

## Microhardness Product Line

Measurement Systems for Nanoindentation



**fischer**<sup>®</sup>



 Coating Thickness



 Material Analysis



 Microhardness

## Knowledge, Competence, Experience

Since 1953, FISCHER has created and produced increasingly innovative, powerful and versatile technologies for measuring coating thickness and microhardness, as well as for material analysis and testing. Today, FISCHER instruments are used all around the globe – wherever trueness, precision and reliability are essential.

## Research and Development

Building leading-edge products requires a strong focus on research and development. All FISCHER products are developed and manufactured at the company's facilities in Germany, where one in five employees works in R&D.

Highly qualified specialists – with advanced degrees in physics, chemistry, electronics, engineering and computer science – continually develop new products and processes to meet the ever-changing demands of the market. FISCHER also cooperates closely with universities and research institutes.

## "Made in Germany" Quality

Keeping its manufacturing predominantly in-house allows FISCHER to fulfil its customers' expectations with truly superior products. In FISCHER's modern, high-tech production facilities, close attention is paid to even the tiniest of details in order to ensure consistently high quality. Here at FISCHER, "Made in Germany" is more than just a merchandise mark: it is a point of employee pride and an integral part of the company philosophy.

## Product Assortment

FISCHER instruments cover a comprehensive range of measuring and analysis tasks that are relevant in diverse industries. For each application, the appropriate method is employed for maximum precision and accuracy: whether magnetic induction or eddy current, beta-backscatter, coulometry, microhardness or x-ray fluorescence – FISCHER always has the right technology for the purpose. Industrial enterprises, research centres and academic institutions worldwide depend on the reliability and accuracy of their instruments. FISCHER meets this challenge with its rigorous quality standards and commitment to develop and produce the most technically advanced – yet easy to use – measurement systems and software on the market.

## Pioneer in Microhardness Measuring Technology

A true pioneer in measuring technologies, FISCHER recognised very early on the immense potential of Instrumented Indentation Testing for determining microhardness. The first commercially-produced microhardness test instrument to employ this method, the FISCHER-SCOPE® H100, was launched onto the market in 1985; many of these first-generation instruments are still being used today.



 Material Testing

# Instrumented Indentation Test – Determining Mechanical Properties in the Nanometre Range

The exacting requirements of today's advanced surface technologies require increasingly more powerful measurement techniques to characterise the various coatings and materials. Regardless of whether the coatings are very hard, very thin, or viscoelastic – FISCHER provides the optimum measurement system for practically every application with its range of high-precision and user-friendly instruments for Instrumented Indentation Testing.

In addition to Martens Hardness, characteristics such as the Elastic Modulus of Indentation, Creep, and other material parameters can be determined reliably. The main areas of application are testing paint layers, electroplated coatings, hard material coatings, polymers, metals and coatings on glass. FISCHER's microhardness measurement systems excel where classic methods reach their limits: measuring quickly, precisely and effectively – in the nanometre range.

## Standards for Hardness Measurements

**1987:** First efforts to standardise the hardness measurement under load. Seminal work by W. Weiler and Helmut Fischer on the topic of "Microhardness measurements at the push of a button"

**1999:** DIN Standard 50359-1 to 3, with significant contributions from FISCHER

**2000:** International Standard DIN EN ISO 14577. Participation in Standards Committee: FISCHER

**Current:** Revision of the Standard DIN EN ISO 14577-1 to -3 with significant cooperation by FISCHER

## Instrumented Indentation Test

All FISCHER instruments employ Instrumented Indentation Test method – often called nanoindentation – for determining the Martens Hardness (HM). In contrast to other hardness tests, this method not only determines the plastic behaviour of the material, but additional material parameters can also be read from the measurement plot, such as the Elastic Modulus of Indentation ( $E_{IT}$ ), the Indentation Hardness ( $H_{IT}$ ) and the Indentation Creep ( $C_{IT}$ ), as well as the plastic and elastic deformation energies.

## Operating Principle of the Instrumented Indentation Test

In the Instrumented Indentation Test, an indenter penetrates the specimen's surface using a specified load. During this process, the depth of the indentation is measured continuously. Because of their high resolutions for load ( $\leq 100$  nN or  $\leq 400$  nN) and distance (40 to 100 pm), FISCHER microhardness instruments can be used for a broad range of applications and materials. The Instrumented Indentation Test can determine material properties on thin coatings, for example on sensors, on glass or on data storage media, and even on very soft materials such as elastomers.

