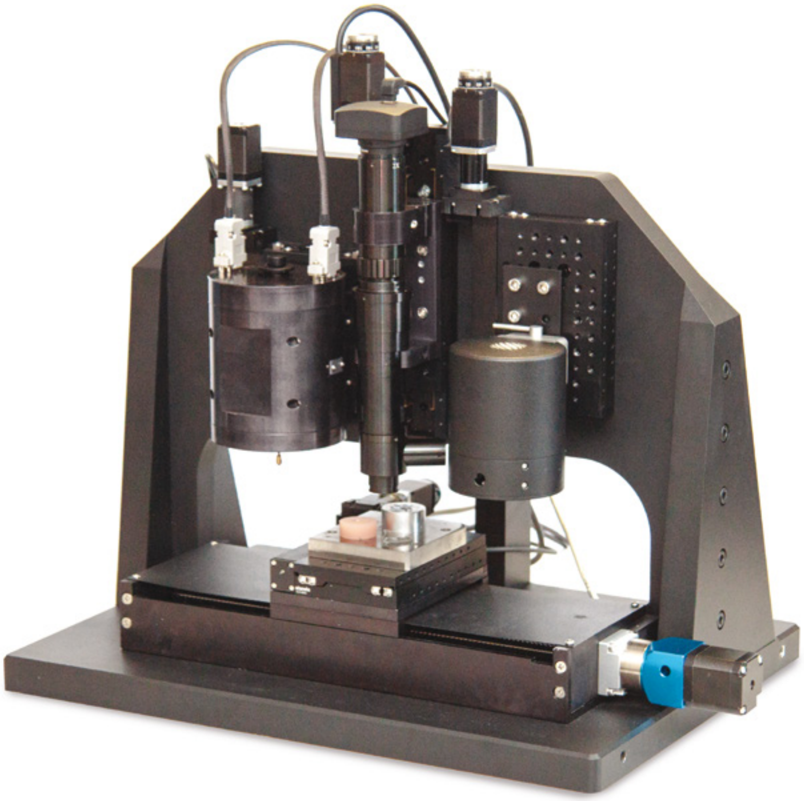
ostec
corporate group

future's
in the making

Nanomechanical Testers
NIOS

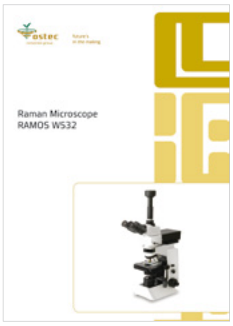


Ostec Instruments produces and offers hi-tech innovative scientific and analytical equipment.

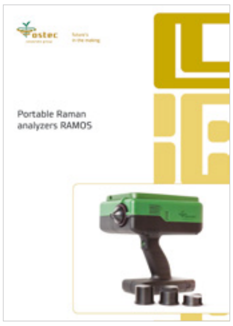
Our mission is to be a company that finds, selects, protects and develops cutting-edge ideas to create new products and technologies and deliver technological progress. That is why the symbol of our company is a growing sprout.

We provide complete solutions for our clients: the best equipment to meet customer's requirements, deep knowledge of customer's applications, qualified and reliable maintenance support.

OUR other products:


Raman Microscope
RAMOS W532

Raman Microscope
RAMOS W532


Portable Raman
analyzers RAMOS

Portable Raman
analyzers RAMOS


Optical components
OCOS

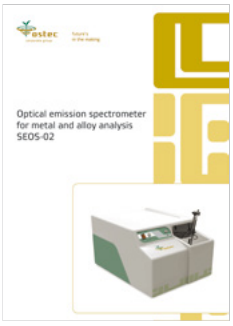
Optical components OCOS


Laser elemental analyzer
LIOS-500N

Laser elemental analyzer
LIOS-500N


Vibration Control
Solutions AVOS

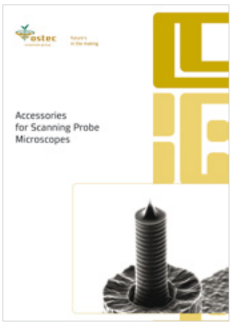
Vibration Control
Solutions AVOS


Optical emission spectrometer
for metal and alloy analysis
SEOS-02

Optical emission
spectrometer for metal
and alloy analysis SEOS-02


Confocal Raman
Microscope RAMOS

Confocal Raman
Microscope RAMOS


Accessories
for Scanning Probe
Microscopes

Accessories for Scanning
Probe Microscopes


FTIR spectrometers
and microscopes IROS

FTIR spectrometers
and microscopes IROS


Analytical metallographic
systems OMOS M-series

Analytical metallographic
systems OMOS M-series

About NIOS

The technologies underlying NIOS nanomechanical instruments have been developed by russian materials scientists and engineers since 1995. Technical solutions employed on NIOS instruments are protected by patents under Russian Federation.

The modular design of NIOS series allows end-users to configure a nanomechanical tester specifically for their needs and requirements. Configurations of NIOS nanomechanical tester can consist of the following modules:

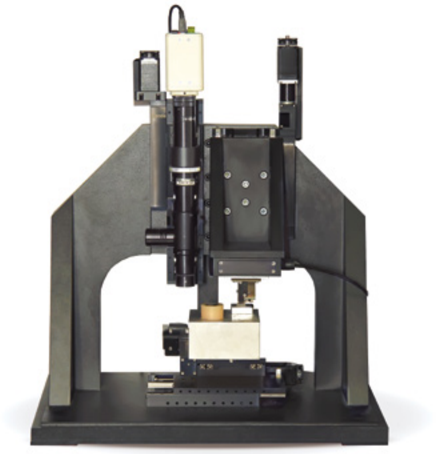
- Wide-range nanoindenter
- Optical Microscope
- Atomic Force Microscope
- Scanning nanomechanical tester
- Electrical Properties Measurement
- Lateral Force Sensor
- In-situ Topography Imaging
- Heating Stage

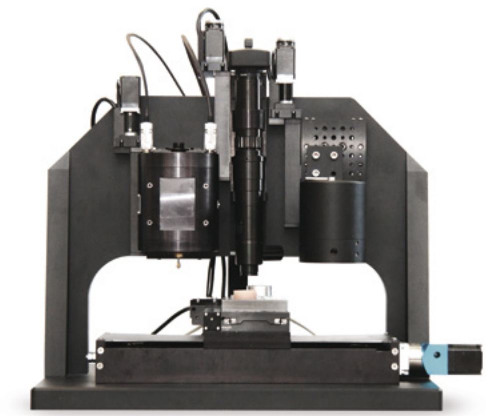
NIOS Advanced is the flagship model, which implements more than 30 different measuring techniques covering the product line widest range of physical and mechanical properties measurements at the submicron and nanometer scale.

With NIOS control software a high degree of automated measurements can be achieved allowing the end-user to configure any set of measurements recipes to be performed without operator intervention. This feature is particularly useful for the technical control of materials quality. With this added functionality, NIOS can be used for research work as well as for industrial applications.

Standard

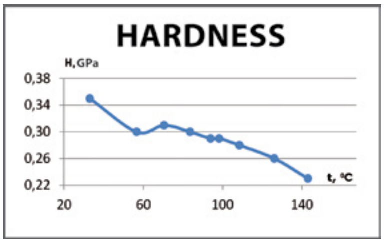
Advanced

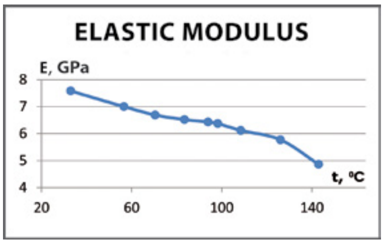


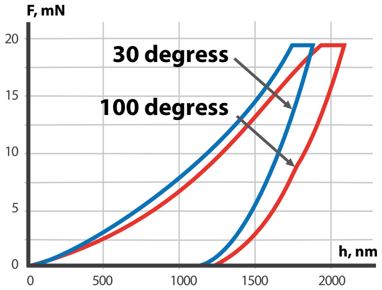


NIOS Standard

NIOS Advanced







Application Areas
Measurement modes and methods
Measured characteristics

Application Areas

The unique features of NIOS allows the instrument to be used in traditional nanomechanical testing and in areas that require SPM techniques to get mechanical properties characterization.

Materials science: scientific research and engineering:

- Nanophase and composite materials
- Ultra dispersive hard alloys
- New hard and superhard materials
- Structural nanomaterials: alloys, composites, ceramics
- Thin films and coatings
- Carbon nanomaterials and fibers

Energy:

- Nanostructured materials for nuclear energy
- Coatings for turbine blades

Instrumentation engineering:

- New semiconductor materials
- Optical components
- Micro- and nanoelectromechanical systems (MEMS and NEMS)
- Microchannel plate for night vision devices
- Storage devices (i.e., Hard Disk Drive)
- Nanolithography

Medicine:

- New materials in dentistry
- Nanostructured materials implants
- Bioactive coatings
- Stents

Industry: automobile, aircraft construction; space research and mechanical engineering

- New structural and functional nanomaterials
- Wear resistant coatings for machine elements
- Coatings for cutting tools
- Carbide tools quality control
- Diamonds and diamond powders

Metrology:

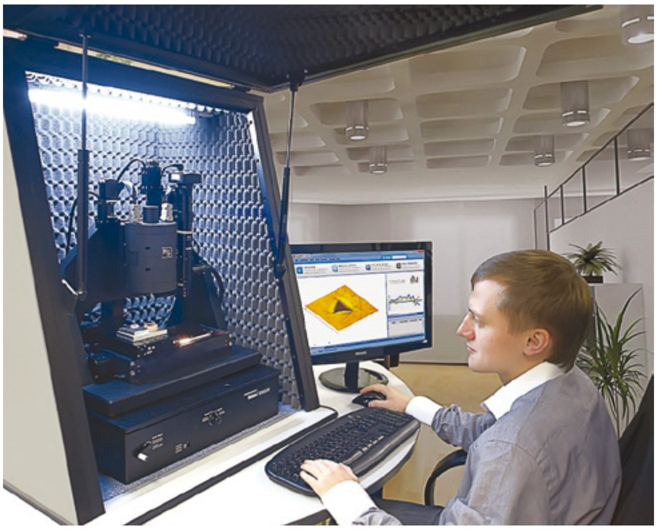
- Linear dimension measurements at the nanometer scale using 3-axis laser interferometry

Packaging:

- Protective coatings for plastic products
- Glass and metal decorative and functional coatings

Education:

- Nanoindentation & Scanning Probe Microscopy laboratory courses
- Advanced research



All NIOS measurements are performed in an open environment (i.e. without the use of special vacuum or heat treatment). NIOS instruments are designed with features and functionalities that allows its use for research and industrial applications.

