Zwick / Roell

Intelligent Testing

ZHN Nanoindentation – an application overview

testXpo 2018, October 15 - 18

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ZwickRoell GmbH & Co. KG

Agenda

Introduction

ZHN: Our Solution, Portfolio & News

Customer Applications

Introduction

What do we mean by nanoindentation and nanoindenter?

ISO 14577 makes the following distinction:

- macro range for forces from 2 to 30000 N and indentation depths greater than 6 µm
- micro range for forces below 2 N
- nano range for indentation depths less than 0.2 µm
- ... and includes determination of hardness and other material parameters.

→ Instrumented indentation test in which test load and indentation depth are measured continuously; **the displacement resolution is in the nanometer range**

Background :

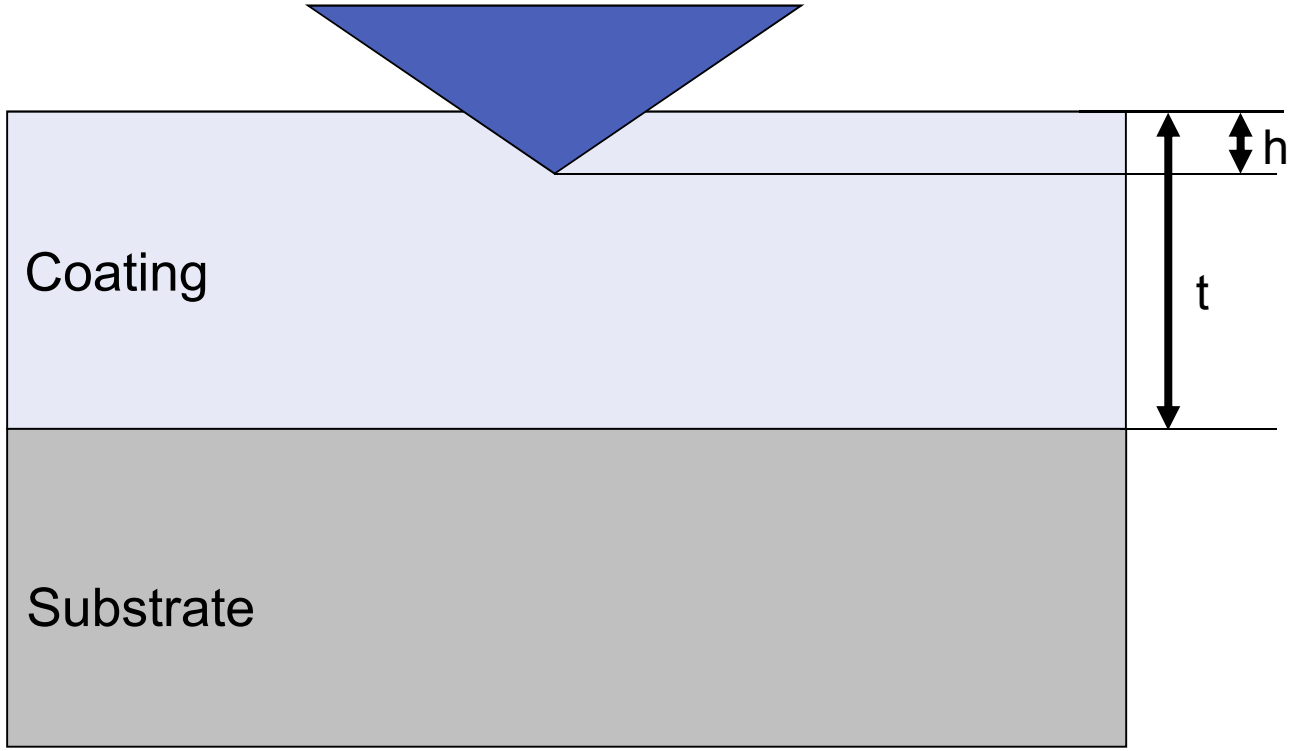
- The dimension of an indentation is too small for optical measurement
- Vickers indenter: diagonal length d ≈ 7 * depth
- Min. diagonal length for optical measurement: 20µm → min. depth around 3µm
- With 1/10th rule: minimum measurable film thickness 30µm

Additionally there are other “Nano-Applications” like scratch, wear, adhesion, friction, …

Introduction

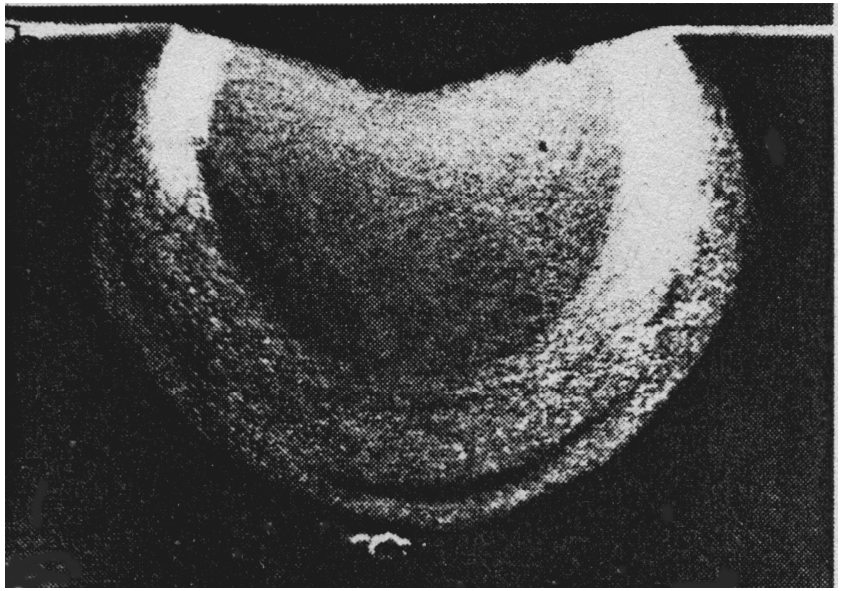
Depth limit for coatings hardness:

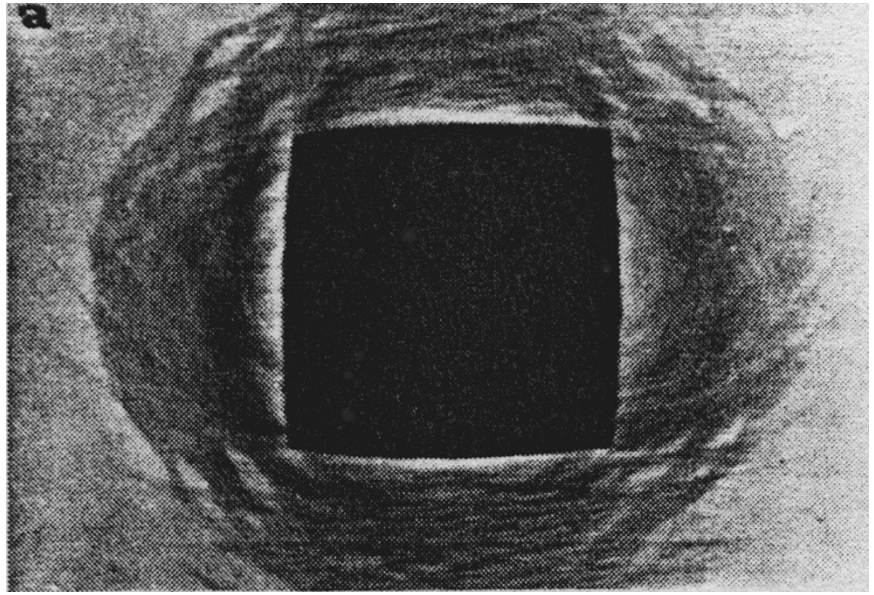
It is: One tenth rule → h < 1/10 x t



Introduction

Reason for depth limit: The plastic zone is much larger and deeper than the indent. Therefore the information in the load-displacement curve comes from a depth of up to 10 times the indentation depth.

