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# APPLICATION OF POWDER METALLURGY TO AN ADVANCED-TEMPERATURE NICKEL-BASE ALLOY, NASA-TRW VI-A

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16. Abstract

Bar stock of the NASA-TRW VI-A alloy was made by prealloyed powder techniques and its properties evaluated over a range of temperatures. Room temperature ultimate tensile strength was 1894 MN/m$^2$ (274 500 psi). The as-extruded powder product showed substantial improvements in strength over the cast alloy up to 649$^{\circ}$ C (1200$^{\circ}$ F) and superplasticity at 1093$^{\circ}$ C (2000$^{\circ}$ F). Both conventional and autoclave heat treatments were applied to the extruded powder product. The conventional heat treatment was effective in increasing rupture life at 649$^{\circ}$ and 704$^{\circ}$ C (1200$^{\circ}$ and 1300$^{\circ}$ F); the autoclave heat treatment, at 760$^{\circ}$ and 816$^{\circ}$ C (1400$^{\circ}$ and 1500$^{\circ}$ F).

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## SUMMARY

One of the strongest high temperature cast nickel-base alloys, NASA-TRW VI-A, was made by prealloyed powder techniques (argon atomization followed by extrusion) and its mechanical properties determined in the as-extruded condition and after conventional and autoclave heat treatments.

The as-extruded powder product demonstrated significant improvements in tensile strength over cast VI-A at room temperature and $649^{\circ}$ C ($1200^{\circ}$ F). The values were 1894 and 1628 MN/m$^2$ (274 500 and 236 000 psi) as against 1049 and 1139 MN/m$^2$ (152 000 and 165 000 psi) for the cast alloy. Tensile elongations were 12 and 5 percent compared to 4.2 and 3.9 percent for the cast alloy. At $982^{\circ}$ C ($1800^{\circ}$ F) and above, however, superplasticity was observed.

Application of a conventional solution and aging heat treatment to the powder product did not affect grain size but resulted in tensile strengths and elongations intermediate to those of the as-extruded powder product and the cast alloy at room temperature and $649^{\circ}$ C ($1200^{\circ}$ F). However, stress-rupture life was increased at temperatures of $649^{\circ}$ and $704^{\circ}$ C ($1200^{\circ}$ and $1300^{\circ}$ F). Lives of 2012 and 1763 hours were obtained for the conventionally heat treated powder product at these temperatures and at stresses that resulted in 100 and 1000 hour lives for the strongest wrought nickel-base alloys in use.

A heat treatment that involved heating the sample above the incipient melting temperature followed by application of high pressure in an autoclave increased grain size from ASTM number 16 to 7 (0.0014 to 0.032 mm diameter). This heat treatment resulted in low stress-rupture life at $649^{\circ}$ C ($1200^{\circ}$ F) and higher lives at $760^{\circ}$ and $816^{\circ}$ C ($1400^{\circ}$ and $1500^{\circ}$ F). At the latter two temperatures and at stresses which gave 1000 hour life for the strongest wrought nickel-base alloys in use, lives of 2877 and 1567 hours were obtained.

# INTRODUCTION

Compressor and turbine disks and the latter stage compressor blades of modern jet engines operate at temperatures as high as 540° to 704° C (1000° to 1300° F). Wrought superalloys are currently used to meet the design requirements of these components. However, further increases in the strength of conventional wrought superalloys are inherently limited, because segregation of alloying constituents which occurs during solidification from the melt prevents the use of strong, highly alloyed compositions. These tend to yield unforgeable ingots which cannot be processed into finished products.

Prealloyed powder techniques afford a means of overcoming the segregation and forming problems inherent in conventional casting and working of highly alloyed compositions. Atomizing a molten alloy with inert-gas jets subjects each metal droplet to rapid solidification rates. This, in turn, upon subsequent powder consolidation, results in a substantially homogeneous material with a fine grain size and a structure free from macrosegregation. This general concept has been applied in a number of investigations (refs. 1 to 5) and has been shown to significantly improve the room temperature and intermediate temperature strength of a number of superalloys. It has also been shown (refs. 4 and 5) that highly alloyed casting alloys, when made by prealloyed powder techniques, can readily be deformed by hot pressing at high temperature. These results suggest that the powder metallurgy approach should have considerable potential in transforming even the strongest cast nickel-base alloys into workable, turbine disk materials with substantial strength advantages over current wrought alloys used for that purpose.

The present investigation was intended to evaluate the NASA-TRW VI-A alloy, one of the strongest recently developed cast nickel-base alloys (refs. 6 and 7), when made by prealloyed powder techniques. The alloy was made by extrusion of prealloyed powders obtained from inert-gas atomization of vacuum melted ingots. Tensile and stress-rupture tests were made at various temperatures with the as-extruded and heat treated material. Impact tests were made at room temperature with the as-extruded powder product. The effects of heat treatments on the mechanical properties and microstructure were evaluated. Data comparisons are presented with the strongest conventionally wrought nickel-base alloys in use.

# MATERIALS AND PROCEDURE

## Materials

Vacuum melted ingots of the NASA-TRW VI-A nickel-base alloy which was originally developed for investment casting were obtained from an alloy manufacturer. This material was remelted under vacuum and then atomized by a stream of high pressure

TABLE I. - CHEMICAL ANALYSES OF VI-A POWDER PRODUCTS

| Element | Specified composition range | Ingot[a] | Powder[b] | Extrusion[b] |
|---|---|---|---|---|
| | Weight percent of element | | | |
| Tantalum | 8.5 to 9.5 | 9.0 | 8.68 | 8.81 |
| Columbium | 0.25 to 0.75 | .47 | .56 | .53 |
| Zirconium | 0.05 to 0.15 | .10 | .10 | .12 |
| Hafnium | 0.20 to 0.70 | .41 | .48 | .50 |
| Rhenium | 0.10 to 0.70 | .41 | .40 | .34 |
| Tungsten | 5.3 to 6.3 | 5.92 | 5.24 | 5.04 |
| Cobalt | 6.5 to 8.5 | 7.52 | 7.78 | 7.58 |
| Titanium | 0.75 to 1.25 | .97 | 1.00 | .90 |
| Molybdenum | 1.5 to 2.5 | 1.98 | 1.72 | 2.17 |
| Chromium | 5.6 to 6.6 | 6.20 | 5.94 | 5.86 |
| Carbon | 0.08 to 0.18 | .15 | .140 | .141 |
| Silicon | ------------- | <.10 | .05 | ----- |
| Manganese | ------------- | <.02 | .004 | ----- |
| Iron | ------------- | ----- | .06 | ----- |
| Sulfur | ------------- | .005 | 10 ppm | ----- |
| Aluminum | 5.0 to 5.8 | 5.53 | 5.41 | 5.25 |
| Boron | 0.012 to 0.024 | ----- | ----- | ----- |
| Nickel | Balance | Balance | Balance | Balance |
| Oxygen | ------------- | ----- | 90 ppm | 59 ppm |

[a]Vendor's analysis.
[b]Analysis by independent laboratory.

argon. Table I shows a comparison of the chemical analyses of the ingot stock, the powder, and the extruded powder product. The oxygen content of the powder and extruded powder material was less than 100 ppm. The analysis of the extruded powder product was in all instances within the specified composition range except for tungsten content which was 5.04 weight percent compared to the 5.3 minimum weight percent specified. The apparent difference in molybdenum content of the powder and the extrusion is unexplained.

The powder was handled under argon until consolidation was completed to avoid contamination of the loose powder by air. Approximately one-third of the atomized material was not used because of geometry and size considerations. The remaining powder was screened (under argon) through screen openings less than 0.210 millimeter square (65 mesh). Only the fraction smaller than 100 mesh (opening of 0.149 mm square) was used for extrusions. The size distribution of the screened powder is shown in table II. Over 40 percent of the powder was finer than 400 mesh (opening of 0.037 mm square).

