HOMOGENIZATION AND THERMOMECHANICAL PROCESSING

OF CAST ALLOY 718

Paul J. DiConza*, Ronald R. Biederman* and
Rishi P. Singh**

*Worcester Polytechnic Institute, Worcester, MA 01609
**Wyman-Gordon Company, North Grafton, MA 01536-8001

Abstract

The microstructural response displayed by a cast and homogenized Alloy 718 ingot during subsequent homogenization and elevated temperature deformation treatments was investigated to better understand the effects of chemical segregation on the resulting microstructure of converted billet. In particular, secondary precipitation and its effects upon recrystallization behavior were studied. The study contained three major areas of investigation. First, straight thermal treatments in the temperature range of 1650°F to 2150°F were performed over a range of times. An automated EDXS technique was used to measure reductions in segregation in the heat treated material.

The second area involved isothermal deformation over the same temperature range as above. Cylindrical specimens were deformed in compression at a strain rate of 0.1 sec^{-1}, to various strains ranging from 5% to 20% engineering strain. Finally, compression was combined with post-deformation thermal treatment. Microstructural characterization was carried out using optical, scanning and transmission electron microscopy.

It has been shown that nucleation of strain free grains first occurs at the carbide clusters/high angle grain boundaries. Recrystallization was rapid above the δ solvus, but inhibited at lower temperatures. It appears to be due to precipitation of secondary phases. In addition, the effects of prior segregation were observed in the form of nonuniform phase precipitation, which occurred in many of the deformed samples. This was attributed to a concentration of niobium in the prior boundaries. Implications of these results are discussed in light of uniform billet conversion.

Superalloys 718, 625 and Various Derivatives
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Introduction

Alloy 718 continues to lead the other superalloys in terms of production because its excellent workability and mechanical properties lend themselves to a wide variety of applications. As the limits of these properties are approached in today's advancing technologies, there has developed an interest in obtaining precise control of the Alloy 718 microstructure during all phases of processing. To achieve optimum properties in an Alloy 718 part, control of such factors as chemical segregation, precipitate size and morphology, and grain size becomes very important in every phase of production, from the initial creation of ingot through the final forging operations [1,2].

The objective of this study was to document the flow response of ingot Alloy 718 to thermal/mechanical processing in the temperature range between 1650°F and 2150°F and to characterize microstructural changes that occur. This temperature range is important to vital processes (homogenization and cogging) used to convert the inherently inhomogeneous 718 ingot to a uniform billet suitable for forging. This study investigated the effects of various microstructural responses, such as precipitation, recovery, recrystallization, and grain growth.

Experimental Procedure

Slices from a triple-melted Alloy 718 ingot which had undergone a proprietary homogenization treatment were used as a starting condition, and all samples were taken from the columnar oriented section of the slices for repeatability.

After characterization of the starting condition, six different homogenization treatments were studied. Heat treatments of 8, 24, and 48 hours were performed at temperatures ranging from 1650°F to 2150°F in intervals of 100°F. This wide range of temperatures was selected to span both above and below the nominal δ solvus temperature of 1825°F [3]. For characterization, the samples had 0.060 in. machined from a surface to avoid surface effects such as oxidation [4].

For the compression testing, cylindrical test samples were machined from the ingot to final dimensions 0.4 in. diameter x 0.6 in. height. All compression tests were done on a servohydraulic testing system equipped with a three-zone split-type furnace. Upon reaching temperature, samples were held for at least 10 minutes before deformation to equilibrate sample temperature. Strain rate was kept constant at 0.1 sec^{-1}. A lubricant was used to minimize barrelling effects.