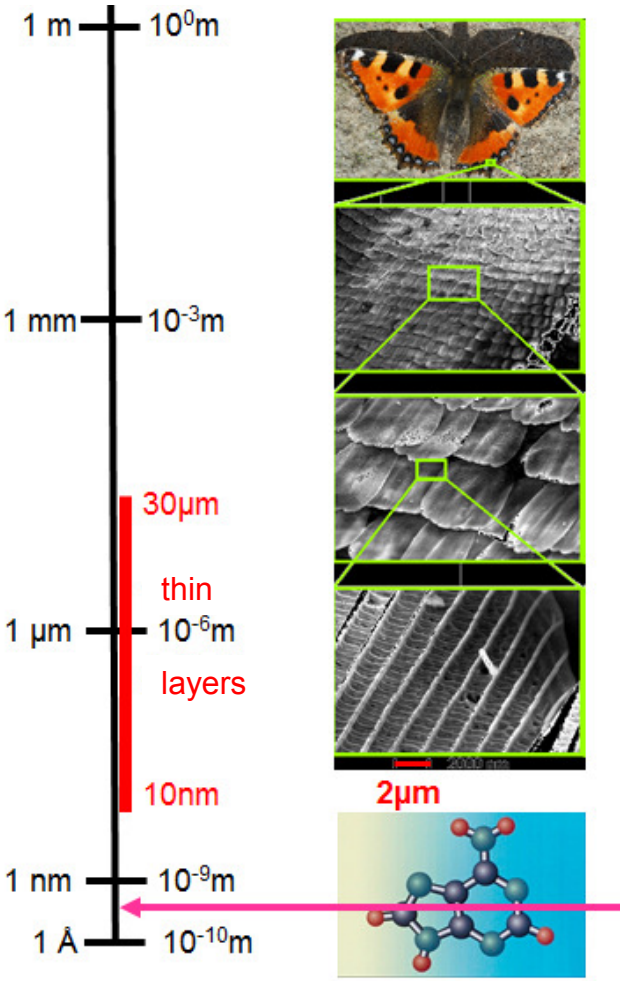




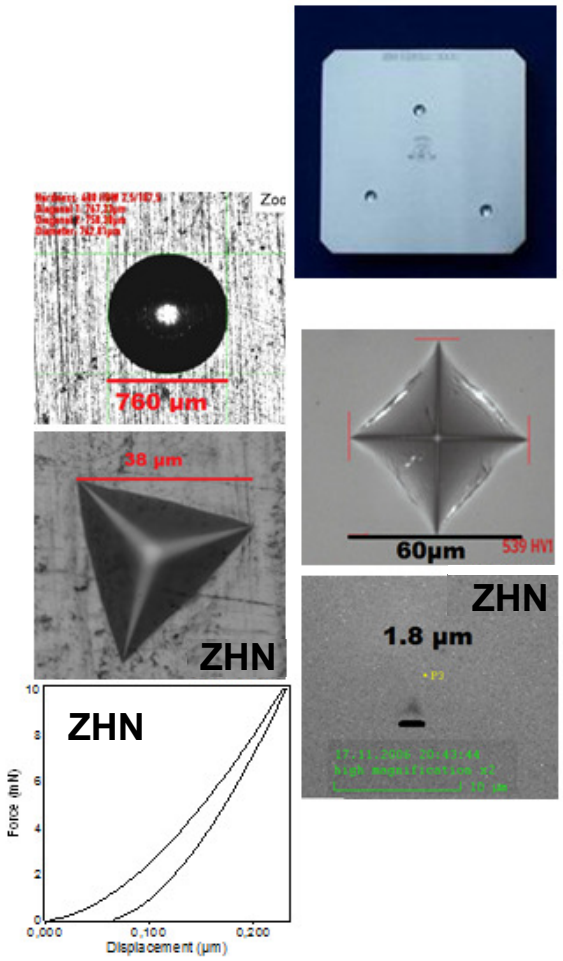
Presentation of the plastic zone in steel using a special etching technique

Introduction

The thickness of thin layers is between 10 nm and 30 µm. Too thin to measure optically…



Noise level displacement measurement of ZHN



Agenda

Zwick / Roell

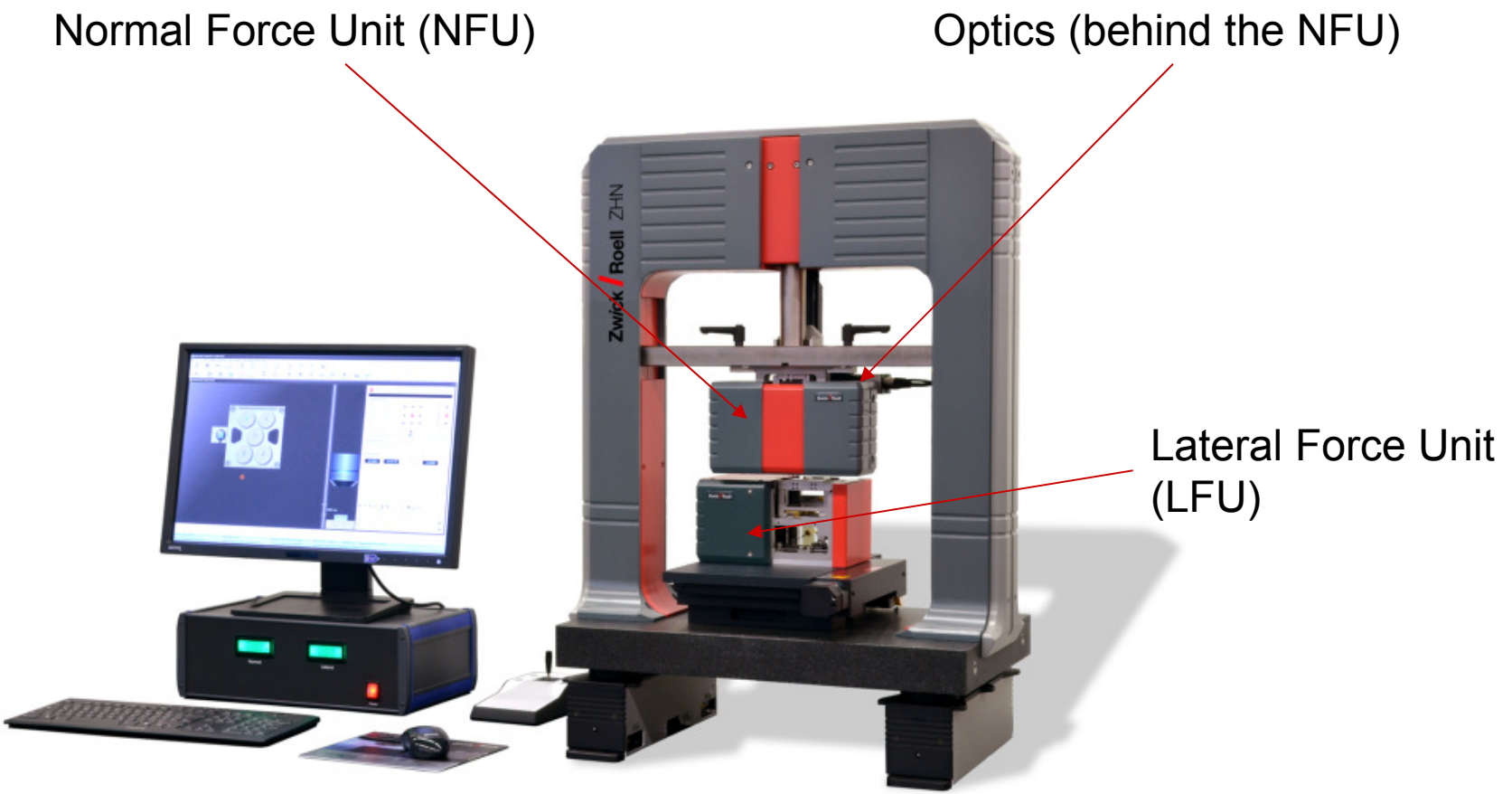
Introduction

ZHN: Our Solution, Portfolio & News

Customer Applications

ZHN: modular measuring heads

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



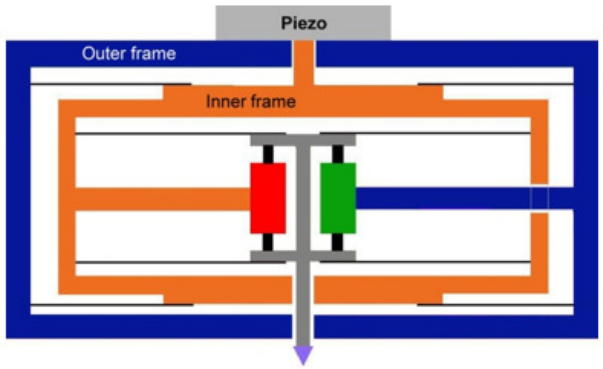
Our NFU: CVP

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

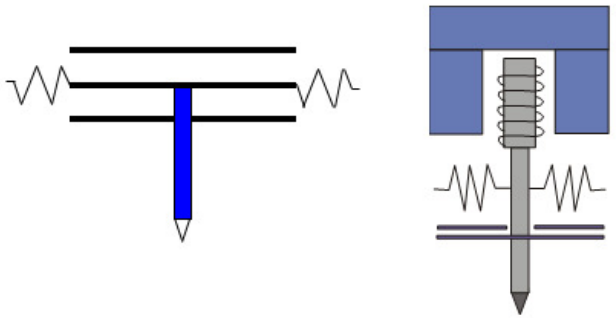


Force generation and force measurement are completely decoupled.
The force measuring spring is only bended after contact with the surface

