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

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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 $n = 1/2 \sim 2/3$ (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 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 $(100 \pm 10) \mu\text{m}$ for pure Mg and Mg9Al and $(85 \pm 15) \mu\text{m}$ 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 24h @ 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 $\pm 90^\circ$ 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 $1 \times 10^{-3} \text{ s}^{-1}$ 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 $\frac{1}{4} \mu\text{m}$ diamond paste and finally etched in 3% Nital solution.

Results and discussion

2.1 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:

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

where σ_0 and k are parameters of Hall-Petch equation (we used values $\sigma_0 = 11$ 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 twin nucleation is followed by rapid twin growth in compression [10]. The twin growth was found inaudible for

AE owing to their slow speed [26]. It is noteworthy that the decrease of count rate is more rapid with increasing solute content, most probably due to faster accommodation of dislocations. If the count rate is plotted against the true stress (Fig. 5b), it can be seen that the peak values of the count rate are shifted towards higher stresses with increasing Al content, which is a clear sign that the twin nucleation stress depends on the alloy content.

In summary the mechanical tests and the AE indicate that the solute atoms harden the basal plane, significantly increase the dislocation density and influence the twin nucleation stress. In the next chapter these assumptions are verified using the neutron diffraction technique.

2.2 Neutron diffraction measurements

The neutron diffraction experiments have been used for estimation of the twinned volume, calculation of the lattice strain as function of the applied stress and determination of fraction of dislocations in particular slip systems.

2.1.1 Solute content dependence of twinned volume

As it was shown by seminal work of Gharghouri et al. [11], the {10.2} extension twinning causes significant texture changes, which manifests with intensity changes of diffraction peaks. Since *in compression* the grains having their *c*-axis perpendicular to the loading axis are optimally oriented for extension twinning the {10.0}–(00.2) and {11.0}–{10.3} parent (i.e. grains undergoing twinning) - daughter (i.e. twinned fraction of the parents) are those, which characterize the most the twinning activity in the *axial* detector. Thus if the extension twinning is active, the {10.0} and {11.0} peaks decreases and the (00.2) and {10.3} increases. Such a behavior can be seen in Fig. 6, where the relative intensity changes determined by single peak fits with respect to the initial (stress-free) state of the parent-daughter orientations presented. The evaluation of the twin volume fraction can be performed by a 2-bank Rietveld refinement assuming an axisymmetric texture [17], [27], [28]¹. Fig. 7 shows the development of the axial distribution function for the 00.2 peak during deformation. It is obvious that the 00.2 intensity is almost random at the start, and strongly increases at the center during compression, consistent with the expected texture change due to extension twinning. From the changes in the area under the curve below the cross-over point we can directly determine the

¹ Since the initial texture is random and the uniaxial deformation is an axisymmetric operation, the radial symmetry is warranted. The refinements were done using the GSAS [26] and SMARTSware [27] software packages developed at Los Alamos National Laboratory.

twin volume fraction (TVF). Figure 8 shows the twin volume fraction as a function of the composition, applied strain (a) and stress (b). It is obvious that the strain dependence of the TVF for pure Mg and Mg-2%Al is the same within the experimental error (Fig. 8a), which fits well with the AE data. In contrast, the TVF for Mg-9%Al is smaller, which indicates that there is another significant strain accommodation mechanism besides the twinning. Similar dependence of twinned volume on Al content was observed in the same set of specimens by Nagarajan et al. [7] using analysis of metallographic pictures.

If we plot the same quantity against the applied stress, we find that the stress, at which the twin volume starts to grow, increases with increasing Al content. This result can be used for an estimation of the solute strengthening of the twin growth. If we subtract, similarly to Eq. (1), the contribution of the grain size from the experimentally measured data, we obtain a dependence of critical stress for twin growth on the Al content. The influence of the solute content on the twin growth was recently studied by Ghazisaeidi et al. [6]. They calculated the solute – twin dislocation interaction energy by density functional theory (DFT) and predicted the solute strengthening of the twin growth for Mg-Al binary system. As it is obvious from Fig. 9., our experimental data are in good agreement with their theoretical predictions.

2.1.2 Lattice strain evolution

The elastic lattice strains were calculated using the Bragg's law, which gives relation between the scattering angle (θ) and lattice spacing (d) as follows:

$$2d \sin \theta = n\lambda \quad (2)$$

The relative strain (ε) is then given by differentiating of Bragg's equation:

$$\varepsilon = \frac{d-d_0}{d_0} = -\cot \theta (\theta - \theta_0) \quad (3)$$

In the present work, we considered d_0 and θ_0 as the lattice spacing and the Bragg angle of the corresponding lattice planes in the stress-free material. The activation of specific deformation mechanisms emerges on the lattice strain – applied stress curves as a deviation from the straight line of Hooke's elasticity. In Fig. 10 the stress evolution of the (00.2)-{10.0} lattice strains, related to extension twinning, are shown with respect to the both axial and radial detector. When the twinning become active, the “soft-oriented” grains (e.g. {10.0} in axial and (00.2) in radial detector, respectively) starts to behave plastically and their curve deviate above the ideal straight line. At the same time the conjugated curves of “hard-grains” fall under this line, since they have to accommodate larger portion of the elastic loading [29]. The

ideal elastic behavior is violated approximately at the same stress level, at which the both twin volume starts to grow and a maximum of the AE response is observed (c.f. Fig. 5b).

