

1 Article

## 2 Thermal conductivity of an AZ31 sheet after 3 accumulative roll bonding

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12 **Abstract:** Accumulative roll bonding (ARB) is one of methods of severe plastic deformation which  
13 is relevant for industrial production of sheets. While mechanical properties of several magnesium  
14 alloys subjected to ARB process have been studied, the physical properties have been reported only  
15 for some magnesium alloys. These properties are influenced by the texture developed during the  
16 ARB process and the temperature load. In the presented contribution, we studied thermal  
17 diffusivity and thermal conductivity of an AZ31 magnesium alloy after 1 and 2 passes through the  
18 rolling mill. Thermal diffusivity was measured with the laser-flash method in the temperature  
19 range between 20 and 350 °C. Thermal conductivity depends on the number of rolling passes. The  
20 microstructure and texture of sheets are significant factors influencing the thermal properties.

21 **Keywords:** magnesium alloy; accumulative roll bonding; thermal conductivity; texture  
22

### 23 1. Introduction

24 Mg-Al-Zn alloys due to their low density and high specific strength belong to the most used  
25 magnesium materials. The strength of these alloys increases with increasing Al content. An AZ31  
26 alloy can be applied as wrought alloy. The mechanical properties of Mg-Al-Zn alloys can be  
27 improved using methods of severe plastic deformation (SPD) due to Hall-Petch strengthening.  
28 Several SPD methods have been described among them methods based on the rolling process as  
29 differential speed rolling (DSR) [1, 2], single roller drive rolling (SRDR) [3] or accumulative roll  
30 bonding (ARB) [4-6]. ARB allows preparing big sheets with a predetermined thickness. On the other  
31 hand, such materials exhibit developed planar anisotropy of mechanical properties as a consequence  
32 of the texture formed during the rolling process. The texture influence on properties of hexagonal  
33 magnesium materials is significant due to large anisotropy of magnesium itself. The anisotropy of  
34 mechanical properties has been reported several times in the literature [7-9]. On the other hand, the  
35 developed texture affects not only mechanical properties (strength, modulus) but also physical  
36 properties (thermal expansion coefficient, internal friction, electrical resistivity). The effect of the  
37 texture on the thermal properties was studied only rarely [10-13].

38 Thermal properties of alloys belong to the important characteristics of structural materials.  
39 Dimensional stability and distribution of the heat in the thermally loaded devices are problems of  
40 the highest priority. In this study the temperature dependence of the thermal conductivity of a  
41 magnesium alloy AZ31 is measured with the aim to estimate the effect of the ARB process on the  
42 thermal conductivity.  
43

## 44 2. Materials and Methods

45 AZ31 (commercial purity) magnesium alloy sheets with initial thickness of 2 mm were used in  
 46 this study. Table 1 shows the chemical composition of the used AZ31 alloy. Before the rolling, the  
 47 surfaces of the sheets were wire brushed and clean with acetone to obtain free from oxide surface  
 48 with sufficient roughness for high-quality joints. To set the cleaned surfaces in contact the sheets  
 49 were riveted along one side. Then the sheets were annealed at 400 °C for 15 min. Four-high rolling  
 50 mill configuration with the rolling speed of 0.4 ms<sup>-1</sup> and 50% rolling reduction in each pass was used.  
 51 We performed up to 2 passes through the rolling mill. ARB\_2, resp. ARB\_1 indicates the samples  
 52 subjected to 2, resp. 1 pass of the ARB process. ARB\_0 stands for the as-received sheet.

53  
54 Table 1: Chemical composition of the alloy in wt%.

Al	Zn	Mn	Si	Ce	Fe	Mg
3.16	1.29	0.41	0.01 <sub>5</sub>	0.05 <sub>5</sub>	0.02	Bal.

