Product information ZHN-S

Robust nanoindenter for mechanical testing in industry and research

ZHN-S Nanoindenter

Detailed view with sample holder and measuring head

Scope of application

The nano- and micromechanical testing system ZHN-S is ideal for measuring indentation hardness and indentation modulus according to DIN ISO 14577 (Determination of hardness and other material parameters of metallic materials and layers) as well as many other mechanical parameters. There are three measuring heads to choose from with maximum forces of 0.2 N; 2 N or 20 N, which can be easily exchanged by the user. This means that the device covers the nano to macro range. The extremely robust measuring heads for this device class behave well even when overloaded.

All measuring heads can be used to carry out dynamic measurements with an oscillating tip at frequencies up to 300 Hz. This enables depth-resolved measurements of hardness and elastic modulus, fatigue tests or measurements of viscoelastic properties.

With spherical tips, the profile of the surface or existing impressions can be scanned tactile like with a profilometer. Roughness values can be determined as well.

Using the 2 N or 20 N measuring heads, the device can carry out scratch and wear tests, but in contrast to the larger ZHN without measuring friction values.

A comprehensive, easy-to-use and flexible software allows automatic data analysis depending on the selected measurement method, the creation of reports and the export of data in all common formats.

Advantages and features

- Predefined applications in the *InspectorX* instrument software can be selected by a click or called up as saved apps. This reduces operator errors and makes training easier.
- Useful presets make operation so easy that no specialists are required. This reduces testing costs.
- Changing the indenter is quick and easy possible without additional protective measures or recalibration. Stored indenter calibration data is available on demand.
- Very high-resolution optics with a 20 megapixel color camera allow 4x zoom without loss of resolution. Changing lenses is not necessary.
- Intelligent measurement of layers through automatic fit function and range selection of the depth-resolved hardness and elastic modulus curves.
- The rigid frame construction with the indenter in the axis of movement of the height drive prevents any moment of tilt and leads to very low device compliance.
- A precise definition of the measurement positions in the camera image and an easy shift or rotation of point groups allows a quick adaptation to the sample geometry with a positioning accuracy of the indentations of ≈ 1 µm.
- A stable work bench, a cabin for thermal and acoustic insulation and an active vibration damping system (recommended) are available as options.

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Functional description

Modular device consisting of

- 2-column load frame with central spindle drive, precision guide and granite base
- Programmable motorized cross table (X, Y)
- 3-axis stepper motor control by a PCIe plug-in card
- Control electronics for sensors and actuators
- Interchangeable measuring heads
- Control and analysis software *InspectorX*
- Autofocus function
- Dynamic module for tip oscillations up to 300 Hz (QCSM / CSM) optional
- Optional passive or active vibration damping

In contrast to devices from other manufacturers, the measuring heads work with the same measuring range in both tension and compression directions. This also makes micro tensile tests possible.

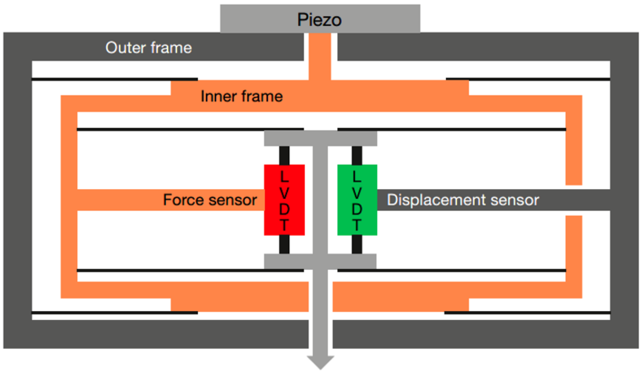
The device can work with force or displacement control in “open loop” mode (only maximum force/displacement is controlled) or “closed loop” (every individual measuring point is controlled). A constant strain rate loading can be programmed as well. The maximum acquisition rate is 4000 readings per second. The internal control (feedback) works with over 50 kHz.

The dynamic module generates sinusoidal oscillations of the measuring tip. This enables continuous stiffness measurements, fatigue tests and the measurement of viscous material properties. The robust measuring head structure allows the use of self-made indenters or counterparts of any shape. With a shaft extension, measurements can be carried out in liquids or in holes.

The software allows a quick and flexible programming of the measuring process (application) and the measuring positions. The measurement positions can also be defined in the camera image by clicking on the desired location. In addition, a variety of unique evaluations are available in the software modules.

