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EVALUATION OF A COBALT-BASE ALLOY, HS-31, MADE BY EXTRUSION OF PREALLOYED POWDERS

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16. Abstract

Bars extruded from prealloyed powders of HS-31 made by argon gas atomization were evaluated by tensile and stress-rupture tests in the as-extruded and heat-treated conditions. Significant improvements in tensile strength over the as-cast condition were obtained with the as-extruded powder product up to 1400° F (760° C), but lower strengths were observed in the 1500° to 1800° F (816° to 982° C) temperature range. Heat treatments involving temperatures above the solidus and 30 000-psi (207-MN/m^2) pressure substantially increased the stress-rupture life of the extruded powder product over that of cast HS-31 at intermediate temperature (1200° F, 649° C) and resulted in comparable life at high temperature (1800° F, 982° C).

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SUMMARY

Prealloyed powders of a cobalt-base alloy, HS-31, made by argon gas atomization and extruded into bar stock were evaluated by tensile and stress-rupture tests. Metallographic studies were made to relate properties to microstructure.

Tensile strength of the as-extruded powder product was almost double that of the cast alloy at temperatures to 1200° F (649° C), but only about two-thirds that of the cast alloy at 1800° F (982° C). Stress-rupture life of the as-extruded powder product at 1200° F (649° C) and 61 000 psi (420 MN/m^2) was 340 hours, compared to 10 hours for the cast alloy; at 1800° F (982° C) and 13 000 psi (90 MN/m^2) it was less than 1 hour, compared to 10 hours for the cast alloy.

Conventional heat treatments failed to achieve good high temperature (1800° F, 982° C) stress-rupture life. However, by exceeding the incipient melting point it was possible to increase the grain size of the extruded powder product. Subsequent simultaneous application of high pressure (30 000 psi, 207 MN/m^2) and high temperature (2200° F, 1205° C) in an autoclave closed the voids formed during exposure to temperatures above the solidus and restored structural integrity. Grain size was increased approximately from ASTM micrograin size number 10 to number 3.5. The prealloyed powder product heat treated in this way substantially exceeded the average stress-rupture life of the cast alloy at intermediate temperature (1200° F, 649° C) and resulted in comparable life at high temperature (1800° F, 982° C). The solidification structure resulting from partial melting at the grain boundaries, as well as the coarser grain size, may have contributed to the improved stress-rupture life.

INTRODUCTION

Prealloyed powder techniques afford a means of overcoming the segregation and forming problems inherent in conventional casting and hot-working operations of superalloys which are used in the hot components of gas turbine engines. These techniques also can substantially improve superalloy strength and ductility. Atomizing the molten alloy with an inert-gas jet subjects each metal droplet to rapid solidification rates. This, in turn, results in a substantially homogeneous powder and a structure free from macrosegregation upon subsequent powder consolidation. Oxygen content of the powders should be kept to a low level to prevent the formation of tightly adherent oxide films on the powders, and this can be accomplished by use of inert gas in the atomization process. 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 5).

One approach involves the slip casting of prealloyed powders followed by sintering (refs. 1 and 2). Of the materials considered in the latter investigations, some of the most promising results were obtained with the cobalt-base alloy HS-31. For example, tensile properties essentially equivalent to those of the cast counterpart of the alloy were obtained over the entire temperature range in both these investigations (room temperature to 1900° F (1038° C) in ref. 1 and room temperature to 1800° F (982° C) in ref. 2). By the addition of small quantities of ZrB_2, however, the average room temperature tensile strength of the slip-cast, sintered product was increased by approximately one-fifth, and the 1300° F (705° C) tensile strength was increased by approximately one-half compared to the cast alloy (ref. 1). The high temperature (1800° F, 982° C) stress-rupture properties exceeded those of the cast alloy in the investigation of reference 1, but both the 1400° and 1800° F (760° and 982° C) stress-rupture properties were substantially lower than those of the cast alloy in the investigation of reference 2.

Another 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 5). We have shown (ref. 5) the tensile strengths of the as-extruded prealloyed powder products of two normally cast nickel-base alloys, Alloy 713C and TAZ-8A, to be almost twice those of their cast counterparts to 1200° F (649° C). In the 1800° F (982° C) range, however, the as-extruded powder products of both alloys had significantly lower tensile and stress-rupture strengths than the cast alloys, and superplastic behavior was observed with TAZ-8A. It was also shown, however, that it is possible to take advantage of such superplasticity to deform the material to a desired shape by hot pressing under relatively low applied pressure. Attempts to achieve high temperature strength comparable to that of cast alloys by conventional heat treatments were unsuccessful. However, the application of high pressure together with temperatures above the minor-phase melting point of the as-extruded TAZ-8A prealloyed powder product resulted in a coarser-grained product without voids. This suggested that a combination of autoclave and high temperature heat treatments might make it possible to achieve microstructures with extruded prealloyed powders that would have good elevated temperature (1800° F or 982° C, and above) properties as well. If suitable high temperature strengths could be achieved in this way, the technique of using prealloyed powders that involves compaction, forming to a desired shape, and heat treatment could result in powder products with substantial strength increases over the entire temperature range compared to conventional cast or wrought products.

The present investigation was intended to evaluate the commonly used cobalt-base alloy HS-31 made by extrusion of prealloyed powders obtained from inert-gas atomization. The effects of various heat treatments on mechanical properties and microstructure were determined. Tensile and stress-rupture tests were made over a range of temperatures with as-extruded and heat-treated materials. The effect of different heat treatments, including autoclave heat treatments, was evaluated by stress-rupture tests.

MATERIALS AND PROCEDURE

Materials

Extruded bars of a cobalt-base alloy, Haynes Stellite-31 (X-40), were obtained from a manufacturer of high temperature alloys. To make the bars, air-melted HS-31 was remelted under argon and then atomized with a high pressure stream of argon. The bars were extruded at 2200° F (1205° C) directly from -30 mesh powders canned in mild steel. An extrusion ratio of 11:1 produced bars having a decanned diameter of about 9/16 inch (1.43 cm).

A screen analysis of the -30 mesh powder used is given in table I. The photomicrographs of figure 1 show the generally spheroidal shape of the powder particles. Some voids are evident, and the powder particles exhibit a dendritic structure.

Table II shows the nominal chemical composition for HS-31 and analyses of the -30 mesh powders as well as of the extruded powder product. The analysis of the loose powder was made by the supplier, and that of the extruded powder product was made by an independent laboratory. The oxygen content reported for the as-received powder was 110 ppm and that for the extruded bar stock was 244 ppm. Essentially no increase in oxygen content after extrusion was observed for Alloy 713C and TAZ-8A prealloyed powder products (ref. 5). Oxygen content after extrusion in this early investigation was less than 200 ppm.

The extruded bars were inspected by radiographic techniques using an iridium source. After surface grinding they were also inspected with a fluorescent dye penetrant. These inspections showed the bars to be generally sound, although a longitudinal microsection of an as-extruded bar at a magnification of ×100 showed elongated voids and inclusions but equiaxed grains (see fig. 2). The measured density of 0.312 pound per cubic inch (8.65 g/cm^3) was essentially the same as that reported for the cast alloy in reference 6, 0.311 pound per cubic inch (8.61 g/cm^3).

Heat Treatments

The heat treatments applied to the as-extruded powder product are listed in table III, as well as the grain size determined for each condition. All heat treatments were conducted in an inert atmosphere. Most of these were intended to achieve a large grain size in order that high-temperature properties equivalent to the cast alloy might be obtained. To determine a temperature appropriate for promoting grain growth, a bar of extruded HS-31 powder was heated in argon for 1 hour in a thermal-gradient furnace. The temperature from one end of the bar to the other ranged from about 2030° to 2550° F (1110° to 1399° C). The hotter end of the bar is shown in figure 3. Temperatures above the solidus (~2340° F (1282° C)) produced substantial grain growth as well as voids and swelling of the bar. This is discussed in detail in the section Microstructure. Metallographic examination of the thermal-gradient bar after exposure led to the selection of step 1 of autoclave treatment A (table III), which was applied to several extruded bars. Microstructural examination of portions of the thermal-gradient bar which were at 2400° and 2450° F (1316° and 1342° C) showed no significant grain size difference. Therefore, both temperatures were considered to be acceptable for step 1 of autoclave heat treatment A. In order to obtain additional material for mechanical testing, more bars of extruded HS-31 powder were autoclave heat treated. Due to a furnace breakdown, the heat-treat cycle in the autoclave for these bars became that shown in table III as autoclave heat treatment B. It should be noted that the first step of autoclave heat treatment A involved an air cool; whereas, the first step of autoclave heat treatment B involved a furnace cool.

