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STUDY OF A COLUMNAR GRAIN STRUCTURE PRODUCED IN A SUPERALLOY BY GRADIENT-ANNEALING PREALLOYED POWDER EXTRUSIONS

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16. Abstract

Annealing in a temperature gradient was used to produce a structure of continuous columnar grains in alloy 713C made by extrusion of prealloyed powder. The columnar-grained specimens had tensile strengths within ±5 percent of those of the as-cast alloy at 760° and 980° C but were significantly stronger at room temperature. Stress-rupture tests at 760° and 980° C showed lives scattered from 17 to 376 percent of those of as-cast 713C tested under the same conditions. X-ray diffraction studies showed the large grains formed upon annealing to have a well-defined texture, although the as-extruded material had none.

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SUMMARY

The present work is part of an effort to achieve good strength at temperatures above 750° C in superalloys produced from prealloyed powder. A microstructure of long columnar grains was produced by annealing in a temperature gradient extrusions made from alloy 713C prealloyed powder. Mechanical test specimens without transverse grain boundaries were produced in this way and tested in tension and stress-rupture.

The columnar-grained powder product had tensile strengths within ±5 percent of those of the as-cast alloy at 760° and 980° C but was significantly stronger at room temperature. The yield and ultimate tensile strengths at room temperature were 841 and 1048 MN/m^2, which may be compared with 738 and 848 MN/m^2 for the as-cast alloy.

The stress-rupture lives of the columnar-grained specimens were scattered about those of as-cast 713C tested under the same conditions. Specimens had lives at 760° and 980° C from 17 to 376 percent of those of as-cast 713C tested under the same conditions.

Growth of abnormal grains in the prealloyed powder product was very rapid at temperatures above about 1205° C but was preceded by an incubation period probably associated with the dissolution of the gamma prime phase. The fact that the incubation time is inversely related to the temperature allows columnar growth of the abnormal grains in a temperature gradient.

There was a [110] fiber texture symmetric about the extrusion axis in the large-grained microstructures produced by either isothermal or gradient annealing. However, this texture was sharpest in the columnar grains grown along the length of the extruded bars.

INTRODUCTION

The application of powder-metallurgy techniques to the production of superalloys holds great promise. The use of prealloyed powders imparts a homogeneous distribution of the solute elements in an alloy and allows their full strengthening effect to be obtained. In cast parts or even parts forged from cast ingots segregation during solidification leaves some regions deficient in solute element concentration. In other areas solute enrichment occurs, and the normal secondary phases can precipitate in massive form and become ineffectual in strengthening, if not detrimental. Also, other phases not desired may form because of the local solute enrichment.

While significant improvements in the properties of superalloys produced from prealloyed powders as compared to their cast or wrought counterparts can be obtained at temperatures below about 750° C (refs. 1 to 6), the achievement of properties suitable for higher temperature use presents substantial difficulties. Powder-metallurgy superalloys generally have very fine grain sizes. While this benefits low and intermediate temperature properties, it leads to low strengths at high temperature and often even to superplastic behavior. For high-temperature service it is necessary to produce large grain sizes in the powder-metallurgy alloys.

Achieving grain growth has been especially difficult in advanced highly alloyed compositions which contain large amounts of insoluble phases, particularly eutectic nodules of the gamma prime phase, in nickel-base alloys. Some success with such alloys has been achieved at this laboratory by annealing at temperatures above the incipient melting point under high pressure to close any voids due to entrained argon or to partial melting (refs. 1 to 3).

Grain growth below the incipient melting point can be achieved readily, however, in some nickel-base alloys in which the gamma prime phase can be entirely dissolved. Abnormal grain growth was observed in a previous study of alloy 713C produced from prealloyed powder (ref. 1). Large equiaxed grains a few millimeters in diameter grew upon annealing the as-extruded material at 1230° C; the large grain size was not sufficient for high-temperature strength. The stress-rupture strength of this large-grained material was greatly improved over that of the as-extruded powder product, but it was still considerably less than that of as-cast 713C. The best stress-rupture life obtained for the large-grained powder product at 1038° C and 68.9 MN/m^2 was 98.1 hours, while the as-extruded powder product had only a 1.2-hour life under the same conditions and elongated 60 percent. As-cast 713C has a 250-hour life at this temperature and stress.