56  
57 Laser flash method (LINSEIS LFA 1000) was applied for the thermal diffusivity and specific heat  
 58 measurements from 20 up to 350 °C in vacuum. The dimensions of the specimens were 12.7 mm in  
 59 diameter and 2 mm in thickness and the measurement was performed across the sample thickness.  
 60 The samples were coated with graphite on both sides to ensure homogenous absorption of the laser  
 61 energy. At the same time four samples were measured three times, for each sample the median of  
 62 the three values was taken and then the mean value and the standard deviation was calculated. The  
 63 main principle of the thermal diffusivity measurements is to measure the temperature rise on the  
 64 rear side of the samples with liquid-nitrogen cooled infrared detector after the laser pulse. Then, the  
 65 thermal diffusivity coefficient  $a$  is calculated according to the formula:  $a = 0.1388\ell^2/t_{0.5}$ , where  $\ell$  is the  
 66 thickness of the specimen and  $t_{0.5}$  is the time at 50% of temperature increase. For the specific heat  
 67 measurement comparison method was used. Pure Molybdenum (3N8) was used as a reference  
 68 sample. Then, the specific heat coefficient corresponds to the total temperature increase according to  
 69 the formula:  $c_p = c_{pr}(\Delta T_r / \Delta T)(m_r / m)$ , where  $c_p$ ,  $c_{pr}$  are the specific heats of sample and reference  
 70 sample;  $\Delta T$ ,  $\Delta T_r$  are temperature increases induced by laser pulse;  $m$ ,  $m_r$  are the masses of the sample  
 71 and reference sample, respectively.

72 The thermal expansion coefficient,  $\alpha$ , was measured using a LINSEIS L75PT-1600 dilatometer at  
 73 heating and cooling rates of 1 °C/min in a resistance furnace in an argon protective atmosphere.  
 74 Length changes were measured by linear variable differential transformer (LVDT) sensor through  
 75 quartz push-rods. Al<sub>2</sub>O<sub>3</sub> reference was used for correction measurements.

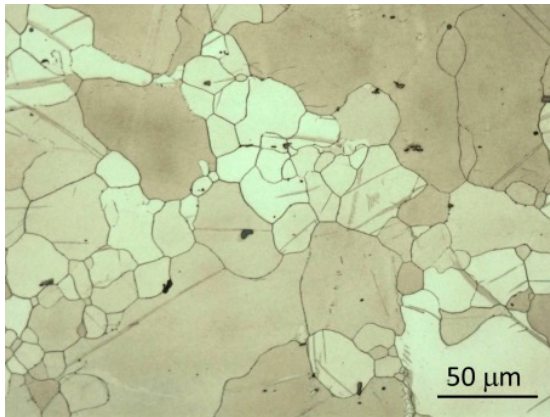
76 Density of samples was estimated by the hydrostatic weighing at room temperature.  
 77 Microstructure characteristics and texture of samples were studied using light microscopes  
 78 (OLYMPUS and NEOPHOT) and by a FEI Quanta 200 FX scanning electron microscope equipped  
 79 with EDAX EBSD system; orientation imaging map software was utilized for EBSD observations.  
 80 The step size used for EBSD measurements was 0.8 μm.

## 81 3. Results

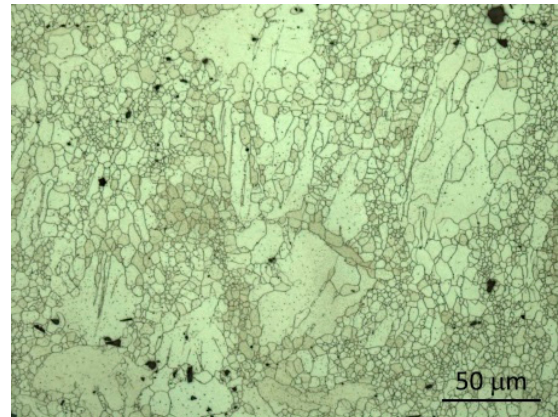
### 82 3.1. Microstructure of samples

83 Light micrographs taken from the sheets surface of the ARB\_0, ARB\_1 and ARB\_2 samples are  
 84 depicted in Figs. 1, 2 and 3. Non-uniform grain structure consisting of bigger grains surrounded by  
 85 small grains was found in the rolled sheet (Fig. 1). After the first ARB process the grain structure was  
 86 refined as it is obvious from Fig. 2, anyway it remained still non-uniform. The result of the second  
 87 ARB pass is shown in Fig. 3. As can be seen the microstructure is nearly uniform with fine grains.