The powder finer than 100 mesh (opening of 0.149 mm square) was canned at room temperature under vacuum in type 304 stainless steel cans 7.62 centimeters (3 in.) in

TABLE II. - PARTICLE SIZE DISTRIBUTION OF ALLOY

VI-A ATOMIZED POWDER[a]

[Screen analysis.]

| Mesh opening, mm square | Tyler screen size | Particle size distribution, percent |
|---|---|---|
| >0.210 | >65 | 0 |
| .210/.149 | 65/100 | 1.0 |
| .149/.105 | 100/150 | 9.7 |
| .105/.074 | 150/200 | 17.0 |
| .074/.053 | 200/270 | 14.2 |
| .053/.037 | 270/400 | 14.9 |
| <.037 | <400 | 43.2 |

[a]As reported by vendor.

diameter. The sealed cans were then preheated for 1 hour at 816° C (1500° F) followed by 1 hour in a salt pot at the extrusion temperature of 1191° C (2175° F). The cans were extruded at a reduction ratio of 17 to 1 through a die having a 90° included angle to a final diameter of 1.9 centimeters (0.75 in.).

For comparison of material properties, investment cast bars were obtained from the supplier of the master remelt heat.

Density measurements of the extruded alloy gave values of $8.71\times10^3$ kg/m$^3$ (3.14 lb/in.$^3$). This compares to $8.74\times10^3$ kg/m$^3$ (3.16 lb/in.$^3$) measured for the cast alloy.

## Material Conditions

The conditions in which the NASA-TRW VI-A alloy was evaluated are listed in table III. In addition to the as-cast condition, the alloy was tested in the powder product form in the as-extruded condition and after two different heat treatments were applied. One heat treatment which we have designated the autoclave heat treatment, involved heating above the solidus at 1299° C (2370° F) for 1 hour and air cooling, followed by application of pressure of 82.5 or 138 MN/m$^2$ (12 000 and 20 000 psi) in an autoclave at 1232° C (2250° F) for 2 hours.

The significant aspects of the autoclave heat treatment have been discussed in detail in reference 5. Briefly it is intended to close voids which form at the temperatures above the incipient melting point. Such high temperatures are required to achieve grain growth. The voids are attributable both to entrained argon and incipient melting.

Another more conventional three step heat treatment similar to one developed for

TABLE III. - MATERIAL CONDITIONS OF ALLOY VI-A INVESTIGATED

| Form | Condition | Step 1 | Step 2 | Step 3 | Final grain size | |
|---|---|---|---|---|---|---|
| | | | | | ASTM number | Average grain diameter, mm |
| Extruded powder | Autoclave heat treatment | 1 hr, 1299° C (2370° F), air cooled | 2 hr, 1232° C (2250° F) at 82.5 or 138 $MN/m^2$ (12 000 or 20 000 psi), furnace cooled | ------------ | 7 | 0.032 |
| Extruded powder | Conventional heat treat- | 24 hr, 871° C (1600° F), air cooled | 1 hr, 1093° C (2000° F) oil quenched | 16 hr, 760° C (1400° F), air cooled | 16 | 0.0016 |
| Extruded powder | As-extruded | ------------ | -------------------- | ------------ | 16 | 0.0014 |
| Cast | As-cast | ------------ | -------------------- | ------------ | 000 to 0000 | 2 to 5 |

TABLE IV. - TENSILE DATA FOR ALLOY VI-A

| Form | Condition | Test temperature | | Ultimate tensile strength | | Elongation, percent |
|---|---|---|---|---|---|---|
| | | °C | °F | $MN/m^2$ | psi | |
| Extruded powder | As-extruded | R.T.[a] | R.T.[a] | 1894 | 274 500 | 12.0 |
| | | 649 | 1200 | 1628 | 236 000 | 5.0 |
| | | 760 | 1400 | 1294 | 187 500 | 5.0 |
| | | 871 | 1600 | 573 | 86 900 | 10.0 |
| | | 982 | 1800 | 93 | 13 500 | 220.0 |
| | | 1093 | 2000 | 19.3 | 2 800 | >300.0 |
| Extruded powder | Conventional heat treatment | R.T.[a] | R.T.[a] | 1456 | 211 000 | 7.0 |
| | | 649 | 1200 | 1277 | 185 300 | 6.5 |
| | | 760 | 1400 | 1049 | 152 100 | 2.5 |
| | | 1093 | 2000 | 10.4 | 1 500 | >160.0 |
| Extruded powder | Autoclave heat treatment | 760 | 1400 | 1015 | 147 500 | 2.0 |
| Cast | As-cast[b] | R.T.[a] | R.T.[a] | 1049 | 152 000 | 4.2 |
| | | 649 | 1200 | 1139 | 165 000 | 3.9 |
| | | 760 | 1400 | 1097 | 159 000 | 4.4 |
| | | 871 | 1600 | 869 | 126 000 | 2.5 |
| | | 1013 | 1875 | 490 | 71 000 | 5.5 |
| | | 1093 | 2000 | 324 | 47 000 | 4.8 |

[a]Room temperature.
[b]Cast data from ref. 6.

thermomechanically processed material (ref. 8) was also used. In this heat treatment an age at 871° C (1600° F) for 24 hours was used before solution treating at 1093° C (2000° F) for 1 hour followed by 16 hours at 760° C (1400° F).

## Mechanical Testing

All tensile and stress-rupture tests were made in air. The material and test conditions are listed in tables IV and V. Generally, because of the amount of material available only single tests were run at specific test conditions. The tensile and stress-rupture specimens had cylindrical gage sections 0.64 centimeter (0.250 in.) in diameter and 3.18 centimeters (1.25 in.) long with conical shoulders having a 20° included angle. All tests were run in accordance with recommended ASTM practice.

A standard Charpy impact tester was used to measure impact strength of the extruded powder product. V-notched (ASTM Type A) as well as unnotched specimens were

TABLE V. - STRESS-RUPTURE DATA FOR ALLOY VI-A

| Form | Condition | Test temperature | | Stress | | Life, hr | Elongation, percent |
|---|---|---|---|---|---|---|---|
| | | °C | °F | $MN/m^2$ | psi | | |
| Extruded powder | As-extruded | 593 | 1100 | 1069.5 | 155 000 | 7 000+ | ---- |
| | | 593 | 1100 | 1069.5 | 155 000 | 7 000+ | ---- |
| | | 649 | 1200 | 724.5 | 105 000 | 11 000+ | ---- |
| | | 649 | 1200 | 724.5 | 105 000 | 1 039.9 | 8.0 |
| | | 649 | 1200 | 862.5 | 125 000 | 2 326.5 | 9.0 |
| | | 649 | 1200 | 1035.0 | 150 000 | 618.3 | 4.5 |
| | | 704 | 1300 | 586.5 | 85 000 | 1 432.0 | (a) |
| Extruded powder | Conventional heat treatment | 649 | 1200 | 1035.0 | 150 000 | 2 012.9 | (a) |
| | | 704 | 1300 | 586.5 | 85 000 | 1 763.3 | 7.0 |
| | | 760 | 1400 | 427.8 | 62 000 | 82.8 | (a) |
| | | 982 | 1800 | 51.6 | 7 500 | <1 | >300 |
| Extruded powder | Autoclave heat treatment | 649 | 1200 | 1035.0 | 150 000 | 2.8 | 4.0 |
| | | 760 | 1400 | 427.8 | 62 000 | 2 877.2 | 6.0 |
| | | 816 | 1500 | 310.5 | 45 000 | 1 567.1 | 4.5 |
| | | 982 | 1800 | 51.6 | 7 500 | 756.1 | 33.0 |
| Cast | As-cast | 649 | 1200 | 724.0 | 105 000 | 9 000+ | ---- |
| | | 649 | 1200 | 1035.0 | 150 000 | 14.0 | 6.0 |
| | | 649 | 1200 | 1035.0 | 150 000 | 15.8 | (a) |
| | | 760 | 1400 | 427.8 | 62 000 | 4 100+ | ---- |

[a]Elongation not measurable because fracture faces damaged during specimen removal from test grips.

