

*AZ31 magnesium alloy, PACVD technology,
carbon coating, optical profiler,
roughness parameters*

Marcin GOLABCZAK¹
Philippe JACQUET²

THICKNESS EVALUATION OF THE CARBON COATINGS MANUFACTURED ON MAGNESIUM ALLOYS USING OPTICAL PROFILER

One of the problems in identification of carbon nanocoatings is measurement of their thickness. There are a lot of methods which allow for determination of thickness i.e. SEM analysis of cross section of sample with manufactured coating, nanoindentation analysis with using DSP (Digital Signal Processing) method, roughness analysis with using different kind of profilers. In this paper the technology of manufacturing of carbon coatings on AZ31 magnesium light alloy has been depicted and optical profiler has been used for thickness evaluation of these coating. Obtained results allowed for precise estimation of manufactured carbon coatings thickness. In this way it was also possible to compare obtained thickness results with thickness evaluated by other methods.

1. INTRODUCTION

Carbon coatings have been characterized by very attractive functional properties, especially by decorative and protective, which predestine them for application in many fields. At present, there are many methods and techniques which are used for their manufacturing, among them dominate techniques exploitative plasma, ion beams and methods of unconventional synthesis [1-3]. Diversity of these methods and wide range of applied parameters have essential influence on quality of manufactured carbon coatings. For investigations AZ31 magnesium alloy samples have been used. AZ31 (ASTM designation) is very commercial alloy used in die casting and plastic forming. The chemical composition in wt% of AZ31 is: 3% Al, 1% Zn, 0.2%Mn, Mg-balance [4-6]. Presented investigation results concern measurements of geometrical microstructure of AZ31 magnesium alloy surface layer with manufactured carbon coating and without it with using optical profiler.

The differences in geometrical microstructure of coated and uncoated sample has determined the thickness of carbon coating. The obtained thickness results have been compared with earlier investigations.

¹ Technical University of Lodz, 90-924 Lodz, Poland

² École Catholique d'Arts et Métiers, 69321 Lyon, France

2. PREPARATION OF AZ31 MAGNESIUM ALLOY SAMPLES FOR INVESTIGATIONS

Polishing process of samples made of AZ31 magnesium alloy has been carried out using Phoenix Beta 2 Buehler-Germany dual platen grinder-polisher machine equipped with Vector power head and specimen holder for single force for 3-6 specimens up to max \varnothing 25 mm, according to holder selected. Thus 3 to 6 specimens can be prepared under reproducible conditions. The Buehler grinder-polisher machine has had stepless rotation speed from 30 to 600rpm and the power head settings of control time, pressure up to 200N, speed and direction and automatic start and stop system [7].

Vector power head upgrades the Beta 2 grinder-polisher machine to from manual operation to semi-automatic operation, increasing productivity and specimen consistency. This stand is on equipment of Department of Production Engineering of Technical University of Lodz - Poland laboratory.

The technological conditions of realized operations of grinding, lapping and polishing have been estimated basing on experimental investigations and shown in Table 1.

Elaborated technological process has ensured suitable preparation of samples, in range of required roughness parameters and their proper purity (including removal of machining products). The surface of polished samples has had silver, glossy colour and no visible tool marks.

Table 1. Conditions of technological process of AZ31 magnesium alloy samples preparation

Process stages	Abrasive surface	Type of abrasive material	Lubricant type	Process time [min]	Feed force [N/cm ²]	Rotation speed of platen V [m/s]
Grinding	BuehlerMet silicon carbide abrasive paper	Silicon carbide SiC P 600 (grits size \varnothing 26 μ m)	Wax	5	10	6
	BuehlerMet silicon carbide abrasive paper	Silicon carbide SiC P 1200 (grits size \varnothing 15 μ m)	Wax	1	5	3
Lapping	Medium hard woven silkcloth VerduTex	Monocrystalline diamond suspension MetaDi -oil based (grains size \varnothing 3 μ m)	Oil-based polishing extender Buehler AutoMet Lapping Oil	5	2.5	2
Polishing	Soft synthetic pad ChemoMet	Aluminium oxide (Al ₂ O ₃) final polishing suspension MasterPrep (grains size \varnothing 0,05 μ m)	–	3	2,5	1

