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Investigation of the surface layer of wheel steel by indentation method

Abstract. *The article considers methods of measurement of microhardness and modulus of elasticity of plasma hardened wheel steel by kinetic indentation method. It provides features of measurement of microhardness, modulus of hardness, elastic recovery, influencing on wear resistance of surface layers of steel. Measurement of these characteristics of material allows estimating and choosing optimum technology of surface modification by surface plasma hardening. It is confirmed that objectivity of determination of microhardness, modulus of elasticity, elastic recovery and flow stress depends on strict observance of imprint depth requirements depending on thickness of hardened layer. It is noted that in spite of the increased amount of factual information, obtained by indentation method, the physical substantiation of hardness micro-mechanisms remains poorly understood, which necessitates substantiation of physical representations of the nature of hardness of metallic materials.*

It is established that the measurement of microhardness, elastic modulus and elastic recovery by the kinetic indentation method is expedient to use for certification of the surface layer of plasma-hardened wheel steel according to the parameters of physical and mechanical characteristics.

Keywords: *wheel ridge and rim, Young's modulus, elastic recovery, micromechanisms of hardness, indentation, microhardness.*

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Introduction

Reliability and durability of heavily loaded parts of machines and mechanisms operating under conditions of friction and wear are in many cases ensured by increasing the hardness of the surface layer. In fact, in real operating conditions, minimization of wear depends on elasticity and resistance to deformations of the surface layer no less than on hardness. These properties are determined by the main physical and mechanical characteristics of the surface layer, namely microhardness, elastic modulus and elastic recovery. In this work, the measurement of these parameters was carried out using the method of testing by tool indentation according to GOST R 8.748-2011 (ISO 14577-1:2015), the micro range of application of this method is $2 \text{ N} > F; h > 0.2 \text{ }\mu\text{m}$. The results obtained when determining Vickers, Rockwell and Brinell hardness are determined, as is known, after the test load is removed. Therefore, the influence of elastic deformation of the material under the influence of the indenter (TIP) is not taken into account. GOST R 8.748-2011 provides determination of hardness and other mechanical characteristics of the material by joint measurement of load and tip movement during indentation. By tracing the complete cycle of loading and removal of the test load, it is possible to determine hardness values equivalent to those measured by classical hardness measurement methods. In addition, this method makes it possible to determine additional material properties such as indentation modulus and elastic-plastic hardness.

It is also important to note that with the advent and development of the method of continuous measurement nanoindentation, it has become possible to quantitatively assess some important characteristics of metallic materials within individual submicroscopic zones, in particular, Vickers hardness, Young's modulus, flow stress, and others. Thus, in [1], the hardness of submicrocrystalline aluminium alloys has been measured by nanoindentation method. The authors of established the limit values of hardness, elastic deformation and corresponding stress by automatic indentation method. Experimental studies have been carried out to improve the wear resistance of hardened structural steel by nanostructured friction treatment. A number of researchers have applied scanning probe microscopes and nanohardness meters to study the mechanical properties of materials at the nanoscale.

The aim of the work is to measure microhardness, elastic modulus and elastic recovery in the micro range by the kinetic indentation method to certify the surface layer of plasma-hardened wheel steel by the parameters of physical and mechanical characteristics. In this case, there is a continuous introduction of a diamond tip into the test specimen under the action of a smoothly increasing load with its subsequent removal and registration of the dependence of the tip movement on the load [2].

Methodology for measuring physical and mechanical properties by tool indentation

Microhardness and Young's modulus of plasma-hardened wheel steel grade 2 (GOST 398-2010) were measured by the method of kinetic indentation. The measurements were carried out on the UPNN-170 unit of the research and production company PlasmaCentre (Saint-Petersburg).

Technical characteristics of the UPNN-170 unit: rated current - 120 A, rated operating voltage - not more than 42 V, argon flow rate 5 l/min, cooling water flow rate 180-220 l/hour. Hardness was measured by the kinetic indentation method in the Centre for Research of Material Properties of Tomsk Polytechnic University (Tomsk). Samples for the study with dimensions of 20x30 mm were subjected to grinding and polishing on a LaboPol-5 machine manufactured in Denmark. As a result of electrochemical polishing the height of surface irregularities did not exceed 10 nm. SPM images were obtained for each selected surface of the sample. The SPM images were processed and analysed using Nova software. During the experiment, a probe sensor of SPMProdeNSC 15/AIBS type was used. The Vickers hardness determined in this way is equal to the average pressure on the contact surface "indenter-sample". An equilateral 4-faceted diamond pyramid was used as an indenter. Hardness measurement was carried out under conditions of continuous loading with linearly increasing load up to 150 mN at room temperature.

