Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 53
ISSUE 2
August
2012

Surface layers on the Mg-Al-Zn alloys coated using the CVD and PVD methods

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Received 11.06.2012; published in revised form 01.08.2012

Manufacturing and processing

ABSTRACT

Purpose: The aim of this paper was investigated structure and properties of gradient coatings produced in PVD and CVD processes on MCMgAl9Zn1 magnesium alloys.
Design/methodology/approach: The following results concern the structures of the substrates and coatings with the application of electron scanning microscopy ZEISS SUPRA 35; phase composition of the coatings using X-ray diffraction and grazing incident X-ray diffraction technique (GIXRD); microhardness and wear resistance.
Findings: The deposited coatings are characterized by a single, double, or multi-layer structure according to the applied layers system, and the individual layers are coated even and tightly adhere to the substrate as well to each other. The analysis of coatings obtained on the surface of cast magnesium alloys by the PVD and CVD processes show a clear - over 100% - increase of the microhardness, compared to the base material microhardness. The best results of the sliding distance were obtained for the DLC coatings.
Practical implications: Achieving of new operational and functional characteristics and properties of commonly used materials, including the Mg-Al-Zn alloys is often obtained by heat treatment, ie, precipitation hardening and / or surface treatment due to application or manufacturing of machined surface layer coatings of materials in a given group of materials used for different surface engineering processes.
Originality/value: The paper presents the research involving the PVD and CVD coatings obtained on an unconventional substrate such as magnesium alloys. Contemporary materials should possess high mechanical properties, physical and chemical, as well as technological ones, to ensure long and reliable use. The above mentioned requirements and expectations regarding the contemporary materials are met by the non-ferrous metals alloys used nowadays, including the magnesium alloys.
Keywords: Thin & thick coatings; PVD and CVD coatings; Structure; Properties

Reference to this paper should be given in the following way:
T. Tański, Surface layers on the Mg-Al-Zn alloys coated using the CVD and PVD methods, Journal of Achievements in Materials and Manufacturing Engineering 53/2 (2012) 89-96.

1. Introduction

Modern technological development makes it necessary to find new design solutions for improving efficiency as well product quality, to minimize size and to increase the reliability and dimensional stability under operating conditions. The selection of proper materials is always preceded by an analysis of many factors including following requirements: mechanical, physicochemical, design, environmental, cost-relating requirements, availability and weight. The problem of overweight of the produced elements verifies the applicability of particular groups of materials. Therefore, over the past few decades there has been increased significantly the demand for materials with low density and high relatively strength, such as titanium, aluminium and magnesium alloys. Within this group of materials, particularly magnesium alloys reveals - in addition to low densities - also other advantages such as good damping capacity - the best among all currently known structural materials - as well high dimensional stability, good castability, low cast shrinkage, the combination of low density and high strength, appliance of machine components working at temperatures up to 300°C, and ease recycling possibility ensuring a repeatable use of alloys with very similar quality to the original material, in order to replace elements, produced from Mg alloys and applied for responsible structural elements [1-9]. Moreover, these alloys reveals the lack of aggressiveness in relation to the die material and have a low melting heat. The high damping capacity and relatively low weight allows to use magnesium alloys for fast-moving elements in cases where there are high accelerations and speed present. However, the unquestionable advantage of magnesium is above all its low density (1.7 g/cm^3), which is about 35% lower than the density of comparable aluminium alloys (2.7 g/cm^3) and over four times smaller than the density of steel (7.86 g/cm^3 .) At present, approximately 70% of the magnesium alloy casts is applied in the automotive industry [1-3,7]. In case of transportation, the general rule is to reduce weight of vehicles because of fuel savings. Together with mass reduction there will be also improved the driving characteristics of vehicles assembled with elements made from Mg alloys, what is mainly related to physical dynamics parameters. The need for weight reduction of vehicles is more and more important, because of the increasing number of vehicles equipped with additional accessories, which are designed not only to improve driving safety, but also increase the attractiveness of these vehicles in use. It should be noted, that the reduction of car weight of 100 kg can save up to 0.5 litres of petrol per 100 km, this has a significant contribution to the reduction of global fuel consumption, especially in view of rapid decrease in the availability of oil and its derivatives.