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

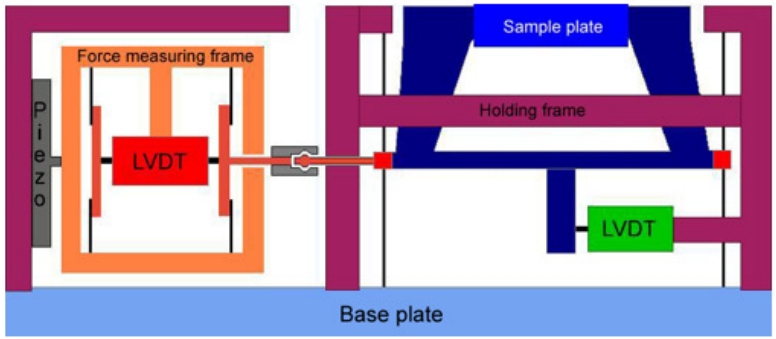
Low lateral stiffness

Difficult exchange of indenter

Our LFU: CVP

Our strong CVPs: measurement of friction force due to decoupled sample movement (green LVDT) and force measurement (red LVDT).

Lateral force head – the new component



- Nanometer resolution like in normal direction
- The sample is moved laterally (not the tip)
- High stiffness in normal direction
- No height change during lateral movement

Patent for NFU & LFU

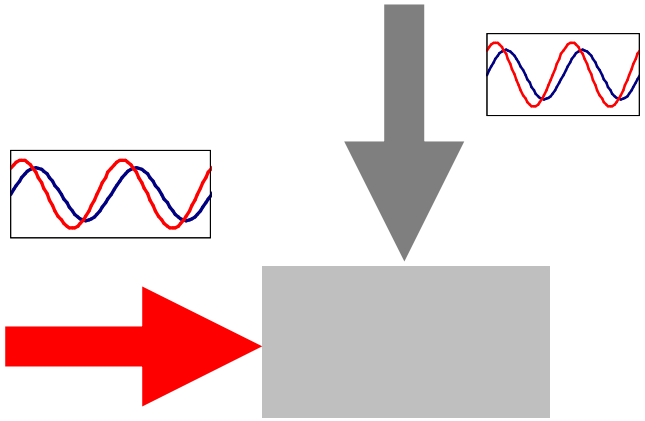
- A force can be applied and measured without any movement of tip or sample
- No rolling motion of the tip due to bending of the indenter shaft
- Transition sticking- sliding friction highly resolved

The interaction of normal and lateral forces due to static and sliding friction can be considered with the ZHN according to the conditions in the application without loss of resolution.

4-degrees-of-freedom loading

The Nanoindenter ZHN can load the sample in 4 degrees of freedom simultaneously which mostly reflects real loading

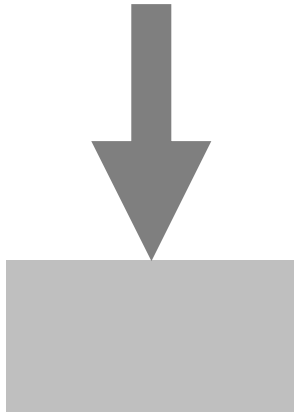
Zwick / Roell Nanoindenter



4 degrees of freedom:

- normal force-displacement curve
- lateral force-displacement curve
- vibration normal (dynamic)
- vibration lateral (dynamic)

Standard Nanoindenter
Standard Scratchtester



1 degree of freedom:

- normal force-displacement curve

ZHN: wide range of application

As well as its main application as a 'hardness and Young's modulus tester', the nanomechanical tester ZHN covers the applications of wear-tester, scratch-tester, profilometer and fatigue tester

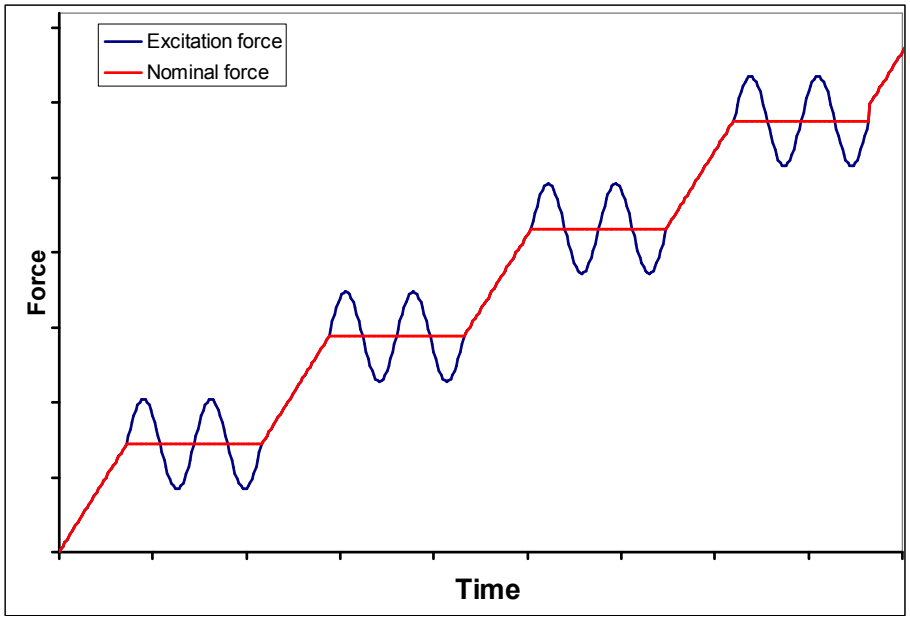
ZHN nanomechanical tester with 2 measuring heads

- hardness + Young's modulus tester
- micro wear tester
- micro scratch tester
- profilometer (roughness measurement)
- fatigue tester

Standard nanoindenter with 1 measuring head

- hardness + Young's modulus tester

QCSM Method (Quasi continuous stiffness measurement, developed by ASMEC)



A sinusoidal oscillation is used in superposition to the force signal and is switched on during short dwell times of 1-4 s. Average normal force is kept constant at every point.

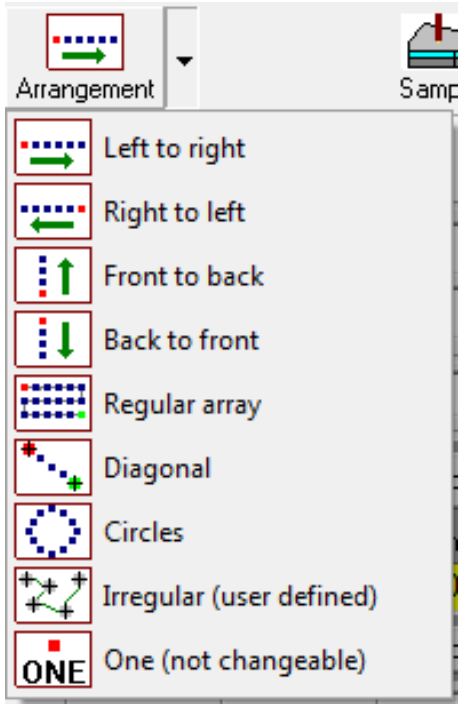
First 20% of the dwell time is not used to reduce creep influence.

