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future's in the making

Nanoindentation and sclerometry for coated cutting tools studying

NIOS Nanoscan Scanning Nanohardness Tester

NIOS Nanoscan scanning nanohardness testers are designed to study the topography and surface structure and measure the mechanical properties (hardness and elastic modulus) of materials and thin films on a submicron and nanometer scale.

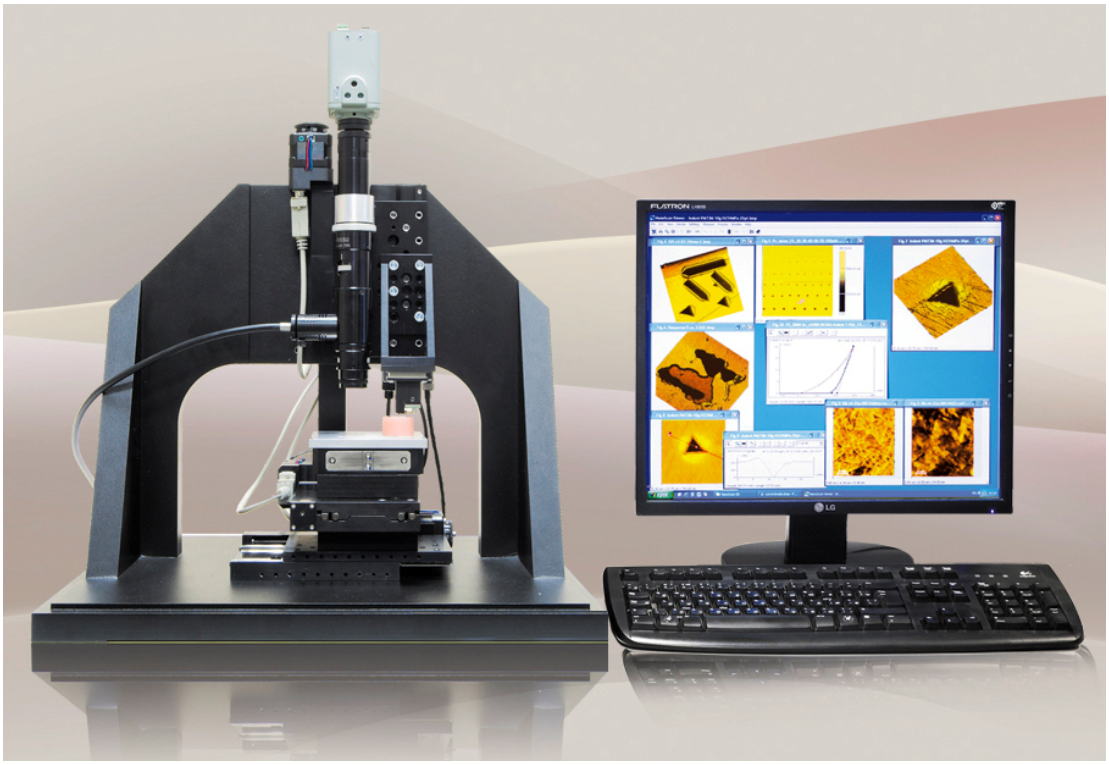


Fig.1. NIOS Nanoscan scanning nanohardness testers

NIOS Nanoscan scanning nanohardness tester works on the principles of scanning force microscopy. The main difference between this device and classical scanning probe microscopes (SPM) is the use of a piezoresonance cantilever tuning fork design with high bending stiffness of the console. Using the resonance mode allows you to control the surface–probe tip contact by two ways: change the amplitude and frequency of the probe. The probe resonant mode provides high amplitude and frequency stability of oscillations and guarantees a fairly soft contact of the tip with the solid material surface during scanning. At the same time, the high bending stiffness of the probe console allows the probe tip to penetrate through the viscous layer until it comes into contact with the elastic surface, and also modify the surface — indentation and scratching (sclerometry). The probe design allows the use of diamond tips of various types and sizes. The bend of the piezoresonance probe is monitored using a high-precision displacement sensor, which allows measuring the loading force during the nanoindentation process. The

listed features significantly distinguish **NIOS Nanoscan** from other commercial devices. In particular, **NIOS Nanoscan** is capable of carrying out a controlled force effect on the structures under investigation ranging from several micronewtons to hundreds of millinewtons, which is hundreds of times higher than the capabilities of other SPMs. Not inferior to classical nanoindenters in measuring hardness and elastic modulus, **NIOS Nanoscan** significantly surpasses them in the quality of the surface images of the material being studied during scanning.