Measurement modes and methods

Mechanical properties:

- Instrumented indentation in accordance with ISO 14577
- Vickers microhardness measurements
- Sclerometry tests (hardness measurements by scratching) with constant or variable load
- Force spectroscopy
- Mechanical nanolithography
- Beams and membranes stiffness measurements
- Hardness and elastic modulus dependency of the indentation depth
- Automated mapping of two- and three-dimensional hardness and elastic modulus distribution over the surface within the area of 50x50 mm
- Adhesion characterisation by the scratch test
- Measurements in liquids

Nanotribology:

- Cyclic surface abrasion with an applied load
- Nanotribological tests with a lubricate on an investigated surface

Optical microscopy:

- Selection of the field for nanomechanical testing
- Objects' sizes measurements and high-precision positioning

In-situ modes of scanning:

- Surface profiles on a linear basis up to 50 mm measurements
- Surface topography semi-contact dynamic scanning by the diamond indenter

Local electrical properties:

- Current-voltage characteristics measurements with a controlled load or an indentation depth
- Current Spreading measurements in the nanomechanical tests

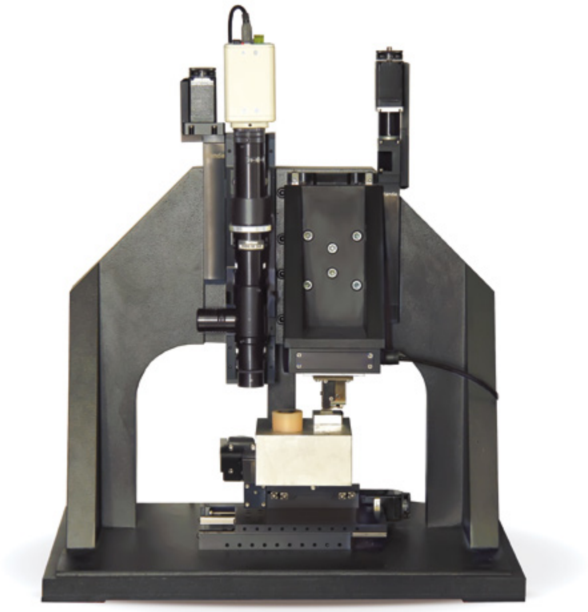
Atomic Force Microscopy:

- Contact Atomic Force Microscopy (AFM)
- Vibrational (semi-contact) Atomic Force Microscopy (VAFM)
- High magnetic fields microscopy (M-AFM)
- Lateral Force Microscopy (LF-AFM)
- Hardness measurements according to residual imprint
- The extended set of roughness parameters calculation for two- and three-dimensional images of the surface relief in accordance with international standards ISO 3274, ISO 4287, ISO 13565 and ISO 16610

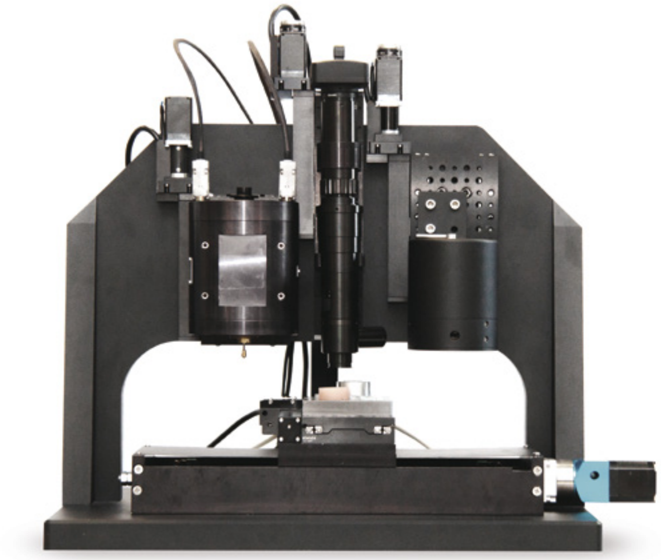
Measured characteristics

- Hardness from imprint (microhardness)
- Hardness from instrumented indentation (nanohardness)
- Elastic modulus (reduced young's modulus)
- Coefficient of elastic recovery
- Adhesion
- Coating thickness
- Mechanical properties mapping
- Mechanical properties vs depth
- Mechanical properties vs three coordinates (tomography)
- Microstructures stiffness and displacement
- Fracture resistance
- Durability
- Linear wear intensity
- Coefficient of friction
- Lateral force during scratching
- Surface topography
- Roughness parameters
- Local voltage characteristics
- Electrical resistivity

NIOS Products



NIOS Standard



NIOS Advanced

NIOS series of nanomechanical testers is available in three platforms: NIOS Compact, NIOS Standard and NIOS Advanced. Depending on the size of the selected platform the device may comprise one, two of three of the following measurements modules:

- Wide-range nanoindenter
- Scanning nanomechanical tester
- Atomic force microscope
- Optical microscope

Special modification of NIOS Standard instrument can be equipped with three-axis heterodyne interferometer used for application of linear displacements nanometrology.

Depending on the type of the platform, one extends the capabilities of the instrument with additional options such as a lateral force sensor, heating stage, high load option, acoustic emission sensor, reciprocating wear module, etc. For more details about additional units and sensor see page 18.

Wide-range nanoindenter module

Purpose:

The wide-range or instrumented nanoindenter module is designed to measure the mechanical properties of various materials using a wide range of applied loads and depths. The range of application spreads from sufficiently hard materials (sapphire and harder) to quite soft polymeric materials, plastics and some sorts of rubbers. This capability to conduct a measurement for the different types of materials is provided by the big displacement range of the shaft and by the method of load application.

This module is also used for the wear resistance measurements as well as for the scratch testing, which can be used for the mechanical and adhesive properties measurements. Using lateral force sensor it is possible to measure a lateral force and a friction coefficient.

Modes and methods:

- Instrumented indentation in accordance with ISO 14577
- Vickers hardness measurements
- Scratch tests (hardness measurements by scratching) with constant or variable load
- Dynamic stiffness measurements
- Beams and membranes stiffness measurements
- Determination of hardness and elastic modulus vs depth dependencies with partial unloading (fig. 1 and p. 20) and dynamic measurement analysis (p. 29) techniques
- Automated mapping of two- and three-dimensional hardness and elastic modulus distribution over the surface within the area of 50x50 mm
- Adhesion characterisation by the scratch method
- Measurements in liquids
- Additional option for the loads up to 30 N

Technical data:

The indentation module has 4 basic operating modes (Table 1).

Table 1. Force and displacement ranges.

		Displacement			
		10 µm	30 µm	100 µm	300 µm
Load range	30 mN	films and coatings		soft materials	
	150 mN				
	400 mN	hard materials		micro-objects, bulk materials	
	2 N				

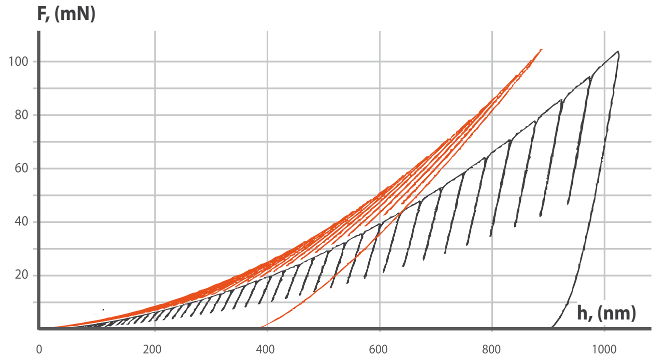


Fig. 1. Load displacement curve for the partial unloading indentation. Red curve: fused silica, black curve: steel.

Optical microscope measurement module

Mono-zoom video microscope with CCD camera. The microscope is used for selection of the measurements position for the AFM and the wide-range nanoindenter / scanning nanomechanical tester modules. It is also used for the measurements of the dimensions of indentation imprints, microelements, microcrystals within metals, composites, powder grains, electrical boards paths, MEMS and others.

Modes and methods:

- Hardness measurements according to the residual imprint area or scratch width
- Crack analysis (fracture toughness)
- Grain size analysis
- Grains size distribution function

Technical data:

- Optical zoom change up to 1500X
- Smooth changing of optical zoom: from 0.58x to 7x
- Field of view: from 1.57 x 2.09mm to 0.13 x 0.17mm
- Working length: 35mm
- Digital USB camera

