Features of Measuring the Hardness of a Metal Surface Modified with Ultrafine Particles of Minerals

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Abstract

One of the important characteristics of the surface properties of metal parts subjected to friction is hardness. Hardness measurements are important for determining the operational characteristics of parts and monitoring the technological regimes of surface modification. However, hardness measurements of thin modified layers made by different methods can lead to differences in measurement results. The aim of the article was to study the hardness of a metal surface modified with ultrafine particles of minerals by two different methods (instrumental indentation and Vickers hardness measurement) and a comparative analysis of the measurement results obtained by these methods.

Standard Vickers hardness measurements at loads of 0.025, 0.1 and 0.5 kgf showed a qualitative difference between the hardness values of the two samples modified with different mixtures of ultrafine particles of minerals and a large heterogeneity of the hardness values over the area. By the method of instrumental hardness, standard measurements were performed without preliminary selection of the indentation site (at a load of 1.05 N) and measurements during indentation into even sections (at low loads of 10 mN).

It is noted that the high precision of measurements implemented by instrumental indentation, due to the large roughness of the samples, leads to large values of the error in calculating the measurement results. An additional difference in the results of measurements performed by two methods at shallow indentation depths may be due to the fact that the object under study has a complex structure consisting of a metal matrix and particles distributed over the depth of the sample. A possible way out of the situation lies in the transition from the use of hardness measures when calibrating instruments to standard samples of properties for which the constancy of mechanical properties in the measured range of indentation depths will be ensured, but which are not yet available in research practice. Therefore, at present, when carrying out work related to the search for optimal conditions for obtaining thin wear-resistant layers on the surface of metals modified with ultrafine particles of minerals, comparative measurements performed by one measurement method are recommended.

Keywords: hardness, metal surface, Vickers measurements, industrial indentation, mineral coatings.

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Особенности измерения твёрдости металлической поверхности, модифицированной ультрадисперсными частицами минералов

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Одной из важных характеристик свойств поверхности металлических деталей, подвергающихся трению, является твёрдость. Измерения твёрдости важны для определения эксплуатационных характеристик деталей и контроле технологических режимов модификации поверхности. Однако измерения твёрдости тонких модифицированных слоёв, выполненные разными методами, могут приводить к различию результатов измерений. Целью данной работы являлось исследование твёрдости поверхности металла, модифицированной ультрадисперсными частицами минералов, двумя различными методами (инструментального индентирования и измерения твёрдости по Виккерсу) и сравнительный анализ результатов измерений, полученных этими методами.

Стандартные измерения твёрдости по Виккерсу при нагрузках 0,025, 0,1 и 0,5 кгс показали качественное отличие значений твёрдости двух образцов, модифицированных разными смесями ультрадисперсных частиц минералов и большую неоднородность значений твёрдости по площади. Методом инструментальной твёрдости выполнены стандартные измерения без предварительного выбора места индентирования (при нагрузке 1,05 Н) и измерения при индентировании в ровные участки (при малых нагрузках 10 мН).

Отмечено, что высокая прецизионность измерений, реализуемая методом инструментального индентирования, из-за большой шероховатости образцов приводит к большим значениям погрешности при расчёте результатов измерений. Дополнительную разницу результатов измерений, выполненных двумя методами на малых глубинах индентирования, может вносить то, что исследуемый объект имеет сложную структуру, состоящую из матрицы металла и частиц, распределённых по глубине образца. Возможный выход из ситуации заключается в переходе от использования мер твёрдости при калибровке приборов к стандартным образцам свойств, для которых будет обеспечено постоянство механических свойств в измеряемом диапазоне глубин индентирования, но которые пока отсутствуют в исследовательской практике. Поэтому в настоящее время при проведении работ, связанных с поиском оптимальных условий получения тонких износостойких слоёв на поверхности металлов, модифицированных ультрадисперсными частицами минералов, рекомендуются сравнительные измерения, выполненные одним методом измерения.

Ключевые слова: твёрдость, металлическая поверхность, измерения по Виккерсу, индустриальное индентирование, минеральные покрытия.

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Introduction

The properties of the surface layers of the metal, modified with ultrafine particles of minerals, depend on the technological conditions of production, the composition of mineral mixtures and can vary widely. For example, changing only some technological regimes using the technology of mineral coatings [1], which ensure that microparticles of minerals enter the metal, it is possible to obtain modified layers of various roughness Ra (average roughness) on structural steel samples, differing by almost an order of magnitude [2]. The interest in modified layers enriched with microparticles of minerals is due to their prospects as wear-resistant and/or antifriction coatings on the surfaces of friction pairs of various metals – steels [1], titanium [3], aluminum [4] operating in an aggressive environment – abrasive, marine water, in the presence of gases, acid solutions, under thermocyclic loads [5, 6]. Studies show that the thickness of the modified layer, the coefficient of friction, the hardness of the layers depend both on the composition of the mineral mixtures and on the modes of particles entering the metal and the properties of the modified metal surface. In this case, a huge role is played by the quantitative determination of some tribological parameters of the created modified layers, in particular, the determination of such a parameter as hardness for comparison with the macroscopic properties of the part itself and the evaluation of the practical benefits of creating layers [7].

