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AN EBSP INVESTIGATION OF ALTERNATE MICROSTRUCTURES FOR SUPERPLASTICITY IN ALUMINUM-MAGNESIUM ALLOYS

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Introduction

The presence of grain boundaries capable of supporting superplastic flow by grain boundary sliding (GBS), in addition to a fine grain size (~10μm or less) and the presence of a dispersed second phase to inhibit grain growth during elevated temperature deformation, is generally recognized to be a principal prerequisite for fine-grained superplasticity in metals. High-angle, disordered boundaries are thought to be necessary due to the mobility required to support sliding and rotation processes during GBS. The objective of this study is to examine the microtexture and mesotexture associated with grain boundary development during thermomechanical processing (TMP) and post-TMP superplastic deformation of two aluminum-magnesium alloys. Ultimately it is of interest to consider the importance of disordered boundaries as a prerequisite for superplastic flow in these alloys. Interactive electron back-scatter pattern (EBSP) methods will be utilized to obtain grain-by-grain orientation measurements of as-processed, processed and annealed, and superplastically deformed specimens of the two materials. Such measurements, when expressed in the form of microtexture and grain misorientation data, may provide insight into mechanisms of microstructural transformation during processing and the evolution of grain orientation and boundary misorientation during elevated temperature deformation.

This study proposes to provide insight into alternative grain boundary structures in two aluminum-magnesium alloys processed to achieve superplastic behavior. A commercially processed superplastic 5083 aluminum alloy, SKY5083, and a laboratory processed, non-commercial superplastic Al-10Mg-0.1Zr alloy have been selected for examination. Although alloy content, processing routes, and deformation conditions vary for each material, a comparison of results may provide evidence that alternate grain structures and boundary misorientation distributions may support superplasticity in the GBS regime, depending on the TMP processing and alloy system chosen.

Experimental Procedure

Table 1 provides the alloy composition data. The thermomechanical processing schedule for TMP6 Al-10Mg-0.1Zr included unidirectional warm rolling to an equivalent strain of 2.5 in a series of 12 rolling passes at 300°C with a 30 minute interpass anneal at 300°C between successive rolling passes. Further

TABLE 1
Composition of Alloys Investigated in This Study

Alloy	Mg	Zr	Mn	Cr	Fe	Si	Al
Al-10Mg-.1Zr	9.89	0.09	-	-	0.02	0.02	bal.
SKY 5083	4.48	-	0.65	0.11	0.07	0.05	bal.

details have been presented previously [1]. Processing of the SKY5083 material is proprietary to the manufacturer. Tensile coupons were extracted from the as-processed sheet materials and were deformed in uniaxial tension at constant strain rate with the tensile direction aligned in the rolling direction. Specimens chosen for examination in this study were those which exhibited the highest elongations. Mechanical test data indicated that both specimens chosen were deforming in the GBS regime with the strain rate sensitivity exponent, $m \geq 0.4$.

Table 2 provides a summary of the test data for the two alloys examined. Fractured tensile specimens were sectioned longitudinally along the tensile axis to reveal the through-thickness direction. Mechanical and electro-polishing in preparation for BSE/EBSP and details of the interactive EBSP methodology utilized here has been presented recently [2]. Grain size (MLI) determination was conducted using standard back-scatter electron (BSE) microscopy involving low accelerating voltage (~5kV) in a TOPCON SM-510 SEM to reveal grain contrast. EBSP examination was conducted using the spot mode of the SEM to capture of successive diffraction patterns at a magnification of 1000× and at a working voltage of 20kV. Pattern and data analysis of neighbor orientations to obtain boundary information was obtained utilizing software from TexSEM Laboratories, Inc. running on a Silicon Graphics INDY workstation. Nominally, 500 orientations were captured in each data set to yield microtexture information and analyzed to provide boundary information.

