

Grain Refinement in 7075 Aluminum by Thermo-Mechanical Processing

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A thermomechanical process for grain refinement in precipitation hardening aluminum alloys is reported. The process includes severe overaging, deformation, and recrystallization steps. Microstructural studies by optical and transmission electron microscopy of grain refinement in 7075 aluminum have revealed that precipitates formed during the overaging step create preferential nucleation sites for recrystallizing grains. The relationship between precipitate density following severe overaging and recrystallized grain density has been investigated; the results show that the localized deformation zones associated with particles larger than about $0.75\ \mu\text{m}$ can act as preferential nucleation sites for recrystallizing grains. The density of particles capable of producing nucleation sites for new grains is approximately ten times greater than the density of recrystallized grains. A close relationship between dislocation cell size after the deformation step and recrystallized grain density has also been established. Both quantities saturate for rolling reductions larger than approximately 85 pct. The grain size produced in 2.5 mm thick sheet by the optimum processing schedule is approximately $10\ \mu\text{m}$ in longitudinal and long transverse directions and $6\ \mu\text{m}$ in the short transverse direction.

1. INTRODUCTION

GRAIN refinement frequently results in beneficial changes of mechanical properties in metals. Since the pioneering work of Hall¹ and Petch,² many investigators have verified the increase in macroscopic yield strength which is caused by grain refinement.³ Properties such as fatigue life, ductile-to-brittle transition temperature and fracture toughness are also affected by grain refinement,³⁻⁵ although grain size effects on these properties have received less attention than yield strength.

Another significant change produced by grain refinement is its effect on the character of deformation at high temperatures. At temperatures above about half of the absolute melting temperature and slow strain rates, grain boundary sliding can make a substantial contribution to the total strain, provided that the grain boundary area is large.^{6,7} Under the proper conditions of temperature and strain rate, such materials exhibit large elongations prior to failure during tensile deformation; these materials are termed superplastic. The phenomenon of superplasticity requires a fine grain size; typically the mean grain diameter must be less than about $10\ \mu\text{m}$ for superplastic behavior to be observed.^{6,7}

Previously developed processing techniques for grain refinement of medium to high strength aluminum alloys are of two basic types.* One class of

*Several eutectic aluminum alloys such as Al-Cu and Al-Mg may be processed to achieve fine grain sizes but their room temperature strengths are low.

thermomechanical treatments is capable of reducing the size of certain aluminum alloys during the first stages of high temperature deformation.^{8,9} These processes begin with casting procedures and heat treatments which produce special distributions of solute species and precipitates. Subsequent warm working (rolling) produces a complex, partially polygonized dislocation structure. Prior to the onset of deformation at high temperatures, the precipitate and solute distributions resist discontinuous recrystallization. Instead, the dislocation cell structure recovers continuously to form a network of sub-grain boundaries which is stabilized by the particle dispersion. High temperature deformation is necessary to complete the continuous recrystallization process.

The other class of grain refining processes has been applied to standard, high-strength aluminum alloys. This class of processing techniques involves a series of heat treatment and deformation steps intended to produce a fine grain size by discontinuous recrystallization. These processing schedules are often termed "Intermediate Thermal Mechanical Treatments" or ITMT. A number of ITMT processes have been applied to 7000 series aluminum alloys by investigators who sought improved ambient temperature properties through grain refinement.¹⁰⁻¹⁵ Changes in grain size produced by ITMT processes have been attributed to differences in the distribution of the solute elements Zn, Mg and Cu (FA-ITMT processing¹¹⁻¹³ and to differing amounts and distributions of fine, Cr-bearing precipitates (ISML-ITMT processing¹⁰).

Subsequent to development of the ITMT processes, substantial progress has been made in understanding the role of particle dispersions on recrystallization, as reported in several recent reviews.¹⁶⁻¹⁹ In particular, it has been found that large, widely-spaced particles stimulate nucleation of recrystallizing grains, producing a relatively high density of active nucleation sites. Small, closely-spaced particles have been found to

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Manuscript submitted July 8, 1980.

decrease the density of active nucleation sites and impede growth of recrystallizing grains. Quantitative definition of "large" and "small" particles depends on factors such as severity of deformation, strength of the particles and volume fraction of particles.¹⁶⁻²⁰ For nondeformable particles and reductions of more than 50 pct, the division between large and small particles is about 1 μm in aluminum.¹⁶⁻²⁰

The goal of the present investigation was to control both nucleation and subsequent growth of recrystallizing grains through control of the size and density of precipitates. Based on the improved understanding of the effects of dispersed particles on recrystallization, a thermomechanical process was developed for manipulating the density of large particles that serve as nucleation sites for recrystallizing grains.²¹ The approach used here is similar to the previously developed FA-ITMT processes in that both control the distribution of Zn, Mg and Cu in order to refine the recrystallized grain size.¹¹⁻¹³ The FA-ITMT processes were developed to eliminate the original grain boundaries in the cast ingots prior to conventional hot working of the material into final product form, either sheet or plate, with enhanced ductility at room temperature. In contrast, the thermomechanical process described in this paper is designed to convert conventionally processed plate material into fine-grain sheet, primarily for superplastic forming applications. For this application, it is desirable to achieve the finest possible grain size. The grain sizes obtained with the present process are finer than those reported for the FA-ITMT processes.^{10,11} The purpose of this investigation was to understand the origin of grain refinement by this process and to understand how precipitate particles influence the recrystallized grain size. The present paper describes the results of this investigation, concentrating on the relation between microstructure and the recrystallization process. The widely used alloy 7075 has been utilized for all of the experiments described in this paper.

2. EXPERIMENTAL PROCEDURE

A 25.4 mm thick plate of 7075 aluminum was used for all of the experimental work described in this paper. The chemical composition of the plate is presented in Table I. As discussed later (Section 3.1), the thermomechanical processing sequence involved both heat treatment and deformation steps. Heat treatments were carried out in molten salt baths where the temperatures were controlled to within ± 2 K of the desired temperature. Deformation was carried out by rolling at temperatures between 200 and 220 $^{\circ}\text{C}$. Between passes through the rolling mill, specimens were replaced in the furnace for periods ranging from 1 to 4 min while the roll gap was being reset. The duration of the reheating periods over the range of 1 to 4 min had no effect on the recrystallized grain size.