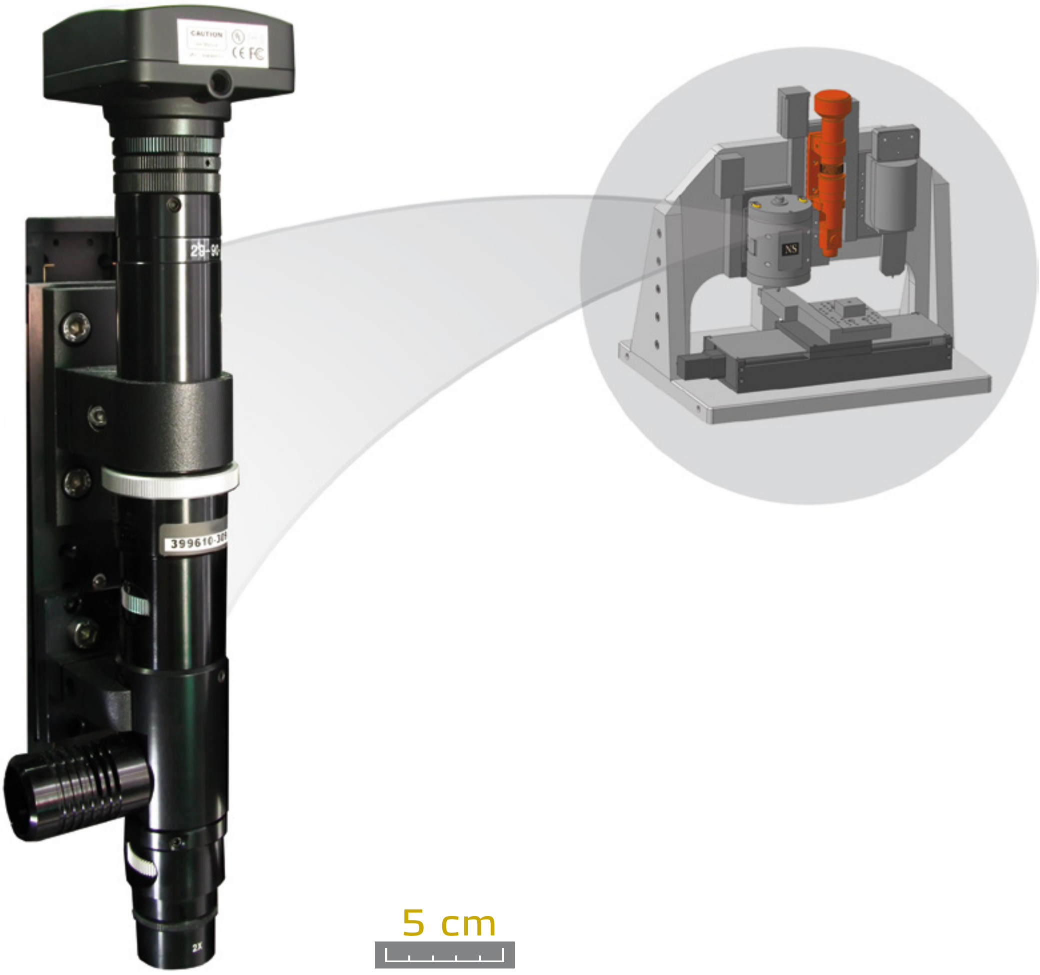
Wide range of illumination options are possible: unshaded circular, optical fiber, fluorescent, coaxial, LED. Polarization set option is possible.

Zoom adjustment:

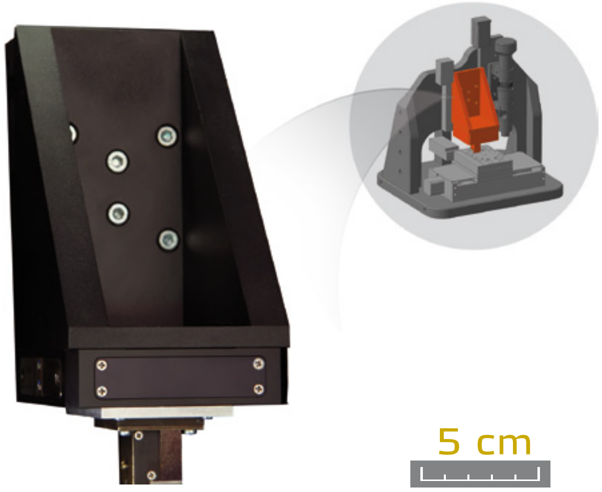
- Manual click-stop zoom
- Automatic zoom

Additional functionalities:

- Auto focus
- Automatic field of view scaling during zoom changing
- Illumination irregularities correction



Scanning nanomechanical tester module



This module is purposed for complex research of mechanical properties in loads range up to 100 mN by using indentation and scratch methods. It's also used for materials surface research in a semi-contact SPM method.

Modes and methods:

- Semi-contact dynamic topography scanning
- Indentation and scratching with a given load or depth
- Scratch hardness measurements
- Hardness measurements by residual indentation imprint
- Mechanical properties measurements by instrumented indentation in accordance with ISO 14577
- Elastic modulus measurements by force spectroscopy
- Materials and thin coatings mechanical properties measurements (hardness, adhesion, coating thickness) by scratching with variable loads
- Wear resistance measurements of thin coatings
- Surface profiling

Technical data:

- Measurement range of X and Y axis no less than: 100 µm
- XY positioning resolution: 2 nm
- Measurement range of Z axis no less than: 10 µm
- Z axis resolution: 0.2 nm
- Maximum load: 50 mN
- Load resolution: 0.5 µm

Three-axis heterodyne laser Interferometer module

The interferometer module is intended for the metrological measurements of surface structures. A source of radiation is a single-frequency stabilized He-Ne laser (power 1 mW, wave length 632,991084 nm, relative instability of optical frequency no more than 3.10^{-9} during 8-hour work).

This module is purposed for a metrological characteristics determination of other SPMs, for providing accurate measurements of the linear dimensions at the nanometer scale and for a nanotechnology products control.

Modes and methods:

- Hardware and software compatible with a Scanning nanomechanical tester module
- Hardware and software compatible with an AFM module
- Surface topography mapping during SPM and AFM modes of scanning

Technical data:

- measurement range of the XYZ axis: 500 µm
- resolution of all three axes (no less than): 0.01 nm
- noise level of the interferometer, RMS in frequency band from 1 Hz to 1 kHz (no more than): 1 nm
- nonorthogonality of the axis displacement measurement: 0.01 radian
- phase displacement range: $\pm 1*10^4$ radian
- phase displacement resolution: 10^{-4} radian
- time measurement resolution: 1 ms
- maximum rate of the scanning: 100 µm/s
- heat release in the working area (no more than): 5 W

Atomic force microscope

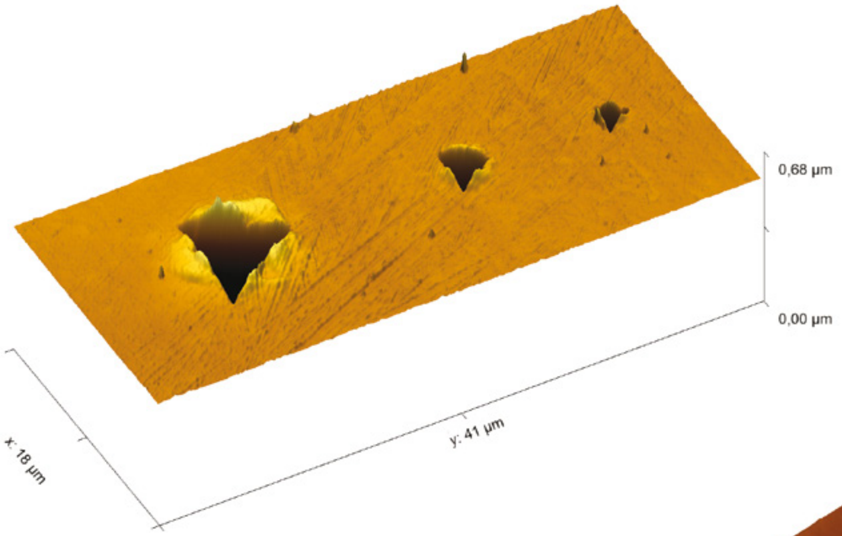
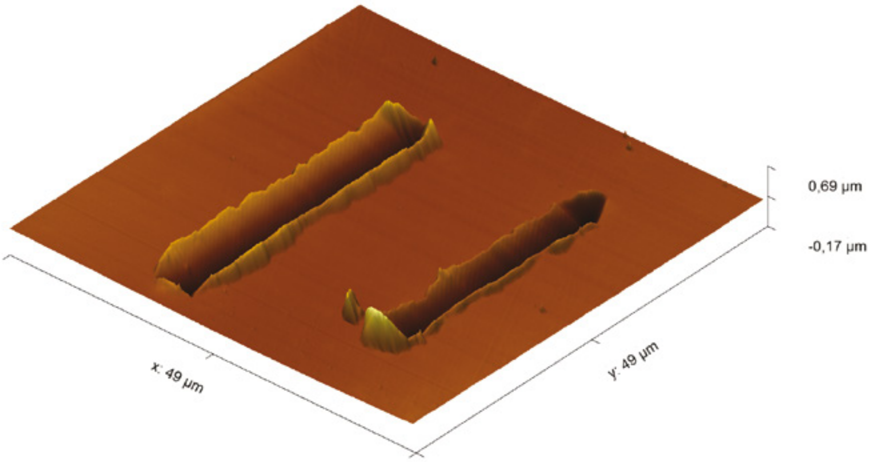


Fig. 1. Plastic indents in steel. 10, 30, 100 mN load.

Fig. 2. Traces made by triangular pyramid Berkovich indenter during the face-forward and edge-forward scratching.



Functionality:

- 3D surface topography imaging with nanometer-scale spatial resolution
- Surface roughness measurement
- Direct method of hardness measurements by images of plastic imprints of indents and scratches. Evaluation of plastic pile-ups and elastic sink-in for mechanical properties measurements correction.

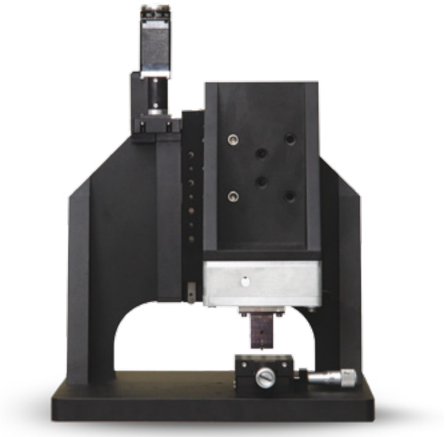
There is a built-in optical microscope for accurate positioning of the scanning area.

Techniques:

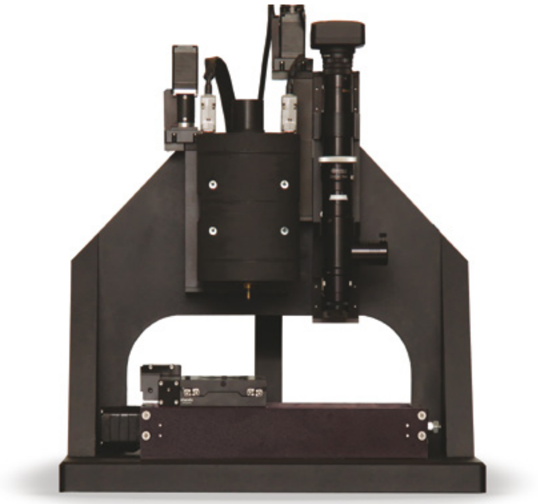
- Contact, semi contact, atomic force phase microscopy;
- Magnetic force microscopy and lateral force microscopy;
- Hardness measurements over the images of indents;
- Roughness parameters estimation by 2D and 3D images of surface topography in accordance with ISO 3274, ISO 4287, ISO 13565 and ISO 16610.