This is where the classic Vickers Hardness measurement method quickly reaches its limits: where the indenter is pressed into the specimen under specified conditions and the geometry of the resulting indentation is measured optically. The hardness value obtained using the Vickers Method is only a measure of the plastic material properties and does not contain any information about the elastic properties.



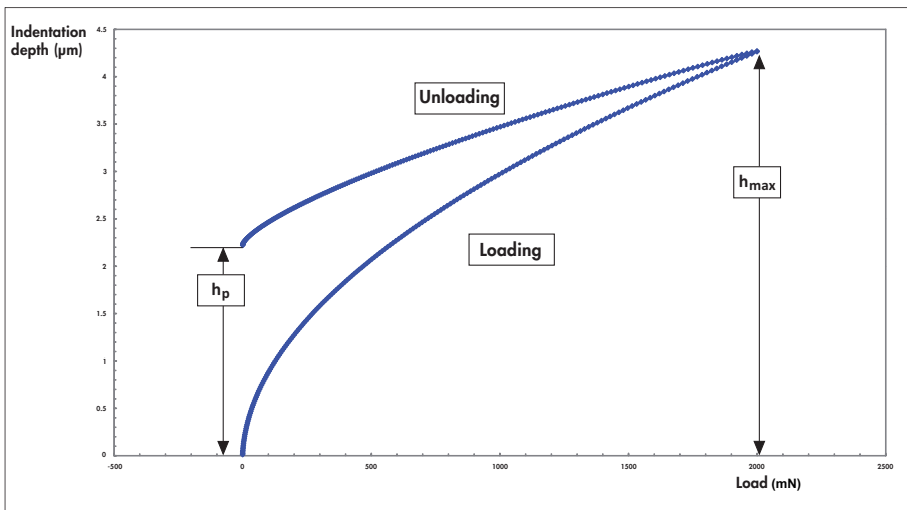
**1985 FISCHERSCOPE® H100:**  
First microhardness test instrument



**1999 FISCHERSCOPE® H100 C:**  
Newly designed, with improved distance measurement, higher load resolution, integration of XY-control, new software



**2004 FISCHERSCOPE® HM2000 and PICODENTOR® HM500:**  
Wider load range (HM2000) and increased vibration stability for industrial applications



$h_{max}$ : Maximum indentation depth during the test

$h_p$ : Displacement at the end of the test procedure, plastic portion

Instrumented Indentation Test sequence

#### Advantages of the Instrumented Indentation Test

- Measures elastic and plastic material properties in a single pass
- Measures thin coatings without any influence from the substrate material
- Precludes operator errors through partially automated measuring
- Provides depth-related information about the material characteristics

Additionally, all instruments feature ESP (Enhanced Stiffness Procedure). This allows for the depth-related determination of  $H_{IT}$  and  $E_{IT}$ .

#### Measurable Material Characteristics

Measurement and computation of material characteristics according to DIN EN ISO 14577-1 and ASTM E 2546:

- Martens Hardness  $HM$
- Indentation Hardness  $H_{IT}$  (convertible to  $HV$ )
- Modulus of Indentation  $E_{IT}$
- Indentation Creep  $C_{IT}$
- Percentage of the Indentation Energy  $W_{elast}/W_{total}$  attributable to Elastic Deformation  $\eta_{IT}$
- Additional characteristics such as the Martens Hardness at a specific test load, plastic deformation energy, etc.



**2011 New generation of instruments** feature a compact and dimensionally stable design, high-precision, programmable XY-stages, motor-driven Z-axes and improved optics



**2013 Continual enhancements**, such as the integration of an AFM (Atomic Force Microscope) or a heated sample support, make these measurement systems ever more powerful and versatile



The compact **FISCHERSCOPE® HM2000 S** is the cost-effective entry model for determining the microhardness of coatings in the micrometre range, i.e. for coatings thicknesses greater than 1 – 2  $\mu\text{m}$ . Samples with a simple shape are positioned manually. A twist prevention mechanism on the stand ensures safe sample positioning while its stable construction, with stone (cf. granite) base, help avoid measurement errors and reduce influences of e.g. vibration. The instrument can be equipped with a programmable positioning aid for

accommodating more challenging specimens or for taking semiautomatic measurements. In addition to the Martens Hardness, other plastic and elastic properties can be determined, such as indentation hardness, modulus of indentation or creep.

The HM2000 S is used by preminent vehicle and paint manufacturers in both development and quality control. It is also used frequently in the electroplating industry or for testing hard material coatings.