These autoclave heat treatments were performed in a cold-wall autoclave typical of those used for gas-pressure bonding (ref. 7). The specimens were placed in the hot zone of a furnace which was heavily insulated from a water-cooled pressure vessel. The furnace was heated electrically. High pressures were achieved by pumping helium into the pressure vessel. In this way, the charge was isostatically hot pressed.

An intermediate temperature heat treatment, 1350° F (732° C) for 50 hours, has been reported to increase low and intermediate temperature strength of cast HS-31 without adversely affecting strength at high temperatures (ref. 6). The same heat treatment (nonautoclave heat treatment C, table III) was applied to as-extruded material as well as to extruded material which had first been solution treated at 2240° F (1226° C) for 1/2 hour (nonautoclave heat treatment D, table III). Because of the limited quantity of material, only previously aged specimens were available for solution treating at

2240° F (1226° C). This solutioning temperature was chosen as being safely below the solidus.

Mechanical Testing

Tensile and stress-rupture tests were made in air. Tensile tests were conducted at temperatures to 2100° F (1149° C) and stress-rupture tests to 1800° F (982° C) with as-extruded and heat-treated material. The material and test conditions, as well as the results, are listed in tables IV and V. Generally, only single tests were run at a particular test condition due to the limited amount of extruded powder product available. The specimens had cylindrical gage sections 0. 250 inch (0. 64 cm) in diameter and 1. 25 inches (3. 18 cm) long with conical shoulders having a 20° included angle. 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. The specimens of consolidated powders were etched electrolytically in a 5 percent aqua regia (3 parts hydrochoric acid, 1 part nitric acid) water solution. The loose powders were etched by swabbing with a solution of 92 parts hydrochloric acid, 3 parts nitric acid, and 5 parts sulfuric acid.

Working

Swaging. - To impart cold work a limited amount of as-extruded material was cold swaged. The swaged material which was tested was subjected to a rounding rather than a reducing operation. For example, a typical, as-extruded bar cross section was elliptical with major and minor diameters of 0. 547 and 0. 567 inch (1. 49 and 1. 44 cm). Passing such bars through a 0. 565-inch (1. 43-cm) diameter die resulted in a final diameter of 0. 575 (1. 46 cm) and did not cause cracking. A second pass through a 0. 535-inch (1. 36-cm) diameter die reduced the diameter to 0. 547 inch (1. 39 cm) and cracked the bar. This material was not tested. It is believed that by hot swaging, or by cold swaging with intermediate annealing, more reduction could have been obtained.

Hot pressing. - To determine formability, sections of bars of the prealloyed powder product were hot pressed in a hydraulically operated press with a graphite-susceptor induction furnace. A section of extruded bar 0. 535 inch (1. 36 cm) high and about 9/16 inch (1. 43 cm) in diameter was isothermally hot pressed at 2000° F (1094° C) using a maximum stress of 6500 psi (45 MN/m^2). The initial deformation rate was 0.015 inch per minute (0.04 cm/min); the final rate was 0.005 inch per minute (0.01 cm/min). A portion of the head of a tested stress-rupture bar which had first been subjected to grain coarsening in autoclave heat treatment A was also hot pressed. The same temperature and deformation rates were used.

RESULTS AND DISCUSSION

The mechanical properties are graphically presented in figures 4 to 6 and are discussed in relation to the microstructures shown in figures 7 to 11.

Tensile Properties

The tensile properties of HS-31 are shown in figure 4, and a compilation of all tensile data for the HS-31 prealloyed powder product is provided in table IV. The extruded powder product had nearly double the tensile strength of the as-cast alloy at room temperature and 1200° F (649° C). For example, ultimate tensile strengths at these temperatures were 175 500 and 134 500 psi (1210 and 927 MN/m^2) for the extruded powder product and 108 000 and 75 000 psi (744 and 517 MN/m^2) for the as-cast alloy. At 1400° F (760° C) the difference in strength was considerably less, 88 200 psi (608 MN/m^2) for the powder product against 72 500 psi (500 MN/m^2) for the cast alloy. At 1800° F (982° C), however, the cast alloy was stronger, 30 000 psi (206 MN/m^2) against 21 700 psi (150 MN/m^2).

Swaging appears to offer additional means of improving the room temperature and intermediate temperature tensile strength. An increase of about 8500 psi (58 MN/m^2) above the tensile strength of the extruded powder product was observed at room temperature and at 1200° F (649° C).

A simple 50-hour age at 1350° F (732° C), heat treatment C, reduced the room temperature tensile strength of the as-extruded powder product by about 10 000 psi (69 MN/m^2) and the 1400° F (760° C) tensile strength by about 6000 psi (41 MN/m^2). This effect is opposite to that observed when the same heat treatment is applied to the cast alloy (ref. 6).

The ductility of the as-extruded powder product at elevated temperatures was higher than that of the cast alloy at temperatures to 1750° F (954° C). At this temperature a crossover occurred in the ductility curves for the as-extruded powder product and the as-cast alloy at an approximate elongation value of 27 percent. Contrary to our experience (ref. 5) with the nickel-base alloy TAZ-8A, superplasticity was not observed in high temperature tensile tests with the HS-31 prealloyed powder product. The larger grain size of the latter product and the greater number of inclusions (fig. 2), as well as differences in chemistry, may have contributed to this fact.

The effect of the swaging and aging treatments on ductility of extruded material was to reduce room temperature elongations to about 10 percent. But the 1200° F (649° C) elongation of the swaged material was 20 percent, essentially unchanged from the as-extruded value. Aging increased the 1400° F (760° C) elongation from 27 to 36 percent.

Insufficient material was available to permit tensile testing of autoclaved material.

Stress-Rupture Properties

The stress-rupture data are summarized in table V. Figure 5 shows a comparison of the stress-rupture lives of the as-extruded and heat-treated powder products and the cast alloy at 1200° F (649° C) and 61 000 psi (420 MN/m^2). At this test condition the cast alloy had an average life of 10 hours (ref. 6). The prealloyed powder products had substantially higher lives: the as-extruded powder product had a 342-hour life and the material that was subjected to the nonautoclave heat treatment C had a 209-hour life. The reason for the lower life of the heat-treated product compared to the as-extruded powder product is not apparent from a comparison of their microstructures which are similar (see figs. 8 and 11). Material subjected to autoclave heat treatment B had a life of 421 hours. Although this heat treatment was intended to provide improved high temperature properties, the coarser-grained material resulting from this heat treatment had better life at this relatively low temperature. It can be seen that the autoclave heat treatment B (fig. 10(b)) resulted in a considerably larger grain size, ASTM number 6, as against ASTM number 10 for the as-extruded material (fig. 2).

A comparison of stress-rupture properties at 1800° F (982° C) and 13 000 psi (90 MN/m^2) is made in figure 6 for the as-extruded HS-31 powder product, the extruded material after being subjected to various heat treatments, and the as-cast alloy. The longest life for the powder product, approximately 20 hours, was obtained after autoclave heat treatment A (table III). This life is double the 10-hour, as-cast life (ref. 6) even though the microstructure of the HS-31 powder product after autoclave heat treatment A (fig. 10(a)) shows that this material still had a grain size (ASTM number 3.5) appreciably finer than that of as-cast sample of the alloy (fig. 7). As would be expected, the strongest material, namely that exposed to autoclave heat treatment A, had the lowest stress-rupture ductility of all powder products tested. However, the stress-rupture ductility values observed (i.e., 4.5 and 7 percent) are not excessively low.

At the test conditions shown in figure 6, the as-extruded powder product had a life of less than 1 hour. The two nonautoclave heat treatments, C and D, both failed to increase life beyond 1 hour. At all these conditions the grain size was less than that of the material subjected to the autoclave heat treatment A. As noted previously, an intermediate grain size, ASTM number 6, was obtained with the powder product subjected to autoclave heat treatment B. This material had approximately 3 hours of life at the test condition.

These results demonstrate that, upon application of a suitable autoclave heat treatment, it is possible to achieve high temperature strength with an extruded prealloyed powder product that is equivalent to or greater than the strength of the alloy's cast counterpart. The results also suggest that not only increased grain size but also the solidification structure of the grain boundaries resulting from the autoclave heat treatments (as will be further discussed in the section dealing with microstructure) may have contributed to the improved high temperature life with the prealloyed powder product.

