MICROSTRUCTURAL REFINEMENT OF AS-CAST ALLOY 718

VIA THERMOMECHANICAL PROCESSING

C. I. Garcia+, D. E. Camus+, E. A. Loria* and A. J. DeArdo+

+Basic Metals Processing Research Institute
Department of Materials Science and Engineering
University of Pittsburgh
Pittsburgh, PA 15261

*Niobium Products Company, Inc.
Pittsburgh, PA 15108

ABSTRACT

Technology demand for improved tensile and fatigue properties in the final components requires Alloy 718 producers to develop a fine grain practice for the cogged billet derived from the ingot. The effect of thermomechanical processing on the microstructural refinement of Alloy 718 was studied. The results of this study revealed that under certain thermomechanical processing conditions, substantial microstructural refinement can be obtained. Large portion of the softening associated with the recrystallization process did not occur by the formation of new, strain free grains via the motion of high angle boundaries, but rather, by the formation of dislocation free annealing twins that grow directly into the deformed matrix.

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INTRODUCTION

In the last three decades the use of Alloy 718 in the aerospace and other demanding environmental applications has gained considerable acceptance. The popularity of Alloy 718 compared to other superalloys originates from its excellent package of mechanical properties and cost-effectiveness. However, in order to maintain its advantageous position with the fabricators and end users, the alloy producers must be able to generate a finer and more uniform microstructure through the conventional forging of large ingots (i.e. 21 in. in diameter). The conventional commercial processing of alloy 718 leads to a very heterogeneous grain structure which is carried through the final forging and heat treatments. This heterogenous structure leads to a marked deterioration in performance.[1] The value of having a uniform and fine microstructure after the ingot-to-billet conversion process is enormous, since, this material will then be used as feedstock for the production of turbine disks and other components. Despite the recognized importance of achieving the proper control (i.e. refinement) of the final microstructure in the cogged billet, there has been little systematic work done on the effect of thermomechanical processing (TMP) during the ingot-to-billet conversion of Alloy 718. Most of the studies conducted on the influence of TMP on Alloy 718 have been conducted using a relatively small initial grain size as the starting condition (i.e. during the billet-to-disk forging conversion process) and the results have been reported in the literature.[2-6].

The major goal of the work described in this paper was directed to define the conditions under which substantial microstructural grain refinement of Alloy 718 can be achieved through proper TMP during the ingot-to-billet conversion practice. The approach taken was to simulate the commercial operation in forging a 21 in. diameter ingot into a 10 in. diameter billet. Of particular interest in this study were: a) the definition of the recrystallization-stop temperatures under dynamic and static conditions, b) the determination of temperature-deformation-microstructure maps, and c) the assessment of the stability of the as-deformed structures.

GRAIN REFINEMENT CONSIDERATIONS

Most engineering materials are required to exhibit certain levels of several different properties in order to be technologically and economically successful. The principal properties of interest in Alloy 718 are strength, ductility, fatigue strength (LCF and HCF), fracture toughness and creep resistance. The fundamental understanding of the relationship between microstructure and properties in Alloy 718 has led to the definition of the critical parameters responsible for strength and fracture toughness. For example, it is well-known that high strengths can be obtained in a simple manner, e.g. through the addition of solute strengthening or precipitation hardening elements. High fracture toughness, on the other hand, cannot be achieved through alloy additions, but must be achieved through the control of the final microstructure. Furthermore, it is well-recognized that tough microstructures exhibit fine grain sizes. Hence, one of the goals of TMP is achieving large amounts of fine grains. The major challenge then becomes how these two goals may be achieved.

The microstructural refinement in plain carbon and microalloyed steels, Cu-alloys, Al-alloys, and Ti-alloys can lead to a substantial improvement in their mechanical, physical and chemical properties. Recent studies[7-15] have shown that the proper combination of TMP followed by heat treatment can lead to significant levels of microstructural refinement provided the ratio of the rates of nucleation (N) to growth (G) is large. Hence, fine final microstructures will be achieved when the N/G ratio is large. Therefore, proper TMP followed by controlled transformation processes is a very cost-effective way of controlling final microstructure and properties.

In general, the level of microstructural refinement by TMP is strongly related to three major factors[9]: a) the reheating behavior, b) the hot deformation schedule, and c) the transformation behavior. During the reheating period a series of events may occur, these include: (1) the nucleation, growth and possible coarsening of the grains, (2) the reduction of inhomogeneities in solute distribution and (3) the dissolution of precipitation species which were inherited from the original as-cast microstructure. The traditional ingot-to-billet conversion practice of Alloy 718 results in a very heterogeneous grain structure prior to final forging and heat treatment. This broad grain size distribution is carried through the forging and heat treatments, and results in higher creep rates and inferior mechanical properties. The application of proper TMP to Alloy 718 will lead to a processing path which will yield structures of desired homogeneity and grain size.

The typical high temperature as-deformed microstructures of most metals and alloys can show three types of microstructures: fully recrystallized, partially recrystallized and fully unrecrystallized. Each type of these microstructural conditions describes a definite grain boundary area per unit volume. In general, since the final grain size is related to the number of sites for the nucleation of new grains, and since this number is related to the grain boundary area per unit volume in the deformed structure, the final grain size after recrystallization will decrease as this boundary area increases. One way to describe this number is by using the parameter S_V.[16] The parameter S_V quantitatively assesses the number of pre-existing or strain-induced heterogeneities introduced into the γ phase during high temperature deformation (i.e. rolling or forging) which could act as sites for the nucleation of new grains. The parameter S_V (mm^{-1}), is formally defined as the effective interfacial area per unit volume. The effect of as-reheated grain size and rolling reductions below the recrystallization-stop temperatures on S_V is shown in Figure 1.[8]