TABLE VI. - CHARPY IMPACT RESISTANCE

| Alloy | Form | Condition | Impact resistance | | | |
|---|---|---|---|---|---|---|
| | | | Unnotched | | Notched | |
| | | | J | ft-lb | J | ft-lb |
| VI-A | Powder product | As-extruded | 109, >150 | 80, >110 | 16 | 12 |
| VI-A[a] | Cast | As-cast | 38 | 28 | ---- | ---- |
| TAZ-8A | Powder product | As-extruded | 34 | 25 | 10 | 7 |
| TAZ-8A[b] | Cast | As-cast | 33 | 24 | ---- | ---- |
| René 41[c] | Wrought | Annealed | >300 | >220 | >150 | >110 |

[a]Ref. 6.
[b]Ref. 12.
[c]Ref. 13.

machined from the extruded bar stock and tested at room temperature. Some additional impact tests were run for comparison. These included TAZ-8A extruded power product and wrought René 41. The materials and test conditions are listed in table VI.

## Metallography

Representative samples of the alloy in various conditions of heat treatment or processing were examined metallographically. Specimens for optical metallography were etched by immersion in a solution of 33 parts each of water, acetic acid, and nitric acid, and one part hydrofluoric acid. Specimens for replication for electron metallography were etched as shown on the electron micrographs.

# RESULTS AND DISCUSSION

The mechanical properties are graphically presented in figures 1 to 4 and listed in tables IV, V, and VI.

## Tensile Properties

The tensile properties of VI-A in the extruded powder product form and in the as-cast condition are compared in figure 1 over a range of temperatures from room temperature to 1093° C (2000° F). The as-extruded powder product had nearly double the ultimate tensile strength of the as-cast alloy at room temperature, 1894 $MN/m^2$

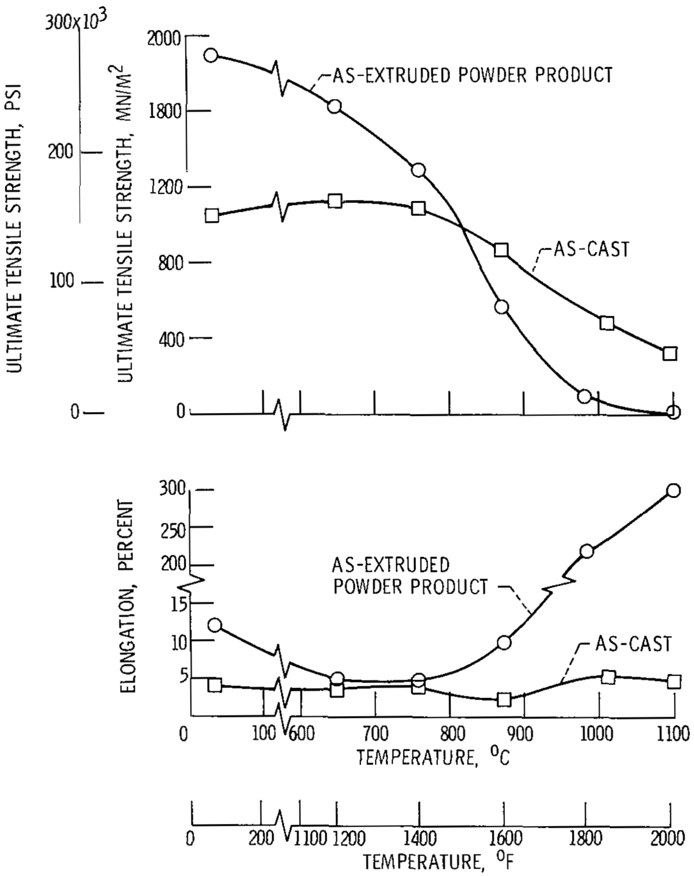
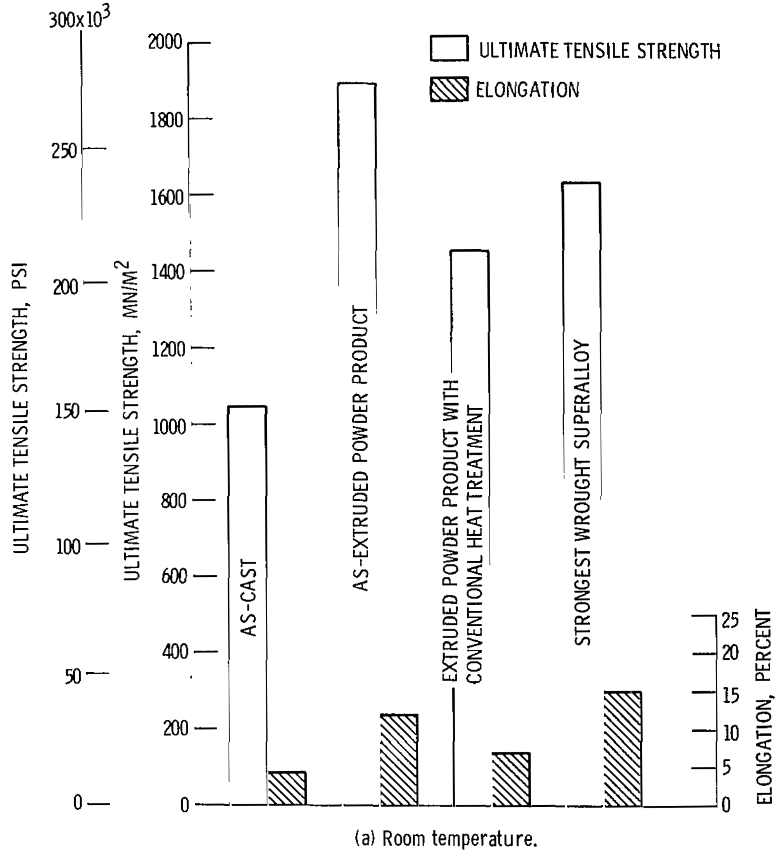
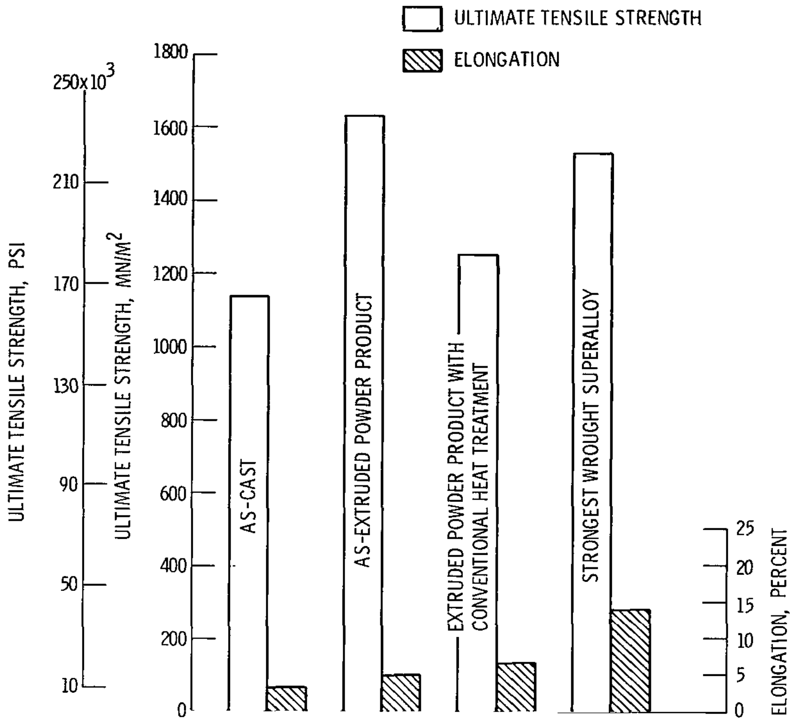



Figure 1. - Comparison of tensile properties of VI-A powder product and cast VI-A.