*Determining the influence of weather on paint coatings*



*Determining the mechanical characteristics of lacquer coatings*



*Testing the wear resistance of thin anodic coatings on extruded aluminium profiles*

### Characterisation of Hard Anodic Coatings

Hard anodic coatings are notable for their excellent abrasion and wear resistance as well as for their imperviousness to corrosion. For this reason, they are used for finishing automotive parts such as pistons, cylinders and gears. Using the HM2000 S, the wear resistance of hard anodic coatings can be determined easily via mechanical properties – not only in the laboratory but even in running production lines.

### Mechanical Characterisation of Paint Coatings

Paints and lacquers find use in a broad range of applications – e.g. to finish metal parts used in household equipment or in the automotive and electrical industries – not only for protection and decoration but also for functional purposes, such as changing surface properties like electrical conductivity. Using the Instrumented Indentation Test to determine the plastic and elastic properties of a paint coating allows conclusions to be drawn about the layer's hardness, its elasticity, the degree of cross-linking and level of resistance to UV radiation. If different paints are to be compared against each other, the same test conditions must be met for all indentation tests – which is quick and easy to accomplish with the HM2000 S.

To test for the impacts of environmental influences such as temperature fluctuations, humidity and aggressive substances, the painted parts are subjected to various conditions using accelerated weathering equipment. Environmental exposure increases the hardness of the paint surface and reduces its elasticity; such effects can be verified easily with the HM2000 S.



Outdoor weathering site for testing how paint reacts to environmental influences, Picture credit: Atlas Material Testing GmbH

#### Features

- Quick measurements without extensive sample preparation, thus suited for both lab and production checks
  - User-friendly; easy to position samples manually
  - Intuitive operation with the WIN-HCU® software
- Optional:
- Enclosed measurement chamber to prevent external influences such as breezes from open windows, fans, laboratory hood, air conditioning, etc.
  - Additional stone plate with anti-vibration feet to further reduce the influences of vibration

#### Typical Fields of Application

- Measurements on specimens with simple shapes
- Paint, plastic or hard material coatings (PVD, CVD)
- Electroplated coatings (decorative, functional)



Hard material coatings on tools



Determining the properties of paints on various materials



The **FISCHERSCOPE® HM2000** is the measuring instrument of choice for determining the microhardness of coatings in the micrometre range, i.e. for coatings thicker than 1-2  $\mu\text{m}$ . Measurements can be taken reliably even on soft materials like rubber or polymers. The device's natural stone base (granite or similar) ensures excellent dimensional stability, is resistant to temperature swings and also reduces the effects of vibration. The programmable XY-stage and the motor-driven Z-axis facilitate semiautomatic measurements on multiple samples with high throughput and easy handling. Its built-in

microscope with three magnification settings makes the HM2000 suited for demanding measuring applications with difficult positioning.

The instrument's modular design allows for customer-specific configuration and for later upgrades, e.g. adding even higher-resolution optics or a measuring stage with greater repeatability precision. It is also possible to equip the system with an Atomic Force Microscope (AFM) or a heated sample support for temperature-independent measurements.



*Hardness determination of hard anodic coatings on pistons*

*DLC coating on a gear wheel*



## Electroplating and Galvanisation

The challenges faced daily by electroplating businesses are complex. While regulations require the replacement of tried-and-tested chemicals with more environmentally-friendly substitutes, the demands of industry on the coatings themselves only increase; processes must be modified and optimised according to these new requirements. So that uniform coating quality can still be ensured, the elastic and plastic properties must be monitored during the coating procedure. This can be done quickly and precisely with the HM2000 in both the lab and in running production.



Electroplating facilities are typical areas of application for the HM2000

## Hard Material Coatings

The expectations placed on the tools used in industrial production also continue to grow. New substrate materials (such as high alloy steel) and faster cutting and processing speeds require new hard coatings that are applied, for example, using PVD (Physical Vapour Deposition). Typical hard materials for this purpose include TiN, TiAlN and CrN – materials that exhibit Vickers Hardness in the range of about 2000-3000 HV0.05 – and coating thicknesses of about 1-15 µm.

Quality assurance for coated tools depends on reliable determination of their mechanical properties. Conventional hardness test instruments are only partially suited to this task because the test loads they work with are too high: The indenters penetrate the top layer and the hardness measured is actually a mix of the coating and the substrate. To determine the hardness of just the coating, the indentation depth must not exceed one tenth of the overall coating thickness (Bücker's Rule). The HM2000 is ideally suited for measuring coatings thicker than 1 µm.