88

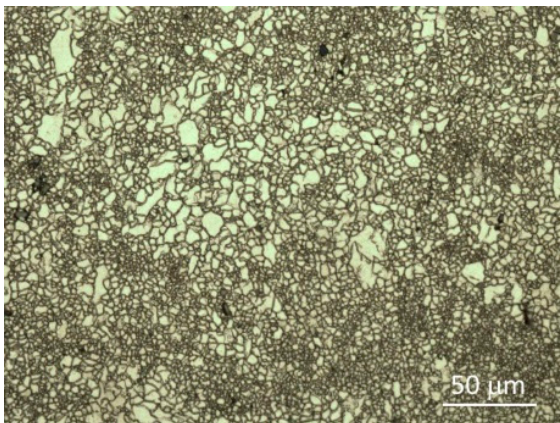


**Figure 1.** Microstructure taken from the ARB\_0 surface

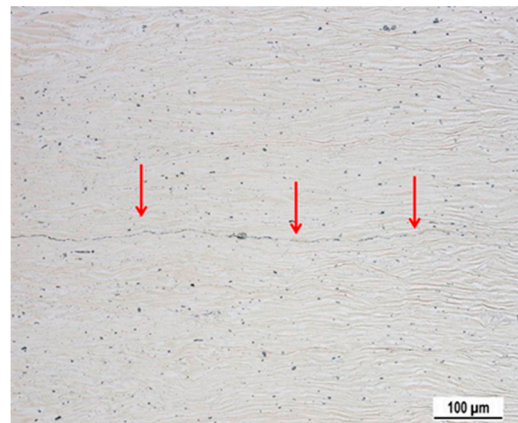


**Figure 2.** Microstructure taken from the ARB\_1 surface.

89



**Figure 3.** Microstructure taken from the ARB\_2 surface.



**Figure 4.** New interface formed in the ARB\_1 sample.

90

91 Particles visible in Fig. 1-3 were analyzed using EDX line spectroscopy. Particles represent an  
 92 Al-Mn binary phase. In the literature, these particles were identified as  $Al_8Mn_5$  and/or  
 93  $Al_{11}Mn_4$  phases [14,15]. New interface formed in the ARB process is visible in the light micrograph  
 94 presented in Fig. 4.

### 95 3.2 Thermal measurements

96 The thermal conductivity  $\kappa$  (W/(m K)) can be calculated according to known relationship

97

$$98 \quad \kappa = a \cdot \rho \cdot c_p, \quad (1)$$

99 where  $a$  is the thermal diffusivity ( $\text{cm}^2/\text{s}$ ),  $\rho$  is the density ( $\text{kgm}^{-3}$ ),  $c_p$  is the specific heat capacity  
 100 (J/kg/K) at constant pressure. Taking into account the thermal expansivity of the sample thickness  
 101 and volume

$$102 \quad \ell = \ell_{20} [1 + \alpha \cdot (T - 20)], \quad (2)$$

103 we obtained for the sample density following relationship

104

$$105 \quad \rho = \rho_{20} / [1 + \alpha \cdot (T - 20)]^3. \quad (3)$$

106 The thermal conductivity can be calculated according to formula:

107

$$\kappa = \frac{0.1388 \cdot t_{20}^2}{t_{0.5}(T)} \rho_{20} c_p(T) / [1 + \alpha \cdot (T - 20)] \quad (4)$$

109 Experimental values of the specific heat,  $c_p$ , and the thermal expansion coefficient,  $\alpha$ , used for the  
 110 calculation, are reported in Table 2.