Magnesium alloys are used not only in the automotive industry, but also for production of helicopters, airplanes, disk reading devices, mobile phones, computers, bicycle parts, home appliances and office equipment, aerospace and electronic, chemical industry, energy, textile and even nuclear industry. The increasing growth of production of magnesium alloys, indicate an increased need for their application in the global construction industry, where magnesium alloys will be one of the most commonly used constructional material of this century [1-9]. According to the statistical data from the Hydro Magnesium company, the production of cast magnesium alloys increased in 1993-2003 in an exponential growth to nearly 180 000 tonnes, a significant share of this growth (about 80 000 tonnes) was detected in Europe. Data published by the U.S. Geological Survey indicate, that the primary magnesium production in 2011 was nearly 670 000 tons in China, and in the United States about 63 thousand tons, in other countries around 110 thousand tons [4]. However, despite the above-mentioned advantages of magnesium alloys, there are also disadvantages, for example - the most frequently mentioned - poor corrosion- and tribological resistance of these materials. For increase of these disadvantage one should look for alternative technologies, particularly in the field of surface engineering for increase of the magnesium alloys properties, and to increase themselves their application attractiveness. Promising technologies are the appliance of physical and chemical vapour deposition PVD and CVD techniques as an alternative to thermal spraying techniques or anodic protection techniques. Among the major advantages characteristic for physical and chemical vapour deposition techniques should be mentioned among others the possibility to obtain a whole range of new generation of high quality materials with unique properties for specific applications, as well the possibility of obtaining composite coatings (multi, multi, multi-phase, gradient, composite , metastable) even in a single process, and also high process efficiency while maintaining of a high level of ecological requirements, the possibility of appliance of these materials as a pure base metal material, unique decorative properties, as well the possibility of obtaining homogeneous layers in a relatively short time, possibility of automation - adapted to the mass production [7,10-18].

2. Experimental procedure

Investigations were performed on samples made of MCMgAlZn1cast magnesium alloys after heat treatment (Table 1). In order to determine the relationship between structure and properties of the achieved hybrid coatings, - a system composed of a soft substrate - transition gradient layer, with a continuous change of one or more components from the substrate to the outer surface - and the outer layer, independent coatings were produced by mind of the cathodic arc evaporation PVD CAE (called: Cathodic Arc Evaporation) as well in the chemical vapour deposition process PACVD (called: Plasma Assisted CVD) on a substrate of magnesium cast alloys, Mg-Al-Zn. PACVD process, where a relatively low temperature of the surface treatment can be achieved, is used to produce the carbon coatings of the DLC type at a defined pressure and in an acetylene C_2H_2 atmosphere. The gradient of the resulting coating was observed as a variable concentrations of silicon (Me) in the intermediate layer, silicon was supplied to the furnace chamber from the gas phase - Ti/aC: H-Me/aC H. The second method was implemented using the device DREVA ARC400 supplied by Vakuumtechnik by the cathodic arc evaporation method. The device is equipped with three pairs of independent sources of metals vapour. Prior to the coating process the substrate was cleaned chemically by washing and rinsing in a ultrasonic cleaner and dried in a stream of hot air. Moreover, the samples were ion-cleaned using Ar ions at a polarisation voltage of 800/200 V for 20 min. For the PVD coating process it were used water cooled discs with a diameter of 65 mm, containing pure metals (Cr, Ti) as well alloys TiAl and TiSi. The samples were coated in an inert Ar atmosphere as well in reactive gases atmosphere N_2 to obtain nitrides, and mixtures of N_2 and C_2H_2 in order to obtain carbonitride layers. The particles knocked out of the target surface react with the reactive gas molecules to form phases with specific of elements concentration ratio. Achieving of a right balance of elements concentration in the phase significantly improves the control of the coating properties. A gradient change of the chemical composition on the coating cross-section was achieved by changing the proportion of reactive gas dose or target evaporation current change on the arc sources. The determined coating process conditions are presented in Table 2. During the PVD coating process the substrates -made of cast magnesium alloys - move relative to the vapour sources, by performing of rotational movements in order to obtain uniform thickness of the layer, and preventing at the same time the phenomenon of so-called “shadow on the coated surfaces”.