Workability

An as-extruded specimen 0.535 inch (1.36 cm) high and 9/16 inch (1.43 cm) in diameter was reduced by hot pressing to a height of 0.315 inch (0.80 cm) without edge cracking. A bar which had first been subjected to autoclave heat treatment A prior to hot pressing was also reduced in height with only minimal edge cracking from 0.500 inch (1.27 cm) to 0.246 inch (0.62 cm). These rather limited data suggest that even after a heat treatment which resulted in considerable grain growth (ASTM micrograin size number 3.5 compared to number 10), the HS-31 prealloyed powder product can be deformed by hot pressing, and the material need not be superplastic to accomplish such deformation.

Microstructure

The microstructure of the prealloyed powder product was examined before consolidation and after various heat treatments. This aspect is discussed in the following sections:

As-cast alloy. - As a frame of reference, the microstructure of investment cast HS-31 is shown in figure 7. A comparison with the structure of the loose powder (fig. 1) shows both to be dendritic. The dendrites in the powder are, of course, much finer. The casting represented by figure 7 was a bar with a 1/2-inch (1.27 cm) square section. Some of the grains extended to the center of the bar.

As-extruded powder product. - Figure 8 shows the microstructure of a transverse section of the as-extruded HS-31 powder product. A series of chains of carbides outline grain boundaries. Scattered inclusions are also present. The average grain size of the as-extruded bars was ASTM number 10 (0.011 mm diam), considerably coarser than that previously observed (ref. 5) for the TAZ-8A alloy (ASTM number 14, ~0.0028 mm diam) and about the same as that observed for the Alloy 713C as-extruded powder product (ASTM number 9. 5, ~0. 012 mm diam). This grain size difference may account for the absence of high elongations in the high temperature tensile tests with HS-31 compared to elongations of 450 percent observed with the as-extruded TAZ-8A prealloyed powder product (ref. 5). Although the grain sizes of HS-31 and Alloy 713C were about the same, the somewhat lower elongations observed with the as-extruded HS-31 powder product compared to those observed with Alloy 713C (ref. 5) may be due, in part, to the greater number of inclusions in the air-melted HS-31 as well as to differences in chemistry and processing.

Grain growth of as-extruded powder product. - As previously described, a thermal-gradient bar was used to establish a suitable heat treatment for achieving grain growth. Figure 3 shows a longitudinal section of the hotter portion of the bar at a low magnification after exposure under argon for 1 hour in a thermal-gradient furnace. Also shown at higher magnifications are two areas of the bar taken above and below the solidus to illustrate the structure. Appreciably more grain growth occurred at the temperatures above the solidus, approximately 2340° F (1282° C), than at the temperatures below the solidus. This is apparent in a comparison of the micrographs of figure 3 and the micrograph of an as-extruded bar not subjected to heat treatment (fig. 2). Exposure to a temperature (~2310° F, 1266° C) slightly below the solidus caused significant growth of selected grains. However, many small grains remained, resulting in a duplex grain size. The thermal-gradient bar also shows that the extruded HS-31 powder product has a wide temperature range (over 200° F, 111° C) between the solidus and liquidus. Thus, although the back wall of the gradient furnace was at a temperature of 2550° F (1399° C), complete melting of the specimen did not occur where it was subjected to this temperature.

As previously noted, the portion of the bar which was heated above the solidus swelled considerably. The darker region of the bar, which was at temperatures below the solidus, had a diameter of approximately 0. 55 inch (1. 40 cm). The lighter region, heated above the solidus, had a diameter of approximately 0. 61 inch (1. 55 cm). The swelling may have been due to gas pressure arising from argon entrained during extrusion and to possible reactions between entrained slag and carbides. Very large voids also formed near the surface of the bar. Near the center of the bar the voids were typical of those found at the junction of several grain boundaries after incipient melting.

Figure 9 shows the microstructure of an extruded bar of HS-31 prealloyed powder after exposure at 2400° F (1316° C). The structure was very similar to that of liquid phase sintered, slip-cast HS-31 (ref. 1). The eutectic-like solidification structure is apparent at the grain interfaces of the semi-melted material, and this may contribute to its good high temperature strength (see previous section Stress-Rupture Properties). This grain boundary structure is somewhat similar to that of cast HS-31 (fig. 7). The grain size of the extruded bars after heating to 2400° F (1316° C) increased to about ASTM number 5. 5 from an ASTM size of 10 for the as-extruded material.

Effect of autoclaving. - Although grain growth was achieved by heat treating above the solidus, it is obvious (fig. 3) that the massive voids left in the material were unacceptable. Consequently, an autoclave heat treatment at 2200° F (1204° C) was applied for 3 hours at a pressure of 30 000 psi (206 MN/m^2) to close the voids. The structure resulting from autoclave heat treatment A (1 hr at 2450° F (1342° C) + 3 hr at 2200° F (1205° C), at 30 000 psi (207 MN/m^2)) is shown in figure 10(a). This heat treatment has affected the microstructure compared to the as-extruded material (fig. 2) by substantially increasing grain size. Also, based on optical microscopy, most voids resulting from exposure above the solidus appear to have been closed. Figure 10(b) shows the structure resulting from autoclave heat treatment B (table III). Comparison of figures 10(a) and (b) indicates that the material subjected to autoclave heat treatment A had a larger grain size (ASTM number 3. 5) than that subjected to autoclave heat treatment B (ASTM number 6). After heat treatment B, carbide precipitation and some recrystallization are apparent. The reason for the recrystallization of the extruded material after heat treatment B and the apparent lack thereof after heat treatment A is not certain. However, it can be postulated that it occurred as a result of the unintentional second step (1/4 hr at 1950° F (1065° C) and 31 600 psi (218 MN/m^2)) of the autoclave heat treatment, which could have caused deformation. This warm work may then have caused partial recrystallization when the 2200° F (1205° C) temperature was reached in step 3 of the heat treatment and may also have produced additional intragranular nucleation sites for carbide precipitation.

The heat treatment in the autoclave described here was limited to 2200° F (1204° C) to avoid the possibility of completely melting the specimen since it is more difficult to control temperature at very high pressure. With better temperature control it should be possible to use a single heat-treating step applied in the autoclave at a temperature on the order of 2400° F (1316° C). Such an approach might eliminate the need for the initial consolidation step applied to the prealloyed powders, namely extrusion. Thus, with appropriate tooling, hardware might be produced in an autoclave directly from loose powders as the authors previously suggested in reference 5.

Nonautoclave heat treatments. - As can be seen from figure 11, the 1350° F (723° C) heat treatment which was applied to as-extruded bars (heat treatment C, table III) resulted in more clearly defined grain boundaries, probably as a result of additional precipitation. No noticeable change in grain size was apparent compared to the as-extruded material. Extruded-and-aged material was subjected to 2240° F (1220° C) for 1 hour and then re-aged at 1350° F (723° C) (heat treatment D, table III). Only very slight grain growth (ASTM number 9. 5) occurred compared to that of the as-extruded powder product. As was shown earlier, there was also no difference in 1800° F (982° C) stress-rupture life for the as-extruded powder product and for the extruded powder product after being subjected to these two aging heat treatments.

CONCLUDING REMARKS

Although the data are rather limited, the results of this investigation reaffirm the ability to achieve substantial gains in room temperature and intermediate temperature strength with superalloys using extruded prealloyed powders. They also indicate that such a prealloyed powder product without suitable heat treatments has substantially lower elevated temperature strength than its cast counterpart. However, heat treatments that exceed the solidus temperature and employ the application of pressure can substantially increase the elevated temperature life of the prealloyed powder product and can achieve lives comparable to those obtainable with the cast product. Such heat treatments not only provide significant grain growth, but they also provide a solidification structure at the grain boundaries, both of which may be necessary for high temperature strength of prealloyed powder products. This is further borne out by the improved high temperature stress-rupture life demonstrated in reference 1 for a slip-cast HS-31 powder product subjected to liquid phase sintering.

Further investigation is obviously required to determine the most suitable combination of temperature and pressure to achieve maximum high temperature stress-rupture properties with extruded prealloyed HS-31 as well as with other superalloys prepared in a similar fashion. However, the concept of heat treating above the solidus and applying high pressure to restore the integrity of the material, thus closing voids formed as a result of minor-phase melting, appears to hold considerable promise for substantially increasing the high temperature strength of compacted prealloyed powder products.

SUMMARY OF RESULTS

Evaluation of a cobalt-base alloy, HS-31, made by extrusion of prealloyed powders gave the following major results:

1. Tensile strengths of the as-extruded powder product were substantially greater than those of cast HS-31, at room temperature and from 1000° to 1400° F (538° to 760° C). The ultimate tensile strengths at room temperature and at 1400° F (760° C) were 175 500 and 88 200 psi (1210 and 608 MN/m^2), respectively, for the powder product, compared to 108 000 and 72 500 psi (745 and 500 MN/m^2) for the cast alloy. At higher temperatures, however, the cast alloy had higher tensile strengths; the comparison at 1800° F (982° C) being 30 000 psi (206 MN/m^2) for cast HS-31 and 21 700 psi (150 MN/m^2) for the as-extruded powder product.

