THE INFLUENCE OF PREHEATING TEMPERATURE ON THE MECHANICAL PROPERTIES OF AZ31 MAGNESIUM ALLOYS AFTER HOT-ROLLING

MILOŠ JANEČEK[1]*, VLADIMÍR ŠUPÍK[2], FRANK HOLLÄNDER[2], JOCHEN WENDT[2]

[1] *Charles University, Faculty of Mathematics and Physics, Department of Metal Physics, Ke Karlovu 5, CZ-121 16 Prague 2, Czech Republic*
[2] *Department of Physical Metallurgy and Materials Technology, Technical University of Brandenburg at Cottbus, D-03010 Cottbus, Germany*

Received 15 February 2005, accepted 19 April 2005

Sheets of magnesium alloys are prospective materials for the mass reduction in car manufacturing. Sheets of the wrought magnesium alloy AZ31 were produced using a hot-rolling mill. Mechanical properties of these sheets were investigated for various preheating temperatures of as-received extruded and cast materials. The optimum preheating temperature resulting in superior mechanical properties was identified. Transmission electron microscopy indicated the occurrence of discontinuous dynamic recrystallization during hot rolling of preheated material. The extruded alloy exhibited better mechanical properties and lower porosity than the cast alloy.

Key words: AZ31 alloy, hot-rolling, mechanical properties, microstructure, transmission electron microscopy

1. Introduction

Magnesium alloys are the lightest metallic structural materials and are therefore very attractive in applications in automobile, railway and aerospace industries where the mass reduction is an important issue [1]. However, owing to their hexagonal close-packed structure and a limited number of slip systems, Mg alloys exhibit only limited ductility and accommodation ability. Parts of these alloys for structural applications are often produced by casting with either no or very limited mechanical pressing [2–3]. This obviously limits the range of possible engineering applications of Mg alloys.

*corresponding author, e-mail: janecek@met.mff.cuni.cz

In the recent years, there has been a renewed interest in Mg alloy sheet products, especially in automotive applications where considerable weight savings can be made by substituting magnesium for aluminium or steel components. Mg--3Al-1Zn alloy (AZ31) is currently the most common Mg alloy used for sheet applications. Due to its poor cold rolling response it has to be rolled at elevated temperatures which considerably increases manufacturing costs of sheet products of this alloy [4]. Optimization of conditions of the hot rolling process is therefore a big challenge. The properties of the rolled sheet obviously depend on the initial state of the material, the temperature of plate before rolling and many other parameters [5, 6]. However, at present there is only a limited knowledge of this area. One of the possible ways how to improve the final properties of the rolled state is to optimize the conditions of the material preheating before rolling. Too high temperature of preheating increases the costs of the final sheet and too low temperature may result in the failure of the material during successive rolling.

The objective of this investigation is twofold:

– to critically evaluate the influence of the initial state of an alloy on the mechanical properties of rolled sheet, and

– to correlate the temperature of preheating with the mechanical properties and the microstructure of the rolled sheet with the aim to find the optimum temperature range of preheating.

2. Experimental

The magnesium alloy AZ31 whose nominal composition is given in Table 1 was received in the form of extruded rectangular bar stocks from Otto Fuchs KG Meinerzhagen, Honsel – Profilprodukte, Soest, Germany (labelled OF) and as a cast ingot from Technion, Haifa, Israel (labelled T). Rectangular samples of the dimensions of 40 mm × 60 mm × 20 mm were machined from both materials. Prior to rolling the samples were first preheated at various temperatures in the range of 325–475 ℃ [4, 7, 8]. The time of preheating was the same at all temperatures. Immediately after preheating the samples were rolled on a pilot hot-rolling mill at the temperature of rolls of 300 ℃. The rolling direction was perpendicular to the extrusion and/or cast direction. Twelve passes of rolling were applied. The thickness reduction between individual passes of rolling was kept constant. The final thickness after the reduction was 1 mm. The stripes had the width of 40 to 43 mm and the length of 950 to 1100 mm. They were compact with limited cracking on both sides of the rolled stripe. Crystallographic texture of rolled stripes was

Table 1. Nominal chemical composition of AZ31 alloy [wt.%]

Element	Al	Zn	Mn	Mg
c [wt.%]	3	0.8	0.2	balance

measured by X-ray diffractometer 3000 Seifert using reflection geometry and Ni--filtered Cu-Kα radiation. The microstructure of individual specimens after rolling was observed by light microscopy using a Zeiss-Axiomant 100 microscope. The observations were conducted in polarized light after coloured etching in picric acid etchant. Polarized light was used to reveal the orientation contrast. Grain size was measured by lineal intercept method.

