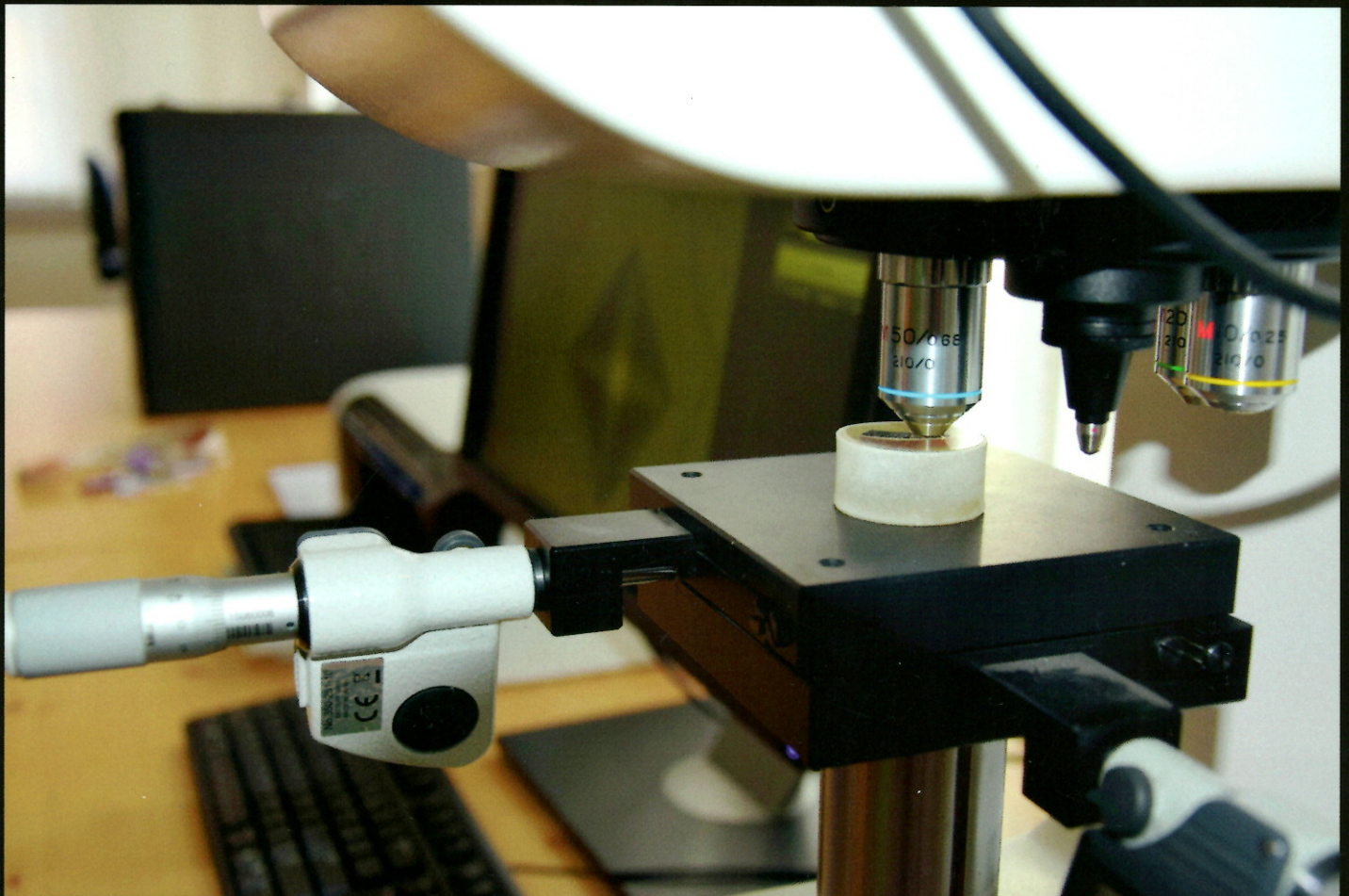


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## Characterization of Zn-Mg-Al Based Drosses from the Continuous Galvanizing

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The present paper deals with the microstructure characterization of drosses originated during production of a new type of coating called Zinkomag. This coating creates during continuous galvanizing process and it is characterized by addition of 0.8 – 1.0 wt. % Mg and 0.8 – 1.0 wt. % Al in the zinc galvanizing bath. The addition of magnesium in such coatings leads to increasing of hardness, corrosion resistance, and better color adhesion than most common used coatings (GI coatings). In this paper drosses were investigated using AAS, LM, SEM with EDX microanalysis, and XRD.

