Product Information

ZHN-S – Robust Nanoindenter for Quality Control and Standard Test Tasks in Industry and Research Applications



ZHN-S nanoindenter for standard test tasks in industry and research

Applications

CTA: 267016 267017

The universal nanomechanical tester is designed for the determination of hardness and Young's Modulus on materials and coating systems. The nano and micro ranges are compliant to the standard EN ISO 14577 (instrumented indentation method for determining hardness and other material parameters of metallic materials and coatings). The nanomechanical tester can also perform cyclic indentation tests and indentation tests with superimposed oscillation.

Due to its high modularity, it is more than a nanoindenter or hardness tester. When used with a measuring head (NFU) it serves as:

- Nanoindenter/hardness tester, depending on the measuring head used, for measurements between 0.05 mN to 20000 mN
- Micro compression testing instrument in the same force range
- Fatigue tester up to 2 Hz quasistatic or up to 300 Hz with dynamic module
- Dynamic mechanical tester (DMA) up to approximately 100 Hz with dynamic module
- For surface profile measurement and with dynamic module, also for stiffness/modulus mapping
- Scratch and wear tester without coefficient of friction measurement
- for fast mapping of hardness and Young's modulus up to 10 measurements per minute



ZHN-S Nanoindenter; detail view

Advantages and features

- Animations and predefined applications in InspectorX support the operator during test preparation and test performance. This reduces operating errors and guarantees/ensures short training periods.
- Trained professionals, rather than only science and research employees, can operate the instrument and evaluate results, which reduces the overall cost of testing.
- Easiest and most robust tip change when compared with any other instrument on the market, without recalibration: The calibration data is clearly assigned to the measuring tip.
- The excellent dynamics module enables particularly accurate and fast calibration of the measuring tip and also the depth-resolved measurements required for coatings.
- The optics with 18 megapixel color camera allows 4x zoom without compromising resolution. This covers a magnification range of up to three classic revolver lenses, eliminating the need to change lenses.
- Very easy evaluation of measurements on layers due to automatic fit function and range selection for the measurement data analysis.
- ZwickRoell has implemented standard compliant radial displacement correction in InspectorX according to ISO 14577:2015.
- Stiff frame design with indenter axis exactly in the movement axis (no tilting moment)
- High positioning accuracy between optics and indenter, better than 1µm.

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CTA:

Function description

Modular assembly, consisting of

- 2-column load frame with central lead-screw drive, precision guidance and granite base
- Programmable motorized X-Y table
- 3-axis step motor control as PCIe slot card
- Microscope with LED incident lighting, white
- Control electronics for machine and measuring head
- Interchangeable measuring heads
- InspectorX control and evaluation software
- Software modules for auto focus function
- Dynamic module for tip vibrations up to 300 Hz (QCSM / CSM), optional
- Passive or active vibration damping, optional

With the two-column load frame with central lead-screw drive and the indenter axis located exactly in the axis of motion, there is no tilting moment during load application and the instrument stiffness is very high.

Unlike instruments from other manufacturers, the two measuring heads work in both the tensile and compression direction with the same measurement range. The instrument can operate with both force and displacement control in open-loop mode (only maximum force/displacement is controlled) or closedloop mode (each individual measuring point is controlled). The maximum data rate is 4,000 points per second, which also allows for very fast measurements.

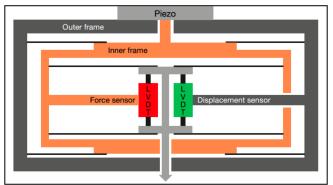
The dynamic module generates sinusoidal measuring tip vibrations, enabling continuous stiffness determination, fatigue testing and the measurement of viscous material properties. The robust design of the measuring head allows the use of basically any customized counterbody. With a shaft extension, measurements can also be performed in liquid media.

Sophisticated software allows fast and flexible programming of the measurement sequence and positions. The positions can be easily configured in the camera image via the point-and-click feature. In addition, a large number of unique evaluation options are available in the software modules.