Table 1.
Chemical composition of the investigated magnesium alloys

Alloy type	The mass concentration of main elements, %						
	Al	Zn	Mn	Si	Fe	Mg	Rest
MCMgAl9Zn1	9.09	0.77	0.21	0.037	0.011	89.79	0.0915

Table 2.
Deposition parameters of the investigated coatings

Coating parameters	Type of the achieved coating and the applied coating technique					
	PVD					CVD
	Ti/Ti(C,N)-gradient/CrN	Ti/Ti(C,N)-gradient/(Ti,Al)N	Cr/CrN-gradient/CrN	Cr/CrN-gradient/TiN	Ti/(Ti,Si)N-gradient/(Ti,Si)N	Ti/DLC-gradient/DLC
Base pressure, Pa	$5x10^{-3}$	5×10^{-3}	5×10^{-3}	$5x10^{-3}$	$5x10^{-3}$	$1x10^{-3}$
Working pressure, Pa	0.9/1.1-1.9/2.2	0.9/1.1-1.9/2.8	1.0/1.4-2.3/2.2	1.0/1.4-2.3/2.2	0.89/1.5-2.9/2.9	2
Argon flow rate measurement, cm^3/min	80*	80*	80*	80*	80*	80*
	10**	10**	80**	80**	20**	-
	10***	10***	20***	20***	20***	-
Nitrogen flow rate measurement, cm^3/min	225→0**	0→225**	0→250**	0→250**	0→300**	-
	250***	350***	250***	250***		
Acetylene flow rate measurement, cm^3/min	0→170**	140→0**	-	-		230
Substrate bias voltage, V	70*	70*	60*	60*	70*	500
	70**	70**	60**	60**	100**	
	60***	70***	60***	100***	100***	
Target current, A	60	60	60	60	60	-
Process temperature, °C	<150	<150	<150	<150	<150	<180

*during metallic layers deposition, **during gradient layers deposition, *** during ceramic layers deposition

Structure investigations of the studies cast materials were performed using scanning electron microscope Zeiss Supra 35 with secondary electron detection. Qualitative and quantitative point-wise EDX microanalysis as well the surface mapping of elements on the surface of the cast magnesium samples after heat treatment and surface treatment was performed on the scanning electron microscope Zeiss Supra 35 with X-ray spectrometer Trident XM4 supplied by EDAX. X-ray qualitative phase analysis of the investigated materials was performed using the Philips XPert diffractometer, with 0.05 ° steps and by a count rate of 5-10 s, using filtered $K\alpha1$ X-rays, with the copper anode wavelength of λ=1.54056 nm. The measurements were performed in the 2θ angle range between 20° to 140°. In the case of X-ray investigations of the thin layers obtained by appliance of the PVD and CVD methods - because of the overlapping of reflections coming from the substrate and coating material, as well their intensity, complicating the analysis of results - in order to obtain accurate information of the surface layer of the analysed in material in further investigations the diffraction technique was used at a constant incidence angle of the primary X-ray beam, a collimator was use with a parallel beam prior to the proportional detector. Diffraction patterns of the coatings were detected at different incidence beam angles.

Microhardness investigations of the obtained PVD and CVD coatings were performed using the Vickers ultramicrohardness tester DUH 202 supplied by Shimadzu. The applied load for the measurements was equal 10 mN.

Wear test investigations of the PVD and CVD coatings were performed using the ball-on-disk method according to the requirements of the ASTM G 99 and DIN 50324standards. The tests were carried out in dry friction conditions in horizontal configuration of the disk rotation axis. As a counterpart a tungsten carbide ball was applied with a diameter of 3 mm. The investigations were performed at room temperature, by a defined friction path distance with following testing parameters: Fn-5N load, rotation speed of the disk 200 rot/min, friction path radius 2.5 mm, movement rate v-0.05 m/s.