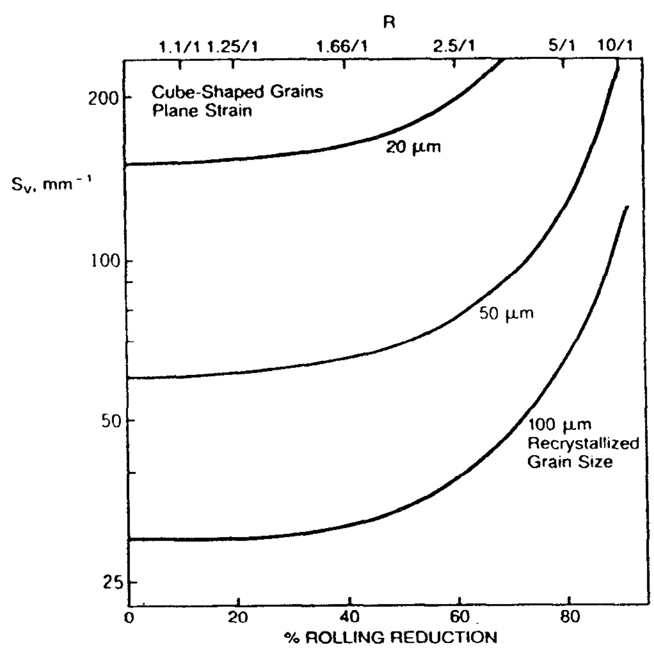
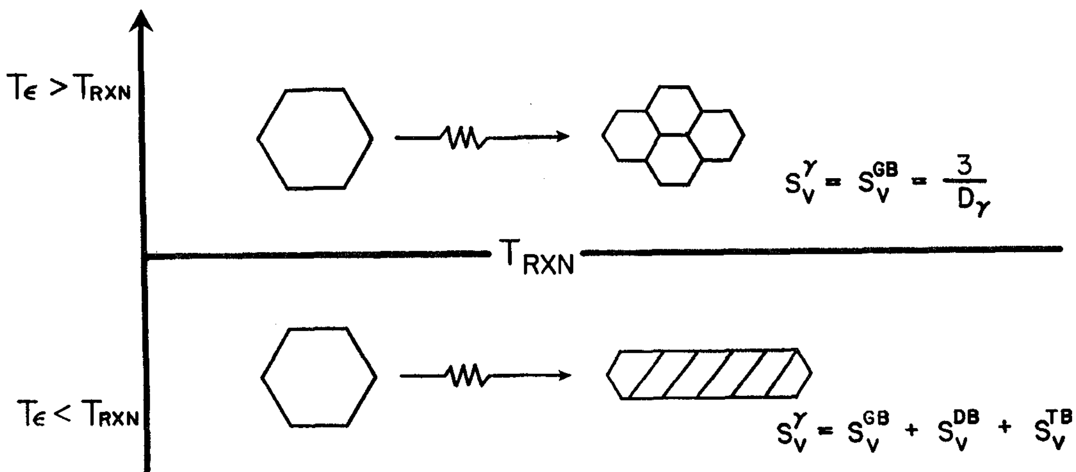


Figure 1. Effect of rolling reduction on S_V for cube-shaped austenite grains.[8]

From the above discussion, it is clear that one of the principal goals of thermomechanical processing is to have well-conditioned microstructures, i.e. high S_V values. Since S_V is comprised of near-planar crystalline defects such as grain boundaries, deformation bands and twin boundaries, the control of recrystallization during thermomechanical processing is critical, because it dictates the size and shape of grains as well as the presence or absence of the intragranular crystalline defects. The ability to produce large S_V values comes from the full understanding of the microstructural behavior during TMP of the given metal or alloy system. For example, two of the most common thermomechanical processing approaches which lead directly to fine, equiaxed grains after subsequent heat treatment are: a) recrystallization controlled rolling or forging (RCR or RCF)[11,12,14,15] and b) conventional controlled rolling or forging (CCR or CCF)[7,10,13]. These two types of TMP illustrating the associated microstructural behavior, are schematically illustrated in Figure 2. This figure also describes the pertinent S_V values. The superscripts GB, DB, TB, and NPD denote the contribution to the total S_V from grain boundaries, deformation bands, twin boundaries and near planar defects, respectively. The use of RCR or RCF involves repeated recrystallization of the grains during deformation above the recrystallization temperature (T_{RXN}) leading to a fine, equiaxed grains. However, to successfully utilize this type of TMP, the alloy system must have a mechanism to inhibit the grain coarsening which follows recrystallization. Alloys not designed for RCR undergo static recrystallization following each high temperature deformation. This is followed by rapid grain coarsening in the remaining interpass time. Hence, this leads to coarse, as-rolled or as-forged grain sizes.



$$S_V^{GB} = \frac{1}{D}\left(1 + \frac{1}{R} + R\right) \quad mm^{-1}$$

$$S_V^{NPD} = S_V^{DB} + S_V^{TB} = 0.63(\%R - 30) \quad mm^{-1}$$

Figure 2. Schematic representation of microstructural behavior after TMP above and below T_{RXN}.

Conventional controlled rolling or forging means that all the deformation takes place below the T_{RXN}; the grains become highly elongated and, at a sufficiently large strain, become filled with intragranular crystalline defects such as deformation bands and twins, i.e. large S_V.

The large number of studies published in the literature leaves no doubt that the proper choice of TMP provides a practical and cost-effective way to optimize the final microstructure of metals and alloys.

EXPERIMENTAL PROCEDURE

HOT DEFORMATION STUDIES

A) Sample Preparation

Transverse 2 in. thick slices were cut from the top, middle and bottom of a 21 in. diameter VAR ingot of Alloy 718 which had been homogenized using standard treatment cycles.[17] The chemical composition in wt% of the ingot as well as the overall sample sectioning is shown in Figure 3. From these sections cylindrical compression specimens with dimensions 0.75 in. height x 0.5 in. diameter were machined. In addition, in order to monitor the specimen temperature during the deformation process, a hole 0.0625 in. in diameter was drilled into each cylinder at mid-height. An additional modification was made to the geometry of the compression specimens in order to minimize the effects of friction between the die-specimen surface. The final shape of the compression samples is what is known as the modified Rastegaev's[18] design. The combination of the sample geometry and the use of proper lubricant for the range of testing temperatures is very effective in decreasing friction and ,hence, barreling effects during high temperature compression testing.

B) Testing

The hot deformation testing was performed using a computer-controlled MTS machine dedicated to high temperature deformation studies. The MTS system has a load capacity of 50,000 pounds and stroke ranges from $\pm$ 0.5 to $\pm$ 5.0 inches. The in-situ furnace equipped with atmosphere controls can achieved temperatures up to 2460^{o}F (1350^{o}C) at heating rates of 900^{o}F (500^{o}C) per minute. The dies and the specimens to be deformed are heated and maintained at the same temperature. The computer generates the crosshead movement algorithm such that constant or desired changes in strain rate can be produced throughout the deformation whether a single or multi-hit test is being performed. Strain rates in the range 0.001 s^{-1} to 75 s^{-1} are attainable. During all deformation sequences, up to 9000 data points per second (i.e. load, stroke, temperature) can be recorded and stored. The hot deformation schedule conducted in this study is shown in Figure 4. The samples were deformed at various temperatures from 2150^{o}F (1177^{o}C) to 1550^{o}F (843^{o}C). The deformations studied were single hits at ϵ= 0.2, 0.5, and 1.0, with a constant strain rate of 0.5 s^{-1}. The samples after each deformation were either immediately quenched in water or held at temperature from 10 to 100 seconds prior to quenching. For comparison purposes a typical commercial forging schedule for the conversion of Alloy 718 from a 21" diameter ingot to 10" billet is also shown in Figure 4.

