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Nanoindentation Testing of the Microstructure of AZ91 Alloy Castings with a WCCoCr Coating

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Abstract

The paper presents the results of nanoindentation testing of AZ91 magnesium alloy and a WCCoCr coating deposited by Air Plasma Spraying (APS). In order to determine the material properties of individual components of the microstructure of the AZ91 alloy and the WCCoCr coating, i.e., Vickers hardness – HV_{IT}, indentation hardness – H_{IT}, and indentation modulus - E_{IT}, nanoindentation testing of the individual microstructural components of the AZ91 alloy and the WCCoCr coating was performed. The analysis showed significant differences in material property values between the α-Mg matrix, the γ-Mg17(Al,Zn)12 intermetallic phase, the β(α+γ) eutectic, and the WCCoCr coating. The highest values of indentation hardness and indentation modulus were found in the case of the microstructure (grains) of the WCCoCr coating. The obtained results indicate that the application of the WCCoCr coating significantly improves the tribological properties of the surface layer of AZ91 alloy castings, especially scratch resistance. The results of these tests complement the previously performed scratch tests of the WCCoCr coating.

Keywords: AZ91 magnesium alloy, WCCoCr coating, Microstructure, Nanoindentation test

1. Introduction

Magnesium alloys represent a group of structural materials of great engineering significance, primarily due to their highly favorable strength-to-weight ratio and low density (approx. 1.74 g/cm^3), which makes them the lightest metals for applications in the aerospace, automotive, electronic, and medical industries. Additional advantages of magnesium alloys include good vibration damping capacity, high thermal conductivity, and recyclability [1-5].

Despite numerous advantages, magnesium alloys are characterized by significant performance limitations. One of them is low corrosion resistance resulting from the high electrochemical activity of magnesium and the presence of microstructural inhomogeneities that promote the formation of galvanic corrosion cells. Additionally, these materials exhibit relatively low resistance to tribological wear and limited strength at elevated temperatures. All of this significantly limits the application of magnesium alloys for components operating in aggressive environments or under intensive abrasive wear without modification of their microstructure or surface [6,7].

One of the most frequently used magnesium alloys is AZ91. Compared to other magnesium alloys, this alloy is characterized by good castability, favorable mechanical properties, and high creep resistance. It finds wide application in the production of gear housing castings, engine components, and many other structures in the automotive and aerospace industries [8,9].

In order to improve the properties of the surface layer of magnesium alloy components, including the AZ91 alloy, various types of protective coatings are applied. The most important

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coating deposition methods include PEO (Plasma Electrolytic Oxidation), HVOF (High Velocity Oxygen Fuel), and APS (Atmospheric Plasma Spraying).

The authors of work [10] compared the process of producing PEO coatings on AZ91 and AZ31 magnesium alloys. They found that the coatings produced on the AZ91 alloy are characterized by better coating-substrate bond quality compared to the coatings produced on the AZ31 alloy. The authors suggest that this is due to the differing aluminum content, which may affect the stability of the coating formation process.

The authors of works [11-14] investigated the properties of coatings made from tungsten carbide (WC)-based powders deposited by the HVOF method onto magnesium alloy components. In the studies, powders with varying content of WC, Co, and Cr were used. The applied coatings were characterized by significantly higher hardness and a lower friction coefficient compared to the substrate material.

The results of the studies by the authors of work [15] showed that the application of TiB2-TiC-Co cermet coatings, deposited on a magnesium alloy using the APS method, allows for the improvement of the tribological properties of the substrate material. The authors emphasize that they achieved a significant improvement in the quality of the coating-substrate bond by using Ni-P/NiCrAlY type interlayers.

The authors of work [16] analyzed the applications of plasma spraying using the APS method to produce NiAl10 and NiAl40 coatings on an AZ91 magnesium alloy substrate in terms of improving its corrosion resistance. They found that this technology enables effective surface protection of the magnesium alloy component, emphasizing that to achieve this goal, the appropriate selection of technological parameters of the APS method, as well as the course of the surface preparation process and the substrate preheating temperature, is of key importance.

