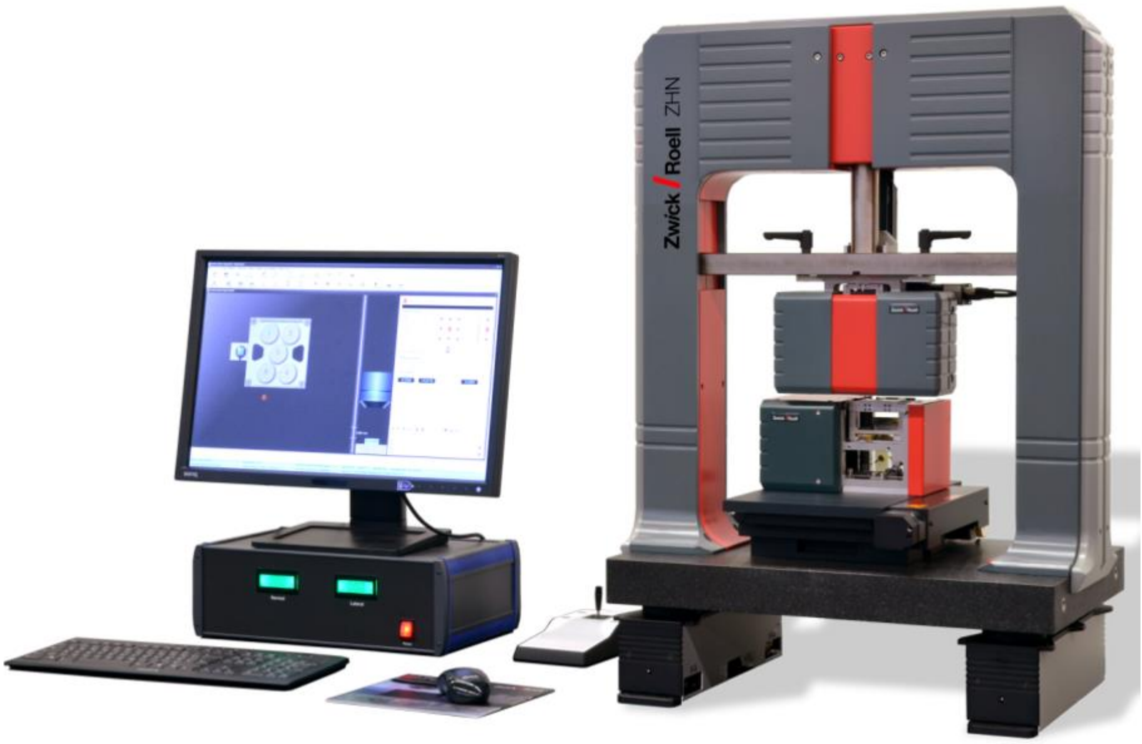
Product information ZHN

Multi-functional Nanoindenter with high modularity



ZHN Nanoindenter with Lateral Force Unit (LFU)

Scope of application

The ZHN is a nano- and micromechanical testing system with very high modularity. A unique feature is the option for the extension with a lateral force unit (LFU), which is a separate second measuring head with an actuator and two sensors. It enables the generation of lateral forces without movement between tip and sample, e.g. for studying static friction. Alternatively, a simple friction force measurement is also available (without an actuator). The large installation space allows additional modules to be accommodated or particularly large samples to be measured. Expansion options include, for example, a sample heater up to 400°C, an atomic force microscope or an optical profilometer, which uses the device's own optics.

With only one measuring head (normal force unit), indentation hardness and indentation modulus can be measured in according to DIN ISO 14577 (Determination of hardness and other material parameters for 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 changed 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 modulus of elasticity, fatigue tests or measurements of viscoelastic properties.

The instrument can be used (amongst others) as:

- Nanoindenter
- Conventional hardness tester
- Scratch tester
- Profilometer
- Wear tester
- Fatigue tester
- Micro-Tensile tester
- Tester for shear strength

The required configuration for the respective use case will be suggested after a consultation.

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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.
- Changing the indenter is quick and easy possible without additional protective measures or recalibration. Stored indenter calibration data is available on demand.
- Several optics variants are available. The highest-resolution optics uses two cameras for magnifications of up to 3350x. Instead of changing lenses, the cameras are simply switched, which leads to greater positioning accuracy.
- 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.

