Mechanical and physical properties of Mg alloys prepared by SPD methods

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Abstract. Magnesium alloys were prepared by severe plastic deformation (SPD). Three methods were used. The mechanical and thermal properties were estimated. The increase in strength could be attributed to the grain refinement and increased dislocation density. The thermal conductivity and expansivity was measured for samples prepared with different methods. Some examples are shown and discussed.

1 Introduction
In recent decades, many papers describing the deformation behavior of Mg alloys under different conditions have been published. It was shown that the yield strength of Mg alloy polycrystal $\sigma_y$ depends on the grain size $d$ according the following Hall-Petch relation

$$\sigma_y = \sigma_0 + k_y d^{-1/2}.$$  (1)

Here $k_y$ is a constant and $\sigma_0$ is a friction stress. According to this relation, ultra-fine-grained materials (UFG) can exhibit a considerable higher strength. The micrometer or nanometer sized material may be produced by different methods.

Over the last decades, a significant effort has been devoted to obtaining UFG materials using several plastic deformation (SPD) methods. The processing mechanisms and technological parameters are especially important for the materials microstructures. The dislocation density, grain size, and texture are produced. Recently, Estrin and Vinogradov [1] have presented a review on extreme grain refinement by SPD techniques. The principal processing scheme and mechanisms were described. The samples processed by SPD have enhanced mechanical properties.

Besides the mechanical strength, also the physical properties of samples after SPD are influenced. This is important for applications [2]. In the present work, some examples concerning the effect of SPD on the mechanical and thermal properties of Mg alloys will be presented.

2 Materials and methods
Two magnesium alloys AZ31 (3wt%Al-1wt%Zn-balance Mg) and AX52 (5wt%Al-2wt%Ca-balance Mg) were processed by SPD. Among the severe plastic deformation techniques, three processes were used: a) Equal-channel angular pressing (ECAP) [3-5] b) accumulative roll bonding (ARB) [6,7] and c) Rotary swaging (RSW) [8]. The microstructure was analyzed by light microscopy and by scanning electron microscopy (SEM). Mechanical properties and thermal properties were estimated in a wide temperature range from RT to 350 °C. Samples were deformed in tension and/or compression.
Thermal diffusivity, \( \alpha \), and specific heat capacity at the constant pressure, \( c_p \), were measured using the laser flash method in a wide temperature range of 20 and 350 °C. The details can be found in references [4, 6]. Thermal conductivity, \( \kappa \), was calculated according the following relationship

\[
\kappa = \alpha \rho c_p,
\]

(2)

where \( \rho \) is the density estimated by the hydrostatic method. Thermal expansion was measured using LINSEIS L75PT-1600 dilatometer with a horizontal dual sample arrangement. Two temperature cycles (heating up to 400 °C and cooling to room temperature) were performed for each sample using heating and cooling rates of 1 K/min in a resistance furnace in an argon protective atmosphere. Thermal expansion coefficient, \( \alpha \), was estimated as an extrapolated value of the temperature dependence to room temperature [6].

3 Experimental results

3.1 Mechanical properties

The grain size of AX52 magnesium alloy processed by ECAP in the route A was substantially refined. The grain size decreased after 8 passes from 60 \( \mu \)m to 3.6 \( \mu \)m (see Table 1). The tensile yield strength (TYS) obtained at two different strain rates 10\(^{-3}\) s\(^{-1}\) and 50 s\(^{-1}\) increased with the increasing number of passes for both strain rates as it is shown in Table 1. Although the tensile tests were performed at room temperature, the sensitivity of the TYS to the strain rate indicates the presence of the thermally activated process/es [3]. The strength of ECAPed AZ31 samples is dependent also on the route used [5]. The TYS and UTS (ultimate tensile stress) of AZ31 samples estimated using ECAP in routes A, BC, and C after different passes are listed in Table 2. The strain rate used was in the order 10\(^{-3}\) s\(^{-1}\). Equivalent strain achieved after one ECAP pass is 1.15.