The grains having their $\{10.2\}$ planes in Bragg position with respect to the axial detector are almost ideally oriented for basal slip in compression (their Schmid-factor for basal slip is: $m_{basal}^{10.2} = 0.43$). Thus, the inflection point of the lattice strain gives estimation for the activation stress for basal $\langle a \rangle$ -slip. As it is shown in Fig. 11a, this value varies with the solute content. If we plot the data into against the $c^{2/3}$ (see Fig. 4), we got a linear dependence, giving a slope of 168.3 MPa (at.) $^{-2/3}$, and $M = 168.3/39.5 = 4.26$, which is almost the same value obtained from the stress-strain curves. In the case of the $\{10.1\}$ “grains”, the activation of the prismatic $\langle a \rangle$ -slip cannot be excluded besides the basal slip ($m_{basal}^{10.1} = 0.36$; $m_{prism}^{10.1} = 0.34$). The stresses at inflection points are higher than those for $\{10.2\}$ grains, but the solute dependence exhibit a similar character (Fig. 11b).

2.1.3 Fraction of dislocations in particular slip systems

The neutron diffraction patterns obtained for the Mg samples deformed up to the strain of 6% were evaluated by the Convolutional Multiple Whole Profile (CMWP) fitting method [30, 31]. Each diffraction pattern is fitted by the sum of a background spline, the convolution of the instrumental pattern and the theoretical line profiles related to crystallite size and dislocations. Finally, the dislocation density (ρ), and the parameters q_1^m and q_2^m are obtained. For magnesium eleven families of slip systems on basal (4), prismatic (2) and pyramidal (5) planes with three different Burgers vectors are considered, which are divided into three groups based on their Burgers vectors: $b_1 = 1/3 \langle \bar{2}110 \rangle$ ($\langle a \rangle$ type), $b_2 = \langle 0001 \rangle$ ($\langle c \rangle$ type) and $b_3 = 1/3 \langle \bar{2}113 \rangle$ ($\langle c+a \rangle$ type). ([14]) in order to reduce the number of variables. The determination of the fractions of dislocations in the different slip system families (f_a , f_c and f_{c+a}) is performed by a computer program, which takes advantages of the fact that the values of particular fractions have to be positive and their sum must be equal to 1 [32]. Further details about the CMWP fitting procedure can be read in Refs. [14, 31, 32]

The dislocation density as a function of strain is shown in Fig. 12. The dislocation density increases with increasing Al content. This result indicates that at higher solute concentration the accommodation of strain by dislocations becomes more significant. The evaluation of the fractions of the dislocations in the particular slip systems approved this assumption. The strain dependence of the basal $\langle a \rangle$ vs. non-basal $\langle a \rangle$ -dislocations ratio is clearly influenced by the

solutes (Fig. 13a)². If we plot the relative change of BE/(PrE+PyE) ratio against the initial dislocation configuration (Fig. 13b), we can see that the Mg9Al specimen increases the fraction of the non-basal $\langle a \rangle$ at the expense of basal $\langle a \rangle$ dislocations. Further the stress necessary for increasing the non-basal $\langle a \rangle$ fraction is less for alloys than that for pure magnesium (Fig. 13c). The necessity of activation of prismatic $\langle a \rangle$ slip for the macroscopic plasticity is a well known phenomenon, described in numerous theoretical [8, 33] and experimental work [1, 4, 5]. The solute atoms have been found to soften the prismatic slip system through ease the both cross slip of dislocations and ability of dislocations to form a jog-pair [2, 33]. Our results indicate that the solute softening of prismatic slip system increases with increasing Al concentration, which is in good agreement with the findings Akhtar and Teghtsoonian [2] in dilute Mg-Al single crystals.

The influence of the solutes on the pyramidal $\langle c+a \rangle$ -slip have been studied only in few works [34, 35]. The $\{11\bar{2}2\}\langle 11\bar{2}3 \rangle$ slip mode is the only mechanism besides the extension twinning, which provides elongation in crystallographic c direction. Despite of the high critical resolved shear stress for its activation at room temperature, several works found indication for its activity [8, 12]. The strain evolution of the ratio of $\langle a \rangle/\langle c+a \rangle$ dislocation is plotted in Fig. 14. There is a jump at 1% strain for Mg2Al and Mg9Al alloy, caused by massive activation of non-basal $\langle a \rangle$ slip in the vicinity of the macroscopic yield point. For Mg9Al alloys at higher strain level the fraction of $\langle c+a \rangle$ dislocations significantly increases, whereas for Mg2Al and pure Mg the increment is very small. Such a large difference can be given by restricted twin growth in Mg9Al, shown in §2.1.1. Since the strain cannot be accommodated by twinning, an alternative mechanism is required for continuing the plastic deformation, which is realized in the form of $\langle c+a \rangle$ -slip.