DILATOMETRY

Prior to hot compression testing, the samples in the homogenized condition were subjected to dilatometry studies. The goal of this work was to have an understanding of the effect of the prior homogenization treatment and cooling rate on the transformation characteristics of Alloy 718. The results of this study led to the construction of a series of continuous cooling transformation (CCT) diagrams. A complete study of the effect of TMP on CCT and corresponding high resolution microscopy of the resulting microstructures will be presented in a separate paper.[17] The dilatometry studies were conducted in a modified Tetha Dilatronic V system. This system has been modified to obtain cooling rates from 100^{o}C/s to 0.001^{o}C/s.

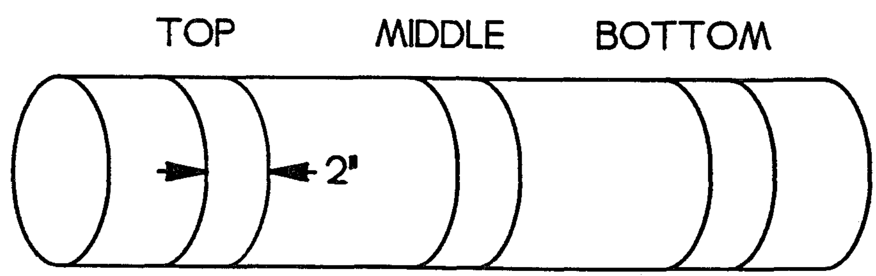
MICROSTRUCTURE

The samples for microstructural examination were prepared using standard metallographic techniques. The etching solution used to reveal the microstructure of the samples prior to and after hot deformation consisted of: 50 ml H_2O (distilled) + 40 ml

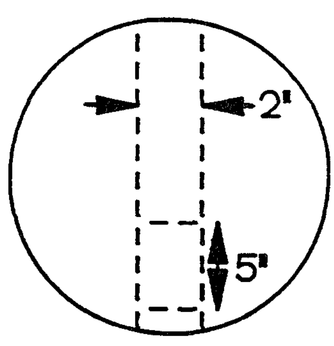
HCl + 10 ml HF + 2-3 ml H_2O_2. The samples were immersed in the solution for 10 to 40 seconds. The quantitative characterization of the microstructure was conducted using a Nikon Epiphot-TME inverted optical microscope. This system is fitted with a video camera which links the microscope to an Automated Bioquant Meg Image Analyzing System.

CHEMICAL COMPOSITION (WT %)

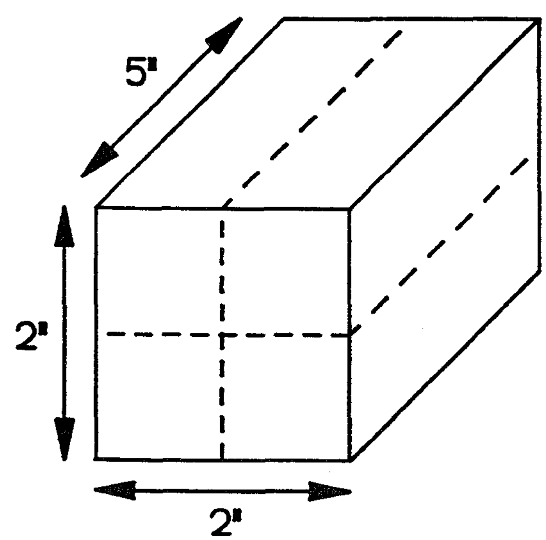
Ni	Fe	Cr	Nb	Mo	Ti	Al	Cr
51.5	19.9	18.2	5.1	2.9	1.15	0.57	0.035



21 inch ϕ As-Cast Ingot.



Slice from center of each disk.



Sample oriented transverse to ingot axis.
three samples per quarter. Total = 12.

Figure 3. Chemical composition (wt%) of Alloy 718 and schematic diagram of 21" diam. ingot and location of samples used in this study.

