MICROSTRUCTURE AND MECHANICAL PROPERTIES

OF ALLOY 718 PROCESSED BY RAPID SOLIDIFICATION

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Abstract

A study was performed on Alloy 718 to determine the influence of rapid solidification processing on the microstructural and mechanical properties of consolidated powders. The powders were produced by inert gas atomization (IGA) using helium, screened into five size groups, and hot extruded into bars. Tensile testing, with and without aging treatments, was performed at room temperature and 650°C. Powder particle size had no apparent influence on the tensile behavior. Very significant increases in yield stress, as well as some improvement in ductility, were observed for the extruded powders compared to their conventionally processed counterpart. The increase in yield stress is attributed to the inherent fine grain size observed in these Alloy 718 powders.

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Introduction

Alloy 718, a nickel-base superalloy that relies on intermetallic precipitates (γ' and γ'') in a fcc matrix (γ) for its strengthening, is conventionally processed as castings and wrought forms from the castings. A major concern associated with conventional processing of this alloy is composition and phase homogeneity. Processing the alloy from powders may greatly improve the homogeneity of the alloy products. If the powders are rapidly solidified, other benefits may also be realized, such as retention of fine microstructural features (1,2). Whether the fine microstructural features can be retained after exposures to high temperatures, i.e., through powder consolidation and subsequent thermal-mechanical processing, is a question that remains to be answered for this specific alloy. There is ample evidence suggesting that retention of a fine grain size in Alloy 718 will provide significant improvements in its mechanical properties (3-6). Although rapid solidification processing (RSP) produces a fine grain size for iron-base alloys (7,8), its effect on nickel-base alloys has not received a great deal of attention. In addition to a fine grain size, development of finer, yet more stable, γ' and γ'' precipitates may also be attained through RSP. This is highly desirable as submicron stability has the potential of extending the service temperature of Alloy 718.

A major drawback for effective application of RSP to alloys such as 718 is current lack of a fundamental understanding of the rapid crystallization behavior, particularly in terms of solute distribution, interaction, and control. This, in part, is due to an limited understanding of the liquid state, which is more complicated for alloy systems with a relatively large number of solute additions.

A study has been initiated on RSP of Alloy 718, using inert gas atomization (IGA) with helium, to evaluate the potential of this approach for enhancing microstructural fineness and stability and to establish correlations with mechanical behavior. For this study, the IGA processed powders were screened into various particle sizes before consolidation to establish whether the solidification rate of the particles has an influence on the microstructure and mechanical properties after powder consolidation. This paper reports the findings on the influence of powder particle size on the tensile properties of the consolidated powders before and after an aging heat treatment.

Experimental

The Alloy 718 melt stock used for the IGA powders was wrought bars obtained from the Department of Energy's reference heat (6,9). The composition of the alloy is as follows (in wt%): Ni-18.4Fe-18Cr-5Nb-2.7Mo-1.0Ti-0.6Al-0.3Mn-0.13Si-0.05C. In addition, 0.009 and 0.007 wt% levels for nitrogen and oxygen, respectively, were determined.

A laboratory-scale IGA system was used to atomize ~7 kg melts (10). Prior to melting, the chamber was evacuated and back filled with argon four times. The melt temperature prior to initiating atomization was 1630°C, which provided a 250 to 270°C superheat. The atomization used helium at a nozzle pressure of ~600 psi. The gas-molten metal interaction involved a close-couple configuration. In order to obtain sufficient quantities of powder for extrusion consolidation of a range of particle sizes, six atomization runs were performed, and the powders from each batch were mechanically screened. The specific particle size fractions for each of the six powder runs were blended (or mixed).

Characterization assessments were performed on the blended particle size fractions. These included particle size distributions, optical microscopy evaluations on polished and etched cross sections for several particle sizes, and entrained helium levels. Additional studies involving TEM/STEM examinations on the powder particles are also underway.