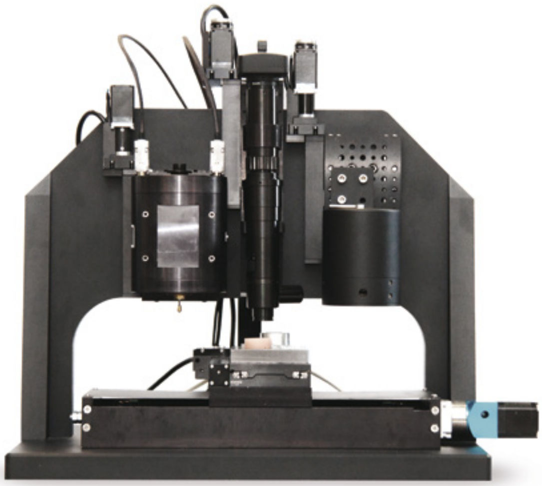
Specification:

- Scanning field of atomic force microscope: XY 40x40 µm, Z 4 µm
- Types of AFM probes: contact, semi contact, magnetic
- Optical microscope resolution: 2 µm; field of view: 390x230 µm



NIOS





NIOS Compact

NIOS Compact is designed for small samples surface mechanical properties research. This device uses the methods of scanning probe microscopy, instrumented indentation and scratching with a load range up to 100 mN. This model is intended for physical and mechanical properties study at submicron and nanometer linear dimensional scale.

NIOS Standard

NIOS Standard incorporates hardness, elastic modulus (and other mechanical parameters) measurement methods. Scratching, static and dynamic indentation are also implemented in this instrument. This model provides the possibility of semi-contact surface topography profiling. Optical microscope ensures high accuracy of positioning of the indenter and the sample. Additional units and sensors available for this model are shown on page 18.

NIOS Advanced

NIOS Advanced is a fully equipped system that implements widest range of methods in the product line. Capabilities and modes of the indentation head are enhanced by the AFM functionality, which allows an investigation of indent's imprints with nanometer resolution. The system provides capability of automated testing and batch data processing.

Configuration Wizard (1)

Step 1: Frame size

Compact (1 head)
200×300 mm

Standard (2 heads)
450×400 mm

Advanced (3 heads)
550×450 mm

Step 2: Heads

Wide-range
nanoindenter module
(NIOS)

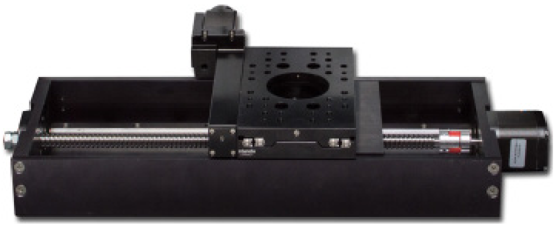
Scanning
nanomechanical
tester module
(NIOS)

Atomic force
microscope

Optical
microscope

Configuration Wizard (2)

Step 3: Sample stage



XY motorized

Manual

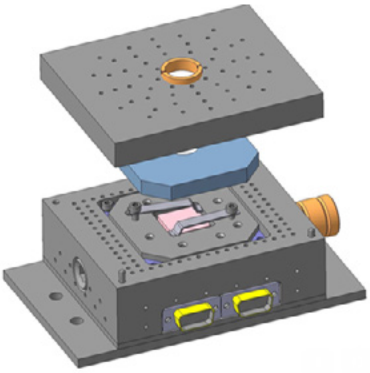
X motorized

Rotation

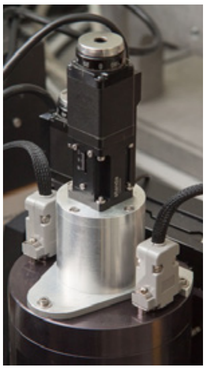
Step 4: Extensions

XYZ scanning stage

Lateral force sensor



Heating stage



Load extension unit

Step 5: Accessories & other

Vacuum chuck

Probes

Indenters

NIOS Configurations Table

Heads:

	Compact	Standard	Advanced
Wide-range nanoindenter module	•	•	•
Optical microscope		•	•
AFM			•
Scanning nanomechanical module	•	•	•

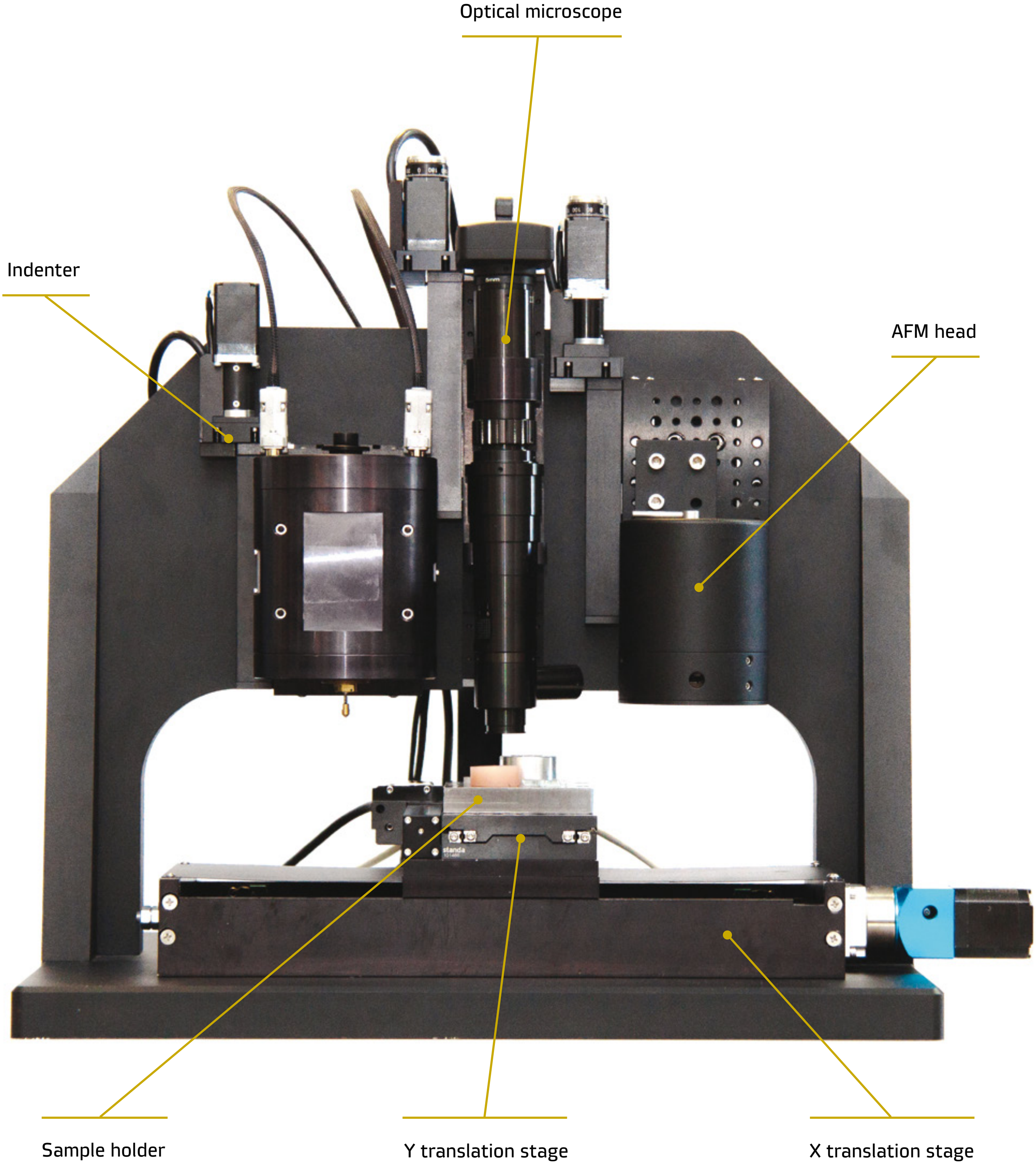
Positioning stage:

	Compact	Standard	Advanced
Manual	optional		
X	•		
XY		•	•
Rotational			optional

Extension:

	Compact	Standard	Advanced
Lateral force sensor		optional	optional
Heating stage		optional	optional
XYZ scanning stage		optional	optional
Electrical properties		optional	optional
High load option	optional	optional	optional

Scanning nano-hardness tester NIOS



Additional units and sensors

NIOS nanomechanical testers have a lot of additional units and sensors. This extends the functionalities of the measurement system and provides a maximum level of device adaptation for customer's needs.

Measurement platform final configuration is selected depending on the customer's research tasks.

In order to deal with unusual research tasks it is possible to create new units, modify existing units and sensors and install on NIOS units from other manufacturers.

Lateral force sensor:

- Lateral force measurements during sclerometry and polycyclic abrasion
- Friction coefficient measurements during tribology tests

In-situ scanning unit:

- SPM mode for a surface topography visualization by diamond indenter

Heating stage:

- Maximum temperature: 400 ^{0}C
- Maximum heating rate: 1 ^{0}C/s
- Temperature stabilization: 0.1 ^{0}C
- Maximum sample dimensions (WxLxH): 25x25x10 mm

Measurement module of electrical properties:

- Current-voltage characteristics and current spreading measurements during mechanical tests

Sample holders:

- Vises
- Clamps
- Supports
- Vacuum cups

Rotary table:

- Mechanical properties anisotropy research
- Sample positioning expanded functionality

Indenters and reference samples

Indenters:

- Indenter tips made of doped or pure high quality synthetic diamonds monocrystals
- Berkovich triangular pyramid
- Knoop four-sided pyramid
- Vickers four-sided pyramid
- Flat punch with a given diameter from 50 μm to 2 mm
- Spherical tip with a given radius

Reference samples:

Reference Sample (RS) is produced from a well-known material with a special surface preparation. Reference samples are purposed for calibration of NIOS devices and inspected to be compliant with established standards. Every RS has a passport of the RS containing standard metrology characteristics, application instruction of transportation and storage conditions.

