MICROSTRUCTURE EVOLUTION IN AI-Mg ALLOYS DURING AND AFTER HOT DEFORMATION

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Abstract

Controlling the microstructure developed during hot rolling is of great importance to controlling final material properties. Changes in processing parameters and chemical composition alter the recovery-recrystallization-grain-growth processes that control microstructure evolution. To better understand these processes in two Al-Mg alloys, cylindrical specimens were subjected to hot compression at temperatures from 300 to 500°C with a fixed strain rate of 1.0 s⁻¹. Upset specimens were immediately quenched by He gas to preserve their deformed microstructures. Specimen sections were then annealed to separate the dynamic and static components of microstructure evolution. Specimen microstructures were characterized by optical and electron microscopy. Grain size and the degree of recrystallization were measured as functions of specimen chemistry, compression-test conditions and annealing conditions. The experimental results are interpreted to better understand the mechanisms of microstructure evolution and to evaluate new paths to microstructure refinement during hot rolling.

Introduction

Aluminum alloys containing magnesium as the primary alloying element, the Al-Mg or 5000-series alloys, are of special interest for hot forming because they can achieve very high ductilities at elevated temperatures. This characteristic can be taken advantage of to form geometrically complex components that are attractive to the transportation industries for vehicle mass reduction. Microstructures that control the ductility of Al-Mg alloys are initially developed during the hot rolling process. Better understanding of microstructure development during hot rolling can help us better control final sheet microstructure to more easily achieve a fine grain size, for example. Fine-grained Al-Mg alloys can consistently achieve tensile ductilities exceeding 300% at elevated temperatures [1-3]. Such great ductilities are possible at elevated temperatures and slow strain rates because of the grain-boundary-sliding (GBS) creep mechanism. However Al-Mg alloys with coarse grains (> 10µm) are still capable of tensile ductilities exceeding 200% at lower temperatures and faster strain rates [4,5]. Components formed

under such conditions take advantage of the solute-drag (SD) creep mechanism [6,7].

The microstructure developed during hot rolling, or other elevated-temperature deformation processing, is controlled by the processing parameters and chemical composition, often through controlling the rates of recovery, recrystallization and grain growth during and immediately after deformation [8-9]. Recrystallization can increase or decrease the final grain size, depending on the details of its occurrence [10]. Controlling the recrystallization process is important to best control the final material microstructure and to engineer improved materials. This study considers the effects of magnesium content and processing temperature on microstructure evolution in aluminum alloys deformation and subsequent during hot annealing. Microstructures were studied through optical microscopy and scanning electron microscopy.

Experimental

Three aluminum alloys were produced for this study by direct-chill casting of ingots into dimensions of $80 \times 200 \times 400$ mm. These were then scalped to 70 \times 200 \times 400 mm. The compositions of the three alloys are provided in Table 1. The ingots were homogenized at 520°C for 5 hour and then air cooled. After homogenization, mechanical test specimens were machined into cylinders with a height of 12 mm and a diameter of 8 mm. Each test specimen was hot upset to a final height of 6 mm at an engineering strain rate of 1.0 s⁻¹, for a total engineering compressive strain of 50%, at temperatures of 300, 400 or 500°C. Immediately after each compression test, the specimen was quenched in He gas to room temperature. This rapid quenching step was intended to preserve the deformation microstructure for subsequent metallographic examination. The microstructures of these specimens were examined in the as-quenched condition and again after annealing for 10 minutes in a salt bath at the tested temperature.

Table I. Chemical composition (wt. Pct)

Alloy	Cu	Si	Fe	Mn	Mg	Zn	Cr	Ti
Al	0.004	0.09	0.11	0.05	0.0	0.016	0.05	0.01
Al-0.5Mg	0.002	0.1	0.11	0.05	0.5	0.006	0.05	0.01
Al-4.5Mg	0.004	0.11	0.11	0.05	4.39	0.004	0.05	0.0



Figure 1. This schematic shows the orientation of a specimen sectioned and mounted for metallographic observations. The image on the sectioned surface demonstrates the plane observed.