The most common method for determining the hardness of thin modified layers is instrumental indentation, which is the basis of the ISO 14577 standard. The essence of the measurement method consists in the process of indenting a trihedral diamond pyramid (Berkovich pyramidal indenter) with recording the indentation diagram and then calculating the hardness of the dependence of the applied force on the implementation in accordance with standard ISO 14577 [8, 9].

Also, to measure the hardness of thin microlayers, the Vickers method of measuring microhardness (ISO 6507 standard) is used, which is methodologically close to the instrumental indentation method [10]. Its essence is in measuring the diagonal of the resulting fingerprint when an indenter is introduced into the metal in the form of a tetrahedral pyramid (Vickers pyramidal indenter) [11]. However, due to the arising effect of elastic indentation of the indentation when the indentation size is less than 10 μm and the resolution of the optical microscope is limited when measuring the diagonal of an imprint of this size, this method is used to measure the hardness of films with a thickness of at least 10 μm [12].

There are common problems for all methods in determining the hardness of thin layers and coatings, such as the effect of the substrate, surface roughness, and the presence of residual stresses, which can make a significant correction in the measurement results [4]. For example, the influence of the base metal substrate in measuring hardness consists in the fact that the recorded response of the material during measurement depends on the modified layer and on the properties of the metal volume [13].

Such features of thin layers modified with ultrafine particles of minerals, such as the absence of a clear interface between the layer and the substrate [1], as well as the requirement to control not only the properties of the surface layer, but also the depth distribution, increase the requirements for hardness determination methods.

It should be noted that, despite the similarity of the industrial indentation method and the Vickers method, namely, that the indenters have the same projection area with the same penetration depths and the imprint geometry is independent of the penetration depth, their difference is also quite significant [14]. The difference between the methods is that in the Vickers method, hardness is defined as the ratio of the applied load to the surface area of the restored fingerprint, and in the industrial indentation method, the hardness is equal to the ratio of the maximum applied load to the projected area of the unrepaired fingerprint [14, 15]. In general, hardness, as a dimensionless quantity, characterizes the behavior of the material under strictly specified test conditions [12], is not a function of primary physical quantities and depends on the measurement technique [8, 11]. Therefore, situations are possible in which differences in the hardness determination procedures by these two methods can lead to differences in the results of hardness measurements of thin modified metal layers with a thickness of several tens of microns, which is typical when microparticles are modified with minerals. On the one hand, the instrumental indentation method provides the largest locality and precision of measurements [8, 9, 13, 14], the largest of all existing methods for measuring the hardness of thin layers, on the other hand, the hardness tester that implements the instrumental indentation method is a complex laboratory complex, and the price of the instrument, implementing this

method, often differs by an order of magnitude from the price of a Vickers microhardness tester, which makes it difficult to use when scaling the technology. It should also be noted that the implementation of both methods in modern instruments provides automatic hardness measurement, touch control, and automatic focusing.

The purpose of this article is to study the hardness of a metal surface (steel 20X13 − Russian analogue of steel X20Cr13 (EU)), modified with ultrafine particles of minerals, by two different methods (instrumental indentation and Vickers microhardness measurement) and a comparative analysis of the measurement results obtained by these methods.

The development of procedures for measuring such a parameter of thin layers as the hardness of a metal surface modified by mineral particles is aimed at developing solutions to such problems of technological control of layer parameters as adapting methods to specific technological processes, developing automation of measurements and possible remote control of measurements.

Materials and methods

Two samples of steel 20X13, with a diameter of 5 (sample No. 1) and 8 cm (sample No. 2), about 1 cm thick, were made by turning with subsequent standard grinding. On the surface of the samples layers were created, modified with ultrafine particles of mixtures of minerals according to the basic technology of SPC "Geoenergetika" [1]. The layers were created using different types of mineral mixtures under the same technological conditions, which implies the difference in their hardness from each other. The thickness of each modified layer, based on the technological parameters during its creation and earlier experiments, was not less than 20 μm [1, 2]. Comparative measurements of surface roughness, hardness, and elastic modulus (Young) were performed on the samples.

The surface roughness was measured on a Model 130 profilometer (PROTON MIET manufacturer), the measurement method was profilometry. Measurement procedure parameters: profile length − 12.5 mm, profile measurement speed − 0.5 mm/s.

