FACTORS AFFECTING DELTA PHASE PRECIPITATION AND GROWTH AT HOT WORK TEMPERATURES FOR DIRECT AGED INCO 718

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Abstract

The billet conversion and forge practise of DA 718 employs temperatures in the range where delta phase may precipitate, may agglomerate, or may redissolve depending on the prior microstructure, time at heat, and chemical homogeneity. The need for microstructural uniformity dictates a need for a positive understanding of the factors influencing this equilibria.

One bar of DA 718 billet was upset to obtain a neutral fine grain microstructure, thermally treated to form five different starting structures, and then exposed for varying times to temperatures marginal for delta precipitation or solution. The delta phase is a very effective barrier to static recrystallization at temperatures at or below 990°C. Above 990°C, delta particles solution and rapid grain growth occurs. Plates of delta phase may co-exist with plates of gamma" at temperatures as low as 870°C, but heating to 990°C will dissolve gamma" and spheroidize the delta.

The temperature to which the material is exposed is more critical than the cooling or heating rate of the material. Overheating the material promotes large grains and grain boundary precipitation of delta phase which may result in embrittlement.

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Introduction

Progress in Inconel 718* disk manufacturing has reached the point where it is now necessary to control the grain size and microstructures of the billet. For reasons of tensile strength, ductility, crack initiation, and sonic penetrability, a fine billet grain size is sought. However, for reasons of crack propagation and creep resistance, the grain size should not be too fine.

A remarkably wide variety of microstructures and grain sizes may occur during billet conversion. The billet may receive homogenization cycles at temperatures near the melting point or it may have a die-chilled, warm-worked surface which may cause accelerated precipitation of Ni_3Cb (delta) or the BCT gamma" phase. Billets of 718 can be massive in cross-section and may weigh up to 5,000 Kg (11,000 lbs); therefore, the metal may be exposed to very slow heating and cooling rates. All or any of these conditions influence the final product's microstructure and mechanical properties.

The ultimate goal of a forge practice is to modify what might be called an "uncontrolled" billet conversion (or at least the later part of this process), to a "controlled" and reproducible process. An understanding of the phase equilibria developed during the billet conversion must serve as the cornerstone of such a "controlled" process.

Inconel 718 is an example of a disk alloy whose microstructures must be tightly controlled during billet conversion in order to develop the optimum mechanical properties. The types of phases present in Inconel 718 are known (1,2,3), but the kinetics of their precipitation and influence upon recrystallization and grain growth at billet conversion temperatures have received little attention. It is generally recognized that temperatures above the Ni_3Cb (delta) phase solvus cause grain growth and that precipitated delta particles inhibit grain growth. The purpose of this paper is to focus more closely upon the phase equilibria and grain growth in 718 in a narrow temperature range near the critical delta solvus.

Material History

The Inconel 718 billet used in this study was of good quality and was from Heat 9-8922. The chemistry of the top slice in w/o is listed: .028 C, 52.82 Ni, 1.05 Ti, .51 Al, 17.93 Fe, 1.96 Mo, 18.17 Cr, 5.27 Cb, .004 B. A 20.3 cm (8") dia., 23.6 cm (9.3") long mult was cut, heated to 990°C (1810°F) for 1½ hours, and upset in one operation to a 7.6 cm (3") thick, 36.8 cm (14.5") diameter pancake and water quenched. The press head speed was about 25.4 cm (10") per minute and the dies had been heated to about 900°C (1650°F) to obtain some, but not excessive, die chill such as is encountered in billet cogging.

After forging, a radial etched slice was cut and examined. The radial flow was symmetric with a friction cone present equally distributed with top and bottom surfaces. A number of radially oriented blanks were cut immediately adjacent to the top and bottom surfaces. Therefore, all of the test material would have a uniform history of moderate flow after heating to a marginal temperature.

In order to understand the structures which might be obtained by billet homogenization, billet soaking, billet preheating, improper preheating and severe die chill, respectively, one set of blanks was exposed to each of the following thermal histories:

*Inconel 718 is a trademark of the International Nickel Company.

1 hour at 1150°C (2100°F), water quenched
1 hour at 1010°C (1850°F), water quenched
1 hour at 970°C (1775°F), water quenched
2 hours at 870°C (1600°F), water quenched
4 hours at 790°C (1450°F), water quenched

Next, additional blanks which were first cycled to the five above described temperatures were exposed to 990°C (1810°F) and 970°C (1775°F) for 1, 4, and 8 hours and water quenched. In order to follow the possible structural changes and kinetics of the 990°C or 970°C exposures, room temperature tensile testing was carried out on all of the thermal conditions and the resultant microstructures in both the thread and gage areas were characterized. Identification of the phases present was carried out by X-ray diffraction analyses of extracted residues.

Because the 990°C temperature is more marginal to the critical delta solvus and a more realistic forge temperature, the discussion of results will focus on this group. Moreover, because the kinetics were found to be unusually rapid in 1 hour at 990°C (i.e., equilibrium was achieved in 1 hour), only the 1 and 8 hour tensile data will be presented while only the microstructures of the 1 hour exposed samples will be presented.

Results

Room Temperature Tensile

Table I shows the results of the room temperature tensile tests. These tests were used solely as a means to monitor the kinetics at the various temperatures and are not to be compared to regular tensile data from fully aged materials.

TABLE I. Tensile Properties at R.T.

