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THE EFFECT OF COLD WORK ON THE PRECIPITATION AND RECRYSTALLIZATION KINETICS IN AL-SC-ZR ALLOYS

C.T. McNamara, S.L. Kampe, P.G. Sanders, D.J. Swenson
Department of Materials Science and Engineering, Michigan Technological University, Houghton, MI 49931

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Abstract

Scandium plus zirconium additions to aluminum offer potent alloy strengthening opportunities with enhanced kinetic stability at elevated temperatures, attributable to a favorable sequence of trialuminide precipitation events which occur. This work examines the additional variable of prior cold work on precipitate aging kinetics, and on the recrystallization of the underlying aluminum matrix. Al-0.07Sc-0.08Zr (at%) alloys have been cast, swaged, and isochronally aged over a range of temperatures to quantify hardening response and degree of recrystallization. Cold-worked specimens are compared to as-cast variants; the recrystallization response of the alloy is compared to pure aluminum. These data demonstrate the expanded and optimized property space attainable via microstructural response to thermomechanical processing.

Introduction

The coherent precipitation of scandium trialuminide from a supersaturated solution occurs both discontinuously at moving grain boundaries, and continuously within FCC grains, the latter being predominant in dilute, work hardened systems [1]. This mode of precipitation provides impressive dispersion strengthening effects, even at low Al_3Sc volume fractions. Furthermore, through the secondary core-shell precipitation of coherent zirconium trialuminide, coarsening resistance is increased at elevated temperatures. A recent publication by Knipling et al. [2] contains an isochronal aging study on an Al-0.10Sc-0.10Zr at% alloy and have determined peak aging conditions. Fuller et al. [3] have performed aging studies on several Al-(Sc,Zr) compositions and have incorporated a two-stage heat treatment which emphasizes the primary and secondary precipitation events. It is well documented that increased dislocation density will lead to increased continuous precipitation within grains, thus forming a finer array of the second phase. For example, Blake and Hopkins [4] have shown that prior cold work leads to a faster age hardening response and increased hardness, due to heterogeneous precipitation within grains and at grain boundaries. In addition, alloying elements typically have an influence on the recrystallization of α-Al following deformation processing. Martin and Doherty [5] present a review of the experimental evidence in favor of the impurity drag theory, which states that recrystallization is slowed due to the attraction of impurity atoms to advancing grain boundaries. Since both Sc and Zr are slow diffusers in aluminum, this effect may be prominent in the current system, even with an overall concentration of 15 parts in 10,000.

Experimental Method

Casting

18mm cylindrical bars are cast in a permanent steel mold from an induction-heated graphite crucible. Graphite acts as a susceptor, and its use allows the melt to retain heat better than a standard alumina crucible. The melting procedure follows that of Johnson [6], and is performed under 99.99% argon (Northern Welding Supply) cover gas flowing into the top of a Dura Line furnace (Inductotherm) at a rate of 2.4 L/min. First, 99.99% aluminum ingot (Belmont Metals) is melted and superheated to 850°C. Degassing is then performed using a rotary degasser with a graphite lance flowing argon at a rate of 2.4 L/min. Scandium (KB Alloys) and zirconium (KBM Affilips) are added in the form of 2.1 wt% and 10.2 wt% master alloys, respectively. The melt temperature is then increased to 900°C for ten minutes to allow full dissolution of the trialuminides in the master alloys through induction stirring. Pouring is performed at 760°C by first transferring the melt to a ladle, and then pouring directly into the permanent mold. An array of thermocouples is incorporated into the mold design giving an initial cooling rate of approximately 40˚C/min at the surface of the casting. The castings are allowed to cool slowly in air post-solidification.

Several compositions are cast during one melting session; an Al-0.07Sc-0.08at% Zr (Al0708) composition is used extensively for the current study. Control samples of 99.99% Al are also used to examine recrystallization response in the absence of alloying additions. Compositions are confirmed through optical emission spectroscopy (ICP-OES).

Wrought Processing, Hardness Measurements, and Optical Microscopy

The as-cast cylindrical samples are cold worked at room temperature using a rotary hammer swage. The as-cast diameter is reduced to fixed die sizes, constituting values of cold work shown in Table 1.

Sections taken from the swaged and as-cast Al0708 bars are subjected to isochronal heat treatment in which the discs are aged for 3.0 hours in air at temperatures of 200, 275, 325, 350, 375, 425, and 500°C. The temperatures are spaced more closely in the 300-400˚C range, since the peak aging temperature is expected to lie within this range [2].

To illustrate the hardening response, the samples are metallographically polished and Vickers microhardness measurements are taken along radial directions on the cross section of the swaged bars. A minimum of 12 measurements are taken per sample, with a load of 200g and a dwell time of 5 seconds.