Patented measuring head (2N and 20N)

- Thanks to the double leaf spring system, flexibility in the normal direction and very high rigidity in the lateral direction is realized
- Independence of force generation and force measurement. Forces that are not generated by the measuring head can be measured without any problems.
- Robust construction
- The inductive sensors have no limit stop and will not be damaged when overloaded



Schematic diagram of the Normal Force Unit (NFU)

The optics

- 20x lens combined with a high-resolution 20 megapixel color camera gives a maximum magnification > 2000 x
- 4x zoom without loss of resolution
- Within the optical image you can
  - Define measuring points
  - Measure distances and circumferences
  - Review and display existing measuring points at the push of a button
  - Control of illumination and image parameters
  - Show scales and recording date and time
- Even slightly reflective surfaces such as glasses can be imaged well
- Autofocus function to find the focal plane for a sharp image
- Automatic creation of images at the measuring points (programmable)
- Calculation of an image with a large depth of focus from many single images at different height positions

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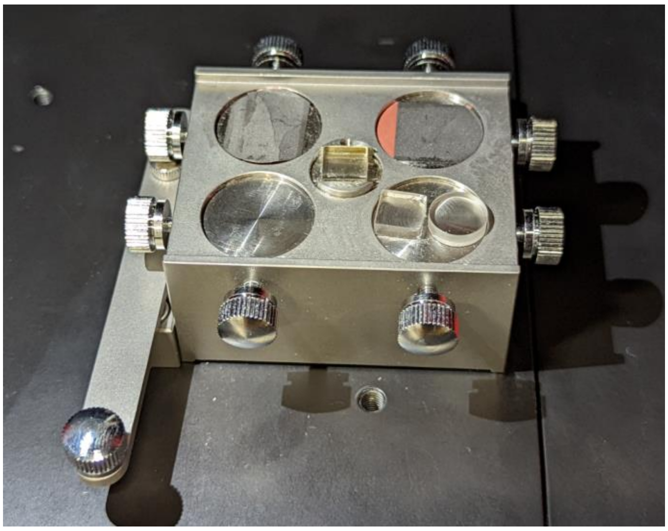
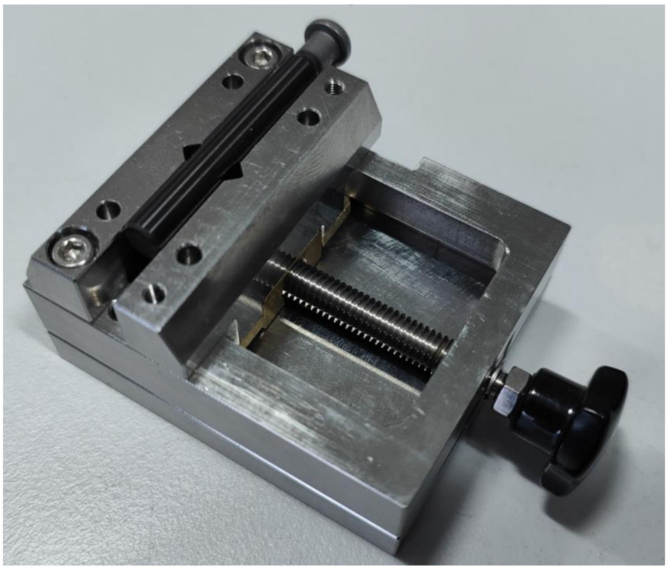
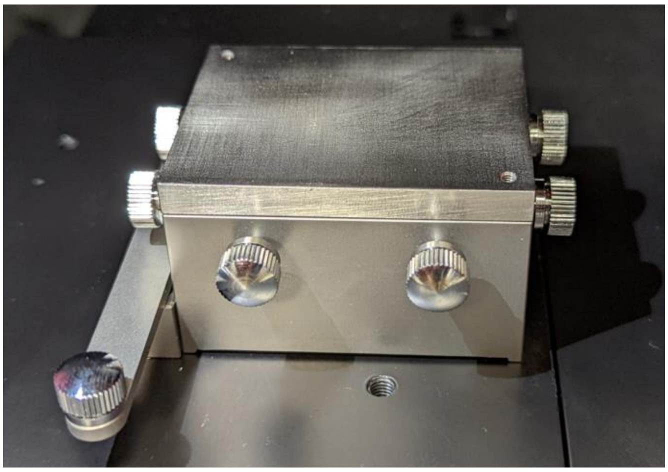


Sample holder with magnetic clamping for four samples and a permanent tip-check sample in the middle

