

THE EFFECT OF THERMO-MECHANICAL PROCESSING ON THE STRUCTURE, STATIC MECHANICAL PROPERTIES AND FATIGUE BEHAVIOUR OF PURE Mg

VPLIV TERMOMEHANSKE PREDELAVE ČISTEGA MAGNEZIJA NA STRUKTURO, STATIČNE MEHANSKE LASTNOSTI IN OBNAŠANJE PRI UTRUJANJU

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Magnesium alloys find important applications in automotive, aircraft and space industries. Magnesium itself is also a key component in biodegradable metallic materials considered for applications in medicine. Although there are many studies that include the mechanical behaviour of pure Mg and its alloys after different thermomechanical processing, there is a huge impact of the processing method on the observed properties. Based on these facts, predicting the final properties is difficult. This paper deals with the preparation of pure Mg using a classic extrusion method with a low extrusion ratio, slow extrusion rate and low temperature. These parameters were selected to prevent an excessive grain size and to obtain optimal mechanical properties. The prepared materials were subsequently heat treated under different conditions and the relations between the grain size, texture strength and mechanical properties were studied. As well as static mechanical tests in tension and compression, fatigue tests were also performed. Finally, the obtained data were fitted using the Hall-Petch relation and the results were compared with the literature. The observed results confirmed the significant effect of processing on the mechanical properties with a strong impact on the Hall-Petch behaviour.

Keywords: magnesium, mechanical properties, texture, Hall-Petch relation

Magnezijeve zlitine so pomembne za uporabo v avtomobilski, letalski in vesoljski industriji. Magnezij je tudi ključna komponenta v biorazgradljivih kovinskih materialih za uporabo v medicini. Čeprav obstaja mnogo študij, ki vključujejo mehansko obnašanje čistega Mg in njegovih zlitin po različnih termomehanskih obdelavah, je velik vpliv metode obdelovanja na opazovane lastnosti. Na osnovi tega je težko napovedovati končne lastnosti. Ta članek obravnava pripravo čistega Mg s klasično metodo ekstruzije z majhnim razmerjem ekstruzije, majhno hitrostjo ekstruzije pri nizki temperaturi. Ti parametri so bili izbrani, da se prepreči prekomerna rast zrn in da se doseže optimalne mehanske lastnosti. Pripravljeni material je bil pozneje toplotno obdelan pri različnih pogojih in študirane so bile odvisnosti med velikostjo zrn, teksturo in mehanskimi lastnostmi. Razen statičnih mehanskih preizkusov, nateznih in tlačnih, so bili izvedeni tudi preizkusi utrujanja. Na koncu so bili dobljeni podatki primerjani s Hall-Petchevim razmerjem in dobljeni rezultati so bili primerjani s podatki iz literature. Dobljeni rezultati so potrdili močan vpliv obdelave na mehanske lastnosti, z močnim vplivom na Hall-Petchevo razmerje.

Ključne besede: magnezij, mehanske lastnosti, Hall-Petchevo razmerje

1 INTRODUCTION

Magnesium is an essential matrix element of many materials used in the traffic industry and also of some biodegradable materials that can be used for medical applications. Both these groups are characterized by high demands on the mechanical properties. These properties are strongly affected by many material factors. One of the very well-known factors is strengthening by grain boundaries, which is simply described by the Hall-Petch Equation (1):

$$\sigma = \sigma_0 + k \cdot d^{-1/2} \quad (1)$$

In this equation " σ_0 " and " k " are the constants representing the intercept and the slope of the Hall-Petch line, respectively. Magnesium as well as other metals with *hcp* structures have already been the subjects of many studies which showed that " σ_0 " is related to the

critical resolved shear stress (CRSS) for the easy slip system operating within the grain volume or in other words it represents the friction stress of mobile dislocations on the slip plane^{1,2}, and " k " is related to the CRSS for the more difficult slip/twinning systems required to operate near grain boundaries in order to maintain the continuity of strain³ or in other words " k " represents the stress-concentration factor.² Both these constants (" σ_0 " and " k ") are texture dependent.

Material texture can be significantly affected by different processing methods such as rolling, extrusion, forging and of course by the parameters of those processes.

