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Dynamic anisotropic grain growth during superplasticity in Al-Mg-Mn alloy



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ABSTRACT

The microstructural mechanisms of dynamic anisotropic grain growth during superplasticity in a quasi-single phase Al–Mg–Mn alloy were characterized. The tensile superplasticity with 320% elongation was mediated by grain boundary sliding accompanied by rigid grain rotation with a limited crystallographic slip. The deformed sample exhibited a bimodal microstructure. Some grains maintained their original size and equiaxed morphology during superplasticity, whereas the others became elongated more than twice in aspect ratio and were composed of equiaxed subgrains that were aligned in the tensile axis. These microstructural features were possibly attributed to a rotation-coupled grain coalescence accompanied by grain boundary sliding.

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Dynamic grain growth during superplasticity is anisotropic in (quasi-) single-phase materials; this unique characteristic was first reported by several studies in the 1990s [1–3] but is attracting only a limited attention today although its mechanism is still unclear. In this study, we characterize microstructural development during superplasticity in a quasi-single phase Al–Mg–Mn alloy using today's advanced microscopy and propose a new phenomenological mechanism for the dynamic anisotropic grain growth. This grain growth is possibly attributed to rotation-coupled grain coalescence accompanied by grain boundary sliding (GBS).

Superplasticity is the ability of a fine-grained polycrystalline solid to exhibit a huge ductility over hundreds of percent of elongation at high temperature and/or low strain rate. This excellent ductility is owing to GBS, the relative translation of neighboring grains with respect to one another along their boundary [4]. Superplasticity involves dynamic grain growth which occurs more rapidly than under static annealing at the same temperature without deformation. Dynamic grain growth plays a critical role in the macroscopic behavior of superplasticity; it results in strain hardening to stabilize neck-free plasticity and also determines the limit of ductility, both of which are owing to grain coarsening [5].

Although dynamic grain growth has been recognized for a long time in many superplastic alloys [1–8], ceramics [9,10], and minerals [11], its microscopic mechanism is still controversial. According to previous theories, the possible mechanisms should be based on (i) the grain boundary migration that is accelerated by superplasticity [1,3–6] and/ or (ii) the rotation and coalescence of grains [2,6]. Thus far, however, the primary process contributing to the dynamic grain growth has not been successfully determined from the analyses of grain size vs. time or strain. Therefore, the mechanisms should be determined from the microstructural features, which provide stronger evidence of their elucidation.

Another important feature is the anisotropic behavior of dynamic grain growth in (quasi-) single-phase materials including aluminum. [1-3,12-15], magnesium [16,17], and nickel alloys [18], in which grains tend to become elongated in a tensile direction during superplasticity, whereas dynamic grain growth occurs in an isotropic manner in dualphase materials [4–8]. This dynamic anisotropic grain growth was first reported in aluminum alloys in the 1990s [1-3]. Rabinovich and Trifonov [1] and Shin et al. [3] attributed this grain elongation to an anisotropic grain boundary migration, whereas Li et al. [2] considered a combination of dislocation creep and rotation-coupled grain coalescence to be the mechanism for grain elongation. However, only limited attention has been given to this feature since the 2000s despite its availability in many types of superplastic materials today [12–18]. This study aims to reinvestigate the mechanism of dynamic anisotropic grain growth using advanced microscopy available today and to obtain new insight into the nature of this anisotropy which is unique to (quasi-) single-phase materials.

In these experiments, a commercial Al–Mg–Mn alloy (ALNOVI–U, UACJ) with a chemical composition of Al–4.94 Mg–1.53 Mn–0.01 Fe–0.03 Si (mass%) was used. The as-received sheet had a thickness of 1.2 mm and was cut into tensile specimens along the rolling direction (RD) in two different gauge dimensions via electric discharging:



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(a) 10 mm in length and 6 mm in width and (b) 5 mm in length and 3 mm in width. All specimens were annealed at 500 °C for 0.5 h, and they then had an average grain size of 4.2 µm in the normal direction (ND) and 7.0 µm in the RD as measured by the linear intercept method using electron backscattering diffraction (EBSD). They contained secondary-phase particles (Al₆Mn) at 3.6 vol% as measured from backscattered electron images using Image J software. The as-annealed samples were deformed in tension to various true strains (0.25, 0.50, 0.75, 1.00, and fracture) at 450 °C and at an initial strain rate of 1×10^{-3} s⁻ ¹; this condition is present in superplastic region II [19]. For specimen (a), the side surface of the gauge section was cut and polished after deformation to view the internal microstructure in the transversedirection (TD) planes. The sample surfaces were finished by electropolishing in a solution of 70% ethanol +20% perchloric acid +10% glycerol at -25 °C and 20 V for 60 s for EBSD characterization. For specimen (b), 20×20 microgrids with intervals of 5 µm were processed on a polished ND surface using focused ion beam (FIB) prior to deformation and were characterized after they were strained to 0.25 via scanning electron microscopy (SEM).