Hardness measurement was carried out by two methods and, accordingly, by two devices:

1. Microhardness meter DuraScan (EMCO-TEST, Austria). Parameters of the measurement procedure: indenter – tetrahedral pyramid of the Vickers type, load range: 0.025 kgf − 0.5 kgf.

2. Nanohardness tester "NanoScan-3D" (manufacturer FGBNU TISNUM). Parameters of the measurement procedure: indenter − a trihedral pyramid of the Berkovich type, loading time – 10 s, unloading time – 10 s, time to maintain maximum load – 3 s, load range: 10mN–1.05 N.

Research results and discussion

The roughness measurement was carried out by measuring the surface profile of the samples, the roughness parameters are shown in Table 1.

Table 1

Comparison of sample roughness parameters

Sample	Ra, μm	Rz, μm	Sm, μm
№ 1	2.03 ± 0.02	10.7 ± 0.3	236 ± 13
№ 2	2.62 ± 0.02	14.3 ± 0.1	206 ± 6

From the Table 1 it follows that the samples have a similar roughness Ra, which is quite predictable, given the constancy of technological conditions. To exclude the influence of such roughness, it is necessary to perform indentation at a depth of ≈ 100 μm, which significantly exceeds the estimated thickness of the modified layer (about 20 μm). At the same time, a large roughness step (Sm ≈ 200 μm) allows one to find sufficiently even sections for the location of the indent, which was done during indentation by the Vickers method.

Before presenting the results of hardness measurements by two methods, it should be noted that the surface after modification is rather heterogeneous due to the fact that the process operations of the technology of mineral coatings lead to the formation of a flat surface (about 90 %) and randomly located microcavities (about 10 %) throughout the entire working sample surfaces [1, Figure 1] (or Figure 6 and 7 of this article, see below). The resulting and existing surface defects of a flat surface, as well as microcavities, are filled with particles of minerals and undergo further technological operations. Particle filling of surface defects of a flat surface, as well as microcracks and microcavities, and changes that occur with defects in a surface hardened layer during further technological operations, increase the wear resistance of the material [2]. But then, when conducting micromeasurements, the question arises about the place of measurements and the

correctness of the obtained parameters for the characteristics of the surface. Given the state of the surface, it is obvious that when carrying out measurements it is necessary to adhere to two measurement strategies:

– taking measurements of consciously selected areas of the modified surface;

– performing measurements on a large number of measurement sites selected at random and statistical processing of the results.

Considering that it is precisely the even sections of the modified surface that play the dominant role in the friction processes, and it is the parameters of the even sections of the surface that are the characteristics of the surface during friction and wear [1], indentation by both methods must be carried out precisely in the even sections of the modified surface. On the other hand, with further automation of the hardness measurement process, indentation locations will be randomly selected, which means that it is necessary to take into account the presence of microcracks and microcavities that will introduce distortions into the final result.

Vickers microhardness measurement

As indicated above, the samples have a significant roughness, and to measure hardness, a flat section was preliminarily selected and then indentation was performed. An example of the image obtained after indentation with a load of 0.5 kgf is shown in Figure 1.

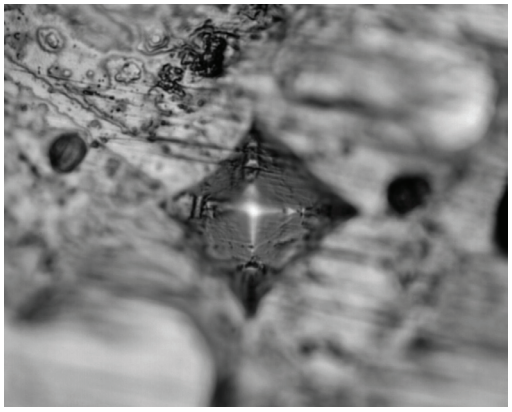


Figure 1 – Optical image of the fingerprint obtained after indentation with a load of 0.5 kgf (magnification ×60)

For measuring the samples, we selected the loads of 0.025 kgf, 0.1 kgf and 0.5 kgf. The measurement results are shown in Table 2.

A graphical representation of the results is presented in Figure 2. Measurement at each load was carried out 5 times, the standard deviation is presented as an error.