Loading and unloading of the indenter, as well as recording of the P-h diagram (applied load and indenter insertion depth) were performed automatically. This method of hardness measurement, called the kinetic hardness method (continuous indentation of the indenter), allows determining the depth of unrecovered h_{max} and recovered (plastic) h indentations, Young's modulus, as well as the work of plastic and elastic deformation during indentation in one cycle "loading-unloading". Figure 1 shows the kinetics of the change in the load P and the indenter penetration depth h in the loading-unloading cycle and the diagram of the dependence of the load P on the penetration depth h .

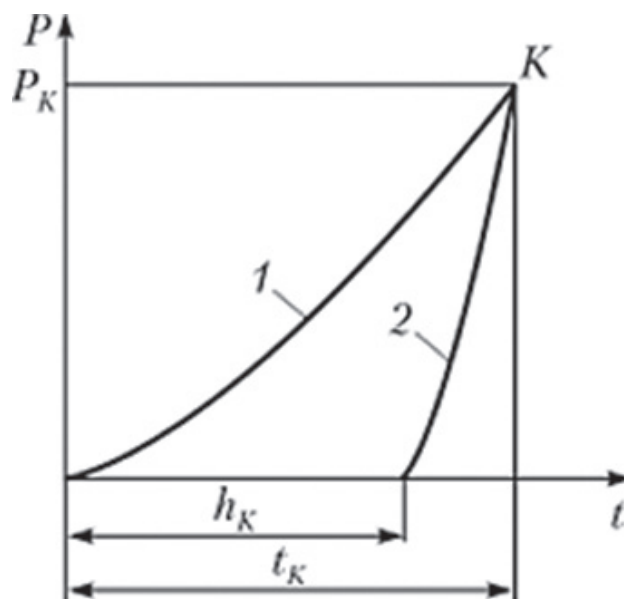


Figure 1. Kinetic hardness method

The indenter indentation size was measured by the maximum indentation depth h_{\max} using the data of semi-contact scanning probe microscopy. The indenter loading and unloading rates were 300 mH/min. The Oliver and Faure method was used to process the test results. Hardness was determined by the maximum load, P_{\max} , divided by the projected contact area after unloading:

$$H = \frac{P_{\max}}{A_{PC}} \quad (1)$$

Vickers hardness is determined by the maximum load P_{\max} divided by the contact area after unloading:

$$H_v = \frac{P_{\max}}{A_c \cdot 9.81} \quad (2)$$

It should be noted that the informative capabilities of this method far exceed those of static indentation; in fact, it is equivalent to the transition from the measurement of a single quantitative characteristic (e.g., yield strength - σ_t , tensile strength - σ_v , etc.) to the continuous registration of loading diagrams.

Improvement of this technique is carried out along the way of full automation of the measurement process and limiting reduction of the indenter load with adequate increase in the sensitivity of the measurement process [3,4].

