THE ANNEALING RESPONSE OF COLD WORKED 6000 AND 7000 SERIES ALUMINIUM ALLOYS

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ABSTRACT: The evolution of microstructure and hardness during processing of commercial AA6061 and AA7032 heat treatable alloys have been investigated. Annealing experiments performed on cold worked material revealed similar recovery and recrystallization temperature regimes for both alloys, however different grain structures were observed after comparable processing conditions: AA6061 consisted of fine equiaxed grains whilst AA7032 exhibited an elongated microstructure. This was attributed to the thermal stability of coarse particles present in the alloys. For example, AA6061 contains Mg_2Si precipitates at the recrystallization temperature, favouring particle simulated nucleation (PSN) as a recrystallization mechanism. In both alloys, salt bath heat treatment resulted in a finer grain structure than that observed in commercial practices. This effect was greater in AA7032 where less PSN occurs at slower heating rates.

Keywords: *grain structure, annealing, recrystallization, PSN, heat treatable alloys*

1. INTRODUCTION

Dislocation density may increase from a typical value of 10^{10} lines per square metre in the undeformed state, to around 10^{16} in heavily deformed work hardened metals [1]. Consequently a deformed structure is thermodynamically unstable and the desire to lower the total energy of the system provides a driving force for the changes taking place during annealing. Softening occurs in two stages, recovery and recrystallization, and whilst the driving force for these two phenomena is the same, the mechanisms and effects on properties are quite different [2]. Partial restoration of mechanical properties occurs during recovery, in which dislocations are rearranged to lower energy configurations, whereas primary recrystallization involves the formation of new, strain-free grains at the expense of the deformed microstructure. This is usually accompanied by more significant restoration of mechanical properties.

Nucleation of primary recrystallization generally occurs in a heterogeneous manner, at regions of the microstructure where locally the extent of deformation is highest [3]. In single phase materials suitable sites may be the original deformed grain boundaries, deformation or transition bands, and shear bands [4]. The nucleation process itself has been the subject of intense research and debate for many years during which numerous models have been proposed. Most hypotheses can usually be attributed to one of the following mechanisms; subgrain growth [5], subgrain coalescence [6], or bulge nucleation [7]. In recent years the effects of large particles on recrystallization behaviour have been studied, giving rise to the mechanism of particle stimulated nucleation or PSN [8]. It is generally accepted that the critical size of particle capable of causing nucleation is approximately 1μm [9], although there is mention of particles as small as 0.5μm causing PSN when spaced more than five diameters apart [10].

2. EXPERIMENTAL PROCEDURE

All specimens used in this investigation were taken from material which was cast via a Direct Chill (DC) process, stress relieved and homogenised to provide a suitable microstructure for subsequent cold deformation processes. Homogenised AA6061 and AA7032 billets were subjected to a cold deformation process, with total reduction in area corresponding to true strains of 1.87 and 1.70 (approximately 85% and 82% cold work) respectively. Small specimens were annealed in an air circulating oven between 150°C and 450°C for a constant time of 30 minutes and allowed to air cool. Vickers hardness tests were carried out using a load of 5 kg, and a minimum of 5 readings were taken to ensure reproducibility. At higher annealing temperatures, duplicate specimens were allowed to cool slowly in the oven in order to inhibit any solid solution strengthening which may have occurred during the more rapid air cooling.

In order to examine recrystallized microstructures, cold worked material was solution heat treated and aged according to the standard practice for each alloy. Specimens ramped at typical commercial oven rates, i.e. reaching temperature in 2-3 hours, were compared to duplicate specimens solution heat treated in a salt bath, taking less than 5 minutes to reach temperature. Metallographic samples were prepared using standard grinding and polishing techniques, before anodising in Barkers Reagent and examining under polarised light to reveal grain structures. Since, for these alloys, optical microscopy is generally only capable of revealing grain structures in the solution heat treated and aged condition, selected annealed specimens were prepared for examination in a scanning electron microscope (SEM) in order to investigate the possible presence of PSN. This involved electropolishing in a chilled solution of 25% nitric acid / 75% methanol and viewing under backscattered electron imaging conditions.

