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The Effect of Precipitation on the Recovery and Recrystallization Behaviour of AA6111

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The present paper provides a systematic experimental study on the interaction of precipitation with recovery and recrystallization in an Al-Mg-Si-Cu AA6111 alloy. After solution treatment, samples were artificially aged to produce three different precipitate conditions: (i) naturally aged (T4) (ii) overaged (OA) and (iii) severely overaged (SOA). Samples with these systematically varied precipitate states were then cold rolled and annealed at 325 and 445°C. The softening behaviour during annealing was measured and detailed microstructure and precipitate characterization was conducted using optical and scanning electron microscopy techniques. These measurements enabled an in-depth analysis of the interaction of precipitates with recovery and recrystallization. The overall recrystallization behaviour is sluggish with the fastest recrystallization rate being observed in the SOA sample and the slowest in the OA sample. At low annealing temperature of 325°C, the majority of softening can be attributed to recovery and precipitate coarsening, particularly in the T4 and OA samples. In general, partially recrystallized microstructures are observed even for annealing time as long as 40 days. This is particularly pronounced in the T4 and OA samples where the fraction recrystallized remains below 0.50. In the OA sample, recrystallization has been correlated with precipitate free zones whereas recrystallization is inhibited in zones with a dense population of fine and coarse precipitates. Increasing the annealing temperature to 445 °C results in complete recrystallization within one hour of annealing and this can be attributed to the dissolution of precipitates.

Keywords: *recovery, recrystallization, precipitation, annealing, AA6111*

1. Introduction

The heat treatable AA6xxx alloys (i.e., Al-Mg-Si-Cu) are a popular choice of aluminium alloys for automotive skin panel applications. In North America, AA6111 is the alloy of choice as it combines high strength with reasonable formability and thus offers automotive makers an attractive option to replace steel sheets in an effort to reduce the weight of the vehicles. However, processing of these alloys must be tightly controlled in order to optimize their performance. Typically, the alloys are processed through a continuous annealing line prior to shipping to customers. Microstructually, recrystallization of the cold rolled structure is the final step of processing during which the texture and grain structure can be controlled. Hence, the sheet metal producers are required to have an increasingly sophisticated understanding of the linkage between processing conditions and the evolution of microstructure in the materials during annealing.

The objective of the present study is to understand the changes in the recovery and recrystallization behavior of AA6111 during annealing by altering systematically the precipitate conditions of the materials prior to cold rolling. With regard to the interaction with recrystallization and precipitation, the traditional emphasis has been on Zener pinning and particle stimulated nucleation (PSN) associated with *stable* secondary phases. However, there have been comparatively few studies aimed at understanding the effect of *unstable* precipitates which may evolve simultaneously with recovery and recrystallization during annealing. It is widely known that fine scale precipitates retards recrystallization by pinning mobile recrystallization fronts resulting in recrystallization kinetics several orders of magnitude different depending on the initial states of precipitation [1]. The other important goal of the present study is to link the evolution of microstructure to the changes in mechanical properties in terms of yield stress. The work is part of a larger study to develop a comprehensive process model to predict the mechanical properties of AA6111 alloys based on microstructural parameters.

2. Experimental Procedure

The starting materials was an industrial hot rolled AA6111 alloy. The composition of the alloy is shown in Table 1. Prior to artificial aging, the hot rolled samples were solution heat treated in a salt bath at 560°C for 10 minutes followed by water quenching to room temperature. The supersaturated solid solution (SSS) samples were then artificially aged to three specific precipitate conditions: (i) naturally aged (T4), (ii) overaged (OA) and (iii) severely overaged (SOA). The corresponding artificial aging conditions are (i) room temperature for 7 days, (ii) 250°C for 7 days and (iii) 325°C for 7 days. For elevated temperature heat treatments, an air furnace was employed. After aging, the samples were cold rolled at room temperature using a laboratory rolling mill to a final thickness of 2.1 mm giving a total of 40% reduction in thickness. Isothermal annealing of the cold rolled samples were conducted at 325 and 445°C for time ranging from 1 minute to 40 days. For short annealing cycle (<24 hours), the samples were annealed in salt baths to ensure short heat up time. For longer annealing cycles, preheated air furnaces were employed. At the end of the annealing cycle, the samples were quenched in water to room temperature.