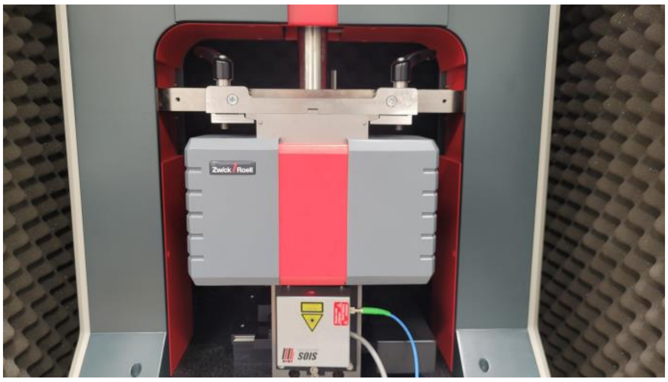


Clamping sample holder for samples up to a maximum width of 32 mm

Tool for changing tips without additional protective measures



Sample holder with adapter plate and a usable measurement area of 60 x 40 mm



Displacement calibration of the measuring head with a Laser-Interferometer

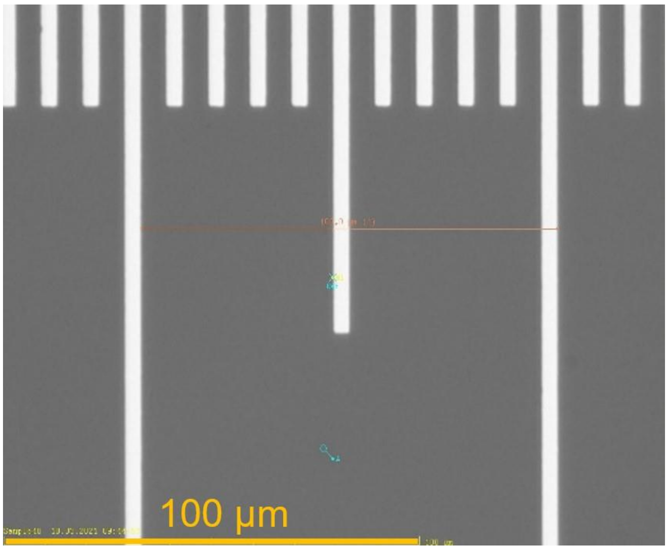


Image of an optical grating with 10 µm bar spacing

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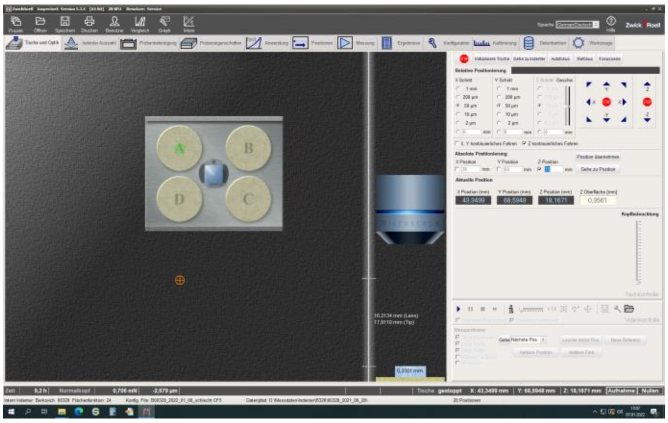
The user interface

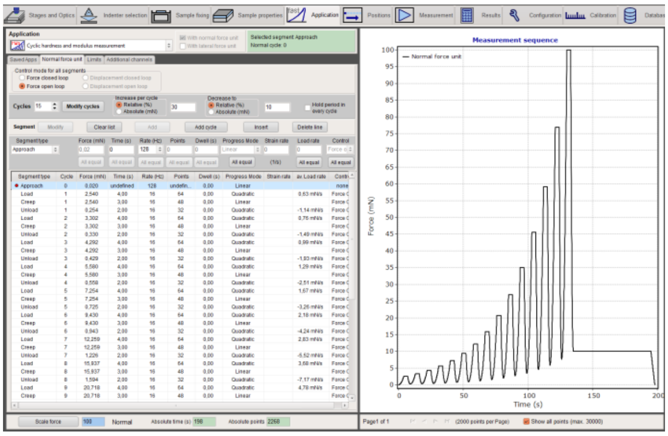
Control of the precision tables

The device is designed for fully automatic measurement series with up to 10,000 measuring positions. The InspectorX control software allows a complete overview of the current position of the precision stages and enables control with step sizes of 50 nm. If the sample is under the lens, an image of the sample surface is displayed in the same window instead of the stage positions.



