Recrystallization Behavior of Cold Rolled Alloy 718

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Abstract

Recrystallization of cold rolled Alloy 718 has been studied in the temperature range of 1575°F - 1725°F as a function of strain and annealing time. Changes in the microstructure have been monitored by measuring microhardness. An increase in hardness with annealing time has been observed for the 30% cold rolled material heated to 1575°F and 1625°F, whereas a continuous decrease in hardness has been observed for the 50% cold rolled material exposed to the same temperatures. Above 1625°F, a decrease in hardness for both 30% cold rolled and 50% cold rolled material has been observed. The increase in hardness in the 30% cold rolled material heated below 1625°F is due to precipitation of γ'' / γ' and inhibition of recrystallization. The decrease in hardness in the 50% cold rolled material appears to be due to recovery and the onset of recrystallization. Changes in microstructure in various conditions have been studied by optical microscopy and SEM.

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Introduction

Recovery and recrystallization are primary processes which govern microstructural evolution in cold worked metallic systems during high temperature anneal. These processes are well studied and documented for pure metals and model alloy systems (1-3). In commercial multiphase alloys, precipitation of the strengthening phases takes place during high temperature anneal. If the recrystallization is not complete before the precipitates have nucleated, precipitation and recrystallization exert a mutual influence upon each other (4,5). Precipitate particles can hinder the formation and migration of recrystallization fronts, and the lattice defects themselves promote the nucleation of precipitates. In Alloy 718, the fcc matrix is strengthened by combined precipitation of γ'' (an ordered DO_{22} structure) and γ' (an ordered $L1_2$ structure). In addition to these phases, delta phase (Ni_3Nb) also precipitates and is primarily used to control creep and stress-rupture properties in Alloy 718. The delta phase is also used in ingot to billet conversion to produce controlled grain structure in the billets. The precipitation kinetics of these phases in Alloy 718 have been studied extensively (6). However, an understanding of the role of these precipitates in recovery and recrystallization processes in this commercial alloy is lacking. In this study, recrystallization behavior of Alloy 718 in the fully solutioned and cold rolled condition has been studied and results are reported.

Experimental:

Samples from commercial Alloy 718 were machined flat to a thickness of 4.75 mm. These strips were fully solutioned at 1900°F for 1 hour followed by a water quench. The solution temperature used in this study was above the delta solvus of 1850°F. The solution anneal resulted in a single phase material with an average grain size of 40 - 50 µm. These 4.75 mm thick strips were further cold rolled to 30% or 50% reduction in thickness in multiple passes. Small samples were sectioned from the rolled strips and heated in the temperature range of 1575 - 1725 °F for various time intervals. At the lowest temperature 1575°F, all the three phases, namely; γ', γ'' and delta can precipitate. Temperatures in the band of 1675°F - 1725°F are above the precipitation range of γ' and γ'', but below the delta solvus. Samples were water quenched from the exposure temperatures. Microhardness measurements using a 300 gram load were made on the samples in as-rolled and rolled + annealed condition. Samples for optical microscopy and SEM were prepared by using standard metallographic techniques (7).

Results and Discussion:

Figures 1(a) and (b) show representative microstructure of the 30% and the 50% cold rolled samples, respectively. For 30% rolling, grains have retained their original equiaxed shape, however, some planar markings are seen in the grain interiors. These planar markings could be slip band and / or deformation bands. With increased deformation to 50%, grains have elongated and the density of the planer markings in the grain interior have increased.

The effect of annealing temperature on these structures was studied using microhardness measurements to show aging, recovery and recrystallization behavior. The variation of microhardness with time at an exposure temperature of 1575°F is shown in Figure 2 for both the 30% and the 50% cold rolled samples. A rapid increase in hardness for the 30% cold rolled material heated up to 1 hour is observed. After one hour hardness decreased slowly, and after 4 hours it is almost the same as the starting hardness of the 30% rolled material. After 30 minutes exposure at 1575°F, the 30% rolled material has attained a higher hardness than the 50% rolled alloy in the as-rolled condition. In contrast to the 30% rolled material, the 50% cold rolled samples showed a rapid loss in hardness after a 15 minutes thermal exposure at 1575°F. Hardness decreased up to 2 hours and then increased slightly.

a) b)

