NASA TECHNICAL NOTE

NASA TN D-7117

NASA TN D-7117

EFFECT OF AUTOCLAVE HEAT TREATMENTS ON THE MECHANICAL PROPERTIES OF THE PREALLOYED POWDER COBALT-BASE ALLOY HS-31

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • FEBRUARY 1973

1. Report No.: NASA TN D-7117
2. Government Accession No.:
3. Recipient's Catalog No.:
4. Title and Subtitle: EFFECT OF AUTOCLAVE HEAT TREATMENTS ON THE MECHANICAL PROPERTIES OF THE PREALLOYED POWDER COBALT-BASE ALLOY HS-31
5. Report Date: February 1973
6. Performing Organization Code:

7. Author(s): John C. Freche and Richard L. Ashbrook

8. Performing Organization Report No.: E-7107

9. Performing Organization Name and Address:
Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio 44135

10. Work Unit No.: 501-21
11. Contract or Grant No.:
12. Sponsoring Agency Name and Address:
National Aeronautics and Space Administration
Washington, D.C. 20546
13. Type of Report and Period Covered: Technical Note
14. Sponsoring Agency Code:
15. Supplementary Notes:

16. Abstract

The cobalt-base alloy HS-31 was atomized into powder and then consolidated by extrusion or by hot isostatic pressing (HIP) in an autoclave over a range of temperatures spanning the solidus, approximately 1280° C (2340° F). Extrusions were subsequently autoclaved at the same conditions. Extrusions autoclaved at 1327° C (2420° F) had a life of 300 hours at 649° C (1200° F) and 30 hours at 982° C (1800° F) at stresses that result in a 10-hour life with cast HS-31. Superior stress rupture lives of autoclaved material are probably related to the solidification structure at the grain boundaries as well as to the increased grain size.

17. Key Words (Suggested by Author(s)):
Cobalt-base
Superalloys
Prealloyed powder
Powder metallurgy
HIP
Autoclave

18. Distribution Statement: Unclassified - unlimited

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price*
Unclassified	Unclassified	23	$3.00

* For sale by the National Technical Information Service, Springfield, Virginia 22151

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SUMMARY

Prealloyed powder processing has been used to achieve improved properties with cast superalloy compositions, particularly up to intermediate temperatures. To fully exploit this approach processing variables must be investigated. This investigation deals with the commonly used cast cobalt-base alloy HS-31, which was made into a powder by argon atomization and then consolidated by extrusion of canned powders or by direct hot isostatic pressing. Hot isostatic pressing was done in an autoclave at 68.9 meganewtons per square meter (10 ksi) and at temperatures from 1227° to 1393° C (2240° to 2540° F), which extended from below to above the solidus temperature (~1280° C (2340° F)) of HS-31. A portion of the extruded material was also subjected to these same autoclave conditions. Some as-extruded material was aged at 732° C (1350° F) for 50 hours, a treatment used to increase the tensile strength of cast HS-31.

At intermediate and high temperatures both the hot isostatically pressed powders and the autoclaved extruded material exhibited similar responses to the autoclave treatment. Intermediate autoclave temperatures resulted in a maximum rupture life at both intermediate and high temperatures for both powder products. An extruded powder product autoclaved at 1327° C (2420° F) had a life of 300 hours at 649° C (1200° F) and a stress of 420.6 meganewtons per square meter (61 ksi) and a life of 30 hours at 982° C (1800° F) and 89.6 meganewtons per square meter (13 ksi). These values contrasted to the 10-hour average life for the as-cast alloy at both of these test conditions. The tensile strength of the extruded powder material autoclaved at 1327° C (2420° F) was slightly higher than that of the as-cast material from room temperature to 982° C (1800° F). The aging treatment reduced the stress-rupture life of the as-extruded material at all temperatures except at 982° C (1800° F) but resulted in virtually no change in tensile strength over the entire temperature range.

The superior stress-rupture lives of materials autoclaved at 1327° C (2420° F) appear to result at least in part from a solidification structure at the grain boundaries, not merely from an increased grain size.

INTRODUCTION

The general concept of prealloyed powders has been used with promising results to produce nickel- and cobalt-base superalloys as well as other materials (refs. 1 to 7). It has been shown that the tensile strengths of as-extruded prealloyed powder products of normally cast nickel-base alloys (refs. 3 to 5 and 7) and a cobalt-base alloy (ref. 6) were almost twice those of their cast counterparts up to 649° C (1200° F). At 982° C (1800° F) and above, however, the as-extruded powder products had significantly lower tensile and stress-rupture strengths than the cast alloys. One promising approach to the use of prealloyed powders involves their compaction by hot pressing or extrusion and the subsequent application of heat treatments to achieve desired properties (refs. 3 to 7). When high pressures and high temperatures were applied in an autoclave after conventional heat treatment at temperatures above the minor phase melting point of the as-extruded powder products, coarser grained powder products without voids were achieved. In one instance (ref. 6), rupture lives greater than those of the cast alloy were obtained over the entire temperature range; in another instance (ref. 7), substantially greater rupture lives were obtained at 760° C (1400° F) and above than were possible by heat treatments below the incipient melting temperature.

The investigations of prealloyed powder material compacted by extrusion followed by combined conventional and autoclave heat treatments (refs. 6 and 7) suggested that varying the autoclave heat treatment temperature could significantly alter the subsequent properties of the powder product. This aspect was therefore investigated using available HS-31 extruded prealloyed powder material. In view of the possibility that prealloyed powders may be used more effectively in fabricating components such as turbine disks or blades by directly hot pressing the powders to the final component configuration, without first compacting them by extrusion, direct hot isostatic pressing of canned HS-31 powders was also investigated.

Mechanical properties of HS-31 prealloyed powder products consolidated by both methods, extrusion of canned powder and direct hot isostatic pressing of canned powder, were obtained. The hot isostatic pressing was done in an autoclave at 68.9 meganewtons per square meter (10 ksi) and at temperatures from 1227° to 1398° C (2240° to 2540° F), a temperature range which extended from below to above the solidus temperature (~1280° C (2340° F) of HS-31. Some of the extruded powder product was also subjected to these same autoclave conditions. The purpose of varying the autoclave temperature in this fashion was to determine if an optimum autoclave heat treatment could be obtained that would result in a powder product with substantial strength increases compared to cast HS-31 over its useful temperature range. In addition to determining the effects of heat treatment variations on mechanical properties (tensile and stress-rupture), the effects on the microstructure of the powder product were also determined.

MATERIALS AND PROCEDURES

Preparation of Powders for Extrusions and Hot Pressing

Prealloyed powder and extruded bars of the cobalt-base alloy HS-31 (X-40) were obtained from a manufacturer of high temperature alloys. The powder was made by argon remelting and atomization of air-melted HS-31.

A screen analysis typical of the -30 mesh powder used is given in table I. This powder was somewhat finer than that used in the investigation of reference 6. Table II shows the nominal chemical composition of HS-31 and a sample analysis of the powder reported by the supplier. The oxygen content was 0.049 percent, almost five times as great as the 0.011 percent reported in reference 6. Analyses for argon were made for three ranges of powder particle size screened from the original -30 mesh powder. The argon contents were all well below 1 ppm. The argon content decreased as the mesh size decreased. For powders falling in the -30 to +100 mesh range, argon content was 0.09 ppm; for the -100 to +325 mesh range, it was 0.03 ppm; and for the -325 mesh powders, it was 0.01 ppm.