Specimens for optical and electron microscopy examination were prepared with standard techniques. Grain size measurements were performed by the linear intercept method on micrographs from specimens

etched with Keller's reagent. Test line length was sufficient to yield at least 100 grain boundary intercepts for each grain size determination. Measurements on micrographs of the L - S plane were used to determine longitudinal grain size (d_L) and short transverse grain size (d_S). For several specimens, measurements were also made on L - T and T - S planes, revealing that the long transverse grain size (d_T) is approximately equal to the longitudinal grain size ($d_T \approx d_L$). Grain density and average grain size were calculated from grain size measurements according to ASTM standard practice.²² By taking advantage of the approximate equality of d_T and d_L , average grain diameter, \bar{d} , was calculated by

$$\bar{d} = 3(2d_L^{-1} + d_S^{-1})^{-1} \quad [1]$$

and grain density, n_v , was found from

$$n_v = 0.566(d_L^{-2}d_S^{-1}) \quad [2]$$

Particle densities were measured on extraction replicas prepared by the following technique. Coupons 1 mm thick by 10 mm square were cut from overaged specimens. One face of each coupon was polished as for standard metallographic examination, etched in a 10 pct NaOH solution, dipped for a few seconds in a concentrated nitric acid bath for cleaning, washed in alcohol and immediately mounted in a standard epoxy metallurgical mount with the etched face inward. The metallurgical mounts with the coupon embedded were ground until the coupon was about 0.5 mm thick. By immersing the mount in a 20 pct NaOH solution, the aluminum matrix was rapidly dissolved; thus particles previously exposed by etching were left adhering to the epoxy. When the coupon was entirely dissolved, the extraction replica was again dipped into nitric acid again for cleaning and was prepared for SEM examination by evaporating Au on the replica.

Dislocation cell thickness was measured by transmission electron microscope of thin foils removed from bulk specimens after the deformation step. The specimens were carefully oriented with the beam parallel to the long transverse direction of the specimen. Measurements of the dislocation cell dimension in the short transverse direction were made from negatives taken of these foils.

3. RESULTS AND DISCUSSION

3.1 Concepts of Grain Refinement by Thermomechanical Processing

The mechanisms of recrystallization in alloys containing particle dispersions are phenomenologically well understood.¹⁶⁻¹⁹ Previous investigators have found that particles of different sizes produce dif-

Table I. Chemical Composition of 7075 Al Used in This Investigation

Element	Cr	Cu	Fe	Mg	Mn	Si	Ti	Zn
Wt Pct	0.20	1.71	0.32	2.44	0.04	0.12	0.04	5.84

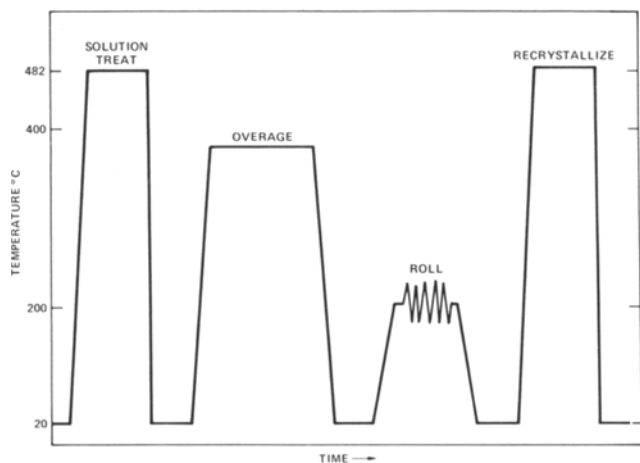


Fig. 1—Schematic diagram showing the four step thermomechanical processing treatment developed for grain refinement in 7075 Al.

ferent effects on the recrystallization process. Dispersions of large particles accelerate recrystallization rates by increasing the density of nucleation sites for recrystallizing grains. Electron microscope investigations have revealed that the intense deformation zones formed around large particles during deformation are preferential sites for nucleation of new grains during recrystallization.^{20,23} Dispersions of small particles retard recrystallization rates by exerting a drag force on migrating grain or subgrain boundaries. The small particles decrease nucleation rates by interfering with development of recrystallization nuclei and by retarding growth of recrystallizing grains.^{19,24,25} Nes²⁵ has modified a theory originally proposed by Dillamore *et al*²⁶ of nucleation at transition bands in a deformed

Table II. Optimum Thermomechanical Treatment Parameters for 7075 Al

Step	°C	Conditions	
Solution treatment	482 °C	3 h	Water quench
Age	400 °C	8 h	Water quench
Deform by rolling	220 °C	90 pct reduction of thickness in 5 to 10 passes	
Recrystallize	482 °C	30 min	Water quench

matrix to include the drag effect of small particles on migrating sub-boundaries. The modified theory predicts decreasing nucleation rate with increasing values of f/r , where f is the volume fraction of small particles and r is the particle radius. After a successful nucleation event, the small particle dispersion impedes growth of the recrystallizing grains by the same drag mechanism.

In addition to effects of particles on recrystallization rate, several studies have also considered the effects of particles on recrystallized grain size. Those results can be summarized as follows:

1) Large particles increase recrystallized grain density by increasing the density of nucleation sites.^{16–20,23,27,28} Humphreys' results on Al-Si alloys suggest that the recrystallized grain density is nearly equal to the density of large particles.²⁰

2) Small particles have been found to decrease recrystallized grain density in some cases by interfering with nucleation of recrystallizing grains, thus decreasing the overall nucleation rate.^{16–19,25,26}

Based on this fundamental understanding of the effects of particle dispersions on recrystallization and on previous work on ITMT processes,^{10–15} a thermomechanical processing sequence for grain refine-