Lateral Force Unit

- This measuring head has the same digital force and displacement resolution as the measuring head for vertical (normal) loads. The background noise of the displacement signal is better than 1 nm.
- The two directions of movement (normal, lateral) are completely decoupled and do not influence each other. There is no roll motion due to bending of the shaft with the tip on the surface.
- Lateral forces can be generated and measured without moving the sample with the stage system.
- A measurement of the lateral contact stiffness is possible.

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 up to four sensors and up to two actuators
- Three interchangeable measuring heads for measurements normal to the surface
- Two optional measuring heads for the measurement of friction force (and more), not interchangeable
- Control and analysis software *InspectorX*
- Autofocus function
- Dynamic module for tip oscillations up to 300 Hz (QCSM / CSM) optional
- Extendable with (besides others):
  - Special sample holders adapted to component geometries
  - Sample heater up to 400°C
  - Atomic force microscope
  - Optical profilometer (interferometer)

All 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. With a shaft extension, measurements can be carried out in liquids or 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 analysis options are available in the software.

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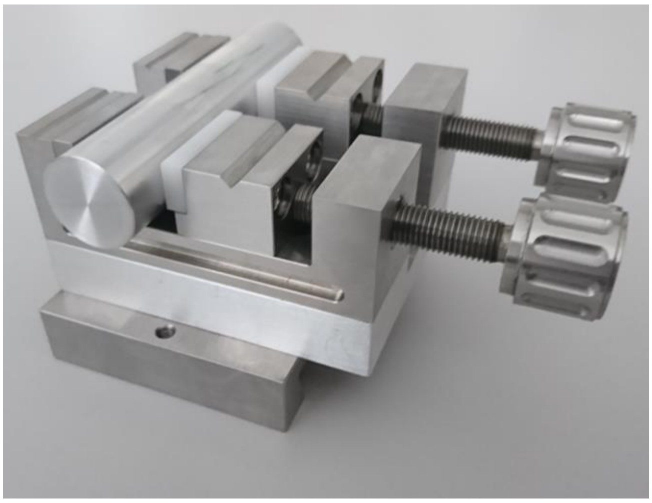
Default sample holder of the ZHN for five samples. It has an isolated surface and a connector for resistance measurements between (conductive) tip and sample



Bench vise sample holder for samples up to max. 40 mm width. More types of sample holders are available.

Tool for changing tips without additional protective measures

Recommended active vibration isolation unit for the ZHN

Recommended cabin for the ZHN

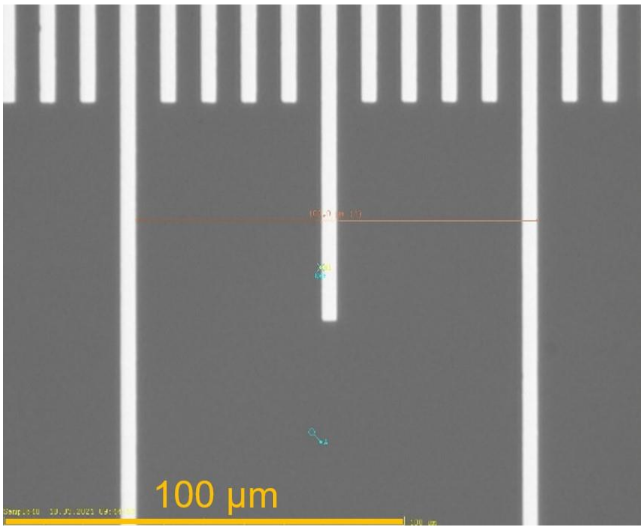


Image of an optical grating with 10 µm bar spacing

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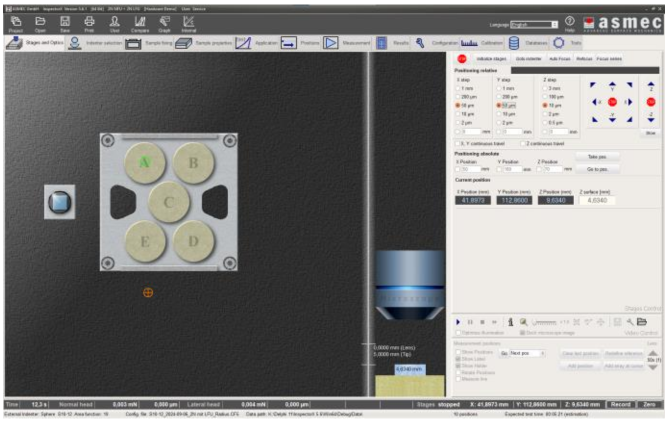
The user interface