Introduction

The relief and mechanical properties of the cutting tools were studied by scanning probe microscopy (SPM), nanoindentation, and sclerometry using **NIOS Nanoscan** scanning nanohardness tester.

Samples description

Sample #1:
Drill. The base has a metallic sheen, the working part is iridescent (*Fig.2*).

Sample #2:
Milling cutter. The base has a metallic luster, the working part is brown (*Fig.3*).

To carry out the measurements, special equipment was made that made it possible to provide rigid fixation of the sample.

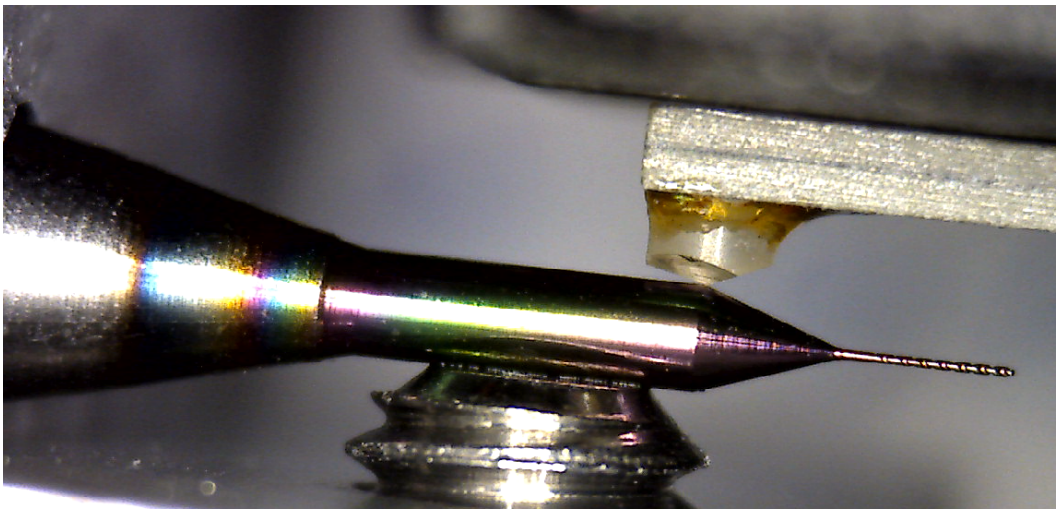


Fig.2. Sample #1. Photo of a drill in a side-view USB videomicroscope

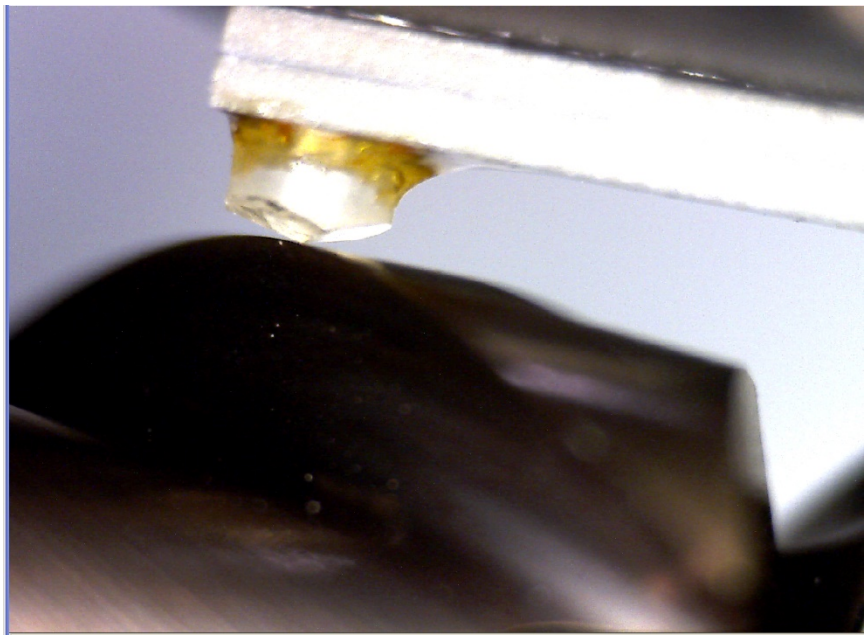


Fig.3. Sample #2. Photo of a milling cutter in a side-view USB videomicroscope

Surface topography and roughness measurement

The surface relief was measured using a **NIOS Nanoscan** scanning nanohardness tester in the mode of semi-contact scanning probe microscopy (see *Appendix 1*).

