OBSERVATIONS ON THE DEVELOPMENT OF DELTA PHASE IN IN718 ALLOY

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Abstract

A wide variety of microstructures may be developed in wrought IN718. While billet chemistry and thermomechanical process history directly relate to the resulting general microstructure it may be difficult at times to understand how particular structures were formed. The appearance, morphology and control of δ phase are of special interest because of its critical influence on grain structure, grain size and mechanical properties. This paper reviews the evolution of microstructure during the deformation and heat treatment of plate rolled samples with the goal of increasing the understanding of how different morphologies of δ may form and how they will respond to further processes. Processing began with a forging heat treated above the δ solvus and then either exposed to a δ precipitation cycle followed by sub δ solvus rolling or rolled below the δ solvus followed by δ precipitation. Qualitative evaluations of the resulting microstructures provide insight into the mechanisms controlling the development and morphology of δ phase. Consideration will also be given to processes for evaluating uniformity in IN718 forgings. The information presented may assist those involved in the manufacture and use of IN718 articles where microstructure control is sought.

Superalloys 718, 625, 706 and Various Derivatives
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Introduction

δ phase plays a significant role in the development of microstructure and properties of IN718. Numerous papers describe using δ phase to control microstructure and related mechanical behavior [1-3]. At times the microstructures of forgings and rolled rings display unusual concentrations of δ phase or non-uniform grain sizes that may be problematic for full life service. Attempts to unravel how the process history contributes to the resultant condition can be difficult to determine when the microstructures developed in prior processing are unavailable. The operations applied in this experiment were not intended to represent a particular part process but instead to form δ and then process it. δ phase was developed along two different process paths and then observations were made during subsequent deformation and annealing treatments. In one series δ was developed first and then subjected to deformation. In the other sequence deformation preceded the intentional δ precipitation and further deformation followed. By following the evolution of δ through precipitation and deformation it was hoped that more knowledge about the phase and how it can be used to control microstructure would result.

Experimental procedure

Material

The starting stock for these deformation studies consisted of sections of a fully heat treated AMS 5663 ring forging. The composition of the 8-in. diameter billet used to produce the part is shown in Table I.

Table I Nominal composition (weight percent) of material used in this study.

Ni	Fe	Cr	Ti	Al	Mo	Nb+Ta	C	Mn	Si
54.12	17.43	17.91	1.01	0.45	2.88	5.45	0.025	0.07	0.07

Processing

Samples were machined from the same region of the ring to provide rectangular slabs that were rolled in the same orientation and direction the metal had experienced during ring rolling. The specimens were approximately 25 mm thick at the start of rolling. All material was annealed at 1054 C for 60 minutes and static air cooled prior to any further processing. A sufficient number of samples were created to allow each slab its own particular level of rolling strain. A variety of process paths were applied including anneals above and below the δ solvus to purposefully solution or generate δ phase. Prior to rolling samples were heated for 1 hour at the rolling temperature. Rolling was conducted in increments of 10% reduction in thickness for each heating cycle on Pratt & Whitney Florida's Material's Laboratory cold rolling mill. The rolls have a diameter of approximately 25.4 cm, the roll speed was about 15 cm/second and the estimated strain rate was 0.4/ sec. Samples that had completed their prescribed amount of deformation were allowed to air cool. Samples that required additional reduction to achieve their particular amount of total strain were immediately returned to the furnace for a 15 minute heating cycle and then rolled again. Table II outlines the various processing conditions used. Both the DR and RD series of specimens were processed sequentially (i.e., DR1 lead to DR2 lead to DR3 and so on). Microstructure evaluations were conducted at each step along the process path to allow the evolution of changes to be observed. The longitudinal sections examined were from mid length and mid width of the rolled coupons.

Process Selection

The conditions applied to the material of this study were chosen for a variety of reasons. Heating the material at 1054 C was intended to normalize the microstructure to a uniform grain condition free from the δ precipitates to be developed in subsequent processing. 899 C heating was applied to give this relatively Nb rich material opportunity to develop δ phase near the precipitation maxima [4]. 982 C heating and rolling exposures were used as sub δ solvus solution cycles that allow relatively fine grains to be formed while partial solutioning δ and full solutioning any γ' phase that may be present. Process sequences were chosen to allow rolling of δ rich structures, and to assess how prior processing affects modes of δ precipitation. Reductions at increments of only 10% were intended to simulate ring rolling and provide marginal forging conditions that may be present in many forming scenarios. Recrystallization during this study is believed to be primarily static, occurring either immediately following rolling or during re-heating. Previous studies indicated a true strain of 0.1, at 982C and moderate rates of strain would be insufficient to allow peak flow stresses to be reached and softening due to dynamic recrystallization was not likely to have occurred during rolling [5,6].