Results and Discussion

Table 2 provides the results of mechanical testing and grain size determination for the two materials. BSE micrographs illustrating the microstructures present in the processed and annealed materials are presented in Figure 1(a) and (b). The microstructure present in the processed and annealed SKY 5083 specimen is an equiaxed, refined grain structure with a low volume fraction of uniformly dispersed Al_6Mn particles. The TMP6 Al-10Mg-0.1Zr processed and annealed microstructure appears as a highly refined microstructure with a larger volume fraction (10 vol.%) of the β phase (Al_8Mg_5) existing as coarse (up to 1.0μ), spheroidally-shaped particles preferentially located near triple junctions. During EBSP examination it was noted that the back-scatter diffraction patterns in the SKY 5083 specimens

TABLE 2
Mechanical Testing and Grain Size Data

Specimen	T_{def}	Strain Rate	Peak Elongation	σ_{flow} at ϵ=0.1	L_{av} (as-pro)	L_{av} (ann)
TMP6 Al-10Mg-0.1Zr	300°C	2.0×10^{-3} s^{-1}	1100%	20.0 MPa	2.3μm	2.4μm
SKY 5083	535°C	1.0×10^{-4} s^{-1}	377%	1.18 MPa	9.0μm	12.3μm

(a) (b)

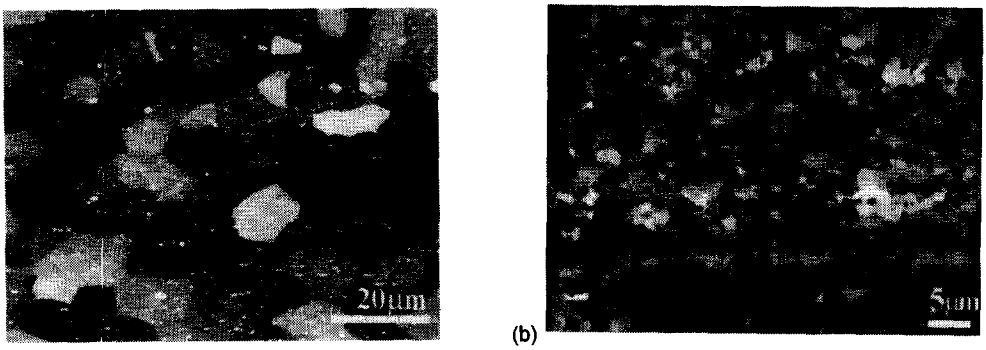

Figure 1. BSE micrographs of processed and annealed microstructures present in (a) SKY 5083 and (b) TMP6 Al-10Mg-0.1Zr. The rolling direction is horizontal. Orientation contrast, no etchant.

were sharper and less difuse than patterns collected in the Al-10Mg-0.1Zr specimens indicative of low residual strain within individual grains and similar to patterns observed in newly recrystallized grains. Grain growth during annealing was more evident in the SKY 5083 material. Evidence of cavitation during the (higher) temperature deformation of SKY 5083 was also observed.

Microtextural information obtained through the collection of succesive orientations in the SKY 5083 specimens is presented in Figure 2. In the as-received condition the SKY 5083 specimen possessed a weak texture comprised of a combination of deformation and recrystallization components. The dominant recrystallization texture is the {011} <100> Goss orientation while most deformation orientations were in the vicintity of the brass, {011} <211>, and S, {123} <634>, components. While TMP has resulted in grain refinement ($La_{s\text{-}rec}$ = 9.0μ), examination of the microtextural results for the processed and annealed material indicated a distinct change toward a moderate cube {100} <001> texture suggesting that grain structure evolution, presumably through static recrystallization during TMP, was incomplete in the as-received material. Examination of microtextural data (not presented here) for specimens which were annealed only briefly (~15 min.) at the same temperature (535°C) indicated that this shift to a cube texture occurs rapidly, suggesting that the TMP scheme involved deformation at a moderate or low temperature. The appearance of a sharp cube texture during annealing of commercial purity aluminum has been examined elsewhere using EBSP techniques and associated with the formation of a nearly random distribution of high angle boundaries during recrystallization [3].

