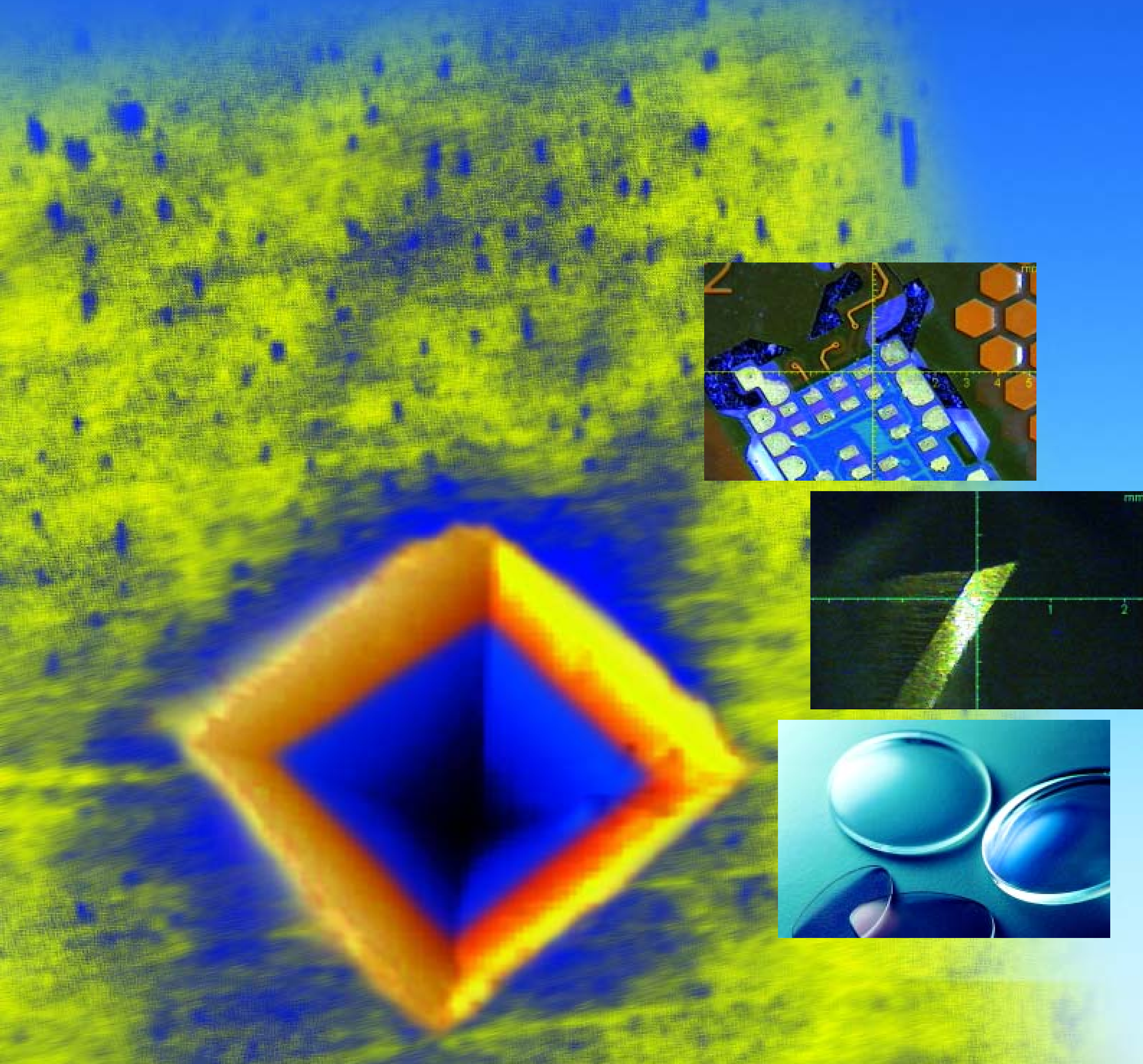
Path-breaking technology in the measuring microhardness and evaluating material properties

Microhardness measurement according
to ISO 14577-1 with the microhardness measurement systems
FISCHERSCOPE® HM2000 and PICODENTOR® HM500



Features, Measurement Principle

The increasingly stringent demands being placed on surface treatment technology has given rise to the need for measuring processes and devices that meet the new requirements. The microhardness measuring systems FISCHERSCOPE® HM2000 and PICODENTOR® HM500 succeed where the classic measuring processes reach their limits – it is fast, precise, user-friendly and effective.