Table 1: Swaged diameters of Al0708 bars, and resulting reductions of area.

	D_0	D_1	D_2	D_3	D_4
Diameter (mm)	18.4	17.5	15.9	14.4	12.8
Reduction of Area	0%	9%	25%	39%	52%

All samples are electrolytically polished and, in many cases, anodized to facilitate an optical assessment. To illustrate the effect of alloying content on recrystallization of the α-Al matrix, images are taken of Al0708 and pure Al in the 25% cold work state at each aging temperature, where the pure Al samples are aged for a maximum of 20 minutes.

Analysis and Discussion

The Vickers hardness measurements, plotted in Figure 1, illustrate two important findings resulting from deformation processing and aging of Al0708.

First, the hardness in the as-cold-worked state (at 25°C in Fig. 1) increases with increasing reduction of area. This effect has been thoroughly explored [7], and is expected as the number density of barriers to dislocation motion (i.e. grain boundaries and deformation dislocations) increases with amount of cold work.

Second, the peak hardness has no significant dependence on amount of deformation, as all of the peak hardness measurements overlap around 460 MPa, within a standard deviation of each dataset. It is noted that the peak hardness in this experimentation is significantly lower than the observed peak hardness of 668 MPa for a Al-0.10Sc-0.10Zr at% alloy observed by Knipling et al. [2].

Additionally, though it cannot be stated with confidence, there appears to be a hardness plateau after the peak aging occurs, possibly attributable to the secondary precipitation of zirconium trialuminide.

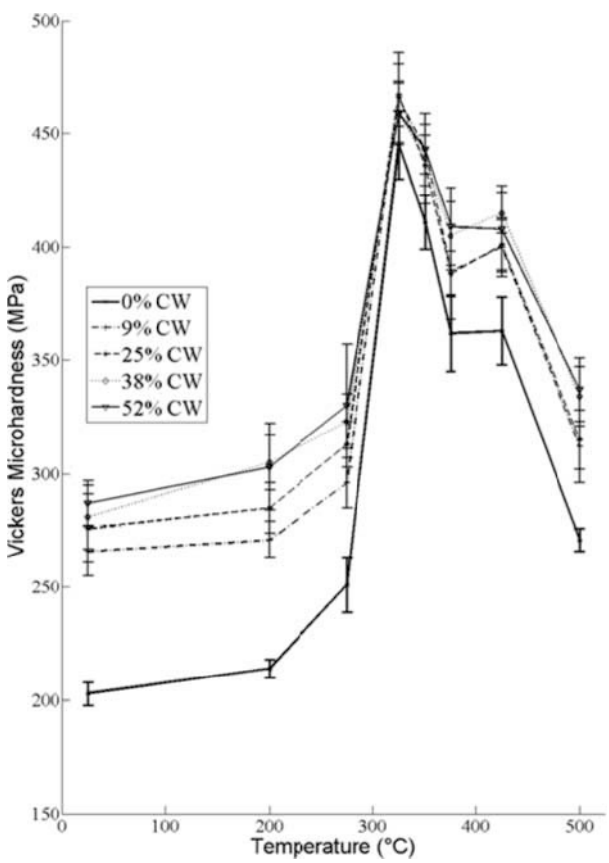

Figure 1: Hardness response to isochronal aging of cold worked Al0708, where the error bars represent one standard deviation from twelve measurements. Peak hardness for all reductions of area is observed at 325°C.

Qualitative conclusions may be drawn from the microstructures in the pure Al and Al0708 25% CW samples. The unalloyed as-swaged aluminum samples exhibit large columnar grains with radial orientations, which indicate slow directional solidification, as shown in Figure 2. For these samples, the columnar grain morphology remains throughout the 20 minute heat treatment temperature range of 100-375°C, and a recrystallized structure is obtained after a 500°C (20 minute) heat treatment; the fully recrystallized structure compared to the as cold-worked structure is shown in Figure 3.