3. RESULTS

Graphs of hardness versus annealing temperature are presented in Figure 1 below for AA6061, and Figure 2 overleaf for AA7032. It can be seen that the cold worked hardness was significantly higher then the undeformed state in both alloys, which exhibited essentially the same two-stage softening behaviour upon subsequent annealing. The as-extruded hardness decreased gradually upon annealing at temperatures above 200°C for AA6061 and 150°C for AA7032. Softening occurred at an increased rate for temperatures higher than 275°C, such that the material was fully soft after annealing at 325°C. Beyond this point, increasing the annealing temperature did not bring about a further reduction in hardness.

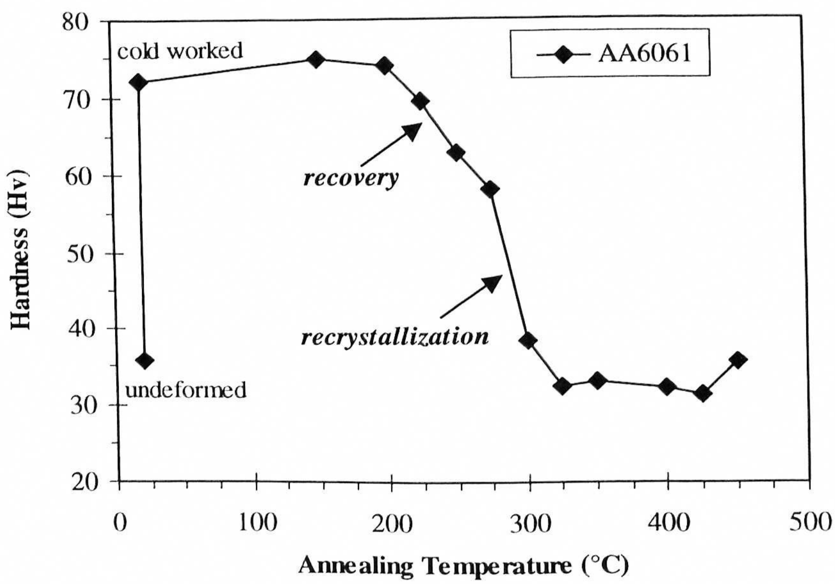

FIGURE 1 - Annealing response of cold worked AA6061 material (30 minutes at temperature)

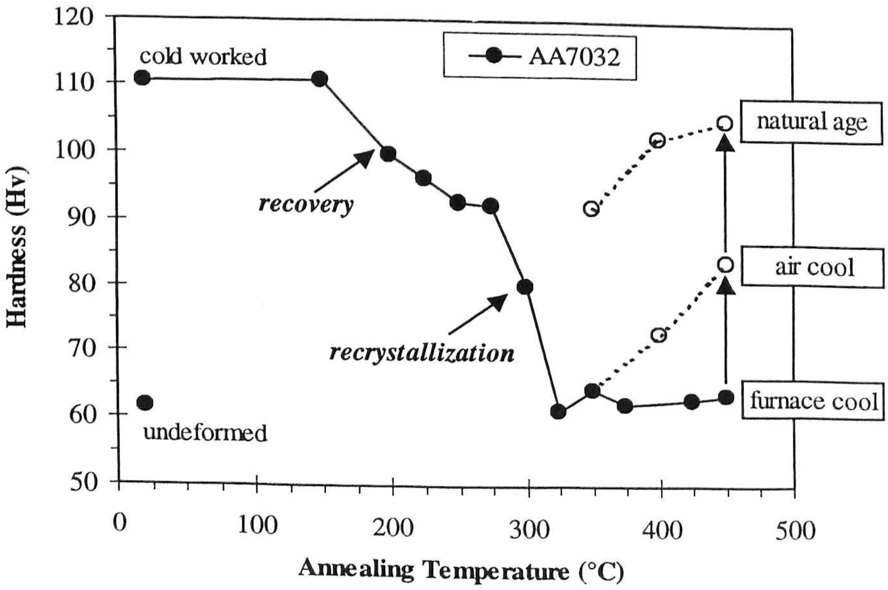

FIGURE 2 - Annealing response of cold worked AA7032 material (30 minutes at temperature)

Results for AA7032 are shown above in Figure 2, which also include hardness values for specimens which were allowed to cool slowly from the higher annealing temperatures. Solid lines represent hardness after slow furnace cooling which can be assumed constant above 325°C, taking into account scatter in the hardness measurements and reproducibility of furnace cooling rates between samples. The hardness of the air cooled samples increased as the annealing temperature increased and furthermore, additional hardening was apparent due to natural ageing after repeat testing one week later. No such effects were observed in the AA6061 specimens.