The bars were extruded at 1204^{o} C (2200^{o} F) directly from -30 mesh powders canned in mild steel. Two canning procedures were used. In the first, extrusion cans were welded shut in a button furnace which, after evacuation, was back filled with enough argon to sustain an arc. Later in the program a second procedure was used in which the extrusion cans were welded shut under vacuum in an electron beam welding chamber. No significant property differences were observed between materials canned by either method. An extrusion ratio of 11.1 to 1 produced bars having a diameter of about 1.43 centimeters (9/16 in.) after decanning in an acid bath. The extruded bars were found to be radiographically sound when examined using an iridium source. Fluorescent penetrant inspection of machined bars also showed them to be sound except for elongated inclusions which may be associated with the high oxygen content.

A portion of the powder was prepared for hot isostatic pressing (HIP) by sealing it in 304 stainless steel 0.75-millimeter (0.03-in.) wall tubes 1.58 centimeters (5/8 in.) in diameter and 8.75 centimeters ($3\frac{1}{2}$ in.) long. The tubes were heated to approximately 538^{o} C (1000^{o} F) for 1 hour in an electron beam welding chamber and welded shut at a pressure of less than 1.33×10^{-2} newton per square meter (1×10^{-4} torr).

Heat Treatments

Autoclave. - Specimen blanks approximately 7.62 centimeters (3 in.) long were cut from extruded bars and were heat treated in an autoclave. They were held for 2 hours at a pressure of 68.9 meganewtons per square meter (10 ksi). The small tubes of powder were simultaneously compacted and heat treated in batches which included the extruded bars. The temperatures selected ranged from 1227° to 1393° C (2240° to 2540° F). Most of the autoclave temperatures employed exceeded the 1280° C (2340° F) (ref. 6) solidus temperature of HS-31.

Conventional. - A portion of the extruded bars were aged at 732° C (1350° F) for 50 hours. This intermediate temperature heat treatment has been reported to increase low and intermediate temperature strength of cast HS-31 (ref. 8).

Mechanical Testing

Tensile and stress-rupture tests were made in air. Tensile tests were conducted at temperatures to 1093° C (2000° F) and stress-rupture tests to 982° C (1800° F) with as-extruded and heat-treated material. The test conditions, as well as the results, are listed in tables III to VI. Generally, only single tests were run at a particular test condition because of the limited amount of extruded powder product available. The 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. Tensile tests were made at an independent laboratory using the same size specimens with threaded ends. All tests were run in accordance with recommended ASTM practice.

Metallography

Representative samples of the various conditions of heat treatment or processing were examined metallographically after being etched electrolytically in a 5-percent aqua regia (3 parts hydrochloric acid, 1 part nitric acid) water solution. An electrolytic mixed acid etch was superimposed on the aqua regia etch to reveal grain boundaries of material autoclaved at the two lowest temperatures considered and also applied to samples of as-extruded and extruded and aged material. The etchant used was 33 parts each of water, acetic acid, and nitric acid and 1 part hydrofluoric acid.

RESULTS AND DISCUSSION

The mechanical properties are presented graphically in figures 1 to 5 and the data are tabulated in tables III to VI. Metallographic results are shown in figures 6 to 8.

Stress-Rupture Properties

Determination of optimum autoclave temperature on the basis of stress-rupture life. - Stress-rupture properties of the HS-31 prealloyed powder material autoclaved at various temperatures from 1227° to 1393° C (2240° to 2540° F) are listed in table III and plotted in figures 1 and 2. These data represent stress-rupture tests run at a high temperature of 982° C (1800° F) and at an intermediate temperature of 732° C (1350° F) and at stresses that would be expected (ref. 8) to result in a 10-hour life with as-cast HS-31. The stress at 982° C (1800° F) was 89.6 meganewtons per square meter (13 ksi), and at 732° C (1350° F) it was 338 meganewtons per square meter (49 ksi). The specimens tested included extruded material that had been canned under argon or under vacuum as well as powder that had been compacted directly by hot isostatic pressing. As mentioned earlier, the autoclave heat treatment and HIP of the loose powders were accomplished at the same time in single autoclave cycles.

In figure 1(a) (test condition - 982° C (1800° F) and 89.6 MN/m^2 (13 ksi)) it can be seen that, considering the scatter and the limited data, there does not appear to be any significant difference in life for the three types of autoclaved material: extruded argon canned, extruded vacuum canned, and HIP. Material autoclaved between 1293° and 1360° C (2360° and 2480° F) had stress rupture lives greater than the life of as-cast HS-31. The maximum lives were obtained with material autoclaved at 1327° C (2420° F).

Rupture ductility is similarly plotted in figure 1(b). The minimum in ductility occurred with the autoclaved material that gave the maximum in rupture life. However, the lowest ductility obtained was 5 percent, which is not considered to be prohibitively low. A comparison with cast material was not made, because no rupture ductility is reported in reference 8.

The maximum stress-rupture life in tests conducted at an intermediate temperature (732° C (1350° F) was also obtained with extruded material autoclaved at 1327° C (2420° F) as shown in figure 2(a). The HIP material exhibited maximum 732° C (1350° F) rupture life after autoclaving at 1293° C (2360° F). Because of possible uncertainty as to the life of the HIP material after it was autoclaved at 1327° C (2420° F), the area between the upper and lower curves in figure 2(a) is shaded. At the 1327° C (2420° F) autoclave temperature the life of argon canned extruded material was 157 hours, the life of vacuum canned extruded material was 173 hours, and the life of HIP material was 31 hours. The average rupture ductility (fig. 2(b)) for the 1327° C (2420° F) autoclaved material was about 10 percent, although rupture ductility generally decreased with increasing autoclave temperature.

The results of rupture tests at 982° C (1800° F) suggest that, for high temperature use, the extrusion step could be eliminated. It is evident that direct hot isostatic pressing can provide, in a single step, rupture properties equivalent to the properties obtained by a combination of extrusion and autoclave heat treatments. It does not appear that the extrusion step can be eliminated at an intermediate temperature, 732° C (1350° F), because the maximum rupture life achieved by HIP material was considerably less than that of the autoclaved extruded material.

At both high and intermediate temperatures it is evident that autoclave heat treatments of prealloyed powder HS-31 material can result in rupture lives substantially above the average lives of the cast alloy.

Comparison of stress-rupture properties over a range of test conditions. - In figure 3 and table IV, the rupture properties of as-extruded material, extruded material aged at 732° C (1350° F) for 50 hours, and extruded powder autoclaved at the optimum temperature of 1327° C (2420° F) are compared. The test temperatures extended from 649° to 982° C (1200° to 1800° F). The stresses applied at each temperature were those that would result in 10-hour lives for cast HS-31. Some of the extruded powder product specimens were from argon canned and some from vacuum canned material. Up to a test temperature of 732° C (1350° F), the powder product in the three conditions compared had a life advantage over cast HS-31 (see fig. 3(a)). For example, at 732° C (1350° F), the autoclaved extruded material has a fifteenfold life advantage. At 649° C (1200° F), lives were 20 to 30 times as great as those of the as-cast alloy. At 816° C (1500° F) and above, the lives of the as-extruded and extruded and aged materials were less than the life of the as-cast alloy. At 982° C (1800° F) their rupture lives were less than one-tenth that of the cast alloy. The life of the autoclaved material however remained greater than that of the cast alloy up to 982° C (1800° F), the highest test temperature considered. At the latter temperature, the autoclaved extruded powder product had a 30-hour life compared to 10 hours for the cast alloy. Aging at 732° C (1350° F) had a slightly deleterious effect on the life of the extruded material over almost the entire temperature range.

The rupture ductility (fig. 3(b)) of the as-extruded material and the material extruded and aged at 732° C (1350° F) was consistently greater (two to five times) than that of the autoclaved material. Elongations of the autoclaved extruded material ranged from 3 percent at 649° C (1200° F) to 13 percent at 732° and 816° C (1350° and 1500° F), and dropped to 6 percent at 982° C (1800° F).