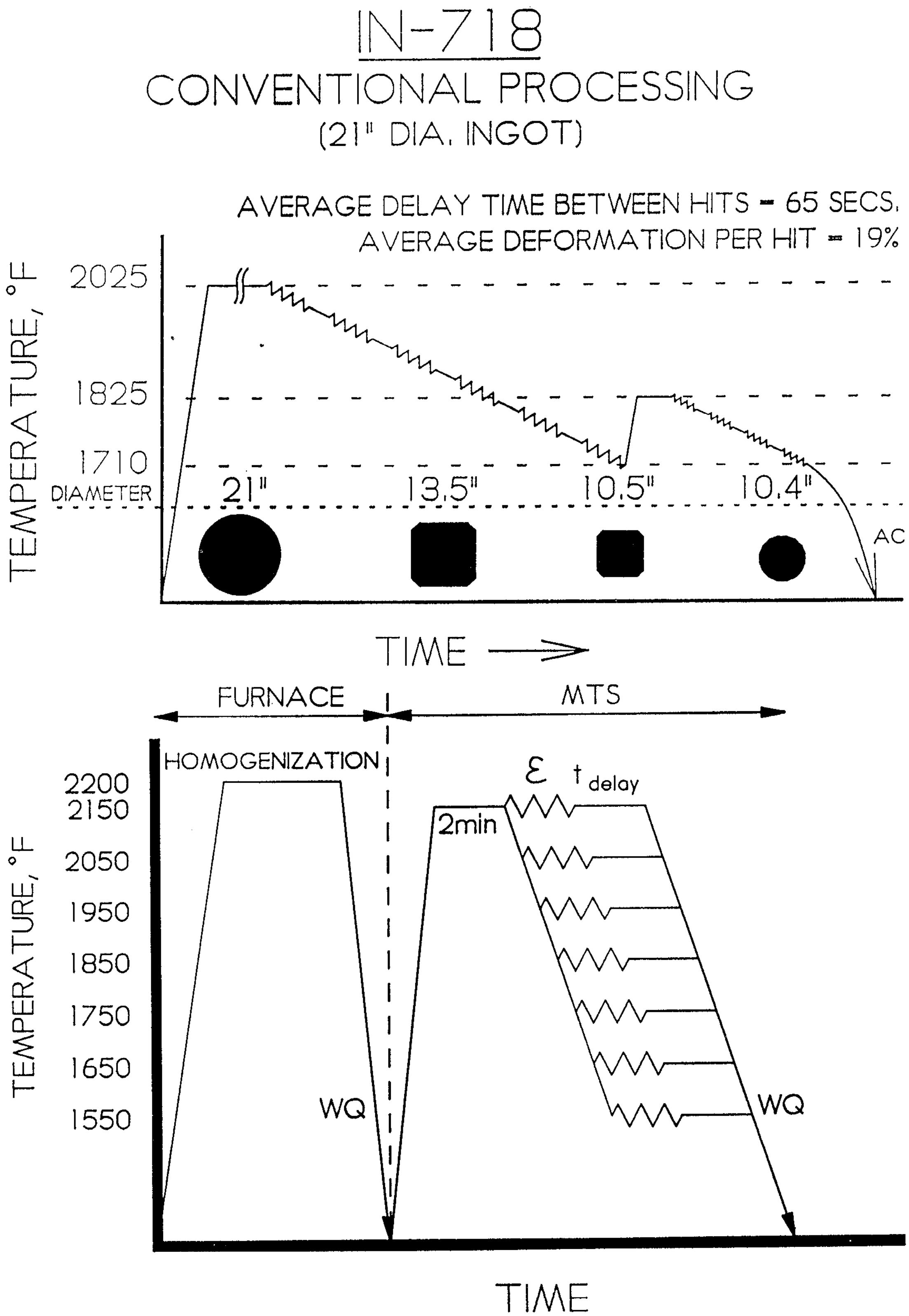


Figure 4. Schematic representation of experimental TMP and typical commercial forging process.

RESULTS AND DISCUSSION

DILATOMETRY

The knowledge of the as-reheated microstructure and subsequent transformation characteristics after cooling in terms of the constituents present in the structure (e.g. composition, grain size, precipitates, etc.) is extremely important. These conditions will affect the response of the material to TMP. Hence, a first hand knowledge of the microstructural conditions prior to TMP is necessary, since this information will aid in the understanding of the recrystallization behavior of the microstructure during and after TMP. One of the tools available for this purpose, is the CCT diagrams. In the present study, the CCT diagram for Alloy 718 was determined and is illustrated in Figure 5. The CCT diagram indicates that in the typical temperature range where the forging of Alloy 718 takes place (see Figure 4), the microstructural constituents are γ, δ, large undissolved NbC (inherited from solidification) and minute amounts of Laves phase. The presence of small amounts of Laves phase was also observed in samples which were homogenized 72 hrs at 2156°F (1180°C).[18] It seems that the Laves phase and the large NbC particles are not fully dissolved during typical or extended homogenization treatments.

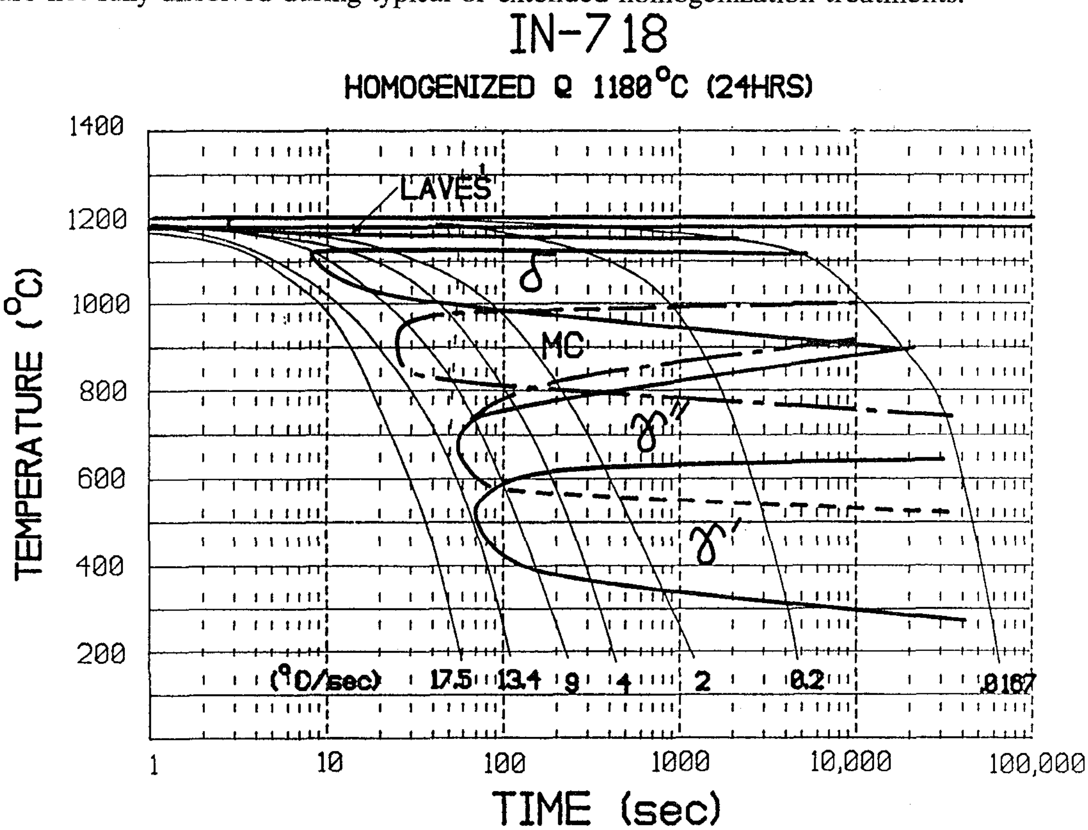


Figure 5. Continuous cooling transformation diagram of Alloy 718.

FLOW STRESS BEHAVIOR

Hot flow curves from continuous compression testing are shown in Figure 6. The flow stress behavior of Alloy 718 with temperature shows similar behavior described in previous studies.[4,6] That is, at deformation temperatures ≥ 2050°F (1121°C) there is a sharp yield drop. As the deformation temperature decreases ≤ 1950°F (1065°C) the yield point starts to disappear and substantial work hardening takes place. The presence of a yield point in Alloy 718 has been attributed to carbides or γ" precipitation on dislocations or stacking faults[4,6] as well as to short range ordering[19]. However, these previous studies did not present microstructural evidence of the pinning forces (i.e. carbides or γ'' precipitates).[4,6] The dilatometry results presented in Figure 5, indicates that in temperature range where the yield drop is observed, the only particles present are δ, large NbC and perhaps fine NbC which reprecipitated during TMP.