Another Equation (2), which characterizes the dependence of polycrystalline strength " σ " on the critical shear strength " τ_0 " and the stress concentration required to propagate slip across a grain boundary " k_s " has to be

modified if we consider textured materials since the orientation constants relating " σ_0 " and " k " to the corresponding single-crystal resolved shear stresses will be different and depend on the intensity of the respective textures. In such case, Equation (2) can be modified to Equation (3), where " M " is the orientation factor relating " σ_0 " to the CRSS for an easy slip, and " N " is the orientation factor relating " k " to " k_s ":

$$\sigma = m \cdot (\tau_0 + k_s \cdot l^{1/2}) \quad (2)$$

$$\sigma = M \cdot \tau_0 + N \cdot k_s \cdot l^{1/2} \quad (3)$$

On the basis of the pile-up model, the Hall-Petch slope " k " is related to the resolved shear stress " τ_c " for the difficult slip system controlling plastic flow in the grain-boundary regions, Equation (4):

$$k = N \cdot k_s = C \cdot N \cdot [m \cdot Gb/2\tau a]^{1/2} \cdot \tau_c^{1/2} \quad (4)$$

In this Equation (4) " C " is a constant, " m^* " is the Sach's orientation factor that takes into account the differences in the grain-to-grain orientation, " a " is a numerical constant depending on the screw or edge character of the dislocation pile-up, " G " is the shear modulus, and " b " is the Burgers vector. Therefore, " k " is related to the CRSS for prismatic slip and the $\{10\bar{1}0\}$ texture can be expected to affect the value of the slope " k ". It is evident that the material properties are strongly texture dependent and the Hall-Petch behaviour can be different for materials prepared by different processing routes.

Due to the limited number of slip systems in *hcp* structures, other mechanisms such as twinning also play an important role in the deformation behaviour of magnesium and magnesium-based alloys. It has been shown that twin boundaries act as strong barriers to dislocation motion.⁴ Therefore, in practice, the mean free path of the dislocations should incorporate terms representing the effects of both the grain size and the presence of any twins. Several types of single- and double-twinning such as $\{10\bar{1}1\}$, $\{10\bar{1}2\}$, $\{10\bar{1}3\}$, $\{10\bar{1}1\}$ – $\{10\bar{1}2\}$ and $\{10\bar{1}3\}$ – $\{10\bar{1}2\}$ are formed in magnesium and its alloys,^{5–7} and the crystallographic relation of the lattice rotates around the $\langle 1\bar{1}20 \rangle$ direction by 56° , 86° and 64° for these types of single twinning formation. Among these types of twinning, the $\{10\bar{1}2\}$ -type ($\langle c \rangle$ -axis tension) twins form easily at the beginning of plastic deformation, since its CRSS is the lowest and has a similar order as that in the basal plane.^{5,6} The twinning is formed by the concentrated stress due to the dislocation pile-up, as reported in³. When the magnitude of the stress concentration is larger than the nucleation stress, twinning is thought to form at an adjacent grain.⁸ It has also been reported that twinning tends to form at grain boundaries with lower misorientation angles, which are characterized by a higher grain-boundary energy in magnesium.^{9,10}

Different methods can be used for the manufacture of magnesium alloys with a low grain size, and different textures are prepared during these processes. The main

examples of such methods are extrusion, ECAP (equal channel angular pressing) and HPT (high pressure torsion). Material prepared by original straight extrusion has a typical texture consisting of $\langle 10\bar{1}0 \rangle$ and $\langle 11\bar{2}0 \rangle$ fibres (i.e., basal poles perpendicular to the extrusion axis). ECAP textures and its strength are dependent on ECAP routes and also on the number of ECAP passes.

Final texture affects measured mechanical properties. After extrusion, if the tensile axis lies within the basal plane of the majority of grains, the grains are forced to deform by either non-basal cross-glide of basal dislocations or pyramidal slip of large Burgers vector $\langle c+a \rangle$ dislocations.¹¹ These hard deformation modes possess a high critical resolved flow stress, and ultimately result in the premature failure of properly oriented samples. In contrast, if the basal axes are oriented close to the tensile stress axis, there is very strong signature of the $\{10\bar{1}2\}$ tensile twinning $[11\bar{1}3]$. Twinning re-orientates a portion of the crystallites, and only a small plastic strain is required to ensure that a major volume fraction of grains becomes poorly oriented for slip or twinning. Therefore, the anisotropy of the mechanical properties observed in magnesium alloys with specific texture and both tensile and compressive properties are a distinctive base on the tests' orientation.

The present work is focused on a classic extrusion process with a low extrusion ratio equal to 10 and an extrusion rate only 5 mm/min. Together with a low temperature (200 °C), such conditions were selected to obtain fine-grained structures and proper mechanical properties.

2 MATERIALS AND METHODS

2.1 Materials

In this study, pure Mg (99.9 %) in an extruded state was investigated. Cylindrical ingots of Mg were prepared by melting pure Mg (99.9 %) in an induction furnace under a protective argon atmosphere (99.996 %). The melt was homogenised at 750 °C for 15 min and cast into a brass mould of 100 mm in length and 20 mm in diameter. Magnesium was directly extruded at a temperature of 200 °C, an extrusion rate of 5 mm/min, and an extrusion ratio of 10:1 to prepare rods of 6 mm in diameter. Such specimens were annealed at temperatures of (200, 300 and 400) °C for different times to prepare magnesium with different grain sizes and properties.