Mechanical tests were carried out on the universal testing machine FP-100 (Rauenstein) at room temperature with the initial strain rate of 8.3×10^{-4} s^{-1}. Flat specimens of the gauge length of 20 mm were machined from hot-rolled stripes parallel to the rolling direction. For comparison, cylindrical specimens of the same gauge length were fabricated from the as-received material.

Transmission electron microscopy (TEM) investigations were performed with a Philips CM 200 electron microscope operated at 200 kV. Specimens for TEM were first mechanically polished on fine abrasive papers to the thickness of approximately 150 μm. Thin foils for TEM were finally electropolished in Tenupol 5 double jet polishing unit in the solution of LiCl + Mg perchlorate + methanol + buthyloxyethanol at -45 °C.

3. Results and discussion

3.1 Microstructure and texture of the initial state

The detail of the microstructure of the cross section of both as-received materials is shown in Fig. 1. Figure 1a presents the typical deformed inhomogeneous microstructure consisting of polyedric grains of the solid solution of Al and Zn in Mg. The grains are elongated in the direction of extrusion. Twins were found in some grains. On the other hand, regular equiaxed grains typical for the cast material are seen on the micrograph, Fig. 1b. Extensive porosity in the as-cast state is seen in this micrograph.

Slightly smaller grains were found in the extruded alloy (OF) than in the cast alloy (T). The average grain size measured in the middle of the cross section of extruded and cast material was about 200–300 μm (Fig. 1a) and 250–400 μm (Fig. 1b).

Texture measurements were conducted to determine the preferential lattice orientation of the initial extruded state and its change after rolling. Figure 2a presents typical (0002) pole figure of the extruded alloy (OF). The pole figure clearly indicates that in the as-fabricated state most grains are oriented with the basal planes parallel to the extrusion direction in the extrusion plane. In the as--rolled material (see Fig. 2b) the preferential orientation of the lattice with the basal plane parallel to the rolling plane can be observed. An isotropic distribution of the mechanical properties in the rolling plane may be therefore expected after rolling.

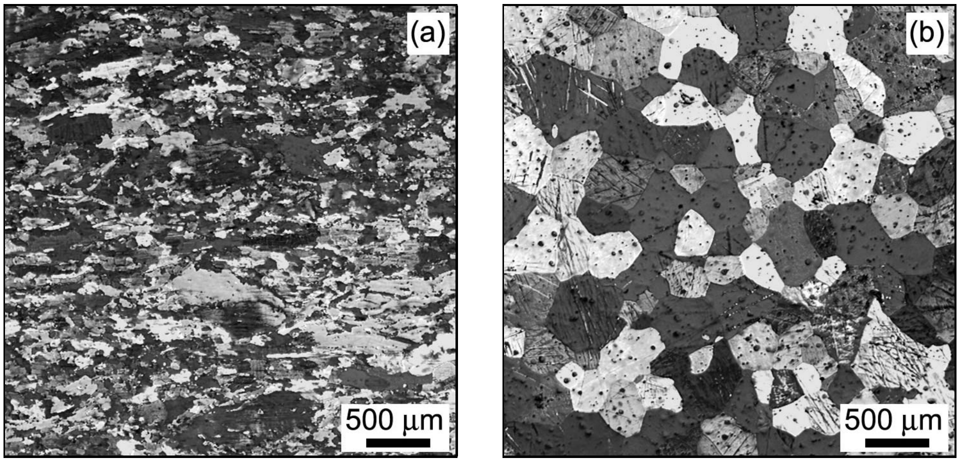

Fig. 1. Microstructure of the initial state of AZ31 alloy (polarized light). a) Extruded alloy (OF), b) cast alloy (T).