Table II Outline of process path used during the microstructure development study.

Baseline 1054 C Anneal, 1 hour, Air cool (1930 F)	
DR1 899 C, 8 hours, air cool (1650 F)	RD1 982 C Rolling (1800 F)
DR2 982 C Rolling (1800 F)	RD2 899 C, 8 hours, air cool (1650 F)
DR3 982 C, 1 hour, air cool (1800 F)	RD3 982 C Rolling (1800 F)
DR4 899 C, 8 hours, air cool (1650 F)	RD4 982 C, 1 hour, air cool (1800 F)

Results and Discussion

Hot solution with δ precipitation followed by rolling

Baseline - Solutioning at 1054 C generated a uniform grain size approximately ASTM 3 with scattered carbides (Figure 1).

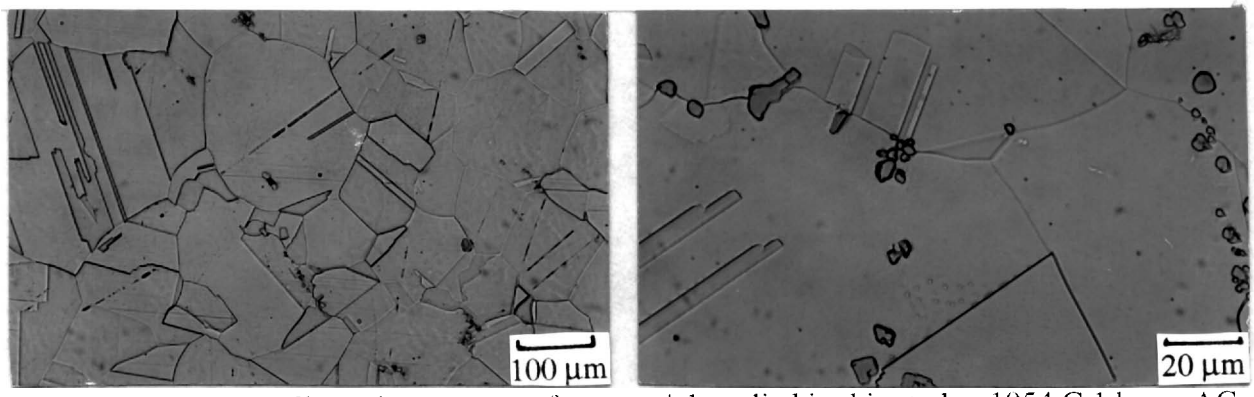

Figure 1: The Baseline microstructure for material applied in this study. 1054 C,1 hour, AC.

Condition DR1 - The 899 C exposure developed grain and twin boundary δ precipitates with what appears to be a background of γ' consistent with previous work [6] (Figure 2). Heterogeneous precipitation of δ may be expected in a relatively strain free system [7]. The lack of appreciable δ within the grains is likely due to the reduced number of dislocation sites of sufficient energy to facilitate development. The development of a more uniform distribution of Widmanstatten δ throughout a grain structure can be related to quenching strains or the remnant strains within unrecrystallized grains [3,4,8].

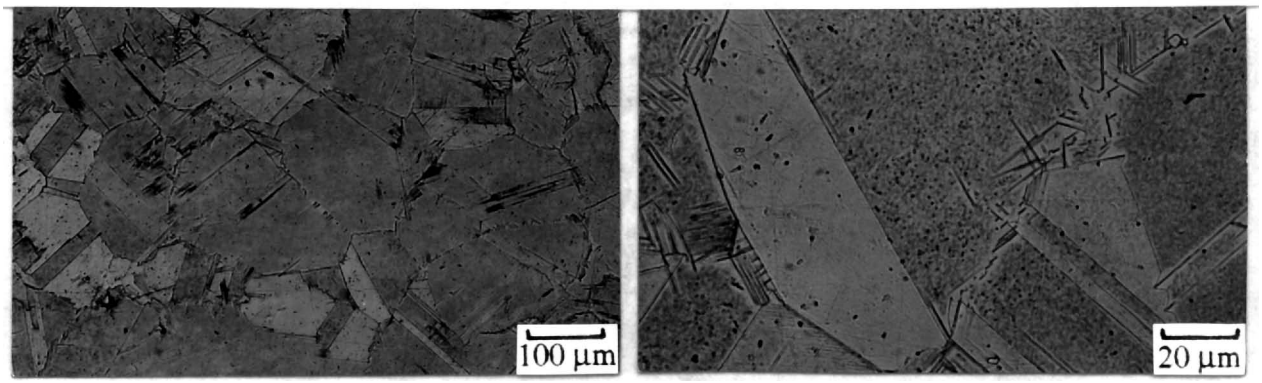

Figure 2: Microstructure of material condition DR1. 899 C for 8 hours, AC.