Test #	Thermal History	.2 YS		UTS		Elong	RA
		MPa	ksi	MPa	ksi	%	%
1	1 hr 1150°C (2100°F)WQ	303	44.0	703	102.0	68	66
2	" " " + 1 hr 990°C, WQ	310	4.50	721	104.5	72	57
4	" " " + 8 hr 990°C, WQ	296	43.0	721	104.5	75	60
9	1 hr 1010°C (1850°F)WQ	448	65.0	941	136.5	51	64
10	" " " + 1 hr 990°C, WQ	490	71.0	979	142.0	48	54
12	" " " + 8 hr 990°C, WQ	445	64.5	959	139.0	42	51
17	1 hr 970°C (1775°F)WQ	559	81.0	1007	146.0	25	44
18	" " " + 1 hr 990°C, WQ	538	78.0	1010	146.5	42	48
20	" " " + 8 hr 990°C, WQ	510	74.0	986	143.0	44	58
29	2 hr 870°C (1600°F)WQ	779	113.0	1110	161.0	28	40
26	" " " + 1 hr 990°C, WQ	545	79.0	1014	147.0	41	45
28	" " " + 8 hr 990°C, WQ	510	74.0	993	144.0	42	52
33	4 hr 790°C (1450°F)WQ	1062	154.0	1372	199.0	24	39
34	" " " + 1 hr 990°C, WQ	517	75.0	1000	145.0	38	48
36	" " " + 8 hr 990°C, WQ	510	74.0	993	144.0	43	51

All tests were conducted on an Instron at a crosshead speed of .127 cm (.05")/min to fracture.

The RTT data shows that as the initial exposure temperature is increased from 790°C to 1150°C the strength falls. Such strength changes signify that structural changes are occurring at each of the temperatures of exposure. The highest strength value would signify smaller and more plentiful precipites such as were found at 790°C and 870°C.

When the samples are subsequently given the 990°C heat treatment, all the high strength levels drop and become nearly the same except in the case of the material which was originally exposed at 1150°C. Thus, the 990°C exposure has apparently solutioned the strengthening particles found at the lower temperatures but has not produced any strengthening particles in the materials originally exposed at 1010°C or 1150°C. The finer grain structure developed at 1010°F has superior strength to the coarse grain material annealed at 1150°C.

Metallographic Study

Figure 1 shows the as-forged starting structure. The grain size is mixed and contains both spherical and platelet shaped delta phase particles. Figure 2 shows the structure after solutioning at 1150°C (2100°F). All the delta phase is gone and the grains have grown immensely. Exposure at the 990°C temperature does not precipitate any delta phase in the grains but a semi-continuous precipitation which probably is delta phase has occurred in the grain boundaries, Fig. 3. This precipitation is embrittling at room temperature since the % RA falls and the fracture follows coarse grain boundaries.

The 1 hour exposure at 1010°C did not dissolve all the delta phase formed during forging. Other heat treat studies on the same material have shown that some delta phase particles still were present at 1022°C, Fig. 4. However, all the delta phase was dissolved when the forged material was heated at 1040°C.

