Zwick / Roell

Intelligent testing

Applications for the Instrumented Indentation Test according ISO 14577 – from Macro to Nano

testXpo 2018, October 15 - 18

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Agenda

ISO 14577 - Introduction in the standard

ZHU/zwicki: our solution for the macro range

Macro range: results & applications

ZHN: our solution for the micro & nano range

Micro & Nano range: customer applications

Structure of DIN EN ISO 14577

The Standard of instrumented indentation (DIN EN ISO 14577) contains of three parts:

Part 1:
Test procedure
Macro (F > 2 N; h > 6 µm), Micro, Nano (h < 200 nm)
Different geometries of indenters
(Vickers, Berkovich, Balls)

Part 2:
Verification and calibration of the testing machine
Direct & indirect verification

Part 3:
Calibration of reference materials
Examples

DIN EN ISO 14577-1

Part 1: Test procedure

- A: Material parameter
  HM, HM_S, H_{IT}, E_{IT}, C_{IT}, R_{IT}, indentation work
- B: Types of control
- C1: Compliance of machine
- C2: Area correction for indenter (h < 6 µm)
- D: Diamond indenter
- E: Influence of surface roughness
- F: Theoretical correlation to Vickers hardness

DIN EN ISO 14577-2

Part 2: Verification and calibration of the testing machine

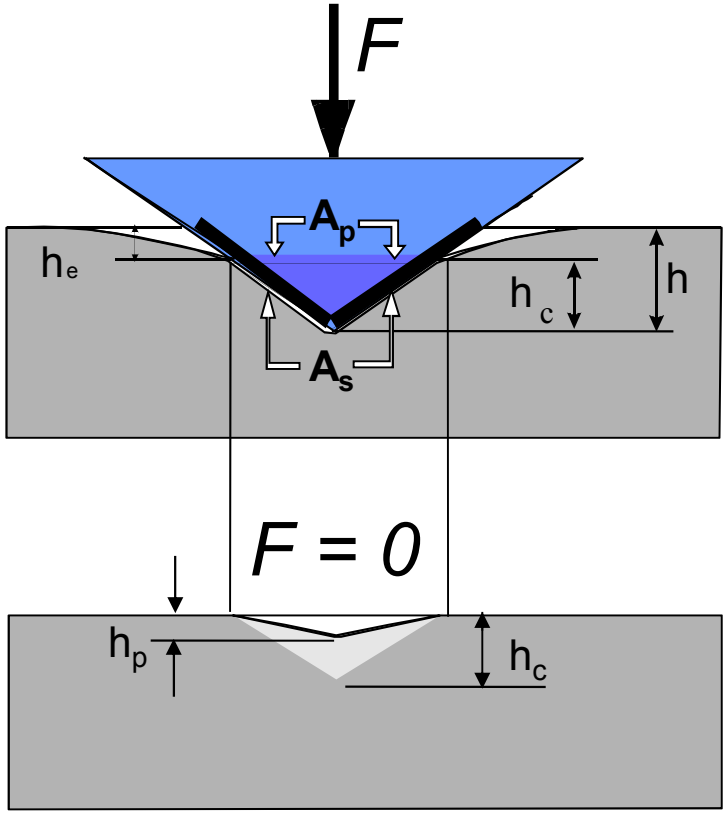
General conditions
- Preparation
- Functional installation

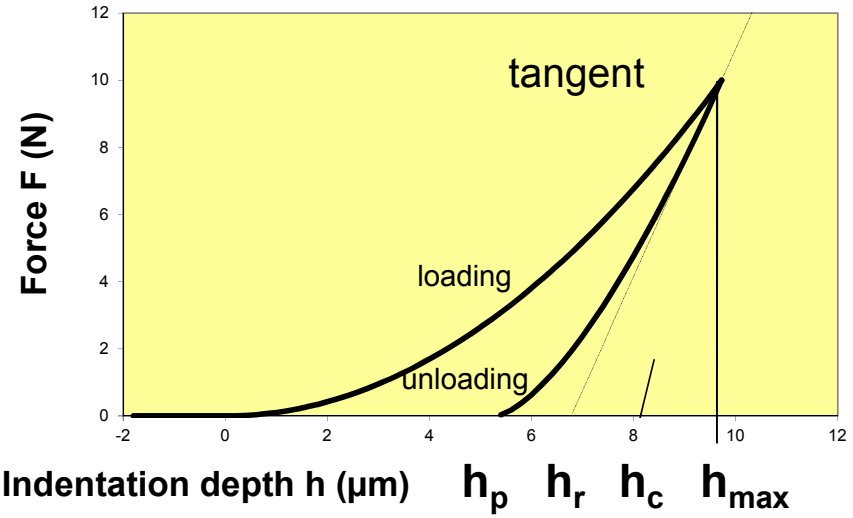
Direct testing and calibration
- Testing the test cycle
- Calibration of test load
- Check of indenter
- Calibration of the distance measuring system
- Testing the test cycle

Periodic (after 1 year) → **Indirect testing with reference samples** ← **Test blocks for hardness or indentation modulus, with 2 test loads**

Test method – instrumented indentation

$$Hardness = \frac{Force\ F}{Area\ [A_S(h)\ or\ A_P(h_c)]}$$





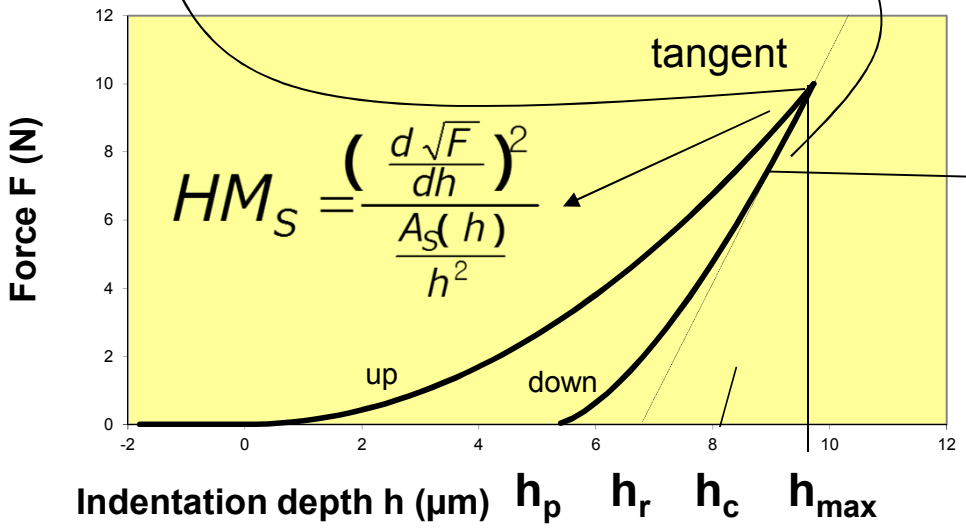
Material parameter of Martens hardness

Martens hardness

$$HM = \frac{F}{A_S(h)}$$

Indentation hardness

$$H_{IT} = \frac{F_{max}}{A_P(h_c)} \; ; \quad h_c = h_{max} - \frac{\varepsilon F_{max}}{\left(\frac{dF}{dh}\right)_{h=h_{max}}}$$



