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Influence of grain size on strain rate sensitivity in rolled Mg–3Al–3Sn alloy at room temperature

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The strain rate sensitivity (SRS) of a rolled Mg–3Al–3Sn (AT33) alloy increased with decreasing grain size at room temperature, owing to a change in the deformation mechanism from dislocation-dominated to twinning-mediated. Moreover, as deformation proceeds the SRS for slip-dominated grains (~6 μm) decreases gradually due to continuous activation of ⟨c + a⟩ slip, while that for twinning-mediated grains (~41 μm) decreases rapidly because of the combined action of ⟨c + a⟩ slip, contraction twinning ($\{10\bar{1}1\}\langle10\bar{1}2\rangle$) and extension twinning ($\{10\bar{1}2\}\langle10\bar{1}1\rangle$).

Keywords: Strain rate sensitivity; Grain size; Twinning; Work hardening; Magnesium alloys

Owing to the only two independent slip systems provided by basal slip traditional magnesium sheet alloys exhibit poor formability at room temperature [1–3]. To improve the plasticity of Mg alloys, therefore, it is necessary to investigate the deformation mechanisms, to which the mechanical properties of Mg alloys are closely related [1–5]. It has been reported that the strain rate sensitivity (SRS), frequently used to describe the variation inflow stress corresponding to increasing strain rates, defined as $d\ln\sigma/d\ln\dot{\varepsilon}$ where σ and $\dot{\varepsilon}$ are the flow stress and strain rate, respectively, can be used to explore deformation mechanisms in Mg alloys [2]. Moreover, the responses of the microstructure and stress–strain to changes in strain rate are of significance in understanding dynamic deformation [6].

In addition to the strain rate, other parameters, e.g. the deformation temperature, texture and grain size, also clearly exert an effect on SRS in Mg alloys. Generally, increasing the deformation temperature produces an increase in SRS [2–4]. The texture also plays an important role in SRS by determining crystal orientations and the types of deformation modes which experience preferential activation [6–8]. However, the effect of grain size on SRS seems more complicated because of the variation in deformation mechanism at different grain sizes. For ultrafine grained (submicron) Mg alloys the SRS values are usually very high (about 0.5), because of grain boundary sliding at moderate temperatures [9,10]. For micro-grained Mg alloys, however, dislocation slip and deformation twinning are two major plastic deformation modes during room temperature deformation [11,12]. Specifically, for the traditional Mg–3Al–1Zn (AZ31) alloys the effect of grain size on SRS was mainly explained here to fore by: (i) a transition from slip to grain boundary sliding with decreasing grain size [4]; (ii) evolution of the material to an orientation favoring slip caused by twinning, which in itself is considered to have negligible rate sensitivity [3]; (iii) more non-basal slip near grain boundaries produced by grain refinement [13].

However, tension perpendicular to the *c*-axis in Mg alloys could only be accommodated by pyramidal ⟨c + a⟩ slip and deformation twinning [14,15], and slip and twinning responses to changes in strain rates and grain sizes are significantly different [16,17], owing to differences in the critical resolved shear stress (CRSS) [18]. In a previous study [19] it was found that deformation mechanisms for rolled AT33 alloys with a strong (0001) basal texture were greatly dependent on grain size at room temperature. For a grain size of ~6 μm the deformation in tension was mainly dominated by

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dislocation slip, while for a grain size of ~41 μm, however, it was mediated by deformation twinning. Moreover, the activation of non-basal slip was mainly attributed to a decrease in stacking fault energy (SFE) of the ($\{10\bar{1}1\}\langle 11\bar{2}0\rangle$) and ($\{11\bar{2}2\}\langle 11\bar{2}3\rangle$) slip systems caused by simultaneous Al and Sn doping [20]. Thus deformation twinning itself should play an important role in SRS during room temperature deformation, especially in large sized grains.

In this study we aim to examine the influence of grain size on SRS of an AT33 alloy at room temperature, with particular attention on the role of twinning in SRS. In addition, the dependence of strain rate on work hardening for different grain sizes will also be discussed.

Based on a chemical composition of Mg–3Al–3Sn (wt.%), commercially pure Mg (99.85%), Al (99.90%) and Sn (99.90%) were selected to prepare the alloys. Alloying components were first melted at about 730 °C, and then directly cast-rolled as thin slabs with a thickness of ~4 mm. After homogenization at 400 °C for 2.5 h the slabs were hot rolled to 0.66 mm (reduction ~84%) after five passes with reduction rations of ~26%, ~35%, ~35%, ~30% and ~23%, respectively. Heat preservation was prepared at 350 °C for 1 h after every pass. Specimens with different grain sizes of ~6 and ~41 μm were obtained by altering the annealing temperature and time, i.e. 290 °C for 1 h and 450 °C for 40 min, respectively. The detailed preparation process has been reported elsewhere [19]. Tensile samples with a gage size of $30 \times 10 \times 0.66\ mm^3$ were tested along the rolling direction (RD) in an MTS 810 testing machine at strain rates of 10^{-4}, 10^{-3} [19], 10^{-2} and $10^{-1}\ s^{-1}$, respectively, at room temperature. Repeated tensile tests were conducted at least three times, giving results with good repeatability. X-ray diffraction (XRD) (2500PC, Rigaku, Japan) was employed to examine the initial textures of the two grained samples. To study the microstructural evolution tensile tests were also terminated at various flow stresses ($\sigma_{0.01}$ and $\sigma_{0.10}$ at true strains of $\varepsilon = 0.01$and 0.10, respectively). A simple point counting technique (ATSM E562-02) was used to calculate the volume fractions of twins, as suggested by Jiang et al. [15]. Five micrographs of each sample were taken at a magnification of 200×. Next, a 21 × 28 point grid was superimposed on the images. The volume fraction of twins was determined by calculating the ratio of the number of points located within such twins to the total number of points.