Characterization of the annealed samples was carried out using a variety of imaging techniques including optical and scanning electron microscopy. The volume fraction of recrystallized grains was estimated using image analyzing software. Recrystallized grains in partially recrystallized microstructures were identified based on their shape: grains with an aspect ratio of 3 or less were considered recrystallized grains.

The isothermal softening behaviour of the samples was quantified by measuring the yield stress of the samples using standard tensile tests. From the engineering stress-strain curves, the 0.2% offset method was applied to extract the value of the yield stress. For each annealing condition, two tensile samples were tested and the averaged values of the two measurements were reported.

Table 1. Chemical composition of the investigated AA6111 in wt. %.

Mg	Si	Cu	Fe	Mn	Cr	Ti	Al
0.75	0.63	0.75	0.25	0.20	0.05	0.06	bal.

3. Experimental Results

As shown in the bar charts of Fig. 1, the T4, OA and SOA samples experienced markedly different magnitudes of yield stress increase resulting separately from the artificial aging and cold rolling processes. Among the three artificial aging processes, the OA samples had the largest increase in the yield stress, i. e. 95 MPa. It can also be observed that the severe overaging process resulted in a significantly reduced precipitation hardening effect, i.e. 34 MPa. After 40% cold rolling, the yield stress of the T4 samples increased by a drastic amount of 217 MPa compared to 78 and 102MPa for the OA and SOA samples respectively.

Fig. 2a shows the isothermal softening behaviour of the three samples at 325°C. The corresponding recrystallization curves are given in Fig. 2b. From Fig. 2a, it can be seen that the yield stress of all three samples decreased substantially in the initial stage of annealing. The T4 samples showed a particularly steep softening rate: the yield stress decreased from 356 (CR yield stress) to 239 MPa in the first 60s of annealing. After nearly 1000 hours of annealing, the yield stress of the T4 and OA samples eventually reached a value of ~70 MPa while the SOA sample which was fully recrystallized reached a final yield stress of 40 MPa. In terms of recrystallization, Fig. 2b shows that significant recrystallization (>0.1) occurred only after about 90 hours of annealing irrespective of prior precipitate condition. Complete recrystallization after 1000 hours of annealing was only achieved in the SOA sample. Given the same amount of soaking time, the volume fraction of recrystallized grains was less than 0.50 in both T4 and OA samples. Optical micrographs showing the partially recrystallized T4 and OA samples and the fully recrystallized SOA sample are given in Fig. 3.

In an effort to understand the effect of annealing temperature, both the OA and SOA samples were also annealed at a higher temperature of 445°C. The results are shown in Figs. 4a and 4b for the softening and recrystallization curves respectively. In comparison to the results depicted in Figs. 2a and 2b, it is evident that both the softening and recrystallization rate were significantly enhanced by increasing the annealing temperature. The increase rate of recrystallization is most significant in the OA samples which showed complete recrystallization after 30 minutes of annealing (0% at 325°C). The SOA samples achieved full recrystallization after only 1 minute of annealing. From Fig. 4a, it can be seen that the yield stress did not decrease further as soon as the samples achieved full recrystallization in both OA and SOA samples reaching a plateau of 50 MPa.