Indentation modulus

$$E_{IT} = \frac{1-\upsilon_s^{\,2}}{\frac{1}{E_r} - \frac{1-\upsilon_i^{\,2}}{E_i}}$$

$$E_r = \frac{\sqrt{\pi}}{2\sqrt{A_P(h_c)}}\left(\frac{dF}{dh}\right)_{h=h_{max}}$$

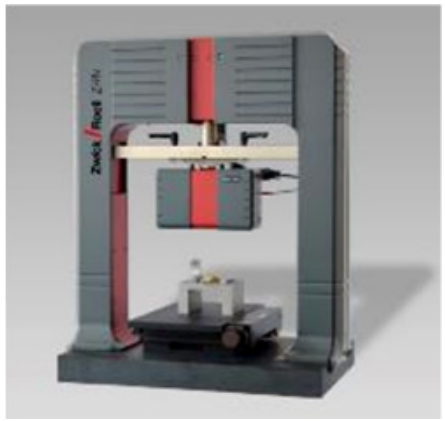
E_r - reduced Modulus

From Macro to Nano

With the ZHU/zwicki plus the ZHN Nanoindenter we can offer customers a complete portfolio from the macro to the nano range with a wide range of applications

ZHU/zwicki

- Loading: static, cyclic
- Indentation test for IIT hardness & Young's modulus
- + classical hardness testing (HR, HV, HB ...)
- Range: 2 to 2500N, greater than 6µm (depth)



ZHN

- Loading: static, cyclic, dynamic
- Indentation test for IIT hardness & Young's modulus
- + tribology, scratch tests, roughness ...
- Range: depth < 0.2 µm or force < 2 N

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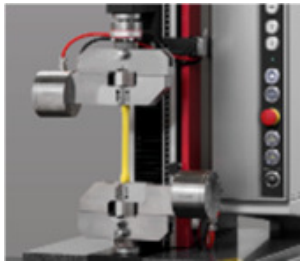
Micro & Nano range: customer applications

Hardness testers

The combination of the new zwickiLine+ with the hardness measuring head and testXpert hardness edition results in an innovative test system with a wide range of applications.

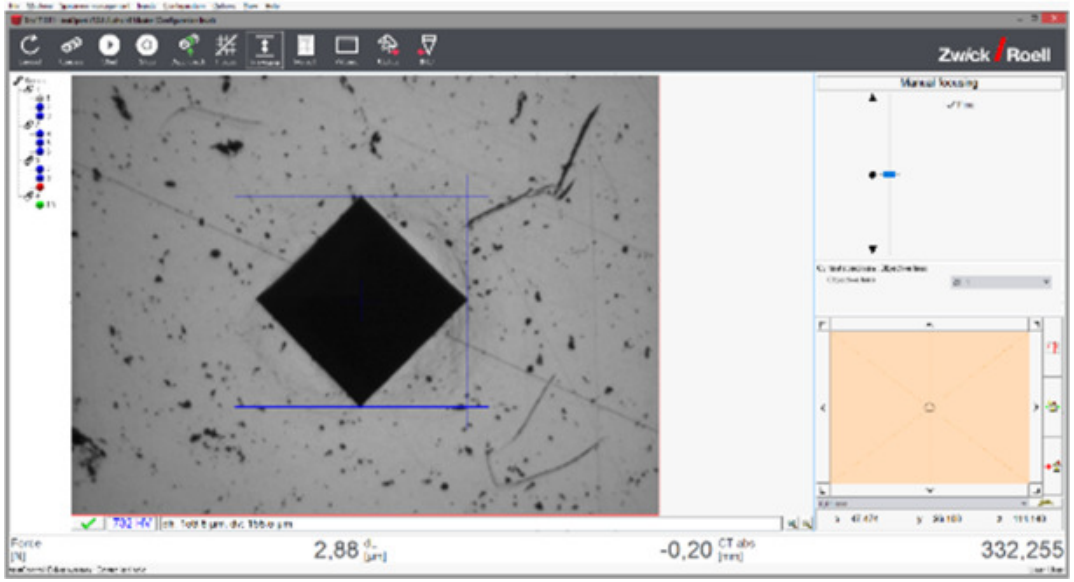
- Automatic display of force-indentation-depth curve regardless of method, for comprehensive materials characterization in instrumented indentation test to ISO 14577
- High operating comfort with changing test conditions due to **fast and precise** AC drive and large test area, e.g. for different specimen sizes (max. speed: 3000 mm/min with < 1 nm travel resolution)
- Extended application range:
  - Fully automatic Vickers and Rockwell hardness traverse tests (Jominy end-quench test, Vickers tests on ceramics)
  - Ball indentation on plastics
  - Cyclic tests on paper or ceramics
  - **Materials testing** in general (e.g. tensile/compression tests)

+

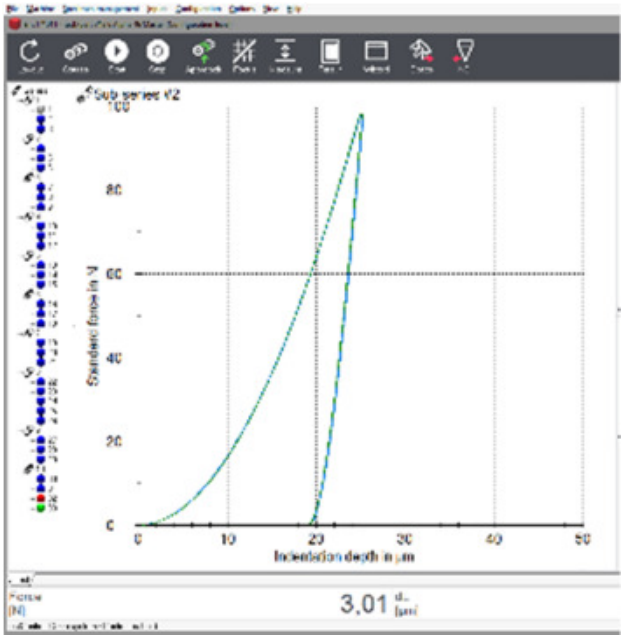


Hardness testers

The intuitive version of testXpert – hardness edition – allows deep insight in the tested material and ensures reliable test results.



testXpert®
hardness edition



- Intuitive one-button operation for starting and fully automatic evaluation of single and sequence testing
- Versatile result presentation: single and statistical values, graphics, on-screen display, and test reports can be varied as required

