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## Mechanical properties and structure evolution of the AZ91 magnesium alloy after hot rolling and annealing

### Właściwości mechaniczne oraz struktura stopu AZ91 po walcowaniu na gorąco i wyżarzaniu

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#### **Abstract**

The AZ91 magnesium alloy was processed up to 87.5% of total thickness reduction in several thermodynamic routes, consisted of hot rolling and intermediate annealing. The hot-rolling process was performed at a high strain rate equal to  $1.6\text{ s}^{-1}$  and at a temperature of  $430^\circ\text{C}$ . The intermediate annealing was performed at  $430^\circ\text{C}$  for 15 minutes after each route. It was found that, during hot rolling, the hardness of the material increased from 32 HV to 40 HV, and the structure investigations showed a huge amount of twins formed inside the grains (which were not observed after annealing). Tensile tests have shown strong anisotropy in mechanical properties of the “as-rolled” samples dependent on the orientation between tension direction (TD) and rolling direction (RD). The samples with TD perpendicular to RD proved higher ultimate tensile strength (UTS) and (on the other hand) worse plastic properties as compared to the samples with TD parallel to RD. The annealing has an effect on the reduction of mechanical properties anisotropy. X-ray investigations have shown texture changes from the basal type with the additional (0001)  $\langle 11\bar{2}0 \rangle$  component for “as-rolled” samples to the texture with the main (0001)  $\langle 10\bar{1}0 \rangle$  component for annealed samples. The texture changes had a great impact on the anisotropy of mechanical properties of the investigated AZ91 magnesium alloy.

**Keywords:** magnesium AZ91, hot rolling, texture, annealing, mechanical properties

#### **Streszczenie**

Stop magnezu AZ91 był walcowany na gorąco ze zgniotem 87.5% w kilku operacjach walcowania-wyżarzania. Walcowanie na gorąco było przeprowadzone w temperaturze  $430^\circ\text{C}$  z prędkością odkształcenia  $1.6\text{ s}^{-1}$ . Wyżarzanie międzyoperacyjne przeprowadzano w temperaturze  $430^\circ\text{C}$  przez 15 minut. Po walcowaniu twardość stopu AZ91 wzrosła od wartości 32 HV do wartości 40 HV, a obserwacje struktury wykazały obecność wewnątrz ziaren dużej liczby bliźniaków. Po

przeprowadzeniu wyżarzania (15 minut, 430°C) nie zaobserwowano obecności bliźniaków. Wyniki badań z próby rozciągania wykazały silną anizotropię własności mechanicznych w zależności od ułożenia kierunku rozciągania (KR) próbek wtórnych względem kierunku walcowania (KW). Przeprowadzone wyżarzanie spowodowało zmniejszenie anizotropii właściwości mechanicznych w badanym materiale. Przeprowadzone badania rentgenowskie wykazały zmiany w teksturze z typu bazalnego z dodatkowym komponentem (0001)  $\langle 11\bar{2}0 \rangle$  dla próbek po walcowaniu na typ tekstury z głównym komponentem (0001)  $\langle 10\bar{1}0 \rangle$  dla próbek wyżarzanych. Zmiany tekstury miały silny wpływ na zaobserwowaną anizotropię właściwości mechanicznych badanego stopu AZ91 po walcowaniu na gorąco i wyżarzaniu.