The literature analysis presented above indicates that the modification of the surface layer through the application of protective coatings allows for a significant improvement in the performance properties of magnesium alloy components. The authors of this publication demonstrated in work [17] that the application of the WCCoCr coating allows for a significant improvement in the wear resistance, and especially the scratch resistance (scratch test), of AZ91 alloy castings. The article complements these studies in terms of determining the values of selected material properties of the WCCoCr coating microstructure, in comparison with the material properties of the AZ91 alloy.

2. Materials and Research Methodology

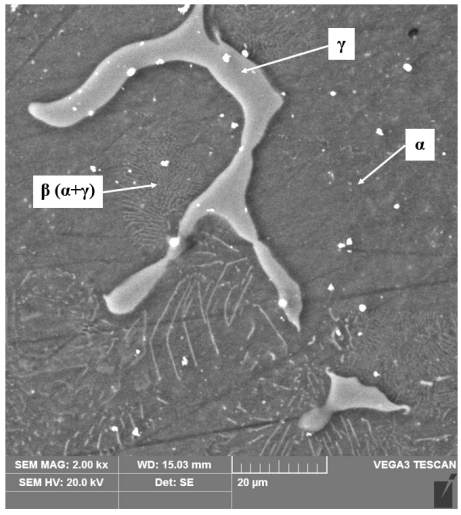
AZ91 magnesium alloy castings were used to prepare the samples, from which plates with dimensions of 50x50x10 mm were cut. Table 1 presents the results of the chemical composition analysis of this AZ91 alloy.

Table 1.
Chemical composition of the AZ91 alloy

Element content, wt. %			
Al	**Zn**	**Mn**	**Mg**
8.73	0.48	0.21	balance

Figure 1 presents the microstructure of the AZ91 alloy. This alloy belongs to the group of Mg-Al-Zn alloys, whose microstructure consists of a metallic α-Mg matrix, β(α+γ) eutectic, and large precipitates of the γ-Mg17(Al,Zn)12 intermetallic phase.

a)



b)

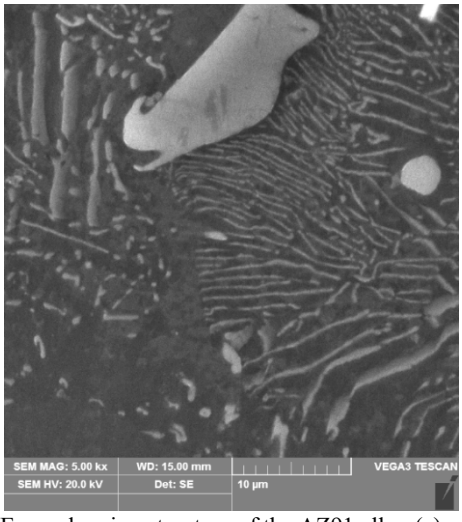
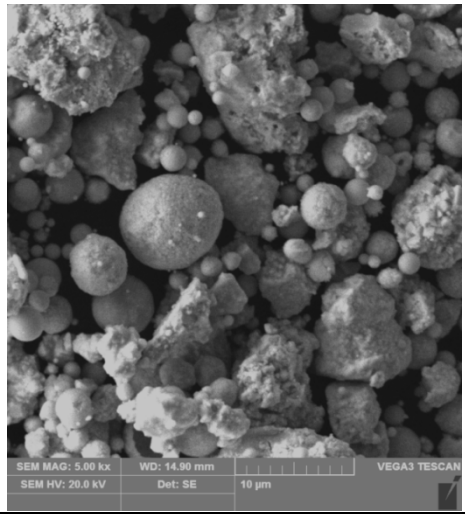


Fig. 1. Example microstructure of the AZ91 alloy (a) and high magnification of the eutectic β (b)

Prior to the APS (Air Plasma Spraying) process, the AZ91 alloy samples were subjected to grit blasting (abrasive blasting) to achieve a higher degree of surface development for better coating adhesion. The value of the Ra parameter (average arithmetic deviation of the profile from the mean line) after sandblasting was Ra = 29 µm. Then, the samples were cleaned with acetone in an ultrasonic cleaner.