**Keywords:** CGL, Zinkomag, Coating, Dross, Structure

### 1 Introduction

Zinkomag or zinc-aluminum-magnesium coatings are produced by hot-dip galvanizing process in zinc alloyed melt with content 0.8 – 1.0 wt. % Al and 0.8 – 1.0 wt. % Mg. In comparison with well known and most common used GI coatings, Zinkomag contains a higher amount of Al (0.8 – 1.0 wt. % Zinkomag, 0.15 – 0.19 wt. % GI) and magnesium in addition. This new type of coating provides excellent corrosion resistance, superior coating adhesion, high scratch resistance and other. Galvanizing process with Zinkomag coatings, the same as the traditional coatings, produce a byproduct – top dross. Dross is defined as a byproduct of the galvanizing process that forms by creations between molten zinc and loose particles of iron in the galvanizing kettle. Dross formation is influenced by aluminium, respectively magnesium content at given temperature. Phase and structure of recycled aluminium alloy was studied in research *Đuriničová et al. [1]*. In the present paper the top dross from Zinkomag continuous galvanizing process is investigated [2 - 9].

### 2 Experimental material and procedure

For experiments three dross samples from continuous galvanizing process in company U. S. Steel Košice, s. r. o. were provided and labeled as sample A, B and C.

Chemical composition was determined by AAS (Atomic absorption spectrometry), phase analysis by

XRD (X-ray diffraction) using analyzer (X-ray diffractometer PANalytical X'pert PRO MPD).

The sample of drosses was further investigated by metallographic analysis. As etching agent the nitric acid and distilled water in ratio 1 : 100 was used. Microstructures were evaluated using a light microscopy (LM) and a scanning electron microscopy (SEM) in connection with EDX microanalysis (Energy dispersive X-ray spectroscopy). For LM analysis the microscope OLYMPUS XC50 was used. The scanning microscope TESCAN MIRA 3 FE was used for SEM analysis. The hardness values of Zinc matrix and intermetallic phase were measured by employing the micro hardness procedure with WILSON TUKON – 1102 where load was 50 g. With the purpose of getting an arithmetic mean, 5 measurements were conducted [10].

Microstructure of all samples was similar and therefore the only one sample was chosen for detailed analysis. LM analysis was used for un-etched (Fig. 2, 3), and etched (Fig. 4, 5) samples.

### 3 Experimental results

Chemical composition of all samples is shown in the Tab. 1. The highest content of magnesium, aluminium and iron has a dross sample B. The chemical composition of all samples was similar. Zinc was not analyzed, but it makes the rest of the amount.

Tab. 1 Chemical composition of dross samples

Sample		Metal content [wt. %]			Arithmetic means [wt. %]
		Mg	Al	Fe	
A	A1	0.69	1.03	0.52	Mg = 0.68 Al = 0.99 Fe = 0.52
	A2	0.72	0.97	0.54	
	A3	0.62	0.98	0.50	
B	B1	0.95	1.3	0.59	Mg = 0.75 Al = 1.16 Fe = 0.54
	B2	0.79	1.02	0.43	
	B3	0.52	1.16	0.59	
C	C1	0.70	1.09	0.57	Mg = 0.62 Al = 0.99 Fe = 0.47
	C2	0.55	1.02	0.53	
	C3	0.60	0.85	0.30	

The data, given in Fig. 1, show XRD of dross sample which consist likely of several phases. The XRD analysis confirmed metal zinc presence and likely presence of the phase  $Al_{12}Mg_{17}$ . The latter phase presence can be observed in the microstructure of dross shown in the Fig. 10, where the concurrent presence of Al and Mg was analyzed using mapping EDX. This phase occurs among zinc crystals. According Commenda [11], and Nishimura [12], intermetallic phases like  $MgZn_2$  and  $Mg_2Zn_{11}$  could exist in grain boundaries. The most negative value of standard free Gibbs energy has the intermetallic phase  $Mg_2Zn_{11}$ . This implies that presence of this phase in the grain boundaries of dross is thermodynamically most probable. This fact was also confirmed by XRD. Based on the weight percentages of iron and aluminium (determined from EDX analysis), it seems that intermetallic phase  $Fe_xAl_y$  could be very likely phase  $FeAl_2$ .