Tensile Properties

The effect of autoclave temperature on the room temperature tensile properties of HS-31 powder metallurgy products is shown in figure 4. These tensile properties are also listed in table V. As the autoclave temperature increased from 1227° to 1361° C (2240° to 2480° F), ultimate tensile strength decreased from 1062 to 724 meganewtons per square meter (154 to 105 ksi). The latter value is the reported ultimate tensile strength for as-cast HS-31. Except for a specimen autoclaved at 1260° C (2300° F) which had a room temperature elongation of 12 percent, all of the autoclaved specimens had elongations less than the 10 percent reported for the as-cast alloy. For the 1327° C (2420° F) autoclave temperature, the elongation was $2\frac{1}{2}$ percent.

In figure 5 tensile properties of extruded powder metallurgy HS-31 (see table VI) are compared over a range of temperatures from room temperature to 1093° C (2000° F) with the properties of as-cast HS-31. HIP material was not included in this comparison because of a lack of material. The powder products are in the as-extruded, extruded and aged at 732° C (1350° F), and extruded and autoclaved at 1327° C (2420° F) conditions. Figure 5(a) shows that the tensile strength of the autoclaved material was slightly higher than that of as-cast HS-31 from room temperature to 982° C (1800° F). Aging at 732° C (1350° F) did not increase the tensile strength compared to that of the as-extruded material. The strengths of the as-extruded and the aged materials were very similar but were significantly greater than those of the as-cast material up to 760° C (1400° F). Above 871° C (1600° F) their strengths fell slightly below the strength of the as-cast alloy and of the autoclaved extruded material.

Figure 5(b) shows a similar plot of the tensile elongations. The autoclaved extruded powder product ductility ranged from $2\frac{1}{2}$ percent at room temperature to 32 percent at 1093° C (2000° F), and it was usually about half that of the as-cast alloy.

Microstructure

The microstructures of material made from prealloyed HS-31 powders are discussed in the next sections.

As-extruded. - Figure 6(a) shows the microstructure of as-extruded prealloyed powder HS-31 at ×100. At a higher magnification (not shown), chains of carbides were observed at the grain boundaries. The average grain diameter was 0.010 millimeter (approximately ASTM number 10).

Aged at 732° C (1350° F). - The microstructure of extruded powder material aged at 732° C (1350° F) for 50 hours is shown at ×100 in figure 6(b). This structure is very similar to the as-extruded structure, although aging caused the grain boundaries to be somewhat more sharply defined. The grain size measured was slightly smaller, 0.008 millimeter diameter (ASTM number 10.5), than that of the as-extruded structure. Generally, the mechanical properties of the aged material were slightly inferior to those of the as-extruded material.

Autoclaved extruded powder product. - Each photomicrograph in figure 7 shows the microstructure at ×100 of the extruded prealloyed powder product that had been autoclaved for 2 hours at 68.9 meganewtons per square meter (10 ksi) at one of several temperatures.

The material autoclaved at 1227° C (2240° F) had a duplex grain size (fig. 7(a)). The fine grained regions had an average grain diameter of 0.013 millimeter (approximately ASTM number 9.5), while the coarse grains were as large as 0.25 millimeter (ASTM number 1). Partial recrystallization had occurred, and no incipient melting was evident.

After autoclaving at 1260° C (2300° F) the material was completely recrystallized (fig. 7(b)). The largest grains were only slightly smaller than the largest grains in the material autoclaved at the lower temperature. However, the average grain diameter was 0.094 millimeter (approximately ASTM number 3.5), which is considerably smaller than the grain size of the recrystallized portion of the material autoclaved at 1227° C (2240° F). The finer grain size may have resulted from a greater number of competing nuclei at the higher temperature.

It is evident in the comparison of the autoclaved extruded material (fig. 7(b)) to the as-extruded material (fig. 6(a)) that recrystallization is complete and that a significant amount of grain growth has occurred (0.010 mm for an as-extruded material against 0.094 mm after the 1260° C (2300° F) treatment). This large amount of grain growth occurred below the solidus in the absence of any liquid phase. It is interesting to note, however, that this amount of grain growth did not significantly increase either the high temperature or the intermediate temperature stress-rupture life.

After autoclaving at 1293° C (2360° F) (fig. 7(c)), the grain size was 0.21 millimeter (ASTM number 1.5), which was significantly larger. The grains were surrounded by a continuous carbide network. Patches of eutectic at triple points marked regions where considerable melting had occurred. Despite the larger number of grains which probably nucleated at the higher temperature, grain growth resulted in a larger final grain size. The interior of the grains was dotted with small angular regions where melting may also have occurred in the vicinity of carbides. A few small voids were evident within the grains as well as at grain boundaries.

After autoclaving at 1327° C (2420° F) (fig. 7(d)) similar but somewhat larger features were observed. The average grain size was 0.187 millimeter (approximately ASTM number 2.0).

At 1361° C (2480° F) (fig. 7(e)), the grain boundary carbides were substantially thicker and a cellular precipitate developed. The average grain diameter was 0.180 millimeter (ASTM number 2.0). A similar structure and grain size were observed after autoclaving at 1393° C (2540° F) (not shown herein).

Figure 8 is shown as an example of the structure of the hot isostatically pressed material. This powder was pressed at 1327° C (2420° F). It appeared to be very similar to the extruded and autoclaved material except that it did not exhibit the features within the grains shown by the extruded material, and it had a finer grain size, 0.151 millimeter in diameter (approximately ASTM number 2.5), than extruded material autoclaved at the same temperature.

SUMMARY OF RESULTS

Argon atomized prealloyed powder of the commonly used cast cobalt-base alloy HS-31 was consolidated by two methods. Evacuated cans of powder were extruded into bars at 1204° C (2200° F) and at an extrusion ratio of 11.1 to 1. Evacuated tubes of powder were isostatically hot pressed at temperatures (1227° to 1393° C (2240° to 2540° F)) ranging from below to above the solidus, approximately 1280° C (2340° F). A portion of the extruded material was also heated in this temperature range in an autoclave at 68.9 meganewtons per square meter (10 ksi) for 2 hours. Another portion of the extruded material was aged at 732° C (1350° F) for 50 hours. The following major results of an evaluation of this material were obtained:

1. Compaction of canned HS-31 prealloyed powder by direct hot isostatic pressing in an autoclave, a process that combined compaction and heat treating in a single step, was about as effective as compaction by extrusion followed by an autoclave heat treatment in achieving good high temperature (982° C (1800° F)) stress-rupture properties.

2. The high and intermediate temperature stress-rupture properties of the material extruded and autoclaved as well as of powders compacted directly in an autoclave were maximum when the autoclave temperature was in the 1293° to 1327° C (2360° to 2420° F) range.

3. The intermediate autoclave temperature 1327° C (2420° F) resulted in maximum stress-rupture life at both an intermediate temperature of 732° C (1350° F) and 338 meganewtons per square meter (49 ksi) and at a high temperature of 982° C (1800° F) and 89.6 meganewtons per square meter (13 ksi) for the autoclaved extruded material. Stress-rupture life of the extruded prealloyed powder material autoclaved at 1327° C (2420° F) was greater than the life of cast HS-31 over the 649° to 982° C (1200° to 1800° F) temperature range. The room temperature tensile strength of the autoclaved extruded material decreased as the autoclave temperature was increased from 1227° to 1361° C (2240° to 2480° F).

4. As-extruded powder material and extruded material given a conventional aging heat treatment at 732° C (1350° F) had stress-rupture lives greater than those of the as-cast alloy at 649° and 732° C (1200° and 1350° F). Above these temperatures the as-cast alloy had substantially greater lives.