2.2 Structure

The microstructure of the Mg was studied by optical microscopy (Olympus PME3) and scanning electron microscopy (SEM – TescanVega3 LMU) equipped with an energy-dispersive spectrometer (EDS, Oxford Instruments Inca 350) and an EBSD detector – NordlysMax and AZtecHKL software. For SEM analysis, the samples

were mechanically grinded using SiC abrasive papers P180–P4000, polished by diamond pastes with 2 μm and 0.7 μm particles and etched in a 2 % Nital solution. For EBSD additional mechanical polishing was performed on a neutral alumina suspension (OP-AN Suspension). Finally, samples were prepared by electro-polishing in 15 % nitric acid in ethanol at 12 V for 15 s.

2.3 Mechanical properties

The mechanical studies were performed using Vickers hardness measurements with a loading of 5 kg (HV5). The mechanical properties were also characterised by tensile tests and compression tests (LabTest 5.250SP1-VM). For the tensile tests of the as-cast alloys, rod samples of 4 mm in diameter and 60 mm in length were used. The strain rate during the tensile tests was set to 0.001 s^{-1} . Compression tests were performed on rod samples of 6 mm in a diameter and 12 mm height at a strain rate of 0.001 s^{-1} as was used for the tensile tests.

Fatigue tests were performed on a device SCHNCK PHG. Smooth fatigue specimens with a diameter of 6 mm and a gauge length of 18.7 mm were machined from the extruded materials. Before the fatigue test they were mechanically polished using progressively finer grades of emery paper and buff-finished. The fatigue tests were performed at a frequency of 50 Hz in laboratory air at ambient temperature.

3 RESULTS AND DISCUSSION

3.1 Structure

Typical examples of microstructures of pure Mg obtained by different thermomechanical processing are

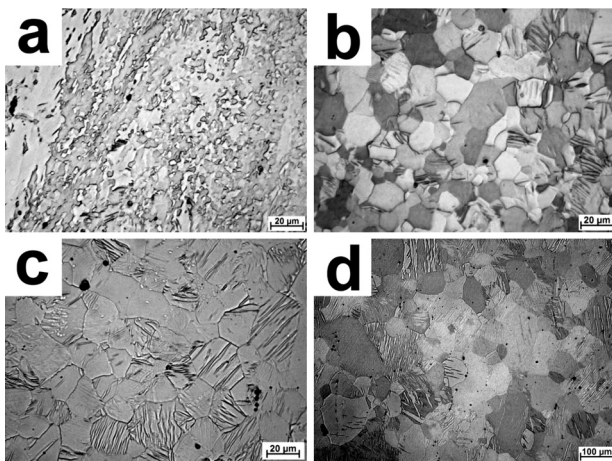


Figure 1: Structure of Mg after different thermo-mechanical processing: a) extruded at 200 °C, b) extruded at 200 °C and annealed at 200 °C for 1 h, refer to extrusion direction, c) extruded at 200 °C and annealed at 200 °C for 2 h, refer to extrusion direction, d) extruded at 200 °C and annealed at 400 °C for 2 h, refer to extrusion direction
Slika 1: Mikrostruktura v smeri ekstrudiranja Mg po različnih termomehanskih obdelavah: a) ekstrudirano pri 200 °C, b) ekstrudirano pri 200 °C in 1 h žarjeno na 200 °C, c) ekstrudirano pri 200 °C in 2 h žarjeno na 200 °C, d) ekstrudirano pri 200 °C in 2 h žarjeno na 400 °C