3. Discussion of experimental results

In order to investigate the structure and the existing relationship between the type of the substrate made from cast magnesium alloy, the type and technological conditions of the hybrid coatings production process, that means - a soft ground system - transition gradient layer, with a continuous change of one or more of its components reached from the ground to the outer surface - and the outer layer, - in the cathodic arc evaporation process as well in the plasma assisted chemical vapour deposition process - metallographic tests were carried out. Coatings produced using the CAE-PVD technique are characterized by a clear heterogeneity connected to a number of microparticles in form of droplets occurred the structure (Figs. 1-2). The highest heterogeneity of the surface area compared to other investigated coatings is characteristic for the coatings of the type: Ti/Ti(C,N)/(Ti,Al)N and Ti/Ti(C,N)/CrN, where a number of solidified droplets of the vaporized metal were identified (Fig. 1). The occurrence of these morphological defects is related to the cathodic arc evaporation process itself. Depending on the process conditions, including the kinetic energy of the drops sputtered into the metal substrate and the nature of the metal vapour source, the observed particles are clearly different in terms of shape and size. It was also confirmed, that the crystallised droplets are often of a spheroid and irregular shape, or they form agglomerates composed of a few droplets (Figs. 1,2). Moreover, it was also observed that on the surface of the obtained coatings, there are present characteristic cavities, which are formed as a result of falling out of droplets after finishing the PVD process. On the basis of metallographic investigations it was found out, that the cavities, coming from falling out of the droplets, does not reach the magnesium substrate. In the case of the DLC coating obtained using the PACVD process, on the surface are also identified small droplets, often in a spheroidal form. Surface morphology of the obtained DLC coatings differ significantly from the surface morphology obtained in the classical high-temperature CVD processes, where characteristic feature occurs, like a network of microcracks, wave-like surfaces or surfaces with spherical shapes.

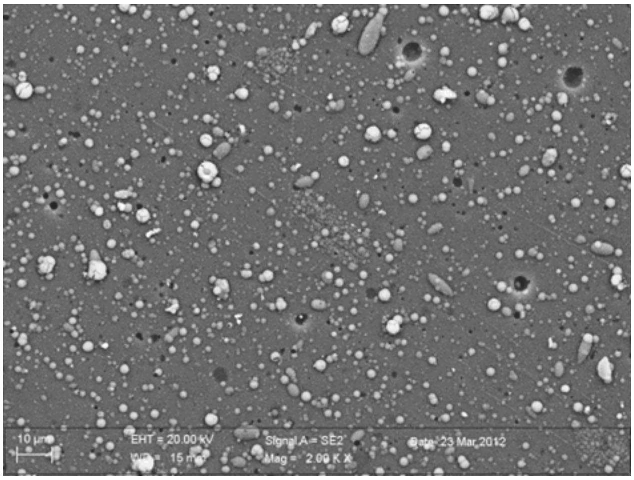

Fig. 1. Surface morphology of the Ti/Ti(C,N)/(Ti,Al)N layer coated on the MCMgAl9Zn1cast magnesium substrate

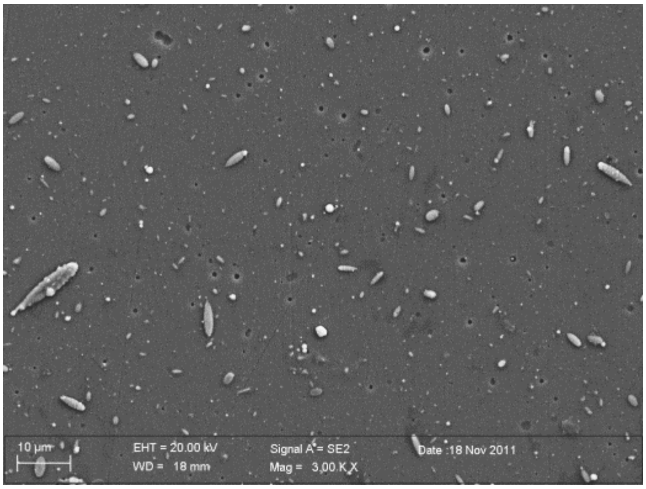

Fig. 2. Surface morphology of the Cr/CrN/CrN layer coated on the MCMgAl9Zn1cast magnesium substrate