2. Application of additional cold work to the extruded powder product affords a method of further increasing both room temperature and intermediate temperature tensile strength. For example, limited cold swaging increased both the room temperature and 1200° F (649° C) ultimate tensile strengths by 8500 psi (58 MN/m^2).

3. Superplasticity was not observed with the as-extruded powder product in high temperature tensile tests.

4. Heat treatments above the incipient melting point increased the grain size of the extruded prealloyed powder product, and the subsequent application of pressures of 30 000 psi (206 MN/m^2) at 2200° F (1204° C) in an autoclave achieved a structurally sound product. Grain size was changed from ASTM number 10 to ASTM number 3.5.

5. An extruded powder product which had been heat treated above its incipient melting temperature and then consolidated in an autoclave showed substantially improved stress-rupture life over the cast alloy at an intermediate temperature (1200° F, 649° C) and life comparable to that of the cast alloy at high temperature (1800° F, 982° C). Maximum stress-rupture lives of 421 and 21 hours, respectively, were obtained with the autoclave-heat-treated product at 1200° F (649° C) and 61 000 psi (420 MN/m^2) and at 1800° F (982° C) and 13 000 psi (90 MN/m^2). These lives compared to 10 hours for as-cast HS-31 at each condition.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, July 15, 1970,
129-03.

REFERENCES

1. Fischer, George W.; et al.: Complex Shapes by Slip Casting. Rep. R65FPD185, General Electric Co. (AFML-TR-65-257, DDC No. 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, DDC No. 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, DDC No. 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, DDC No. 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. Anon.: Haynes Stellite Alloy No. 31, Union Carbide Stellite Co., Apr. 1963.

7. Paprocki, S. J.; Hodge, E. S.; and Gripshover, P. J.: Gas-Pressure Bonding. DMIC Rep. 159, Battelle Memorial Inst., Sept. 25, 1961.

TABLE I. - PARTICLE SIZE DISTRIBUTION OF HS-31 ATOMIZED POWDERS[a]

Tyler screen size	Particle size distribution, percent
>30	0.01
30/60	8.69
60/100	14.86
100/200	30.98
200/270	16.26
270/325	7.24
<325	20.44

[a]Analysis by supplier.

TABLE II. - CHEMICAL ANALYSES OF HS-31 POWDER PRODUCTS

Element	Nominal composition	-30 Mesh powder[a]	Extrusion[b]
	Weight, percent		
Chromium	24.5 to 26.5	25.76	26.20
Nickel	9.5 to 11.5	10.68	10.25
Tungsten	7.0 to 8.0	7.50	7.17
Manganese	1.00 max.	.56	.66
Iron	2.00 max.	.19	.17
Silicon	1.00 max.	.54	.80
Carbon	0.45 to 0.55	.55	.54
Sulphur	.040 max.	.005	---
Phosphorous	.040 max.	.002	.002
Molybdenum	---------	.01	.022
Oxygen	---------	.011	.0244
Hydrogen	---------	.0003	-----
Nitrogen	---------	.040	-----
Cobalt	Balance	Balance	Balance

[a]Analysis by supplier.
[b]Analysis by an independent laboratory.

TABLE III. - MATERIAL CONDITIONS OF HS-31 EXTRUDED

POWDER PRODUCTS INVESTIGATED

Designation	Step 1	Step 2	Step 3	Final grain size, ASTM number
Autoclave heat treatment A	1 hour at 2400° F (1316° C) or 2450° F (1342° C); air cool	3 hours at 2200° F (1205° C) and 30 000 psi (207 MN/m^2)	-----------------	3.5
Autoclave heat treatment B	1 hour at 2400° F (1316° C); furnace cool	1/4 hour at 1950° F (1065° C) and 31 600 psi (218 MN/m^2)	3 hours at 2200° F (1205° C) and 30 000 psi (207 MN/m^2)	6
Nonautoclave heat treatment C	50 hours at 1350° F (732° C)	-----------------	-----------------	10
Nonautoclave heat treatment D	50 hours at 1350° F (732° C)	1/2 hour at 2240° F (1226° C)	50 hours at 1350° F (732° C)	9.5
As-extruded	-----------------	-----------------	-----------------	10

TABLE IV. - TENSILE DATA FOR HS-31 POWDER PRODUCTS

Condition	Test temperature		Ultimate tensile strength		Elongation, percent
	°F	°C	psi	MN/m^2	
As extruded	(a)	(a)	175 500	1210	18
	1000	538	157 800	1085	34
	1200	649	134 500	927	21
	1400	760	88 200	608	27
	1500	815	58 200	401	33
	1600	872	43 400	299	30
	1800	982	21 700	150	27
	2000	1094	9 800	68	20
	2100	1149	5 900	41	18
	2225	1219	2 400	16	21
Extruded + Swagged	(a)	(a)	184 000	1269	10
	1200	649	143 000	985	20
Extruded + Nonautoclave heat treatment C	(a)	(a)	165 000	1137	9
	1400	760	82 400	567	36

[a]Room temperature.

TABLE V. - STRESS-RUPTURE DATA FOR HS-31 POWDER PRODUCTS

Condition	Test temperature		Stress		Life, hr	Elongation, percent
	^{o}F	^{o}C	psi	MN/m^2		
As extruded	1200	649	61 000	420	341.9	21
	1200	649	105 000	724	4.1	21
	1800	982	13 000	90	.9	20
Extruded + Nonautoclave heat treatment C	1200	649	61 000	420	209.1	25
	1800	982	13 000	90	.8	18
Extruded + Nonautoclave heat treatment D	1800	982	13 000	90	0.7	12
Extruded + Autoclave heat treatment A	1800	982	13 000	90	20.9	7
	1800	982	13 000	90	18.1	4.5
Extruded + Autoclave heat treatment B	1200	649	61 000	420	420.5	9
	1500	815	35 000	241	8.31	--
	1800	982	13 000	90	2.7	23
	1800	982	13 000	90	3.0	21

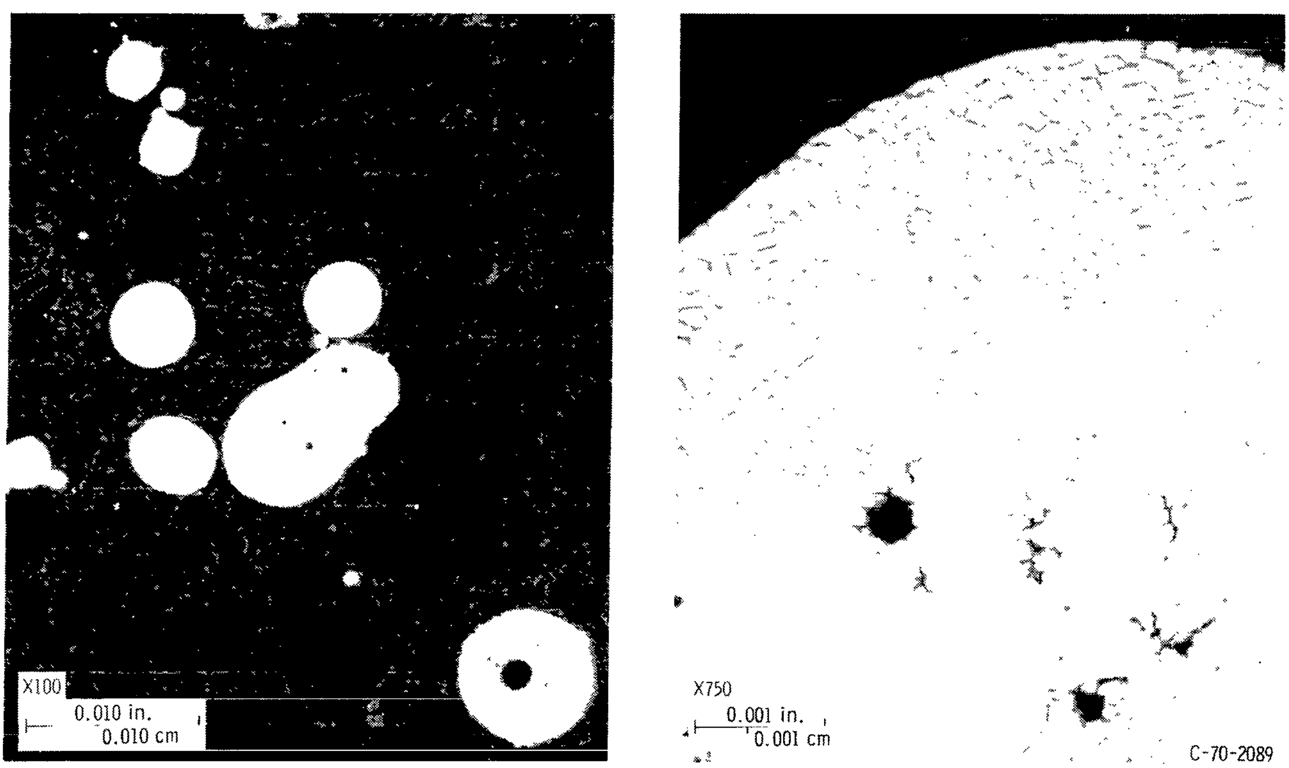


Figure 1. - As-received HS-31 powders.