Precise measurements

The specified test load is built up with high precision. The measurement of the indentation depth is carried out with a resolution in the picometer or nanometer range. The extremely sensitive touch-down of the indenter allows for the exact determination of the zero point. This is significantly below the measurement uncertainty prescribed by the standards (< 1 %). The tip roundness of the indenter is determined using a reference measurement and is taken into account accordingly in the results. The microhardness determination is computer-controlled, free of any subjective influences, and thus independent of the operator.

Meaningful measuring results

In one single measurement, the user obtains information about the hardness of the surface, and the hardness pattern within boundary layers near the surface, the elastic and plastic properties, as well as the creep properties of the material. Additional interesting characteristic qualities of the material, such as the modulus of indentation and the elastic-plastic energy portions can be computed from the recorded measurement plot.

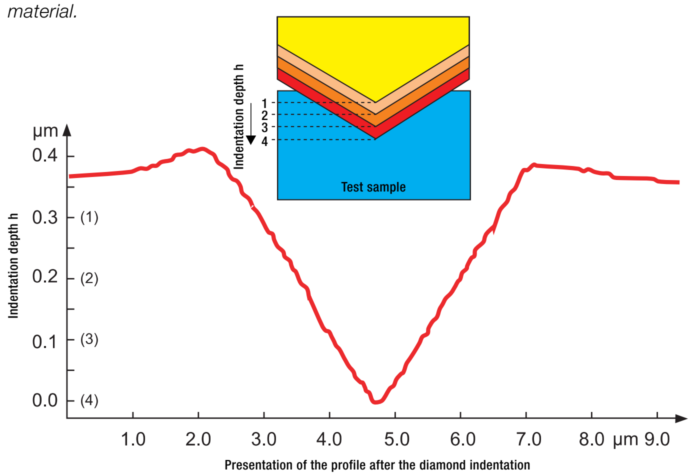
Measuring head

The measuring head contains the indenter, the test load generating unit, and the position measurement unit for determining the indentation depth, as well as the entire electronic system. The indenter is generally diamond pyramid as per Vickers with 136° plane angle, to meet stringent industry requirements. Indenters with a diamond pyramid according to Berkovich or with hard metal spheres are available as well. The measuring head is equipped with a special support ring in order to ensure damage-free touch-down on the specimen surface.

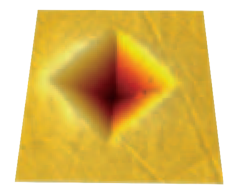
Measurement principle

The microhardness measurement systems FISCHERSCOPE® HM2000 and PICODENTOR® HM500 utilize the load/indentation depth method according to ISO 14577-1. With this method, the indenter – typically a Vickers or Berkovich pyramid – is essentially continuously pressed into the material tested with an increasing test load, and then unloaded. The respective indentation depth is measured at the same time. Taking into account the geometric relationship between the indentation depth and the shape of the indenter, this measurement produces the physically meaningful Martens hardness HM = F/A, where A = f(h) = the surface of the penetration. Important characteristic technological quantities can be obtained from the resultant load/unload cycle. The previously common universal hardness can also be computed from the recorded measurement plot.

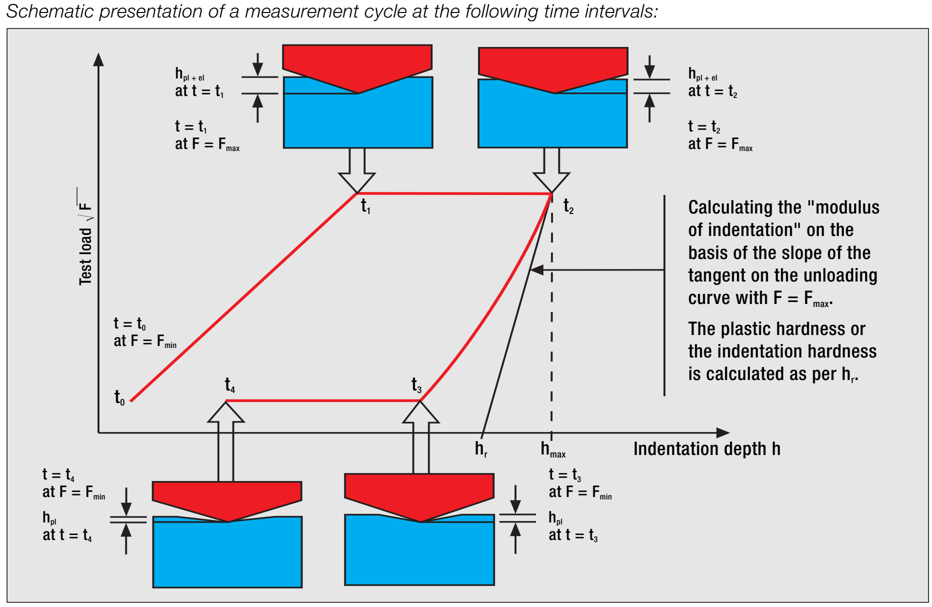
Schematic presentation of the penetration of a diamond indenter into the material.