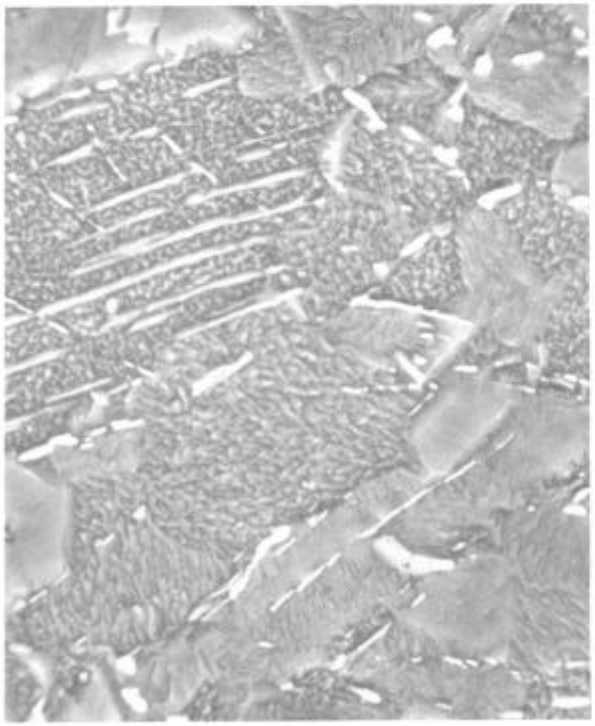

Fig. 1. As-Forged - 3000x

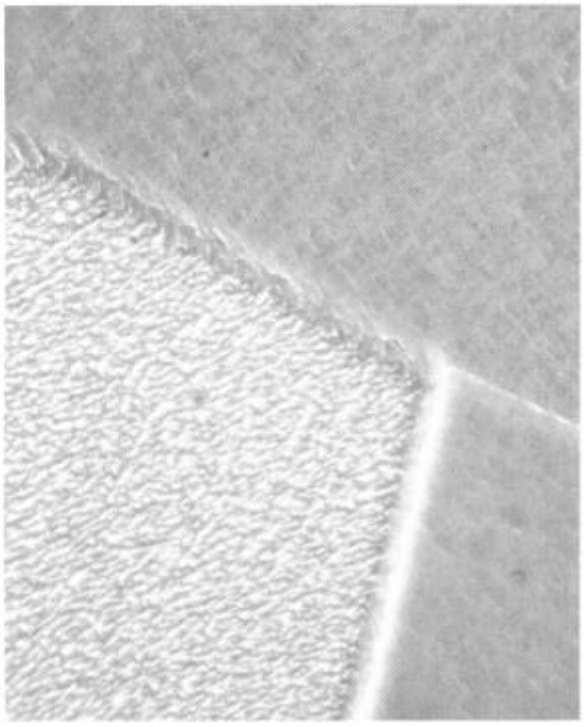

Fig. 2. Solutioned at 1150°C - 3000x

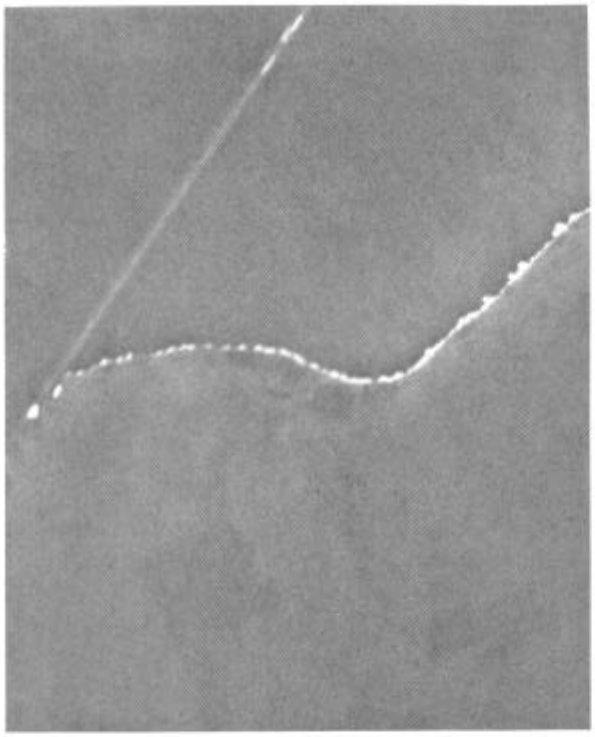

Fig. 3. Solutioned at 1150°C + 1 hr at 990°C - 3000x

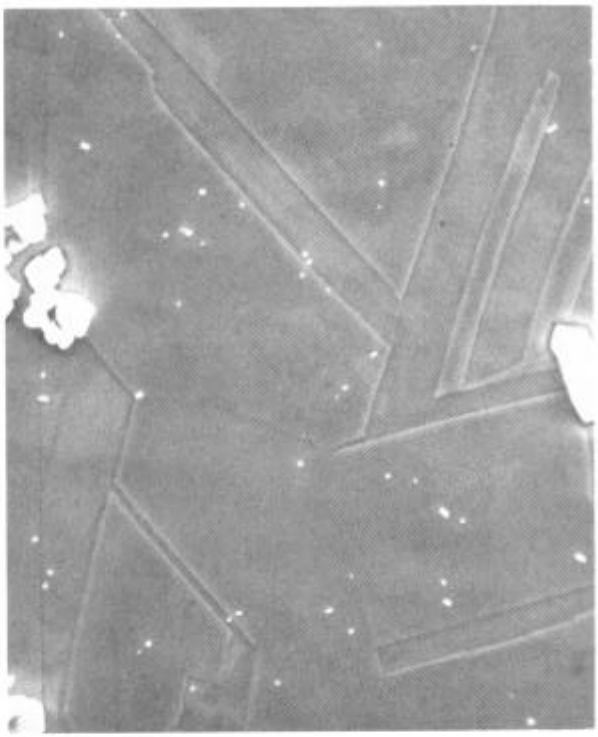

Fig. 4. Solutioned 1 hr at 1025°C - 1000x

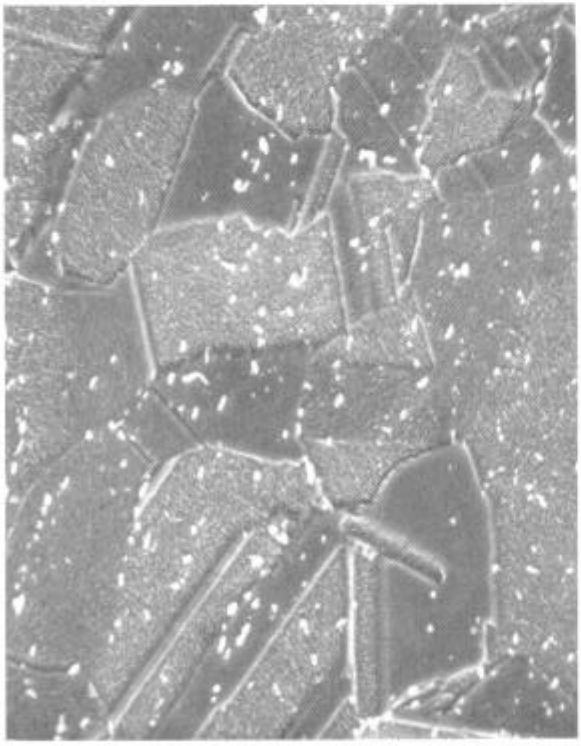

Fig. 5. 1 hr at 1010°C - 1000x.

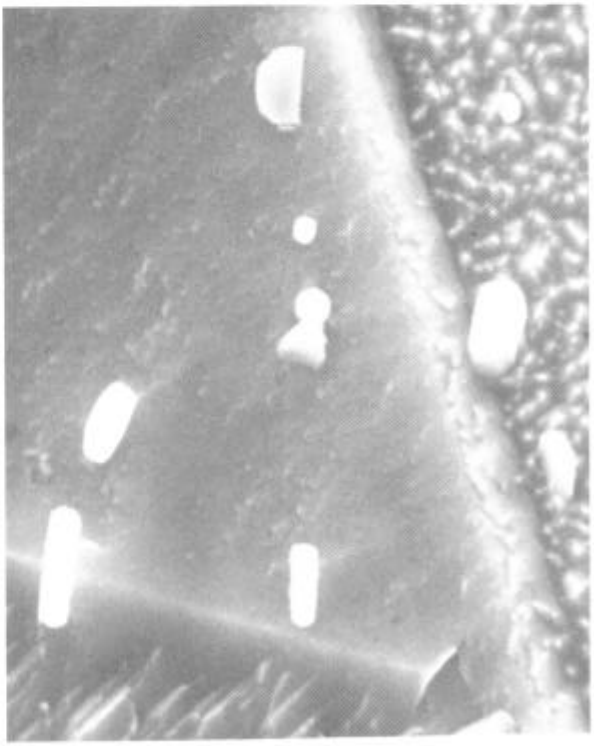

Fig. 6. Same as Fig. 5 - 10,000x.