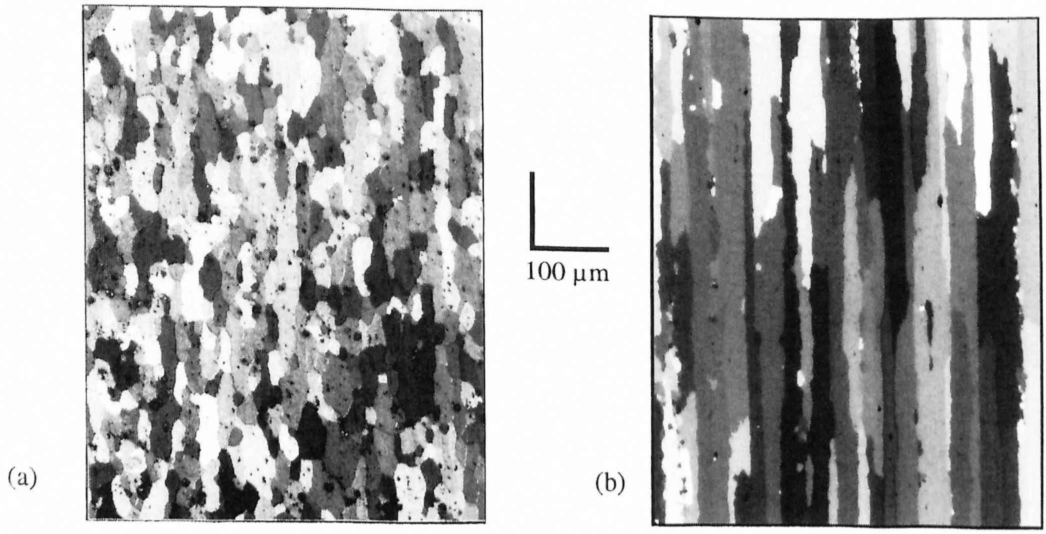

FIGURE 3 – Commercial oven heat treated and aged microstructures: (a) AA6061; (b) AA7032.

(a) (b)

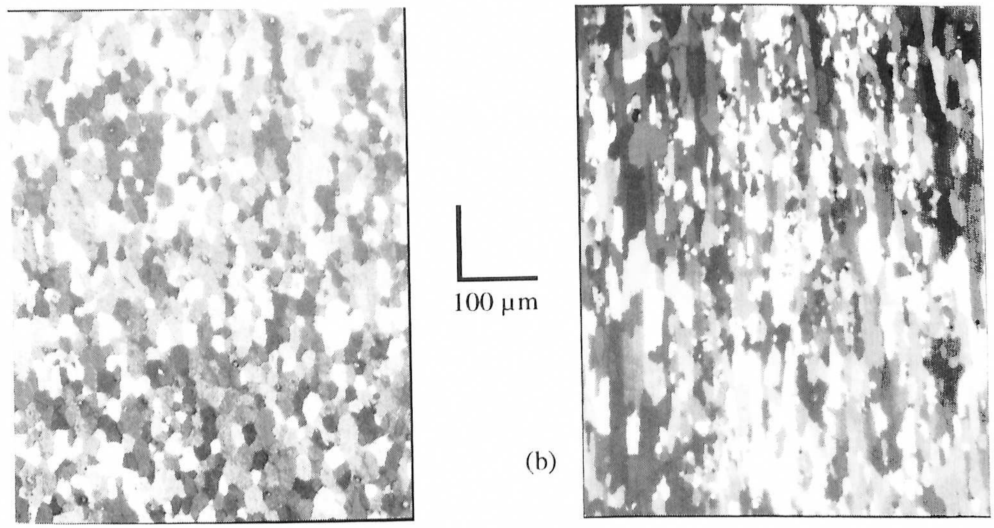

FIGURE 4 – Salt bath solution heat treated and aged microstructures: (a) AA6061; (b) AA7032.