The powders were hot extruded into bars. The particle size fractions used for the extrusions were >10 to <30, >30 to <50, >50 to <75, >100 to <150, and >10 to <150 μm. For the last fraction, the median particle size was 45 μm, typical for the overall particle size distribution. The powders were hermetically sealed in mild steel containers after degassing (300°C). The preheated (1100°C) powder-filled billets were extruded to a ratio of 10.5 to 1, which produced ~2 cm diameter bars after cladding removal. The consolidated powders were found to be fine-grained and in a recrystallized microstructural condition. There was no evidence of porosity in the extrusions.

Isothermal heat treatments were performed on the five consolidated powders and their conventionally processed counterpart (CPC) for grain growth evaluation. Aging studies after an 1100°C - 1 h anneal were also performed using hardness determinations. TEM/STEM examinations were performed on the IGA >30 to <50 μm extruded powder and the CPC material after 1 h heat treatments at 1100°C followed by a water quench. In addition, specimens for the two materials were examined with TEM/STEM after receiving a 750°C - 4 h + 650°C - 16 h aging treatment.

Room temperature tensile tests were performed on the unaged and aged powder series and the CPC. Tests at 650°C were performed on the aged specimens.

Results

Powder Characteristics

The screened powder yields <150 μm (100 mesh) for the six batches ranged from 92 to 95%. The median particle size for the six batches was 50 μm, based on a cumulative weight fraction of 0.5. The particles were predominantly spherical, with some evidence of attached satellites on some of the larger particles. The morphology of the microstructure was primarily dendritic, with dendrite arm spacing (DAS) dependent on the particle size. The DAS ranged from 0.8 to 1.8 μm for particle sizes of >20 to <30 and >100 to 150 μm, respectively.

Entrained helium was detected in the powder. The amount entrained increased with increasing particle size, with 1.2 atomic parts per million (appm) observed for the largest particle size fraction (>100 to <150μm) used for powder consolidation. The entrained helium appears to be associated with porosity. The helium concentrations were retained during powder consolidation.

Composition

Composition was determined on each powder particle size after extrusion consolidation. No composition differences were observed for the various particle sizes except for the helium noted in the previous section and oxygen. The oxygen levels increased with decreasing particle size as shown in Figure 1. The oxygen behavior is attributed to oxide films on the particles acquired during IGA processing.

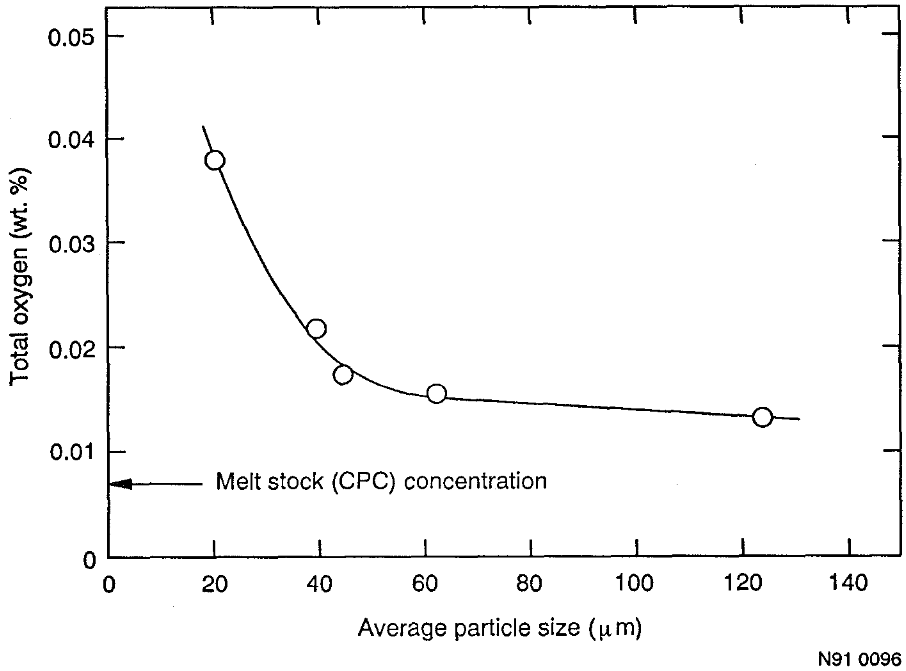

Figure 1 - Influence of powder particle size on the total oxygen content for extrusion consolidated Alloy 718 powders.