The goal of the present work was to produce a microstructure in powder-metallurgy alloy 713C suitable for high-temperature service. The abnormal growth behavior was controlled to produce continuous columnar grains by annealing in a temperature gradient. The use of gradient annealing to produce large grains through critical straining or secondary recrystallization is well known and is discussed thoroughly in reviews by Aust and Burgers (refs. 7 and 8). Mechanical test specimens without transverse grain boundaries were prepared and tested in tension and stress-rupture. The mechanical property data obtained are compared with those for conventionally cast 713C. Also, the mechanism of this abnormal grain growth is discussed on the basis of the results of X-ray and optical metallography.

MATERIALS AND PROCEDURE

Materials

Bars of alloy 713C produced by extrusion of inert-gas-atomized, prealloyed powder were obtained from a commercial alloy manufacturer. For extrusion, the -100 mesh fraction of the powder was sealed under vacuum in type 304 stainless-steel cans. All prior handling of the powder was done in an argon atmosphere. The extrusion cans had an outside diameter of 7.62 centimeters and a 3.2-millimeter wall thickness. They were extruded through a die having a rectangular opening and an included angle of 90°. The cross section of the extrusions (not including the thickness of the can) was 2.46 by 1.14 centimeters. Thus, a reduction of about 13.6:1 was experienced by the powder. Four extrusions were made at 1190° C, one at 1160° C, and one at 1120° C. All bars were inspected radiographically with a minimum 2 percent sensitivity and were found to be for the most part sound.

The vendor's chemical analysis of the alloy 713C powder and the desired composition from AMS 5391 (ref. 9) are presented in table I. The oxygen concentration was 93 ppm.

Gradient-Annealing of Material for Mechanical Testing

Long columnar grains were produced in samples of the 713C powder extrusions by annealing in a commercial gradient furnace so that the temperature gradient was established along the extrusion axis. During the anneal, the power applied to the furnace was slowly increased. This produced an effect analogous to moving the specimen toward the hotter end of the furnace and allowed growth of columnar grains longer than possible if the temperatures at each point in the specimens were held constant with time.

The commercial gradient furnace used was heated by resistance elements at the back wall of a muffle designed to produce a nearly linear temperature gradient. The gradient was about 28° C per centimeter with the back wall temperature at 1315° C.

TABLE I. - CHEMICAL ANALYSIS OF

ALLOY 713C POWDER

Element	Specified concentration, wt. % (a)	Actual concentration, wt. % (b)
Aluminum	5.5 to 6.5	5.89
Carbon	0.08 to 0.20	0.11
Columbium and tantalum	1.8 to 2.8	2.23
Chromium	12.0 to 14.0	13.22
Molybdenum	3.8 to 5.2	4.88
Titanium	0.50 to 1.0	0.8
Boron	0.005 to 0.015	0.011
Zirconium	0.05 to 0.15	0.08
Cobalt	<1.0	0.10
Copper	<0.5	0.034
Iron	<2.5	0.88
Manganese	<0.25	0.01
Phosphorous	(c)	0.004
Sulfur	<0.015	0.003
Silicon	<0.50	0.25
Oxygen	[c]<0.01	0.0093
Nickel	Balance	Balance

[a]AMS specification 5391A.
[b]Vendor's analysis.
[c]Not specified in AMS 5391A.

Specimens of the 1190° C extrusions 8 centimeters long were placed in the furnace with one end in contact with the back wall, which had a temperature of 1205° C. Thus, initially the temperature of the bar varied from 1205° C at one end to about 980° C at the other. The temperature of the furnace was then increased at a rate of about 28° C per hour until the back-wall temperature reached 1315° C. The temperature along the bar then varied from 1315° to about 1090° C. The back wall was held at 1315° C for 30 minutes, and then the specimens were removed and air-cooled. None of the material from the 1120° or 1160° C extrusions was gradient-annealed for mechanical testing.

Mechanical Testing

Two mechanical test specimens were machined from each gradient-annealed bar. The bars were first cut in half lengthwise and macroetched in order to observe the length of the columnar grains developed. The gage section of the test specimens was

6.4 millimeters in diameter, and the total length was adjusted so that the columnar grains extended well into the gripping ends. Because of the limited length of columnar grains grown in the gradient furnace, the gage lengths were as short as 1.91 centimeters. The four-times-diameter gage length recommended by the ASTM would have been 2.54 centimeters. Thus, the elongations reported may be slightly greater than if the specimens were of standard length. Except for the short gage lengths of the specimens, ASTM recommended practice was followed in the mechanical testing.

Study of Abnormal Grain Growth

The mechanism of abnormal grain growth was investigated both as it occurred in isothermal annealing and in a temperature gradient. In the isothermal annealing study, small pieces of the material extruded at 1190° C were annealed for various times at several temperatures from 1190° to 1260° C. The specimens were water-quenched after annealing. Finally, they were sectioned and examined metallographically.

