

EVALUATION OF THE PVD AND CVD COATINGS' STRUCTURE USING THE MODIFIED METHOD OF SPHERICAL METALLOGRAPHIC MICROSECTION (BALTEST-M)

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ABSTRACT

The Baltest-M testing method developed at the Institute of Precision Mechanics allows a precise metallographic analysis of the structures of the coating and substrate materials subject to a strong local plastic strain. The material structure of locally strained areological systems (coating: PVD, CVD – substrate: steel, carbides) is revealed on precise spherical polished sections. The essence of this new testing method is the fact that the spherical polished sections are made in the area of indentations formed during hardness testing (HB, HRC, HV, HK of the surface) of the areological system. Material structures of substrate and coating in the indenter strain area are analysed quantitatively and qualitatively. The Kulotester test rig with a high-quality optical system, a rotary x-y bench for a sample holder and a microprocessor controller allows a precise preparation of polished sections within the indentations formed during the hardness test.

INTRODUCTION

The Baltest-M testing method is related to the research conducted at the Institute of Precision Mechanics on hybrid surface strengthening technologies. One of topics investigated during this research is the research on the areological systems obtained using the hybrid disjoint technology [1,3]. The areological system is formed as a result of gas or ion nitriding of tool steels and independent synthesis of coatings, such as TiN, TiAlN, CrN, CrAlN, using the PAPVD methods. Modern, multifunctional coating materials have a complex multilayered structure. [1-3]

One of the key criterions which determine the quality of the PVD coatings on the surface of tools and machine parts and allow them to be used in industrial environments are good results of hardness tests, adhesions tests and fracture resistance tests. Qualitative and quantitative evaluation criteria result from correlations associated with wear and tear [1-4].

A popular adhesion and cohesion test of coating materials is a standardized test of elastic and plastic strain implemented using the spot method as described in the VDI 3198 German industry standard (Bechichten von Werkzeugen der Kaltmassivumformung CVD- und PVD-Verfahren) [4]. This test is recommended for quenched and tempered hot- and cold-work tool steels (steel hardness > 52 HRC) and for sintered carbides covered with 1÷5 µm TiN, CrN, TiC, TiAlN coatings. The method involves evaluation of the deformed surface and corresponding classification of surface quality on a six-point scale [4].

Being spot and static, the Rockwell test method (VDI 3198) does not give information about the behaviour of material structures in their volume (i.e. in cross-section). Other tests of the quality of areological systems involve spot dynamic tests [14], [16]. They allow to obtain the fatigue evaluation of the system and its resistance to cracking, spalling and chipping. The features of damage, i.e. evolution of fractures and delamination depend, among other things, on geometrical parameters of the coating structure, thickness in case of multilayered coatings, coating modulation parameter [4-12].

The new Baltest-M method of evaluation of the substrate-coating system conforms to strict quality evaluation standards of industrial coating materials. The Baltest-M method is a combination of two available testing techniques: hardness measurement (e.g. HV, HRC, HK) and a precise spherical polished section in the plastic strain area of the coating around the indentation. A precise spherical polished section in the indentation area, formed e.g. during the HRC hardness test, is used to perform a metallographic analysis of strained structures of the substrate and coating materials. The method allows a quick and precise evaluation of the quality of PVD, CVD coatings with a complex multilayered structure. A precise spherical polished section in the hardness test indentation area is made on the Kulotest test rig designed at the Institute of Precision Mechanics.

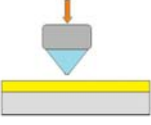
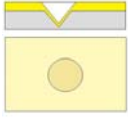


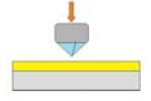


DESCRIPTION OF THE *Baltest-M* TESTING METHOD

The essence of the Baltest-M testing method is combination of two standard available testing techniques:

- hardness measurement (e.g. HV, HRC, HK), and
- a precise spherical polished section in the indentation area.

The diagram of the method used for coatings and layers on flat surfaces is presented in a table 1.