5. The extruded HS-31 powder products autoclaved at 1327° C (2420° F) had tensile strengths that were slightly above those of as-cast HS-31 from room temperature to 1093° C (2000° F). Both the as-extruded material and extruded and conventionally aged material (aged at 732° C (1350° F)) had tensile strengths significantly greater than did the cast material from room temperature up to 760° C (1400° F).

6. The tensile elongations of all powder products except the as-extruded product increased from room temperature to 1093° C (2000° F).

7. No significant differences were observed in the properties of extruded material canned under vacuum or under sufficient argon pressure to sustain an arc. The as-extruded product tensile elongations varied from 18 percent at room temperature to a high of 33 percent at 816° C (1500° F) and then down to 20 percent at 1093° C (2000° F); its elongations exceeded those of the as-cast alloy to at least 871° C (1600° F). The heat-treated powder products had lower elongations than the cast alloy up to 982° C (1800° F).

8. There was evidence of melting at the grain boundaries in material autoclaved at 1293° C (2360° F) and above. This suggests the possibility that the solidification structure as well as the larger grain size resulting from the autoclave heat treatment contributed to the improved high temperature strength.

CONCLUDING REMARKS

An examination of the microstructure of the powder products showed that appreciable grain growth in the autoclaved extruded material occurred at the autoclave temperatures of 1227° and 1260° C (2240° and 2300° F). Both temperatures are below the solidus temperature, which is approximately 1280° C (2340° F). The increase in observed life as the autoclave temperature was raised from 1227° to 1327° C (2240° to 2420° F) may be related to the increased amount of "cast structure" at the grain boundaries (figs. 7(c) and (d)) as well as to the increased grain size. The decrease in rupture life as autoclave temperatures were increased above 1327° C (2420° F) may have been related to the presence of the cellular precipitate noted in figure 7(e).

High temperature strength in prealloyed powder HS-31, which is equivalent or superior to the strength of as-cast HS-31, has been achieved by heat treating above the solidus temperature while simultaneously applying pressure in an autoclave. By appropriate adjustment of the autoclave heat treating parameters, it is possible to obtain processing conditions for the prealloyed powder product HS-31 that provide rupture lives considerably in excess of those available for the cast product over a temperature range from 650° to 982° C (1200° to 1800° F).

The improved strength of the autoclaved extruded powder product compared to that of the as-extruded product was achieved at some expense in ductility and possibly in fracture toughness. The carbide film at the grain boundaries is, in all probability, the cause of the lower ductility observed in the autoclaved material.

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Cleveland, Ohio, October 11, 1972,
501-21.

REFERENCES

1. Fischer, George W., et al.: Complex Shapes by Slip Casting. Rep. R65-FPD-185, General Electric Co. (AFML-TR-65-257, AD-469379), Aug. 1965.
2. Lyon, Dean L., et al.: Continuous Slip Casting Process for the Production of Metal Strip and Sheet. Rep. E-175 (IV), Amplex Div., Chrysler Corp. (AFML-TR-67-387, AD-831843), Jan. 1968.
3. Parikh, N. M.; Farrell, K.; and Spachner, S. A.: Improved Production of Powder Metallurgy Items. Rep. IITRI-B247-25, IIT Research Inst. (AFML-TR-65-103, AD-464368), Mar. 1965.
4. Friedman, Gerald I.; and Loewenstein, Paul: Processing Techniques for the Extrusion of Superalloy Powders. Rep. NMI-9709.19, Whittaker Corp. (AFML-TR-68-321, AD-845185), Oct. 1968.
5. 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.
6. 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.
7. Freche, John C.; Ashbrook, Richard L.; and Waters, William J.: Application of Powder Metallurgy to an Advanced-Temperature Nickel-Base Alloy, NASA-TRW VI-A. NASA TN D-6560, 1971.
8. Anon.: Haynes Alloy No. 31. Stellite Div., Cabot Corp., 1970.

TABLE I. - TYPICAL PARTICLE SIZE

DISTRIBUTION OF HS-31

ATOMIZED POWDERS

Tyler screen size	Particle size distribution, percent
>30	0
30/60	.10
60/100	16.20
100/150	13.75
150/200	15.55
200/270	14.10
270/325	8.50
<325	31.30

TABLE II. - TYPICAL CHEMICAL ANALYSIS

OF HS-31 POWDER

Element	Weight, percent	
	Nominal composition	Powder[a]
Chromium	24.5 to 26.5	26.15
Nickel	9.5 to 11.5	10.65
Tungsten	7.0 to 8.0	7.45
Manganese	1.00 max.	.68
Iron	2.00 max.	.74
Silicon	1.00 max.	.58
Carbon	0.45 to 0.55	.52
Sulfur	0.040 max.	.009
Phosphorous	0.040 max.	.001
Molybdenum	----------	<.01
Oxygen	----------	.049
Boron	----------	.009
Nitrogen	----------	.08
Cobalt	Balance	Balance

[a]Analysis by supplier.

TABLE III. - STRESS-RUPTURE PROPERTIES OF PREALLOYED POWDER HS-31

AUTOCLAVED AT VARIOUS TEMPERATURES AT 68.9 MN/m^2 (10 ksi)

Compaction procedure	Autoclave temperature		Life, hr	Elongation, percent	Reduction in area, percent
	°C	°F			
Tested at 982° C and 89.6 MN/m^2 (1800° F and 13 ksi)					
Extruded, A[a]	1393	2540	9.1	15.7	----
HIP	1393	2540	3.8	19.0	----
Extruded, A	1361	2480	18.0	9.4	----
HIP	1327	2420	44.0	8.0	6.2
Extruded, A	↓	↓	29.8	5.6	2.7
Extruded, A	↓	↓	24.7	BOG[b]	----
Extruded, A	↓	↓	30.3	5.0	2.0
Extruded, V[c]	↓	↓	41.9	8.5	----
Extruded, A	↓	↓	39.6	6.3	5.0
HIP	↓	↓	22.4	8.5	2.0
HIP	↓	↓	16.9	6.3	6.3
Extruded, V	1293	2360	33.8	4.7	2.1
Extruded, A	1293	2360	25.5	----	----
Extruded, V	1260	2300	1.7	6.0	8.7
Extruded, V	↓	↓	.7	6.0	7.9
HIP	↓	↓	1.3	15.0	10.0
Extruded, A	1227	2240	.2	22.8	15.7
HIP	1227	2240	.4	10.6	10.0
Tested at 732° C and 338 MN/m^2 (1350° F and 49 ksi)					
Extruded, A	1393	2540	9.7	6.4	6.3
HIP	1393	2540	12.4	8.8	6.3
Extruded, A	1327	2420	157.1	12.8	7.9
Extruded, V	1327	2420	172.4	11.2	9.5
HIP	1327	2420	30.7	6.4	4.8
Extruded, V	1293	2360	62.1	4.8	2.3
HIP	1293	2360	67.3	6.4	----
Extruded, V	1260	2300	30.3	20.8	16.7
HIP	1260	2300	26.7	19.5	14.6
Extruded, V	1227	2240	10.8	30.4	27.2
HIP	1227	2240	7.8	19.6	16.6

[a]Canned under 1/5 atmosphere of argon.

[b]Broke outside gage.

[c]Canned under vacuum.