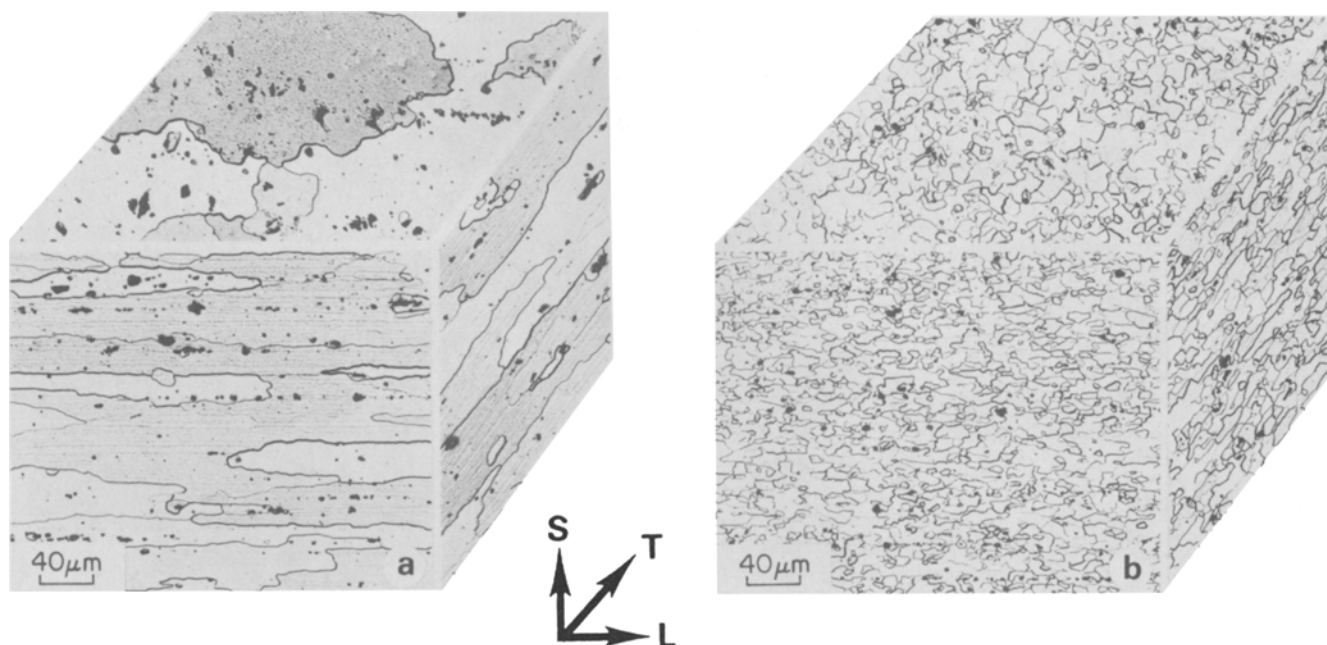


Fig. 2—Typical microstructures of 7075 Al: (a) commercially produced 25.4 mm thick plate before grain refinement and (b) after thermomechanical processing to 2.5 mm thick sheet. *L* indicates longitudinal direction, *T* indicates long transverse direction and *S* indicates short transverse direction.

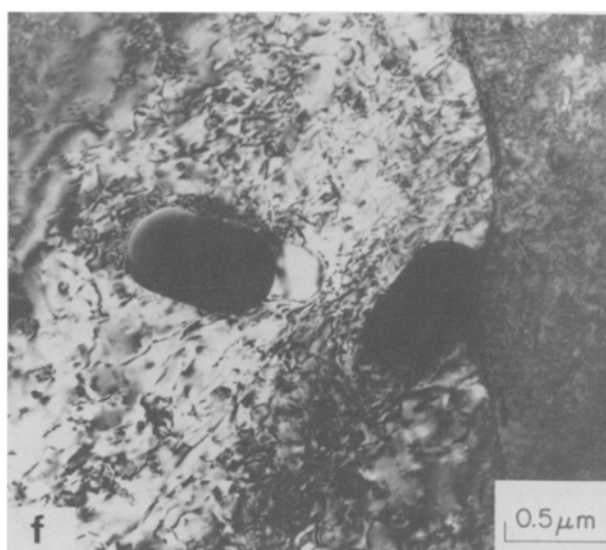
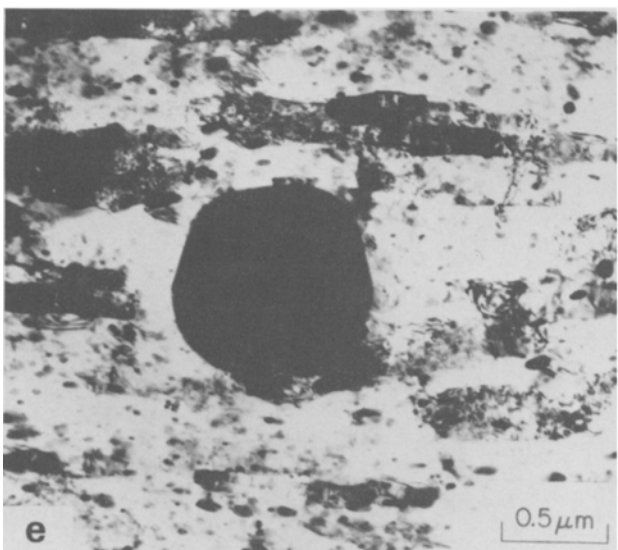
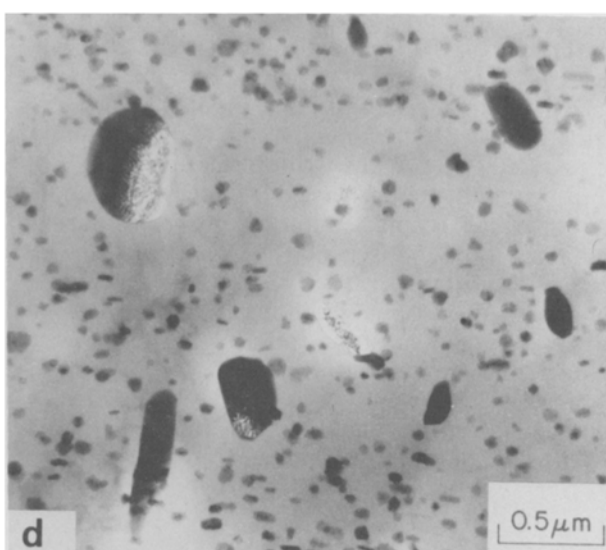
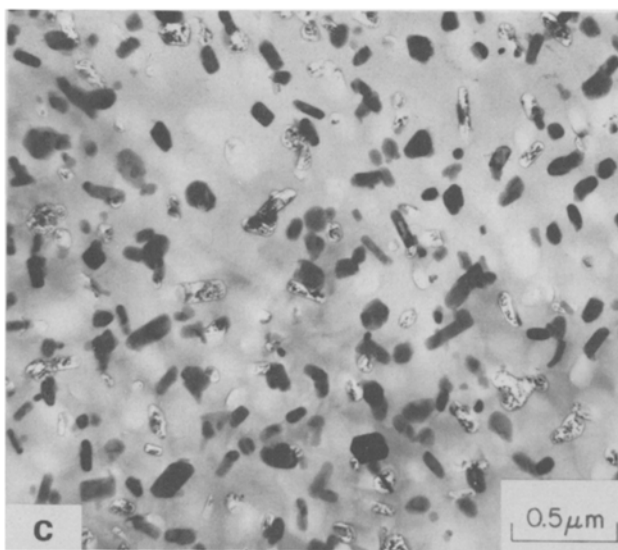
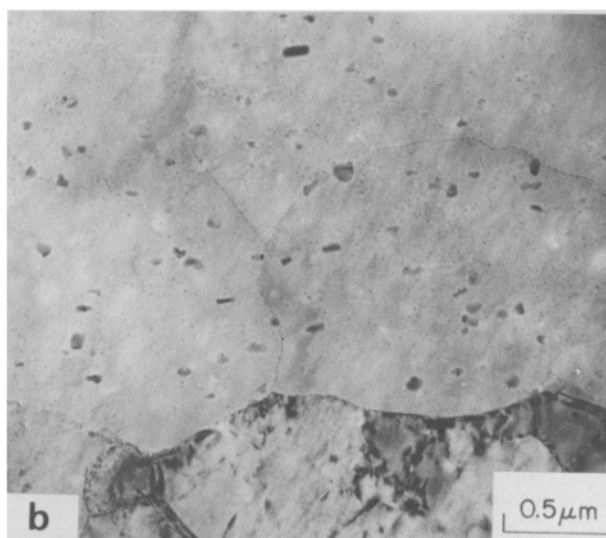
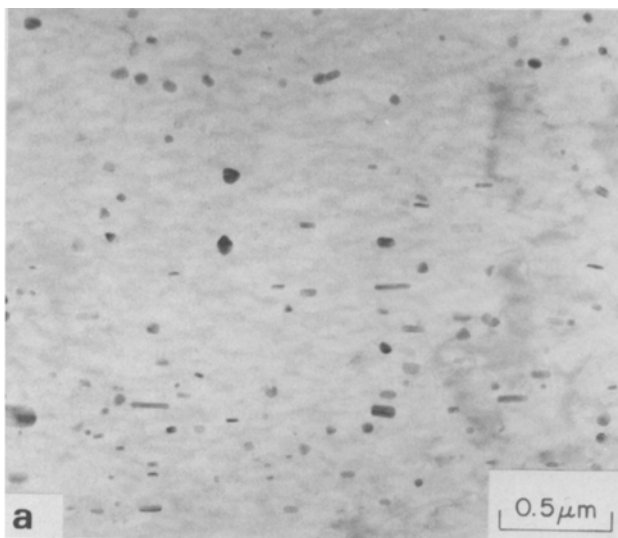


