AD-A125 345 COPPERHEAD ALTERNATE MANUFACTURING PROCESS(U) MARTIN MARIETTA AEROSPACE ORLANDO FL G I FRIEDMAN NOV 81 DAAK10-78-C-0070 1/2

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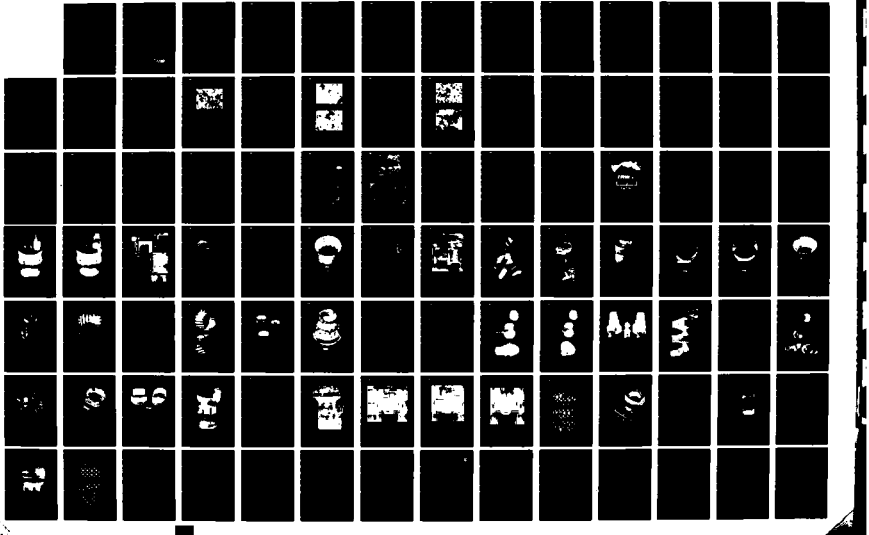

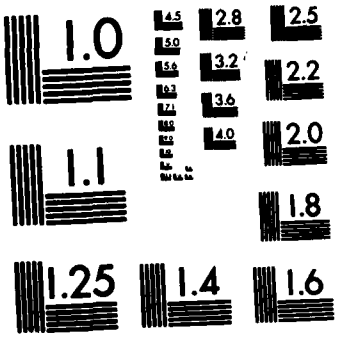

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Gov't Contract DAAK-10-78-C-0070 Vol. I
Cross Reference with Volume I,
"Application of Powdered Metal
Technology to Produce Titanium Gyro
Parts", Mar 82, AD No. A118064

# COPPERHEAD ALTERNATE MANUFACTURING PROCESS

**MARTIN-MARIETTA CONTRACT No. 740907**

**FINAL REPORT**

**NOVEMBER 1979 TO SEPTEMBER 1981**

**G. I. FRIEDMAN**

**Prepared for:**
**MARTIN-MARIETTA AEROSPACE**
**ORLANDO DIVISION**
**ORLANDO, FLORIDA**



**NOVEMBER 1981**



**TRW**
**EQUIPMENT**
**MATERIALS TECHNOLOGY**

ER-8192

# COPPERHEAD ALTERNATE MANUFACTURING PROCESS

Martin-Marietta Contract No. 740907

Final Report

November 1979 to September 1981

Gerald Friedman

November 1981

TRW Materials Technology
TRW Inc.
23555 Euclid Avenue
Cleveland, Ohio 44117

Prepared for:

Martin-Marietta Aerospace
Orlando Division
Orlando, Florida

Gov't Contract DAAK-10-78-C-0070 Vol. II
Cross Reference with Volume I,
"Application of Powdered Metal
Technology to Produce Titanium Gyro
Parts", Mar 82, AD No. A118064

## FOREWORD

This document is the final report under Martin-Marietta Aerospace Contract 740907 and covers work performed from November 1979 through September 1981.

The program was assigned to the Powder Technology Section of TRW Materials Technology under Mr. J. N. Fleck, Section Manager. Engineering responsibility resided with Mr. G. Friedman. Contributions were made by the following: Messrs. L. B. Jones and C. A. Tyndall, Specimen Fabrication (powder blending, briquetting, and sintering), and Mr. D. J. Engeman, Metallography.

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_____
T. S. Piwonka
Department Manager

## ABSTRACT

A demonstration was made of manufacturing five Copperhead gyroscope components via titanium powder metallurgy techniques. Commercially pure titanium was used for one part; the remaining four parts were made of the Ti-6Al-4V alloy, which was produced through the heat treatment of a blend of master alloy powder with the titanium. Two parts were made by isostatic compaction techniques in urethane rubber bags; steel mandrels were used to precisely define the interior details. Three parts were made by die pressing followed by sintering. Of these, one was left as-sintered, one was hot forged, and one was subsequently HIP (hot isostatically pressed). Test bars representing the different material-process combinations were used to demonstrate that the chosen combination provided adequate mechanical properties for the intended applications.

The strongest samples were made of Ti-6Al-4V which had been forged; these had an ultimate tensile strength of 950 MPa (138 ksi) with 5% elongation. The best combination of properties for Ti-6Al-4V was achieved in samples that were HIP after sintering. They had ultimate strengths in excess of 895 MPa (130 ksi) with 12% elongation. Pure titanium samples that were sintered and HIP had tensile strengths of 550 MPa (80 ksi) with over 20% elongation. Samples which were tested under dynamic (high-g) conditions exhibited tensile strengths 10-25% higher than comparable samples tested at one g.

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# 1.0 INTRODUCTION

## 1.1 Concept

This program was performed to demonstrate the feasibility of manufacturing five titanium Copperhead gyroscope components--the base, gotcha lock, gimbal ring, inner gimbal, and actuator housing--by P/M (powder metallurgy) techniques. The reason for doing so was a TRW cost analysis that concluded that a considerable savings could be made over the present practice of machining these components from bar, while retaining or enhancing the properties desired in this application. The program has been successful in meeting its goals, which were:

1. Demonstrate that a "machining preform" for each part could be made, such that one or more difficult-to-produce surfaces would require little or no machining.

2. Demonstrate, through an economic analysis based on the process routings developed in the program, that P/M parts represent a considerable saving over machined-from-bar parts.

3. Achieve considerable savings in input weight of titanium for each part.

4. Select a material-processing combination that achieves the required mechanical properties for each part.

5. Deliver sixty-six pieces of each of the five components: six pieces to demonstrate that a manufacturing process had been arrived at, twelve additional pieces for a process demonstration and subsequent testing by Martin-Marietta Corporation, and forty-eight pieces for inventory. In one case (gimbal ring), the machining preform was functionally satisfactory but did not meet blueprint dimensions; in the case of the gotcha lock, some early inventory pieces had defective teeth. For both these cases, a simple tool or process modification, as described below, would suffice to correct the problem in production.

## 1.2 Program Plan

Four material-process combinations were planned for the five gyro parts:

| | CP Ti | Ti-6Al-4V |
|---|---|---|
| Die Press | Actuator Housing | Gimbal Ring<br>Inner Gimbal |
| Isostatic Press | Gotcha Lock | Base |

The composition of the five parts was determined by Martin-Marietta to satisfy requirements of strength and corrosion resistance. The selection of the primary consolidation technique, i.e., die pressing vs. isostatic pressing, was made by TRW on the basis of the configuration of the individual parts. The thinness of the gotcha lock wall with respect to its height and the hemispherical bowl at the top of the base made these two parts natural candidates for isostatic pressing, whereas the relative simplicity of the gimbals and actuator housing dictated die pressing for them.

The program was planned so that the following steps would occur in the order indicated:

1. Order titanium and master alloy powders.
2. Design and order forge die nest and die inserts.
3. Characterize the titanium, master alloy, and blended powders.
4. Determine density and shrinkage for CP Ti and Ti-6Al-4V as a function of processing conditions.
5. Design and order briquetting tools for the three die-pressed parts (actuator housing, gimbal ring, inner gimbal).
6. Prepare and evaluate test bars representing the significant material-process combinations.
7. Prepare for Martin-Marietta evaluation thirty test bars for each of three major material-process combinations.
8. Develop a manufacturing process for each part and deliver six of each of the gyro parts to Martin-Marietta as proof.
9. Conduct a process demonstration and deliver twelve of each gyro part.
10. Produce 48 pieces of each part.

# 2.0 PROCESS DESCRIPTION

## 2.1 Ti P/M - A Discussion

The basis for this work is the fact that, in the industrial production of titanium ingot, there is an intermediate product known as sponge fines. The "sponge" is formed through the reaction of liquid titanium tetrachloride with magnesium, sodium or calcium. The fines occur as a result of breaking up the titanium sponge to wash it prior to consolidating it into an ingot for consumable-electrode arc melting. The fines are washed, dried, graded and sold as commercial-purity titanium powder (CP/Ti). This powder is soft, irregular in shape, of moderately high purity, and is one half to one third as expensive as titanium bar, thus making it a good P/M raw material. On the debit side, the powder contains a residual amount of alkali metal chloride trapped within the particles; unlike the sponge processed into ingot, the sponge fines have not been arc melted, and therefore there has been no opportunity to drive off the chlorine.

Because of its irregular shape, CP titanium powder has a low apparent density, only 20% that of solid titanium. With tapping, the powder density can be increased about 10%; manipulation of the powder particle size distribution can also influence the powder density so that it is possible to achieve a "tap" density of about 35% of theoretical. Depending upon the means and pressure employed, cold consolidation will transform the loose powder mass to a green compact with a density up to 90% of theoretical. This compact can be quite strong and, at densities above $\approx$80%, can be machined.

The next stage in the processing of P/M titanium is sintering, which is usually performed in vacuum, at $10^{-5}$ to $10^{-4}$ torr. During the sintering of sponge-fines titanium, three things occur:

1. The compact density increases proportionally with the temperature and time employed.

2. The boundaries between individual particles disappear as a result of diffusion, i.e., particles diffusion bond to one another.

3. The elemental mixture of powders is converted to a true alloy wherein all locations have the same composition. Thus an alloy such as Ti-6Al-4V can be made by adding an aluminum-vanadium master-alloy powder to the sponge fines in the ratio 90% Ti-10% (60Al-40V).

Depending on sintering temperature and time (in the range of 1200-1260°C 2-4 hours), the density of sintered CP Ti or Ti-6Al-4V will be between 90 and 97% of true density. The range of density between approximately 86% and 94% is of great significance. Below 86% density, all pores are interconnected; above 94% density, all pores are isolated, and in the range 86-93% there is, therefore, a continuum starting at no closed pores and ending with all closed pores. When a sintered body has no interconnected porosity, it can be subjected without

fear of internal oxidation to the same high temperature and/or high pressure processing conditions that are used for fully dense, wrought bodies. Therefore, if it is useful or necessary to change the shape of the sintered body by hot forging, the piece can be coated with lubricant and heated in air. If it is desired to further densify the sintered body without changing its shape, then it can be HIP (Hot Isostatic Pressed) at 930°C (1700°F) under argon gas pressure at 100 MPa (15,000 lb/in$^2$) to collapse virtually all the remaining pores. Both forged and HIP sintered bodies are over 99% dense.

In summary, the density progression in Ti P/M is as follows:

1. Loose powder - 20-25% of theoretical density
2. Tapped powder - 30-35% " " "
3. Green compact - 75-90% " " "
4. Sintered compact - 80-97% " " "
5. Forged compact - >99% " " "
6. HIP compact - >99% " " "

## 2.2 Powder Characteristics

Two hundred pounds of Grade 020 sponge fines CP Ti were purchased from the RMI Company, Ashtabula, Ohio. Its composition is shown in Table I; note the 0.1% sodium chloride content. Table II presents the particle size distribution, apparent density, and tap density for this powder. In contrast to Nuclear Metals' prealloyed titanium powders,which have a size range of 500 to 50 μm, all of this is finer than 150 μm; 16% is finer than 44 μm. In Figure 1 the titanium particle size distribution is plotted as a cumulative percent finer than the indicated screen size. $D_{50}$ denotes the size mid-range, i.e., half the particles in this powder lot are finer than 85 μm. Particle morphology is illustrated in Figure 2.

Nine kilograms (20 lb) of 60 Al-40V master alloy, finer than 6-1/2 mm (1/4 inch) were purchased from Reading Alloys, Reading, PA. The composition of the Al-V and its as-received size distribution are shown in Table III. In order to blend the master alloy with the matrix powder, it is important for the master alloy to be as fine as possible. Fineness not only enhances the uniformity of mix but aids in sintering and promotes interdiffusion of the two metal species. Accordingly, the fraction of powder coarser than 10 mesh was removed, and using a specially prepared ball mill, the Al-V was ground into fine powder (Figure 3 and Table IV). A comparison of Figures 1 and 3 clearly shows the difference in morphology of the two powders.

The Al-V was added to the CP Ti in the ratio 90 Ti-10(Al-V); the characteristics of a typical blend are illustrated by Figures 4 and 5, a scanning electron micrograph and a size distribution curve, respectively.

## TABLE I

Composition of RMI Ti-020, -100 Mesh CP Ti Powder, Lot 12179-33

| | Percent | | | | | | |
|---|---|---|---|---|---|---|---|
| Ti | Fe | Na | Cl | C | O | H | N |
| Balance | 0.032 | 0.085 | 0.046 | 0.032 | 0.15 | 0.067 | 0.014 |

## TABLE II

Size and Density of RMI Grade 020, -100 Mesh CP Ti Powder

| Size | | Fraction Retained | |
|---|---|---|---|
| US Mesh | µm | Percent | Cumulative Percent |
| 80 | 180 | 0.33 | 0.33 |
| 100 | 150 | 1.42 | 1.75 |
| 140 | 106 | 25.6 | 27.3 |
| 200 | 75 | 32.3 | 59.6 |
| 270 | 53 | 20.2 | 79.8 |
| 325 | 44 | 3.9 | 83.7 |
| Pan | <44 | 16.3 | 100.0 |

Apparent Density: 0.962 $g/cm^3$ = 21% Theo.
Tap Density: 1.293 $g/cm^3$ = 29% Theo.

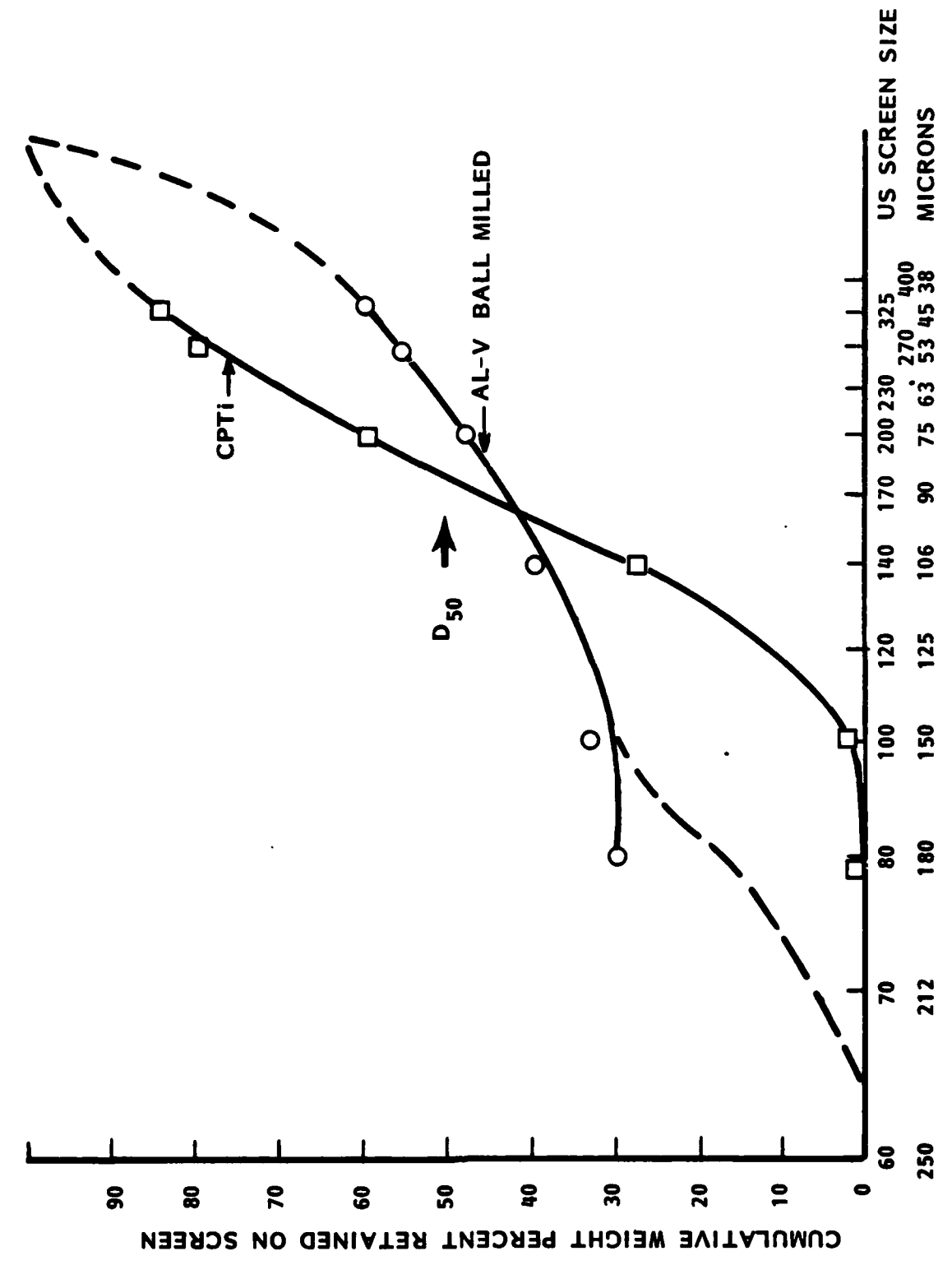



FIGURE 1

PARTICLE SIZE DISTRIBUTION FOR C.P. Ti & [60% Al-40%V] POWDERS

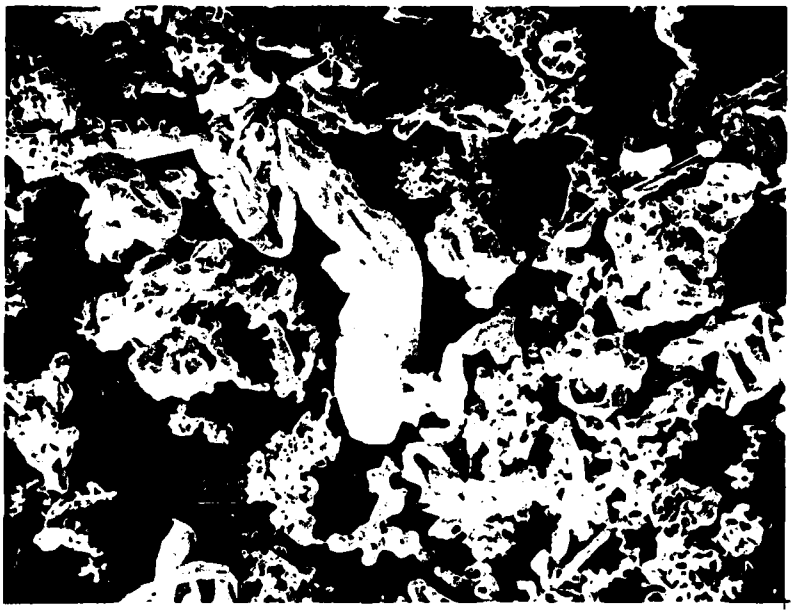


Figure 2. RMI Ti-020, -100 Mesh CP Ti Powder, SEM.

TABLE III

# READING ALLOYS, INC.

MANUFACTURERS OF FERROUS AND NON-FERROUS MASTER ALLOYS

Robesonia, Pa. 19551 • Phone (215) 693-5822

Telex: 83-6422

Mr. Gerald Friedman
TRW, Inc.
TM3044
23555 Euclid Avenue
Cleveland, OH 44117

## CERTIFICATE OF ANALYSIS

DATE 5 November 1979

ORDER NO. 88X800346 (Complete) ALLOY 60% ALUMINUM - 40% VANADIUM

SHIPPED: 7 November 1979 LOT NO. 33-194 (28 lbs. alloy)

CHEMISTRY:

| | | | |
|---|---|---|---|
| Al | 55.55% | | |
| V | 43.62% | | |
| B | 0.0016% | Ni | 0.002% |
| C | 0.05% | P | 0.007% |
| Cr | 0.007% | Si | 0.23% |
| Cu | 0.004% | S | 0.002% |
| Fe | 0.34% | Ti | 0.02% |
| Pb | 0.002% | W | <0.005% |
| Mg | 0.003% | | |
| Mn | 0.008% | | |
| Mo | 0.05% | | |

SPECTROGRAPHIC:

GASES:

| | |
|---|---|
| H | 0.0027% |
| N | 0.007% |
| O | 0.068% |

SCREEN:

| | | | | | |
|---|---|---|---|---|---|
| - 1/4" | x + | 4 mesh | = | 11.05% |
| - 4 mesh | x + | 5 mesh | = | 8.15% |
| - 5 mesh | x + | 7 mesh | = | 17.34% |
| - 7 mesh | x + | 20 mesh | = | 43.46% |
| - 20 mesh | x + | 30 mesh | = | 7.48% |
| - 30 mesh | x + | 50 mesh | = | 10.62% |
| - 50 mesh on pan | | | = | 1.90% |
| | | TOTAL | = | 100.00% |

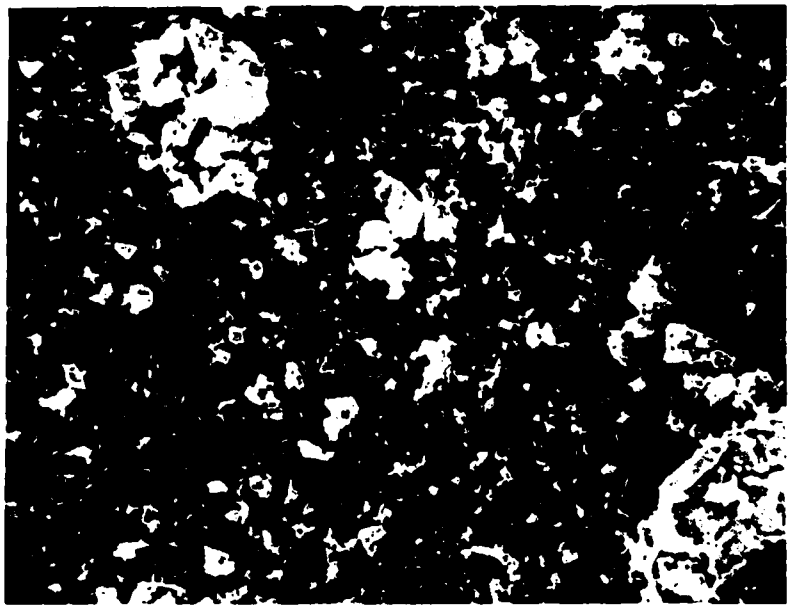
SCREEN:

C: TRW
Mr. Friedman - 2 + S.N.
W/Shipment - 1
R.A. Files - 2
(5559)

READING ALLOYS, INC.

*"Metals Progress Through Applied Research"*

RA8)



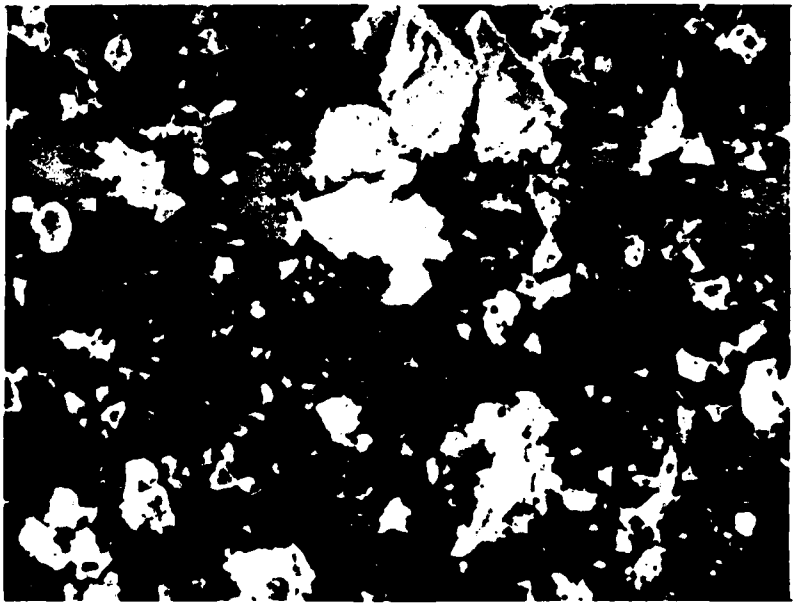


Figure 3. Ball-milled Al-V, SEM.

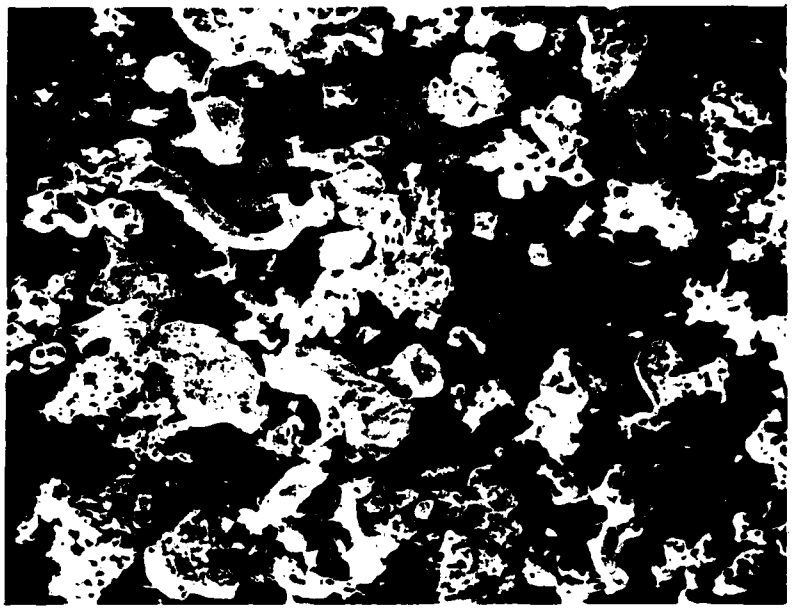
TABLE IV

Particle Size Distribution and Density of Ball Milled Al-V Powder

| Screen Size | | Fraction Retained | |
|---|---|---|---|
| US Mesh | μm | Percent | Cumulative Percent |
| 70 | 212 | 0 | 0 |
| 80 | 180 | 0.33 | 0.33 |
| 100 | 150 | 1.42 | 1.75 |
| 140 | 106 | 25.59 | 27.34 |
| 200 | 75 | 32.26 | 59.60 |
| 270 | 53 | 20.17 | 79.77 |
| 325 | 45 | 3.89 | 83.66 |
| Pan | <45 | 16.34 | 100.00 |

Apparent Density: 0.962 $g/cm^3$ = 22% Theo.

Tap Density: 1.27 $g/cm^3$ = 29% Theo.



100X

500X

Figure 4. Blend of 90% Ti - 10% (60Al-40V) Powder SEM.

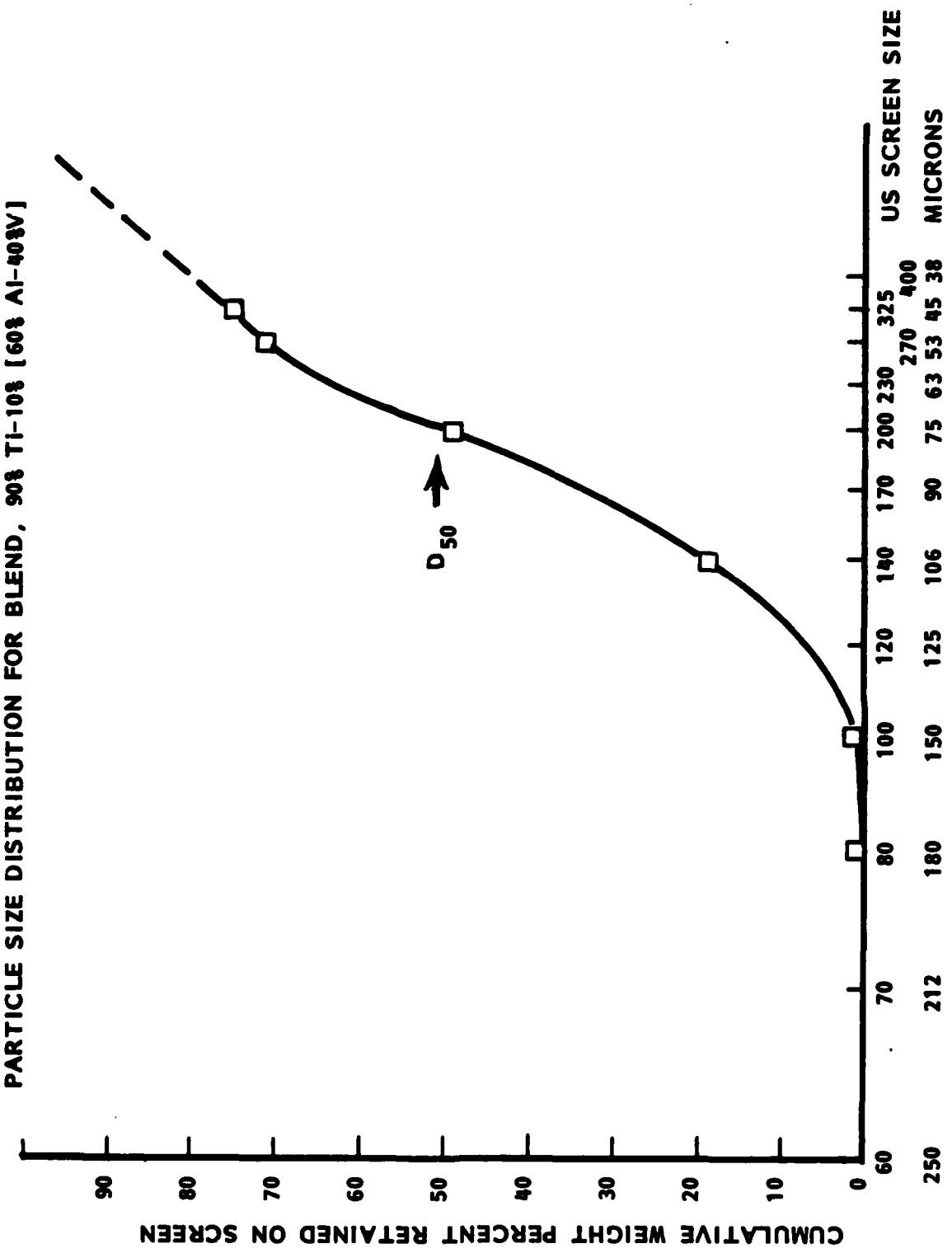



FIGURE 5
PARTICLE SIZE DISTRIBUTION FOR BLEND, 90% TI-10% [60% Al-40%V]

## 2.3 Consolidation Behavior

### 2.3.1 CP Ti

As noted, sponge fines titanium is a relatively soft, compressible material. In order to establish the relationship between consolidation conditions and compact density, a series of CP Ti test bars were made. The bars were briquetted in a floating-die sub-press that produces specimens 60 mm long x 15 mm wide; 900 $mm^2$ (2.353 in. long x 0.596 in. wide; 2 $in^2$). Since titanium pressure welds to steel, the die wall was lubricated by a solution of zinc stearate in acetone before every pressing; the acetone was allowed to evaporate before the powder was added to the die. Sintering was carried out in vacuum in the pressure range of $10^{-4}$ to $10^{-5}$ torr. The relationship between compact density and processing conditions for this powder is shown in Table V and Figure 6. Recall that, at densities below approximately 87% of theoretical, all pores are interconnected, while at densities greater than 95%, all pores are isolated. A 96% dense compact has 4% porosity in which none of the pores are interconnected. These relationships assume great significance with respect to the ability of sintered compacts to be further processed by HIP or hot forging. The data indicate that, for CP Ti to attain 95% density, it is necessary to die press at pressures of 480 MPa (35 ton/$in^2$) or above, and sinter at 1260°C; 1100°C sintering achieves the same end-point density for compacts pressed to a higher green density. It should be noted that, because of the absence of die-wall friction in isostatic pressing, higher green densities are achieved for a given consolidation pressure; isostatically pressed bodies are 1-2% more dense than die-pressed bodies.

### 2.3.2 Ti-(6Al-4V)

CP Ti was blended with milled Al-V in the ratio 90:10 and briquetted at pressures from 415-690 MPa (30-50 ton/$in^2$) by die and by cold isostatic compaction. Because of the additional requirement of the sintering cycle to also produce alloying, the temperature range investigated was increased from 1100-1260°C to 1200-1300°C for the four hours. The relationships are shown in Table VI and Figure 7 from which two observations can be made: the powder mixture can easily be pressed to high densities, and the sintering conditions employed produced a decreasing rate of densification with increasing green density, such that the densest green compact, which had been pressed at 690 MPa (50 ton/$in^2$), expanded instead of shrinking.

This unfavorable sintering response was caused by a too-rapid heating rate combined with high initial densities, so that trapped gas in the closed pores expanded the compacts before the gas could escape or react with the metal. When sintering is preceded by an outgassing period at intermediate temperatures, the sintered densities again reflect a proportionality to consolidation pressure, green density, and sintering temperature. For example, Ti-6Al-4V test bars, isostatically pressed at 415 and 550 MPa (30 and 40 tons/$in^2$), when outgassed at 850°C, sinter to 96 and 97% of theoretical density, respectively, at 1300°C.

