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

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

**Part 1:**
Test procedure
**Macro** \((F > 2 \text{ N}; \ h > 6 \text{ µm})\), Micro, Nano \((h < 200 \text{ nm})\)
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
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
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
**Introduction**

**Test method – instrumented indentation**

\[ \text{Hardness} = \frac{\text{Force } F}{\text{Area } [A_s(h) \text{ or } A_p(h_c)]} \]

![Diagram showing a test method with force (F) and depth (h) relationships, including terms for hardness calculation.](image)

- **Force** \( F \)
- **Depth** \( h \)
- **Loading**
- **Unloading**
- **Tangent**
- **Indentation depth** \( h \) (µm)
- **Hardness** calculation components: \( A_s(h) \) and \( A_p(h_c) \)

The diagram illustrates the force-depth relationship during loading and unloading, with the hardness calculation equation shown alongside.
Material parameter

Material parameter of Martens hardness

Martens hardness

\[ HM = \frac{F}{A_s(h)} \]

Indentation hardness

\[ H_{IT} = \frac{F_{\text{max}}}{A_p(h_c)} ; \quad h_c = h_{\text{max}} - \frac{\varepsilon F_{\text{max}}}{(\frac{dF}{dh})_{h=h_{\text{max}}}} \]

Indentation modulus

\[ E_{IT} = \frac{1 - \nu_s^2}{E_r} - \frac{1 - \nu_i^2}{E_i} \]

\[ E_r = \frac{\sqrt{\pi}}{2\sqrt{A_p(h_c)}} (\frac{dF}{dh})_{h=h_{\text{max}}} \]

\[ E_r - \text{reduced Modulus} \]
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.

- 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)

- 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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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.

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

Results:
- \( h_{\text{max}} \approx 220 \, \mu\text{m} \)
- Negligible elastic fraction of indentation work, \( \eta_{\text{it}} \approx 10.5\% \)

HM-quantity:
- Little dependence of indentation depth (< 30 \( \mu\text{m} \))
F & HM - h diag. (steel)

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

Results:

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

HM-quantity:

- higher dependence of indentation depth (< 50 $\mu$m)
Vickers test results

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

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)

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

- Normal Force Unit (NFU)
- Optics (behind the NFU)
- Lateral Force Unit (LFU)
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

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

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)

- 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

Piston pin (DLC coated)

Cam follower (CrN + DLC coated)

Chain pin (DLC coated)

Note: DLC = Diamond-like Carbon
Typical application

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

- Application at automotive supplier
- Left: 3D-picture of the needle tip by the use of a white light Interferometer
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
Investigation of the adhesion strength of fibers in a composite material.

- Example: Composite industry
- Used in: Research

Source: Dr. Müller, UNI Augsburg

Einzelfaser-Push-out Versuch
Many thanks for your attention