Tested specimens were sectioned for metallographic examination as shown in Figure 1. The sectioned specimens were prepared for metallography through standard grinding and polishing procedures. For optical metallography, specimens were electrolytically etched in Barker's reagent (1.8 vol. pct. HBF4 in H2O) at 25 V for approximately 60 s. Optical microscopy (OM) used polarizing filters to produce color contrast between grains. Grain sizes were measured at the center of each specimen, where deformation was greatest. Grain sizes were measured using the directional lineal-intercept method [11] along the compression axis and radial direction. Additional metallographic observations used scanning electron microscopy (SEM) with electron channeling contrast imaging (ECCI). For SEM-ECCI observations, one selected specimen was prepared by ion milling with Ar after preparation by grinding and polishing.

Results

Figure 2 shows microstructures at the centers of upset specimens for each alloy at 300, 400 and 500°C. The largest grain sizes observed are in the pure Al material, Figure 2a-c. The finer grain sizes in the Al-0.5Mg material, Figure 2d-f, demonstrate the significant effect of just 0.5 wt. pct. Mg. The 4.5 wt. pct. Mg addition in the Al-4.5 Mg leads to the finest grain sizes, Figure 2g-i. Figure 2 demonstrates the effectiveness of Mg for increasing microstructure refinement during hot deformation.

Unique among the deformed microstructures shown in Figure 2 is that shown in Figure 2i for the Al-4.5Mg material deformed at 500°C. This specimen exhibits several small equiaxed grains that suggest partial recrystallization [12,13]. This interesting recrystallization behavior was further investigated using ECCI, which produced the image shown in Figure 3. The very small recrystallized grains evident in Figure 2i appear among the elongated grains shown in Figure 3b. The small recrystallized grains are identified with arrows. Figure 3 also reveals some intermetallic particles, likely associated with the minor alloy additions/impurities Si, Fe and Mn. The intermetallic particles show as light specks in Figure 3.

The recrystallization behavior observed in the Al-4.5Mg alloy reflects the effect of Mg on the kinetics of recovery and recrystallization. At high Mg concentrations, from 0.5 to 4.5 wt. pct, dynamic recovery slows and microstructural refinement is increased. This effect is most pronounced at the highest Mg concentration. The test of Al-4.5Mg material at 500°C is the only one to show any indications of recrystallization nuclei. The suppression of recovery by the high Mg concentration in this

alloy makes it most susceptible to recrystallization. More deformation energy, dislocation density, is retained as recovery is reduced. It is this stored deformation energy that initiates recrystallization. In this case, the recrystallization likely occurred following deformation, i.e., under static conditions. It should be noted that there is some contention in the literature as to whether recrystallization is static or dynamic in Al-Mg alloys, but the later case is far better supported by experimental data [5]. The lack of any recrystallization at lower temperatures is likely a result of both the rapid quenching denying sufficient time for recrystallization and the slowed recrystallization response at lower temperatures. The small equiaxed grains shown in Figure 2i and 3 might form from cross-linked arrays of dislocations between elongated grains [14] and/or from pinched off subgrains [5].



Figure 2. Polarized-light optical photomicrographs are shown from the centers of upset specimens as functions of temperature and composition. Specimens were tested in compression at 1.0 s^{-1} and immediately quenched. The compression axis is vertical and the radius/diameter is horizontal.

Figure 4 shows microstructures of the specimens after annealing for 10 min. at the same temperatures as the compression tests. The photomicrographs in Figure 4 are from approximately the same locations within test specimens as those shown before annealing in Figure 2. The effects of annealing on the specimen microstructures vary with temperature. The specimens tested and then annealed at 300°C evidence recovery and grain growth but do not show evidence of significant recrystallization. At 400 and 500°C all three materials obtain microstructures after annealing that suggest recrystallization. As the annealing temperature increases from 400 to 500°C, the respective grain sizes of the materials increase. The most immediate evidence for recrystallization at these two highest temperatures is an alteration of the grains from shortened along the compression axis and elongated along the diameter of the test specimen, as shown in Figure 2, into approximately equiaxed shapes after annealing. The deformed morphologies are, however, retained after annealing at 300°C.



