CONTROL OF RECRYSTALLIZATION IN Al-Mg-Sc-Zr ALLOYS

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ABSTRACT

A series of aluminum alloy ingots containing between 0-3.5 wt% Mg with combinations of Sc (0, 0.1, 0.2 wt%) and Zr (0, 0.12 wt%) were cast using controlled solidification rates (5K/s) comparable to commercial practice. Following a preheating routine to homogenize solute and precipitate the Al_3Sc-$L1_2$ and Al_3Zr-$L1_2$ dispersoids the ingots were hot rolled at 425°C to 40, 63, and 80% total thickness reduction. In some cases the 80% hot rolled ingots were reduced further by cold rolling to 40, 63, and 76% of the hot deformed thickness. Stereological techniques were used to determine the volume of recrystallized material after both isothermal annealing for extended time and isochronally annealing for 1hr. at various temperatures. It is shown that the Al_3Sc dispersoid is much more effective in inhibiting recrystallization than Al_3Zr but the combination of Al_3Sc and Al_3Zr is better than either dispersoid acting alone. The morphology of recrystallized grains differs between Al-Sc and Al-Zr alloys. Al-Sc alloys tend to recrystallize only after a lengthy recovery process by consumption of low angle subgrain material. This is probably due to well dispersed stable Al_3Sc dispersoids and their ability to maintain coherency for long times at temperatures near the Al-Sc solvus boundary. On the other hand Al_3Zr coarsens rapidly at much lower temperatures and may lose coherency allowing recrystallization to occur by nucleation and growth of equiaxed grains.

KEYWORDS

Scandium, Dispersoid, Recrystallization, Hot Work, Cold Work

INTRODUCTION

The 5XXX series Al-Mg alloys exhibit good formability, high resistance to general corrosion, and have superior weldability. The strength of alloys in this system is controlled by solid solution and strain hardening but is relatively low compared to heat treatable alloys such as 2024, 6061 and 7075. Exposure to elevated temperatures provides a mechanism to remove the high energy state of a strained microstructure through recovery and recrystallization of the alloy. Associated with recrystallization is a dramatic decrease in mechanical properties. Typical recrystallization temperatures for aluminum alloys range from 340-410°C [1]. The presence of finely dispersed high number density phases, referred to as dispersoids, suppress recrystallization by providing a pinning force to retard an advancing boundary. The most potent dispersoid reported is the metastable Al_3Zr. The particles are coherent with the aluminum matrix forming an $L1_2$ phase. Ryum[2] demonstrated that an Al-0.5wt%Zr alloy was resistant to recrystallization up to 500°C when Al_3Zr dispersoids were present.

Recently the advantages of adding scandium to aluminum alloys have been documented When present in aluminum alloys, scandium combines with aluminum to form Al_3Sc which, like the metastable Al_3Zr, has $L1_2$ structure and is also coherent with the aluminum matrix. Surprisingly, only limited work has been published to demonstrate the effectiveness of Al_3Sc in retarding recrystallization in wrought commercial products. The main thrust of this work is focused on quantifying the interactions between the amount of hot and cold deformation, annealing time and temperature, magnesium content and the presence of zirconium in retarding recrystallization in ingots containing from 0-0.2wt% scandium.

EXPERIMENTAL

Ingots measuring 15 x 7 x 2 cm were prepared by melting high purity aluminum (99.99%) in a resistance type furnace at 720°C with specific alloy additions. Master alloys used for the ingots were Al-6.3Zr, Al-6Mg, and Al-2Sc. Melting was done in clay-bonded graphite crucibles coated with boron nitride to reduce chemical reactions between the crucible and the melt. The melt was poured into room temperature steel molds also coated with boron nitride. Cooling rate under these conditions has been previously reported to be 5K/s[3]. All alloy compositions are reported in weight percent. To homogenize the as-cast ingots a ramp heat-up rate of 50°C/hr to a soak temperature of 450°C, hold for 4 hours, then quench in cold water was used. The solidification conditions and preheating schedules used are typical of those used on commercial scale ingots.