Advantages:

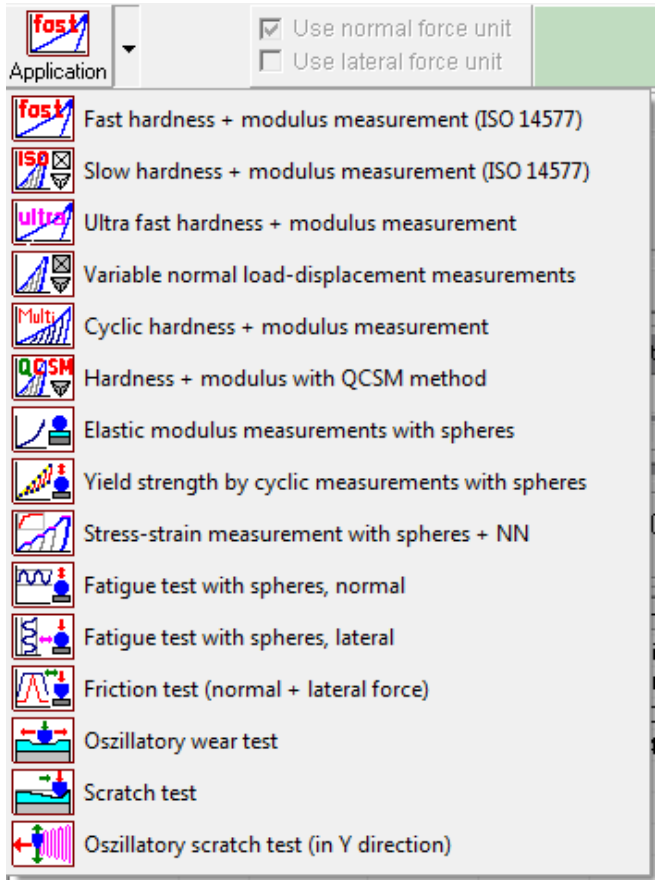
- The result can be averaged over several oscillations and is therefore more accurate.
- The effect of creep on the result is significantly reduced.
- The corresponding force values can be specified.

Software InspectorX: test procedures

Predefined applications make the definition of test procedures easy.



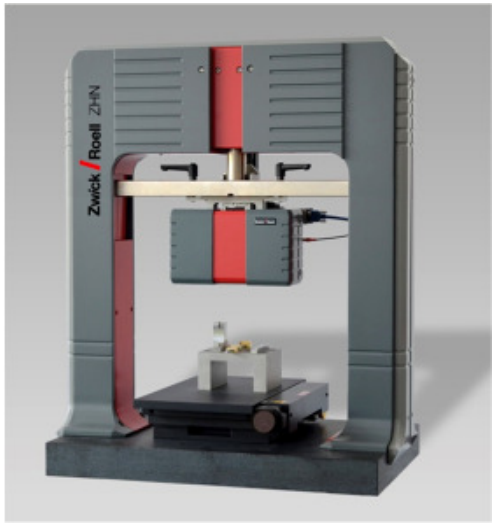
Selection menu for point alignment



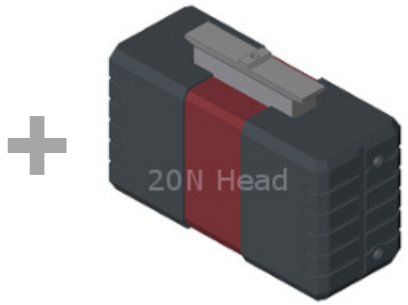
Selection menu for the application

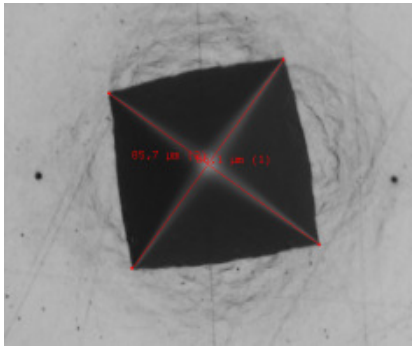
At testXpo 2018 we will show…

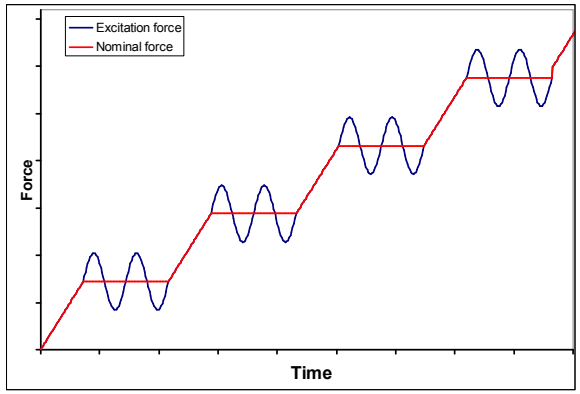
…. the new 20 N NFU Normal Force Unit



- Extended application range with new 20 N measuring head:
  - Every time when the conventional **micro Vickers** is used, but with additional information about modulus of elasticity, energies and creep behavior
  - Measuring “thick” coatings
- The new 20N head has excellent dynamic capabilities an can measure according to QCSM or CSM method.
  This is a unique feature for this load range.





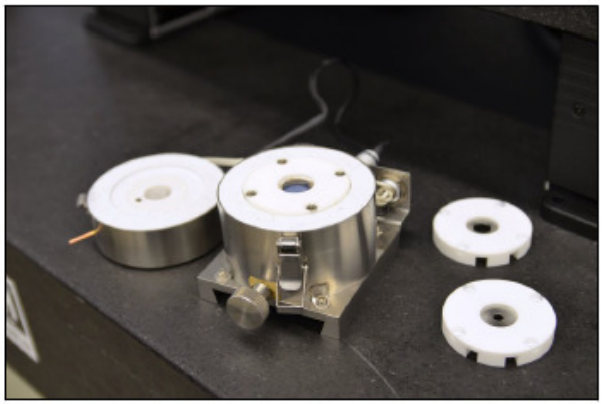


New sample heater to ZHN

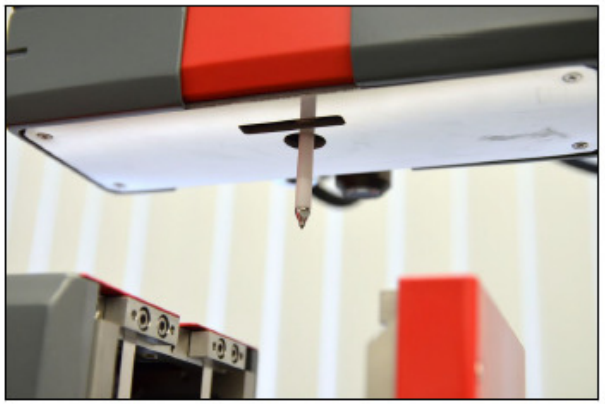
The new sample heater for the ZHN enables testing at elevated temperatures up to 400°C **in an inert gas atmosphere**

What is new?

