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Method for determining the composite and real hardness of hardened surfaces considering the effect of retained stresses in the coating

M S Pugachev and N A Voronin
Mechanical Engineering Research Institute of the Russian Academy of Sciences, 4 Maly Kharitonyevsky Pereulok, Moscow, 101990, Russia

pugachevmax@mail.ru

Abstract. A computational and experimental method for determining the real hardness of the coating material and the effective (composite) hardness of the surface of engineering products hardened with hard protective coatings is developed. This method can be used to control the quality and reproduction of the technology of constructing parts with protective coatings.

1. Introduction
Hardness value of the surface layers is of a significant interest to predict the performance and tribotechnical properties of materials used in friction units. Hardness of the materials is determined by the method of indentation in the surface of the test material. Brinnel hardness (BH) is widely used for metal surfaces of the low and medium hardness and for hard and superhard compact materials, as well as coatings, including thin one – Vickers hardness (VH) [1].

Method for determining the Vickers hardness (VH) is to indent a four-sided diamond pyramid with a square base into the surface of the test material. Berkovich triangular pyramid of is widely used. Nowadays the method of evaluation of mechanical characteristics of materials by the kinetic (instrumental) diagram "load-introduction" is widely used. The method consists in continuous indentation of the indenter into the surface of the sample with recording of the diagram of indenter introduction during loading and unloading [2]. Most of the research is conducted using diamond pyramids such as Vickers or Berkovich. The loading branch is used to determine the plastic characteristics of the material, in particular hardness.

Hardness (microhardness) of the coated surfaces is determined by the size of the imprint left by the diamond indenter after its indentation with a given load or by the depth of penetration under normal load.

If it is necessary to obtain information about the hardness of the coating material only, the measurement procedure is performed when small loads are applied to the indenter, which ensure the introduction of an indenter into the coating surface to a depth of not more than a tenth of the coating thickness. In the technique of measuring the hardness of thin coatings on the basis of numerous experimental data it is assumed that the values of the measured hardness corresponds (or approaches) to the value of the hardness of the coating material only when the thumb rule is fulfilled, which requires that the depth of the imprint under the diamond prism will be 0.08 – 0.1 of the coating thickness. At large depths of penetration the calculated microhardness of the surface is the value of the
composite (effective) one considering the hardness not only of the coating, but also of the base material.

The technology of producing thin coatings and surface modified layers is continuously developing. Technologies based on the effect of highly concentrated flows of energy and matter on the surface are widely used in industry over the last years. In some cases, if not most, the surface layers may differ significantly from the base material (substrate) in physical and mechanical characteristics. During operation of products with thin coatings the retained stresses affect the performance inevitably arising in the process of obtaining the coating. They make their own sometimes significant contribution to the formation of the stress field in the surface volume of the coated product material. The depth, type and magnitude of retained stresses depend on the type and mode of processing. The strength of the surface layer will be largely determined by the presence and type of retained stresses. If the deformation of the material in contact is elastic, the stress at any point is a superposition of the retained microstresses and stresses generated by the external load. It is necessary to avoid the occurrence of retained stresses in the parts of the same sign as the voltage from the operating loads.

The problem of assessing the quality of the coating after its manufacture is inextricably associated with the problem of assessing the reproduction of the process. In modern technologies based on the impact of high-energy corpuscular fluxes on the surface, the synthesis of chemical compounds in the form of a thin coating occurs on the working surface of the hardened product under extremely non-equilibrium thermodynamic conditions, as a rule in a volume isolated from the external environment (reactor) at a reduced working gas pressure or in vacuum. Processing in most cases is multiprocess. The products (machine parts or tools) are located in the volume of the reactor (chamber) at different distances and not with the same orientation with respect to the generator of the corpuscular flux. Tools affect the temperature distribution in the chamber during the processing of products. All this results in a fairly high percentage of defective processed products, even for the waste of the production process of hardening (e.g., carbide profile cutting of plates, drills, rings, mechanical seals, plungers, fuel pumps). It is important to have indicators that assess the quality of coated products and ways to quantify such indicators.

The objective of the study is to develop a method for assessing the real hardness of the coating and the composite surface hardness of the coated product, which takes into account the contribution of the magnitude and type of retained stresses in the coating.

2. Experimental details

The following types of coatings are used as the targets of research: diamond-like coating (DLC) applied to the substrate of 12X18H10T steel, titanium nitride coating on the substrate of D16T aluminum alloy, aluminum nitride coating on the substrate of 12X18H10T steel. All coatings are applied using ion-plasma method.

Coating thickness was measured by S3P profilometer (Germany) by scanning the step obtained as a result of local screening of the treated surface with a metal screen.

The hardness of the coated surface was measured by PMT3 microhardness tester with an indenter in the form of Vickers pyramid.