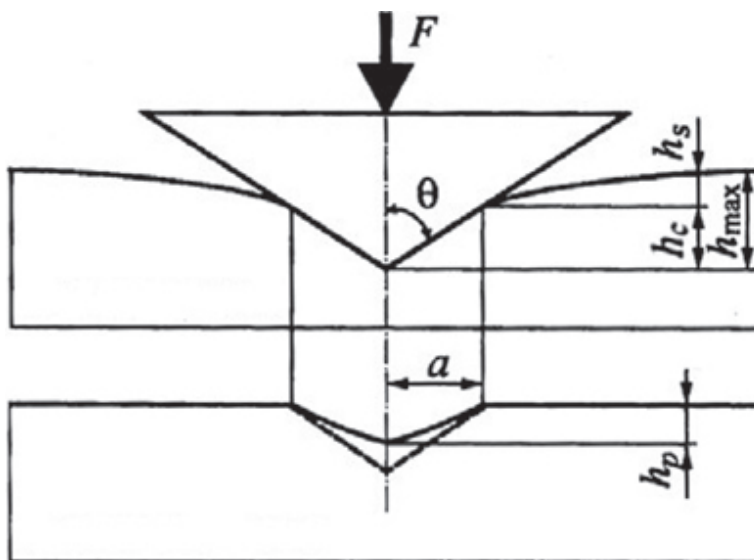


Figure 2. Footprint cross-section at maximum load and after unloading

The operation of the NanoHardness Tester nanohardness tester consists in the fact that as a result of passing a current pulse through the coils located in the magnetic field of a permanent magnet, an electric field is created, which presses on an indenter with a diamond tip. The indenter is lowered to a point on the surface of the specimen with a predetermined load. After the load has reached its maximum value and the direction of the coil current is reversed, it returns to its original position. The sapphire ring is used to check the perpendicularity of the sample position relative to the indenter. If this condition is not met and the sample is tilted, a signal is generated on one of the capacitive sensors that prohibits the indentation process. In this case it is necessary to reinstall the sample under test. The springs are necessary to hold the indenter and the magnetic shield protects the instrument from electromagnetic interference.

Results obtained and their discussion

Measurement of some mechanical characteristics of wheel steel was carried out using the method of testing by tool indentation according to GOST R 8.748-2011 (ISO 14577-1:2015), as noted above, the micro range of application of the method is $2 \text{ N} > F; h > 0.2 \text{ }\mu\text{m}$.

The indentation method consists in pressing an indenter with a diamond tip, with a load acting on it, into the near-surface layer of the material under study and determining the thickness of this layer using the nanohardness meter software. Processing of experimental data is carried out based on the results of measurements of at least 3 prints obtained under the same experimental conditions. Figure 3 shows indenter imprints in the near-surface layer of the material at some distance from the surface.

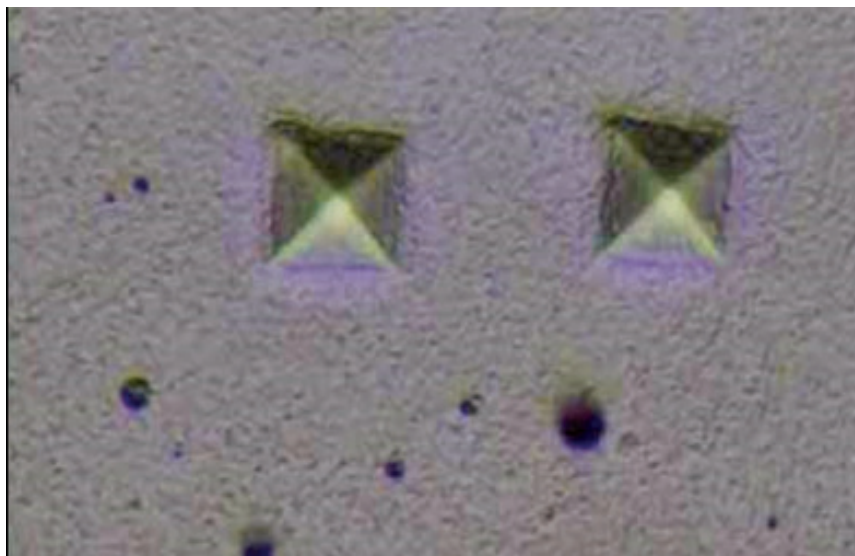


Figure 3. Indenter imprints in the near-surface layer of the material

Before the indenter begins to descend onto the surface of the material, the load with which it will press on the sample is set. Figs. 4 a) and b) show the interfaces of the nanohardness tester, where the loading and unloading curves showing the indentation process can be seen. After the load value reaches the maximum value, the indenter starts unloading, the load acting on it is gradually reduced to zero and it returns to its original position. The unloading curve is obtained, which shows that during indentation the specimen deforms elasto-elastically, i.e. the material from under the indenter does not fully return to its former position. The misalignment of the loading and unloading lines is probably due to inelasticity.