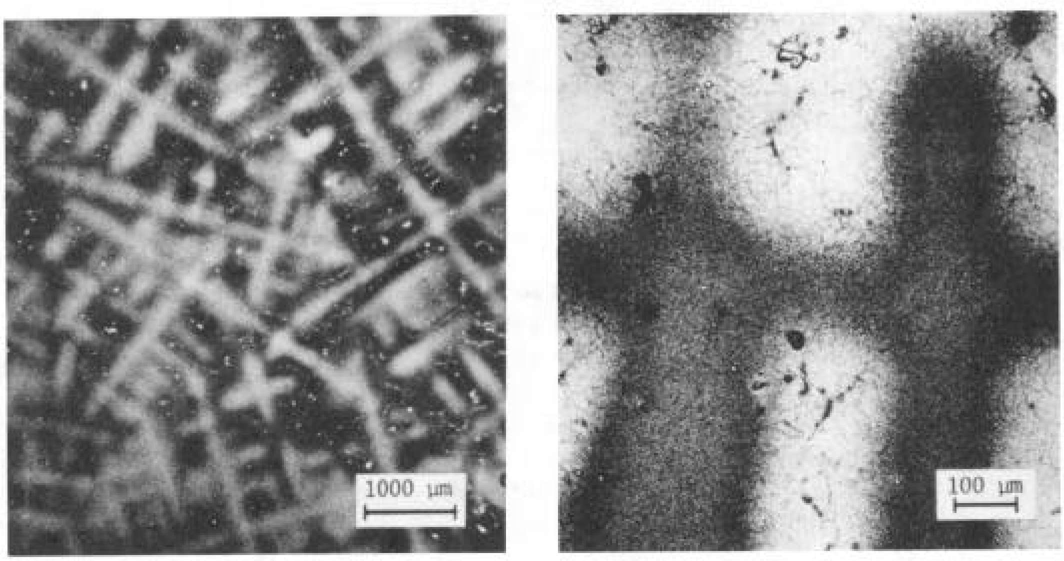
After completion of the specified process, the samples were sectioned longitudinally for characterization. Post-deformation heat treatments were carried out in the same arrangement used for the straight thermal treatments.

Optical and electron microscopy were employed for the purpose of documenting the Alloy 718 microstructures. The dendritic pattern of the starting condition was etched with a 50-50 solution of HCl and water with a volumetric 4-8% hydrogen peroxide addition. Compression samples were electropolished and etched with a 10% HCl-methanol solution to show grain boundaries and δ phase. Selected samples were prepared for TEM using the twin-jet electropolishing technique to manufacture thin foils [5].

To quantify chemical segregation in the untreated state as well as in the thermally treated state, a technique was developed which utilized EDXS to determine the variation of alloy composition as a function of position in the sample. EDXS spectra were collected in discrete spots along a linear array and were analyzed quantitatively using theoretical standards. The distance between spots was approximately 300 μm, and the beam spot was approximately 500Å in diameter. The quantitative analysis performed for each spot (30 spots per specimen) was stored in spreadsheet form, where it was analyzed to provide mean compositions and standard deviations for each element. The standard deviation was used as a quantitative measure of segregation, where a high standard deviation about the mean composition would indicate a segregated condition. This technique has previously been used with success to measure segregation in cast superalloys [4].