Heat Treatments and Microstructure

Since the powders were hot extruded at 1100°C with no subsequent cold working, the extruded materials were given a 1 h 1100°C solution anneal prior to aging. This heat treatment produced a fine grain size of 0.011 mm that was independent of the powder particle sizes used for consolidation. The same heat treatment produced a grain size of 0.13 mm for the CPC. Optical micrographs of the IGA processed and extruded Alloy 718 powder and the CPC after this 1 h heat treatment are shown in Figure 2. TEM examinations on the two specimens shown in Figure 2 revealed that the heat treatment had produced a solution anneal condition (See Figure 3).

The aging treatment for the extruded powder series and the CPC involved a duplex age from 1100°C. The specimens were furnace cooled to 750°C at 83°C/h, held at 750°C for 4 h, furnace cooled to 650°C where they were held for 16 h, and air cooled to room temperature. The hardnesses observed (in diamond pyramid units using a 200 g load) before (1100°C-1 h) and after the duplex aging are shown in Table I for the powder series and the CPC. The powder particle size used for the extrusions has no obvious influence on hardness before or after aging.

TEM examinations were performed on the aged >30 to <50 μm extruded powder and the CPC. Typical TEM photographs are shown in Figure 4.

Tensile Properties

The room temperature tensile properties (0.2% offset yield stress, ultimate stress, total elongation, and reduction in area) for the unaged and aged Alloy 718 specimen series are shown in Figure 5. The stress results (Figure 5a) are independent of powder particle size in both the unaged and aged conditions. CPC values are shown along the ordinates of Figure 5 for comparison. The yield stress for the IGA extruded powder series is about

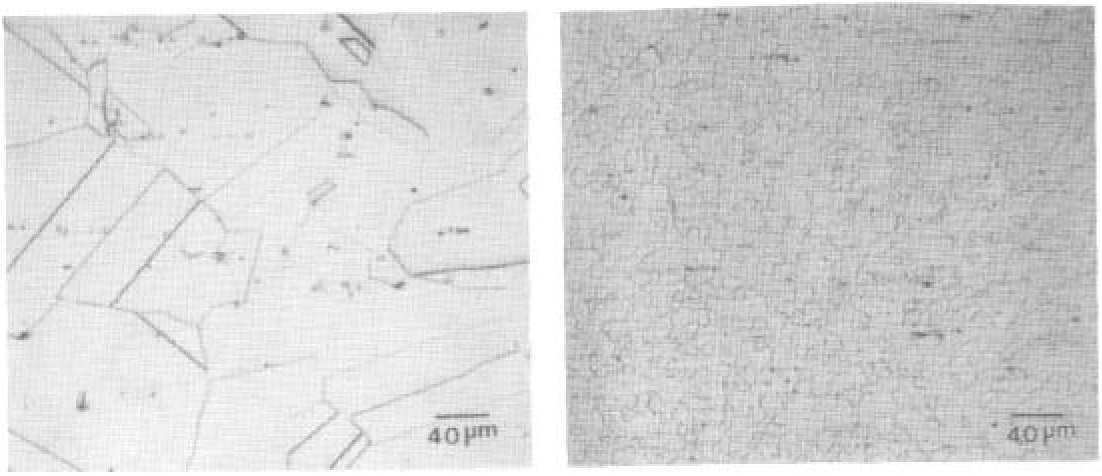

Figure 2 - Optical micrographs for Alloy 718 CPC (left) and IGA extruded powder (>30 to <50 µm, right) after 1100°C-1 h heat treatment.