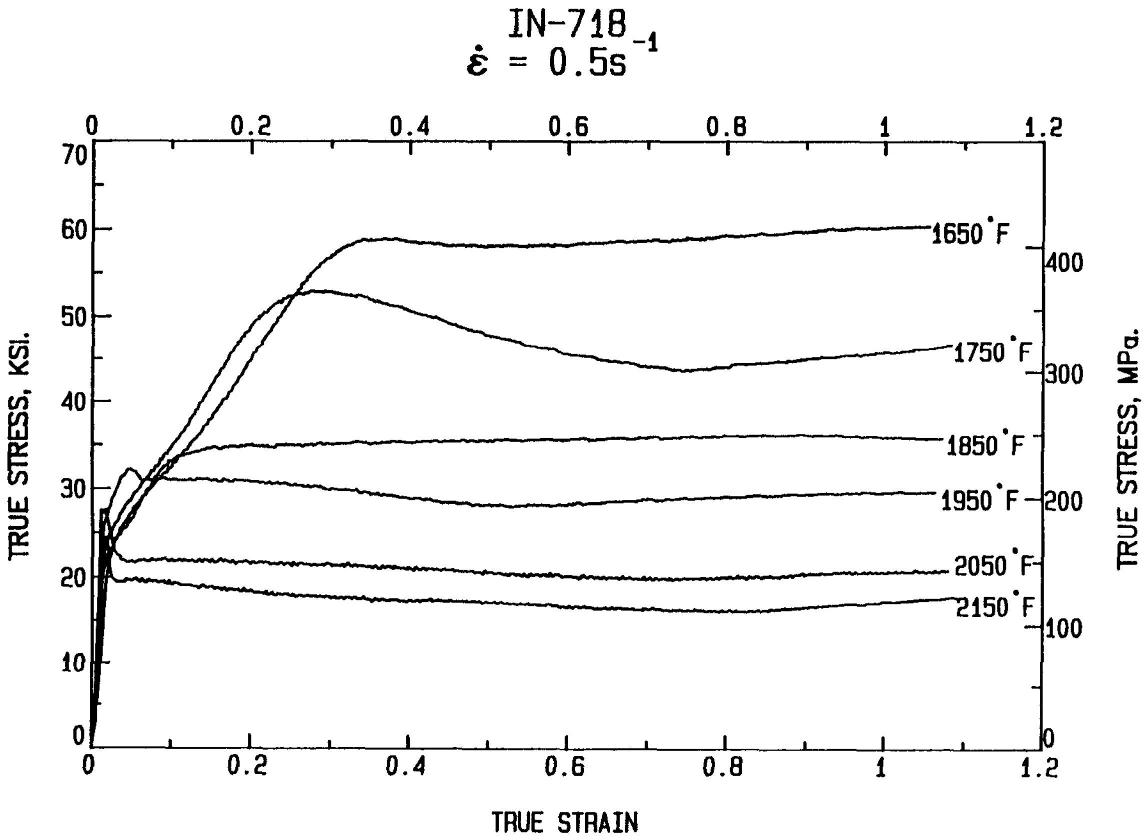


Figure 6. Hot flow curves of Alloy 718 during isothermal compression.

<u>RECRYSTALLIZATION BEHAVIOR</u>

It is well-known that the restoration process (i.e. recovery[20], recrystallization[21] and grain growth[20]) which occur during TMP is dependent on the balance between the driving force (i.e. stored energy of deformation) and the retarding forces (i.e. solute drag and particle pinning) for restoration. When the driving force for a restoration process exceeds all retarding forces, the process will proceed. However, when the retarding forces exceed the driving force, the restoration process will not occur, and the as-deformed condition will prevail. The attainment of substantial microstructural refinement, high S_V values, clearly depends on the ability to control the restoration processes which could otherwise occur during TMP. Obviously, for a given TMP, this means that the level of retarding force must be controlled during the process. This point is clearly illustrated in Figure 7.[22] This figure shows the temperature-deformation-recrystallization map for a Nb-Ti steel. One of the major results presented in Figure 7 is the significant enhancement in microstructural refinement achieved when the initial grain size is small. For example, for a deformation per pass of 20%, the $T_{RXN}^{100\%}=R_f$ for the condition with large initial grain size (160 μm) is about 2102°F (1150°C) and the resulting recrystallized grain size is about 70 μm. This is to be compared with the case where the steel with smaller initial grain size (20 μm) leads to a R_f temperature which is about 234°F (112°C) lower and has a recrystallized grain size of about 22 μm. Figure 7 also shows that the recrystallized grain size for a given rolling reduction is almost independent of the deformation temperature. In summary, the reduction in initial grain size causes the recrystallization to be accomplished at lower temperatures and with lighter deformations.

The importance of having a small initial grain size prior to TMP is clearly seen in Figure 7. This is a similar condition where most of the studies on the recrystallization behavior of Alloy 718 have been conducted. That is, the average initial grain size has been about 200 μm or smaller.[2-6] Hence, under these conditions, it is relatively easy to obtain substantial microstructural refinement, since most likely a large portion of the deformation process takes place above the T_{RXN}. While achieving microstructural optimization through TMP in metals or alloys with small initial grain sizes is rather simple, the opposite is true when the initial grain size is large. This is especially true when practical limitations are imposed (i.e. equipment loads, deformation per pass, etc.).