Condition DR2 - Heating for rolling at 982 C does little to the δ laths while dissolving the γ' completely (Figure 3). Rolling at 982 C progressively distorts the grain structure with the δ laths becoming more disturbed and interrupted at 30% reduction and degenerating largely into chains at 50% reduction. The combination of hot mechanical strain and intermittent annealing breaks up the long range lath δ and set the stage for creating spheroidal or tablet shaped δ (Figure 4).

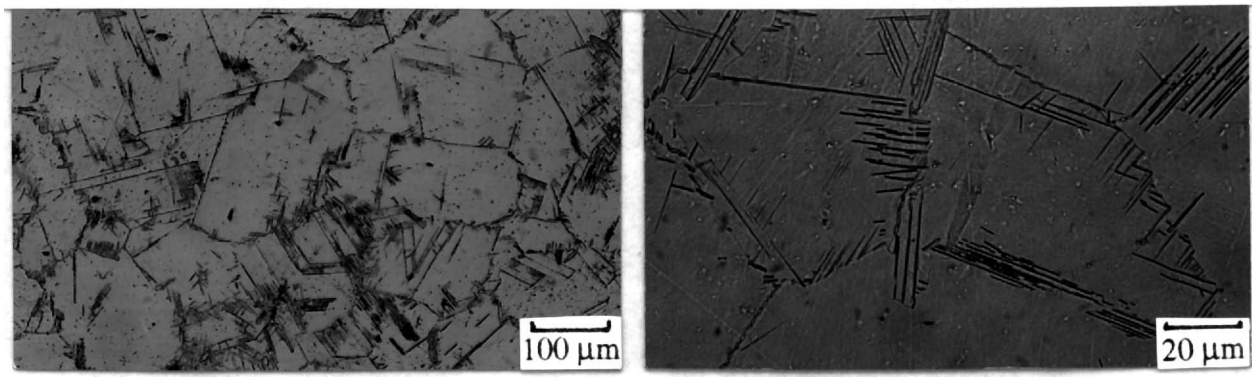

Figure 3: Microstructure of material condition DR2 prior to deformation.

