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Investigation of the dependence of deformation mechanisms on solute content in polycrystalline Mg–Al magnesium alloys by neutron diffraction and acoustic emission

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ABSTRACT

Influence of aluminium content on the deformation mechanisms in Mg–Al binary alloys has been studied using *in-situ* neutron diffraction and acoustic emission technique. It is shown that the addition of the solute increases the critical resolved shear stress for twinning. Further, the role of aluminium on the solid solution hardening of the basal plane and softening of non-basal planes are discussed using results of the convolutional multiple peak profile analysis of diffraction patterns. The results indicate that the density of both prismatic $\langle a \rangle$ and pyramidal $\langle c + a \rangle$ dislocations increases with increasing alloying content.

1. Introduction

The application of magnesium alloys as lightweight structural elements in the transportation industry has been significantly increased in the last two decades. However, there are many technological challenges, as e.g. limited room temperature formability or asymmetric response on the strain path changing, which still waiting on the proper solution. The specific deformation behavior of the hexagonal closed packed (hcp) structure of magnesium is often in the background of these problems. It is well known that the basal slip $(0001)\langle 11\bar{2}0\rangle$ (BE) and $\{10\bar{1}2\}$ extension twinning requires the lowest activation stress at room temperature. The further slip systems, as first-order $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$ prismatic (PrE), followed by the first- $(\{10\bar{1}1\}\langle 11\bar{2}0\rangle)$ (PyE) and second-order $(\{11\bar{2}2\}\langle \bar{1}\bar{1}23\rangle)$ pyramidal generally requires either higher applied stress and/or elevated temperatures to be activated. As it was shown by numerous authors, the addition of solute elements significantly influences the deformation mechanisms both in single- [1,2] and polycrystals [3–7]. There is a general agreement that addition of Al and Zn increases the critical resolved shear stress (CRSS) for basal slip and concurrently decreases that for prismatic slip. The concentration dependence of CRSS usually follows the $\Delta\tau_s \propto c^n$ equation, where c is the atom concentration and $n = 1/2$–$2/3$ (for dilute and concentrated alloys, respectively). The works dealing with the influence of solute content on the twinning mechanisms are less frequent [3,4,6,7]. The majority of above listed works evaluates the CRSS of particular slip systems from the stress–strain curves and the input parameters of theoretical calculations are usually also based on the single crystal data [8,9]. Nevertheless, in the case of concentrated polycrystalline alloys such an approach often leads to ambiguous results, since the microstructural parameters, as grain size or initial texture have to be taken into account. In the last decade, the *in-situ* neutron diffraction (ND) has been proved as proper method for investigation of deformation mechanisms in the magnesium alloys [10–12]. The large penetration depth of the thermal neutrons facilitates the investigation of relatively large sample volumes, which is a key feature in the case of coarse-grain materials. In combination with the acoustic emission (AE) technique, the twin nucleation (AE measurement) and growth (ND measurement) can be successfully investigated [10,12]. Further, the activation stresses of particular deformation mechanisms can be deduced from the stress dependence of lattice strains. Finally, the recent results show that the evolution of the dislocation structure can be obtained using the diffraction line profile analysis [13–15].

In this work the effect of Al on the deformation mechanisms in binary Mg–Al alloys has been studied *in-situ* using neutron

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diffraction and acoustic emission techniques during uniaxial compression tests at room temperature. The influence of the solute content on both dislocation slip in particular systems and extension twinning is discussed in detail.