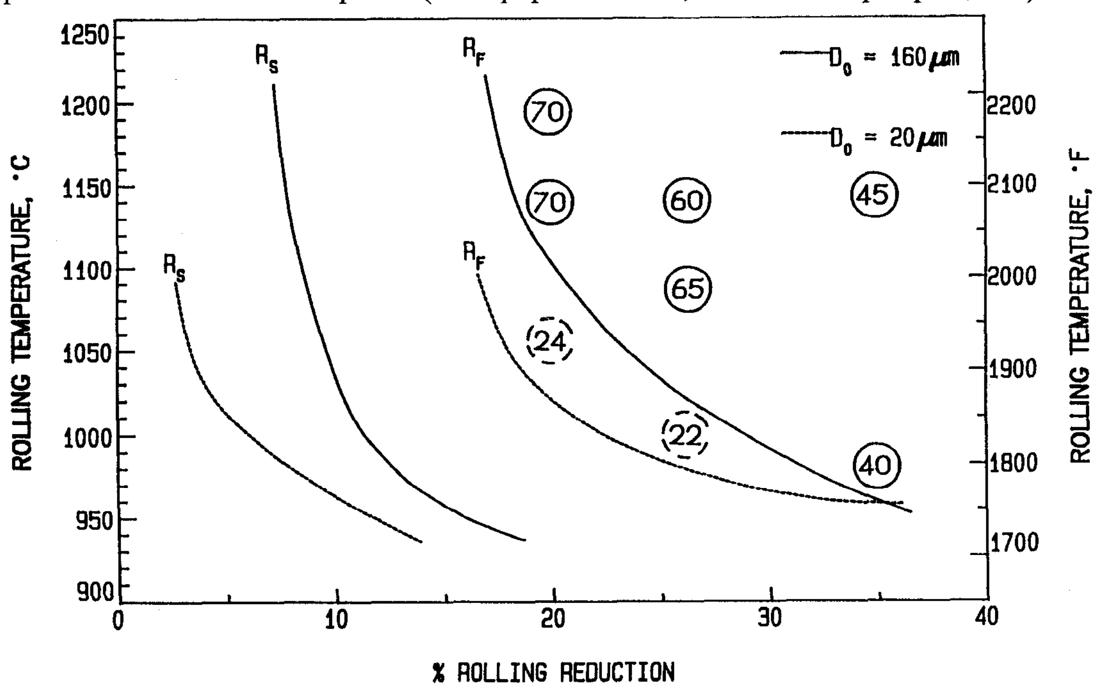


Figure 7. Relationship between deformation temperature, %deformation and recrystallization behavior for a Nb-Ti Steel. Circled numbers are recrystallized grain size in μm.[22]

The temperature-recrystallization-time surface map of Alloy 718 determined at a ϵ=0.2 and $\dot{\epsilon}$=0.5 s^{-1} at all temperatures is presented in Figure 8. This figure describes the recrystallization behavior of Alloy 718 under dynamic and static conditions. The time coordinates represent the holding time at temperature prior to quenching. The results shown in this figure indicate a series of points: (1) at deformations equal to or less than ϵ=0.2, full dynamic recrystallization can take place if the deformation process occurs at very high temperatures, that is above 2150°F (1177°C); (2) at lower temperatures, about 1960°F (1071°C), full recrystallization can be obtained under static conditions; (3) Partial recrystallization is obtained between 1960 and 1780°F (962°C); (4) below 1780°F the fully unrecrystallized zone is observed. Similar type of recrystallization maps were obtained for a different TMP conditions and a summary of these results is presented in Figure 9. This figure schematically defines the recrystallization-stop temperatures, $T_{100\%}$ and $T_{0\%}$, for Alloy 718 under dynamic and static conditions for the TMP used in this study.

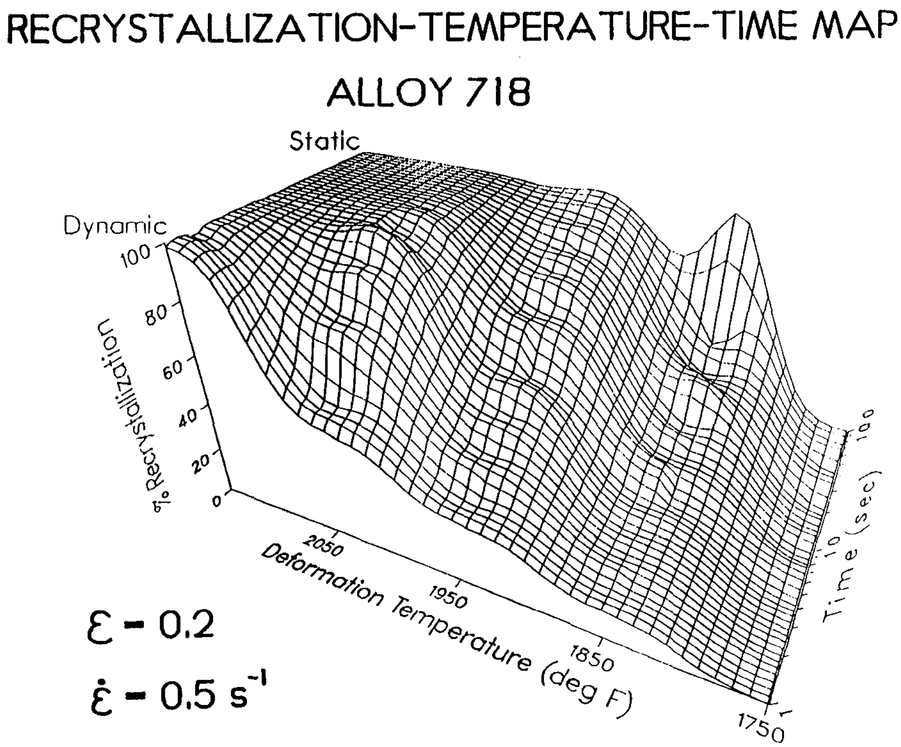


Figure 8. Recrystallization-Temperature-Time surface map for Alloy 718 under dynamic and static conditions.

