Influence of the grain size on the strain rate sensitivity in a Mg-Al-Zn alloy at moderate temperatures

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In a previous work [1], it was found that a decrease in grain size below approximately 15 μm produces a decrease in work hardening (WH) rate at room temperature in the AZ31 and AM60 magnesium alloys. Conversely, current WH models predict an increase in WH for decreasing grain sizes due to an extra-storage of dislocations. Therefore, the effect of grain size on the deformation mechanism is not well understood.

A plausible explanation of the decrease in WH with decreasing grain size was given on the basis of a grain boundary sliding (GBS) contribution, according to determinations performed by Koike et al.[2]. Moreover, it is well known that the GBS mechanism is associated to high strain rate sensitivity values. It is expected, therefore, that the analysis of the deformation kinetics allow additional information on GBS as a possible mechanism to operate in the present case.

A useful tool for analyzing thermally activated processes is the strain rate sensitivity (SRS) parameter, defined as $\partial\sigma/\partial\ln\dot{\epsilon}$, which is usually measured by strain rate change tests. A representation of SRS as a function of the flow stress, often called

“Haasen Plot”, gives information about the dependence of the apparent activation areas on the microstructural parameters.

In the following, the data of the grain size dependence of the SRS in rolled AZ31 alloy with different grain sizes are presented. The AZ31 alloy (Mg-3wt%Al-0.75wt%Zn) was received from Magnesium Elektron.

In a previous study [1], complete information about thermomechanical processing used on the preparation of the AZ31 samples, metallographic preparation, texture, etc., was given. The alloy was processed via hot rolling described in Ref [1]. The final grain size was close to 2 μ m. Subsequently, annealing treatments were performed to produce the samples with coarse grain sizes, from 2.6 to 55 μ m studied here. After the hot rolling process the microstructure was almost completely recrystallized and it is assumed that the successive annealing treatments, performed to produce the grain size growth, contributes to anneal even more the dislocations inside grains. The alloy after the hot rolling presents a basal texture characteristic of rolled magnesium alloys. In previous works we have found that the textures produced by rolling or ECAP [1] remains very stable under the used thermal treatments.

Concerning the SRS measurements, dog-bone tensile samples of 15 mm gage length and a radius of 3 mm were machined out of the processed AZ31 alloy with the tensile axis parallel to the rolling direction. Tensile tests were performed in a screw driven Servosis testing machine in the range of temperatures 300 to 400K at constant true strain rates. The strain rate sensitivity was measured using strain-rate-change tests with jumps from 10^{-4} s^{-1} to 10^{-3} s^{-1} . During the test, data of load-displacement-time were stored in the computer memory at frequencies appropriate to the strain rate range. The change in flow stress, $\Delta\sigma$, was determined by the method shown in Ref. [3]. The strain rate before and after the change was obtained from true plastic strain-time plots.

Fig. 1(a) shows the SRS as a function of the flow stress for the AZ31 alloy with grain sizes ranging from 2.6 to 55 μm at 300K. Fig. 1(b) shows the SRS as a function of the flow stress at 300, 325 and 350 K, for the grain sizes of 2.6 and 55 μm .

It is interesting to compare the grain size dependence of both the SRS and the WH. The WH behavior of the AZ31 alloy with different grain sizes is shown in Fig. 2 by means of representations $\theta(\sigma - \sigma_{0.2})$ vs. $(\sigma - \sigma_{0.2})$, where $\theta = d\sigma/d\epsilon$ and $\sigma_{0.2}$ is the stress measured at the 0.2% proof strain.

Fig. 1(a) shows that a strong increase in the sensitivity occurs with decreasing grain size. It can be seen, that the data can be well fitted by straight lines of increasing slopes. From these linear fits, a back extrapolation to the yield stress, $\sigma_{0.2}$, can be performed, obtaining $\Delta\sigma_{0.2} / \Delta \ln \dot{\epsilon}$. Fig 2(a) shows near parabolic curves at 300K for all grain sizes. It can be seen that the first part of the curves at low stresses can be fitted by straight lines passing through the origin with a slope θ_0 that decreases with decreasing grain size. The value of θ_0 is commonly related to the athermal hardening in most of the studies on fcc metals [4, 5]. Comparison of Fig 2(a) with Fig 1(a) shows a correlation between decreasing θ_0 values and increasing SRS slopes with decreasing grain size.