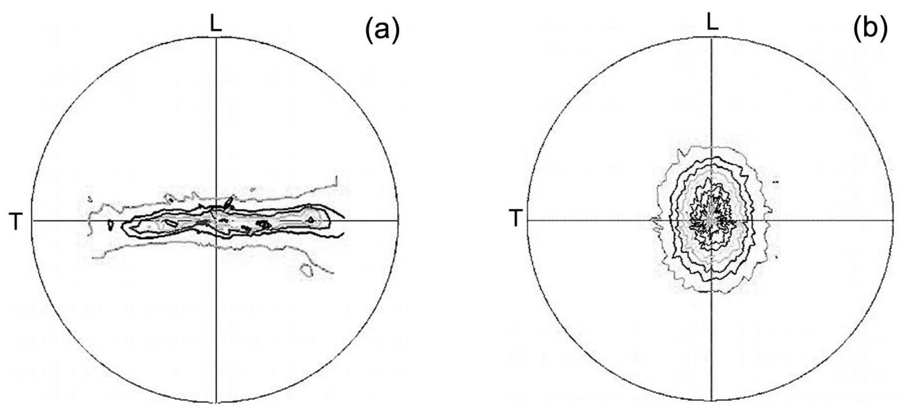

Fig. 2. (0002) pole figures of extruded AZ31 alloy (OF). a) As-extruded, b) as-rolled.

3.2 Mechanical properties – as-received state

In order to evaluate the anisotropy of mechanical properties tensile tests were performed at room temperature. Specimens for tensile tests were machined parallel to the longitudinal direction (marked L) of extrusion/casting and parallel to the long-transverse direction (marked LT). The results of the tensile tests are summarized in Table 2. The extruded alloy exhibits better mechanical properties. Both the

Table 2. Tensile properties of as-received AZ31 alloys (L – longitudinal direction, LT – longitudinal-transverse direction)

Material	$\sigma_{0.2}$ [MPa]		UTS [MPa]		ε_F [%]	
	L	LT	L	LT	L	LT
OF	210	140	280	255	15	11
T	170	150	250	230	16	11

yield stress and the ultimate strength of the extruded alloy are much higher than of the cast alloy. On the other hand, the ductility of both alloys was approximately the same.

Strong anisotropy of mechanical properties was found in the extruded alloy. For example, the yield stress of the L specimen was 1.5 times higher than of LT specimen. The difference in ultimate strength between these two specimens was 25 MPa. Anisotropic behaviour was also observed in the cast alloy. In this case, however, the difference of the yield stress and the ultimate strength between the L and LT specimen was much smaller. Anisotropic values of ductility were also found in both materials. Specimens parallel to the longitudinal direction exhibited higher elongation to fracture.

Anisotropic behaviour of the extruded alloy can be explained by the crystallographic texture which was confirmed by X-ray diffraction measurements – see Fig. 2. Weaker anisotropy of mechanical properties of the cast alloy is in accord with the weaker texture which can be expected in this material.

3.3 Mechanical properties – rolled states

The influence of the preheating temperature on the yield stress in both alloys after rolling is shown in Fig. 3a. Significantly higher stresses were found in the hot-rolled extruded alloy preheated at lower temperatures. The difference between both materials decreases with increasing temperature of preheating. At high temperatures above 425 ℃ the difference disappears and practically the same values of yield stress were observed in both alloys. The maximum of the yield stress was found at 350 ℃ in the extruded alloy. On the other hand, in the cast alloy the yield stress remains almost constant up to the temperatures of 400 ℃.

The dependence of the ultimate tensile strength on the temperature of preheating is presented in Fig. 3b. At lower temperatures ($T < 400$ ℃) the UTS of the extruded alloy is higher while above 400 ℃ it becomes lower than in the cast alloy. The UTS of the extruded alloy decreases monotonously in the whole temperature range. On the other hand, in the cast alloy a maximum of UTS was found at about 350 ℃. Above 350 ℃ the UTS in the cast alloy decreased with temperature at a slower rate than in the extruded alloy.