Conclusions

The influence of solute content on the deformation mechanisms was investigated in Mg-Al binary alloys using advanced *in-situ* methods. The following conclusion can be drawn:

- The stress necessary for both twin nucleation and growth increases with increasing Al content.
- The softening effect of Al on the prismatic plane was proven. The macroscopic yielding is accompanied by onset of prismatic $\langle a \rangle$ slip, which magnitude increases with increasing Al content.

² Owing to the similar average contrast factors of prismatic and pyramidal $\langle a \rangle$ dislocations is difficult to make difference between them, therefore they are treated together

- As a consequence of limited twin growth, the pyramidal $\langle c+a \rangle$ slip plays a key role in the plasticity of Mg-9 wt.%Al alloy.

Acknowledgement

The authors are grateful for the financial support of the Czech Science Foundation under the contract P204/12/1360. JČ acknowledges the support from the Grant Agency of Charles University under contract Nr. 513512. This work has benefited from the use of the Lujan Neutron Scattering Center at LANSCE, funded by the US Department of Energy's Office of Basic Energy Sciences. Los Alamos National Laboratory is operated by Los Alamos National Security LLC under US DOE Contract DE-AC52-06NA25396. KM is grateful to Pavel Lukáč (Charles University, Prague) and Carlos Cáceres (The University of Queensland, Brisbane) for fruitful discussions.

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Figure captions

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

Fig. 2 – The initial microstructure of samples for a)Mg; b)Mg-2 wt.% Al; c)Mg-9 wt.% Al alloy

Fig. 3 – The true stress – true strain curves as measured in compression for various Al concentration

Fig. 4 – Experimental yield strength σ_{02}^{exp} (solid symbols); values corrected to grain size $\Delta\sigma_{ss}$ (empty symbols) and inflection stress $\sigma_i^{(10.2)}$ for {10.2} lattice strain from the ideal elastic response as a function of $c^{2/3}$ (c - Al concentration)

Fig. 5 – The AE count rates measured during compression tests as a function of a) strain; b) stress for all solute concentrations

Fig. 6 – Relative changes of normalized integrated intensity for {10.0}-(00.2) and {11.0}-{10.3} intensity pairs

Fig. 7 - Axial distribution function variation for the 00.2 pole during for Mg-2 wt.% Al alloy measured at different strains

Fig. 8 – Evolution of twin volume fraction for all examined Al concentrations as a function of a) strain; b) stress

Fig. 9 – Comparison of the concentration dependence of the critical stress for twin growth as measured experimentally (symbols) and calculated using theoretically (theoretical data are replotted from [6]). The CRSS for basal slip is indicated as well.

Fig. 10 – Stress evolution of the (00.2)-{10.0} lattice strains as it was measured in a) axial; b) radial detector. The ideal elastic response is indicated with a dash line.

Fig. 11 – Stress evolution of a) {10.2}; b) {10.1} lattice strains as it is measured in axial detector. The ideal elastic response is indicated with a dash line.

Fig. 12 – Evolution of dislocation density with applied strain for all alloy compositions.

Fig. 13 – a) Strain dependence of the - Basal edge (BE) - Prismatic + Pyramidal Edge (PrE+PyE) dislocations ratio; b) Evolution of the relative change of BE/(PrE+PyE) ratio with the strain; c) Evolution of the relative change of BE/(PrE+PyE) ratio with the strain.

Fig. 14 – Evolution of the ratio of $\langle a \rangle$ and $\langle c+a \rangle$ dislocations as a function of the applied strain and Al concentration.

Figure01a
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Mg

Max: 1.06

Min: 0.88

Mg2Al

Max: 1.03

Min: 0.97

Mg9Al

Max: 1.12

Min: 0.88

Figure02a
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Figure02b
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Figure02c
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Figure03

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Figure04

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Figure05a
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Figure05b
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Figure06a
[Click here to download high resolution image](#)

a)

Figure06b
[Click here to download high resolution image](#)

b)

Figure07
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Figure08a
[Click here to download high resolution image](#)

Figure08b
[Click here to download high resolution image](#)

b)

Figure09

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Figure10a
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Figure10b
[Click here to download high resolution image](#)

Figure11a
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10.2 Compression Axial

a)

Figure11b
[Click here to download high resolution image](#)

10.1 Compression Axial

b)

Figure12
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Figure13a
[Click here to download high resolution image](#)

a)

Figure13b
[Click here to download high resolution image](#)

b)

Figure13c
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Figure14

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