

## Instrumented Indentation Set-ups for Determination of Hardness and Other Plastic-elastic Materials Characteristics (Test Force < 1 $\mu$ N to 1000 N)

### Key words

Instrumented indentation test, hardness, modulus of elasticity, plastic-elastic behaviour, visco-elastic and visco-plastic behaviour, parts of plastic and elastic deformation

### Fields of application

Development and certification of reference materials and coatings; reference procedure for mechanical property testing; homogeneity testing; stability testing

### Methodology and instrumentation

**Testing Methodology:** Instrumented indentation test according to DIN EN ISO 14577 Parts 1-3 (2003) and Part 4 (2007) and ISO/TR 29381 (2008)

**Test equipment:** Hardness heads with force transducers up to 200 N (Z005) and 2500 N (4S) and incremental displacement sensor (resolution 20 nm), Microindenter Fischerscope H100 (max. test force 1 N), Nanoindenter XP (max. test force 500 mN), Nano DCM (max. test force 15 mN)

### Items tested

Thin films (thickness > 5 nm), compound materials, composite and bulk materials

### Quantities / characteristics tested

Indentation depth  $h$  ( $h > 0.01 \mu\text{m}$ ) as a function of testing force  $F$  using an indenter of well defined shape (e.g. Vickers pyramid) that is punched into the surface of a (uncoated or coated) testing item. By means of a model, material quantities such as Martens hardness, indentation hardness, and elastic indentation modulus are derived. Testing forces between 1  $\mu\text{N}$  and 1000 N allow the determination of film quantities for thicknesses beyond 5 nm and of bulk quantities.

### Uncertainty / reliability of results (Test force 10 $\mu\text{N}$ to 1000 N)

Martens hardness $HM$ :	2 N/mm <sup>2</sup> to 5 x 10 <sup>4</sup> N/mm <sup>2</sup>	from 1.0 %	to 1.5 %
indentation hardness $H_{IT}$ :	20 N/mm <sup>2</sup> to 10 <sup>4</sup> N/mm <sup>2</sup>	from 1.0 %	to 3.0 %
indentation modulus $E_{IT}$ :	10 <sup>3</sup> N/mm <sup>2</sup> to 5 x 10 <sup>5</sup> N/mm <sup>2</sup>	from 1.0 %	to 5.0 %

### Qualification and quality assurance

Nationally unique combination of different equipment with overlapping measuring ranges into one test system with a test force range covering 8 orders of magnitude.  
Long-term experience, leading cooperation in the development of the respective international standard.

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## Further information

### Principle of the instrumented indentation testing

An indenter of well defined shape (e.g. Vickers-pyramid) is punched into the surface of a test item. While testing force  $F$  is being increased, indentation depth  $h$  is being measured continuously both for loading and unloading sequences. As a result and under given testing conditions, a functional correlation between  $F$  and  $h$  typical of the materials is determined.

From this experimental force-indentation depth-curve, the following quantities can be derived: Martens hardness  $HM$ , ( $HMs$  from the slope of the force-indentation depth-curve: hardness vs. indentation depth) indentation hardness  $H_{IT}$  (for several materials there is a close correlation between indentation hardness and Vickers-hardness), indentation modulus  $E_{IT}$  (this modulus may be compared with the modulus of elasticity of the material under investigation), creep behaviour, relaxation behaviour, plastic and elastic parts of indentation work.

Using the unique possibilities of a combination of different testing machines in a force range from  $< 1 \mu\text{N}$  up to 1000 N mechanical properties can be investigated in the micro-, macro- and nano-range.

Contrary to visual inspection under a microscope (off-line with respect to indentation), error sources such as relaxation and visual inspection are avoided in case of instrumented indentation test as the measurement of indentation in course of indentation is realised fully automated and on-line. This circumstance is a prerequisite for the investigation of thin film materials.

### Requirements on testing items

Smooth surfaces of sufficiently low roughness ( $< h/10$  to  $h/100$ ) without foreign particles. Testing and bearing surfaces have to be even and parallel to each other. Usually, the determination of thin film quantities requires a film thickness ten times higher than indentation depth  $h$ .

The requirements on reference plates for the instrumented indentation test are given in DIN EN ISO 14577 Part 3 (2003).

### Accuracy of the testing equipment

Requirements on the testing equipment and on the calibration are described in DIN EN ISO 14577 Part 2 (2003).