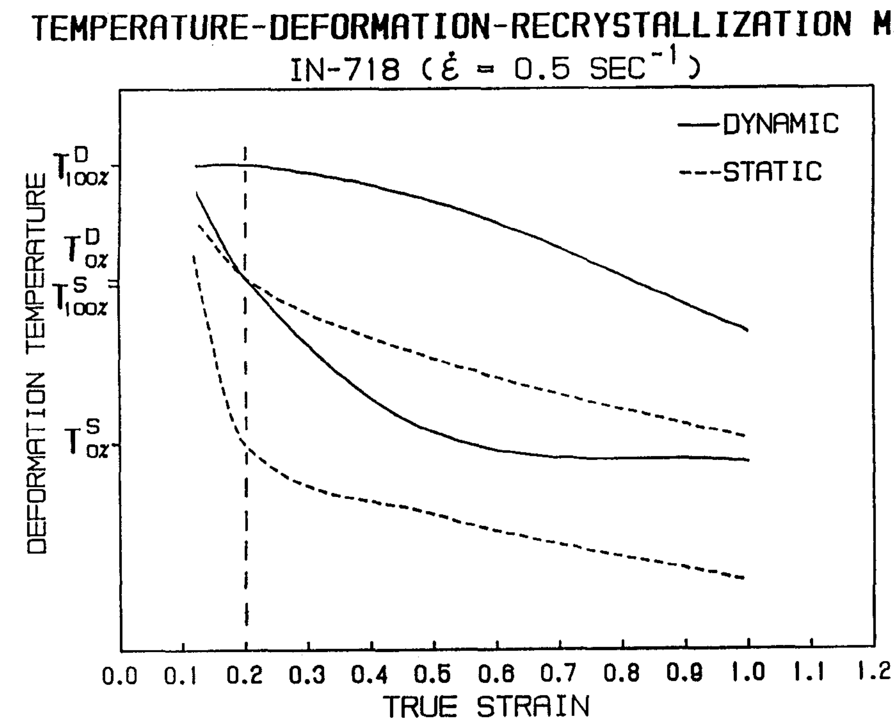
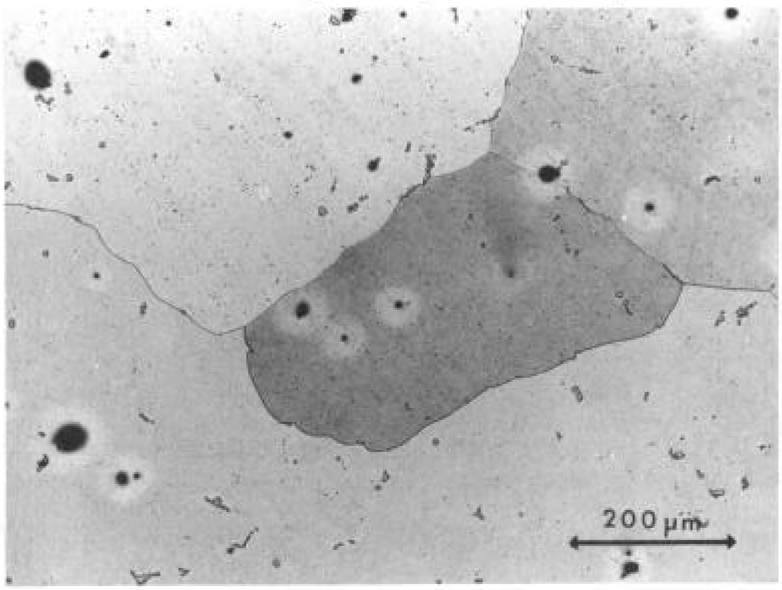


Figure 9. Schematic Temperature-Deformation-Recrystallization map for Alloy 718 under dynamic and static conditions.

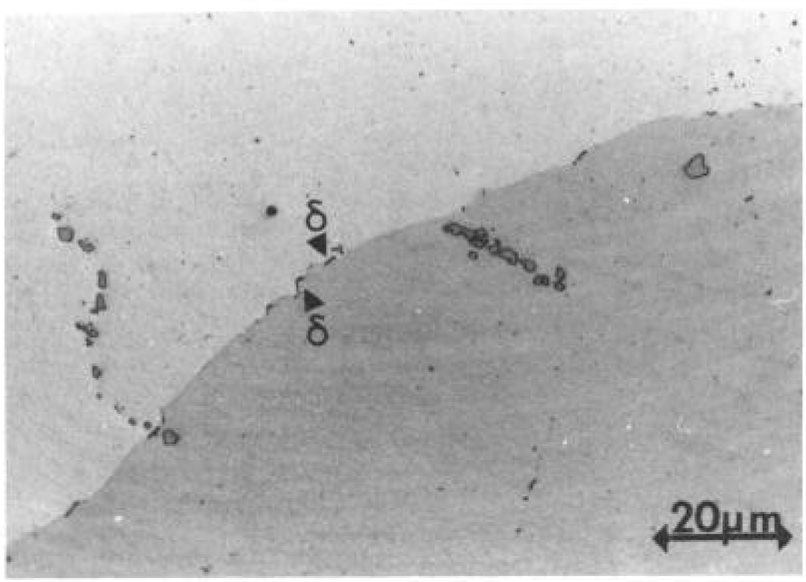
MICROSTRUCTURE

One of the most important microstructural features is the size of the as-reheated grains, since good microstructural conditioning is a direct result of proper TMP. That is, each element of TMP (reheating, rolling or forging, and cooling) must be controlled. Since these elements may each contribute to the success or failure in obtaining optimum microstructural conditioning.

The typical optical microstructures after the homogenization treatment prior to TMP are illustrated in Figures 10a and 10b. These microstructures are characterized by large γ grains (about 750 μm), large NbC particles and plate-like δ (Ni_3Nb). The higher optical magnification micrograph shown in Figure 10b clearly illustrates the plate-like δ at the γ grain boundaries and the large NbC particles in the γ matrix.



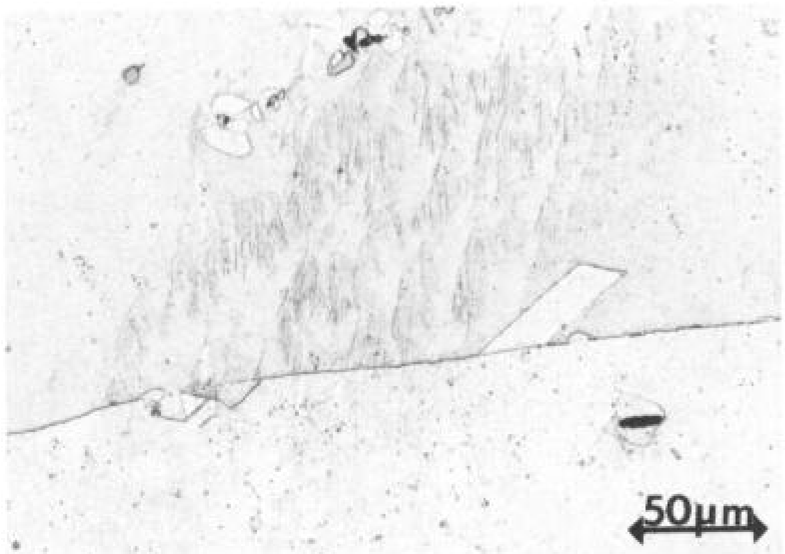
(a)



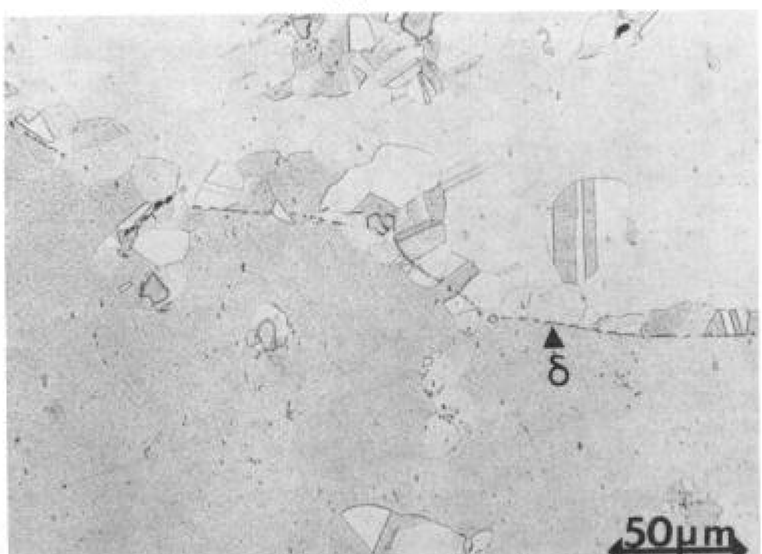
(b)

Figure 10. Optical micrograph of as-homogenized structure.