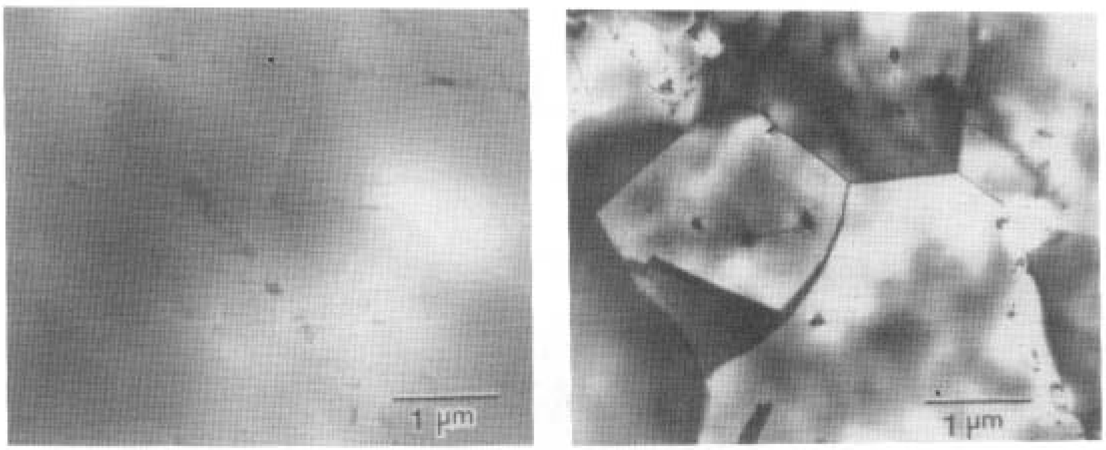

Figure 3 - TEM photographs of Alloy 718 after an 1100°C-1 h heat treatment: CPC material (left) and extruded powder with a 30 to 50 µm particle size (right).

Table I. Hardness of Alloy 718 Before and After Aging

Material	Hardness (DPH)	
	Unaged	Aged
IGA (10-30 µm)	253	459
IGA (30-50 µm)	249	467
IGA (50-75 µm)	242	465
IGA (100-150 µm)	242	461
IGA (10-150 µm)	254	462
CPC	188	462

150 MPa higher than that of the CPC after equivalent heat treatments for both the unaged and aged conditions. The ultimate tensile stress for the unaged IGA series is also about 150 MPa higher than that of the CPC; however, aging

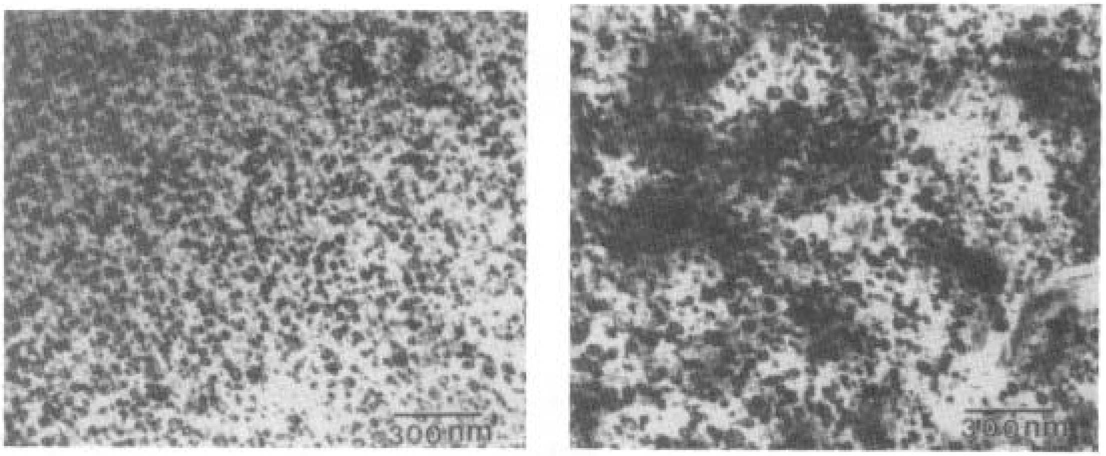

Figure 4 - TEM photographs of Alloy 718 after 1100°C-1 h solution annealed followed by a 750°C-4 h and 650°C-16 h aging treatment: CPC material (left) and extruded powder with a >30 to <50 µm particle size (right).

reduces the difference to about 30 MPa. The ductility represented by total elongation and reduction in area (Figure 5b) shows no obvious dependence on particle size before or after aging. Except for the total elongations associated with the unaged conditions, the ductility appears to be comparable at room temperature for the IGA extruded powder series and the CPC material.