Ingots were hot rolled at 425°C in three passes. The first pass through the hot mill reduced the thickness by 40%, the second by 63%, and the third by 80% of the original thickness. After each pass the ingots were quenched to room temperature in cold water and sectioned for metallographic analysis. To maintain a constant deformation temperature the ingots were rapidly reheated to 425°C in a resistance-type furnace within 15 minutes then rolled for another pass.

For some alloys it was necessary to provide further deformation by reducing the sheet thickness an additional 76% of the thickness of the hot rolled material. These alloys were reduced in three stages by cold rolling 40%, 63%, and 76% from the hot rolled thickness. No annealing was given to the cold rolled ingots between passes so that the cold deformation would be cumulative. Table 1 includes the average thickness resulting from the as-cast state to each rolling stage.

Table 1: Average alloy thickness after each step of rolling

Condition	thickness (cm.)
As-Cast	1.97
40% Hot Roll	1.18
63% Hot Roll	0.73
80% Hot Roll	0.39
40% Cold Roll	0.24
63% Cold Roll	0.14
76% Cold Roll	0.09

Samples taken along the rolling direction at the centerline of the deformed ingots were annealed at various temperatures and times to investigate recrystallization behavior. Annealing was done in a resistance type furnace between large slabs of aluminum plate. The plates were preheated to the desired annealing temperature. Samples and a thermocouple were placed between these plates to ensure the annealing temperature was known and maintained at a constant value. All annealings were followed by an immediate quench in cold water.

Metallographic samples for optical microscopy were prepared by mounting the plane containing the long and short transverse directions for inspection in bakelite, grinding, and polishing to 0.04μm finish using standard techniques. The samples were anodized using a solution of 0.5L H_2O, 23mL fluoboric acid, and 35g boric acid for approximately 20 seconds. A piece of 304 stainless steel was used as the anode under an applied voltage of 18VDC. The anodization developed good grain and subgrain contrast under polarized light for studying recrystallization. Optical microscopy was done using a Reichert-Jung MeF3 microscope under polarized light. Determination of the volume fraction of recrystallized grains in annealed samples was performed using the point method outlined by Underwood[4].

RESULTS AND DISCUSSION

I. Phase Diagrams

Binary phase diagrams of Al-Sc and Al-Zr were gathered from literature to help interpret the precipitation behavior during elevated temperature annealing. The equilibrium Al-Zr binary phase diagrams taken from Wright and Wiley[5] combined with Al-Sc solvus data from Gschneidner *et al.*[6]and Fujikawa *et al.*[7] provided an estimate for the limits of maximum solubilities for each of the binary systems. Fig.1 is a composite diagram of equilibrium solvus boundaries for Al-Sc and Al-Zr. This information combined with the recrystallization studies helped to interpret the individual and combined roles of Al_3Sc and Al_3Zr in these alloys.

II. Comparison of Al-0.2Sc, Al-0.12Zr, & Al-0.12Zr-0.2Sc

A study of binary Al-0.2Sc and Al-0.12Zr systems after 80% hot deformation shows a dramatic difference in recrystallization behavior of the two systems. Fig.2 shows the volume fraction of recrystallized material versus time behavior of Al-0.12Zr, Al-0.2Sc, and Al-0.12Zr-0.2Sc when hot deformed to 80% reduction. It was found that recrystallization in the Al-0.12Zr alloy occurred very rapidly at this level of deformation. Therefore annealing was done at 400°C for very short times to measure volume fraction of recrystallized material versus time. At 550°C the Al-0.2Sc alloy is still much slower to recrystallize than Al-0.12Zr at 400°C. When the ternary Al-0.12Zr-0.2Sc system is annealed at 590°C little recrystallization occurs after 24hrs.