Solution heat treated and aged microstructures are presented in Figures 3 and 4 above, viewed under polarised light. For conventional oven processing, AA6061 (Fig. 3a) exhibits a finer and more equiaxed grain structure than that observed in AA7032 (Fig. 3b), which is elongated in the direction of cold working. Salt bath treatment, Figs. 4a and 4b, resulted in a finer structure in both alloys, although the difference with respect to oven processing is greater in AA7032, Fig. 4b.

Figures 5a and 5b respectively show backscattered electron images of fully and partially annealed AA6061 material. The fully annealed grain structure in Figure 5a is fine and equiaxed, of the order 10µm. Figure 5b shows quite clearly that the limited number of recrystallized grains present in the partially annealed specimen have nucleated preferentially in the vicinity of coarse Mg_2Si particles.

(a) (b)

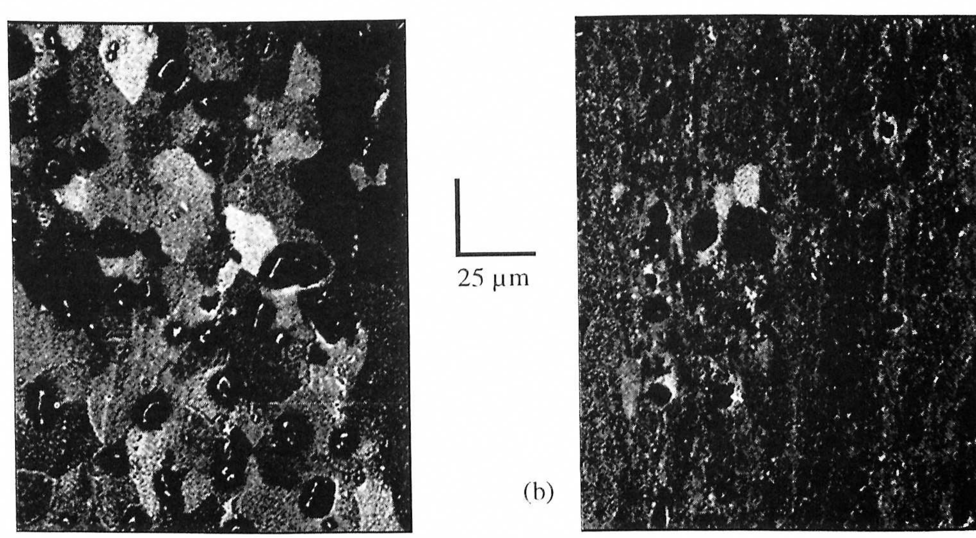

FIGURE 5 - Backscattered electron images of (a) fully annealed; (b) partially annealed AA6061.

4. DISCUSSION

Material hardness increases significantly with cold deformation for both AA6061 and AA7032, as shown in Figures 1 and 2. The microstructural and hardness changes occuring during subsequent annealing can be understood by considering the nature of these curves, which are essentially the same for both alloys. Cold worked material undergoes two distinct softening reactions. The first is quite gradual and occurs at relatively low temperatures up to 275°C, causing a reduction of approximately 40% in the total work hardening from cold extrusion. The second stage brings about a more rapid decline in material hardness, with the remaining 60% work hardening diminishing over a fairly narrow temperature range, between 275°C and 325°C. This two-stage behaviour is characteristic of recovery and recrystallization phenomena frequently observed in aluminium and its alloys.

It is interesting to note that the recovery process in AA7032 appears to start at a lower temperature than in AA6061, 150°C compared to 200°C. A possible explanation for this may be found in the heat evolved during the 'cold' deformation process. AA6061 material reached a temperature of 150°C during deformation whereas AA7032, due the inherent higher strength of the alloy, was measured at 200°C. It is quite feasible therefore that rearrangement of dislocations, i.e. dynamic recovery, could be taking place during processing at these temperatures. In effect, recovery is already in progress in AA7032 before annealing commences and consequently subgrain formation and growth is able to continue at low temperatures. This does not affect the total reduction in hardness due to recovery, however, the curve simply flattens out before the onset of recrystallization indicating that there is clearly a limit to the reduction in stored energy which can be achieved by this mechanism alone. Full restoration of the undeformed mechanical properties is only achieved after recrystallization, which for both AA6061 and AA7032 is complete after annealing at 325°C. Annealing of AA7032 at temperatures above 350°C can result in solid solution strengthening and subsequent natural ageing unless equilibrium slow cooling rates are employed. This is due to the instability of the $MgZn_2$ precipitates formed during post-homogenisation cooling, and may lead to misinterpretation of as-annealed results unless testing is carried out immediately after heating. This behaviour was not exhibited by AA6061 to any significant extent, which can be attributable to the greater stability of coarse Mg_2Si particles formed during homogenisation.