A WCCoCr-type powder was used to produce the coating, containing particles with both near-spherical and irregular shapes. Powder morphology analysis indicates that for spherical powder particles with a size of 1.5–35 µm, their percentage was approximately 65%. For irregularly shaped powder particles with

a size of 8–38 μm, their percentage was 35%. Figure 2 presents the results of the X-ray microanalysis of the chemical composition of the WCCoCr powder.



Element, wt. %	Powder particle shape	
	irregular	spherical
W	71.8-82.5	78.1-81.6
Co	7.8-13.4	8.6-10.5
Cr	1.2-3.4	1.7-4.2
C	5.6-8.6	6.8-11.1

Fig. 2. View and chemical composition analysis results of WCCoCr powder particles

The WCCoCr coating deposition process was performed on a robotic station for APS plasma spraying (Fig. 3).



Fig. 3 View of the APS coating spraying station

The technological parameters of WCCoCr coating spraying by the APS method are presented in Table 2.

Material property testing (nanoindentation test) of individual microstructural components of the substrate, i.e., the AZ91 alloy, and the WCCoCr coating, was performed using a Nanoindentation Tester NHT and in accordance with the standard PN-EN ISO 14577-1:2005: Metallic materials – Instrumented indentation test for hardness and materials parameters. Part 1: Test method.

Table 2.
Technological parameters of the APS spraying process

Parameter	Value
Torch traverse speed	160 mm/s
Current intensity	630 A
Gas flow rate	Ar – 60 l/min
	H – 1 l/min
Powder feed rate (powder feeder disk speed)	0.45 rpm
Mixer	90%
Torch distance from the sprayed element	100 mm
Powder carrier gas	Ar – 3 l/min
Cooling gas type	Air, pressure 6 bar
Number of passes (coating layers)	20

A Berkovich diamond indenter and a loading force of 20 mN were used in the nanoindentation tests. The dwell time of the loading force was 15 s, while the loading and unloading rate of the indenter was 40 mN/min.

Material properties were determined for all three microstructural components of the AZ91 alloy (α-Mg matrix, γ-Mg17(Al,Zn)12 intermetallic phase, β(α+γ) eutectic). Due to the similar chemical composition of the spherical and irregular WCCoCr powder particles, material property measurements were performed in randomly selected areas of the coating. For each structural component, 5 indentations were made, and the results were averaged after discarding the lowest and highest values.

The nanoindentation test analyzed:

- Vickers hardness – HV_{IT},
- indentation hardness – H_{IT},
- indentation modulus – E_{IT}.

The indentation hardness H_{IT} is a measure of the material's resistance to permanent deformation. The difference between HV_{IT} and H_{IT} lies in the method of calculating these material properties. The HV_{IT} hardness is calculated based on an expression that considers the actual contact area between the indenter and the tested material, whereas the H_{IT} hardness is calculated based on the projected contact area between the indenter and the tested material.

The indentation modulus E_{IT} is determined from the expression:

$$E_{IT} = \frac{1-\nu_s^2}{\frac{1}{E_r} - \frac{1-\nu_i^2}{E_i}} \quad (1)$$

$$E_r = \frac{\sqrt{2}}{2}\frac{S}{\sqrt{A_p}}$$
$$S = \frac{dF}{dh} \quad (2)$$

where: E_{IT} – indentation modulus of the tested material [GPa], ν_s – Poisson's ratio of the tested material, ν_i – Poisson's ratio of the indenter material, E_r – reduced Young's modulus of the tested material [GPa], S = dF/dh – elastic unloading stiffness, determined at the initial stage of unloading, E_i – elastic modulus of the indenter material (1.14 TPa for diamond), S – contact compliance evaluated from the unloading curve at its maximum force value equal to dh/dF, Ap – projected contact area of the Berkovich indenter with the tested material, $Ap = 24.50 \cdot h_c^2$ [mm^2].

3. Research Results and Their Analysis

Microstructural analysis of the WCCoCr coating, deposited on the AZ91 alloy substrate, indicates a laminar structure typical of the APS plasma spraying process (Fig. 4). Grains formed from partially melted or completely melted powder particles of various sizes are visible in the microstructure of this coating.