Fig. 3—Transmission electron micrographs of structures produced during each step of the thermomechanical treatment. (a) Solution treated 3 h/482 °C. (b) Solution treated, aged 24 h/121 °C. (c) Solution treated, aged 4 h/288 °C. (d) Solution treated, aged 8 h/400 °C. (e) Solution treated, aged 8 h/400 °C, deformed 90 pct at 220 °C. (f) Treatment (e) recrystallized 60 s at 482 °C. (g) Treatment (e) recrystallized 100 s at 482 °C. (h) Treatment (e) recrystallized 30 min at 482 °C.

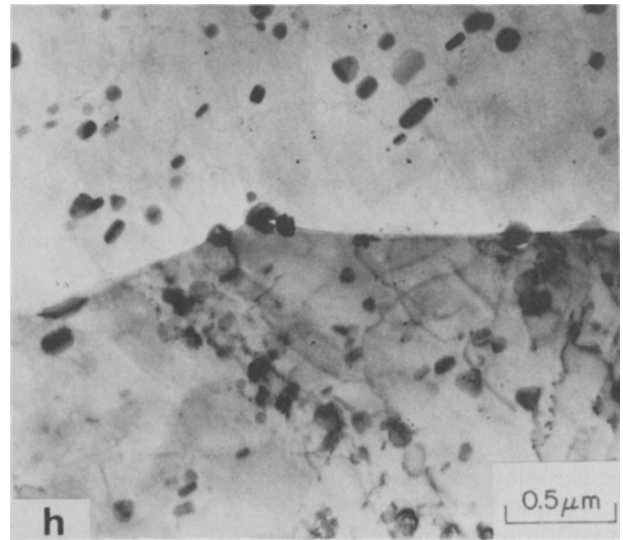
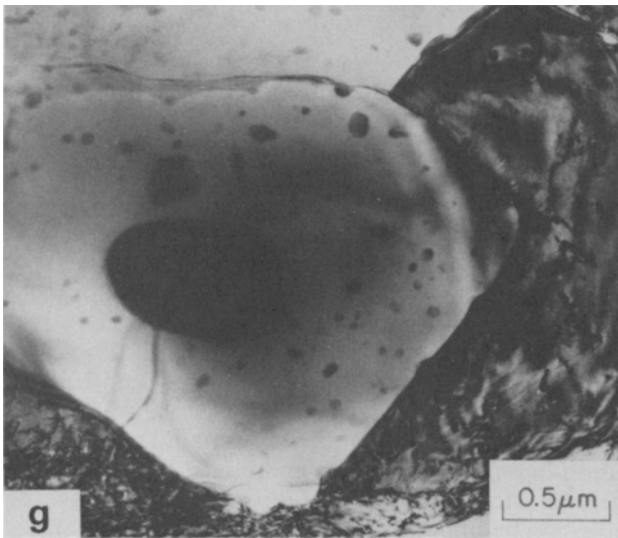


Fig. 3—Continued.

ment has been devised for age-hardening aluminum alloys. By suitable overaging treatments, large particles that act as nucleation sites for recrystallizing grains are introduced into the microstructure. These particles are in the $1\ \mu\text{m}$ size range. Subsequent steps in the process are deformation at temperatures below the recrystallization temperature followed by recrystallization.

The thermomechanical treatment for grain refinement of 7075 Al is therefore a four step process as illustrated schematically in Fig. 1.

1. Solution treat: produces a standard initial condition.
2. Overage: produces a dispersion of large particles.
3. Deform below the recrystallization temperature.
4. Recrystallize and solution treat.