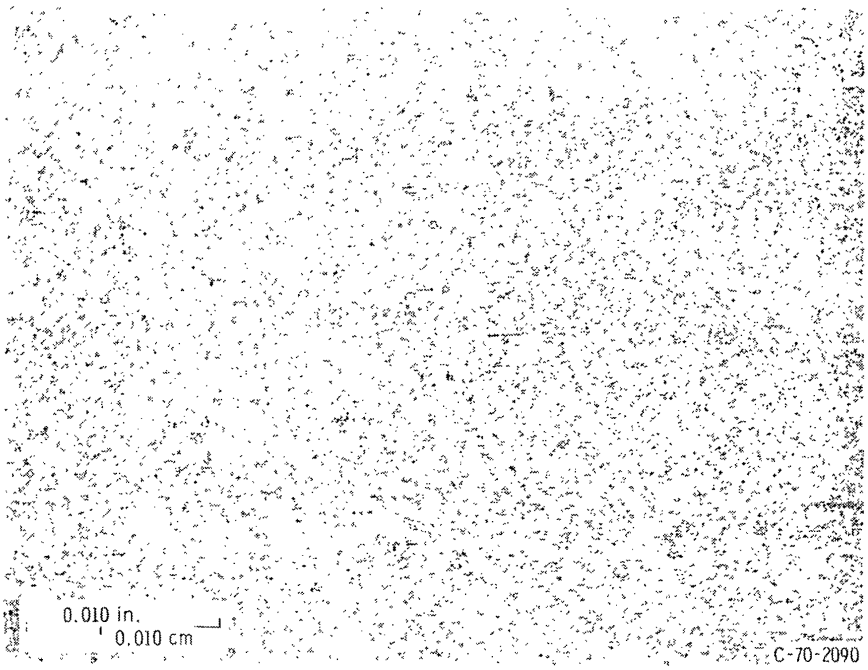


Figure 2. - Longitudinal section of as-extruded bar of HS-31 prealloyed powder, showing stringered inclusions. X100.

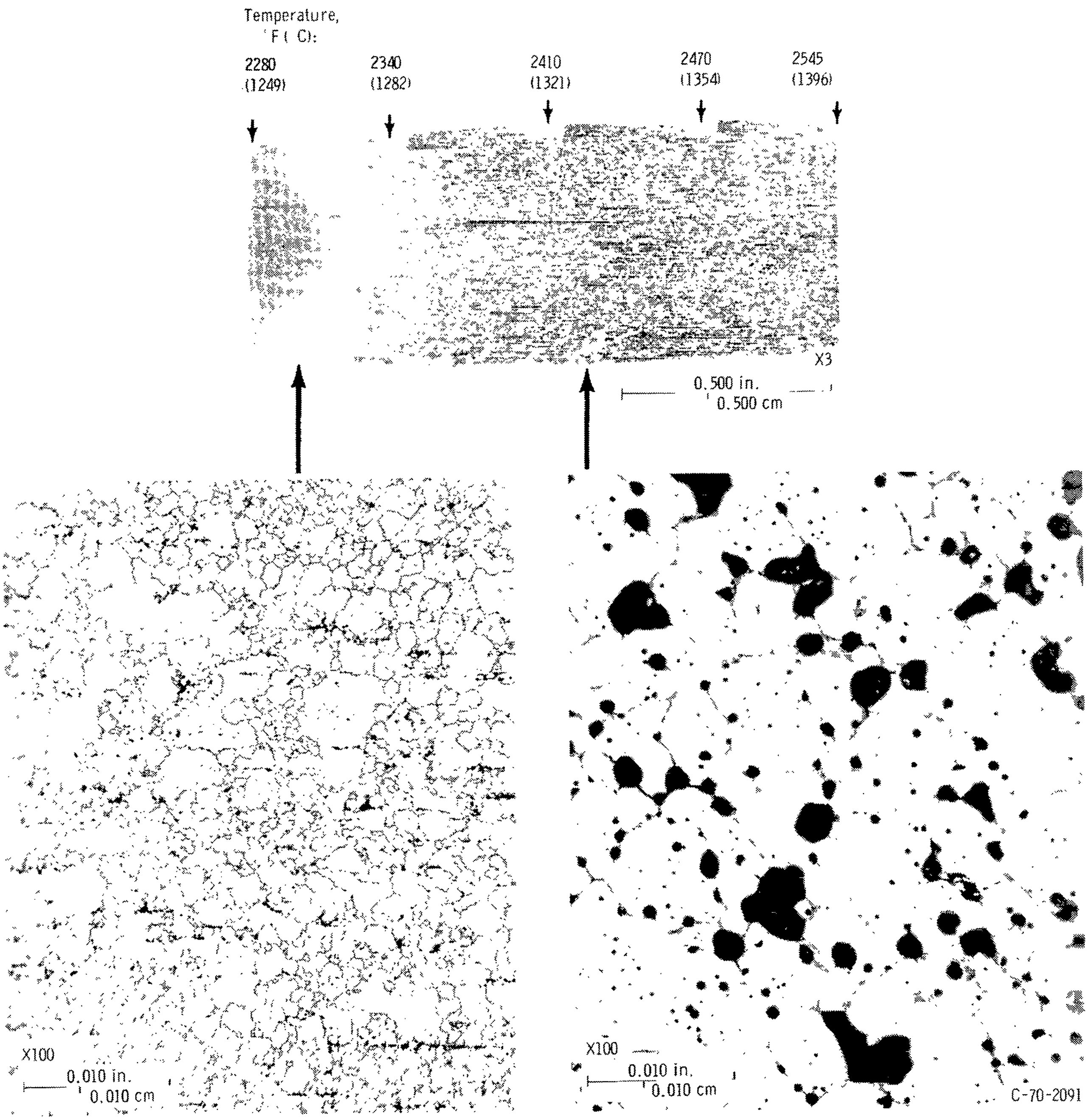


Figure 3. - Thermal gradient bar of as-extruded HS-31 prealloyed powder, showing effect of temperature on microstructure.

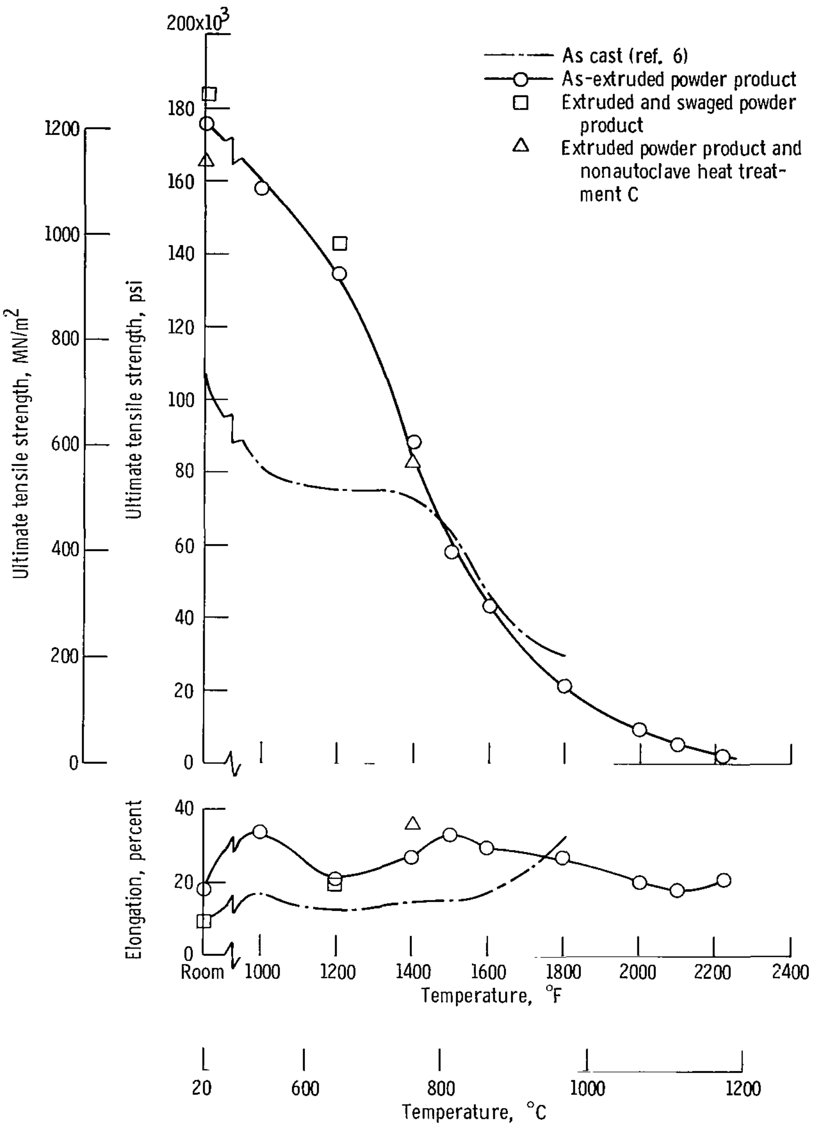


Figure 4. - Comparison of tensile properties of HS-31 powder product and as-cast HS-31.