**Słowa kluczowe:** stop magnezu AZ91, walcowanie na gorąco, tekstura, właściwości mechaniczne

## 1. Introduction

Magnesium alloys are widely used for many applications as functional materials; e.g., in industries such as automotive, aerospace, or electronics [1] because of their low density and high specific strength. Recently, more and more new applications for magnesium alloys have been found [2, 3]. Magnesium alloys enable us to supply materials for medical applications or hydrogen storage, especially when the material is processed by severe plastic deformation (SPD) methods; e.g., high-pressure torsion (HPT) or equal-channel angular pressing (ECAP) [2]. However, low ductility (especially at room temperature), relatively poor mechanical properties at higher temperatures, and poor corrosion resistance limit the use of magnesium alloys [4]. Magnesium is a light metal with a low-temperature melting point and, therefore, is rather soft, so plastic deformation processes can be applied to improve the mechanical properties of magnesium alloys. On the other hand, magnesium alloys have limited ductility due to the anisotropy of their hexagonal structure [5]. Therefore, strong textures (e.g., basal during rolling [6] or double peaked in the basal plane during extrusion [7]) are formed. This is an important factor that effectively reduces the use of magnesium alloys [5–7]. To enhance the ductility and reduce the anisotropy of the mechanical properties of magnesium alloys by means of weakening the basal texture, the temperature of the process can be increased. Thus, processes like hot rolling or hot extrusion may be applied [8, 9]. But the high temperature of processing annihilates the hardening effects of plastic deformation [10]. To enhance the ductility of magnesium alloys at lower temperatures, solutions such as changing the deformation path [11], high speed rolling [12, 13], or SPD [14] can be applied. A very interesting method was developed by Zhu [12], where magnesium alloy ZK60 was processed by rolling at high strain rates, resulting in the activation of twins. This method proves very promising, because twins are more sensitive to strain rate than temperature. Thus, magnesium alloys may be processed at high temperatures to achieve good plastic properties and at high strain rates to produce twins having an impact on mechanical properties. In addition, enhancing the mechanical properties of magnesium alloys can be achieved by solid-solution strengthening or second-phase hardening [15, 16].

In this paper, the AZ91 magnesium alloy was processed by hot rolling at a high strain rate equal to  $1.6 \text{ s}^{-1}$ , where twinning plays an important role in deformed magnesium alloys. The tensile tests, hardness, structure observations, and texture investigations are aimed at checking the impact of the structure and texture on the anisotropy of the mechanical properties of AZ91 hot-rolled at high-strain rate.

## 2. Experiments

Magnesium alloy AZ91 (with nominally 9% aluminum and 1% zinc) was hot rolled up to 87.5% of total thickness reduction in several thermodynamic treatments. Before processing, the “as-cast” material was pre-heated for an hour at a temperature of  $430^\circ\text{C}$ . After that, the material was hot rolled in three passages. Each route consisted of annealing for 15 minutes at  $430^\circ\text{C}$  followed by rolling at a  $1.6 \text{ s}^{-1}$  strain rate with 50% thickness reduction in the current passage. The rolling direction (RD) remained the same during the whole rolling process. After rolling, several investigations were performed, such as observation microstructure, tensile and hardness tests, and X-ray measurements of texture. The investigations were performed on samples “as-rolled” and annealed at  $430^\circ\text{C}$  for 15 minutes and 1 hour respectively. To observe the structure, a GX51 Olympus optical microscope was used. Based on the data, the mean grain size was calculated using the straight-line method. The hardness properties were investigated by using a Vickers micro-hardness Instron Wolpert Testor 2100, with an applied load of 50gf. Tensile test samples were cut from the “as-rolled” and annealed material in the way that rolling direction (RD) was parallel with tensile directions (TD) in the first case, and in the second, RD was perpendicular to TD (as shown in Figure 1).

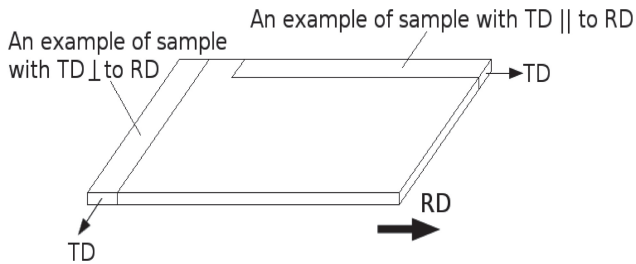


Fig. 1. A method cutout samples  $\text{TD} \parallel \text{RD}$  and  $\text{TD} \perp \text{RD}$  to the tensile tests