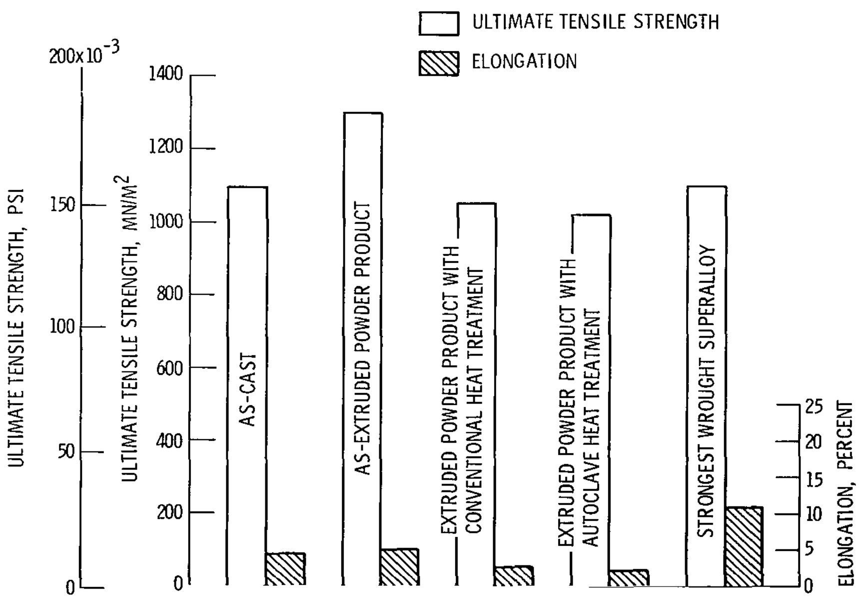



(a) Room temperature.

Figure 2. - Comparison of tensile properties of VI-A powder products, cast VI-A, and strongest conventional wrought nickel-base alloy in use.




(b) 649° C (1200° F).




(c) 760° C (1400° F).

Figure 2. - Concluded.

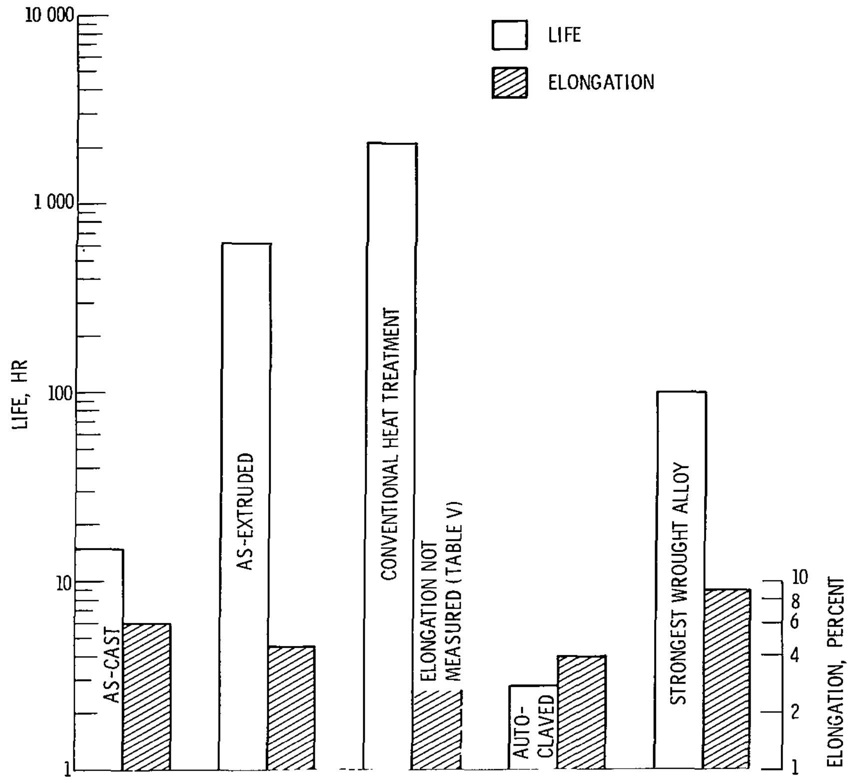
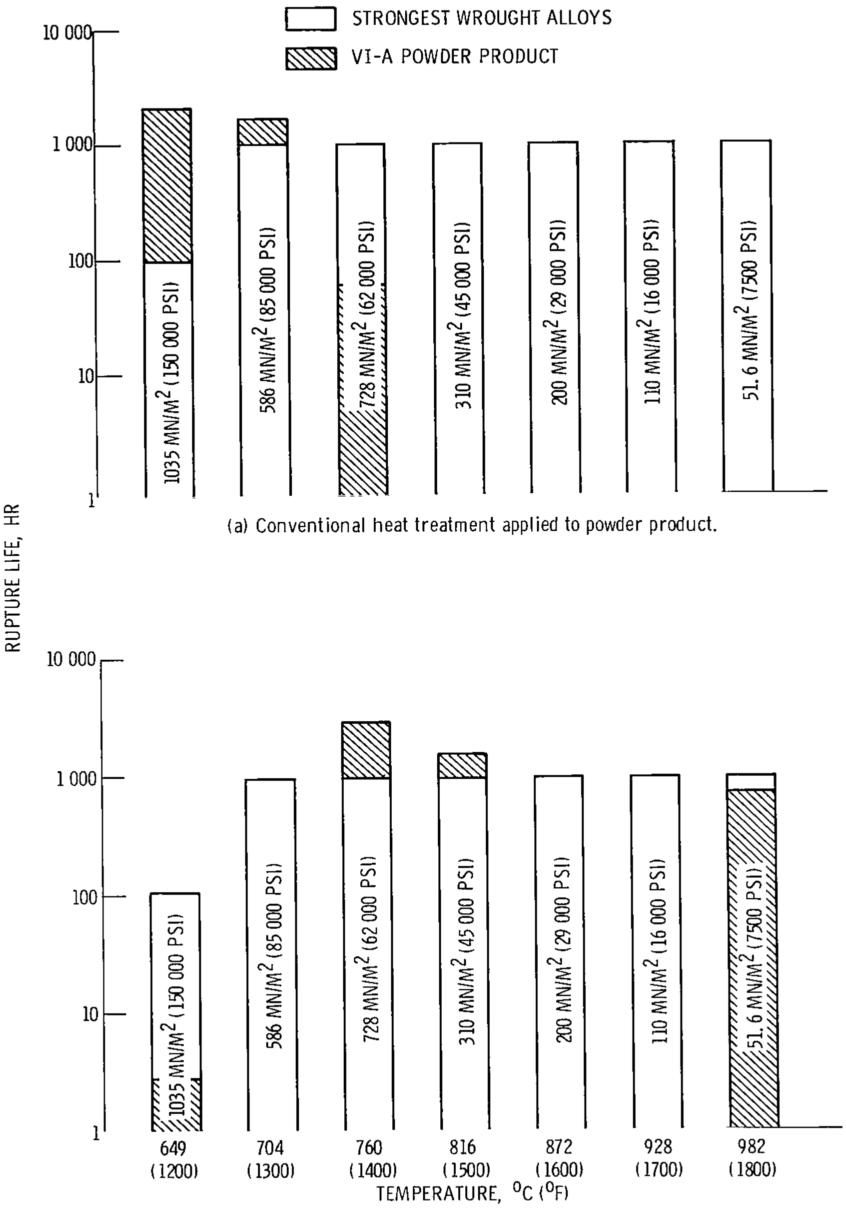



Figure 3. - Comparison of rupture properties of VI-A powder products, cast VI-A, and strongest conventional wrought nickel-base alloy in use, at 649° C (1200° F) and 1035 MN/m² (150 000 psi).