TABLE V

Density of CP Ti as a Function of Consolidation Pressure (Die Pressing) and Sintering Temperature, Percent of Theoretical

| Sintering Temp., °C (4 Hour Cycle P = $<10^{-4}$ millitorr) | Consolidation Pressure, MPa (tons/in$^2$) | | | |
|---|---|---|---|---|
| | 275(25) | 415(30) | 550(40) | 690(50) |
| | Density, Percent of Theoretical | | | |
| As Pressed | 83.8 | 86.4 | 90.1 | 93.2 |
| 1100 | 91.1 | 93.3 | 95.7 | 97.5 |
| 1200 | 92.1 | 93.8 | 95.9 | 97.6 |
| 1300 | 92.1 | 93.7 | 95.8 | 97.3 |

FIGURE 6

DENSITY OF RMI GRADE Ti-020, -100 MESH C.P. Ti
AS A FUNCTION OF CONSOLIDATION PRESSURE AND SINTERED TEMPERATURE
(ALL SINTERING IN VACUUM FOR 4 Hr, $P=1.5\times10^{-5}-3.2\times10^{-4}$ TORR)

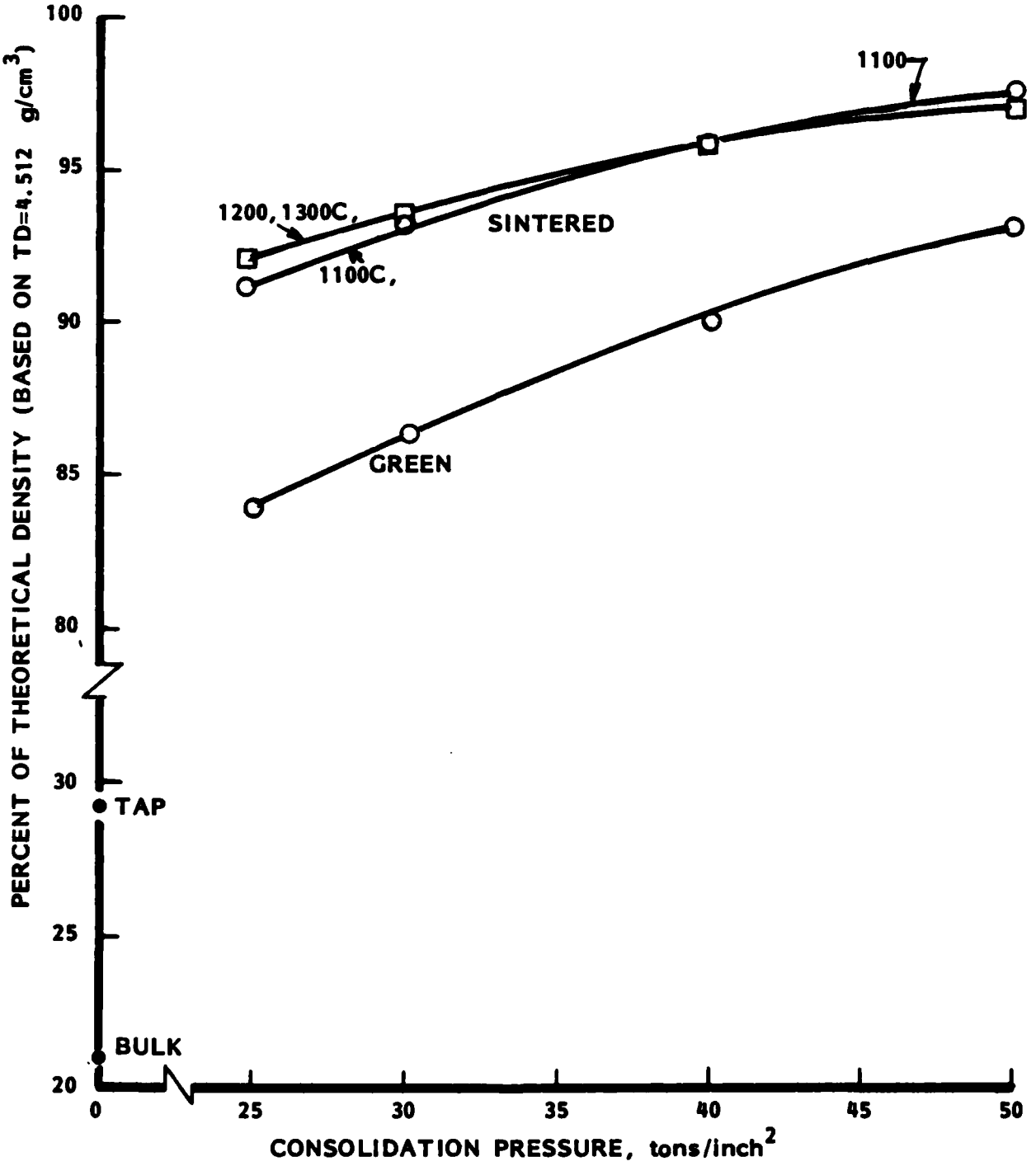


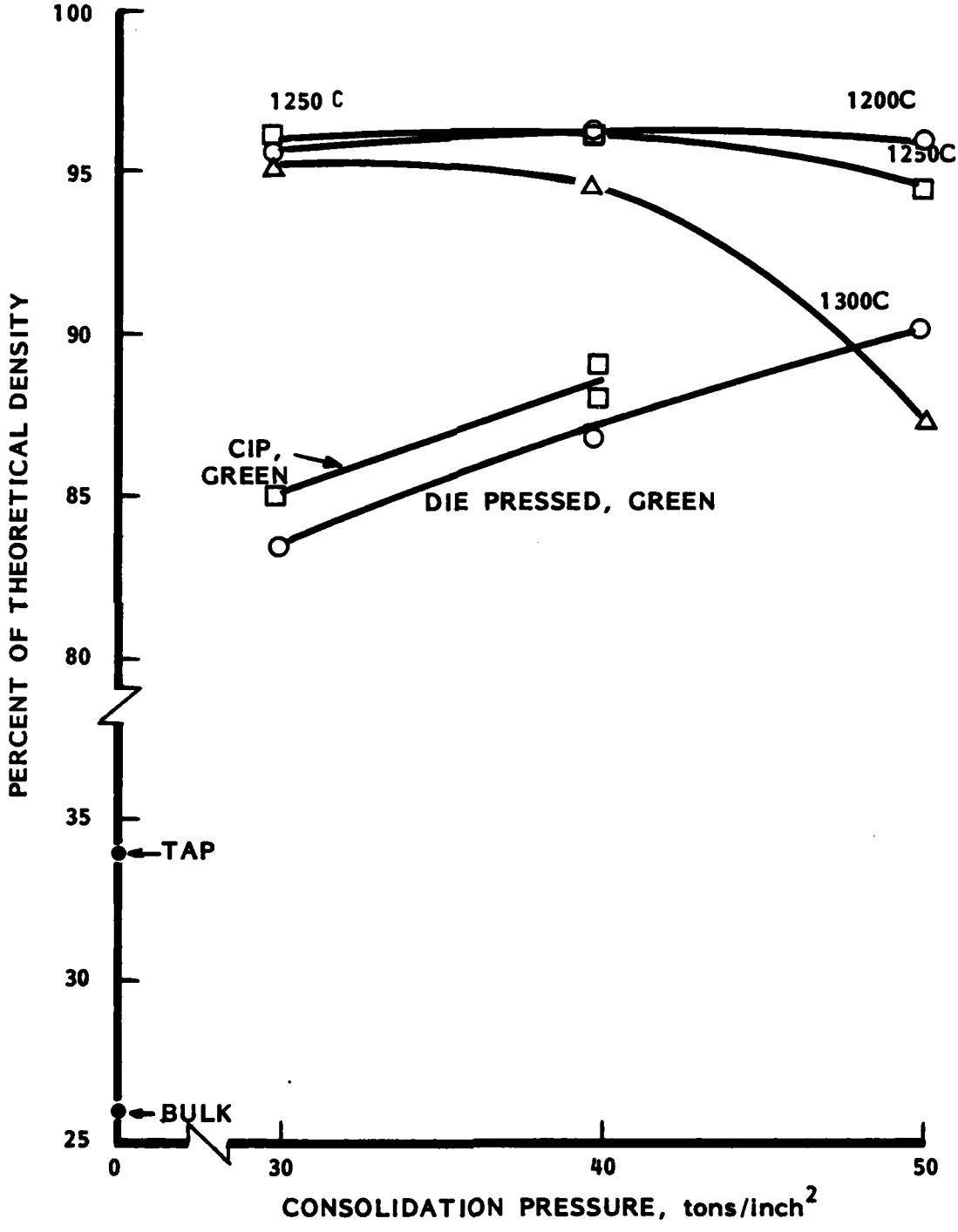
TABLE VI

Density of Ti-6Al-4V as a Function of Consolidation Pressure (Die Pressing) and Sintering Temperature

| Sintering Temp., °C (4 Hour Cycle P = $<10^{-4}$ torr) | Consolidation Pressure, MPa (tons/in$^2$) | | |
|---|---|---|---|
| | 415(30) | 550(40) | 690(50) |
| | Density, Percent of Theoretical | | |
| As Pressed | 83.5 | 86.9 | 90.1 |
| 1200 | 95.7 | 96.2 | 95.9 |
| 1250 | 95.9 | 96.2 | 94.7 |
| 1300 | 95.1 | 94.6 | 87.4 |

FIGURE 7

DENSITY OF Ti-[6AL-4V] BLENDED POWDER
AS A FUNCTION OF CONSOLIDATION PRESSURE AND SINTERED TEMPERATURE
(VACUUM SINTERED FOR FOUR HOURS @ P=1.5x10$^{-5}$ -3.2x10$^{-4}$ TORR)



## 2.4 Post-Sinter Operations

Three post-sinter operations--coining, HIP and forging--were deemed useful or necessary. These will be discussed in general terms first and, more specifically, in connection with the descriptions of individual part processing.

### 2.4.1 Coining

Coining is a room temperature sizing operation performed in hard tool steel or carbide tools for the purpose of imparting dimensional precision to a sintered part. The initial forming or briquetting die is deliberately made undersize so that the sintered part will fit into the coining die. The coining die is made to virtually blueprint dimensions, i.e., except for an allowance for a very small size change due to springback after the part is ejected from the die, the coining die has the same dimensions as the desired part. A coined part is sometimes resintered to improve properties, but such an operation adds uncertainty to the part dimensions.

The actuator housing, a part subjected to only low stress and consequently to be made from soft, ductile CP Ti, was considered to be a good candidate for coining. A series of discs were pressed, sintered, coined, and re-sintered to determine the size and density changes that would accompany these operations (see Tables VII, VIII and IX). These data show: 1) the higher the sintered density the less density increase is achieved by coining; 2) in the density range of interest, i.e., above 95% of theoretical, coining at 690 MPa (50 ton/in$^2$) produces less than 0.4% increased density; 3) the change in sample height by coining is two to three times greater than change in sample diameter; 4) for samples initially 95% or more dense, coining produced lateral movement ranging from 0.7-1.7%; 5) for half the samples, re-sintering had no effect or caused swelling, thus reducing their density. In the case of the remaining samples, however, there were density increases ranging from slight to considerable (2%).

### 2.4.2 HIP

Hot Isostatic Pressing is a high-temperature pressurization treatment in pure argon. It is used to consolidate encapsulated high-density spherical powders or to further densify almost solid objects in which only closed pores remain. Subjecting a 95% dense titanium body to a two-hour HIP cycle at 930°C (1700°F) reduces the remaining porosity to between 0.5-1.0%. In the present case, it was intended to HIP the CP Ti gotcha lock and the Ti-6Al-4V base. No fixtures, coating or cannisters were needed or used to protect the sintered bodies during HIP.

### 2.4.3 Forging

Forging is employed to shape, densify and strengthen the sintered parts. In the case of the gimbal ring, forging provides an upward extrusion of the two long ring walls. At the same time, density is increased to approximately 99% of theoretical; there is also a small amount of grain refinement that results from the metal deformation.

TABLE VII

Density of CP Ti Coined at 50 Ton/in$^2$ as a Function of Initial Consolidation Pressure and Sintering Temperature

| Sintering Temperature °C | Initial Consolidation Pressure, MPa(ton/in$^2$) 340(25) | 415(30) | 550(40) | 690(50) |
|---|---|---|---|---|
| | Sintered Density/Coined Density (Percent Change) | | | |
| 1100 | 91.3/92.0(.8) | 93.4/93.6(.2) | 95.8/96.1(.3) | 97.6/97.6(0) |
| 1200 | 92.1/92.6(.5) | 93.8/94.2(.4) | 96.0/96.3(.3) | 97.5/97.6(.1) |
| 1300 | 92.0/92.6(.7) | 93.7/94.1(.4) | 96.0/96.3(.3) | 97.4/97.7(.3) |

TABLE VIII

Dimensional Change for CP Ti Coined at 690 MPa (50 Ton/in$^2$) as a Function of Initial Consolidation Pressure and Sintering Temperature

| Sintering Temperature °C | Initial Consolidation Pressure, MPa(ton/in$^2$) 340(25) | 415(30) | 550(40) | 690(50) |
|---|---|---|---|---|
| | Percent Change, Diameter/Height | | | |
| 1100 | 2.75/5.97 | 2.29/4.65 | 1.73/4.8 | 1.14/2.25 |
| 1200 | 2.6/5.4 | 2.4/5.0 | 1.7/4.7 | 1.1/2.9 |
| 1300 | 2.1/4.7 | 1.6/3.4 | 0.7/117 | 0.7/1.6 |

TABLE IX

Effect of Post-Coin Sintering at 1300°C, 4 Hours on the Density of CP Titanium

| Initial Sintering Temp. °C | Initial Consolidation Pressure, MPa (ton/in$^2$) | | | |
|---|---|---|---|---|
| | 340(25) | 415(30) | 550(40) | 690(50) |
| | Coined Density/Re-sintered Density (Percent Change) | | | |
| 1100 | 92.0/93.5(2.0) | 93.6/95.1(1.6) | 96.1/96.9(.8) | 97.6/97.5(-.1) |
| 1200 | 92.6/93.0(.4) | 94.2/94.7(.5) | 96.3/96.4(.1) | 97.6/97.6(0) |
| 1300 | 92.6/90(-2.6) | 94.1/92.7(-1.5) | 96.3/95.2(-1.1) | 97.7/96.8(-.9) |

In the context of these P/M titanium gyro parts, forging is an elevated temperature operation performed in a closed-die arrangement. The parts to be forged are coated to minimize oxidation and for insulation, heated in air, and deformed by the action of opposed steel punches while under constraint in a steel die. The die is heated slightly to avoid cracking due to thermal shock, but it is far cooler than the workpiece which, in the case of titanium, may be heated to 700-1040°C (1300-1900°F). A coating, which serves as a lubricant and additional thermal insulator, is applied to the die before each piece is formed.

## 2.5 Property Evaluation

### 2.5.1 TRW Test Data

A series of test specimens, some die pressed into bars 90 mm long x 15 mm wide x 11 mm high (353 x 0.6 x 0.45 in.) and some cold isostatically pressed (CIP)* into 12.5 x 12.5 x 20 mm (0.5 x 0.5 x 8 inch) bars were made and tested at TRW to provide data on porosity and mechanical properties for the different material-process combinations. CP Ti bars were CIP only, representing the combination to be used for the gotcha lock; at the time of testing, the material for the actuator housing was changed from CP Ti to Ti-6Al-4V. Ti-6Al-4V bars were made by CIP and die pressing; some samples were subjected to post-sinter operations of forging and of HIP. Table X lists the results of these tests which show:

1. CP Ti can be CIP + HIP to over 99% density, producing ductile bodies of strength levels essentially equivalent to ASTM B348, Grade 4,
2. The strongest samples were forged Ti-6Al-4V.
3. The most ductile Ti-6Al-4V samples were those that had been die pressed and HIP.

The microstructures of representative specimens are shown in Figure 8.

These micrographs show the transformed beta (Widmanstätten) structure of titanium that has been exposed to temperatures above the alpha/beta transformation temperature, 990°C (1820°F) for Ti-6Al-4V. Particle boundaries have all been obliterated, and the presence of mostly spherical pores indicates a high degree of sintering has taken place. (At an earlier stage in sintering, the inter-particle voids would be angular, reflecting the geometric positioning of particle-next-to-particle.)

### 2.5.2 MMC Test Data

In addition to the samples tested at TRW, ninety test bars were made and delivered to Martin-Marietta for testing. The test bars represented three material-process combinations:

---

* Isostatic compaction of powder performed by sealing the powder into a rubber bag of specific internal configuration and immersing the bag in a high-pressure fluid. A fuller description of this technique is given below in section 3.2.

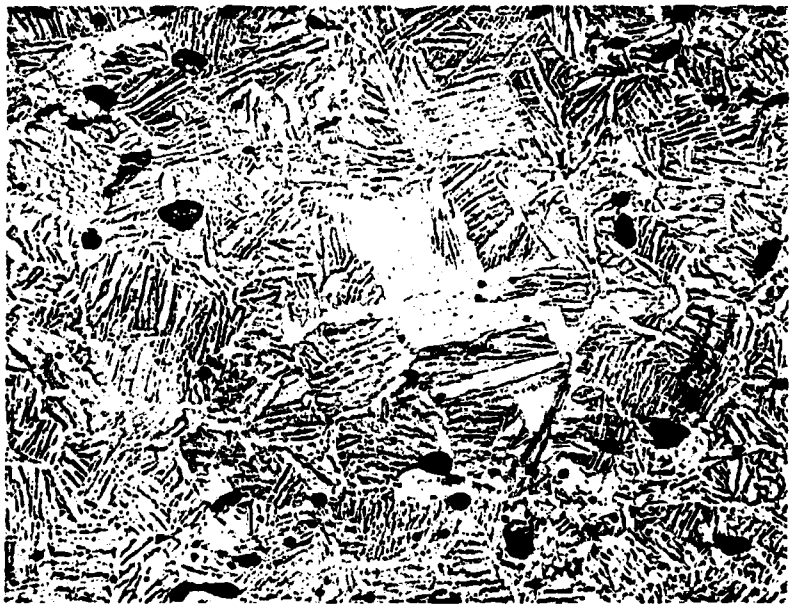
TABLE X

Properties of P/M Sponge Fines Titanium

| S/N | Composition | Consolidation Mode | Consolidation Pressure ton/in$^2$ | Post-Sinter Treatment[1] | UTS MPa (ksi) | .2% Y.S. MPa (ksi) | Elong. % | RA % | Porosity[2] % | $O_2$ % | Na % | Cl % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 100 | CP Ti | CIP | 30 | HIP, 1700°F/15ksi/4 hr | 1101(80) | 860(62) | 22.8 | 29.2 | - | - | - | - |
| 101 | " | " | " | " " " " | 1122(81) | 929(67) | 23.0 | 25.9 | 0.5 | 0.23 | 0.10 | 0.003 |
| 102 | " | " | " | " " " " | 1061(77) | 847(62) | 22.1 | 29.8 | - | - | - | - |
| 103 | Ti-6-4 | DP[3] | 40 | ------- | 1743(126) | 1528(111) | 9.0 | 9.9 | 3.5 | 0.17 | 0.09 | 0.004 |
| 104 | " | " | " | | 1742(126) | 1552(113) | 7.3 | 9.5 | - | - | - | - |
| 98 | Ti-6-4 | DP | 40 | HIP, 1700°F/15ksi/4 hr | 1816(132) | 1626(118) | 13.0 | 19.7 | 0.5 | 0.17 | 0.09 | 0.015 |
| 99 | " | " | " | " " " " | 1812(132) | 1616(117) | 11.5 | 19.1 | - | - | - | - |
| 91 | Ti-6-4 | DP | 30 | Forge, 1200°F/20%+SR[4] | 1756(127) | 1600(116) | 4.9 | 9.8 | - | - | - | - |
| 107 | Ti-6-4 | DP | 30 | Forge, 1350°F/20% Reduct. | 1902(138) | 1775(129) | 5.0 | 8.2 | 1.1 | - | - | - |
| 108 | " | " | " | " " " " | 1911(139) | 1796(130) | 4.7 | 8.0 | - | 0.19 | 0.08 | 0.007 |
| 92 | Ti-6-4 | DP | 30 | Forge, 1500°F/20%+SR | 1750(127) | 1652(120) | 8.0 | 9.8 | - | - | - | - |
| 93 | " | " | " | " " " | 1827(133) | 1506(109) | 5.1 | 8.5 | - | - | - | - |
| 105 | Ti-6-4 | CIP | 40 | ------- | 1819(132) | 1707(124) | 6.3 | 9.5 | 1.8 | 0.2 | 0.07 | 0.002 |
| 106 | " | " | " | | 1550(112) | 1525(111) | 2.6 | 4.6 | - | - | - | - |
| 94 | Ti-6-4 | CIP | 40 | HIP, 1700°F/15ksi/4 hr | 1815(132) | 1634(119) | 7.3 | 14.8 | - | - | - | - |
| 95 | " | " | " | " " " " | 1819(132) | 1503(110) | 9.0 | 16.6 | - | - | - | - |
| 96 | Ti-6-4 | CIP | 30 | HIP, 1700°F/15ksi/4 hr | 1735(126) | 1625(118) | 4.2 | 7.9 | - | - | - | - |
| 97 | " | " | " | " " " " | 1822(132) | 1634(119) | 8.8 | 14.8 | - | - | - | - |

NOTES: (1) All samples were degassed at 850°C and sintered for four hours at 1200°C.
(2) Determined by Omnicon scan of polished specimens using a known-porosity sample for calibration.
(3) Consolidated in a double-acting "floating die."
(4) Stress relieve, 700°C (1300°F)/1 hr/air cool.

60 ksi DP/S/HIP S/N 101



Ti-6-4
80 ksi DP/S S/N 103

Figure 8. Microstructures of P/M Ti and Ti-6Al-4V.

Ti-6-4 S/N 98
80 ksi DP/S/HIP @1700°F

S/N 108 Ti-6-4
80 ksi DP/S/Forge @1350°F

Ti-6-4 S/N 105
80 ksi CIP/Sinter

Figure 8 (continued)

1. CP Ti, isostatically pressed at 415 MPa (30 ton/in$^2$), sintered* and HIP, 1700°F/15 ksi/4 hr.

2. Ti-6Al-4V, die pressed at 550 MPa (40 ton/in$^2$), sintered*, forged 20% at 800°C (1475°F), and stress relieved for one hour at 700°C.

3. Ti-6Al-4V, CIP at 103 MPa (15 ksi), 210 MPa (30 ksi), and 275 MPa (40 ksi), sintered, and HIP, 930°C/103 MPa/4 hr.**

The results of these tests, performed at -15, 20, and 60°C (-25°F, 70°F and 140°F) under static conditions and at 8000 g dynamic conditions, are presented in Table XI.

* Outgas, 850°C, +1200°C, 4 hours.

** 1700°F/15 ksi/4 hr

## TABLE XI

## Mechanical Properties of P/M Titanium at Different Temperatures and Velocities*

| | Composition | Processing Conditions | UTS MPa(ksi) | 0.2% Y.S. MPa(ksi) | Elong. % |
|---|---|---|---|---|---|
| A. Static Tests | | | | | |
| Test Temp. °C(°F) | (1) CP Ti | CIP+HIP | | | |
| -15 (-25) | | | 664(96) | 558(81) | 21.3 |
| +20 (+70) | | | 566(82) | 470(68) | 20.9 |
| +60 (+140) | | | 498(72) | 408(59) | 19.8 |
| | (2) Ti-6Al-4V | DP + Forge | | | |
| -15 (-25) | | | 1050(152) | 978(142) | 7.3 |
| +20 (+70) | | | 925(134) | 874(127) | 6.7 |
| +60 (+140) | | | 883(128) | 820(119) | 6.0 |
| | (3) Ti-6Al-4V | CIP + HIP | | | |
| -15 (-25) | | | 1050(152) | 985(143) | 13.2 |
| +20 (+70) | | | 946(137) | 861(125) | 7.5 |
| +60 (+140) | | | 881(128) | 806(117) | 5.3 |

B. Dynamic (high g) Tests

(1) CP Ti CIP + HIP Apparent UTS = 690 MPa (100 ksi)

(2) Ti-6Al-4V DP + Forge
Apparent UTS = 1066 MPa (155 ksi)
@8570g

(3) Ti-6Al-4V CIP + HIP
Apparent UTS = 970 MPa (141 ksi)
@8780g

* Property values are the average of 7 tests.

# 3.0 DEVELOPMENT OF MANUFACTURING PROCESSES FOR FIVE GYRO COMPONENTS

## 3.1 Actuator Housing, P/N 408MP

The machined actuator housing is shown in Figure 9. Figure 10 shows the initial design for the P/M machining preform. However, after discussions with the tool designers selected to manufacture the briquetting tools, it was decided that the 0.035 inch (0.9 mm) wide lever arm (on the right) was too narrow to permit good flow of powder into this region of the die. The design was, therefore, modified to that shown as 408B, Figure 11. A CP Ti disc was then briquetted (i.e., cold pressed), machined (in the green state) to the shape and size of Drawing 408B MP, and traced at 10X using an optical comparitor. The compact was then sintered and returned to the comparator, where the new, smaller outline was traced inside the earlier one. This drawing, Figure 12, was then sent to the tool vendor* who used it to construct the outline of the briquetting and the coining dies shown in Figures 13 and 14.

A dozen parts were pressed, sintered, and measured. Pressing was performed in a 690 MPa (50 ton) hydraulic press. The output of a pressure transducer connected to the oil line of the press was fed to a strip-chart recorder, thus providing a means of recording both the compaction and the ejection forces, as shown in Figure 15. It was found that only by using a low compaction pressure, 415 MPa (30 ton/in$^2$), and a high sintering temperature, 1260°C (2300°F), could the sintered part be fitted into the coining die, and even then a small amount of filing was required in order for the piece to drop into the die. At about this time, TRW was advised by Martin-Marietta of a design change for this part, which required that it be made of Ti-6Al-4V. It was then found that,with the change in composition, the parts were now too strong to flow laterally in the coining die. It was, therefore, agreed between Martin-Marietta and TRW that the Ti-6Al-4V version of the part would be made as-sintered.

Since the briquetting die had been made to produce a "coining preform," i.e., an undersize part, the briquetting tools were reworked. In addition to increasing the width of the tools, they were also modified to entirely eliminate the corner where the lever arm meets the long curve (Figure 16), since die fill was still a slight problem in this region.

The modified tool performed satisfactorily and was used to make six (Process Demonstration), twelve (Process Verification), and forty-eight (Prototype Production) actuator housing machining preforms.

A process description for the manufacture of the actuator housing machining preform is included as Appendix A.

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* Major Gauge & Tool Company, Livonia, MI.

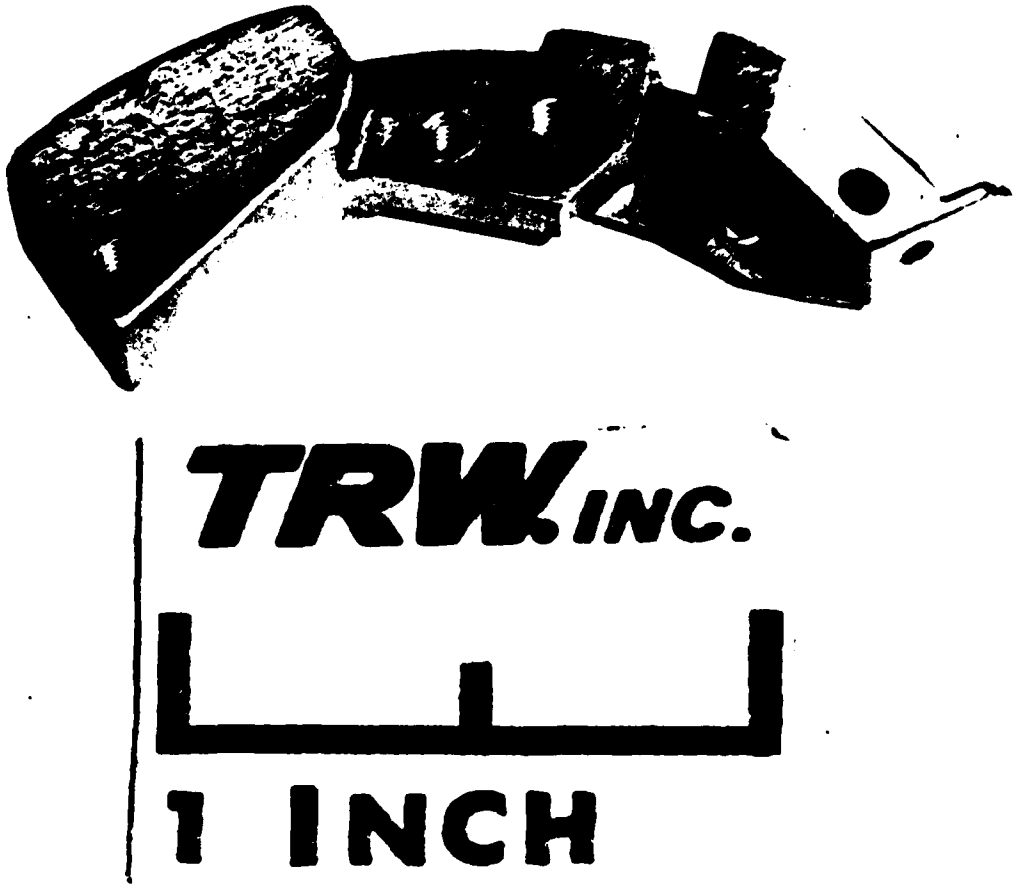



Figure 9. Actuator Housing, P/N 93066408.

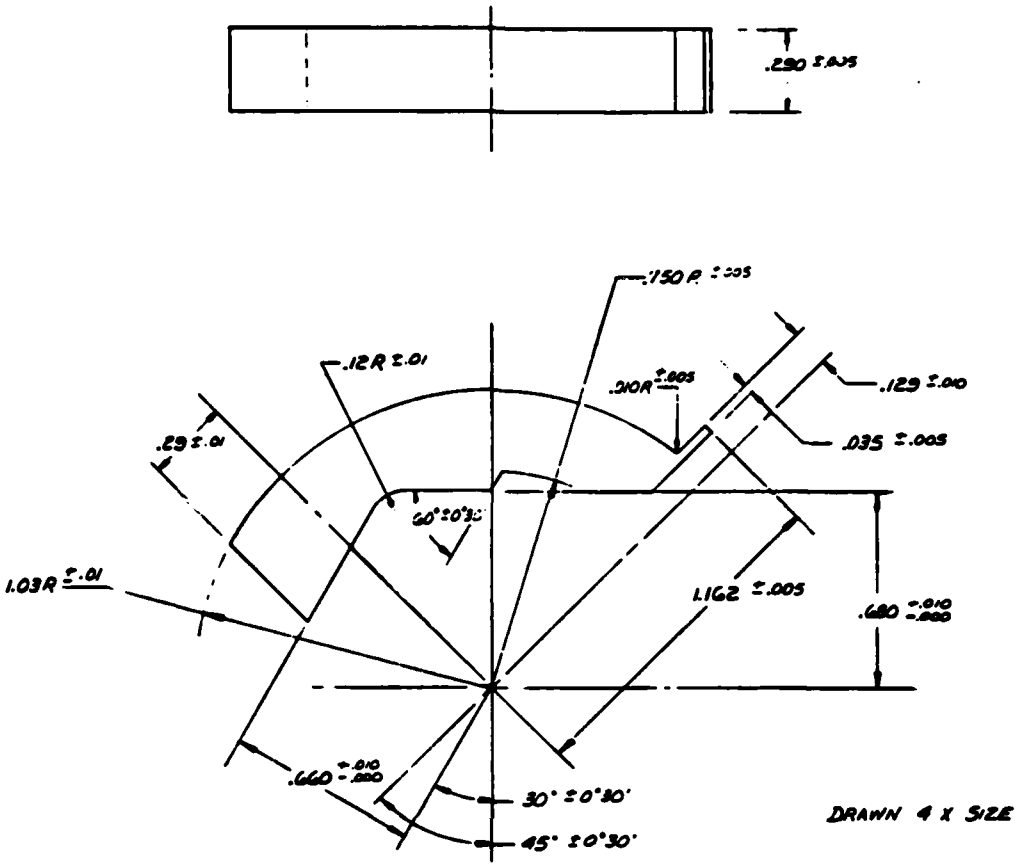



Figure 10. Actuator Housing Machining Preform, 408A.

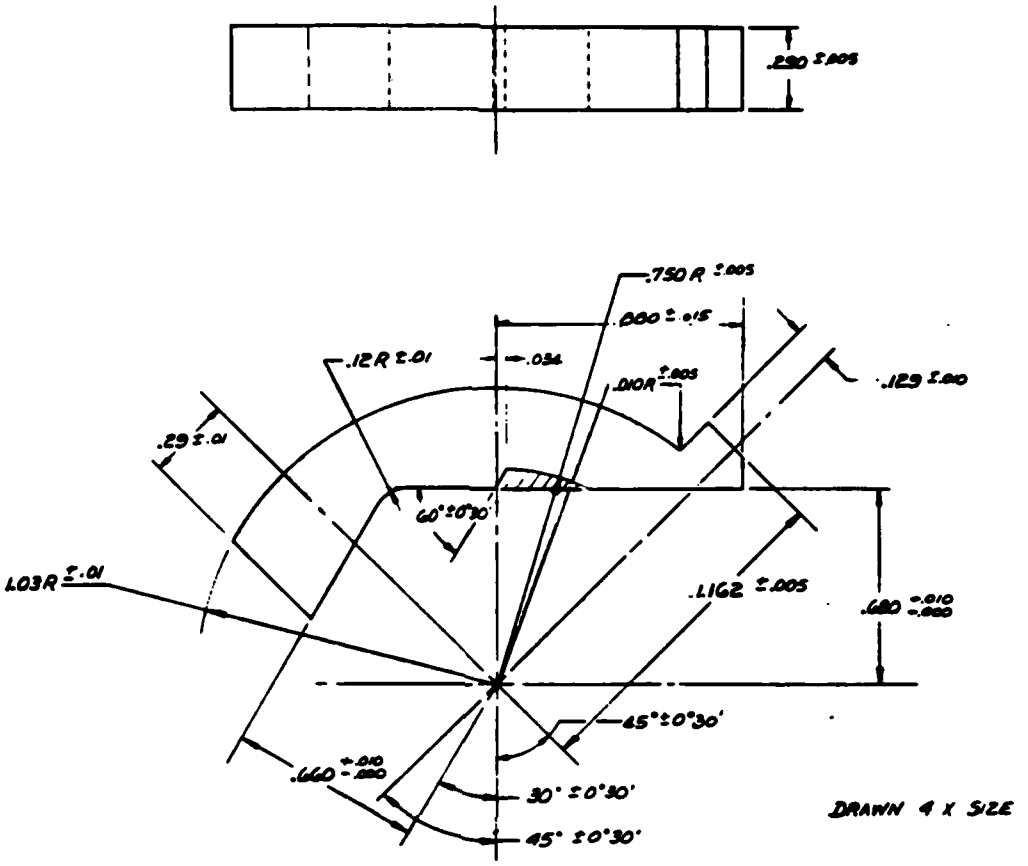



Figure 11. Actuator Housing Machining Preform, 408B

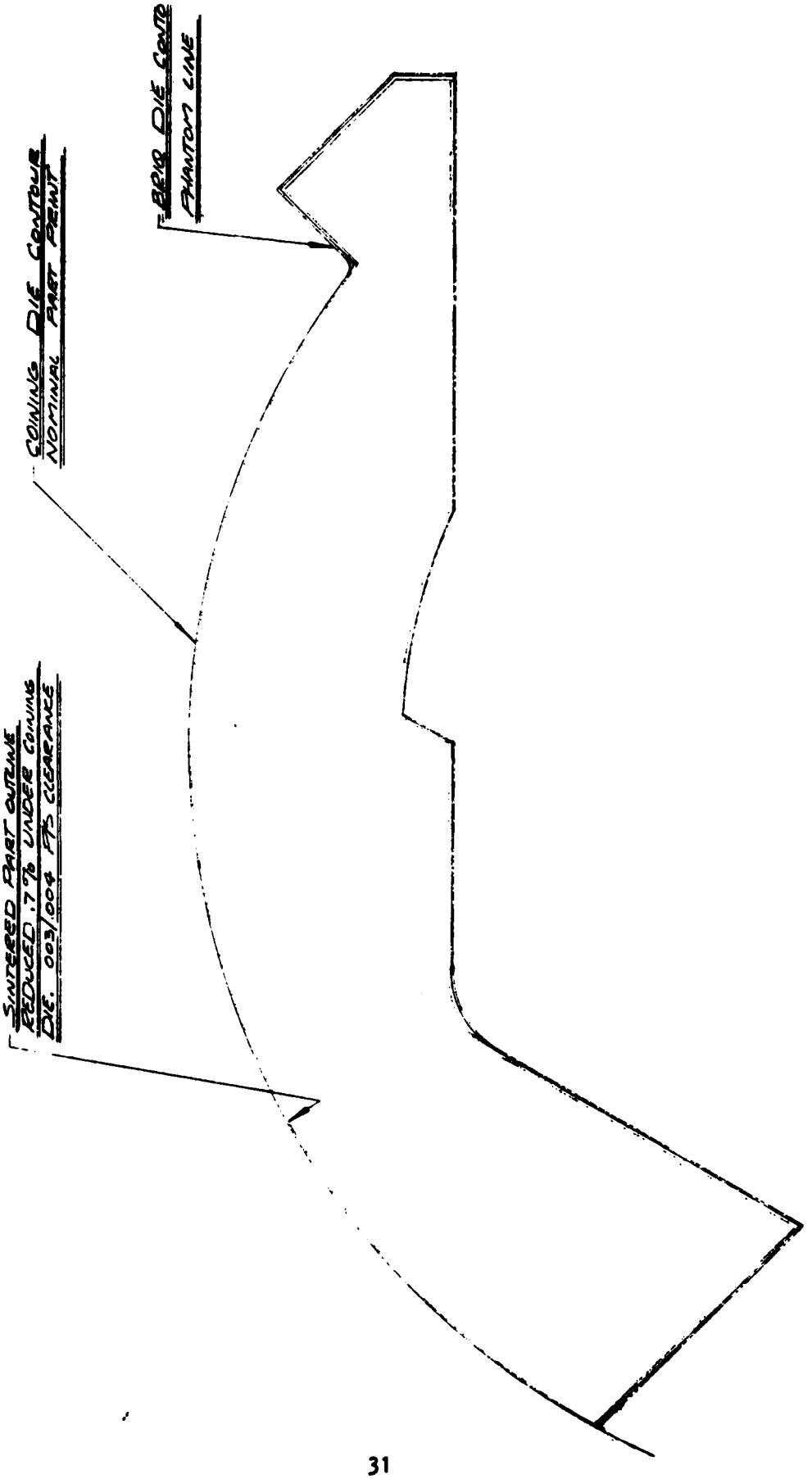



Figure 12. Actuator Housing Machining Preform Die Outlines

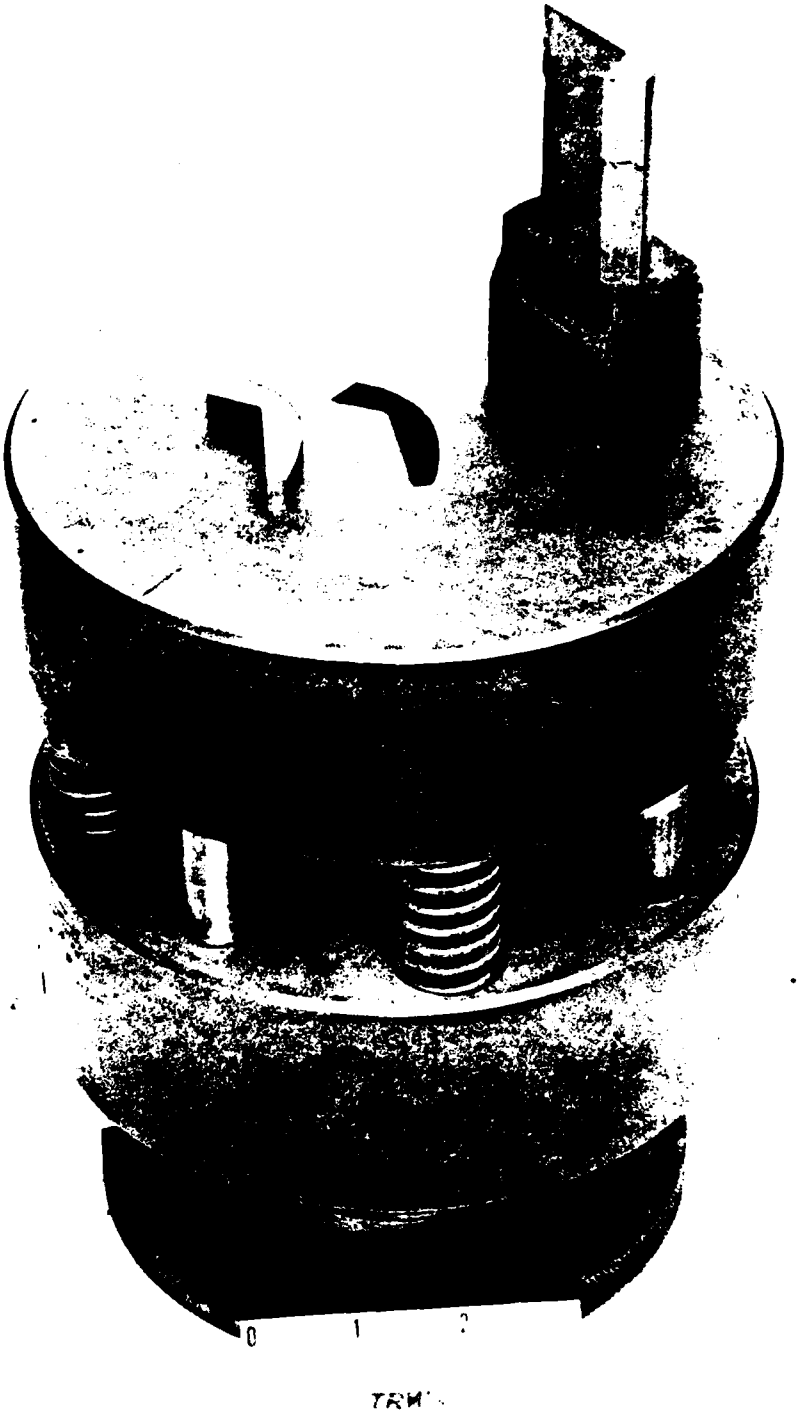


Figure 13. Actuator Housing Machining Preform Briquetting Tool.