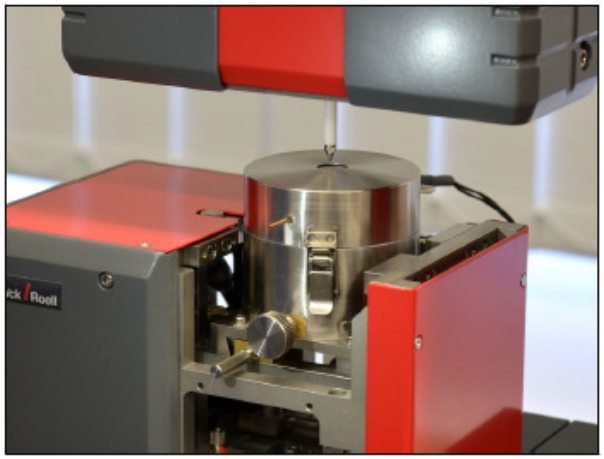
- Gas feed connection for testing at elevated temperature in an inert gas atmosphere to eliminate oxidation on the sample surface
- Predecessor had only heating functionality (oxidation occurred)



Picture 40: Specimen heater, open, with additional inserts



Picture 41: Shaft extension



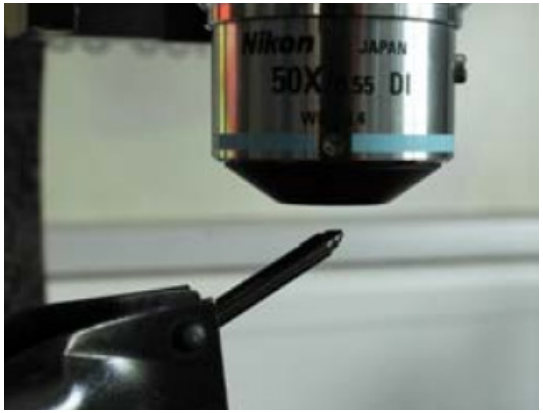
Picture 42: Specimen heater mounted in a ZHN

ZHN: Optic options

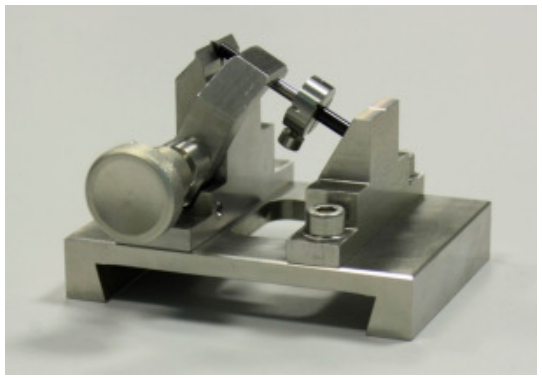
Integration of a WLI (White Light Interferometer) with Mirau objective in the standard optics of the ZHN:

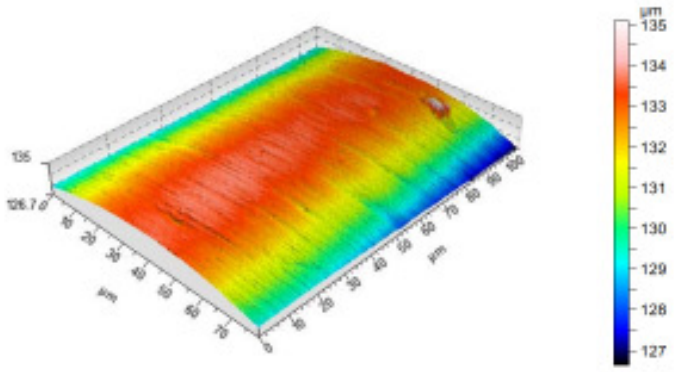
Advantage:

- 2 cameras = 2 magnifications
- economical solution + upgradeable
- Excellent analysis options with MountainsMap software



Injection needle for diesel engines (DLC coated)

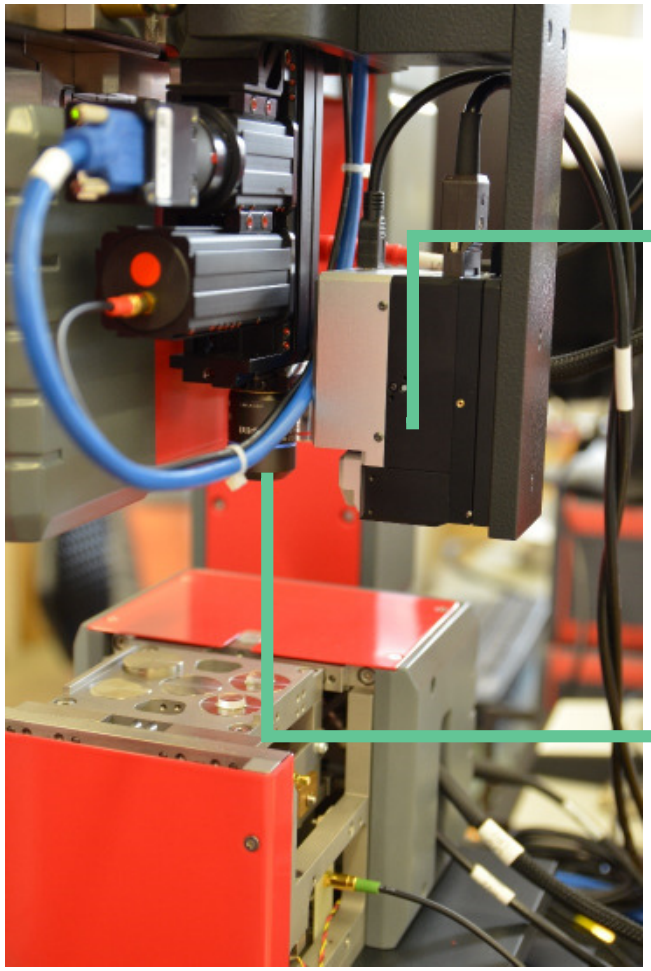


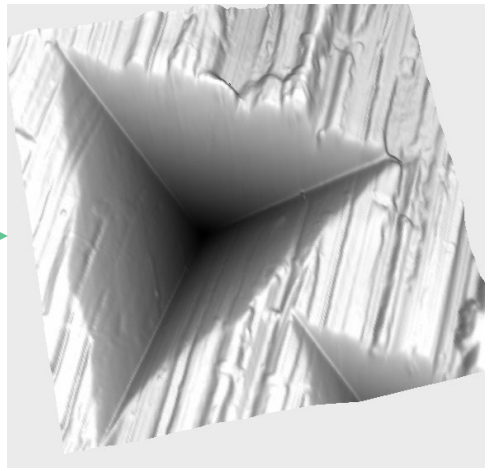


- Application at automotive supplier
- Left: 3D-picture of the needle tip by the use of a white light Interferometer

ZHN: Optic options

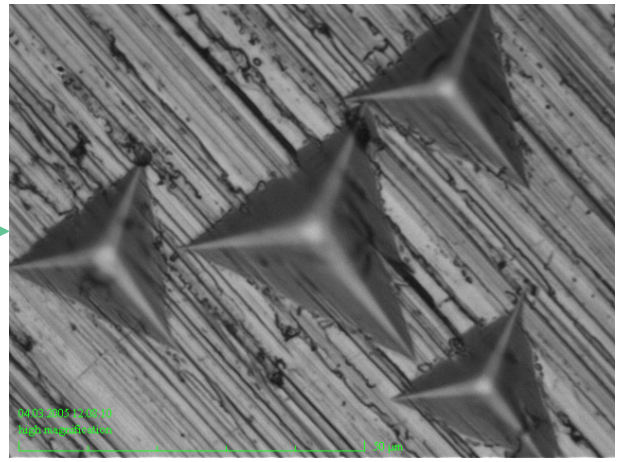
Integration of an AFM (Atomic Force Microscope) into the ZHN:





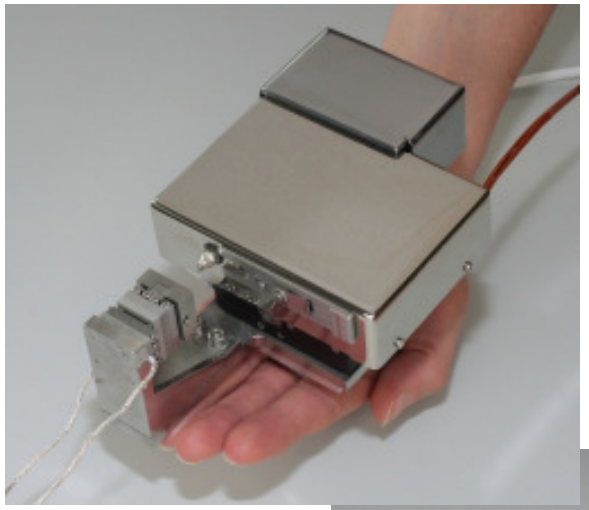
nanoSurf®

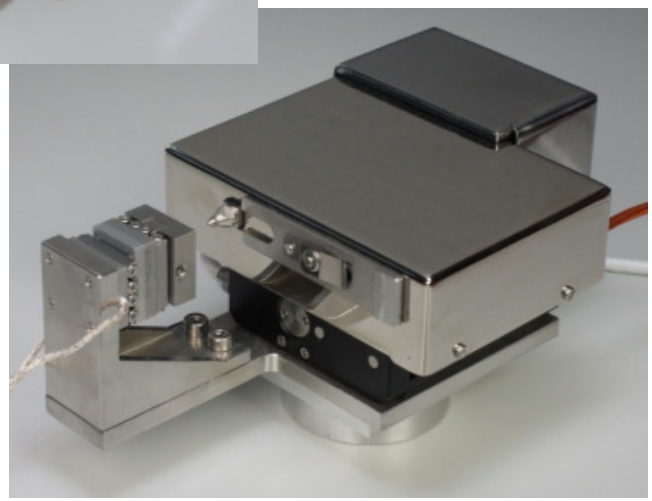
AFM

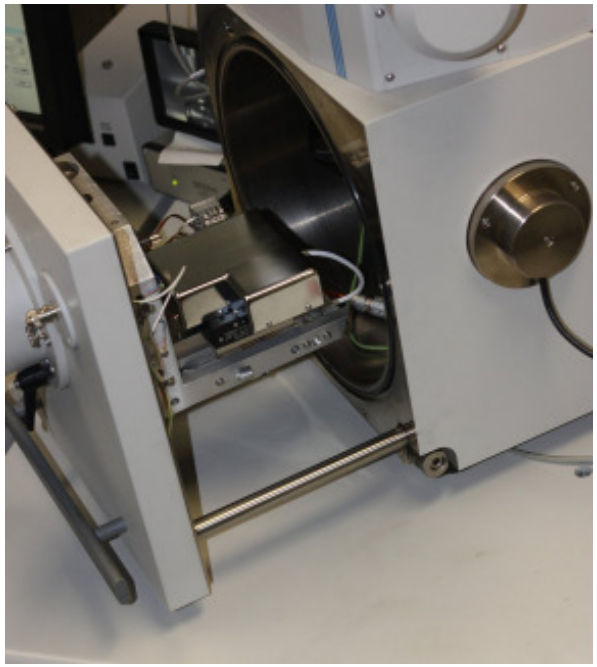


Microscope

Nanoindenter ZHN for installation in a scanning electron microscope (SEM)







ZHN/SEM
mounted in the chamber of a scanning electron microscope

Agenda

Motivation & Introduction

ZHN: Our Solution, Portfolio & News

Customer Applications

Typical application

The durability and frictional behavior of coated components were optimized.

Piston pin (DLC coated)

Cam follower (CrN + DLC coated)

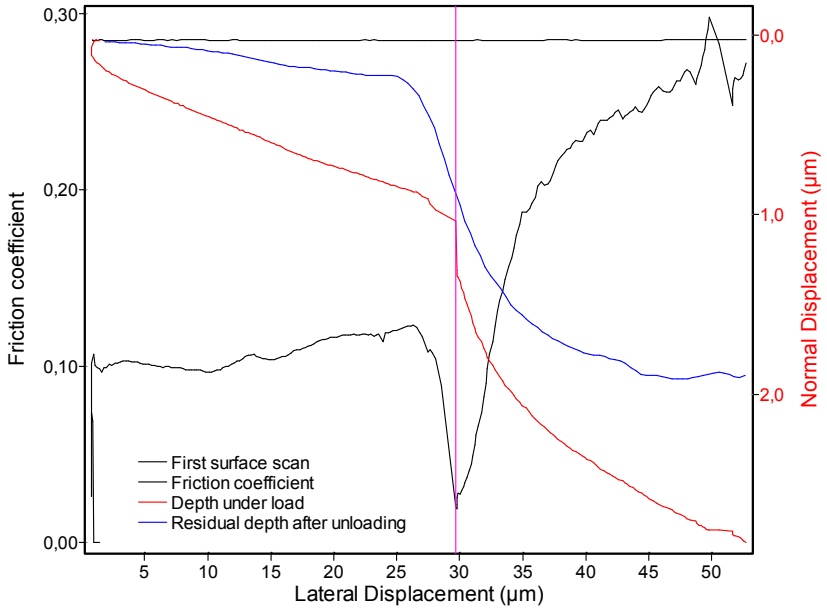
Chain pin (DLC coated)

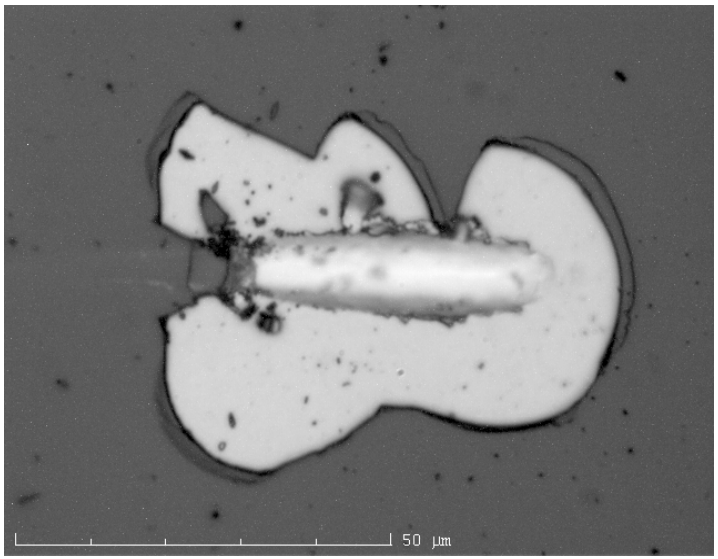
Note: DLC = Diamond-like Carbon

- 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 adhesion of the coating is qualified by the micro scratch test

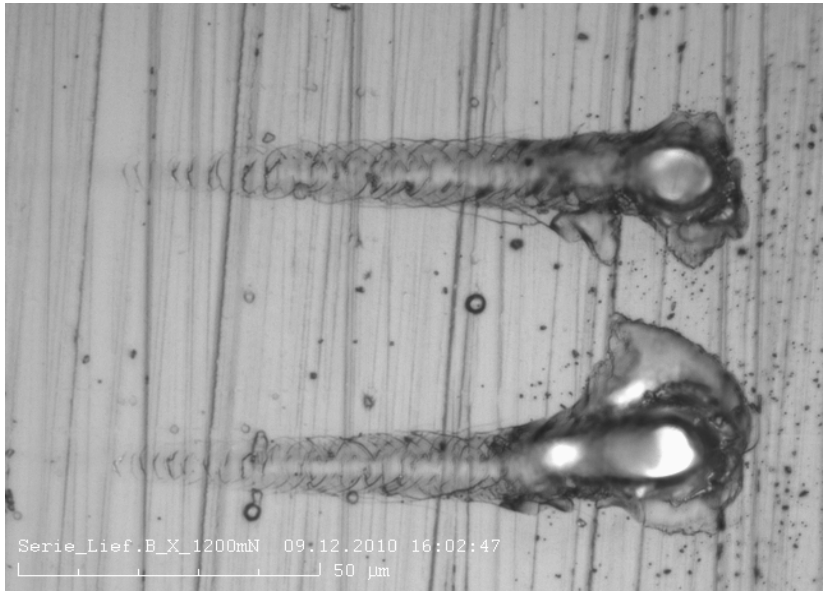


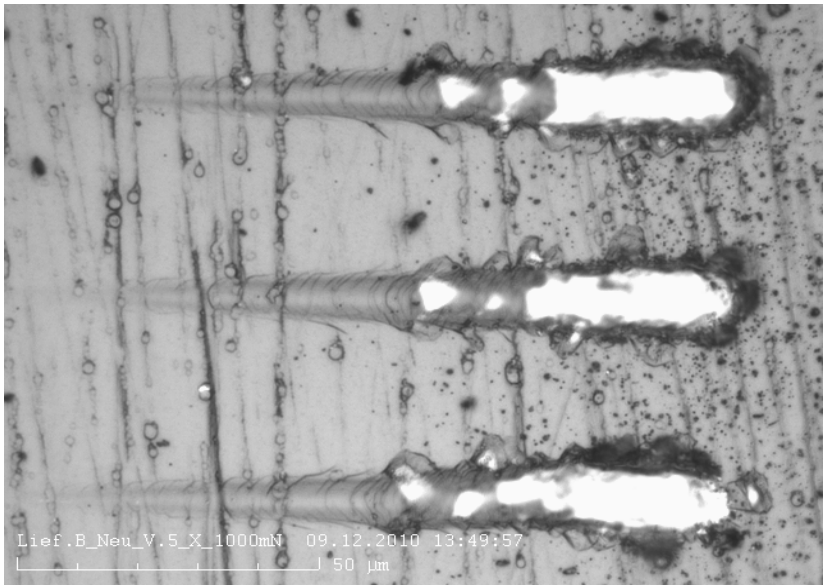


Dark area: DLC coating; bright area: failure of the coating, seen is the steel substrate with scratch