The tensile tests were performed at room temperature at a strain rate of  $10^{-3} \text{ s}^{-1}$ . The samples had  $30 \times 6 \times 2 \text{ mm}^3$  in dimensions, and the gauge length was 20 mm. An Instron 5566 testing machine was used. The texture measurements were performed on samples cut from “as-rolled” and annealed pieces, respectively. An X-ray diffractometer Bruker D8 Advance with Cu-K $\alpha$  radiation operating at 30 kV was used. For each sample subjected

to texture measurements, the  $\{0001\}$ ,  $\{10\bar{1}0\}$ ,  $\{11\bar{2}0\}$ , and  $\{10\bar{1}1\}$  pole figures were measured (with step size  $5 \times 5$ ). Using the MTEX application under MATLAB, the calculated pole figures were generated and corrected by the defocusation data.

### 3. Results and discussion

The initial Vickers micro-hardness for “as-cast” material (for “as-rolled” AZ91 and for “as-rolled” followed by annealing at  $430^\circ\text{C}$  for 15 minutes and for an hour respectively) are shown in Figure 2. This proves that, during annealing, hardness does not change significantly. The grain size for “as-rolled” is  $228 \mu\text{m}$ , and the increase in grain size was observed up to  $251 \mu\text{m}$  and  $376 \mu\text{m}$  during annealing at  $430^\circ\text{C}$  for 15 and 60 minutes, respectively.

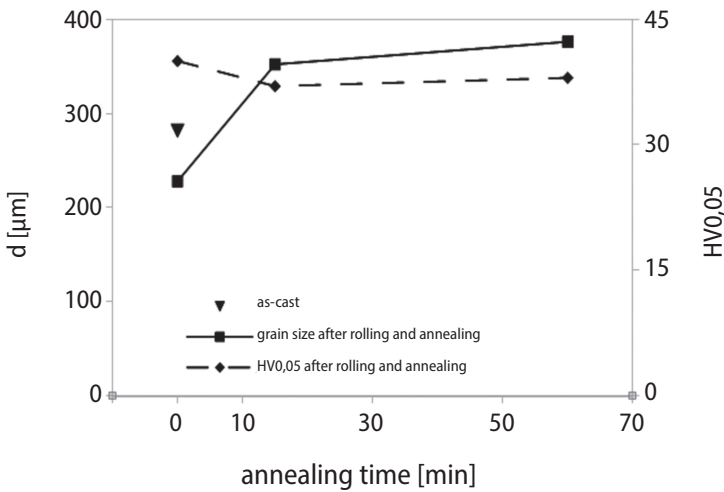


Fig. 2. Average grain size  $d$  and Vickers micro-hardness  $HV0.05$  for “as-rolled” AZ91 and annealed at  $430^\circ\text{C}$

The increase in hardness for the “as-rolled” sample can be attributed to the formation of a large number of twins during deformation, which can be seen in Figure 3a. Most of the twins is arranged perpendicular to the RD.

After annealing at  $430^\circ\text{C}$  for 15 minutes, the twins are not observed (as can be seen in Figure 3b). In Figure 3b, large grains are surrounded by small grains, while in Figure 3c, only big grains can be observed. Very similar effects were observed by del Velle et al. [17], where the small grains resulted from the dynamic recrystallization process (DRX) during the hot rolling of AZ61. Ion et al. [18] found that the areas with small grains play an important role in the plasticity of magnesium as “ductile shear zones” where the deformation is localized. However, in this study, such a structure is a result of the recrystallization after rolling. The nature of the formation-observed structure needs further investigation.