As a result of metallographic fracture investigations of samples made from magnesium alloys coated with the analysed coatings, performed using scanning electron microscopy, there were identified a clear transition zone between the substrate and the coating. The obtained coatings reveals a compact structure, without visible delamination and defects, they are uniform and tightly adhere to each other as well to the substrate (Figs. 3-6). Investigations of fractures confirm that the shells of the type Ti/Ti(C,N)/(Ti,Al)N and Ti/Ti(C,N)/CrN show a layered structure, with a clearly visible transition zone between the coating and the gradient wear resisting coating, achieved by appliance of separate sources of the metal vapour (Fig. 3). In the case of the Cr/CrN/CrN, Ti/(Ti,Si)N/(Ti,Si)N coatings, where the same set of chemical elements in the gradient coating as well in the wear resistant coating and in the coatings of the Ti/DLC/DLC type was applied, any differences on the cross section has been found. (Figs. 4-6). Moreover, in case of the thin adhesive coating, which improves the adhesion of DLC coating to the substrate, there was identified a characteristic bight continuous titanium layer, which was also confirmed using EDS microanalysis (Fig. 7). In the case of the titanium nitride layer of the Cr/CrN/TiN type, there was confirmed the characteristic nature similar to a columnar growth of crystallites characteristic of coatings based on titanium nitride obtained in the cathodic arc evaporation process (Fig. 6).

As a result of quantitative and qualitative X-ray microanalysis performed using the EDS spectrometer the presence of major alloying elements Mg, Al, Zn, Ti, Cr, C, N, Si was confirmed, both the investigated cast magnesium alloys as well the tested layers (Fig. 7, Table 3), also information were obtained about the mass and atomic concentration of elements in point-wise tested microareas of the substrate and of the obtained coatings.

The applied qualitative X-ray analysis method for phase composition performed in the Bragg-Brentano geometry confirmed the presence of the following phases: TiN, (Ti,Al)N, (Ti,Si)N, Ti(C,N), CrN, α-Mg, γ-$Mg_{17}Al_{12}$ in the investigated coatings as well in the substrate (Fig. 8). Some of the identified reflections are shifted relative to the reflection angle given in the

JCPDS tables and their intensity is also different from the values given in these tables, which may indicate the presence of internal stresses in the investigated coatings, what is characteristic for the PVD process. Because of reflections overlapping coming from the substrate and the coating material, their intensity and a relatively small thickness of individual layers up to 3.5 μm, as well a similar 2Θ angle values for the coatings of the Ti(C,N) and Ti(Al,N) type, the phase identification was difficult to perform. The presence of reflections from the substrate material was confirmed on all diffraction patterns made from the coating, because of the thickness of the obtained coatings, smaller than the X-ray penetration depth of the investigated material. To obtain more accurate information concerning the analysed layers of the surface coatings in further investigations, there was applied a diffraction technique performed with a constant angle of the incident X-ray beam, what leads to achieving of diffraction lines coming from thin films by increasing the tested material volume. At different incidence beam angles, chosen experimentally and individually for each type of coating, there were reflections registered only from the thin surface layers (Fig. 8).

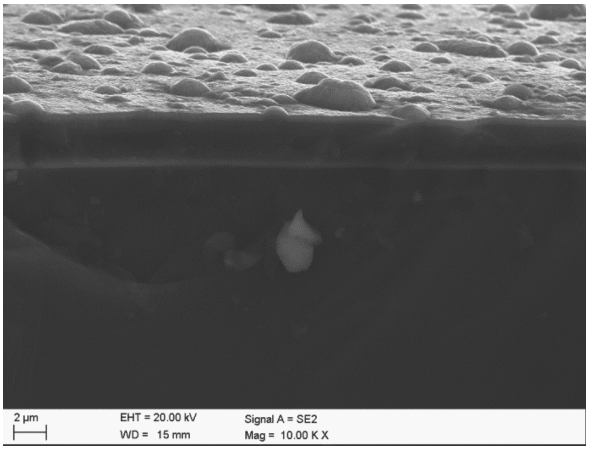

Fig. 3. Fracture of the Ti/Ti(C,N)/(Ti,Al)N coating on the MCMgAl9Zn1 cast magnesium alloy