### Features

- Quick measurements without extensive sample preparation, thus suited for lab and production checks. The HM2000 requires only 30 seconds for its travel to the measuring position and the zero point determination.
- Programmable XY-stage for automated measurements with a re-positioning accuracy of  $\pm 2 \mu\text{m}$
- User-friendly handling through motor-driven Z-axis
- Microscope with three different magnification settings for accurate positioning on the measurement location
- Natural stone base provides dimensional stability, prevents temperature swings and buffers against vibrations

### Optional:

- Active anti-vibration table and enclosed measurement chamber to further reduce the influence of vibrations
- Intuitive handling with the individually configurable software WIN-HCU®

### Typical Fields of Application

- Paint, plastic or hard material coatings (PVD, CVD)
- Electroplated coatings (decorative, functional)
- Materials specifically for medical applications
- Electronic components, bond wires, etc.
- Automated measurements on multiple samples



Electroplated coatings



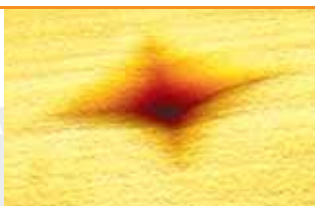
Microhardness determination on a gear housing



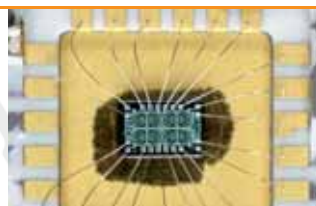
The **PICODENTOR® HM500** is optimised for Instrumented Indentation Testing on layers in the nanometre range, i.e. for coatings less than 1 µm thick. Besides the proven stone base (granite or similar), the standard fittings on the measurement system include a high-precision programmable XY-stage, a fast microscope with three different objectives, an active piezo-based anti-vibration table as well as an enclosed measurement chamber.

Thus, the PICODENTOR HM500 is suited for sophisticated measuring applications such as the determination of plastic and elastic properties on polished micro-sections or on bond wires in the semiconductor industry.

Additionally, for an exact characterisation of sample surfaces, the measuring system can be equipped with an Atomic Force Microscope (AFM), making even small material changes in the nanometre range visible.



*Indentation left by a Vickers diamond*



*Microhardness determination on bond wires*

## Plug Contacts

Important properties of plug contacts such as abrasion resistance or bondability can be determined using the Instrumented Indentation Test. The contact areas are plated with gold (depending on the alloy, the Martens Hardness ranges between 1200 and 6000 N/mm<sup>2</sup>) very selectively and, to control costs, less than 1 µm in thickness. The measuring task is challenging in many ways: the extremely thin coating requires a very delicate indenter touchdown on the sample for setting the initial zero point, which is the foundation for correct measurement results. At the same time, the small dimensions of the plug contacts require extremely precise positioning.

Using the PICODENTOR HM500, even such demanding measuring applications can be solved with the highest degree of accuracy. Repeated measurements of the Martens Hardness on 0.2 µm thick gold coatings can achieve a coefficient of variation smaller than 5%.

## Plastic Optical Lenses

Plastic lenses in eyeglasses are coated with multiple layers of various thickness in the nanometre range to attain a surface that is scratch-resistant, dirt repellent and anti-reflective. Testing the mechanical properties of such thin coatings requires a measuring system with high-precision distance measurement in the picometre range and load generation restricted to just a few micro-Newtons.



Testing the protective coatings on plastic optical lenses

## Features

- Quick measurements without extensive sample preparation, thus suited for both lab and production checks: requires only 60 seconds to travel to the measuring position and determine the zero point
- Measurements even on the smallest structures due to a high-precision XY-stage with a re-positioning accuracy of  $\leq 0.5 \mu\text{m}$
- User-friendly handling through auto lens recognition and motor-driven Z-axis with auto-focus
- Microscope with three different magnification settings for accurate positioning of the measurement location
- Natural stone base provides dimensional stability, prevents temperature swings and buffers against vibrations
- Active anti-vibration table and enclosed measurement chamber to further reduce the influence of vibrations
- Intuitive operation with the individually configurable software WIN-HCU®

## Typical Fields of Application

- Hard material coatings and ultra-thin DLC coatings
- Dirt-repellent coatings (e.g., Sol-Gel coatings)
- Coatings on PC hard disks / CDs
- Super-thin paint coatings
- Ion-implanted surfaces
- Nano-coatings on sensors
- Implants / medical applications
- Matrix effects in alloys
- Biological materials
- Ceramic materials
- Hardness determination on polished micro-sections
- Automated measurements on multiple samples