(274 500 psi) as against 1049 MN/m² (152 000 psi), and about $1\frac{1}{2}$ times greater strength at 649° C (1200° F). At the latter temperature the values were 1628 MN/m² (236 000 psi) against 1139 MN/m² (165 000 psi). Tensile elongations were also higher than those of the cast alloy at these temperatures, 12 and 5 percent against 4.2 and 3.9 percent, respectively. A crossover in the strength curves occurred between 760° and 871° C (1400° and 1600° F). At 982° and 1093° C (1800° and 2000° F) the as-extruded powder product exhibited superplastic behavior, having elongations of 220 and more than 300 percent, respectively. Ultimate tensile strengths were 93 and 19.3 MN/m² (13 500 and 2800 psi), whereas the cast alloy had tensile strengths of 490 MN/m² (71 000 psi) at 1013° C (1875° F) and 324 MN/m² (47 000 psi) at 1093° C (2000° F).

A bar chart comparison of the tensile properties of the VI-A alloy in several conditions with those of the strongest representative wrought alloys at room temperature, 649° and 760° C (1200° and 1400° F) is provided in figure 2 (refs. 9 and 10).

As shown in figure 2(a) the room temperature tensile strength of the as-extruded VI-A powder alloy is approximately 1894 MN/m² (274 500 psi). This is about 250 MN/m² (35 000 psi) greater than that of the strongest conventional wrought nickel-base superalloy in use. The as-extruded VI-A has an elongation of 12 percent. This is only slightly lower than the 15 percent elongation of the strongest conventional wrought alloy.




(a) Conventional heat treatment applied to powder product.

(b) Autoclave heat treatment applied to powder product.

Figure 4. - Rupture life comparison of VI-A alloy heat treated powder products and strongest conventional wrought nickel-base alloys in use.

A conventional heat treatment, table III, intended to raise elevated temperature stress-rupture strength of the powder product, decreased tensile strength and ductility at room temperature.

As shown in figure 2(b) this conventional heat treatment increased tensile elongation of the extruded material at $649^{\circ}$ C ($1200^{\circ}$ F) to 6.5 percent but lowered the tensile strength to 1277 $MN/m^2$ (185 300 psi). Both strength and elongation of the conventionally heat treated extruded bar were less than those of the strongest conventional wrought alloy which has a strength of 1530 $MN/m^2$ (220 000 psi) and an elongation of 14 percent.

However, the strength of the heat treated powder product was still considerably higher than that of the cast alloy which was 1139 $MN/m^2$ (165 000 psi). The as-extruded material had the greatest tensile strength, 1628 $MN/m^2$ (236 000 psi), of the materials tested at $649^{\circ}$ C ($1200^{\circ}$ F). Its elongation was 5 percent.

Figure 2(c) indicates that at $760^{\circ}$ C ($1400^{\circ}$ F) the tensile strength of the as-extruded material which was 1294 $MN/m^2$ (187 500 psi), was greater than the strength attained at this temperature by VI-A in any of the other conditions. The strength of the heat treated material, however, was slightly lower than that of the cast alloy. For example, conventionally heat treated material had a 1049 $MN/m^2$ (152 000 psi) tensile strength. This compared to 1097 $MN/m^2$ (159 000 psi) for the cast alloy. The strength of the material with the autoclave treatment was 1017 $MN/m^2$ (147 500 psi). The ductilities of the heat treated material followed the trends of the tensile strength and fell below the ductility of the cast or as-extruded material. The strength of the strongest conventional wrought alloy was less than that of the as-extruded VI-A powder product, 1100 $MN/m^2$ (160 000 psi), but its elongation was about twice as great, 11 percent.

## Stress-Rupture Properties

The stress-rupture data are summarized in table V. Figure 3 shows a comparison at $649^{\circ}$ C ($1200^{\circ}$ F) of the stress-rupture properties of VI-A in the as-extruded powder product form, after application of a conventional heat treatment (table III), an autoclave heat treatment (table III), and in the as-cast condition with the stress-rupture life of one of the strongest conventionally wrought superalloys in use (ref. 9). The stress was 1035 $MN/m^2$ (150 000 psi). At this condition the cast alloy had a life of 14.9 hours, the as-extruded powder product 618 hours, the conventionally heat treated powder product 2013 hours, and the wrought alloy 100 hours. The autoclave heat treated material had substantially lower life, 2.8 hours, lower even than that of the as-cast alloy. The grain size was increased by the autoclave heat treatment to ASTM number 7, but it was still substantially smaller than that of the cast alloy.

Figure 4 provides a bar chart comparison at several temperatures and stresses of rupture lives of the VI-A powder product after the conventional heat treatment and after the autoclave heat treatment. The stresses selected were those which provide 100 hour life at 649° C (1200° F) and 1000 hour life at higher temperatures with the strongest wrought nickel-base alloys in use (refs. 9 to 11). Such a comparison with several wrought alloys is necessary, since no one alloy demonstrates maximum strength at all temperatures. Figure 4(a) shows that the conventionally heat treated powder product gives about twenty times the life at 649° C (1200° F) and $1\frac{3}{4}$ times the life at 704° C (1300° F) of the strongest wrought alloy. At 760° C (1400° F) however, rupture life is about an order of magnitude lower, 83 hours. And at 982° C (1800° F) the life drops to less than 1 hour and elongation exceeds 300 percent. These results suggest that the VI-A alloy when made by the prealloyed powder process and subsequently subjected to a conventional heat treatment has a substantial strength advantage over the strongest wrought nickel-base alloys, precisely in the temperature range of interest for advanced turbine disk applications. However, this type of heat treatment will not impart strength to the VI-A powder product above this temperature, principally because the grain size has not been increased. Figure 4(b) dramatically illustrates the effect of the autoclave heat treatment on the intermediate and high temperature strength of the VI-A prealloyed powder product. At 649° C (1200° F) rupture life is almost two orders of magnitude less than that of the strongest wrought alloy. However, at 760° and 816° C (1400° and 1500° F) it is about 3 and $1\frac{1}{2}$ times greater, respectively. Even at as high a temperature as 982° C (1800° F) it approaches the 1000 hour wrought alloy life with a life of 756 hours. However, it should be noted that the useful life at this temperature could be significantly shorter, because the rupture elongation was quite large, 33 percent (table V).

Clearly the grain size increase to ASTM number 7 resulting from the autoclave heat treatment must be a major factor contributing to increased life. The conventionally heat treated material is unusable at high temperatures. At 982° C (1800° F) and a stress of 51.6 $MN/m^2$ (7500 psi) it is superplastic, with an elongation greater than 300 percent, and a life of less than 1 hour. This superplasticity, however, makes it possible to work the alloy to achieve a desired product such as a turbine disk, which functions in the intermediate temperature range where the high strength of the powder product can be used to advantage.

With respect to the elongations observed after rupture testing at lower temperatures it should be noted that in the 649° to 816° C (1200° to 1500° F) temperature range, values ranging from 4 to 9 percent were obtained. On the basis of the limited data obtained, the type of heat treatment applied did not appear to have a noticeable effect on rupture ductility.

## Impact Resistance

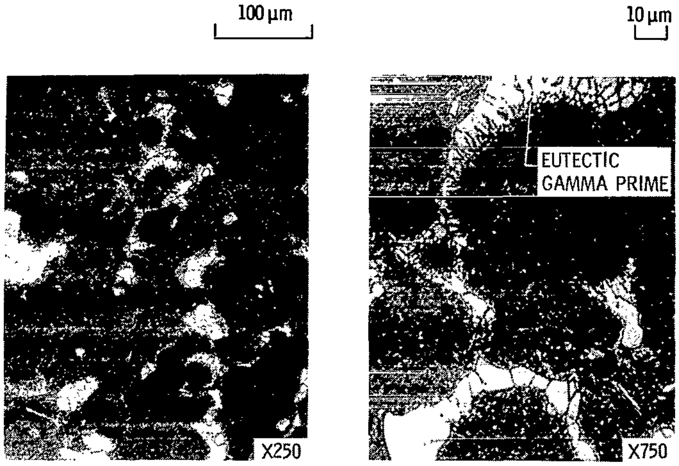
Impact data for VI-A are given in table VI. Included in the listing are data for cast TAZ-8A, TAZ-8A powder product, and a conventional wrought alloy, René 41. The cast TAZ-8A and wrought René 41 unnotched data are taken from references 12 and 13, respectively. In the unnotched condition the as-extruded VI-A powder product has greater than 109 joules (80 ft-lb) impact strength. This is substantially higher than that of cast VI-A which has an unnotched impact strength of 38 joules (28 ft-lb) (ref. 6), as well as the TAZ-8A powder product (34 J or 25 ft-lb). However, its unnotched impact strength is still considerably lower than that of the conventional wrought alloy, René 41, which has an impact strength of more than 300 joules (220 ft-lb).