2. Experimental procedure and processing methods

Pure Mg, binary Mg–2 wt.% Al and Mg–9 wt.% Al (further referred as Mg2Al and Mg9Al) were used for the experiments. Since the grain size significantly influences the twinning activity, the authors endeavored to prepare specimens with similar grain sizes. The final (after heat treatment) grain sizes (measured by linear intercept method according to ASTM E112) of (100 ± 10) μm for pure Mg and Mg9Al and (85 ± 15) μm for Mg2Al were achieved as follows: in the casting phase 1 wt.% Zr was added to the melt in the case of pure Mg, whereas different cooling rates were used in the case of Mg–Al binary alloys. The as-cast specimens were then solution heat treated for 24 h@413 °C and quenched into water. Since the proper tuning of cooling rate was difficult, there is a difference between the grain sizes of Mg2Al and Mg9Al alloys. Nevertheless, such a difference is not significant from the point of view of the deformation mechanisms studied. The inverse pole figures (IPF) and the microstructures after the heat treatment are showed in Figs. 1 and 2. As it is obvious from IPFs (Fig. 1), all of specimens exhibited a random initial texture. The initial microstructure is similar for pure Mg and Mg9Al samples (Fig. 2a and c): the grain size varies; both coarser and finer grains are present. In contrast, the Mg2Al specimen has more uniform grain size distribution and generally finer grains (Fig. 2b). The testing was carried out using cylindrical specimens with a diameter of 9 mm and gauge length of 20 mm. The *in-situ* neutron diffraction (ND) measurements were carried out at the SMARTS engineering instrument [16] in the Lujan Neutron Scattering Center. The mutual orientation of the longitudinal axis of the sample and the incident beam was 45°. The two detector banks were positioned at ± 90° to the incident beam in order to record diffraction pattern in both along and perpendicular to the loading direction (for scheme of the experimental setup see [17]). The compression testing were carried out using a horizontal 250 kN capacity load frame at a strain rate of $1 \times 10^{-3}\ s^{-1}$ in strain control mode. In order to collect ND data with good enough statistics, the test were stopped at predefined strain levels (0.1%, 0.5%, 1%, 2%, 3%, 4%, 5%, 6%) for approx. 70 min.

The acoustic emission (AE) testing was performed using a Physical Acoustics PCI-2 acquisition board and a broadband AE sensor from Dakel company was mounted on the outside the gauge length using vacuum grease and an elastic band. The AE amplified by 60 dB in the frequency range 100–1200 kHz. The threshold level was set as 30 dB. The AE was recorded separately during uniaxial deformation at the same condition, as during ND testing.

The samples for metallography were first grinded, than polished step-by-step 3, 1 and ¼ μm diamond paste and finally etched in 3% Nital solution.