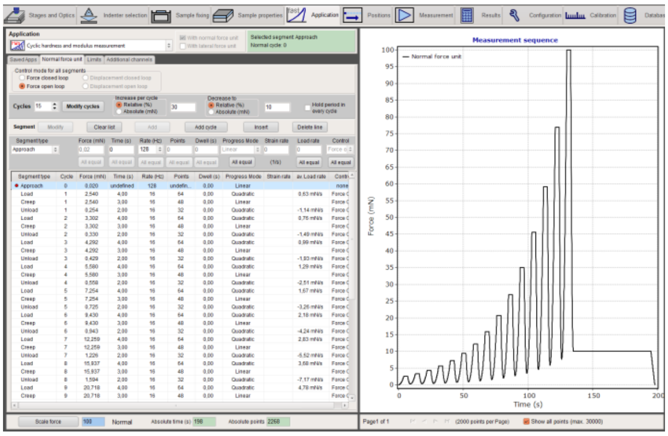
Control of the precision tables

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.



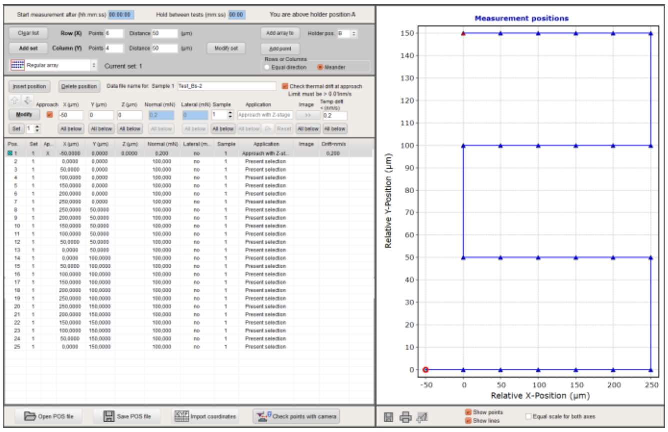
Definition of the measurement sequence

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.



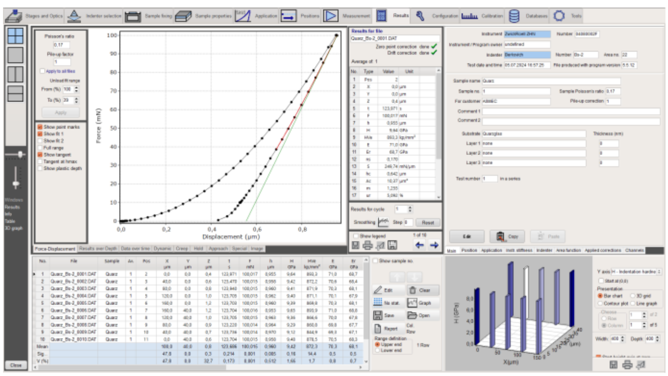
Definition of the measurement positions

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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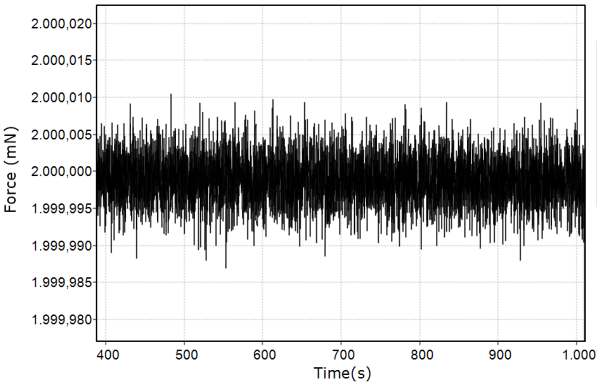
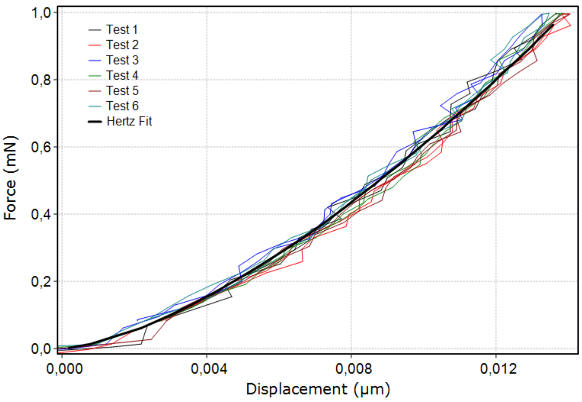
Measurement precision

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.

