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USABLE PROPERTIES OF MAGNESIUM ALLOY ZK60A BY PLASTIC DEFORMATION

WŁAŚCIWOŚCI UŻYTKOWE PRZERABIANEGO PLASTYCZNIE STOPU MAGNEZU ZK60A

The object of this study was to develop parameters of plastic deformation process of magnesium alloy – ZK60A. The tests have showed that for this alloy it is possible to use the temperature of the plastic deformation process ranging from 350°C to 450°C. Samples were characterized by mechanical properties and structure in different heat treatment tempers. This magnesium alloy obtained in the T5 temper higher mechanical properties than T6 temper. The paper also presents research results of investigation of conversion coating on ZK60A magnesium alloy by anodic oxidation method in non-chromium solutions. It was found that the coating produced in non-chromium solutions show considerable increase of corrosion resistance of tested alloy.

Keywords: magnesium alloys, extrusion, mechanical properties, structure, corrosion

Przedmiotem prezentowanej pracy było określenie możliwości kształtowania plastycznego na gorąco półwyrobów ze stopu magnezu ZK60A. Z przeprowadzonych prób wynika iż dla tego stopu można stosować temperaturę przeróbki plastycznej materiału z zakresu 350°C÷450°C. Otrzymane półwyroby scharakteryzowano pod kątem właściwości mechaniczne i struktury w różnych stanach umocnienia wydzieleniowego. Stop ten uzyskuje wyższe właściwości mechaniczne w stanie T5 niż dla stanu T6. W pracy przedstawiono wyniki badań dotyczące wytwarzania powłok konwersyjnych na stopie magnezu ZK60A metodą utleniania anodowego w roztworach bezchromianowych. Stwierdzono, że wytworzone powłoki w roztworach bezchromianowych powodują wzrost odporności na korozję badanego stopu.

1. Introduction

Magnesium alloys most frequently used nowadays are alloys of aluminium, zinc and manganese [1,2]. Aluminium preferably improves the tensile strength and hardness. Zinc in magnesium alloys is used to raise the strength of the alloy at room temperature, while manganese increases the corrosion resistance. The phase equilibrium diagram of Mg-Al alloys shows a variable increasing solubility of aluminium in magnesium up to a maximum content of 12.7% in the solid state and to a eutectic temperature of 437°C. Therefore, these alloys are subjected to a heat treatment to improve the mechanical properties by precipitation hardening (conditions T5 and T6) [3,4,5,6].

Magnesium alloys with manganese (MgMn) are not precipitation hardened. On the other hand, manganese has positive effect on the corrosion resistance of these alloys [7,8]. Magnesium alloys included into the MgZnZr family, which also incorporates the examined ZK60A alloy, are in the precipitation-hardened condition placed among the alloys of the highest strength (~ 350-380MPa).

The vast majority of magnesium alloy products are nowadays manufactured by casting methods. The process of magnesium alloy casting is very difficult to control, mainly due to

the high affinity of magnesium to oxygen, which demands the use of various types of protective atmospheres. Despite these difficulties, as the demand for magnesium alloy products increases, a rapid development in the fabrication techniques of these alloys is observed. Currently, the technology dominant in the manufacture of these items is shape casting, especially pressure die-casting in hot- and cold-chamber machines. Popularity are gaining the processes of plastic forming of magnesium alloys [9]. Thus, despite the fact that wrought materials are much more expensive, for many applications the processes of plastic forming are indispensable to yield the material with the assumed parameters. Generally, products from wrought magnesium alloys offer higher strength and plastic properties than the products obtained by casting. This difference is due to the fact that wrought materials have finer grains as a result of dynamic recrystallisation [10]. The advantages offered by wrought alloys as compared to alloys cast are shown in Figure 1.

Global aerospace and automotive industry, in an effort to reduce the weight of its components, has a very high demand for products made from wrought magnesium alloys. Meeting these expectations is possible only through the development of new complex technology of the plastic forming [12].