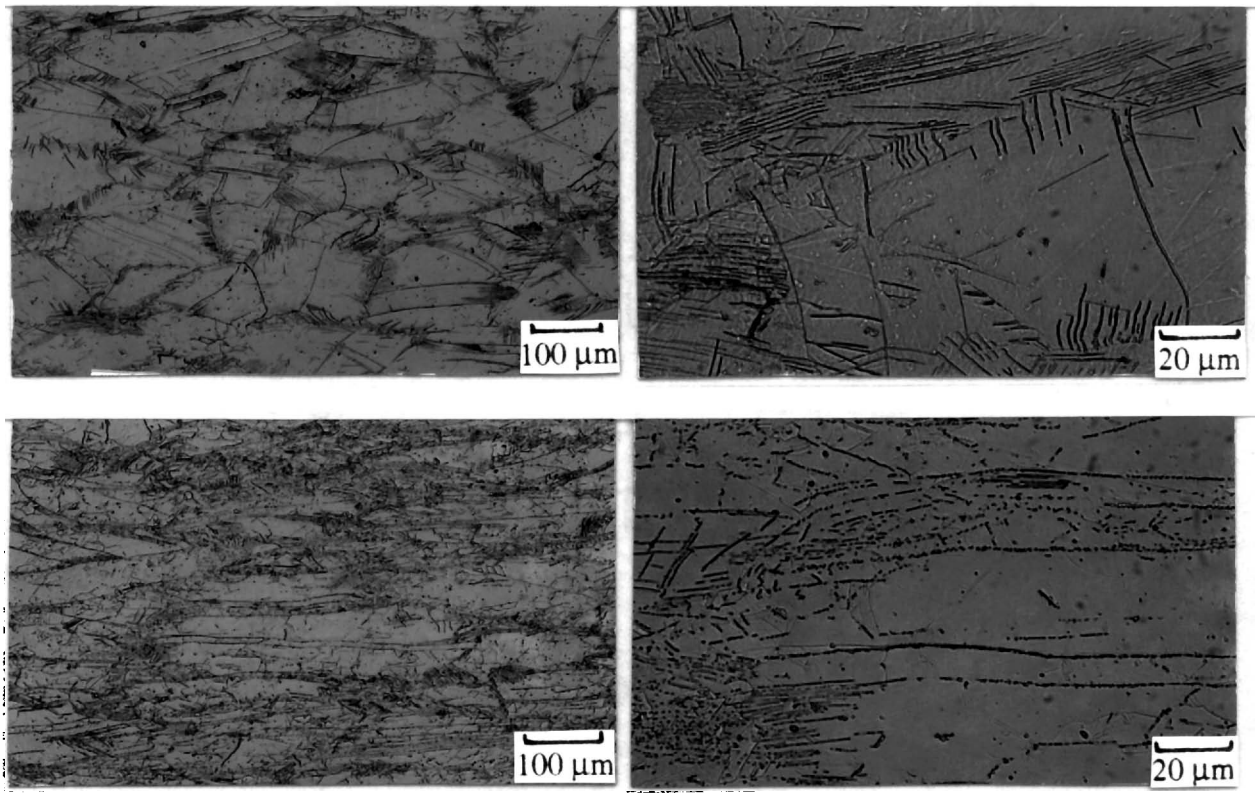

Figure 4: Microstructure of material condition DR2 at 30% (Top) and 50% (Bottom) total reduction at 982 C.

Condition DR3 - Subsequent annealing at 982 C for one hour appears to recrystallize much of the material that has been through at least 30% reduction to a very fine grain size ASTM 12. However, the original grain structure highlighted by broken δ is still very evident (Figure 5) and it is difficult to see the new grains.

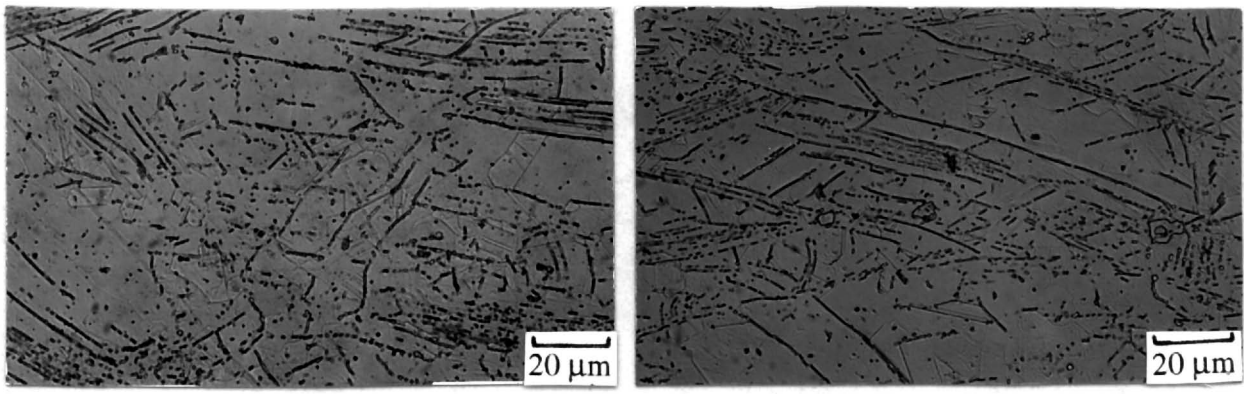

Figure 5: Microstructure of material condition DR3 after 30% (left) and 50% (right) total reduction, 982 C followed by 982 C, 1 hour, AC.

Condition DR4 - The final anneal at 899 C shows that lath δ can be readily developed again and the microstructure becomes very congested with various forms of δ phase (Figure 6). Close inspection of the specimens at 20 to 50% prior reductions reveals the nature of the different δ morphologies. Spheroidal δ appears in those locations favored by higher rolling strains and the concentrations of δ produced in the original 899 C precipitation, the grain boundaries of the original 1054 C solution anneal. The volume fraction of spheroidal δ is low

for the lower reductions but also low in general because not much δ was formed in the initial 899 C treatment prior to rolling. A few particular grain and twin boundaries show nearly continuous δ phase that has stubbornly resisted deformation and solutioning and outline the original grains formed at 1054 C. Distorted δ phase is present in roughly parallel layers that appear to be on the verge of becoming spheroidal. There are patches of fresh lath δ that are forming within the spaces not occupied by the spheroidal or distorted δ. The appearance of this new or fresh δ lath may indicate areas of strain within the microstructure that have not been fully recrystallized. At the 50% reduction level the scale or span of the δ lath is much more limited than at 20%. Also, a background decoration of γ' particles appears among the more widely separated δ laths where Nb had remained in solution.