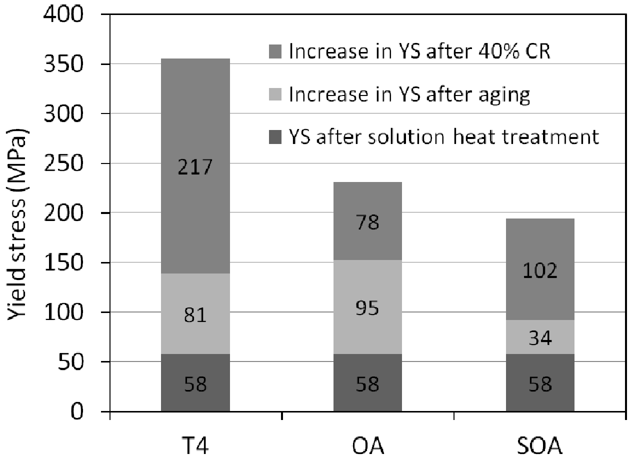

Fig. 1. A bar chart showing the magnitudes of the yield stress obtained in the T4, OA and SOA samples after artificially aging and 40% cold rolling reduction.

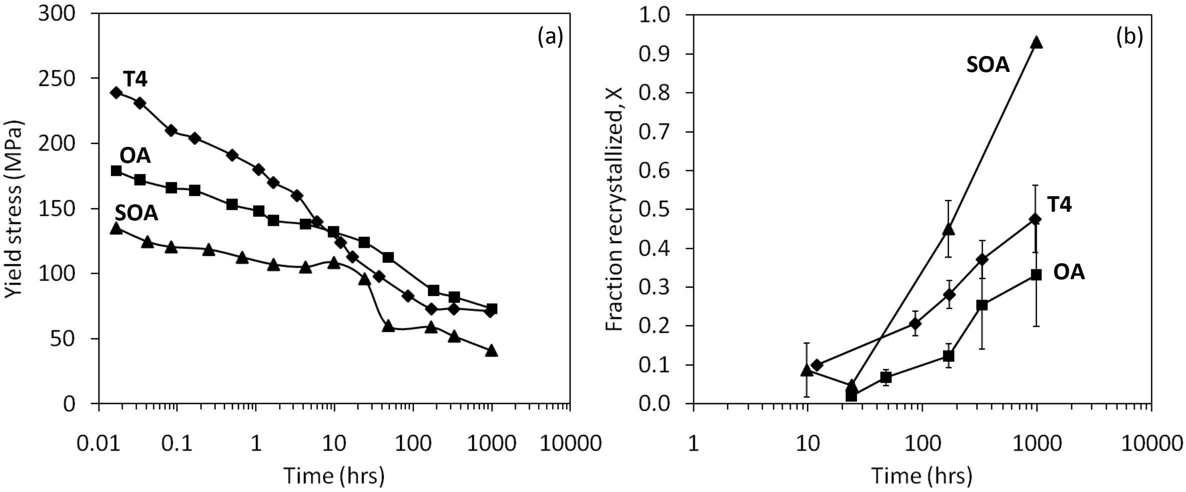

Fig. 2. Isothermal (a) softening and (b) recrystallization curves of the T4, OA and SOA samples obtained after annealing at 325°C.

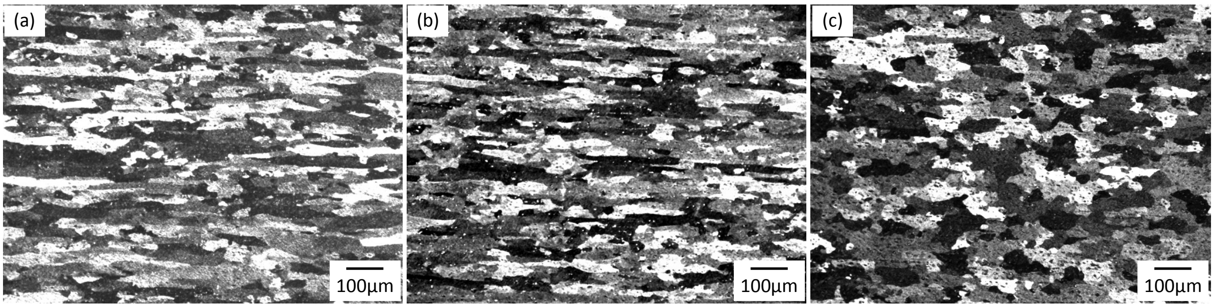

Fig. 3. Optical micrographs showing the microstructures of the (a) partially recrystallized T4, (b) partially recrystallized OA and (c) fully recrystallized SOA samples. The corresponding annealing conditions were 325°C/330h for the T4 and OA samples and 325°C/960h for the SOA sample.