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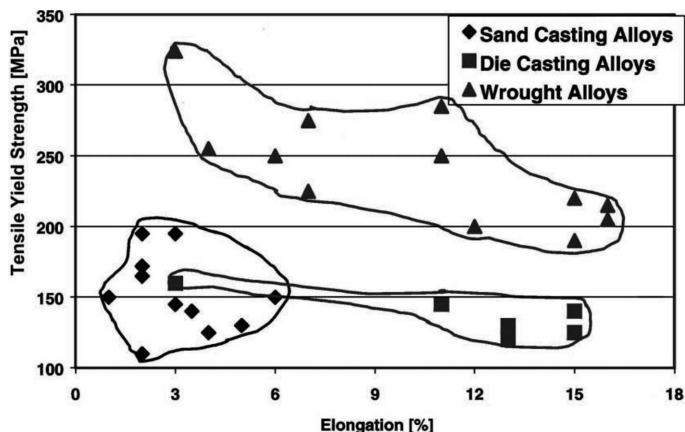


Fig. 1. Mechanical properties compared for products cast and subjected to plastic forming [11]

2. Test material and methodology

Studies of the direct extrusion process were conducted on MgZnZr (ZK60A) magnesium alloys belonging to the group of alloys characterized by the highest strength. The materials were in the form of 100 mm diameter ingots. The chemical compositions of these alloy were: Zn – 5,1%; Zr – 0,56%; Mg – rest. Tests of direct extrusion were conducted on a laboratory press of maximum 0.6MN force using specially designed and constructed tooling. Tests on pilot-scale were conducted on a horizontal direct-indirect press of maximum 5MN force [13]. For the purpose of laboratory tests, from billets of 100 mm diameters, rods of 29 mm diameter were cut out and were next hot extruded to φ 8 mm bars. For pilot-scale tests, 96 mm diameter billets were prepared, extruded next in a direct system

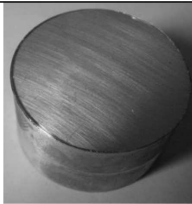

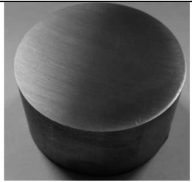
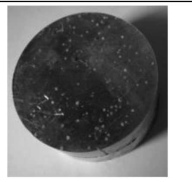
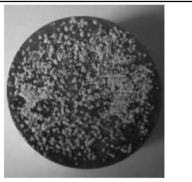
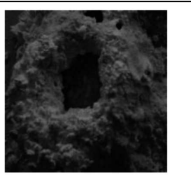

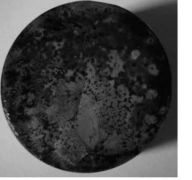
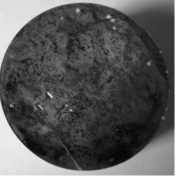

to φ 26 mm bars. Due to the application of relatively high, as for magnesium alloys, preheating temperatures, rods were protected with chromate coating to minimise oxidation.

Making magnesium alloy products in T5 condition requires the process of extrusion or forging to be conducted at a sufficiently high temperature to obtain full effect of solutionising after cooling the alloy directly from the temperature of plastic forming. Unfortunately, heating of magnesium alloys to the temperature of plastic forming causes a lot of problems with their surface oxidation. Therefore, in the described studies, the use of chromate coatings was tested. Applied on-to products made of metals and their alloys, they act as a protective layer when the product is used in the conditions of internal atmospheres, as a protection during transport and storage (the method of temporary protection), and as primer coats for organic top coats (varnishes). In the case of magnesium and its alloys, these coatings prevent the oxidation of alloy during preheating before the plastic working process.

Chromate coatings on magnesium products are produced in solutions of chromium, both tri- and hexavalent. The quality of chromate coatings applied on magnesium and its alloys depends not only on the type of the chromating technology but also on the chemical composition of magnesium and its alloys, and on the surface condition of products subjected to this treatment. Proper surface preparation is also important (the removal of fat or fat-like substances and corrosion products). The relationship between the chemical composition, structure and surface condition of products made from magnesium and magnesium alloys and the quality of chromate coatings results from the fact that these are the conversion coatings produced during a reaction that takes place between the surface layer of products and the components of chromating solutions (Fig. 2).