Polycarbonate
Reference characteristics:

- hardness: 0.21 ± 0.02 GPa
- elastic modulus (Young's modulus): 3 ± 0.3 GPa
- roughness: <5 nm
- dimensions: 10x10x7 mm
- surface preparation: —

Aluminum
Reference characteristics:

- hardness: 0.5 ± 0.1 GPa
- elastic modulus: 70.0 ± 7.0 GPa
- roughness: <5 nm
- dimensions: 10x10x8 mm
- surface preparation: polishing, electrolytic etching

Fused Silica
Reference characteristics:

- hardness: 9.5 ± 1.0 GPa
- elastic modulus: 72,0 ± 3,0 GPa
- roughness: <5 nm
- dimensions: 7x10x4 mm
- surface preparation: deep grinding-polishing

Sapphire
Reference characteristics:

- hardness: 24.5 ± 2.5 GPa
- elastic modulus: 415.0 ± 35.0 GPa
- roughness: <5 nm
- dimensions: 25x5 mm
- surface preparation: epi-polishing

Measurement analysis suite

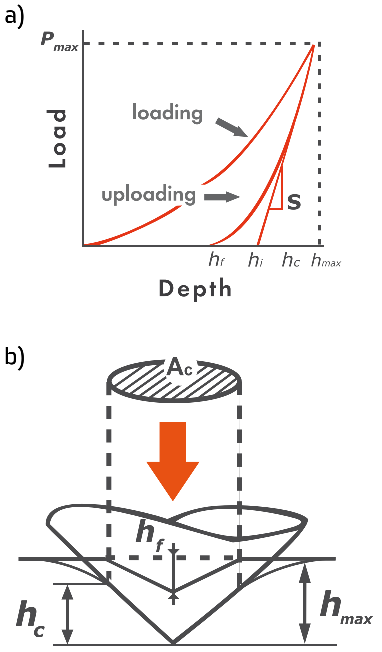


Fig. 2. Typical load-displacement curve with main parameters for hardness and elastic modulus calculation (a) scheme of the indenter and the surface contact (b).

Hardness and elastic modulus measurements by instrumented nanoindentation

Quasistation instrumented intentation testing is the basic function of NIOS devices. The algorithm is based on measuring and analysis of indentation load-displacement data. This technique underlies the international standard of hardness testing ISO 14577. For typical expiremental curve of load (P) versus depth (h) see Fig. 2.

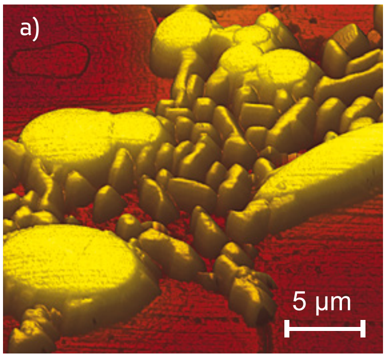
Reduced elastic modulus E_r is calculated from the initial slope of the unloading curve S and contact area A_c. Contact area is defined by the dependency of the pre-calibrated area A_c (h_c) on contact depth h_c, which is in turn calculated from maximum indentation depth h_{max} and unloading slope S. Additional parameter β accounts for the non-axisymmetry of the indenter tip. Reduced Young's modulus of the sample is calculated from E_r according to indenter tip material properties and sample's Poisson ration.

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

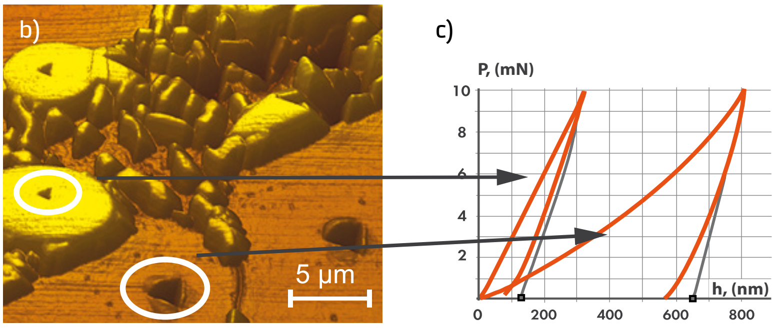
$$E_r = \frac{1}{ß} \cdot \frac{\sqrt{\pi}}{2} \cdot \frac{S}{\sqrt{A_c}} \; .$$

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

Multiphase materials research



Multiphase materials properties study involves precise indenter positioning in the specified areas of the surface corresponding to the individual components. NIOS nanomechanical testers combine the functions of a scanning probe microscope and a hardness tester.
Device allows to obtain three-dimensional surface topography image of a multiphase sample, and then specify measurement location with a connection to the resulting image.



Positioning accuracy of the indenter relative to the surface at measurements is about 10 nm in the XY plane.

Example: D16 aluminum alloy sample (Fig. 3). Topography images of the same surface area before and after a series of measurements.

Fig.3. D16 aluminum alloy. Surface topography: Before indentation (a), After indentation (b), The load-displacement curve for phases with different properties (c).

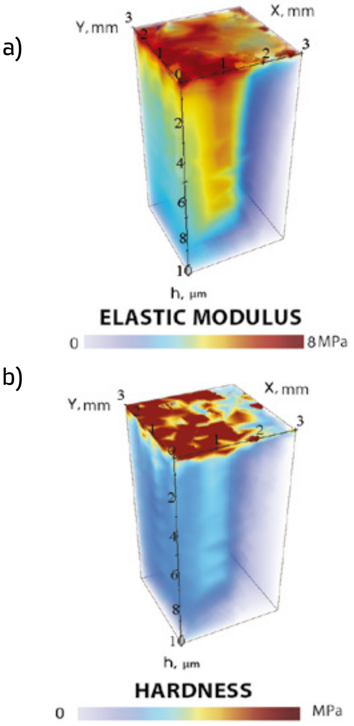


Fig. 4. Elastic modulus tomogram (a), hardness tomogram (b).

Tomograms of mechanical properties

Basic instrumented indentation test (ISO 14577) contains single loading-unloading cycle and thus gives hardness and elastic modulus that correspond to one depth. NIOS testers can perform an indentation with partial unloading (PUL): at a given position on the surface tip penetrates the sample, partially goes back and penetrates further penetrates deeper again. Such kind of penetration repeated within multiple cycles allows to profile mechanical properties along sample depth.

Tests that allow to profile mechanical properties (PUL or DMA) along depth can be arranged in the grid that partially covers sample area, which gives the opportunity to map mechanical properties along three axes (X,Y & Z). Corresponding data are used to construct tomograms of elastic modulus and hardness. For examples of such kind of properties volumetric distribution see Fig. 4. Maximum surface area for the tomogram can be up to 10 cm x 10 cm, maximum depth is limited by sample properties but cannot be bigger than 200 um.

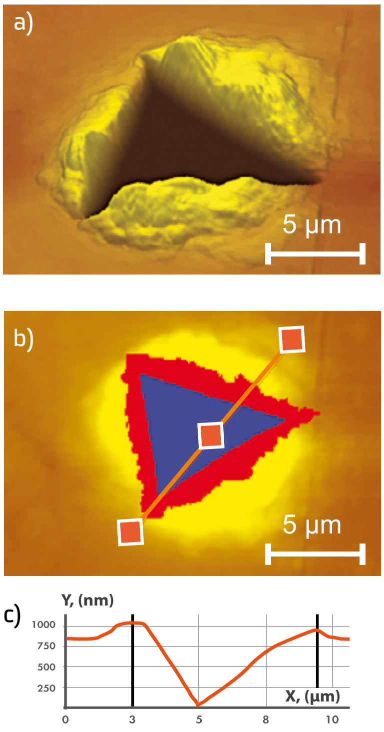


Fig. 5. Indentation imprint on the titanium surface 99% (a); example of automated area calculation (b); indentation imprint profile (c).

Hardness measurements by the residual indentation imprint

NIOS nanomechanical tester provides hardness testing by the residual imprint method (ISO 6507-1:2005). In contrast to traditional micro-hardness testers, the imprint dimensions measurement is made in scanning probe microscopy (SPM) mode. Indentation and corresponding surface topography imaging are performed using the same probe sensor and the same tip. Three-sided Berkovich diamond pyramid is used as an indenter, tip apex angle is 140º and the radius of curvature is ~ 50 nm. According to residual imprint method the hardness is defined as the ratio of the maximum applied load to the area of the residual imprint, measured from the SPM image.
In case of the plastic pile-ups formation on the residual imprint perimeter, a three-dimensional image allows to determine the pile-ups area and take it into account when calculating the hardness value. NIOS software allows automated measurement

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

of indentation area and takes the pile-up effect into account (Fig. 5).

Scratch hardness testing

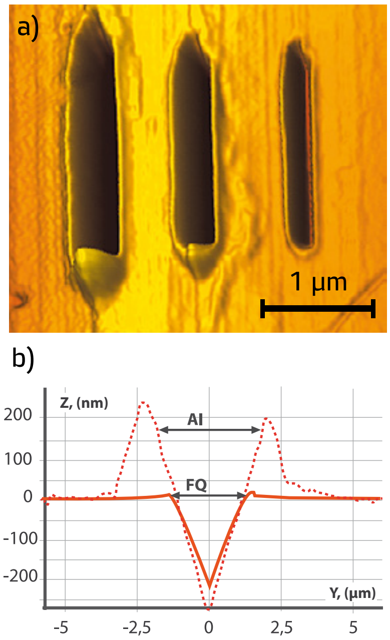
Determination of hardness by scratching implies making a scratch on the sample surface and measuring its width. One can use different NIOS modules to measure this value: optical microscope, AFM, or scanning nanomechanical tester, which scans the surface in SPM mode and makes the scratches with the same probe.

Similarly to instrumented indentation scratch measurements require pre-calibrated function for tip shape. This is achieved by measuring widths b of the scratches, performed on the surface of the reference sample with the different (increasing) loads (Fig. 6). For the given load P hardness H is inversely proportional to the scratch width b, according to the given below equation. Ideal pyramid tip requires single coefficient k for calibration.