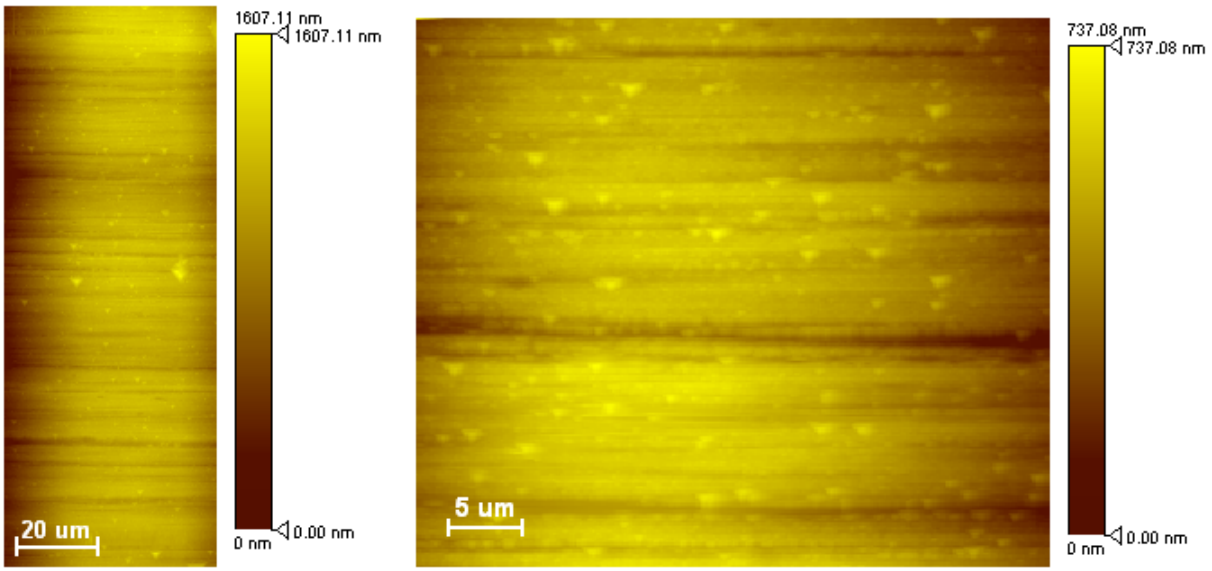


Fig.4. Sample #1. SPM image of the surface relief of the drill in the area covered by the film

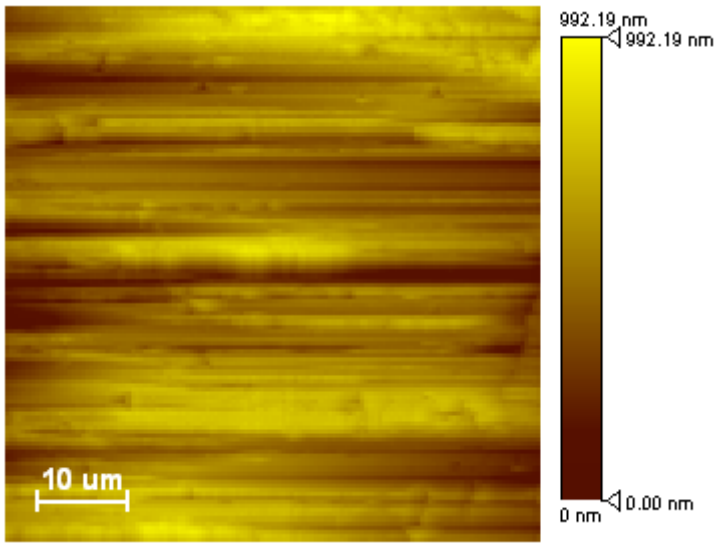


Fig.5. Sample #1. SPM image of a surface topography in an area without a film

Table 1. Sample #1. Surface roughness

Roughness parameter	Area with film	Area without film
Ra, nm	70	120
Rms, nm	86	146
Rz, nm	232	294