Table 2

Vickers (HV) measurement results with different loads

Sample	0.025 kgf	0.1 kgf	0.5 kgf
№ 1	1215 ± 180 (HV)	1080 ± 225 (HV)	382 ± 58 (HV)
№ 2	600 ± 120 (HV)	530 ± 140 (HV)	320 ± 40 (HV)

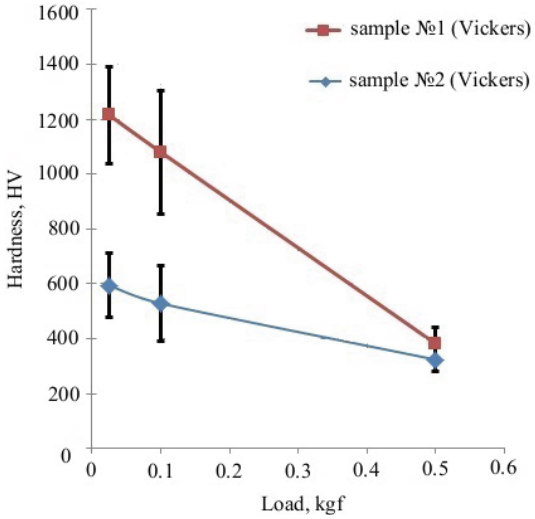


Figure 2 – Dependence of the measured Vickers hardness (HV) on the indentation load

The results of Tables 2 and 3 show a qualitative difference in hardness measured on samples 1 and 2, and a large heterogeneity of hardness (greater than 100 %), measured over the area of the samples.

Table 3

Hardness (HV), measured according to Vickers in different places of the samples

Sample	Hardness in the center of the sample (HV)	Hardness in the middle of the sample, between center and edge (HV)	Hardness at the edge of the sample (HV)
№ 1	933 ± 90	500 ± 145	1133 ± 275
№ 2	563 ± 210	519 ± 50	732 ± 175

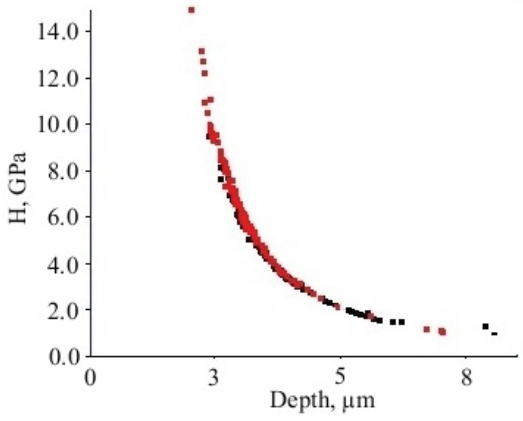
When measuring hardness by instrumental indentation (nanoindentation), an attempt was made to measure with the two measurement

strategies indicated above. It should be noted that the instrumental indentation method can determine the values of hardness and elastic modulus of a material under loads of micronewtons or more. However, it is precisely at these load values that the quality requirements of the modified surface increase.

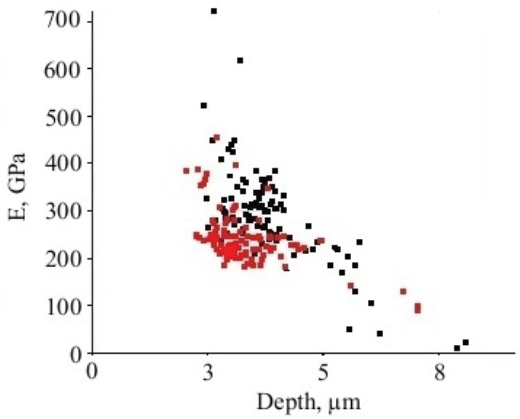
Measurement of tool hardness (hardness determined by instrumental indentation method) and elastic modulus (nanoindentation): processing of a large number of indentes.

Measurements in the nanoindentation mode were carried out as follows: an array of injections was applied to each of the obtained samples with a load of 1.05 N. The measurements were performed without first selecting the indentation site and, thus, were significantly affected by roughness and the presence of microcavities. The array contained 10×10 points with a distance of 300 μm between the points (total size of the indented surface: 3×3 mm). The measurements were carried out with a tip in the form of a Berkovich pyramid, (for estimates: the transverse size of the indent is ≈ 6 times the contact depth, which in this case of small elastic recovery is close to the maximum depth). The results of measuring hardness H and elastic modulus E are shown in the Figure 3 below.