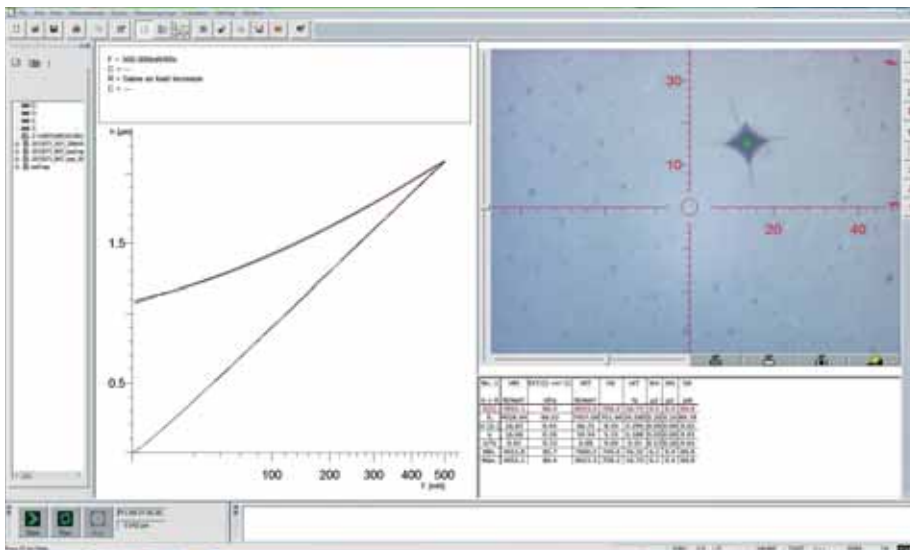
MICROHARDNESS



Contacts on a printed circuit board



Automated measurements on a wafer



The main WIN-HCU window shows a video image of the measurement location (except with the HM2000 S), a graphical presentation of the current measurement and a table with the current measurement parameters

All FISCHER hardness measuring instruments are supplied in conjunction with a Windows® PC on which the WIN-HCU® software is installed. The software controls the instruments and evaluates the signals resulting from the measurement procedure. The readings are displayed on the monitor and saved to memory. Using WIN-HCU, the results can be formatted and printed conveniently as paper records, and they can be exported to other programs (e.g. Excel). The software is intuitive to use and can be configured individually, for example, to set up users with different permission levels. Furthermore, in addition to German and English, Chinese and Japanese are available as operating languages. The software is based on the DIN ISO 14577-1 and ASTM E 2546 standards.

### Programmable Test Cycle

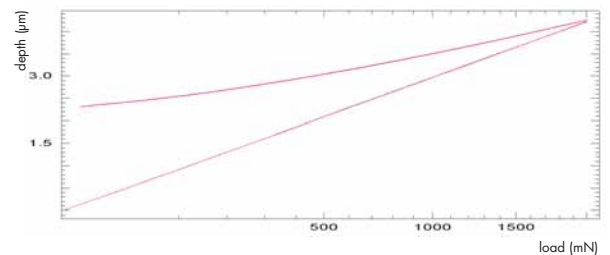
The parameters for the test cycle can be defined individually, e.g.:

- Maximum test load
- Load reduction, creep
- Loading and unloading duration

This way, the user can easily define measurement sequences and obtain significantly more information about a material than with conventional hardness measurement systems. The settings can be stored so that the measurement can be repeated later with identical parameters, for example to test different materials.

### Measurements According to the Standard Method

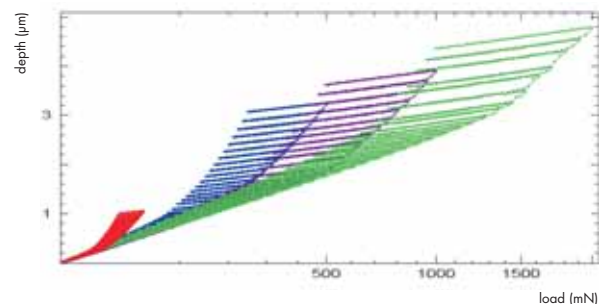
When measurements are taken according to the standard method, the indenter presses into the specimen under a defined load for a specified length of time (load/force increase) and is then unloaded again over another specified time period (load reduction).



Indentation depth / load diagram for a measurement using the standard method

### Measurements According to the ESP Method

When measuring according to the ESP method (Enhanced Stiffness Procedure), loading and unloading are incremental. This allows the quick for depth- and force-dependent determination of characteristics such as  $E_{IT}$ ,  $H_{IT}$  or HV – all at the same location.