Despite scratching does not give an information about the elastic modulus the method has its own advantages. In contrast to the instrumented indentation determination of scratch hardness makes an account for the pile-up effect and, what is of particular importance for the thin and rough films, is less sensitive for roughness.

Table 2. Comparative hardness tests

Material	P, mN, normal load	$R^{scr}_{xy''}$,% creep recovery of scratch width	R^{scr}_{Z},% creep recovery of scratch depth	R^{NI}_{Z},% creep recovery of indentation depth	H^{scr}, GPa scratch hardness	H^{NI}, GPa nanoindentation hardness
Fused Quartz	20	15	47	46	Ref.	10,1
Glass	20	16	49	44	9,7	9,3
Bi_2Te_5	7,6	13	23	30	2,6	2,8
Ni	15	10	15	13	4,7	4,8
Al	1,7	1	3,2	4,2	0,5	0,6



Calibration:

$$H_{ref} = k\frac{P_1}{b_1^2} \quad => \quad k = \frac{H_{ref} b_1^2}{P_1}$$

Measurement: $H_2 = k\frac{P_2}{b_2^2}$

Fig. 6. Examples of the cross-section profile of the residual scratch groove for fused quartz (solid line) and aluminum (dashed line). Arrows indicate the width of the area of contact between the indenter and the material during the scratch test.

Mechanical properties measurements for materials and thin films (hardness, adhesion, thickness) by a variable load scratch test

Thin films are used extensively as protective and wear resistant coatings for a wide range of objects. Accurate mechanical properties measurements of these films without the substrate influence is an important task in the modern quality control systems. NIOS nanomechanical testers allow films hardness measuring by different methods for a wide range of thicknesses. Instrumented indentation is the most common method of measuring physical and mechanical properties of thin films. However, there are several factors that lead to methodological errors in this measurement method. The most critical are surface roughness, residual stresses and so-called "substrate effect" (for the film-substrate system the response of the material depends both on the film properties and the substrate properties). The scratch test method (scratching and scratches profile analysis) has several advantages over indentation methods for measuring the films hardness at the nanoscale. Direct SPM observation of the residual scratch trace allows to minimize the influence of the dominant elastic deformation which is typical for the indentation methods. Scratching at variable load (Fig. 7) makes it possible to define several film parameters in a single measurement procedure: load of plastic deformation (value that corresponds to a visible trace on the surface), positions of film separation and delamination.

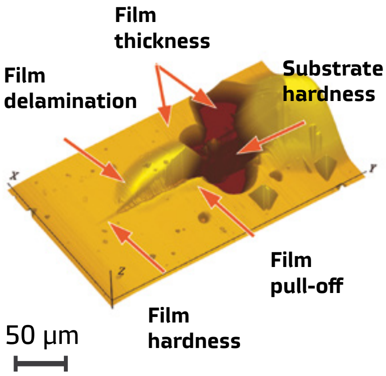


Fig. 7. Scratch with a linear zoom of a load on a surface of a diamond-like film on a silicon substrate.

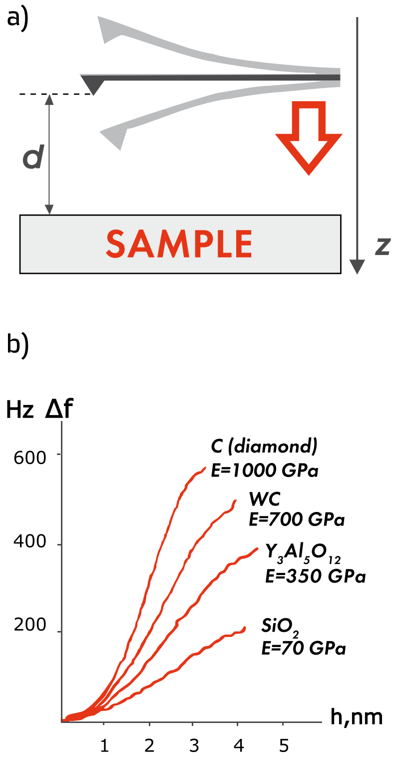


Fig. 8. Approach-retraction curve measurement schema (a); the slope of Δf curve characterizes the elastic modulus of the material (b).

Elastic modulus measurements by force spectroscopy

NIOS nanomechanical testers are capable of measuring the quantitative value of the elastic modulus. According to this method oscillating probe is loaded into the surface. The oscillation amplitude is less than 10 nm, frequency is around 10 kHz. When the diamond indenter contacts the surface, the frequency increases with increasing load.

According to the analytical description based on the Hertz model, the slope of the frequency dependence versus probe displacement (approach-retraction curve) is proportional to the elastic modulus of the material.

Prior to the test the device is calibrated on reference materials with known elastic modulus values. The resulting elastic modulus value is evaluated as a proportion between approach-retraction curves slopes and reference elastic modulus (Fig. 8). This method is nondestructive.

The material layer involved in the test can be as small as 100 nm. This makes it possible to measure thin films elastic modulus without substrate influence. The comparative measurements on different materials showed accurate elastic modulus values in the wide range.

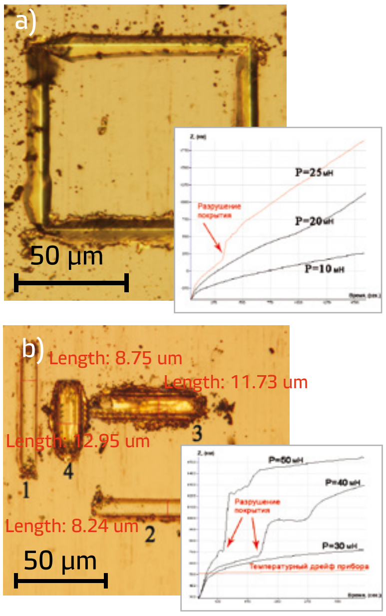


Fig. 9. "Square" tests by a pyramidal diamond indenter (a); wear test results for a spherical sapphire indenter (b). Overlaid graph axes label: horizontal axis T indicates time in seconds, vertical axis Z indicates average penetration into the sample surface.

Wear resistance measurements

Wear resistance testing of coatings is implemented in NIOS devices.

The test principle is based on specified indenter movement while keeping the constant normal load and recording the normal displacement of the indenter. Indenter will deepen inside the surface due to material wear. After some time the indenter will break the coating and reach the substrate, which is indicated by change in the diagram slope.

In case of standard triangular tips being used indenter moves in a «square" path taking account of the indenter asymmetry (Fig. 9a). In case of spherical indenters made of different materials being used reciprocating indenter movement is realized (Fig. 9b)

Mapping of mechanical properties during surface topography scanning

Semi-contact surface scanning and quasi-static mechanical testing are two basic options of NIOS scanning nanomechanical tester module. Measurement head (which uses a piezo ceramic probe working in self-oscillation circuit) construction allows to simultaneously measure not only surface topography but characterize its mechanical properties as well.

Such kind of additional information that can be recorded during any surface scanning provides rapid mechanical characterization. For simultaneous surface topography and elastic properties mapping example see Fig. 10. The resolution is about 10nm in XY plane and about 1 nm along Z axis.

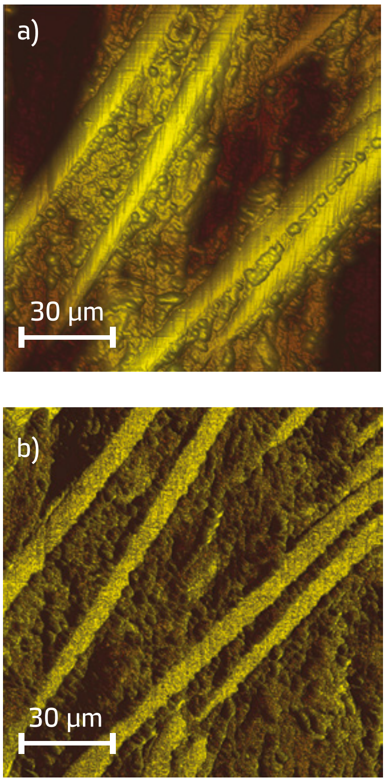


Fig. 10. Composite carbon fibers. Surface topography (a); stiffness map (b)

Dynamic hardness measurements

Dynamic hardness measurement is implemented in NIOS devices. This method is based on a simultaneous processing of oscillating and direct movement of the indenter. This method is less influenced by surface roughness compared with quasistatic nanoindentation. However it requires information about elastic modulus value. The final equation used for such kind of measurements is the following:

$$\frac{H}{E^2} = \frac{F}{\pi}\left(\frac{f_0}{\Delta f}\cdot\frac{1}{k}\right)^2$$

where F and Δf – force and resonance frequency shift, both are measured during the scanning, f_0 and k - probe resonance frequency and dynamic stiffness. Last two parameters are determined during calibration procedure and considered to be constant in future measurements. This equation leads to H/E^2 determination, (the value of H or E if the other one known) as a function of the depth or coordinates of the surface. For hardness map of a hard fiberglass in soft matrix and fused silica hardness versus depth examples see Fig. 11a and Fig. 11b accordingly. In both cases elastic modulus was taken from the other sources.

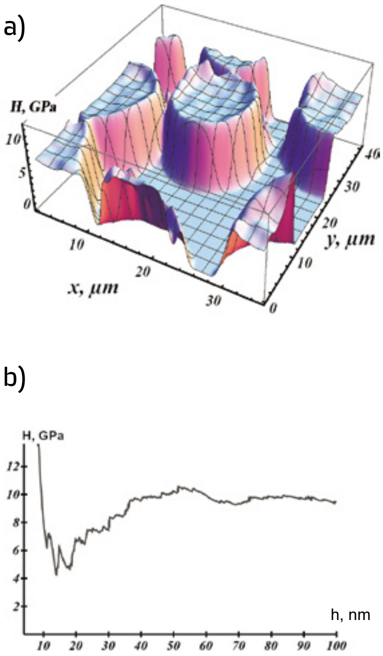


Fig. 11. Hardness map of a hard fiberglass in soft matrix (a); fused silica hardness versus depth of indentation (b).

Surface profiling with the mechanical tester module

Fig. 12. A sleeve (a) and the corresponding profile of cylindrical surface (b).