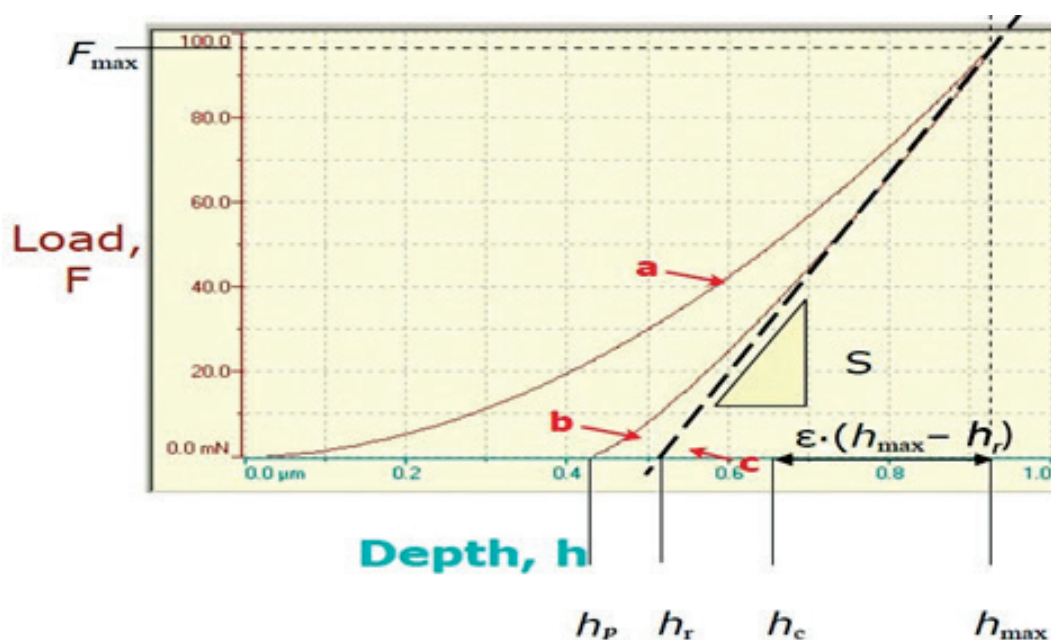


Figure 4. Analysing the F-h diagram using the Oliver-Farr

Note that Sample 1 is cut from the rim and Sample 2 - from the wheel ridge, subjected to surface plasma hardening under the same conditions. By comparing the data in Tables 1 and 2,

it can be seen that the HV and HIT of the wheel rim are greater than the corresponding values of the wheel crest (388.8-354.6), while the Young's modulus values of the wheel crest, on the contrary, are greater in the wheel crest compared to the rim (246.8-231.7). It can also be seen that surface plasma hardening of the wheel rim was more effective than that of the wheel crest, which may be due to the different cooling rates of these wheel elements during the hardening and self-tempering process. Probably, the different efficiency of plasma hardening of the wheel rim and wheel ridge is influenced by the difference in the angle between the plasma jet and the wheel rim, on the one hand, and between the plasma jet and the wheel ridge, on the other hand, as it leads to different heat flux density into the hardened surface of the wheel [5,6].

It is also noteworthy that the numerical values of the measured mechanical characteristics (HV, HIT, EIT) differ from the corresponding values of the interface of the nanohardness tester displaying the indentation process during loading and unloading. This may be due to the pliability of the device, since the applied test load acts not only on the surface of the test specimen, but also on the device parts, which are elastically deformed in the process. The accuracy and correctness of determining the hardness and other characteristics of the tested steel depends on strict compliance with the requirements for measuring the indentation depth [7].

Table 1. Measured mechanical characteristics of the wheel rim

#	Mechanical characteristics of the wheel rim at 20 μm from the edge of the specimen deep into the metal			Mechanical characteristics of the wheel rim at 1700 μm from the edge of the specimen deep into the metal		
	HV	H _{IT} , MPa	E _{IT} , GPa	HV	H _{IT} , MPa	E _{IT} , GPa
1	380.0	4222.86	217.32	466.22	4939.52	254.11
2	375.32	4082.4	226.33	378.08	4005.66	245.44
3	351.22	3827.01	211.70	373.25	3954.54	254.95
4	375.10	4122.39	221.29	354.25	3753.25	245.98
5	358.02	3793.15	229.06	383.95	4067.88	230.76
6	395.26	4187.66	252.30	397.99	4216.62	248.90
7	366.76	3885.8	243.07	385.83	4087.81	230.62
8	386.15	4191.21	235.22	369.62	3916.01	261.58
9	406.59	4307.7	233.89	398.50	4222.04	253.07
10	463.92	3855.66	236.25	386.23	4092.04	251.74
11	366.71	3885.21	242.59	-	-	-
tot.	388.8	4032.8	231.7	384.38	4072.39	241.41

Table 2. Measured mechanical characteristics of the wheel ridge

#	Mechanical characteristics of the wheel ridge at 40 μm from the edge of the specimen to the depth of the metal			Mechanical characteristics of the wheel ridge at 100 μm from the edge of the specimen deep into the metal		
	HV	H _{IT} , MPa	E _{IT} , GPa	HV	H _{IT} , MPa	E _{IT} , GPa
1	350.74	3715.99	287.72	392.01	4153.16	210.41
2	354.93	3760.39	254.69	356.29	3774.81	210.65
3	334.65	3545.52	219.53	387.87	4109.37	230.99
4	365.16	3568.82	232.74	370.25	3922.27	241.84
5	367.65	3895.16	239.30	375.79	3981.44	247.86
6	-	-	-	366.42	3882.17	337.77
tot.	354.6	3697.2	246.8	374.77	3970.54	246.59

Note: 1 N/mm² = 1MPa, according to GOST R 8.748-2011 it is allowed to use multiples and fractions.