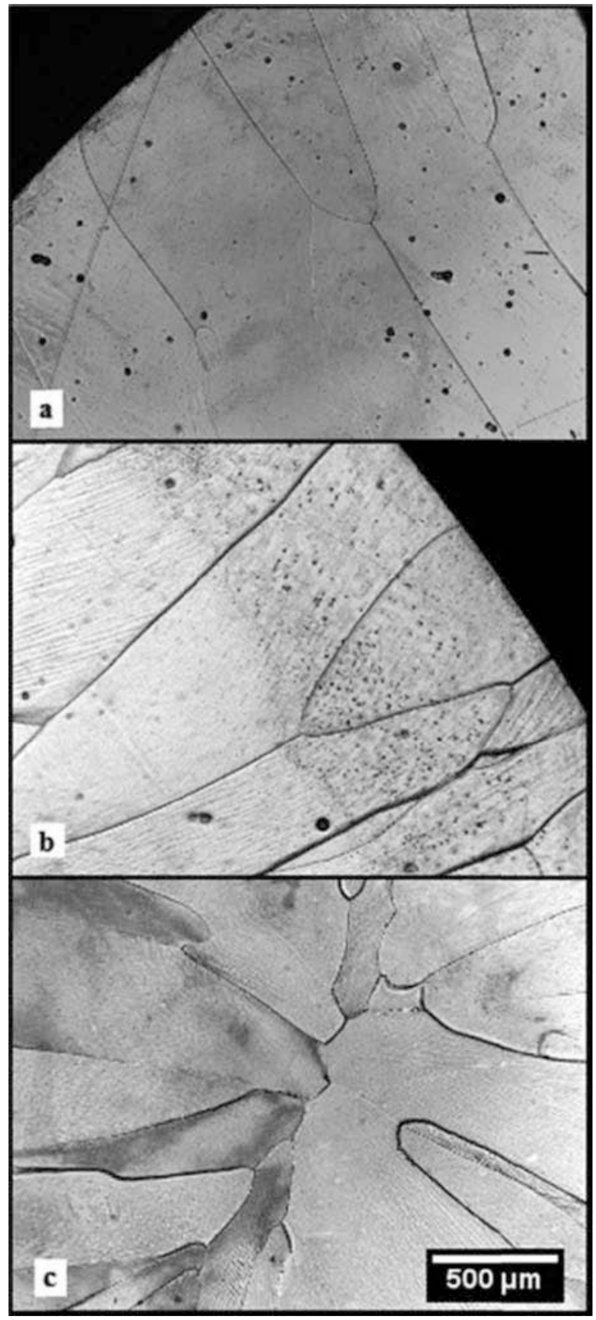

Figure 2: Microstructure of pure Al, 25% CW at heat treatment of (a) As cold-worked edge grains (b) 20 minutes at 325°C, edge grains and (c) 20 minutes at 375°C, center grains. All images are taken at the same magnification.

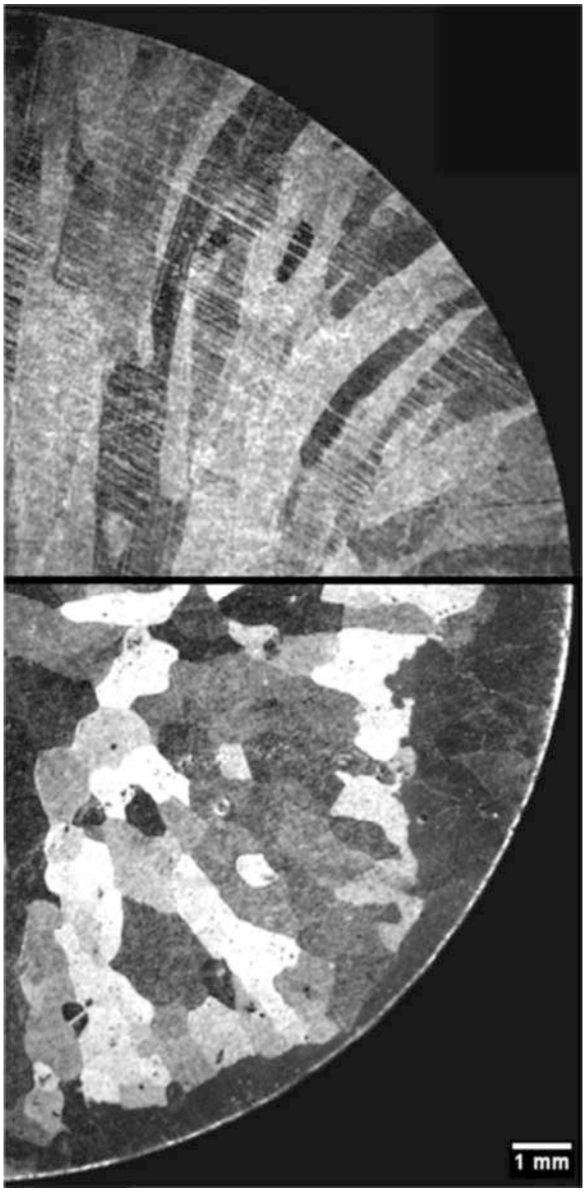

Figure 3: Pure aluminum in as-cold worked state (top) and after 20 minutes at 500°C (bottom) showing retained directionality in the fully recrystallized structure.