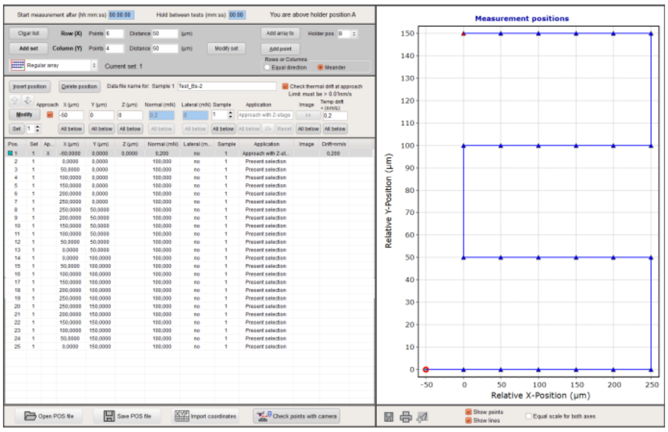
Definition of the measurement sequence

A variety of predefined applications are available, which can be selected in a pull-down menu. Each sequence (test cycle) can be flexibly programmed with any number of load-unload cycles. In the “open loop” mode, force or distance, the time of a segment and the data rate can be specified; in the “closed loop” mode the number of data points and the holding time per point can also be specified.



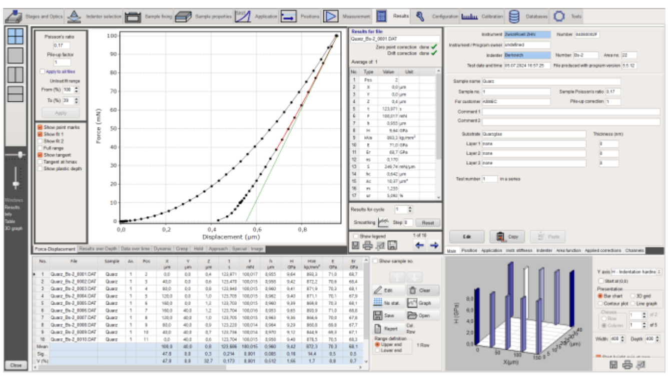
Definition of the measurement positions

Up to 10,000 positions can be programmed in the form of lines, uniform grids or any desired arrangement. For every position a different force or a different test cycle (application) can be defined. Images can be generated automatically before and after the measurement using the auto focus function. Extensive specimen information can be assigned to the individual positions, which is also stored in the data file.



Analysis of measurement data

Measurement data can be graphically displayed, compared, averaged or exported in various forms (ASCII, EXCEL, BMP ...). Extensive and flexible correction routines are available for data evaluation. Once parameters have been set for the evaluation and the presentation of the results in the output, they can be saved in configuration files. Almost any number of files can be read and evaluated at the same time. The data corrections (zero-point correction, thermal drift correction) and the averaging of measurement curves with the same load can be carried out manually or automatically. Averaged curves are saved in a new file so the steps do not have to be repeated. The results appear summarized in a table and in a graphic as a function of the measuring position.



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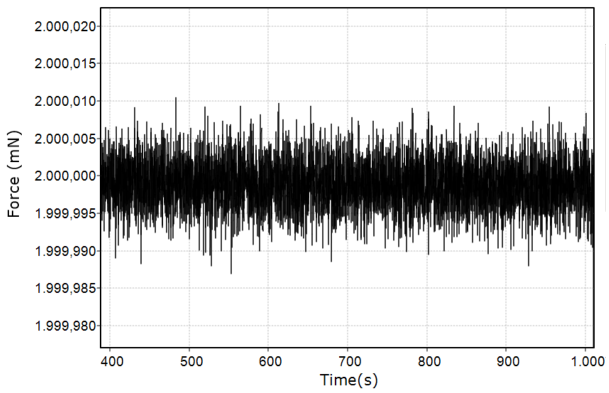
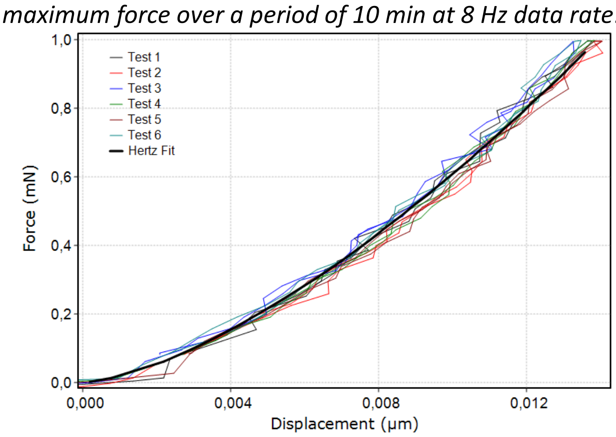
Measurement precision