TABLE IV. - STRESS-RUPTURE PROPERTIES OF EXTRUDED PREALLOYED POWDER HS-31 TESTED AT THE 10-HOUR STRESS-RUPTURE LIFE CONDITIONS FOR CAST HS-31 (REF. 8)

Test conditions				Life, hr	Elongation, percent	Reduction in area, percent
Temperature		Stress				
^{o}C	^{o}F	MN/m^2	ksi			
As-extruded						
982	1800	89.6	13.0	0.5	----	----
982	1800	89.6	13.0	.9	20.0	----
899	1650	155.2	22.5	1.6	28.0	20.2
816	1500	227.5	33.0	6.7	31.0	24.2
732	1350	337.8	49.0	24.1	26.2	23.0
649	1200	420.6	61.0	341.9	21.0	----
Extruded and aged at 732° C (1350° F) for 50 hours						
982	1800	89.6	13.0	0.8	18.0	----
899	1650	155.2	22.5	1.0	24.8	15.8
816	1500	227.5	33.0	3.1	26.4	23.0
732	1350	337.8	49.0	19.1	32.0	27.8
649	1200	420.6	61.0	206.4	25.6	21.5
649	1200	420.6	61.0	209.1	25.0	----
Extruded and autoclaved at 1327° C (2420° F)						
982	1800	89.6	13.0	29.8	5.6	2.7
↓	↓	↓	↓	24.7	----	----
				30.3	5.0	2.0
				41.9	8.5	----
				39.6	6.3	5.0
899	1650	155.2	22.5	16.1	9.6	7.9
816	1500	227.5	33.0	52.3	12.8	11.1
732	1350	337.8	49.0	157.1	12.8	7.9
649	1200	420.6	61.0	367.9	3.2	2.4

TABLE V. - ROOM TEMPERATURE TENSILE PROPERTIES OF PREALLOYED POWDER HS-31

AUTOCLAVED AT VARIOUS TEMPERATURES

Compaction procedure	Autoclave temperature		Ultimate tensile strength		0.2-Percent yield strength		Elongation, percent	Reduction in area, percent
	°C	°F	MN/m^2	ksi	MN/m^2	ksi		
Extruded, A[a]	1361	2480	724	105	498.5	72.3	6.0	5.5
Extruded, V[b]	1327	2420	779	113	583.3	84.6	2.5	3.0
Extruded, V	1293	2360	765	111	659.8	95.7	.8	.1
Extruded, A	1293	2360	772	112	621.9	90.2	1.5	1.2
HIP	1293	2360	696	101	534.4	77.5	5.8	6.0
Extruded, V	1260	2300	1007	146	553.0	80.2	12.0	11.2
Extruded, V	1227	2240	1062	154	551.6	80.0	7.0	6.8

[a]Canned under 1/5 atmosphere of argon.
[b]Canned under vacuum.

TABLE VI. - TENSILE PROPERTIES OF EXTRUDED HS-31

PREALLOYED POWDER PRODUCTS

[See ref. 6 for as-extruded properties, ref. 8 for as-cast properties.]

Condition	Test temperature		Ultimate tensile strength		0.2-Percent yield strength		Elongation, percent	Reduction in area, percent
	°C	°F	MN/m^2	ksi	MN/m^2	ksi		
Aged at 732° C (1350° F) for 50 hr	RT[a]	RT	1213.0	176.0	730.8	106.0	5.5	7.5
	649	1200	910.0	132.0	471.6	68.4	10.5	11.3
	871	1600	324.0	47.0	-----	-----	----	----
	982	1800	164.0	23.8	-----	-----	19.0	----
	1093	2000	61.4	8.9	39.3	5.7	56.0	37.8
Autoclaved at 1327° C (2420° F)	RT	RT	779.0	113.0	583.3	84.6	2.5	3.0
	649	1200	587.5	85.2	317.2	46.0	8.0	8.5
	871	1600	366.0	53.1	293.1	42.5	8.0	10.7
	982	1800	235.8	34.2	194.5	28.2	16.0	18.0
	982	1800	210.9	30.6	-----	-----	19.5	21.0
	1093	2000	136.5	19.8	97.9	14.2	32.0	29.2

[a]Room temperature.

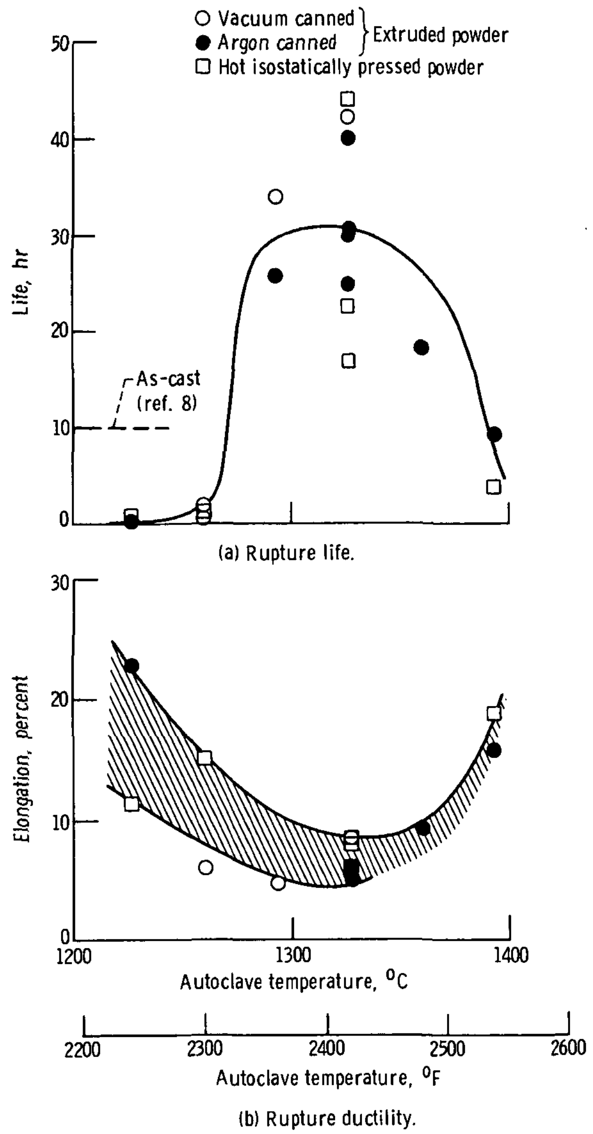


Figure 1. - Effect of autoclave heat treatment on stress-rupture properties of HS-31 powder metallurgy material at temperature of 982° C (1800° F) and stress of 89.6 meganewtons per square meter (13 ksi).