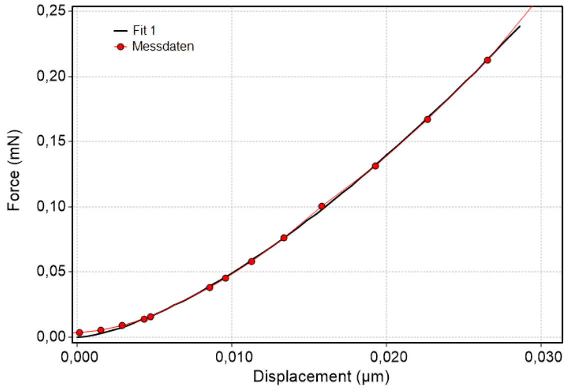


Example 1: Noise and stability of the force signal at maximum force over a period of 10 min at 8 Hz data rate.

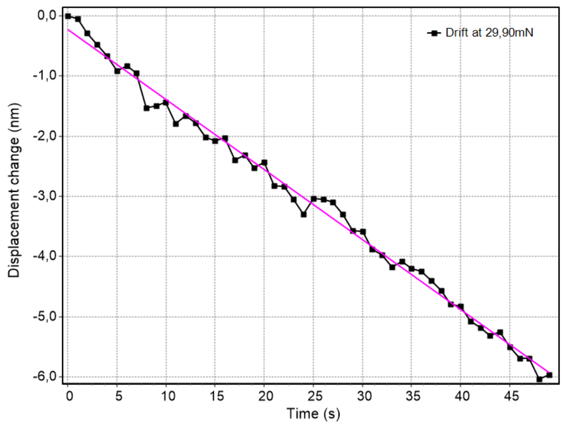


Example 2: Comparison of six purely elastic measurements on fused silica compared to a calculated curve for 36.6µm tip radius.

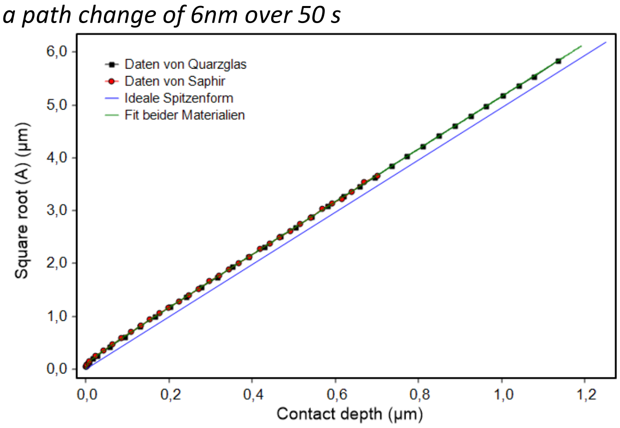
Even more important than the signal-to-noise ratio are the precision of the zero-point determination (position of the surface), of the thermal drift correction and the accuracy of the area function (shape of the indenter). The *InspectorX* software has particularly precise routines, the quality of which 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

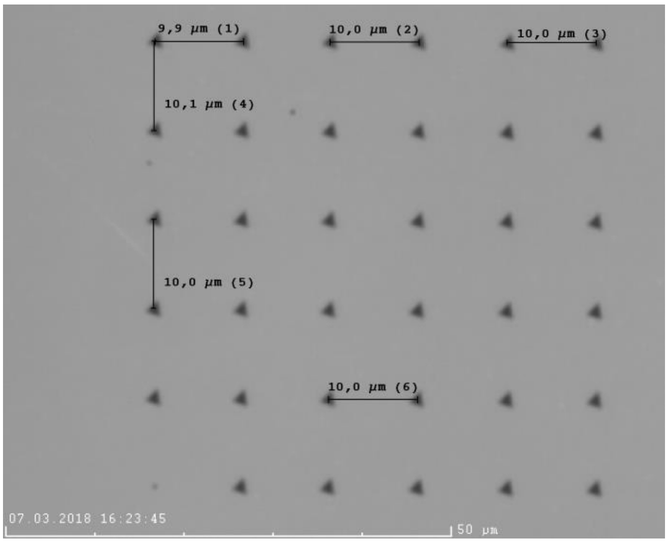
Measurement of hardness and modulus according to DIN ISO 14577

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.