The resolution of a force or displacement measurement is a purely theoretical value that is based on the number of bits of the AD converter and the measuring range. It is not suitable for comparing different devices. The noise of the measurement signals is much more important, although this depends on the environmental conditions. The ZHN-S has an extremely high signal-to-noise ratio of six orders of magnitude, allowing measurements over four orders of magnitude of force.

In example (1), the force was maintained constant at the maximum force of 2000 mN over a period of 10 min and at a data rate of 8 Hz. The mean is 1999.999 mN and the standard deviation is 3 µN. Example (2) compares six purely elastic measurements in fused silica with a ball indenter of a radius of 36.6 µm at a maximum force of 1 mN and a data rate of 8 Hz. The depth difference at a maximum penetration depth of 13.7 nm is only 0.6 nm despite different measuring positions. For comparison, the fit curve according to the Hertzian contact model is shown for this radius.

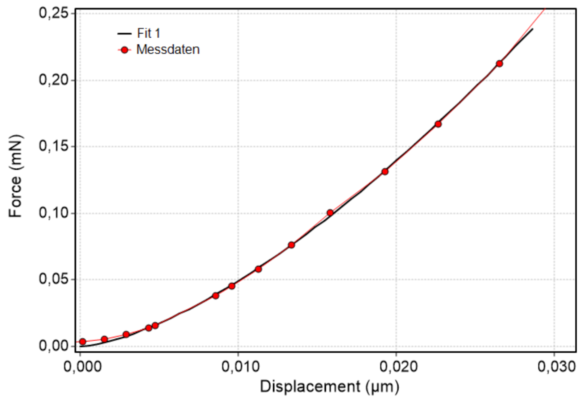


Example 1: Noise and stability of the force signal at maximum force over a period of 10 min at 8 Hz data rate.

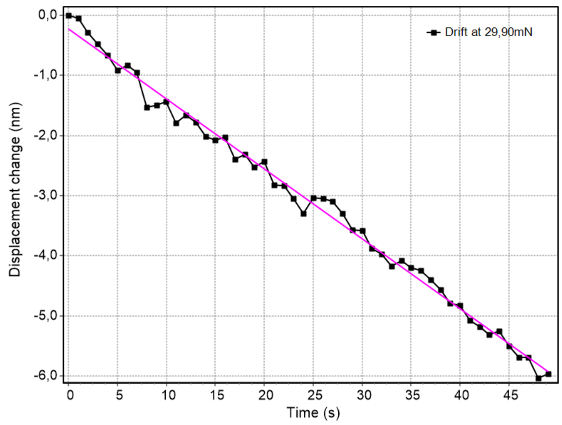


Example 2: Comparison of six purely elastic measurements on fused silica compared to a calculated curve for 36.6µm tip radius.

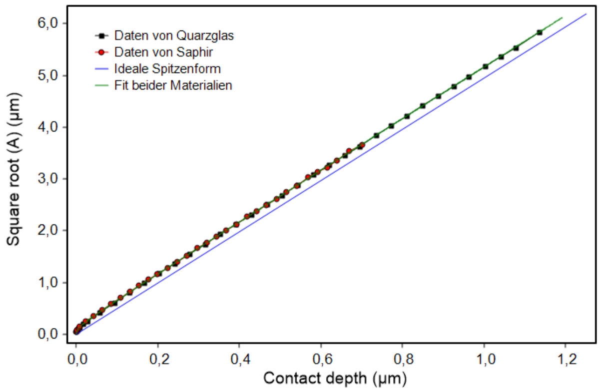
Even more important than the signal-to-noise ratio are the precision of the zero-point determination (position of the surface), of the thermal drift correction and the accuracy of the area function (shape of the indenter). The *InspectorX* software has particularly precise routines, the quality of which has been proven, for example in comparative measurements with the Physikalisch-Technische Bundesanstalt (PTB) or in various interlaboratory tests.



Example 3: Zero-point correction with extrapolation method using the data from the first 30nm and the approach segment.



Example 4: Determination of thermal drift with linear fit with a path change of 6nm over 50 s.