3. MANUFACTURING OF CARBON COATINGS BY PACVD METHOD

Carbon coatings have been deposited on AZ31 magnesium alloy by Plasma Activated Chemical Vapour Deposition method PACVD, which has relied on decomposition of methane in electric field with high frequency of 13.56MHz, obtained at the pressure of approximately 12Pa in a working chamber [3],[8]. Processes of PACVD have been realized with using the stand which is on equipment of Laboratory of Material Science of Ecole Catholique d'Arts et Metiers ECAM - France and presented in Fig. 1. It has consisted of the chamber of water cooled plasma reactor, the high frequency electrode fixed to the plate of the base and connected through the condenser (the latter provided the negative potential of self-polarization), generator of high frequency (facilitated production of plasma with high density and maintained the frequency at the constant level), vacuum system and systems of measurement and control. Carbon coatings have been deposited on polished AZ31 HP magnesium alloy sample of diameter \varnothing 50mm and 10mm thickness in two steps comprising the process of ionic digestion of their surface followed by the process of synthesis of these coatings. Parameters of these steps are shown in Table 2. Moreover the sample surface layer has been partially covered by silicon carbide plate in order to obtain after deposition process area with carbon coating and without it. The view of the sample after PACVD process has been presented in Fig. 2.



Fig. 1. The view of the system used for deposition of carbon coatings by PACVD method

Table 2. Optimum parameters of PACVD process

Parameter	Ionic digestion of the surface	Process of coating deposition
Feed gas	CH ₄	CH ₄
Pressure in a working chamber	8 ÷ 10 Pa	12 Pa
Time of process - t	4 min	5 ÷ 9 min
Gas flow rate - V	5 cm ³ /min	20÷60 cm ³ /min

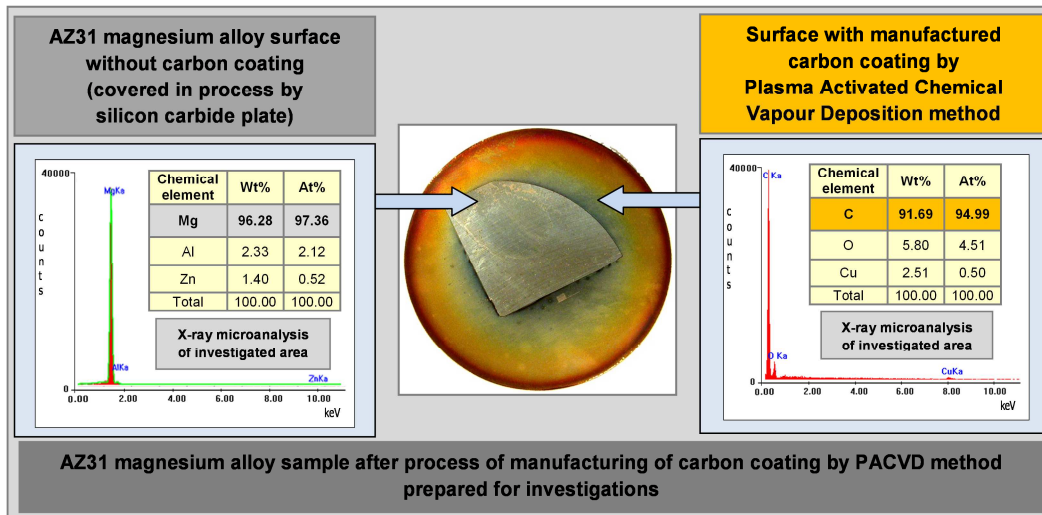


Fig. 2. View of the AZ31 magnesium alloy sample after process of manufacturing of carbon coating by PACVD method

The X-ray microanalysis EDX of prepared AZ31 magnesium alloy sample has confirmed chemical composition of this alloy and that in uncovered area magnesium has been predominant chemical element however in area with manufactured PACVD coating carbon has been predominant element - Fig. 2.

4. EXEMPLARY METHODS OF THICKNESS ESTIMATION OF THIN COATINGS

Basing on earlier investigations of thin coatings we are able to estimate the thickness of such a coatings using different methods: SEM analysis of cross section of sample with manufactured coating - Fig. 3, with using mechanical profiler - Fig. 4 and basing on data from nanoindentation investigation processed with using Digital Signal Processing - DSP methods i.e. digital filters - Fig. 5.