Ref. Code	Score	Compound Name	Chemical Formula
03-065-3358	63	Zinc	Zn
01-089-4186	17	Iron	Fe
03-065-0523	11	Zinc Oxide	Zn O
00-034-0570	14	Aluminum Iron	Fe Al2
01-073-1148	6	Aluminum Magnesium	$Al_{12}Mg_{17}$
00-003-1021	7	Magnesium Zinc	$MgZn_{5.31}$
03-065-1853	6	Magnesium Zinc	$Mg_2Zn_{11}$
00-001-1185	7	Magnesium Zinc	$Mg_7Zn_3$

Fig. 1 XRD analysis of dross sample

The microstructure of un-etched samples is shown in Fig. 2, 3, and etched samples in Fig. 4, 5. The zinc matrix with presence of intermetallic compounds as well as dispersed oxide layers can be observed in the structure. In the etched samples the grain boundaries and surface relief were visualized.

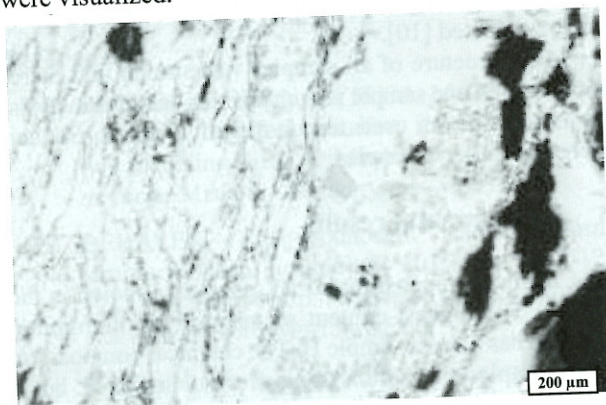


Fig. 2 The dross microstructure



Fig. 3 The dross microstructure

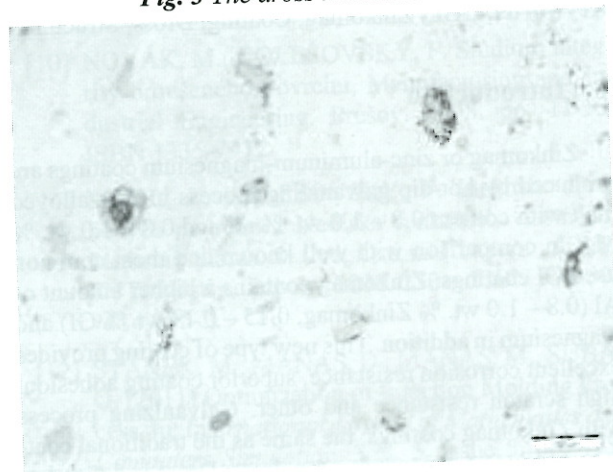


Fig. 4 The dross microstructure

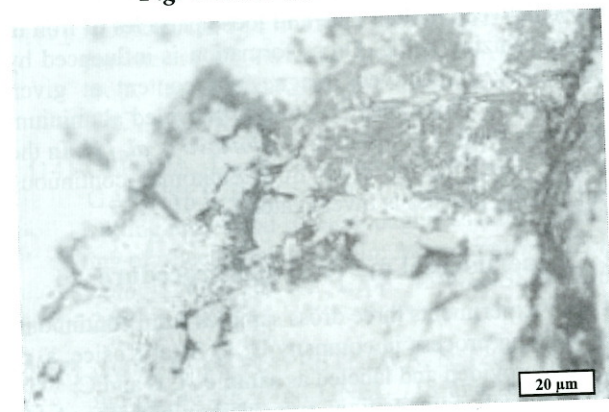


Fig. 5 The dross microstructure

The microstructure of dross sample observed by SEM is shown in Fig. 6 and Fig. 7. In the sample there is a matrix that consists of zinc. The intermetallic compounds dispersed in this matrix are displayed by dark gray color. The existence of the phase between zinc crystals is also observed.