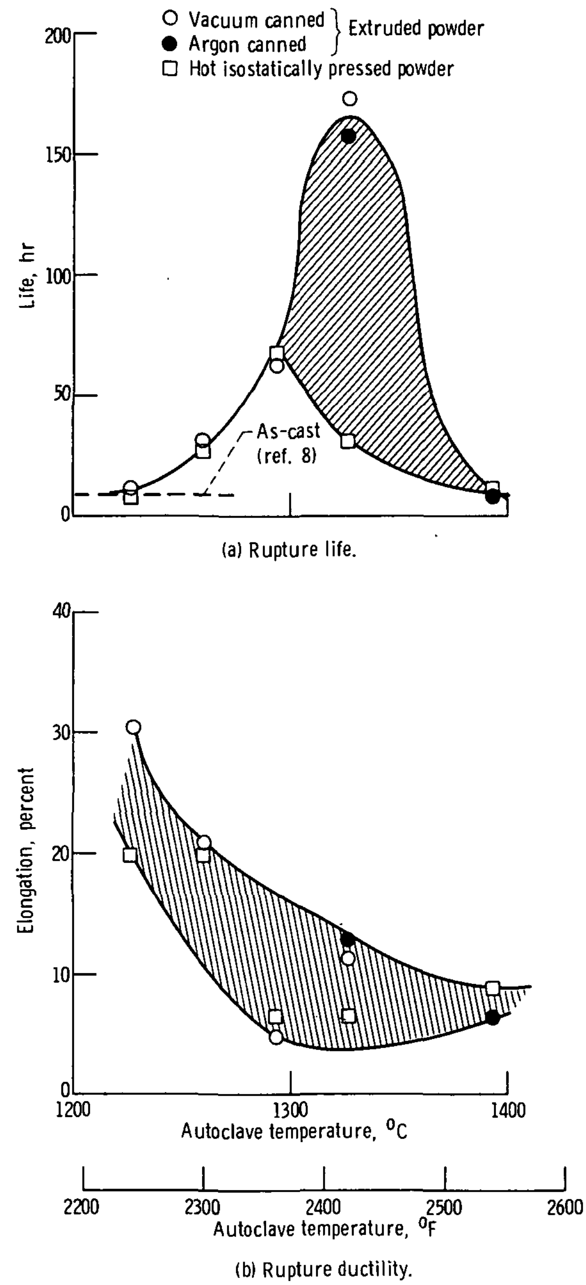


Figure 2. - Effect of autoclave heat treatment temperature on stress-rupture properties of HS-31 powder metallurgy material at temperature of 732° C (1350° F) and stress of 338 meganewtons per square meter (49 ksi).

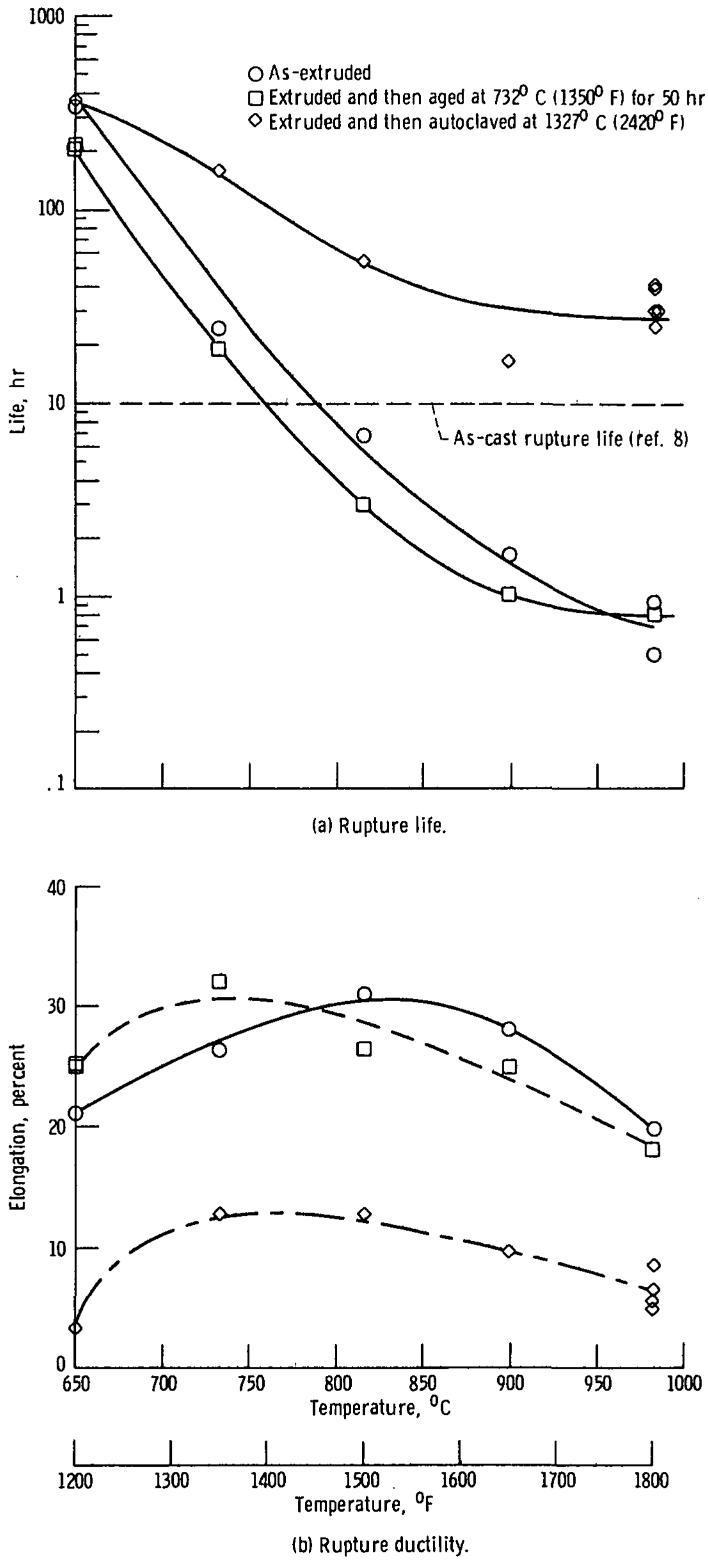


Figure 3. - Stress-rupture properties of prealloyed powder HS-31 at 10-hour life conditions for cast HS-31 (ref. 8).

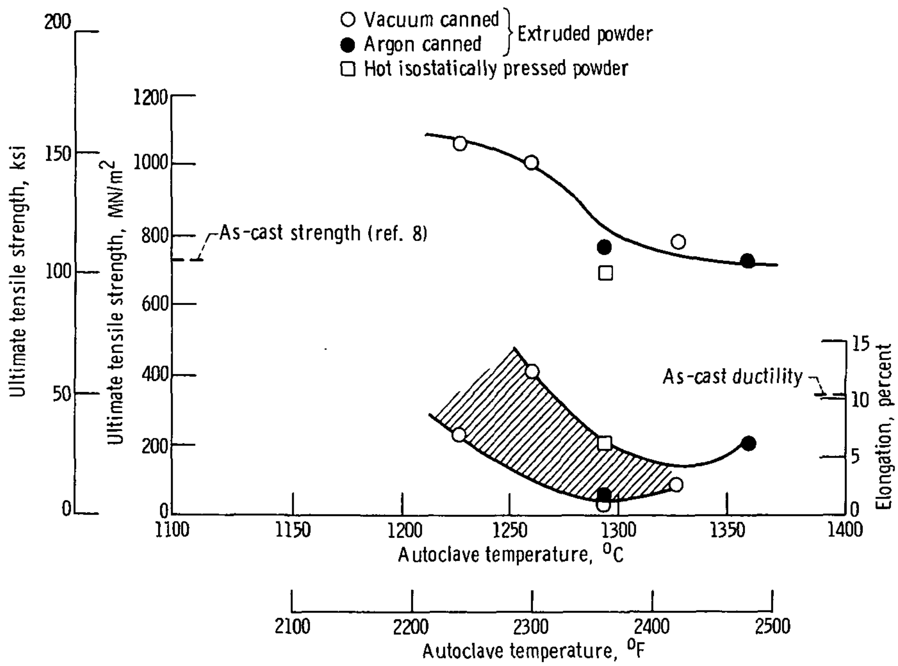
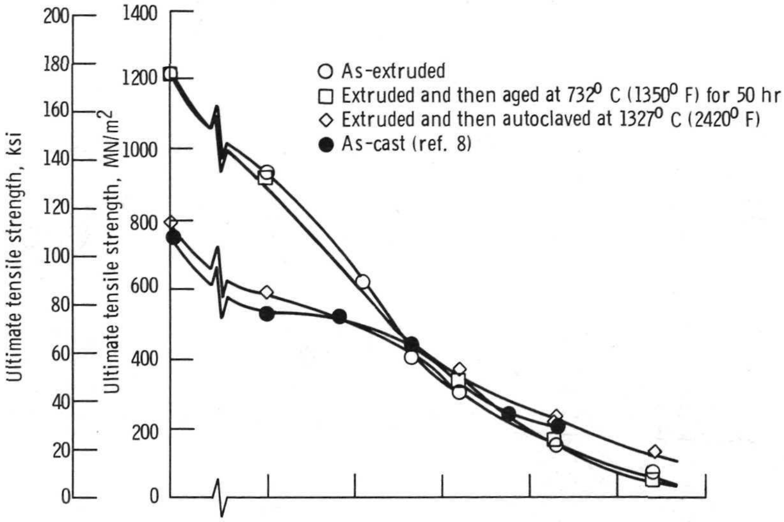
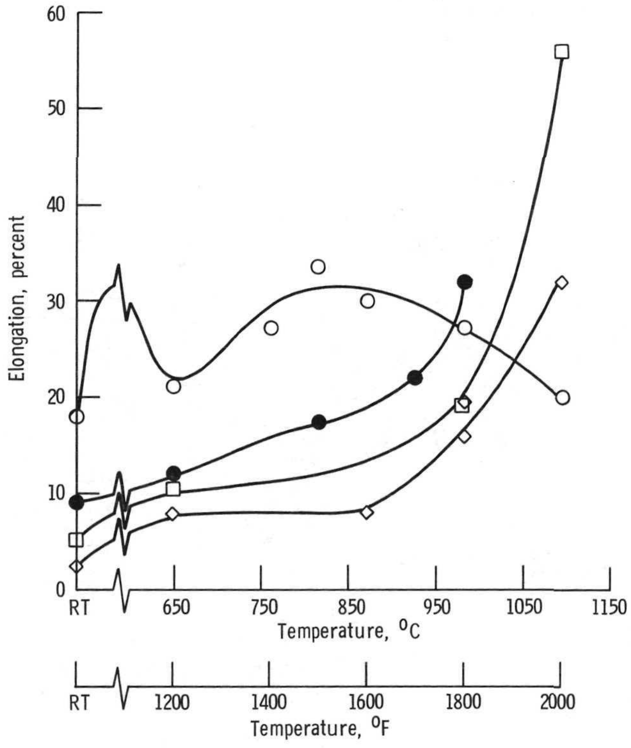