Scanning nanomechanical tester and wide range indenter modules can measure surface profiles. Maximum length of measurement makes up to 10 mm and 100 mm correspondingly. Maximum measurable slope for the standard indentation tip (Berkovich pyramid) is 10 degrees, optional tips are available. Horizontal resolution depends on the type of the module used, scanning speed and has a limit of 10 nm in horizontal direction and 10 nm in vertical direction. All profiles are obtained in semi-contact scanning mode. For the example of sleeve profile see fig. 12

Application:

- Surface roughness measurement
- Part's shape control
- Small objects location
- Surfaces flatness

Mechanical Nanolithography

NIOS devices provide ample opportunities for precise mechanical micromachining and nanolithography. Diamond tip can cut almost all known materials. By controlling the load during the cutting process with a resolution of 10 µN, one can steadily get scratches with 100 nm width and a several nanometers depth (Fig. 13). The maximum scratch depth reaches several microns.

By using high-precision piezoceramic nanopositioners and mechanical linear translation stages, the diamond tip positioning accuracy reaches 10 nm in a 100 x 100 µm area and about 1 µm in a 100 x 100 mm area.

The result of a surface micro treatment can be controlled by the same diamond tip via scanning in the SPM mode or via digital optical microscope.

The mechanical nanolithography mode can be used to create regular structures on the surface (Fig. 13b), remove oxide films, clean the coatings in selected areas (Fig. 13a) and adjust the microelectronics and micromechanical systems (MEMS) elements' geometry.

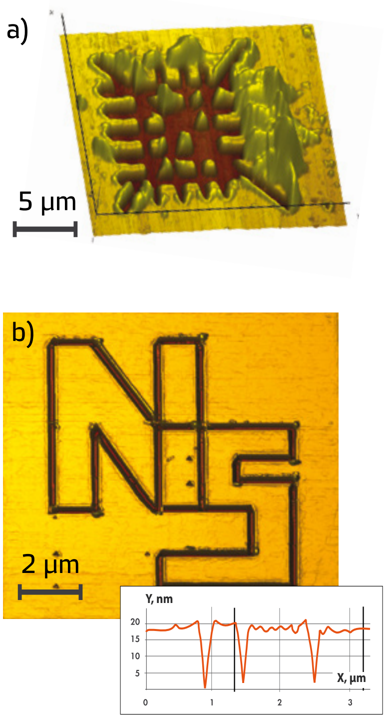


Fig. 13. Golden coating removed from a diamond substrate (a); inscription made by scratching the fused silica surface; scratch profile (b).

Micromechanical stiffness measurements

NIOS devices are capable to control stiffness of different objects: cutting tools (Fig. 14a), beams, MEMS and NEMS beams and membranes. The value is calculated according to the force-displacement diagram, i.e. the same curve as it is used in the instrumented indentation analysis (see page 19). Different modes of loading are possible, including multiple loading-unloading mode, which allows to get average stiffness value as well as to determine a number of cycles to failure.

For scheme of membrane properties measurement see Fig. 14 b,c.

NIOS systems allow to determine the measurement location by in-situ SPM pre-scannning (scanning nanomechanical tester module) or with the help of optical microscope that works with the wide range indenter module.

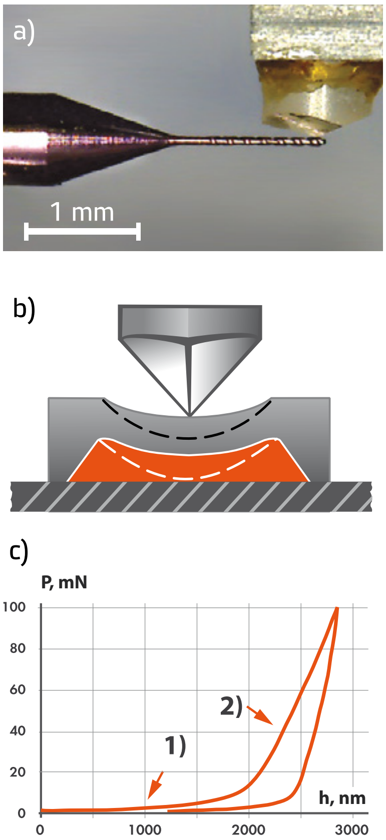


Fig. 14. Stiffness measurement of the cutting tool (a). Membrane propeties measurement scheme (b). Loading-unloading curve (c): 1 - membrane bending (stiffness measurement); 2 – contact with the substrate.

Surface electrical properties measurements

Boron-doped diamond tip allows to conduct electrical properties analysis: measure sample's specific resistivity, current spreading during the surface scanning or indentation. Measurement of volt-ampere characteristics with the given load or penetration depth is also possible. Current spreading map is measured with the constant voltage bias between the tip and the sample. Measurement of current spreading at the different surface locations allows to identify location with the different conductivity and to compare them with the surface structure and inclusions. For ALCuO conductivity map with the quasicrystal phase see Fig. 15a. Areas of different colors correspond to different crystal structures of the alloy. For electron microscopy image of the same surface area see Fig. 15b.

Measurement of the current spreading during the indentation allows to investigate material's heterogeneity along its depth, control coating thickness and study phase transitions in semiconductors under pressure. Simultaneous processing of force and current vs depth dependencies allows specific electrical resetivity calculating. Measurement of volt-ampere characteristics is conducted in the contact with the material with the force ranging from 0.1 mN up to 100 mN. Voltage range is ±10V, current range is ±30uA, current measurement resolution is better than 10 pA.

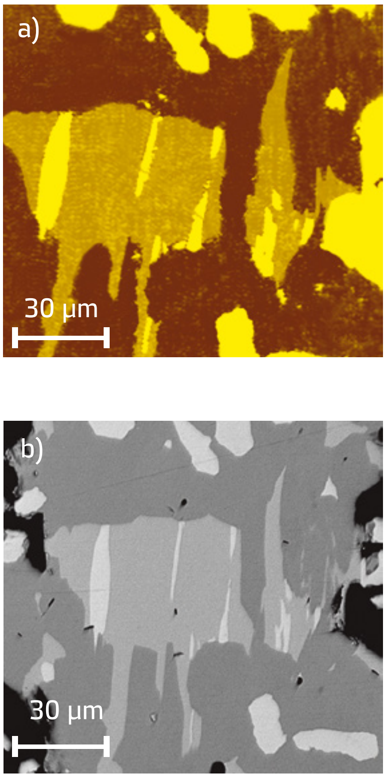


Fig. 15. Surface's micro-structure of AlCuCo alloy. Map of conductivity (a); electron microscope image (b).

Crack resistance measurements

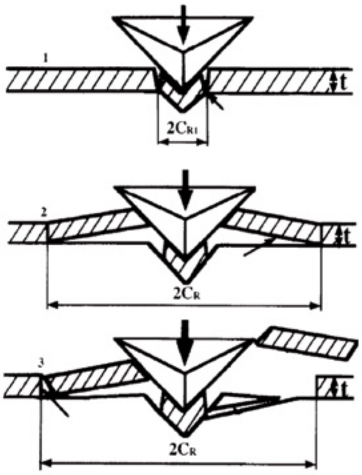
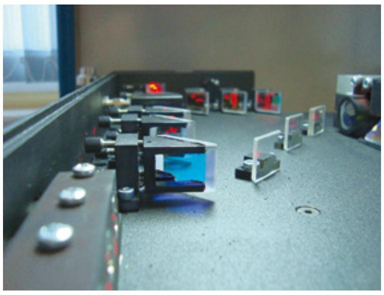


Fig. 16. Scheme of coating destruction with cracks formation

Material's ability to resist crack propagation is often described by the fracture toughness Kc. The parameter is particular important for brittle bulk materials and coatings. Nanoindentation provide a possibility to determine this property with high spatial resolution. There are several tens of indentation-based relations to get Kc value, all of them use the load and crack dimensions, the parameters that are measured by NIOS system. Each approach requires the determination of cracking mode, which can be performed NIOS modules: optical microscope, AFM or in-situ SPM scanning depending on the problem scale.

NIOS systems provide a possibility to investigate cracks both with the instrumented indentation and scratch method, these methods in combination with the methods to get elastic value and surface imaging, give the researcher set of tools to get elastic, plastic and fracture properties of the material.

Metrological support of linear dimensional measurements at the nanoscale



Meas-ure	Step Height, nm	
	NIOS	PTB (Germany)
TGZ1	18,1± 0,2	18,4 ± 1,0
TGZ2	100,0 ± 0,4	101,1 ± 1,6

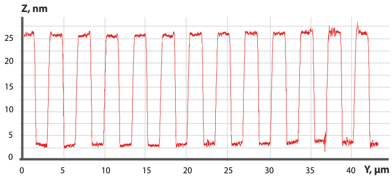


Fig.17. A profile of the TGZ1 measure (21,4 nm) measured by NIOS.

Metrological characteristics of SPMs and metrological support of linear dimensional measurement at nanoscale are very important for equipment dealing with technological and certification tasks and for nanotechnology products control. The interferometer module of NIOS is designed as a compact plug-in instrument for the real time measurements. A source of radiation is a single-frequency stabilized He-Ne laser (power 1 mW, wave length 632,991084 nm, relative instability of optical frequency no more than 3x10-9 during 8-hour work).

Metrological characteristics inspection was conducted with linear measures TGZ1, TGZ2, TGZ3 calibrated by SPM in PTB (Germany). For obtained results comparison see Table 3. For all three measurements results are included in 95% confidence interval estimated by PTB equipment. Obtained results certify the designed device as an etalon of linear dimensional measurements at nanoscale that allows to support a traceability of linear dimensional measurements of nanostructures by scanning probe microscopy methods.

Mechanical testing of micro-objects

A high accuracy of mutual positioning of an indenter and a sample, and using indenters with different geometry allow to implement in NIOS devices mechanical properties testing of micro-objects. In particular there is a method of mechanical durability determination of polyelectrolyte microcapsules produced with LBL technology based on a consecutive adsorption of poly-cations and poly-anions on a charged substrate.