TABLE 1. Sequence of technological operations in the Baltest-M method, flat surfaces

I - Make an indentation with the hardness tester	II - Elastic and plastic strain of coating and substrate	III - Make a precise spherical polished section	IV - Microscopic analysis of revealed structures
 <p data-bbox="256 488 478 517">Rockwell HRC indenter</p>			
 <p data-bbox="256 656 478 685">Vickers HV indenter</p>			

Correct application of this technique to evaluate the structure of layers and coatings requires:

- a hardness measurement using one of the methods, e.g. HB, HRC, HV, with parameters allowing the plastic strain of the coating,
- an analysis of the coating material structure around the indentations, at the edge and inside,
- making a precise spherical polished section in the indentation area – in the indentation axis (Fig.1a) or asymmetrically (Fig.1 b, c).
- an analysis and quantitative evaluation of the revealed coating and substrate material structures on spherical polished sections using optical or electron microscopes.

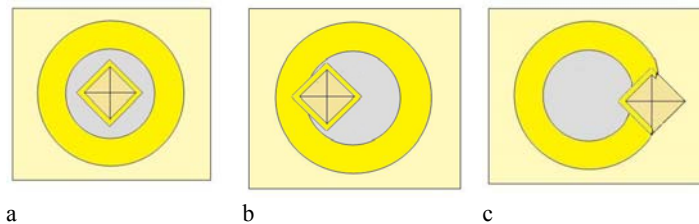


Fig. 1. Structures revealed on the spherical polished section surface in various positions in relation to the Vickers hardness indentation: a – centrally, b – from side inwards, c – from side outwards

During the coating material quality evaluation using the Rockwell hardness test (VDI 3198), in case of the best adhesion quality classes HF1 and HF2, Baltest-M allows to obtain more detailed description. The description relates to the fracture propagation structure inside the substrate and coating materials. The diagram of the proposed classification for Rockwell indentations is presented in Fig. 2. The cases B I – B IV correspond to the following situations:

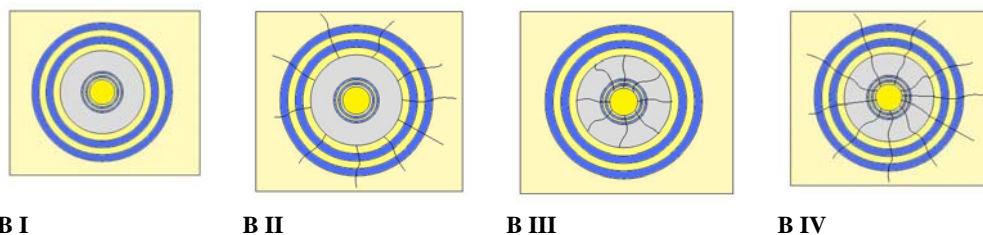


Fig. 2. Structures revealed on the spherical polished section surface of the substrate + multilayered coating areological system in the Rockwell indentation axis: B I – absence of fractures, B II – fractures in the coating, B III – fractures in the substrate, B IV – fractures in the substrate and the coating

- B I shallow, very short fractures, or absence of fractures, in the coating and the substrate,
- B II propagating fractures in the coating,
- B III propagating fractures in the substrate,
- B IV propagating fractures in the coating and the substrate.

The fractures can be linear, curvilinear or mixed, singular or propagating to a number of smaller fractures.

A spherical polished section in the area of indentation formed during the Vickers hardness test, with fractures in the corners visible on the coating surface, allows to observe the character of the fracture propagation in the volume and in the substrate material, Fig. 3, 4.