It can be seen that the dependence of hardness on depth is easily described by a curve of the form ~$1/h^2$, which is due to the effect of roughness. The value of hardness and elastic modulus of the material can be determined by the peaks of the distribution density of the measured values N (the maximum value on the histogram) in Figures 4 and 5.



a



b

Figure 3 − Measurement of hardness (*a*) and elastic modulus (*b*) depending on the maximum indentation depth. Red dots − sample № 1; black dots − sample № 2

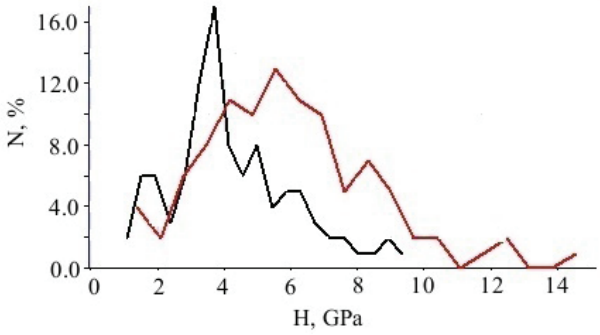


Figure 4 − Distribution of measured hardness. Red dots – sample № 1; black dots – sample № 2

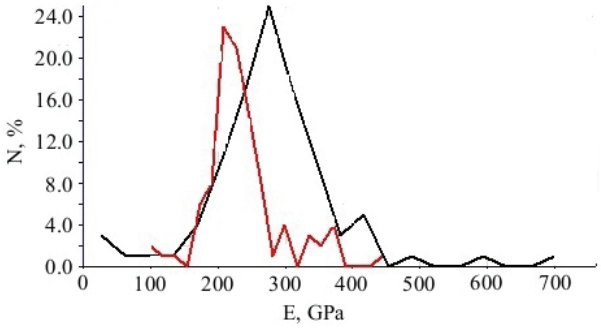


Figure 5 − Distribution of the measured elastic modulus. Red dots – sample № 1, black dots – sample № 2

The positions of the maxima in Figures 4 and 5 correspond to the following values of the hardness H_{hyst} and the elastic modulus E_{hyst}, shown in the Table 4. It also presents the average values of hardness and modulus (index "media"), as well as extreme values.

Table 4

Hardness and elastic modulus of the samples, determined by the distribution maxima of the corresponding quantities

Sample	H_{hyst}, GPa	H_{mean}, GPa	$H_{\min}$, GPa	$H_{\max}$, GPa	E_{hyst}, GPa	E_{mean}, GPa	$E_{\min}$, GPa	$E_{\max}$, GPa
№ 1	5.6	6.2	1.07	14.9	215	243	92	363
№ 2	3.7	4.4	0.89	9.6	275	292	10.4	720

Measurement of tool hardness and elastic modulus (nanoindentation): indentation in flat areas

In both samples, indentation was performed in preselected surface areas. The indentation load was 10 mN, an example of the location of indentation sites for sample No. 1 is shown in Figure 6. A similar image for sample No. 2 is shown in Figure 7. In these photographs, even areas that were selected for nanoindentation, as well as microcavities, which indicated in [1]. Identification with such a small load into preselected flat surface areas is logical, since measurement with such a load in arbitrary places, due to the presence of cavities, caverns and other defects, can lead to measurement results very different from the main population. On the other hand, it is the results of measurements with such a small load that are closest to the values of surface hardness.

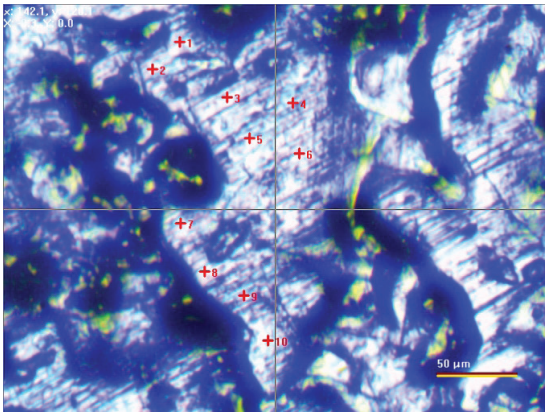


Figure 6 – Optical micrograph of the distribution of indentation sites in sample № 1 (magnification ×60, aperture 0.85)

As a result of processing the obtained data, the dependences of hardness and elastic modulus were obtained, presented in Figure 8 and in Table 5. Measurements significantly different from the main population were deleted.

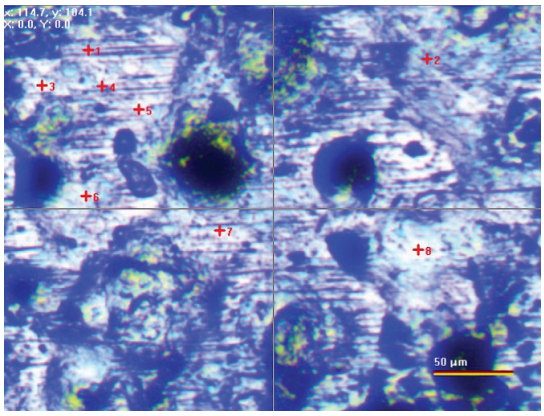


Figure 7 – Optical micrograph of the distribution of indentation sites in sample № 2 (magnification ×60, aperture 0.85)

Table 5

Hardness and modulus of elasticity of the samples, determined when indented in flat areas with a load of 10 mN

Sample	H, GPa	E, GPa
№ 1	14.2 ± 2.1	268 ± 47
№ 2	9.5 ± 2.2	260 ± 33

A comparison of the measured hardness values by the two methods is shown in the Figure 9.