3. Experimental procedure

There are usually high retained stresses of different types on the surface of the samples with the test coatings. To remove these stresses the authors propose a method that is performed as follows:

- coating thickness \( h \) on the surface of the product is determined;
- based on the geometry of the pyramidal indenter the size of the diagonal \( d_h \) of indenter is calculated at a distance \( s \) equal to the coating thickness from the tip of the indenter;
- two slots identical in width and length are cut in the coating perpendicular to the surface with a depth that equals not less than the coating thickness figure 1, in the local place of the test surface of the coated product at a distance \( l \) from each other not less than the triple value of the calculated diagonal \( d_h \), but not more than four times of its value;
the coated product is placed in microhardness tester;
• a local place is combined with slots relative to the tops of the pyramidal indenter of microhardness tester in such a way that the indenter tip gets into the center of the local area under study between the two slots on further loading of the indenter;
• procedure of indentation in the test area of the surface is carried out by the method of the restored print by step increase in the load;
• load values \( P_i \) are recorded at each step of loading fixing the average value of the diagonal of the imprint \( d_i \) (according to the results of the measurement of two diagonals of the imprint); Step loading keep to the diagonal of the imprint value equal to or close to \( d_i \);
• depth of penetration \( s_i \) is calculated at each step by formulas (1) and (2):
    - for a quadrangular pyramid with a square base (Vickers pyramid):
      \[
      s_i = \frac{d_i}{7}
      \]  
    - for a triangular pyramid with a base in the form of an equilateral triangle (Berkovich pyramid):
      \[
      s_i = \frac{d_i}{6.43}
      \]  
• the current values \( HV_i \) of the surface layer material are calculated using the known formulas [2]:
    - for a quadrangular pyramid with a square base (Vickers pyramid):
      \[
      HV_i = \frac{P_i}{26.43s_i^2}
      \]  
    - for a triangular pyramid with a base in the form of an equilateral triangle (Berkovich pyramid):
      \[
      HV_i = \frac{P_i}{26.44s_i^2}
      \]  
• the curve is built on the base obtained from experiments for penetration into the surface with a coating of array data on hardness and the corresponding penetration depth \( HV_i = \varphi(s_i) \) in coordinates \( HV_i - s_i \) or curve \( HV_i = \varphi(s_i/h) \) in coordinates \( HV_i - (s_i/h) \);
• within the penetration depth values of the obtained diagram from ~ 0.1 \( h \) up to ~ 1 \( h \) the curve \( HV_i = \varphi(s_i) \) is the curve of change of dependence of composite hardness of the surface with the coating considering the absence of retained stresses in the coating;
• within the penetration depth values (0.08 – 0.1) \( h \) hardness value on the curve \( HV_i = \varphi(s_i) \) is the evidence of real hardness of the coating considering the absence of retained stresses in the coating.

The method provides the use of parallel slots as alternative options, the use of rectilinear slots converging at an angle to each other or curved, including annular one, figure 1.

4. Verification of experimental procedure
All the samples studied in this work the coating thickness is ~ 10 µm and the initial microhardness varies in the coating depth from the maximum value on the surface to the minimum one closer to the substrate figure 2, 3, 4 curve 1.

Since drilling we cut through only the coating, the release from retained stresses occurs in it; therefore, the maximum depth of the penetration of Vickers indenter should not exceed the coating thickness. We will determine DLC coating, using a known ratio between the height and diagonal of the Vickers pyramid, we find that the value of the maximum depth of the indenter penetration, i.e. 10 µm, corresponds to the diagonal of the imprint of 70 µm and multiply it by 4, to obtain the size \( l \) of the distance between the slots, which will be 280 µm.

We drill the coating 3 see (figure 1) up to the substrate (base) with a core drill with a diamond abrasive at the end with an external diameter of 1 mm, moreover, an internal diameter is approximately 0.8 mm, figure 1. To obtain the required diameter of the inner platform 6 of the coating remaining after drilling, it is necessary to move the drill along one axis 4 in parallel, changing the
distance between the drilling centers. You can just change this distance arbitrarily and find a suitable size of the platform 5 or 6 after drilling. To obtain an inner platform 6 coated with a transverse axis size of 280 microns it is necessary that the distance between the drilling centers will be approximately 520 microns (depends on the cartridge beat with a core drill). After drilling we place the sample in the hardness tester and choose the place of hardness measurement, the center of the coated area 6. We measure the hardness at a depth of 0.9 to 1 coating thickness to determine the composite hardness of the coating corresponding to the obtained size of the platform 6, i.e. 280 microns. Its own platform 5 or 6 with the calculated size between the slots is created for each diagonal of the imprint and accordingly the penetration depth of the indenter. After drilling it is necessary to make sure that the coated area 6 does not have cracks and damages on the surface.

Figure 1. Drawing of types of slots of the surface layer obtaining different sizes of platforms of coating. 1 - sample (product), 2 – substrate, 3 - thin coating, 4 – displacement axis of the core drill, 5 – coating platform with a large size, 6 - coating platform with a smaller size.

Figure 2. Hardness dependence of DLC coating on the relative depth of indentation. 1 - initial, 2 - after the release of the coating from retained stresses, • - values of the real hardness of coating.