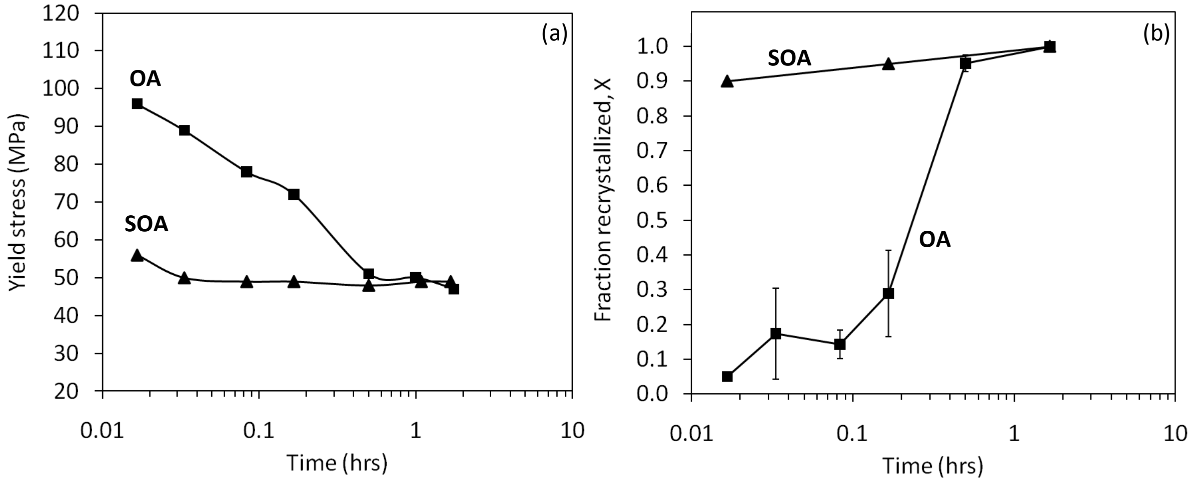

Fig. 4. Isothermal (a) softening and (b) recrystallization curves of the OA and SOA samples obtained after annealing at 445°C.

4. Discussion

In a previous article, it has been shown that the softening behaviour depicted in Fig. 2a is the consequence of complex interaction between recovery, recrystallization and precipitation [2]. In the case of the T4 samples, recrystallization was severely retarded due to the reprecipitation of the Q' precipitates on grain boundaries in the early stage of annealing. These precipitates pinned indiscriminately all grain boundaries thus making recrystallization a particularly difficult process. Consequently, the recovery process was greatly prolonged as can be observed by the large decrease in yield stress seen in the T4 samples after only 1 minute of annealing (Fig. 2a). Although the pinning effect induced by the Q' precipitates gradually decreased due to precipitate growth and coarsening as the annealing proceeded, recrystallization was extremely sluggish as much of the stored energy was already consumed by the recovery process. Note the softening curve resembled the typical logarithmic decay of yield stress due to recovery [3]. Hence, the majority of the softening in the T4 samples is attributable to the combined effect of recovery and precipitate coarsening. The softening behaviour of the OA samples is similar to the T4 samples. However, the recrystallization kinetics of the OA samples was even more sluggish. This point will be further considered later from the vantage point of recrystallization-precipitation interaction. In contrast to the T4 and OA samples, the overall softening of SOA samples was the direct consequence of combined recovery and recrystallization processes. By correlating the softening curve of the SOA samples in Figs. 2a to the recrystallization curve in Fig. 2b, it can be seen that recrystallization was responsible for the softening that occurred after ~20 hours of annealing. In this case, the precipitate coarsening kinetics was slow since the overaging temperature (325°C) before cold rolling and the annealing (325°C) temperature were identical.