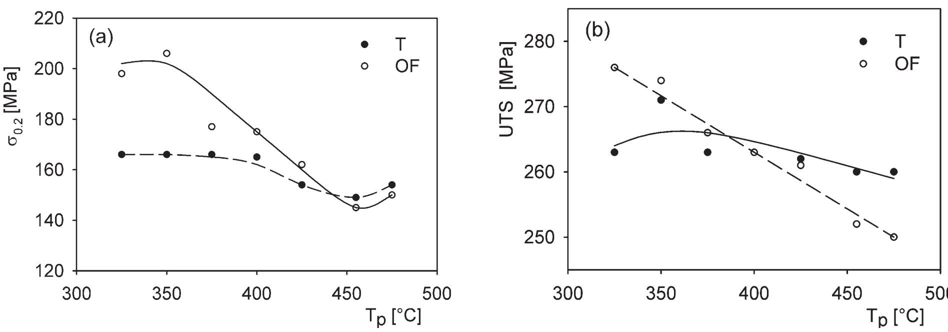

Fig. 3. Mechanical properties of AZ31 alloy. a) The dependence of the yield stress on the temperature of preheating, b) the dependence of the ultimate tensile stress on the temperature of preheating.

T a b l e 3. Tensile properties of the hot-rolled AZ31 alloys after preheating at various temperatures

Preheating T [°C]	$\sigma_{0.2}$ [MPa]		UTS [MPa]		ε_F [%]	
	OF	T	OF	T	OF	T
325	166	198	263	276	17	15
350	166	206	271	274	21	15
375	166	177	263	266	17	16
400	165	175	263	263	21	14
425	154	162	262	261	18	19
455	149	145	260	252	18	19
475	154	150	260	250	19	18

From the mechanical properties viewpoint, the temperature of 350 °C seems to be the optimum temperature for attaining the best mechanical properties of hot rolled sheets both in the extruded and the cast material.

All values evaluated from mechanical tests in both materials are summarized in Table 3. As seen from this table, elongation to fracture ε_F ranged between 17–21 % and 15–19 % in the extruded and cast alloy, respectively. Except for highest temperatures of preheating ($T > 400$ °C) the extruded alloy exhibited higher elongation to fracture.

The values in Table 3 also indicate that significant strain hardening occurs during room temperature deformation of hot-rolled specimens and is almost independent of the temperature of preheating before rolling. This is true in the extruded alloy where the difference between the UTS and the yield stress remains almost constant for all temperatures of preheating. On the other hand, in the cast alloy this difference slightly increases with increasing temperature of preheating. For higher temperatures of preheating, however, the difference remains almost constant and is approximately the same as in the extruded alloy ($\sim$ 100 MPa). Twinning is the most probable mechanism of plastic deformation at low temperatures and causes the strain hardening in these alloys [7, 8]. Further TEM study of deformed states is necessary to confirm this assumption. Such investigation is however beyond the scope of this paper.

3.4 Microstructure of rolled states

A comprehensive metallographic study of all specimens after rolling at 300 °C was conducted. Significant grain refinement after rolling was observed in all specimens. Two specimens corresponding to the lowest (325 °C) and highest temperature (475 °C) of preheating were selected as the representative states in which the microstructural changes during rolling are discussed.

The optical micrograph showing the microstructure of these two specimens is presented in Fig. 4. With increasing temperature of preheating the linear increase of grain sizes in the structure after rolling was found. The characteristic grain size after preheating at the lowest temperature (Fig. 4a (extruded alloy), 4b (cast alloy)) is almost two times smaller than in the specimen preheated at the highest temperature (Fig. 4c (extruded alloy), 4d (cast alloy)). The microstructure of all specimens is inhomogeneous containing two populations of grains which are typically arranged in bands. For example, the average grain size of fine grains in the extruded alloy preheated at 325 °C (Fig. 4a) and 475 °C (Fig. 4c) was 9 μm and 17 μm, respectively. The average grain size of coarse grains in the same material preheated to 325 °C was approximately 33 μm and increased to 71 μm in the specimen preheated to 475 °C. The finer grains of the average size of 10 μm were located in shear bands. These bands were aligned parallel to the rolling direction and/or inclined of approximately 45° with respect to the rolling direction. Two distinct directions of bands in hot rolled microstructures were also reported by other authors [11, 12].