The VI-A extruded powder product had greater notched impact strength (16 J or 12 ft-lb) than the TAZ-8A powder product (10 J or 7 ft-lb). The large difference between unnotched and notched impact strength for the as-extruded VI-A powder product as contrasted to the much smaller difference noted for the TAZ-8A powder product, suggests that it is more notch sensitive than the latter alloy. Compared to mill annealed René 41, the notched impact resistance of the as-extruded VI-A powder product is much lower, 16 joules (12 ft-lb), as against more than 150 joules (110 ft-lb). The notched impact strength of the VI-A powder product is superior to that of the better cast nickel- and cobalt-base alloys reported in reference 14. The notched impact resistance values of the cast materials ranged from approximately 3 to 14 joules (2 to 10 ft-lb). The effect of the various heat treatments that were applied to the VI-A powder product to enhance strength was not investigated with respect to impact strength due to lack of sufficient material.

## Metallography

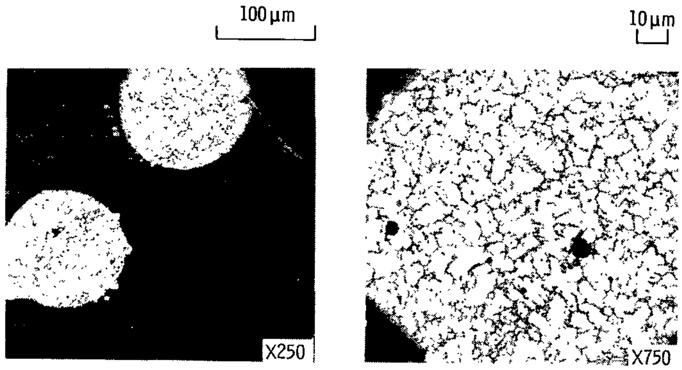
The structure of cast VI-A is shown in figure 5. The matrix has a precipitate of gamma prime that is not resolved at a magnification of 750. The massive gamma prime particles exhibit a eutectic-like structure. Some carbides are also evident. The grain size determined from the surface of etched tensile bars is 3 to 5 millimeters in diameter (ASTM no. 000 to 0000).

Figure 6 shows the structure of the atomized powder at three magnifications. The powder particles generally have a round cross section, a few protuberances, and some voids. If one assumes that each white region surrounded by a dark constituent is a grain, the grain diameter is 0.009 millimeter (approximately ASTM no. 11). At a higher magnification, 26 000, no precipitate can be seen in the matrix itself. Regions as small as 0.0015 millimeter appear to be surrounded by grain boundaries. In the bound-

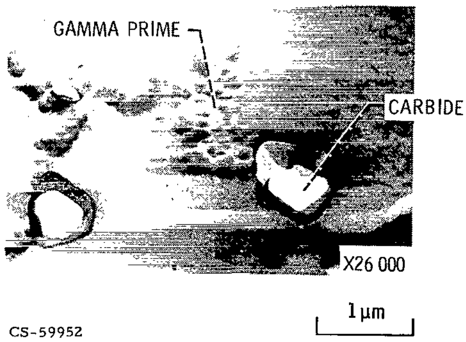



CS-59954

Figure 5. - As-cast VI-A alloy. Etchant: 33 parts water, 33 parts glacial acetic acid, 33 parts nitric acid, 1 part hydrofluoric acid. (Reduced 50 percent in printing).



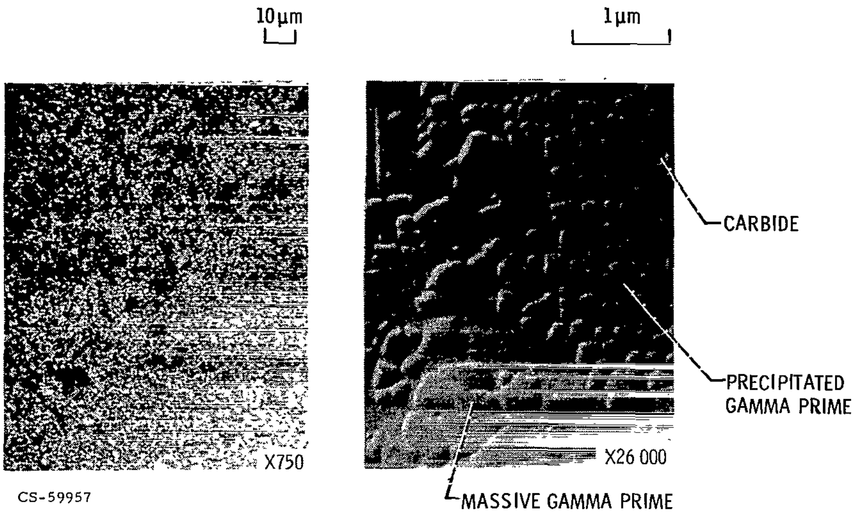

(a) Etchant: 33 parts water, 33 parts glacial acetic acid, 33 parts nitric acid, 1 part hydrofluoric acid.




CS-59952

(b) Etchant: 40 parts lactic acid, 25 parts water, 25 parts hydrochloric acid, 10 parts nitric acid.

Figure 6. - As-received powders of VI-A alloy. (Reduced 50 percent in printing).




CS-59957

(a) Etchant: 33 parts water, 33 parts glacial acetic acid, 33 parts nitric acid, 1 part hydrofluoric acid.

(b) Etchant: 40 parts lactic acid, 25 parts water, 25 parts hydrochloric acid, 10 parts nitric acid.

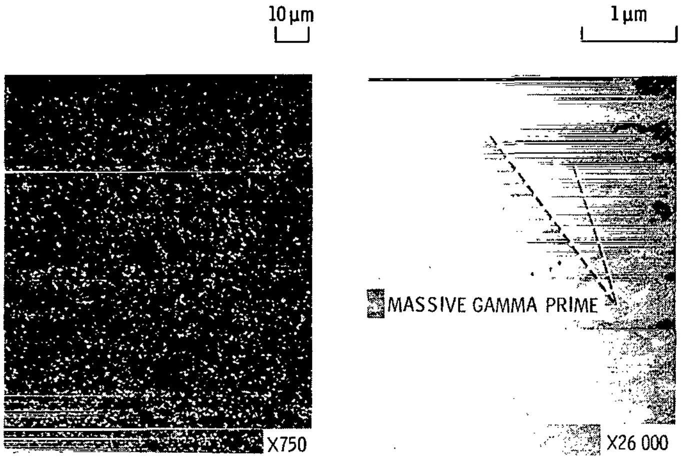
Figure 7. - As-extruded VI-A alloy powder product. (Reduced 50 percent in printing).

aries is a cuboidal appearing constituent presumed to be gamma prime. Carbides are also evident, primarily at the boundaries.

After extrusion (fig. 7) the grains are much finer, having a diameter on the order of 0.0014 millimeter (ASTM no. 16). Very little detail can be resolved at a magnification of 750. At a magnification of 26 000, however, it is apparent that a gamma prime precipitate is present throughout the matrix. Only a few carbides are evident and these are considerably smaller than the carbides in the powder.