It is known that ultrahigh heating and cooling rates (~1500-3000 0C/s) during plasma quenching leads to strong structure refinement with the formation of nanostructured elements of phase and structural components of the quenched material [13-14]. Hence, one of the important and urgent tasks of tribology is to determine the relationship between wear resistance and mechanical properties of the structure of contacting materials. High friction wear resistance is known to be achieved due to the stability of the surface layer structure, which is stabilised by increasing hardness, for example, by plasma hardening. Therefore, changing the structure of the material under heavy friction conditions is of great interest (work is ongoing in this direction), because of the plastic deformation of the surface layer and the increase in its temperature. In this case, the surface layer is deformed by the mechanism of low-cycle fatigue and therefore the structure is not stable. Increasing the temperature of the surface layer leads to a decrease in its hardness and, consequently, wear resistance.

Speaking about the physical nature of microhardness in general, nanohardness in particular, it should be said that, despite the increased amount of factual information obtained by nanoindentation, the physical substantiation of hardness micromechanisms remains poorly satisfactory. Thus, an example of the insufficiency of the existing ideas about the nature of hardness is the problem of explaining the causes of the scale dimensional factor, which manifests itself in the increase of the hardness number with decreasing load and indentation size, especially at depths $h < 1 \mu\text{m}$. Explanation of the causes of the dimensional factor by the dislocation mechanism of plasticity by introducing the necessary number of dislocations did not lead to the removal of the issue [8]. Formally, it is possible to introduce the necessary number of dislocations into the material under study, but their density at $h < 100 \text{ nm}$ becomes unrealistically large ($> 10^{14} \text{ cm}^{-2}$).

In addition, it contradicts the results of microstructural studies, which do not detect such a number of dislocations. Obviously, the mechanical properties and behaviour of materials in nano-volumes for a number of reasons may be very different from those obtained in traditional macroscopic tests, since with the reduction of the size of the loaded area by many orders of magnitude, many new factors affecting the material properties begin to act. The localisation of the load leads to a strong hardening of the material in the deformation zone and the resulting large stress gradients can strongly influence the plastic flow mechanisms. For example, it is not clear in which direction the material moves from under the indenter. It is believed that in plastic materials the material flows out from under the indenter towards the free surface. In reality, mass transfer is directed into the volume, which compacts the material in the local deformation zone [9].

At present, there is a lot of direct evidence of the growing role of non-equilibrium point defects in mass transfer as the contact spot size decreases. There is also convincing evidence of significant changes in the structure of materials under the indenter as a result of amorphisation, phase transformations, and formation of nanocrystalline structure [10]. However, in the existing hardness theories based on dislocation mechanisms of plastic deformation, these circumstances are not taken into account and explained.

In addition, due to the small size of the deformed region ($\sim h$), large relative strain rates $\varepsilon \sim v/h$ are realised even at small absolute embedding velocities- v . As a result, the nanohardness of the material can exceed the yield strength by two or more orders of magnitude. Hence the necessity to substantiate the physical ideas about the nature of hardness, in general, and nanohardness, in particular.

Conclusion

1. Microhardness, elastic modulus and elastic recovery of plasma-hardened wheel steel have been measured by the method of instrumental indentation, in which there is a continuous introduction of a diamond tip into the test specimen under the action of a smoothly increasing load with its subsequent removal and registration of the dependence of the tip displacement on the load. The values of these mechanical characteristics of the wheel rim and ridge at different distances from the edge of the specimen deep into the metal are given. It is noted that sharp

fluctuations in the numerical values of the modulus of elasticity of the wheel ridge (287.72GPa; 254.69GPa; 219.53GPa) can be associated with methodological error, in particular, the change of the indentation step (Fig.4a and b). It is confirmed that the objectivity of determining the hardness and other elasto-mechanical characteristics of the tested steel depends on strict adherence to the indentation depth measurement requirements.