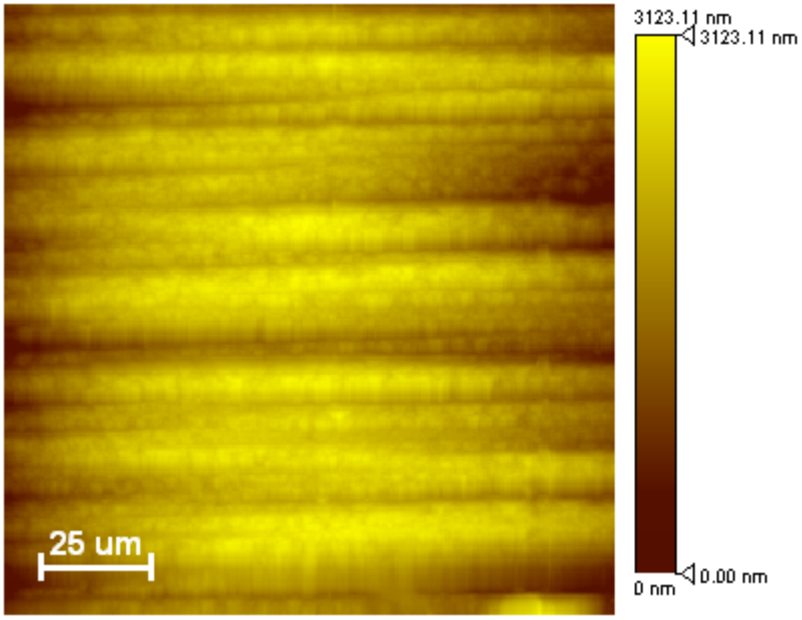


Fig.6. Sample #2. SPM image of the surface relief of the milling cutter in the area covered by the film

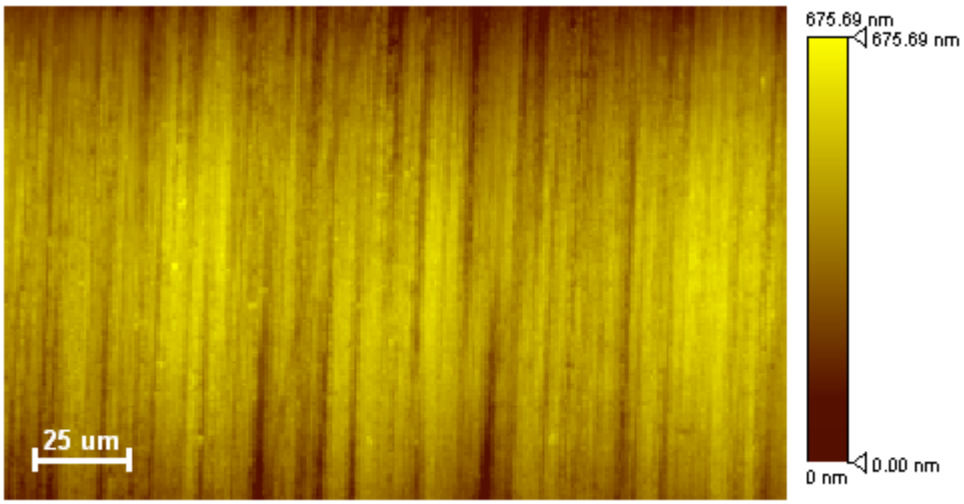


Fig.7. Sample #2. SPM image of a surface topography in an area without a film

Table 2. Sample #2. Surface roughness

Roughness parameter	Area with film	Area without film
Ra, nm	265	40
Rms, nm	345	52
Rz, nm	785	156

Hardness and elasticity modulus measurement

Measuring dynamic nanoindentation

The hardness and elasticity modulus were measured using a **NIOS Nanoscan** scanning nanohardness tester by the measuring indentation method in accordance with the recommendations of the international standard ISO 14577 (see Appendix 2).

Table 3. Sample #1. Measurement of hardness and elasticity modulus of a sample areas with film

Measurement Series No.	Max. depth, nm	Load, mN	Hardness, GPa	Elasticity modulus, GPa
1	68 (8.2%)	5 (0.2%)	36.4 (19.5%)	594.8 (14.5%)
2	106 (9.0%)	10 (0.1%)	31.7 (22.2%)	663.1 (20.2%)
3	183 (10.2%)	20 (0.1%)	23.0 (20.8%)	704.8 (7.8%)
4	211 (4.9%)	30 (0.0%)	26.1 (11.5%)	754.5 (11.4%)

Elasticity modulus: **675** ± 105 GPa (15.8%)
Hardness: **30.8** ± 6.5 GPa (21.0 %)

Table 4. Sample #1. Measurement of hardness and elasticity modulus of a sample areas without film

Measurement Series No.	Max. depth, nm	Load, mN	Hardness, GPa	Elasticity modulus, GPa
1	138 (12.7%)	5 (0.3%)	8.1 (19.3%)	380.9 (18.3%)
2	185 (7.6%)	10 (0.1%)	9.6 (10.4%)	395.3 (9.2%)
3	278 (1.5%)	15 (0.0%)	6.2 (2.0%)	482.3 (10.2%)
4	312 (25.3%)	19.3 (5.0%)	7.6 (47.4%)	451.6 (17.9%)
5	334 (14.4%)	21.9 (8.9%)	7.5 (40.3%)	442.7 (27.5%)