Results and Discussion

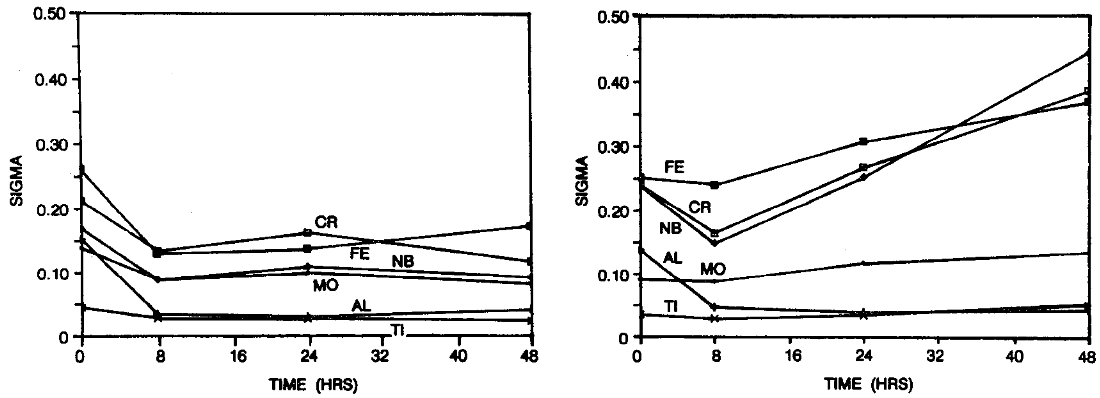
The dendritic structure of the as-received material is shown in Figure 1. Large amounts of interdendritic phases were present in the matrix. The concern was that the homogenization treatment performed prior to receipt of the ingot had failed to dissolve Laves phase, which would seriously degrade mechanical properties during subsequent deformation [6]. One of the most important goals of homogenization of this alloy is the removal of all of this phase [3]. Analysis showed, however, that the interdendritic phases



A. Dendrites Light B. Dendrites Dark

Figure 1 - Dendritic Structure of the As-Received Alloy 718.

in this ingot were carbides and nitrides, which cannot be dissolved by typical homogenization. Further evidence as to the relative homogeneity of the as-received ingot was given by the low standard deviations obtained through spot-by-spot analysis. The highest deviation measured was below 0.5% by weight. These two observations indicated that the as-received condition was suitable for deformation testing.



A. At 2150°F.   B. At 1650°F.

Figure 2 - Isothermal Homogenization of Ingot Alloy 718, Compositional Standard Deviation vs Time.

Two separate trends were observed in the thermally treated samples, as represented in Figure 2. When the heat treatment temperature was above the δ solvus, as in Figure 2a, the amount of segregation decreased after 8 hours treatment, with little subsequent improvement. Figure 2b shows that the lower temperature response was radically different, with segregation of Cr, Fe, and Nb increasing as time increases. This effect is due to δ precipitation during treatment. Because it is comparatively rich in Nb and lean in Cr and Fe relative to the matrix, δ will cause a large spread in the chemistry data to occur when it appears in the microstructure. The spread will increase with the amount of δ phase in the matrix.

Figure 3 shows true stress-true strain curves at 1650°F, 1850°F and 2050°F. The yield stress displayed typical inverse dependence with temperature. At lower temperatures i.e., 1650°F and 1850°F, it shows significant work hardening. At 2050°F, work hardening is almost negligible. It is interesting to note here that these curves don't show any cyclic stress-strain behavior as has been reported for fine grain material [7]. The cyclic stress-strain behavior represents the region of work hardening and softening due to dynamic recrystallization. Absence of cyclic stress-strain curves suggest that dynamic recrystallization under these test conditions has not started.

At 2050°F, serrated flow or yield drop was also observed. Serrated flow at 1650°F and 1850°F was not observed. This is in agreement with other studies [8,9]. Strain rate and temperature dependence of serrated flow in Alloy 718 was not a concern of this study.