Figure 3. Hardness dependence of TiN coating on the relative depth of indentation. 1 - initial, 2 - after the release of the coating from retained stresses, • - values of the real hardness of the coating.

Figure 4. Hardness dependence of TiAlN coating on the relative penetration depth of the indenter. 1 - initial, 2 - after the release of the coating from retained stresses, • - values of the real hardness of the coating.

5. Theoretical background
According to the proposed method, the study area is allocated on the surface of the coated product see figure 1, the local area of coating 6, where two slots identical in width and length with a depth equal to the minimum coating thickness are formed perpendicular to the surface of the coating. Slots can be
obtained mechanically, through electrosparse or ultrasonic methods, chemical or physical etching. The width of the slots is not subject to strict requirements. They must create safely two free side surfaces in the coating that are not in contact with each other after their formation. An important condition for the formation of the slots is the choice of the method of their manufacture and processing modes, providing the minimum possible force effect and the minimum depth of plastic deformation of the surface on the side walls of the slots.

Slot length is free, but not less than 4 diagonals of the maximum indenter imprint in the test surface during a specific experiment for penetration. It was established experimentally that slot length is from 5 diagonals of the imprint and more reliably ensures the reproduction of the results of the experiment for one point of indentation. When conducting a study in a local place with a multiple number of imprints, the slot length should be increased by a multiple of the number of experimental imprints.

The most important and essential parameter is the distance between parallel slots. Distance value between the slots must ensure removal of retained stresses from previous operations (coating application) and create the conditions for conducting the test without any additional retained stresses from the indentation process.

It is known that the ratio between the maximum penetration depth of the indenter $s$ in the elastic drive (typically $s = 5-50$ nm) and the equivalent radius $R$ of the tip of the real indenter (usually $R > 100-200$ nm) is such that the elastoplastic transition occurs under conditions under which the indentation is actually carried out by a quasi-sphere radius slightly larger than the diagonal of the imprint. The stress state is characterized by the presence of an area of almost pure hydrostatic compression adjacent to the contact surface and propagating to a depth of several tenths of the imprint diagonal [3, 4]. Therefore, the best conditions for the initiation of plastic deformation on indentation exist at a depth $= 0.5$ of the radius of the quasi-sphere from the contact surface. This depth of the initiation point for surfaces with thin coatings is already in the substrate material [5]. This suggests that the deformation of the surface of the coated product is carried out due to the elastic deformation of the coating and the elastoplastic deformation of the substrate, if loading is increased. Consequently, plastic deformation in the coating material during indentation to a depth of more than 0.1 of the coating thickness up to the depth equal to the coating thickness does not occur and deformation of the coating does not create additional retained stresses, which could distort the values of the measured hardness.

Further increase in the indentation force results in the development of elastoplastic deformation of the substrate material and the increase in the size of the locally deformed coating area to comparable with the diagonal of the imprint. Therefore, if the imprint with a diagonal of the imprint $d$ is positioned so that the diagonal of the imprint is perpendicular to the longitudinal direction of the future slot, then the distance between the imprint and the side wall of the slot should be about the size of the diagonal of the imprint. From the symmetry of the local place of study it follows that the distance $l$ between the side walls of the slots should be approximately equal to $3d$ at the central, symmetrical location of the indenter imprint relative to the slots. Moreover, a significant increase in the distance $l > 3d$ causes the increase in the measurement error, since there are conditions when the deformation of the coating begins to experience a hindered deformation from the available material that complicates its deformation. A smaller value of the distance $l < 3d$ is also not preferable, but its effect is less significant, since it is a question of reducing the elastic deformations of the coating and they are much less than the plastic deformations of the substrate. Theoretically it is preferable to place the slots at a distance from each other on the value $(3-3.5)d$. Experience has shown that the upper limit of the distance range $l$ is not more than $4d$ functional in this method.

6. Measurement results

Figure 2, 3 and 4 show the measurement results of the real hardness of the coating and the composite hardness of the surface coating for all samples with coatings under study.
This method, according to the above dependencies, allows comparing the coatings among themselves by the type of the available retained stresses. Figure 2 shows that microhardness falls at all indentation depths after drilling, which indicates the presence of compression stresses in the initial coating. Figure 3 shows on the contrary that tensile stresses are present on the surface of the initial coating, since microhardness increases after drilling and this is true only up to the middle of the coating thickness. When the depth of indenter penetration is more than 5 µm, the microhardness does not change almost, which suggests the absence of retained stresses at a depth below the middle of the coating. Figure 4 shows that the initial coating contains also tensile stresses, but over the entire depth.

These dependences confirm the well-known statement that the greatest retained stresses affect the coating surface; this is evidenced by the large differences between the hardness of the initial hardness and after drilling at the low load and small differences at the high load.

7. Conclusions
Method for determining the real hardness of the coating material and the composite hardness of the surface of engineering products hardened with hard protective coatings is developed.

This method of determining the hardness of hardened surfaces can be used to control the quality and reproduction of the technology of construction parts with protective coatings, including friction units. The proposed method for determining the real hardness of the coating and the effective surface hardness of the coated product is applied for the invention.

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