TABLE 1

Examples of ZK60A alloy samples after annealing with and without the coating

Output samples (not annealed)				
		uncoated	coated	
Sample	Anneal temperature			
	400 ⁰ C	425 ⁰ C	450 ⁰ C	475 ⁰ C
uncoated				
coated				

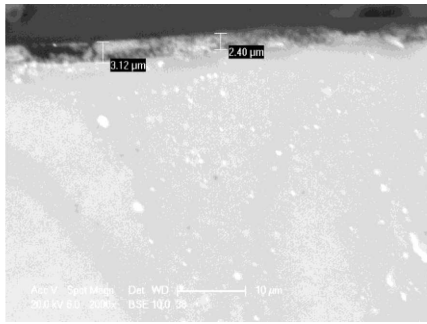


Fig. 2. Coating formed on the sample of ZK60A alloy (coating thickness $2 \div 3 \mu\text{m}$)

Samples with and without coatings were heated at temperatures ranging from 350°C to 475°C . Examples of samples with and without the coating after annealing are given in Table 1. Tests have proved that in the case of the ZK60A alloy annealed at 350°C to 400°C there was no major difference between the samples with and without the coating (absence of the effect of intensive oxidation). However, in the temperature range of 425°C to 475°C , the uncoated samples showed the effect of oxidation in the form of spongy oxide clusters which, as a result of plastic working, might be pressed inside the product, causing defects and loss of strength. At 475°C , the sample without coating suffered complete degradation. Samples with coatings did not show the oxidation effect, only colour of the coating became darker. Therefore, the application of protective coatings before preheating of the wrought ZK60A alloy is recommended for temperatures above 400°C .

2.1. Extrusion process

Magnesium alloys ZK60A were extruded in laboratory scale in direct process with extrusion ratio $\lambda = 14$, using two ram speeds, i.e. 0.8 mm/s and 2.8 mm/s . The materials were preheated from 350°C to 450°C . This alloys, next were extruded using 5MN force horizontal press and applying the extrusion ratio of $\lambda = 14.7$, similar to the value applied in laboratory tests, and ram travel speeds from 1 mm/s to 3 mm/s . The materials for pilot-scale trials were always preheated to 400°C . As a criterion in an assessment of the extruded rod quality, the surface condition was adopted, i.e. the occurrence of hot cracks, and the macro- and microstructure. Figure 3 shows the example of a extruded bars.

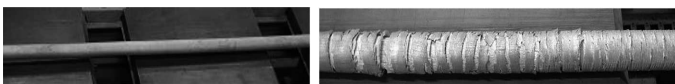


Fig. 3. Examples of extruded rods ZK60A alloy: extrusion temperature 400°C , $V_T = 1 \text{ mm/s}$ (A), $V_T = 3 \text{ mm/s}$ (B)

The laboratory extrusion tests have showed that for ZK60A alloy it is possible to use the temperature of the extruded material ranging from 350°C to 450°C when the process is run with an extrusion ratio of $\lambda = 14$ at the ram speed of 0.8 mm/s . Higher temperature of extrusion is beneficial as it enables reducing the process power and is essential for the rod solutioning heat treatment in an "on-line" system, i.e. on the press handling equipment (the treatment to T5 temper). For higher ram speed of 2.8 mm/s at a temperature of 450°C ,

cracks began to show in the surface, the rod was breaking. It is thus important for the ZK60A alloy to preheat the material to a temperature not exceeding $400\text{--}420^\circ\text{C}$.

The, selected on the basis of laboratory tests, temperature of 400°C to which the billets of Mg alloy were preheated for extrusion on a 5 MN press was correct at the ram speed values comprised in a range of $1 \div 2 \text{ mm/s}$ which, in the case of extrusion ratio $\lambda = 14.7$. With the ram speed increased to 3 mm/s , cracks appeared in the extruded rods (Fig. 3), which means that, as a result of deformation, the temperature of the extruded material was too high.

The extruded rods were subjected to multi-variant heat treatment, first, and examined for mechanical properties and structure, next. The extruded material was subjected to further plastic forming in the process of die forging.

2.2. The process of die forging

Tests of die forging were carried out on a vertical hydraulic 2.5 MN press using a specially designed set of tools with interchangeable shaping inserts (Fig. 4). The stock was heated in a temperature range of 350°C to 400°C and forged in dies in the shape of connecting rod. The ready forgings were subjected to a multi-variant heat treatment. Then the mechanical properties of forgings were tested and their structure was examined by optical and scanning microscopy.