The presence of delta phase particles at 1010°C resulted in much less grain growth. The delta particles are well spheroidized and show no strong propensity for location at grain boundaries, Figs. 5 and 6. Figure 7 shows that exposure at 990°C slightly increased the number of delta particles. Figure 8 shows several long thin delta particles lying in large angle boundaries.

Figures 9 and 10 show the effects of a 970°C cycle superimposed on the as-forged structure. The unrecrystallized grains show more precipitation of delta phase platelets. Recrystallized grains also showed an increase of delta particles but these are thicker and more blocky and reside mostly in the grain boundaries.

Figure 11 shows that the thin platelets in unrecrystallized regions tend to spheroidize at 990°C but their density is still high enough to impede complete recrystallization. The density of delta phase particles in recrystallized regions is reduced and they become even more blocky. Figure 12 shows portions of the same field of Fig. 11 at higher magnification.

The exposure of 718 at 870°C can produce variations in microstructural reponses. This temperature is the lowest at which the delta phase may begin to form, while also being near the upper temperature at which the gamma" goes in solution. Depending on the material chemistry and the furnace temperature control, delta and gamma" phases may co-exist. In our initial program survey, a

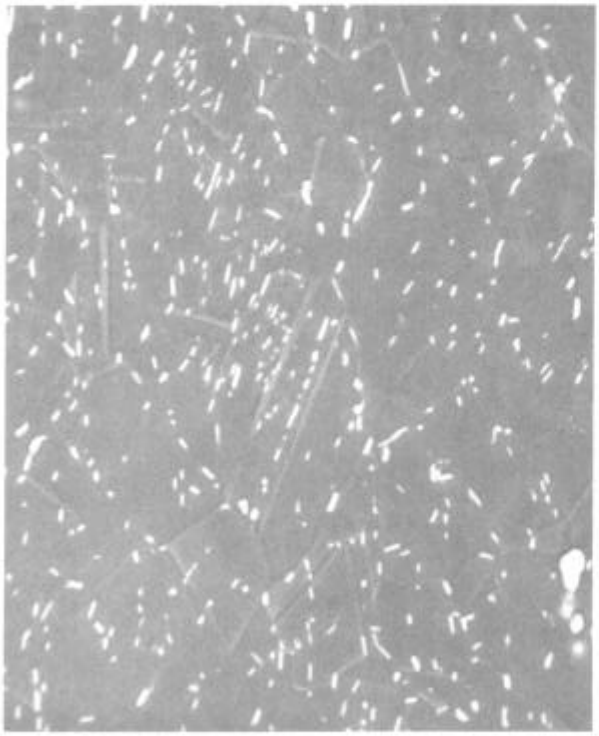

Fig. 7. 1 hr at 1010°C + 1 hr at 990°C - 1000x.

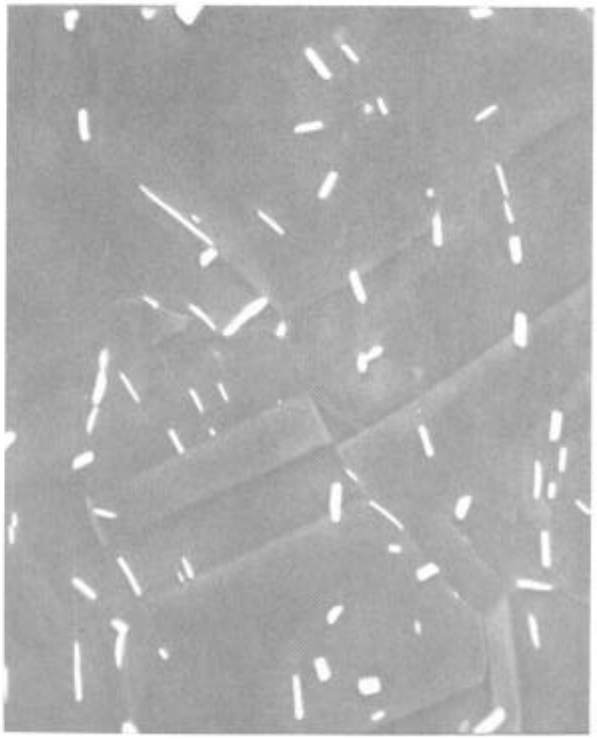

Fig. 8. Same as Fig. 7 - 3000x.