- Example for a micro scratch test on a 2 µm DLC coated steel
- The red curve is the indentation depth under loading during the scratch, the delamination can be recognized by a vertical step at 30 µm lateral displacement
- The black curve is the friction coefficient with a minimum at 30 µm lateral displacement (when the coating fails) and an increase in the friction on the steel substrate

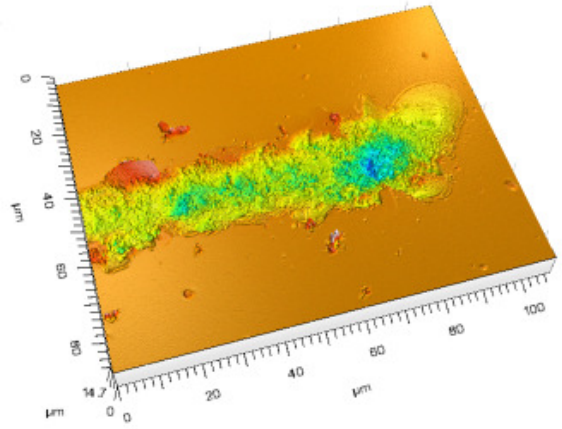
Typical application





Critical force for failure: left: 970mN

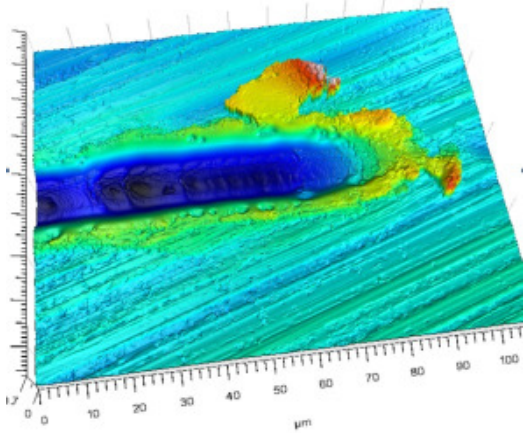
right: 650mN



3D profiles

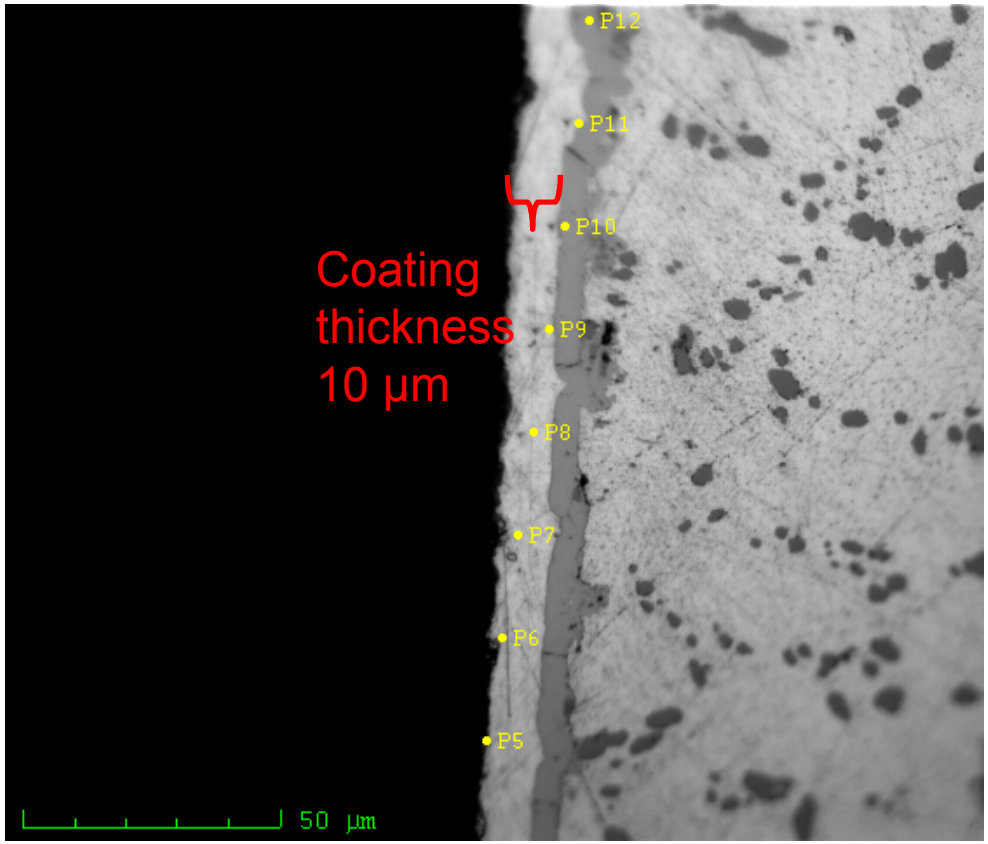
← Fused silica

Steel →



Typical application

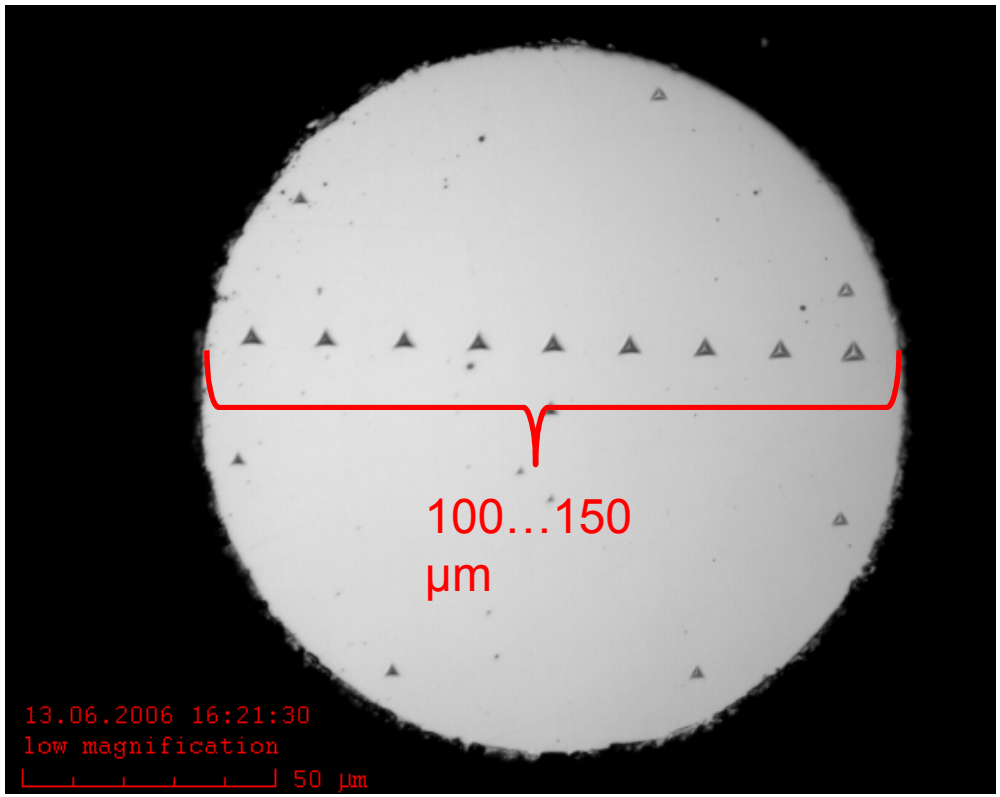
Wear/compound (white) layer of a component investigated in transverse cross-section by means of a hardness test.