Fig. 1a displays the true stress–true strain curve at 450 °C and 1 $\times 10^{-3}$ s⁻¹ with a fracture elongation of 320%. This flow curve is accompanied by distinct strain hardening that was possibly due to grain growth and a resultant increase in the resistance to GBS, which is typical of superplastic materials [5] but not to an increase in dislocation density as seen in ordinary plasticity. Fig. 1b shows the grain growth behaviors in the RD (tensile direction) and the ND during deformation and annealing at the same temperature. The grain growth during deformation was strongly anisotropic and dynamic; the growth rate was more rapid in the RD than in the ND and almost negligible during static annealing.

Fig. 2 shows SEM images of the ND surface covered with microgrids after deformation to a true strain of 0.25. The local elongation was 24%, and the major part of the plastic strain was mediated by GBS but with a limited intragranular deformation due to a crystallographic slip; for example, the dotted line in Fig. 2b was originally straight but slid at grain boundaries C/E or E/I, whereas the microgrids inside grains remained almost square during deformation. GBS was apparently directed in the resolved shear stress (i.e. almost 45° to the tensile axis) and accommodated by a rigid grain rotation (e.g. compare grains C and E) and possibly by atomic diffusion resulting in local volumetric changes near the grain boundaries (e.g. new matter was deposited at grain boundary A/E in a tensile state, whereas the matter diffused out of grain boundary E/F in a compressive state, as observed more precisely by Rust and Todd [20] previously). These surface micrographs show the typical phenomena of superplasticity [20,21], although "floating

grains," which slid and floated out of the free surface in tensile superplasticity, could prevent a more quantitative study [22].

Fig. 3 shows the evolution of the internal microstructure, inverse pole figure (IPF) maps (a, c) and 2D histograms of the minor grain size, d_m , and aspect ratio, a, measured by the elliptic approximation of each grain with its boundaries defined as pixel borders larger than 2° (b, e), all reconstructed from the EBSD characterization in the TD plane before and after deformation to fracture. The grain size and morphology were rather homogeneous and equiaxed before deformation; the peak in the histogram is positioned at $d_m = 5.6 \,\mu\text{m}$ and a = 1.5(white triangle, Fig. 3b). However, the grain size and morphology became bimodal after deformation; the original peak shows only a slight translation from the initial state (gray triangle, Fig. 3d) whereas a distinct second peak appeared at $d_m = 7.2 \,\mu\text{m}$ and a = 2.8 (black triangle, Fig. 3d). These bimodal peaks imply that some grains became elongated via anisotropic growth to generate the second peak, whereas the others almost maintained their original size and equiaxed morphology during deformation. The aspect ratio of the second peak almost doubled from the first one (Fig. 3d) and these elongated grains have a substructure composed of rather equiaxed subgrains that connects with each other along the tensile axis as shown by circles in the IPF map (Fig. 3c). However, the grain elongation and the substructural formation may not be due to a crystallographic slip, as confirmed from the surface SEM images with a limited intragranular deformation (Fig. 2).

According to these observations, the dynamic grain growth in this material was bimodal and anisotropic. Some grains maintained their original size and equiaxed morphology, whereas other grains became elongated in the tensile axis with only a small growth in the tensile transverse. This grain elongation may not be due to a crystallographic slip but may involve GBS, rigid grain rotation, and the equiaxed subgrains that are connected together with along the tensile axis.

Fig. 4 shows a series of schematics illustrating the new possible mechanism for dynamic anisotropic grain growth that does not contradict any microstructural feature observed in this study. First, a pair of neighboring grains is rearranged along the tensile axis by GBS as the primary straining mechanism during superplasticity (Fig. 4a and b); this rearrangement occurs for almost any couple of neighboring grains. Second, GBS is accompanied by rigid grain rotation (Fig. 4b). Finally, if this rotation results in a coincidence of crystal orientations with a certain probability, the couple of grains coalesce with each other along the tensile axis (Fig. 4c). This rotation-coupled grain coalescence can explain the anisotropic grain growth, which involves GBS, rigid grain rotation, and the equiaxed subgrains that are connected along the tensile axis without any contribution from crystallographic slip. The second peak position in the histogram (Fig. 3d) in which the aspect ratio is almost



Fig. 1. (a) True stress-true strain curve at 450 °C and 1×10^{-3} s⁻¹ (fracture elongation was 320%); (b) average grain size in the RD (circles) and the ND (squares) vs. time during deformation at 450 °C and 1×10^{-3} s⁻¹ (solid lines) and upon annealing (dotted lines).

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