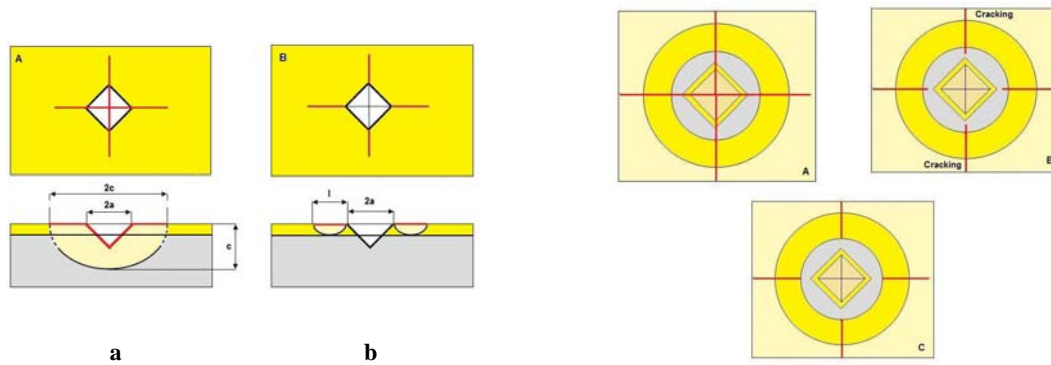


Fig. 3. Propagation of dry cracking initiated by a spot strain of the areological system (substrate + coating) by the Vickers indenter: a – fracture propagation in the substrate and coating material, b – fracture propagation in the coating material

Fig. 4. Structures revealed on the spherical polished section surface of the substrate + multilayered coating areological system in the Vickers indentation axis: a – fractures propagating in the substrate and coating material, b – fractures propagating partially in the substrate and in the whole coating, c – fractures propagating in the coating material

The fractures' propagation analysis gives a possibility of unambiguous determination of their route. A fracture can propagate in the entire volume of areological system (Fig. 2a, Fig. 3a) or only in the layer and coating material (Fig. 2b, Fig. 3b). The obtained data can be used as a basis to find the methodology of determining the K_{Ic} factor (Fig. 6) for coating materials based on the length measurements of fractures formed in the corners of the Vickers indentations.

This data is necessary to test the resistance to the layer dry cracking in order to choose the methodology of determining the K_{Ic} factor [15], [19].

Figure 4 presents three diagrams of the coating structure around the Vickers indentation revealed on the spherical polished section.

- the first one corresponds to the fracture propagation through the indentation corners, the maximum fracture depth h_c (Fig. 3a) is greater than the indentation depth h_{HV} ($h_c > h_{HV}$) (Fig.4a.)
- the second one illustrates a situation where the fractures are located in the substrate and the coating, the maximum fracture depth is less than the indentation depth ($h_c < h_{HV}$) Fig. 4b.
- the third one shows a situation where the fractures (Fig. 3b) are located only in the coating Fig. 4c.

Application examples of the Baltest-M testing method

Analysis of the TiN coating – HSS steel areological system in the area of Rockwell (HRC) and Vickers (HV5) indentations with the spherical polished sections in order to illustrate the Baltest-M testing method.

Strain made using the Rockwell HRC indenter

The analysis was performed on the TiN coating - SW7M steel areological system. The 4 μ m titanium nitrides coating was made using the magnetron technique (Fig. 5, Fig. 6). The coating has a gradient structure with varying nitrogen concentration (Fig7). The fractures visible on the surface around the Rockwell indentation seen on the spherical polished section propagate only in the TiN coating (Fig.5 b). The fracture propagation within the coating is linear. Very small local chipping on the substrate-coating boundary are visible in the indentation edges area (Fig.7 a, b). Such a behaviour of the coating material proves its good adhesion.

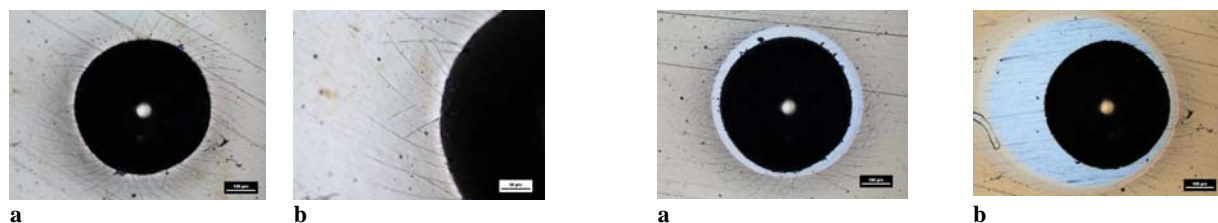


Fig. 5. Rockwell indentation on the surface of the SW7M (1.3343) steel with the TiN coating: a-micro-fractures around the indentation edge, b- spherical polished section within the Rockwell indentation revealing fracture propagation in the coating