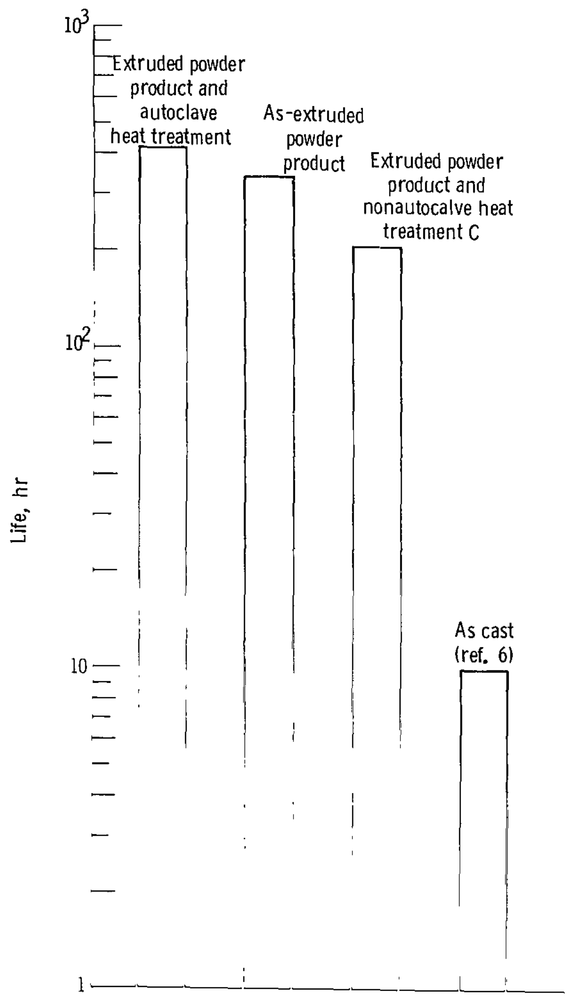


Figure 5. - Comparison of rupture lives of HS-31 powder products and cast HS-31 at 1200° F (649° C) and 61 000 psi (420 MN/m^2).

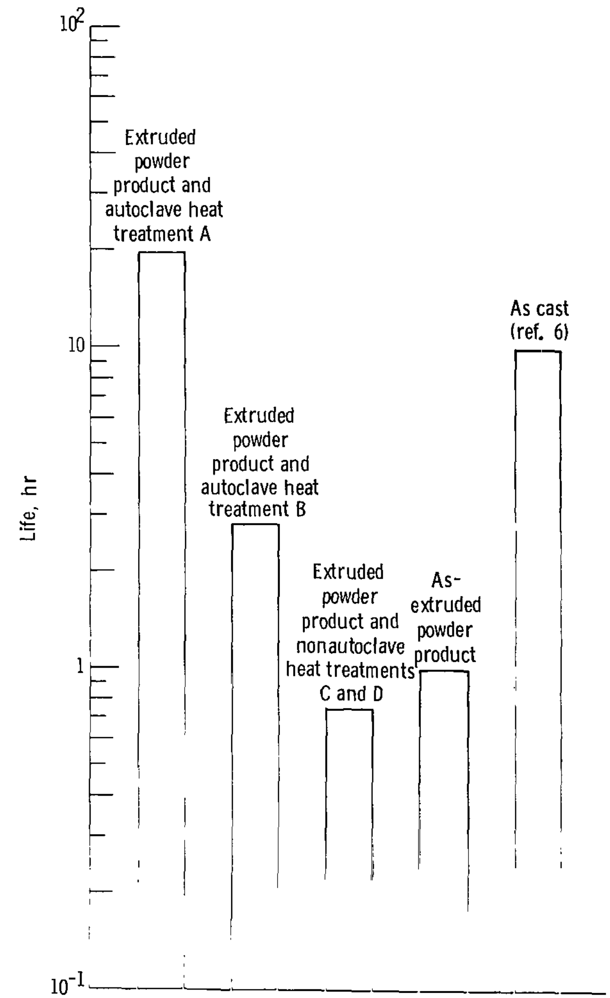


Figure 6. - Comparison of rupture lives of HS-31 powder products and cast HS-31 at 1800 F (982 C) and 13 000 psi (90 MN/m^2).

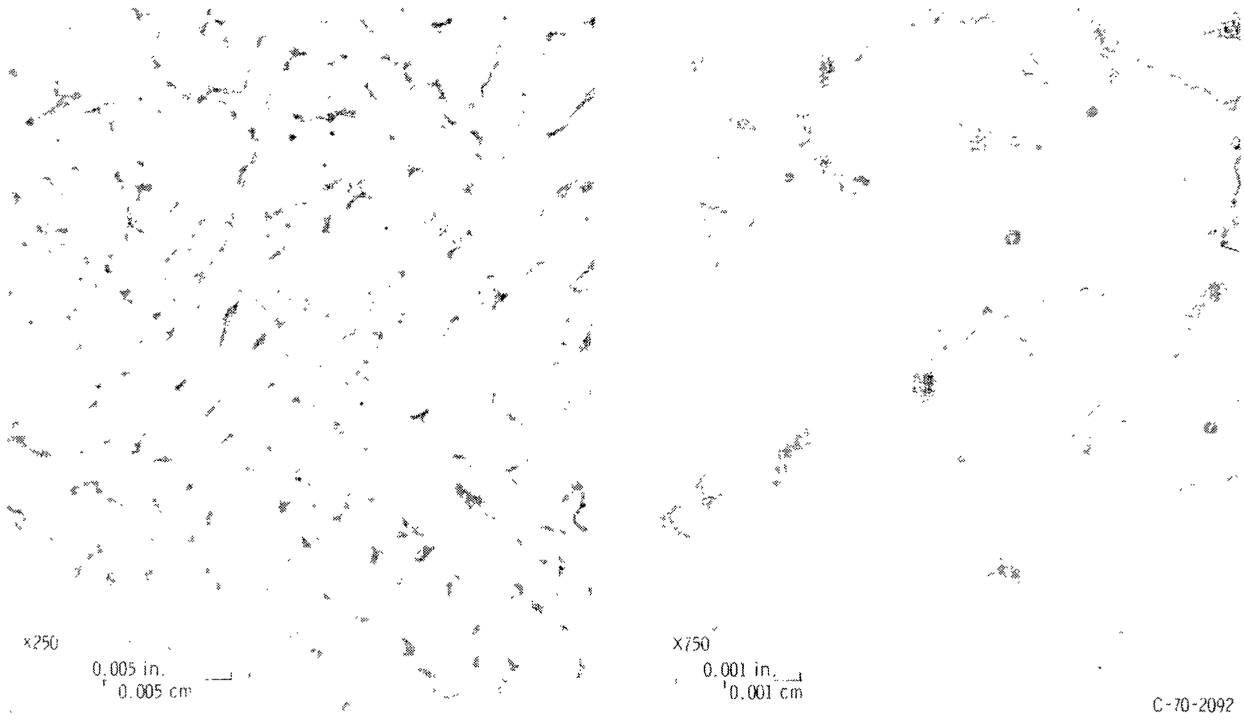


Figure 7. - Microstructure of as-cast HS-31.