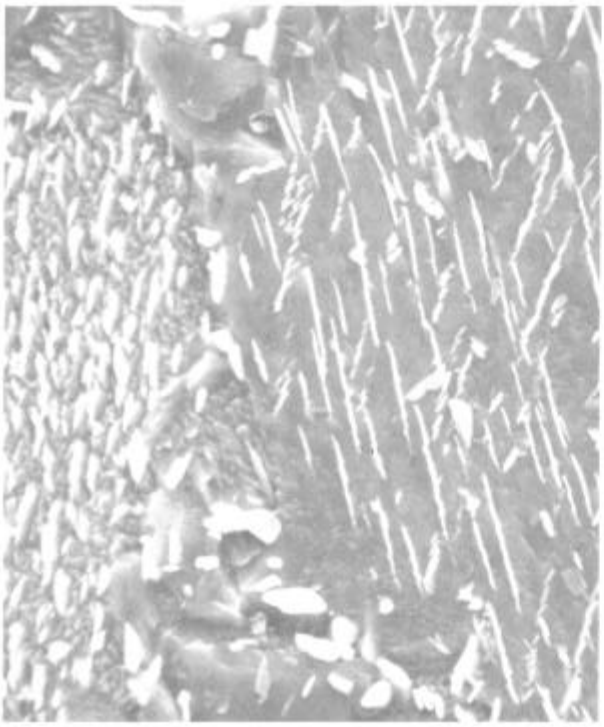

Fig. 9. 1 hr at 970°C - 3000x
Unrecrystallized Region.

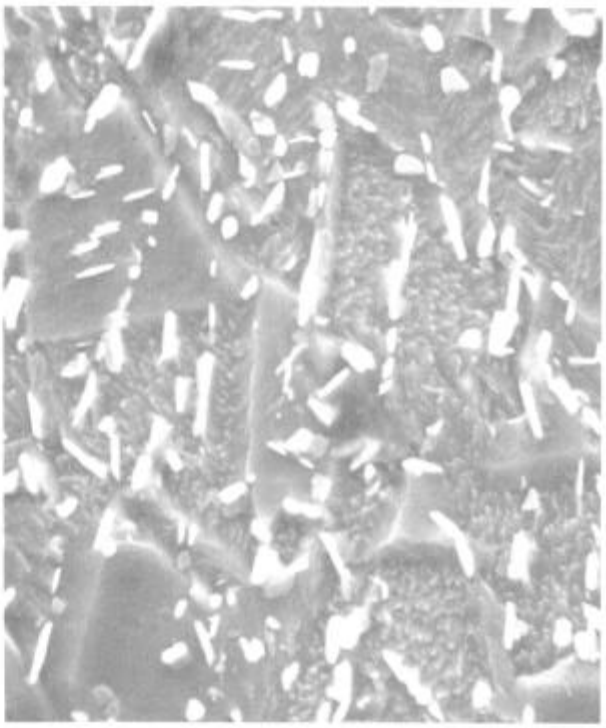

Fig. 10. Same as Fig. 9 - 3000x
Recrystallized Region.

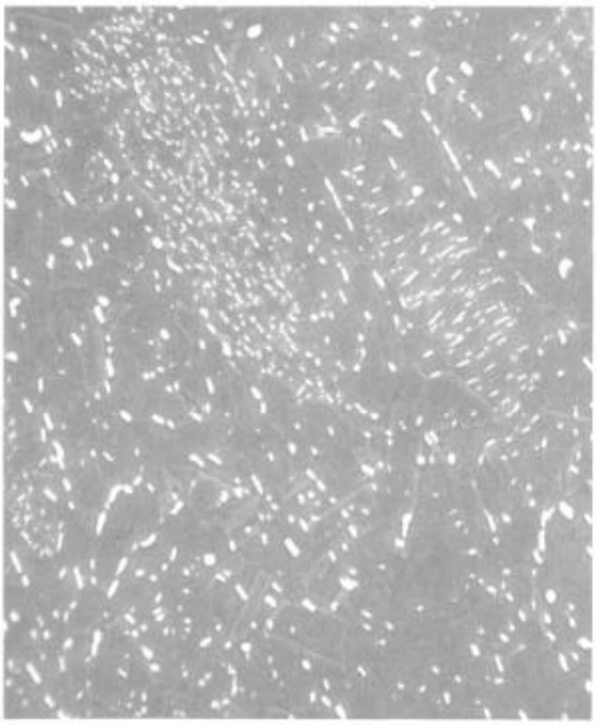

Fig. 11. 1 hr at 970°C + 1 hr at
990°C - 1000x.

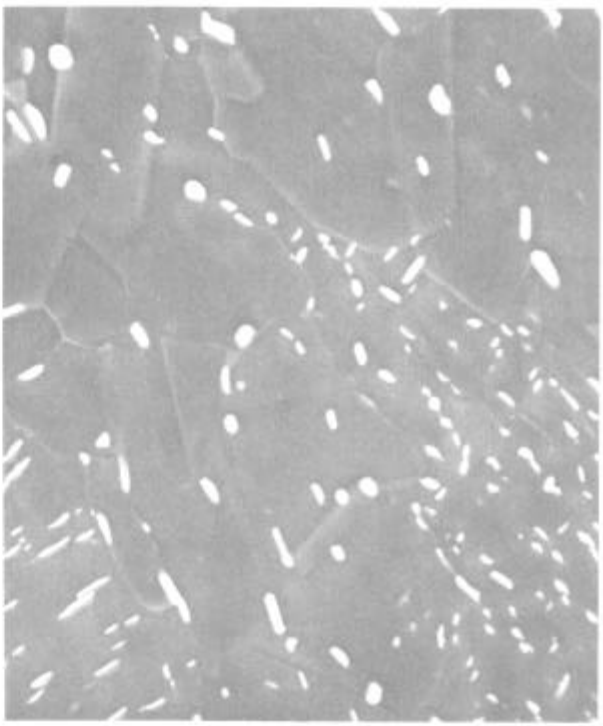

Fig. 12. Same as Fig. 11 - 3000x.

sample exposed at 870°C showed more gamma" precipitation than did the later series of tensile blanks. Figure 13 shows the large numbers of delta phase particles with fewer gamma" particles in the tensile sample. Figure 14 shows the large quantity of finely distributed gamma" in the original trial heat treat sample. This indicates a precipitation sensitivity not pursued further because of the easy dissolution of the gamma" during subsequent billet processing. In our study, the materials initially exposed to 870°C showed more gamma" precipitation than in the tensile samples.