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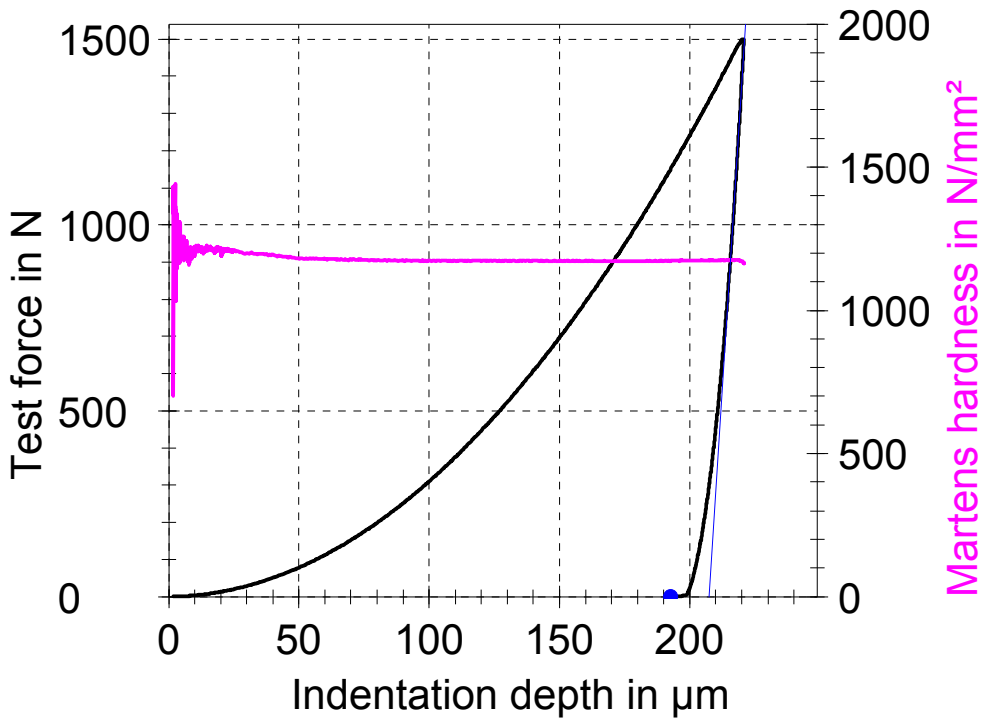
F & HM - h diag. (brass)

Force and Martens hardness - indentation depth diagram (tests on brass)



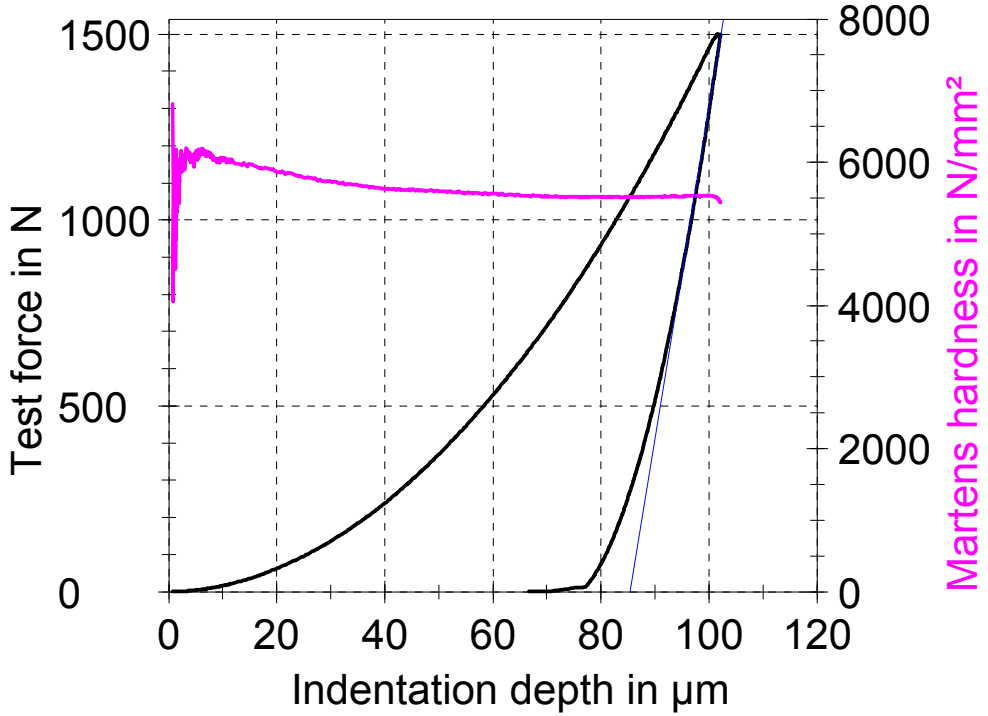
Results:

- $h_{max} \approx 220$ µm
- negigible elastic fraction of indentation work, $\eta_{it} \approx 10{,}5\%$

HM-quantity:

- little dependence of indentation depth (< 30 µm)

Force and Martens hardness - indentation depth diagram (tests on steel)



Results:

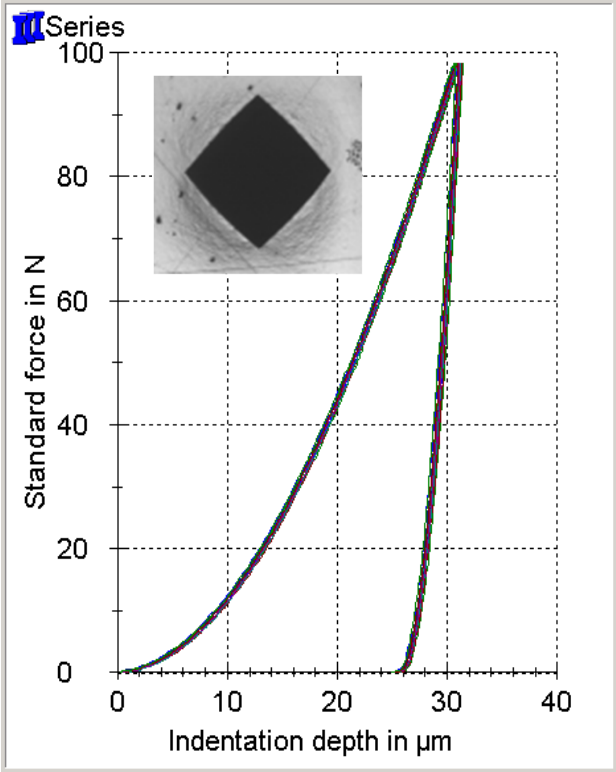
- $h_{max} \approx 100$ µm
- pronounced elastic fraction of indentation work, $\eta_{it} \approx 28\%$

HM-quantity:

- higher dependence of indentation depth (< 50 µm)

Vickers test results

Typical result performing a Vickers test with ZHU/zwickiLine (done on a HV10 test block)



Nr	HV 10	d µm	d_h µm	d_v µm
1	416	211,1	211,9	210,3
2	415	211,3	211,4	211,1
3	416	211,1	210,9	211,4
4	416	211,1	210,9	211,4
5	418	210,6	209,8	211,4
6	422	209,6	209,6	209,7
7	425	208,8	208,3	209,4
8	422	209,6	208,8	210,3
9	424	209,1	209,6	208,7
10	424	209,1	209,5	208,8
11	425	208,8	208,5	209,1
12	419	210,3	210,4	210,1