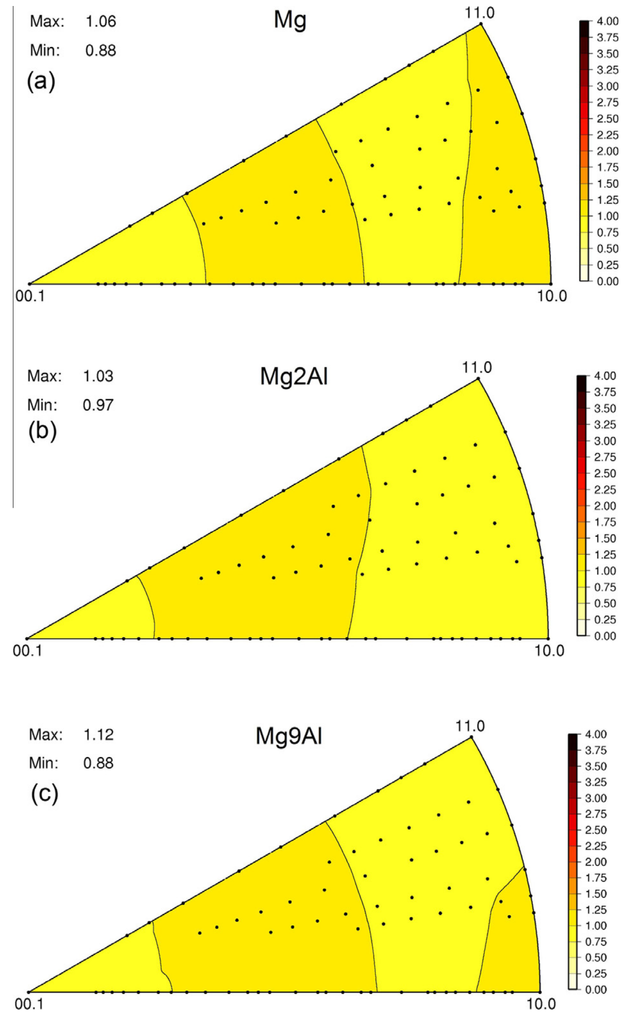

Fig. 1. The IPF of initial texture of deformed samples measured by ND in axial direction for (a) Mg; (b) Mg–2 wt.% Al; and (c) Mg–9 wt.% Al alloy.

3. Results and discussion

3.1. Stress–strain curves and the corresponding AE responses

The influence of Al content on the mechanical properties is presented in Fig. 3. In agreement with the literature data [1,5], the strength of alloys increased with the increasing Al content. As it was discussed in detail by Cáceres and Rovera [5], the effect of solid solution on the yield strength in polycrystals can be roughly deduced similarly to the single crystals, when the experimental yield stress data are corrected for the grain size. They suppose to express the combined effect of the grain size and solid solution strengthening on the experimentally established yield stress σ_{02}^{exp} as:

$$\sigma_{02}^{exp} = \Delta\sigma_{HP} + \Delta\sigma_{ss} = (\sigma_0 + kd^{-1/2}) + MB_n c^n \tag{1}$$

where σ_0 and k are parameters of Hall–Petch equation (we used values σ_0 = 11 MPa [18], k = 0.39 MPa/m$^{-1/2}$ [19]), M is the Taylor orientation factor and $B_n = d\tau_R/dc^n$ is the solid solution hardening rate on the basal plane. In Fig. 4, both the concentration dependence of σ_{02}^{exp} as well as the values corrected to grain size $\Delta\sigma_{ss}$ are plotted as a function of the $c^{2/3}$ (i.e. we assuming Labusch's theory for concentrated alloys, describing dependence of resolved shear stress $\Delta\tau_s$ on solute concentration c as $\Delta\tau_s \propto c^{2/3}$ with B_n = 39.5 MPa (at.)$^{-2/3}$ [5]). The dependence is clearly linear and the value for M = 4.7 obtained from the linear fit (slope = 185.6 MPa (at.)$^{-2/3}$, thus M = 185.6/39.5) is in the range (4–6) suggested by theoretical calculations [18]. Thus, it seems that the strengthening of the basal planes by solutes significantly contributes to the yield strength. This conclusion is in agreement with results on other binary systems (e.g. Mg–Zn [4,20]). Nevertheless, both single crystals studies [2] and the recent modeling data [8,9] indicates that the prismatic $\langle a \rangle$-slip plays also a significant role in the plasticity around the macroscopic yield. This mechanism is enhanced by pile-up of basal dislocations at the end of easy glide stage, since the cross-slip through prismatic plane becomes easier due to the stress concentration from pile-ups [21]. Hence, the investigation of the influence of the solutes on this system is also crucial. The AE count rates (i.e. number of crossing of threshold level per second) corresponding to the stress–strain curves are depicted in Fig. 5a. It is obvious that the Mg9Al specimen has the lowest AE response, whereas the pure Mg and Mg2Al behave similarly. The characteristic peak in the vicinity of the yield point can be attributed to synergic effect of twin nucleation and massive dislocation motion [22,23]. Discriminating of the twinning and dislocation density is a complex task requiring statistical analysis of the AE parameters, which is beyond of the scope of this paper. The recent results of Vinogradov et al. [24] proved that the straining starts with a slip of dislocation in basal plane and the twinning is the major contributor to the above mentioned peak of count rate. Above the yield point the count rate rapidly decreases. This effect is caused by several issues: (i) the dislocation density increases with increasing stress, which causes reduction of the mean free path of dislocations. Since this parameter is proportional to the released AE energy [25], the count rate decreases. (ii) The