Characteristic diameter of objects can be from several to hundreds microns. Accurate geometry of the object is defined by using an optical microscope. Diamond flat stamp with a specified diameter is used as a tip. The load, after which capsule destruction is occurring, is defined from a registered load-displacement curve (see fig. 18 c). Mechanical durability is defined from ratio of the load to the capsule diameter.

This method is widely used for biological objects, pieces of dye used in the toner and during testing of particulate abrasive materials.

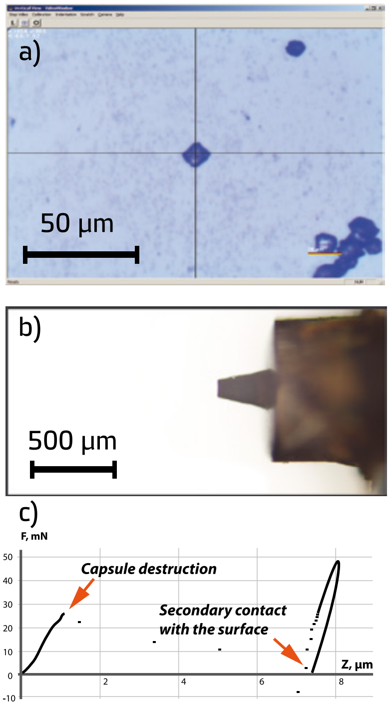


Fig.18. Object positioning by microscope (a), micrograph of a diamond flat stamp indenter (b), load versus displacement registered while capsule compression (c).

Mechanical properties profiling

Due to automatization of conducting series of measurements along specified line or area, the mapping and profiling methods of mechanical properties are implemented in NIOS devices.

This method is important for studying object which structure has inhomogeneous mechanical properties. For example, a golf ball consists of a core and a several layers with different properties with thickness range from microns to millimeters (Fig. 19a). Measurement can be conducted in the open air and in liquid.

NIOS software allows automatical measuring, profiling dozens mapping of hardness and elastic modulus distribution over the area from dozens tens microns to 100 millimeters with specified step between points.For measured profiles examples see Fig. 19b and Fig. 19d. Results of properties measurements are shown in Table 4.

Table 4. Properties of different layers in the golf ball.

Parameter \ area	Core	Inner protective layer	Polyurethane layer	Inner ink layer	Outer ink layer
Thickness, um	-	1100	800	12	15
Hardness, MPa	15	45	15	10	5
Elastic modulus, MPa	100	600	150	80	60

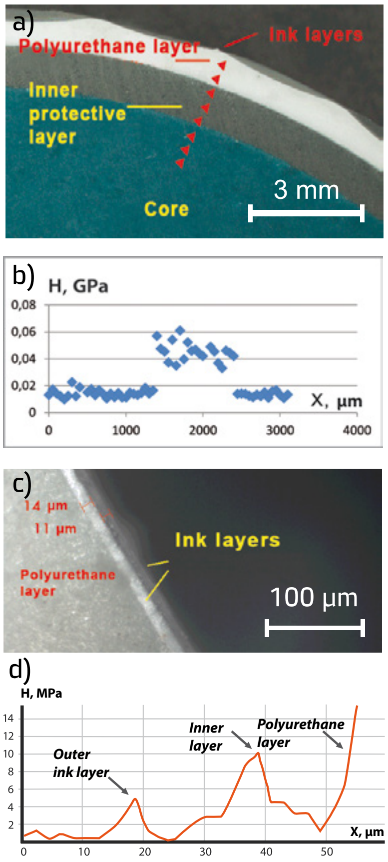


Fig. 19. Optical images of the sample and hardness profile. Core, inner layer, polyurethane layer (a), (b); polyurethane layer and two ink layers (c), (d).

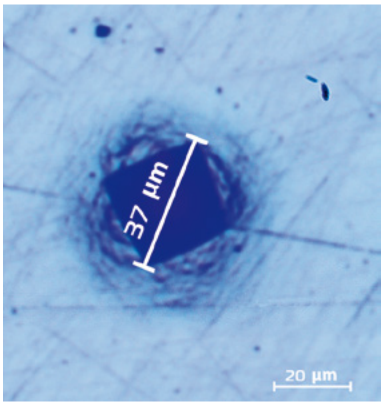


Fig. 20. Imprint in a standard hardness measure. Load is 200g. Hardness is 270 HV 0.2.

Vickers microhardness measurements

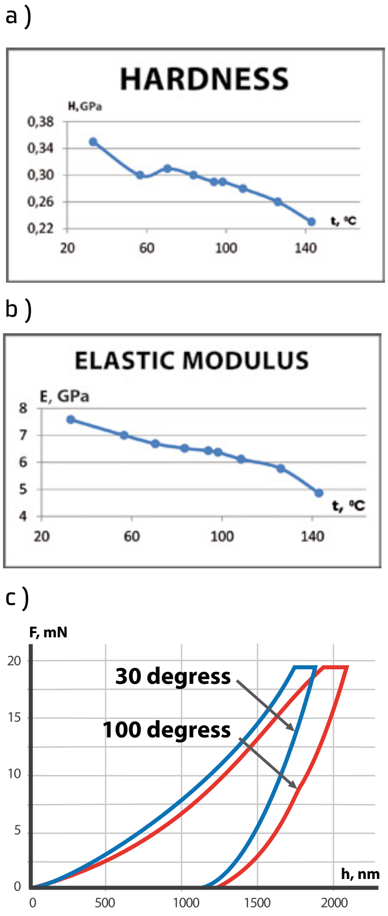
NIOS nanomechanical tester provides microhardness measurements over the residual indent image in accordance with ISO 6507. This method is used in usual microhardness testers.

Four-side Vickers pyramid is used as an indenter (the angle between opposite sides is 136⁰). Measurements are provided with optical micro-images (see fig. 20). Hardness HV is calculated as a maximum of an applied to indenter load divided to an area of residual imprint measured with its image:

$$HV = \frac{P}{F_{пов}} = \frac{1{,}8544 \cdot P}{d^2} \quad .$$

d – medium length of a quadrangular imprint (mm), P – maximum load (kgf).

Vickers hardness is one of the commonly used methods of a hardness measurement. Combination of that and other methods of a nanohardness measurement allows to compare and define hardness at different scales.



Measurement of temperature dependence of mechanical properties

The heating stage with a heating control is applied for materials mechanical properties measurements at high temperature. The heating stage allows to heat a sample up to 400 ⁰C and conduct all types of mechanical tests implemented in NIOS. Temperature control accuracy is 1 ⁰C.

Test results include hardness, elastic modulus, creep recovery, crack resistance, wear resistance and other characteristics at a specified temperature.

Typical sample dimensions for temperature testing in NIOS are 25x25x10 mm.

For example, Fig. 21a and Fig. 21b show hardness and elastic modulus versus temperature graphs of polymethylmethacrylate (PMMA) at 140 ⁰C.

Fig. 21. Hardness and elastic modulus versus temperature graph of PMMA (a); Load-displacement curve at 30⁰C and 100⁰C, measured on a PMMA sample (b).

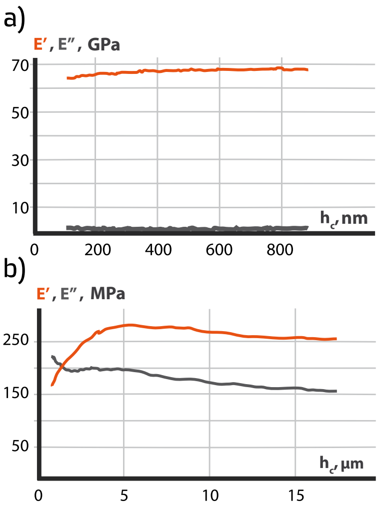


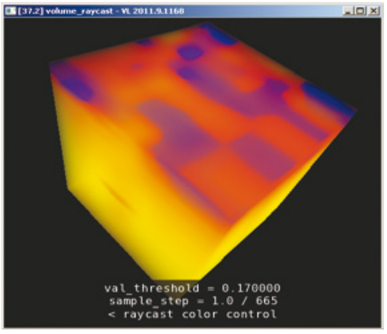
Fig. 22. Measurement of elastic modulus E$^{|}$ and loss modulus E$^{||}$ of a fused silica (a); bitumen (b).

Dynamic measurement analysis

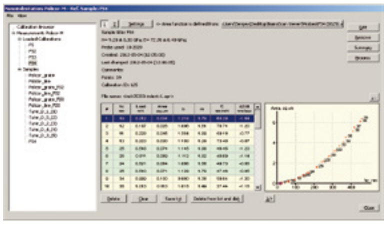
NIOS supports measurements of mechanical properties by Dynamic Mechanical Analysis.
According to the method oscillating force is imposed over the linear increasing load and applied to the surface while corresponding in-phase and 90 degrees out of phase components are measured.

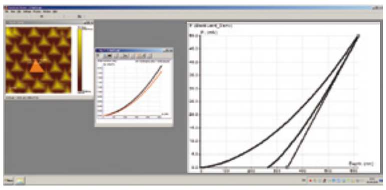
Obtained data is used for the calculation of real and imaginary components of signal, which in turn are used for the calculation of storage and loss elasic moduli E′ and E′′ (Fig. 22). Hardness value is calculated as well.

Supported range of frequencies goes up to 50 Hz, oscillation without the analysis may be performed up to 250 Hz.



val_threshold = 0.170000
sample_step = 1.0 / 665
< raycast color control





NIOS software

- Automatic measurements by instrumented indentation, sclerometry, force spectroscopy, AFM and SPM methods
- Parameters setting using optical microscope or surface topography image obtained with AFM or SPM method
- High performance of experimental nanoindentation data (using new parallel calculations) batch processing
- Flexible setting of the indentation reports
- Macro-commands language allows to perform a random sequence of the measurements in the automatic mode
- A wide set of mathematical functions allowing to perform a variety of the conversions, plotting of the spectra, filtering, obtaining two- and three-dimensional data and approximation of the curves
- Two- and three-dimensional mapping of hardness and elastic modulus distribution versus coordinates
- Measured mechanical properties and the roughness parameters calculation in accordance with appropriate standards

For notes

ostec
corporate group

future's
in the making

Nanomechanical Testers
NIOS

Official dealer in your country

Ostec Instruments

+ 7 (800) 700-65-55

info@ostec-instruments.com
www.ostec-instruments.com