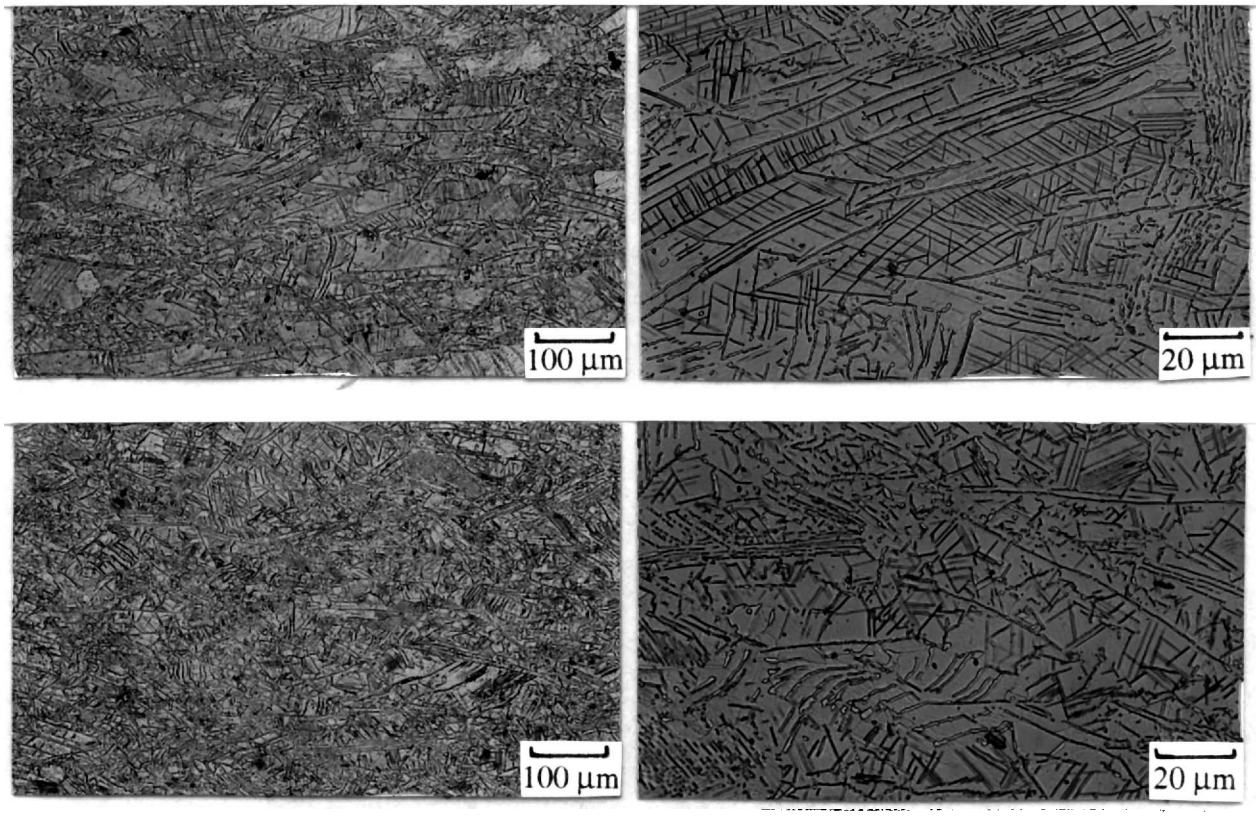

Figure 6: Microstructure of material condition DR4 after 899 C, 8 hours, AC for material rolled 20% (Top) and 50% (Bottom) and heat treated at 982 C.