For study of abnormal grain growth in a temperature gradient, specimens were annealed in the gradient furnace with the temperature at the back wall held constant, not increased with time as was done with the material for mechanical testing. Thus, each point in these specimens was exposed to a constant temperature. Three 7-centimeter-long pieces, one each from the 1120°, 1160°, and 1190° C extrusions, were placed side by side in the gradient furnace. The piece in the center had thermocouples attached to both ends and at midlength. After annealing the pieces were sectioned for metallographic examination.

Several of the specimens annealed as just described were examined by X-ray diffraction. The crystallographic orientations of the abnormal grains in these specimens were determined from back-reflection Laue patterns. Also, Laue patterns were obtained from the as-extruded material to determine if any deformation texture had been produced by the extrusion.

RESULTS AND DISCUSSION

Tested Material

Shown in figure 1 is a mechanical test specimen failed in stress-rupture at 760° C. It is sectioned and macroetched to show the large columnar grains developed by the gradient anneal. In each gradient-annealed bar, the columnar grains developed were about 5 centimeters long and 1 to 5 millimeters in diameter. Each test specimen ground from

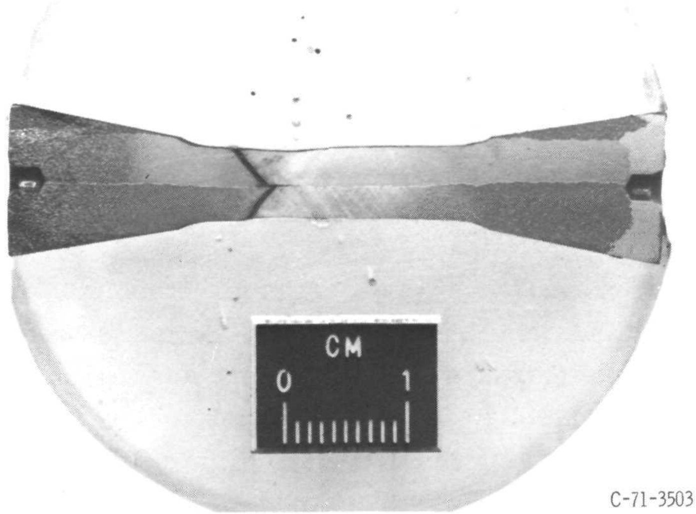

Figure 1. - Failed stress-rupture specimen sectioned and etched to show columnar grains developed by gradient-annealing.

one of these bars contained from 1 to 7 grains in the test section, and none contained transverse grain boundaries in the test section. All the mechanical test specimens were produced from the bars extruded at 1190° C.

Tensile Properties

The results of the tensile tests at room temperature and 760° and 982° C are given in table II together with the reported properties of as-cast alloy 713C at those tempera-

TABLE II. - TENSILE DATA FOR ALLOY 713C

Structure	Test temperature, °C	Yield strength, MN/m^2	Ultimate tensile strength, MN/m^2	Elongation, percent	Gage length, cm
Columnar-grained	Room temperature	841	1048	34	2.29
	760	772	896	11	2.29
	980	---	490	20	2.29
[a]As-cast	Room temperature	738	848	7.9	----
	760	744	938	5.9	----
	980	303	469	20	----

[a]Ref. 10.

tures (ref. 10). The properties of the columnar-grained material are within ±5 percent of those of the as-cast material except at room temperature, where the columnar-grained material was clearly superior. Yield and ultimate tensile strengths were 841 and 1048 MN/m^2, compared to 738 and 848 MN/m^2, respectively, for the cast alloy. The elongation was also improved over the 7.9 percent for the cast alloy, although the 34 percent measured in the 2.29-centimeter gage length may be slightly greater than if the gage length were the full 2.54 centimeters recommended by ASTM.

Stress-Rupture Properties

The stress-rupture data obtained in the present study and data for as-cast 713C (ref. 10) are given in table III. A graphical comparison is made in figure 2. There was large scatter in the results of the stress-rupture tests. Some specimens showed lives considerably greater than that of cast 713C tested under the same conditions, and other h had lives considerably less. At 573 MN/m^2, a stress which gives 100-hour life at 760° C for as-cast 713C, lives of 122.5 and 16.9 hours were obtained for the columnar-grained material. Lives of 154.8 and 75.5 hours were obtained at 980° C and 145 MN/m^2, the 100-hour stress for the as-cast alloy. The single test at 980° C and 103 MN/m^2 resulted a 1391.2-hour life as compared to a life of 370 hours for the as-cast alloy.

