Anisotropy of mechanical and thermal properties of AZ31 sheets prepared using the ARB technique

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Abstract. In the accumulative roll bonding (ARB) technique, repeated stacking of material followed by conventional roll-bonding is carried out. For this process the surfaces are cleaned with ethanol and then joined together by rolling. The rolled material is then cut into two halves, again surface treated and roll-bonded. This process may be repeated several times. For the magnesium alloy AZ31 (Mg-3Al-1Zn) rolling at an elevated temperature of 400 °C is necessary for ARB because of the low plasticity of hexagonal magnesium alloys at lower temperatures. Samples for this study were prepared using 1 to 3 ARB passes through the rolling mill. It was found that the ARB substantially refined the grain size of sheets to the micrometer scale. The microstructure and texture of the deformed samples were studied by light and electron microscopy. The mechanical properties of the ARB samples were explored using tensile test-pieces cut from the sheets with the tensile axis taken either parallel or perpendicular to the rolling direction, where a significant anisotropy in both mechanical properties and Young’s modulus was found. Anisotropy is explained on the basis of the specific microstructure and texture formed during the ARB process.

1. Introduction
Accumulative roll bonding is very effective approach to refine the grain structure and improve mechanical properties of a metallic material. Relatively big sheets may be prepared using this technology. In comparison with ECAP this method is also cheaper [1]. Severe plastic deformation of magnesium alloys is real problem because of the low plasticity at lower temperatures of hexagonal magnesium materials. For compatible deformation of polycrystals the activity of five independent slip systems is necessary [2]. In the hexagonal close packed cell with the basal slip (0001)/(1120) only two independent crystallographic equivalent systems exist and therefore non-basal slip (prismatic, pyramidal) or mechanical twinning must be activated. Shortage of easy glide slip systems in
magnesium is the reason of the limited plasticity of hexagonal magnesium alloys. Originally, the ARB process should be performed at temperatures below the recrystallization temperature. Because of low plasticity in magnesium materials this is however not possible and a higher process temperature must be used to achieve good formability. Despite this, substantial grain refinement and improvement of mechanical properties has been achieved via ARB in pure Mg [3], and in magnesium alloys of the AZ and AM series [4-7]. The ARB process influences not only the mechanical properties of magnesium materials but also other physical properties and a distinctive anisotropy is observed. The objective of this paper is to show this anisotropy and to attempt to explain observed phenomena.

2. Experimental results

2.1. Material and methods

Commercially available sheets of AZ31 magnesium alloy (nominal composition 3Al-1Zn-0.2Mn-balance Mg in wt%) were used in this study. Two sheets were rolled together in a four-high rolling mill. The rolling speed was 0.4 ms\(^{-1}\). Prior to rolling, the sheets were ground and cleaned. The resulting surfaces were thus free from oxides and had sufficient roughness, which is essential to making a high-quality joint. In addition, the sheets were riveted together along one side. The initial sheet thickness was 2 mm and the rolling reduction was 50% in each pass. The rolling process took place at 400 °C. We performed one, two and three passes through the rolling mill. Samples were hereafter designed as ARB\(_0\) (as received sheet) ARB\(_1\) (after one pass through the rolling mill), ARB\(_2\) (two passes) and ARB\(_3\) (three passes). Samples from sheets were cut so that the longer sample axis was either parallel (L orientation) or perpendicular (T) to the rolling direction.

![Figure 1. Three planes used for microstructure observation.](image)

The microstructure of the samples was studied using light microscopy (OLYMPUS and NEIPHOT) and using a FEI Quanta 200 FX scanning electron microscope (SEM) equipped with EDAX EBSD system; OIM software was utilized for to analyse EBSD data. The step size used for EBSD measurements was 0.8 μm. Intermetallic phases were analysed in a TESCAN VEGA LMU II SEM.

Tensile tests were performed in an INSTRON 5486 machine with an initial strain rate in the order 10\(^{-3}\) s\(^{-1}\) at room temperature on miniature tensile test specimens using a recently developed test method [8-15].

The thermal diffusivity was measured with the laser-flash method (Linseis LFA 1000) in the temperature range between 20°C and 350°C. The measurements were done in vacuum with a Nd:YAG laser; the temperature rise on the sample surface was measured with a liquid-nitrogen cooled infrared detector. Thermal diffusivity coefficient \(a\) corresponds to the halftime of temperature increase according to the following formula: \(a = 0.1388 \cdot \ell^2 / t_{0.5}\), where, \(\ell\), is the thickness of the specimen and \(t_{0.5}\) is the time at 50% of temperature increase.

The Young’s modulus was measured in resonant frequency and damping analyser (RFDA) system. Samples were excited to vibrations in the resonant frequency using a small striker. Free vibrations of the sample were registered with a microphone and a fast Fourier transform was used for estimation of the resonant frequency.