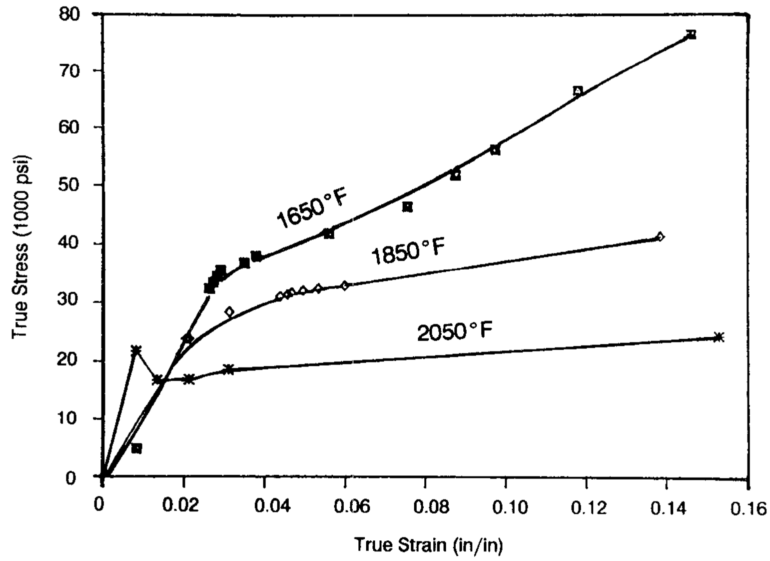


Figure 3 - Isothermal Compression Response for Homogenized Alloy 718.

The microstructural response of the isothermally deformed samples was strongly temperature dependent. Deformation at the lower temperatures (Figures 4a and 4b) yielded only deformed grain boundaries, pinned by precipitates. Figure 4c shows that the high temperature sample, free of any secondary precipitation, responded with recrystallization localized at carbide clusters. The equiaxed nature of the newly formed grains makes a case supporting their origin was through static, rather than dynamic, recrystallization. At such a high temperature, the 10 second (approximately) delay between deformation and quenching is sufficient to allow nucleation for static recrystallization [10].

Figure 5 shows that while both the samples deformed 12% and 20% at 1950°F had precipitate free matrices, the more heavily deformed specimen showed a continuous "necklace" of small recrystallized grains running along the prior grain boundaries. Again, there is evidence that recrystallization began at carbide clusters. This is most likely due to a stress concentration factor at the carbide/matrix interface, which would locally increase the driving force for recrystallization in the regions immediately surrounding such areas [11]. After deformation at 1950°F heat treatments at 1650°F caused little progression in the recrystallization process. Figure 6 shows that the precipitates formed during treatment seemed to pin the existing structure to its original form. The TEM photo in Figure 6c particularly shows the pinning action of δ and γ'' on dislocations.

Heat treatment at 1800°F resulted in a slowly developing recrystallization response. Table I shows that the more heavily strained sample responded more quickly, as would be expected. Some δ precipitation was observed in the microstructure resulting from this treatment. The precipitation seemed to occur along prior high angle grain boundaries and not along newly formed recrystallization boundaries. The δ followed along regions corresponding to prior boundaries, indicating that the precipitation was responding to a chemistry difference localized in these areas. More will be said concerning this effect.