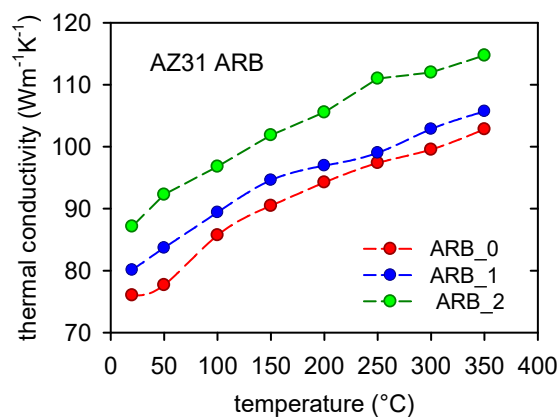
111

112 **Table 2.** Specific heat,  $c_p$ , and thermal expansion coefficient,  $\alpha$ , estimated at various temperatures.  
 113

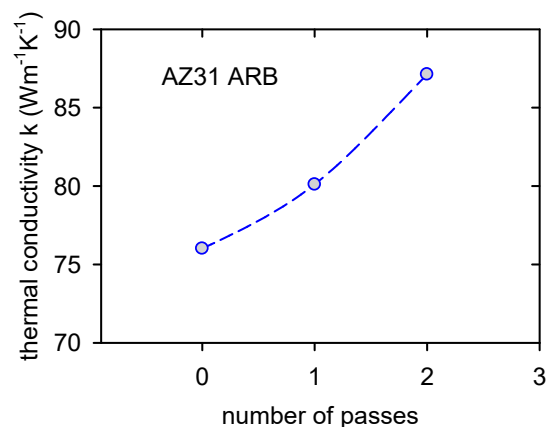
Material	ARB_0		ARB_1		ARB_2	
T (°C)	$c_p$ (Jkg <sup>-1</sup> K <sup>-1</sup> )	$\alpha \times 10^{-6}$ (K <sup>-1</sup> )	$c_p$ (Jkg <sup>-1</sup> K <sup>-1</sup> )	$\alpha \times 10^{-6}$ (K <sup>-1</sup> )	$c_p$ (Jkg <sup>-1</sup> K <sup>-1</sup> )	$\alpha \times 10^{-6}$ (K <sup>-1</sup> )
20	1010	26.53	1002	26.27	1036	25.73
50	1012	26.72	1037	26.47	1087	25.94
100	1060	27.04	1070	26.80	1101	26.31
150	1090	27.36	1107	27.12	1133	26.67
200	1112	27.68	1120	27.45	1159	27.04
250	1130	27.99	1113	27.77	1186	27.40
300	1161	28.31	1146	28.10	1186	27.77
350	1184	28.63	1166	28.43	1203	28.13

114

115 The temperature dependence of the thermal conductivity is shown in Fig. 5. The thermal  
 116 conductivity increases with increasing number of passes at all temperatures. This is better seen in  
 117 Fig. 6 where the thermal conductivity, measured at room temperature, rapidly increases with  
 118 increasing number of passes.  
 119