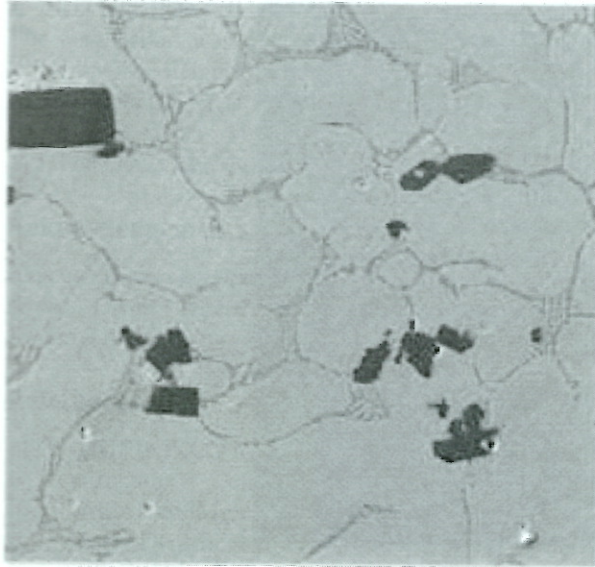


Fig. 6 The microstructure of sample A

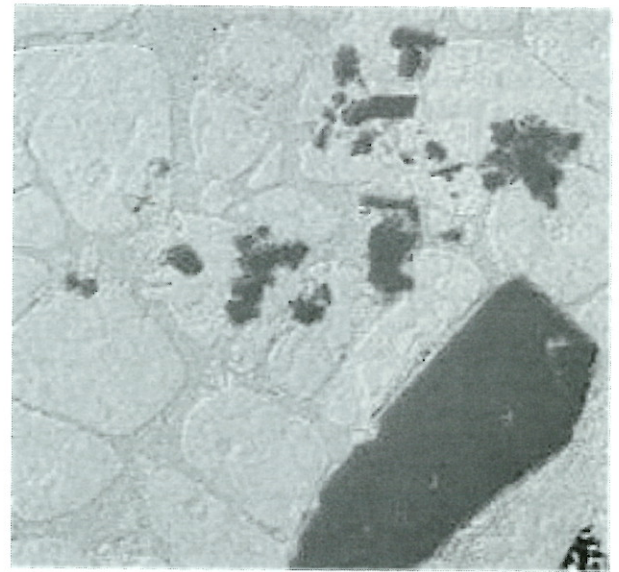


Fig. 7 The microstructure of sample B

In the Fig. 8 and Fig. 9, there can be seen a mapping EDX analysis for particular elements (Fe, Al, Mg), which are present in the sample. Beside zinc matrix, concurrent presence of aluminium and iron can be observed, that quadrate with presence of intermetallic phase. The magnesium is clearly segregated along the grain boundaries and its presence is not bound to iron. A concurrent presence of magnesium along with aluminium is observed.