Examination of the failed specimens indicated that, in the two shortest lived specimens in particular, the two or three grains in the test sections were oriented so that mutual strain accommodation was difficult. Deformation was limited to sharply defined

TABLE III. - STRESS-RUPTURE DATA FOR ALLOY 713C

Structure	Test temperature, $^\circ$C	Stress, MN/m^2	Life, hr	Elongation, percent	Gage length, cm
Columnar-grained	760	573	16.9	11	1.91
			122.5	21	1.91
	980	145	154.8	7	2.29
			75.5	--	----
	980	103	1391.2	--	----
[a]As-cast	760	573	100	--	----
	980	145	100	--	----
	980	103	370	--	----

[a]Ref. 10.

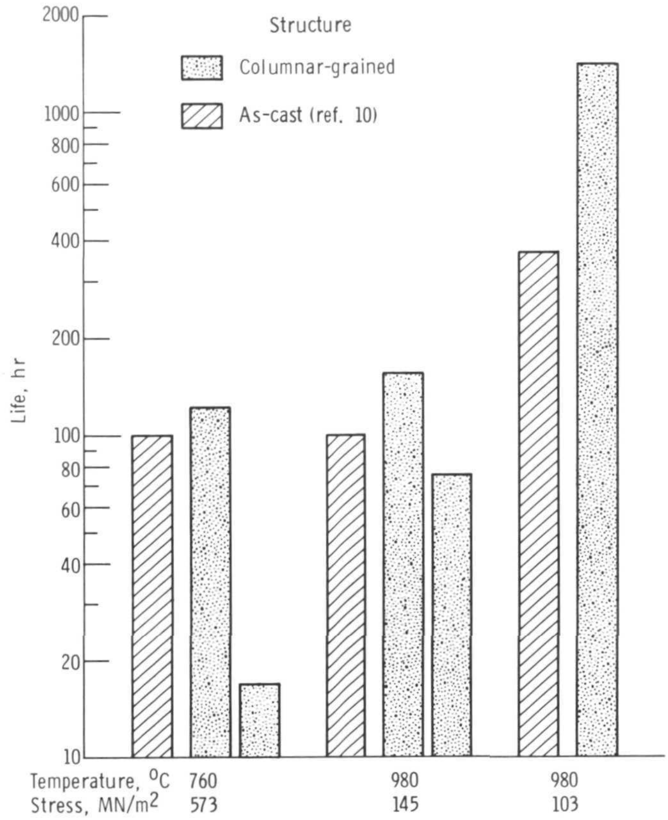

Figure 2. - Stress-rupture lives of columnar-grained alloy 713C produced from prealloyed powder compared with those of as-cast 713C.

slip planes. This behavior can produce dislocation pileups where slip bands meet the internal grain boundaries. Such areas would provide crack initiation sites and embrittle the material.

It is expected that reducing the problem of strain accommodation would lead to more uniformly high properties than exhibited by the material produced in this study. This might be accomplished in two ways. First, the problem should be eliminated entirely if single crystals could be produced. Second, the problem should be reduced by decreasing the average diameter of the grains in a columnar structure like that produced in this work. Decreasing the grain diameter would reduce the number of dislocations piled up at slip-band - grain-boundary intersections at a given strain and thus reduce the local stress concentrations.