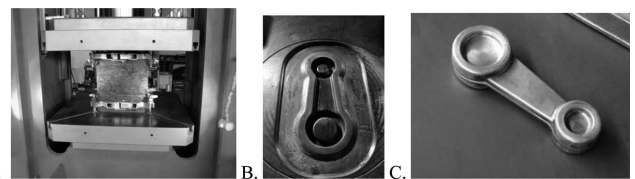


Fig. 4. A set of tools for die forging fitted in a 2.5 MN press (A), die insert in the shape of connecting rod (B), connecting rod forged from Mg alloy (C)

2.3. Heat treatment

Magnesium alloys are heat treated to conditions T6 (solution heat treatment and artificial aging) and T5 (solution heat treatment from the temperature of plastic forming and artificial aging). The T5 condition is used more commonly and allows achieving higher mechanical properties. Studies was carried out to select the best time-and-temperature regime for this alloys. For condition T6, the following parameters were adopted: solution heat treatment temperature of 460°C and aging temperature of 175°C [3,6,9,14]. To determine the optimum aging time, the precipitation hardening curves were plotted for the variant of solutioning heat treatment in a furnace and from the temperature of plastic forming (rods were extruded into water). The resulting curves are shown in Figures 5.

From the curve of natural aging it follows that the ZK60A alloy is sensitive to a limited extent only (slight increase in hardness only). In the case of artificial aging at 175°C , alloys ZK60A, achieve higher hardness during solution heat treatment from the temperature of plastic forming (T5 condition) and reach maximum hardness after approximately 12 hours.

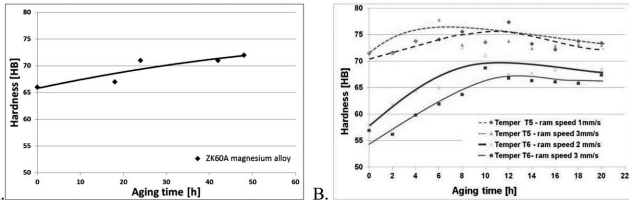


Fig. 5. The hardening curves plotted for ZK60A alloys after solution heat treatment from the temperature of plastic forming and natural aging – condition T1 (A) and after solution heat treatment in furnace and from the temperature of plastic forming and artificial aging at 175°C (B)

3. Structure of ZK60A alloy after plastic deformation

For the investigated alloys, microstructural analysis was performed also on the scanning electron microscope and optical microscope. The data are presented in Figures 6÷9.

The tested material after the forging process (F condition) and after rapid cooling from the temperature of plastic working and artificial aging (T5 condition) was characterised by a fine-grain structure. However, after re-heating for the solution heat treatment and after aging (T6 condition), examinations of the forged microstructure revealed the grain growth which occurred as a result of recrystallisation.

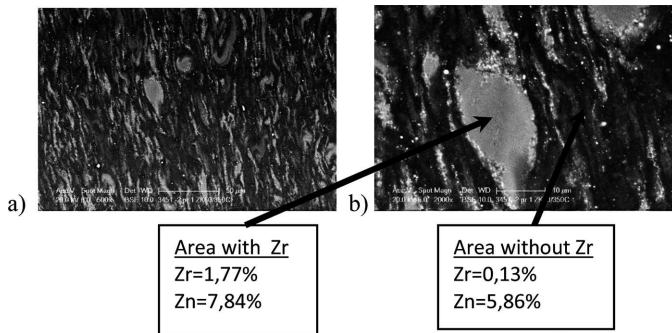


Fig. 6. SEM microstructure of ZK60A alloy forged at 400°C, T5 temper (a – mag. x500, b – mag. x2000) with the EDS chemical analysis in microregions

The structure of alloy reveals the presence of lenticular zirconium-rich areas about 1.7 wt.% (Fig. 6). Borders of these areas are additionally enriched in zinc, and there are fine zinc-zirconium precipitates (Fig. 7). These phases precipitate during either hot plastic forming or heat treatment.

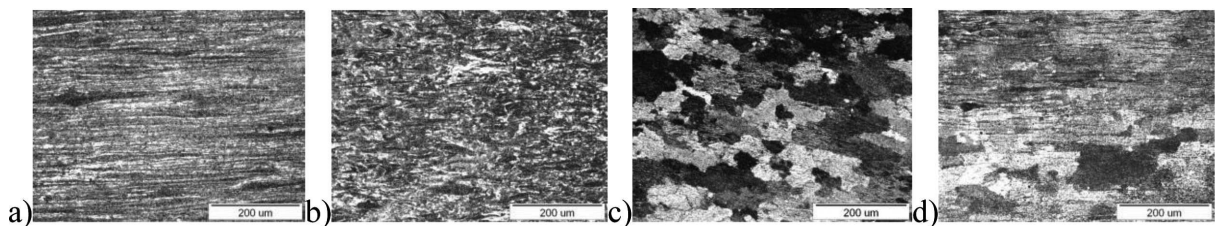


Fig. 8. Microstructures showing the grain size in ZK60A alloy forged at 350°C and heat treated to various conditions, a) T5 – non-recrystallised structure, b) T5 partially recrystallised structure c) T6 (solutioning at 430°C) – completely recrystallised structure, d) T6 with local large grain size variations