Hot solution with rolling followed by δ precipitation

Condition RD1 - Starting with the same base microstructure as described above, rolling at 982 C again progressively distorts the grain structure. However, for this process path, as time and the amount of work are increased at 982 C, more and more δ phase appears (Figure 7). At 20% reduction (two heating cycles at 982 C) δ is marginally present. At 50% total reduction (five heating cycles and associated higher strain) small chopped up particles of δ phase appear within the grains and along grain boundaries. Lower magnification viewing reveals a layered morphology of fine δ traversing grain boundaries indicating an overall segregation of Nb inherent in the original barstock. This "banded" distribution of solute complicates microstructure interpretation in that metal with two levels of Nb effectively has two δ solvii and associated thermo-mechanical process responses. Studies within Pratt & Whitney have documented variations of 1.4 wt. % Nb sufficient to develop banded structures [9].

Segregation in highly alloyed materials is not unexpected but considering the uniformity of the grain structure at the start of this study it was surprising to see after rolling at 982 C. However, considering that deformation was at a relatively constant temperature just below the δ solvus, only those areas rich enough in Nb were able to develop δ. Forging within a falling temperature range or at a lower temperature would not likely show this condition due to the greater volume of δ that could be developed.

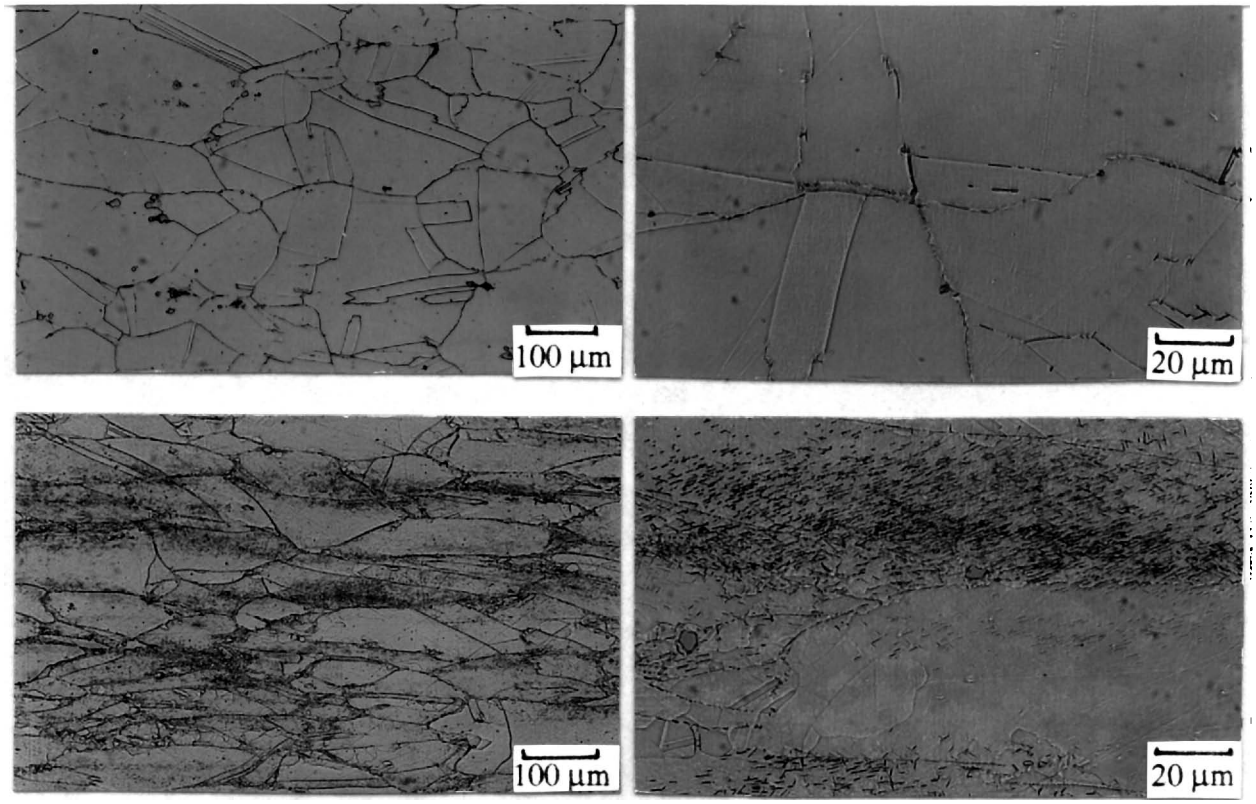

Figure 7: Microstructure for material condition RD1 at 20% (Top) and 50% (Bottom) total reduction, 982 C.