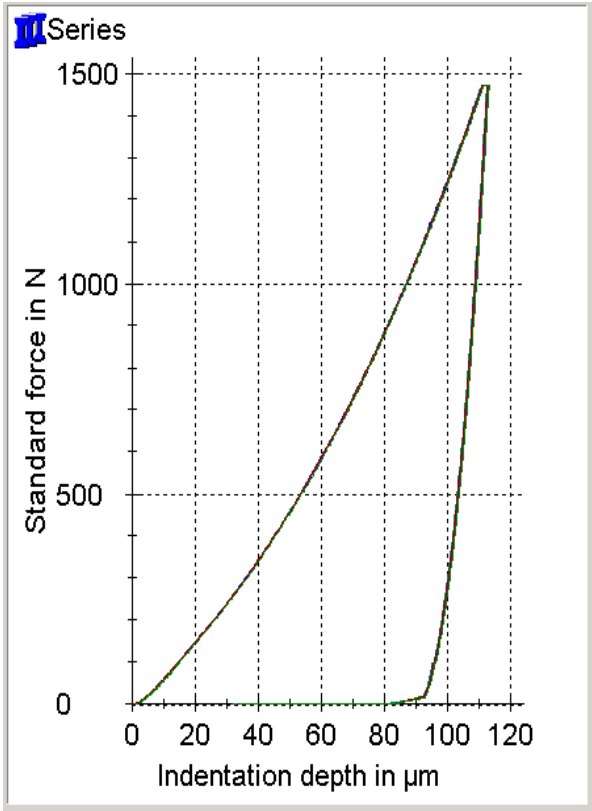
Series n = 12	HV 10	d µm	d_h µm	d_v µm
$\bar{x}$	420	210,0	210,0	210,1
s	4	1,0	1,1	1,0
v	0,92	0,46	0,54	0,49

Results:

- F-h curve measurement
- congruent behaviour
- high reproducibility of machine
- standard deviation of results 1 %

Rockwell test results

Typical result performing a HRC test with ZHU/zwickiLine (done on a HRC test block)



Nr	HRC	h (depth) µm	h1 (pre-load) µm	h2 (pre-load) µm
1	59,5	80,9	14,9	95,8
2	59,6	80,8	14,8	95,6
3	59,6	80,8	14,7	95,5
4	59,6	80,8	14,6	95,4
5	59,6	80,7	14,6	95,4
6	59,6	80,8	14,6	95,4
7	59,6	80,9	14,5	95,4
8	59,6	80,7	14,5	95,2
9	59,7	80,7	14,5	95,2
10	59,5	81,0	14,6	95,6
11	59,6	80,9	14,5	95,3
12	59,6	80,9	15,0	95,8

Series n = 12	HRC	h (depth) µm	h1 (pre-load) µm	h2 (pre-load) µm
$\bar{x}$	59,6	80,8	14,7	95,5
s	0,0	0,1	0,2	0,2
v	0,08	0,12	1,16	0,23

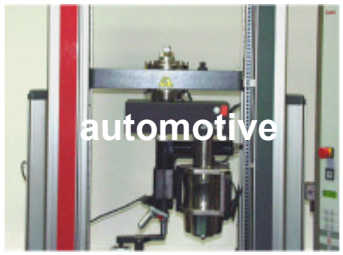
Next specimen: Mandatory inputs

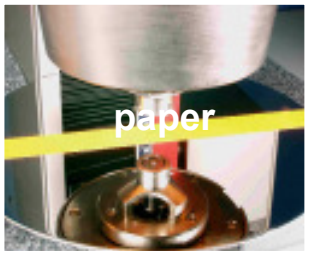
Results:

- F-h curve measurement
- congruent behaviour
- high reproducibility of machine
- standard deviation of results 0.1 %

Application examples

The ZHU/zwickiLine supports in research and development tasks and is working successfully in different industries.





- Materials:
  steel, metals, NF-metals, high strength materials, dental materials, stiff plastic, glass, ...
- Test methods:
  instrumented indentation & classical hardness test methods
- Application:
  Institutes, Research & Development, Rapid Prototyping, Laboratory Application
- Stand-alone systems or fully automated test systems

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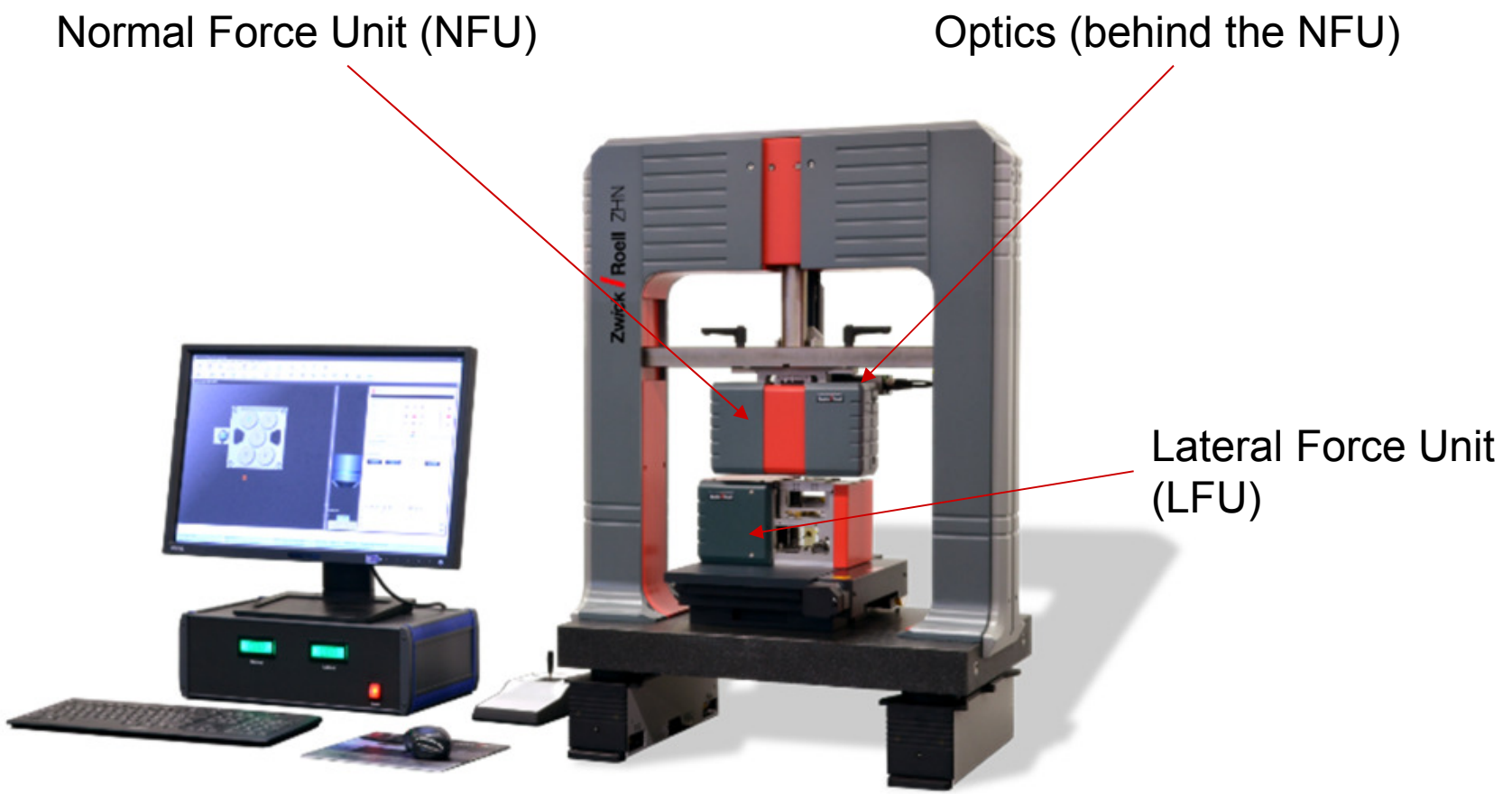
ZHN: modular measuring heads