Patented measuring head (2N and 20N)

- Movement in the normal direction and high stiffness in the lateral direction thanks to the double leaf-spring system
- Robust construction

- No inductive sensor stop in the event of an overload and thus no damage
- The shaft can bear heavier weights without leaving the measurement range Any type of customer-specific measuring tip can be easily used



Principle of normal force unit (NFU)

Optics

- 20x lens in combination with a high-resolution 18 megapixel color camera allows 4x zoom without compromising resolution
- Within the optical image, it is possible to:
 - Define measuring points
 - Measure distances and perimeters
 - Review and display existing measuring points at the push of a button
 - Control lighting and image parameters
 - Show scales and recording times
- High-quality imaging is possible even for low-reflection surfaces such as glass
- Auto focus function establishes the correct height for a sharp image
- Automatic generation of images of measuring points (programmable)
- Overview image composed of individual images with large depth of field



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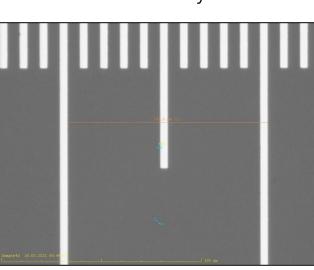


Image of an optical grid with 10 µm bar spacing

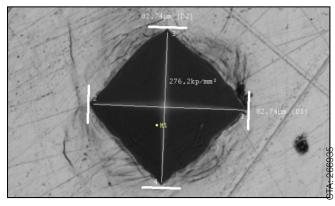




Berkovich indentations with 500 mN in steel and specimen grip position superimposed

CTA: 267023

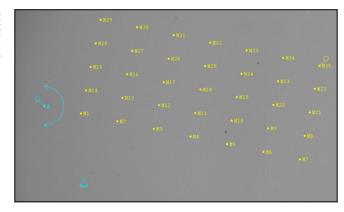
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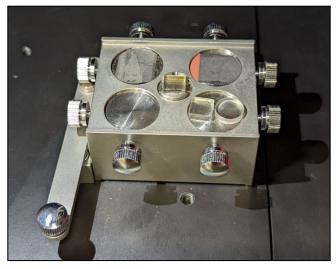
Vickers indentation in steel with measuring lines



Tool for changing the tip without additional safety measures



Point fields or lines can be rotated around an approach point by dragging them with the mouse



Specimen grip with magnetic holder for four specimens and a permanent tip-check specimen in the center



Specimen grip with adapter plate and a usable surface of 50 x 40 mm



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The user interface

Control of the precision tables

The instrument is designed for fully automated measurement series with up to 10,000 measuring positions. The corresponding control software InspectorX provides a full overview of the current position of the three precision tables and enables control with 50 nm step sizes. If the specimen is located under the lens, instead of the positions of the tables, an image of the specimen surface is displayed in the same window.

Definition of the measurement sequence

You have access to a wide range of predefined applications, which can be selected from a pull-down menu. Every sequence (test cycle) can be flexibly programmed with any number of load cycles required. When in open loop mode, the force or displacement, the time of a segment and the data rate can be preset; in closed loop mode the number of data points and the hold time per point can also be preset.

Definition of the measurement positions

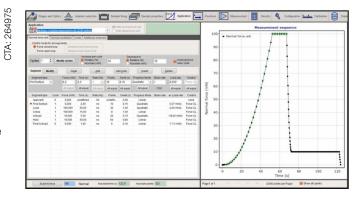
Definition or use Up to 10,000 positions can be programmed in the form of lines, uniform grids or any desired arrangement. CTA: can be generated automatically before and after the measurement using the auto focus function. Extensive specimen information can be assigned to the individual positions, which is also stored in the data file.