The time and temperature combinations that produce the highest grain density in 7075 with this thermomechanical treatment are listed in Table II. The similar FA-ITMT process discussed previously used multi-step furnace cooling and overaging treatments to produce the desired distribution of precipitates.¹¹⁻¹³

Figure 2 illustrates the grain structure of 7075 Al before and after the thermomechanical treatment specified in Table II. The grain structure typical of commercial 7075 plate is shown in Fig. 2(a). The grain size of 25.4 mm thick commercial plate material measured by the mean linear intercept method is approximately $25\ \mu\text{m}$ in the short transverse direction and 250 to $300\ \mu\text{m}$ in the longitudinal and long transverse directions. The grain structure of the same material after thermomechanical processing to 2.5 mm thick sheet is shown in Fig. 2(b). The grain size after grain refinement is $6\ \mu\text{m}$ in the short transverse direction and $10\ \mu\text{m}$ in the longitudinal and long transverse directions. In comparison, the grain size of 1.6 mm thick commercial sheet was measured to be about $8\ \mu\text{m}$ in the short transverse direction and $40\ \mu\text{m}$ in the longitudinal and long transverse directions.

3.2 Microstructural Observations

In order to understand the roles of the precipitate particles in grain refinement, the microstructure of 7075 was examined after each step in the four-step thermomechanical processing sequence. This section describes the results of those microstructural observations. Thin foils used for transmission electron microscopy were removed from the bulk specimens parallel to the plane containing the longitudinal and short transverse directions and thus represent transverse sections.

Solution Treated. After solution treatment, 7075 contains a dispersion of insoluble particles as shown in Fig. 3(a). These are dispersoid particles formed by the Cr and Mn additions to the alloy. As found by previous studies, these particles are approximately $0.1\ \mu\text{m}$ in diam and are fairly homogeneously dispersed.²⁹ No attempt was made to further identify these particles.

Overaged. The goal of the overaging step is to produce a dispersion of particles sufficiently large to act as nucleation sites for recrystallizing grains. Based on the results of previous investigators, the minimum particle diameter is between 0.5 and $2\ \mu\text{m}$ for heavily deformed material,^{16-20,28} although the lower value seems to be applicable to polycrystalline, commercial aluminum alloys.²⁸ Overaging experiments were performed to establish the conditions required to produce a dispersion of precipitates of approximately $1\ \mu\text{m}$ diam. Several results are shown in Fig. 3(b) through (d).

Conventional precipitation hardening treatments produce the microstructure shown in Fig. 3(b). The fine, metastable precipitates which form during aging at $121\ ^\circ\text{C}$ are not resolved at the magnification of this micrograph. Fig. 3(c) and (d) illustrate the particle dispersions produced by overaging 4 h at $288\ ^\circ\text{C}$ and 8 h at $400\ ^\circ\text{C}$, respectively. Some of the particles produced by overaging for 8 h at $400\ ^\circ\text{C}$ appear to be

sufficiently large to act as preferential nucleation sites during recrystallization. Evidence detailed in Section 3.3 reveals that 8 h at 400 °C is the optimum overaging treatment for grain refinement.

Composition analysis of the large precipitates, such as those shown in Figure 3(d), by STEM microscopy revealed that there were two types of particles in this size category.

1) The majority of the particles analyzed contained Al, Zn and Mg. X-ray diffraction from overaged specimens confirmed the presence of the *M*-phase which formed during overaging.

2) Some of the particles analyzed contained Al, Fe and Si. These particles were presumed to be constituent particles formed by Fe and Si impurities.²⁹ They were not dissolved during solution treatment. These particles play a small role in recrystallization, as discussed in Section 3.3.

In addition to increasing the density of large equilibrium precipitates, overaging for 8 h at 400 °C increases the density of smaller precipitates which are in the 50 to 100 nm size range. These precipitates are either dispersoid particles formed from Cr and Mn in solution after solution treatment or small *M*-phase equilibrium precipitates dissolving during coarsening of the particle dispersion.

The optimum overaging treatment for grain refinement thus produces a bimodal particle dispersion. Large particles in the size range 0.5 to 1 μm are equilibrium precipitates formed during overaging. Dispersoid particles in the size range 50 to 100 nm are also present. Some of the dispersoid particles did not dissolve during solution treatment, others appear to have precipitated during the overaging treatment.

In comparison, the thermal treatments employed in the FA-ITMT process produce a dispersion of precipitates of larger size.¹¹⁻¹³ Details of the precipitate dispersions were not published by the FA-ITMT investigators. However, estimates from Fig. 6(a) of Ref. 11 suggest an average precipitate diameter in the range of 2 to 4 μm prior to deformation in the FA-ITMT process. While this may not seem greatly different from the 0.5 to 1 μm diam achieved in the present investigation, it will be seen in Section 3.3 that precipitate density is an important parameter in grain refinement. Assuming approximately equal volume fractions of precipitate in the two cases, the precipitate density achieved by the 8 hour overage used in the present investigation is more than 10 times higher than that achieved by the FA-ITMT process.

Deformed. After overaging, the alloy was deformed as described in Table II. The heavily dislocated microstructure created by this step is shown in Fig. 3(e). Away from the large particles, the dislocations form an elongated cell structure characteristic of heavily worked aluminum alloys.³¹ In the case of 90 pct reduction of thickness, the cell size was typically 0.2 μm in the short transverse direction by several μm in the longitudinal direction. The presence of dispersoid particles did not disrupt the cell structure, although the particles may have influenced formation of the dislocation cells as discussed by other authors.¹⁸

Near the large particles, the situation is quite dif-

ferent. Strong deformation zones form around these particles as shown in Fig. 3(e). Previous investigators have found that deformation zones around particles consist of dislocation cells considerably smaller than those found in the matrix far from the particles.^{18,20,23} In addition, many of the small dislocation cells near large particles have a lattice orientation quite different than the general orientation away from the particle. These features were not quantitatively investigated in the present study because the excellent studies of Humphreys' and others^{18,20,23} are directly analogous to this situation. However, the small dislocation cell size near the particle can be seen in Fig. 3(e) and tilting experiments in the microscope qualitatively confirmed Humphreys' observations concerning lattice rotations.

Small dislocation cells in the deformation zone around large particles have been observed to be nucleation sites for new grains during recrystallization.^{18,20,23} The important effect of large particles on recrystallization is therefore the formation of deformation zones in the surrounding matrix surrounding the particles. The relationships between overaging time, particle size and density and recrystallized grain density are quantitatively explored in Section 3.3.

Partially Recrystallized. The potential difficulties encountered in *in situ* recrystallization experiments in thin foils with conventional electron microscopes have been well documented.³⁰ To avoid such problems, deformed specimens were heated to the recrystallization temperature for short periods, then rapidly quenched. The goal was to partially recrystallize the specimens but to arrest recrystallization in progress. Removing thin foils from partially recrystallized bulk specimens permitted observation of the early stages of recrystallization.