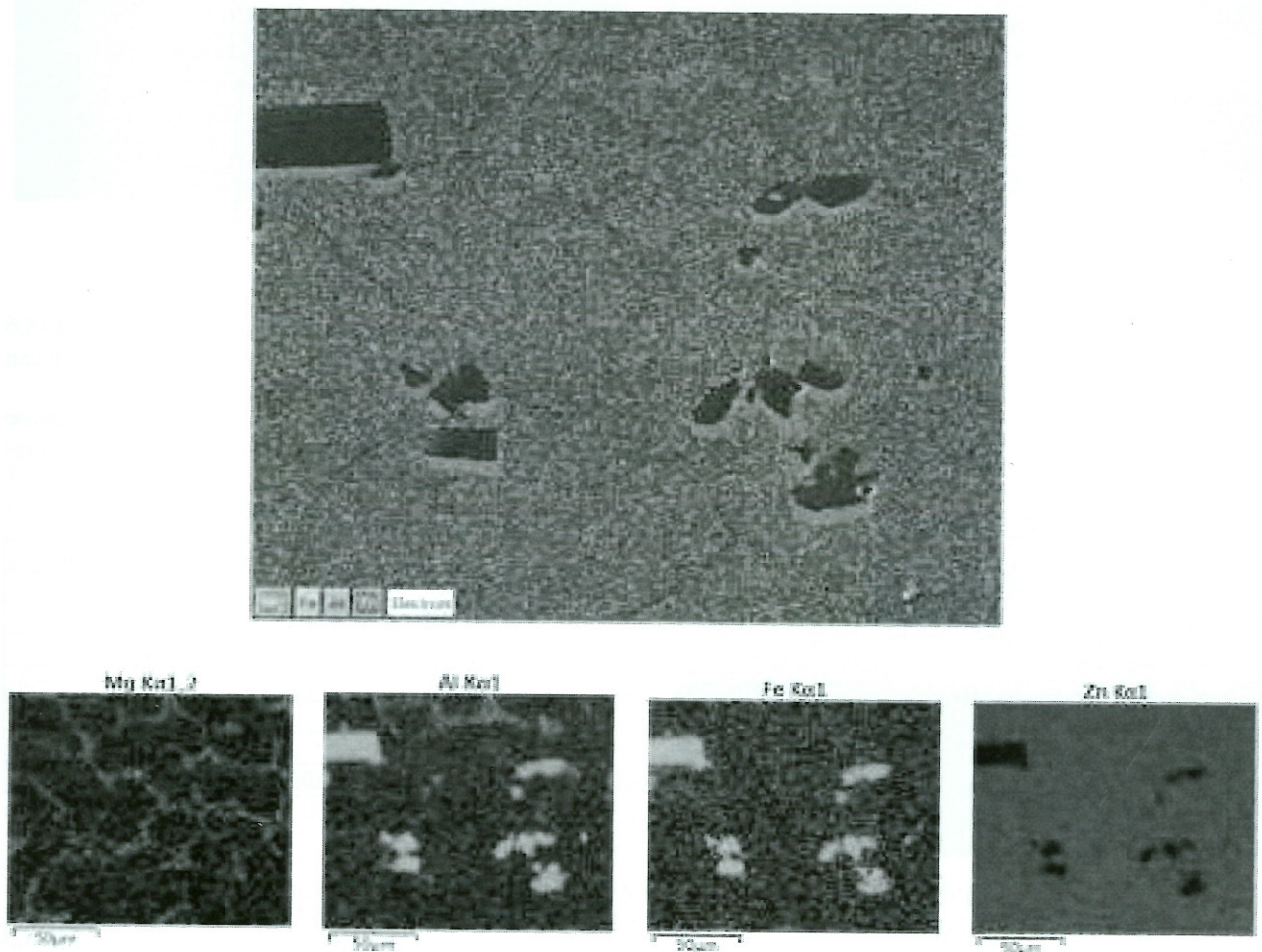


Fig. 8 The microstructure of sample A

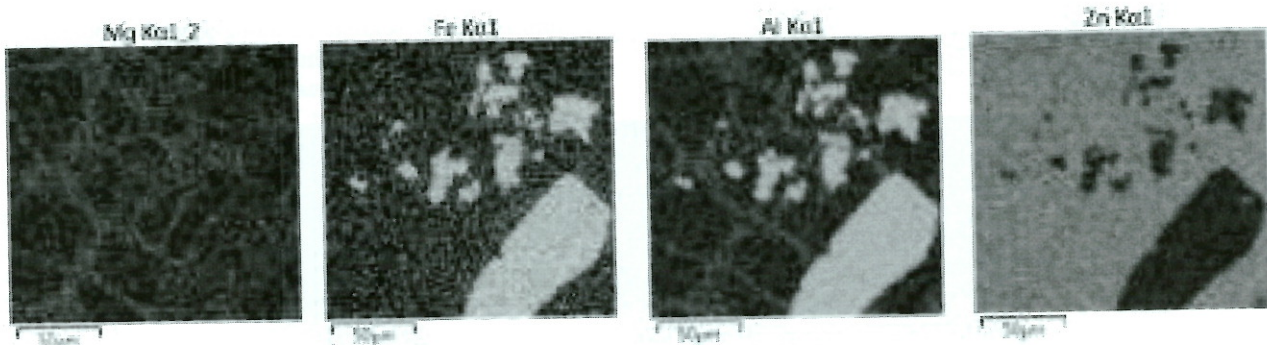
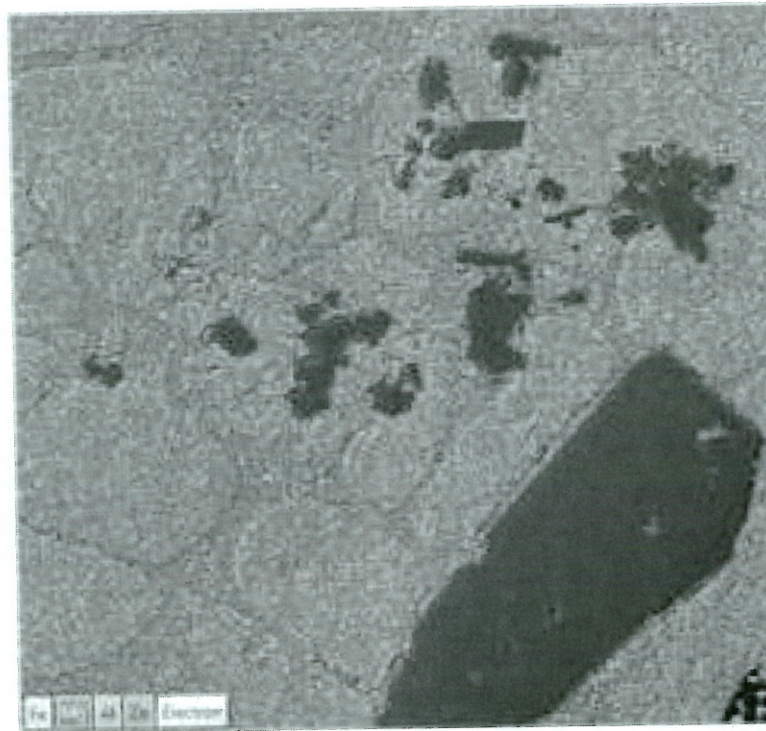