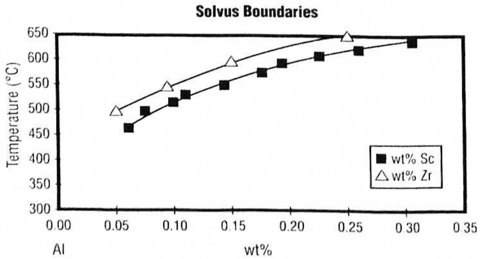


Fig.1: Al-Sc, Al-Zr solvus boundaries

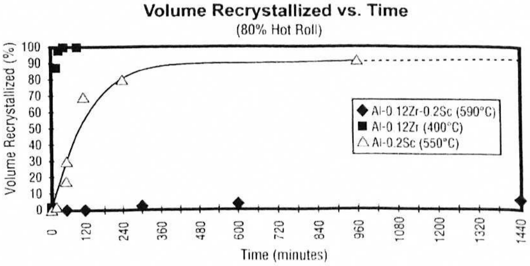
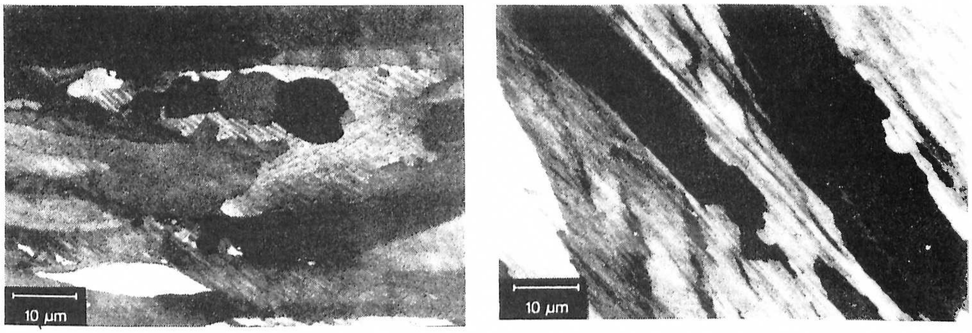


Fig.2: Al-0.12Zr (80%Hot Roll) at 400°C,
Al-0.2Sc (80%Rot Roll) at 550°C
Al-0.12Zr-0.2Sc (80%Hot Roll) at 590°C

Optical microscopy of these alloys shows a difference in recrystallized grain morphology between Al-0.12Zr and Al-0.2Sc. In Fig.3a an Al-0.12Zr alloy has annealed for 1 minute at 400°C. At the interior of grains and near grain boundaries equiaxed, recrystallized grains are beginning to consume the surrounding deformed grains. The deformed grains still contain well developed subgrain bands. Fig.3b shows the growth of recrystallized grains in Al-0.2Sc after 30 minutes at 550°C. Recovery of the deformed grains precedes recrystallization, as noted by a decrease in contrast between subgrains under polarized light. The growth of recrystallized grains in this alloy tends to preferentially consume low angle bounded subgrains up to, but not beyond, the original deformed high angle grain boundary. Only after most of the subgrain material has been consumed by the growth of recrystallized grains does penetration through original high angle boundaries into neighboring grains occur.



(a) (b)

Fig. 3: a) Al-0.12Zr (80% Hot Roll) annealed 1min., 400°C
b) Al-0.2Sc (80% Hot Roll) annealed 30min., 550°C