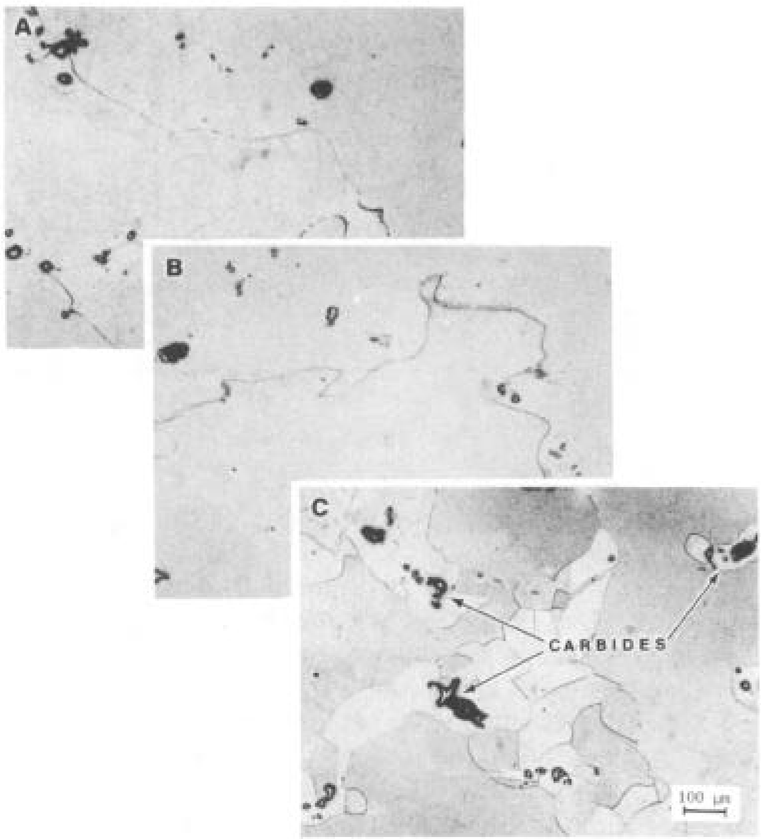
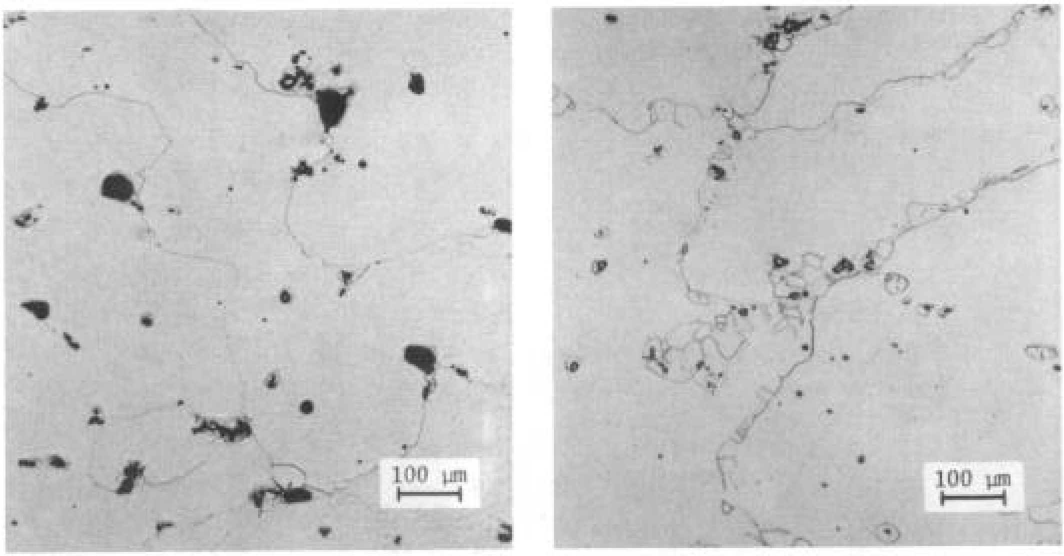


Figure 4 - Microstructure Resulting from 20% Isothermal Deformation at:
A. 1650°F., B. 1850°F., C. 2050°F.

Rapid recrystallization and grain growth were observed in the samples heated at 1950°F. Unhindered by precipitates, the recrystallization was complete after only 30 minutes in both samples.

Although the samples deformed at 1750°F showed very little initial recrystallization, the subsequent heat treatments triggered very rapid responses due to the high temperatures. Only the 1850°F treatment showed any sort of delay, due in part to the pinning action of a residual amount of δ phase present in prior boundaries.

Table I presents an overview of the recrystallization behavior for samples which underwent a post-deformation heat treatment. Note the rapid response for those samples heat treated above the δ solvus.



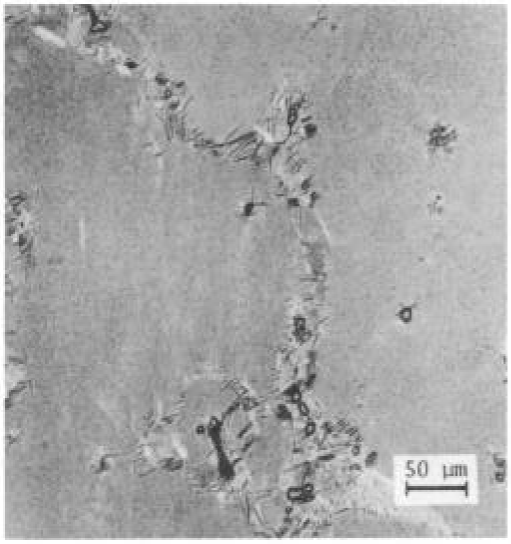
A. 12% Deformed. B. 20% Deformed.