2. The mechanical properties and behaviour of the material in the nano-volume differ significantly from those determined by traditional macroscopic testing. Knowledge of the physical and mechanical characteristics of the material (hardness, Young's modulus, elastic recovery, etc.) affecting the wear resistance of surface layers makes it possible to evaluate and select the optimal technology for surface modification by plasma quenching. The objectivity of hardness, elastic modulus and elastic recovery determination depends on the parameters of the measuring equipment used and strict compliance with the requirements for the depth of hardness, elastic modulus and elastic recovery. It is found that the hardness (Vickers HV and H) of the rim is greater, and Young's modulus, on the contrary, is less than the corresponding characteristics of the ridge. Measurement of microhardness, elastic modulus and elastic recovery by the method of kinetic indentation is reasonable to use for certification of the surface layer of plasma-hardened wheel steel according to the parameters of physical and mechanical characteristics.

3. It is noted that despite the increased amount of factual information obtained by nanoindentation method, the physical substantiation of hardness micromechanisms remains poorly studied, which necessitates the substantiation of physical ideas about the nature of hardness, in general, and nanohardness, in particular. The mechanical properties and behaviour of materials in nano-volumes can be very different from those obtained in traditional macroscopic tests, since as the size of the loaded region decreases (by many orders of magnitude), many new factors affecting the material properties begin to act. Localisation of the load leads to a strong hardening of the material in the deformation zone and the resulting large stress gradients can strongly influence the mechanisms of plastic flow, and the structure of materials under the indenter can change significantly as a result of amorphisation, phase transformations, formation of nanocrystalline structure.

Confirmations

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Дөңгелек болаттың беткі қабатын индеттеу арқылы зерттеу

Аңдатпа. Кинетикалық индеттеу әдісімен плазмалық шыңдалған дөңгелекті болаттың микроқаттылығын және серпімділік модулін өлшеу әдістемесі қарастырылған. Болаттың беткі қабаттарының тозуға төзімділігіне әсер ететін микроқаттылықты, серпімділік модулін, серпімді қалпына келтіруді өлшеу ерекшеліктері келтірілген. Материалдың аталған сипаттамаларын өлшеу беттік плазманы шыңдау арқылы бетті модификациялаудың оңтайлы технологиясын бағалауға және таңдауға мүмкіндік береді. Микроқаттылықты, серпімділік модулін, серпімді қалпына келтіруді және ағын кернеуін анықтаудың объективтілігі шыңдалған қабаттың қалыңдығына байланысты индеттеу тереңдігіне қойылатын талаптардың қатаң сақталуына байланысы расталды.

Индеттеу арқылы алынған нақты ақпараттың көбеюіне қарамастан, қаттылықтың микромеханизмдерінің физикалық негіздемесі әлі де аз зерттелген, бұл металдық материалдардың қаттылығының табиғаты туралы физикалық идеяларды негіздеуді қажет ететіні атап өтілген. Плазмамен шыңдалған доңғалақ болатының беткі қабатын физикалық-механикалық сипаттамалары бойынша сертификаттау үшін микроқаттылықты, серпімділік модулін және серпімді қалпына келтіруді кинетикалық индеттеу арқылы өлшеуді қолдану орынды екені анықталды.

Түйін сөздер: тарақ және доңғалақтың жиегін наноиндеттеу, Юнг модулі, қаттылықтың микромеханизмі, қаттылық, серпімді қалпына келтіру.

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Исследование поверхностного слоя колесной стали методом индентирования

Аннотация. Рассмотрена методика измерения микротвердости и модуля упругости плазменно-закаленной колесной стали методом кинетического индентирования. Приведены особенности измерения микротвердости, модуля упругости, упругого восстановления, влияющие на износостойкость поверхностных слоев стали. Измерение этих характеристик материала позволяет

оценить и выбрать оптимальную технологию модификации поверхности путем поверхностной плазменной закалки. Подтверждено, что объективность определения микротвердости, модуля упругости, упругого восстановления и напряжения течения зависит от строгого соблюдения требований по глубине отпечатка в зависимости от толщины закаленного слоя.

Отмечено, что, несмотря на возросший объем фактической информации, полученный методом индентирования, физическое обоснование микромеханизмов твердости остается слабо изученным, что вызывает необходимость обоснования физических представлений о природе твердости металлических материалов.

Установлено, что измерение методом кинетического индентирования микротвердости, модуля упругости и упругого восстановления целесообразно использовать для сертификации поверхностного слоя плазменно-закаленной колесной стали по параметрам физико-механических характеристик.

Ключевые слова: гребень и обод колеса, модуль Юнга, микромеханизм твердости, индентирование, микротвердость, упругое восстановление.

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