Indentation depth / load diagram for a measurement using the ESP method

### Statistics and SPC Chart

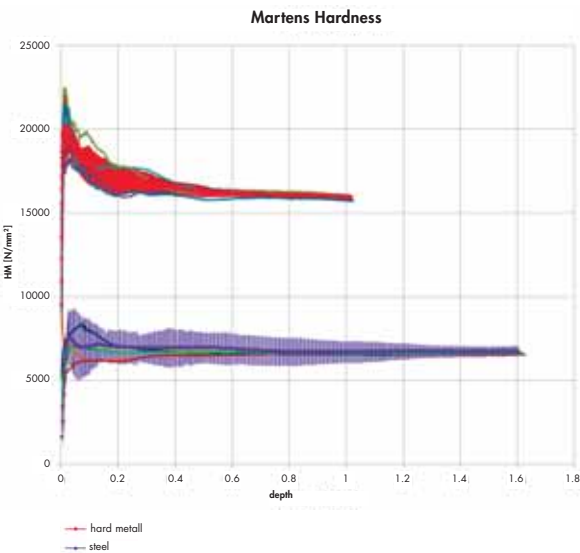
The WIN-HCU software computes such statistical characteristics as mean value, standard deviation, coefficient of variation, expected value, maximum, minimum, and range, among others.

For a quick and concise quality check, e.g. of a coating process in running production, WIN-HCU offers presentation of the measurement results in an SPC control chart with control limits (UCL and LCL) drawn in. In addition, the specification limits (USL and LSL), and the computed process capability factors  $C_p$  and  $C_{pk}$ , are given as numeric values. The group size can be adjusted.

### Graphical Presentation

The software makes it possible to plot the measurement results with any definition and scaling of the diagram axes, e.g.:

- Indentation depth / load diagrams
- Hardness / load diagrams
- Load / time diagrams
- Mean value plots including standard deviation
- Comparison of different test series within one chart



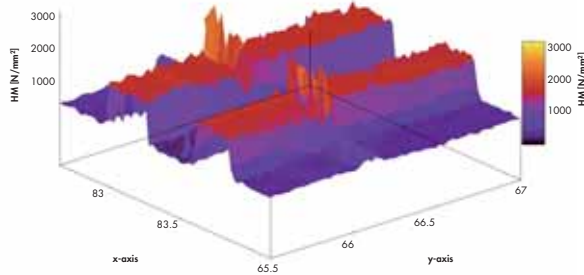
Martens Hardness indentation depth diagrams of hard metal and steel

### Determination of the Vickers Hardness (HV) and Knoop Hardness (HK) via Optical Measurements

Besides computation of the Indentation Hardness ( $H_{IT}$ ), the Vickers Hardness can also be directly measured optically. A special indenter also allows for determination of the Knoop Hardness (except with the HM2000 S).

### 3D Presentation

In addition to 2D graphs, the measured data can also be presented in 3D, which depicts the individual quantities along a line or across an area.



3D presentation of the Martens Hardness measured on the polished micro-section of a cardiac valve

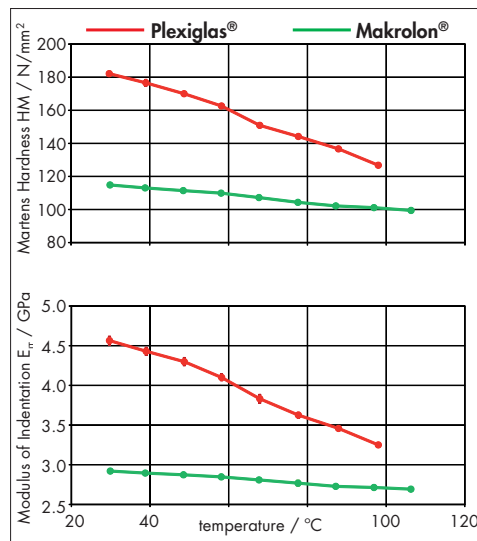
### Correcting for Deviations in the Shape of the Indenter

Deviations in the shape (line of conjunction, tip radius) of the indenter can be detected by WIN-HCU through a simple investigation with the correction function. Two methods are available for this purpose: determination of the shape correction via the material constant (Modulus of Elasticity) or by using the HM (Martens Hardness) characteristics based on a reference material (e.g. BK7).