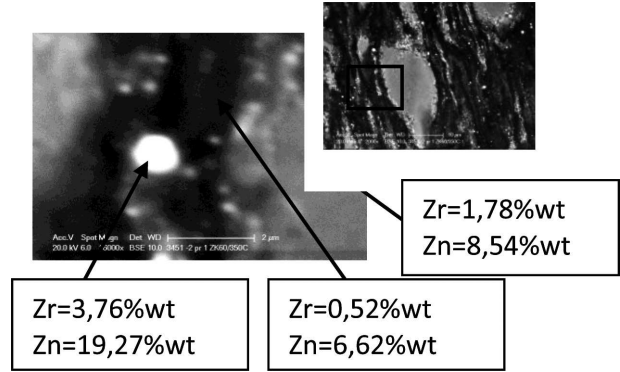


Fig. 7. EDS chemical analysis in microregions (ZK60A alloy forged at 400°C, T5 temper)

As a result of plastic forming (extrusion or forging) or heat treatment (solutioning), at the temperature of 350°C, the recrystallisation of zirconium-free areas has occurred (Fig. 8b). Areas rich in zirconium do not recrystallise at this temperature, since zirconium in these alloys increases the recrystallisation temperature. Heating to the temperature of solution heat treatment, i.e. to 430°C (T6 condition), has resulted in complete recrystallisation of the material (Fig. 8c), i.e. including the grains with and without the zirconium particles, causing local large variations in the grain size (Fig. 8d). Large variations in the size of recrystallised grains were due to the fact that in the areas with zirconium-free grains, the growth of grains took place earlier during recrystallisation, while in the grains with zirconium, zirconium acted as a grain refiner, and grains in those areas were finer after recrystallisation.

4. Mechanical properties

Rods extruded and forged materials from Mg alloys tests were subjected to multi-variant heat treatment. The heat treatment to T6 condition consisted in solutioning of the examined alloys at two temperatures 430°C and 460°C and aging at 175°C/20h. For T5 and T6 conditions also the effect of extrusion and forging temperature on the mechanical properties was studied in combination with the ram speed. In the case of materials forged to the T5 condition, the material after forging was immediately cooled in water from the temperature of the plastic forming process and then artificially aged. On the other hand, the T6 condition was obtained by application of the same parameters that were used for the extruded products. The extrusion process parameters of samples and parameters of the heat treatment as well as the obtained respective mechanical properties are compiled in Tables 2, 3.

TABLE 2

The mechanical properties of the ZK60A alloys heat treated to the T6 condition

Extrusion temp. [°C]	Solutioning temp. [°C]	Rp 0,2 [MPa]	Rm [MPa]	A [%]
350	430	263	323	15,5
	460	270	325	13,9
400	430	269	325	14
	460	276	324	10,1

TABLE 3

The mechanical properties of the extruded ZK60A alloys heat treated to the T1 and T5 condition

Alloy	Temper	Extrusion temp. [°C]/ Ram speed [mm/s]	Rp 0,2 [MPa]	Rm [MPa]	A [%]
ZK60A	T1	450 / 0,8	258	329	15,9
ZK60A	T5	420 / 2,8	267	339	15,2
		450 / 0,8	293	341	18,6

The results of mechanical tests carried out on forgings made at the extreme temperatures of 350÷400°C with the following heat treatment to the T5, T6 condition are shown in Table 4.

TABLE 4

Static tensile test – the results obtained after heat treatment

Alloy	Forging temperature [°C]	Temper	Rm [MPa]	R _{p0,2} [MPa]	A [%]
ZK60A	350	T5	325	282	15,3
ZK60A		T6	292	231	11,6
ZK60A	400	T5	318	278	14,4
ZK60A		T6	290	226	10,3

The results of structure examinations were confirmed by the mechanical properties determined in static tensile test (Tables 2,4). The mechanical properties of forgings were higher for the T5 condition, because the resulting material was only partially recrystallised (Fig. 8 a, b). Aging has no longer had any effect on the recrystallisation process, only on the decomposition of the solid solution and precipitation of fine-dispersed phases of Mg-Zn and Zn-Zr, which resulted in a slight increase of the properties.