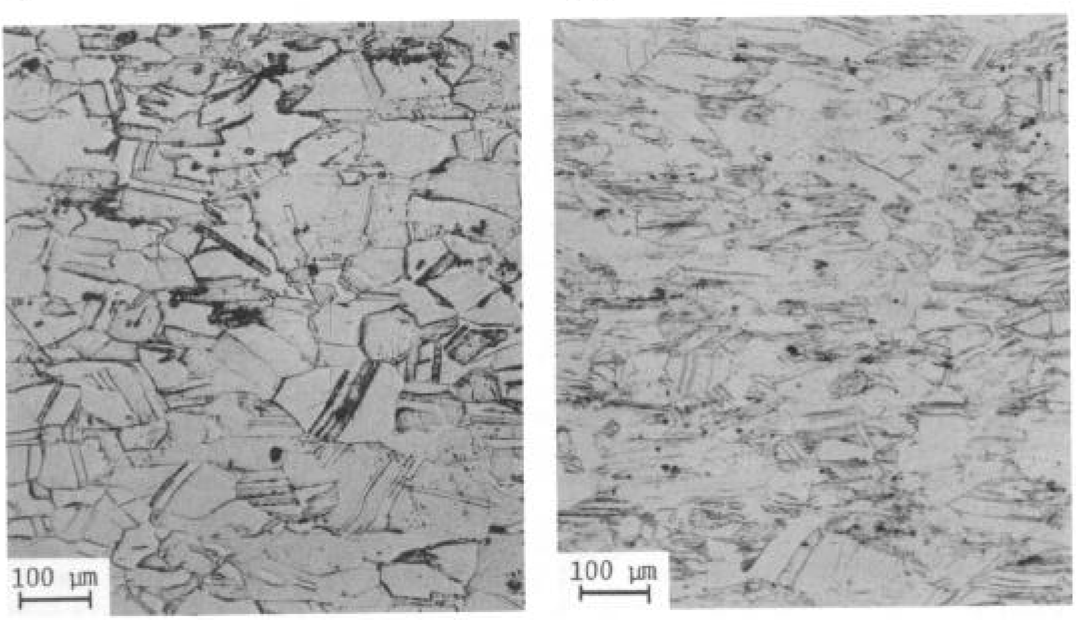

Figure 1: Optical micrograph of as-rolled material (a) 30% cold rolled, and (b) 50% cold rolled

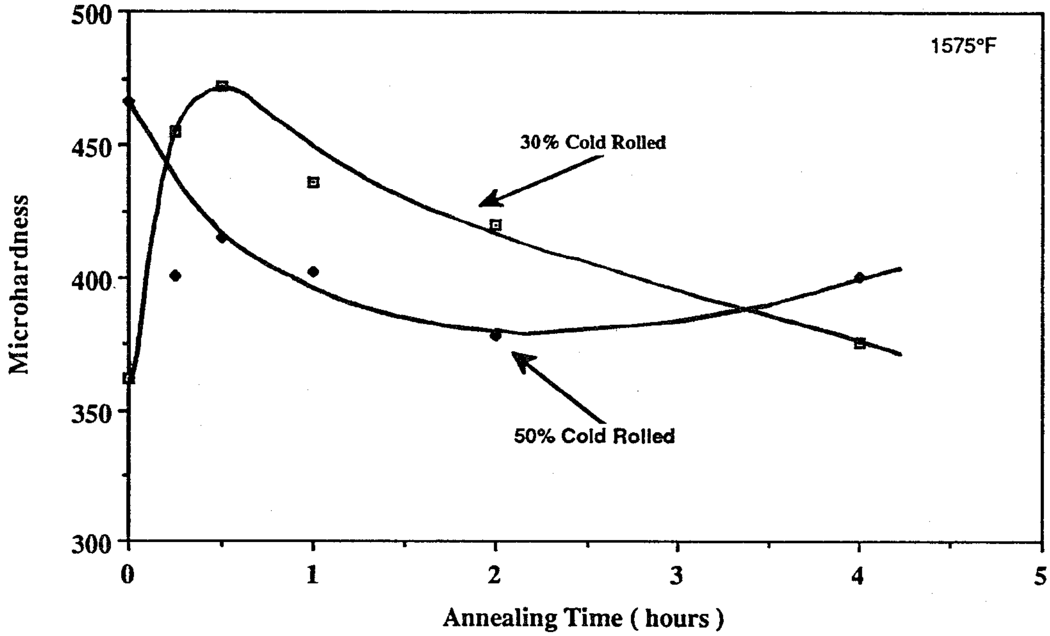

Figure 2: Microhardness Vs. annealing time at 1575°F

Figures 3(a) and 3(b) show optical micrographs of material rolled 30% and 50%, respectively after thermal exposure at 1575°F for 30 minutes. There is no apparent change in the grain structure for the 30% cold rolled material after 30 minutes exposure although an increase in hardness was observed. For the 50% cold rolled material, nucleation of strain free grains is observed at the grain boundaries and at some of the coarse carbide particles.