At a magnification of 750 (fig. 8) the microstructure of the conventionally heat treated powder product (table III) looks very like that of the as-extruded material. However, at a magnification of 26 000 differences are evident. Primarily the amount of massive gamma prime has increased at the expense of the precipitated gamma prime from the matrix. The remaining precipitate has coarsened. The contours of the massive gamma prime are somewhat more angular. No fine precipitate is evident in the matrix even at a magnification of 26 000.

A heat treatment of 1 hour at 1299$^\circ$ C (2370$^\circ$ F), the first step of the autoclave heat treatment, increased the grain size of the extruded powder product to 0.032 millimeter (ASTM no. 7). A number of voids are present (fig. 9). However, only minor indications of incipient melting appear, evidenced by eutectic gamma prime. At the periphery of some of the massive gamma prime islands, intrusions of gamma (observable at a magnification of 10 000) suggest that incipient melting had begun. The voids are presumed to be largely the result of entrained argon. Although the extrusion cans were evacuated before they were welded shut, they were not heated before sealing to drive off entrained

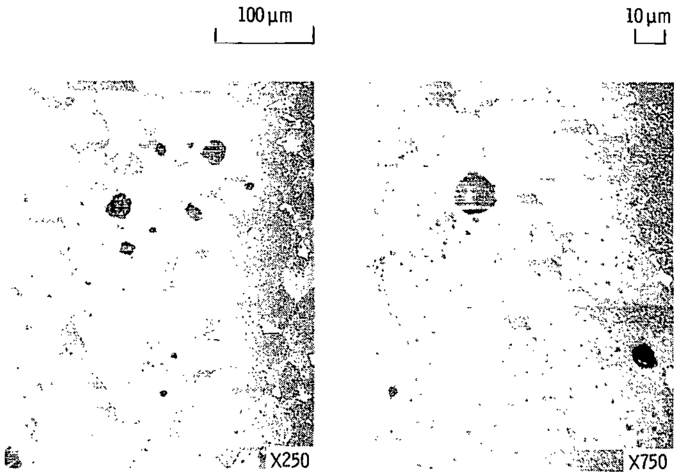



CS-59955

(a) Etchant: 33 parts water, 33 parts glacial acetic acid, 33 parts nitric acid, 1 part hydrofluoric acid.

(b) Etchant: 75 parts glacial acetic acid, 15 parts hydrochloric acid, 5 parts sulphuric acid, 5 parts nitric acid.

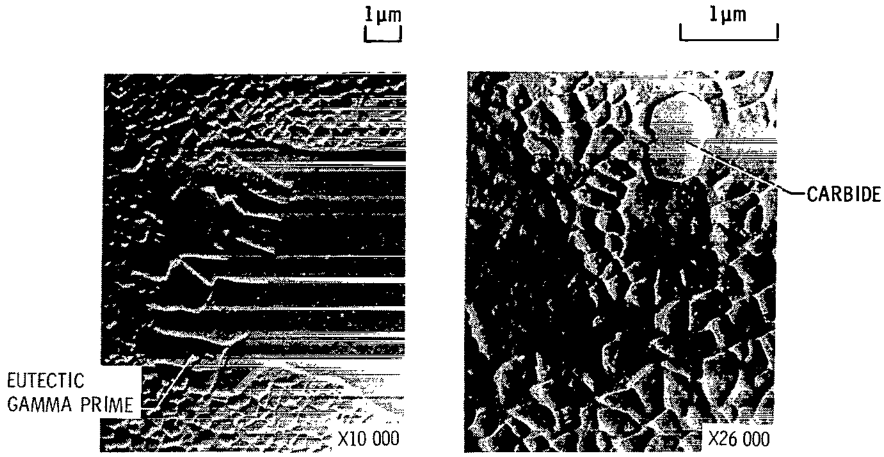
Figure 8. - Conventionally heat treated VI-A alloy powder product. 816° C (1600° F) for 16 hours (air cooled), 1093° C (2000° F) for 1 hour (oil quenched), and 760° C (1400° F) for 16 hours (air cooled). (Reduced 50 percent in printing).




CS-59953

(a) Etchant: 33 parts water, 33 parts glacial acetic acid, 33 parts nitric acid, 1 part hydrofluoric acid.

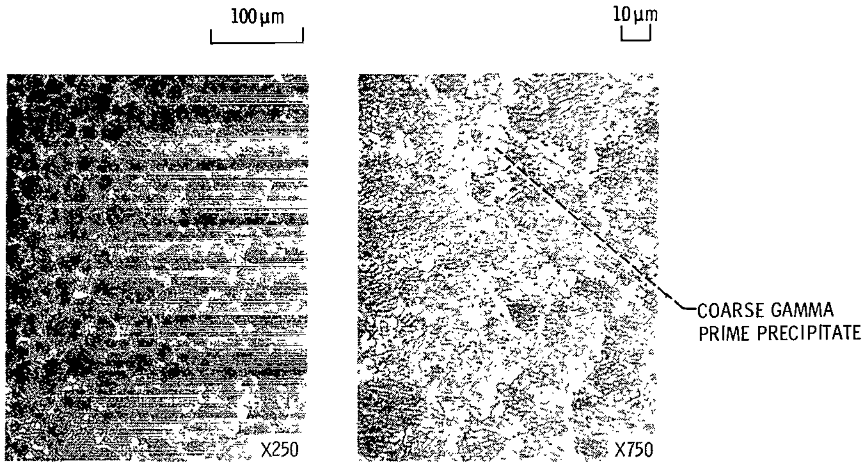
Figure 9. - Microstructure of VI-A powder product after first step of autoclave heat treatment for 1 hour at 1299° C (2370° F). (Reduced 50 percent in printing).



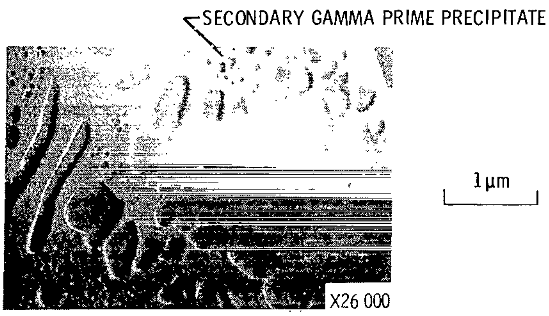

CS-59956

(b) Etchant: 10 percent hydrochloric acid (electrolytic).

Figure 9. - Concluded.




(a) Etchant: 33 parts water, 33 parts glacial acetic acid, 33 parts nitric acid, 1 part hydrofluoric acid.




CS-59951

(b) Etchant: 10 percent hydrochloric acid (electrolytic).

Figure 10. - Alloy VI-A powder product after autoclave heat treatment at 1299° C (2370° F) for 1 hour (air cooled) and at 1232° C (2250° F) for 2 hours at 138 $MN/m^2$ (20 000 psi) (furnace cooled). (Reduced 50 percent in printing).

gas. Compared to the as-extruded powder (fig. 7), the carbides also increased in size. The second step of the autoclave heat treatment at 1232° C (2250° F) had little effect on grain size. In fact, comparing figures 9 and 10 reveals that the grain size measured after the autoclave heat treatment is slightly less (0.032 mm diameter) than that of the material heated only to 1299° C (2370° F). The grains of gamma are ringed with massive gamma prime. Because of the slow cooling in the autoclave the gamma prime precipitate is relatively coarse (fig. 10). The gamma contains a fine secondary precipitate of gamma prime. A few small carbides are evident. There was no significant difference in the appearance of the microstructure of the powder product after autoclave treatments applied at 82.8 $MN/m^2$ (12 000 psi) and those applied at 138 $MN/m^2$ (20 000 psi).