Figure 3 shows corresponding microtextural results for the TMP Al-10Mg-0.1Zr alloy. The as-processed texture resulting from the warm rolling schedule at a moderate (300°C) temperature is weak and dominated by deformation components primarily near the copper orientation, {112} <111>, with some brass, {011} <211>, also evident. During annealing at the test temperature, 300°C, the texture sharpens moderately but is still dominated by these same deformation components. No evidence of the development of cube or other recrystallization texture components is observed. In the deformed region of the tensile specimen, microtextural data illustrated a weakening and randomizing of texture characteristic of deformation by GBS. BSE micrographs of deformed gage specimens of both alloys showed the retention of an equiaxed grain morphology, also indicative of deformation by GBS mechanisms.

Histograms describing grain boundary misorientation distributions present in all specimens examined are illustrated in Figure 4. For the SKY 5083 material the as-received, processed and annealed, and deformed gage sections all exhibited essentially a random misorientation distribution. In contrast, the grain misorientation distribution histograms for the Al-10Mg-0.1Zr alloy the as-processed and processed and annealed data plots show a large fraction of moderately misoriented boundaries.

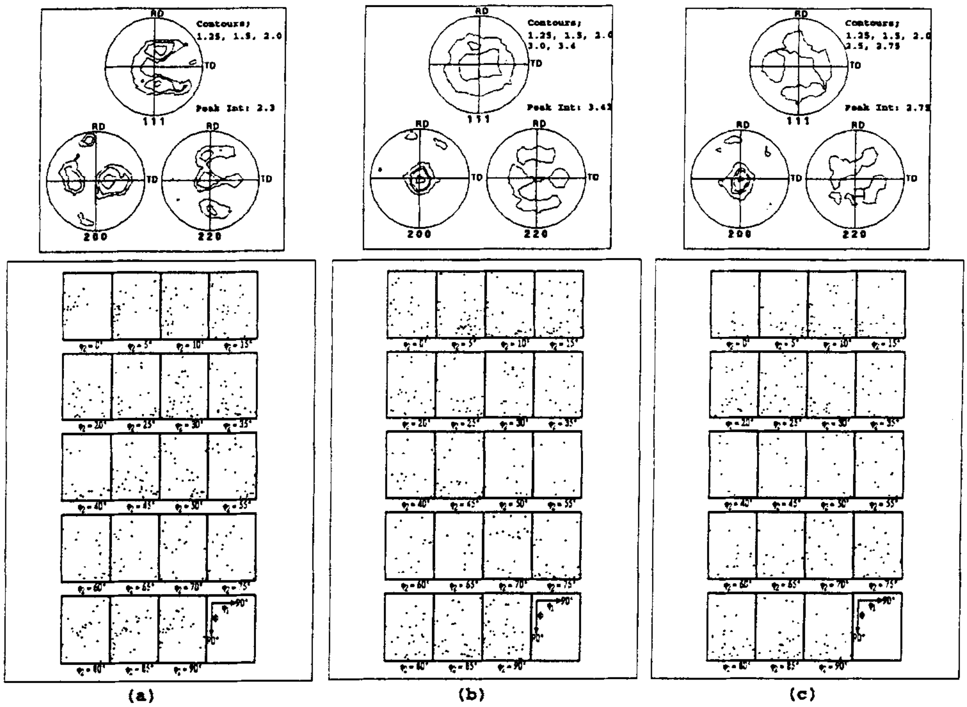

Figure 2. Microtextural plots for (a) as-received, (b) processed and annealed and (c) deformed specimens of SKY 5083.

The distribution of boundary misorientation is distinctly different from random and, essentially, is a bimodal distribution in the processed and annealed state. The low-angle boundaries present here are complex in nature with an average misorientation of 10.6°. In the deformed gage region, a decline in the fraction of boundaries of the lowest misorientations may be attributed to coalescence occurring during GBS and rotation. A recent study by the authors [4] describing the microstructural evolution of the TMP6 processed alloy attributed the development of moderately misoriented boundaries to extended recovery processes during TMP. There is no evidence of any such process in the SKY 5083 material.