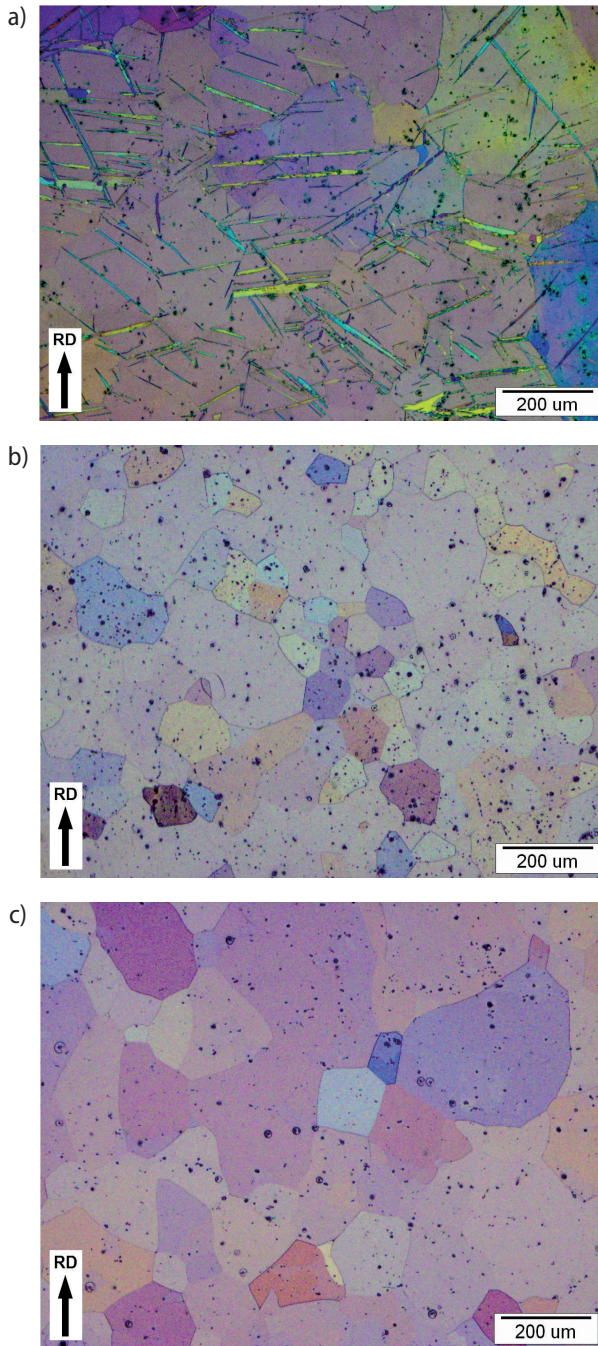


Fig. 3. The microstructure of: a) for “as-rolled”; b) “as-rolled” followed by annealing for 15 minutes at 430°C; c) “as-rolled” followed by annealing for an hour at 430°C

In Figure 4, the work-hardening curves obtained from tensile tests of the samples from AZ91 processed by hot rolling and annealed are presented.

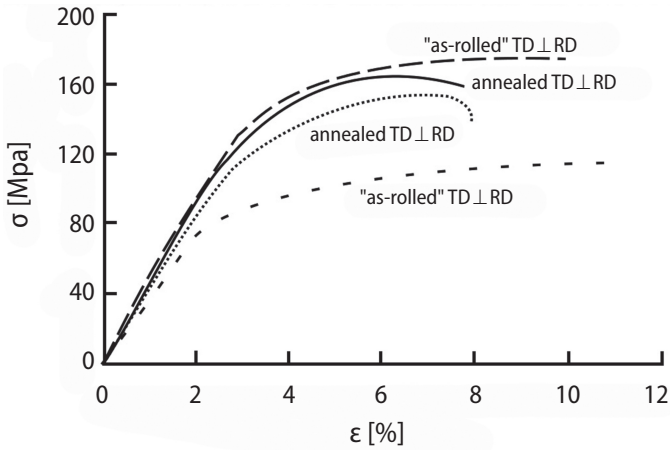


Fig. 4. Work-hardening curves from tensile tests of investigated AZ91 after hot rolling and annealing for an hour

The best mechanical properties, such as unlimited tensile strength (UTS) equal to 175 MPa and yield point ( $R_{p0.2}$ ) equal to 130 MPa, present the “as-rolled” sample where TD is perpendicular to RD. Moreover, the “as-rolled” sample where TD is parallel to RD presents the lowest values of  $R_{p0.2}$  and UTS. Such strong anisotropy of mechanical properties for magnesium alloys has been previously reported [19, 20]. Partially, the reason for this is the formation of very strong textures that reduce the activation of non-basal slip systems and twinning [6–12, 19, 20]. The effect of texture on mechanical properties will be discussed later in this paper. Furthermore, after annealing for 60 minutes, the anisotropy was reduced while only slight differences in  $R_{p0.2}$ , UTS and elongation ( $A_5$ ) were observed. The data is summarized in Table 1, where  $R_{p0.2}$ , UTS, and  $A_5$  are shown.