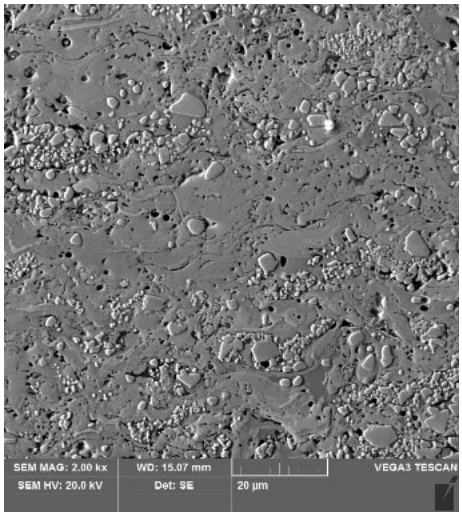


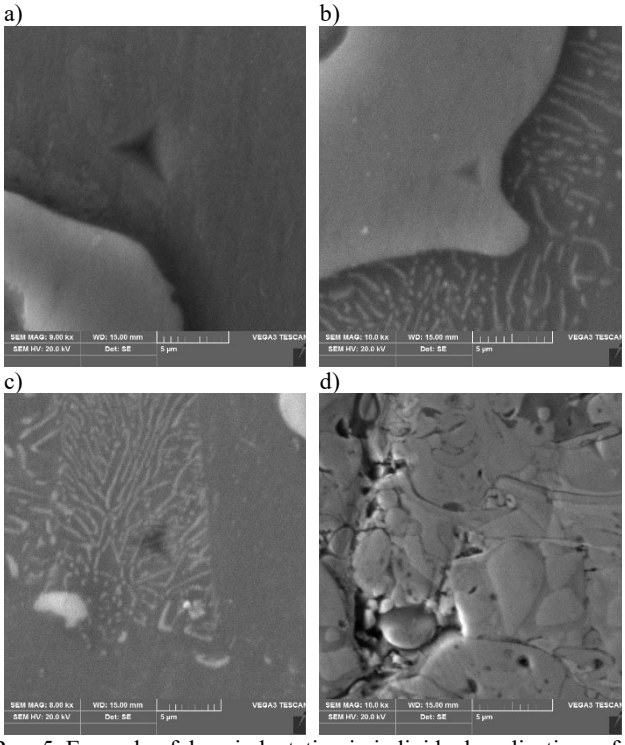
Fig. 4. Example view of the WCCoCr coating microstructure

An example view of the indenter impression in individual areas of the AZ91 alloy microstructure and in the WCCoCr coating is shown in Figure 5.

The indentation path of the indenter during loading and unloading in the nanoindentation test of the AZ91 alloy substrate material and the WCCoCr powder coating is presented in Figures 6-9. The values of the material properties are given in Table 3.

The obtained results indicate that depending on the analyzed structural component, varied material property values occur within the microstructure of the AZ91 alloy. The α-Mg matrix was characterized by lower material property values, which is associated with its high susceptibility to plastic deformation. The higher hardness values recorded for both types of hardness for the γ-Mg17(Al,Zn)12 phase confirm its brittle nature. The β(α+γ) eutectic area exhibited intermediate values depending on the share of the γ-Mg17(Al,Zn)12 intermetallic phase and the α-Mg matrix.

The results of the nanoindentation tests of the WCCoCr coating indicate much higher values (approximately 10-fold) of HV_{IT} and H_{IT} hardness, as well as the indentation modulus E_{IT}, compared to the AZ91 alloy.



Rys. 5. Example of deep indentation in individual applications of the AZ91 microstructure: α matrix (a), γ precipitate (b), β eutectic (c), and WCCoCr coating (d)

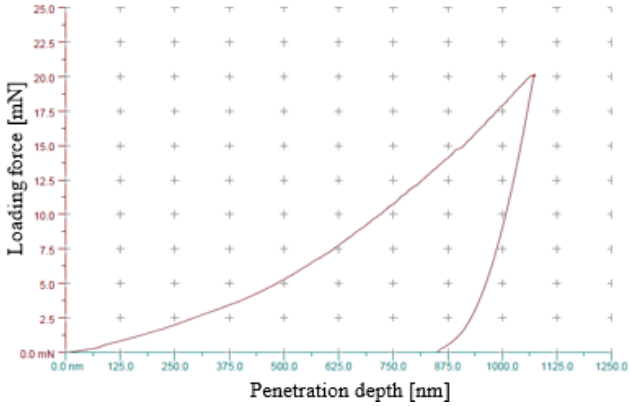


Fig. 6. Example indentation path of the indenter in the α-Mg matrix within the AZ91 alloy microstructure