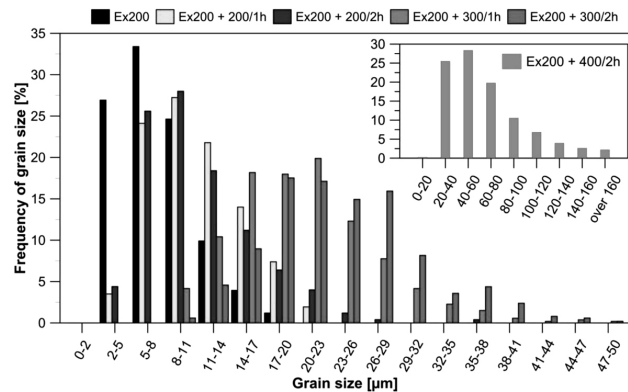


Figure 2: Grain size distribution in the Mg treated under different conditions

Slika 2: Razporeditev velikosti zrn v Mg, obdelanem pri različnih pogojih

given in **Figure 1**. Magnesium after the extrusion at 200 °C (**Figure 1a**) was fully recrystallized with the grain size ranging from 2 μm to 18 μm and with the dominant equivalent grain diameter of 4 μm . No elongated grains were observed in the structure. Different thermal treatment leads to an obvious grain coarsening. **Figure 2** shows the distribution of grain sizes for pure Mg treated at different conditions – extruded material and extruded material exposed to the subsequent thermal treatment at 200, 300 and 400 °C for 1 h and 2 h. It is clear that the coarsening of Mg is strongly dependent on both temperature and time. However, it seems that at lower temperatures such as 200 °C coarsening is not so significant with prolonged time on the temperature. At higher temperatures (300 °C, 400 °C) the grain size is increased significantly, also with a prolonged exposure time. **Figure 3a** shows EBSD maps of pure Mg extruded at 200 °C. The analysis was performed on the surface parallel to the extrusion direction. As can also be seen in **Figure 4**, the majority of grains is oriented in the structure with the basal planes parallel to the extrusion direction, which is a known effect of the extrusion process on magnesium-based alloys. Texture orientation and strength are evident in **Figure 4a**. A specimen subjected to the subsequent annealing at 400 °C for 2 h was analysed using EBSD (**Figures 3b** and **4b**) for a comparison with the extruded Mg. The grain size ranged from 20 μm to 160 μm and the dominant equivalent grain diameter reached about 40 μm . It can be clearly seen that texture was maintained similar to the extruded condition and only texture strength was slightly reduced from 10.2 to 10 for extruded Mg and extruded + annealed Mg, respectively (**Figure 4**). This confirms that the texture created during thermomechanical treatment is maintained also during the subsequent thermal treatment.

Dynamic recrystallization is supposed to manage the recrystallization process during the hot extrusion. In this case, new fine grains are formed at the original grain boundaries and subsequent nucleation occurs at the recrystallized grains.

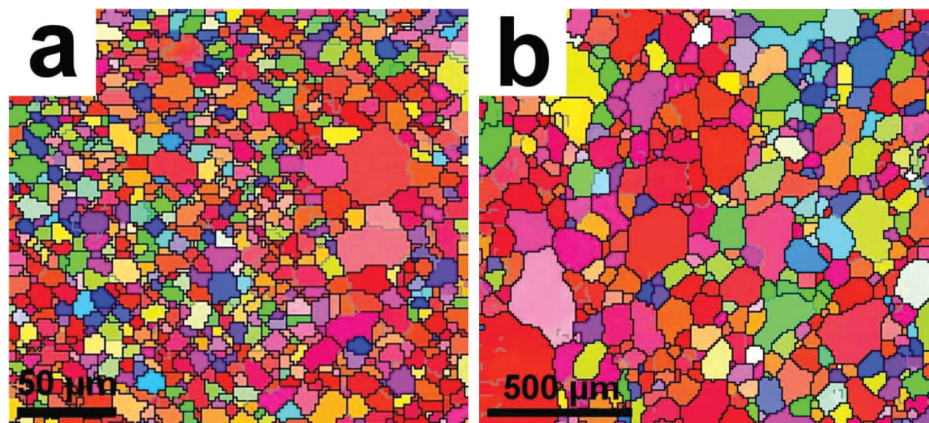


Figure 3: IPF maps of pure Mg: a) extruded at 200 °C, b) extruded at 200 °C and annealed at 400 °C for 2 h, refer to extrusion direction
Slika 3: IPF-mape pri čistem Mg v smeri ekstruzije: a) ekstrudirano pri 200 °C, b) ekstrudirano pri 200 °C in 2 h žarjeno na 400 °C

Discontinuous and continuous dynamic recrystallization (DDRX, CDRX) are considered for magnesium depending on the initial grain size and also the temperature. DDRX is operative when the initial grain size of magnesium alloys is large enough for the crystallographic slip to be heterogeneous, which is the case of the as-cast Mg. Pure magnesium in the as-cast state was characterized by the grains with about 100 µm in thickness and nearly 1 mm in length. It is known that extrusion ratio (ER) has a significant effect on the grain size. Although an ER equal to 10 seems to be low, it is possible to reach a very fine-grained structure after extrusion at 200 °C. According to the empirical Equation (5), strain during extrusion process corresponds only to 2.3:

$$\varepsilon = \ln(ER)^{14} \quad (5)$$

Therefore, other important factors, such as temperature and extrusion velocity, have to affect the final grain size significantly. In this case the extrusion velocity is only 2 mm/min. At a lower extrusion velocity, a lower strain rate can lead according to the Zener–Hollomon parameter to the coarsening of the structure. Moreover, it means prolonged times at temperature during the extrusion process and subsequent cooling, which can also lead to the coarsening. However, a lower strain rate also means only a smaller temperature increase in the material during deformation, which prevents an excessive grain growth.¹⁵

Texture development is global process that includes slip in the basal system and also other slip systems and