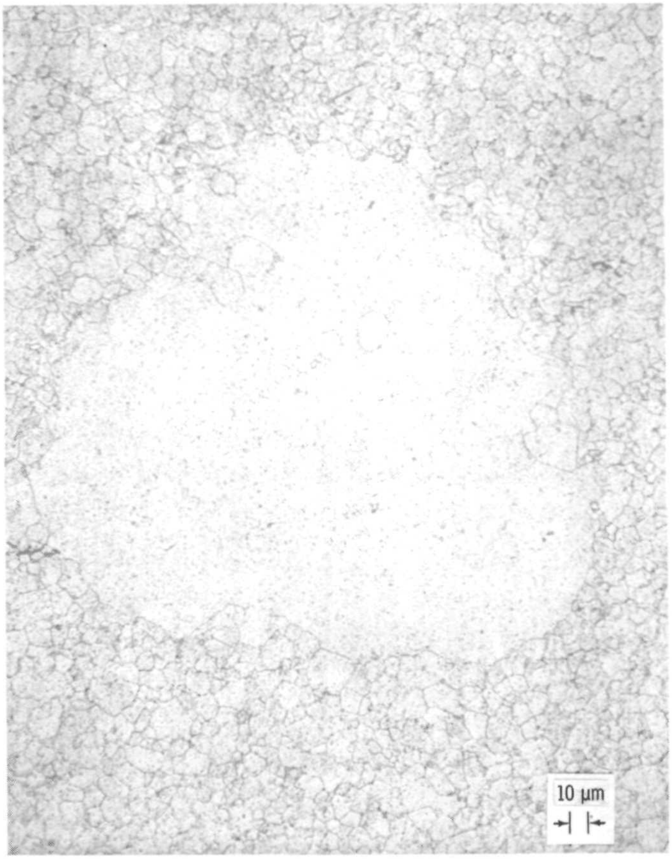

Figure 3. - Abnormal grain in specimen annealed 3 minutes at 1260° C and water-quenched.

Abnormal Growth Under Isothermal Conditions

Abnormal grain growth occurred upon annealing at or above 1190° C in alloy 713C produced from prealloyed powder. Figure 3 shows an abnormal grain grown in a specimen isothermally annealed 3 minutes at 1260° C and water-quenched. The average grain diameter of the fine-grained matrix is 9.0 micrometers (ASTM grain size 10.6), still the same as that of the as-extruded material. Thus, there was little normal coarsening of the matrix and no general primary recrystallization preceding the abnormal grain growth.

Growth of the equiaxed abnormal grains was extremely rapid once it had started but was preceded by an incubation period which is the rate-controlling step of the overall process. The incubation period increased with decreasing temperature. Abnormal grains were first seen in specimens annealed 3 minutes at 1260° C, 10 minutes at 1205° C, and 15 minutes at 1190° C. As will be explained in the next section, it is the fact that the incubation period increases with decreasing temperature that allows columnar growth in a temperature gradient.

The incubation period is probably related to the time required for the gamma prime phase to dissolve. The carbides in the alloy, however, seemed to offer little resistance to the growth of the large grains. The fine carbide distribution in the as-extruded material was little changed in the short annealing times required for the abnormal growth.

At both 1205° and 1260° C, the fine-grained matrix was almost entirely consumed by the abnormal grains in less than 1 minute after the incubation period. Although a few large grains formed in 15 minutes at 1190° C, their rate of growth was much slower than at the higher temperatures. Only about 20 percent of the fine-grained matrix had been consumed after 1 hour at 1190° C.

Once the fine-grained matrix was consumed, the size of the large grains was stable. The average diameter of the large grains grown at 1205° C was about 2 millimeters. For those grown at 1260° C, the average diameter was about 0.5 millimeter. The decreasing size with increasing temperature is typical of abnormal growth (ref. 11). This occurs because the density of abnormal grains formed during the incubation period increases with increasing temperature and allows less volume finally for each grain.

Abnormal Growth in Temperature Gradient

Columnar growth mechanism. - Specimens from the 1120°, 1160°, and 1190° C extrusions were annealed in the gradient furnace with the temperature at the back wall held constant. Shown in figure 4 is a specimen from the 1190° C extrusion. It is sectioned

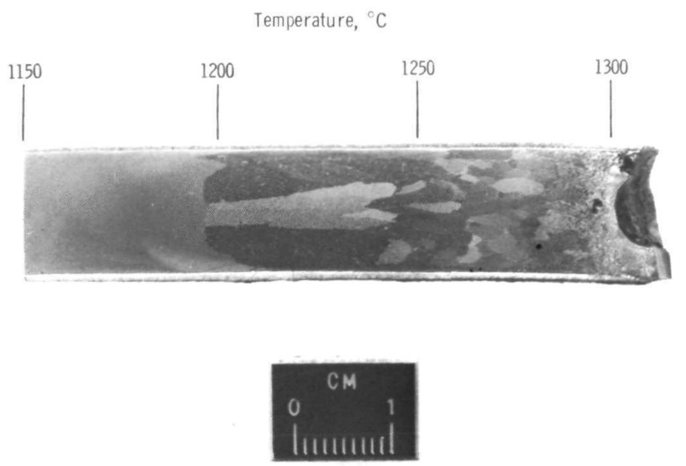

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Figure 4. - Gradient-annealed specimen of 713C sectioned longitudinally and etched to show grain structure developed.

and macroetched to show the grains developed. Approximate temperatures at four locations on the bar are indicated in the figure. Note the columnar grains which have grown from the 1250° C region to the 1190° C region. This same behavior was observed in the specimens of the 1120° and 1160° C extrusions.