The tensile test results at 650°C for the IGA extruded powder series and the CPC are shown in Figure 6 for aged specimens. Similar to the results shown in Figure 5, there appears to be no influence from powder particle size for the IGA extruded series. The significant increase in yield stress for the IGA series is retained at this temperature; however, the ultimate stress values are comparable for the IGA and CPC materials. Total elongation and reduction in area results, shown in Figure 6b, indicate that powder particle size does not significantly affect ductility. A marked improvement in ductility is apparent for the IGA Alloy 718 extruded powders compared to the CPC material.

Discussion

IGA processing of Alloy 718 powders, using a close-coupling configuration with helium as the atomizing gas, produced high quality powders. The cooling rate associated with the IGA processing is difficult to determine; however, based on previous studies of Alloy 718 (11,12), the solidification rate can be estimated from the dendrite arm spacing (DAS) determinations. An empirical expression such as the following can provide an estimate of the solidification rate (11):

$$R = (A/d)^{-n},$$

where **R** is the solidification rate (°K/S) and **d** represents the dendrite arm spacing (µm). **A** and n are empirical constants, for Alloy 718 these have been reported as **A**=141 and n=0.40 (11). Measurements of DAS for the IGA Alloy 718 powders showed values of 0.8 and 1.8, for particles with average diameters of 25 and 125 µm, respectively. The solidification rates calculated from these DAS values are $4x10^5$ and $5x10^4$ °K/S, respectively, which indicates that rapid solidification did occur.