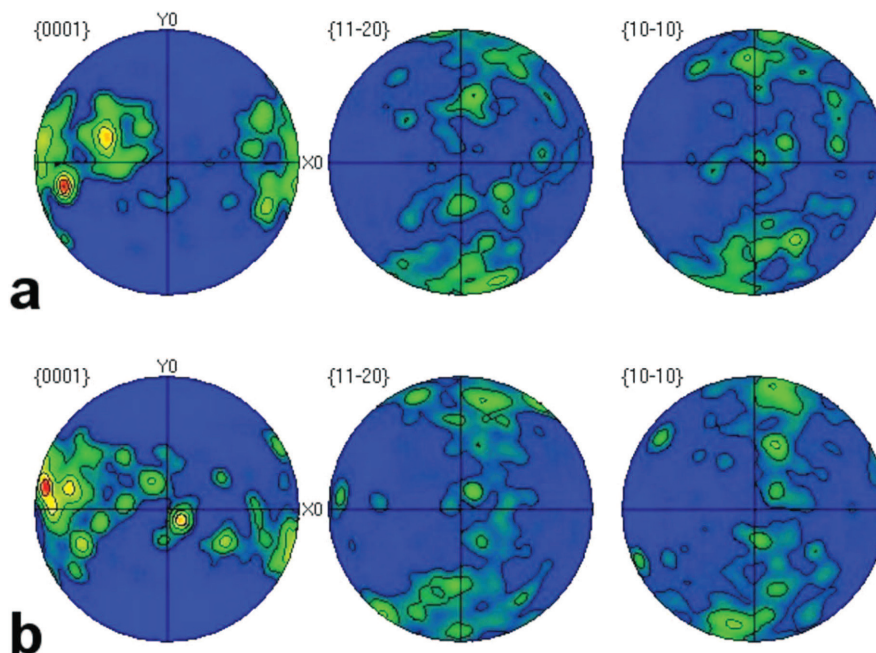


Figure 4: Pole figures of pure Mg: a) extruded at 200 °C (max mud = 10.14), b) extruded at 200 °C and annealed at 400 °C for 2 h (max mud = 9.91), X0–Y0 area is parallel to the extrusion direction and Y0 represents the extrusion direction

Slika 4: Slike polov za čisti Mg: a) ekstrudirano pri 200 °C (max mud = 10,14), b) ekstrudirano pri 200 °C in 2 h žarjeno na 400 °C (max mud = 9,91), X0-Y0 je področje, ki je vzporedno s smerjo ekstruzije, Y0 predstavlja smer ekstruzije

twinning. At low temperatures, the dominate slip system is basal slip. Tensile twinning also plays an important part in the accommodation of strain during the deformation, especially in the coarse-grained magnesium alloys. Due to the fact that coarse grains are presented in the structure especially at the beginning of deformation process, tensile twinning is favoured at this stage and helps to reorient the grains to a basal orientation. It was also proved that other mechanisms, such as double twinning and shear and kink banding, also operate to accommodate c-axis compression when non-basal slip is limited at low temperature.¹⁶ Texture can be partially suppressed by the slip in non-basal slip systems; however, the critical resolved shear stresses (CRSS) of non-basal slip systems for Mg are significantly higher compared with the basal slip system. This can be partially compensated by stress concentration at the grain boundaries, which is effective for the activation of non-basal slip. A higher extrusion ratio can be partially responsible for the activation of non-basal slip and finally also a weaker texture. Also, a higher temperature can lead to an easier activation of non-basal slip, which can significantly affect the texture evolution.

The DRX process itself does not lead to the texture change in the case of magnesium-based alloys exposed to the extrusion or a combination of extrusion and annealing (**Figure 4**); conversely, it serves to strengthen the basal texture. Due to the low temperature of the extrusion process, the low extrusion velocity and the extrusion ratio, it is really difficult for non-basal slip to be activated. There are only signs of twins in the struc-

ture, which can be responsible for a slight texture weakening. Overall, all the samples were characterized by similar and strong textures.

3.2 Mechanical properties

Mechanical properties in both compression and tension were measured. Based on the conditions of thermal treatment, magnesium is characterized by different compressive yield strength (CYS) and ultimate compressive strength (UCS) and also different behaviour after the onset of plastic deformation (**Figure 5**). The CYS of extruded Mg reached the maximum value of nearly 130 MPa. After that, the deformation strengthening is obvious; however, the ultimate tensile strength reaches a value as low as 190 MPa. On the contrary, magnesium exposed to the subsequent thermal treatment is characterized by lower values of CYS. Moreover, CYS is decreased with increased temperatures and prolonged times at these temperatures. When the CYS is overcome, strong deformation strengthening take place and finally, UCS reaches values between 240 MPa and 270 MPa (**Figure 5**).