**Table 1.** The grain size, tensile yield stress, ultimate tensile strength, and thermal conductivity of the AX52 alloy depending on the number of ECAP passes.

| Nr of passes | Grain size (\( \mu \)m) | TYS(10\(^{-3}\)s\(^{-1}\)) (MPa) | TYS(50s\(^{-1}\)) (MPa) | \( \kappa \) (20 °C) (W/m.K) | \( \kappa \) (250°C) (W/m.K) |
|--------------|-------------------------|---------------------------------|-------------------------|----------------------------|--------------------------|
| 0            | 63                      | 77.1                            | 85.2                    | 70.2                       | 68.4                     |
| 1            | 28                      | 137.3                           | 313.2                   | 87.0                       | 84.9                     |
| 2            | 21                      | 170.2                           | 346.0                   | 79.1                       | 68.3                     |
| 4            | 4                       | 197.8                           | 358.9                   | 73.9                       | 61.3                     |
| 8            | 3.4                     | 205.9                           | 358.8                   | 75.5                       | 71.0                     |

Mechanical properties of AZ31 magnesium alloys were also investigated using samples processed by rotary swaging. The yield strength and ultimate strength determined in tensile (TYS, UTS) and compression tests (CYS, CPS) at room temperature are presented in Table 3 depending on the number of the swaging steps. The yield strength values estimated in tension are higher than the values found in compression tests; the tensile compression anisotropy is observed. Plastic anisotropy was also observed on AZ31 sheets submitted to accumulative roll bonding [7]. Values of the TYS measured in the rolling direction (L) are higher compared to the TYS in the transversal direction (T) (see Table 4). The deformation stresses increased with the number of passes in the rolling mill for both samples of orientation L or T.

**Table 2.** The stress characteristics of AZ31 alloy estimated for the three processing ECAP routes: A, BC, C.
### Table 3: The stress characteristics estimated in tension and compression (strain rate of $10^{-3} \text{s}^{-1}$) for AZ31 alloy submitted to rotary swaging.

| Nr of RSW | Grain size ($\mu$m) | TYS (MPa) | UTS (MPa) | TYS (MPa) | UTS (MPa) | TYS (MPa) | UTS (MPa) |
|-----------|---------------------|-----------|-----------|-----------|-----------|-----------|-----------|
|           | 20-300              | 60.5±55.2 | 41.5±28.6 | 40.3±26.8 | 26.3±13.9 | 16.2±12.6 |
|           | 1                   | 102       | 105       | 120       | 123       | 106       | 154       |
|           | 2                   | 341       | 331       | 343       | 343       | 342       | 329       |
|           | 3                   | 98        | 103       | 123       | 131       | 130       | 186       |
|           | 4                   | 374       | 374       | 382       | 390       | 378       | 428       |
|           | 5                   | 0         | 0.54      | 0.83      | 1.16      | 1.39      | 1.83      |

### Table 4: Tensile yield strength and thermal expansion coefficient estimated for AZ31 alloy after ARB in the rolling direction (L) and transversal direction (T).

| Nr of passes | TYS(L) (MPa) | TYS(T) (MPa) | UTS(L) (MPa) | UTS(T) (MPa) | CTE(L) $\times 10^{-6} \text{K}^{-1}$ | CTE(T) $\times 10^{-6} \text{K}^{-1}$ | Equivalent strain |
|--------------|--------------|--------------|--------------|--------------|--------------------------------------|--------------------------------------|--------------------|
| 0            | 162.5        | 85.0         | 307.0        | 297.0        | 25.92                                | 27.16                                | 0                  |
| 1            | 223.0        | 172.4        | 344.9        | 365.6        | 26.05                                | 26.49                                | 1.86               |
| 2            | 227.0        | 212.0        | 356.6        | 370.0        | 25.04                                | 26.41                                | 3.72               |

### 3.2 Thermal properties

Thermal properties of magnesium alloys are especially important for their industrial applications. In the following, we like briefly to summarize the effect of SPD processing, as mentioned in subsection 3.1, on the thermal conductivity of AX52 and AZ31 alloys. Table 1 lists the thermal conductivity of AX52 magnesium alloy samples after ECAP using the route A after 1, 2, 4, and 8 passes estimated at 20 and 250 °C. The thermal conductivity at a higher temperature is lower than that at room temperature.

### Table 5: Thermal conductivity of AZ31 alloy submitted to accumulative roll bonding.

| $\kappa$ (Wm$^{-1}$K$^{-1}$) | 20 °C | 100 °C | 200 °C | 300 °C |
|-----------------------------|-------|--------|--------|--------|
| 0 pass                      | 76.0  | 85.7   | 92.4   | 99.5   |
| 1 pass                      | 80.1  | 89.4   | 96.9   | 102.9  |
| 2 passes                    | 87.1  | 96.8   | 105.5  | 112.0  |

The differences were found at all passes (at all grain sizes). The effect of temperature on the thermal conductivity of AZ31 specimens processed by accumulative roll bonding is summarized in Table 5.