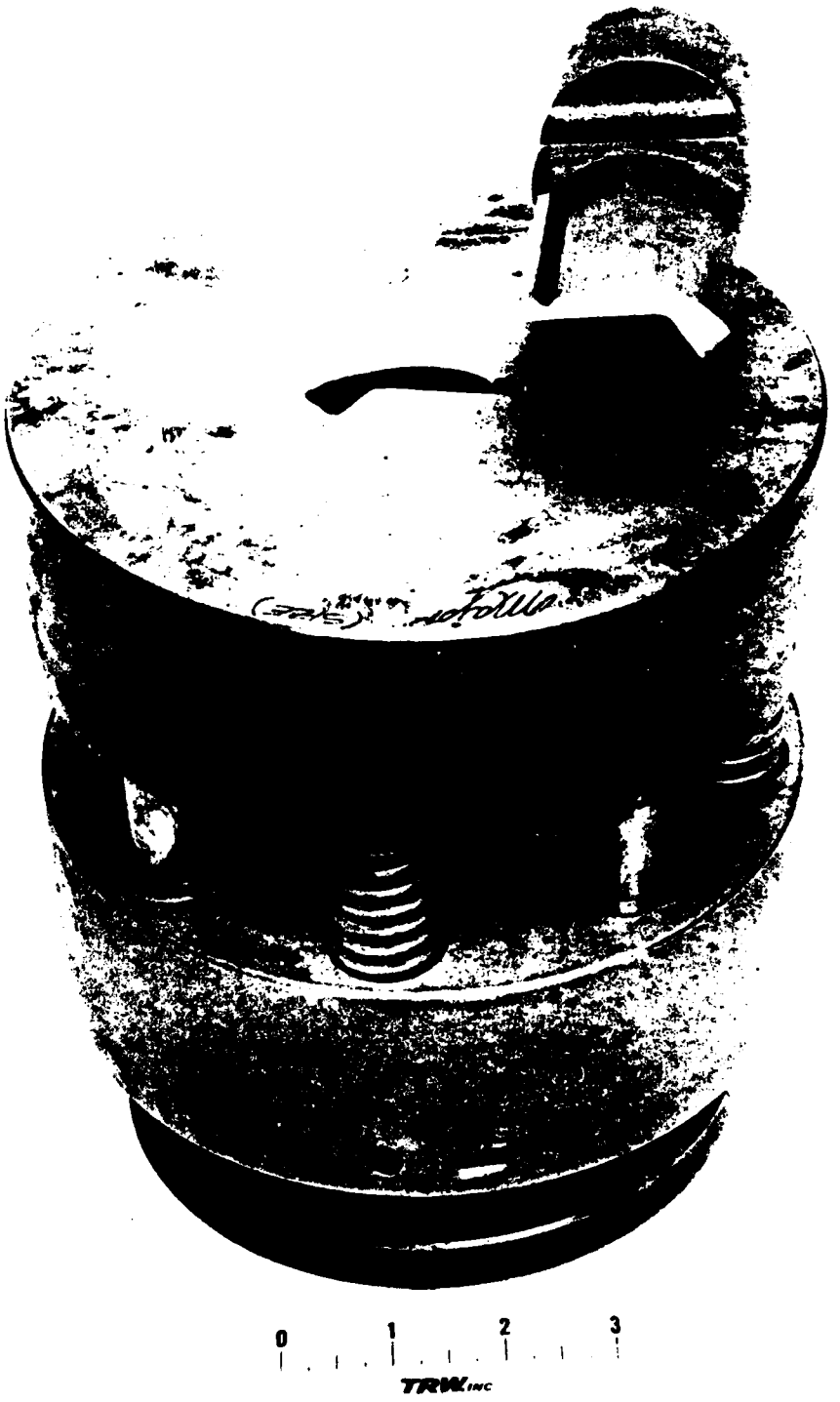



Figure 14. Actuator Housing Machining Preform Coining Tool.

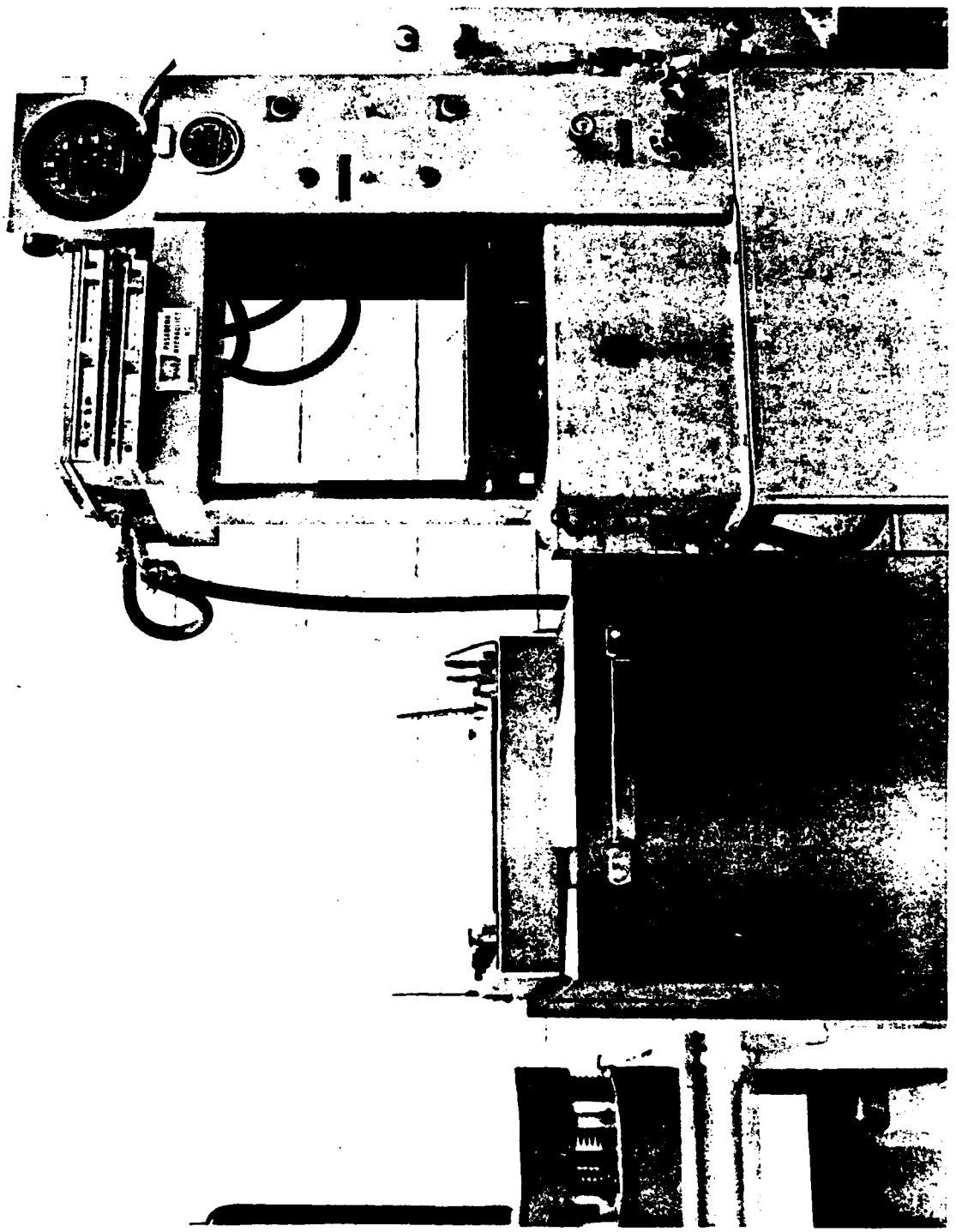


Figure 15. Fifty-ton Hydraulic Press with Pressure Transducer and Strip Chart Recorder.

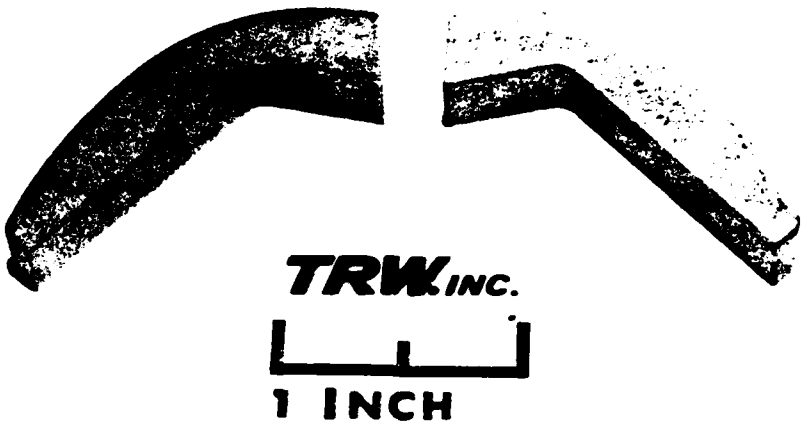



Figure 16. Ti-6Al-4V Actuator Housing Machining Preform, As-Sintered.

## 3.2 Gotcha Lock, P/N 407MP

The gotcha lock is a thin-walled splined cylinder with 16 teeth, made of CP titanium (see Figure 17). It was decided that this machining preform should be made by cold isostatic pressing because of the density gradient in the 2.5 mm (0.100 inch) wall that would occur if it were die-pressed; i.e., because of die-wall friction, the top and bottom of the cylinder would be more dense than the center.

In CIP of a hollow object, one can use an outer bag to press the powder onto a machined hard mandrel, producing a precise inner surface in the compact, or preform the reverse process and use an inner bag to press the powder outwards against a surrounding hard tool. The former philosophy was followed in the production of this part from powder so as to obviate the difficult and expensive operation of machining the sixteen teeth. At the same time, CIP against the mandrel saves the two-inch diameter cylinder of metal which is machined away when the part is made from bar. For any hollow object to be CIP, a choice must be made as to whether to press inward or outward, for it is not possible to do both simultaneously.

Work on CIP of the gotcha* started with isostatic pressing of test bars. Figure 18 illustrates schematically the relationship of pressure vessel, pressurizing medium (water), elastomeric container (bag), and powder. An illustration of the linear relationships between the inner dimensions of the bag and the consolidated titanium powder is given by Figure 19. The bag cavity is 19 x 19 x 200 mm (3/4 x 3/4 x 8 inch); the bars are 12.7 x 12.7 x 150 mm (1/2 x 1/2 x 6 inch).

Using the linear change data from the test bars as a starting point, a set of tools for a prototype gotcha was designed and built (see Figure 20). The tool set consisted of a steel mandrel, a one-quarter-inch thick wall urethane rubber cylinder, and urethane rubber caps incorporating recesses to position the mandrel.

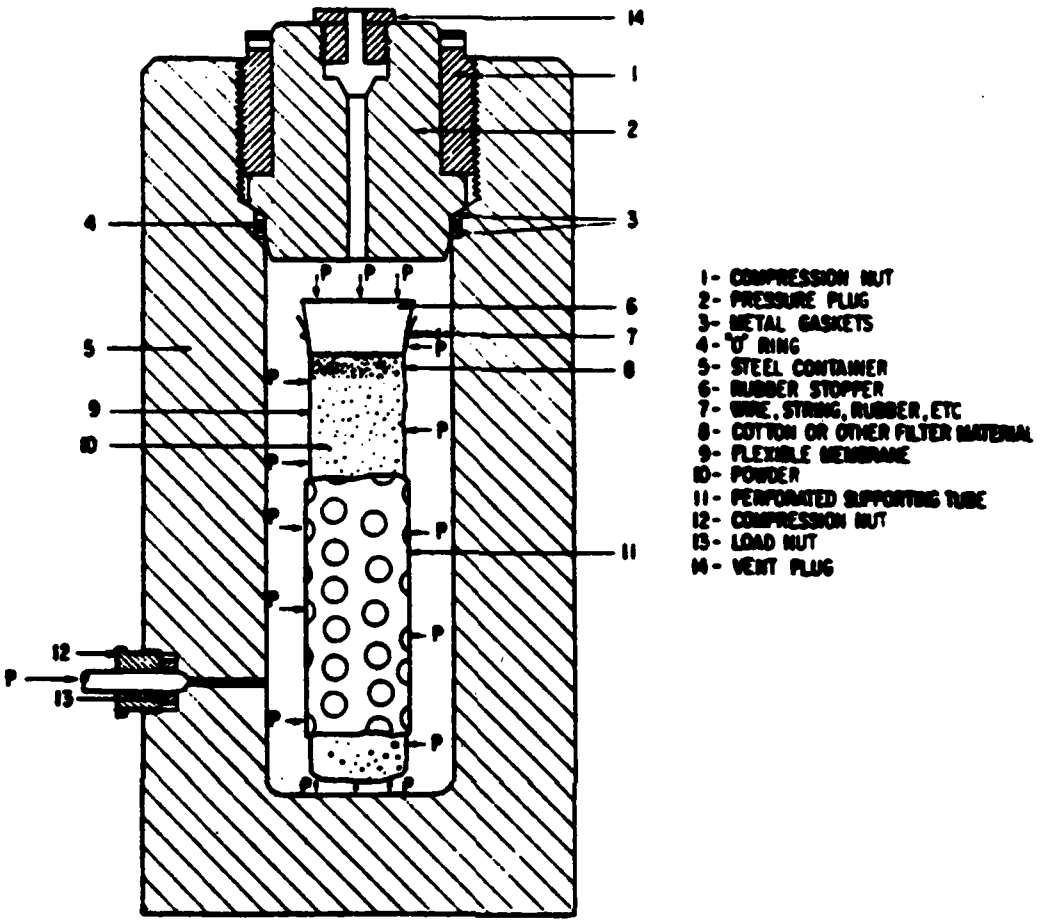
Figure 21 shows a pressing made at 205 MPa (30,000 lb/in$^2$) with these tools. The tearing along the tooth edge indicates that a larger annulus (gap between mandrel and outer urethane cylinder) is needed to feed more powder into the tooth region.

A second urethane bag and caps with a larger inner diameter were made and used to produce the sound prototype gotcha shown in Figure 22. The appearance of this piece after its outer diameter was turned in the green state is shown in Figure 23, which illustrates both the surface produced by the mandrel and after green machining.

A sixteen-tooth mandrel was then manufactured and used to produce the green compact shown in Figure 24. This piece was pressed at 415 MPa (60,000 lb/in$^2$) pressure. Note the depressions at the tooth locations. The appearance of the piece after removal of the mandrel is shown in Figure 25. The layer of titanium

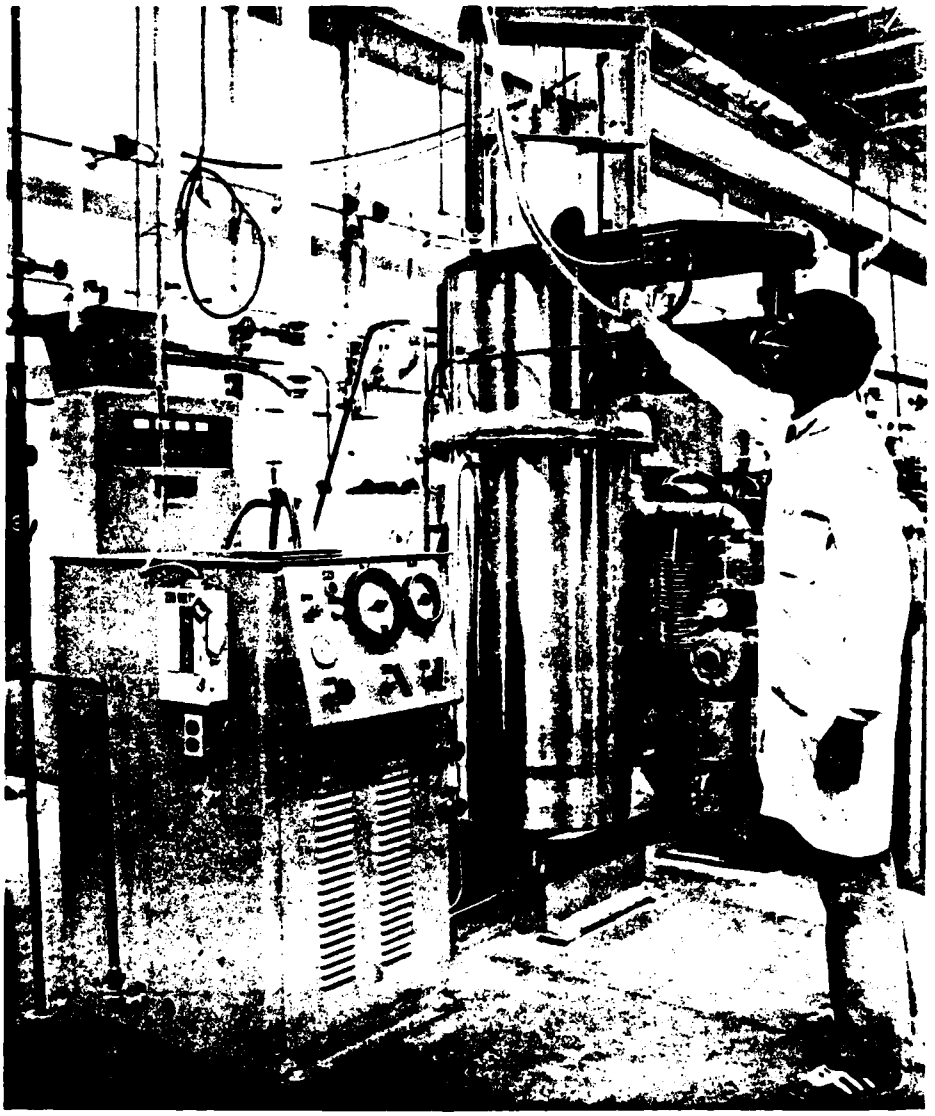
* From this point onwards, "gotcha" will be used to mean "gotcha lock machining preform."


Figure 17. Gotcha Lock, P/N 9306407.




**Sketch of a typical hydro-pressing assembly. The pressure is supplied by an auxiliary pumping unit.**

Figure 18. Tool Arrangement in CIP of Powders.



b) TRW 60,000 lb/in$^2$ Hydroclave.

Figure 18 (continued)

Figure 19. CIP Bags and Test Bars.

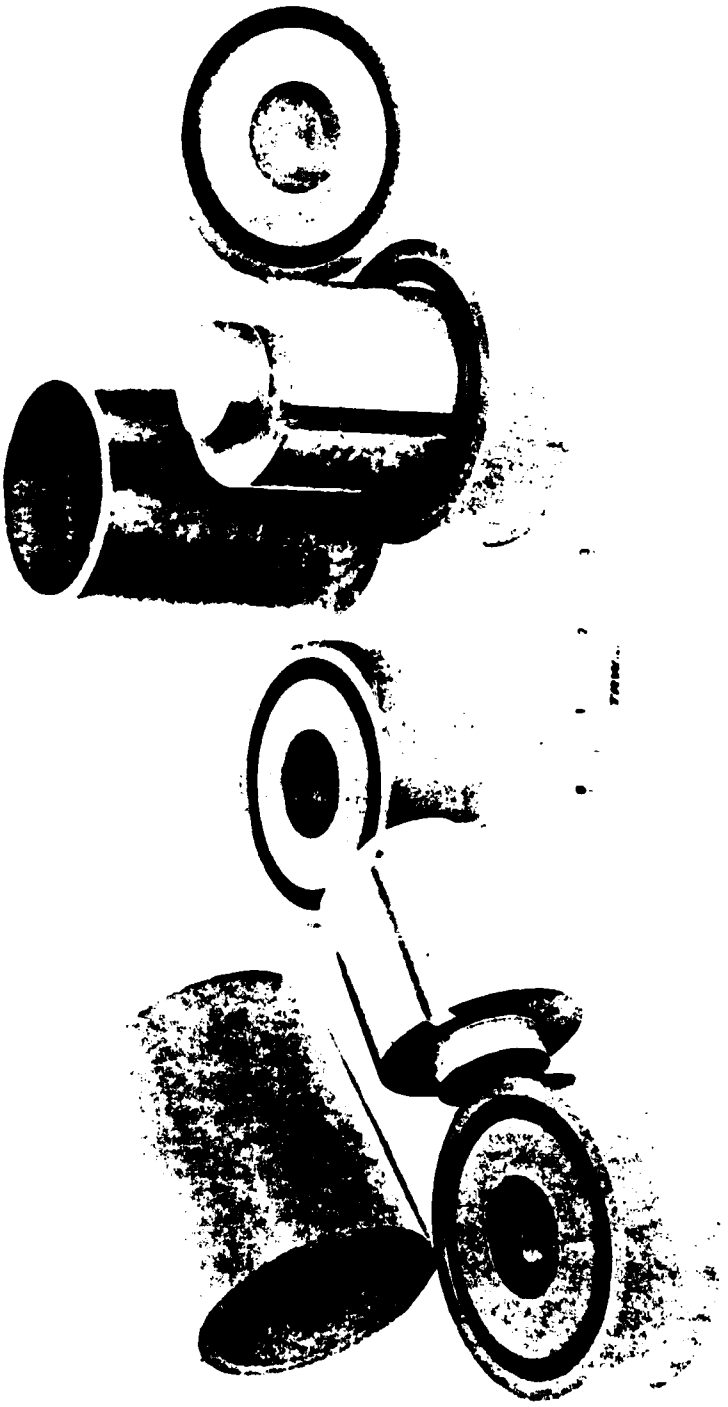


Figure 20. CIP Tools for Prototype Gotchas.

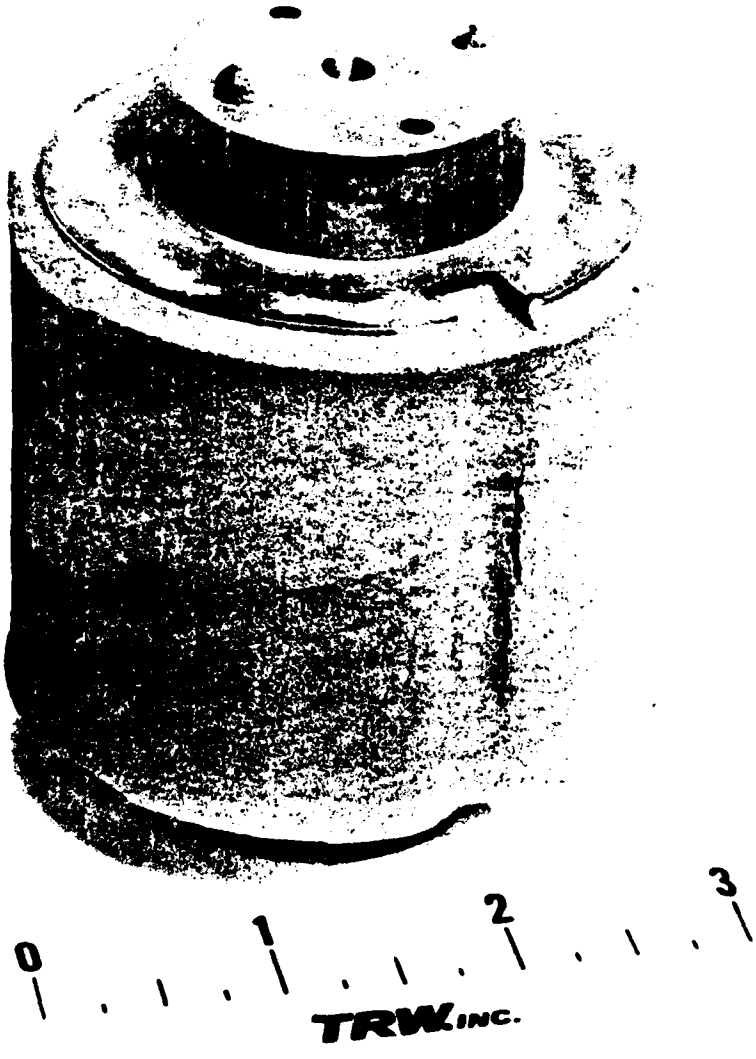



Figure 21. Prototype Gotcha Made in First Tool Set.

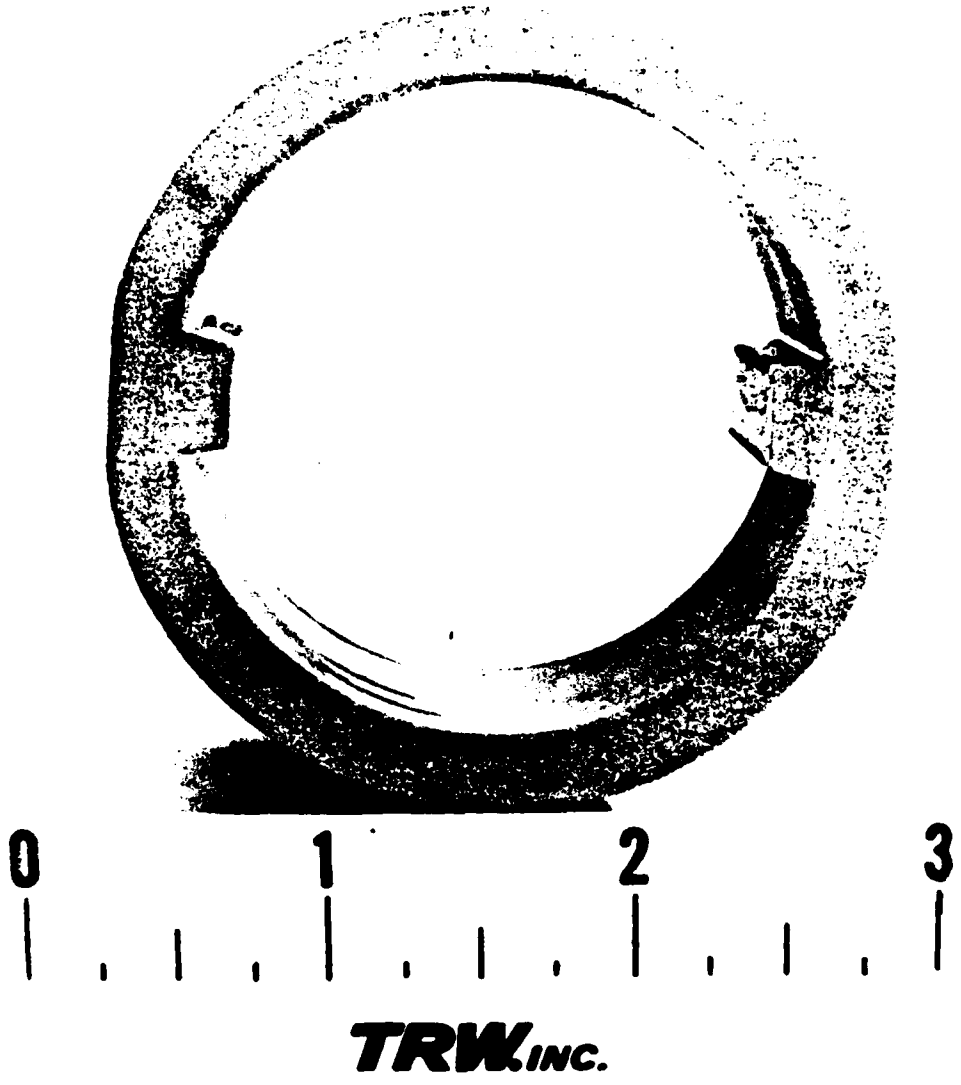



Figure 22. Prototype Gotcha from Larger Bag.

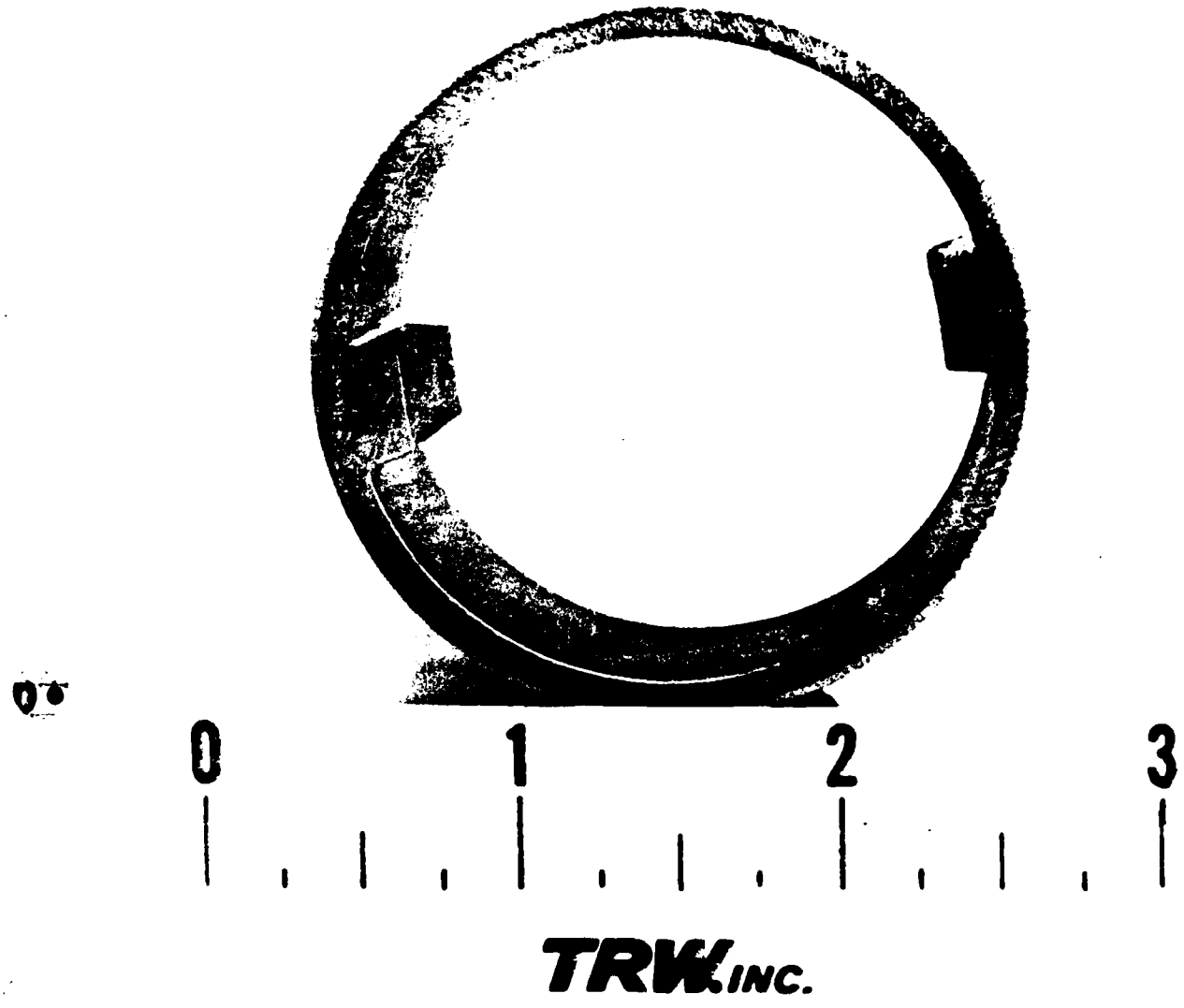



Figure 23. Prototype Gotcha, Turned.

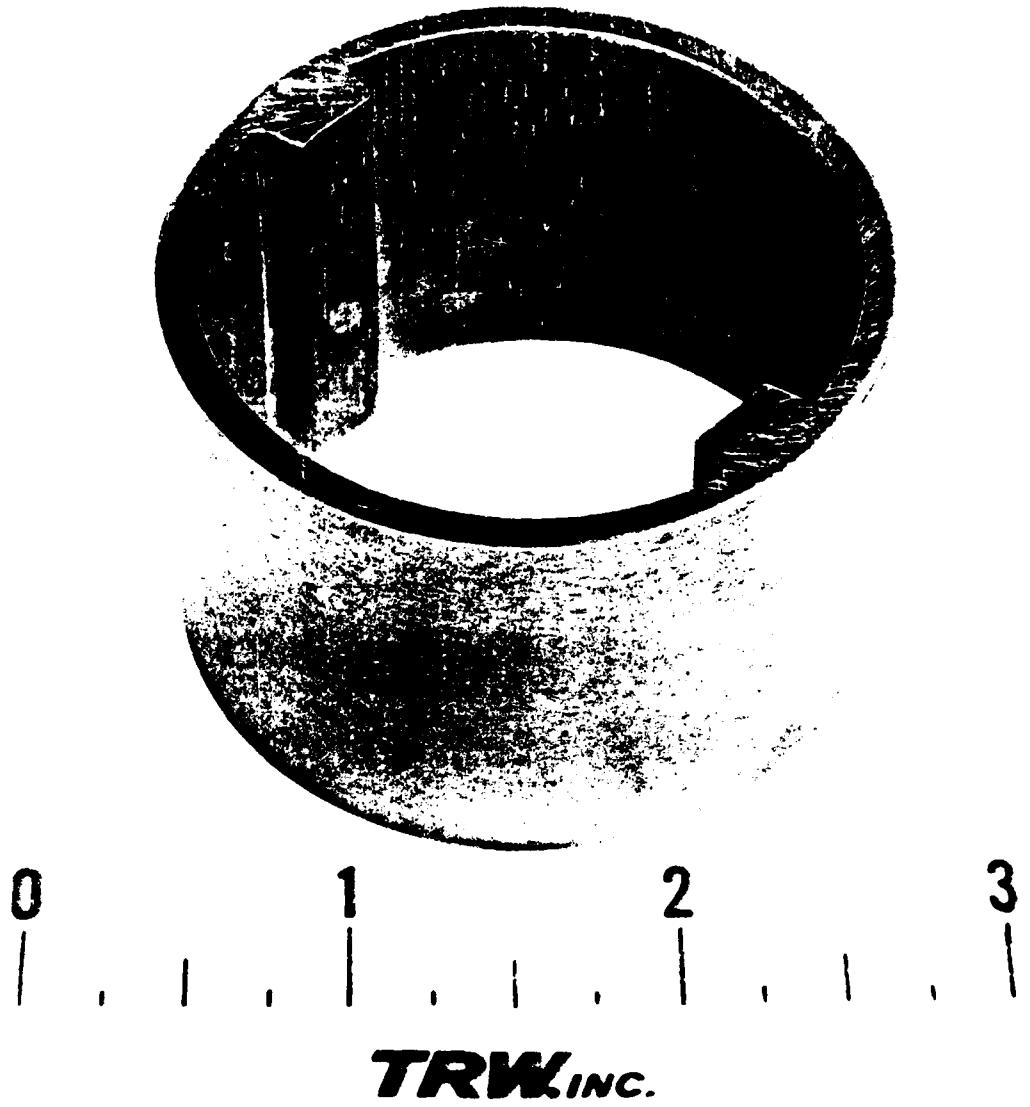



Figure 23 (continued)

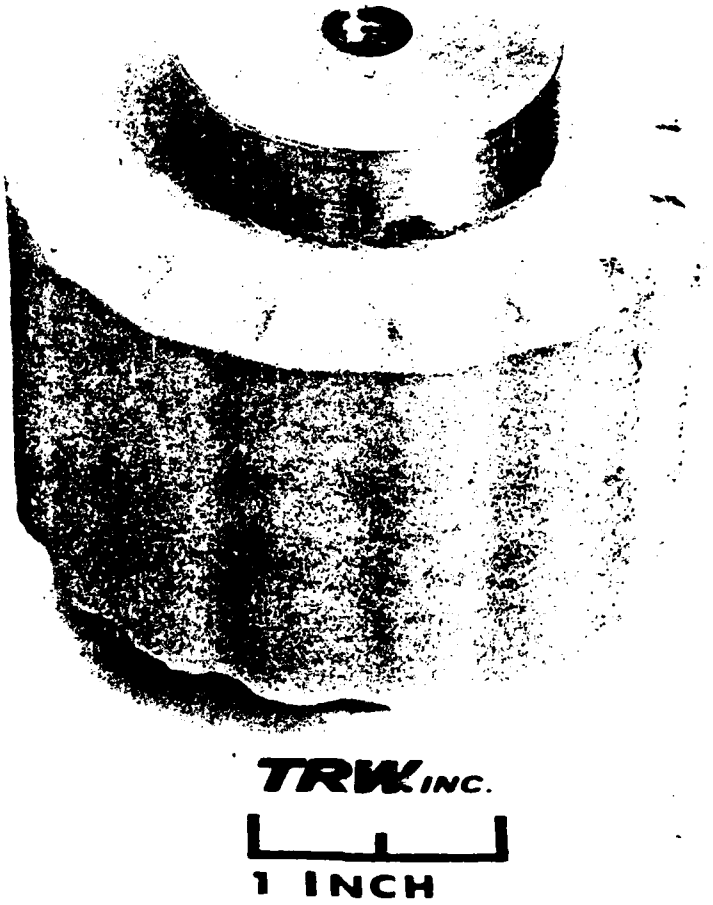



Figure 24. First Gotcha, As-Pressed, with Mandrel in Place.