The abnormal grains develop a columnar shape because of the increasing incubation period with decreasing temperature. In the highest temperature region abnormal grains form rapidly and, because of their great growth rate, are able to grow longer into the cooler region, where competing abnormal grains have not yet nucleated. Transverse growth of the grains is restricted by the presence of other large grains growing down the thermal gradient from the hottest region. Growth continues down the gradient until the temperature is too low to supply enough thermal energy to drive the process. Continued growth of the columnar grains to long lengths can be achieved, however, by continuously increasing the temperature in the region just ahead of the advancing grains to above 1190° C.

Texture studies. - Two specimens annealed in the gradient furnace with the back-wall temperature held constant at 1320° C were studied by X-ray diffraction. The columnar grains developed have a definite preferred orientation which can be simply described as a [110] fiber texture. That is, a [110] direction in each grain tends to lie near the axis of the extrusion, but there is no preferred orientation in the plane perpendicular to this axis. Figure 5 is an inverse pole figure showing the orientation of the extrusion axis within the stereographic triangle for each of the seven large columnar grains visible in the specimen in figure 4. A still sharper [110] fiber texture was observed for the columnar grains grown in a specimen from the 1.43-centimeter-diameter round extrusions produced for the study reported earlier (ref. 1). An inverse pole figure for the six grains that filled the circular cross section is presented in figure 6.

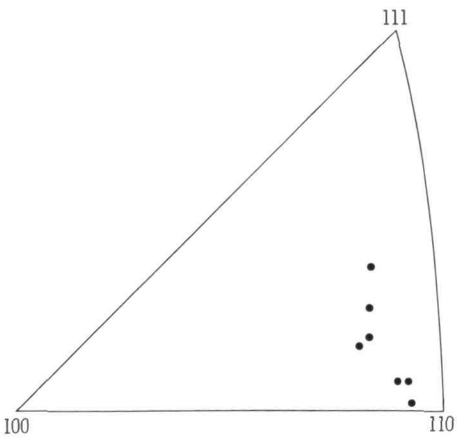

Figure 5. - Inverse pole figure showing orientation of extrusion direction in large columnar grains in specimen shown in figure 4.

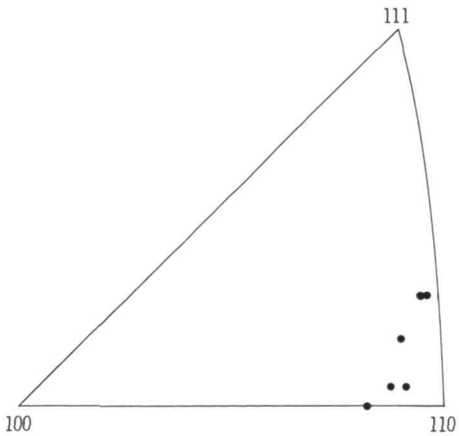

Figure 6. - Inverse pole figure showing orientation of extrusion direction in large columnar grains grown in round extrusion.

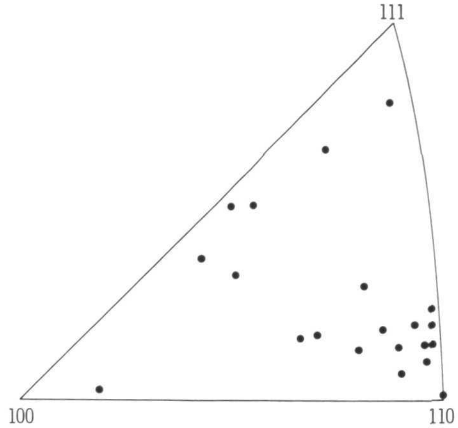

Figure 7. - Inverse pole figure showing orientation of extrusion direction in 21 equiaxed grains grown in specimen annealed isothermally 15 minutes at 1190° C.

The [110] fiber texture is only partly a result of the columnar growth of the abnormal grains in the temperature gradient. The [110] fiber texture is even present to some degree in the large equiaxed grains grown isothermally in the 713C powder product. Figure 7 shows the relative orientation of the extrusion direction in an inverse pole figure for 21 grains of approximately 70 that fill the rectangular cross section of a specimen isothermally annealed 15 minutes at 1190° C. It is apparent that there is a higher density of orientations near the (110) than in the other regions of the stereographic triangle.