Figure 3(f) shows a partially recrystallized region in a specimen recrystallized to 60 s at 482 °C. The recrystallized region nucleated in the deformation zone of one of the arrowed particles. Closely spaced particles have been found to share a common deformation zone in copper alloys. Porter and Humphreys²³ report that such a joint deformation zone may be a more potent nucleation site for recrystallization than the deformation zone around an isolated particle. Figure 3(f) supports that proposition, because no other recrystallized regions were found in the specimen shown in Fig. 3(f), neither at single particles nor any other sites.

Note that the matrix has undergone extensive dislocation rearrangement during the brief heating represented by Fig. 3(f). The cellular dislocation structure has coarsened, and the dislocation cell walls are narrower than in the as-deformed microstructure. Such changes are typical of dislocation rearrangements which occur in many metals during recovery.³¹

A more advanced stage of recrystallization is shown in Fig. 3(g). This micrograph shows a specimen that was recrystallized 100 s at 482 °C. The recrystallizing grain has grown to completely surround the particle that formed the preferential nucleation site. Although it is not certain that the recrystallizing grain nucleated in the deformation zone of the particle, this situation

is similar to *in situ* experiments in HVEM by other investigators where recrystallized regions were observed to nucleate at or near particles and grow to surround the particle.^{18,20,23,27}

The role of the small dispersoid particles in the recrystallization process is also illustrated in Fig. 3(g). At many locations along the migrating interface between the recrystallized region and the deformed matrix, the interface bows out between particles. Since the dispersoid content was an invariant factor in this investigation, the results do not clarify the effect of dispersoid particles on recrystallized grain size.

Completely Recrystallized and Solution Treated.

Figure 3(h) shows the microstructure after the complete recrystallization treatment of 30 min. In addition to recrystallization of the matrix, the large precipitates have dissolved, leaving the dispersoid particles in the recrystallized, fine grain matrix.

3.3 Effect of Overaging Time on Recrystallized Grain Density

One of the many external variables in the four-step thermomechanical treatment presented in Section 3.1 is overaging time. From the microstructural observations presented in the previous section, it is clear that overaging time influences recrystallized grain size by affecting the size and density of equilibrium precipitates formed during overaging. The results of a quantitative study of the relations between overaging time, precipitate size and density and recrystallized grain density are reported in this section. For this study, the solution treatment, deformation and recrystallization steps were carried out as prescribed in Table II and the overaging temperature was held constant at 400 °C; overaging time was the only variable.

The first relationship examined in this section is between overaging time and precipitate size and density. Particle densities were measured by scanning electron microscopy of extraction replicas prepared by the technique described in Section 2. Micrographs covering an area of at least 2×10^{-3} mm² and containing at least 230 particles were analyzed for each overaging condition. Particle diameters were measured and classed into size ranges; in the case of noncircular particles, the diameter was taken to be that of a spherical particle covering the same projected area as the particle being measured. Histograms of the area density of particles in each size range were prepared, using 0.25 μm size intervals. The area densities of particles were converted into volume densities using the average particle size in each size interval, as described by Ashby and Ebeling.³² The standard deviation of the number of particles counted in each interval was taken as the square root of the number counted.

The results of the study of particle density as a function of overaging time are shown in Fig. 4(a) for aging times of 0, 1, 8 and 128 h. The results are presented as the density of precipitates with diameters greater than d , $N_v(d)$, for different values of d . This method of presenting precipitate densities is unconventional, but useful for the present investigation. For a constant amount of deformation, it is reasonable to

assume that virtually all particles larger than a critical size will produce sufficiently intense deformation zones to potentially act as preferential nucleation sites for recrystallizing grains. Measurements of $N_v(d)$ permit easy correlation of particle densities with recrystallized grain densities. Uncertainty about the critical particle size required to produce a preferential nucleation site necessitated measurement of particle densities for several values of d . The error bars shown in Fig. 4(a) represent standard deviations calculated from the standard deviations in all of the intervals combined to find $N_v(d)$.³³

The trends for precipitate density as a function of overaging time illustrated in Fig. 4(a) conform to expectation. The sample of unaged material examined in this study was found to contain insoluble constituent particles which ranged in diameter from approximately 1 to 10 μm; few of the constituent particles were less than 1 μm in diam. Therefore, in the unaged material, the density of particles larger than 1 μm, $N_v(1)$, is nearly equal to the density of particles larger than 0.25 μm, $N_v(0.25)$. Hence, the points representing $N_v(0.25)$ and $N_v(1)$ after 0 aging time are virtually superposed on Fig. 4(a).

Overaging for 1 h at 400 °C causes precipitation to occur in the supersaturated solid solution, producing a very high density of small particles ($d \sim 0.25$ μm), although $N_v(1)$ also increases during this short aging

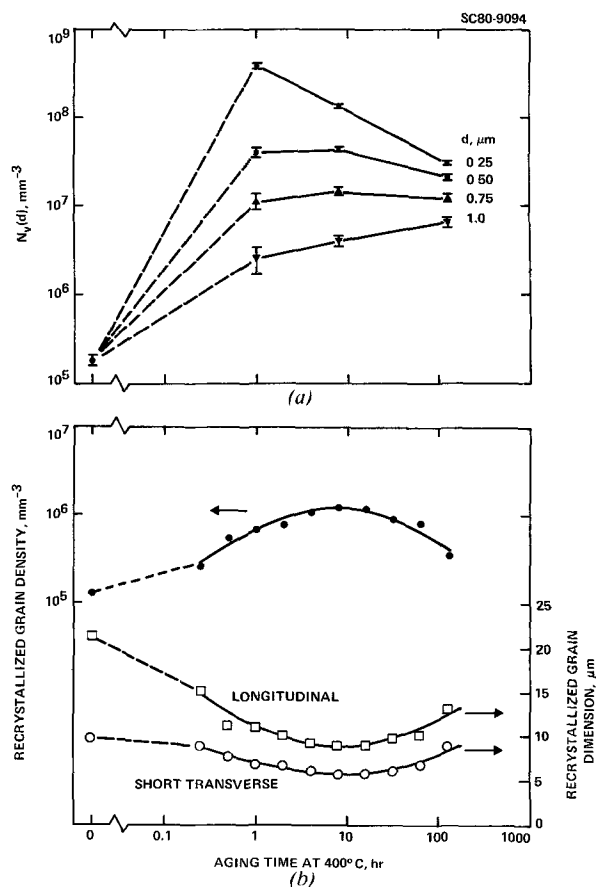


Fig. 4—(a) Density of particles with diameters larger than a minimum size as a function of aging time at 400 °C. The minimum size, d , is given in μm. (b) Linear grain sizes and grain density for recrystallized 7075 Al, as a function of aging time in the thermomechanical treatment.

period. Longer overaging times cause coarsening of the precipitate dispersion in the usual manner: the average particle size increases but the overall particle density decreases. From theoretical arguments concerning coarsening of precipitate dispersions, it is possible to show that $N_v(d)$ first increases with overaging time, reaches a maximum value and subsequently decreases for long overaging times. The overaging time required to attain the maximum in $N_v(d)$ depends on d : for small d , the maximum occurs at short times; for large d , the maximum occurs at long times. The results shown in Fig. 4(a) qualitatively agree with the theoretical prediction. The maximum value of $N_v(1)$ apparently occurs for overaging times longer than 128 h.