Upon addition of scandium and zirconium, the as-cast grain structure is refined, with columnar grain widths on the order of 100µm. Several circular cross sectional grains with diameters on the order of 50µm are also observed, residing between columnar grains. The reduction in columnar grain width for the as-swaged Al0708 sample can be attributed to a slower coarsening rate due to scandium and zirconium in solution. The several minutes that it takes the castings to cool from the melting temperature to room temperature allows the grains in the pure aluminum castings to coarsen significantly, while the Al0708 grains remain relatively slender. It is noted for future work that castings such as these should be quenched as soon as they can be removed from the mold.

Recrystallized grains are identified in Al0708 at all temperatures, primarily on grain boundaries. Therefore, at the observed peak hardness temperature of 325°C, strain-free grains are present and the corresponding nominally low peak hardness is observed. The recrystallized grains do not coarsen significantly throughout the temperature range; however, the number density increases appreciably. No scandium or zirconium trialuminide precipitates are identified through optical examination.

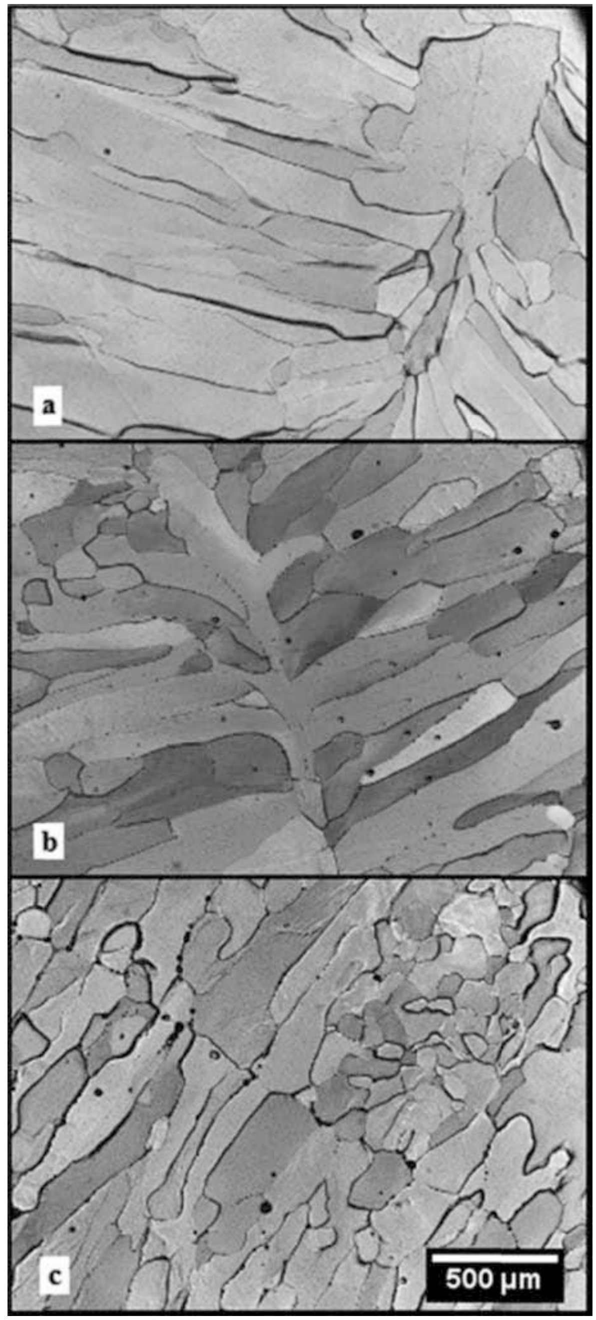

Figure 4: Microstructure of Al0708, 25% CW at heat treatment of (a) As-cold worked (b) 3.0 hours at 325°C and (c) 3.0 hours at 375°C. All images are taken at the center of the samples, at the same magnification.

Conclusions

From the obtained hardness measurements and optical images of cold worked and aged Al-0.07Sc-0.08at% Zr bars, the following observations have been presented:

- Peak hardness in the current system is not significantly dependent upon amount of deformation.
- The as-cast grain structure is refined upon addition of scandium and zirconium to aluminum, due to increased coarsening resistance during solidification/cooling of the castings.
- In the 25% CW Al0708, lower than expected Vickers microhardness measurements are observed due to recrystallization occurring at and below 325°C.
- Recrystallized grains are resistant to coarsening in Al0708, but coarsen significantly in the absence of alloying additions.
- It has been noted for future work that the castings from this and similar molds must be quenched as soon as possible after pouring to minimize grain coarsening and relaxation.

Future work in similar aluminum-scandium-zirconium systems will include the use of SEM imaging in conjunction with atomic force microscopy (AFM) to examine trialuminide precipitate properties and their effect on macro-scale mechanical properties. Also, a study is planned which will investigate the dependence of trialuminide precipitate density on dislocation density following thermomechanical processing.

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