Table 3 presents the fraction of boundaries in the as-processed materials, catagorized by interfacial energy based on boundary character following Watanabe and others [6-7]. Here, low-angle boundaries ($\theta < 15°$) and high-angle boundaries satisfying criteria for nearness to coincident site lattice (CSL) relations up to $\Sigma 29$ are assumed to have lower interfacial energy than random, disordered high-angle boundaries. It is recognized that, in the case of the CSL boundaries, this is a catagorization of convenience as grain boundary energy may not follow in such a simple manner [8]. Additionally, the correlation of interfacial energy with rates of boundary migration and GBS is not conclusive and has been shown to be sensitive to impurity content and specific boundary orientation [8-11].

The summary of data presented in Table 3 shows that, for the specimens examined, SKY 5083 material possessed grain boundaries of higher interfacial energy with over 90% of all boundaries examined being random, disordered boundaries. Such distributions would fit with the conventional thesis that random misorientation distributions and disordered boundaries facilitate the GBS process. Alternately, TMP6 processed Al-10Mg-0.1Zr boundaries were of different character with nearly 1/3 of all boundaries either low-angle or satisfying nearness criteria to a CSL relation. The Brandon criteria ($\Delta\theta = 15/\sqrt{\Sigma}$) for maximum deviation from CSL coincidence was utilized. Random distributions of the

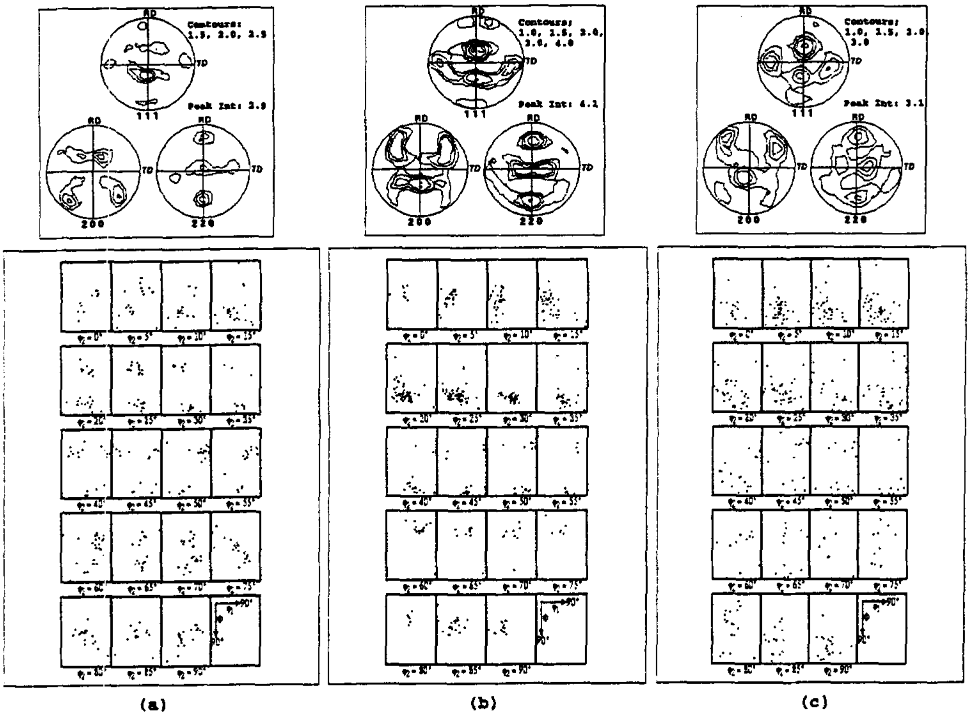

Figure 3. Microtextural plots for (a) as-processed, (b) processed and annealed, and (c) deformed specimens of TMP 6 Al-10Mg-0.1Zr.

high-angle, disordered boundaries present in the SKY 5083 processed material would be expected to result in faster grain boundary migration and grain growth rates than those of the Al-10Mg-0.1Zr given that the fraction of second phase particles to restrict boundary motion is lower in the SKY 5083 alloy and the homologous deformation temperature is higher. Additionally, evidence of faster grain growth rates for grains of cube orientations in recrystallized aluminum has been presented [3] recently. This growth may compete with presumably faster GBS rates for disordered boundaries in the superplastic response of SKY5083. Lower energy boundaries, such as those present in the TMP6 Al-10Mg-0.1Zr material, have been shown to exhibit enhanced ductility attributed to greater resistance to intergrannular cracking [6].

