# The Interaction Between Recovery, Recrystallization and Precipitation During Annealing of Cold Rolled AA6111

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#### Abstract

The annealing behaviour of commercial precipitation hardened alloys is complex as it may involve the simultaneous occurrence of recovery, recrystallization and precipitation. The present work examines the effect of different initial precipitation states on the recrystallization behaviour of a cold rolled aluminum alloy, AA6111. It is found that the recrystallization kinetics are extremely sluggish irrespective of the starting precipitation state. Re-precipitation of Q phase during annealing is found to occur primarily along grain boundaries and on shear bands. Recrystallized grain size is controlled by the spacing of precipitates in the structure. Larger precipitate spacing allows recrystallized grains to grow coarser resulting in a wider distribution of recrystallized grain size.

### 1. Introduction

The increased use of advanced aluminum sheet alloys to replace steels in the construction of light weight automobiles has resulted in a significant reduction in fuel consumptions. The heat treatable AI-Mg-Si-Cu alloy AA6111 is one of the main aluminum choices for automotive sheet skin due to its excellent combination of paint bake hardening response and high formability. In the commercial processing of this alloy, precipitation occurs during the hot rolling and subsequent coiling processes. After hot rolling, the sheets are cold rolled to the final gauge. The cold rolled sheet is then heat treated on a continuous annealing line in order to recrystallize and solution treat the material. Hence, recrystallization of the cold rolled sheets occurs concurrently with the solution heat treatment process. The recrystallization process determines the final microstructure of the sheets, i.e. the grain size, texture, and the associated mechanical properties. Therefore, it is imperative to understand how precipitates influence or interact with the recrystallization process during annealing in order to achieve optimum properties.

To date, the majority of studies on AA6111 have focused on improving the strength of this alloy through precipitation [1-3]. In contrast, there have been relatively few studies aimed at understanding the role of precipitates in the recrystallization process. Pre-existing precipitates can play a number of different roles during the recrystallization process depending on the

precipitate size and spacing [4]. Particles with a size larger than 1 µm can promote recrystallization by introducing additional nucleation sites. This phenomenon is commonly known as particle-stimulated-nucleation (PSN) [5]. On the other hand, fine scale precipitates which are situated along grain boundaries and on substructures can severely impede the progress of recrystallization by pinning grain boundaries. The pinning force is commonly known as the Zener pinning force, and it is a function of the volume fraction of precipitates and precipitate size. To date, the most extensive experimental investigation into the effect of precipitation state on recrystallization in aluminum alloys was carried out by Lillywhite et al. [6]. Using a high purity AI-Mg-Si alloy with varied precipitate conditions, they have shown that the recrystallization kinetics can be changed by several orders of magnitude. Burger et al. have also demonstrated that the final recrystallized grain structure in a 6000 series alloy is controlled by the initial precipitate distribution [7]. However, a comprehensive study on the interaction between precipitation and recrystallization in AA6111 which is an Al-Mg-Si-Cu alloy system is still lacking. The objective of the present study is to examine the effect of different initial precipitation states on the recrystallization kinetics of cold rolled AA6111 during annealing.

### 2. Experimental Procedures

The AA6111 sheet alloy used in this investigation was received in the form of a hot rolled sheet with gauge of 3.5 mm. The chemical composition of the alloy is given in Table 1.

Mg	Si	Cu	Fe	Mn	Cr	Ti	AI		
0.75	0.63	0.75	0.05	0.05	0.06	0.06	bal.		

Table 1: Chemical composition of AA6111 in wt%.

Rectangular specimens were sheared from the hot rolled sheets with the longitudinal section parallel to the rolling direction. The specimens were then solution heat treated at 560°C for 10 min followed by guenching in water at room temperature. After guenching, the samples were aged to produce specimens with three different precipitate conditions: i) aging at room temperature for 8 days (T4), ii) aging at 180°C for 7 hours (T6) and iii) aging at 250°C for 7 days (OA). After aging, the specimens were cold rolled to a reduction of 40% in thickness using a laboratory scale rolling mill. Annealing of the cold rolled specimens was conducted at 325°C for times ranging from 1 minute to 2 weeks in a salt bath. In order to reveal the microstructures in the ND-RD section, the samples were first anodized in Barker's solution. Microstructures were then revealed under cross polarized light in an optical microscope. The micrographs showing the precipitate morphology were taken without polarization. Samples for EBSD were electropolished in a perchloric-methanol solution at temperatures below -10°C. EBSD mapping of the structures was carried out with a step size of 2 µm in a Hitachi S-570 scanning electron microscope operating at 20keV. Using EBSD generated microstructures, recrystallized grains were outlined and then analyzed using image tool software. Fraction recrystallized was quantified based on area measurements whereas the recrystallized grain size was calculated as equivalent area diameter assuming spherical grains. A minimum of 300 grains were included in the analysis of grain size for each specimen.

## 3.1 Precipitate Conditions

The precipitation sequence of AA6111 involves the formation of various complex metastable phases [8, 9]:

Supersaturated solid solution  $\rightarrow$  GP zones/clusters  $\rightarrow \beta$ " + Q  $\rightarrow$  equilibrium Q +  $\beta$ 

The formation of GP zones and clusters provides the initial increase in strength during natural aging at room temperature (T4 condition). The strength of the alloy can be further enhanced by aging at elevated temperatures due to the precipitation of the two main hardening phases,  $\beta$ " and Q. The excellent age hardening response of this alloy is illustrated by the six fold increase in yield stress after peak aging for 7 hours at 180°C (T6 condition) [10]. During overaging one observes the formation and coarsening of the quaternary Q phase and as a result the yield stress decreases.