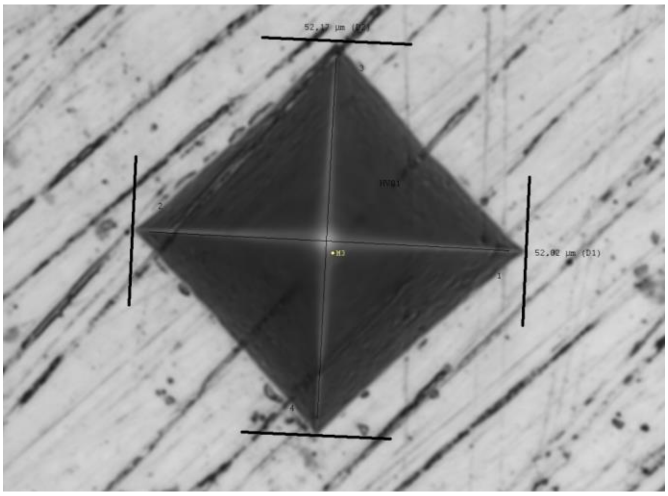
Grid of measurements in fused silica with a maximum force of 25 mN and a regular spacing of 10 µm at the highest optical resolution.

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]

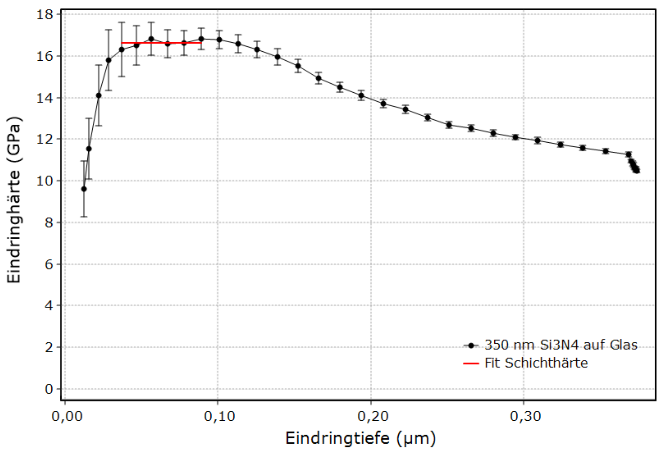
In the instrument software, the Vickers hardness can also be determined conventionally by measuring the diagonals.



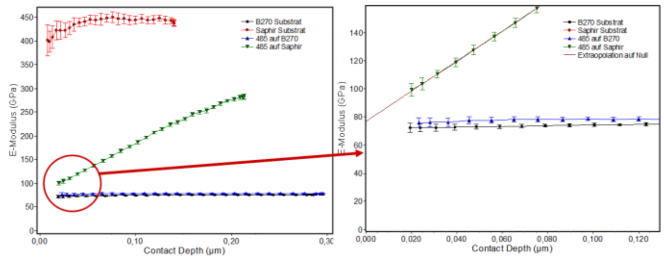
Vickers impression in a steel sample with a hardness of 672 HV1 using the 20 N measuring head

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



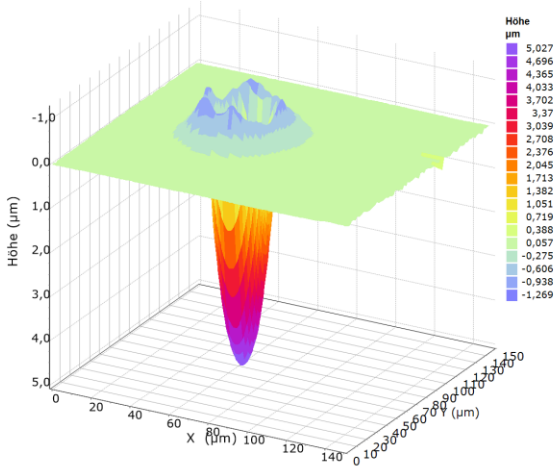
Hardness curve of a 350 nm thick Si_3N_4 coating on glass



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.