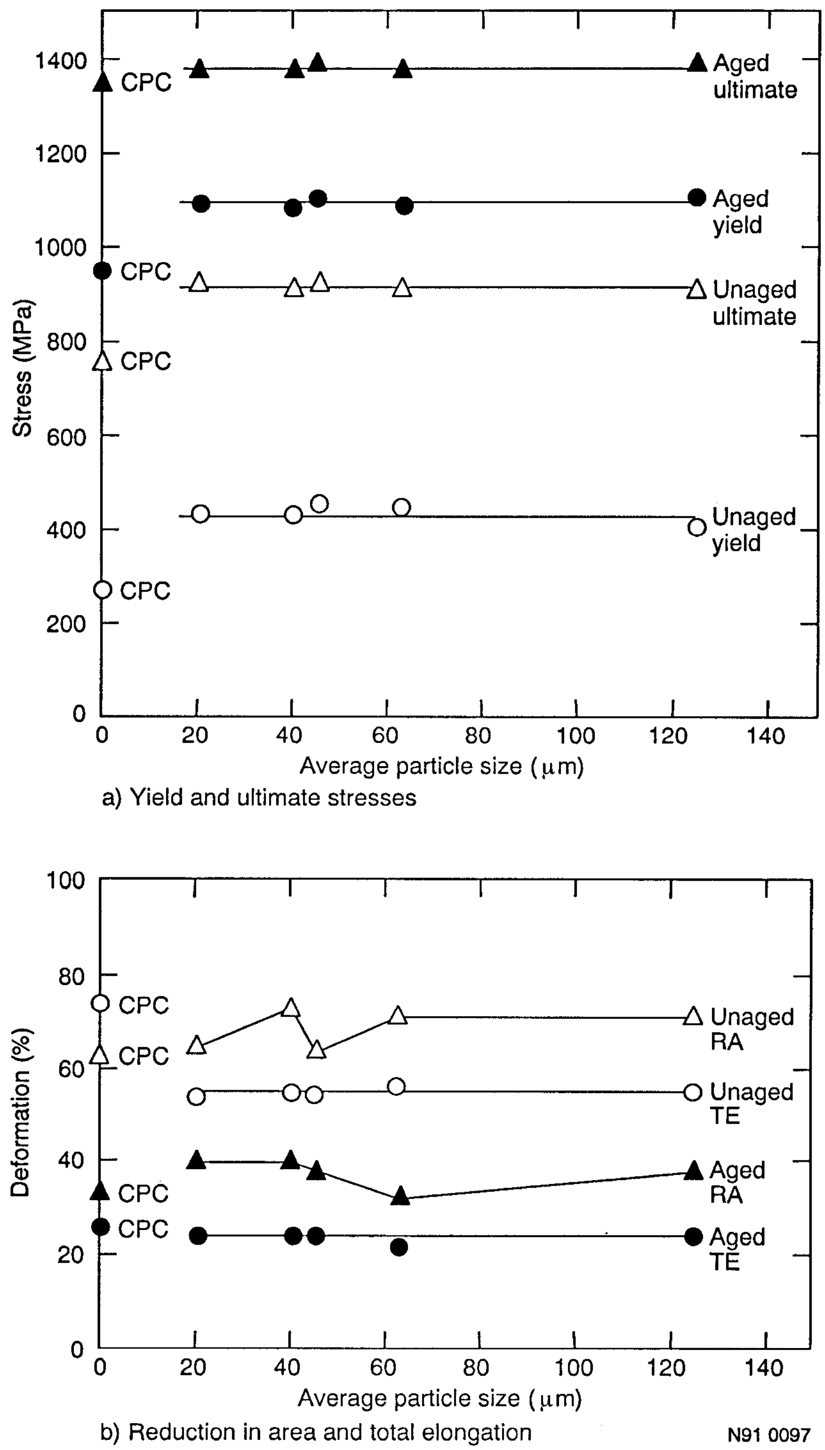

Figure 5 - Room temperature tensile properties for unaged and aged IGA Alloy 718 powders after extrusion consolidation and CPC comparisons.

Further characterization studies on the IGA Alloy 718 powders, now underway, involve TEM/STEM examinations. These examinations are intended to establish the influence of particle size, hence solidification rate, on microchemical segregation and submicron defects, e.g. dislocations.