Microstructures after 4 hours at 1575°F are shown at 100X in Figure 4 and at 5,000X in Figure 5. The optical microstructures in Figure 4(a) and 4(b) show variations in the delta phase precipitation after 4 hours exposure for the 30% and the 50% rolled material, respectively. It is interesting to note that more of the delta phase has precipitated in the 50% cold rolled material compared to 30% cold rolled material. Precipitation of γ'' / γ' and delta phase is observed in Figure 5 at higher magnification. In the 30% rolled material precipitation of γ'' / γ' precipitates have been observed on a very fine scale and this appears to inhibit the recrystallization. In contrast, for the 50% rolled material, precipitation of γ'' / γ' is less, and delta precipitation is higher. The precipitation of delta phase does not appear to inhibit recrystallization. It seems that the lower strain enhances the precipitation of γ'' and γ' phase whereas higher strain in the 50% rolled samples appears to promote precipitation of delta phase. This behavior can be rationalized based on the slip behavior in this alloy. It is plausible that with increasing strain wavy slip may change over to planar slip forming slip bands and deformation twins as has been seen in nickel-cobalt based alloys (8). These slip

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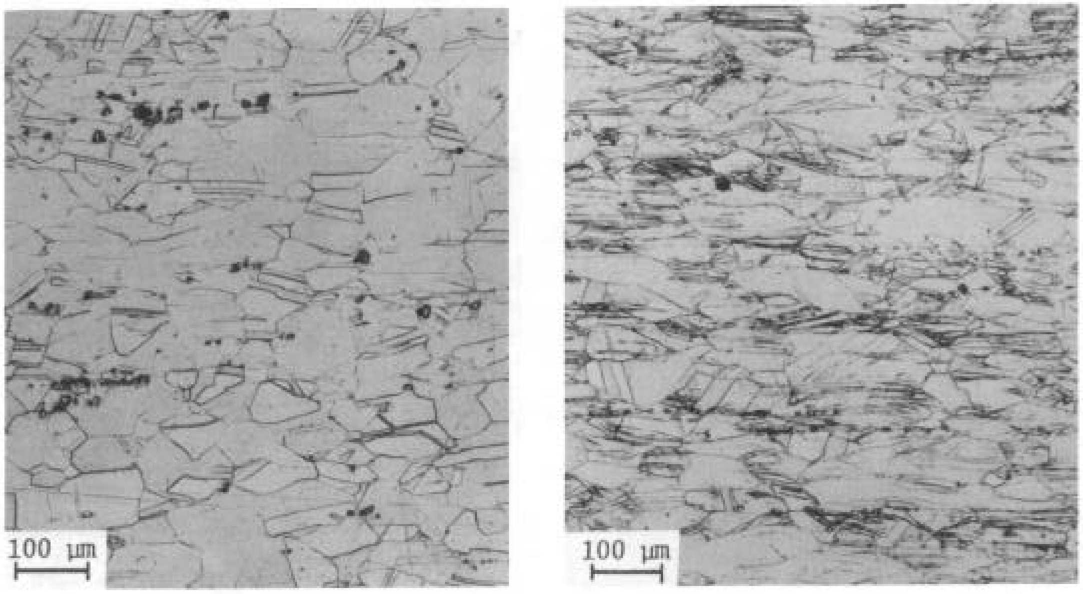

Figure 3: Microstructures after thermal exposure of 30 minutes at 1575°F (a) 30% cold rolled, and (b) 50% cold rolled (Optical)

bands and / or deformation twins may act as nucleation sites for delta precipitation and will decrease the amount of γ'' phase since there will be less niobium present to form γ''. Planar markings in the optical micrographs are indications of planar slip, however, to confirm this hypothesis transmission electron microscopy work will be carried out.

The above microstructural observations suggest that the increased hardness shown in Figure 2 for the 30% cold rolled samples heated for short times at 1575°F is due to precipitation of γ'' / γ' and some delta precipitation. There is also precipitation of γ'' / γ' and delta in the 50% cold rolled material heated to 1575°F, but rapid recovery and the on-set of static recrystallization leads to rapid softening. There is more of a driving force for the nucleation to occur in the 50% cold rolled samples than the 30% rolled. The gradual increase in the hardness for the 50% cold rolled samples as a result of exposure times longer than 2 hours appears to be due to precipitation of delta phase (see Figure 4(a) and 4(b)).