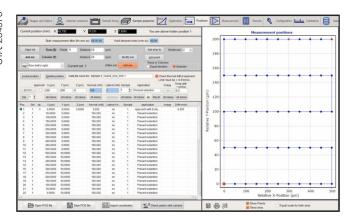
Evaluation of measurement data

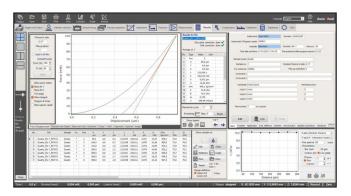
Measurement data can be displayed graphically, compared, averaged or exported in various forms (ASCII, Excel, BMP, etc.). A wide range of flexible correction routines are available for data evaluation. Once defined, parameters for the evaluation and display of the results can be saved in configuration files.

26497 Almost any amount of data can be entered and evaluated simultaneously. Data corrections (zero point correction, thermal drift correction) and the averaging of measurement curves of the same load can be carried out manually or automatically. Averaged curves are saved in a new file so that the steps do not have to be repeated. A summary of the results is displayed in a table and in a graph as a function of the measurement position.









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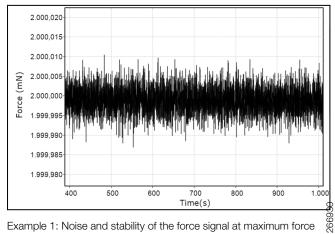
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CTA:

Precision measurement

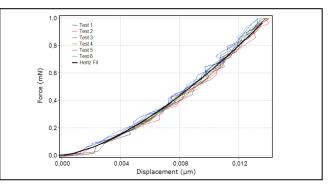
Resolutions for force or displacement measurement are a purely theoretical values based on the bit number of CTA: the A-D converter and the measurement range. They are not suitable for comparing different instruments. Considerably more important is the measurement signal noise, which can however be dependent on environmental conditions. The ZHN features an extremely high signal-to-noise ratio of six orders of magnitude, which permits measurements over four orders of magnitude of the force.

In example (1), the force was maintained constant at a maximum set force value of 2000 mN over a period of 10 minutes and at a data rate of 8 Hz. The set force is very stable. The mean value is 1999.999 mN and the standard deviation is 3 µN. Example (2) compares six purely elastic measurements in quartz glass using a spherical indenter with a 36.6 µm radius at a maximum 266924 force of 1 mN and a data rate of 8 Hz. The depth differential at a maximum indentation depth of 13.7 CTA: nm is only 0.6 nm, despite the different measuring positions. For comparison, the fit curve according to the Hertzian contact theory is shown for this radius.



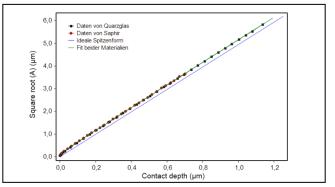
Example 1: Noise and stability of the force signal at maximum force over a duration of 10 minutes at data rates of 8 Hz

Even more significant than the signal-to-noise ratio when small indentation depths are involved, however, is the accuracy of the correction functions for the specimen shape and for the zero point (position of the surface) and thermal drifts. InspectorX software features particularly realistic routines, the quality of which was demonstrated in comparative measurements by the National Metrology Institute of Germany (PTB) and in various round-robin tests.



Example 2: Comparison of five purely elastic measurements on quartz glass in relation to a modeled curve

Example (3) shows the determination of the area function of a Berkovich indenter. The calibration method using two reference materials (most commonly quartz glass and sapphire) is unique and particularly accurate.



Example 3: Area function of a Berkovich indenter at 300 mN maximum force

Example (4) Zero-point determination using the extrapolation method Data from before positioning of the test tip (approach) is also available for the determination.

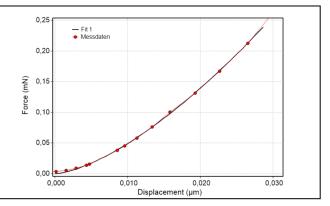


Fig. 4: Zero-point determination using the extrapolation method



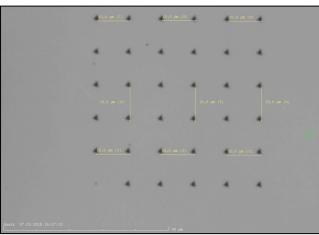
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Applications

Measuring hardness and the Young's modulus to DIN EN ISO 14577 Measurement is typically performed with a Berkovich

Measurement is typically performed with a Berkovich indenter under force control. A typical measurement takes approximately 20 seconds for the measurement itself, and approximately 30 seconds for careful approach of the tip to the surface. Very fast measurements with a duration of only 2 seconds are also possible.