It is emphasized that the grain size decrease produces effects that are very different to those generally attributed to an increase of dynamic recovery: on one hand the SRS maintains the linear dependence against the stress, Fig. 1(a), on the other hand the grain size affects the extrapolation θ_0 , Fig 2(a).

With regard to the temperature influence on SRS and WH, Fig 1(b) shows that the slope increases with increasing temperature by a factor of about three, for $d = 2.6 \mu\text{m}$. Moreover, an increase in SRS by a factor of about two at the yield stress, $\sigma_{0.2}$, is observed. Simultaneously, Fig. 2(b) shows that a moderate increase in temperature produces a strong effect on the WH through a decrease in θ_0 , for $d = 2.6 \mu\text{m}$.

Conversely, for $d = 55 \mu\text{m}$, no large changes are observed in the SRS at the yield stress in Fig 1(b). In this case, an increasing upward curvature with increasing temperature is observed. The data for $d = 55 \mu\text{m}$ cannot be fitted by a straight line at higher temperatures as in the case of $d = 2.6 \mu\text{m}$. Simultaneously, the extrapolated hardening θ_0 remains approximately constant against these changes of temperature, Fig. 2(b) for $d = 55 \mu\text{m}$.

It can be noted that the behavior of the curves for $d = 55 \mu\text{m}$, in Fig. 1(b) and 2(b), resembles the behavior of fcc crystals for increasing temperatures [5] where an increase of dynamic recovery is induced. A similar correlation between WH and SRS data was also shown in fcc metals by Mecking and Kocks [6] and it was attributed to dynamic recovery, although this opinion is not unanimously accepted, Ref. [5].

In Fig. 3, the value of $\Delta\sigma_{0.2} / \Delta \ln \dot{\epsilon}$ is represented against $d^{-1/2}$ for the data of Fig. 1. In addition the slope of the data in Fig.1 is also plotted. The slope and $\Delta\sigma_{0.2} / \Delta \ln \dot{\epsilon}$ follows a linear dependence on $d^{-1/2}$.

In the following, an attempt to explain these findings on the basis of the thermal activation theory for dislocation slip is given. We will show that the dependence of $\Delta\sigma_{0.2} / \Delta \ln \dot{\epsilon}$ on the grain size is well explained by these models. However the strong influence of the grain size on the slope of the Haasen plots is out of the predictions. It is our contention that this difference between the experimental data and the models occurs because the contribution of GBS is not considered. Finally, a brief discussion, making a comparison with published data of cadmium, helps to give some qualitative guidelines about the reported grain size effects on the SRS and WH.

In order to perform a quantitative analysis of the SRS data it is necessary to deal with the superposition of hardening mechanisms leading to the flow stress [3]. Let us suppose a microstructure with a set of glide obstacles such as precipitates, grain

boundaries, forest dislocations, etc. In the most common approach, individual stress contributions are combined to obtain a constitutive equation. It is expected that this equation give the SRS of the alloy in terms of the SRSs of each contribution. Also, it is expected that this equation, in combination with a set of evolution equations for the microstructural parameters, give a description of the WH of the alloy.

The earlier attempts to account for the grain size effect on WH and SRS consisted in the extension of the Hall-Petch law to larger strains. Armstrong [7] proposes a phenomenological model of WH given by:

$$\sigma = \sigma_{cg}(\varepsilon) + c[m \tau_c(\gamma) Gb]^{1/2} d^{-1/2} = \sigma_{cg}(\varepsilon) + k(\varepsilon)d^{-1/2} \quad (1)$$

where $\sigma_{cg}(\varepsilon)$ corresponds to the stress-strain curve of the coarse grained material, c is a constant, m is an orientation factor, b is the Burgers vector, and $\tau_c(\gamma)$ is the resolved shear stress required for slip in an adjacent grain. However, according to the arguments discussed in Ref. [1], it is more justified to assume the following linear superposition rule:

$$\sigma = \sigma_o + \sigma_{HP} + \sigma_d \quad (2)$$

where σ_o is a frictional contribution, $\sigma_{HP} = kd^{-1/2}$ is the Hall-Petch contribution and $\sigma_d = M\alpha Gbp^{1/2}$ is the Taylor dislocation contribution, where ρ is the total dislocation density, G is the shear modulus, M is the Taylor factor, and α is a constant.