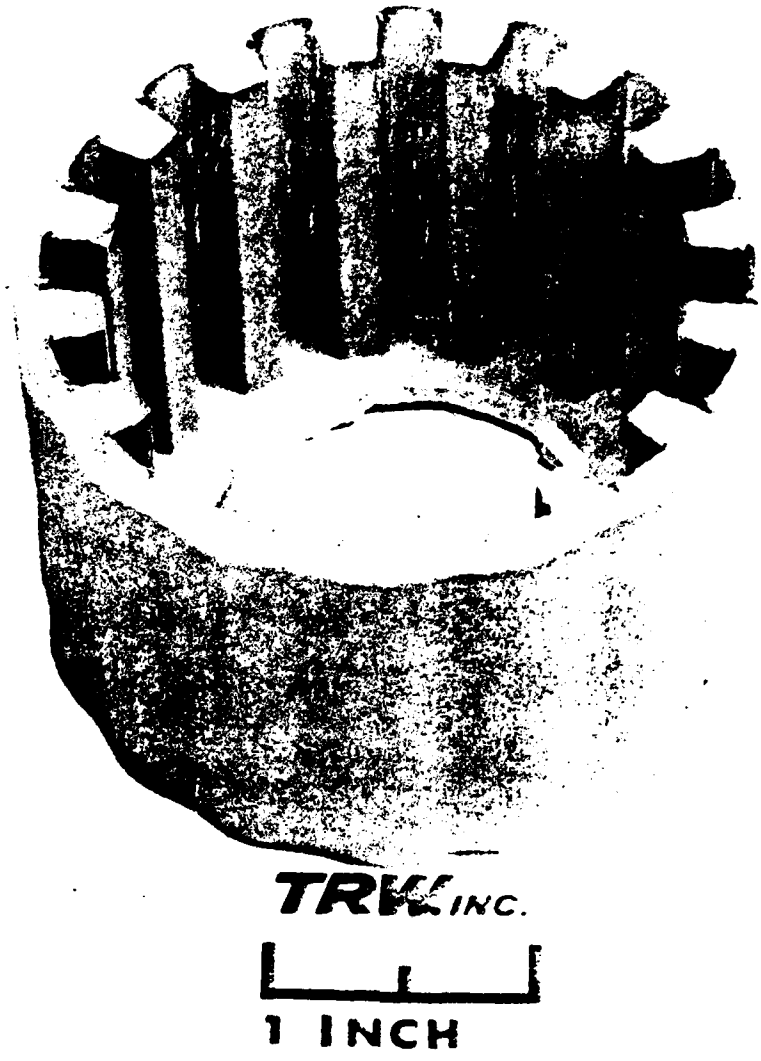



Figure 25. First Gotcha, As-Pressed, with Mandrel Removed.

at the bottom of the piece results from powder sifting under the mandrel when the bag and mandrel are vibrated to achieve maximum powder density.

This compact was sintered and turned to an outer diameter of 2.7 inches. It is shown in Figure 26 alongside a machined (wrought) gotcha. The P/M gotcha was made with an 82 mm (3-1/4 inch) ID bag, and, as the illustration shows, it does not quite "clean up" to its required 68.6 mm (2.7 inch) diameter. A satisfactory diameter was achieved when the bag was enlarged to 85.7 mm (3-3/8 inch) diameter, producing the parts shown in Figure 27.

Measurements of the gotcha's diameters after sintering revealed an ovality of from 0.25 to 1 mm (0.010 to 0.040 inch), which could not be eliminated through attempts to increase the uniformity of powder-filling in the bag. The subsequent HIP cycle did not change the ovality appreciably, and so it was decided to correct it afterwards. Subsequent to HIP, therefore, each gotcha is thermally sized. The sizing operation consists of heating the gotcha to 700°C (1300°F) and then fitting it onto a chilled, high-coefficient-of-expansion steel mandrel. The gotcha and mandrel, which is a right circular cylinder (no teeth), are returned to the furnace for two hours. The size of the mandrel is such that, as it heats to sizing temperature, it presses outward against the small diameter of the oval part. When the mandrel-part pair are cooled to room temperature, the mandrel shrinks away from the gotcha and is easily removed. The gotcha is subsequently treated to remove the slight oxidation layer that results from the sizing operation.

Details of the gotcha lock manufacturing process are contained in the process description in Appendix B.

## 3.3 Base, P/N 406MP

The Ti-6Al-4V base, Figure 28, is a complex part with both inner and outer details that could benefit from being isostatically pressed against a mandrel. Pressing outward against a hard tool or former would eliminate the need for machining the teeth, while pressing inward against a mandrel would produce the flat-sided hemispherical bowl at the top. Our initial decision was to press outward since we would, at the same time, produce a hollow interior, although not to net dimensions.

Forming a hollow object by pressing outward is referred to as employing a "dilating bag" procedure and presents two difficulties: 1) the bag is placed in tension rather than compression, and 2) it is more difficult to seal against the ingress of water.

Figure 29 illustrates the tool arrangement that was designed and built for the dilating bag CIP of the gyro base. The essential features of this tool are:

1. The hollow axial rod aligns the upper and lower bag supports, serves as a conduit for the water to act on the bag and, with the top and bottom nuts, clamps the upper and lower end plates against the horizontal portions of the bag.

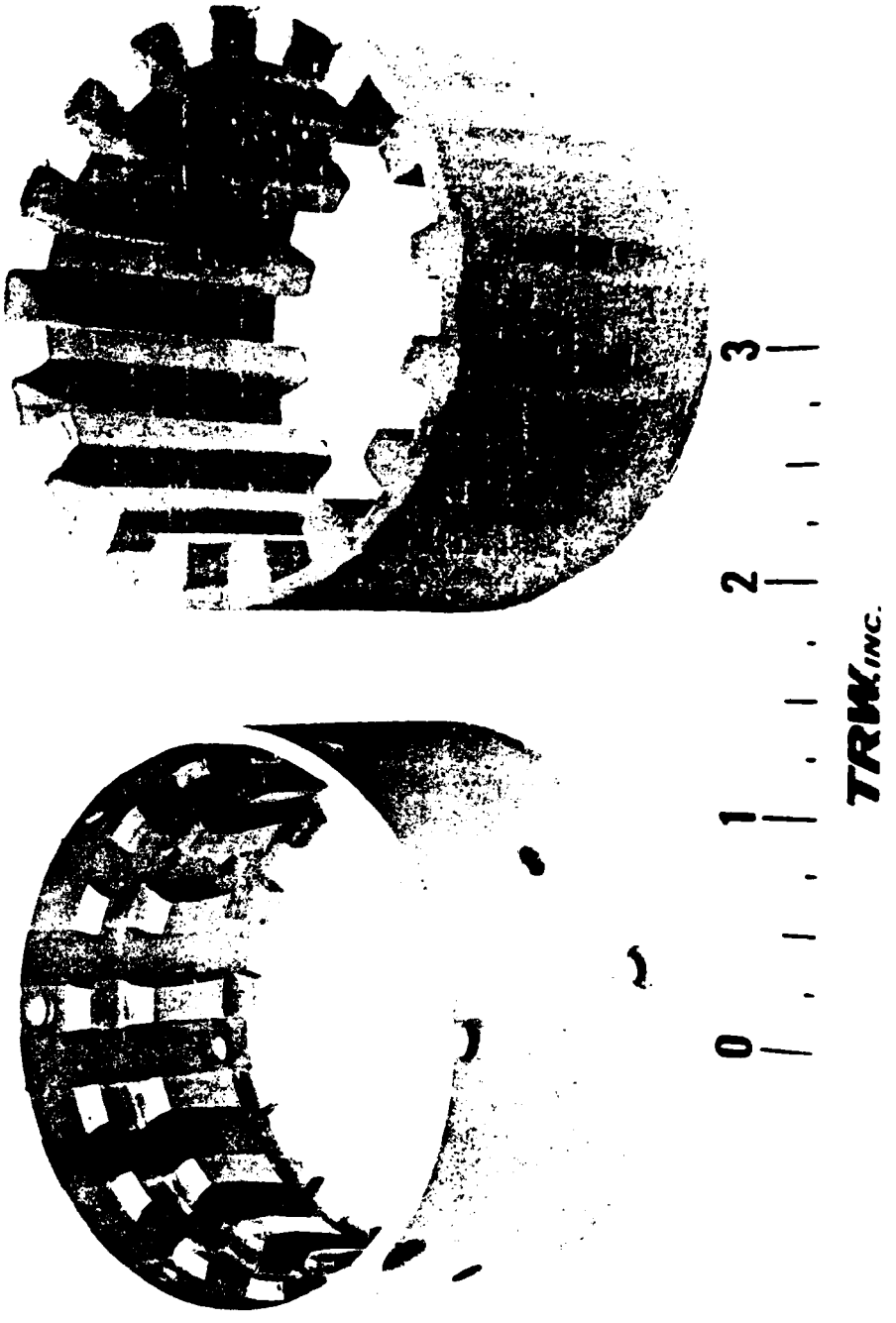



Figure 26. Wrought and an Undersize P/M Gotcha Lock.

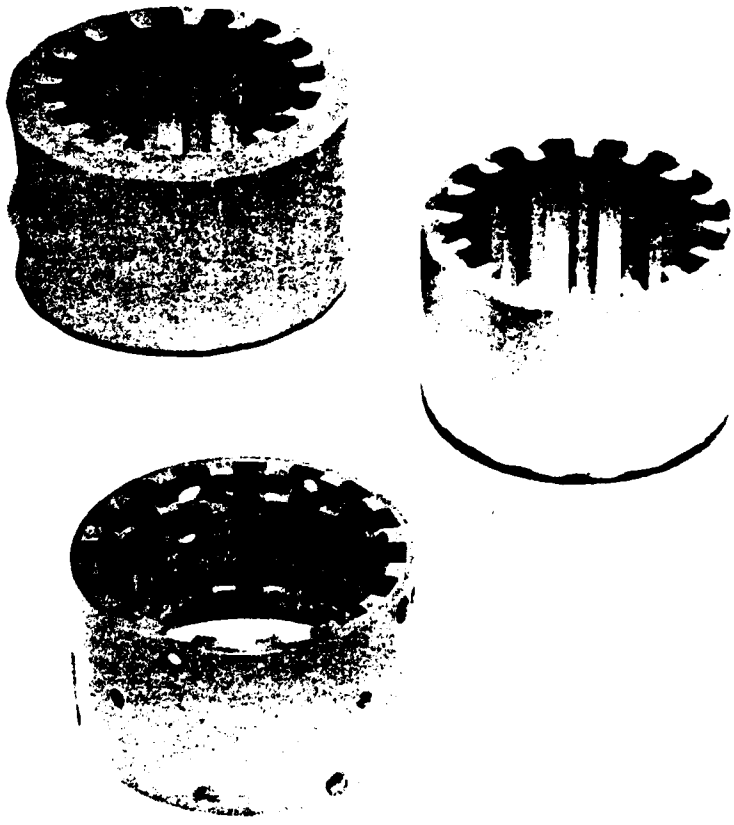


Figure 27. Wrought Gotcha and P/M Gotchas, As-Pressed and After Sintering.

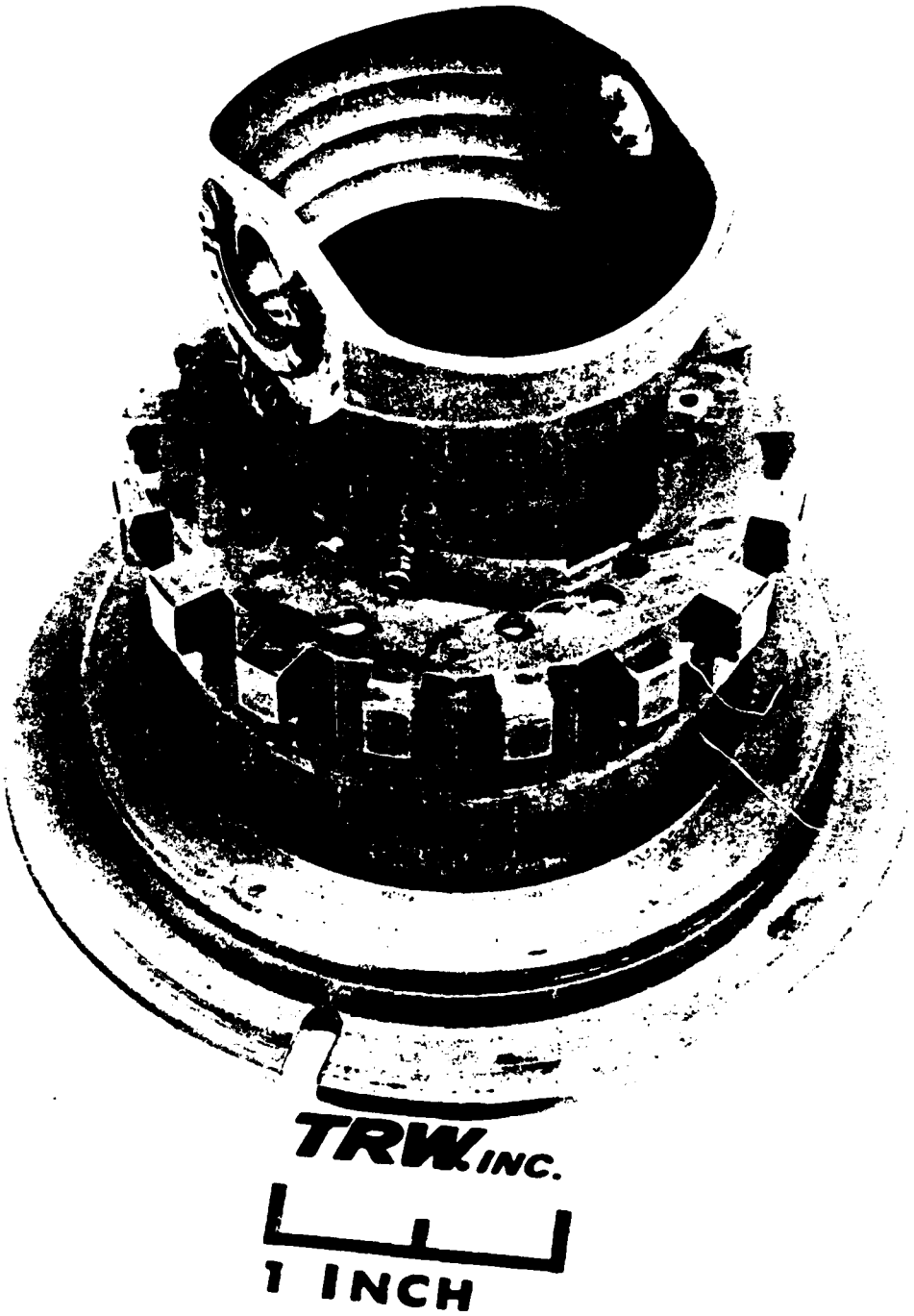



Figure 28. Gyro Base.

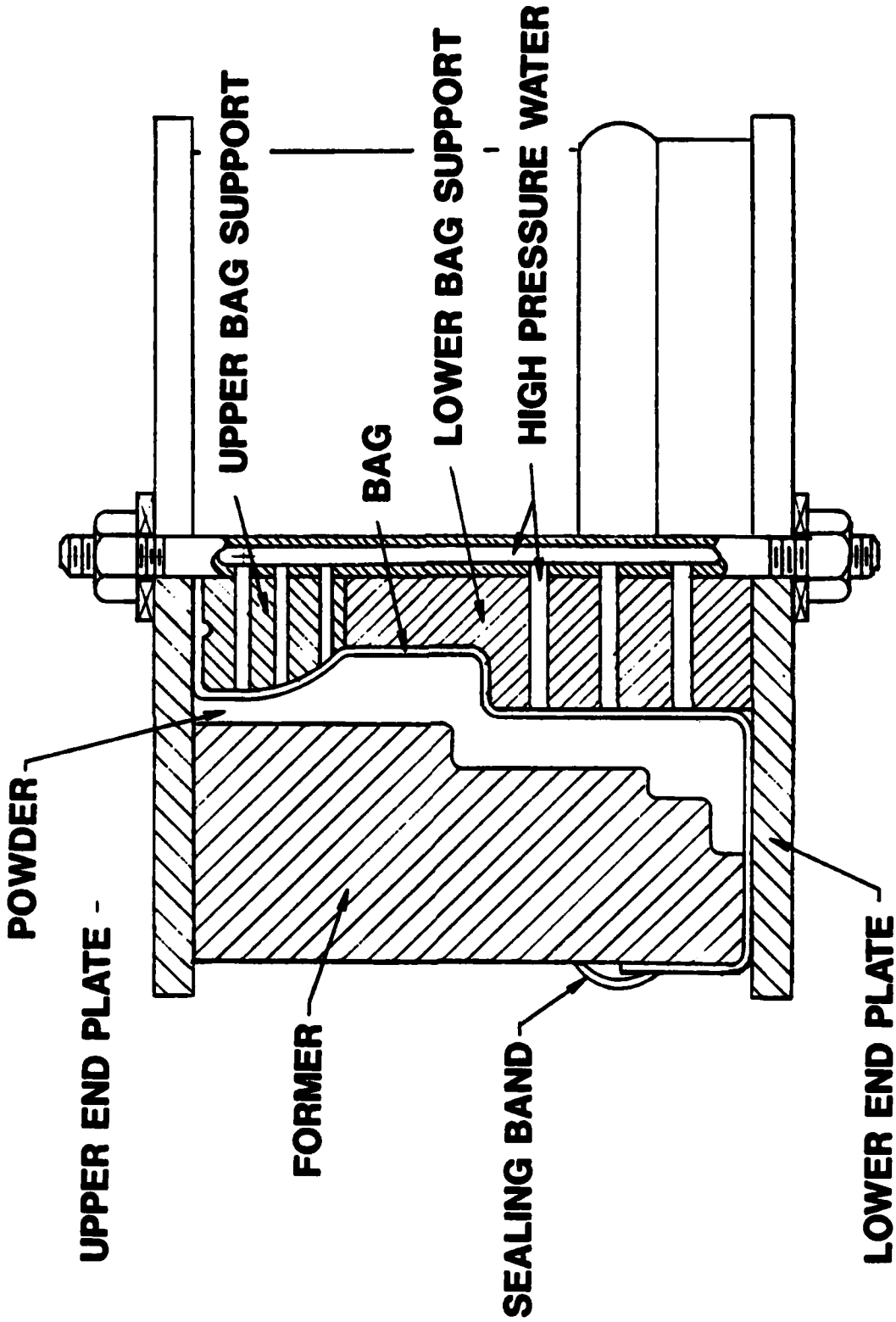



Figure 29. CIP Assembly for Gyro Base.

2. The bag used in this technique is of thin, 1-1/2 mm (1/16 inch) latex, since urethane, though stronger, lacks sufficient ductility to undergo the required dilation.

3. The upper and lower bag supports are made of aluminum, since they carry no significant load, and serve the dual purpose of supporting the thin bag while it is being filled and pressurized and of introducing the high pressure water to the inner bag surface.

4. The former is a circular steel block which duplicates the desired gyro base outer contour on its inner surface. Although not shown in the sketch, it contains an O-ring groove in its upper surface to provide for sealing against the upper end plate.

5. The end plates' purpose is to keep the water from contacting the powder by squeezing the bag against the former, at the bottom, and against the upper bag support at the top. An additional water seal is provided through the combination of upper end plate, O-ring, and former.

This tool arrangement could not be made leak-tight, although additional O-rings were introduced and the bag was redesigned. The origins of the problem are twofold: 1) as the bag is pressurized, it stretches, not only against the powder but in such a manner as to become thinner at the sealing surfaces, so that the initial thickness of rubber sandwiched between the metal components is reduced; and 2) the lower plate has an unsupported region subjected to an enormous force (415 MPa/60,000 psi water pressure x 6500 $mm^2$/10 $in^2$), which is only partially counterbalanced by the powder compact on the inside. The effect of this unbalanced load is to deflect the plate inward, thus reducing the area in contact with the bag. A decision was therefore made to CIP the base radially inward.

In addition to the savings in material represented by the titanium formerly machined from the interior of the base, the P/M CIP-inward approach solved the difficult problem of machining the bowl. Because of the bowl's two flat sides, it cannot be bored or turned in a lathe, but instead is made by sculpting with a ball mill, as can be seen in Figure 28.

A two-piece steel mandrel and a cast urethane bag assembly were designed and built. It was quickly demonstrated that this approach was not subject to the water leakage problem. The bag is shown in Figure 30, along with the two-piece mandrel. Although a sound flange was produced, it fractured near the body joint. A new bag, shown on the left in Figure 31, incorporates a chamfered region between the body and flange. This bag produced the piece shown, which has a crack at the junction between body and flange. The problem shown here is related to the need to supply sufficient powder to the junction, while the powder is separately moving radially inward to form the body and axially upward to form the flange. When the CIP pressure is released, the compressed (shortened) bag returns to its original length and, if the flange-body junction is too thin (indeed, even if it meets blueprint dimensions), the flange is pushed away from the body and either cracks or breaks off. The problem existed also at the next higher 90° junction on this piece. The cracks are more visible in Figure 32,

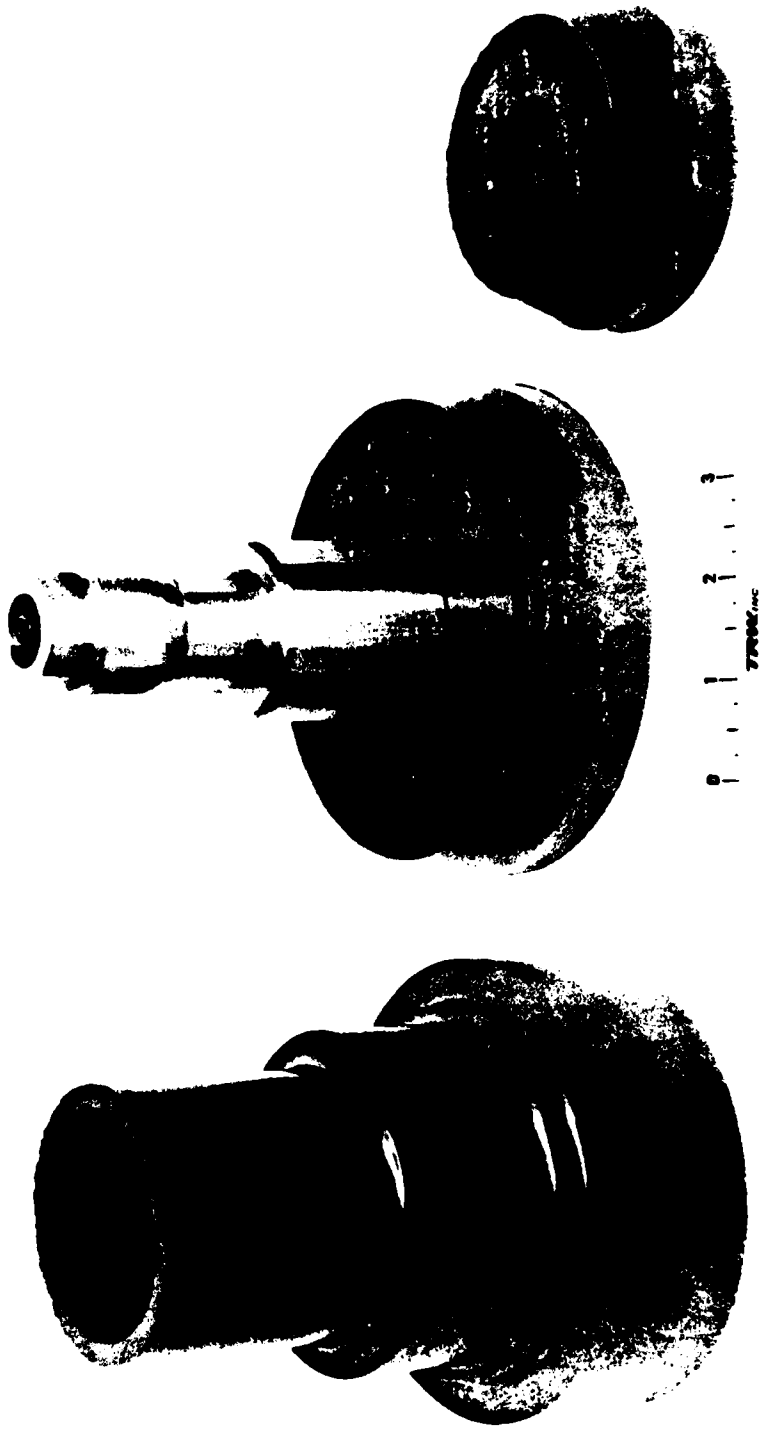


Figure 30a. Urethane Rubber Bag, Closures and Steel Mandrel for CIP of Base.

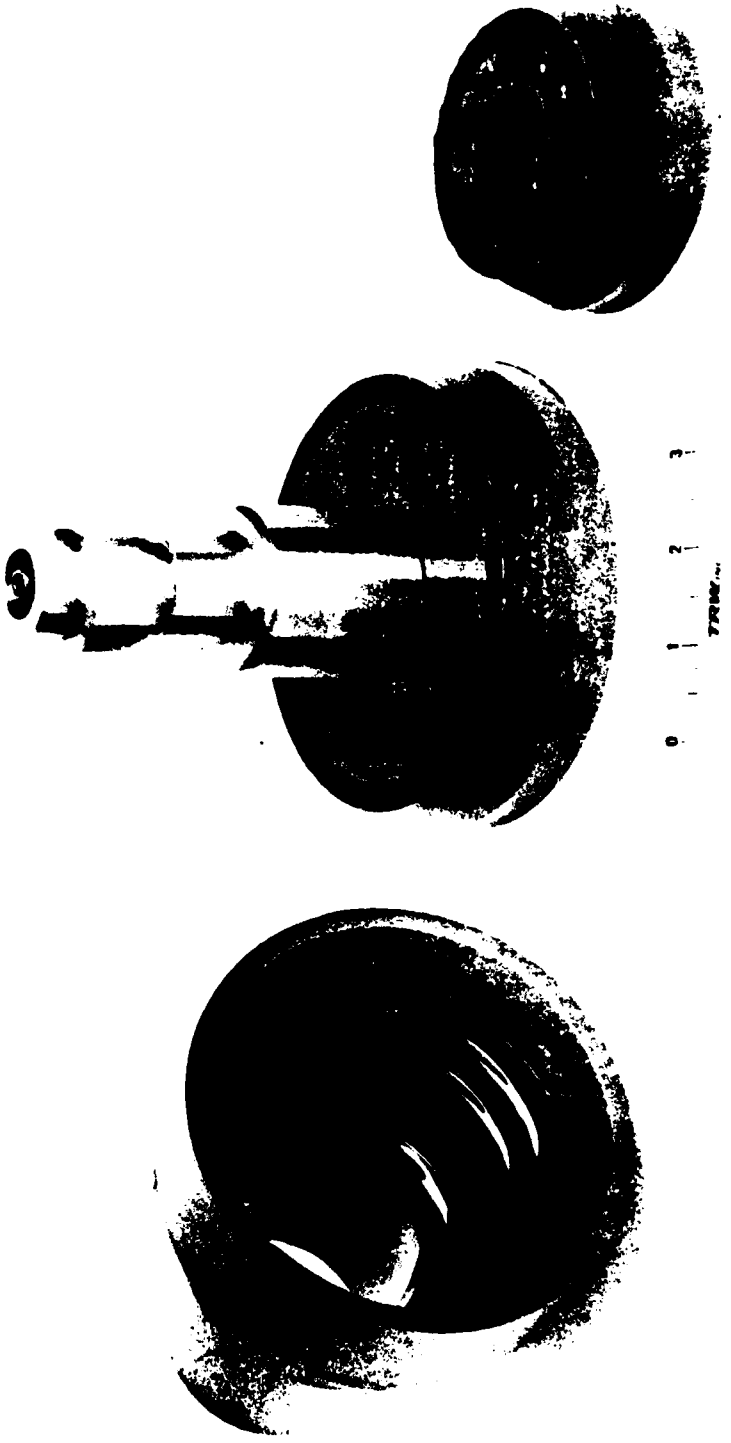


Figure 30b. Urethane Rubber Bag, Closures and Steel Mandrel for CIP of Base.

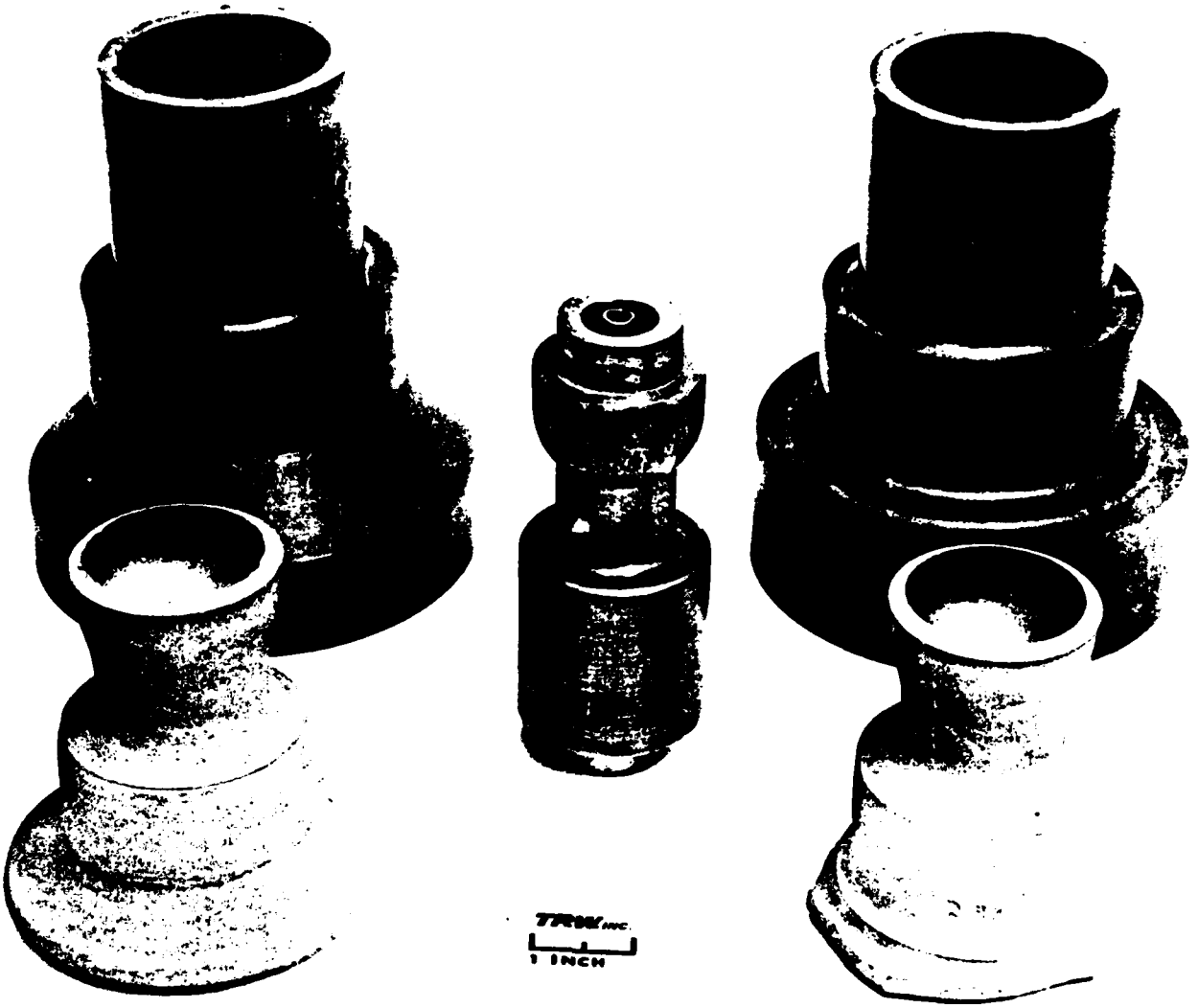



Figure 31. CIP + Sinter Base, Ti-6Al-4V made in original (right) and modified (left) bags. (Two-piece steel mandrel in center).

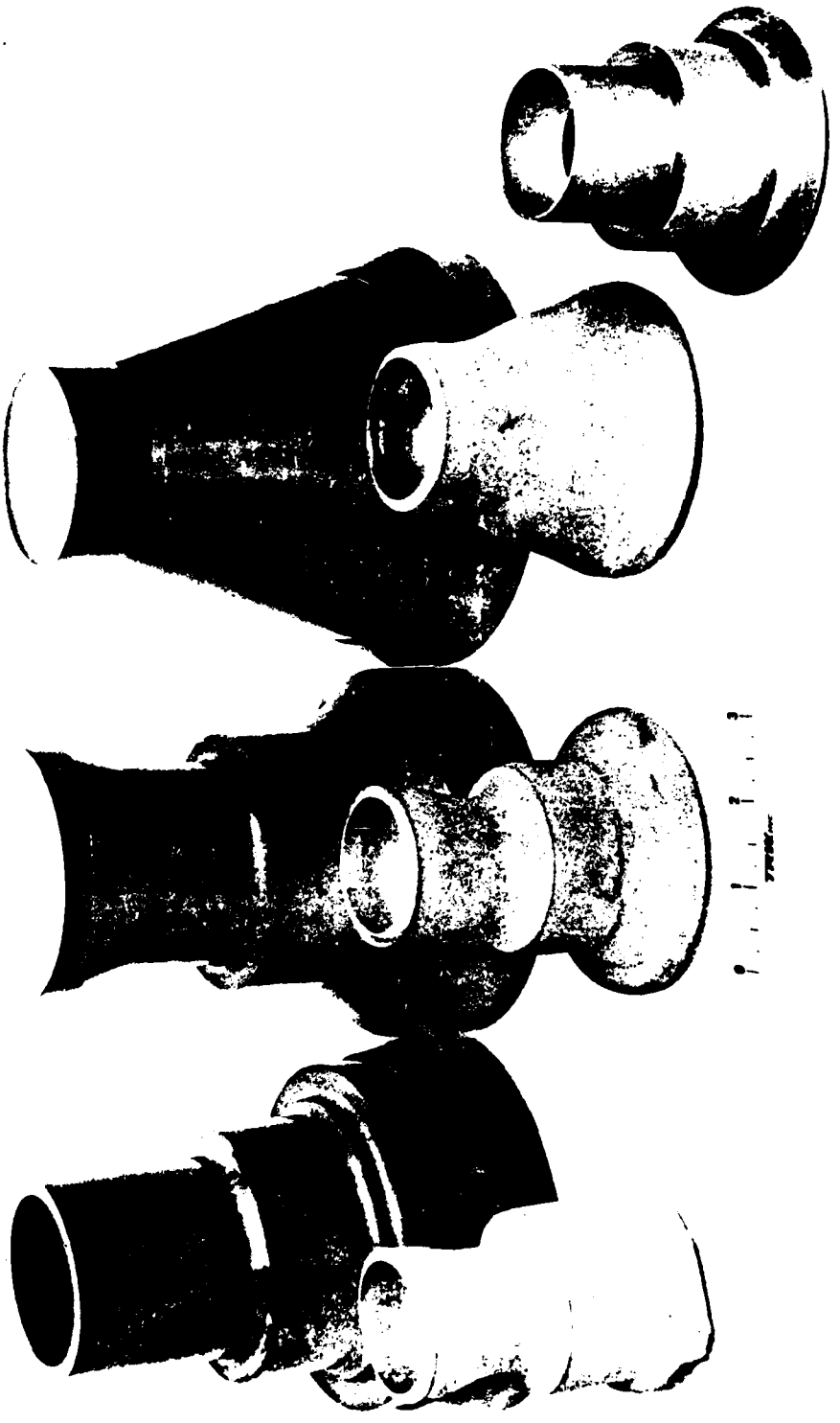


Figure 32. Evolution of CIP Bag Design and CIP Product for Gyro Base. Bags/Base 406 T1, T2, T3.

which also illustrates the solution to the problem. Although the bag shown on the right of this figure is not optimum with respect to minimal consumption of powder, the base pressed from it is structurally sound. At the extreme right in this figure is a sintered base which was machined to confirm that sufficient stock was available to clean up to the required outer dimensions. When this was confirmed, a pair of flats was machined on the upper portion of the mandrel, producing the base with a flat-sided bowl shown in Figure 33. The figure also illustrates the inner detail of the lower end of the base. Two P/M base preforms, CIP, machined and sintered, are shown in Figure 34 along with a machined standard cast and wrought base.

Subsequent to sintering, the gyro base is HIP (hot isostatically pressed) at 930°C and 103 MPa (1700°F and 15,000 lb/in$^2$) to further increase density and enhance properties. It was found that considerable economies could be realized by performing the outline machining shown in Figures 33 and 34 for, in this condition, the bases can be nested together during the HIP operation, which is a batch process, while in the unmachined state they cannot.

A process description for the manufacture of the gyro base machining preform is found in Appendix C.

## 3.4 Gimbal Ring, P/N 409MP

The shape and size of the finished Ti-6Al-4V gimbal ring is shown in Figure 35. This part was designed as a ring forging, and therefore the briquetting operation served to produce a forging preform. Initially, the briquetting punches were shaped to make the forging preform illustrated in Figure 36. Two difficulties were experienced with this design. Whereas in an automatic powder compacting press it would be possible to use one pair of punches (one upper, one lower) to make the ring's flat sides and a second pair to make the long gimbal sides, only one pair of punches can be built into a subpress of the type used in this work (see Figure 37). The result of this restriction is that, after the powder is leveled with the top of the die, the upper punch must force some powder to move laterally from the "flat" region into the "gimbal" region. Since green strength and particle friction have a direct relationship, this type of powder, which produces high green strength compacts, resists lateral flow and powder redistribution does not occur.