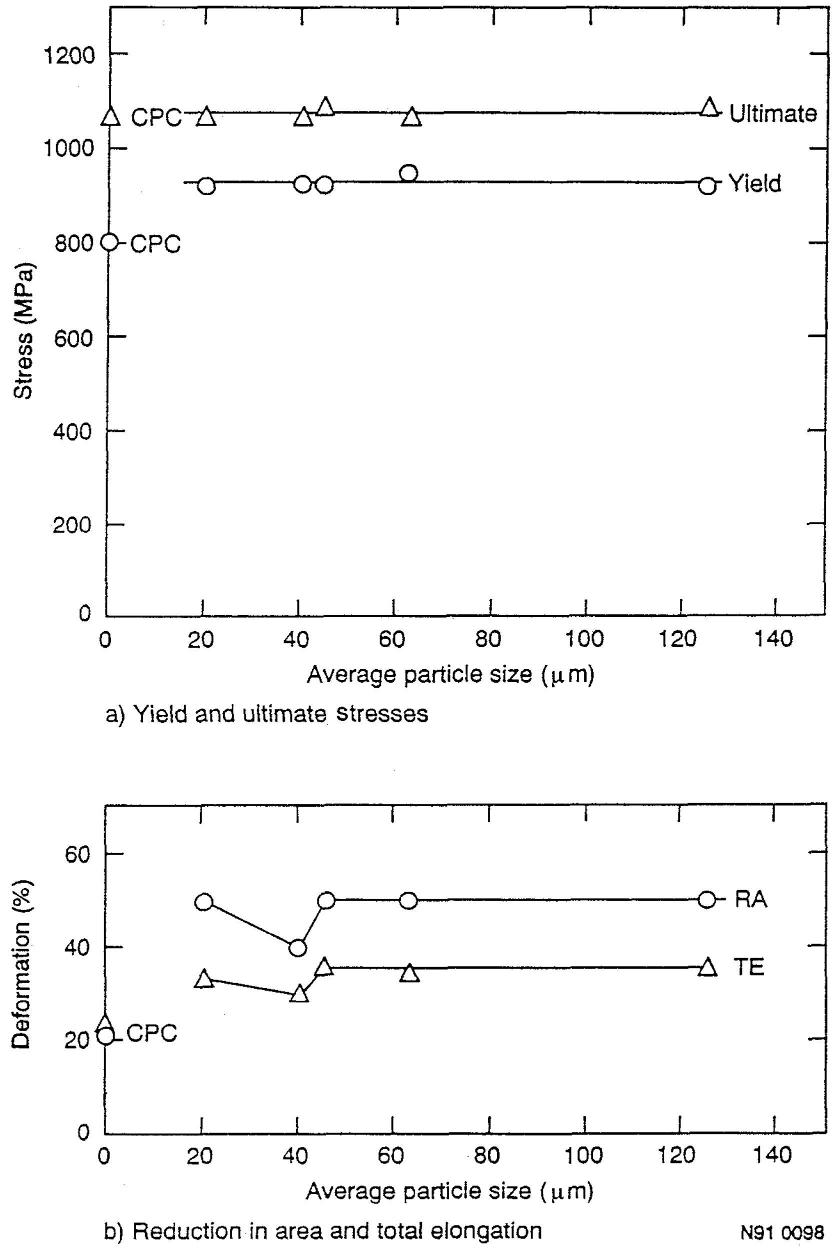

Figure 6 - Influence of powder particle size on the tensile properties at 650°C for IGA Alloy 718 extruded powders (with CPC comparisons).

The influence of particle size on macroscopic composition had no surprises except for the oxygen behavior. Although not covered in this paper, an analysis of the results of Figure 1 shows that the oxygen increase for the powders compared to the melt stock (CPC material) is due to oxide films on the particles (13). Furthermore, the oxide film thickness, calculated to be 5.3 nm, is independent of particle size (13). The oxygen entrained inside the particles, i.e., ~0.007 wt%, appears to be that associated with the melt stock material.

The two particle size dependent characteristics (i.e., DAS, which indicates solidification rate effects, and the amount oxide film from the particles that potentially could be dispersed during extrusion consolidation) would initially be suspects for influencing the grain size and tensile properties for the consolidated Alloy 718 powders. This study has shown that there is no apparent influence from the powder particle size on these two properties. The very fine, and powder particle size independent, grain size observed after the 1100°C-1 h heat treatment suggests that a pinning and/or a stabilization mechanism is operating at the grain boundaries. However, the study has not resolved the reason for the small grain sizes observed in the extruded powder series. It was shown in Figure 3b that the extruded Alloy 718 powder (>30 to <50 μm powder particle size) contains a fairly high population of niobium-rich particles. A common interpretation would tend to attribute the fine grain size to a pinning influence from the residual niobium-rich dispersions. However, these dispersed particles appear to reside predominately in the grain interiors; there is very little evidence of their presence on grain boundaries. In addition, supporting evidence from a very large data base obtained on RSP iron-base alloys suggests that a mechanism other than dispersion pinning is responsible for the grain sizes and their retardation of growth (8). This mechanism has not yet been identified; however, there seems to be considerable evidence that oxygen is playing an important role in stabilizing the grain boundaries (8).

The tensile strengths observed for the unaged and aged extruded Alloy 718 powders and the independence from powder particle size provide fairly clear evidence that the fine grain size is responsible for the increase in strength. The tensile behavior shows no evidence that the matrix strengthening from the γ' and γ'' precipitates was influenced by the RSP. The preliminary TEM observations indicate that there are some differences in the submicron microstructural features between the IGA and CPC Alloy 718 materials. Forthcoming examinations should identify these differences. In addition, more mechanical property tests are underway, including creep and strain-controlled, low cycle fatigue.

Conclusions

Inert gas atomization of Alloy 718, using helium, produced high quality powders having fine dendritic microstructures with arm spacings that decreased with decreasing particle size. The powders for five different particle sizes were consolidated by hot extrusion. Characterization of the consolidated materials showed a powder particle dependence on oxygen content that decreased with increasing particle size and is due to oxide films from the particle surfaces that were acquired during powder processing. A fine grain size that was independent of powder particle size was observed after 1100°C-1 h heat treatment for the IGA extruded powders. Tensile properties for the IGA extruded powders before and after a duplex aging treatment were independent of powder particle size. Significant increases in tensile stresses were observed for the IGA extruded powder series compared to their conventionally processed counterpart. The increases in tensile stresses are attributed to the fine grain size associated with the IGA Alloy 718 extruded powders.

Acknowledgments

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