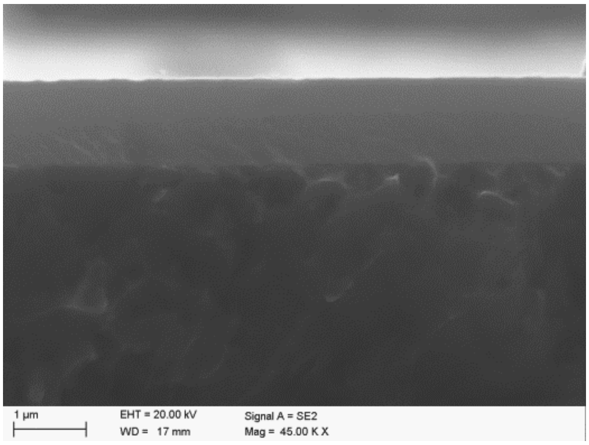

Fig. 4. Fracture of the Ti/(Ti,Si)N/(Ti,Si)N coating on the MCMgAl9Zn1 cast magnesium alloy

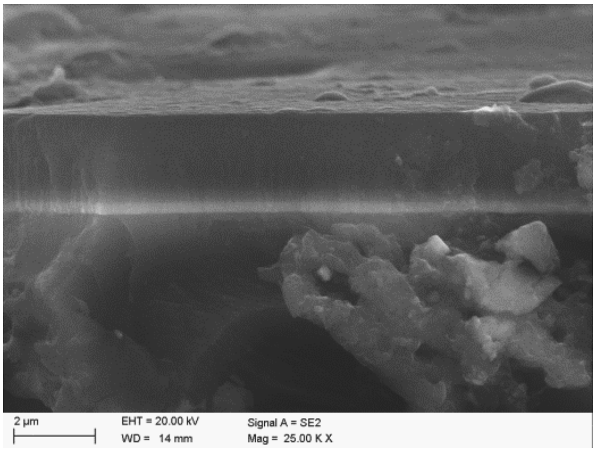

Fig. 5. Fracture of the Ti/DLC/DLC coating on the MCMgAl9Zn1 cast magnesium alloy

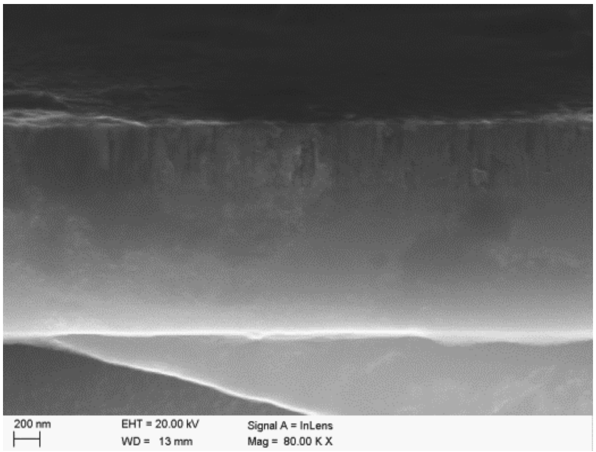

Fig. 6. Fracture of the Cr/CrN/TiN coating on the MCMgAl9Zn1 cast magnesium alloy

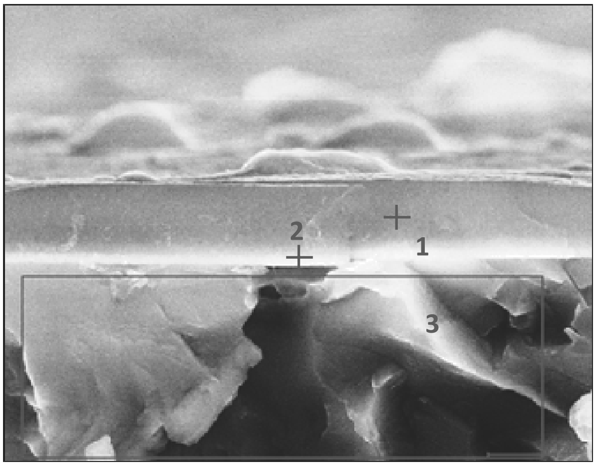

Fig. 7. Cross-section SEM images of the Ti/DLC/DLC coating deposited onto the MCMgAl9Zn1 substrate