The modular designed ZHN can be equipped with two measuring heads: Normal Force Unit and Lateral Force Unit



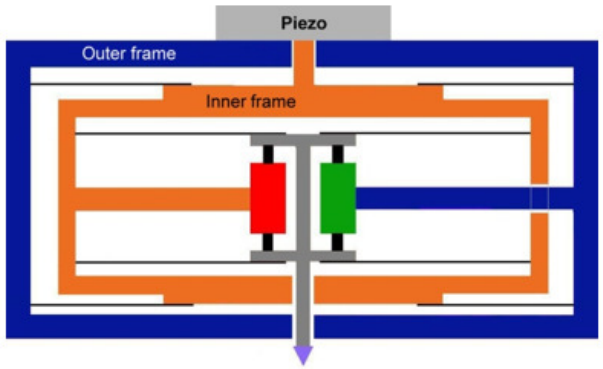
Our NFU: CVP

Our strong CVPs: measurement of adhesion force due to decoupled force generation and force measurement or head can be used in compression and tensile direction.

Normal force head – the difference to other nanoindenters

Patent for NFU & LFU

ZHN principle

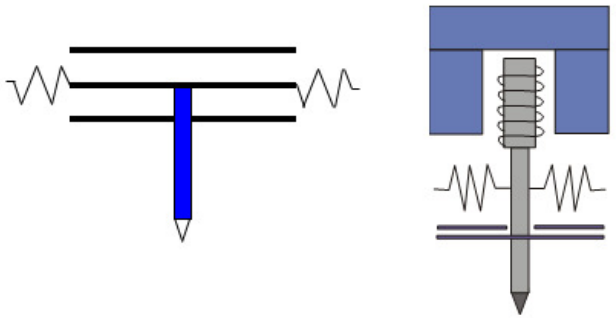


Force generation and force measurement are completely decoupled.
The force measuring spring is only bended after contact with the surface

High lateral stiffness

Easy exchange of indenter

Other principles



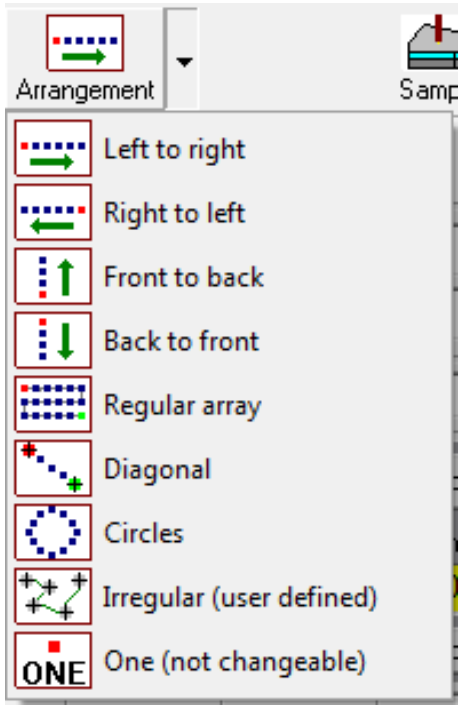
Force generation and force measurement are done with the same signal.
An increase of the force requires a bending of springs already during approach of the surface

Low lateral stiffness

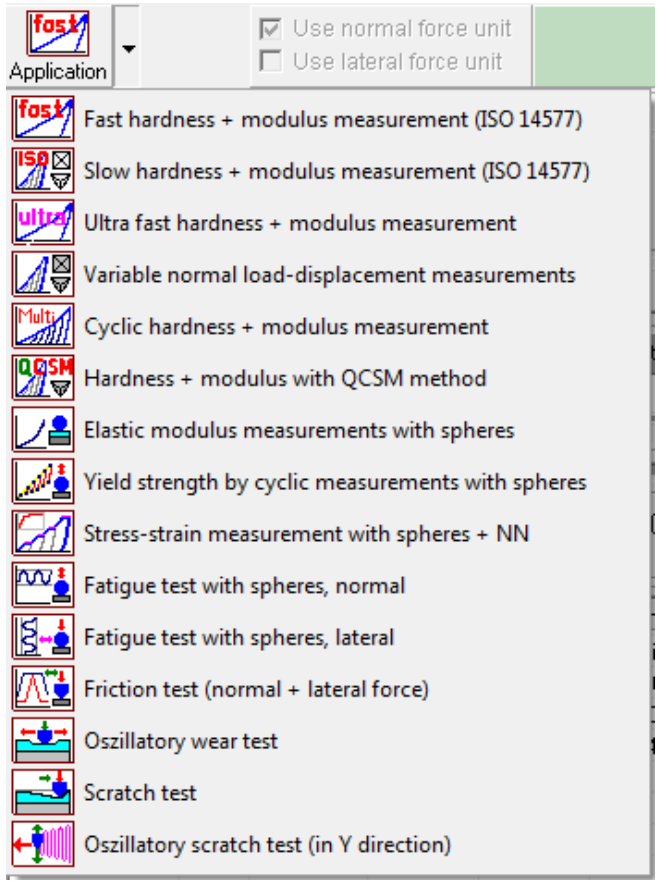
Difficult exchange of indenter

Software InspectorX: test procedures

Predefined applications make the definition of test procedures easy.



Selection menu for point alignment



Selection menu for the application

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Typical application

The durability and frictional behavior of coated components were optimized. (I)

Piston pin (DLC coated)

Cam follower (CrN + DLC coated)

Chain pin (DLC coated)