In this brief study it is not possible to ascertain or describe the effect of grain boundary energy on superplastic deformation in these alloys. Rather, it is to present two distinctly different distributions of grain boundary misorientation which result from TMP and annealing, both supporting superplastic deformation in the GBS regime. Ongoing research to investigate the evolution of grain boundary character in dynamically recrystallizing aluminum alloys may contribute further to the understanding of the influence of disordered boundaries as a prerequisite for superplastic flow by GBS.

Conclusion

Evidence has been presented that suggests that alternate distributions of (grain) boundary structure may support superplastic flow in the GBS regime in aluminum-magnesium alloys. This suggests that

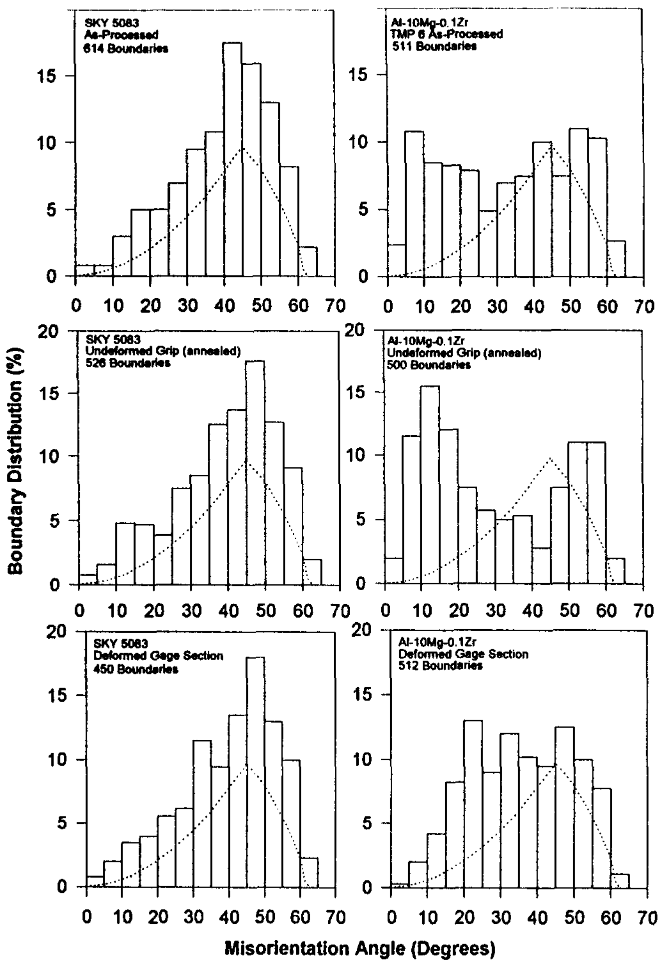

Figure 4. Grain misorientation histograms for specimens examined. The dotted line indicates the *continuous* plot of the probability distribution of misorientations for randomly oriented cubes predicted by Mackenzie [5].

the GBS mechanism(s) may not require random distributions of boundary misorientation or a predominance of disordered, high-angle boundaries.

TABLE 3

Distribution of Boundaries by Energy Character (% Predicted for a Random Polycrystal Shown in Parenthesis)

Specimen	Boundaries <15° (2.3%)	CSL Boundaries >15° [$3 \leq \Sigma \leq 29$] (11.3%)	Random Boundaries >15° (86.4%)
SKY 5083 (as-processed)	4.6 %	4.8 %	90.6 %
TMP6 Al-10Mg-0.1Zr	21.7 %	9.0 %	69.3 %

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