2.2. Microstructure of samples

The microstructure of samples was studied on the three principal planes indicated in figure 1. An EBSD map of the as prepared sheet is shown in figure 2(a). The microstructure (estimated from the A plane) of the as prepared sheet (figure 2(a)) contains a non-uniform grain structure - bigger grains are surrounded by small grains. A typical feature of this rolled sheet is an abundance of twins. Substantial
Refinement was achieved by the ARB procedure. Very fine grains are visible in figure 2(b, c, d). The similar colour of grains in the map indicates that during the rolling process a certain texture was developed. The grain size decreased to several µm. The microstructure of the as-prepared sheet taken from the B plane is shown in figure 3. Bigger particles elongated in the rolling direction were analysed using line EDX spectroscopy, as shown in figure 5. Bright particles represent an Al–Mn binary phase. In the literature, these particles were identified as Al₈Mn₅ and Al₁₁Mn₄ phases [16,17]. Also, particles containing Si were found. No particles of γ-phase Mg₁₇Al₁₂ were found in ARB sheets. Bonding of sheets was visible on both B and C plane. After three passes through the rolling mill 7 new interfaces were formed as seen in figure 4 (viewed on the C plane).

![Figure 2](image1.png)

**Figure 2.** Microstructure of sheets taken from plane A: ARB_0 (a), ARB_1 (b), ARB_2 (c), ARB_3 (d).

![Figure 3](image2.png)

**Figure 3.** SEM micrograph taken from plane B of the ARB_0 sample.

![Figure 4](image3.png)

**Figure 4.** New interfaces formed in the ARB_3 sample visible on the C plane.

Pole figures documenting the texture developed during rolling and ARB procedure in samples are shown in figure 6. The calculated textures show that the basal planes (0001) of the grains are preferentially oriented parallel to the rolled sheet surface (A plane) with the ⟨11̅20⟩ direction oriented along the rolling direction.

2.3. Mechanical tests

True stress-true strain curves obtained at room temperature for ARB_0, ARB_1 and ARB_2 samples cut in the L orientation (tensile axis parallel to the rolling direction) are shown in figure 7(a). ARB results in an increased deformation stress. The difference between curves for ARB_1 and ARB_2 samples is smaller compared with the difference between ARB_0 and ARB_1. Similar curves were obtained for samples cut in the T orientation (see figure 7(b)). The strengthening effect of the ARB
process is more significant for this orientation. The strengthening effect of the ARB process is also obvious from table 1. Both the yield and ultimate tensile strength increase with the number of passes for each sample orientation. Note, that the ductility of samples is relatively high, much higher compared with cast materials.

![Diagram]

**Figure 5.** Mapping of chemical elements in bright particles (a) visible in (b).

![Diagram]

**Figure 6.** Pole figures for ARB_0 (a), ARB_1 (b), ARB_2 (c) and ARB_3 (d); samples, taken from the A plane.
Table 1. Yield stress (YS) and ultimate tensile strength (UTS) estimated for T and L sample orientations.

| Number of passes | YS (L) | YS(T) | UTS(L) | UTS(T) |
|------------------|--------|-------|--------|--------|
| 0                | 162.5  | 85.0  | 307.0  | 297.0  |
| 1                | 223.8  | 172.4 | 344.9  | 365.6  |
| 2                | 227.0  | 212.0 | 356.6  | 370.0  |

Figure 7. True stress-true strain curves obtained for ARB_0, ARB_1 and ARB_2 samples in the L orientation (a) and T orientation (b).

Figure 8. Dependence of Young’s modulus on the number of passes for samples in the L and T orientations.

Figure 9. Dependence of thermal diffusivity on the number of passes.

2.4. Young’s modulus

The dependence of the Young’s modulus on the number of passes is shown in figure 8 for samples in both the L and T orientations. A significant planar anisotropy is obvious. While the modulus decreases with increasing number of passes, the modulus for the L orientation decreases only for 1 and 2 passes, and after the third pass in the rolling mill it increases again.
2.5. Thermal diffusivity
Thermal diffusivity was measured perpendicular to the surface of sheets, i.e. perpendicular to the L-T plane. The capability of the measuring apparatus did not enable the measurements in the B and C planes to be made. Results of the diffusivity dependence on the number of passes are shown in figure 9. Surprisingly thermal diffusivity increases with increasing number of passes even though new interfaces were introduced into samples during ARB.

3. Discussion
The von Mises equivalent strain, $\epsilon$, accumulated during the ARB process can be calculated [18] based on fact that the von Mises strain for 50% rolling is 0.8, for three passes the equivalent strain is relatively high at a value of 2.4. The grain size decreases with the number of passes. The majority of this decrease is observed after the first pass. The influence of the second and third pass is weaker, and the grain size decreased down to a stabilised value of 4.5±0.5 µm. The microstructure after ARB process was significantly more homogenous than in the as-prepared sheet. Grain refinement contributed to the observed increase of deformation stresses due to Hall-Petch strengthening:

$$\sigma = m (\tau_0 + k_\sigma d^{-1/2})$$

where $m$ is the Taylor orientation factor and $k_\sigma$ gives a measure of the shear stress concentration required in the average instance to propagate the plastic flow across grain boundaries [19, 20]. The main grain refinement occurred between the ARB 0 and ARB 1 sheets and therefore the highest strength increase was observed after the first ARB pass. Subsequent passes in the roll mill refined the grains structure only slightly and the strength increase is also only moderate. The grain refinement influenced deformation stresses of samples cut in the rolling as well as perpendicular directions. The grain refinement may be described as continuous rotational dynamic recrystallisation (RRX). This mechanism involves dynamic polygonisation of rotated lattice regions adjacent to the grain boundaries [21, 5]. Deformation characteristics, introduced in table 1, show significant planar anisotropy. Values estimated for the T orientation are lower compared with the L orientation. This anisotropy decreases with the number of passes. While the yield stress in the T orientation increased after first pass two times, in the L orientation it increased only by 23 %.