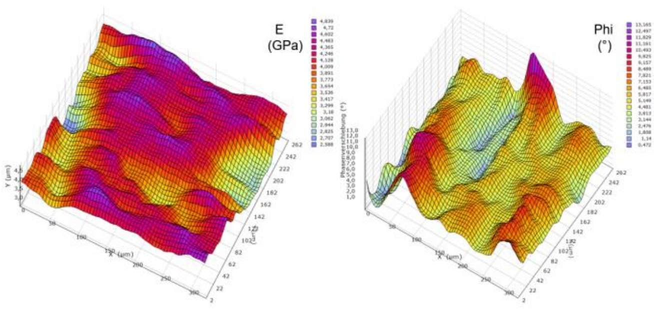
Measurement of surface profiles

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.

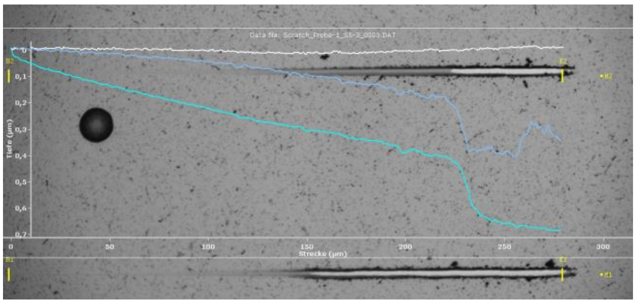
Mapping of modulus and phase shift by a scan with oscillating tip



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

Micro scratch tests

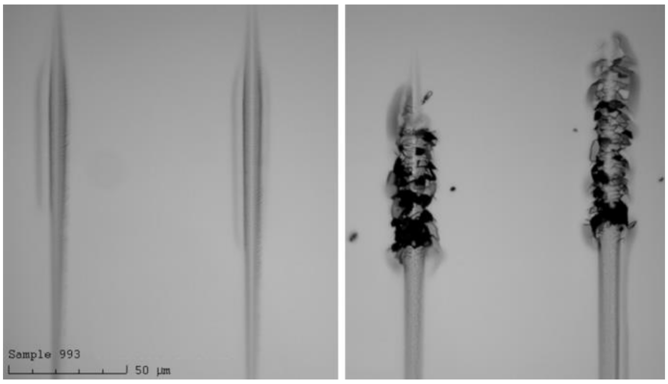
The tests are usually carried out with spherical tips between 5 and 10 µm radius. This means that the stress maximum is typically in the layer and not in the substrate. With the help of a pre- and a post-scan of the surface, a distinction can be made between elastic and plastic deformation.



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.

Oscillatory scratch tests

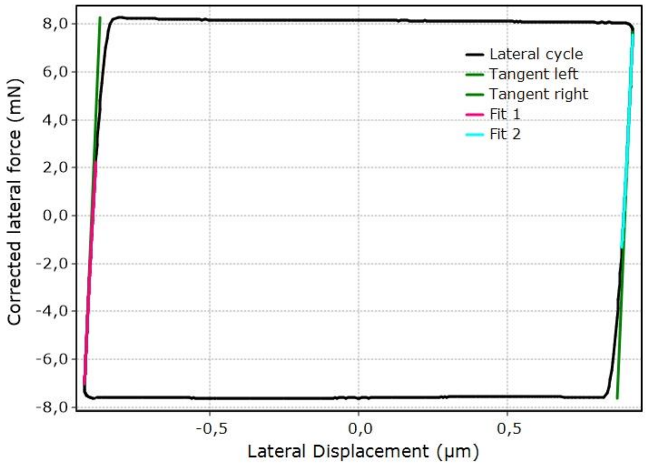
In these tests, the scratch is performed in the Y-direction using the mechanical stage. The sample oscillates vertically (in X-direction) with the help of the LFU (typically at 20 Hz) and generates an additional force component that makes it possible to remove layers that would otherwise only deform plastically and are difficult to remove from the substrate.