Grid of measurements in quartz glass with 25 mN maximum force and regular spacing of 10 μm at high optical resolution.

Measurable values:

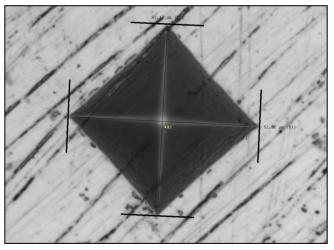
- Indentation hardness HIT (convertible to HV)
- Martens hardness HM or HMs
- Indentation modulus EIT (Young's modulus)
- Indentation creep C IT or relaxation RIT
- Ratio of elastic deformation to indentation energy n_{IT}

A total of more than 60 values can be determined.

Vickers hardness

Vickers hardness can be calculated from the indention hardness. A comprehensive study conducted by the Federal Institute for Materials Research and Testing (BAM) used 20 materials to make a comparison between the conventional Vickers hardness method and Vickers hardness calculated using InspectorX algorithms converted with $H_{\rm IT}$. It showed a mean difference of less than 10% vs. 25-30% with other software packages.

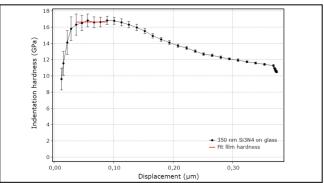
[T. Chudoba, M. Griepentrog, International Journal of Materials Research 96 (2005) 11 1242 – 1246] However, Vickers hardness can also be determined conventionally by measuring the diagonals.



Vickers indentation in a steel specimen with a hardness of 672 HV1 using the 20 N measuring head

Hardness and Young's modulus depth profiles using the QCSM module

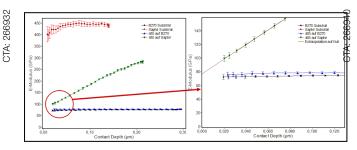
The "Quasi Continuous Stiffness Measurement" method is a dynamic measurement procedure, which makes it possible to determine hardness and Young's modulus with depth resolution at one and the same measuring position. It is ideal for coatings to detect and eliminate substrate influence. In addition, measurement sensitivity is increased, enabling the determination of accurate values for very low forces and indentation depths. With the QCSM module, the load increase is paused for a short time (0.5-3 seconds) and a sinusoidal vibration is superimposed on the static force. The amplitude and vibration phase are determined using a lock-in filter, and from that the local contact stiffness is determined, which in turn can be used to calculate the hardness and Young's modulus.



Hardness gradient for a 350 nm thick Si3N4 coating on glass

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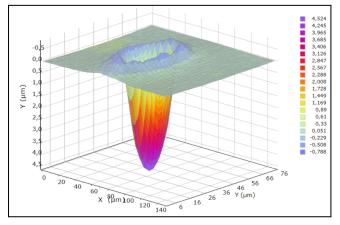
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Young's modulus gradient for 260 nm thin oxide coatings on sapphire and glass substrate, measured with a maximum force of 18 mN. Only after extrapolation to zero indentation depth do we obtain the same Young's modulus value for the same coating on different substrates.

Measurement of surface profiles

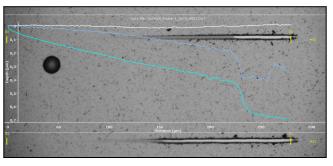
Topography scans of the surface can be performed using the X-Y tables with 50 nm resolution. Roughness values such as Ra, Rq or Rt are thereby measured. Either line scans or surface scans can be taken.