Figure 6 shows variation of microhardness with time at 1625°F. A slight initial increase in hardness for the 30% cold rolled material and a gradual decrease in hardness for the 50% cold rolled samples are observed. For the 50% rolled material, the loss in hardness at 1625°F is less rapid than the loss of hardness at 1575°F for the same heating time. Also the

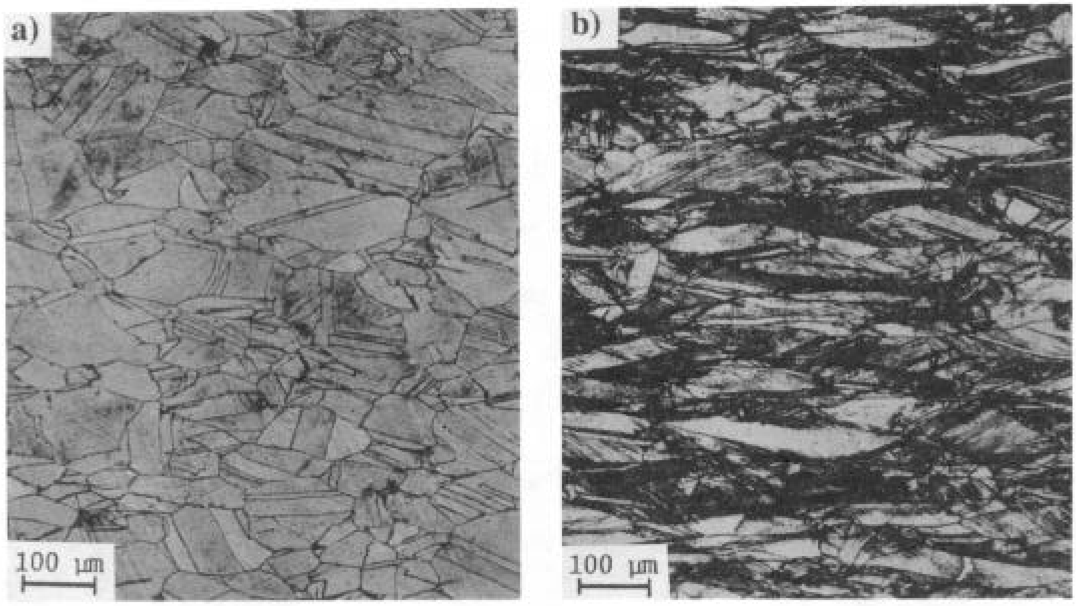

Figure 4: Microstructures after thermal exposure of 4 hours at 1575°F (a) 30% cold rolled, and (b) 50% cold rolled (Optical)

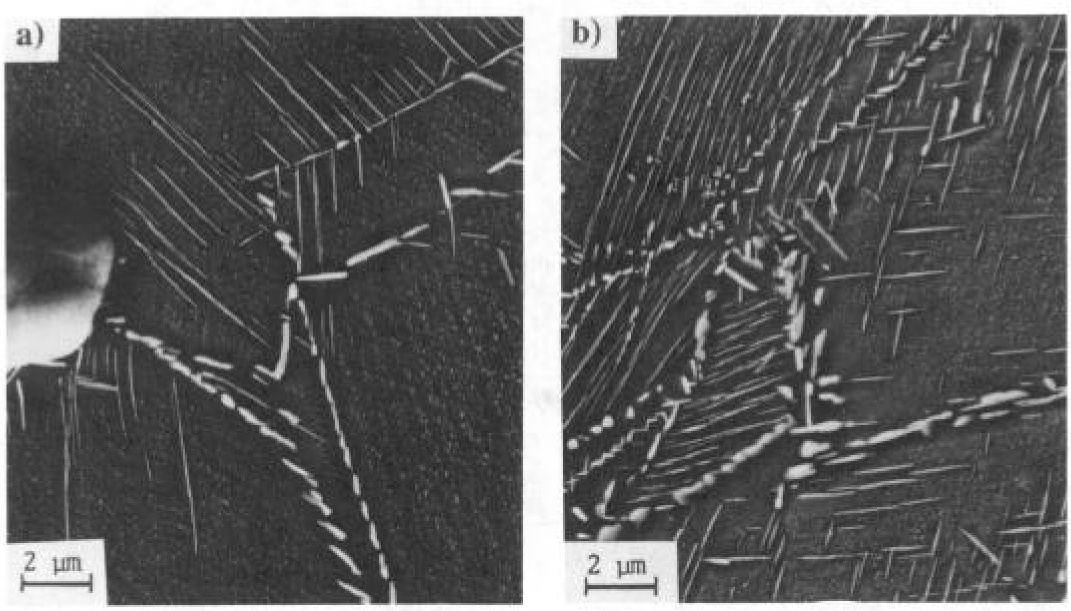

Figure 5: Microstructures after thermal exposure of 4 hours at 1575°F (a) 30% cold rolled, and (b) 50% cold rolled (SEM)