## CONCLUDING REMARKS

The results of this investigation reaffirm results previously obtained with other nickel- and cobalt-base alloys (NASA TN's D-5248 and D-6072) that substantial gains in room temperature and intermediate temperature strength can be obtained with extruded prealloyed powder products compared to their cast counterparts. The 1894-$MN/m^2$ (274 500-psi) room temperature tensile strength obtained with the highly alloyed VI-A extruded prealloyed powder product certainly represents a significant increase in strength compared to that of the strongest conventional wrought nickel-base alloys in use. These are inherently limited in alloy content because of potential segregation problems. The results also indicate that conventional heat treatments, even though they do not affect grain size, can further substantially increase the intermediate temperature stress-rupture life of an extruded powder product, at least up to 704° C (1300° F). For the VI-A alloy the rupture properties at these temperatures are considerably higher than those of the strongest wrought nickel-base alloys in use. However, this increase in stress-rupture life was accompanied by a decrease in tensile strength. The latter was lower than that of the strongest conventionally wrought nickel-base alloy, but greater than the cast version of VI-A.

Autoclave heat treatments can significantly increase the stress-rupture life of the extruded powder product at 760° C (1400° F) and above. This is in part associated with the increased grain size resulting from such heat treatments. This result substantiates earlier data obtained with the application of an autoclave heat treatment to the cobalt-base alloy, HS-31, described in NASA TN D-6072.

Finally, this investigation has demonstrated that there is a wide latitude in the strength and temperature combinations that can be achieved with prealloyed powder products depending upon the heat treating technique employed. Further investigation is required to determine the optimum heat treatment needed to achieve any particular set of properties. However, guidelines have been established that suggest a conventional heat treatment for improved intermediate temperature rupture strength and an autoclave heat treatment for improved rupture strength at higher temperatures.

## SUMMARY OF RESULTS

Prealloyed powders of the NASA-TRW VI-A alloy were made by inert gas atomization and extruded into bar stock. The powder product was evaluated by tensile and stress-rupture tests in the as-extruded condition and after two heat treatments. The following major results were obtained:

1. Up to intermediate temperatures, tensile strengths and elongations of the as-extruded powder product were substantially increased compared to those of the cast alloy. At high temperatures the as-extruded powder product had lower strength and substantially higher elongation than the cast alloy. For example, ultimate tensile strengths of the extruded powder product ranged from 1894 MN/$m^2$ (274 500 psi) at room temperature to 93 MN/$m^2$ (13 500 psi) at 982$^\circ$ C (1800$^\circ$ F) compared to 1049 MN/$m^2$ (152 000 psi) and 560 MN/$m^2$ (83 000 psi) for the cast alloy. Tensile elongations at these two temperatures for the extruded powder product were 12 and 220 percent (indicative of superplasticity), respectively, compared to 4.2 and 5 percent for the cast alloy.

2. At an intermediate temperature, 649$^\circ$ C (1200$^\circ$ F), and high stress, 1035 MN/$m^2$ (150 000 psi), such as might be encountered in advanced turbine engine compressor and turbine disks, the stress-rupture life of the extruded powder product was 618 hours. This exceeded the life of the strongest conventionally wrought nickel-base alloys in use (approximately 100 hr) as well as that of as cast VI-A (14 hr).

3. Application of a conventional heat treatment involving a solution and aging sequence did not affect grain size. Stress-rupture life at 649$^\circ$ C (1200$^\circ$ F) and 1035 MN/$m^2$ (150 000 psi) was substantially increased, both compared to the as-extruded powder product and the strongest conventional wrought alloy in use (2012.9 hr versus 618 and 100 hr, respectively). Above 704$^\circ$ C (1300$^\circ$ F), the conventionally heat treated powder product had lower stress-rupture life than the strongest conventional wrought nickel-base alloys. At intermediate temperatures of 649$^\circ$ and 760$^\circ$ C (1200$^\circ$ and 1400$^\circ$ F) tensile strength was decreased compared to that of the as-extruded powder product.

4. Application of an autoclave heat treatment increased grain size from ASTM number 16 to 7. At 760$^\circ$ and 816$^\circ$ C (1400$^\circ$ and 1500$^\circ$ F), the autoclaved material had a higher stress-rupture life than the strongest conventional wrought alloys in use. At 982$^\circ$ C (1800$^\circ$ F) and 51.6 MN/$m^2$ (7500 psi) the autoclaved material had a life approach-

ing that of the strongest wrought nickel-base alloy, but had a rupture elongation of 33 percent. The autoclave heat treatment, however, reduced rupture life compared to the as-extruded powder product and the strongest conventional wrought nickel-base alloys at $649^{\circ}$ C ($1200^{\circ}$ F) and 1035 $MN/m^2$ (150 000 psi).

5. The VI-A as-extruded powder product exhibited a room temperature notched Charpy impact resistance of 16 joules (12 ft-lb). This is higher than impact strengths obtained with representative cast nickel- and cobalt-base alloys, but substantially less than that obtained with a conventional wrought nickel-base alloy, René 41, in the mill annealed condition. Unnotched impact values for as-extruded VI-A were greater than 109 joules (80 ft-lb); this compares to 38 joules (28 ft-lb) for the cast version of VI-A.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 8, 1971,
134-03.

## REFERENCES


1. Parikh, N. M.; Farrell, K.; and Spachner, S. A.: Improved Production of Powder Metallurgy Items. Rep. IITRI-B-247-25, IIT Research Inst. (AFML-TR-65-103, DDC. No. AD-464368), Mar. 1965.
2. Friedman, Gerald I.; and Loewenstein, Paul: Processing Techniques for the Extrusion of Superalloy Powders. Rep. NMI-9709-19, Whittaker Corp. (AFML-TR-68-321, DDC No. AD-845185), Oct. 1968.
3. Friedman, Gerald; and Kosinski, Edward: High-Performance Material From a Hot-Worked Superalloy Powder. Metals Eng. Quart., vol. 11, no. 1, Feb. 1971, pp. 48-50.
4. Freche, John C.; Waters, William J.; and Ashbrook, Richard L.: Evaluation of Two Nickel-Base Alloys, Alloy 713C and NASA TAZ-8A, Produced by Extrusion of Prealloyed Powders. NASA TN D-5248, 1969.
5. Freche, John C.; Ashbrook, Richard L.; and Waters, William J.: Evaluation of a Cobalt-Base Alloy, HS-31, made by Extrusion of Prealloyed Powders. NASA TN D-6072, 1970.
6. Collins, H. E.: Development of High Temperature Nickel-Base Alloys for Jet Engine Turbine Bucket Applications. Rep. TRW-ER-7162, TRW, Inc. (NASA CR-54507), June 20, 1967.

7. Collins, H. E.; Quigg, R. J.; and Dreshfield, R. L.: Development of a Nickel-Base Superalloy Using Statistically Designed Experiments. Trans. ASM, vol. 61, no. 4, Dec. 1968, pp. 711-721.

8. Barker, J. F.; Oxx, G. D.; Perlmutter, I.; and Wukusick, C. S.: Effect of Processing Variables on the Properties of René 95. Presented at the Symposium on Thermo-Mechanical Treatment of Metals, London, England, Apr. 30 - May 1, 1970.

9. Barker, James F.; and Calhoun, Clyde D.: AF95 Powder Manufacturing Techniques. Rep. R71AEG122, General Electric Co. (AFML-TR-70-314, AD-881272L), Dec. 1970.

10. Anon.: High Temperature, High Strength Nickel Base Alloys. Rev. ed., International Nickel Co., Inc., 1968.

11. Anon.: Aircraft Engine Material Selector. Aircraft Engine Tech. Div., Aircraft Engine Group, General Electric Co., 1969.

12. Waters, William J.; and Freche, John C.: Investigation of Columbium-Modified NASA TAZ-8 Superalloy. NASA TN-D-3597, 1966.

13. Freche, John C.; and Waters, William J.: Continued Investigation of an Advanced-Temperature, Tantalum-Modified, Nickel-Base Alloy. NASA TN D-1531, 1963.

14. Freche, John C.: Nickel-Base Alloys for Aerospace Applications. Aerospace Structural Materials. NASA SP-227, 1970, pp. 71-89.



NASA-Langley, 1971 —— 17 E-6490