Example 5: Area function of a Berkovich indenter with two reference materials (maximum force 300 mN).

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Applications

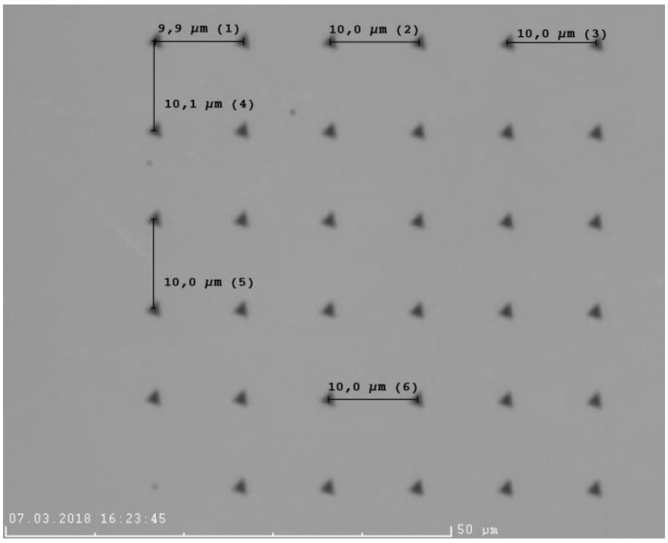
Measurement of hardness and modulus according to DIN ISO 14577

The measurements are usually carried out with a Berkovich indenter under force control. A typical measurement takes about 20 s for the measurement itself and about 20 s for the approach to detect the surface carefully. However, very fast measurements lasting just 2 s are also possible.

Measurable quantities according to the standard:

- Indentation hardness H_{IT} (convertible to HV)
- Martens hardness HM or HMs
- Indentation modulus E_{IT} (Young's modulus)
- Indentation creep C_{IT} or relaxation R_{IT}
- Ratio of elastic deformation to indentation energy η_{IT}

A total of more than 60 values can be determined.



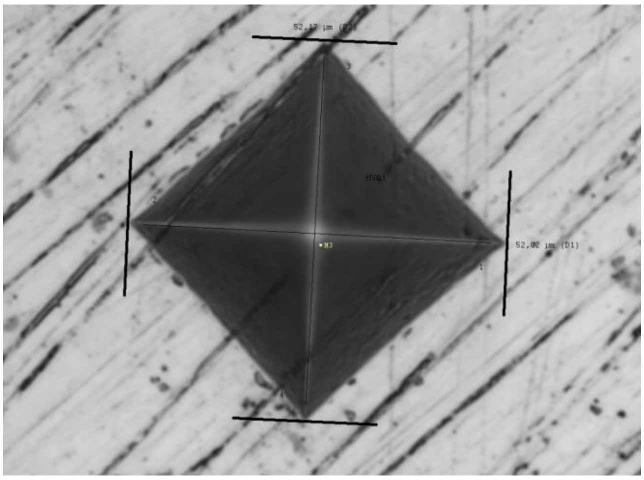
Grid of measurements in fused silica with a maximum force of 25 mN and a regular spacing of 10 µm at the highest optical resolution.

Vickers hardness

The Vickers hardness can be calculated from the indentation hardness according to ISO 14577. An extensive comparison by the Federal Institute for Materials Research and Testing (BAM) with 20 materials between the conventional Vickers hardness and the Vickers hardness calculated using *InspectorX* algorithms and converted from H_{IT} showed an average difference of less than 10 %, in contrast to 25 % - 30 % for other software packages.

[T. Chudoba, M. Griepentrog, International Journal of Materials Research 96 (2005) 11 1242 – 1246]

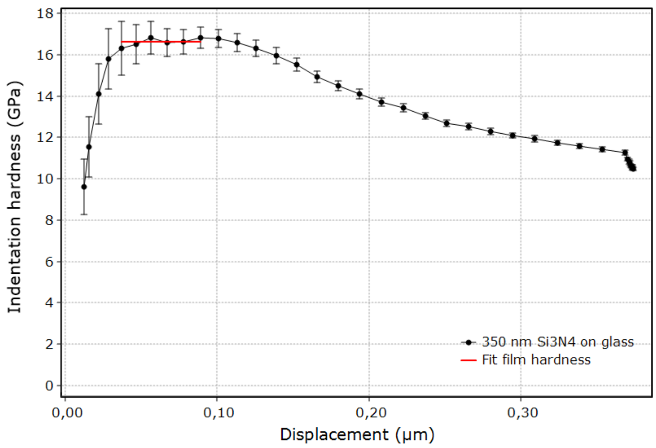
In the instrument software, the Vickers hardness can also be determined conventionally by measuring the diagonals.