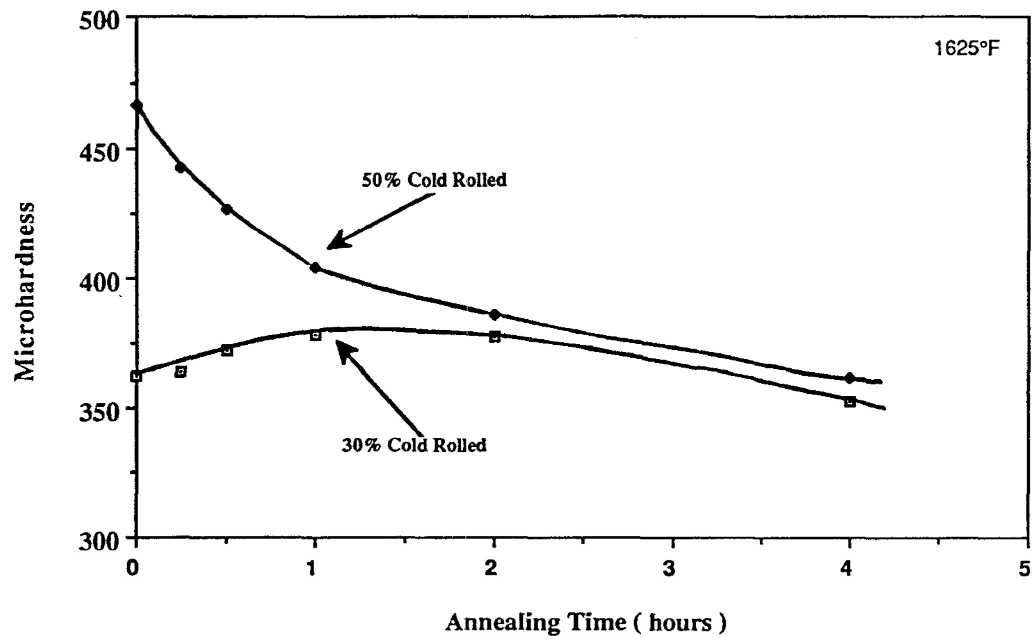

Figure 6: Microhardness Vs. annealing time at 1625°F.

increase in hardness for the 30% cold rolled samples at 1625°F is much less than the hardness gain at 1575°F for the same time. Representative optical microstructures after 30 minute and 4 hour exposures at 1625°F are shown in Figures 7 and 8, respectively. Once again there is no sizeable change in the grain structure with up to 4 hours exposure at 1625°F except that a few isolated strain free grains are seen at the grain boundaries. The precipitation of delta phase is not observed in the optical microstructures of the 30% rolled material heated at 1625°F. Precipitation of delta phase occurred for the longer annealing times, and this is shown in Figures 8(a) and 8(b). For the 50% cold rolled material, more of the strain free grains have nucleated at the grain boundaries, and this results in a necklace grain structure. The precipitation of delta phase primarily occurs at the prior grain boundaries, and this inhibits the grain growth of the newly recrystallized grains. A comparison of the precipitates for 4 hours exposure time at 1625°F in the 30% and the 50% cold rolled material is shown in Figure 9. The γ'' / γ' and delta phase are present in the 30% rolled alloy whereas only delta phase can be resolved at 5,000X in the 50% rolled alloy. The amount of delta phase has increased with increasing hold time.

Microhardness variations with annealing time are shown in Figures 10 and 11 for 1675°F and 1725°F exposures, respectively. At 1675°F, a rapid loss in hardness is observed with time up to 30 minutes for both the deformation levels. After 30 minutes, the hardness increased slightly and then remained constant. Microstructural examination revealed that after 15 minutes exposure, the 50% rolled material is fully recrystallized and precipitation of delta