Numerous cracks were observed in the cast alloy after rolling, particularly in specimens preheated at higher temperatures (above 350 °C) (see Fig. 4d). On the other hand, only fine cracks were found in the extruded alloy. Crack formation is probably caused by porosity which is more extensive in the cast alloy than in the extruded alloy.

A TEM micrograph of the extruded alloy preheated at 325 °C is presented in Fig. 5. It shows a zone of new equiaxed grains with relatively low density of dislocations. Electron diffraction analysis confirmed that most of these grains were aligned along [0001] zone axis. It indicates that the basal plane of hexagonal lattice, which was randomly oriented in the extruded material, rotated during rolling to the position parallel to the rolling plane. Twins were often observed in new grains.

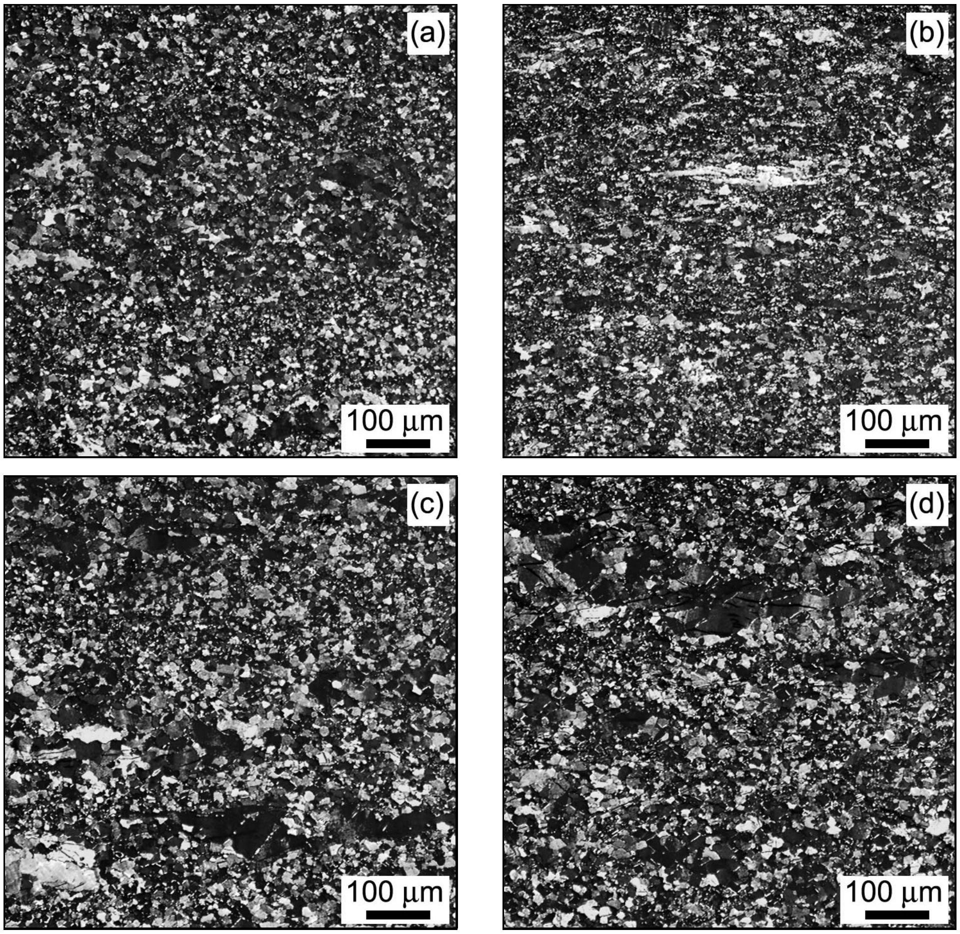

Fig. 4. The microstructure of hot-rolled AZ31 alloys after various preheating temperatures (polarized light). a) AZ31 (OF) 325 °C, b) AZ31 (T) 325 °C, c) AZ31 (OF) 475 °C, d) AZ31 (T) 475 °C.