Condition RD2 - Processing and evaluation was now focused on material which had an initial 20% reduction at 982 C followed by exposure to 899C for 8 hours (Figure 8). In this condition δ has precipitated as medium range laths across grain boundaries and within the grains accompanied by γ'. Comparison with Figure 2 shows that 20% reduction at 982 C was sufficient strain to set up the substructure necessary for δ to come out within the grains.

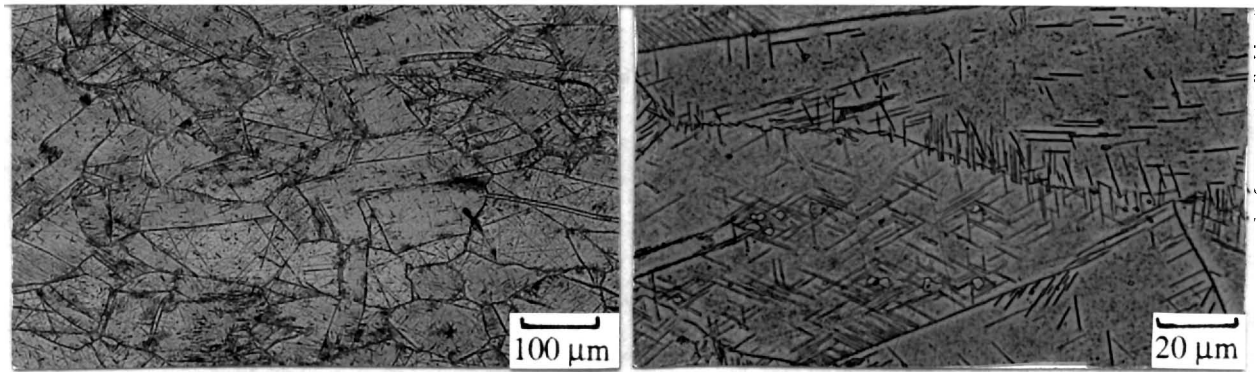

Figure 8: Microstructure for material condition RD2 after 899 C, 8 hours, AC on material rolled 20% total reduction, 982 C.

Condition RD3 - Rolling reductions were resumed at 982 C for material that had been rolled at 20% at 982 C and given the 899C δ cycle. Rolling at 982 C begins to break up the grain boundary δ at 20% but does little to disturb δ within the grains. At 50% reduction only a few laths have not been broken up and a large amount of spheroidal δ is present. Coarser grains are noted in the areas lean in δ (Figure 9).

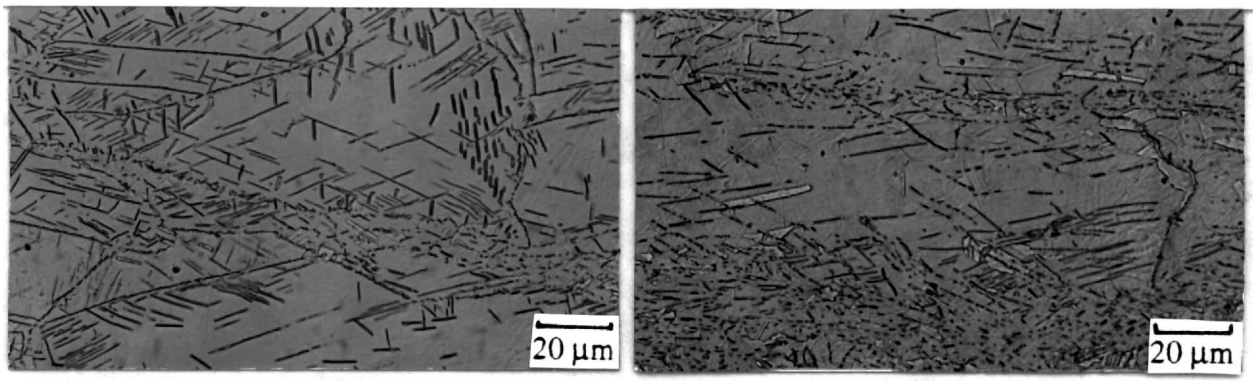

Figure 9: Microstructure for material condition RD3 at an additional 20% (Left) and 50% total reduction (Right), 982 C on material condition RD2.

Condition RD4 - Annealing at 982C after rolling at 982C did little to change the as-rolled microstructure until the 50% prior reduction level where virtually all lath δ has been disrupted into spheroidal particles (Figure 10). The coincident grain size and δ structure gradients within the sample are presumed to be additional evidence of a Nb composition variation within the metal. Many other studies have also noted similar duplex grain size distributions of coarse unrecrystallized or partially recovered grains surrounded by a necklace of fine recrystallized grains [2,3,5-7].