There is evidence of a beneficial interaction between Sc and Zr. An alloy containing Al-0.12Zr-0.2Sc deformed by 80% hot rolling shows superior resistance to recrystallization over both Al-0.12Zr and Al-0.2Sc in the same conditions [Fig.2]. Volume fraction recrystallized versus temperature for these alloys after annealing for 1hr. demonstrates the combined effect of Zr and Sc in Al [Fig.4]. The Al-0.12Zr alloy is completely recrystallized at 400°C. No work below this temperature was done to determine the start and finish temperatures of recrystallization for this alloy. Recrystallization was suppressed to 550°C for Al-0.2Sc. No recrystallization occurred in Al-0.12Zr-0.2Sc to 590°C. Inspection of the binary Al-Zr diagram shows that 400°C is far below the solvus boundary of 572°C for Al-0.12Zr. This suggests that recrystallization in Al-0.12Zr is not due to dissolution of the Al_3Zr dispersoid at this level of deformation. Rapid coarsening and loss of coherency of Al_3Zr in this alloy are most likely responsible for the decreased effectiveness of this dispersoid to retard recrystallization by Zener drag. From the Al-Sc binary diagram 550°C is only 40°C below the solvus temperature of 590°C for 0.2Sc. The estimated solubility of Sc at 550°C is about 0.12Sc so an appreciable decrease in volume fraction of Al_3Sc should occur resulting in a decrease in recrystallization resistance for heavily deformed alloys. The combination of well distributed Al_3Sc, high level of deformation, and high recrystallization temperature suggests that the Al_3Sc dispersoid maintains coherency and is effective in retarding recrystallization, via the Zener drag mechanism, up to the Al-Sc solvus boundary. At this point rapid growth and/or dissolution of Al_3Sc is expected.

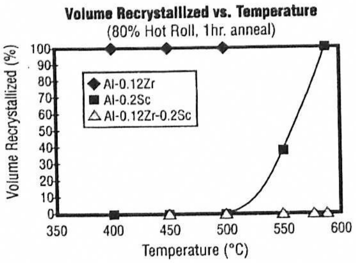


Fig.4: Al-0.12Zr (80% Hot Roll), Al-0.2Sc (80% Hot Roll)
Al-0.12Zr-0.2Sc (80% Hot Roll)

III. Effect of Mg content on Al-0.2Sc & Al-0.12Zr-0.2Sc

The recrystallization resistance for Al-0.2Sc-Mg and Al-0.12Zr-0.2Sc-Mg in the 80% hot rolled condition after annealing for 1hr. at various temperatures was studied. Increasing the Mg content decreases the recrystallization resistance of both alloys. All the alloys containing Sc showed significant increase in resistance to recrystallization compared to alloys without Sc. Volume fraction recrystallized versus temperature for samples annealed 1hr. are shown in Fig.5.

(a)

(b)

Fig.5: a) Effect of Mg on Al-0.2Sc (80% Hot Roll)
b) Effect of Mg on Al-0.12Zr-0.2Sc (80% Hot Roll)

IV. Effect of Sc content on Al-0.12Zr-Sc-2Mg

To study both the potency of Sc and the effectiveness of combining Sc and Zr in Al, the resistance to recrystalliztion for Al-0.12Zr-Sc-2Mg in the 80% hot rolled state was examined [Fig.6]. By lowering the Sc content from 0.2Sc to 0.1Sc the recrystallization start temperature is unaffected at 550°C. At 590°C however the 0.1Sc alloy recrystallizes completely while the alloy with 0.2Sc has recrystallized only 10%. In either case the recrystallization temperature is unusually high for Al alloys. In comparison without Sc the alloy was completely recrystallized at 400°C and probably would have recrystallized at an even lower temperature.

V. Recrystallization behavior of Al-0.12Zr-0.2Sc-2Mg after 80%cold deformation

Since the recrystallization resistance limit of Al-0.12Zr-0.2Sc-2Mg was not reached after 80% hot deformation even at 590°C, further cold rolling was added to the alloy. At 590°C this alloy is below the Al-2Mg solidus boundary by only about 30°C so higher temperature studies would have dissolved Al_3Sc and/or melted the alloy. A total of 76% cold deformation was added to the 80% hot rolled alloy and volume recrystallized versus time was studied at 590°C [Fig.7]. A partially recrystallized microstructure was maintained at 590°C for up to 10hrs. The potency of Al_3Sc, combined with Al_3Zr, is seen even at this very high level of deformation and annealing temperature. After 24 hours of annealing at 590°C this alloy contains an average of 1-3 recrystallized grains across the cross section. Longer annealing times may have yielded a single crystal. Coherency and/or slow dissolution of Al_3Sc are again expected to be responsible for retaining the unrecrystallized state up to the Al-Sc solvus for long times.