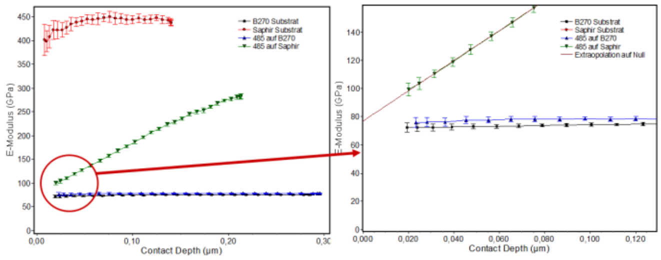
Vickers impression in a steel sample with a hardness of 672 HV1 using the 20 N measuring head

Depth profiles of hardness and modulus using the QCSM module

The **Q**uasi **C**ontinuous **S**tiffness **M**easurement method is a dynamic method that makes it possible to determine depth resolved hardness and modulus functions at one and the same measuring position. It is particularly suitable for coatings to determine and eliminate the influence of the substrate. In addition, the sensitivity of the measurement is increased so that precise values can be determined even for very low forces and penetration depths. With the QCSM module, the load increase is stopped for a short time (0.5 - 3 s) and a sinusoidal oscillation is superimposed on the static force. The amplitude and phase of the vibrations are measured using a lock-in filter and the local contact stiffness is determined, which in turn can be used to calculate the hardness and modulus



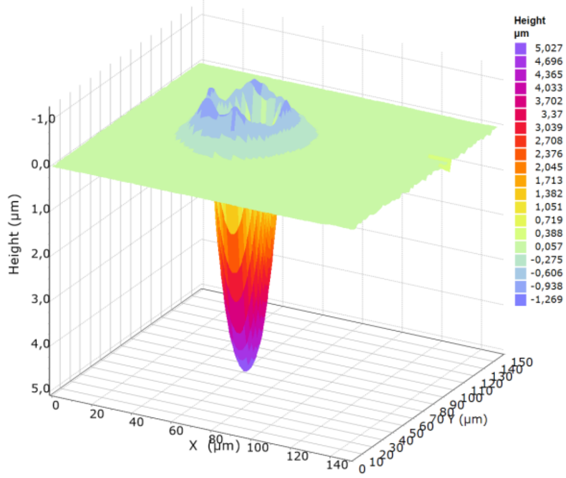
Hardness curve of a 350 nm thick Si_3N_4 coating on glass



Modulus curves for 260 nm thin oxide layers on sapphire and glass substrates, measured with a maximum force of 18 mN. Only after extrapolation to zero depth the same modulus can be obtained for the layer on different substrates.

Measurement of surface profiles

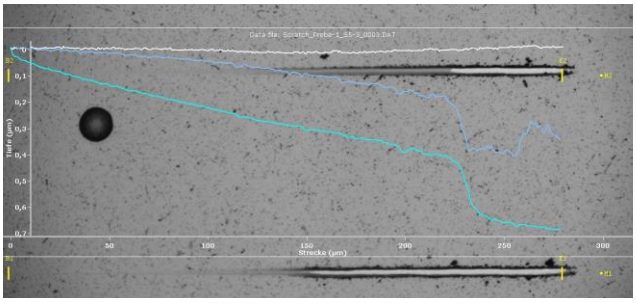
Measurements of surface profiles can be carried out with the XY stages with 50 nm resolution. Roughness values such as Ra, Rq or Rt are measured. Line or area scans are possible.



Area scan of an impression with a 30 µm radius ball in steel, scanned with a 5 µm radius indenter to determine the pile-up behavior.

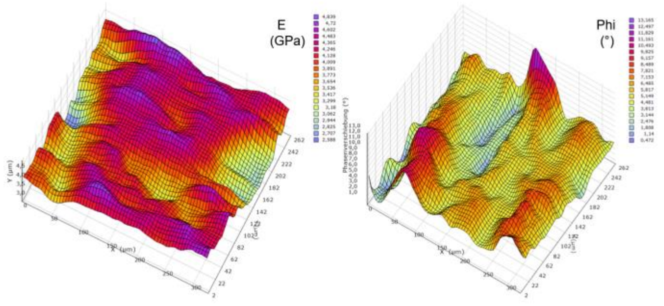
Micro scratch tests

The tests are usually carried out with spherical tips between 5 and 10 µm radius. This means that the stress maximum is typically in the layer and not in the substrate. With the help of a pre- and a post-scan of the surface, a distinction can be made between elastic and plastic deformation.