In Figure 9, the values of "nanoindentation" of samples No. 1 and No. 2, determined at a load of 1 H ≈ 0.1 kgf, refer to the indices located arbitrarily.

The values of "nanoindentation 10 mN" on samples No. 1 and No. 2 refer to the indices located on a flat surface area (load 10 mN ≈ 0.001 kgf).

There are a number of methodological sources of uncertainty in the results of measurements of hardness by the above methods: 1) hardware related to the calibration of the measuring installation [8]; 2) methodological associated with assumptions in the calculation methodology [8, 15]; 3) sources associated with the physicomechanical properties

of the studied material [16, 17]. Without considering the hardware and methodological components of the uncertainty of the measurement results by the Vickers and industrial indentation methods, we note the sources of uncertainty associated with the physical properties of the mineral coatings of the metal surface. In particular, the high precision of measurements implemented by the instrumental indentation method leads to the fact that the minimum deviation of the interfering parameters, in particular, the roughness, leads to large error values when calculating the measurement results [13, 14], which is clearly recorded graphically in Figure 9 when comparing results measured by two methods. The role of roughness as an uncertainty factor and the associated error is affected by the actual contact area with the indenter [14, 18], which is especially noticeable when measuring at shallow indentation depths (at a load of less than 0.1 kgf in Figure 9).

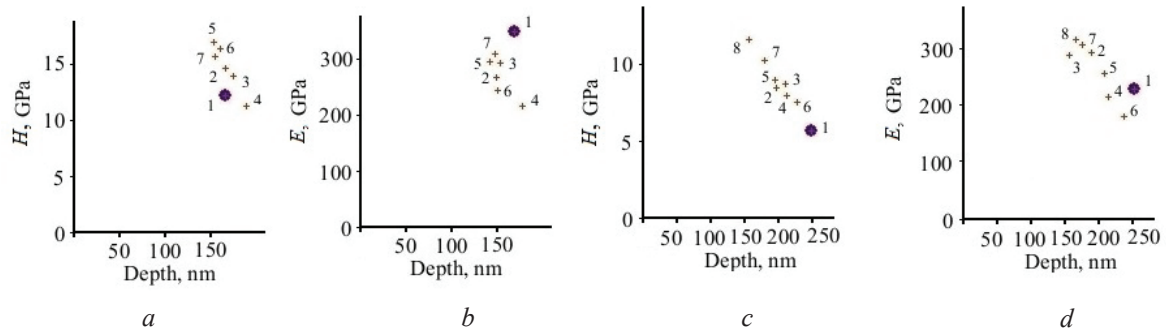


Figure 8 − The dependence of hardness *H* and elastic modulus *E* on the maximum indentation depth for sample No. 1 (*a*, *b*) and sample No. 2 (*c*, *d*)

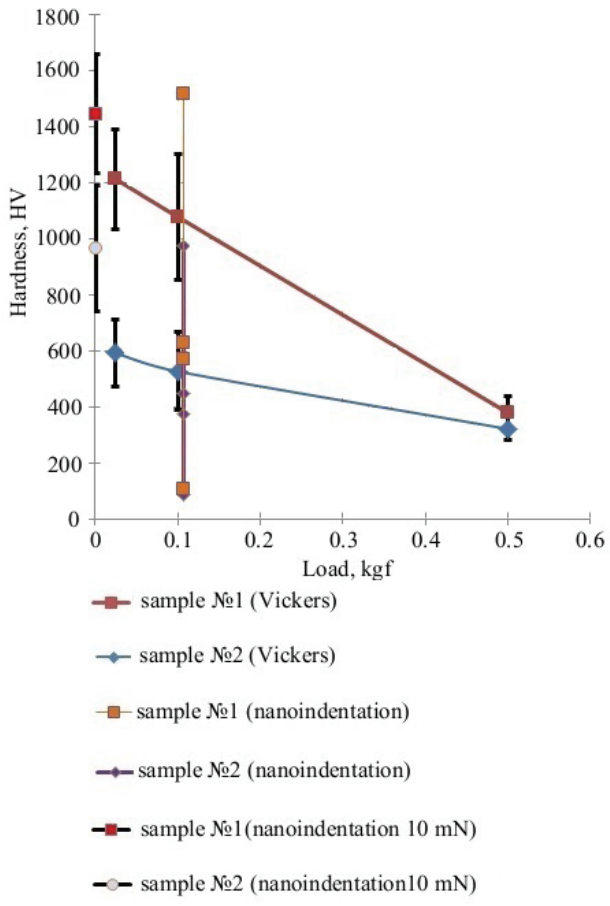


Figure 9 − The dependence of hardness on the indentation load, measured by two methods