Table 3.
The results of quantitative chemical analysis from third 1, 2, 3 areas of coating Ti/DLC/DLC deposited onto substrate from MCMgAl9Zn1 alloy marked in Fig. 7

Chemical element	The mass and atomic concentration of main elements, %	
	mass	atomic
	Analysis 1 (point 1)	
C	92.80	95.92
Mg	3.38	1.04
Al	0.52	0.24
Si	3.30	2.8
Matrix	Correction	ZAF
	Analysis 2 (point 2)	
C	76.59	89.31
Zn	00.84	00.18
Mg	12.56	7.23
Al	1.55	00.81
Ti	8.46	2.47
Matrix	Correction	ZAF
	Analysis 2 (point 3)	
Zn	5.67	2.25
Mg	67.38	71.85
Al	26.95	25.90
Matrix	Correction	ZAF

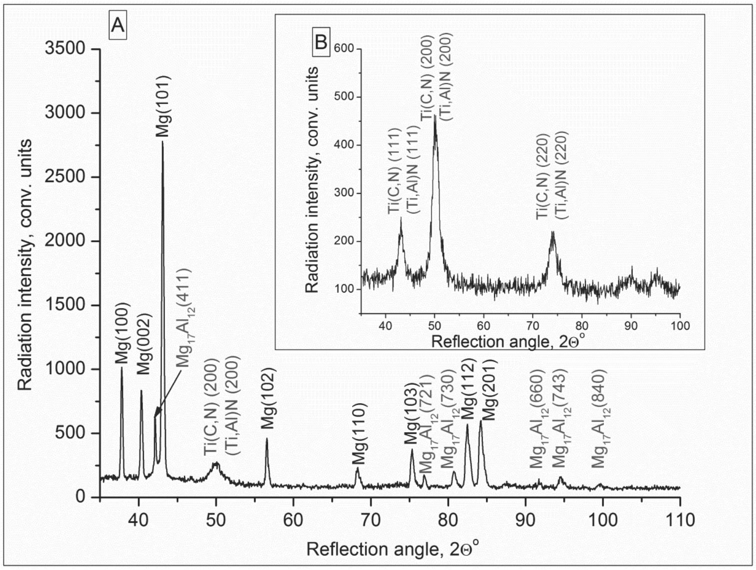

Fig. 8. (A) X-Ray diagrams of the Ti/Ti(C,N)/(Ti,Al)N layers coated on the MCMgAl9Zn1cast magnesium alloy, obtained using the Bragg-Brentano method (B) X-Ray diagrams of the coating performer in a stable angle of α=4°

The layers obtained in the PVD arc process as well with plasma-assisted PACVD on the substrate of magnesium alloy shows significantly higher hardness (Fig. 9). The microhardness increase in the investigated cases should be associated with change of the chemical and phase composition of the coatings, as well with conditions and type of the applied PVD or CVD method, the substrate material as well the combination of the obtained layers. In case of coatings produced in the cathodic PVD process in a nitrogen atmosphere of the N_2 type: Cr/CrN/CrN; Cr/CrN/TiN; Ti/(Ti,Si)N/(Ti,Si)N it was found a clear increase - over 100% - in microhardness compared to the microhardness of the substrate material (after precipitation hardening). The results of microhardness measurement of the abtained coatings did not exceed in this case the value of 2000 HV (Fig. 9). Whereas for the nitride and nitro-carbide coatings obtained in an environment containing CH_4 and N_2 of the type Ti/Ti(C,N)/CrN, Ti/Ti(C,N)/(Ti,Al)N the highest microhardness increase of the surface was higher than 2000HV. For the case of DLC coatings produced by the chemical vapour deposition process, the measured microhardness was about 2000 HV (Fig. 9).