The testing equipment in use meets all these requirements. For several important parameters even much better values are proved:

- The tolerance of the determination of testing force  $F$  (max. deviation of the actual value determined by calibration from the nominal value) for  $1 \mu\text{N} < F < 1 \text{ N}$  is smaller than 0.1 % (required  $< 1.5 \%$ ).
- The error of the determination of indentation depth  $h$  measurement (max. deviation of the actual value determined by calibration from nominal value) for  $0.005 \mu\text{m} < h < 10 \mu\text{m}$  is smaller than 1 % (required  $< 1 \%$ rel.).
- The repeatability of the testing equipment (variation coefficient of the hardness measured on a certified reference sample) is smaller than 1 % (required  $< 5 \%$ ).
- The error of the testing equipment (difference between the arithmetic mean value of hardness calculated from 5 individual measurements on a certified reference sample and the certified hardness of the reference sample) is smaller than 3 % (required 5 %).

The figure on the next page shows the good reproducibility of the different testing machines.

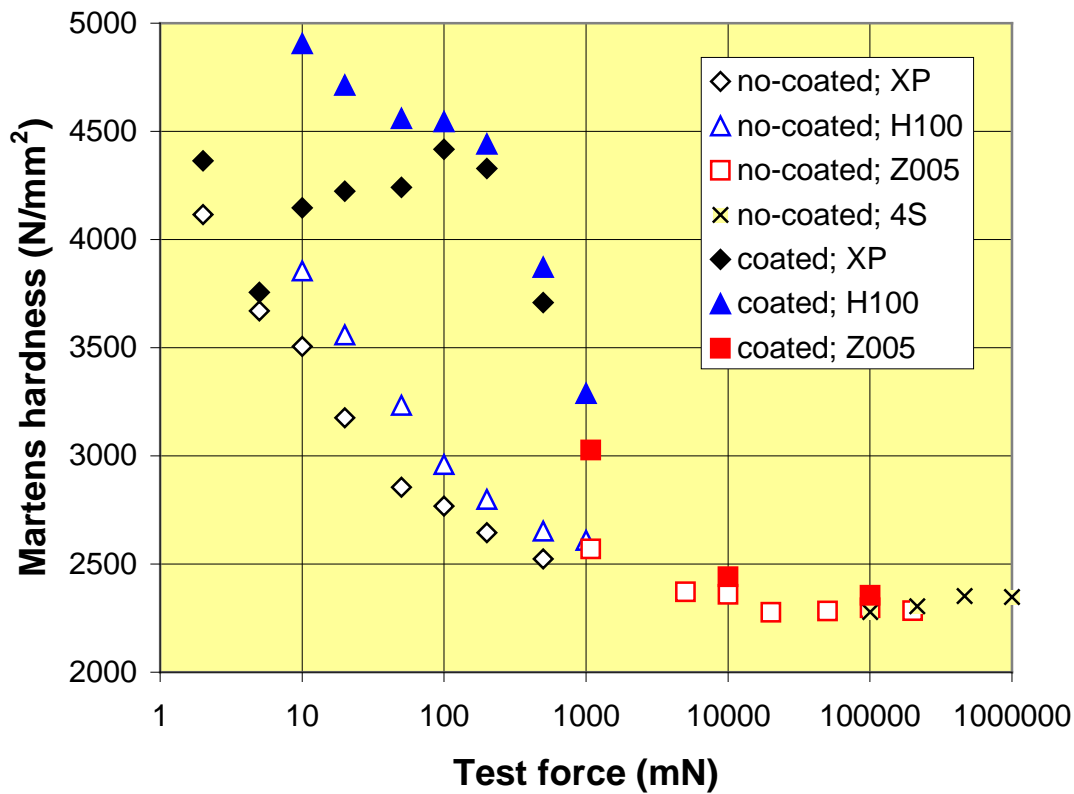
### Uncertainty of testing results

In general, the uncertainty of the result depends on the accuracy of the testing equipment, on the properties of the testing item, and on the testing range as well. Surface roughness has a strong influence at all. Uncertainties of the results mentioned before refer to a certified reference sample.

### Further developments

BAM is performing many pre-normative activities for further development of the test procedure:

- Procedures for determination of machine compliance and measurement uncertainty comparison of Vickers-hardness and indentation hardness
- Determination of E-modulus of films with a thickness < 10 nm
- Development of layered reference materials (VAMAS TWA 22 activity; round robin BAM, NIST, AIST)
- Co-working in revision of DIN EN ISO 14577 Parts 1-3



Martens hardness of coated and no-coated steel samples in dependence on test force. The use of four testing machines of different construction allows the measurement over a test force range of six orders of magnitude so that the substrate properties of the coated samples can be estimated, too.