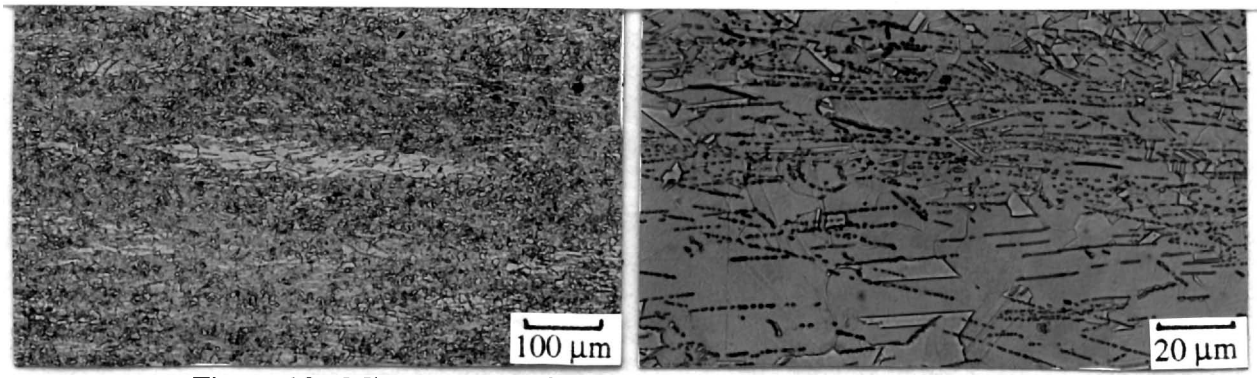

Figure 10: Microstructure for material condition RD4 after 982 C, 1 hour, AC on material RD3 rolled 50% total reduction.

In light of the composition and microstructure gradients observed in the RD series, the DR series specimens were revisited and some evidence of banding was found in the DR2 and DR3 material at the 50% reduction level (Figures 4 and 5). Below 40% total reduction the grain boundary concentration of δ appears evenly distributed. At 50% reduction the variations in δ phase and grain size begin to appear. It may be theorized that when a fine grain envelope is established around a coarse grain, that grain will be insulated from much of the nominal deformation if the reductions are slight or incremental in nature. The fine grains may accommodate the majority of plastic strain and only enough strain to provide for annealing recovery with elongation but no refinement of the coarser grains. The subtle banding of these samples may also be due to composition gradients where differences in local δ solvus temperature set up non-uniform δ barriers to grain size changes during forging and annealing.

This author suggests a combination of both mechanisms may be acting. The composition gradient sets the stage for coarse grains and once they become trapped in the field of fine grains they appear to resist refinement because the fine grains preferentially use up the strain. Perhaps the microstructures of DR4 show a way to achieve uniform grain structure in the presence of composition gradients. Figure 6 shows that with a second δ precipitation treatment, material with a 50% prior reduction of an initial grain boundary δ structure now develops a much more uniform distribution of δ, albeit of various morphologies.

A review of the observations made on the development of δ phase in IN 718 finds δ is formed initially as laths of varying densities and span depending on strain level, composition, system strain and prior process history. Deformation processing at temperatures below but near the δ solvus distorts δ. Additional time at temperature, where partial solutioning of δ occurs, and mechanical work combine to break up the continuity of the lath and chains of tablet and spheroidal shaped δ begin to appear. When sufficient working and partial solutioning are applied, stable spheroidal δ particles will result.

Summary

The objective for this study was to create various δ morphologies and then observe how further processing evolves the microstructure. The following conclusions can be made from this effort.

Basic origins of complex combinations of δ phase morphology can be understood through systematic sampling and interpretation of material during the course of its process history.

Residual strain in the system is important to uniform precipitation of δ phase.

A δ precipitation heat treatment may be used to understand the existing state of strain in an article of wrought IN 718.

Hot solutioning followed by limited rolling and δ precipitation heat treatment may reveal segregation patterns if present.

Nb segregation and near δ solvus processing may be responsible for the development of duplex grain size distributions in IN 718. Processing to generate more uniform precipitation of δ phase can be used to develop more uniform grain structures.

A 50% reduction at 982C combined with an 8 hour treatment at 899C is sufficient to convert a partially developed δ structure into a more complete distribution of short range δ suitable for further processing.

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