Elasticity modulus: **420** ± 65 GPa (15.5%)
Hardness: **8.2** ± 2.0 GPa (24.8 %)

Table 5. Sample #2. Measurement of hardness and elasticity modulus of a sample areas with film

Measurement Series No.	Max. depth, nm	Load, mN	Hardness, GPa	Elasticity modulus, GPa
1	95 (7.3%)	5 (0.3%)	19.1 (24.2%)	180 (9.6%)
2	140 (5.3%)	10 (0.1%)	17.3 (4.5%)	210 (11.2%)
3	180 (11.1%)	15 (0.1%)	18.5 (27.4%)	215 (12.7%)
4	200 (4.7%)	20 (0.0%)	20.2 (7.9%)	200 (10.9%)
5	245 (6.6%)	25 (0.1%)	17.9 (16.6%)	195 (7.2%)
6	260 (5.5%)	30 (0.0%)	20.1 (15.7%)	195 (9.3%)
7	300 (6.1%)	35 (0.0%)	18.5 (20.3%)	190 (7.1%)

Elasticity modulus: **196** ± 20.3 GPa (15.8%)
Hardness: **18.9** ± 3.5 GPa (21.0 %)

Table 6. Sample #2. Measurement of hardness and elasticity modulus of a sample areas without film

Measurement Series No.	Max. depth, nm	Load, mN	Hardness, GPa	Elasticity modulus, GPa
1	75 (3.2%)	5 (0.2%)	21.9 (5.5%)	500 (9.6%)
2	105 (7.5%)	10 (0.1%)	20.2 (12.1%)	720 (8.5%)
3	135 (10.4%)	15 (0.1%)	20.4 (18.5%)	790 (17.3%)
4	160 (4.6%)	20 (0.1%)	21.2 (12.0%)	760 (11.6%)
5	185 (5.1%)	25 (0.0%)	21.9 (8.6%)	710 (16.7%)

Elasticity modulus: **700** ± 125 GPa (17.5%)
Hardness: **21.5** ± 2.2 GPa (10.2 %)

Sclerometry

In addition, the hardness was measured using **NIOS Nanoscan** scanning nanohardness tester by sclerometry — by scratching and analyzing profile of the scratches from the image of the residual trace (see Appendix 3).

Table 7. Sample #1. Measurement of hardness of a sample areas with film

Measurement Series No.	Load, mN	Hardness, GPa
1	20	35.5 (25.4%)
2	30	29.3 (26.8%)
3	40	30.7 (20.1%)

Hardness: **31.8** ± 7.7 GPa (24.2 %)