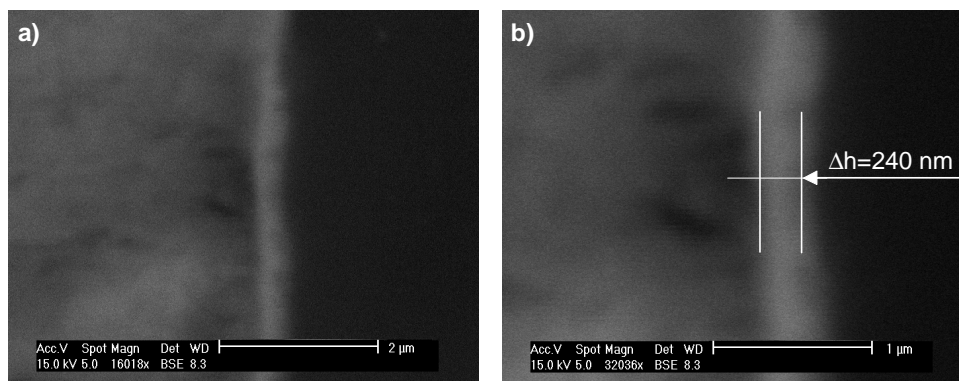


Fig. 3. SEM pictures of carbon coating manufactured on AZ31 magnesium alloy: a) magnification 16000 x, b) magnification 32000 x

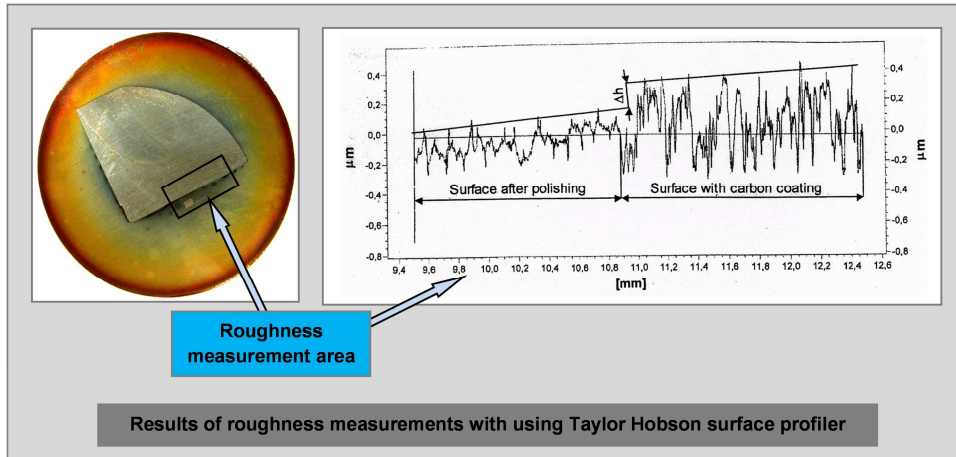


Fig. 4. Results of measurements of the thickness of hard carbon coating deposited on magnesium alloy AZ31 carried out by the method of direct profilography

The thickness of manufactured carbon coatings has been determined using method of simultaneous profiling of two areas of sample surface layer - with carbon coating and without it - Fig. 4. On obtained profilograms we have drawn tangents to roughness vertexes. Distance between them has been the thickness of carbon coating and has been equal to $\Delta h=240\text{nm}$.