Surface scan of an indentation with a 30 μ m radius steel sphere, scanned with a 5 μ m radius sphere (scales of the two axes are not equal) to determine the pile-up behavior.

Micro scratch tests

The tests are typically performed using spherical tips with a radius between 5 and 10 µm. The stress maximum is most often in the coating and not in the substrate. By performing pre- and post-scans of the surface, a differentiation can be made between elastic and plastic deformation.



Insertion of the graphic from the scratch test of a 300 nm thick coating on hard metal with a maximum force of 300 mN into the camera image with the corresponding (upper) scratch mark. The coating's point of failure can be seen at the point where the curve bends.

Additional applications

- Determination of yield point from tests using a spherical indenter
- Purely elastic measurements with spherical indenter to determine the Young's modulus, including for very thin, hard coatings less than 50 nm thick
- Mapping 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)
- Micro tensile tests
- Push-out tests

Typical areas of use

- Coating development from soft (polymer) to hard (diamond-type coatings)
- Determination of critical stresses for cracking or plastic deformation
- Development and testing of hard material coatings for tools and as scratch protection
- Protective coatings on glass
- Paints and sol-gel coatings
- Automated measurement of hardness traverses on transverse cross-section
- Nano 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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Technical data

Туре	ZHN-S automatic			
Item No.	1100664			
Dimensions, without vibration damping				
Height	635	mm		
Width	524	mm		
Depth	358	mm		
Weight, approx.	65	kg		
Voltage	230	V		
Optics				
Camera	Color, 4912 x 3684 pixels (18	Color, 4912 x 3684 pixels (18 MP), USB 3.0 connection		
Lighting	White LED			
Lens	20 x NA 0.4 ¹⁾			
Operating distance	8.5	mm		
Optical magnification to 24" min-max	535x - 2140x			
Image field when displayed in InspectorX	617 x 462 - 161 x 129	μm		
Pixel resolution	503 - 126	nm		
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 specimen size (H x W x D)	45 x 60 x 50	mm		
Maximum length of a scratch test	50 ²⁾	mm		
Maximum length of a scratch test	50 ²⁾	mm		

1) Included in the standard scope of delivery

2) Dependent on the smoothness of the specimen surface

NFU (normal force unit) measuring head

Item No.	1050945	1016415	1016416					
Test load F _{max} , standard ¹⁾	± 20	± 2	± 0.2	Ν				
Test load, min. Fmin, standard ¹⁾	Approx. 2	Approx. 0.2	Approx. 0.05	mN				
Digital resolution, force measurement	≤ 0.2	≤ 0.02	≤ 0.002	μN				
Background noise, force measurement	$\leq 30^{2)}$	$\leq 3^{2)}$	$\leq 0.3^{2)}$	μN				
Displacement, max.	± 200 ¹⁾	± 200 ¹⁾	± 200 ¹⁾	μm				
Digital resolution, displacement measure- ment	≤ 0.002	≤ 0.002	≤ 0.002	nm				
Background noise, displacement meas- urement index 2	≤ 0.4	≤ 0.3	≤ 0.3	nm				
Background noise, displacement meas- urement (1 σ for closed-loop mode)	≤ 0.15	≤ 0.15	≤ 0.15	nm				
Dimensions								
Height	162.6	163	158	mm				
Width	232	232	200	mm				
Depth	109	109	50	mm				

Subject to change in the course of further development.

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Item No.	1050945	1016415	1016416	
Dynamic module ³⁾				
Oscillation frequency, max.	300	300	100	Hz
Frequency, max. for stiffness evaluation	90	70	30	Hz
Data acquisition rates	40	40	40	kHz
Max. force amplitude of oscillation	> 500	> 50	> 13	mN
Weight, approx.	4	4	1.5	kg

1) Compression (e.g. instrumented indentation test) and tensile (e.g. adhesion measurements on liquids)

2) All 1 σ at 8 Hz and for use of active vibration damping. With passive vibration damping, approximately 4x greater.

3) Only in conjunction with the QCSM software module