Figure 5 - Isothermal Deformation Behavior at 1950°F.

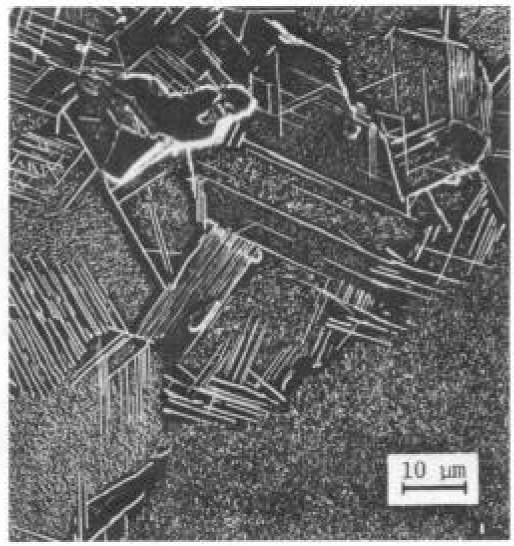
The Behavior of δ

The behavior of δ in the warm working temperature regime is an important concern during forging operations. In particular, control of grain size depends upon control of the δ size and morphology. The importance of this was observed in this study, where samples deformed and heat treated above the δ solvus quickly developed coarse grains, while lower temperatures resulted in a finer structure. Some conditions generated by this investigation resulted in interesting δ phase behavior, which merits further discussion.

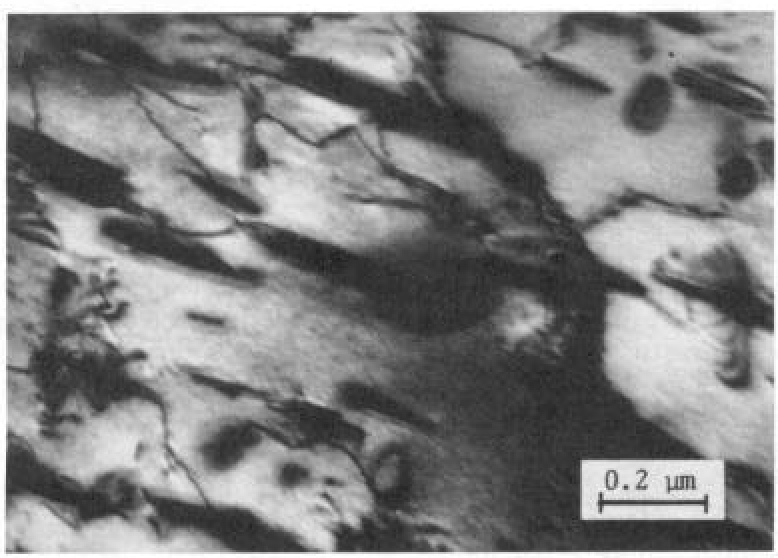
Under certain conditions, the δ precipitated and grew along a prior boundary, rather than the newly formed grain boundaries Figure 7. The explanation may be provided by the characteristics of Nb diffusion prior to the actual deformation. Figure 8 outlines the mechanism. Prior to deformation above the δ solvus temperature, all of the Nb-rich secondary phases dissolve as the temperature is equilibrated in the section. This frees a large amount of Nb, and since the grain boundaries and subboundaries were sites of concentrated precipitates in the prior condition, they will now be very rich in Nb. This Nb is free to diffuse via "pipe diffusion" along the boundaries much more readily than through the lattice. Upon deformation and heat treatment, when recrystallization occurs, the high Nb concentration will foster the formation of δ [3], because it is very likely that the new grain boundaries will correspond to regions which are relatively depleted of Nb. The regions which are more favorable for δ formation remain in the prior boundary areas, due to the chemistry of the region.