Fig. 6. Spherical polished section within the Rockwell indentation edge revealing the TiN coating structure and the fracture propagation features: a – polished section in the indentation axis, - offset in relation to the indentation axis

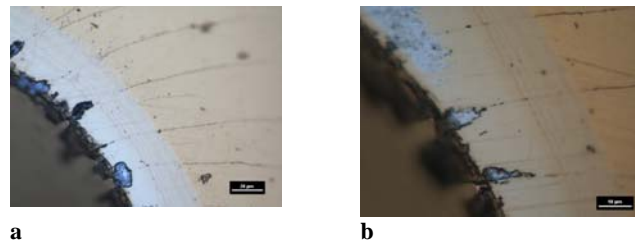


Fig. 7. Spherical polished section within the Rockwell indentation SW7M (1.3343) steel edge with the multi-gradient TiN coating: a- fracture propagation inside and on the coating, b- fracture propagation in the TiN coating initiated on the substrate-local micro-chipping boundary

Strain made using the Vickers HV5 indenter

Strain of the areological system made using the Vickers HV5 indenter is less invasive. The load range can be adapted to control the indenter penetration depth and corresponding strain size. Examples of surface defect structures and the cross-section of a multi-gradient TiN coating on the spherical polished section is shown in Fig. 8 and Fig. 9. The HV5 coating strain generates numerous fractures in the indentation corner and parallel to its edge Fig.8 b. The coating structure revealed in the corner area does not show distinct fractures and chippings (Fig.9b) which proves its good fracture resistance in the conditions of the generated HV5 load.



Fig. 8. Defects of the TiN coating on the SW7M (1.3343) steel around the indentation after the HV5 hardness test: a - fractures and micro-chipping of the TiN coating on the indentation edge, b - coating fractures propagating in various directions

Fig. 9. Structure of the multi-gradient TiN coating in the HV indentation area revealed on the spherical polished section surface: a - coating structure, b - brittle fracture propagating in the coating in the Vickers indentation corner

CONCLUSIONS

The Baltest-M method allows to evaluate the behaviour of the internal structure of the coating and substrate materials subject to a strong elastic and plastic strain generated during the HB, HRC, HV hardness tests.

The coating materials structure revealed on the spherical polished section allows a precise analysis of material cracking and chipping phenomena in the areological system, particularly in its cross-section.

The Baltest-M testing method requires further research, especially in the area of its systematic qualitative and quantitative description.

LITERATURA

- [1] Burakowski T.: Areologia , Instytut Technologii Eksploatacji -PIB, Radom 2007.
- [2] Dobrzański L.: Significance of materials science for the future development of societies. "Journal of Materials Processing Technology" 175 (2006) pp 133-148.
- [3] Verein Deutscher Ingenieure Normen, VDI 3198, VDI-Verlag, Dusseldorf (Coating (CVD, PVD) of cold forging tools) 1991.
- [4] Mathias C., Ernst G., Fleischmann W, Christian H. Konrad, Schuetz A., Glatzel U.: Abrasion resistance of oxidized zirconium in comparison with CoCrMo and titanium nitride coatings for artificial knee joints. "Journal of Biomedical Materials Research B": applied biomaterials 2010 vol 93B, 24 pp 4 -251.
- [5] Taktak S., Tasgetiren, S.: Identification of delamination failure of boride layer on common Cr-rased steels. "Journal of Materials Engineering and Performance". Volume 15(5) October 2006, pp. 570-574.
- [6] Wiklund U., Hedenqvist P., Hogmark S.: Multilayer cracking resistance in Bendig. "Surface and Coatings Technology", 97 (1997) pp 773-778.
- [7] Stallard J., Poulat S., Teer D.: The study of the adhesion of a TiN coating on steel and titanium alloy substrates using a multi-mode scratch tester. "Tribology International", 39 (2006) pp.159-166.
- [8] Harris S., Doyle E.D., Vlasveld A.C., Audy J., Quick D. A.: Study of the wear mechanisms of Ti1-xAlxN and Ti1-x-yAlxCrYn coated high-speed steel twist drills under dry machining conditions. "Wear" 254 (2003) 7 pp 23-734.