The significant strain hardening in compression for materials with a coarse-grained structure is known and can be ascribed to the formation of $\{10\bar{1}2\}$ twins.^{8,17} The formation of deformation twins causes a significant strain-hardening tendency, because the twins block the propagation of dislocations. With a decrease in the grain size the decrease in the strain hardening is caused because the twinning cannot develop in the same manner as for materials with larger grains.¹⁸

The TYS and UTS increased gradually with grain refinement (**Table 1**), because magnesium has a large Taylor factor due to the lack of slip systems.¹⁹ It is also clear that uniform elongation is decreased with subsequent thermal treatment, in other words with increased grain size.

Table 1: Mechanical properties of magnesium in different states estimated from the tensile tests

Tabela 1: Z nateznimi preizkusi določene mehanske lastnosti magnezija v različnih stanjih

	Proof stress 0.2 % (TYS) (MPa)	UTS (MPa)	Elongation (%)
Mg Ex 200 °C	108±10	173±10	6.7±0.8
Mg Ex 200 °C + 200/2 h	91±8	168±7	6.2±0.8
Mg Ex 200 °C + 300/2 h	79±5	160±5	5.3±1.1
Mg Ex 200 °C + 400/2 h	72±5	150±7	3.6±0.9

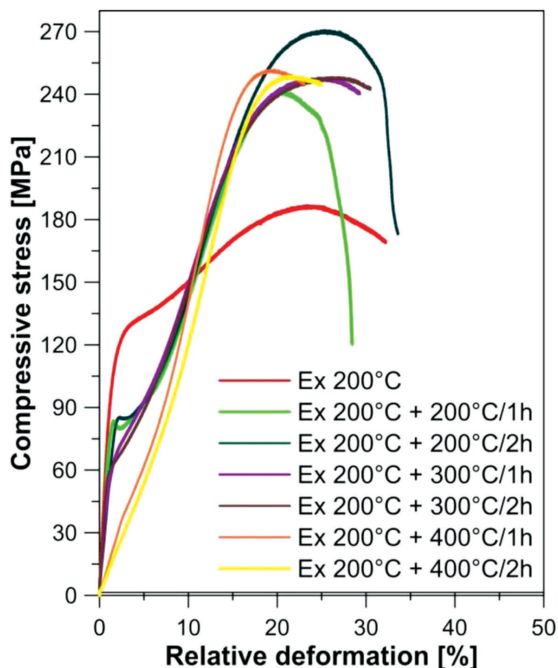


Figure 5: Compressive curves of Mg processed by extrusion and different subsequent heat treatments

Slika 5: Tlačne krivulje za Mg izdelan z ekstruzijo in z različnimi toplotnimi obdelavami po njej

Although there are many studies about the effect of texture and related effect on the Hall Petch behaviour, the obtained values were properly fitted by linear function, because all the samples seems to be characterized by a very similar texture and also its strength. **Figures 6**

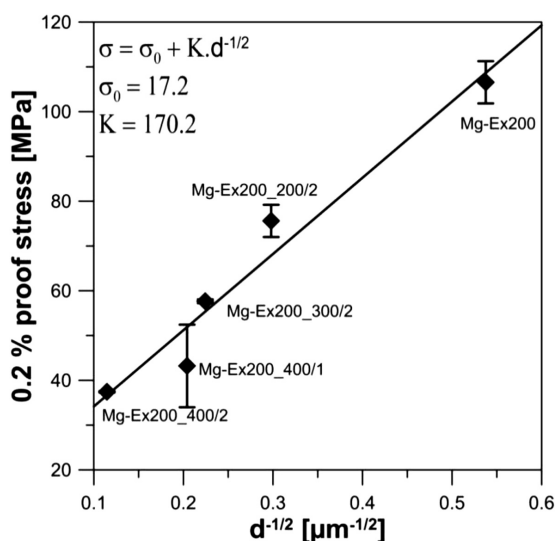


Figure 6: Experimental change of the compressive yield strength (CYS) with the grain size

Slika 6: Spreminjanje tlačne meje plastičnosti z velikostjo zrn

to 8 show the Hall-Petch relations based on the compressive tests, tensile tests and hardness measurements for pure Mg. It is worth mentioning that in all cases the deviation of different points from linear behaviour was negligible. Based on these facts it seems that the measured mechanical properties are dependent on the grain size according to the Hall-Petch relation. The obvious differences in the estimated constants for different measurement methods are connected with different types of mechanical loading and also texture of the material. It is known that during the extrusion or hot-rolling process, the basal planes are strongly oriented parallel to the rolling direction. In the case of extrusion, the texture strength is often weaker compared to the rolling; however, it is still there, as can be seen from