Figure 1 shows the coefficient of thermal expansion (CTE) extrapolated to room temperature of AZ31 alloy after ECAP at different routes (A, BC, and C) as a function of the route number. The permanent strain estimated after the temperature cycle (heating to 400 °C and cooling to 20°C) is shown in Figure 2.
Thermal expansion characteristics of magnesium alloys depend on the temperature range, chemical composition, and processing conditions. The thermal conductivity of AZ31 sheets increases with increasing temperature. Similar results were found by Lee et al. [9]. The thermal conductivity consists of two components: an electron component and a lattice (phonon) component. It is important to stress that the electron component strongly depends on the crystal defects.

5 Discussion

The yield strength and deformation mechanisms of magnesium alloys are extremely sensitive to the microstructure (grain size, dislocation density, texture) achieved by severe plastic deformation. Magnesium materials submitted to severe plastic deformation exhibit the pronounced texture which depends on the SPD type. This texture development is due to the fact that the magnesium contains only two easy slip systems. To fulfill the von Mises criterion, five independent slip systems must cooperate to secure the compatible deformation [10]. Besides of basal slip, non-basal prismatic or pyramidal slip systems should be activated. The further possibility for compatible deformation is mechanical twinning, which is usually observed at the beginning of the plastic deformation at lower temperatures. The texture observed in ECAPed samples depends on the processing route. In the AZ31, three different textures were developed for A, BC, and C routes. In the samples extruded via A and B, the pyramidal planes \{10 \bar{1}2\} and \{10 \bar{1}1\} tend to be aligned nearly parallel to the extrusion direction (ED). Such orientation is favourable for motion of \(1/3 <1 \bar{1} 2 0>\) dislocations in the basal or prismatic planes. After the processing by route C, the \(c\)-axis is nearly oriented to ED than the deformation process starts with the twinning. Generally, it is possible to conclude that the strengthening effect of the ECAP and the TYS increase are saturated in the 4th pass and the changes estimated after the 8th pass were found to be marginal. Comparing the TYS estimated for cast AZ31 alloy (TYS(0)=57 MPa), we may conclude that the strengthening effect of the ECAP is significant already after the first pass; further passes increase the deformation stress only marginally even decrease. A significant increase of TYS was observed in the AX52 alloy after the ECAP using the route A. The highest effect of ECA extrusion was observed just after the first pass.

Observed texture in the AX52 alloy was mostly developed after the first pass via route A. The basal planes are parallel to the extrusion direction, while the prismatic planes rotate along the \(<c>\) axis. A part of the basal planes, oriented 45° to the ED, lay near the maximum shear stress plane. The observed
increase of the yield strength corresponds to a decrease in the grain size. It indicates that the main reinforcing mechanism is the Hall-Petch strengthening. The main thermally activated mechanism is very probably the motion of $<a>$ dislocations in the prismatic planes [3]. Pronounced planar anisotropy was observed in the AZ31 sheets submitted to the ARB. The yield strength of L-samples where the tension axis was parallel to the ED was higher than that measured in T-samples with the axis perpendicular to the ED [7]. This anisotropy decreased with the increasing number of passes and increasing deformation temperature. The rolled sheets of the AZ31 alloy after the ARB exhibited a typical fibre texture with the splitting of the basal poles in the transversal direction. The plastic anisotropy consists in the different deformation mechanisms occurring during straining. While the deformation in L samples occurs mostly by the dislocation mechanism, the beginning of straining in the T samples was realised by the mechanical twinning. Formation of twins during the deformation of T samples was confirmed by the acoustic emission detection. The twinning activity decreases with decreasing grain size [11]. The tension – compression asymmetry was observed in the rotary swaged AZ31 samples. The reason for this asymmetry consists again in the different deformation mechanisms in tension and compression. The fibre texture in the swaged samples oriented basal planes parallel to the ED. Straining of samples in the $<c>$ axis of the hexagonal magnesium cell is possible by the twinning mechanism. While in tension tests six twins’ variants may be formed, in compression are only two.

Similarly, the thermal properties depend on the microstructure and defects in the crystal structure. The thermal conductivity of the AX52 alloy increased after the first ECAP pass and then again decreased up to the 4th pass, eventually again slightly increased in the 8th pass. The thermal conductivity of the AZ31 sheets after the ARB increased with the measurement temperature and the number of passes in the rolling mill. The thermal conductivity of metallic materials, $\kappa$, may be written as a sum of two components - the electronic part, $\kappa_e$, and phonon part, $\kappa_{ph}$:

$$\kappa = \kappa_e + \kappa_{ph}.$$  \hspace{1cm} (3)