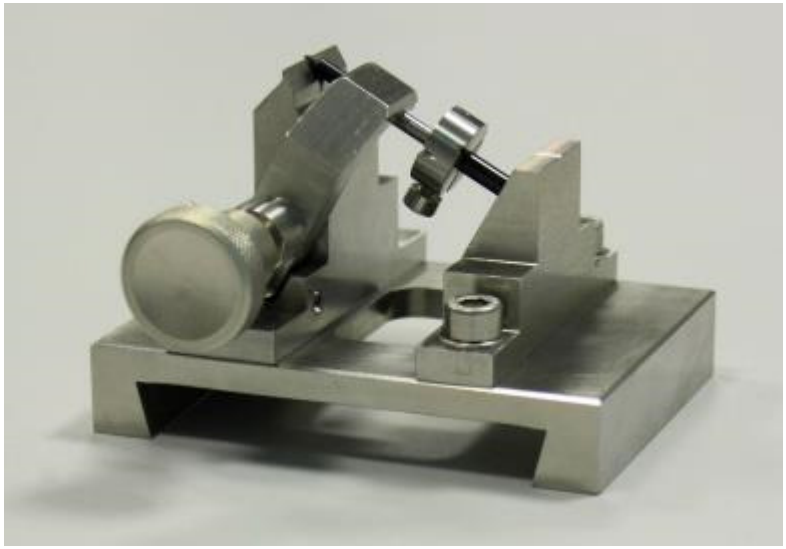
Note: DLC = Diamond-like Carbon

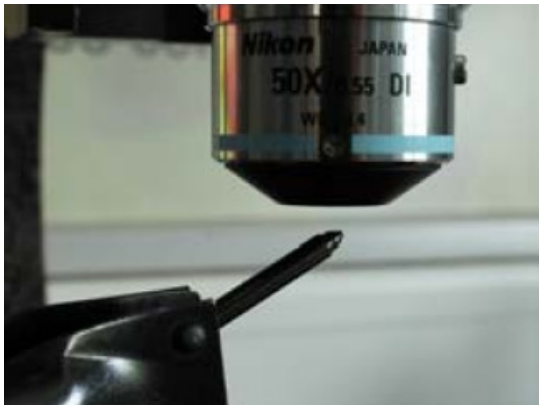
- Example: automotive industry
- Customer's product: piston pin (left), cam follower (middle), chain pin (right)
- Objective: improvement of friction and durability
- Used in: development, failure analysis and quality control
- Testing system: ZHN nanomechanical tester

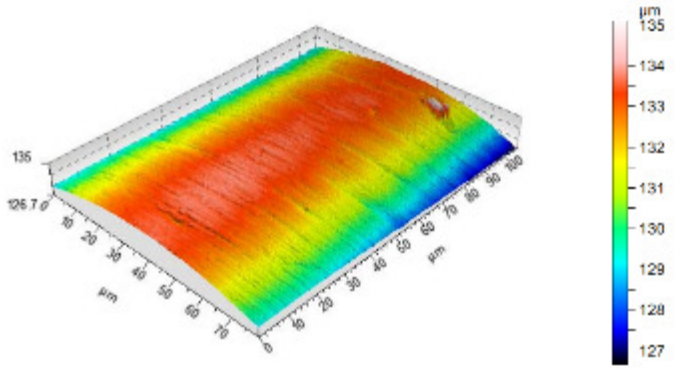
Typical application

The durability and frictional behavior of coated components were optimized. (II)

Injection needle for diesel engines (DLC coated)

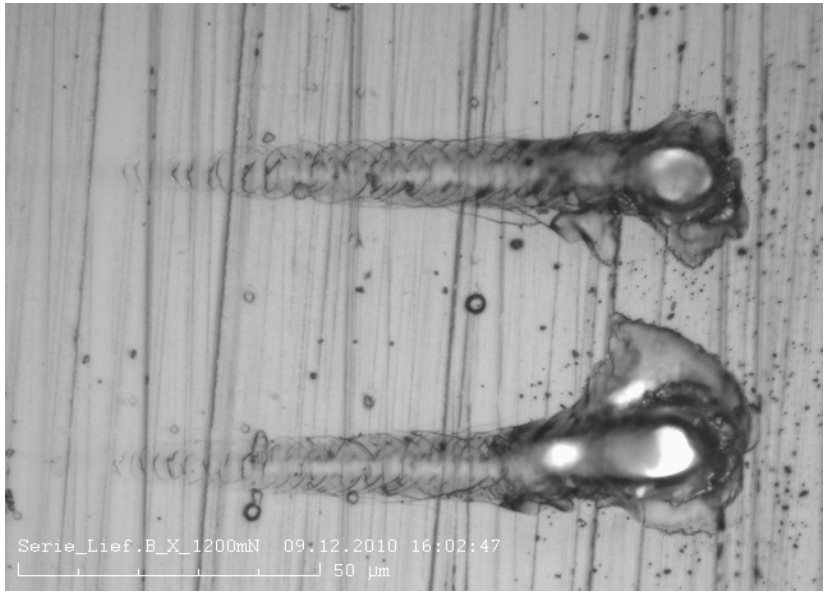


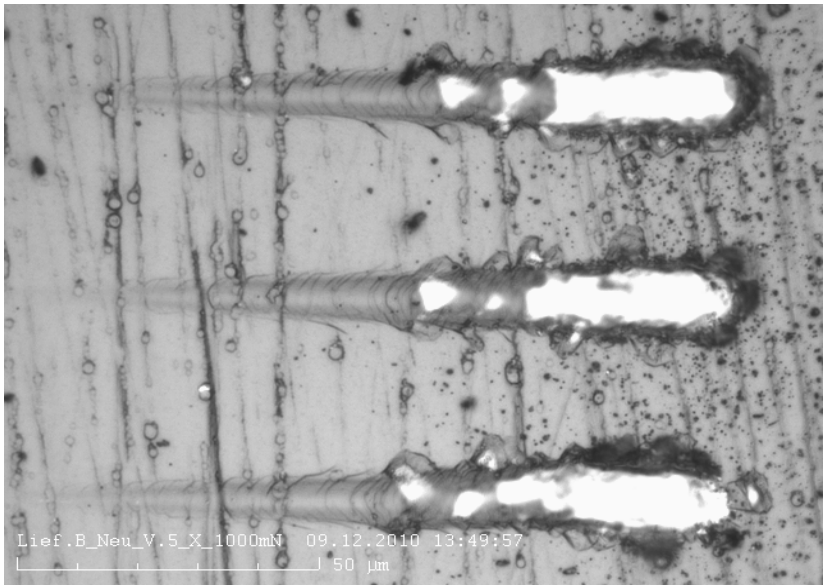




- Application at automotive supplier
- Left: 3D-picture of the needle tip by the use of a white light Interferometer