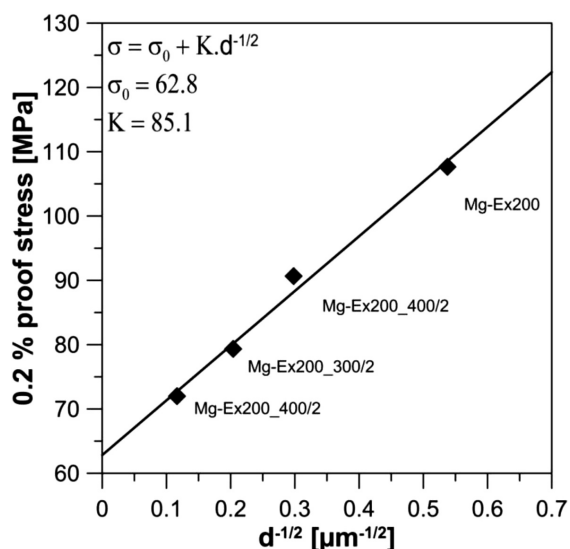


Figure 7: Experimental change of the tensile yield strength (TYS) with the grain size

Slika 7: Spreminjanje natezne meje plastičnosti z velikostjo zrn

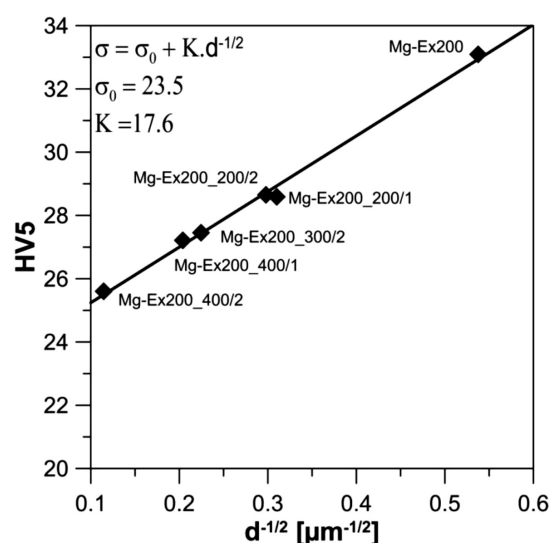


Figure 8: Experimental change of the Vickers hardness (HV5) with the grain size

Slika 8: Spreminjanje trdote po Vickersu (HV5) z velikostjo zrn

Figure 4. The observed mechanical properties are then strongly dependent on the direction of loading during the mechanical testing. If the basal planes are parallel to the axis of tension, so the directions (10 $\bar{1}0$) are parallel to the extrusion direction, the slip on basal planes is complicated. Therefore, a high tensile strength is observed along the extrusion direction.²⁰

The flow stress of Mg strongly depends on the grain size; although there is also a strong texture dependence due to the large difference in critical shear stress between the basal planes and the non-basal planes. Such differences are decreased with increased temperature. The effect of texture on the Hall-Petch slope or in other words the grain size dependence of "K" is significant.²¹ It is known that the misorientation of grain boundaries affects the grain size dependence of the flow stress. As a result, the value of "K" is lower for low-angle grain boundaries. Based on these facts, extruded Mg is characterized by a stronger dependence of the yield strength on the grain size compared to the Mg with a weaker texture.

Different works study the Hall-Petch behaviour of Mg after different thermomechanical processing. Some results are indicated in **Figure 9**. N. Ono et al.¹ used rolled Mg sheets as a starting material for a determination of the Hall-Petch relation using tensile tests. G. S. Rao et al.³ studied the Hall-Petch behaviour on tensile specimens from a hot rolled sheet in the longitudinal and transverse directions and also from hot-rolled square rods. H. Somekawa et al.⁸ prepared magnesium plates using extrusion with an extrusion ratio of 10 at 200 °C. The compression tests on these samples were performed with the compression axis parallel to the extrusion direction, which is the same as in the present research. A. Yamashita et al.²² studied magnesium and magnesium alloys prepared by ECAP with an internal angle of 90° at