An additional difference in the results of measurements performed by two methods at shallow indentation depths, which is clearly fixed in Figure 9, can be made by the fact that the object under study is a complex structured material [1], consisting of a metal matrix and fine particles distributed over the depth of the sample. Given that when calculating the values of hardness and elastic modulus from the load–injection diagram according to the standard method [8, 9], all the calculation formulas are deduced from the assumptions about the interaction of the indenter with a homogeneous isotropic half-space [15, 17], the complex structure of the distribution of particles and how consequence, properties, can lead to distortion of the results. A similar overestimation of hardness values at a small indentation depth, measured on thin modified layers of various metals and alloys, has been observed in many works (see, for example, [18]).

All of the above leads to problems in determining the actual properties of thin layers modified by ultrafine particles of minerals. A possible way out of the situation was identified in several works and consists in the transition from the use of hardness measures in calibrating instruments that implement the instrumental indentation method to standard property samples for which the mechanical properties will be constant in the indentation depth

range in which measurements are carried out (see, for example [19, 20]). However, at the moment, such reference samples are absent both in the markets of materials and research instruments, and in wide research practice. Therefore, the comparison of the results of measurements of the hardness of the layers of the metal surface modified with ultrafine particles of minerals, performed by two methods, the Vickers method and the industrial indentation method, can be carried out with a considerable degree of conventionality, especially at loads less than 0.1 kgf. When carrying out experimental design work related to changes in technological conditions and the search for optimal conditions for obtaining thin wear-resistant layers on the surface of metals modified with ultrafine particles of minerals, comparative measurements performed by one of the measurement methods are preferable.

Conclusion

A comparative study of the hardness of a metal surface (steel 20X13) modified with ultrafine particles of minerals was performed using two different methods (instrumental indentation and Vickers hardness measurement), taking into account the features of measuring the hardness of thin layers modified with ultrafine particles of minerals. Given the state of the surface after modification, indentation by both methods was carried out in flat sections of the modified surface. Additionally, taking into account the perspective automation of the measurement process, the hardness was measured by instrumental indentation at randomly selected locations. Standard Vickers hardness measurements at loads of 0.025, 0.1 and 0.5 kg showed a qualitative difference between the hardness values of the two samples modified with different mixtures of ultrafine mineral particles (1215 ± 180 HV and 600 ± 120 HV for samples No. 1 and 2, respectively) and a large heterogeneity of values hardness by area. By the method of instrumental hardness, standard measurements were performed without preliminary selection of the indentation site (at a load of 1.05 N) and measurements during indentation into even sections (at low loads of 10 mN). In addition, measurements of roughness and elastic modulus were performed.

In discussing the differences in the measurement results performed by different methods, emphasis is placed on the sources of uncertainty in the results associated with the physicomechanical properties of the material under study. In particular, the high precision of measurements implemented by the instrumental indentation method leads to the fact that the minimum deviation of the interfering parameters, in particular, roughness, leads to large values of the error in calculating the measurement results. An additional difference in the results of measurements performed by two methods at shallow indentation depths can be made by the fact that the object under study is a material with a complex structure. All of the above leads to problems in determining the actual properties of thin layers modified by ultrafine particles of minerals.

A possible way out of the situation lies in the transition from the use of hardness measures in the calibration of instruments that implement the instrumental indentation method to standard samples of properties for which the constancy of mechanical properties will be ensured in the range of indentation depths in which measurements are carried out. Given the absence of such samples, it is preferable, when conducting experimental design work, related to changes in technological conditions and the search for optimal conditions for obtaining thin wear-resistant layers on the surface of metals modified with ultrafine particles of minerals, are comparative measurements performed by one of the measurement methods.

References

1. Kislov S.V., Kislov V.G., Skazochkin A.V., Bondarenko G.G., Tikhonov A.N. Effective mineral coatings for hardening the surface of metallic materials. *Russian Metallurgy (Metally)*, 2015, no. 7, pp. 558–564.
DOI: 10.1134/S0036029515070095

2. Skazochkin A., Bondarenko G., Kislov S. Research of Tribological Features of Steel Surface by Creating Mineral Coatings. *Journal of Engineering Science and Technology Review*, 2018, no. 11(6), pp. 138−143.
DOI: 10.25103/jestr.116.17

3. Skazochkin A.V., Useinov A.S., Kislov S.V. Surface hardening of titanium alloy by minerals. *Letters on Materials*, 2018, no. 8(1), pp. 81−87.
DOI: 10.22226/2410-3535-2018-1-81-87