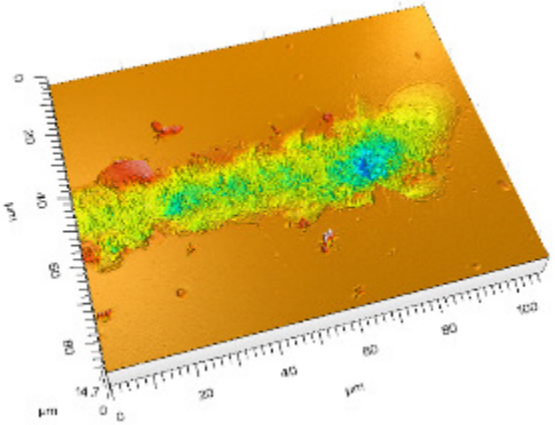
Typical application





Critical force for failure: left: 970mN

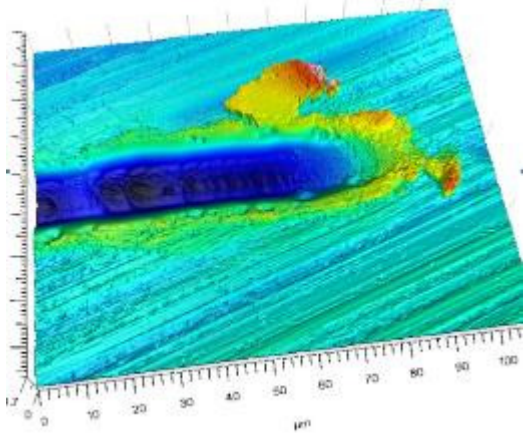
right: 650mN



3D profiles

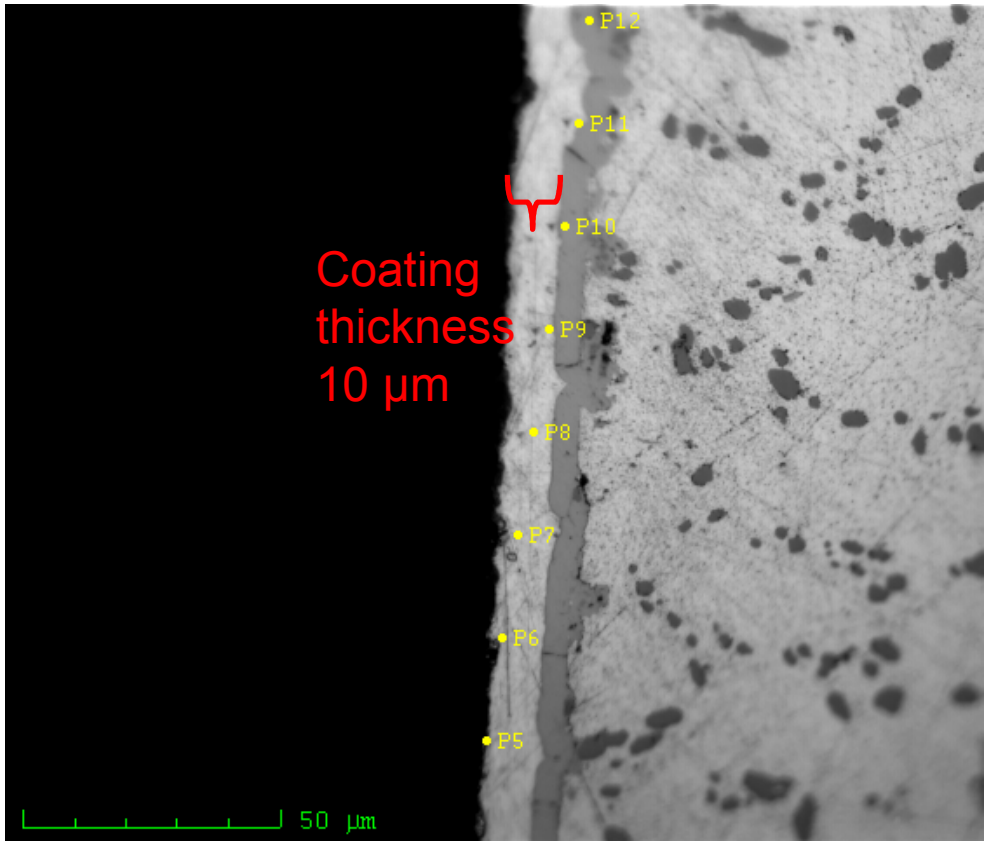
← Fused silica

Steel →



Typical application

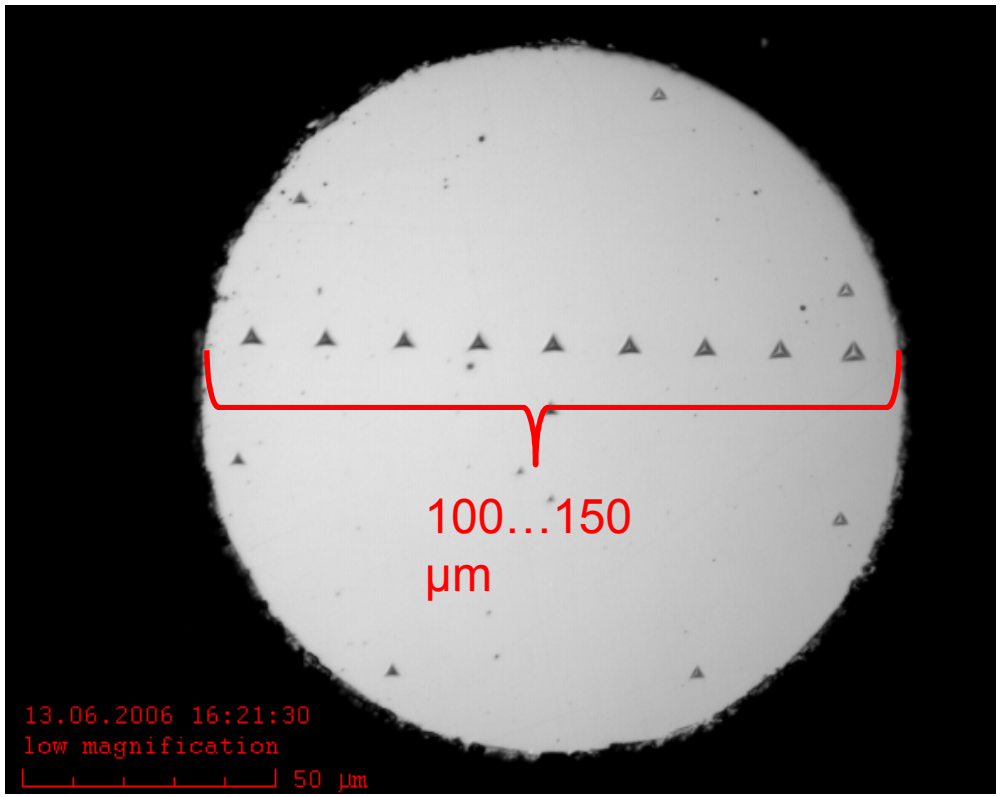
Wear/compound (white) layer of a component investigated in transverse cross-section by means of a hardness test.



- Example:
  metalworking industry
- Customer's product:
  component with 10-20µm wear-layer
- Objective:
  measurement of the hardness profile in the wear/ compound layer (tested in transverse cross-section)
- Used in:
  development, quality control
- Testing system:
  Nanoindenter ZHN

Typical application

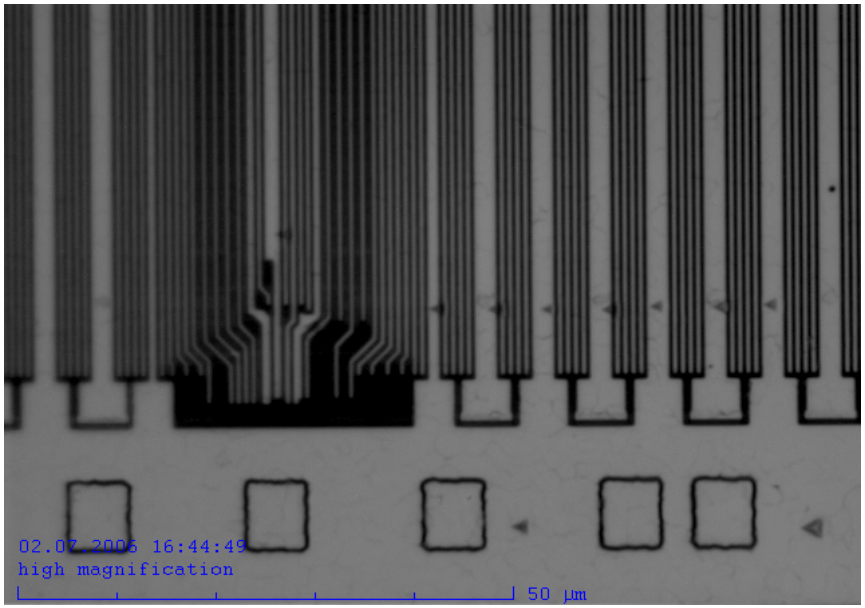
Hardness distribution of a cutting-wire used in the manufacture of silicon wafers was tested in transverse cross-section