The first consequence of this situation is an unbalanced pressure in the die. When a 550 MPa (40 ton/in$^2$) compacting pressure is applied to the punches, the long sides of the punches (which produce the "flat" gimbal sides) contact the powder first and will eventually produce a pressure greater than 550 MPa, whereas the recessed portions of the punches will lag in compressing the powder and will deliver an effective consolidation pressure less than 550 MPa. In the case of titanium powder, which has a tendency to gall the sides of the core rod and die at high pressure, the unbalanced pressure caused particles of titanium to weld to these "higher-pressure" tool surfaces, thus increasing the ejection forces.

The uneven consolidation pressure produced a lower density along the long gimbal sides than along the short flat sides of the gimbal ring. As noted

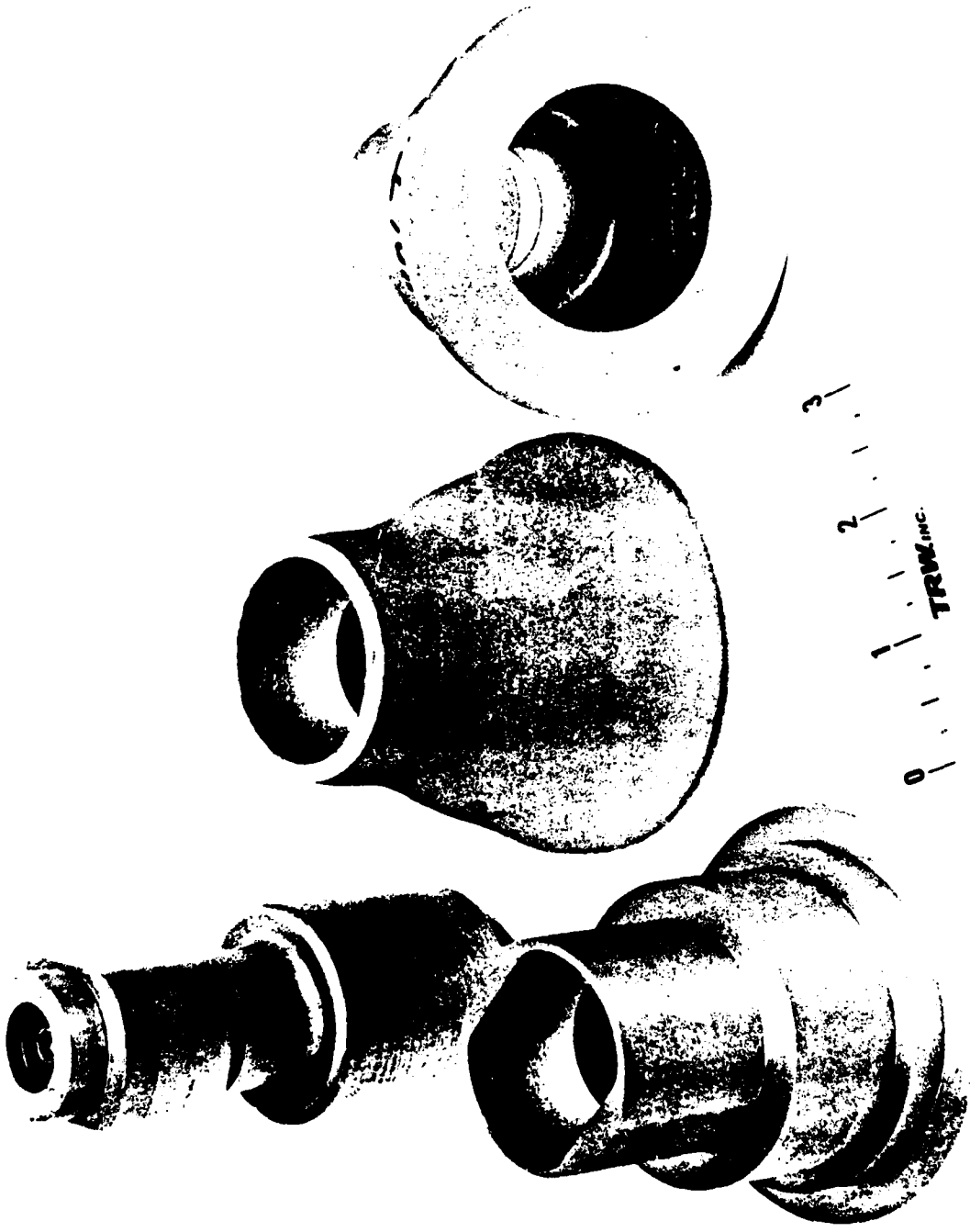



Figure 33. Gyro Base before and after incorporation of flats in bowl.

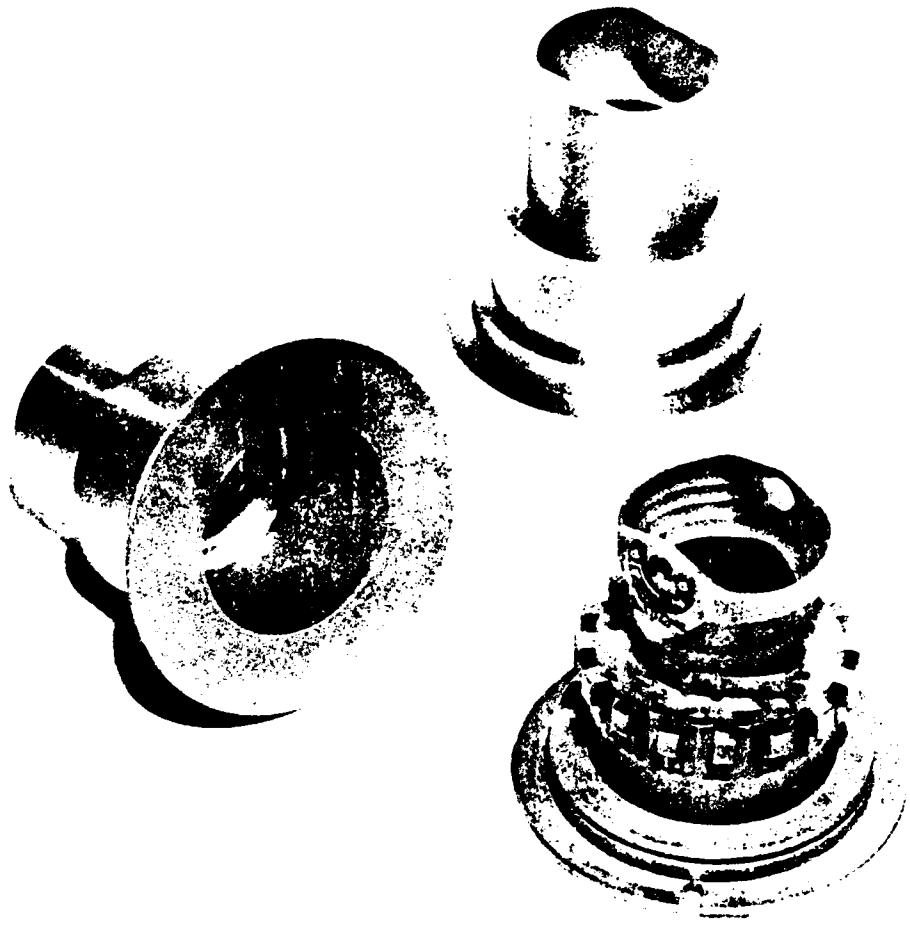


Figure 34. CIP-Machined-Sintered Gyro Base Machining Preforms.

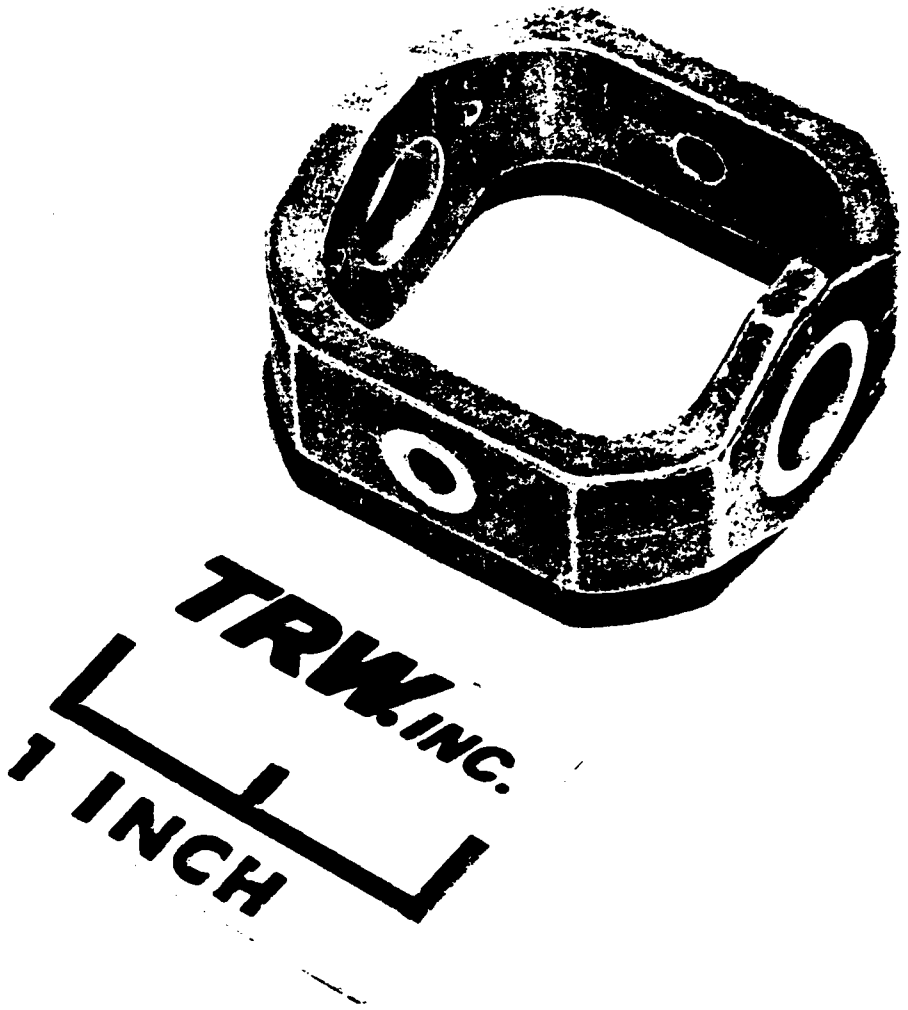



Figure 35. Gimbal Ring, P/N 9306409.

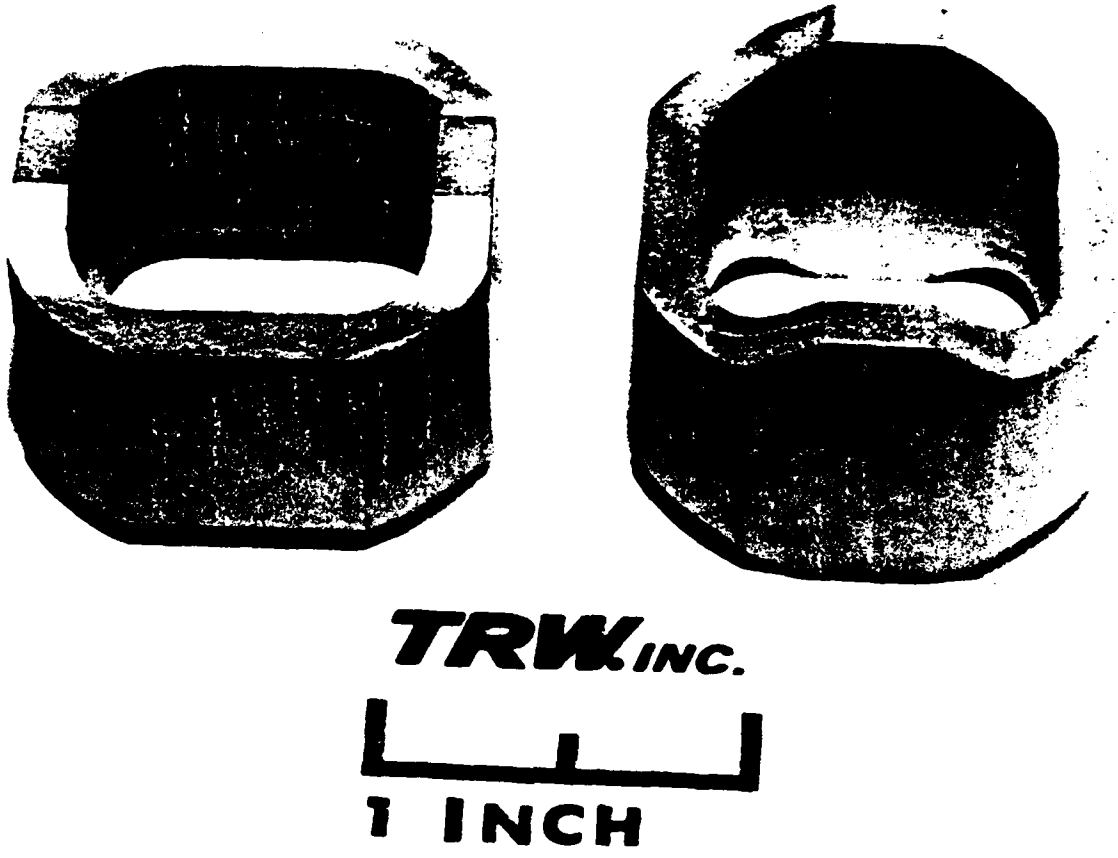



Figure 36. Gibal Ring Forging Preform, First Design.

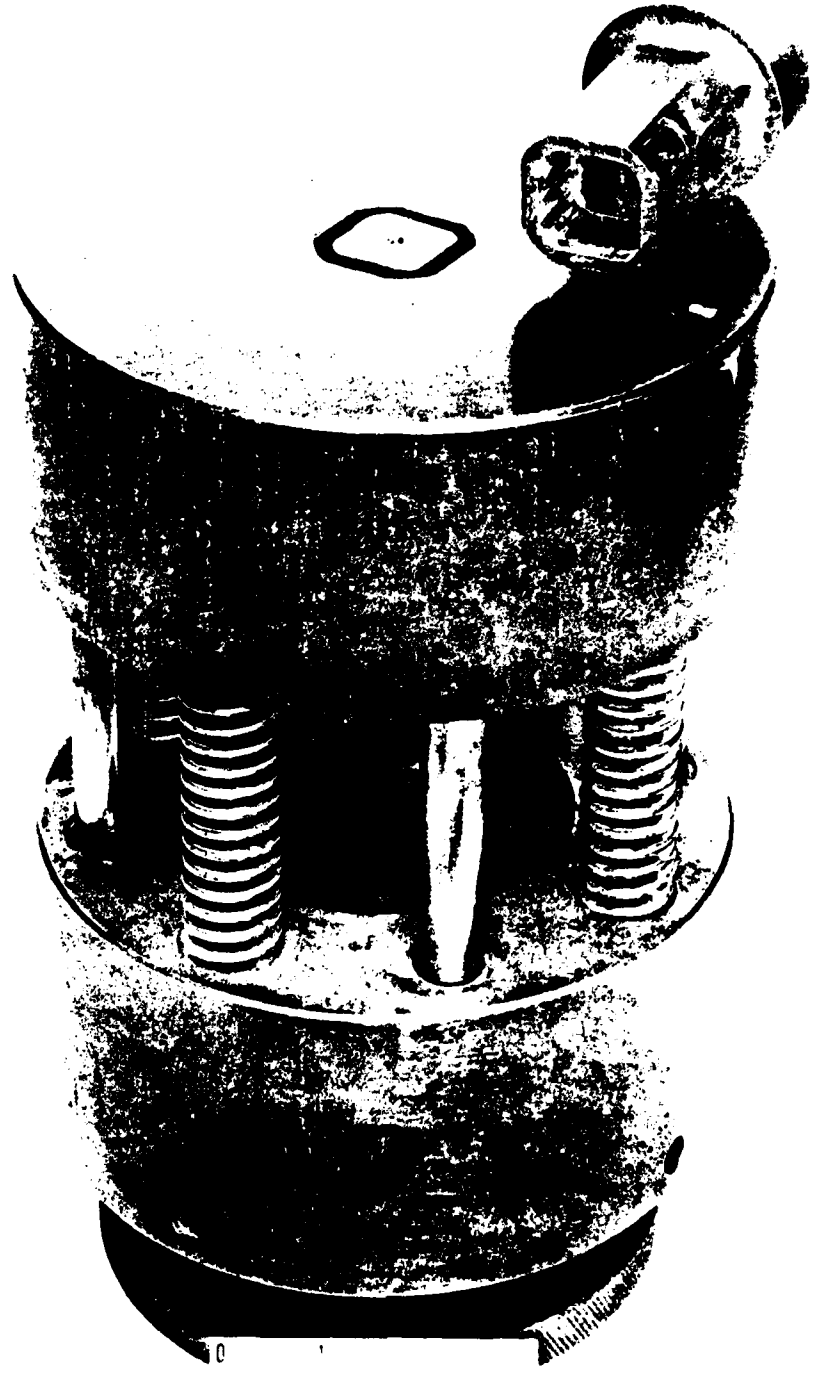


Figure 37. Gimbal Ring Briquetting Die Set with Modified Punches.

earlier, low-density compacts shrink more during sintering than high-density compacts. The practical effect of this phenomenon was for the square green compact to sinter into a rectangle, and the short side of the rectangle would then not fit over the forge die core rod.

The problem was solved by removing the gimbal details from the briquetting punches, i.e., they were ground square (see Figure 37) so that they produced rings with flat top and bottom surfaces.

After the gimbal ring forge preforms were vacuum sintered, they were coated, heated to forging temperature, and forged in the apparatus shown in Figures 38-41. Figure 39 illustrates the tooling concept employed whereby the die is in a "nest" which, independent of the condition of the press ways, provides its own precision of alignment through the use of guideposts and bushings.

The arrangement of cartridge heaters and insulation used to heat the tools to approximately 180°C (350°F) in order to minimize die chill is shown in Figure 40.

Figure 41, a closeup of the tool arrangement, shows the gimbal profile in the upper punch, the projecting core rod, which provides exact positioning of the preform and defines the inner dimensions of the forging, and the insulation on the sides and top of the die.

Although the test bars discussed earlier were forged at 650-810°C (1200-1500°F), it was found that the gimbal ring's large surface-to-volume ratio caused such great heat loss that it would not flow when heated to that temperature (transfer time from furnace to forging operation was approximately six seconds). The furnace temperature was increased in steps until satisfactory forgings were made at 980°C (1800°F) using a forge pressure of 880 MPa (64 ton/in$^2$).

The forged gimbal rings were air cooled, after which they were acid treated to remove the surface oxide layer. A set of cleaned gimbal ring machining preforms is illustrated in Figure 42.

The detailed process description for this part is included as Appendix D.

## 3.5 Inner Gimbal, P/N 410MP

The finished inner gimbal, P/N 9301410, is shown as the drawing of Figure 43. The initial manufacturing decision was to produce this part by forging it from a simplified preform shown in Figure 44. Forge preforms were die-pressed and sintered and forge trials were conducted with the outcome that, using a part temperature of 980°C (1800°F) and forge pressures as high as 965 MPa (70 ton/in$^2$), the compact could not be made to flow inward enough to satisfactorily form the slot. The inner gimbal presented a very different forging requirement from the gimbal ring. The gimbal ring's major metal movement during forging is vertical, which occurs as and after the walls have thickened sufficiently to take up the clearance gap. The inner gimbal, however, has a single height but, with the forge preform first attempted, requires a large lateral (inward) flow of metal to form the circular bore and slot around the forging core rod. A cross section of this forging is illustrated in Figure 45. It was concluded that, with its small size and large surface-to-volume ratio, the piece

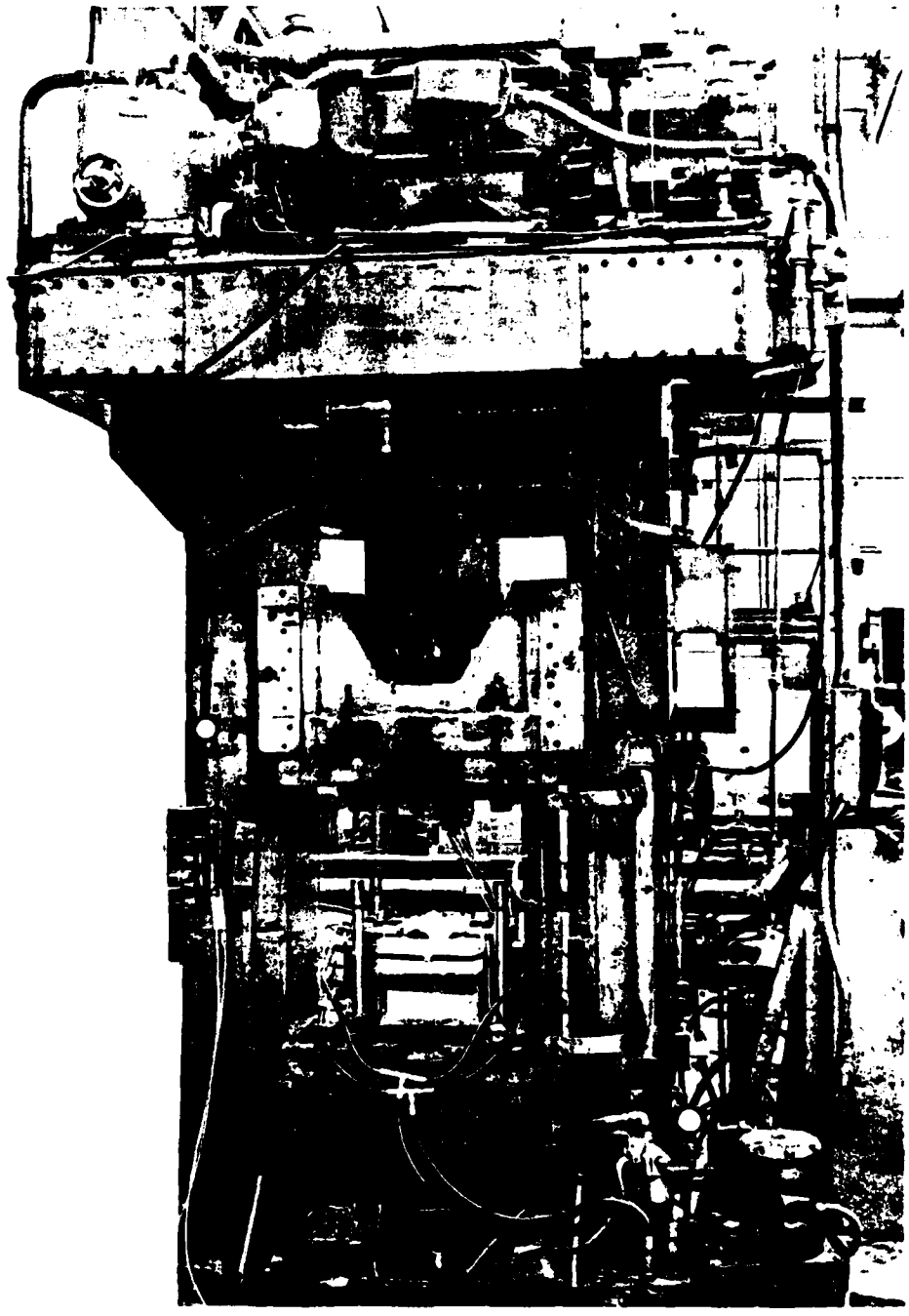


Figure 38. 150 Ton Forge Press with Die Nest in Place.

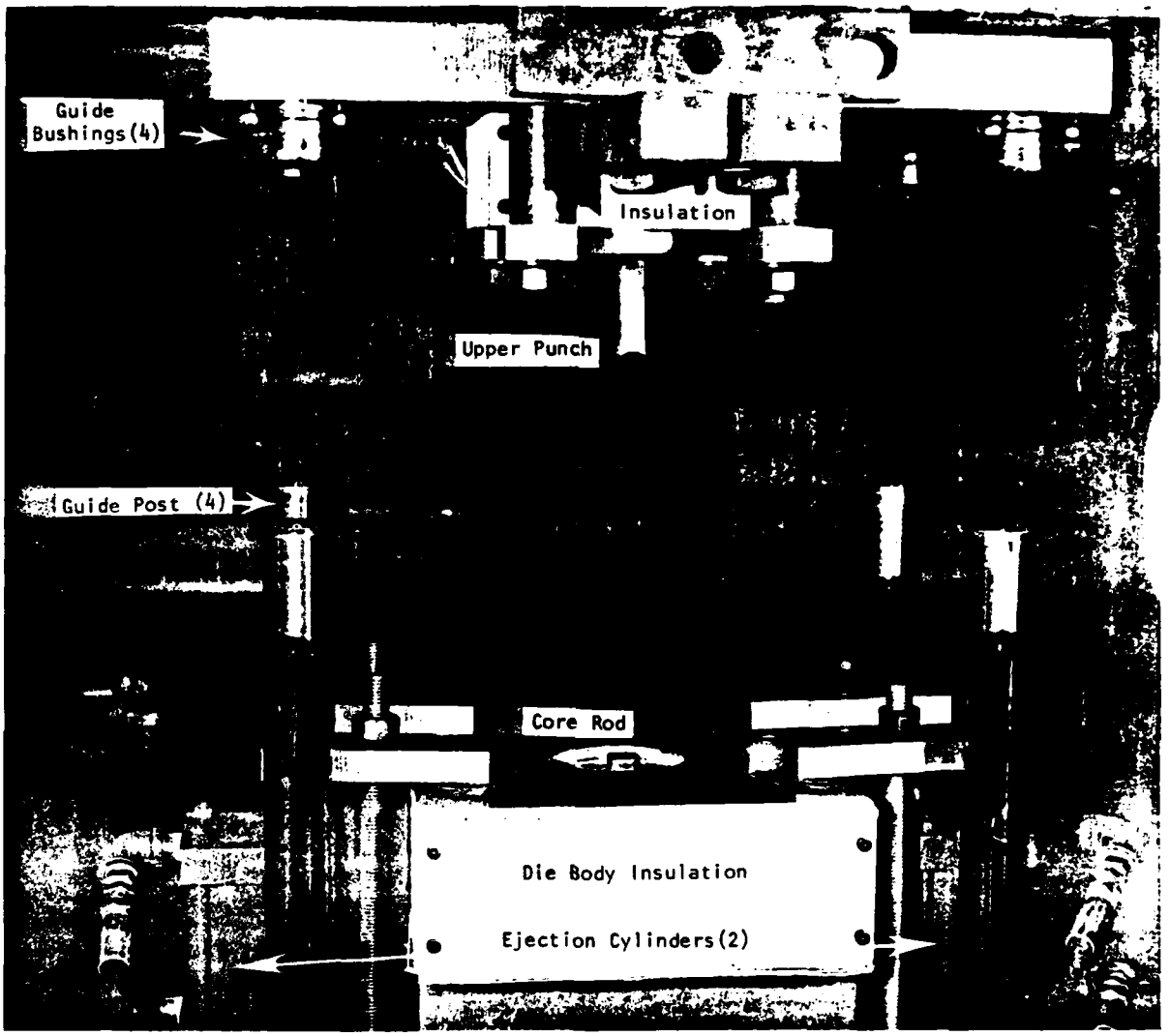



Figure 39. Forging Die Nest for Ti P/M Gyro Gimbals, Front.

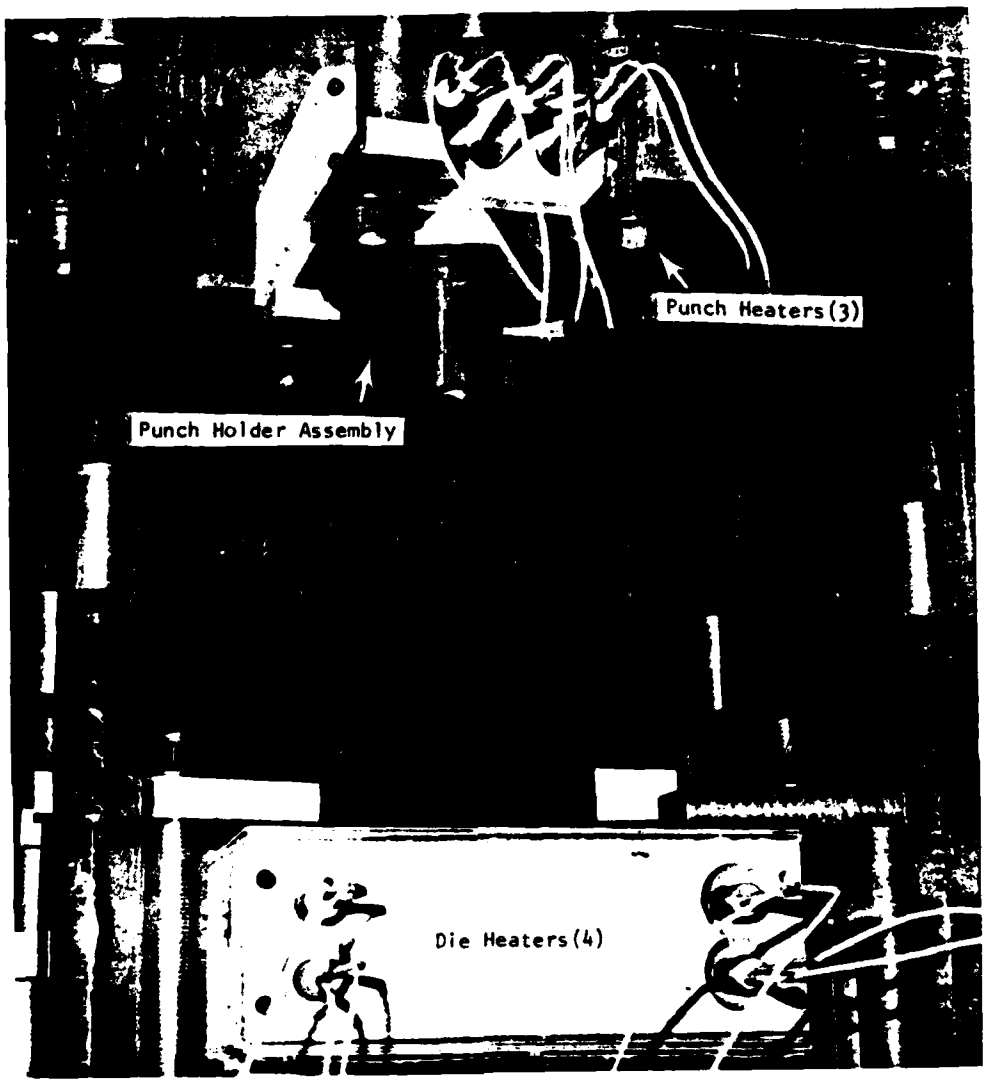



Figure 40. Forging Die Nest for Ti P/M Gyro Gimbals, Rear.

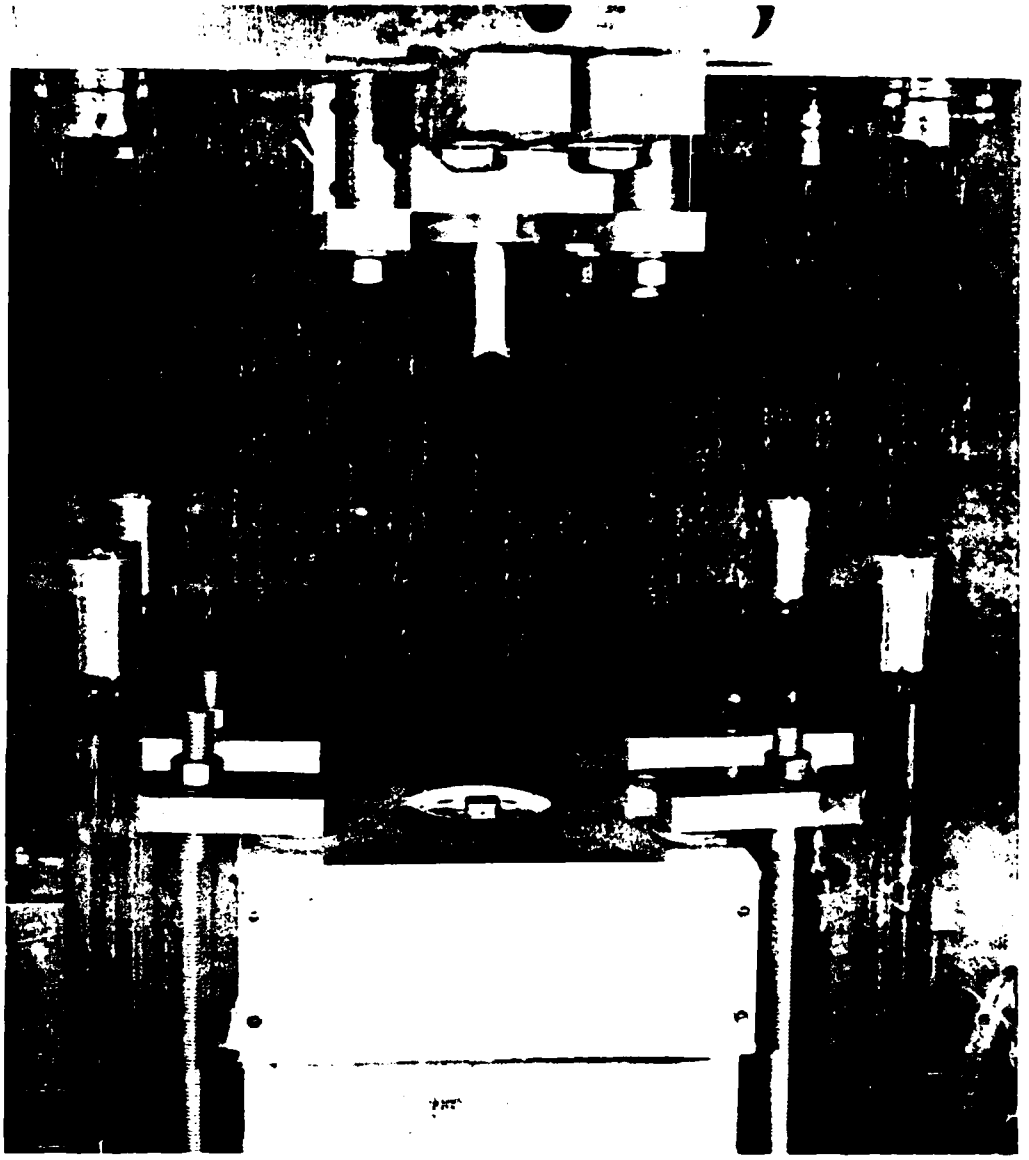


Figure 41. Forging Die Nest for Ti P/M Gyro Gimbals, Closeup.

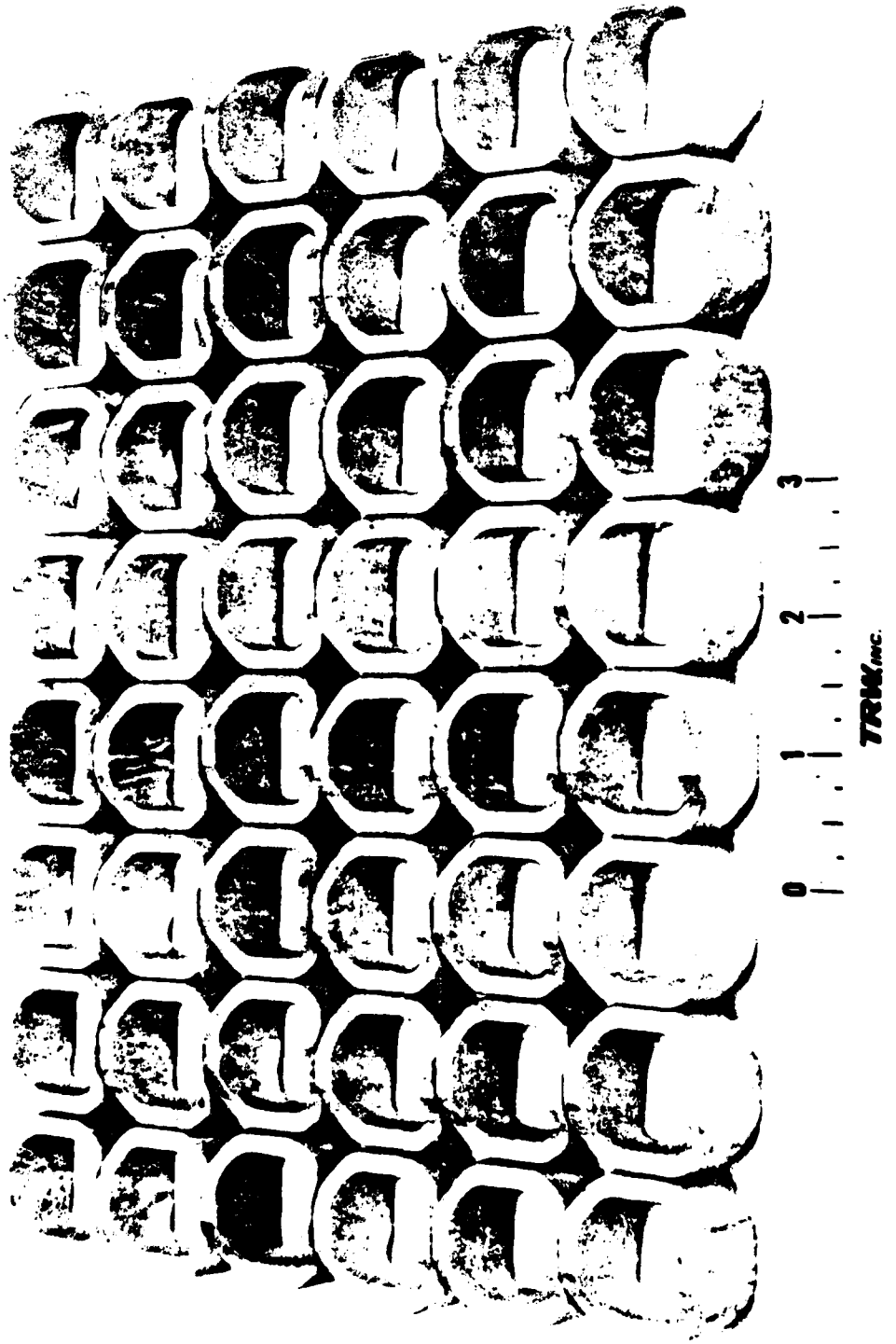



Figure 42. Forged and Cleaned Gimbal Ring Machining Preforms.