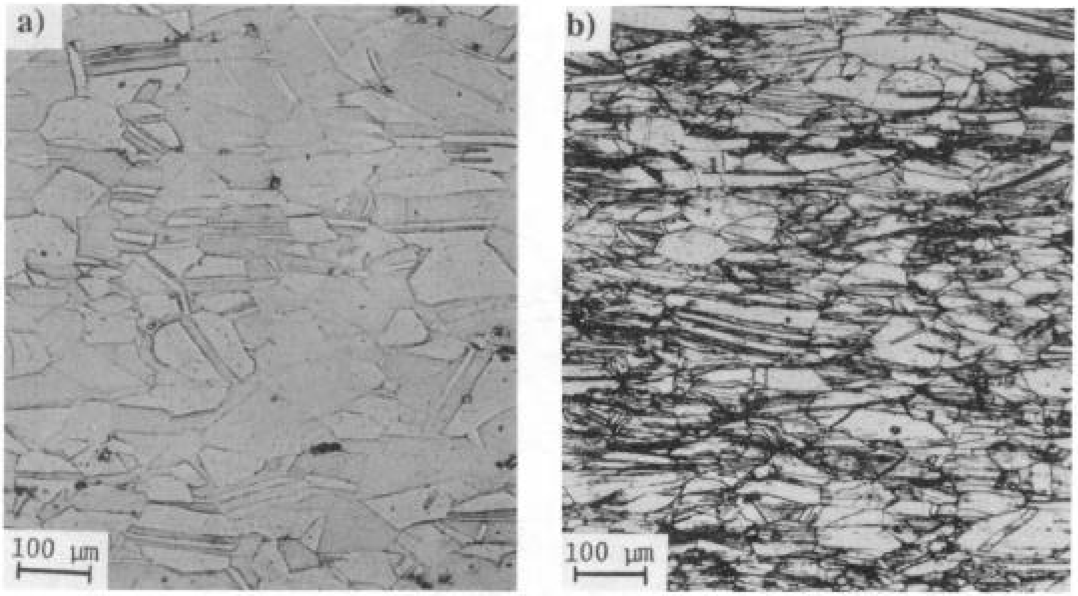

Figure 7: Microstructures after thermal exposure of 30 minutes at 1625°F (a) 30% cold rolled, and (b) 50% cold rolled (Optical)

a) b)

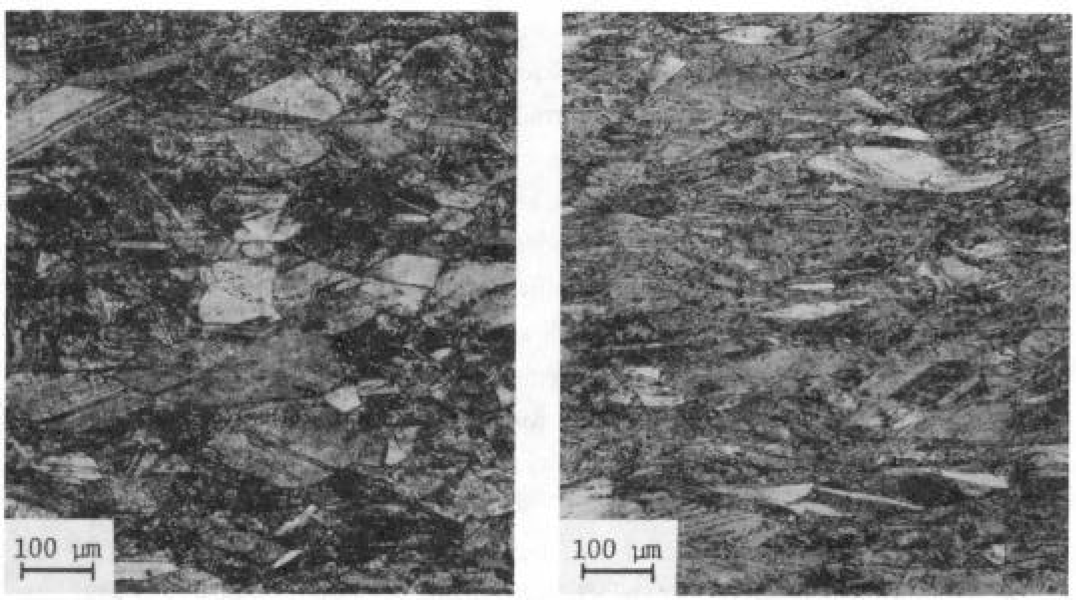

Figure 8: Microstructures after thermal exposure of 4 hours at 1625°F (a) 30% cold rolled, and (b) 50% cold rolled (Optical)