Table 1. The values of  $R_{p0.2}$ , UTS, and  $A_5$  for the samples of AZ91 subjected for tensile tests

Property	“as-rolled” TD    RD	annealed TD    RD	“as-rolled” TD ⊥ RD	annealed TD ⊥ RD
$R_{p0.2}$ [MPa]	75	110	130	111
UTS [MPa]	117	152	175	163
$A_5$ [%]	7,9	4,5	6,1	4,3

Shown in Figures 5a and 5b are the orientation distribution (ODF) obtained from the texture investigations, sections  $\varphi_2$  for the “as-rolled” and annealed for an hour samples, respectively.

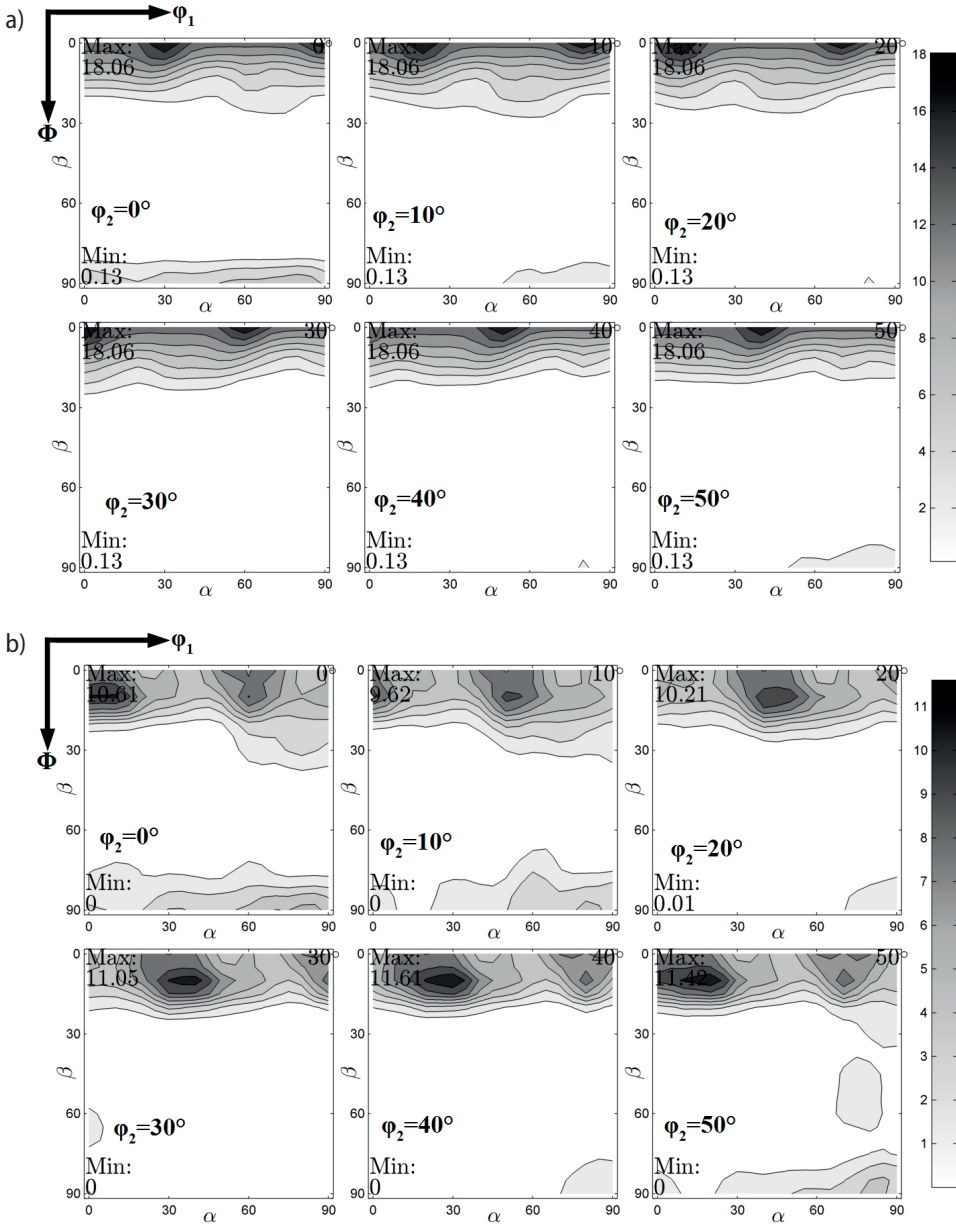


Fig. 5. ODF of the AZ91 “as-rolled” (a) and annealed-for-an-hour (b) samples, section  $\varphi_2$

From Figure 5a (according to Wang [21]), it can be seen that the texture for “as-rolled” samples is a basal-fiber type with an additional  $(0001) \langle 11\bar{2}0 \rangle$  component, while after annealing, the texture developed to the type where the main component is  $(0001) \langle 10\bar{1}0 \rangle$ .

During the deformation along RD, magnesium alloys with a basal fiber texture basal slip system is the most active (the case of “as-rolled” sample with RD  $\parallel$  TD in Figure 4) [5, 22]. In the case of the deformation where TD is perpendicular to RD, twinning is the most active deformation mechanism [22]. Twins enhance the plasticity of magnesium and its alloys; after the twinning is exhausted, pyramidal slip systems are activated, leading to increase the work-hardening characteristics [22]. However, in “as-rolled” samples, twins are already introduced by the rolling process, and while RD  $\perp$  TD, deformation must be accomplished by hard pyramidal slip systems (the case of “as-rolled” sample with RD  $\perp$  TD in Figure 4). On the other hand, in the annealed samples, most of the grains are oriented in such a way that the  $\langle 11\bar{2}0 \rangle$  direction is parallel and  $\langle 