Table 3.
Nanoindentation test results of the AZ91 alloy microstructure and the WCCoCr coating

Material	Microstructural component	Material property values		
		HV_{IT} [kG/mm^2]	H_{IT} [MPa]	E_{IT} [GPa]
AZ91 alloy substrate	α phase	81.57	880.73	31.56
	γ phase	169.37	1828.9	41.93
	β eutectic	95.66	1032.9	32.74
Coating	WCCoCr phase	1237	13357	243.44

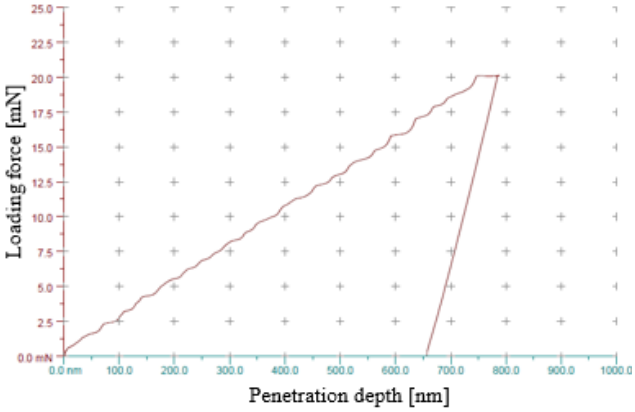


Fig. 7. Example indentation path of the indenter in the γ-Mg17(Al,Zn)12 phase within the AZ91 alloy microstructure

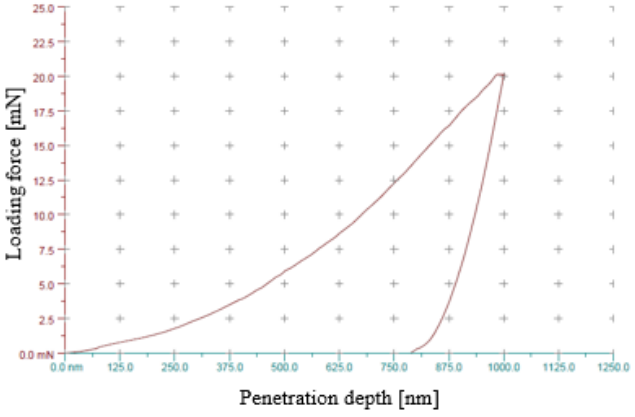


Fig. 8. Example indentation path of the indenter in the β(α+γ) eutectic within the AZ91 alloy microstructure

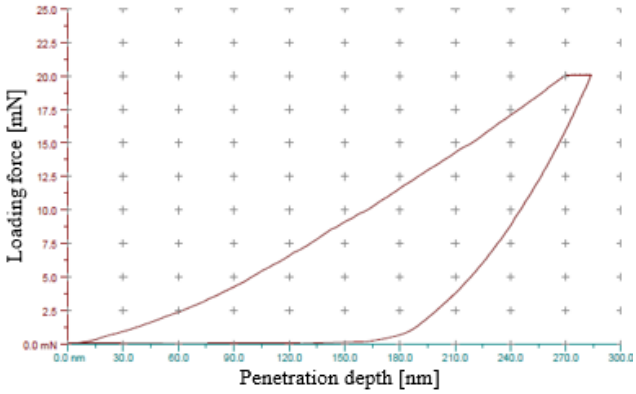


Fig. 9. Example indentation path of the indenter in a WCCoCr grain

4. Conclusions

The obtained nanoindentation test results indicate definitively higher material property values for the microstructure of the WCCoCr coating compared to the AZ91 alloy substrate material. This confirms the feasibility of applying plasma-sprayed coatings using the APS method to improve the abrasive wear resistance of AZ91 alloy castings, especially scratch resistance.

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