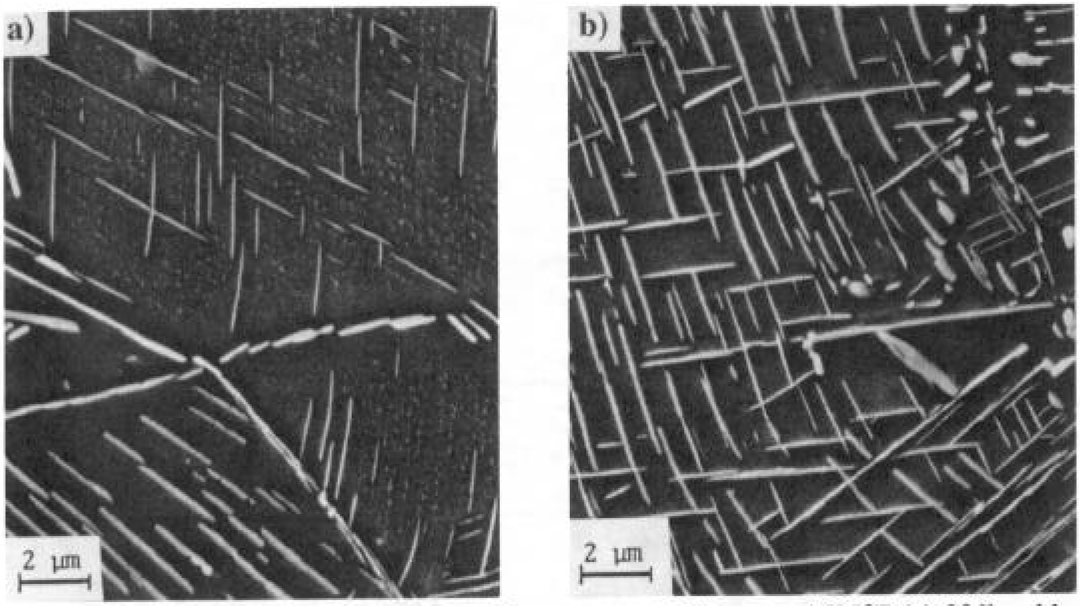

Figure 9: Microstructures after thermal exposure of 4 hours at 1625°F (a) 30% cold rolled, and (b) 50% cold rolled (SEM)

phase occurred at the prior grain boundaries. For longer holding time, delta phase precipitated in other areas as well. For the 30% rolled material, recrystallization started after 15 minutes exposure and a necklace grain structure developed. With increasing time the size of the necklace grains increased until their growth was inhibited by delta precipitation.