Figure 4. - Effect of autoclave heat treatment temperature on room temperature tensile properties of powder metallurgy HS-31.

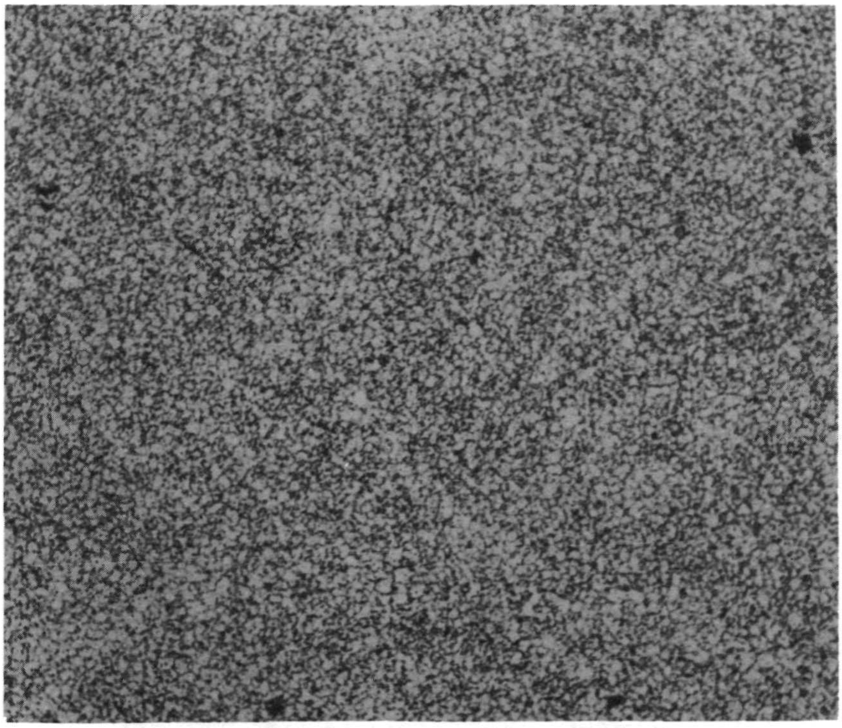


(a) Ultimate tensile strength.

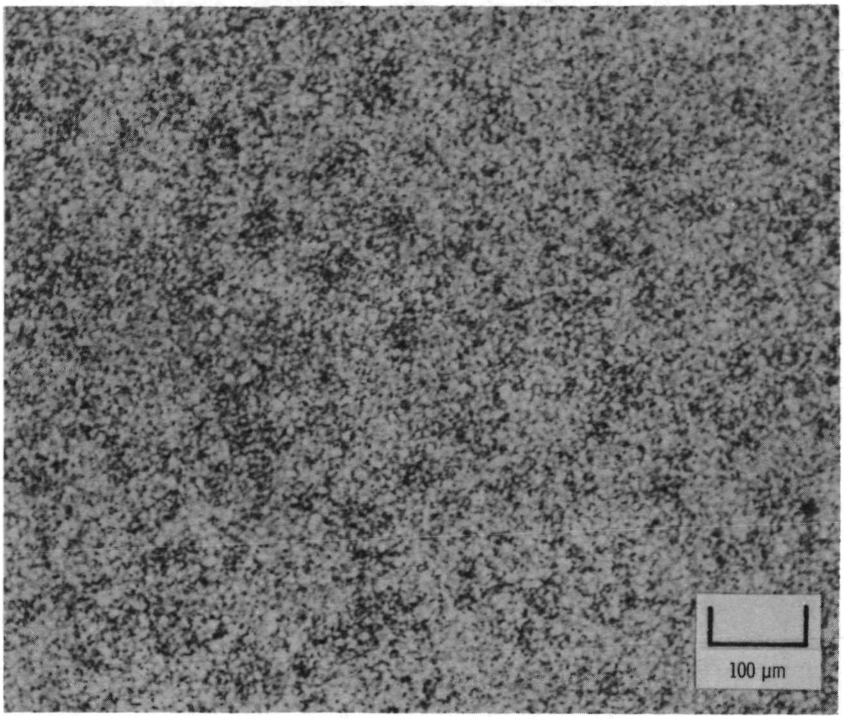


(b) Ductility.

Figure 5. - Effect of test temperature on tensile properties of extruded powder metallurgy HS-31 in several conditions.

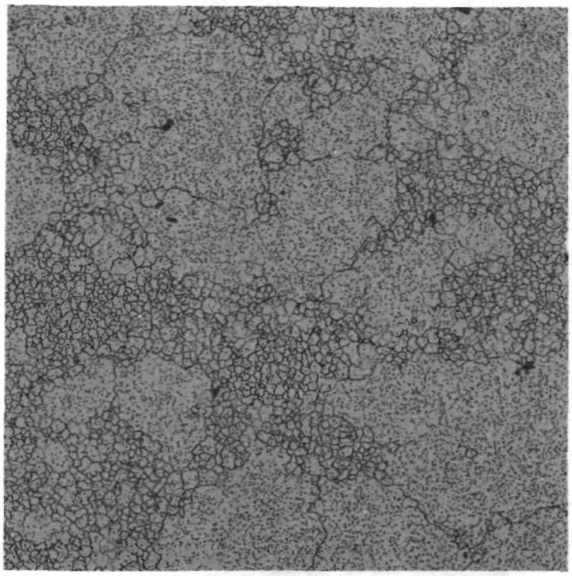


(a) As-extruded.

