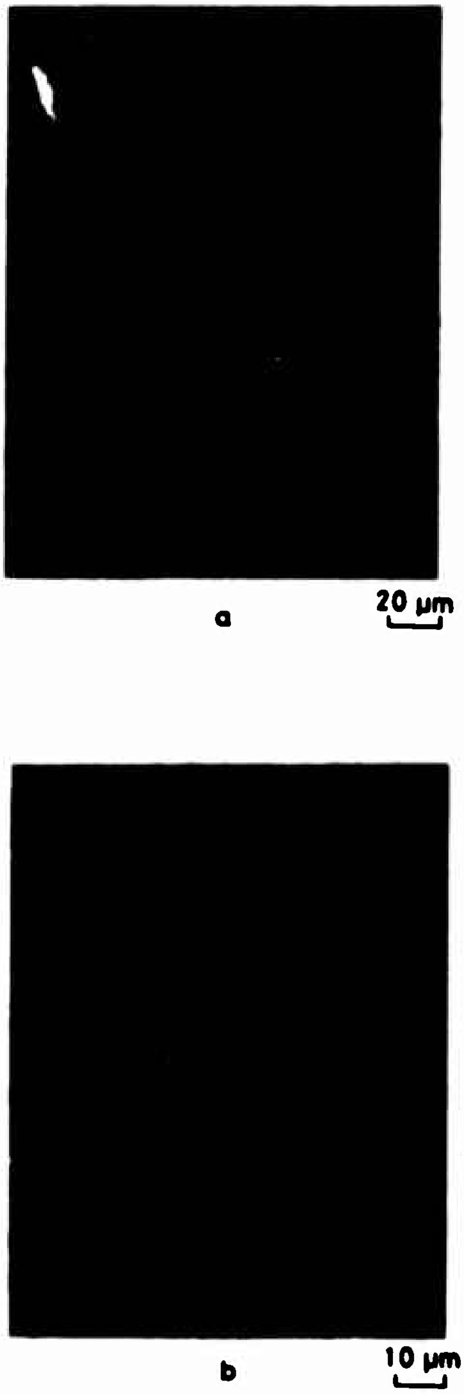

Figure 2. Transverse sections of γ-γ'-Cr_3C_2 directionally solidified in (a) a conventional silica-bound shell mould and (b) recrystallised alumina.

Figure 4. Comparison of sections of (a) γ-γ'-Cr_3C_2 and (b) IN713LC after burner rig tests at 850°C for 300 h.

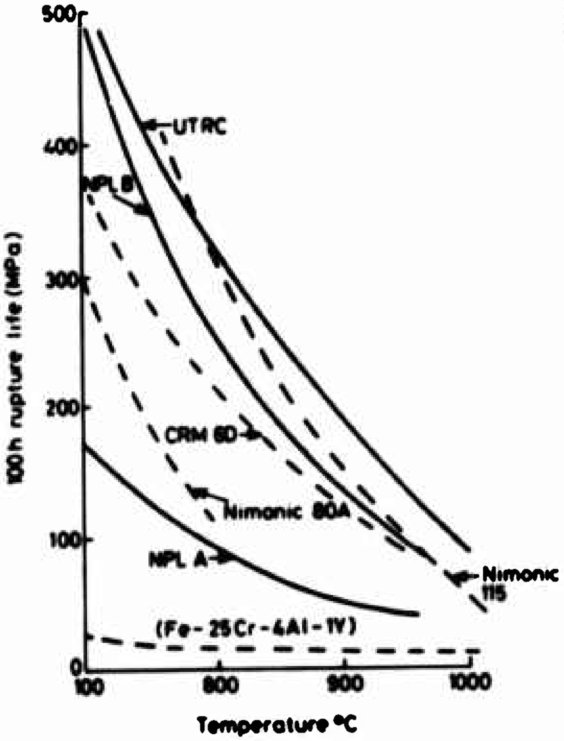

Figure 8. Stresses to cause rupture in 100h as a function of temperature for ferrous in-situ composites. Data for conventional iron- and nickel-base alloys are included for comparison.