However, it can be seen that the [110] fiber textures in the columnar-grained specimens (figs. 5 and 6) are sharper than that of the isothermally annealed specimen (fig. 7). This is a result of the columnar growth process. In the region of the specimen shown in figure 4 which experienced temperatures above 1240° C, there are a few abnormal grains which do not have [110] near the extrusion direction. However, these grains have been pinched out in the growth race down the temperature gradient. It appears that the abnormal grains with [110] nearly parallel to the direction of the maximum temperature gradient are able to grow fastest. Thus, after columnar growth has progressed a short distance only the grains with [110] near the direction of maximum gradient remain in the growth contest.

Why even the isothermally annealed specimens have a fairly well defined [110] fiber texture is not understood. This annealing texture does not seem to be derived from a deformation texture in the as-extruded material. Figure 8 is a back-reflection Laue pattern taken of a section of the as-extruded material cut parallel to the extrusion direction. Note there is no apparent radial variation in the intensity of the (111) or (200) Debye rings. Thus, if there is any texture in the as-extruded material, it is only very weak.

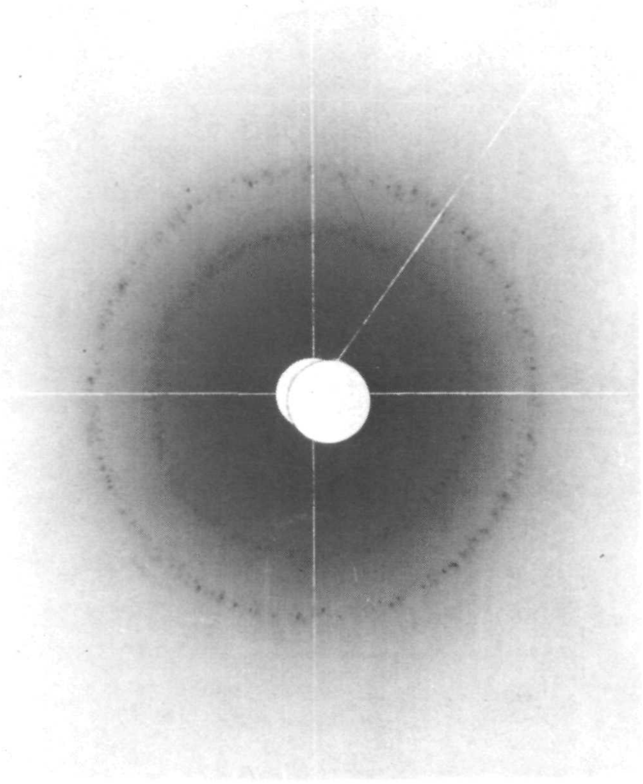

Figure 8. - Laue pattern from longitudinal section of as-extruded 713C. Extrusion direction is vertical; width direction is horizontal.

Whatever the mechanism of [110] fiber texture formation is, it was strong enough to produce this texture even when columnar growth was made to be transverse to the extrusion direction. Grains elongated in the width direction of the extrusion, as shown in figure 9, were produced when the temperature gradient was established across the width of the specimen. Even in this case, the grains have a weak [110] fiber texture about the extrusion axis. An inverse pole figure for the eight largest grains in the specimen in figure 9 is shown in figure 10.

The [110] fiber texture appears to come into being with the nucleation of the abnormal grains regardless of whether in a temperature gradient or isothermal conditions. During subsequent columnar growth transverse to the extrusion direction (the axis of the initial [110] fiber texture) any tendency for grains with [110] near the direction of the maximum temperature gradient to grow fastest was not able to alter the initial texture significantly. This was probably because there was only a low density of abnormal grains with [110] near the width direction, and thus few grains were able to receive the growth advantage due to the gradient. This situation is much different than that when

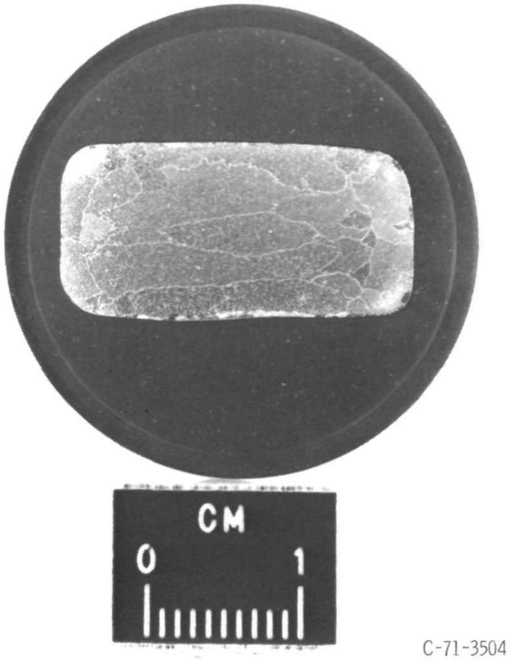

Figure 9. - Columnar grains grown in width direction of extrusion by imposing temperature gradient in that direction.