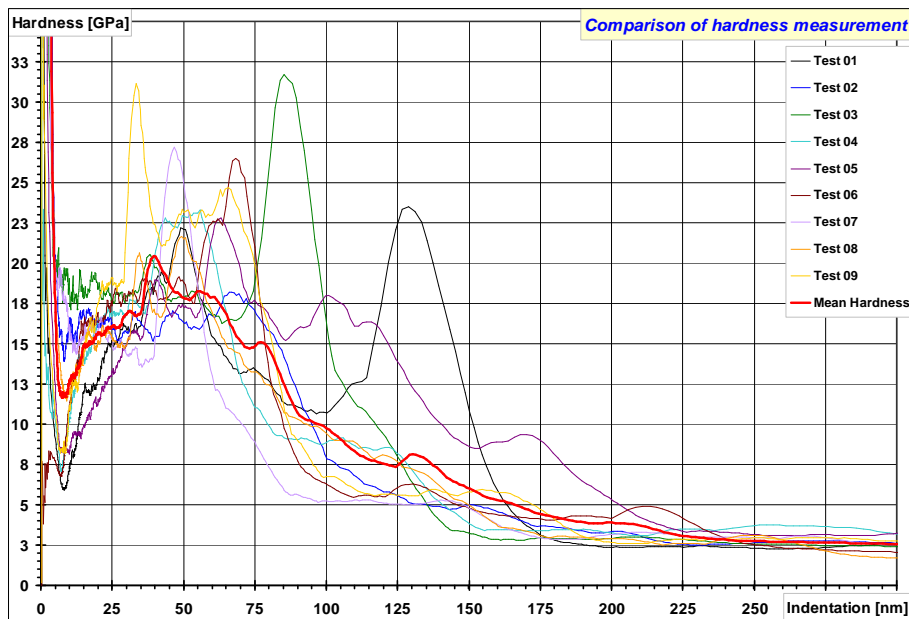


Fig. 5. Estimation of thickness basing on a comparison of coating hardness measurement, DSP elaborated, from different tests performed on the same sample [9]

The nanohardness measurements of manufactured carbon coatings have been carried out using NANO G200 (MTS Nanoindenters - USA) nanohardness tester, equipped with diamond pyramidal penetrator so-called Berkovich penetrator – Fig. 5. For investigations the CSM

(Continuous Stiffness Measurement) method has been used. This method allows for continuous measurement of microhardness in function of depth penetrator. It is possible to estimate the TiN surface thickness basing on data from nanoindentation investigation processed with using DSP (Digital Signal Processing) methods ie. digital filters [9]. Accuracy of DSP processing, and thus carbon coating thickness, could be strongly enhanced by including data related to the mechanical realisation of indentation measurement eq. LVDT actuator.

5. THICKNESS ESTIMATION OF CARBON COATINGS MANUFACTURED ON AZ31 MAGNESIUM ALLOY USING OPTICAL PROFILER

For estimation of thickness of carbon coatings the optical profiler WYKO NT 1100 Veeco Instruments-USA has been used which is on equipment of LaBoMaP Arts et Métiers ParisTech-Cluny in France. WYKO NT 1100 optical profiler is fast and repeatable, provides high resolution 3D surface measurement, from sub-nanometer roughness to millimeter-high steps. The small-footprint NT1100 offers all the advantages of industry-standard WYKO optical profiling, including the full Vision[®]32 analytical software package. Advanced optics ensure sub-nanometer vertical resolution at all magnifications. The Data Stitching option adds a motorized stage for high resolution measurements over a larger field of view. The NT1100 enables accurate, cost-effective metrology for R&D and production of MEMS, thick films, optics, ceramics, and advanced materials [10]. The view of the stand used in investigations has been presented in Fig. 6.