A thermal cycle of 990°C, Figs. 15 and 16, dissolves all of the fine particles and some of the blocky delta phase in the recrystallized regions. In the unrecrystallized regions, the delta particles are agglomerated but the density of delta particles is still high enough to retard complete recrystallization. The structures of Figs. 15 and 16 look very much like those in Figs. 11 and 12, and the tensile properties confirm that they are equivalent structures.

When the as-forged material is exposed to 790°C, a radically different structure is found. The delta phase formed during forging is still present, but a very fine precipitation of gamma" and gamma" has formed everywhere, Figs. 17 and 18. Figures 19 and 20 show that the 990°C thermal cycle dissolves the gamma' and gamma", dissolves some of the delta phase, and spheroidizes the rest of the delta phase into blocky particles. Again, the unrecrystallized grains remain with the higher density of delta phase.

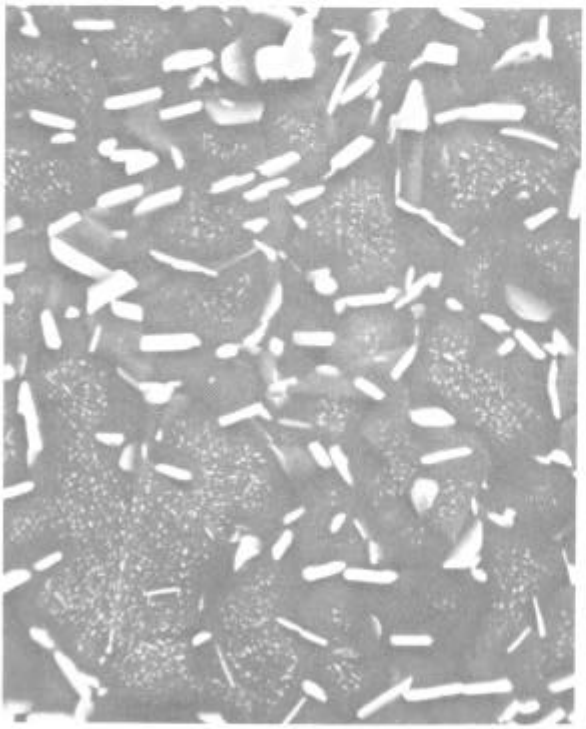

Fig. 13. 2 hr at 870°C - 3000x.

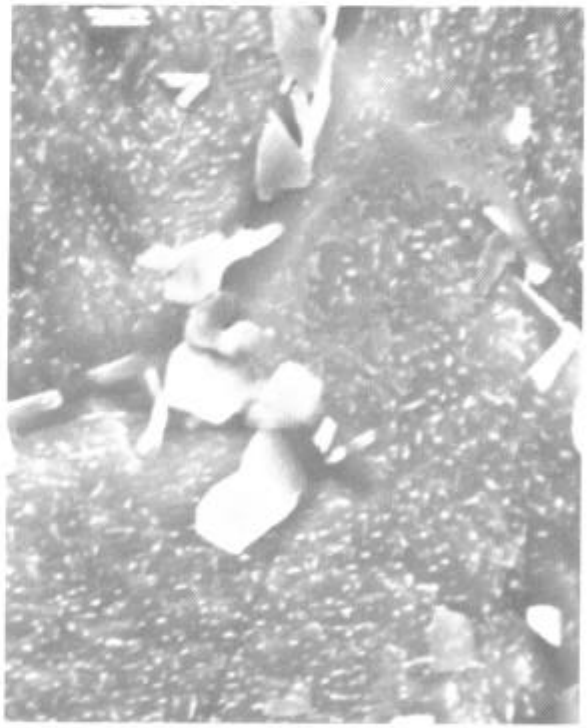

Fig. 14. 2 hr at 870°C - 10,000x. First heat treated sample.

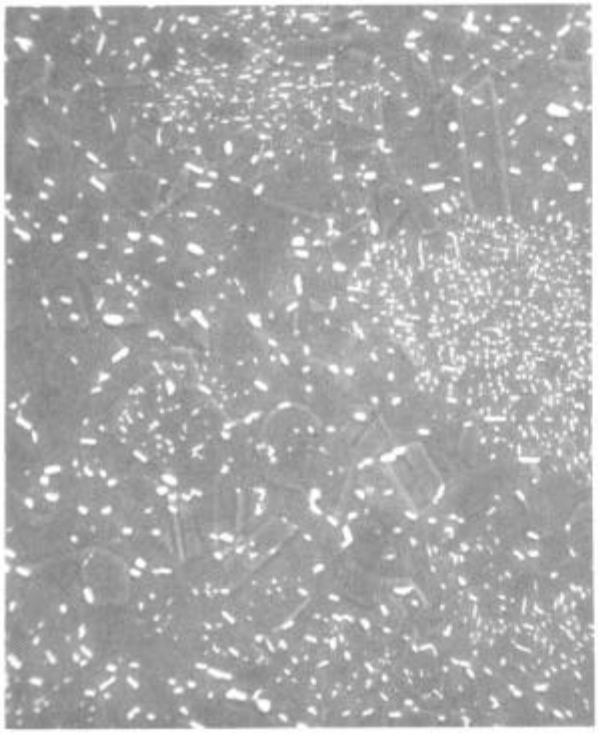

Fig. 15. 2 hr at 870°C + 1 hr at 990°C - 1000x.