**Figure 5.** Temperature dependence of thermal conductivity.

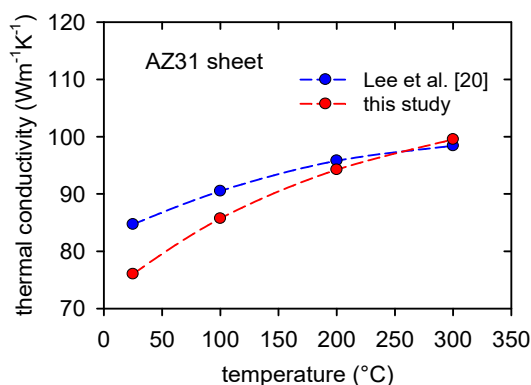


**Figure 6.** Thermal conductivity estimated at room temperature depending on number of passes.

#### 120 4. Discussion

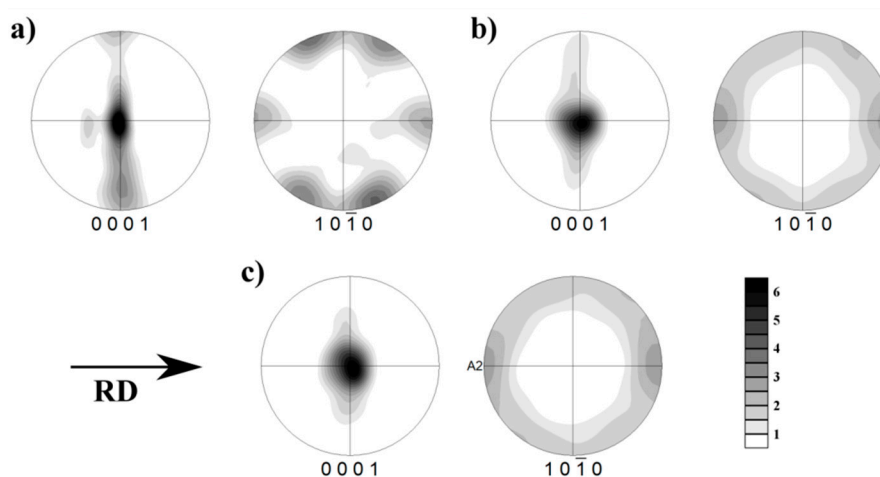
121 The conductivity of magnesium alloys has been reported in many papers. Mostly, binary and  
 122 ternary magnesium alloys consist of  $\alpha$ -grains containing the solid solution of the alloying elements  
 123 in Mg and various phases. Generally, the thermal conductivity depends on their composition and  
 124 thermal history [9,11,13-25]. Results of these studies were ambiguous. Ying et al [9] studied extruded  
 125 Mg-Al alloys in the temperature interval from room temperature up to 250 °C. While the thermal  
 126 conductivity of pure Mg and Mg-0.5at.%Al decreases with increasing temperature, the thermal  
 127 conductivity of Mg-0.9at.%Al remains almost unchanged, and the thermal conductivity of

128 Mg-1.5at.%Al decreases with increasing temperature. Similar results were found for Mg-Zn alloys  
 129 [11]. For Zn contents higher than 1wt.%, the thermal conductivity increases with increasing  
 130 temperature from 303 up to 513 K. Precipitation of the second phase particles increases the thermal  
 131 conductivity due to purification of the matrix [21]. The thermal conductivity of an AZ31 alloy  
 132 prepared by twin roll casting was measured in a wide temperature range from -125 °C up to 400 °C  
 133 [21]. The thermal conductivities estimated by Lee et al. in [20] and our results of ARB\_0 are shown in  
 134 Fig.7. It can be seen that obtained results are very close even of different manufacture conditions  
 135 were used.



136 **Figure 7.** Thermal conductivity of rolled sheets.

137 As it was shown in several papers, the microstructure and microstructure changes influence the  
 138 thermal conductivity [12,13,16-25]. The main changes in the microstructure occurred during the ARB  
 139 process are grain refinement and development of the texture. While the grain refinement influence  
 140 on the thermal conductivity was found as an effect of the second order [15], the influence of texture  
 141 was observed only rarely in the literature [10-13,26]. Rolled sheets from magnesium materials  
 142 exhibit a typical texture, where basal planes (0001) are mostly parallel to the sheet surface. Observed  
 143 textures of our samples are presented in Fig. 8a for ARB\_0, Fig. 8b (ARB\_1) and Fig. 8c (ARB\_2)..



144  
 145 **Figure 8.** Texture of ARB\_0 (a), ARB\_1 (b) and ARB\_2 samples (c).

146 After the repeated rolling in the first and second pass the grain refinement occurred in a  
 147 continuous rotational dynamic recrystallization (RRX) process. This mechanism involves dynamic  
 148 polygonization of rotated lattice regions adjacent to the grain boundaries [26,27]. The texture after  
 149 the first and the second pass is nearly perfect (Fig. 8), i.e. the most grains are oriented with their basal

150 planes parallel to the sheet surface. The results presented in this study show that discussion of the  
151 thermal conductivity of magnesium sheets exhibiting certain texture in terms of polycrystalline  
152 quasi-isotropic materials is not correct.