10\bar{1}0 \rangle$  is perpendicular to RD. In the case of deformation along  $\langle 10\bar{1}0 \rangle$  or  $\langle 11\bar{2}0 \rangle$  while  $[0001]$  is parallel to normal direction (ND), pyramidal slip systems are activated followed by twinning, and the work-hardening curves are similar in these two cases [5, 22] (both cases of “annealed” samples with RD  $\parallel$  TD and RD  $\perp$  TD in Figure 4).

The relation between strong basal texture and the anisotropy of mechanical properties in magnesium alloys has been previously observed by many researchers, and the reduction of anisotropy was related to the reduction of a strong basal texture [8, 17]. However, in this study, the reduction of mechanical property anisotropy is related to the change of the main texture components and its effect on the activation of twinning and pyramidal slip systems.

## 4. Conclusions

- Twins formed during hot rolling of AZ91 at 430°C caused an increase of hardness.
- “As-rolled” samples show strong anisotropy of mechanical properties dependent on the relation in orientation between TD and RD.
- The texture for “as-rolled samples” is a basal-fiber type with the additional  $(0001) \langle 11\bar{2}0 \rangle$  component.
- The main component of the texture for annealed samples is  $(0001) \langle 10\bar{1}0 \rangle$ .
- The change of texture from basal-fiber with  $(0001) \langle 11\bar{2}0 \rangle$  component to the  $(0001) \langle 10\bar{1}0 \rangle$  type texture had an effect on the reduction of mechanical property anisotropy of hot-rolled and annealed AZ91 magnesium alloy.

## Acknowledgment

The work was supported from the statutory activity of the Department of Non-Ferrous Metals, University of Science and Technology in Krakow, Poland (under agreement 11.11.180.653).



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