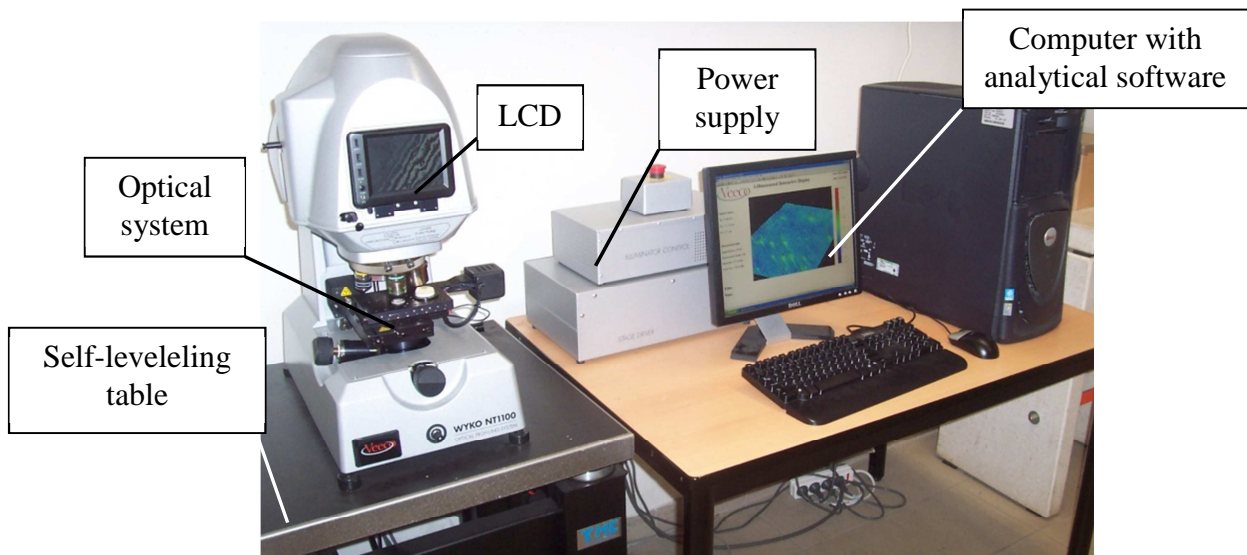


Fig. 6. The view of the stand used in investigations

In the investigations we have used WYKO NT 1100 optical profiler for measurement of roughness of AZ31 magnesium alloy sample with manufactured carbon coating and

without it. Basing on differences in roughness parameters we have been able to estimate thickness of this thin carbon coating and obtain images in 2D and 3D configuration of investigated areas. Exemplary investigation results of roughness measurements on the limit of manufactured carbon coating in different places and obtained 3D images in different magnifications have been presented in Fig. 7.