Fig. 9 The microstructure of sample B

The chosen places on the sample were analyzed by point EDX microanalysis and results can be seen in Fig. 10. The Fig. 10 shows point EDX microanalysis of intermetallic phases. The content of magnesium in this intermetallic phase was not confirmed by this analysis. The low amount of aluminium was specified.

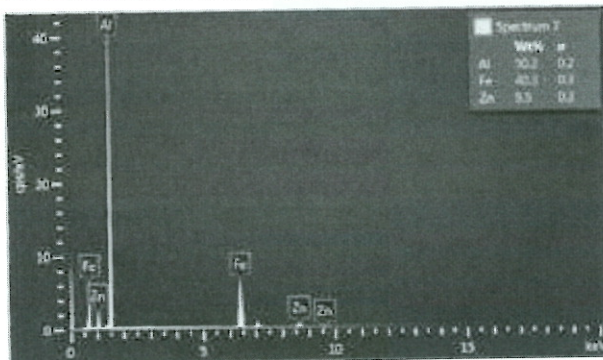


Fig. 10 EDX spectrum of intermetallic phase

The EDX analysis showed, that in the intermetallic phase of sample A there was specified 40.3 wt. % Fe and

50.2 wt. % Al and in the sample B 41.5 wt. % Fe and 48.6 wt. % Fe. On the basis of these weight percentages it can be assumed the presence of the phase FeAl<sub>2</sub>.

From the Tab. 2 it can be seen that intermetallic phase has a higher hardness in comparison to zinc matrix (approximately more than 44 %).

Tab. 2 Micro hardness HV0.05

Zn matrix	Intermetallic phase
46.4	75.8
56.9	80.6
62.5	95.7
56.6	82.5
53.1	63.4
55.1	79.6
Load HV0.05 = 50 g HV	

#### 4 Conclusion

The performed research showed that dross from continuous galvanizing process using Zinkomag coatings contains iron, aluminium and magnesium besides major

zinc. From the analyzed elements (Fe, Al, Mg), the highest element content of aluminium (0.99 – 1.16 wt. %) was determined. The iron was present in the amount of 0.47 – 0.54 wt. %, aluminium 0.99 – 1.16 wt. %, and magnesium 0.62 – 0.75 wt. %. The iron present in form of intermetallic compounds was bound with aluminium. On the base at XRD and EDX analyses it is likely the FeAl<sub>2</sub> phase. The magnesium was not a part of intermetallic phases with iron. The magnesium created an intermetallic phase Al<sub>12</sub>Mg<sub>17</sub>, as the XRD analysis revealed. The intermetallic phase FeAl<sub>2</sub> has 44 % higher hardness than zinc matrix. The phase Al<sub>12</sub>Mg<sub>17</sub> occurs among grain boundaries.

### Acknowledgements

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## Printing of Thin Walls using DMLS

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**This work deals with the problematics of 3D printing. Additive manufacturing (AM) covers a lot of principles of producing products and prototypes, for example, Direct Metal Laser Sintering (DMLS). This principle is based on sintering metal powder in thin layers, layer by layer. This theme is very extensive and a very popular research**