Figure 3. SEM-ECCI photomicrographs are shown from the center an Al-4.5Mg specimen tested at 500° C and 1.0 s^{-1} . Arrows point out the small equiaxed grains

The largest grains are found in the Al specimens, Figure 4a-c, and the specimens of magnesium containing alloys retain the finer microstructures, Figure 4d-i. The Al-4.5Mg specimen tested and annealed at 300°C produced a particularly interesting microstructure. Its microstructure after annealing, Figure 4g, is somewhat similar to the microstructure after quenching from the compression test at 500°C, Figure 4i. Similarity is found in the very small equiaxed grains that appear. This suggests that recovery was sufficiently suppressed by the Mg content in the Al-4.5Mg alloy for it to retain enough deformation energy to start the recrystallization process after testing and annealing at 300°C. This was not possible in the other alloys that contained less Mg. The question of the recrystallization mechanism, however, remains. While particle stimulated nucleation (PSN) of recrystallization [15,16] is a possibility, the pinching off of subgrains during deformation is a more likely process [5]. It should be noted that the mechanism of nucleation at slip band intersections, as is hypothesized for static recrystallization during annealing following significant cold rolling reduction, is unlikely for this case of hot-deformed materials.

Figure 5 displays lineal intercept grain size measurements made along the radial direction (width) and compression axis (height) in specimens after upsetting (a and b) and after annealing (c and d). Measurements were made at the center of each specimen and are plotted as a function of temperature. The Al material consistently exhibits the largest grain sizes, and the Al-4.5Mg material consistenly exhibits the smallest grain sizes. Grains in the upset specimens are elongated along the radial direction, Figure 5a. Grain sizes in the upset specimens generally increase with temperature, with grain width (Figure 5a) increasing more rapidly with temperature than grain height (Figure 5b). The lone exception is grain width in the Al-4.5Mg material at 500°C, which is slightly decreased from the test at 400°C. This is an artifact in the measurements that results from the small equiaxed recrystallized grains that appear in this specimen only after upsetting. Studies of hot upsetting other Al alloys containing Mg





Figure 4. Polarized-light optical photomicrographs are shown from the centers of upset specimens as functions of temperature and composition. Specimens were tested in compression at 1.0 s^{-1} , immediately quenched and then annealed for 10 min. at the test temperature.



Figure 5. Lineal intercept grain sizes are shown along the radial (width) and compression axis (height) in specimens before (a and b) and after (c and d) annealing. The temperatures shown are those for hot upsetting and annealing.

The specimens upset and then annealed at 300°C exhibit essentially no change in grain width from the annealing step, but annealing slightly increases the grain height. The specimens upset and annealed at 400°C exhibit a significant reduction in grain width from annealing. The grain height in both the Al and Al-0.5Mg increase after annealing at 400°C, while the Al-4.5Mg retains approximately the same small grain height before and after annealing. These effects are a result of recrystallization during the 400°C annealing treatment. Annealing at 500°C produces larger grain sizes than does annealing at 400°C. It is of note that the Al-4.5Mg exhibits only a slight increase in crystallized grain size as testing and annealing temperatures increase from 400 to 500°C, while the Al and Al-0.5Mg materials exhibit much more significant increases in grain size.

Conclusions

The effects of Mg on grain refinement during hot upsetting and on recrystallization during subsequent annealing were investigated. Mg at a concentration of 0.5 wt. pct. reduced recovery and enhanced grain refinement during upsetting at 300, 400 and 500°C. A concentration of 4.5 wt. pct. was even more effective. For the higher Mg concentration, recovery was suppressed sufficiently to induce the nucleation of recrystallization immediately after upsetting at 500°C, despite rapid quenching. The initial stages of recrystallization were evident from small isolated recrystallized grains in the microstructure. Annealing after upsetting produced full recrystallization at 400 500°C, confirming and that recrystallization is a static phenomenon in these alloys, not dynamic. For annealing at 300°C, the Al and Al-0.5Mg alloys gave no indications of recrystallization. However, the Al-4.5Mg material upset then annealed at 300°C contained very small recrystallized grains within a microstructure dominated by recovery. Mg additions to Al suppress recovery during hot deformation and promote static recrystallization during annealing after hot deformation.

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