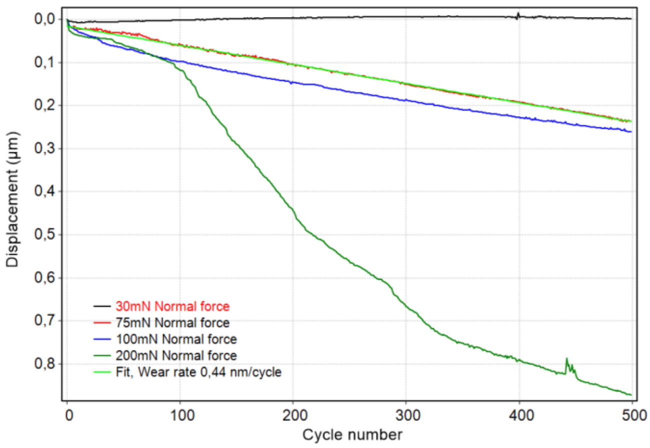
Scratch tracks in an approximately 300 nm thin Mo layer on glass. Without oscillation, the layer does not come off (left picture), whereas with oscillation the layer can be removed reproducibly.

Lateral contact stiffness and friction measurement

With the LFU, a sample can be moved laterally very precisely during contact with a spherical tip and the required forces can be measured with high resolution. The displacement can be a few 10 nm or up to over 100 µm. A cycle consists of a back and forth movement. The slope of the lateral force-displacement curve at the turning points corresponds to the lateral contact stiffness. The Poisson's ratio can be estimated from the ratio of lateral and normal contact stiffness and the coefficient of sticking and sliding friction can be determined from the ratio of lateral and normal force at certain positions. Elastic measurements are necessary to determine the lateral stiffness, while the coefficients of friction also characterize plastic deformation and surface erosion. When averaging across the width of the friction track, the depth resolution is so good that the removal of individual atomic layers can be observed.



Lateral force-displacement curve with an amplitude of about 1µm. At the turning points on the left and right a fit is carried out like for the unloading curve in a hardness measurement and the tangents indicate the lateral contact stiffness.



Average wear depth per cycle for oscillatory wear measurements with different normal forces and with an about 5 µm radius diamond ball on DLC (wear length 80 µm). At 30 mN there is no wear while at 75 mN 0.22 nm of material is removed per movement at a constant rate during 500 cycles (0.44 nm for forward + backward movement)

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 of viscous materials
- Oscillatory wear tests
- Micro tensile tests
- Push-out tests of fibers in a matrix
- Shear 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

Technical data

Type Item number	ZHN Nanoindenter 10.01-01
Dimensions (W x H x D)	640 x 790 x 390 mm (without anti-vibration system)
Weight	ca. 105 kg
Voltage supply	230 V
Cabin dimensions (W x H x D)	932 x 1094 x 730 mm

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Measuring heads - Normal force unit (NFU) 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
Measuring heads - Lateral force unit (LFU) Item number	**2 N LFU 10.08-01**	**2 N simple LFU 10.09-01**	
Maximum force	± 2 N	± 2 N	
Background noise force measurement (1σ @ 8 Hz)	≤ 10 µN	≤ 140 µN	
Maximum displacement	± 70 µm	---	
Background noise depth measurement (1σ @ 8 Hz)	≤ 0,5 nm	---	
Actuator	with	without	
Dynamic module [1]	**30.01-01**	**30.01-01**	**30.01-01**
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
Optik	**11.03-01**		
Tandem microscope with two cameras	Black/white, 1280 x 1024 Pixel (1,3 MP); USB 3.0		
Objective	50 x NA 0.65	additional 5x as option	
Working distance	8 mm	10 mm	
Illumination	green LED		
Optical magnification at 24“ screen; camera 1 / 2	1000 x / 3350 x	100 x / 335 x	
Image field	324 x 259 µm / 96 x 77 µm	3,2 x 2,6 mm / 0,97 x 0,77 mm	
Pixel resolution	254 nm / 76 nm	2540 nm / 760 nm	
Stage system			
X-stage travel distance	100 mm	step size 50 nm	
Y-stage travel distance	200 mm	step size 50 nm	
Z-stage travel distance	75 mm	step size <10 nm	
Maximum sample size (measuring area) (X x Y x Z)	80 x 80 x 70 mm		
Maximum scratch length [2]	50 mm		

[1] Separate module, not in basic configuration
[2] Depending on flatness and 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