Recrystallized grain size in the short transverse and longitudinal directions as well as grain density are shown as functions of overaging time at 400 °C in Fig. 4(b). If the majority of recrystallized grains nucleated in the deformation zones of particles larger than some critical size, d' , then grain density and $N_v(d')$ should similarly vary as functions of overaging time. The maximum in $N_v(0.25)$ occurs at an overaging time of approximately 1 h or less, and the maximum in $N_v(1)$ occurs at an overaging time of 128 h or more; neither correlates well with the observed variation of grain density with overaging time. The maximum value of $N_v(0.5)$ occurs for overaging times between 1 and 8 h, and the maximum value of $N_v(0.75)$ occurs at approximately 8 h. The best correlation between particle density and recrystallized grain density is found for $N_v(0.75)$, suggesting that the critical particle diameter necessary to form a nucleation site for a recrystallizing grain is approximately 0.75 μm . A value of 0.75 μm as the minimum particle diameter for particle-assisted nucleation of recrystallizing grains is in general agreement with the results of many other studies.^{16-20,28}

In addition to determining an approximate value for the minimum particle diameter required for particle-assisted nucleation, the results presented in Fig. 4 reveal the relation between recrystallized grain density and absolute particle density. For the optimum overaging time of 8 h, there are approximately 10 particles larger than the critical size per recrystallized grain. For the other overaging times, similar values are found. This result is very different from the results of Humphreys²⁰ who found approximately one critically-sized-particle per recrystallized grain in experimental Al-Si alloys deformed 90 pct.

The result obtained in the present investigation can have two possible explanations: either there was only one successful nucleation event for every 10 particles, or grain growth occurred during the 30 min recrystallization period so that the recrystallized grain diameter shown in Fig. 4 is actually the Zener diameter determined by the dispersoid particles. The latter explanation seems unlikely because the recrystallized grain density would not vary with overaging time, as shown in Fig. 4, if that were the case. A more likely explanation involves the results of Nes,^{19,23,24} which show that a dispersion of small particles can decrease the overall nucleation rate of recrystallizing grains at large par-

ticles. In the present investigation, the dispersoid particles may prevent nucleation at many critically-sized particles, so that the ratio of $N_v(0.75)$ to recrystallized grain density is higher than in the study by Humphreys²⁰ where no dispersoid particles were present.

The relative importance of precipitate particles and constituent particles in forming nucleation sites for new grains is also illustrated in Fig. 4. After overaging for 8 h at 400 °C, the density of precipitates more than 0.75 μm in diam is more than fifty times the density of constituent particles with diameters larger than 0.75 μm . Assuming that constituent particles and precipitates are equally effective as nucleation sites, the number of grains nucleating at precipitates is more than fifty times the number nucleating at constituent particles; this suggests that constituent particles do not make a substantial contribution to the grain size in optimally aged material. Thus, alloys with reduced Fe and Si content, such as 7475, should be capable of achieving approximately the same recrystallized grain density as 7075.

The results of this study of recrystallized grain density and precipitate densities as functions of overaging time support the following conclusions.

- 1) A value of 0.75 μm for the minimum particle diameter capable of nucleating a new grain is approximately correct. This observation is consistent with the microstructural observations and the results of previous investigations.^{15-19,27}
- 2) Only about 10 pct of the particles greater than 0.75 μm in diam nucleate a recrystallized grain.

3.4 Effect of Severity of Reduction on Recrystallized Grain Size

Severity of reduction is related to recrystallized grain size by the formation of deformation zones around precipitate particles. Increasing reduction causes intense deformation zones to form around smaller particles, thus providing a higher density of nucleation sites for new grains. As suggested by Nes,¹⁸ the ability of particles to nucleate new grains can be assessed by comparing particle size with dislocation cell thickness. To establish a relationship between dislocation cell thickness and recrystallized grain density, both quantities were measured as a function of severity of reduction. Solution treatment, overaging and recrystallization variables were held constant at the values listed in Table II; the only variable was the severity of reduction.

For increasing severity of reduction, the specimens tended to widen more during rolling, so that reduction of thickness was not a satisfactory measure of deformation. A better quantity to use for the amount of deformation is the effective strain, $\bar{\epsilon}$, defined as³⁴

$$\bar{\epsilon} = \frac{\sqrt{2}}{3} (\epsilon_1 - \epsilon_2)^2 + (\epsilon_2 - \epsilon_3)^2 + (\epsilon_1 - \epsilon_3)^2)^{1/2} \quad [3]$$

where ϵ_i is the true strain in direction i . The effective strain accounts for widening of the specimen during rolling and can be used to measure deformation by other processes, such as swaging or forging. The ideal reduction, R_I , is defined as the reduction in thickness

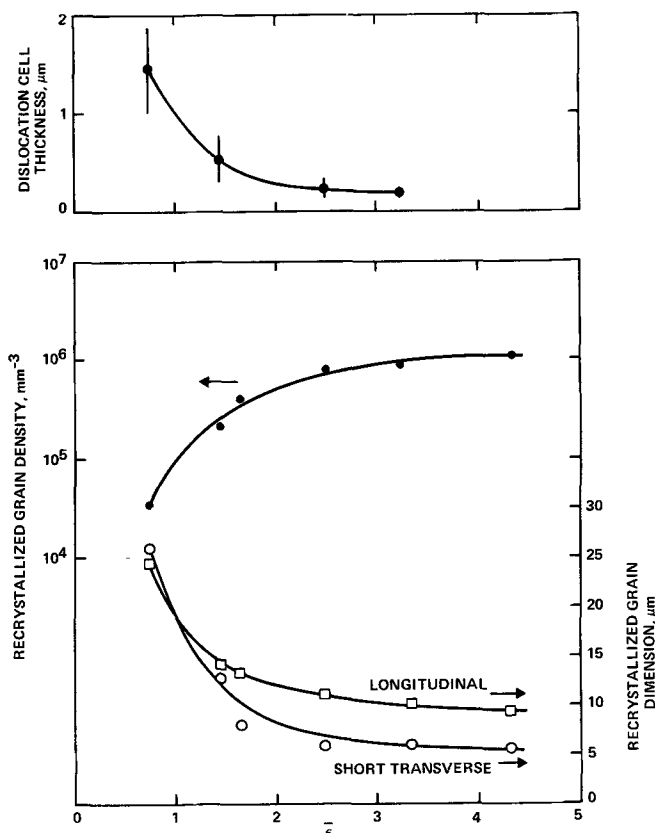


Fig. 5—Dislocation cell thickness, recrystallized grain sizes and recrystallized grain density as functions of the amount of deformation during the third step in the thermomechanical processing sequence.