Figure 6 represents a dark field image of such grain with a twin passing through it. Dislocations are clearly seen to pile-up against grain boundaries. In approximately 30 % of the observed area heavily deformed zones containing high density of tangled dislocations or subgrains with many dislocations in their interior were found in this specimen. A typical example of such subgrain is shown in Fig. 7. All observed microstructural features indicate that during rolling the dynamic recrystallization (DRX) must have occurred in this specimen.

In the rolled extruded alloy preheated at 475 °C only recrystallized grains were

Fig. 5. Bright field TEM micrograph of recrystallized grains in hot-rolled AZ31 (OF) preheated at 325 ℃ ($\mathbf{g} = (10\bar{1}0)$, $\mathbf{B} = [0001]$).

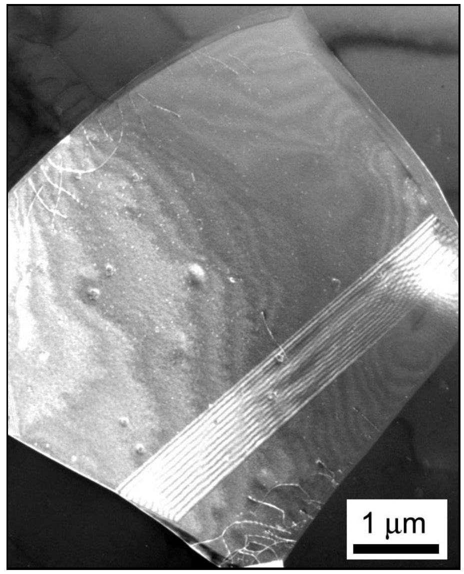

Fig. 6. Weak beam dark field TEM micrograph of a twin in the recrystallized grain in hot-rolled AZ31 (OF) preheated at 325 ℃ ($\mathbf{g} = (0\bar{1}11)$, $\mathbf{B} = [1\bar{5}49]$).

present in all areas of the specimen observed by TEM. Rather inhomogeneous structure consisting of grains of various sizes was found in this specimen. In some zones clusters of relatively small grains of the average size not exceeding 5 μm were observed. However, big grains of the size exceeding 20 μm were predominantly present in the microstructure. Dislocation free grains were predominantly found in this specimen. It indicates that the recrystallization process was completed after rolling.

The microstructure of the cast alloy after rolling is very similar to the microstructure of the rolled extruded alloy. In the specimen preheated at 325 ℃ the mixture of new grains and deformed zones with dislocations and subgrains were observed. Both these regions were in balance in this specimen. Figure 8 shows an example of the interface between the recrystallized and deformed zone. Extensive porosity was observed in all specimens of this cast alloy which contrasts to the extruded alloy, where the porosity was limited.

Completely recrystallized structure very similar to that of the rolled extruded alloy was found in the cast alloy preheated at 475 ℃ and rolled down.

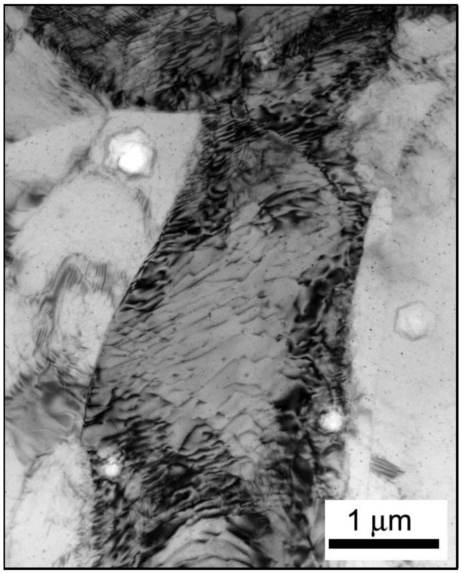

Fig. 7. TEM micrograph showing a subgrain in the hot-rolled AZ31 (OF) preheated at 325 ℃.