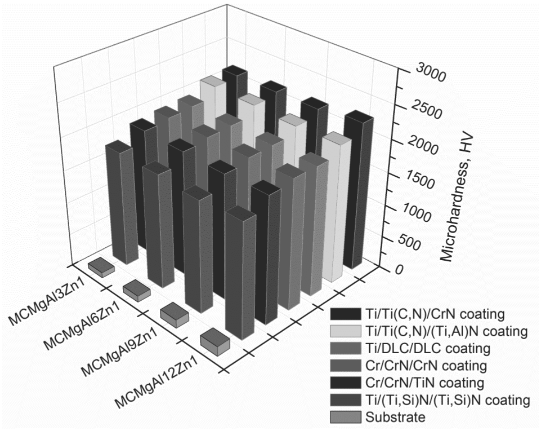

Fig. 9. Diagram of the microhardness results of the investigated layers onto the cast magnesium alloys

Hardness of the obtained coatings correlated tightly with their wear resistance (Fig. 10). However, it is not always important and crucial for the friction pair, that the surface hardness increase; a good example for that are self-lubricating DLC coating, which reduces friction (friction coefficient), and that fore protect the surfaces against wear. According to the applied load of 5N, the average friction coefficients for DLC coatings, obtained with the sliding speed of 0.05 m/s, will be in the range from 0.08-0.15 (Fig. 10), one order of magnitude lower - compared to the friction coefficient of the other examined coatings. This condition is characteristic for DLC coatings composed of graphite, which behaves in the abrasion process similar like a lubricant, also settled on the counterparts. Moreover, a high movement rate with the associated heat accumulation makes it easier to create a self-lubricating layer, what leads to a lower friction coefficient [7,19-21]. The results of sliding distance for the DLC coatings were measured at a level exceeding even 70 times the results of the sliding distance of the coating - for example Cr/CrN/CrN (Fig. 10). The obtained values of the sliding distance for all investigated coatings were measured in a wide range of 6 to 630 m (Fig. 10).

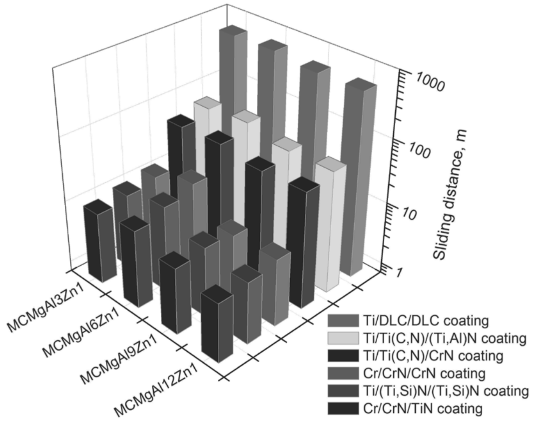

Fig. 10. Diagram of the wear path length until a breakthrough of the layer during the wear test (ball-on-disk) of the investigated layers coated onto the cast magnesium alloys

4. Summary

Increase of the exploitation life and stability of elements produced from the Mg-Al-Zn alloy is also possible using the processing techniques of surface layer refinement in the vapour deposition process. The achieved coatings are characterized by a clear heterogeneity of the surface associated with the occurrence of a number of microparticles in the structure in the shape of droplets sputtered during the deposition process and some cavities occurring due to falling out of some droplets during solidification. As a result of fracture investigation performed using the scanning electron microscope, of the analysed PVD and CVD coatings it was found, that the deposited coatings are characterized by a single, double, or multi-layer structure according to the applied layers system, and the individual layers are coated even and tightly adhere to the substrate as well to each other. Moreover, the analysis of coatings obtained on the surface of cast magnesium alloys by the PVD and CVD processes, show a clear - over 100% - increase of the microhardness, compared to the base material microhardness. The increase of the surface microhardness of magnesium alloys as a result of the coating deposition from gas phase often corresponds with increased wear resistance of the investigated substrates, like in the case of in of Ti/Ti(C,N)/CrN and Ti/Ti(C,N)/(Ti,Al)N coatings. According to the applied load of 5N, the best results of the sliding distance were obtained for the DLC coatings, and were measured on a level exceeding even 70 times the results of the sliding distance, for example for the Cr/CrN/CrN coating, which was comparable with the hardness of diamond coatings.

Acknowledgements

Research was financed partially within the framework of the Polish State Committee for Scientific Research Project No. 4688/T02/2009/37 headed by Dr Tomasz Tański.

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