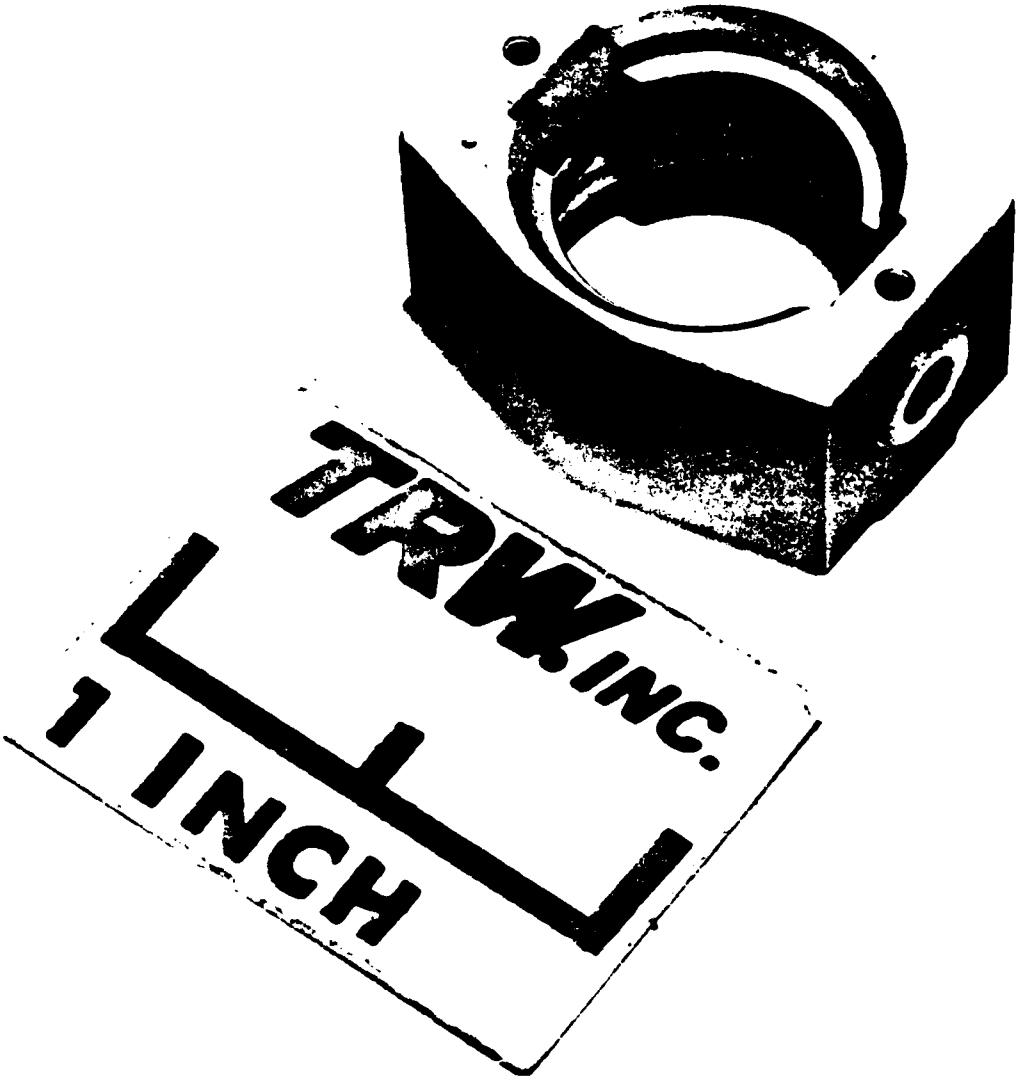



Figure 43. Inner Gimbal, P/N 9301410.

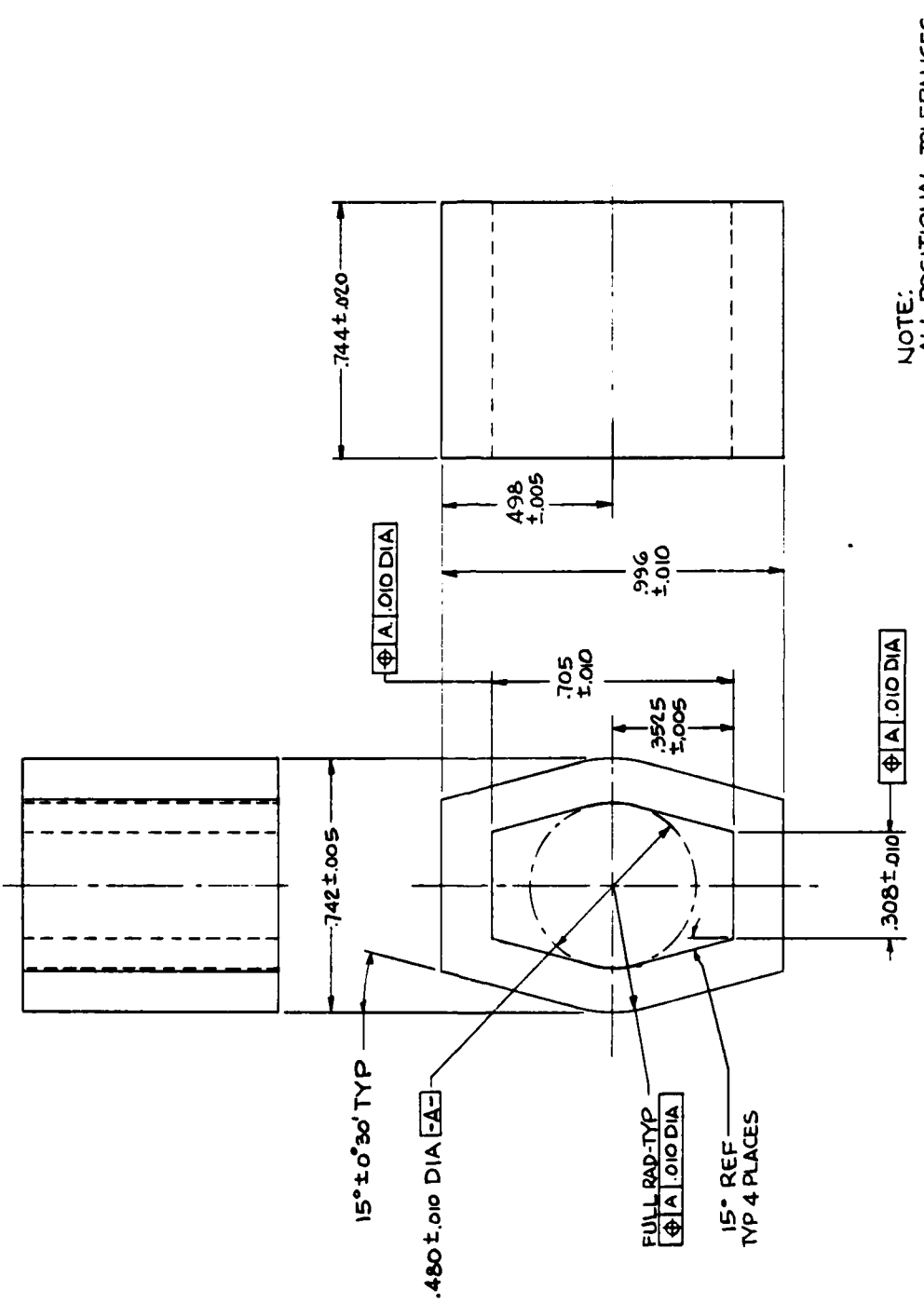



Figure 44. Inner Gimbal Forge Preform, First Design.

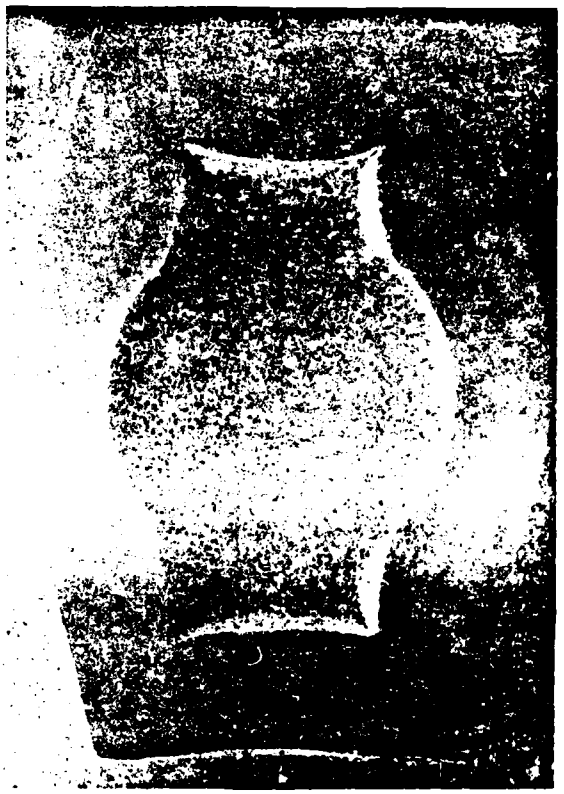


Figure 45. Cross Section Forged from "Simplified" Forge Preform.

lost too much of its heat to the 180°C (350°F) tools to be plastic enough for the required deformation. The briquetting tools were therefore modified to produce a forge preform similar in shape to the desired final part, i.e., with bore and slot, but oversize (see Figure 46).

While this concept appeared sound, it failed in execution when the upper forging punch snapped while forging the first piece. Time constraints did not permit the manufacture of a new punch (its keyhole bore was extremely difficult to manufacture), and therefore it was decided to HIP the forge preforms in the same run as the bases; since the inner gimbals fit easily inside the bases, they are, in effect, HIP at no additional cost. Their processing sequence therefore became: Die Press-Sinter-HIP. Figure 47 illustrates the production run of sixty die-pressed-sintered-HIP inner gimbals.

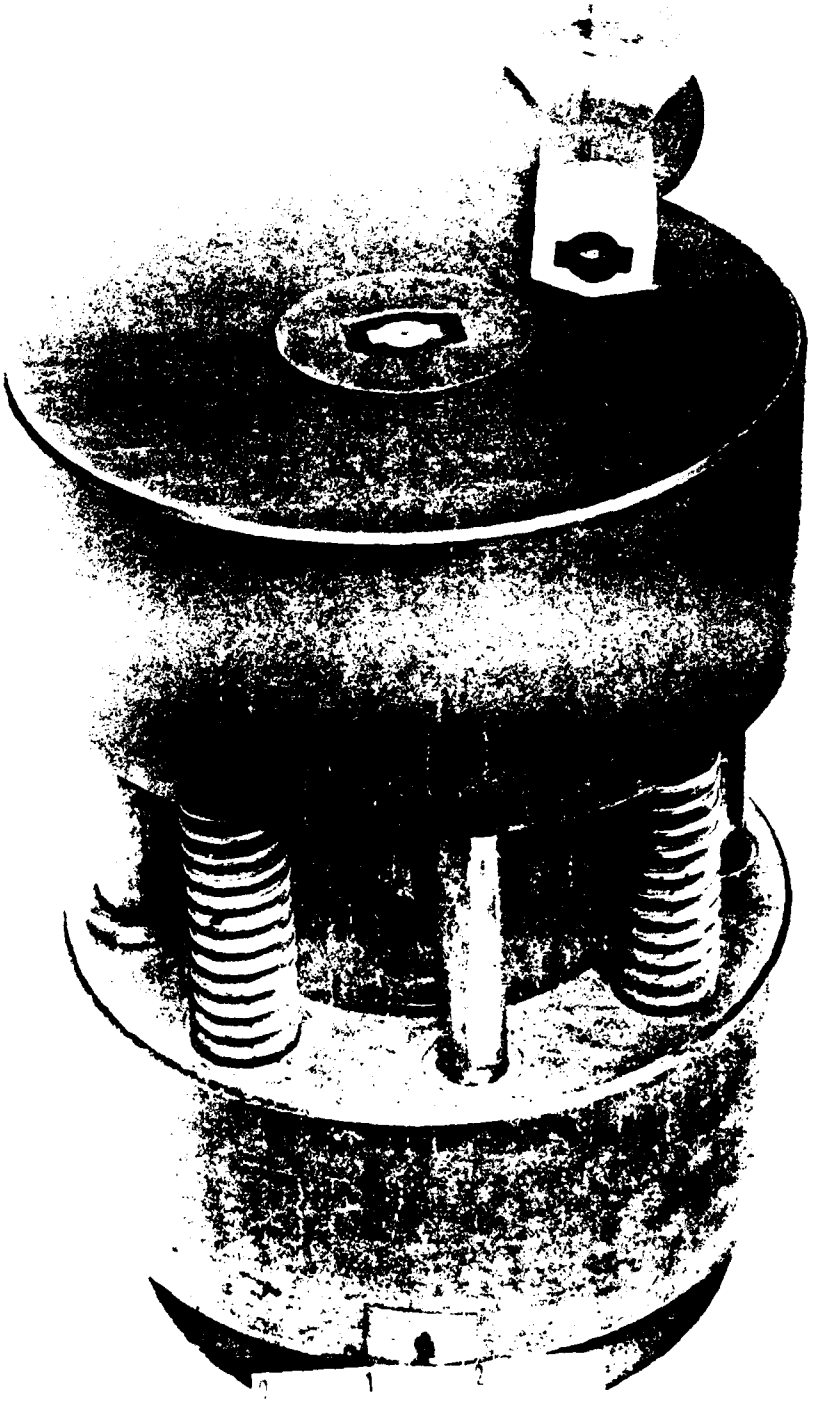


Figure 46. Inner Gimbal Forge Preform, Second Design.

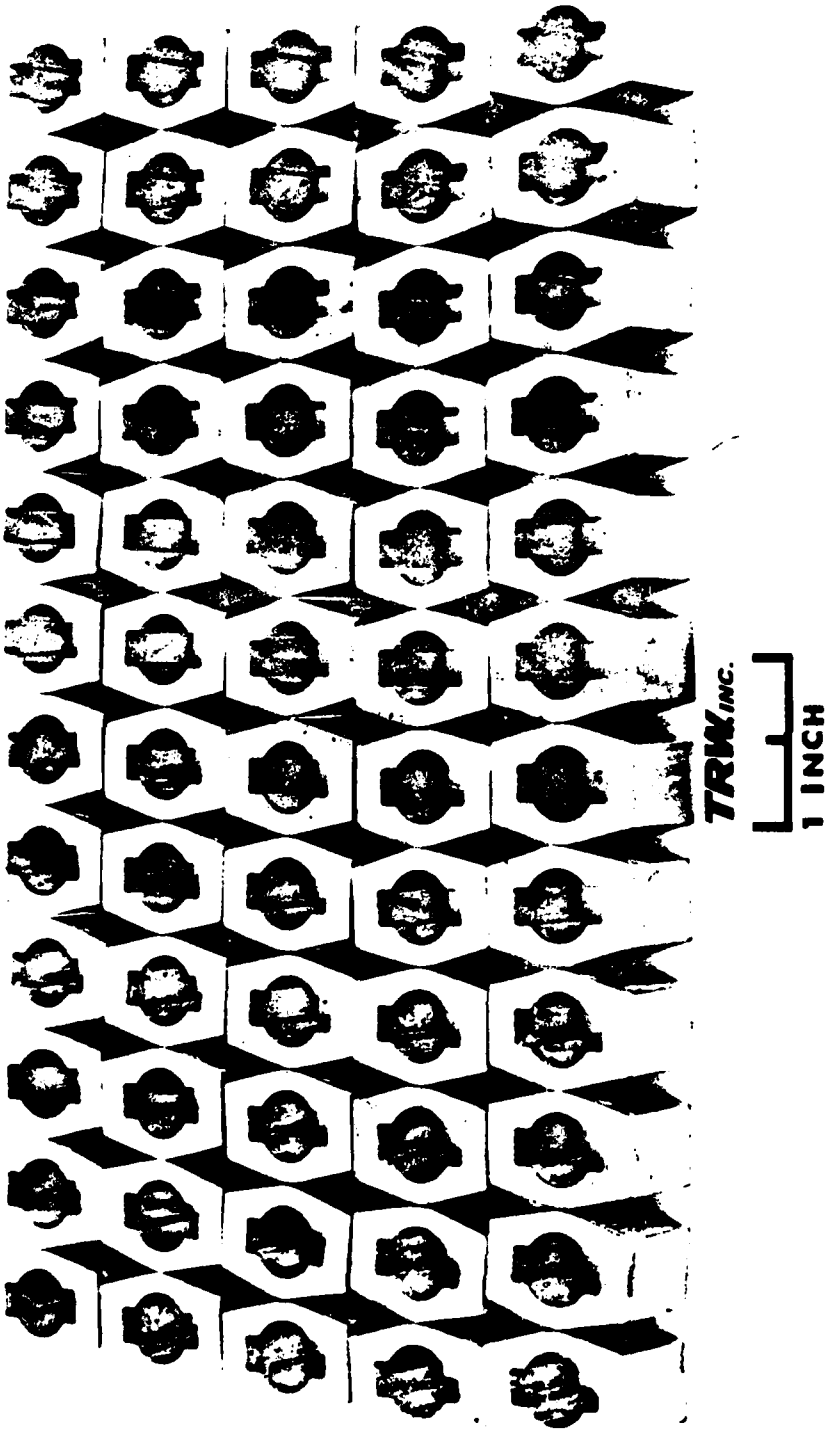



Figure 47. Prototype Production Run of 60 Inner Gimbals.

## 4.0 CONCLUSIONS

The program has demonstrated the validity of two significant premises upon which this work was based:

1. Properties adequate for the intended application(s) can be obtained from blended elemental titanium and master alloy powders through the selection of appropriate processing conditions. Although bench tests and test firings had not been concluded at the time of writing, the static and dynamic test data indicated that the selected material-process combinations would produce strength levels above those needed for each part according to the MMC project personnel.

2. The P/M techniques of die pressing and isostatic pressing made it possible to produce these titanium parts from less material and also eliminated expensive machining operations.

The program can be deemed successful in that its objectives were accomplished. Each of the five gyro machining preforms has been produced from powder.

## 5.0 RECOMMENDATIONS

The knowledge gained through the performance of this program should be utilized in reducing the material input and manufacturing costs for similar types of parts in other ordnance applications. The techniques employed for the manufacture of these gyro parts can be used for other gyros, as well as for such parts as fins, canards, missile bodies, actuating arms, etc. To have the largest effect on cost, such a P/M program should be conducted early in the life of the ordnance system, while material choices are still flexible and new materials are more easily introduced.

# APPENDIX A

## PROCESS DESCRIPTION: ACTUATOR HOUSING

## MACHINING PREFORM

TRW Materials Technology
Process Description
Ti P/M Gyro Machining Preform: Actuator Housing: P/N 408 MP

Material: Ti-6Al-4V Process: Die Press/Sinter

| Operation Number | Operation | Equipment or Facility | Qualified Operator | Process Sheets Spec., Drwg. |
|---|---|---|---|---|
| 408-00 | Mill Al-V master alloy to -100 mesh | Ball mill, 10 mesh SS Screen<br>" " jar, gallon<br>Steel balls | LJ | 8851-01 |
| 408-05 | Determine PSD, AD | Tyler Ro-Tap<br>80-325 mesh SS Screens<br>Balance, Hall apparatus | LJ | MPIF 04<br>MPIF 05 |
| 408-07 | Mill Titanium | Ball mill, jar, balls, as in 408-00 | LJ | 8851-05 |
| 408-10 | Determine PSD, AD for Ti powder | As above, for 408-05 | LJ | MPIF 04,05 |
| 408-12 | Blend Al-V into Ti | Ball mill, jar, balls, as in 408-00; balance | LJ | 4203-12<br>8851-05 |
| 408-20 | Press green compact | Briquetting die, 408-BD-1<br>Hydraulic press | LJ | 8851-120<br>4203-408-20 |
| 408-25 | Inspect compact | Bench<br>0-1 & 1-2" micrometers<br>Balance | LJ<br>GF | Dwg 408-B |
| 408-30 | Sinter | Brew Vacuum Furnace<br>Recorder | LJ<br>JS | 8851-30<br>4203-30 |
| 408-35 | Inspect | Bench, 0-1, 1-2"<br>Micrometer,<br>Balance | LJ<br>GF | Dwg. 408-MP |

TRW Materials Technology

Procedure 8851-01: Powder Milling

1. Use new steel jar, one that has been last used for titanium or one that has been bored out to remove any metal contamination. If new or machined, "condition" with scrap titanium powder for 1 hour.
2. Screen Al-V to -10 mesh.
3. Fill jar 1/4 full with powder ($\approx$1200 g for 6 inch jar).
4. Add 3/4-7/8 inch Ø steel balls either new or previously used for this material so that powder plus balls half fill the jar.
5. Seal jar.
6. Place jar on mill and run for 2 hours.
7. Remove jar from mill.
8. Tap lid to settle powder.
9. Open jar.
10. Use 10 mesh screen to separate balls from powder.

MPIF STANDARD NO. 04

METAL POWDER INDUSTRIES FEDERATION
Method for

# DETERMINATION OF APPARENT DENSITY OF FREE-FLOWING METAL POWDERS USING THE HALL APPARATUS

MPIF Standard 04
Issued 1945, Adopted 1948, Revised 1972



## 1. SCOPE

1.1 This standard describes a method for determining the apparent density of free-flowing metal powders and is only suitable for those powders which will flow unaided through the specified Hall flowmeter funnel. See MPIF Standard No. 28 for apparent density of non-free-flowing metal powders.

## 2. APPARATUS

2.1 The following apparatus is required to perform this determination.

2.1.1 Hall Flowmeter Funnel: A standard flowmeter funnel (Fig. 1) having an orifice of 0.10 in. in diameter (2.54 mm).

2.1.2 Density Cup: A cylindrical cup (Fig. 2) having a capacity of 25 ± 0.05 cc.

2.1.3 A support (Fig. 3) to hold the flowmeter funnel concentric with the density cup so that the bottom of the orifice is 1.0" (approx. 25 mm) above the top of the density cup when assembled, (Fig. 4).

2.1.4 Base: A vibration-free table to support the flowmeter assembly.

2.1.5 Balance: A balance having a capacity of at least 200 g and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen shall consist of a volume of approximately 30 to 40 cc of dry metal powder.

Note 1 – The powder is dry when there is no weight loss as the result of conditioning it for one hour in a drying oven at 215 to 225 F (102 to 107 C) and cooling to room temperature in a dessicator.

3.1.1 The test specimen shall be a representative sample obtained in accordance with MPIF Standard No. 01.

## 4. PROCEDURE

4.1 The dry test specimen shall be carefully loaded into the flowmeter funnel and permitted to run into the density cup through the discharge orifice. Care must be taken not to move the density cup.

4.2 When the powder completely fills and overflows the periphery of the density cup, the funnel shall be rotated approximately 90° in a horizontal plane so that the remaining powder falls away from the cup.

4.3 Using a non-magnetic spatula with the blade held perpendicular to the top of the cup, the powder shall be leveled off flush with the top of the density cup. Care must be taken to avoid jarring the apparatus at any time.

4.4 After the leveling operation, the density cup should be lightly tapped on the side to settle the powder to avoid spilling in transfer.

4.5 The powder shall be transferred to the balance and weighed to the nearest 0.05 g.

## 5. REPORT

5.1 The weight in grams of the powder from the leveled density cup, multiplied by 0.04, shall be reported as the apparent density to the nearest 0.1 g/cm³.

## APPENDIXES

### A1. REPORT ON PRECISION OF MPIF STANDARD NO. 04

A1.1 A planned testing program was carried out among users of the Hall flowmeter apparatus to obtain data to determine the precision of this method. The complete report on this work is available in the offices of the Metal Powder Industries Federation.

A1.2 The report concludes that MPIF Standard No. 04 is operable, in the testing of free-flowing metal powder, with a precision of V = 2.1%.

### A2. COMPARABLE STANDARDS

ASTM B 212
Japanese JIS Z 2504–1966
German 83–69

Metric system conversion factors for all dimensions referred to in this Standard are available in the ASTM Metric Practice Guide, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

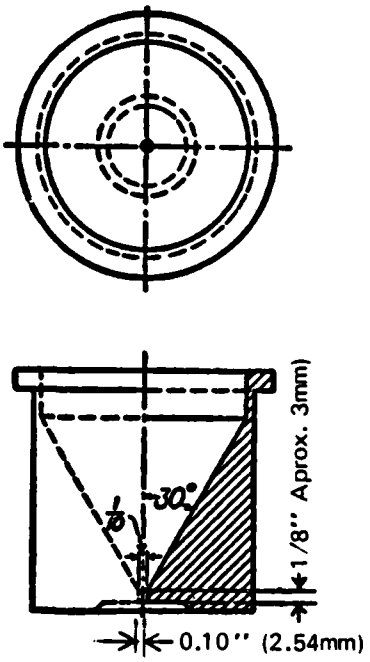



Fig. 1 Hall Flowmeter Funnel

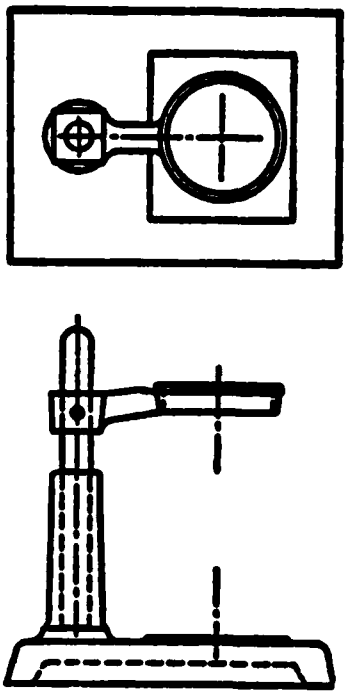


Fig. 3 Stand

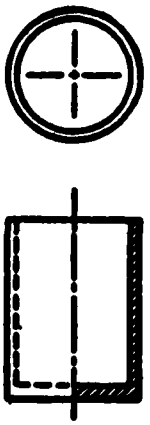


Fig. 2 25cc Density Cup

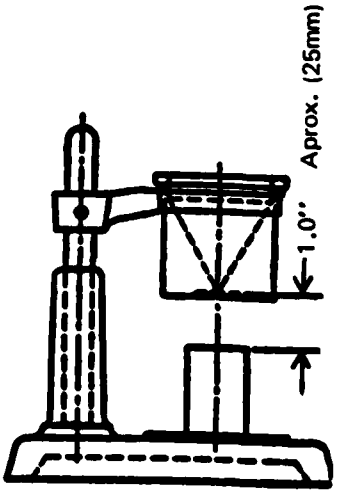



Fig. 4 Assembly

weight of the fraction collected in the pan.

5. REPORT

5.1 The weights of the fractions retained on each sieve, and the weight of that fraction collected in the pan, shall be expressed as percentages of the test specimen weight to the nearest 0.1 percent, and reported in the following form. Any screen fraction whose percentage of the test specimen weight is less than 0.1 percent shall be reported as "trace."

Table II – Form for Reporting Test Data

| Mesh (Tyler or U.S.S.) | Percentage by Weight |
|---|---|
| + 80 | ..... |
| – 80 +100 | ..... |
| –100 +150 | ..... |
| –150 +200 | ..... |
| –200 +325 | ..... |
| –325 | ..... |

APPENDIX

A1. Method for Obtaining Sieve Correction

Table III –

| Mesh | Certified Sieve | Work Sieve | Factor by Which to Multiply Work Sieve to Convert to Standard |
|---|---|---|---|
| + 80 | 0.1 | 0.1 | 0.1/0.1 = 1. |
| – 80 +100 | 3.0 | 3.0 | 3.0/3.0 = 1. |
| –100 +150 | 10.0 | 10.0 | 10.0/10.0 = 1. |
| –150 +200 | 20.0 | 20.0 | 20.0/20.0 = 1. |
| –200 +325 | 5.0 | 4.0 | 5.0/4.0 = 1.25 |
| –325 | 21.9 | 27.9 | 21.9/27.9 = 0.785 |

A2. Certified Sieve

Sieves conforming to ASTM Specification E11 can be obtained from the sieve manufacturers, and arrangements can be made through them to have the sieves certified by the Bureau of Standards. If used continually, the sieves will, after a period of time, become less accurate and might no longer be acceptable as certified sieves. The common practice, which would be considered acceptable according to this standard, would be to use the certified sieves as a master set for checking other working sets of sieves. By comparing sieve tests on the same sample, run in both the master set and working set, a factor can be established for correcting results on the working sieves. These factors will vary with the coarseness of the powder and should be established for powders of different particle size distribution. The method for obtaining this factor is illustrated by the example given in Table III. All results obtained on the working sieves them would be multiplied by the factor so obtained before reporting.

A3. Comparable Standards

ASTM – B 214

German – 81-69

MPIF STANDARD NO. 05

METAL POWDER INDUSTRIES FEDERATION

Method for

# DETERMINATION OF SIEVE ANALYSIS OF METAL POWDERS

MPIF Standard 05

Issued 1945, Revised 1949, Adopted 1949, Revised 1962, 1973



## 1. SCOPE

1.1 This method covers the test for the sieve analysis of standard compacting grade granular metal powders.

## 2. APPARATUS

2.1 Sieves: A set of standard sieves selected from Table No. 1, ASTM Specification E11, or the equivalent Tyler Standard Screen Scale Sieves. The sieves shall be 8 in. (200 mm) in diameter and either 1 or 2 in. (25 or 50 mm) in depth and fitted with brass, bronze, stainless steel or other suitable wire. The sieves shall conform to ASTM Specification E11. The following sieves shall be used for the sieve analysis of metal powders 80 mesh or finer:

Table I – Testing sieves according to Tyler Standard Screen Scale - U.S. Standard or ASTM Series.

| ASTM Sieves, Size and U.S. Standard Sieve Designation | Tyler Sieves, Size and Tyler Sieve Series Designation |
|---|---|
| 177 micron (No. 80) | 175 micron ( 80 mesh) |
| 149 micron (No. 100) | 149 micron (100 mesh) |
| 105 micron (No. 140) | 104 micron (150 mesh) |
| 74 micron (No. 200) | 74 micron (200 mesh) |
| 44 micron (No. 325) | 44 micron (325 mesh) |

2.2 Sieve Shaker: A mechanically operated single eccentric sieve shaker which imparts to the nest of sieves a rotary motion of 285 rpm plus or minus 15, and a tapping action of 150 taps per minute plus or minus 10. The sieve shaker shall be fitted with a plug to receive the impact of the tapping device. The entire apparatus shall be mounted rigidly, and preferably shall be provided with a time switch to insure accuracy of duration of the test.

2.3 A balance having a capacity of at least 100 g. and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen, obtained in accordance with MPIF Standard 01, shall be 100 g. of any powder the apparent density of which, determined by MPIF Standard 04, is greater than 1.50 g/cm³.
If the apparent density of the powder is less than 1.50 g/cm³, a 50 g. sample shall be used.

## 4. PROCEDURE

4.1 The group of sieves selected shall be assembled in consecutive order as to size of openings, with the sieve having the largest opening at the top, the assembly being completed by a solid collecting pan below the bottom sieve. The test specimen shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly then shall be fastened securely in a suitable mechanical sieve shaking device and operated for a period of 15 minutes.

4.2 The sieved fractions shall be removed from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them upon a glazed paper. Any material adhering to the bottom of the sieve and frame shall be brushed with a soft brush into the next finer sieve. The sieve just removed then shall be tapped, upside down, on the paper containing the portion that had been retained on it. This fraction shall be weighed to within 0.1 g. This process shall be repeated for each sieve in the nest and the fraction collected in the pan shall also be removed and weighed. The sum of the weights of all the fractions shall be not less than 99 percent of the test specimen weight, and the difference between this sum and 100 (or 50 in case of powders the apparent density of which is less than 1.50 g/cm³) shall be added to the

Metric system conversion factors for all dimensions referred to in this Standard are available in the ASTM Metric Practice Guide, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

TRW Materials Technology

Procedure 8851-05: Mill powder to increase its apparent density and flow rate.

A. Equipment

1. Ball mill.
2. Ball mill jar.
3. Steel balls, 3/4-7/8 inch diameter.
4. Hall apparatus.
5. Top-loading electronic balance.

B. Procedure

1. Mill powder, as in Procedure 8851-01 for 1/2, 1, or 2 hours, as required, to increase its apparent density to 1.35-1.58 $g/cm^3$ (30-35% of theoretical).

TRW Materials Technology

Procedure 4203-12: Powder Blending
(To be used in conjunction with Procedure 8851-01 "Powder Milling")

1. Weigh out Ti and Al-V in ratio of 90:10 using electronic top-loading balance, Ex 1200 g charge = 120 g Al-V
   1080 g Ti

2. Fill jar with powder and balls as per 8851-01 and blend for 1/2 hour.

TRW Materials Technology

Procedure 8851-120: Die Pressing (Briquetting) of Metal Compacts

A. Equipment:

1. Briquetting die set.
2. Powder compaction press, hydraulic.

B. Procedure:

1. Inspect wall of die and core rod, if any, and remove any adherent metal.
2. Adjust lower punch to provide correct powder fill depth.
3. Use 3.35 inch test bar die to check press for platen parallelism left-to-right and front-to-back. Shim as required, with a 1/4 inch steel plate over the shims.
4. When platens are parallel within 0.002 inch over 3.35 inch, place required briquetting die in center of lower platen. Connect pressure recorder to press.

(4a. Lubricate die wall.)

5. Add powder, tap die, and level off the powder.
6. Fit upper punch into die so that top of punch is horizontal and punch is at least 1/64 inch into die.
7. Apply required pressure to top punch. Hold 5 seconds.
8. Release pressure.
9. Remove to punch.
10. Use ejector ring to eject the compact. Note ejection force.

AD-A125 345 COPPERHEAD ALTERNATE MANUFACTURING PROCESS(U) MARTIN MARIETTA AEROSPACE ORLANDO FL G I FRIEDMAN NOV 81 DAAK10-78-C-0070 2/2

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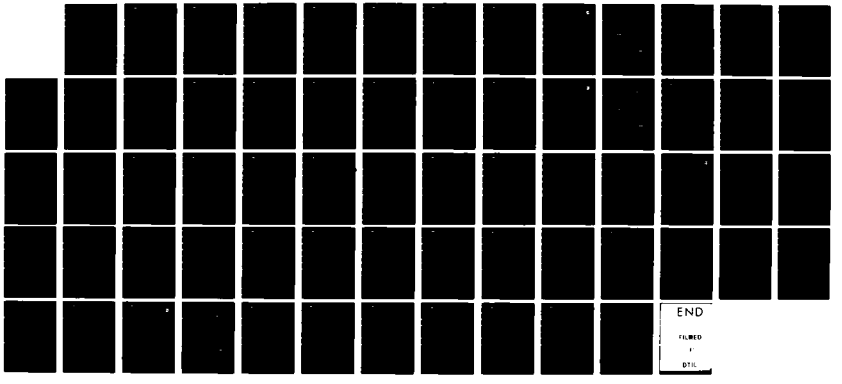

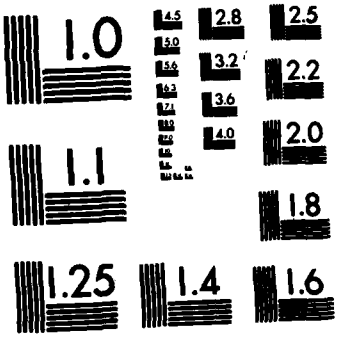


MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

TRW Materials Technology

Procedure 4203-408-20, Rev. 0: Briquetting of Actuator Housing

(To be used in conjunction with Procedure 8851-120, "Die Pressing (Briquetting) of Metal Compacts.")

1. Briquetting pressure for this part is 30 ton/in$^2$.
   Die area 0.45 in$^2$; briquetting force = 13.5 tons ± 1 ton.

TRW Materials Technology

Procedure 4203-408-20, Rev. 0: Briquetting of Actuator Housing

(To be used in conjunction with Procedure 8851-120, "Die Pressing (Briquetting) of Metal Compacts.")

1. Briquetting pressure for this part is 30 ton/$in^2$.
   Die area $\approx$0.45 $in^2$; briquetting force = 13.5 tons ± 1 ton.

TRW Materials Technology

Procedure 8851-30: Sintering Green Compacts

A. Equipment:

1. Brew Model 426 vacuum furnace or equivalent.
2. L-H Penningvac cold cathode vacuum gauge with electrical readout.
3. Strip-chart recorder.

B. Procedure:

1. Place an outgassed BN-coated moly foil on furnace hearth, BN facing upward.
2. Place compacts on BN surface, with space between compacts, and not compact closer than 1/4 inch from the furnace heating element.
3. Close furnace and pump down.
4. Blank chamber off from pump and leak rate. Do not start run if leak rate exceeds 10 microns/minute.
5. Connect thermocouple and vacuum gauge to recorder. Identify channels, FSD values, and chart speed on Recorder Data Sheet.

TRW Materials Technology

Procedure 4203-30: Sintering Titanium Powder Compacts

(To be used in conjunction with 8851-30, "Sintering Green Compacts.")

1. Initial chamber pressure should be $<10^{-4}$ torr.
2. Heat sample to 1000°F at rate such that chamber pressure does not exceed $10^{-4}$ torr. Hold ≥15 minutes at this temperature.
3. Heat to 1600°F with same pressure control as in (1). Hold at least 15 minutes at this temperature.
4. Heat to 2250 ± 25°F with same pressure control as in (1). Hold at temperature for 4 hours.