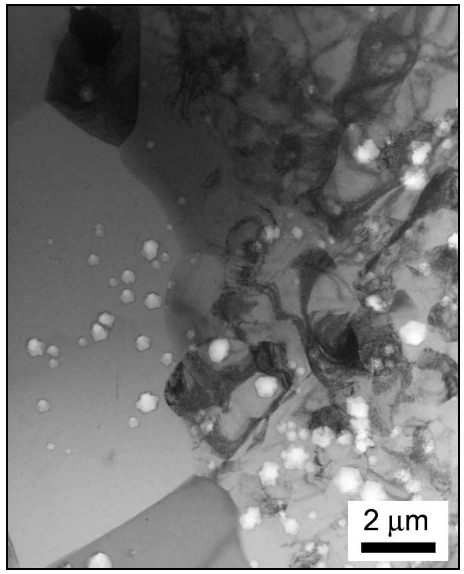

Fig. 8. Interface between recrystallized grains and still deformed zone in the hot-rolled AZ31 (T) preheated at 325 ℃.

4. Conclusions

The following conclusions may be drawn from this experimental investigation:

– Strong anisotropy of mechanical properties was found in the extruded alloy. Much weaker anisotropy was found in the cast alloy. After rolling the anisotropy was smeared out.

– The temperature of 350 ℃ seems to be the optimum preheating temperature for attaining the best mechanical properties of hot rolled sheets both from the extruded and the cast material.

– Significant strain hardening due to twinning occurs during room temperature deformation of hot-rolled specimens.

– Significant grain refinement was found after rolling. The temperature of preheating influences the final rolled structure which tends to coarsen with increasing T.

– Numerous cracks were observed in the cast alloy after rolling due to extensive porosity.

– Dynamic recrystallization occurs during rolling. The rate of DRX is enhanced by the temperature of preheating.

Acknowledgements

We would like to dedicate this paper to Prof. RNDr. Pavel Lukáč, DrSc., Dr.h.c. on the occasion of his 70^{th} birthday.

This work was financially supported by the research program MSM 0021620834 of Ministry of Education (M. J.) and by the BMBF through the project Magnesium im Automobilbau (MIA).

The authors would like to thank Otto Fuchs KG, Honsel – Profilprodukte, Germany and Technion, Haifa, Israel for providing the material for this investigation.

REFERENCES

[1] FRIEDRICH, H.—SCHUMANN, S.: Materialwissenschaft und Werkstofftechnik, *32*, 2001, p. 6.
[2] FRIEDRICH, H.—SCHUMANN, S.: Magnesium 2000. Eds.: Aghion, E., Eliezer, D. Beer-Sheva, Israel, MRI 2000, p. 9.
[3] LUO, A.: Magnesium Technology 2000. Eds.: Kaplan, H., Hryn, J., Clow, B. Warrendale, USA, TMS 2000, p. 89.
[4] EMLEY, E. F.: Principles of Magnesium Technology. Oxford, Pergamon Press 1966, p. 550.
[5] YIN, D. L.—ZHANG, K. F.—WANG, G. F.—HAN, W. B.: Mat. Sci. Eng., *A392*, 2005, p. 320.
[6] JÄGER, A.—LUKÁČ, P.—GÄRTNEROVÁ, V.: Kovove Mater., *42*, 2004, p. 165.
[7] ROBERTS, C. S.: Magnesium and its Alloys. New York, Wiley 1960.
[8] POLMEAR, I. J.: In: Magnesium alloys and their applications. Eds.: Mordike, B. L., Hehmann, F. Oberursel, DGM 1992, p. 201.
[9] TROJANOVÁ, Z.—GÄRTNEROVÁ V.—PADALKA, O.: Kovove Mater., *42*, 2004, p. 206.
[10] LAMARK, T. T.—CHMELÍK, F.—ESTRIN, Y.—LUKÁČ, P.: Kovove Mater., *42*, 2004, p. 293.
[11] BULIAN, W.—FAHRENHORST, E.: Metallographie des Magnesiums. Berlin, Springer-Verlag 1942.
[12] BECK, A.: Magnesium und seine Legierungen. Berlin, Springer-Verlag 2001.