4. Skazochkin A.V., Bondarenko G.G., Zukowski P. Research of Surface Wear Resistance of Aluminum Alloy Modified with Minerals using Sclerometry Method. *Devices and Methods of Measurements*, 2019, vol. 10, no. 3, pp. 263–270.
DOI: 10.21122/2220-9506-2019-10-3-263-270

5. Khodabakhshi F., Simchi A., Kokabi A.H. Surface modifications of an aluminum-magnesium alloy through reactive stir friction processing with titanium

oxide nanoparticles for enhanced sliding wear resistance. *Surface and Coatings Technology*, 2017, vol. 309, pp. 114−123. **DOI:** 10.1016/j.surfcoat.2016.11.060

6. Adebisi A.A., Maleque M.A., Rahman Md.M. Metal matrix composite brake rotor: historical development and product life cycle analysis. *International Journal of Automotive and Mechanical Engineering*, 2011, vol. 4, pp. 471−480. **DOI:** 10.15282/ijame.4.2011.8.0038

7. Oliver W.C., Pharr G.M. Measurement of hardness and elastic byinstrumented indentation: Advances in understanding and refinements to methodology. *J. Mater. Res*., 2004, vol. 19, no. 1, pp. 3−20.

8. Potapov A.I., Gogolinskiy K.V., Syasko V.A., Umanskiy A.S., Kondratiev A.V. Methodological and metrological aspects of materials mechanical properties measurements by instrumented indentation. *Testing. Diagnostics*, 2016, no. 8, pp. 16−21.
DOI: 10.14489/td.2016.08.pp.016-021

9. Useinov S., Solov'ev V., Gogolinskiy K., Useinov A., L'vova N. [Measurement of the mechanical properties of materials with nanometer resolution]. *Nanoindustriya* [Nanoindustry], 2010, no. 2, pp. 30−35 (in Russian).

10. Morozov E.M., Zernin M.V. *Kontaktnye zadachi mekhaniki razrusheniya* [Contact problems of fracture mechanics]. M: Mashinistroenie, 1999, 543 p.

11. Oliver W.C., Pharr G.M. An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. *J. Mater. Res*., 1992, vol. 7, no. 6, pp. 1564−1583.

12. Bhushan B. Modern Tribology Handbook, Two Volume Set, USA, CRC Press Inc, 2000, p. 1760.

13. Useinov A., Gogolinskiy K., Reshetov V. Mutual consistency of hardness testing at micro- and nanometer scales. *Int. J. Mater. Res.*, 2009, vol. 100, pp. 968–972.
DOI: 10.3139/146.110138

14. Potapov A.I., Gogolinskiy K.V., Kondratiev A.V., Umanskiy A.S. Indirect assessment of indenter area function for measuring mechanical properties by instrumented indentation. *Testing. Diagnostics*, 2017, no. 2, pp. 28–32. **DOI:** 10.14489/td.2017.02.pp.028-032

15. Maslenikova I., Reshetov V.N., Useinov A.S. Mapping the Elastic Modulus of a Surface with a NanoScan 3D Scanning Microscope. *Instruments and Experimental Techniques*, 2015, vol. 58, no. 5, pp. 711–717. **DOI:** 10.1134/S0020441215040223

16. Bhushan B., Li X. Micromechanical and tribological characterization of doped single-crystal silicon and polysilicon films for microelectromechanical systems devices. *Journal of Materials Research*, 1997, vol. 12, no. 1, pp. 54–63.

17. Zhang P., Li S.X., Zhang Z.F. General relationship between strength and hardness. *Materials Science and Engineering A*., 2011, vol. 529, pp. 62–73.
DOI: 10.1016/j.msea.2011.08.061

18. Grishin A.M., Khartsev S.I., Bohlmark J., Ahlgren M. Ultra-hard $AlMgB_{14}$ coatings fabricated by RF magnetron sputtering from stoichiometric target. *Journal of Experimental and Theoretical Physics Letters*, 2015, vol. 100, pp. 680–687.
DOI: 10.1134/S0021364014220056

19. Gogolinskiy K.V., Reshetov V.N., Useinov A.S. [On the unification of the determination of hardness and the possibility of transition when measuring it to dimensional values]. *Izmeritel'naya tekhnika* [Measuring technique], 2011, no. 7, pp. 28–34 (in Russian).

20. Aslanyan A.E., Aslanyan E.G., Gavrilkin S.M., Doinikov A.S., Temnitskiy I.N., Shipunov A.N. [State primary standard of rigidity according to Martens scales and indentation scales GET 211-2014]. *Izmeritel'naya tekhnika* [Measuring technique], 2016, no. 6, pp. 3–6 (in Russian).