Overlay of the graphic from the scratch test of a 300 nm thick coating on hard metal with a maximum force of 300 mN with the camera image of the corresponding (upper) scratch track. The coating's point of failure is at the position of the depth step.

Mapping of modulus and phase shift by a scan with oscillating tip



Determination of the elastic modulus distribution and the phase shift on a mouse bone with a ruby ball of 500 µm diameter, oscillating at 28 Hz

Additional applications

- Determination of the yield point of brittle materials from tests with a spherical indenter
- Purely elastic measurements with spherical indenter to determine the Young's modulus of very thin and hard coatings with thicknesses less than 100 nm
- Mapping of mechanical properties with high point density within a specific surface area
- Fatigue measurements with cycle numbers up to one million
- Long creep tests, even at constant pressure (instead of constant force)
- Measurement of storage and loss modulus
- Micro tensile tests
- Push-out tests of fibers in a matrix
- Oscillatory wear tests

Typical areas of use (examples)

- Coating development from soft (polymer) to hard (diamond-type coatings)
- Determination of critical stresses for cracking or plastic deformation
- Development and testing of hard coatings for tools and surfaces and for scratch protection
- Protective coatings on glass
- Paints and sol-gel coatings
- Automated measurement of hardness profiles on cross-sections
- Coatings for sensors and MEMS/NEMS
- Biological materials
- Matrix effects in alloys
- Ceramic materials and composites
- Ion-implanted surfaces
- Damage analysis in microelectronics

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ADVANCED SURFACE MECHANICS

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Technical data

Type Item number	ZHN-S Nanoindenter 10.02-01		
Dimensions (W x H x D)	540 x 634 x 358 mm (without anti-vibration system)		
Weight	ca. 65 kg		
Voltage supply	230 V		
Cabin dimensions (W x H x D)	800 x 932 x 730 mm		
Measuring heads **Item number**	**0,2 N** **10.07-01**	**2 N** **10.05-01**	**20 N** **10.06-01**
Test load maximum	± 0,2 N	± 2 N	± 20 N
Test load minimum	± 0,05 mN	± 0,2 mN	± 2 mN
Digital resolution force measurement	≤ 0,002 µN	≤ 0,02 µN	≤ 0,2 µN
Background noise force measurement (1σ @ 8 Hz)	≤ 0,2 µN	≤ 2 µN	≤ 20 µN
Maximum displacement	± 200 µm	± 200 µm	± 200 µm
Digital resolution displacement measurement	≤ 0,002 nm	≤ 0,002 nm	≤ 0,002 nm
Background noise depth measurement (1σ @ 8 Hz)	≤ 0,3 nm	≤ 0,3 nm	≤ 0,4 nm
Background noise depth meas. (closed loop, 1.4 s)	≤ 0,15 nm	≤ 0,15 nm	≤ 0,15 nm
Dynamic module (1			
Maximum oscillation frequency	300 Hz	300 Hz	300 Hz
Max. frequency for stiffness evaluation	25 Hz	60 Hz	75 Hz
Intern acquisition rate	> 40 kHz	> 40 kHz	> 40 kHz
Maximum force amplitude of oscillation	10 mN	110 mN	600 mN
Maximum displacement amplitude of oscillation (in air)	10 µm	25 µm	25 µm
Optics			
Camera	Color, 5536 x 3692 Pixel (20 MP); USB 3.0		
Objective	20 x NA 0.4		
Working distance	8,5 mm		
Illumination	white LED		
Optical magnification at 24“ screen; Zoom min - max	574 x – 2300 x		
Image field	615 µm x 440 µm (max.) – 154 µm x 123 µm (min.)		
Pixel resolution	481 nm (max.) – 120 nm (min.)		
Stage system			
X-stage travel distance	60 mm	step size 50 nm	
Y-stage travel distance	100 mm	step size 50 nm	
Z-stage travel distance	55 mm	step size <10 nm	
Maximum sample size (measuring area) (X x Y x Z)	60 x 40 x 50 mm		
Maximum scratch length (2	50 mm		

(1 Separate module, not in basic configuration
(2 Depending on flatness of sample surface and horizontal alignment

Measurements with the ZHN-S fulfill the following standards: ISO 14577, ISO 6507, ISO 19278, ASTM E2546, ASTM E384, ASTM B578