A. Optical Photomicrograph,
20% Strain, 8 Hrs. Age.



B. SEM Photomicrograph,
20% Strain, 8 Hrs. Age.



C. TEM Photomicrograph of Dislocation Pinning by δ and γ'' Particles.

Figure 6 - Isothermally Deformed at 1950°F., Isothermally Aged at 1650°F.

Table I - Recrystallization Response of Deformed and Aged Samples.

PERCENT RECRYSTALLIZED

1950 F (1066 C)

Deformed	12%			20%		
Hours	0.5	2	8	0.5	2	8
1650 F	5	5	5	10	15	15
1800 F	10	15	25	25	40	98
1950 F	95	100	100	100	100	100

1750 F (954 C)

Deformed	12%			20%		
Hours	0.5	2	8	0.5	2	8
1850 F	20	25	100	35	50	100
1950 F	75	100	100	100	100	100
2000 F	100	100	100	100	100	100

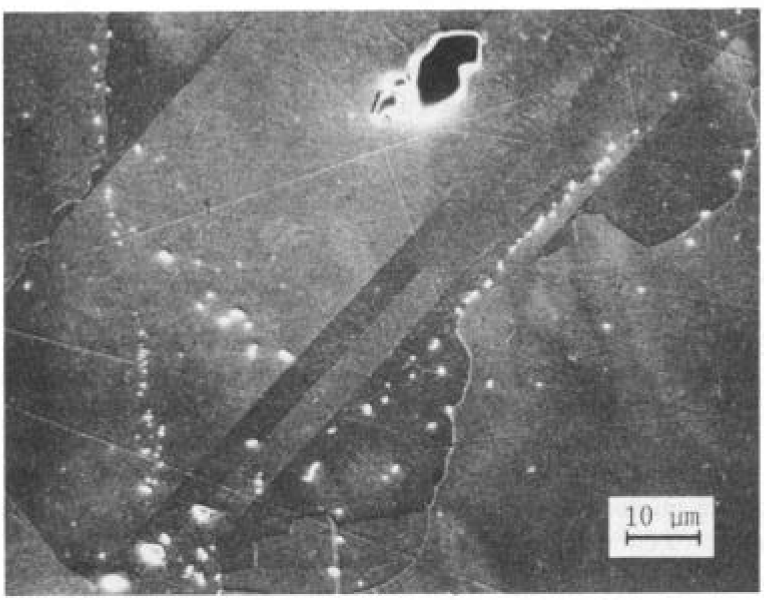
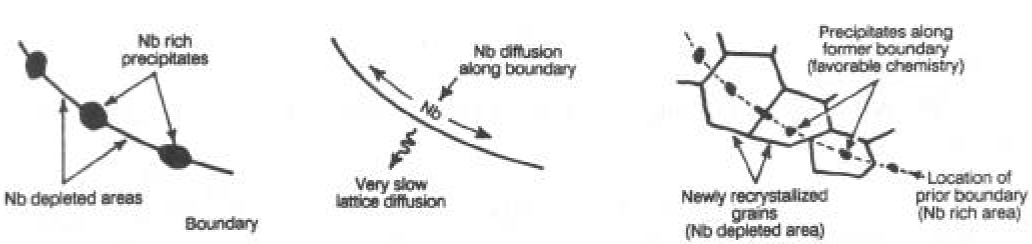


Figure 7 - 20% Deformation at 1950°F, Aged for ½ Hrs. at 1800°F.



A. As-Received Condition **B. At Temperature, Prior to Deformation** **C. Aging Response, After Deformation**

Figure 8 - Schematic of Nonuniform Precipitation.