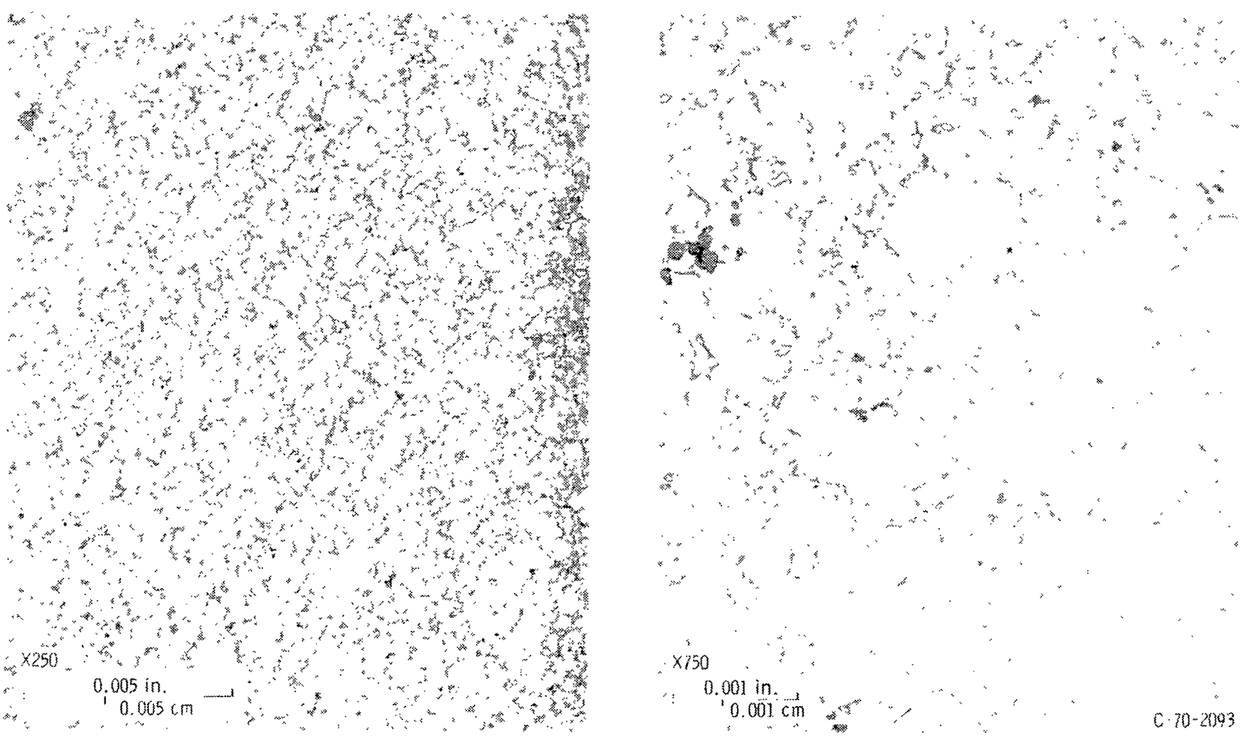


Figure 8. - Microstructure of as-extruded HS-31 powder product.

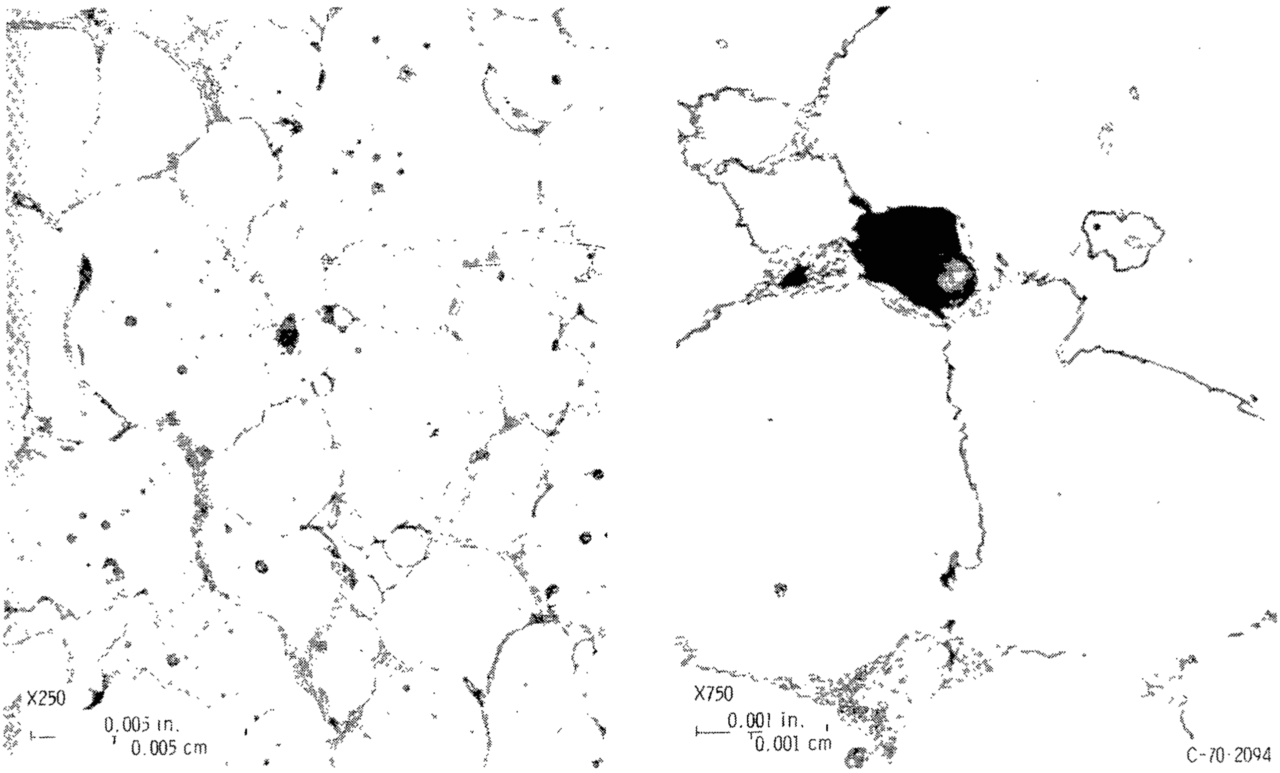
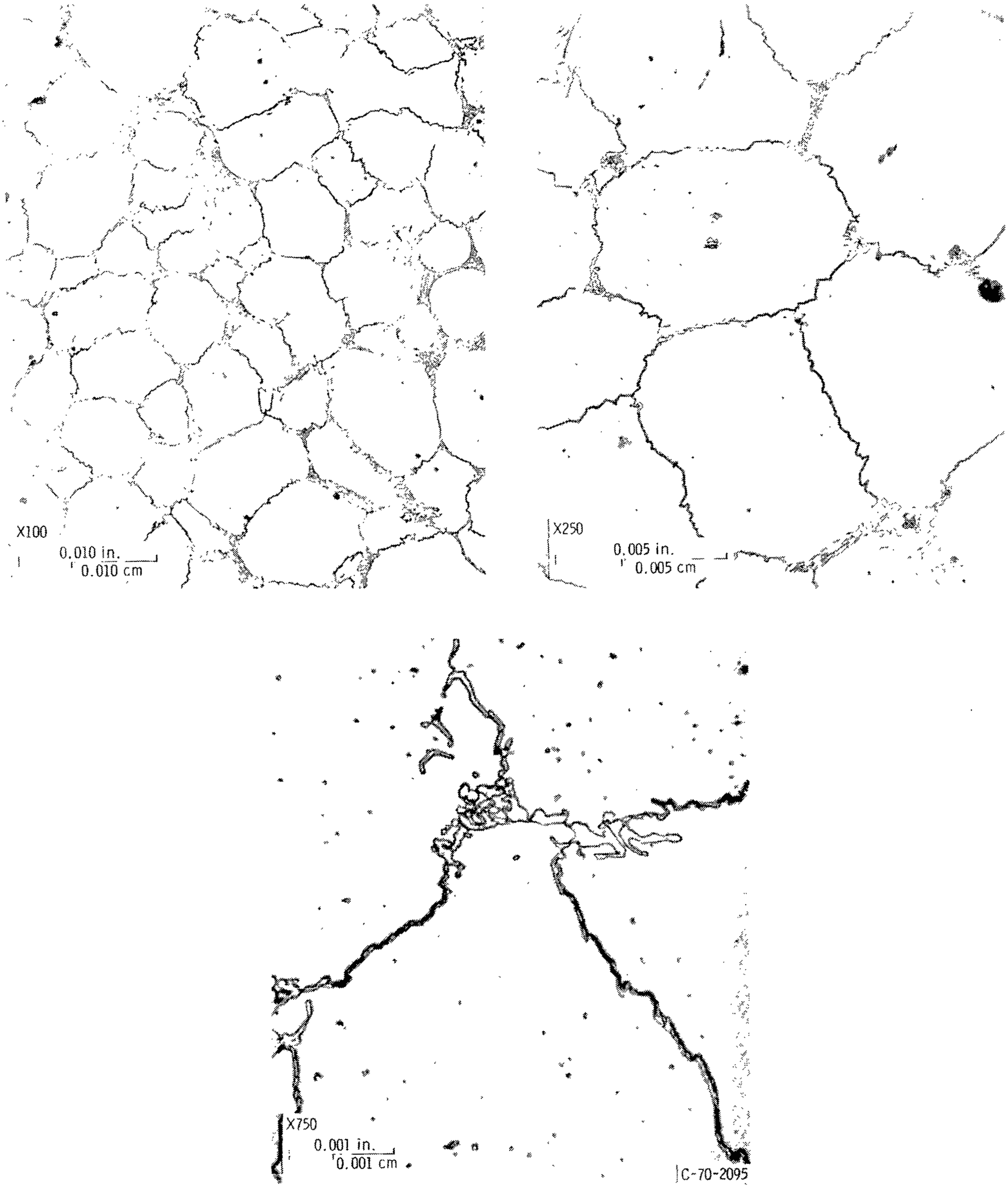
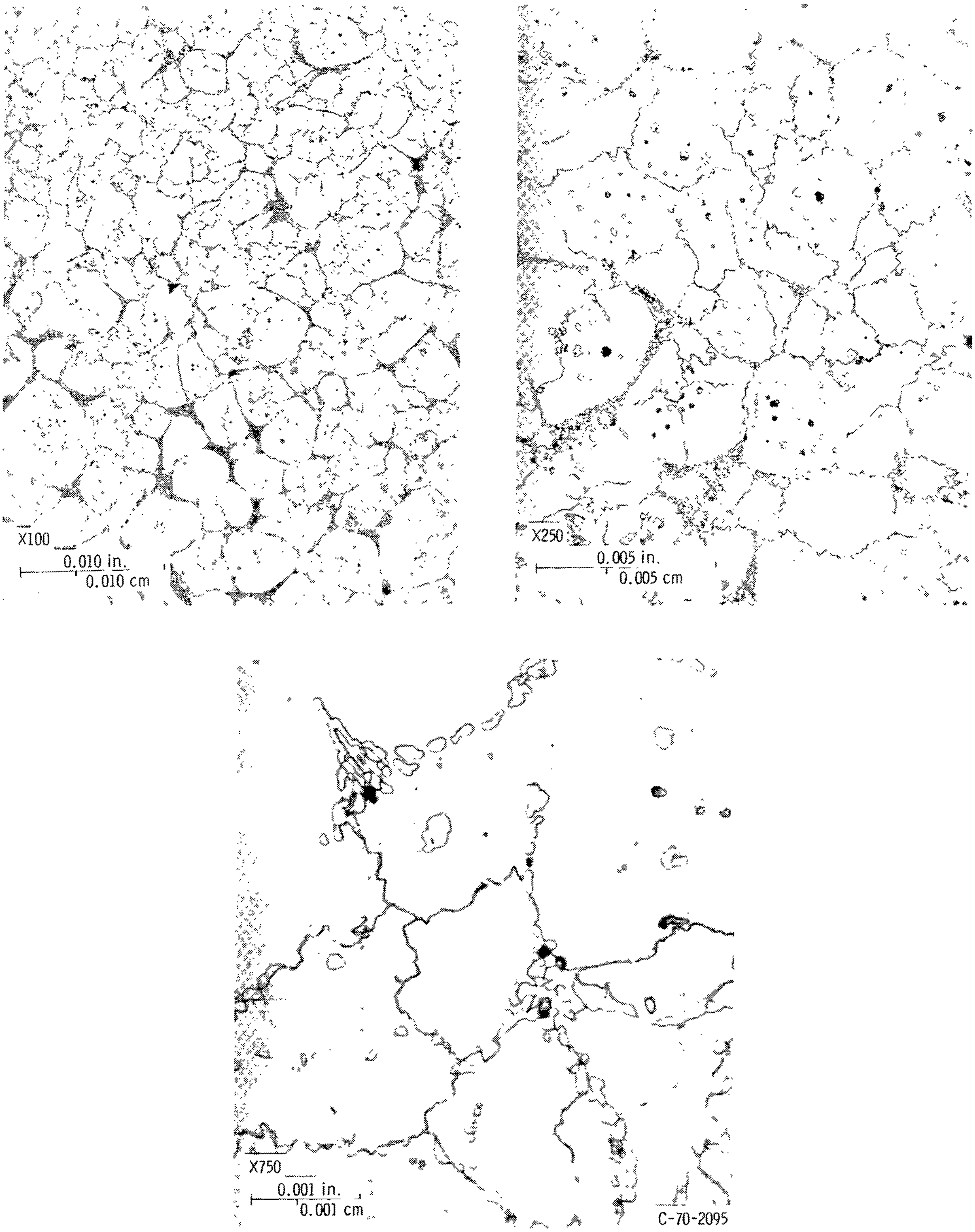