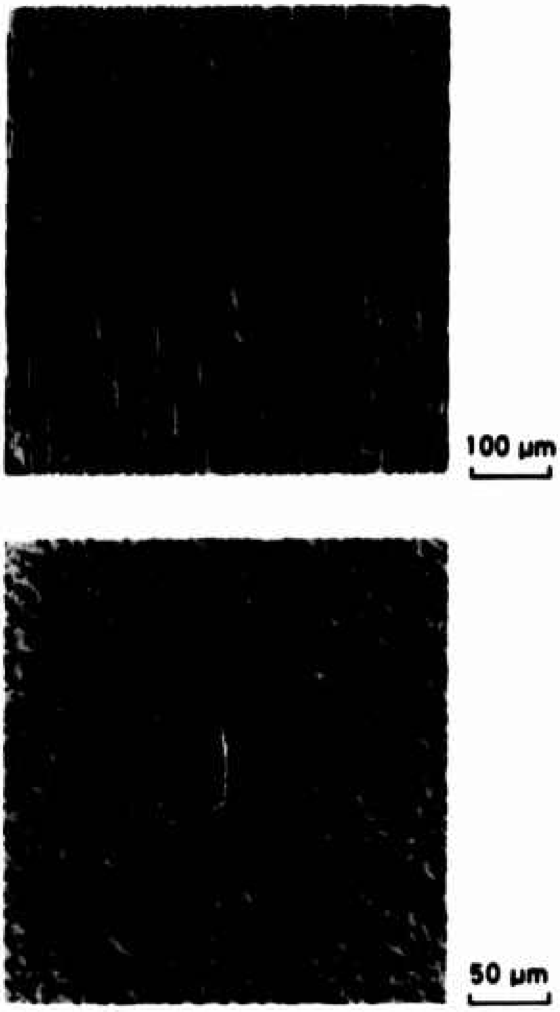

Figure 6. Micrographs of (a) longitudinal and (b) transverse sections of the FeCrAlY-Cr_7C_3 in-situ composite (NPL A) directionally solidified at 300 mmh^{-1}.

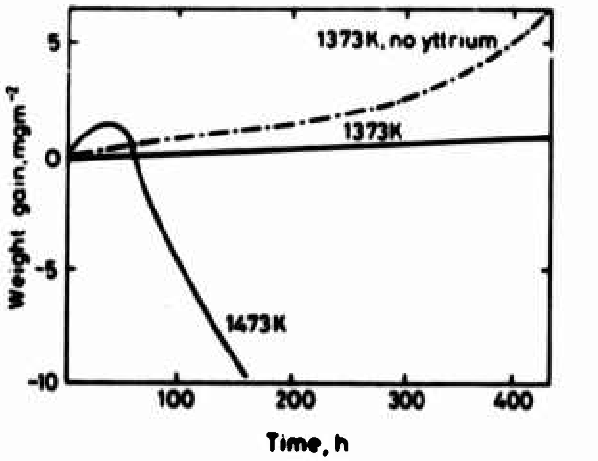

Figure 7. Comparative weight changes during cyclic oxidation testing of ferrous in-situ composites.