The observations reported reinforce the idea that a one step deformation/homogenization treatment is insufficient to achieve a desired level of microstructural uniformity. The ultimate goal of TMP research into Alloy 718 is to utilize the grain size controlling properties of δ while simultaneously providing a sufficient amount of Nb for strengthening precipitates. This study has isolated various conditions which can exist during forging operations, and the results indicate that iterations of homogenization, deformation at various temperatures, and aging treatments all need to be used to achieve final processing goals. Of course, this idea is well known and utilized by today's manufacturers, but each step in the process and its effects on Alloy 718 microstructure must be more fully understood if alloy optimization is ever to come about.

Conclusions

This study focused on the microstructural response of an Alloy 718 ingot to thermal and mechanical treatments. The following conclusions can be drawn:

1. The small amounts of measured segregation in the as-received condition, in addition to the small improvement gained as a result of homogenization heat treatments, and the absence of Laves phase, indicate that the proprietary heat treatment performed after casting significantly reduced segregation from the as-cast condition.

2. Recrystallization initiated at carbide clusters and continued at high angle boundaries, is probably due to stress concentrations in the matrix adjacent to these particles.

3. Recrystallization at temperatures below 1850°F was delayed, and sometimes prevented, by the pinning action of precipitates (mostly δ). Above this temperature, recrystallization was rapid, due to the absence of these precipitates.

4. Residual segregation resulted in non-uniform precipitation of secondary phases, and ineffective microstructural control, in many of the deformed specimens. This was attributed to concentrations of Nb in prior boundaries.

5. The initial distribution of Nb in Alloy 718 ingot has direct effects upon the microstructure of the converted billet, and it must be more carefully controlled to optimize Alloy 718 properties.

References

1. N. A. Wilkinson, "Forging of 718 - The Importance of TMP," Superalloy 718, TMS, Warrendale, PA, 1989.

2. T. E. Howson, and W. H. Couts, "Thermomechanical Processing of Superalloys", Superalloys, Supercomposites, & Superceramics, edited by J. K. Tien & T. Caufield, Academic Press, N.Y., pg. 183, (1989).

3. J. F. Radavich, "The Physical Metallurgy of Cast and Wrought Alloy 718", Superalloy 718, TMS, Warrendale, PA, 1989.

4. C. B. Bunis, "Characterization of Segregation and Homogenization in Cast Udimet 720" (Master of Science, Thesis, WPI, 1990).

5. P. J. DiConza, R. R. Biederman, and J. L. Burger, "Microstructural Characterization of Alloy 718", Microstructural Science, Volume 18, Editors: T. A. Place et al. IMS, Columbus, Ohio and ASM International, Materials Park, Ohio, 1990.

6. E. E. Brown, and R. Muzyka, "Nickel-Iron Alloys", Superalloys II, John Wiley and Sons, New York, 1987.

7. T. Sakai, and J. J. Jonas, "Dynamic Recrystallization Mechanical and Microstructural Considerations", Acta Metall., Vol. 32, #2, pp. 189-209, (1984).

8. M. J. Weis, "The Hot Deformation Behavior of As-Cast Alloy 718" (Master of Science Thesis, #T - 3382, Colorado School of Mines, 1987).

9. A. A. Guimaraes, and J. J. Jonas, "Recrystallization and Aging Effects Associated with the High Temperature Deformation of Waspaloy and Inconel 718", Met. Trans. A, Vol. 12A, Sept. 1981, pp. 1655-1666.

10. J. L. Burger, R. R. Biederman, and W. H. Couts, "The Effect of Starting Condition on the Aging Response of As-Forged Inconel 718", Superalloy 718, Metallurgy and Applications, Edited by E. A. Loria, TMS, Warrendale, Pa., (1989), pp. 207-218.

11. F. J. Humphreys, "The Nucleation of Recrystallization at Second Phase Particles in Deformation Aluminum", Acta Metall., Vol. 25, (1977), pp. 1323-1344.