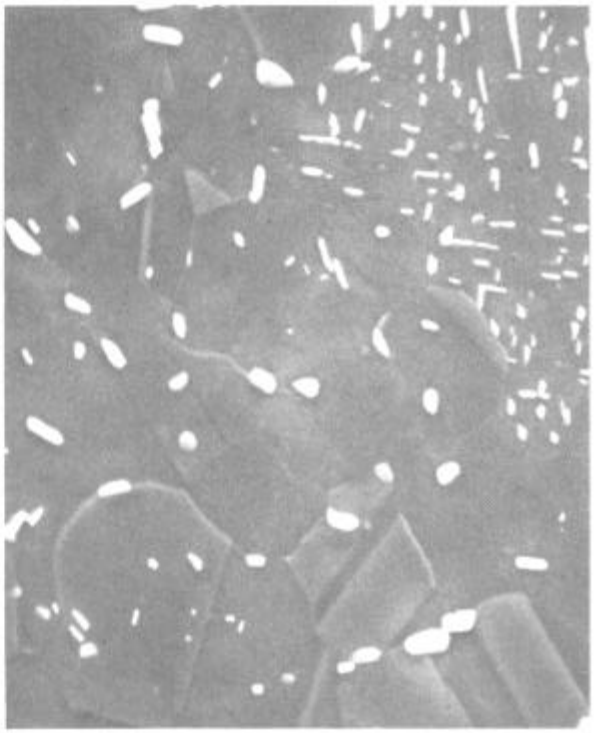

Fig. 16. Same as Fig. 16 - 3000x.

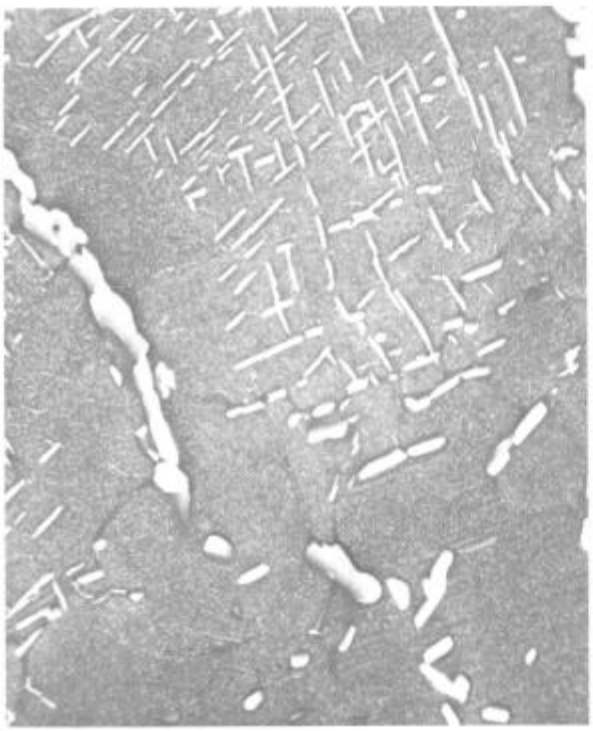

Fig. 17. 4 hr at 790°C - 3000x.

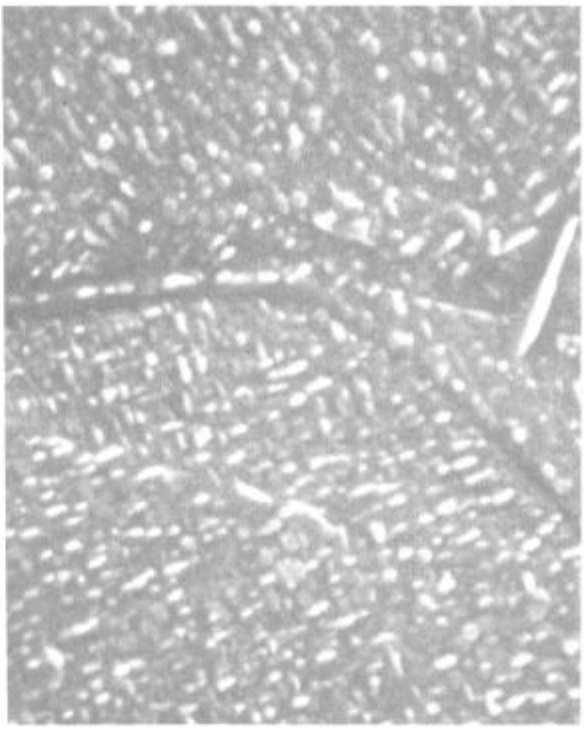

Fig. 18. Same as Fig. 17 - 30,000x.

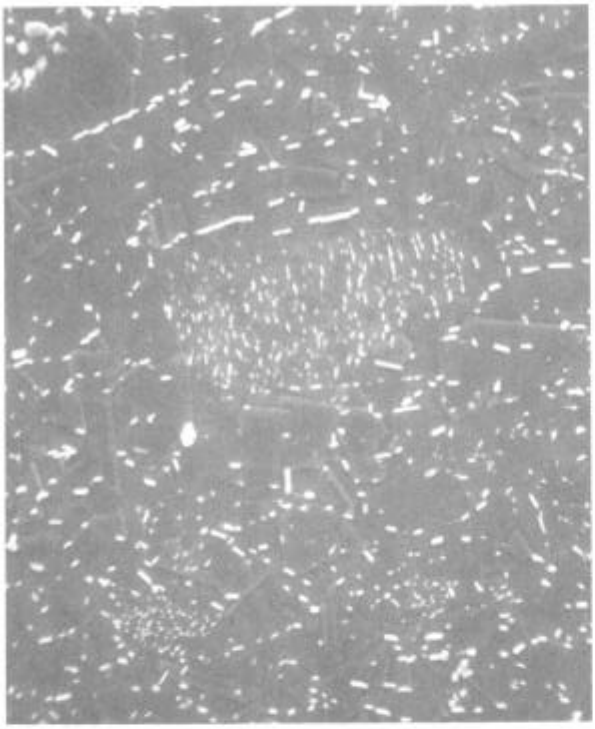

Fig. 19. 4 hr at 790°C + 1 hr at 990°C - 1000x.