As shown in Fig. 2b, the fraction of recrystallized grains in the OA samples was only about 0.3 after 1000 hours of annealing at 325°C. The prior overaging process of 250°C/7 days produced a relatively stable population of coarse and widely spaced particles in the microstructures. Upon annealing at 325°C, due to increased solid solubility in the matrix, some of the precipitates will dissolve but the majority of the precipitates will undergo growth and/or coarsening. Fig. 5a shows a backscattered electron micrograph obtained from the OA samples after 330h of annealing at 325°C. It can be clearly observed that the microstructure was composed of two zones: (i) precipitate free zones and (ii) precipitate zones. The precipitate zones were filled with dense precipitate clusters which were made up of fine scale precipitates in the order of 1-2μm oriented perpendicular to each other. The orientation of these precipitates is consistent with the typical lath shaped Q' precipitates observed in AA6111 [5-7]. On the other hand, homogeneously distributed precipitates were observed in the T4 and SOA samples after the same annealing conditions. As indicated in Fig. 5b, a growing recrystallized grain which nucleated in the vicinity of a Fe-containing particle located in the precipitate free zones. Further evidence correlating precipitate free zones to recrystallized grains was obtained by comparing electron backscattered diffraction maps to backscattered electrons micrographs obtained from the same area of the microstructure [7]. The sluggish recrystallization behaviour of the OA samples can be attributed to the inhomogeneous distribution of precipitates in the microstructure: after initiation, recrystallizing grains were allowed to grow freely in the precipitate free zones until they impinged on the boundaries of the precipitate and precipitate free zones. Furthermore, it has also been observed that the recrystallized grain size of the OA samples was considerably larger than in the T4 samples although recrystallized fractions were lower in the OA samples [4].

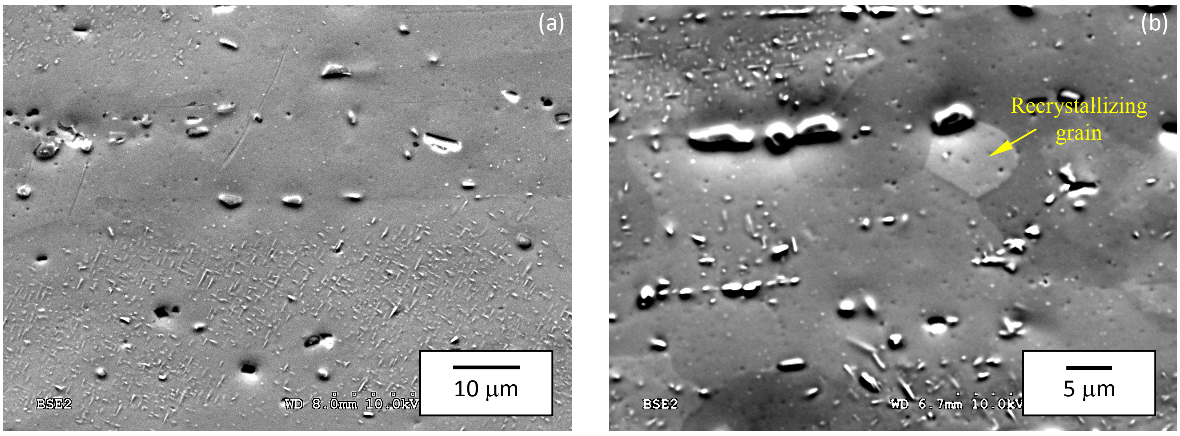

Fig. 5. Backscattered electron micrographs showing (a) the precipitate and precipitate free zones and (b) a recrystallizing grain in the vicinity of a Fe-containing intermetallic particle located in the precipitate free zones in the OA sample after annealing for 330 hours at 325°C.