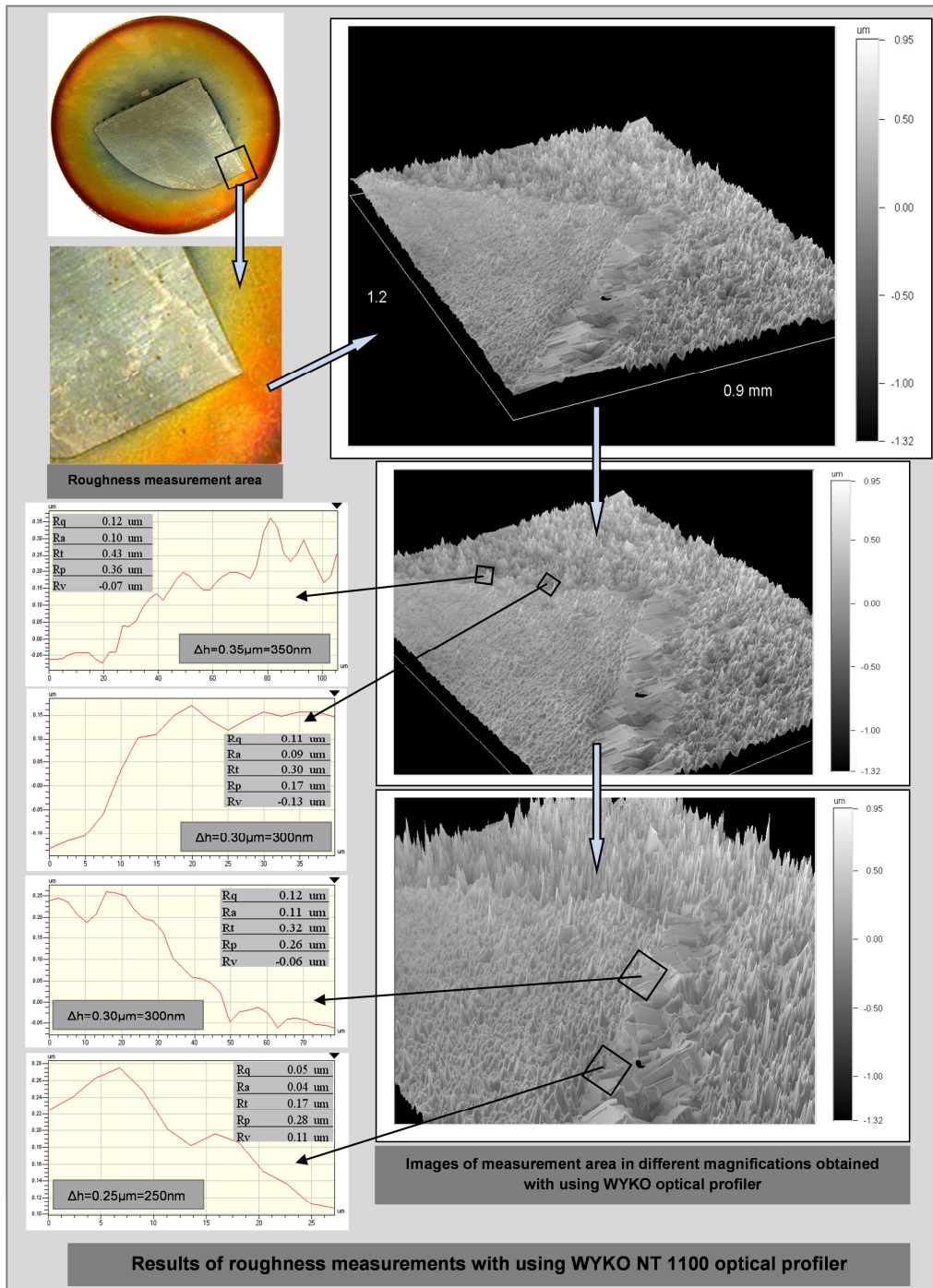


Fig. 7. Exemplary investigation results of thickness evaluation of carbon coating manufactured on AZ31 magnesium alloy with using optical profiler

The thickness measurements of carbon coating and stereometric parameters have been done with using optical profiler - Fig. 7. The thickness of this coating estimated by this method on the basis of R_a parameters has been equal to about 350nm and has been similar to values obtained by direct profiling - Fig. 4.

6. CONCLUSIONS

In the paper the different methods of thickness measurements of carbon coatings manufactured on AZ31 magnesium light alloy by PACVD method, especially with using optical profiler have been depicted. Average thickness of the coatings has been equal to about few hundred nanometers depending on the method used for investigation. Because of this it is very difficult to estimate appropriate their real thickness. Investigations of carbon coating basing on SEM images of cross section has revealed that their thickness has been equal to about 240nm, however basing on method of direct profilography 220nm.

Investigations carried out by nanoindentation method has shown the lowest values of coatings thickness (only about 100nm). This method has shown that it is possible to estimate the carbon surface thickness basing on data from nanoindentation investigation processed with using Digital Signal Processing - DSP methods i.e. digital filters.

The estimated thickness of carbon coatings with using optical profiler has been equal to 250-350nm. It seems that this method gives the most proper results. Basing on this method we can easily observe the measured area in visualization of a configuration space in 3D and measure differences in roughness parameters between area with manufactured carbon coating and uncovered. This method is also very quick and easy to use. It doesn't scratch or destroy the sample comparing to other methods and can be applied for surfaces of high roughness parameters and of complicated shapes i.e. drills, mills, etc.

REFERENCES

- [1] GRAY J.E., LUAN B., 2002, *Protective coatings on magnesium and its alloys-a critical review*, Journal of Alloys and Compounds, 336/1, 88-113.
- [2] WENDLER B.G., 2011, *Functional Coatings by PVD or CVD Methods*, ITeE-PIB, Radom.
- [3] GOLABCZAK M., 2011, *Estimation of carbon coatings manufactured on magnesium alloys*, Special Issues on Magnesium Alloys, InTech, Croatia.
- [4] NAKAURA Y., OKORI K., 1993, *Properties of AZ31 magnesium alloy sheet produced by twin roll casting*, Materials Science Forum, 488-489, 419-426.
- [5] MORDIKE B.L., EBERT T., 2001, *Magnesium Properties - Application- Potential*, Material Science and Engineering, 302, 37-45.
- [6] KHRAISHEH M.K., ABU-FARHA F.K., WEINMANN K.J., 2007, *Investigation of Post-Superplastic Forming Properties of AZ31 Magnesium Alloy*, Mechanical Engineering, 2/289-292.
- [7] <http://www.buehler.com/>
- [8] GOLABCZAK M., 2011, *Characteristics of TiN and NCD layers deposited on magnesium alloys*, Journal of Nano Research, 16, 29-35.
- [9] GOLABCZAK M., KONSTANTYNOWICZ A., 2010, *Notices on method of thickness evaluation of the TiN layers on magnesium alloys*, Defect and Diffusion Forum, 297-301, 641-649.
- [10] <http://www.veeco.com/>