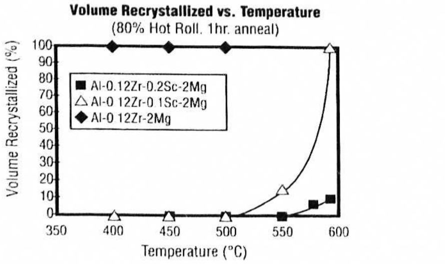


Fig.6: Effect of Sc on Al-0.12Zr-2Mg (80% Hot Roll)

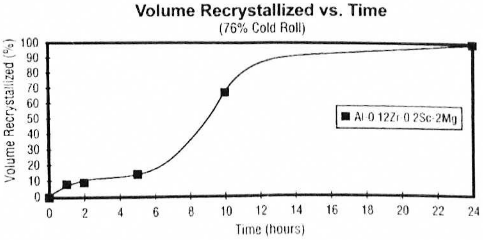


Fig.7: Al-0.12Zr-0.2Sc-2Mg (76% cold rolled)
(annealed at 590°C)

CONCLUSION

The presence of Al_3Sc and Al_3Zr dispersoids in wrought Al-Mg alloys proves to be a more powerful inhibitor of recrystallization compared to either Al_3Sc or Al_3Zr alone. Recrystallization resistance in Al-0.2Sc binary alloys is far superior to that of Al-0.12Zr binary alloys. The presence of very stable cohérent Al_3Sc dispersoids in a heavily deformed Al matrix is responsible for this increase in properties. Low angle subgrain boundaries tend to align during a lengthy recovery before recrystallization occurred in Al-Sc as low as 550°C. Recrystallized grain growth in Al-Sc alloys consumes the subgrain material of the parent grain completely before penetration of as-cast grain boundaries occurs. At 80% hot deformation the Al-0.12Zr alloy showed little resistance to recrystallization at 400°C, the lowest temperature studied in this work. The Al-0.12Zr solvus boundary of 572°C is much higher than 400°C. Recrystallization in Al-0.12Zr is probably not due to dissolution of Al_3Zr but rather coarsening of the Al_3Zr dispersoid and loss of coherency with the Al matrix weakening the effectiveness of this particle under Zener drag forces. This allows recrystallization to take place by nucleation and rapid growth of equiaxed grains.

Alloys containing both Zr and Sc are significantly more resistant to recrystallization than either Al-Sc or Al-Zr binaries. This may be attributed to the distribution of Al_3Sc and Al_3Zr dispersoids after ingot preheating. An Al-0.12Zr-0.2Sc alloy deformed 80% by hot deformation had no recrystallized material in it after 1hr. at 590°C, which is above the Al-Zr solvus temperature and at the Al-Sc solvus temperature.

As magnesium content increases the recrystallization start temperature of Al-Sc and Al-Zr-Sc alloys decreases. Magnesium contents up to 2wt% do not dramatically affect the recrystallization temperature of these alloys. When increased to 3.5wt%, however, partial recrystallization is seen in Al-0.12Zr-0.2Sc-3.5Mg as low as 400°C.

Although it is typically beneficial to use the maximum amount of solid solution solute possible, as with magnesium, in Al alloys it is clear that Sc contents lower than the 0.2wt% maximum are still very effective. Lowering the Sc content from 0.2wt% to 0.1wt% the Al-0.12Zr-Sc-2Mg alloy deformed 80% by hot rolling only reduced the recrystallization start temperature from 575°C to 550°C. Both temperatures are well above the recrystallization limits of typical Al alloys. This work demonstrates a promising capability for Sc to be successfully integrated into commercial 5XXX aluminum alloys.

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