Figure 1: T4 sample of AA6111 which has been cold rolled and then annealed for 6 hours at 325°C showing (a) precipitate distribution and (b) microstructure.

All the pre-existing precipitates in the structure (irrespective of prior aging conditions) enter the coarsening phase of Q particles very quickly at the annealing temperature of  $325^{\circ}C$  [10]. Figure 1a shows the distribution of precipitates in a deformed T4 sample which has been annealed for 6 hours at  $325^{\circ}C$ . The corresponding grain structure is shown in Figure 1b. Fine scale precipitates (less than  $1\mu$ m) can be seen uniformly distributed along grain boundaries and within the matrix on shear bands. One can also observe large ( $\sim 5\mu$ m) Fe rich constituents particles (formed during ingot solidification) which remain stable throughout the entire heat treatment schedule. The microstructure consists of elongated grains typical for a cold rolled material. A few very small recrystallized grains can be observed to nucleate along the grain boundaries of the elongated grains. It should be noted that at this stage, a substantial amount of recovery has occurred in the materials [10].

The structure of Q phase precipitates has been extensively characterized by a number of researchers using conventional as well as high resolution TEM [11, 12]. Weatherly and co-workers were able to show that the structures of grain boundary and matrix Q phase are very

similar with their fully coherent (510) habit plane lying along <100> direction in aluminum [12]. These lath shaped precipitates exerts a significant pinning force on both the substructures and grain boundaries and therefore making recrystallization a particularly difficult process. The recrystallization behaviour is discussed in the next section.

### 3.2 Recrystallization Behaviour

The three partially recrystallized structures generated by EBSD mapping are shown in Figure 2. The samples were obtained after annealing for 2 weeks at  $325^{\circ}$ C. In all cases, nucleation of recrystallized grains occurred preferentially at prior grain boundaries. The most notable feature in Figure 2 is the difference in recrystallized grain size ( $d_{rex}$ ) between the three samples. Colonies of large recrystallized grains with a grain size as large as 100  $\mu$ m can be clearly observed in the OA sample. Smaller and more evenly distributed recrystallized grains are seen in the T4 and T6 specimens. In order to further examine the different recrystallization behaviour, several microstructural parameters have been quantified and listed in Table 2. It is clear that the combined effect of pinning by precipitates and extensive prior recovery severely retards recrystallization since only about 30-40% of the structure was recrystallized after 2 weeks of annealing. Similar studies on conventional hot rolled AA6111 with the same amount of cold work have shown that complete recrystallization can be achieved within 24 hours at 325°C [13].



Figure 2: EBSD maps of partially recrystallized structures obtained after annealing for 2 weeks at 325°C. Black lines represent high angle grain boundaries which are > 15°, grey lines represent boundaries between 2-15°.

Sample	Fraction recrystallized	Ave. recrystallized grain size d <sub>rex</sub> (μm)	No. recrystallized grains/mm <sup>2</sup>	Ave. mean misorientation of unrecrystallized grains $\theta_{unrex}$ (°)
T4	0.37	14.6	1417	5.5
T6	0.32	11.5	1946	5.2
OA	0.32	18.6	673	4.7

Table 2: Microstructural parameters used to characterize the partially recrystallized T4, T6 and OA samples



Figure 3: Recrystallized grain size distribution in partially recrystallized T4, T6 and OA samples after annealing for 2 weeks at 325°C.

Figure 2 clearly shows that the recrystallized grains in the OA sample have grown more extensively than in the other two samples. The average size of the recrystallized grains ranges from 11.5  $\mu$ m for the T6 to 18.6  $\mu$ m for the OA samples. The difference in grain size can be further examined by considering the number of recrystallized grains per unit area as shown in Table 2. The number of recrystallized grains per unit area in the OA samples. While the density of grains is less in this case, these grains have grown to a larger size. This behaviour is not unexpected, since the limiting recrystallized grain size is related to the particle spacing and further coarsening of the precipitates during annealing leads to an even larger particle spacing. In the case of T4 and T6, re-precipitates then coarsen resulting in a much finer

distribution of the precipitate spacing. Finally, it is interesting to compare the recrystallized grain size distribution in the three samples. As shown in Figure 3, it can be seen that the OA sample has a wider distribution of grain sizes while the T6 specimens show a relatively narrow distribution.

In addition to characterizing the recrystallized grains, it is also interesting to calculate the averaged mean misorientation in the deformed grains ( $\theta_{unrex}$ ). Using misorientation data generated from EBSD maps, a critical mean misorientation of 2° was used to separate deformed grains from recrystallized grains, i.e. any grains that possess an internal mean misorientation of greater than 2° are classified as deformed grains. The results are given in Table 2. On average, the mean misorientation angle of the deformed grains in the OA specimen is lower than the T4 and T6 samples.

### 4. Conclusions

Precipitation was found to strongly interact with recrystallization during the annealing of cold rolled AA6111. By varying the precipitate conditions in the materials, a different distribution of the recrystallized grain size was obtained. EBSD provides a direct method to quantify the microstructures of partially recrystallized structures. Work is currently underway to quantitatively correlate the morphology of precipitates with recrystallization kinetics.

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