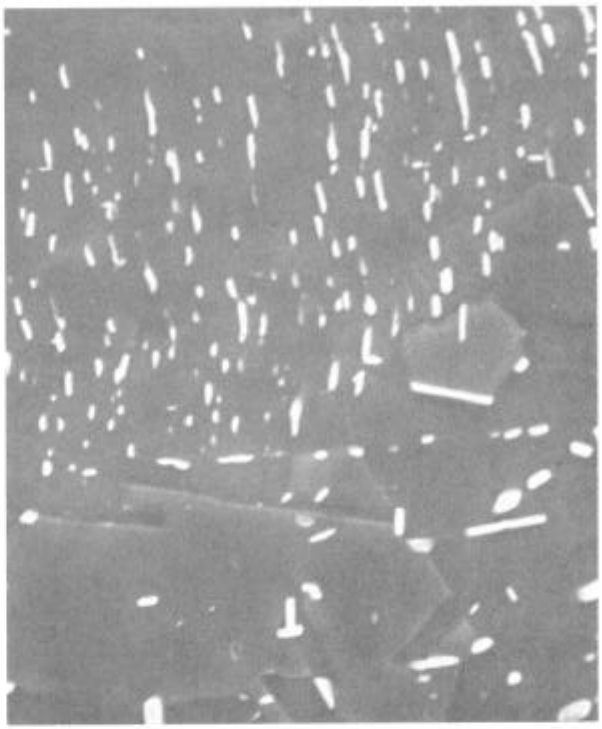

Fig. 20. Same as Fig. 19 - 3000x.

Discussion of Results

Better understanding of metal flow in closed dies leads to recognition of the existence of friction cones. The effective depth may be added to by chill cones. Thus, premium forgings like DA 718 require premium billet to ensure that the whole component has the microstrcture and properties required. This is further emphasized by the spread of near-net forge practices to less expensive alloys like 718.

A fine grain size is desired but is very difficult to maintain through a heating cycle. In this study the as-forged grains were ASTM 9-10 with some residual unrecrystallized as large as 5. But 1 hour at 1010°C grew the grains into the ASTM 3-4 range and 1 hr at 1150°C into the ASTM 0 range. This explains the interest in temperatures very close to the δ solvus. It also explains why a chemically homogeneous matrix is important to obtain a uniform phase equilibria throughout the forging.

The results of this program show that any purposeful or inadvertent over-temperature must be followed by considerable thermo-mechanical deformation to correct the detrimental effect of the over-temperature. The over-temperature at 1150°C delays the precipitation of δ within grains in the 990°C range but caused a precipitation along the grain boundary which is embrittling. This grain boundary precipitation is not observed after heating to 1010°C.

The microstructure and R.T. tensile properties appeared identical after heating at 990°C regardless of the prior participation pattern, if the prior heating was at 1010°C or below. Of course the grain size could be different in material with a different percent reduction than the material in this study, but the phase equilibria should be the same. A 1 hour cycle at heat at 990°C appears to eliminate any differences in precipitation due to heating or cooling rate or pre-heat. It appears that accurate control of the temperature is more important than control of the prior precipitation pattern.

Precipitation of gamma" appears to be a sensitive parameter. However it does not appear to be an important parameter in billet conversions of DA 718 because it is quickly eliminated.

Delta phase is an effective grain size control at 990°C much like gamma' is in other Ni-base superalloys. In fact, unrecrystallized grains are unable to complete recrystallization at 990°C due to the density of δ within the unrecrystallized grains. However, if the structure is recrystallized during forging prior to exposure to 900°C, the δ is well spheroidized and seems to be located only at grain boundaries. It appears desirable to obtain a fully recrystallized structure by controlling the strain and reducing the die chill effect. It does not appear possible to correct a duplex structure by thermal control alone (unless by heating to a temperature above the δ solvus where grain growth quickly occurs).

Delta phase may appear to be a useful phase for controlling grain size. However, other unpublished work has shown that it is undesirable because it keeps the grain size too small for the desired balance of tensile and creep properties. Obviously the temperature range where δ precipitation and grain growth are both within desired limits is extremely restricted, even in a relatively homogeneous piece of metal.

Conclusions

1. The delta phase is a very effective barrier to static recrystallization. The delta phase particles tend to precipitate heavily in worked grains at temperatures at or below 990°C and resist grain boundary migration even if such particles are in a spheroidal form. When the temperature rises well above 990°C, i.e., 1010 to 1025°C, delta particles solution and rapid grain growth occurs.

2. The delta phase may precipitate as low as 870°C and co-exist with the platelike gamma" phase. The gamma" phase undergoes solutioning when the material is held at 990°C for 1 hour, while the delta phase formed at lower temperatures partially solutions and changes its morphology.

3. The rate at which a material is heated or cooled is not as critical as is the temperature to which it is exposed. Overheating the material retards the normal precipitation of delta phase and produces large grains in whose boundaries the delta phase forms as continuous structure and may result in embrittlement.

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