The observed planar anisotropy is a consequence of the deformation texture [22, 23]. Mechanisms of plastic deformation strongly depend on the initial texture [24]. The series of pole figures for ARB samples given in figure 6(a-d) show the texture development during the ARB process. AZ31 magnesium alloys sheets usually exhibit a typical rolling texture in which most grains have their basal planes parallel to the sheet surface. Normally the basal plane texture increases with increasing rolling reduction [25]. From figure 6(b-d) it follows that the basal texture is not ideal; the distribution of basal poles shows a basal pole tilted away from the normal direction towards the transverse direction. The deformation process starts in each grain by the activation of basal slip. Balík et al., studying the in-plane deformation of AZ31 alloy sheets, estimated that rapid hardening took place in the basal plane at the beginning of plastic deformation [22]. The deformation continues in the secondary regime where the double prismatic slip is the dominant mechanism. On the other hand deformation twinning as a prevailing mechanism may be considered at the beginning of deformation process in the transverse direction. Acoustic emission measurements performed in situ confirmed the formation of twins during deformation of samples in the T orientation. The twinning activity depends on the grain size and decreases with decreasing grain size [26]. This is also reason why the planar anisotropy decreases after the first and second pass through the rolling mill.

The Young’s modulus measurements in figure 8 also show significant planar anisotropy. This anisotropy decreases with increasing number of passes. While the modulus measured for the L orientation was lower in comparison with the T orientation, after three passes in the rolling mill the tendency is opposite. This behaviour can be related to the texture development during ARB. Calculation of the elastic modulus for textured materials using Reuss or Voigt models requires a detailed analysis of texture [27, 28]. The elastic modulus of pure Mg and alloys with addition of Al and Zn atoms was studied by Sumimoto et al. in [29]. They estimated the Young’s modulus of pure
Mg to be 43.2 GPa, and that it did not depend on the grain size. Al and Zn solutes with concentration lower than 2 at.% increased the elastic modulus up to approximately 44 GPa. This value is between two limiting values estimated for the samples in L and T orientations. A more developed texture decreases the elastic moduli in both orientations for the first and third rolling pass. The third pass in rolling mill increased again the modulus in the L orientation.

Thermal conductivity may be calculated from thermal diffusivity using the simple relation:

\[ \kappa = a_d \rho c_h \]  

where \( a_d \) is the thermal diffusivity, \( \rho \) is the density and \( c_h \) specific heat. The thermal conductivity can be attributed to heat transported along with the conduction of electrons and from phonons travelling through the lattice: \( \kappa = \kappa_e + \kappa_{ph} \). The lattice conductivity \( \kappa_{ph} \) decreases with increasing disorder in the lattice, i.e., with increasing concentration of lattice defects (vacancies, dislocations, grain boundaries) [30]. In metals the electron thermal conductivity \( \kappa_e > \kappa_{ph} \). Bass [31] estimated that the electrical conductivity of a magnesium single crystal along the \( c \) axis is higher than in the \( a \) direction, i.e., \( \kappa_e(c)/\kappa_e(a) > 1 \). At given temperature the thermal conductivity is proportional to the electrical conductivity, i.e., the same proportionality is expected for the thermal conductivity. In the textured sheet the hexagonal cells are mostly oriented such, that the \( c \) axis is perpendicular to the sheet surface. Thermal conductivity was measured in the same orientation, i.e., mostly in the \( c \) direction. The texture strengthening after the first and second passes increased the number of grains oriented with the \( c \) axis perpendicular to the sheet surface, thus also resulting in an increase in the thermal conductivity.

4. Conclusions

The microstructure and mechanical properties of an AZ31 magnesium alloy deformed by ARB to up to 3 passes have been examined in detail. Typical features of the ARB sheets are a fine grain structure and a developed crystallographic texture. Together these features account for the main influence on the material properties. The following key points can be concluded from the study:

- The ARB process substantially refined the grain structure.
- A planar anisotropy of mechanical properties is observed with the tensile yield strength for the transverse direction lower than that for tension parallel to rolling direction. This anisotropy was less pronounced after the second pass through the rolling mill.
- The Young’s modulus measured in the rolling direction is lower in comparison with the modulus measured in the transversal direction. This tendency was reversed after the third pass through the rolling mill.
- The thermal diffusivity of ARB sheets increases with the number of passes. This is due to development of a pronounced rolling texture, where the materials \( c \) axis is mostly perpendicular to the sheet surface.

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