After tension deformation surface microstructures were observed using an optical microscope (OM) (Carl Zeiss Axio Imager A2m, Germany). The observed samples were first ground with 2000 grit SiC paper, followed by buffing with 0.5 μm diamond paste, and then chemically etched in acetic picral solution (5 g picric acid, 5 ml acetic acid, 10 ml distilled water, 80 ml ethanol) for about 30 s.

The initial optical microstructures of rolled AT33 alloys with two typical grain sizes (~6 and ~41 μm) are presented in Figure 1a and b. Both samples present relatively homogeneous grain size distributions (see insets in Fig. 1a and b). From pole figure analyses of the (0002) and ($\{10\bar{1}0\}$) planes (Fig. 1c and d) it can be seen that the initial textures of the two grained samples

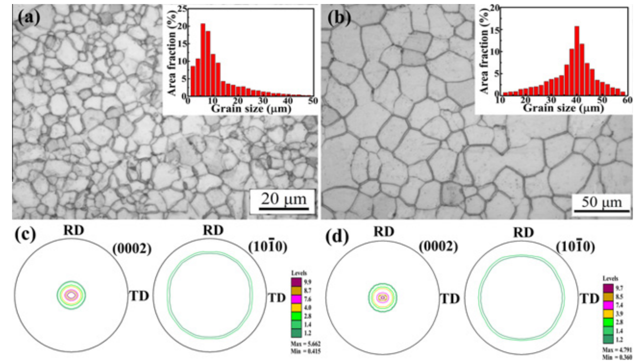

Fig. 1. Optical microstructures of rolled AT33 alloy for grain sizes of (a) ~6 and (b) ~41 μm (upper right insets show grain size distribution), as well as the (0002) and ($10\bar{1}0$) pole figures for grain sizes of (c) ~6 and (d) ~41 μm, respectively.

were very similar, with most of basal planes parallel to the rolling plane. In addition, average 0.2% offset yield strength ($\sigma_{0.2}$), tensile strength (σ_b) and elongation to failure (δ_f) values obtained from engineering strain–stress tests at different strain rates are presented in Table 1. As the strain rate increased from 10^{-4} to $10^{-1}\ s^{-1}$ δ_f decreased from 27.0% to 21.0% for small sized grains (~6 μm), and 16.4% to 11.2% for large sized grains (~44 μm). However, $\sigma_{0.2}$ (σ_b) increased from 195 (265) to 220 (283) MPa for small sized grains, and from127 (223) to 157 (240) MPa for large sized grains. The preliminary result, therefore, is that SRS exhibits a positive value.

The variation in SRS with increasing strain obtained from true strain–stress curves (Fig. 2a) are plotted in Figure 2b. The flow stress at given strains was replotted against strain rate (e.g. 10^{-4}, 10^{-3}, 10^{-2} and $10^{-1}\ s^{-1}$), and then the SRS value at given strains was determined from the slope ($m = \Delta \ln \sigma / \Delta \ln \dot{\varepsilon}$) of linear fits. As deformation proceeds the SRS value decreases slightly for small sized grains, but decreases rapidly for large sized grains. Considering different deformation mechanisms for the two grained samples it is rational to presume that twinning plays an important role in SRS.

It is agreed that a close correlation exists between work hardening and SRS [4]. The work hardening rate ($\Theta = d\sigma_{true}/d\varepsilon_{true}$) is here investigated as $\Theta(\sigma - \sigma_{0.2})$ vs. ($\sigma - \sigma_{0.2}$), as shown in Figure 2c. It is worth noting that the upward curvature of $\sigma - \Theta$ plots can be fitted by homogeneous straight lines and shows linear hardening from the outset, which is mainly due to a forest hardening mechanism [21,22]. In addition, with increasing strain rate the upward curvature of $\sigma - \Theta$ plots decreases for large grained samples but remains approximately constant for small grained samples. Moreover, the work hardening exponent (n) obtained by power law regression of true strain–stress curves (Fig. 2a) decreases with increasing strain rate for large sized grains, but remains relatively constant for small sized grains, as shown in Figure 2d. In other words, work hardening could also be affected by twinning.

To discern changes in twinning during deformation we investigated the microstructural evolution of samples with two typical grain sizes at various tensile stresses