Adhesion and wear resistance

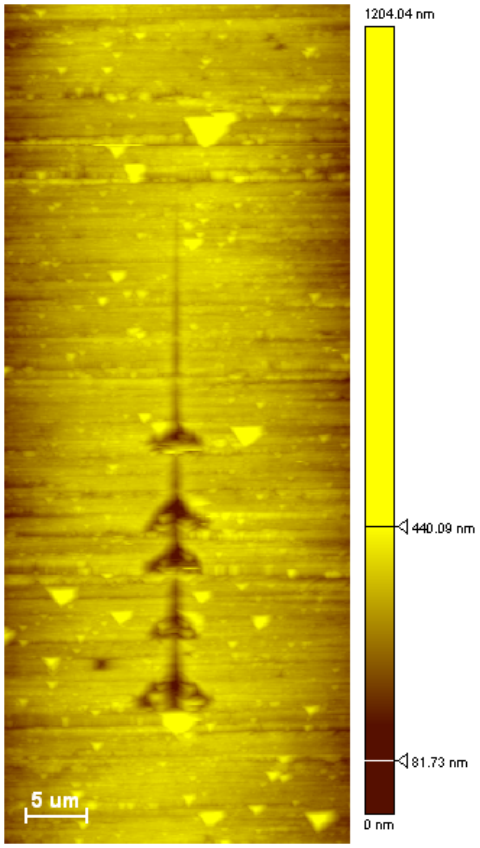


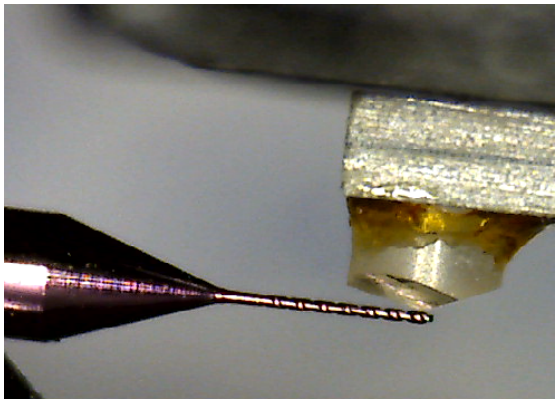
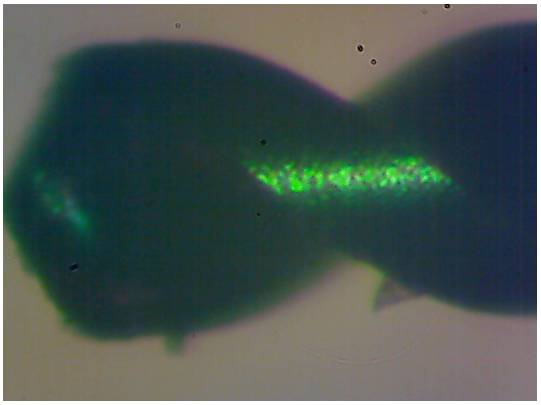
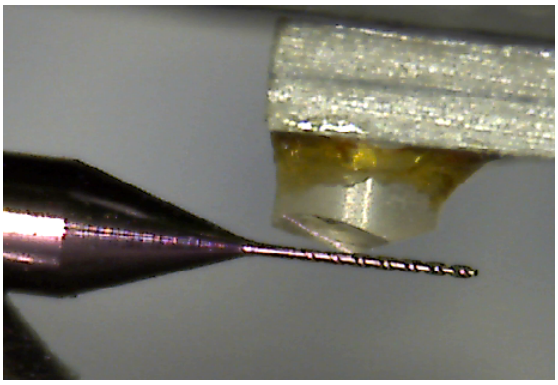
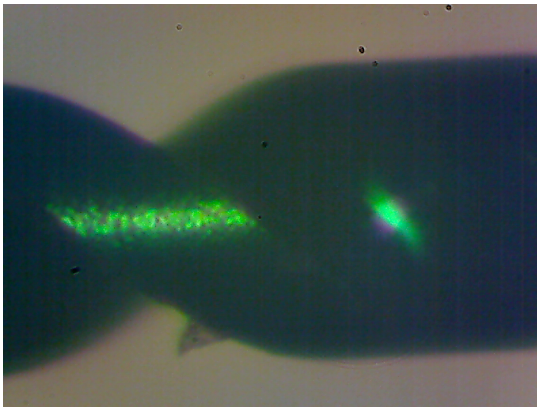
Fig.8. Sample #1. SPM image of the surface topography in the area with film after scratching with variable load: 200μN-60mN

During scratching with a variable load of 200 μN to 60 mN, the film lifted off at a load of 30 mN.

Stiffness measurement

The stiffness was measured using **NIOS Nanoscan** scanning nanohardness tester in the mode of measuring the dependence of loading - displacement (see Appendix 4).

Table 8. Sample #1. Drill stiffness measurement

Photo of a drill in a side-view USB videomicroscope	Microscope drill photo	Stiffness, kN/m
		2.15 (±2%)
		12.5 (±4.4%)

Appendix 1

Surface mapping

Scanning nanoscale hardness testers **NIOS Nanoscan** allow you to obtain images of a three-dimensional surface topography by scanning probe microscopy. Scanning is performed in tapping mode with a diamond tip mounted on a piezoceramic probe. The resonant oscillations of the probe occur with a frequency f ~ 10 kHz and with an amplitude A <50 nm. During the scanning process, the frequency f or the amplitude A of the oscillations is kept constant.

In the frequency scanning mode, a constant stiffness of the indenter contact area with the surface is ensured. In this mode, it is recommended to study materials with relatively high values of hardness and elastic modulus (metals and alloys, crystalline materials, ceramics). In this case, the influence of the presence of contamination on the surface of the sample is eliminated or significantly reduced.

In the amplitude scanning mode, viscous contact of the probe with the surface is maintained constant, which allows one to study soft materials (polymers, plastics).

The size of the maximum scanning window is 100×100×10 μm.

The actual resolution achieved during scanning is limited by the radius of the contact spot of the tip with the surface and is of the order of 10 nm in the XY plane and not worse than 1 nm along the Z axis, which is typical for scanning force microscopes operating in air.