By increasing the annealing temperature to 445°C, the OA samples achieved full recrystallization in about 30 minutes of annealing, as shown in Fig. 4b. Compared to the results obtained at 325°C – 0% recrystallized after 30 minutes – the increase in recrystallization rates was remarkable. This clearly indicated that the precipitation-recrystallization interaction observed at 325°C was strongly reduced. In order to account for this behaviour, one must consider the effect of precipitate dissolution. Burger et al., have provided evidence to show that significant precipitate dissolution took place in AA6111 above 430°C [8]. Precipitate dissolution promoted recrystallization beyond the nominal increase of recrystallization rates due to annealing at higher temperature thereby leading to dramatically decreased recrystallization times for the OA samples at 445°C. Nevertheless, it is noted that significant recovery occurred prior to recrystallization (Fig. 4a) and the combination of both processes contributed to the overall softening. In the case of the SOA samples, annealing at 445°C allowed recrystallization to take place before any significant recovery. Hence, recrystallization was the predominant microstructural process responsible for nearly all of the softening (Fig. 4a). Finally, it is worth noting that the fully recrystallized yield stress did not change appreciably with increasing soaking time indicating that softening due to potential grain growth is not significant in AA6111.

5. Conclusions

The experimental results obtained in the present study clearly indicated that the recrystallization behaviour of AA6111 alloys can be altered considerably by changing the precipitate conditions in the materials. By natural aging or overaging the materials prior to cold rolling (40% reduction), the recrystallization process was completely retarded up to ~90 hours of annealing at 325°C. In fact, the volume fraction of recrystallized grains in both samples remained below 0.5 even for annealing times as long as 40 days. Consequently, the majority of softening can be attributed to the combined effect of recovery and precipitate coarsening. Microstructural evidence was provided to explain the sluggish recrystallization behaviour found in the OA samples. Growth and coarsening of the precipitates in the OA samples during annealing at 325°C resulted in inhomogeneous distribution of precipitate structures. Recrystallized grains were correlated with precipitate free zones and inhibited in the precipitate zones which were filled with dense precipitate clusters. On the other hand, severe overaging the samples prior to cold rolling resulted in precipitates which exerted limited influence on the recrystallization process. By increasing the annealing temperature to 445°C, the recrystallization kinetics of the OA and SOA samples were increased by several orders of magnitude.

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References

[1] S. J. Lillywhite, P. B. Prangnell and F. J. Humphreys, Mat. Sci. Techn., 16, (2000), pp.1112-1120.
[2] J. Go, W. J. Poole, M. Militzer and M. A. Wells, Mat. Sci. Forum, 426-432, (2003), pp. 291-296.
[3] J. Go, W. J. Poole, M. Militzer and M. A. Wells, Mat. Sci. Techn., 19, (2003), pp. 1361-1368.
[4] J. Go, M. Militzer, W. J. Poole and M. A. Wells, Proc. 9th Int. Conf. on Aluminium Alloys, Ed. Nie et al., (Inst. of Mater. Eng. Australasia, 2004), pp. 1276-1281.
[5] A. Perovic, D.D. Perovic, G.C. Weatherly, D. J. Lloyd, Scripta Mater., 41, (1999), pp. 703-708.
[6] D. J. Lloyd, D. R. Evans and A. K. Gupta: Can. Metall. Quarterly, 39, (2000), pp. 475-481.
[7] J. Go, Ph.D. Thesis, University of British Columbia, Vancouver, Canada, (2006), pp. 76.
[8] G. B. Burger, A. K. Gupta, L. Sutak and D. J. Lloyd, Mat. Sci. Forum, 217-222, (1996), pp.471-478.