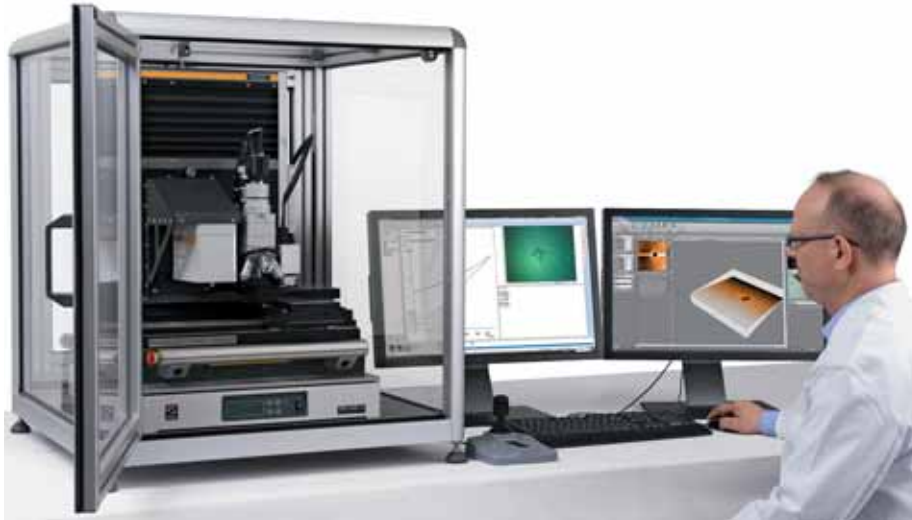
### Temperature-Dependent Measurement

Temperature plays an especially significant role when analysing the mechanical properties of plastics and paint. In combination with the heated sample support SHS200, the software makes it possible to measure and present temperature-related changes in the characteristics.



Temperature-dependent HM and  $E_{IT}$  of Plexiglas® and Makrolon®

## AFM (Atomic Force Microscope)



PICODENTOR® HM500 with measurement chamber and atomic force microscope

### Visualising and Quantifying Structures in the Nanometre Range

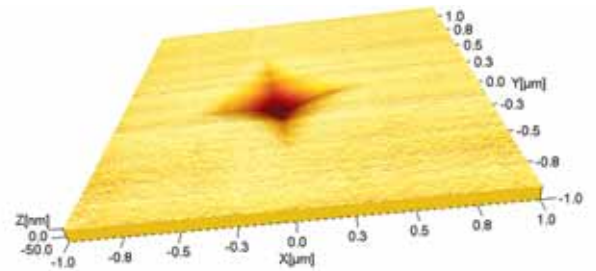
To obtain further information about the material properties, the measuring systems can be optionally upgraded with an Atomic Force Microscope (AFM).

The programmable XY-stage with its repositioning accuracy of  $\leq 0.5 \mu\text{m}$ , the active anti-vibration table and the enclosed measurement chamber offer the ideal conditions for additional AFM measurements.

An AFM physically scans the sample surface. For this purpose, the AFM utilises a cantilever with a very fine silicon tip to measure differences in height. The measurement area is scanned line by line and the height information is recorded point by point with high precision. The resolution in the XY-direction is about 10 nm.

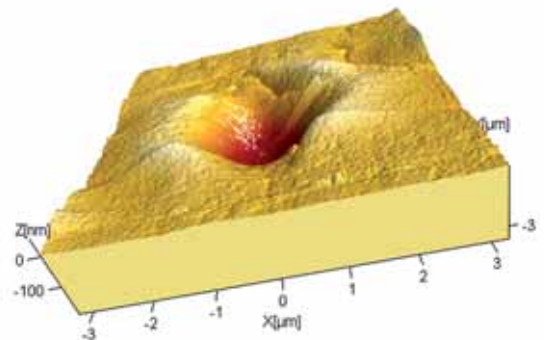
The obtained data can be presented in various ways: In addition to the surface topography, which shows the height profile, the AFM also offers the capability to determine the phase and amplitude of the cantilever's oscillations. These two parameters provide additional information on material properties. In particular, indentations made at the lowest maximum loads can be presented ideally using the AFM.

In the 3D presentation, the typical pyramid shape of the Vickers indenter is quite recognisable. Thus, the AFM offers the ability to observe otherwise invisible structures.



AFM measurement (3D presentation) of an indentation with a maximum load of 5 mN (scale z-axis: 50 nm)

The following example shows a measurement on tungsten ( $F_{\text{max}} = 50 \text{ mN}$ ). The material bulges at the edges of the indenter impression, creating a "pile-up". This behaviour is a known property of tungsten and other materials. Pile-ups increase the contact area at the Instrumented Indentation Test and thus influence the measurements.



Impression in tungsten ( $F_{\text{max}} = 50 \text{ mN}$ ) with clearly visible pile-ups



**A: HM Universal Sample Support incl. Heating Station and Heat Plate**

Sample support for measuring microhardness on up to four different specimens. The specimens are affixed thermally to the sample support.

**B: Universal Vice**

Vice for clamping specimens of different shapes.