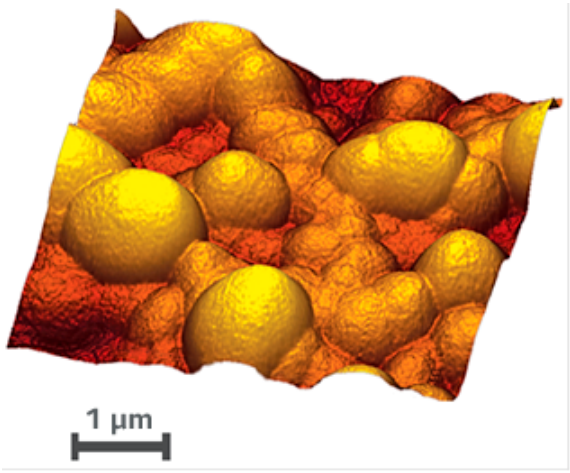


Fig.9. 3D image of the surface relief of polycrystalline SiC

Roughness measurement

Average Roughness, Ra	Arithmetic mean deviation of the profile (average roughness, *ISO 4287/1*), nm. The average deviation of all points of the roughness profile from the midline on the length of the assessment: $R_a = \frac{1}{N}\sum_{j=1}^{N}\lvert r_j\rvert$.
RMS (Root Mean Square), Rms	Root mean square roughness (*ISO 4287/1*), nm. The average value of the measured deviations from the midline on the length of the assessment: $R_q = \sqrt{\frac{1}{N}\sum_{j=1}^{N} r_j^2}$.
Ten point height, Rz	Rz=1/5*(Z_{max1} + Z_{max2} + Z_{max3} + Z_{max4} + Z_{max5} - Z_{min1} - Z_{min2} - Z_{min3} - Z_{min4} - Z_{min5}), ten-point height (*ISO 4287/1*). The parameter expresses the surface roughness at the selected five highest and lowest points of the surface, nm

Appendix 2

Measuring indentation

On the basis of the **NIOS Nanoscan**, a method for measuring hardness is implemented, based on measuring and analyzing the dependence of the load when an indenter is pressed into the surface of the material on the indenter penetration depth. This method is the basis of the standard for measuring hardness *ISO 14577*.

For mechanical tests, an indenter of the Berkovich type is used, which is a trihedral diamond pyramid with an angle at the apex of about 142º.

The method of measuring dynamic indentation is as follows: the indenter is pressed into the surface of the sample at a constant speed, when the specified load is reached, the indenter is retracted in the opposite direction. During this test, the values of the load and the corresponding indenter displacement are recorded.

A typical experimental graph of the dependence of the load (*P*) on the indentation depth (*h*) is shown in *Fig.10*. It consists of two parts corresponding to the loading and unloading process. In the framework of this method, the hardness *H* of the sample is determined by the equation:

$$H = \frac{P_{max}}{A_c}$$

Here, A_c is the area of the fingerprint at the maximum value of the applied load P_{max}.

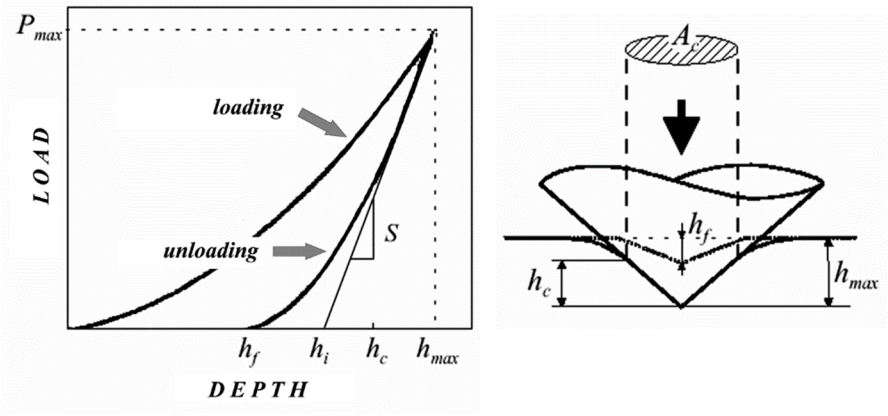


Fig.10. The loading curve, and the contact diagram with values used in the methodology for calculating elasticity modulus and hardness

The value of the reduced modulus of elasticity is calculated as follows:

$$E_r = \frac{1}{\beta} \cdot \frac{\sqrt{\pi}}{2} \cdot \frac{S}{\sqrt{A_c}}$$

Here, the constant β depends on the shape of the indenter, and the stiffness of the contact *S* is determined by the angle of inclination of the tangent to the unloading curve at the point P_{max}:

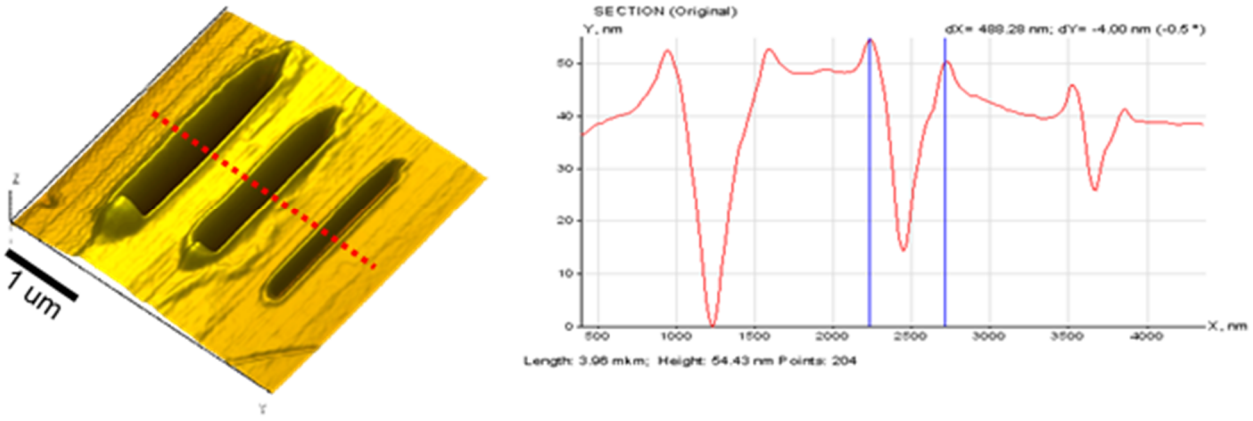
$$S = \left(\frac{dP}{dh} \right)_{P=P_{max}}$$

The contact area at maximum load A_c is determined by the indenter geometry and contact depth h_c and is described by the so-called needle shape function: $A_c = f(h_c)$

Appendix 3

Sclerometry hardness measurement (scratching and analysis)

The hardness measurement using the **NIOS Nanoscan** sclerometry method consists in applying scratches to the surface of the material and scanning the resulting indents. Previously, the shape of the tip is calibrated on the reference material by applying a series of scratches at different loads. The value of the hardness of the material is calculated relative to the hardness of the standard by the ratio of the loads and the widths of the resulting scratches on the studied and reference materials (*Fig.11*).



$$H = k \times \frac{P}{b^2}$$
$$b = b_{ref}$$
$$k = \frac{H_{ref} \cdot b^2}{P}$$
$$\Rightarrow H = H_{ref} \times \frac{P}{P_{ref}}$$

b – scratch width at given load P;

H – hardness to measure,

H_{ref} – hardness of reference material;

Fig.11. Sclerometry hardness measurement

Scanning and surface modification is carried out with the same tip in one measurement cycle. This avoids the difficulty of finding scratches and indentations and significantly reduces the time spent on measurements.

Appendix 4

Measurement of stiffness of beams and membranes

To control the stiffness of beams and membranes using **NIOS Nanoscan** scanning nanohardness tester, the measurement mode of the load – displacement dependence is used, similar to the method of measuring indentation. The mode of multiple loading of an object by an indenter is also implemented. As a result of such a test, it is possible to determine the stiffness (compliance) of the membrane, the number of loading cycles to failure.

For precise positioning of the loading place, **NIOS Nanoscan** uses a high-resolution digital optical microscope as well as a preliminary scan mode of the object's surface before measurements.

Fig.12. Membrane measurement scheme

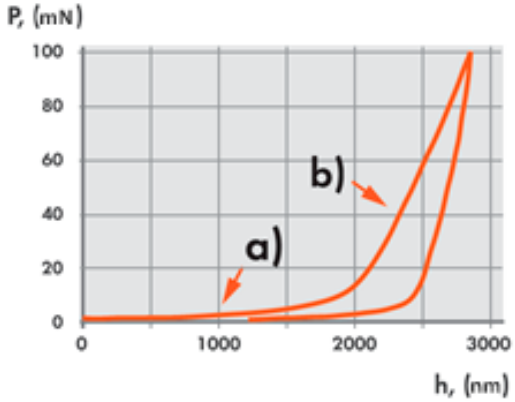


Fig.13. Loading / unloading curve: segment of the free deflection of the membrane (a), abutment of the membrane in the substrate (b)

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