5. Studies of the process of making conversion coatings

Studies of the manufacture of protective coatings were conducted on the ZK60A magnesium alloy. The methods described in literature for the manufacture of conversion coatings on magnesium and its alloys, known as plasma electrolytic oxidation (PEO) and microarc oxidation (MAO), are based on a high-voltage technology. In studies conducted at the IMN OML Skawina it has been decided to use a low-voltage technology, i.e. not exceeding 150V. The study was conducted using a variety of chemical compounds, with attention focussed

on the elimination of harmful chromium (VI) from the proper coating manufacturing process. Toxic compounds commonly used in the process of preparing the metal surface before the step of coating formation were also eliminated by replacing them with the compounds of nitric acid and methyl alcohol. Below (Table 5) are examples of bath compositions for the manufacture of conversion coatings and bath compositions for the chemical surface pretreatment.

TABLE 5

The chemical composition of solutions for production of protective coatings

Surface preparation	The composition of solutions used in the manufacture of coatings		
	A	B	C
HNO ₃ CH ₃ OH	HF H ₃ PO ₄ H ₃ BO ₃	KOH KF	NaOH H ₃ BO ₃ Na ₂ B ₄ O ₇ ·10H ₂ O

Based on the results obtained in studies of the coating manufacture it was found that for the ZK60A alloy the values of the process voltage in a solution designated as A were low (for the ZK60A alloy the process voltage did not exceed 2V), and the obtained coatings were relatively thin (about 6 μm). With the exception of protective coating produced in solution C on the ZK60A alloy, where a relatively low voltage (less than 6 V) was observed for the current densities of both 2 and 3 A/dm², in all other cases, the voltage was increasing with the increasing current density. A maximum voltage of approximately 100 V was obtained for the applied current density of 10 A/dm². At this current density, the thickest coating was also obtained in solution B. The coating-making process was accompanied by the phenomenon of intense sparking, reported in the literature also for high-voltage processes.

TABLE 6

The surface of ZK60A alloy with produced protective coating

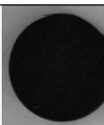
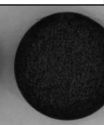
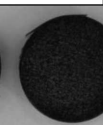
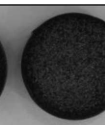



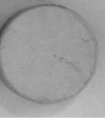
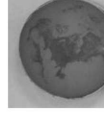
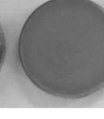
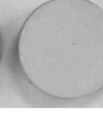
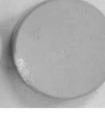
Sample	Current density [A/dm ²]			
	2	3	5	10
A				
B				
C				

Table 6 compares the surface appearance of ZK60A alloy samples with produced protective coatings. Coatings adopt different colours varying from white through grey to black. Colouring of coating depends on the chemical composition of alloy and on the chemical composition of the bath in which













this coating has been produced. The coatings are also characterised by a variable surface roughness. As the current density increases, roughness of the coatings increases, too. The thickness of the coatings varies in a wide range of values. The thickest coatings (about 100 μm) were obtained in solution B. The thinnest coatings had a thickness of about 6 μm.

6. Studies of corrosion process

First, the ZK60A magnesium alloy was tested for corrosion in salt mist. In parallel, studies were conducted on this alloy with protective conversion coatings produced on its surface. Tests were carried out in a HK1000 salt mist spray chamber made by DURA Company according to PN-EN ISO 9227:2012. The exposure to the neutral salt spray was 48 hours for samples without protective coating and 144 hours for samples with protective coating. The test solution consisted of NaCl (50 g/l) of analytical grade and demineralised water with a conductivity of 4.68 -5.10 μ S/cm. The pH value of thus prepared solution was 7.03-7.05. The study was conducted at 35°C in a continuous salt mist spray. From macroscopic observations it followed that most susceptible to corrosion was the material with coating produced in solution A and most resistant to corrosion was the material with coating produced in solution B (Table 7).