153 The thermal conductivity can be attributed to the heat transport by electrons and by phonons. When  
154 both can be considered as independent then:  $\kappa = \kappa_e + \kappa_{ph}$ . The maximum in the phonon part  $\kappa_{ph}$  is  
155 typically at temperature about  $0.1\theta_D$ ,  $\theta_D$  is the Debye temperature of a material ( $\theta_D(\text{Mg})=318$  K) [28].  
156 It means that the measured thermal conductivity in our case can be attributed to the electronic  
157 contribution  $\kappa_e$ . According to the Wiedemann-Franz law [29]

$$158 \quad \kappa_e = L_0 \sigma T, \quad (5)$$

159 where  $\sigma$  is the electrical conductivity and  $L_0$  is the standard Lorentz number and  $T$  absolute  
160 temperature. For highly degenerated systems such as metals  $L_0 = 2.45 \times 10^{-8} \text{ W } \Omega \text{ K}^{-2}$  [30]. Supposing a  
161 weak temperature dependence of electrical conductivity due to temperature independent scattering  
162 on impurities and defects [31], we can simply explain the measured increasing temperature  
163 dependence of the thermal conductivity (Fig. 5).

164 In a conductor, the electronic conductivity  $\kappa_e$  (either electric or thermal) is done as

$$165 \quad J = \kappa_e E, \quad (6)$$

166 where  $J$  is the flow density (electric current density or heat flow density) and  $E$  the field (electric field  
167 or temperature gradient). Generally the conductivity is a tensor. Non scalar conductivity can be  
168 found also in materials with hexagonal symmetry. Bass [32] estimated that the electrical conductivity  
169 of a magnesium single crystal along the  $\langle c \rangle$  axis is higher than that in the  $\langle a \rangle$  direction, i.e. we can  
170 also expect for the thermal conductivity, i.e.  $\kappa(c)/\kappa(a) > 1$ .

171 In the textured sheet, the hexagonal cells are preferentially oriented such that the  $\langle c \rangle$  axis is  
172 perpendicular to the sheet surface. The thermal conductivity was measured in the same orientation,  
173 i.e. in directions preferentially close to the  $\langle c \rangle$  direction. The texture strengthening after the first and  
174 second passes increased the number of grains oriented with the  $c$  axis perpendicular to the sheet  
175 surface, therefore, resulting in an increase in the thermal conductivity. In Mg where basal planes are  
176 close packed, the mean free path of electrons is higher in the  $\langle c \rangle$  direction than in the basal plane.  
177 This simple view of the nature of the conductivity anisotropy is supported by the fact that the  
178 anisotropy of electrical conductivity of hexagonal metals is higher for those with a higher  $\langle c \rangle / \langle a \rangle$   
179 ratio [32].

## 180 5. Conclusions

181 Thermal conductivity of AZ31 magnesium alloy was measured in the temperature range from  
182 room temperature up to 350 °C. Samples for the conductivity measurements were prepared by  
183 accumulative roll bonding. The conductivity measurements were performed in the direction  
184 perpendicular to the sheet surface.

- 185 • ARB process refined the sheets microstructure.
  - 186 • Rolled sheets exhibit developed texture where basal planes (0001) are preferentially parallel to  
187 the sheet surface.
  - 188 • Thermal conductivity increases with temperature and increasing number of rolling passes.
  - 189 • Observed increase of thermal conductivity with the increasing number of rolling passes can  
190 be explained with the texture improvement and anisotropy of thermal properties of  
191 magnesium.
  - 192 • This anisotropy can be of advantage in cases where the heat dissipation occurs in one  
193 direction.
- 194

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198

199 **Author Contributions:** Zuzanka Trojanová conceived and designed the experiments; Kristýna Halmešová and  
200 Ján Džugan performed ARB procedure; Zdeněk Drozd and Kristýna Halmešová measured thermal properties;  
201 Peter Minárik studied microstructure and texture of alloys; Zuzanka Trojanová and Vladimír Šíma analysed the  
202 data; Zuzanka Trojanová and Pavel Lukáč wrote the paper.  
203

204 **Conflicts of Interest:** The authors declare no conflict of interests.

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