Eq. (1) cannot account for the decrease of WH with grain size in AZ31 because this implies a decrease of the slope k with increasing deformation [1] and there is no rationalization of such dependence of τ_c with strain γ . Eq. (2) also cannot account for these effects because there is no rationalization of the decrease in the rate of accumulation of dislocations with decreasing grain size. Therefore, it is unlikely that

Eqs. (1) or (2) leads to an adequate description of the SRS behavior. In the following, an attempt to, at least partially, rationalize our experimental data with the respective predictions of Eq.(1) and Eq.(2) is performed. Differentiating Eq.(1) with respect to $\ln \dot{\epsilon}$:

$$\frac{\partial \sigma}{\partial \ln \dot{\epsilon}} = \frac{\partial \sigma_{cg}}{\partial \ln \dot{\epsilon}} + \frac{c}{2} \left(\frac{mGb}{\tau_c d} \right)^{1/2} \frac{\partial \tau_c}{\partial \ln \dot{\epsilon}} \quad (3)$$

This equation can be rewritten as:

$$\frac{\partial \sigma}{\partial \ln \dot{\epsilon}} = \sigma_{cg} M_{cg} + \frac{k}{2} d^{-1/2} M_c \quad (4)$$

where $M_{cg} = \partial \ln \sigma_{cg} / \partial \ln \dot{\epsilon}$ and $M_c = \partial \ln \tau_c / \partial \ln \dot{\epsilon}$. From Eqs.(1) and (4) the SRS can be obtained as a function of the flow stress:

$$\frac{\partial \sigma}{\partial \ln \dot{\epsilon}} = \frac{k}{2} d^{-1/2} (M_c - 2M_{cg}) + \sigma M_{cg} \quad (5)$$

For the second approach to WH given by Eq.(2), differentiation with respect to $\ln \dot{\epsilon}$ leads to:

$$\frac{\partial \sigma}{\partial \ln \dot{\epsilon}} = \frac{\partial \sigma_o}{\partial \ln \dot{\epsilon}} + \frac{\partial \sigma_{HP}}{\partial \ln \dot{\epsilon}} + \frac{\partial \sigma_d}{\partial \ln \dot{\epsilon}} \quad (6)$$

Moreover, the contribution of the frictional and Hall-Petch stresses can be approximated by $\sigma_{0.2} \approx \sigma_o + \sigma_{HP}$. Therefore, the SRS, Eq.(6), can be rewritten as:

$$\frac{\partial \sigma}{\partial \ln \dot{\epsilon}} = \sigma_{0.2} (M_y - M_d) + \sigma M_d \quad (7)$$

where $M_y = \partial \ln \sigma_{0.2} / \partial \ln \dot{\epsilon}$ and $M_d = \partial \ln \sigma_d / \partial \ln \dot{\epsilon}$.

In general, the parameters M_i may be examined from the standpoint of thermal activation theory [8, 9]. As it is shown elsewhere [1, 8] the apparent activation area, Δa , is related to the SRS parameter by:

$$\frac{\partial \sigma}{\partial \ln \dot{\epsilon}} = \frac{k T M}{b \Delta a} \quad (8)$$

where k is the Boltzman constant and M is the Taylor factor. If there are i -contributions to the flow stress, σ_i , the SRS of the i -set of obstacles is related to their activation area by $\partial\sigma_i/\partial\ln\dot{\epsilon} = kTM/b\Delta a_i$. The M_i parameters are thus given by $M_i = kTM/b\sigma_i\Delta a_i$.

In pure metals, where the largest contribution to the flow stress is σ_d , the SRS parameter is, in some deformation range, proportional to the total flow stress σ . This is equivalent to state that M_d is a constant. This behavior is known as the Cottrell-Stokes law [5,8,9]. Eq. (8) shows that $\partial\sigma/\partial\ln\dot{\epsilon}$ is proportional to the flow stress σ , if Δa^{-1} is also proportional to the flow stress σ . This might be true for forest dislocations, with average spacing l , if $\sigma_d \propto 1/l$ and $\Delta a_d \propto l$. In addition, a dependence of M_d on grain size is not expected at all for glide controlled by the interaction with forest dislocations because it would indicate an additional dependence of σ_d or Δa_d on the grain size.