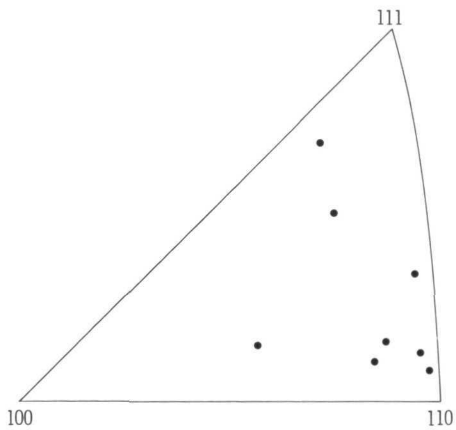

Figure 10. - Inverse pole figure showing orientation of extrusion direction in eight of the large grains in specimen shown in figure 9.

the gradient is applied parallel to the extrusion direction and the growth advantage adds to the initial tendency for [110] fiber texture formation.

CONCLUDING REMARKS

Gradient-annealing of extruded 713C powder can be used to produce continuous columnar grains such as might be achieved by directional solidification. However, a gradient furnace of the type used in this study could not be employed to produce continuous columnar grains of any length desired. It should be possible to do this, though, by drawing the extruded powder product through a furnace open at both ends. The maximum temperature in the furnace must be held below the solidus temperature of the alloy and there must be a suitably steep temperature gradient away from the hot zone. Also, a suitable rate of movement of the work piece through the furnace must be determined.

If columnar-grained alloy 713C, such as produced in this study, is to become a useful material for high-temperature service, it will be necessary to achieve uniformly good stress-rupture properties. It is felt that this result can be achieved if the average diameter of the columnar grains can be reduced from that achieved in the present work. The flexibility allowed by gradient annealing in an open-ended furnace, as just described, should aid in adjustment of the parameters to achieve the desired structure.

The possibility of growing columnar grains in powder-metallurgy extrusions is not limited to the alloy 713C composition. We have observed abnormal growth with several nickel-base superalloy powder products. Abnormal growth is expected to be possible in alloys more advanced than 713C as long as it is possible to dissolve the gamma prime phase at a temperature below the solidus.

SUMMARY OF RESULTS

The following results were obtained in an investigation to achieve high strength at elevated temperatures in alloy 713C produced by extrusion of prealloyed powder:

1. A microstructure of long columnar grains was produced in pieces of the extrusions by annealing in a temperature gradient. Abnormal grain growth occurred in this material at temperatures above about 1190° C.

2. The tensile strengths of the columnar-grained powder product are within ±5 percent of those of the as-cast alloy at 760° and 980° C. However, at room temperature the columnar-grained material is stronger than the as-cast alloy. The yield and ultimate tensile strengths were 841 and 1048 MN/m^2 compared with 738 and 848 MN/m^2 for the as-cast alloy.

3. Stress-rupture lives of the columnar-grained specimens at 760° and 980° C were from 17 to 376 percent of those of as-cast 713C under the same conditions. The large scatter is believed due to differences in strain accommodation among the large-diameter columnar grains in the various specimens.

4. Growth of the abnormal grains at temperatures greater than about 1205° C was very rapid and was preceded by an incubation time inversely related to the temperature.

5. Varying the extrusion temperature from 1120° to 1190° C had no apparent effect on the growth of abnormal grains during subsequent annealing.

6. Formation of the abnormal grains appeared to be permitted by dissolution of the gamma prime phase. Undissolved carbides provided little resistance to grain-boundary motion.

7. The large-grained microstructures produced by annealing the 713C powder extrusions had [110] fiber textures symmetric about the axis of the extrusions. This was true of the equiaxed grain structure produced isothermally as well as of the columnar grain structure grown in the temperature gradient.

8. The [110] fiber texture appeared to be sharpened in the process of the columnar grain growth along the length of the bar. The grains with [110] nearly parallel to the extrusion direction grew fastest and pinched out those with stray orientations.

9. No texture was observed in the as-extruded material, so the texture following abnormal grain growth could not be explained as being derived from a primary deformation texture.

Lewis Research Center,
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