Figure 9. - Microstructure of extruded HS-31 powder product after first step (1 hr at 2400° F (1316° C)) of autoclave heat treatment B.



(a) Autoclave heat treatment A.

Figure 10. - Microstructure of extruded HS-31 powder product after autoclve heat treatments.



(b) Autoclave heat treatment B.

Figure 10. - Concluded.

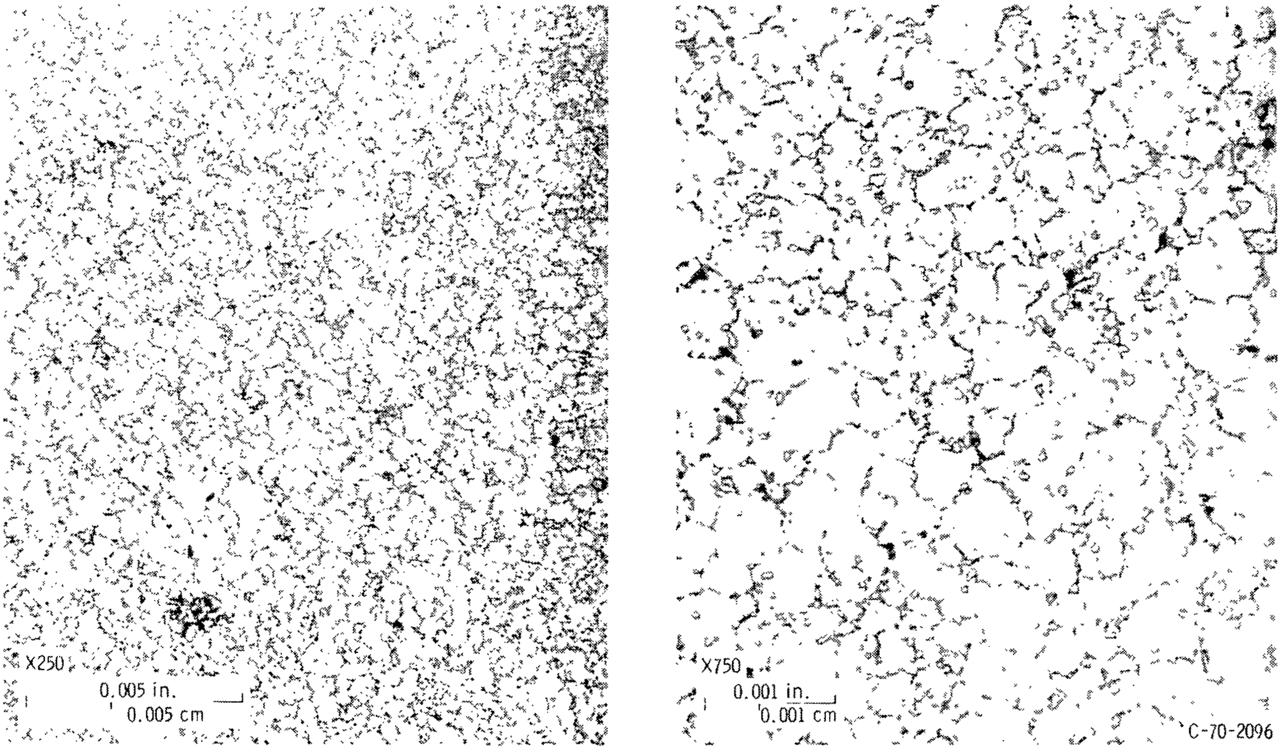


Figure 11. - Microstructure of extruded HS-31 powder product after non-autoclave heat treatment C.

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