# APPENDIX B

## PROCESS DESCRIPTION: GOTCHA LOCK

TRW Materials Technology
Process Description
Ti P/M Gyro Machining Preform: Gotcha Lock: P/N 407 MP

Materials: CP Titanium Process: CIP/Sinter/HIP

| Operation Number | Operation | Equipment or Facility | Qualified Operator | Process Sheets Spec., Drwg. |
|---|---|---|---|---|
| 407-05 | Mill titanium powder | Ball mill, ball mill jar, Steel balls Hall apparatus Balance | LJ | 8851-05 |
| 407-10 | Determine powder size distribution | Tyler Ro-Tap 80-325 mesh screens, balance | LJ | MPIF 04 MPIF 05 |
| 407-11 | Analyze powder: Fe, Na, Cl, C, O, H, N | Chem Lab | | |
| 407-20 | Press Green compact | Rubber bag set 407-B-2 Mandrel 407-T2 CIP Unit . | LJ | 8851-20 4203-407-CIP |
| 407-21 | Turn to 2.8 inch ø | Lathe | | |
| 407-23 | Remove mandrel | Arbor press or Hydraulic press | LJ | |
| 407-25 | Inspect compact | Bench 0-1 micrometer; 6" vernier caliper; Balance | LJ GF | Dwg. 407-CIP |
| 407-30 | Sinter compact | Brew or NRC Vacuum furnace, recorder | LJ JS | 8851-30 4203-30 |
| 407-35 | Inspect compact | Bench; 6" vernier caliper, Balance | LJ GF | Dwg. 407-2 |
| 407-40 | HIP | HIP unit | JA | 8851-40,4203- |
| 407-45 | Inspect | Bench, 6" vernier | LJ | Dwg. 407-MP |
| 407-46 | Hot Size | Sizing Mandrel Furnace | | Dwg. 407-SM 4203-407-HS |
| 407-50 | Clean | Acid Room | LJ | TAP-PS-645A TAP-PS-518B |
| 407-60 | Inspect | Bench 6" vernier caliper | LJ GF | Dwg. 407-MP |

TRW Materials Technology

Procedure 8851-05: Mill powder to increase its apparent density and flow rate.

A. Equipment

1. Ball mill.
2. Ball mill jar.
3. Steel balls, 3/4-7/8 inch diameter.
4. Hall apparatus.
5. Top-loading electronic balance.

B. Procedure

1. Mill powder, as in Procedure 8851-01 for 1/2, 1, or 2 hours, as required, to increase its apparent density to 1.35-1.58 $g/cm^3$ (30-35% of theoretical).

MPIF STANDARD NO. 04

METAL POWDER INDUSTRIES FEDERATION
Method for

# DETERMINATION OF APPARENT DENSITY OF FREE-FLOWING METAL POWDERS USING THE HALL APPARATUS

MPIF Standard 04
Issued 1945, Adopted 1948, Revised 1972



## 1. SCOPE

1.1 This standard describes a method for determining the apparent density of free-flowing metal powders and is only suitable for those powders which will flow unaided through the specified Hall flowmeter funnel. See MPIF Standard No. 28 for apparent density of non-free-flowing metal powders.

## 2. APPARATUS

2.1 The following apparatus is required to perform this determination.

2.1.1 Hall Flowmeter Funnel: A standard flowmeter funnel (Fig. 1) having an orifice of 0.10 in. in diameter (2.54 mm).

2.1.2 Density Cup: A cylindrical cup (Fig. 2) having a capacity of 25 ± 0.05 cc.

2.1.3 A support (Fig. 3) to hold the flowmeter funnel concentric with the density cup so that the bottom of the orifice is 1.0" (approx. 25 mm) above the top of the density cup when assembled, (Fig. 4).

2.1.4 Base: A vibration-free table to support the flowmeter assembly.

2.1.5 Balance: A balance having a capacity of at least 200 g and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen shall consist of a volume of approximately 30 to 40 cc of dry metal powder.

Note 1 — The powder is dry when there is no weight loss as the result of conditioning it for one hour in a drying oven at 215 to 225 F (102 to 107 C) and cooling to room temperature in a dessicator.

3.1.1 The test specimen shall be a representative sample obtained in accordance with MPIF Standard No. 01.

## 4. PROCEDURE

4.1 The dry test specimen shall be carefully loaded into the flowmeter funnel and permitted to run into the density cup through the discharge orifice. Care must be taken not to move the density cup.

4.2 When the powder completely fills and overflows the periphery of the density cup, the funnel shall be rotated approximately 90° in a horizontal plane so that the remaining powder falls away from the cup.

4.3 Using a non-magnetic spatula with the blade held perpendicular to the top of the cup, the powder shall be leveled off flush with the top of the density cup. Care must be taken to avoid jarring the apparatus at any time.

4.4 After the leveling operation, the density cup should be lightly tapped on the side to settle the powder to avoid spilling in transfer.

4.5 The powder shall be transferred to the balance and weighed to the nearest 0.05 g.

## 5. REPORT

5.1 The weight in grams of the powder from the leveled density cup, multiplied by 0.04, shall be reported as the apparent density to the nearest 0.1 g/cm³.

## APPENDIXES

### A1. REPORT ON PRECISION OF MPIF STANDARD NO. 04

A1.1 A planned testing program was carried out among users of the Hall flowmeter apparatus to obtain data to determine the precision of this method. The complete report on this work is available in the offices of the Metal Powder Industries Federation.

A1.2 The report concludes that MPIF Standard No. 04 is operable, in the testing of free-flowing metal powder, with a precision of V = 2.1%.

### A2. COMPARABLE STANDARDS

ASTM B 212
Japanese JIS Z 2504—1966
German 53—69

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Metric system conversion factors for all dimensions referred to in this Standard are available in the ASTM Metric Practice Guide, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

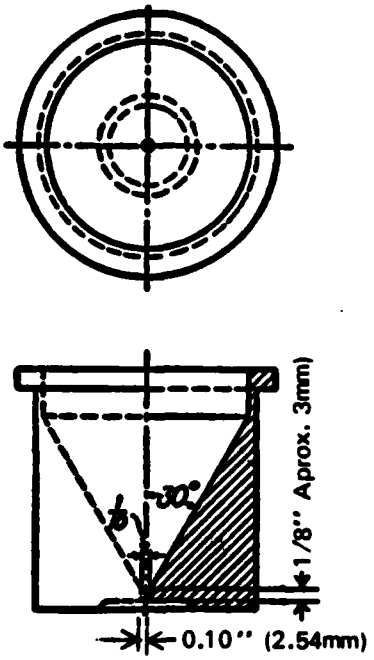


Fig. 1 Hall Flowmeter Funnel

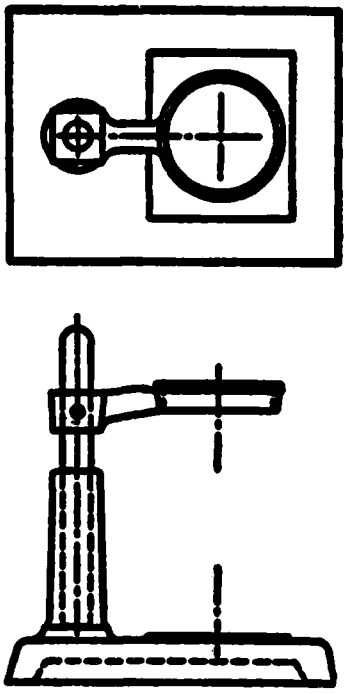

Fig. 3 Stand

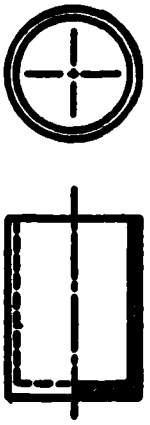

Fig. 2 25cc Density Cup

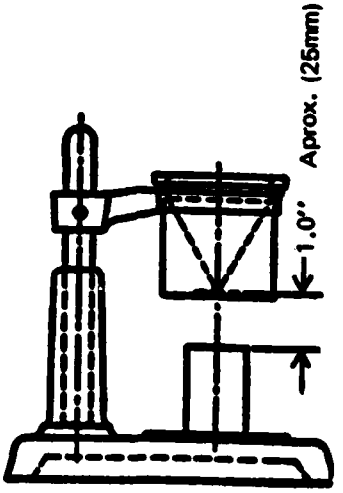


Fig. 4 Assembly

MPIF STANDARD NO. 05

METAL POWDER INDUSTRIES FEDERATION

Method for

# DETERMINATION OF SIEVE ANALYSIS OF METAL POWDERS

MPIF Standard 05

Issued 1945, Revised 1949, Adopted 1949, Revised 1962, 1973



## 1. SCOPE

1.1 This method covers the test for the sieve analysis of standard compacting grade granular metal powders.

## 2. APPARATUS

2.1 Sieves: A set of standard sieves selected from Table No. 1, ASTM Specification E11, or the equivalent Tyler Standard Screen Scale Sieves. The sieves shall be 8 in. (200 mm) in diameter and either 1 or 2 in.(25 or 50 mm) in depth and fitted with brass, bronze, stainless steel or other suitable wire. The sieves shall conform to ASTM Specification E11. The following sieves shall be used for the sieve analysis of metal powders 80 mesh or finer:

Table I — Testing sieves according to Tyler Standard Screen Scale - U.S. Standard or ASTM Series.

| ASTM Sieves, Size and U.S. Standard Sieve Designation | Tyler Sieves, Size and Tyler Sieve Series Designation |
|---|---|
| 177 micron (No. 80) | 175 micron ( 80 mesh) |
| 149 micron (No. 100) | 149 micron (100 mesh) |
| 105 micron (No. 140) | 104 micron (150 mesh) |
| 74 micron (No. 200) | 74 micron (200 mesh) |
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2.2 Sieve Shaker: A mechanically operated single eccentric sieve shaker which imparts to the nest of sieves a rotary motion of 285 rpm plus or minus 15, and a tapping action of 150 taps per minute plus or minus 10. The sieve shaker shall be fitted with a plug to receive the impact of the tapping device. The entire apparatus shall be mounted rigidly, and preferably shall be provided with a time switch to insure accuracy of duration of the test.

2.3 A balance having a capacity of at least 100 g. and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen, obtained in accordance with MPIF Standard 01, shall be 100 g. of any powder the apparent density of which, determined by MPIF Standard 04, is greater than 1.50 g/cm³.
If the apparent density of the powder is less than 1.50 g/cm³, a 50 g. sample shall be used.

## 4. PROCEDURE

4.1 The group of sieves selected shall be assembled in consecutive order as to size of openings, with the sieve having the largest opening at the top, the assembly being completed by a solid collecting pan below the bottom sieve. The test specimen shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly then shall be fastened securely in a suitable mechanical sieve shaking device and operated for a period of 15 minutes.

4.2 The sieved fractions shall be removed from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them upon a glazed paper. Any material adhering to the bottom of the sieve and frame shall be brushed with a soft brush into the next finer sieve. The sieve just removed then shall be tapped, upside down, on the paper containing the portion that had been retained on it. This fraction shall be weighed to within 0.1 g. This process shall be repeated for each sieve in the nest and the fraction collected in the pan shall also be removed and weighed. The sum of the weights of all the fractions shall be not less than 99 percent of the test specimen weight, and the difference between this sum and 100 (or 50 in case of powders the apparent density of which is less than 1.50 g/cm³) shall be added to the

weight of the fraction collected in the pan.

**5. REPORT**

5.1 The weights of the fractions retained on each sieve, and the weight of that fraction collected in the pan, shall be expressed as percentages of the test specimen weight to the nearest 0.1 percent, and reported in the following form. Any screen fraction whose percentage of the test specimen weight is less than 0.1 percent shall be reported as "trace."

Table II – Form for Reporting Test Data

| Mesh (Tyler or U.S.S.) | Percentage by Weight |
|---|---|
| + 80 | ..... |
| – 80 +100 | ..... |
| –100 +150 | ..... |
| –150 +200 | ..... |
| –200 +325 | ..... |
| –325 | ..... |

APPENDIX

A1. Method for Obtaining Sieve Correction

Table III –

| Mesh | Certified Sieve | Work Sieve | Factor by Which to Multiply Work Sieve to Convert to Standard |
|---|---|---|---|
| + 80 | 0.1 | 0.1 | 0.1/0.1 = 1. |
| – 80 +100 | 3.0 | 3.0 | 3.0/3.0 = 1. |
| –100 +150 | 10.0 | 10.0 | 10.0/10.0 = 1. |
| –150 +200 | 20.0 | 20.0 | 20.0/20.0 = 1. |
| –200 +325 | 5.0 | 4.0 | 5.0/4.0 = 1.25 |
| –325 | 21.9 | 27.9 | 21.9/27.9 = 0.785 |

A2. Certified Sieve

Sieves conforming to ASTM Specification E11 can be obtained from the sieve manufacturers, and arrangements can be made through them to have the sieves certified by the Bureau of Standards. If used continually, the sieves will, after a period of time, become less accurate and might no longer be acceptable as certified sieves. The common practice, which would be considered acceptable according to this standard, would be to use the certified sieves as a master set for checking other working sets of sieves. By comparing sieve tests on the same sample, run in both the master set and working set, a factor can be established for correcting results on the working sieves. These factors will vary with the coarseness of the powder and should be established for powders of different particle size distribution. The method for obtaining this factor is illustrated by the example given in Table III. All results obtained on the working sieves them would be multiplied by the factor so obtained before reporting.

A3. Comparable Standards

ASTM – B 214

German – 81-69

TRW Materials Technology

Procedure 8851-20, Rev. 0: CIP Compacting of Powder

1. Inspect bag for tears or adhering metal. Repair tears with RTV rubber.
2. Inspect mandrel; remove any adhering powder.
3. Insert mandrel into bag.
4. Place bag on table and slowly fill and tap, making sure that the powder is evenly distributed around the mandrel, until the powder is one inch from the top of the bag.
5. Fit cap onto bag and adjust until its edge is parallel to the base of the bag, and it is down as far as it will go.
6. Load bag(s) into CIP vessel.
7. Close vessel and pressurize to specified pressure.
8. Hold at pressure one minute.
9. Depressurize by either:
   a) Opening valve completely (fast depressurize), or
   b) Partially opening valve so that it takes 2 minutes to reach 30,000 psi, after which valve may be opened completely.
10. Remove bag and dry with cloth wipers or paper towels.
11. Remove cap.
12. Remove pressed bar (and mandrel) from bag.

TRW Materials Technology

Procedure 4203-407-CIP, Rev. 0: CIP of Gotcha Lock
(To be used in conjunction with Procedure 8851-20, "CIP Compaction of Powder.")

A. Material: CP Ti, AD greater than 1.353 g/cm$^3$ (>30% of theoretical).

B. Equipment:

1. Urethane rubber bag, 407B, hardness Durometer A-70, 3½ inch ID, closed one end.
2. Urethane rubber cap, Durometer A-70 to fit (1).
3. Steel mandrel, 407 M-1, HRC 45, min.
4. CIP apparatus.

C. Procedure:

1. Place clean mandrel, large end down, in recess in base of bag.
2. Fill and seal.
3. Pressurize to 55,000-60,000 lb/in$^2$.
4. Hold at pressure 1 minute.
5. Depressurize by opening valve completely.
6. Remove bag, dry completely, and remove cap.

TRW Materials Technology

Procedure 8851-30: Sintering Green Compacts

A. Equipment:

1. Brew Model 426 vacuum furnace or equivalent.
2. L-H Penningvac cold cathode vacuum gauge or equivalent.
3. Strip-chart recorder.

B. Procedure:

1. Place a moly foil on furnace hearth.
2. Place compacts on foil surface, with space between compacts, and no compact closer than 1/4 inch from the furnace heating element.
3. Close furnace and pump down.
4. Blank chamber off from pump and check leak rate. Do not start run if leak rate exceeds 10 microns/minute.
5. Connect thermocouple and vacuum gauge to recorder, if used. Identify channels, FSD values, and chart speed on Recorder Data Sheet.

TRW Materials Technology

Procedure 4203-30: Sintering Titanium Powder Compacts

(To be used in conjunction with 8851-30, "Sintering Green Compacts.")

1. Heat sample to 1000°F at rate such that chamber pressure does not exceed $10^{-4}$ torr. Hold ≥15 minutes at this temperature.

2. Heat to 1600°F with same pressure control as in (1). Hold at least 15 minutes at this temperature.

3. Heat to 2250 ± 25°F with same pressure control as in (1). Hold at temperature for 4 hours.

TRW Materials Technology

Procedure 8851-40: HIP of Sintered Compacts

A. Equipment:

Instrumented HIP facility.

B. Procedure:

1. Place sample on metal hearthplate.
2. Close unit.
3. Evacuate unit.
4. Pressurize.
5. Heat.
6. Adjust pressure as required.
7. Start timed cycle when required pressure and temperature are achieved.
8. Turn off furnace at end of cycle.
9. Depressurize.

TRW Materials Technology

Procedure 4203-40, Rev. 0, "HIP of Gotcha Lock"

(To be used in conjunction with Procedure 8851-40, "HIP of Sintered Compacts.")

1. Use a titanium or molybdenum foil or sheet as hearthplate.

2. Place a tubular titanium or molybdenum foil or sheet barrier around gotcha lock to insure that compact cannot touch heating element.

3. HIP pressure is 15,000 ±1500 $lb/in^2$.
   HIP temperature is 1700 ±25°F (930 ±10°C).
   Time at 15,000 $lb/in^2$, 1700°F is 2-4 hours.

TRW Materials Technology

Procedure 4203-407 HS, Rev. 0: Hot Sizing of Gotcha Lock

A. Equipment:

1. Hot sizing mandrel, 321 SS, oxidized burr-free.
2. Furnace.
3. Arbor press.

B. Procedure:

1. Place gotcha lock(s) in 1200°F furnace; hold in furnace for 30 minutes.
2. Remove gotcha lock from furnace and press onto the (cold) sizing mandrel.
3. Place gotcha lock/mandrel pair in 1200°F furnace; hold in furnace for two hours.
4. Remove gotcha lock/mandrel from furnace and allow to cool to room temperature.
5. Press mandrel out of gotcha lock.
6. Remove any burrs from surface of mandrel.
7. Spray mandrel with graphite lubricant.

# APPENDIX C

## PROCESS DESCRIPTION: BASE

TRW Materials Technology
Process Description
Ti P/M Gyro Machining Preform: Base; P/N 406 MP
Rev. 1

Material: Ti-6Al-4V Process: CIP/Sinter/HIP

| Operation Number | Operation | Equipment or Facility | Qualified Operator | Process Sheets Spec., Drwg. |
|---|---|---|---|---|
| 406-00 | Mill Al-V master alloy to -100 mesh | Ball mill,10 mesh SS Screen<br>" " jar, gallon<br>Steel balls, inch | LJ | 8851-01 |
| 406-05 | Determine PSD, AD | Tyler Ro-Tap<br>80-325 mesh SS screens<br>Balance Hall apparatus | | MPIF 04<br>MPIF 05 |
| 406-07 | Mill Titanium | Ball mill, jar, balls as in 406-00 | LJ | 8851-05 |
| 406-10 | Determine PSD, AD for Ti powder | As above, for 406-05 | LJ | MPIF 04, 05 |
| 406-12 | Blend Al-V into Ti | Ball mill, jar, balls balance, as in 406-00 | LJ | 4203-12<br>8851-05 |
| 406-15 | Analyze powders: Al, V,Fe,Na,Cl,C,O,H,N | Chemistry Laboratory | | |
| 406-20 | Press Green compact | Rubber bag set, mandrel CIP unit | LJ | 8851-21<br>4203-406-CIP |
| 406-22 | Turn outside diameters concentric with mandrel | Mandrels, lathe | | Dwg. 406-CIP |
| 406-25 | Inspect compact | Bench, 0-1, 1-2" micrometers; 6" vernier | LJ | Dwg. 406-CIP |
| 406-30 | Sinter compact | Brew or NRC vacuum furnace; recorder | LJ or JS | 8851-30<br>4203-30 |
| 406-35 | Inspect | Bench, 0-1, micrometer, 6" vernier caliper, balance | LJ | Dwg. 406-S |
| 406-40 | HIP | HIP unit | JA | 4203-40<br>8851-40 |
| 406-45 | Inspect | Bench<br>0-1" micrometer; 6" vernier caliper | LJ | Dwg. 406-MP |

TRW Materials Technology

Procedure 8851-01: Powder Milling

1. Use new steel jar, one that has been last used for titanium or one that has been bored out to remove any metal contamination. If new or machine, "condition" with scrap titanium powder for 1 hour.
2. Screen Al-V to -10 mesh.
3. Fill jar 1/4 full with powder (≈1200 g for 6 inch jar).
4. Add 3/4-7/8 inch Ø steel balls either new or previously used for this material so that powder plus balls half fill the jar.
5. Seal jar.
6. Place jar on mill and run for 2 hours.
7. Remove jar from mill.
8. Tap lid to settle powder.
9. Open jar.
10. Use 10 mesh screen to separate balls from powder.

MPIF STANDARD NO. 04

METAL POWDER INDUSTRIES FEDERATION
Method for

# DETERMINATION OF APPARENT DENSITY OF FREE-FLOWING METAL POWDERS USING THE HALL APPARATUS

MPIF Standard 04
Issued 1945, Adopted 1948, Revised 1972



## 1. SCOPE

1.1 This standard describes a method for determining the apparent density of free-flowing metal powders and is only suitable for those powders which will flow unaided through the specified Hall flowmeter funnel. See MPIF Standard No. 28 for apparent density of non-free-flowing metal powders.

## 2. APPARATUS

2.1 The following apparatus is required to perform this determination.

2.1.1 Hall Flowmeter Funnel: A standard flowmeter funnel (Fig. 1) having an orifice of 0.10 in. in diameter (2.54 mm).

2.1.2 Density Cup: A cylindrical cup (Fig. 2) having a capacity of 25 ± 0.05 cc.

2.1.3 A support (Fig. 3) to hold the flowmeter funnel concentric with the density cup so that the bottom of the orifice is 1.0" (approx. 25 mm) above the top of the density cup when assembled, (Fig. 4).

2.1.4 Base: A vibration-free table to support the flowmeter assembly.

2.1.5 Balance: A balance having a capacity of at least 200 g and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen shall consist of a volume of approximately 30 to 40 cc of dry metal powder.

Note 1 – The powder is dry when there is no weight loss as the result of conditioning it for one hour in a drying oven at 215 to 225 F (102 to 107 C) and cooling to room temperature in a dessicator.

3.1.1 The test specimen shall be a representative sample obtained in accordance with MPIF Standard No. 01.

## 4. PROCEDURE

4.1 The dry test specimen shall be carefully loaded into the flowmeter funnel and permitted to run into the density cup through the discharge orifice. Care must be taken not to move the density cup.

4.2 When the powder completely fills and overflows the periphery of the density cup, the funnel shall be rotated approximately 90° in a horizontal plane so that the remaining powder falls away from the cup.

4.3 Using a non-magnetic spatula with the blade held perpendicular to the top of the cup, the powder shall be leveled off flush with the top of the density cup. Care must be taken to avoid jarring the apparatus at any time.

4.4 After the leveling operation, the density cup should be lightly tapped on the side to settle the powder to avoid spilling in transfer.

4.5 The powder shall be transferred to the balance and weighed to the nearest 0.05 g.

## 5. REPORT

5.1 The weight in grams of the powder from the leveled density cup, multiplied by 0.04, shall be reported as the apparent density to the nearest 0.1 g/cm³.

## APPENDIXES

### A1. REPORT ON PRECISION OF MPIF STANDARD NO. 04

A1.1 A planned testing program was carried out among users of the Hall flowmeter apparatus to obtain data to determine the precision of this method. The complete report on this work is available in the offices of the Metal Powder Industries Federation.

A1.2 The report concludes that MPIF Standard No. 04 is operable, in the testing of free-flowing metal powder, with a precision of V = 2.1%.

### A2. COMPARABLE STANDARDS

ASTM B 212
Japanese JIS Z 2504–1966
German 83–69

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Metric system conversion factors for all dimensions referred to in this Standard are available in the ASTM Metric Practice Guide, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

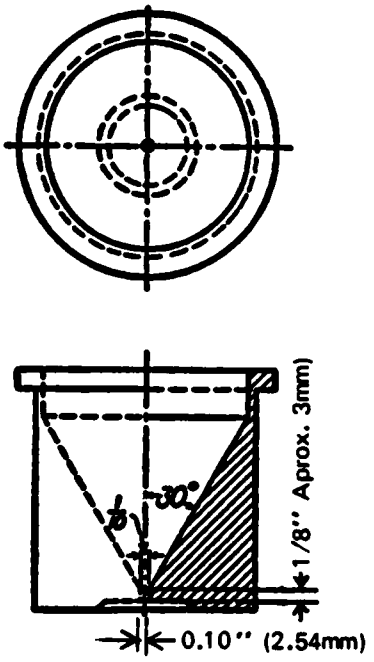


Fig. 1 Hall Flowmeter Funnel

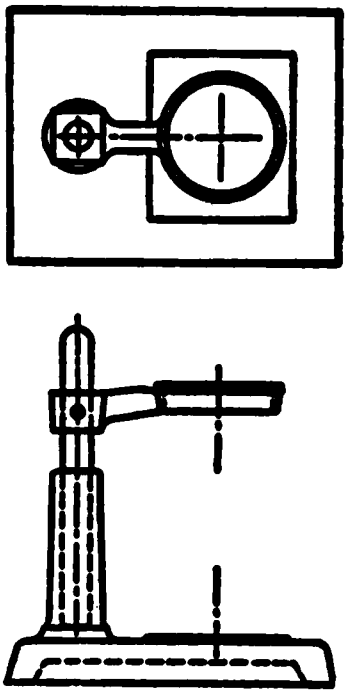

Fig. 3 Stand

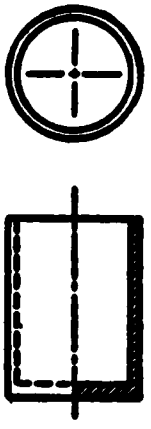

Fig. 2 25cc Density Cup

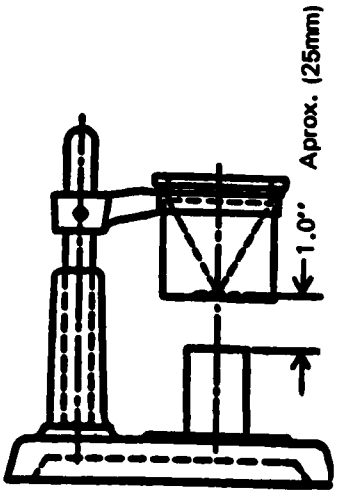


Fig. 4 Assembly

MPIF STANDARD NO. 05

## METAL POWDER INDUSTRIES FEDERATION

Method for

# DETERMINATION OF SIEVE ANALYSIS OF METAL POWDERS

MPIF Standard 05

Issued 1945, Revised 1949, Adopted 1949, Revised 1962, 1973



### 1. SCOPE

1.1 This method covers the test for the sieve analysis of standard compacting grade granular metal powders.

### 2. APPARATUS

2.1 Sieves: A set of standard sieves selected from Table No. 1, ASTM Specification E11, or the equivalent Tyler Standard Screen Scale Sieves. The sieves shall be 8 in. (200 mm) in diameter and either 1 or 2 in.(25 or 50 mm) in depth and fitted with brass, bronze, stainless steel or other suitable wire. The sieves shall conform to ASTM Specification E11. The following sieves shall be used for the sieve analysis of metal powders 80 mesh or finer:

Table I – Testing sieves according to Tyler Standard Screen Scale - U.S. Standard or ASTM Series.

| ASTM Sieves, Size and U.S. Standard Sieve Designation | Tyler Sieves, Size and Tyler Sieve Series Designation |
|---|---|
| 177 micron (No. 80) | 175 micron ( 80 mesh) |
| 149 micron (No. 100) | 149 micron (100 mesh) |
| 105 micron (No. 140) | 104 micron (150 mesh) |
| 74 micron (No. 200) | 74 micron (200 mesh) |
| 44 micron (No. 325) | 44 micron (325 mesh) |

2.2 Sieve Shaker: A mechanically operated single eccentric sieve shaker which imparts to the nest of sieves a rotary motion of 285 rpm plus or minus 15, and a tapping action of 150 taps per minute plus or minus 10. The sieve shaker shall be fitted with a plug to receive the impact of the tapping device. The entire apparatus shall be mounted rigidly, and preferably shall be provided with a time switch to insure accuracy of duration of the test.

2.3 A balance having a capacity of at least 100 g. and a sensitivity of 0.01 g.

### 3. TEST SPECIMEN

3.1 The test specimen, obtained in accordance with MPIF Standard 01, shall be 100 g. of any powder the apparent density of which, determined by MPIF Standard 04, is greater than 1.50 g/cm³.
If the apparent density of the powder is less than 1.50 g/cm³, a 50 g. sample shall be used.

### 4. PROCEDURE

4.1 The group of sieves selected shall be assembled in consecutive order as to size of openings, with the sieve having the largest opening at the top, the assembly being completed by a solid collecting pan below the bottom sieve. The test specimen shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly then shall be fastened securely in a suitable mechanical sieve shaking device and operated for a period of 15 minutes.

4.2 The sieved fractions shall be removed from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them upon a glazed paper. Any material adhering to the bottom of the sieve and frame shall be brushed with a soft brush into the next finer sieve. The sieve just removed then shall be tapped, upside down, on the paper containing the portion that had been retained on it. This fraction shall be weighed to within 0.1 g. This process shall be repeated for each sieve in the nest and the fraction collected in the pan shall also be removed and weighed. The sum of the weights of all the fractions shall be not less than 99 percent of the test specimen weight, and the difference between this sum and 100 (or 50 in case of powders the apparent density of which is less than 1.50 g/cm³) shall be added to the

Metric system conversion factors for all dimensions referred to in this Standard are available in the ASTM Metric Practice Guide, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

weight of the fraction collected in the pan.

5. REPORT

5.1 The weights of the fractions retained on each sieve, and the weight of that fraction collected in the pan, shall be expressed as percentages of the test specimen weight to the nearest 0.1 percent. and reported in the following form. Any screen fraction whose percentage of the test specimen weight is less than 0.1 percent shall be reported as "trace."

Table II – Form for Reporting Test Data

| Mesh (Tyler or U.S.S.) | Percentage by Weight |
|---|---|
| + 80 | ..... |
| – 80 +100 | ..... |
| –100 +150 | ..... |
| –150 +200 | ..... |
| –200 +325 | ..... |
| –325 | ..... |

APPENDIX

A1. Method for Obtaining Sieve Correction

Table III –

| Mesh | Certified Sieve | Work Sieve | Factor by Which to Multiply Work Sieve to Convert to Standard |
|---|---|---|---|
| + 80 | 0.1 | 0.1 | 0.1/0.1 = 1. |
| – 80 +100 | 3.0 | 3.0 | 3.0/3.0 = 1. |
| –100 +150 | 10.0 | 10.0 | 10.0/10.0 = 1. |
| –150 +200 | 20.0 | 20.0 | 20.0/20.0 = 1. |
| –200 +325 | 5.0 | 4.0 | 5.0/4.0 = 1.25 |
| –325 | 21.9 | 27.9 | 21.9/27.9 = 0.785 |

A2. Certified Sieve

Sieves conforming to ASTM Specification E11 can be obtained from the sieve manufacturers, and arrangements can be made through them to have the sieves certified by the Bureau of Standards. If used continually, the sieves will, after a period of time, become less accurate and might no longer be acceptable as certified sieves. The common practice, which would be considered acceptable according to this standard, would be to use the certified sieves as a master set for checking other working sets of sieves. By comparing sieve tests on the same sample, run in both the master set and working set, a factor can be established for correcting results on the working sieves. These factors will vary with the coarseness of the powder and should be established for powders of different particle size distribution. The method for obtaining this factor is illustrated by the example given in Table III. All results obtained on the working sieves them would be multiplied by the factor so obtained before reporting.

A3. Comparable Standards

ASTM – B 214

German – 81-69

TRW Materials Technology

Procedure 8851-05: Mill powder to increase its apparent density and flow rate.

A. Equipment:

1. Ball mill.
2. Ball mill jar.
3. Steel balls, 3/4-7/8 inch diameter.
4. Hall apparatus.
5. Top-loading electronic balance.

B. Procedure:

1. Mill powder, as in Procedure 8851-01 for 1/2, 1, or 2 hours, as required, to increase its apparent density to 1.35-1.58 $g/cm^3$ (30-35% of theoretical).

TRW Materials Technology

Procedure 4203-12: Powder Blending

(To be used in conjunction with Procedure 8851-01 "Powder Milling")

1. Weigh out Ti and Al-V in ratio of 90:10 using electronic top-loading balance, Ex 1200 g charge = 120 g Al-V
   1080 g Ti

2. Fill jar with powder and balls as per 8851-01 and blend for 1/2 hour.

TRW Materials Technology

Procedure 8851-21, Rev. 0: CIP Compacting of Powder

1. Inspect bag for tears or adhering metal. Repair tears with RTV rubber.
2. Inspect mandrel; remove any adhering powder.
3. Insert mandrel into bag.
4. Place bag on vibrator table and slowly fill, making sure that the powder is evenly distributed around the mandrel, until the powder is one inch from the top of the bag.
5. Fit cap onto bag and adjust until its edge is parallel to the base of the bag, and it is down as far as it will go.
6. Load bag(s) into CIP vessel.
7. Close vessel and pressurize to specified pressure.
8. Hold at pressure one minute.
9. Depressurize by either:
   a) Opening valve completely (fast depressurize), or
   b) Partially opening valve so that it takes 2 minutes to reach 30,000 psi, after which valve may be opened completely.
10. Remove bag and dry with cloth wipers or paper towels.
11. Remove cap.
12. Remove pressed bar (and mandrel) from bag.

TRW Materials Technology

Procedure 4203-406-CIP, Rev. 0: CIP of Gyro Base
(To be used in conjunction with Procedure 8851-21,
CIP Compacting of Powder.)

Material: Blended Ti-(6Al-4V), AD $\leq$ 1.16 g/cm$^3$; TD $\geq$ 1.60 g/cm$^3$

1. Check that mandrel halves are securely bolted together.
2. Load mandrel, bowl-end down, into bag. Fill with 950 g Ti-(6Al-4V) powder.
3. Pressurize to 55,000-60,000 lb/in$^2$.
4. Hold at pressure 1 minute.
5. Depressurize by opening valve completely.
6. Dry bag with cloth or paper towels and remove cap.
7. Remove 1/4 inch threaded cap screw from bowl end of mandrel.
8. Screw 3/8 inch bolt into top of bowl mandrel and extract bowl mandrel from compact.
9. Support base of compact on 4" OD x 2 inch ID ejection block and push the lower mandrel body out of the compact.

TRW Materials Technology

Procedure 8851-30: Sintering Green Compacts

A. Equipment:

1. Brew Model 426 vacuum furnace or equivalent.

2. L-H Penningvac cold cathode vacuum gauge with electrical readout.

3. Strip-chart recorder.

B. Procedure:

1. Place an outgassed BN-coated moly foil on furnace hearth, BN facing upward.

2. Place compacts on BN surface, with space between compacts, and no compact closer than 1/4 inch from the furnace heating element.

3. Close furnace and pump down.

4. Blank chamber off from pump and check leak rate. Do not start run if leak rate exceeds 10 microns/minute.

5. Connect thermocouple and vacuum gauge to recorder. Identify channels, FSD values, and chart speed on Recorder Data Sheet.

TRW Materials Technology

Procedure 4203-30: Sintering Titanium Powder Compacts

(To be used in conjunction with 8851-30, "Sintering Green Compacts.")

1. Heat sample to 1000°F at rate such that chamber pressue does not exceed $10^{-4}$ torr. Hold ≥15 minutes at this temperature.

2. Heat to 1600°F with same pressure control as in (1). Hold at least 15 minutes at this temperature.

3. Heat to 2250 ±25°F with same pressure control as in (1). Hold at temperature for 4 hours.

TRW Materials Technology

Procedure 8851-40: HIP of Sintered Compacts

A. Equipment:

Instrumented HIP facility.

B. Procedure:

1. Place an outgassed, BN-coated moly foil on HIP hearth with BN facing upward.
2. Place compact on BN surface.
3. Place an outgassed ceramic barrier around compact to insure that compact cannot touch furnace heating elements.
4. Close unit.
5. Evacuate unit.
6. Pressurize.
7. Heat.
8. Adjust pressure as required.
9. Start timed cycle when required pressure and temperature are achieved.
10. Turn off furnace at end of cycle.
11. Depressurize.

TRW Materials Technology

Procedure 4203-40: HIP of Base and Gotcha Lock

A. Equipment:

Instrumented HIP facility.

B. Procedure:

1. Load and seal unit as per 8851-40.
2. Heat to 1700°F ±20°F.
3. Pressurize to 15,000 lb/in$^2$ ± 500 lb/in$^2$.
4. Hold at these conditions for 3 hours ± 1 hour.