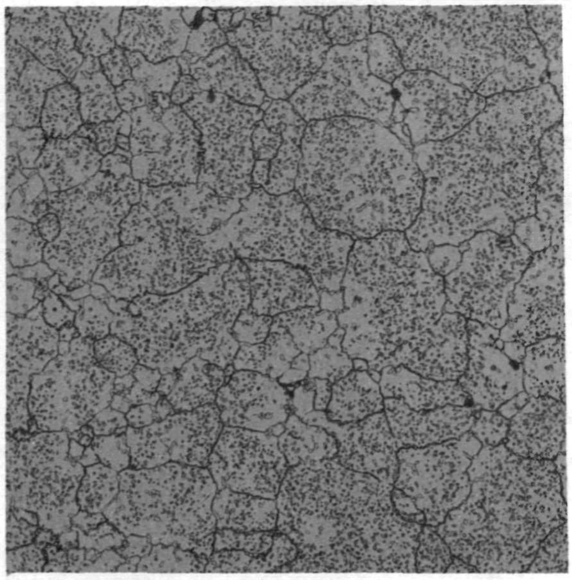


(b) Extruded and aged at 732° C (1350° F) for 50 hours.

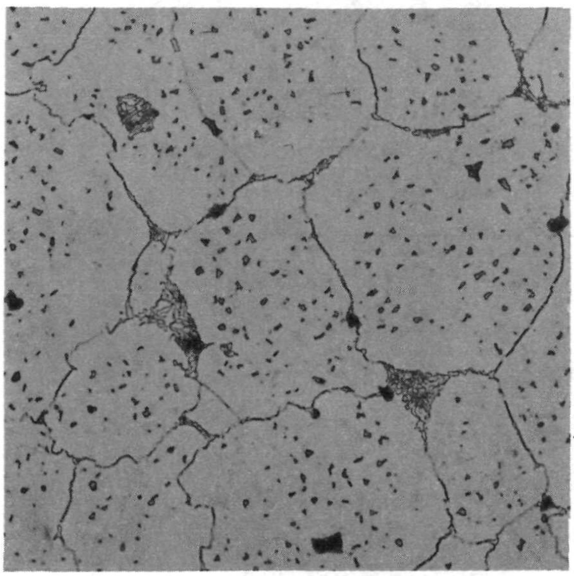
Figure 6. - Microstructure of extruded prealloyed powder HS-31. X100.



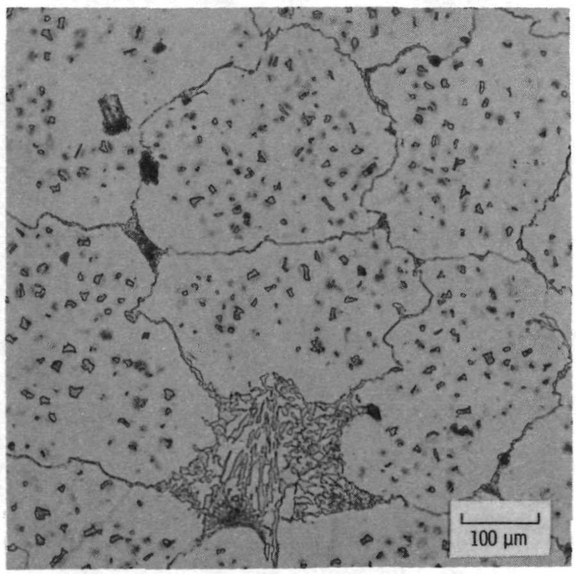
(a) Temperature, 1227° C (2240° F).



(b) Temperature, 1260° C (2300° F).

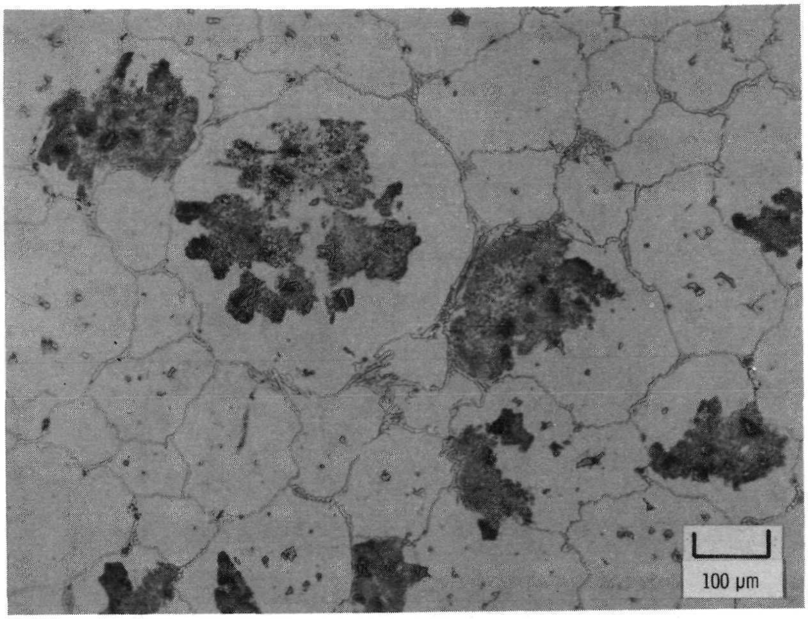


(c) Temperature, 1293° C (2360° F).



(d) Temperature, 1327° C (2420° F).

Figure 7. - Effect of autoclave temperature on extruded HS-31 powder product. Autoclave pressure, 68.9 meganewtons per square meter (10 ksi); time, 2 hours. X100.



(e) Temperature, 1360° C (2480° F).

Figure 7. - Concluded.

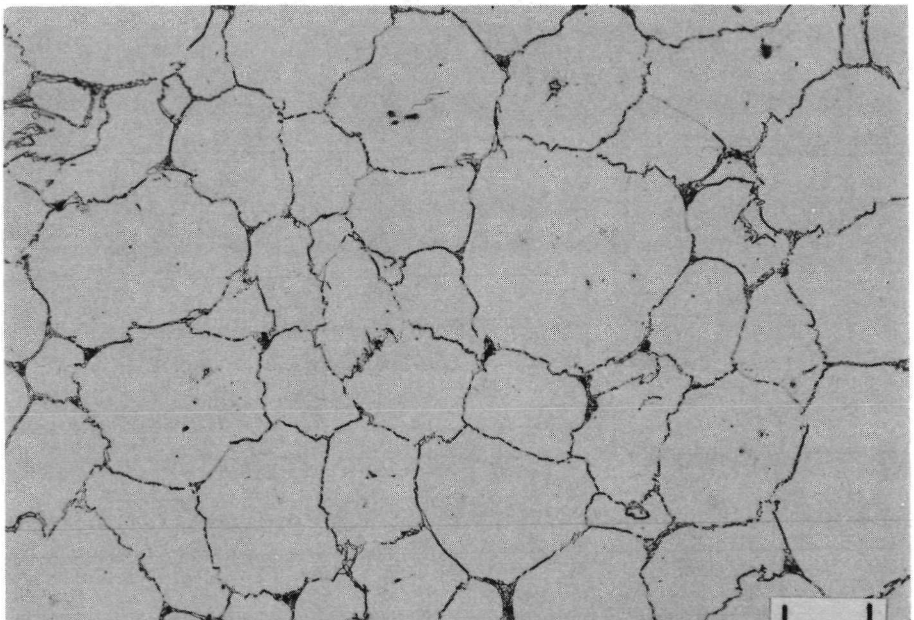


Figure 8. - HS-31 powder isostatically pressed at 1327° C (2420° F) for 2 hours at 68.9 meganewtons per square meter (10 ksi). X100.

NASA-Langley, 1973 — 17 E-7107

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