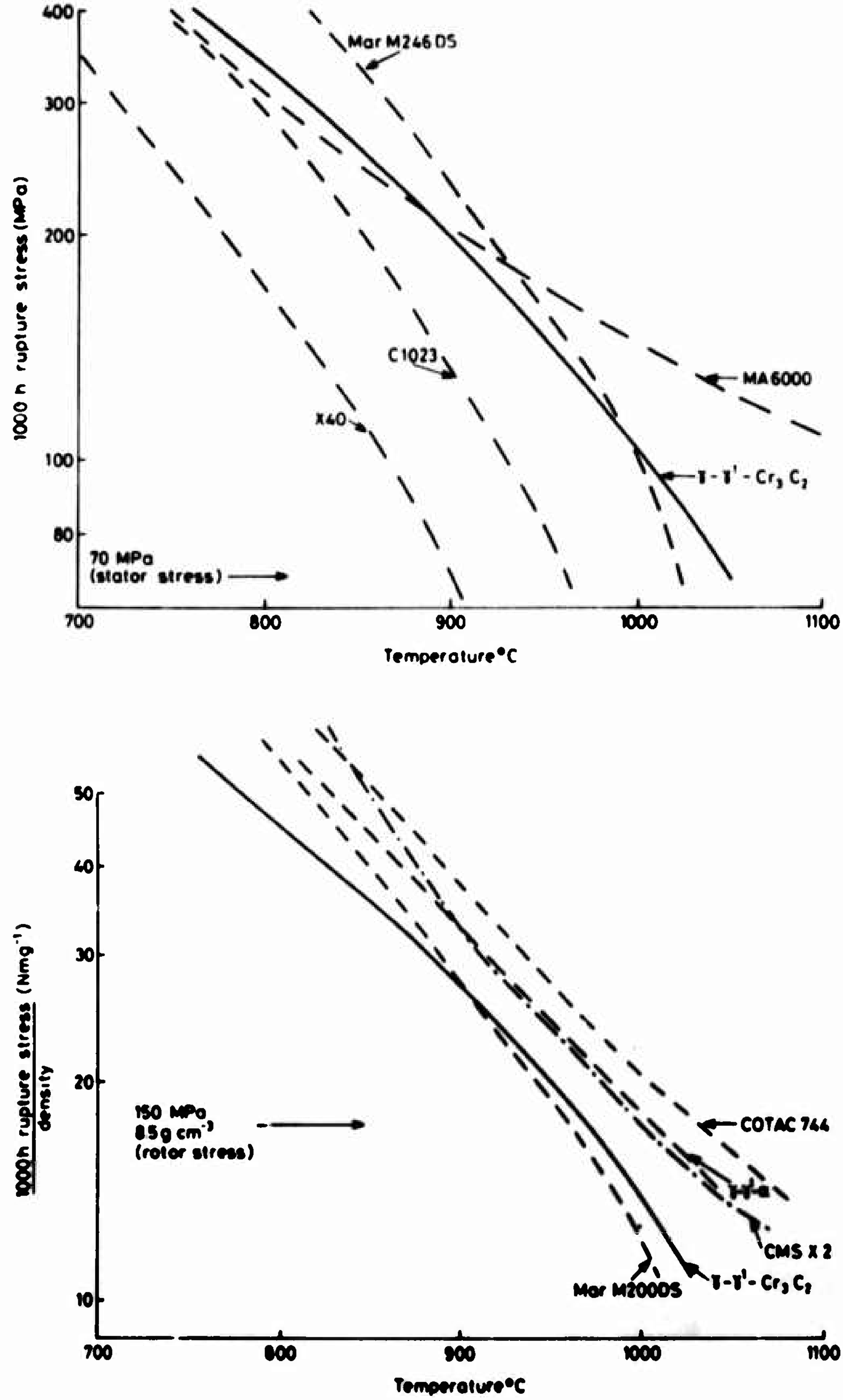

Figure 5. Stresses to cause rupture in 1000 h as a function of temperature for γ-γ'-Cr_3C_2. Data are shown for rival superalloys and <u>in-situ</u> composites for comparison (a) absolute stresses (b) density compensated stresses.

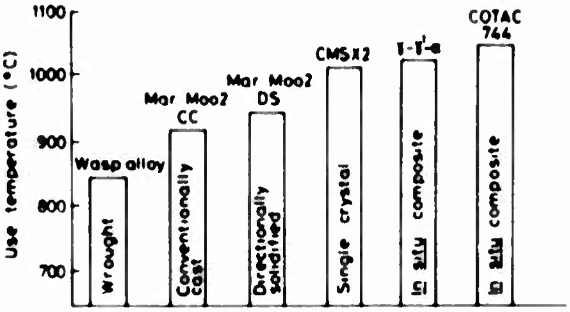

Figure 9. Potential rotor operating temperature for γ-γ'-α in relation to rival alloys.

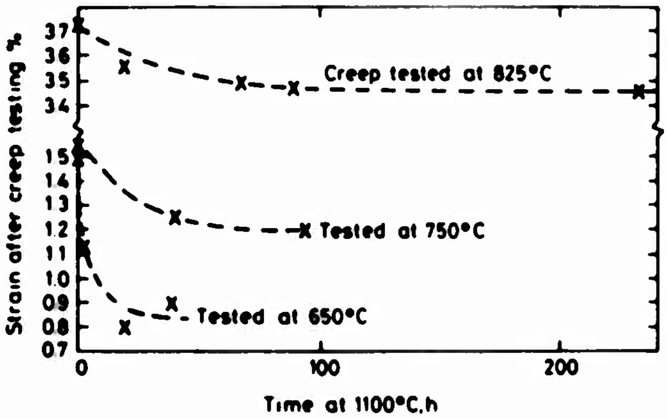

Figure 10. Change in length with time of creep specimens of γ-γ'-Cr_3C_2 during unloaded heat treatment at 1100°C after different prior test conditions.

Figure 11. Finished uncooled blades of the COTAC 74 <u>in-situ</u> composite.

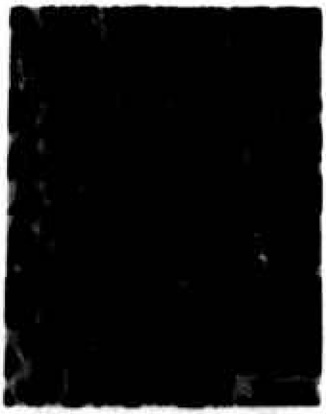

Figure 12. Typical microstructure of a γ-γ' monocarbide reinforced COTAC eutectic (transverse section of COTAC 74 showing NbC fibres embedded in a γ-γ' matrix).