Taking into account that M_d or M_{cg} are independent on strain (during stage II) Eq.(5) predicts a linear relation between the SRS and the flow stress. The slope M_{cg} is the same as for a coarse grained material but the intercept with the ordinate axis has a dependence with the grain size as for the Hall-Petch law. Therefore, if the grain size is varied, a set of parallel curves are expected in the Haasen plot. On the other hand, Eq.(7) predicts almost the same behavior as Eq.(5), parallel lines of slope M_d and the intercept with the ordinate axis, $\sigma_{0.2}(M_y - M_d)$, with a dependence on $d^{-1/2}$ due to σ_{HP} . It is worth noting that the slope M_d , related to dislocation-dislocation interactions in the second model, Eq.(7), must be close to M_{cg} in the first model, Eq.(5).

As it can be seen in Fig. 3, $\Delta\sigma_{0.2}/\Delta\ln\dot{\epsilon}$ follows a linear dependence on $d^{-1/2}$ as it is predicted by Eqs.(5) or (7). In contrast, the slope also depends on $d^{-1/2}$, which is against the models discussed.

It is interesting to compare the present results with those for cadmium hcp polycrystals. Risebrough and Teghtsoonian [10] found an increase in the SRS with a

decrease in grain size in Cd. The SRS of Cd samples with grain sizes ranging from large grained polycrystals ($d = 1250 \mu\text{m}$) down to a grain size of $25 \mu\text{m}$ was measured at 77 K. The first attempt to rationalize these results was performed by Prasad and Armstrong [11] who attributed the grain size dependence to the second term of Eq.(3). The data given in Ref [10] are replotted in Fig. 4. These data can be fitted by parallel straight lines inside the stress region corresponding to the Stage II (marked with arrows). As it is shown, the grain size dependence of the SRS in Cadmium can be well explained by both models of Eq.(5) or Eq.(7).

It is worth noting that the WH behavior was also investigated in Ref. [10]. The stress-strain curves show an increase in WH with decreasing grain size (simultaneously to the SRS increase). It is concluded that, for cadmium, the current models qualitatively explain the effect of decreasing grain size on both, the WH and the SRS.

On the contrary, the behavior of AZ31 differs from these observations on cadmium. However, it is stressed that the range of grain sizes studied in both cases is quite different. It is our contention that these findings on SRS and WH of the AZ31 alloy can be congruently related to an increasing contribution of GBS with decreasing grain size. Indeed, for grain sizes decreasing into the nanometric scale in Mg [12], very low WH rates are observed together with an increase in the SRS. This behavior was attributed to GBS because values close to $m \equiv \partial \ln \sigma / \partial \ln \dot{\epsilon} = 0.5$ has been measured at room temperature for a grain size of 50 nm. In addition, Conrad et al. [13, 14] proposed a GBS mechanism to explain the grain size dependence of the plastic deformation kinetics of Cu and Ag in the grain size range of $10^{-2} \mu\text{m} < d < 1 \mu\text{m}$. In our case, m increases from 0.009 for $d = 55 \mu\text{m}$ to a maximum value of 0.017 for $d = 2.6 \mu\text{m}$. It is proposed, therefore, that the grain sizes studied in our work could be in a transition zone between conventional slip and GBS dominated flow, with $m = 0.5$, in the range of

nanometric grain sizes.

Summarizing, the decrease in grain size produces an increase in SRS; moreover this grain size dependence increases with temperature. A comparison between the present results and the WH data shows that there is a close correlation between both behaviors. The SRS data was analyzed using the framework of the thermal activation theory. The grain size dependence of the SRS at the yield stress is correctly predicted. However, the grain size effect on the slope of the Haasen plots and the WH behavior is not predicted by the analyzed models. This can be a consequence of an increasing contribution of GBS to deformation.

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FIGURES

Fig. 1. (a) The SRS as a function of the flow stress for the AZ31 alloy with grain sizes ranging from 2.6 to 55 μm at 300K, the values of $\sigma_{0.2}$ are indicated. (b) The SRS as a function of the flow stress at 300, 325 and 350 K, for the grain sizes of 2.6 and 55 μm .

Fig. 2. Representation of $\theta(\sigma - \sigma_{0.2})$ vs. $(\sigma - \sigma_{0.2})$ for the AZ31 alloy for: (a) various grain sizes at room temperature and (b) grain sizes of 2.6 and 55 μm at 300, 350 and 400 K.

Fig. 3. Representation of $\Delta\sigma_{0.2} / \Delta\ln\dot{\epsilon}$ and the slope of the Haasen plot, from Fig 1(a), against $d^{-1/2}$.

Fig. 4. Representation in a Haasen plot of results on cadmium hcp polycrystals, taken from Ref. [10].

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