An examination of the microstructures during the early stages of recrystallization revealed that small, new microstructural features were formed at the grain boundaries, large NbC and deformation bands as an indication of recrystallization. These new features are often referred as recrystallized grains. However, in many cases, these new features did not appear to be new grains at all, but were, rather, twinned regions of the initial deformed grains. An example of this type of microstructures is shown in Figure 11. The microstructures presented in Figure 11 were obtained after a ϵ=0.2 at $\dot{\epsilon}$=0.5 sec^{-1} at 1950^{o}F (1066^{o}C) and quenched immediately or held for 10 seconds at temperature prior to quenching. The top optical micrograph shows small twins emerging from the grain boundary, the lower micrograph shows elliptical grains which consists of several new twins.



(a)

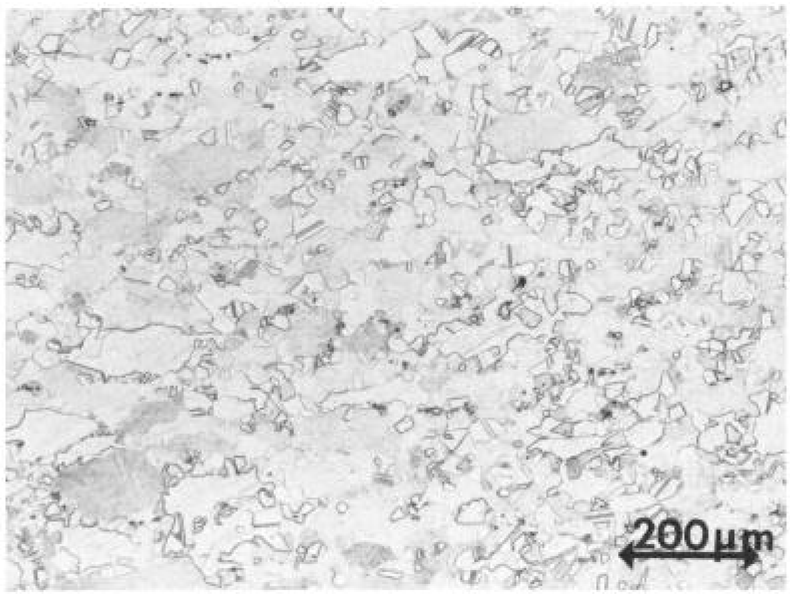


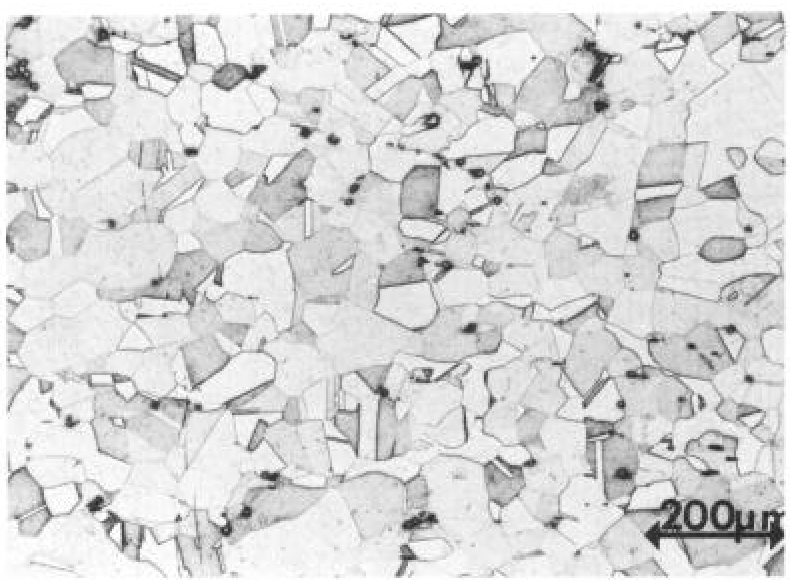
(b)

Figure 11. Optical micrographs showing the early stages of recrystallization. (a) twin formation at γ boundaries, (b) grains filled with twins.

Similar microstructural evidence has been observed and discussed elsewhere.(23-26) These studies have shown the recrystallization process to occur by twinning. This softening mechanism appears to be a common feature observed during recrystallization in the low stacking fault energy materials such as copper and austenitic stainless steels(24-26). Pure Ni has a stacking fault energy value of 128 ergs/cm^2, however, Ni Alloys such as IN-600 have a reported value of 28 ergs/cm^2.(27) Hence, the stacking fault energy in Alloy 718 is not expected to be much different from that of IN-600. Therefore, the mechanism of softening by twinning in Alloy 718 seems to be very possible.

The typical optical micrograph of a fully recrystallized microstructure deformed $\epsilon=0.5$, at $\dot{\epsilon}=0.5$ sec^{-1}, at 2050°F (1121°C) is illustrated in Figure 12a. The average





(b)

Figure 12. Optical micrographs showing fully recrystallized microstructure: (a) after deformation, (b) after 100 seconds holding time at temperature.

recrystallized grain size is about 15 μm. Figure 12b shows a microstructure deformed under the same previous conditions but held 100 seconds at temperature prior to quenching. The final average grain size is about 80 μm. In general, Alloy 718 shows a relatively low rate of grain coarsening when compared to other materials.[12] The relatively low rate of grain coarsening displayed by Alloy 718 appears to be controlled by an inhibition mechanism. In the absence of a substantial pinning force by precipitation, the most likely mechanism appears to be solute drag as the critical element. A separate study[28] found that high levels of soluble Mo and Nb can be very effective in retarding boundary motion at high temperatures.

CONCLUSIONS

1. Through the application of appropriate thermomechanical processing Alloy 718 can show substantial microstructural refinement. The final microstructure is uniform.

2. Recrystallization occurred preferentially in regions of high local strain. Twinning appears to be the major nucleation mechanism for recrystallization.

3. The relatively low rate of grain coarsening exhibited by Alloy 718 under the deformation conditions used in this study, makes this alloy very suitable for microstructural conditioning through different Thermomechanical Processes.

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