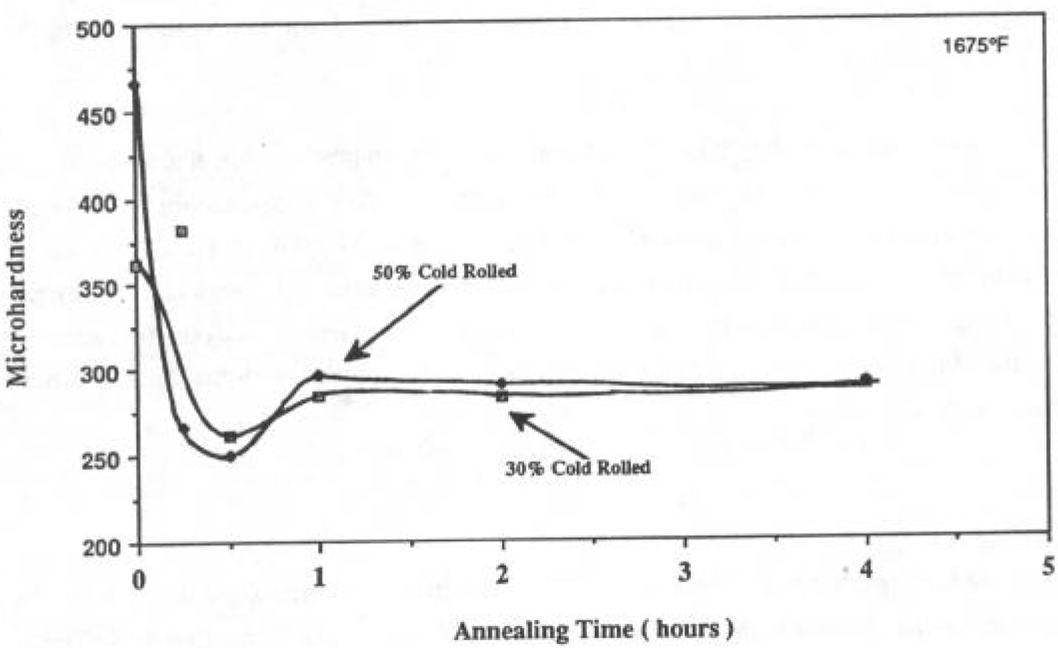

Figure 10: Microhardness Vs. annealing time at 1675°F

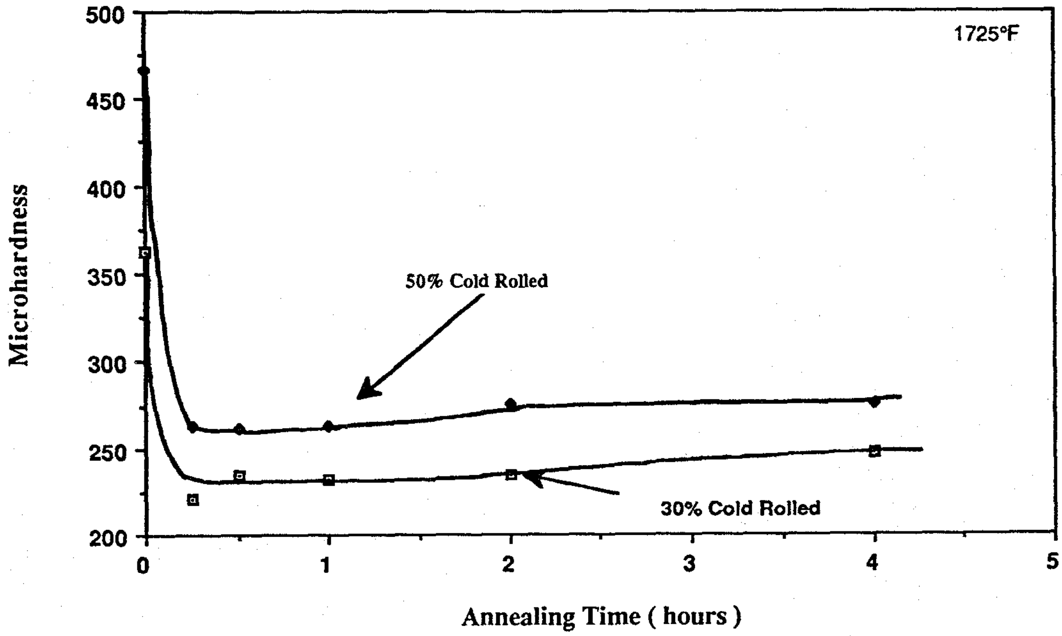

Figure 11: Microhardness Vs. annealing time at 1725°F

At 1725°F, hardness initially decreased sharply, and then remained constant. Microstructural examination showed that the 30% and the 50% rolled samples were fully recrystallized in just 15 minutes at this temperature. Precipitation of delta phase occurs at the prior grain boundaries before recrystallization occurs, and then continues at the grain boundaries of the recrystallized grains and in the grain interiors. It is interesting to note that the hardness after 2 and 4 hours is higher for the 50% rolled alloy compared to the 30% rolled alloy. This appears to be due to finer recrystallized grain size resulting for the 50% rolled material than for the 30% rolled material.

The above results show very interesting interactions between precipitation, and recovery and recrystallization in cold rolled Alloy 718. Precipitation of γ'' / γ' appears to inhibit recovery and recrystallization even at a reletively high temperature of 1575°F (0.75 T_m). Precipitation of delta phase inhibits grain growth as has been demonstrated by others (6). Additional studies of these interactions in Alloy 718 using differential thermal analysis, transmission electron microscopy and scanning electron microscopy coupled with the microhardness changes are proceeding.

Conclusions:

1) At lower temperatures of 1575°F and 1625°F, an initial hardening followed by a softening with increasing annealing time for the 30% cold rolled material, and a continuous softening for the 50% cold rolled material were observed. These changes are attributed to a difference in precipitation of γ'' / γ'. For the 30% cold rolled material, effects of precipitation are more

pronounced than the effects of recovery and recrystallization which are also occurring. For the 50% cold rolled material, the driving force for recovery and recrystallization as evidenced by a continous softening are much stronger than the effects of precipitation of γ'' / γ'.

2) At higher temperatures of 1675°F and 1725°F for both 30% and 50% cold rolled material, hardness initially decreased rapidly, then increased slightly and remained constant. This behavior is a result of rapid initial recovery and recrystallization followed by precipitation of delta phase which inhibits grain growth.

3) Precipitation of γ'' / γ' appears to inhibit recovery and recrystallization, and delta precipitation appears to inhibit grain growth during high temperature anneal of cold rolled Alloy 718.

4) At the lower annealing temperatures, precipitation of γ'' / γ' is more prevalent than precipitation of delta for the 30% cold rolled material. However, precipitation of delta phase was more favorable than precipitation of γ'' / γ' for the 50% cold rolled material.

5) Delta phase first precipitates in the grain boundaries of the cold rolled material, and is observed both at grain boundaries and intergranular locations of the newly recrystallized grains.

References:

1) "Recrystallization of Metallic Materials" Edited by: F. Haessner, Dr. Riederer Verlag GmbH, Stuttgart, (1978)
2) P. Cotterill and P.R. Mould: " Recrystallization and Grain Growth in Metals", John Wiley & Sons, New York, New York, (1976)
3) " Annealing Process - Recovery, Recrystallization and Grain Growth", Edited by: N. Hansen, D.J. Jensen, T. Leffers and B. Ralph, Risø National laboratories, Roskilde, Denmark, (1986)
4) U. Koster: in "Recrystallization of Metallic Materials" (Edited by: F. Haessner), Riederer Verlag GmbH, Stuttgart, (1978), pp 215
5) J.W. Martin and R.D. Doherty: "Stability of Microstructures in Metallic Systems", Cambridge University Press, (1976)
6) " Superalloy 718- Metallurgy and Applications" Edited by E. Loria, TMS, (1989)
7) P.J. DiConza, R.R. Biederman and J.L. Burger: in Microstructural Science, Vol. 18, (Edited by: T.A. Place, J.D. Braun, W.E. White and G.F. VanderVoort), IMS, Columbus, Ohio & ASM International, Materials Park, Ohio, (1990)
8) R.P. Singh: Ph.D. Thesis, Drexel University, Philadelphia, PA , (1987)