TABLE 7

The surface of ZK60A alloy samples with produced protective coatings after testing in the salt mist spray chamber

Current density [A/dm ²]		2	3	5	10
Solution	A				
	B				
	C				

The next step included electrochemical measurements (measurements of corrosion potential and potentiodynamic tests) performed by an "Autolab" potentiostat/galvanostat set made by EcoChemie BV with GPES software version 4.9 for the experiment control, data collection and analysis of results. The examined electrodes were samples of magnesium, while

the comparative electrode was made of platinum (in the case of potentiodynamic tests) and the reference electrode was an Ag/AgCl 3M KCl system. Measurements were carried out at room temperature in 3% NaCl solution naturally aerated.

The measurement of corrosion potential was made in accordance with ASTM G 69-97 (2003) on an AUTOLAB device. The reference electrode was an Ag|AgCl c (KCl) = 3mole/L electrode. The potential of this electrode relative to the normal hydrogen electrode (NEV) was + 211.5. The test time was 1 hour for each sample. The result was an average of the last 30 minutes of the measurement period. A potential vs time curve is shown in Figure 9. The calculated values of corrosion potential are shown in Table 8.

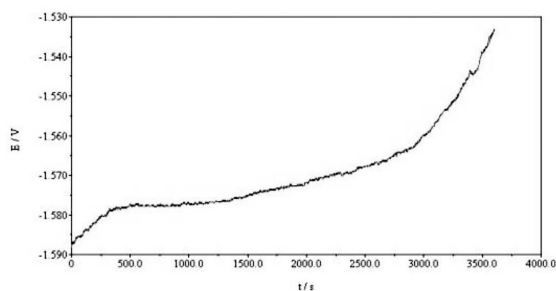


Fig. 9. Potential versus time relationship plotted for the ZK60A alloy

TABLE 8

The corrosion potential of ZK60A alloy – the value obtained in calculations

Alloy	Corrosion potential [mV]
ZK60A	-1562

The potentiodynamic method is an electrochemical method, during which the potential of the electrode changes continuously at a predetermined speed. Polarisation curves were recorded in the range of -1.8 V to +1.0 V relative to E_{corr} with the potential changing rate of 0.01 V/s. Tests were carried out after immersing the samples in naturally aerated 3% NaCl solution. The polarisation was initiated after a ten-minute period of the stationary potential setting up in the conditions of open-circuit potential (OCP).

Following the results obtained in the studies, the polarisation curves were plotted in a logarithmic system from which appropriate values were used to calculate the corrosion potential - E_{corr}, corrosion current - I_{corr} and polarisation resistance - R_p [5-9]. The results of the conducted studies are shown in Table 9.

TABLE 9

The results of electrochemical studies of ZK60A alloy

Electrochemical studies results	uncoated	Solution											
		A				B				C			
	Current density [A/dm ²]												
	0	2	3	5	10	2	3	5	10	2	3	5	10
I _{corr} [μA]	12950	4034	5055	6467	5223	3489	630,2	936,5	14,08	3345	1825	4521	4857
R _p [Ω]	28,72	3,475	4,201	5,713	5,131	5,098	8,782	11,52	1252	9,359	6,699	5,93	7,446
E _{corr} [mV]	-1630	-1508	-1519	-1522	-1522	-1533	-1544	-1544	-1543	-1562	-1550	-1564	-1549

Based on the electrochemical tests carried out, it was found that the highest corrosion resistance had the ZK60A alloy with coating prepared in solution B. The polarisation resistance obtained for this sample differed quite significantly from the other ones (1252 Ω), and the corrosion current was the lowest among all samples tested (14.08 μA).

7. Conclusions

1. Tests of the extrusion and hot forging have proved the possibility of shaping semi-finished and finished products from the ZK60A magnesium alloy and enabled also effective development of the parameters of these processes.
2. The material processed at 350°C had the structure either non-recrystallised or partially recrystallised; at the temperature of 400°C, the structure was already completely recrystallised. In the case of reheating for a heat treatment to the T6 condition, the structure was non-homogeneous as regards the grain size.
3. The wrought ZK60A alloy had higher mechanical properties when solution heat treated from the temperature of plastic working (extrusion, forging) and artificially aged, i.e. in the T5 condition rather than in the T6 condition, which involved re-heating in furnace for the solution heat treatment.
4. Based on the conducted studies of the manufacture of conversion coatings, correct coatings were obtained in solutions of different chemical compositions free from the presence of chromium (VI). The resulting coatings were characterised by good adhesion to the substrate and their thickness varied from several up to tens of micrometers.
5. The corrosion tests in salt mist confirmed the protective nature of the coatings produced and proved that the best protective power had coatings made in solution B.

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