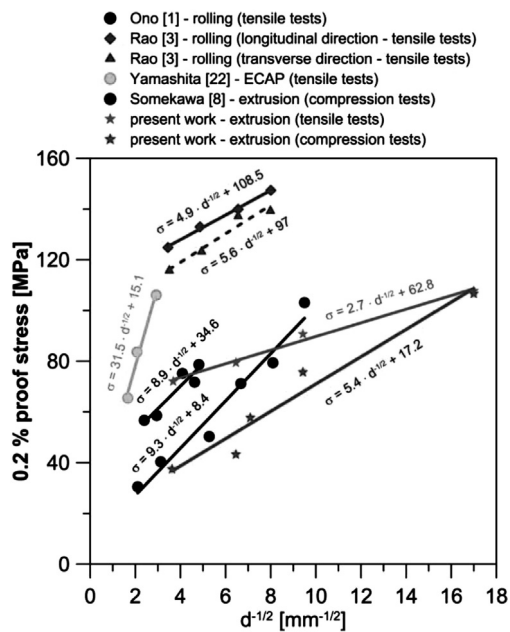


Figure 9: Hall Petch of pure magnesium after different preparation conditions

Slika 9: Hall Petch relacija pri čistem magneziju po različnih pogojih priprave

200–400 °C. The Hall-Petch behaviour was subsequently studied using tensile tests. Compared to our experiments, it is seen that Mg prepared by rolling is characterized by a higher value of σ_0 (Figure 9). Moreover, this value is dependent on the intensity of the rolling, which has a direct effect on texture strength.^{1,3} The same reason can be considered in the case of the difference between the properties of extruded Mg. From all these studies it is clear that there is no general Hall-Petch relation, which could cover different conditions. Instead, it is necessary to include grain size, texture and also other phenomena (basal, non-basal slip and twinning), which are affected by the grain size and texture in a general consideration for the final behaviour of the Mg material.

In Figure 10, stress versus the number of cycles fatigue curves are shown for two extreme states of the studied Mg. Magnesium extruded at 200 °C is characterized by an improved fatigue life. The maximum value of the stress amplitude for a service life longer than 8×10^7 cycles is 30 MPa for extruded Mg and about 25 MPa for Mg with a subsequent thermal treatment at 400 °C. Such a difference is connected with grain refinement, which is very effective for improving the fatigue strength.²³ Another fact that plays an important role during fatigue testing is the texture. Due to the preferential orientation of the basal planes parallel to the extrusion direction, there is an anisotropy between hardening for tensile and compressive loading.²⁴ During tensile loading, twinning is very challenging due to the initial orientation. Pyramidal $\langle a \rangle$ and $\langle c+a \rangle$ pyramidal slips are characterized by higher values of the Schmidt factors (0.33 and 0.49 respectively²⁴). Also, $\langle a \rangle$ prismatic

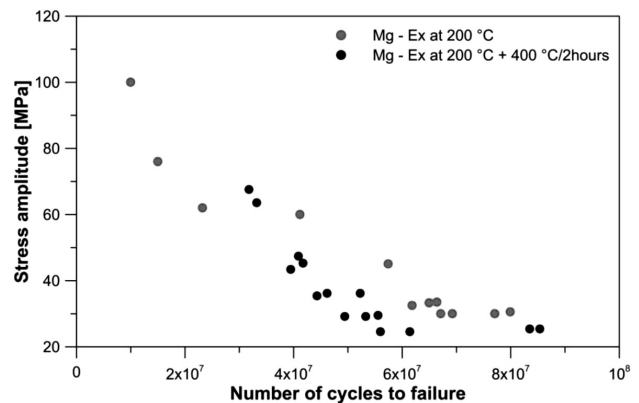


Figure 10: Fatigue behaviour of magnesium extruded at 200 °C and subsequently annealed at 400 °C for 2 h

Slika 10: Utrujanje magnezija, ekstrudiranega pri 200 °C in nato žarjenega 2 h na 400 °C

slip is problematic and a value of Schmidt factor equal to 0.35 has been postulated. As a consequence, especially basal slip with a low Schmidt factor of 0.17 takes place during this stage. In contrast, during the compressive step, $(10\bar{1}2) \langle 1101 \rangle$ twinning and also basal slip are active. Different twin types have been observed during cycling, although some types are presented in the structure after a number of cycles. During cycling, a hardening effect was observed and more stress is concentrated in the structure. As a consequence, more complicated twinning processes and also non-basal slip take place with an increasing number of cycles.²⁵

Tension twinning occurs when the resolved shear stress exceeds the critical resolved shear stress (CRSS). The values of 2.2–3.0 MPa were measured for such behaviour in a single crystal by Q. Yu et al.²⁵ and also the value of 2.4 MPa was published by the same author for a different monocrystal orientation.²⁶

Due to such limitations, complex dislocation structures are rather unlikely in magnesium under cyclic loading.^{24,27} In the case of pure Mg, cyclic hardening is expected as has already been proved in²⁸.

4 CONCLUSION

The present paper is focused on a study of the mechanical properties of pure Mg prepared by an extrusion process at 200 °C with an extrusion ratio equal to 10. Our results confirmed the significant effect of both grain size and texture on the mechanical properties. Both these main characteristics affect the slip and twinning activity. The anisotropy in mechanical properties is documented by different Hall-Petch behaviour based on compressive and tensile testing in the same direction and also by a comparison with literature. It is shown that both σ_0 and k from the Hall-Petch relation are strongly dependent on the mechanical processing. Fatigue tests performed at laboratory temperature confirm the increase in the fatigue life with a decreased grain size. The effect of

texture strength can be, in this case, neglected due to the similar texture strength of the measured samples.

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