**SHS200 Heated sample support**

Newer systems with a positioning aid can be upgraded with the heated sample support SHS200. This allows for analysis of the mechanical properties of various materials at temperatures of up to 200°C. The temperature is controlled very accurately by combining an internal sensor in the heater with a sensor directly on the sample.



**C: HM support for polished micro-section samples**

Support for polished micro-section samples with diameters of 20, 30, 40 and 50 mm and heights of 8.5 to 30 mm.

**D: HM foil clamping device**

Device for clamping thin foils or wires for secure, gap-free fixation.

**Indenters**

All instruments come equipped with Vickers indenters. Optionally available are also: Berkovich or Knoop indenters, hard metal spheres and, by request, custom-specific indenters.

**Sound insulation hood NOAH-M**

The sound insulation hood NOAH-M minimises the impacts of external sounds, allowing for more precise measurements on extremely thin coatings (< 1µm).



## FISCHER worldwide

Knowing what their customers need and want is a must for anyone trying to succeed in today's globalised markets. Because we at FISCHER think of ourselves as partners to our customers, we attach great importance to providing them excellent advice and working in close cooperation with them. This is why the Helmut Fischer Group maintains its worldwide presence through own companies and qualified distribution partners; there is one near you.

In keeping with our high standards of quality and customer satisfaction, all members of the Helmut Fischer Group are certified according to DIN EN ISO 9000.



### Service

Good service and efficient customer support are just as important to FISCHER as technically advanced and innovative products. For this reason, FISCHER has established a dense and tightly-linked global network of service partners staffed with highly qualified personnel. Offering extensive services such as setup, maintenance, training, calibration and so forth, FISCHER supports you in every aspect of your instruments and their use. This is how FISCHER guarantees the reliability and precision of its products. Worldwide.

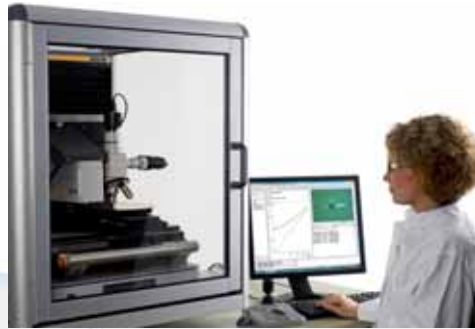




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### Application Laboratories

More and more, demanding applications require highly qualified application advice. FISCHER addresses this need through its strategically located Application Laboratories around the world (Germany, Switzerland, China, USA, India, Japan and Singapore).



### Training and Seminars

Because we want you to derive maximum benefit from our products, FISCHER's specialists are happy to share their practical know-how: starting with seminars and training sessions on metrological basics, through the optimal use of the instruments, to expert symposia on special topics.

**Helmut Fischer GmbH**  
**Institut für Elektronik und Messtechnik**  
71069 Sindelfingen, **Germany**



**Helmut Fischer AG und**  
**Helmut Fischer Technologie AG**  
CH-6331 Hünenberg, **Switzerland**



**IfG-Institute for Scientific Instruments GmbH**  
12489 Berlin, **Germany**

**Fischer Instrumentation (GB) Ltd**  
Lymington, Hampshire SO41 8JD, **England**



**Fischer Technology, Inc.**  
Windsor, CT 06095, **USA**



**Helmut Fischer S. de R.L. de C.V.**  
76230 Querétaro, QRO, **Mexico**

**Fischer Instrumentation Electronique**  
78180 Montigny le Bretonneux, **France**

**Helmut Fischer S.R.L.**  
20099 Sesto San Giovanni (Milano), **Italy**

**Fischer Instruments, S.A.**  
08018 Barcelona, **Spain**

**Helmut Fischer Meettechnik B.V.**  
5627 GB Eindhoven, **The Netherlands**

**Fischer do Brasil**  
04561-001 São Paulo, **Brazil**

**Fischer Instruments K.K.**  
Saitama-ken 340-0012, **Japan**

**Nantong Fischer Instrumentation Ltd**  
Shanghai 200333, **P.R. China**



**Fischer Instrumentation (Far East) Ltd**  
Kwai Chung, N.T., **Hong Kong**

**Fischer Measurement Technologies (India) Pvt. Ltd**  
Pune 411036, **India**

**Fischer Instrumentation (S) Pte Ltd**  
Singapore 658065, **Singapore**

**Helmut Fischer Korea Co., Ltd**  
Seoul City, **Republic of Korea**

**Fischer Technology (M) SDN Bhd**  
47301 Petaling Jaya, **Malaysia**

**Helmut Fischer Thailand Co., Ltd**  
Bangkok 10250, **Thailand**



[www.helmut-fischer.com](http://www.helmut-fischer.com)