producing the same effective strain with no change in specimen width. Ideal reduction can be expressed as

$$R_I = 1 - \exp - \frac{\sqrt{3}\epsilon}{2} \times 100 \text{ pct} \quad [4]$$

Ideal reduction is useful for relating effective strain to values of reduction of thickness normally reported for rolled materials that are wide enough to prevent significant widening during rolling.

Dislocation cell thickness and recrystallized grain density were measured as functions of effective strain. The results are presented in Figs. 5 and 6. In Fig. 5, dislocation cell thickness, recrystallized grain density and linear grain dimensions are plotted as functions of effective strain. The error bars shown for values of dislocation cell thickness represent standard deviations calculated from each measured distribution of dislocation cell thickness. As anticipated, dislocation cell thickness decreases and grain density increases as the severity of deformation is increased. However, both quantities saturate at an effective strain of about 2.5, corresponding to an "ideal" reduction of approximately 85 pct. Saturation of dislocation cell sizes has previously been observed in other materials, although the amount of deformation required to achieve saturation varies greatly.³¹

Figure 6 shows the direct relation between dislocation cell thickness and recrystallized grain size. As seen in the figure, correlation between these two

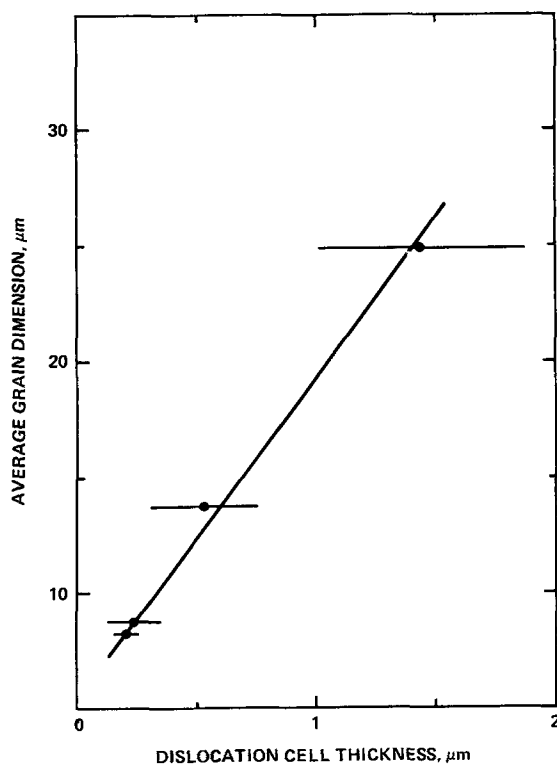


Fig. 6—Relation between dislocation cell thickness and recrystallized grain density.

quantities is excellent. The good correlation suggests that dislocation cell thickness is a reasonable measure of the intensity of deformation zones around particles. This result implies that deformations beyond strains of about 2.5 do not generate intense deformation zones around smaller and smaller particles. The zones already present around larger particles may be intensified but new particles are not activated as nucleation sites for new grains. Increasing the severity of deformation beyond $\epsilon \sim 2.5$ does not produce material with finer grain sizes when the thermomechanical treatment presented in Section 3.1 is used.

3.5 Effect of Heating Rate on Recrystallized Grain Size

All results reported in this paper were obtained by recrystallizing specimens in molten salt baths. This method ensured a rapid heating rate to the recrystallization temperature. Several experiments were performed with slower heating rates, e.g., specimens heated in air furnaces. These results suggest that slow heating rates do not result in the same grain refinement achieved by rapid heating rates. Heating rates of about $2.8 \times 10^{-2} \text{ K} \cdot \text{s}^{-1}$ ($100 \text{ K} \cdot \text{h}^{-1}$) result in grain densities near 10^4 mm^{-3} , while rates of $1.8 \times 10^{-3} \text{ K} \cdot \text{s}^{-1}$ ($6 \text{ K} \cdot \text{h}^{-1}$) produce grain densities of approximately $3 \times 10^2 \text{ mm}^{-3}$ which are indistinguishable from conventional commercial products. Present results thus indicate that rapid heating rates are required to achieve substantial grain refinement by the process illustrated in Fig. 1.

4. CONCLUSIONS

In summary, this study of grain refinement in 7075 Al demonstrated that the basic understanding of recrystallization in particle dispersed alloys can be used to develop processes for grain refinement in commercial alloys. Some of the important conclusions are as follows:

1) A four-step thermomechanical processing sequence has been devised for grain refinement of 7075 Al. An overaging step is used to precipitate a high density of particles larger than approximately 0.75 μm in diam. The deformation zones that form around the particles during subsequent rolling serve as nucleation sites for recrystallizing grains when the alloy is rapidly heated above the recrystallization temperature.

2) The variation of density of particles greater than 0.75 μm in diam correlates well with the recrystallized grain density. This suggests that the density of such particles controls the recrystallized grain density. In contrast to the observations of Humphreys,²⁰ the particle density is about ten times the recrystallized grain density.

3) The variation of dislocation cell thickness after deformation correlates well with recrystallized grain density. The dislocation cell thickness and recrystallized grain density saturate at about 85 pct reduction of thickness. Further deformation does not produce additional grain refinement.

ACKNOWLEDGMENTS

The authors are grateful to both the Rockwell International Independent Research and Development Fund and the Air Force Office of Scientific Research (Contract F44620-76-C-0025) for supporting this work. Thanks are extended to Dr. E. Nes, Dr. C. Bampton, and Dr. W. M. Robertson for helpful discussions and to R. A. Spurling, P. Q. Sauers, and M. Calabrese for experimental assistance.

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