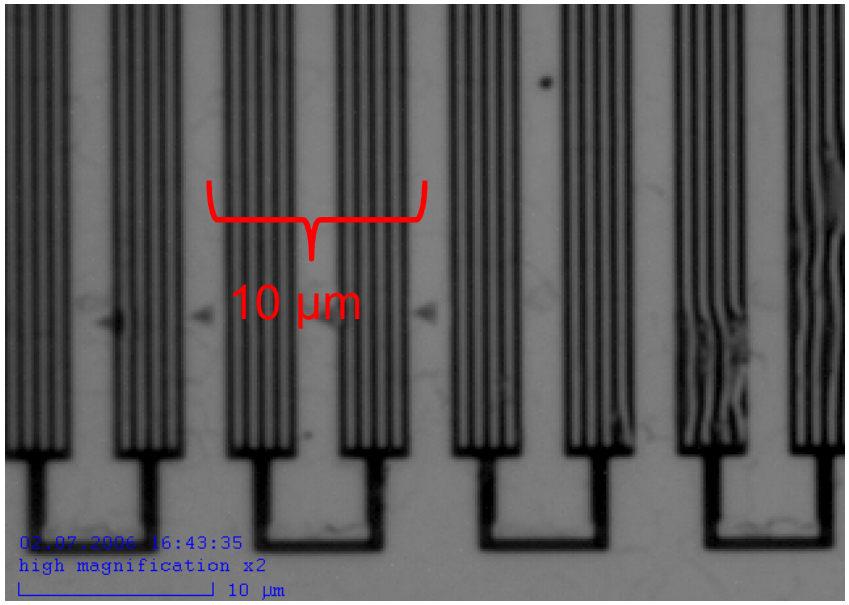


- Example:
  metals/electrical industries (photovoltaic/microelectronic)
- Customer's product:
  diamond cutting-wire (Ø 100-150 µm) for the manufacture of silicon wafers
- Objective:
  measurement of the hardness profile (transverse, contour) of the cutting-wire (tested in transverse cross-section)
- Used in:
  development, quality control
- Testing system:
  Nanoindenter ZHN

Typical application

Tracks on printed circuit boards were optimized via hardness and Young's modulus measurement

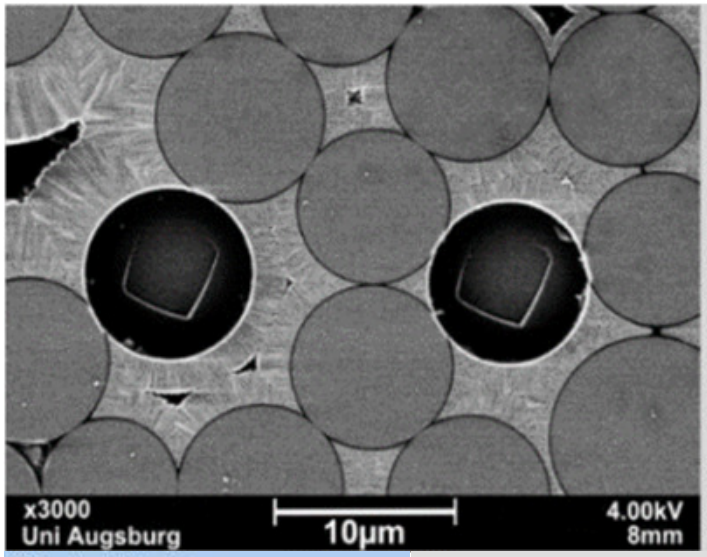


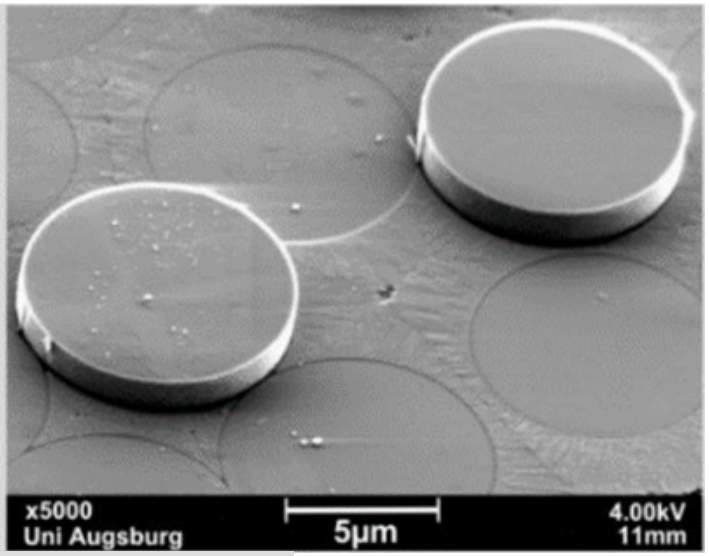


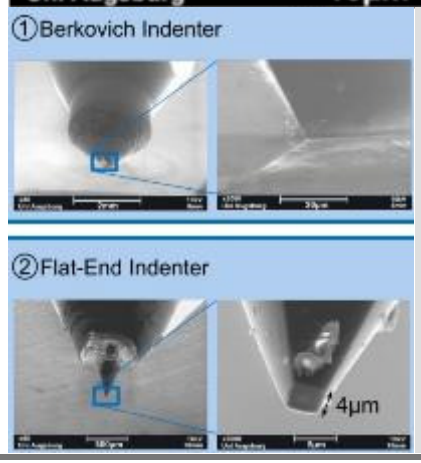
- Example: Electronic industry
- Customer's product: microelectronics, integrated circuits, electronic boards
- Objective: failure analysis
- Used in: development, quality control
- Testing system: Nanoindenter ZHN

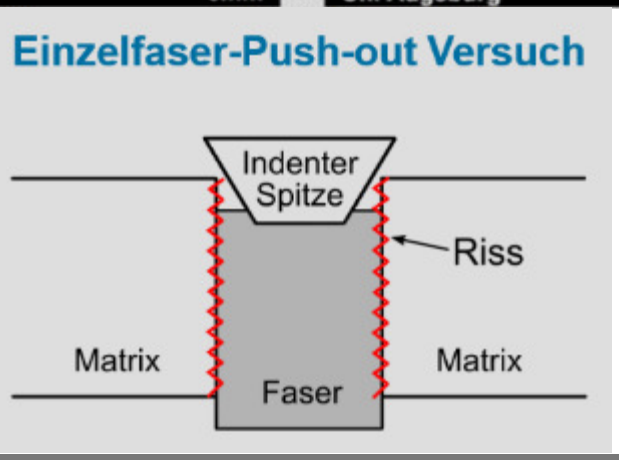
Typical application

Investigation of the adhesion strength of fibers in a composite material.









Source: Dr. Müller, UNI Augsburg

- Example: Composite industry
- Used in: Research

Many thanks for your attention