# APPENDIX D

## PROCESS DESCRIPTION: GIMBAL RING

TRW Materials Technology
Process Description
Ti P/M Gyro Machining Preform: Gimbal Ring: P/N 409 MP

Material: Ti-6Al-4V Process: Die Press/Sinter/Forge

| Operation Number | Operation | Equipment or Facility | Qualified Operator | Process Sheets Spec., Drwg. |
|---|---|---|---|---|
| 409-00 | Mill Al-V master alloy to -100 mesh | Ball mill, 10 mesh SS Screen<br>" " jar, gallon<br>Steel balls | LJ | 8851-01 |
| 409-05 | Determine PSD, AD | Tyler Ro-Tap<br>80-325 mesh SS Screens<br>Balance, Hall apparatus | LJ | MPIF 04<br>MPIF 05 |
| 409-07 | Mill Titanium | Ball mill, jar, balls, as in 406-00 | LJ | 8851-05 |
| 409-10 | Determine PSD, AD for Ti powder | As above, for 406-05 | LJ | MPIF 04, 05 |
| 409-12 | Blend Al-V into Ti | Ball mill, jar, balls balance, as in 406-00 | LJ | 4203-12<br>8851-05 |
| 409-15 | Analyze powder: Al, V, Fe, Na, Cl, C, O, H, N | Chem. Lab | | |
| 409-20 | Press green compact | Briquetting die 409-BD-1 | LJ | 8851-20<br>4203-410-20 |
| 409-25 | Inspect | 0-1" micrometer, balance bench | LJ | Dwg. 409B |
| 409-30 | Sinter | Brew or NRC Vacuum Furnace | LJ or JS | 8851-30<br>4203-30 |
| 409-35 | Inspect | Bench, 0-1" micrometer, Balance | | Dwg. 410-S |
| 409-40 | Lubricate | Spray booth<br>Spray gun<br>Lubricant | LF | 8851-140<br>4203-140 |
| 409-50 | Forge | Die Set 8851-265<br>Die Inserts 8851-265-4<br>Forge press, 50T min.<br>DTI, Type K TC | LJ | 8851-50<br>4203-150 |
| 409-55 | Inspect | Bench, 0-1" micrometer | LJ | Dwg. 409F |

TRW Materials Technology
Process Description
Ti P/M Gyro Machining Preform: Gimbal Ring: P/N 409MP (continued)

Material: Ti-6Al-4V Process: Die Press/Sinter/Forge

| Operation Number | Operation | Equipment or Facility | Qualified Operator | Process Sheets Spec., Drwg. |
|---|---|---|---|---|
| 409-60 | Clean | Sand Blast or Kolene Tank<br>Acid, Rinse Tanks | LJ | 8851-60<br>TAP-PS-645A<br>TAP-PS-518B |
| 409-65 | Inspect | Bench<br>0-1" micrometer | LJ | Dwg. 409-MP |

TRW Materials Technology

Procedure 8851-01: Powder Milling

1. Use new steel jar, one that has been last used for titanium or one that has been bored out to remove any metal contamination. If new or machined, "condition" with scrap titanium powder for 1 hour.
2. Screen Al-V to -10 mesh.
3. Fill jar 1/4 full with powder (≈1200 g for 6 inch jar).
4. Add 3/4-7/8 inch Ø steel balls either new or previously used for this material so that powder plus balls half fill the jar.
5. Seal jar.
6. Place jar on mill and run for 2 hours.
7. Remove jar from mill.
8. Tap lid to settle powder.
9. Open jar.
10. Use 10 mesh screen to separate balls from powder.

MPIF STANDARD NO. 04

METAL POWDER INDUSTRIES FEDERATION
Method for

# DETERMINATION OF APPARENT DENSITY OF FREE-FLOWING METAL POWDERS USING THE HALL APPARATUS

MPIF Standard 04
Issued 1945, Adopted 1948, Revised 1972



## 1. SCOPE

1.1 This standard describes a method for determining the apparent density of free-flowing metal powders and is only suitable for those powders which will flow unaided through the specified Hall flowmeter funnel. See MPIF Standard No. 28 for apparent density of non-free-flowing metal powders.

## 2. APPARATUS

2.1 The following apparatus is required to perform this determination.

2.1.1 Hall Flowmeter Funnel: A standard flowmeter funnel (Fig. 1) having an orifice of 0.10 in. in diameter (2.54 mm).

2.1.2 Density Cup: A cylindrical cup (Fig. 2) having a capacity of 25 ± 0.05 cc.

2.1.3 A support (Fig. 3) to hold the flowmeter funnel concentric with the density cup so that the bottom of the orifice is 1.0" (approx. 25 mm) above the top of the density cup when assembled, (Fig. 4).

2.1.4 Base: A vibration-free table to support the flowmeter assembly.

2.1.5 Balance: A balance having a capacity of at least 200 g and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen shall consist of a volume of approximately 30 to 40 cc of dry metal powder.

Note 1 — The powder is dry when there is no weight loss as the result of conditioning it for one hour in a drying oven at 215 to 225 F (102 to 107 C) and cooling to room temperature in a dessicator.

3.1.1 The test specimen shall be a representative sample obtained in accordance with MPIF Standard No. 01.

## 4. PROCEDURE

4.1 The dry test specimen shall be carefully loaded into the flowmeter funnel and permitted to run into the density cup through the discharge orifice. Care must be taken not to move the density cup.

4.2 When the powder completely fills and overflows the periphery of the density cup, the funnel shall be rotated approximately 90° in a horizontal plane so that the remaining powder falls away from the cup.

4.3 Using a non-magnetic spatula with the blade held perpendicular to the top of the cup, the powder shall be leveled off flush with the top of the density cup. Care must be taken to avoid jarring the apparatus at any time.

4.4 After the leveling operation, the density cup should be lightly tapped on the side to settle the powder to avoid spilling in transfer.

4.5 The powder shall be transferred to the balance and weighed to the nearest 0.05 g.

## 5. REPORT

5.1 The weight in grams of the powder from the leveled density cup, multiplied by 0.04, shall be reported as the apparent density to the nearest 0.1 g/cm³.

## APPENDIXES

### A1. REPORT ON PRECISION OF MPIF STANDARD NO. 04

A1.1 A planned testing program was carried out among users of the Hall flowmeter apparatus to obtain data to determine the precision of this method. The complete report on this work is available in the offices of the Metal Powder Industries Federation.

A1.2 The report concludes that MPIF Standard No. 04 is operable, in the testing of free-flowing metal powder, with a precision of V = 2.1%.

### A2. COMPARABLE STANDARDS

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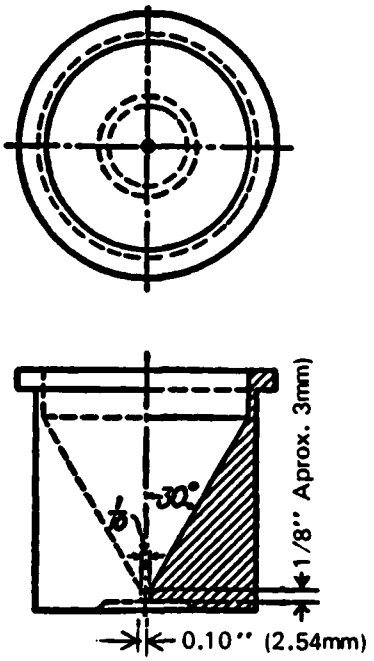


Fig. 1 Hall Flowmeter Funnel

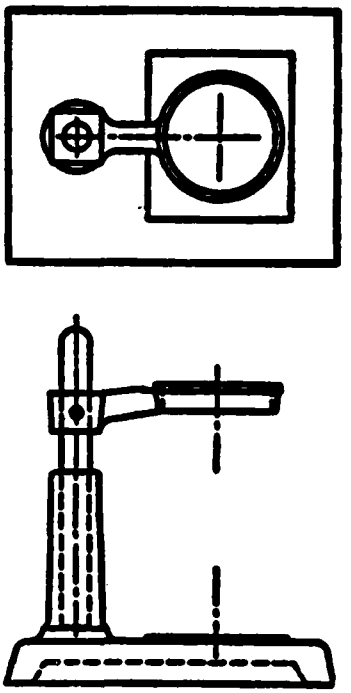

Fig. 3 Stand

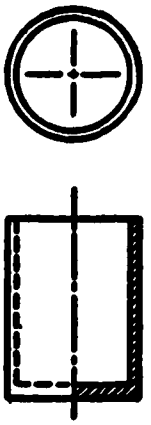

Fig. 2 25cc Density Cup

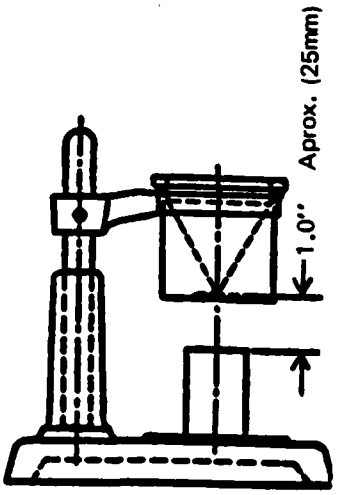


Fig. 4 Assembly

MPIF STANDARD NO. 05

METAL POWDER INDUSTRIES FEDERATION

Method for

# DETERMINATION OF SIEVE ANALYSIS OF METAL POWDERS

MPIF Standard 05

Issued 1945, Revised 1949, Adopted 1949, Revised 1962, 1973



## 1. SCOPE

1.1 This method covers the test for the sieve analysis of standard compacting grade granular metal powders.

## 2. APPARATUS

2.1 Sieves: A set of standard sieves selected from Table No. 1, ASTM Specification E11, or the equivalent Tyler Standard Screen Scale Sieves. The sieves shall be 8 in. (200 mm) in diameter and either 1 or 2 in.(25 or 50 mm) in depth and fitted with brass, bronze, stainless steel or other suitable wire. The sieves shall conform to ASTM Specification E11. The following sieves shall be used for the sieve analysis of metal powders 80 mesh or finer:

Table I – Testing sieves according to Tyler Standard Screen Scale - U.S. Standard or ASTM Series.

| ASTM Sieves, Size and U.S. Standard Sieve Designation | Tyler Sieves, Size and Tyler Sieve Series Designation |
|---|---|
| 177 micron (No. 80) | 175 micron ( 80 mesh) |
| 149 micron (No. 100) | 149 micron (100 mesh) |
| 105 micron (No. 140) | 104 micron (150 mesh) |
| 74 micron (No. 200) | 74 micron (200 mesh) |
| 44 micron (No. 325) | 44 micron (325 mesh) |

2.2 Sieve Shaker: A mechanically operated single eccentric sieve shaker which imparts to the nest of sieves a rotary motion of 285 rpm plus or minus 15, and a tapping action of 150 taps per minute plus or minus 10. The sieve shaker shall be fitted with a plug to receive the impact of the tapping device. The entire apparatus shall be mounted rigidly, and preferably shall be provided with a time switch to insure accuracy of duration of the test.

2.3 A balance having a capacity of at least 100 g. and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen, obtained in accordance with MPIF Standard 01, shall be 100 g. of any powder the apparent density of which, determined by MPIF Standard 04, is greater than 1.50 g/cm³.
If the apparent density of the powder is less than 1.50 g/cm³, a 50 g. sample shall be used.

## 4. PROCEDURE

4.1 The group of sieves selected shall be assembled in consecutive order as to size of openings, with the sieve having the largest opening at the top, the assembly being completed by a solid collecting pan below the bottom sieve. The test specimen shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly then shall be fastened securely in a suitable mechanical sieve shaking device and operated for a period of 15 minutes.

4.2 The sieved fractions shall be removed from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them upon a glazed paper. Any material adhering to the bottom of the sieve and frame shall be brushed with a soft brush into the next finer sieve. The sieve just removed then shall be tapped, upside down, on the paper containing the portion that had been retained on it. This fraction shall be weighed to within 0.1 g. This process shall be repeated for each sieve in the nest and the fraction collected in the pan shall also be removed and weighed. The sum of the weights of all the fractions shall be not less than 99 percent of the test specimen weight, and the difference between this sum and 100 (or 50 in case of powders the apparent density of which is less than 1.50 g/cm³) shall be added to the

Metric system conversion factors for all dimensions referred to in this Standard are available in the ASTM Metric Practice Guide, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

weight of the fraction collected in the pan.

**5. REPORT**

5.1 The weights of the fractions retained on each sieve, and the weight of that fraction collected in the pan, shall be expressed as percentages of the test specimen weight to the nearest 0.1 percent, and reported in the following form. Any screen fraction whose percentage of the test specimen weight is less than 0.1 percent shall be reported as "trace."

Table II – Form for Reporting Test Data

| Mesh (Tyler or U.S.S.) | Percentage by Weight |
|---|---|
| + 80 | ..... |
| – 80.+100 | ..... |
| –100 +150 | ..... |
| –150 +200 | ..... |
| –200 +325 | ..... |
| –325 | ..... |

APPENDIX

A1. Method for Obtaining Sieve Correction

Table III –

| Mesh | Certified Sieve | Work Sieve | Factor by Which to Multiply Work Sieve to Convert to Standard |
|---|---|---|---|
| + 80 | 0.1 | 0.1 | 0.1/0.1 = 1. |
| – 80 +100 | 3.0 | 3.0 | 3.0/3.0 = 1. |
| –100 +150 | 10.0 | 10.0 | 10.0/10.0 = 1. |
| –150 +200 | 20.0 | 20.0 | 20.0/20.0 = 1. |
| –200 +325 | 5.0 | 4.0 | 5.0/4.0 = 1.25 |
| –325 | 21.9 | 27.9 | 21.9/27.9 = 0.785 |

A2. Certified Sieve

Sieves conforming to ASTM Specification E11 can be obtained from the sieve manufacturers, and arrangements can be made through them to have the sieves certified by the Bureau of Standards. If used continually, the sieves will, after a period of time, become less accurate and might no longer be acceptable as certified sieves. The common practice, which would be considered acceptable according to this standard, would be to use the certified sieves as a master set for checking other working sets of sieves. By comparing sieve tests on the same sample, run in both the master set and working set, a factor can be established for correcting results on the working sieves. These factors will vary with the coarseness of the powder and should be established for powders of different particle size distribution. The method for obtaining this factor is illustrated by the example given in Table III. All results obtained on the working sieves them would be multiplied by the factor so obtained before reporting.

A3. Comparable Standards

ASTM – B 214

German – 81-69

TRW Materials Technology

Procedure 8851-05: Mill powder to increase its apparent density and flow rate.

A. Equipment:

1. Ball mill.
2. Ball mill jar.
3. Steel balls, 3/4-7/8 inch diameter.
4. Hall apparatus.
5. Top-loading electronic balance.

B. Procedure:

1. Mill powder, as in Procedure 8851-01 for 1/2, 1, or 2 hours, as required, to increase its apparent density to 1.35-1.58 $g/cm^3$ (30-35% of theoretical).

TRW Materials Technology

Procedure 8851-140: Application of Lubricant to Forging Bar or Preform

A. Equipment:

1. Spray gun.
2. Spray booth.

B. Procedure:

1. Pour designated lubricant into spray gun.
2. Add thinner, if required.
3. Spray sample, one pass only, applying a very light coat of lubricant.
4. Allow sample to air dry.

TRW Materials Technology

Procedure 4203-12: Powder Blending
(To be used in conjunction with Procedure 8851-01 "Powder Milling")

1. Weigh out Ti and Al-V in ratio of 90:10 using electronic top-loading balance, Ex 1200 g charge = 120 g Al-V
   1080 g Ti

2. Fill jar with powder and balls as per 8851-01 and blend for 1/2 hour.

TRW Materials Technology

Procedure 4203-410-20, Rev. 0: Briquetting of Gimbal Ring

(To be used in conjunction with Procedure 8851-120, "Die Pressing (Briquetting) of Metal Compacts.")

1. Briquetting pressure for this part is 30 ton/$in^2$. The die area is 0.6 $in^2$; briquetting force = 18 ± 1 ton.

TRW Materials Technology

Procedure 8851-30: Sintering Green Compacts

A. Equipment:

1. Brew Model 426 vacuum furnace or equivalent.
2. L-H Penningvac cold cathode vacuum gauge with electrical readout.
3. Strip-chart recorder.

B. Procedure:

1. Place an outgassed BN-coated moly foil on furnace hearth, BN facing upward.
2. Place compacts on BN surface, with space between compacts, and no compact closer than 1/4 inch from the furnace heating element.
3. Close furnace and pump down.
4. Blank chamber off from pump and check leak rate. Do not start run if leak rate exceeds 10 microns/minute.
5. Connect thermocouple and vacuum gauge to recorder. Identify channels, FSD values, and chart speed on Recorder Data Sheet.

TRW Materials Technology

Procedure 4203-30: Sintering Titanium Powder Compacts

(To be used in conjunction with 8851-30, "Sintering Green Compacts.")

1. Heat sample to 1000°F at rate such that chamber pressure does not exceed $10^{-4}$ torr. Hold ≥15 minutes at this temperature.

2. Heat to 1600°F with same pressure control as in (1). Hold at least 15 minutes at this temperature.

3. Heat to 2250 ± 25°F with same pressure control as in (1). Hold at temperature for 4 hours.

TRW Materials Technology

Procedure 4203-50: Forging of Gimbals

A. Equipment:

1. Forge press of at least 50T capacity.
2. Die nest 8851-265.
3. Die inserts:
   a) Gimbal Ring: 8851-265-4
   b) Inner Gimbal: 8851-265-5
4. DTI (Digital Temperature Indicator) with Type K thermocouple.
5. Furnace capable of heating to 1800°F and of being moved in close proximity to forge die.
6. Tongs.
7. Die Lubricant: Grafoil 1226.

B. Procedure:

1. Set furnace to 1800°F with hearth plate in place. Check hearth temperature with thermocouple.
2. Set die heater controller to 350°F.
3. Lubricate die, core rod and punches.
4. When furnace hearth has reached 1800°F ±15°F, place forging preform on hearth. Set timer to ten minutes.
5. Lower upper punch into die. Se die force:
   a) Gimbal Ring: 40T
   b) Inner Gimbal: 30T
6. When preform timer rings:
   a) Raise upper punch so that its lower end is 6-10 inches above die.
   b) Remove preform and transfer to die.
7. Forge. Elapsed time from removal of preform from furnace to contact with upper punch must be less than ten seconds.
8. Eject forging from die.
9. Clean die.

TRW Materials Technology

Procedure 4203-140: Application of Forging Lubricant to P/M Ti-6Al-4V Forge Preforms

1. The lubricant to be used for these parts is D1363.
2. Coat preforms with a thin, uniform layer of lubricant.
3. Sprayed parts are to be dried for two minutes with a heat gun, or they may be air dried.

TRW Materials Technology

Procedure 8851-50: Forging Small Titanium P/M Parts

A. Equipment:

1. Forge press capable of accommodating die nest and with enough force to overcome the material flow stress at the forging temperature.
2. Die nest, four column, with integral ejector.
3. Die inserts.
4. Furnace, tongs.

B. Procedure:

1. Mount die nest in press and adjust so that bushings on punch plate move smoothly over columns.
2. Install die inserts in die nest. Align punch or upper die with die.
3. Connect die heaters and die thermocouples to die heater power supply. Heat die to 700-800°F.
4. Move part-heating furnace within arm's reach of die.
5. Lubricate forging preform, lubricate punch and die.
6. Heat forging preform.
7. Transfer quickly to die.
8. Apply forging force; adjust force, as required, to produce required forging pressure.
9. Raise upper punch.
10. Eject from die.

TRW Materials Technology

Procedure 8851-60: Cleaning of Forging

A. Equipment:

1. Sand blast cabinet or Kolene tank.
2. Acid and rinse tanks.
3. Micrometer.

B. Procedure:

1. Remove lubricant by sand blast or by immersion in Kolene bath.
2. Remove 0.001 inch/surface in 3 v/o HF, 30 v/o $HNO_3$, 67 v/o $H_2O$ at 190-210°F.
3. Rinse.

| | TRW INC.<br>23555 EUCLID AVENUE · CLEVELAND, OHIO 44117<br>PROCESS SPECIFICATION | TAP- PS-5180<br>SHEET 1 OF 2<br>ISSUED 3-29-74<br>REVISED 4-27-770 |
|---|---|---|
| PRE-INSPECTION ETCH<br>ALL TITANIUM ALLOYS - CLEAN GLOVE TREATMENT | | |

1.0 TITLE: Titanium Alloy Pre-Inspection Etch --Chemical.

2.0 SCOPE: This specification provides an etch for conditioning titanium hardware surfaces prior to inspection per P&WA EIM-2.

3.0 MATERIALS:

3.1 Alkaline Cleaner, per P&WA PS-101D.
3.1.1 Chemical Composition
P&WA PMC 1256 Alkali Cleaner (Hubbard Hall #200 or equivalent, 9-14 oz./gal.)

3.2 Etch Solution, per P&WA PS-38D
3.2.1 Chemical Composition

| | |
|---|---|
| Nitric Acid (42° Be) | 30.0 - 40.0% by volume |
| Hydrofluoric Acid (70%) | 2.5 - 3.5% by volume |

3.3 Water Rinse Tanks.
3.4 Fluoride waste treatment.

4.0 OPERATING CONDITIONS:

4.1 Rack parts in plastic coated fixtures, or vertically in stainless steel baskets.
4.2 Immerse in alkaline cleaner for 1.0 to 2.0 minutes at 150° - 200°F.
4.3 Rinse in cold water for 2.0 minutes. Air agitation to be provided. Parts must be cooled to room temperature before etching.
4.4 Immerse in titanium etch solution for 30 ± 15 seconds*, however, keep immersion at a maximum time of 45 seconds. Air agitation to be provided.
4.5 Immerse parts in fluoride treatment tank No. 1.
4.6 Immerse parts in fluoride treatment tank No. 2.
4.7 Rinse thoroughly in clean cold water for 2.0 minutes. Air agitation to be provided.
4.8 Rinse thoroughly in clean hot water for 5.0 minutes. Air agitation to be provided.
4.9 All parts are to be handled with clean gloves after the final hot rinse.
4.10 Blow dry with clean filtered air.
4.11 Place cleaned parts in clean containers prepared so that no foreign matter can contaminate the surface.

*NOTE: This process removes approximately .0003" material per side per minute. Care should be taken to assure that finish part dimensions are maintained.

TAP-PS-5188
SHEET 2 OF 2
ISSUED 3-29-74
REVISED 4-27-77 Ø

5.0 SAFETY: The above listed chemicals are hazardous. Protective equipment including face shield shall be worn when making up solutions. In case of bodily contact with chemicals, immediately flush contacted area with water and seek medical attention.

6.0 PROCESS CONTROL: Chemical sampling and additions shall be performed as specified by the Materials Engineering Department.

R. S. Sikora

R.S. Sikora, Chemical Engineer
Materials & Process Control Department

J. C. McKenzie

J. C. McKenzie
Section Manager - Chemical
Materials Engineering Department

H. E. Collins

H. E. Collins, Manager
Materials Engineering Department





PPC-1000 CC REV. PRINTED IN U.S.A.

# APPENDIX E

## PROCESS DESCRIPTION: INNER GIMBAL

TRW Materials Technology
Process Description
Ti P/M Gyro Machining Preform: Inner Gimbal: P/N 410 MP

Material: Ti-6Al-4V Process: Die Press/Sinter/HIP

| Operation Number | Operation | Equipment or Facility | Qualified Operator | Process Sheets Spec., Drwg. |
|---|---|---|---|---|
| 410-00 | Mill Al-V master alloy to -100 mesh | Ball mill, 10 mesh SS Screen<br>" " jar, gallon<br>Steel balls | LJ | 8851-01 |
| 410-05 | Determine PSD, AD | Tyler Ro-Tap<br>80-325 mesh SS Screens<br>Balance, Hall apparatus | LJ | MPIF 04<br>MPIF 05 |
| 410-07 | Mill Titanium | Ball mill, jar, balls, as in 406-00 | LJ | 8851-05 |
| 410-10 | Determine PSD, AD for Ti powder | As above, for 406-05 | LJ | MPIF 04, 05 |
| 410-12 | Blend Al-V into Ti | Ball mill, jar, balls, balance, as in 406-00 | LJ | 4203-12<br>8851-05 |
| 410-15 | Analyze powder: Al, V, Fe, Na, Cl, C, O, H, N | Chem. Lab | | |
| 410-20 | Press green compact | Briquetting die 410-BD-1<br>Hydraulic press | LJ | 8851-120<br>4203-410-20 |
| 410-25 | Inspect | 0-1" micrometer, balance bench | LJ | Dwg. 410-B |
| 410-30 | Sinter | Brew or NRC Vacuum Furnace | LJ or JS | 8851-30<br>4203-30 |
| 410-35 | Inspect | Bench, 0-1" micrometer, Balance | | Dwg. 410-S |
| 410-40 | HIP | HIP unit | JA | 4203-40<br>8851-40 |

TRW Materials Technology

Procedure 8851-01: Powder Milling

1. Use new steel jar, one that has been last used for titanium or one that has been bored out to remove any metal contamination. If new or machined, "condition" with scrap titanium powder for 1 hour.

2. Screen Al-V to -10 mesh.

3. Fill jar 1/4 full with powder (≈1200 g for 6 inch jar).

4. Add 3/4-7/8 inch Ø steel balls either new or previously used for this material so that powder plus balls half fill the jar.

5. Seal jar.

6. Place jar on mill and run for 2 hours.

7. Remove jar from mill.

8. Tap lid to settle powder.

9. Open jar.

10. Use 10 mesh screen to separate balls from powder.

MPIF STANDARD NO. 04

METAL POWDER INDUSTRIES FEDERATION
Method for

# DETERMINATION OF APPARENT DENSITY OF FREE-FLOWING METAL POWDERS USING THE HALL APPARATUS

MPIF Standard 04
Issued 1945, Adopted 1948, Revised 1972



## 1. SCOPE

1.1 This standard describes a method for determining the apparent density of free-flowing metal powders and is only suitable for those powders which will flow unaided through the specified Hall flowmeter funnel. See MPIF Standard No. 28 for apparent density of non-free-flowing metal powders.

## 2. APPARATUS

2.1 The following apparatus is required to perform this determination.

2.1.1 Hall Flowmeter Funnel: A standard flowmeter funnel (Fig. 1) having an orifice of 0.10 in. in diameter (2.54 mm).

2.1.2 Density Cup: A cylindrical cup (Fig. 2) having a capacity of 25 ± 0.05 cc.

2.1.3 A support (Fig. 3) to hold the flowmeter funnel concentric with the density cup so that the bottom of the orifice is 1.0" (approx. 25 mm) above the top of the density cup when assembled, (Fig. 4).

2.1.4 Base: A vibration-free table to support the flowmeter assembly.

2.1.5 Balance: A balance having a capacity of at least 200 g and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen shall consist of a volume of approximately 30 to 40 cc of dry metal powder.

Note 1 – The powder is dry when there is no weight loss as the result of conditioning it for one hour in a drying oven at 215 to 225 F (102 to 107 C) and cooling to room temperature in a dessicator.

3.1.1 The test specimen shall be a representative sample obtained in accordance with MPIF Standard No. 01.

## 4. PROCEDURE

4.1 The dry test specimen shall be carefully loaded into the flowmeter funnel and permitted to run into the density cup through the discharge orifice. Care must be taken not to move the density cup.

4.2 When the powder completely fills and overflows the periphery of the density cup, the funnel shall be rotated approximately 90° in a horizontal plane so that the remaining powder falls away from the cup.

4.3 Using a non-magnetic spatula with the blade held perpendicular to the top of the cup, the powder shall be leveled off flush with the top of the density cup. Care must be taken to avoid jarring the apparatus at any time.

4.4 After the leveling operation, the density cup should be lightly tapped on the side to settle the powder to avoid spilling in transfer.

4.5 The powder shall be transferred to the balance and weighed to the nearest 0.05 g.

## 5. REPORT

5.1 The weight in grams of the powder from the leveled density cup, multiplied by 0.04, shall be reported as the apparent density to the nearest 0.1 g/cm³.

## APPENDIXES

### A1. REPORT ON PRECISION OF MPIF STANDARD NO. 04

A1.1 A planned testing program was carried out among users of the Hall flowmeter apparatus to obtain data to determine the precision of this method. The complete report on this work is available in the offices of the Metal Powder Industries Federation.

A1.2 The report concludes that MPIF Standard No. 04 is operable, in the testing of free-flowing metal powder, with a precision of V = 2.1%.

### A2. COMPARABLE STANDARDS

ASTM B 212
Japanese JIS Z 2504–1966
German 83–69

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Metric system conversion factors for all dimensions referred to in this Standard are available in the ASTM Metric Practice Guide, published by the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103.

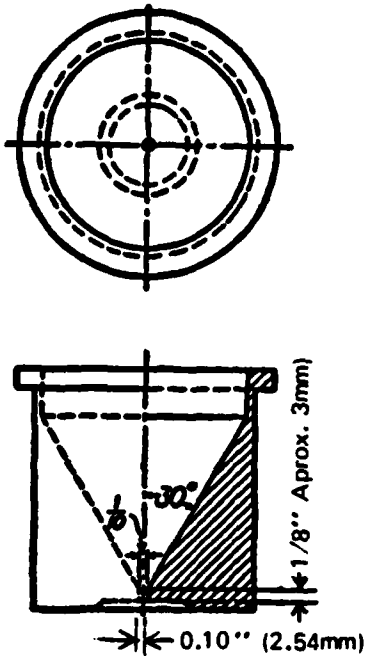


Fig. 1 Hall Flowmeter Funnel

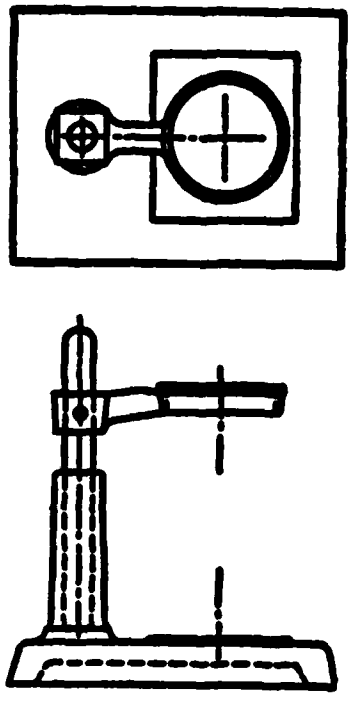

Fig. 3 Stand

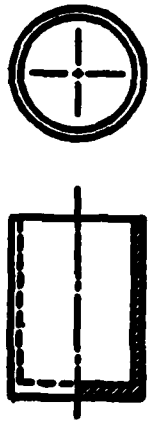

Fig. 2 25cc Density Cup

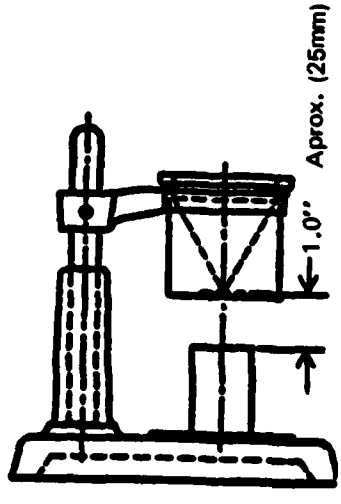


Fig. 4 Assembly

MPIF STANDARD NO. 05

METAL POWDER INDUSTRIES FEDERATION

Method for

# DETERMINATION OF SIEVE ANALYSIS OF METAL POWDERS

MPIF Standard 05

Issued 1945, Revised 1949, Adopted 1949, Revised 1962, 1973



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## 2. APPARATUS

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| ASTM Sieves, Size and U.S. Standard Sieve Designation | Tyler Sieves, Size and Tyler Sieve Series Designation |
|---|---|
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| 149 micron (No. 100) | 149 micron (100 mesh) |
| 105 micron (No. 140) | 104 micron (150 mesh) |
| 74 micron (No. 200) | 74 micron (200 mesh) |
| 44 micron (No. 325) | 44 micron (325 mesh) |

2.2 Sieve Shaker: A mechanically operated single eccentric sieve shaker which imparts to the nest of sieves a rotary motion of 285 rpm plus or minus 15, and a tapping action of 150 taps per minute plus or minus 10. The sieve shaker shall be fitted with a plug to receive the impact of the tapping device. The entire apparatus shall be mounted rigidly, and preferably shall be provided with a time switch to insure accuracy of duration of the test.

2.3 A balance having a capacity of at least 100 g. and a sensitivity of 0.01 g.

## 3. TEST SPECIMEN

3.1 The test specimen, obtained in accordance with MPIF Standard 01, shall be 100 g. of any powder the apparent density of which, determined by MPIF Standard 04, is greater than 1.50 g/cm³.
If the apparent density of the powder is less than 1.50 g/cm³, a 50 g. sample shall be used.

## 4. PROCEDURE

4.1 The group of sieves selected shall be assembled in consecutive order as to size of openings, with the sieve having the largest opening at the top, the assembly being completed by a solid collecting pan below the bottom sieve. The test specimen shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly then shall be fastened securely in a suitable mechanical sieve shaking device and operated for a period of 15 minutes.

4.2 The sieved fractions shall be removed from the nest of sieves by removing the coarsest sieve from the nest, gently tapping its contents to one side and pouring them upon a glazed paper. Any material adhering to the bottom of the sieve and frame shall be brushed with a soft brush into the next finer sieve. The sieve just removed then shall be tapped, upside down, on the paper containing the portion that had been retained on it. This fraction shall be weighed to within 0.1 g. This process shall be repeated for each sieve in the nest and the fraction collected in the pan shall also be removed and weighed. The sum of the weights of all the fractions shall be not less than 99 percent of the test specimen weight, and the difference between this sum and 100 (or 50 in case of powders the apparent density of which is less than 1.50 g/cm³) shall be added to the

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weight of the fraction collected in the pan.

5. REPORT

5.1 The weights of the fractions retained on each sieve, and the weight of that fraction collected in the pan, shall be expressed as percentages of the test specimen weight to the nearest 0.1 percent, and reported in the following form. Any screen fraction whose percentage of the test specimen weight is less than 0.1 percent shall be reported as "trace."

Table II – Form for Reporting Test Data

| Mesh (Tyler or U.S.S.) | Percentage by Weight |
|---|---|
| + 80 | ..... |
| – 80 +100 | ..... |
| –100 +150 | ..... |
| –150 +200 | ..... |
| –200 +325 | ..... |
| –325 | ..... |

APPENDIX

A1. Method for Obtaining Sieve Correction

Table III –

| Mesh | Certified Sieve | Work Sieve | Factor by Which to Multiply Work Sieve to Convert to Standard |
|---|---|---|---|
| + 80 | 0.1 | 0.1 | 0.1/0.1 = 1. |
| – 80 +100 | 3.0 | 3.0 | 3.0/3.0 = 1. |
| –100 +150 | 10.0 | 10.0 | 10.0/10.0 = 1. |
| –150 +200 | 20.0 | 20.0 | 20.0/20.0 = 1. |
| –200 +325 | 5.0 | 4.0 | 5.0/4.0 = 1.25 |
| –325 | 21.9 | 27.9 | 21.9/27.9 = 0.785 |

A2. Certified Sieve

Sieves conforming to ASTM Specification E11 can be obtained from the sieve manufacturers, and arrangements can be made through them to have the sieves certified by the Bureau of Standards. If used continually, the sieves will, after a period of time, become less accurate and might no longer be acceptable as certified sieves. The common practice, which would be considered acceptable according to this standard, would be to use the certified sieves as a master set for checking other working sets of sieves. By comparing sieve tests on the same sample, run in both the master set and working set, a factor can be established for correcting results on the working sieves. These factors will vary with the coarseness of the powder and should be established for powders of different particle size distribution. The method for obtaining this factor is illustrated by the example given in Table III. All results obtained on the working sieves them would be multiplied by the factor so obtained before reporting.

A3. Comparable Standards

ASTM – B 214
German – 81-69

TRW Materials Technology

Procedure 8851-05: Mill powder to increase its apparent density and flow rate.

A. Equipment:

1. Ball mill.
2. Ball mill jar.
3. Steel balls, 3/4-7/8 inch diameter.
4. Hall apparatus.
5. Top-loading electronic balance.

B. Procedure:

1. Mill powder, as in Procedure 8851-01 for 1/2, 1, or 2 hours, as required, to increase its apparent density to 1.35-1.58 $g/cm^3$ (30-35% of theoretical).

TRW Materials Technology

Procedure 4203-12: Powder Blending
(To be used in conjunction with Procedure 8851-01 "Powder Milling")

1. Weigh out Ti and Al-V in ratio of 90:10 using electronic top-loading balance, Ex 1200 g charge = 120 g Al-V
   1080 g Ti

2. Fill jar with powder and balls as per 8851-01 and blend for 1/2 hour.

TRW Materials Technology

Procedure 8851-20: Die Pressing (Briquetting) of Metal Compacts

A. Equipment:

1. Briquetting die set.
2. Powder compaction press, hydraulic.

B. Procedure:

1. Inspect wall of die and core rod, if any, and remove any adherent metal.
2. Adjust lower punch to provide correct powder fill depth.
3. Use 3.35 inch test bar die to check press for platen parallelism left-to-right and front-to-back. Shim as required, with a 1/4 inch steel plate over the shims.
4. When platens are parallel within 0.002 inch over 3.35 inch, place required briquetting die in center of lower platen. Connect pressure recorder to press.

(4a. Lubricate die wall.)

5. Add powder, tap die, and level off the powder.
6. Fit upper punch into die ao that top of punch is horizontal and punch is at least 1/64 inch into die.
7. Apply required pressure to top punch. Hold 5 seconds.
8. Release pressure.
9. Remove to punch.
10. Use ejector ring to eject the compact. Note ejection force.

TRW Materials Technology

Procedure 4203-410-20, Rev. 0: Briquetting of Inner Gimbal

(To be used in conjunction with Procedure 8851-120, "Die Pressing (Briquetting) of Metal Compacts.")

1. Briquetting pressure for this part is 40 ton/$in^2$. The die area is 0.6 $in^2$; briquetting force = 24 ± 1 ton.

TRW Materials Technology

Procedure 4203-30: Sintering Titanium Powder Compacts

(To be used in conjunction with 8851-30, "Sintering Green Compacts.")

1. Heat sample to 1000°F at rate such that chamber pressue does not exceed $10^{-4}$ torr. Hold ≥15 minutes at this temperature.

2. Heat to 1600°F with same pressure control as in (1). Hold at least 15 minutes at this temperature.

3. Heat to 2250 ±25°F with same pressure control as in (1). Hold at temperature for 4 hours.

END

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