The thermal conductivity may be affected by various microstructural factors such as texture, grain size, solute atoms, precipitates, and dislocations [12]. The electron conductivity is proportional to the electrical conductivity, $\sigma$, via the Wiedemann-Franz law

$$\kappa_e = \sigma LT,$$  \hspace{1cm} (4)

where the constant $L$ is the Lorenz number. Thermal conductivity of alloys can be described with the Smith-Palmer equation [13]

$$\kappa = \frac{C}{\rho_e}LT + D,$$  \hspace{1cm} (5)

where $C$ and $D$ are constants and $\rho_e$ the electrical resistance. The Smith-Palmer equation assumes a temperature-independent phonon thermal conductivity via the constant $D$. Bass measured electrical resistivity in magnesium single crystals [14]. He estimated that the electrical conductivity along the $<c>$ axis is higher than that in the $<a>$ direction i.e. $\kappa(c)/\kappa(a)>1$. The ECAP process oriented the basal planes in AX52 samples parallel to the ED and the channel walls. The conductivity is measured mostly in the $<a>$ direction. Similar effect may be observed in the AZ31 alloy submitted to the ARB. In this case, the conductivity is measured perpendicular to the sheet surface and owing to the texture parallel to the $<c>$ axis. Improved texture after the first and second rolling pass increases the conductivity. On the other hand, the conductivity decreases with increasing concentration of crystal defects (dislocations, point defects). Severe plastic deformation by the ECAP may increase the dislocation density and so simultaneously decrease the conductivity, which is also observed in the AX52 samples.

The planar anisotropy of the thermal expansion coefficient in the AZ31 sheets after the ARB is very probably connected with the elastic anisotropy of the hard and soft direction in the basal plane [15].

Note that the samples after the ECAP processing exhibited significant thermal stresses (see Figure 2). These stresses may be different for various processing routes. Shortening of the AZ31 samples (originally 20 mm in length) after the first pass achieved ~ 600 µm, then decreased, and again increased in the 8th pass. The permanent deformation is different for the samples prepared with diverse processing routes. Observed results are a consequence of the different texture developed in various processing routes and elastic anisotropy of the hexagonal magnesium cell.
6 Conclusions

Sever plastic deformation may substantially refine the microstructure of Mg alloys. On the other hand, the significant texture developed in materials depends on the processing technique and composition of alloys. This texture is reason for the plastic anisotropy, tension-compression asymmetry, and anisotropy of thermal properties. It is necessary to bear in mind these findings in various structural applications of magnesium alloys submitted to severe plastic deformation.

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References
[1] Estrin Y and Vinogradov A 2013 Acta Mater. 61 782
[2] Li S, Yang X, Hou J and Du W 2020 J. Magnes. Alloys 8 78
[3] Trojanová Z, Halmeshová K, Džugan J, Palček P, Minárik P. Lukáč P 2018 Letters on Materials 8 278
[4] Trojanová Z, Drozd Z, Máthis K, Köver M, Džugan J, Lukáč P, Halmeshová K 2019 Adv. Mater. Lett. 10 887
[5] Trojanová Z, Halmeshová K, Drozd Z, Šíma V, Lukáč P, Džugan J, Minárik P 2018 Crystals 8 278
[6] Trojanová Z, Halmeshová K, Džugan J, Drozd Z, Minárik P, Lukáč P 2020 Crystals 10 497
[7] Trojanová Z, Džugan J, Halmeshová K, Németh G, Lukáč P, Minárik P, Bohlen J Materials 2018, 11(1) 73
[8] Trojanová Z, Drozd Z, Škraban T, Minárik P, Džugan J, Halmeshová K, Németh G, Lukáč P, Chmelík F 2020 Adv. Eng. Mater. 22 1900596
[9] Lee S, Ham H J, Kwon S Y, Kim S W, Suh C M 2013 Int. J. Thermophys. 34 2343
[10] von Mises R 1928 Z. Anwew. Math. Mech. 8 161
[11] Máthis K, Čapek J, Zdražilová Z, Trojanová Z Mater. Sci. Eng. A 2011 528 5904
[12] Lumley RN, Polmear IJ, Groot H, Ferrier J Scripta Mater. 58, 2008, 1006
[13] Uher C. Thermal conductivity of metals. In: Thermal Conductivity. Theory, Properties and Applications. Tritt, M. Ed. Kuwen Academic/Plenum Publishers, New York, 2004
[14] Bass J, Fisher KH Landolt-Börnstein database 1982 New Series III/15a Springer
[15] Drozd Z, Trojanová Z, Halmeshová K, Džugan J, Lukáč P, Minárik P Acta Physica Polonica A 2018 134 820