- 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

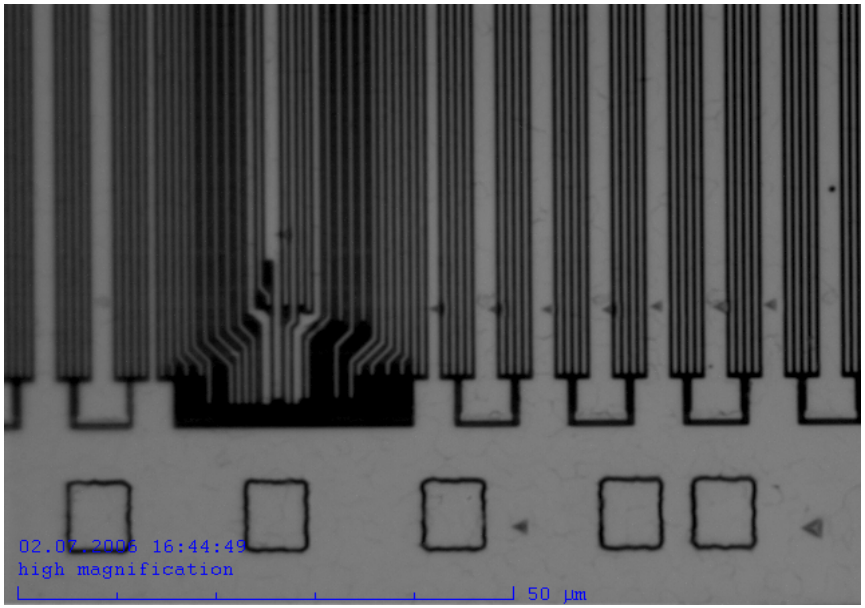
Hardness distribution of a cutting-wire used in the manufacture of silicon wafers was tested in transverse cross-section

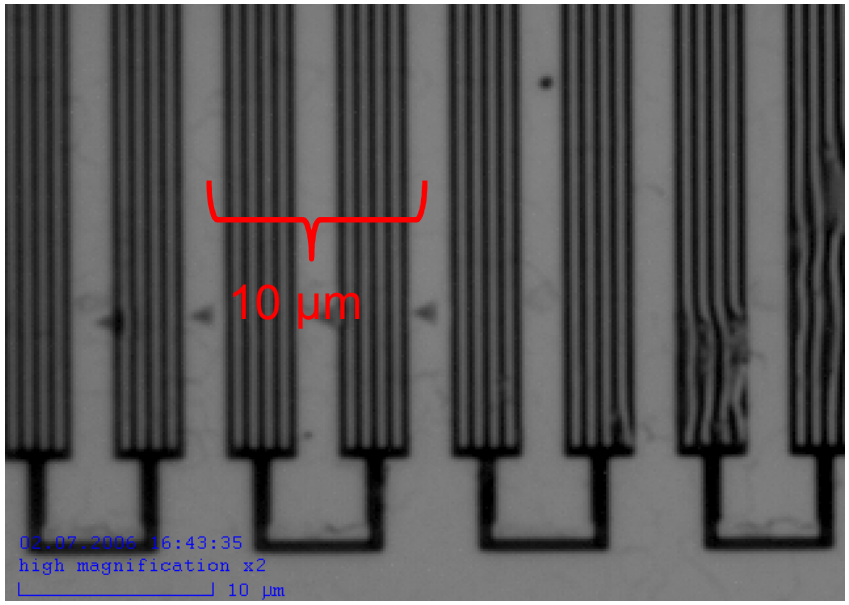


- Example:
  metals/electrical industries (photovoltaic/microelectronic)
- Customer's product:
  diamond cutting-wire (Ø 100-150 µm) for the manufacture of silicon wafers
- Objective:
  measurement of the hardness profile (transverse, contour) of the cutting-wire (tested in transverse cross-section)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

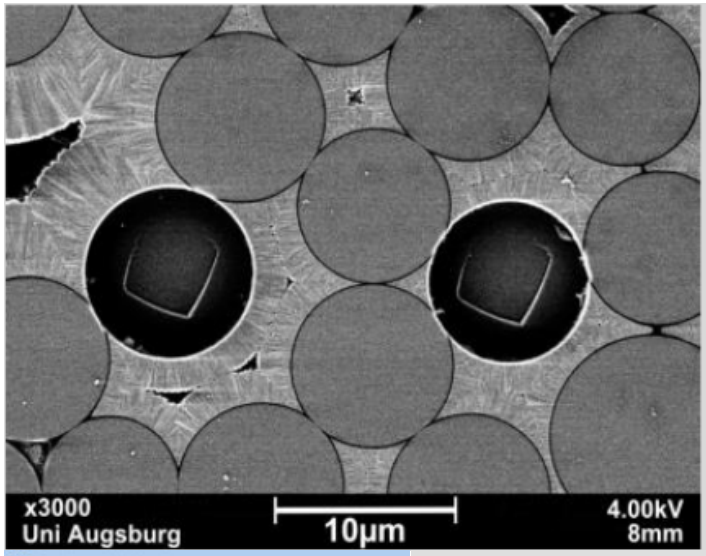


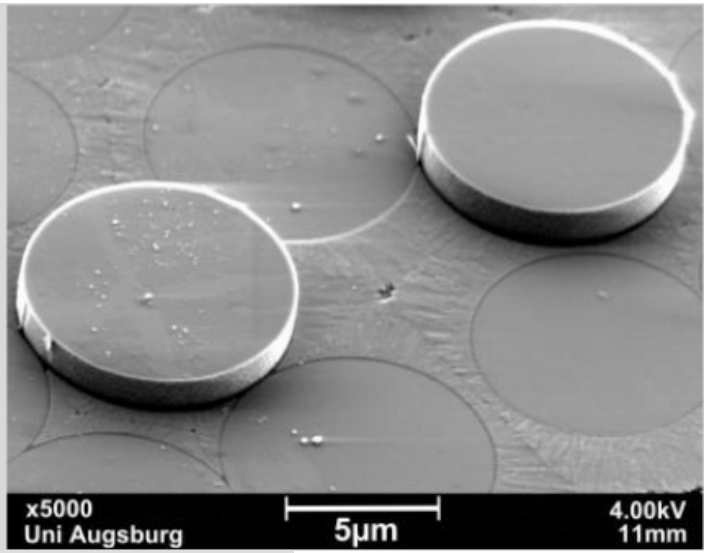


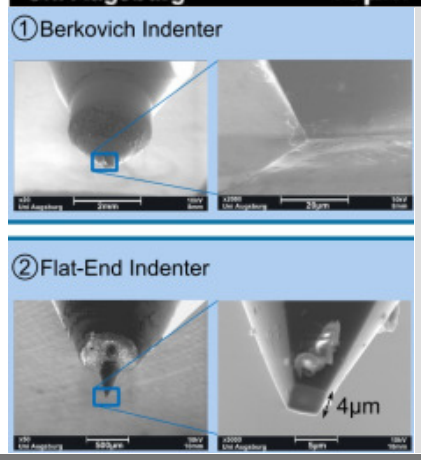
- Example: Electronic industry
- Customer's product: microelectronics, integrated circuits, electronic boards
- Objective: failure analysis
- Used in: development, quality control
- Testing system: Nanoindenter ZHN

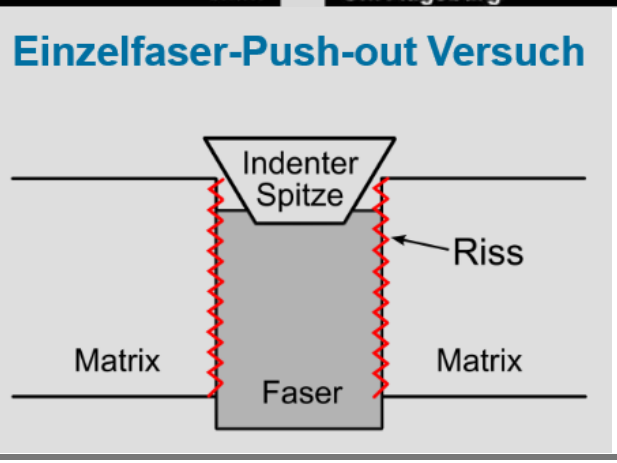
Typical application

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