Microstructures produced after solution heat treatment and ageing at conventional oven heating rates are shown in Figure 3. Significant differences exist between the grain structures of the two alloys: AA6061 consists predominantly of equiaxed grains whilst AA7032 exhibits a more elongated microstructure in the deformation direction. This can be attributed to the difference in coarse particles present in the alloys after homogenisation, i.e. Mg_2Si in AA6061 and $MgZn_2$ in AA7032. AA6061 contains a uniform distribution of precipitates which are stable at temperatures well above the completion of recrystallization, thus providing many sites for PSN despite a reduction in the stored energy of cold working due to recovery. PSN has been reported in 6000 series alloys [11], and further evidence is provided in Figure 5. AA7032, however, contains precipitates which are unstable, as demonstrated by the solution strengthening and natural ageing effects shown in Figure 2. Consequently, dissolution of particles occurs simultaneously with recrystallization during annealing resulting in a decrease in the volume fraction of particles capable of stimulating nucleation as temperature increases. The resultant final grain structure is therefore largely dependent on the volume fraction and distribution of other coarse particles. Previous work has shown that no S-phase ($CuMgAl_2$), which can often occur in commercial 7000 series alloys, is present after homogenisation leaving only soluble $MgZn_2$ precipitates and the Cu_2FeAl_7 constituent phase [12]. This phase forms as long clusters of intermetallic particles strung out along the direction of deformation. The total volume fraction of particles is very small, however, such that the statistical probability of a migrating grain boundary being pinned is much lower in the longitudinal orientation than in the transverse direction. Hence grain growth in AA7032 occurs primarily in the direction of working and the recrystallized structure tends to be elongated, as observed in Figure 3b.

Figure 4 shows that rapid heating to the solution heat treatment temperature can significantly affect the recrystallization behaviour of both alloys. Whilst some of the microstructural changes associated with recovery are in fact a necessary prerequisite of new grain nucleation, it should also be remembered that the concomitant reduction in stored energy effectively lowers the driving force for recrystallization. Rapid heating, however, allows less time for recovery to take place prior to recrystallization. In both alloys, the higher level of retained stored energy afforded by salt bath heat treatment has lead to an increase in nucleation and hence a finer grain structure than that observed in the commercial practice. The effect is more pronounced in AA7032 where there is less particle stimulated nucleation at the slower heating rate.

5. CONCLUSIONS

1) Homogenised material from both AA6061 and AA7032 alloys undergo significant work hardening when cold deformed (80-85% reduction).
2) Cold worked material undergoes two distinct softening processes:
   (i) recovery occurs between 150°C and 275°C, reducing work hardening by approx. 40%, and
   (ii) recrystallization between 275°C and 325°C eliminates the remaining 60% cold work.
3) Commercial oven solution heat treatment practices result in a predominantly equiaxed grain structure in AA6061, whilst AA7032 grains are elongated.
4) Particle stimulated nucleation (PSN) has been observed at coarse Mg_2Si particles in AA6061. Mg_2Si is stable at temperatures well above the completion of recrystallization.
5) Dissolution of $MgZn_2$ precipitates occurs during high temperature annealing of AA7032. The reduced volume fraction of large particles leads to less efficient nucleation and hence a coarser final grain size than in AA6061.
6) Rapid salt bath heating leads to a finer recrystallized grain size in both alloys, and in AA7032 produces a more equiaxed structure. This is due to the suppression of recovery and a resultant increase in nucleation, since a higher level of stored energy exists at the onset of recrystallization.

ACKNOWLEDGEMENTS

This research programme was made possible with 50% funding from the Royal Commission for the Exhibition of 1851. Sincere gratitude is also extended to F.J. Humphreys and I. Brough at Manchester Materials Science Centre, for use of facilities and discussions.

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