Penetration of a Vickers diamond in silicon (Atomic Force Microscope recording).



Schematic presentation of a measurement cycle at the following time intervals:



t_0 - t_1:
Application of the test load from the minimum to the maximum levels. The plastic and elastic deformation contribute to the hardness indentation. The hardness calculation is carried out only in this time interval.

t_1 - t_2:
The maximum test load is kept at a constant level for a duration that can be pre-selected. The change in indentation depth indicates the creep properties of the material.

t_2 - t_3:
Unloading of the test load. The "modulus of indentation" is calculated from the slope of the curve at F_{max}.

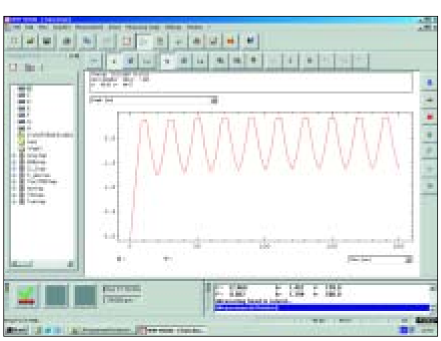
t_3 - t_4:
The minimum test load is kept at a constant level. On the basis of the relaxation found, conclusions can be drawn about, the effect of damage on paint (reflow behavior).

Software WIN-HCU®

The WIN-HCU® software provides all functions for controlling the measuring head. The software enables the user to simply and quickly adjust all the measuring parameters. It also enables the display, evaluation and documentation of measurement series. The WIN-HCU® software also controls the functions of the optional positioning devices. The WIN-HCU® software operates under Windows® beginning with version 98SE or NT and higher. The user-friendly software, which can be individually configured, allows even semi-skilled users to make measurements. If required, e.g., for faster familiarization, Fischer offers individual training for the WinHCU® Software both at the customer's site and Fischer headquarters, or at the respective country's representatives.

Freely programmable test cycle

The test parameter settings such as load or depth control for the hardness penetration by specifying the max. load or indentation depth, the desired times for loading and unloading as well as the hold duration at test load can be stored in a separate file as well and can, in this manner, be recalled at will.
In addition, any measurement sequence can be defined in Microsoft® EXCEL spreadsheets, e.g., cyclical loading and unloading with the same or a different final load. This may provide more information about a material than a conventional hardness test sequence.



Measuring plot of measuring process with cyclic loading and unloading with the same ultimate force.

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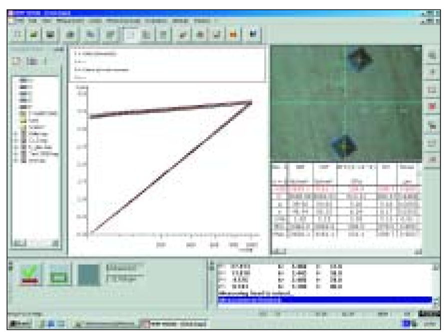
Accounting the indenter shape

Accounting for indenter shape deviations according to applicable standards (top edge, rounding-off of peak values) through simple, automatic determination of correction functions.

Measurement screen

Several simultaneously open information windows enable an overview of all relevant information:

- Online representation of the measuring process. Video image of the measuring point (only for measuring systems with positioning device).
- Table showing the selected material characteristics with statistical evaluation.
- Directory structure of the application and measuring plot files.



Measurement screen.

Measurable characteristic material quantities

Material characteristics computed according ISO 14577-1:

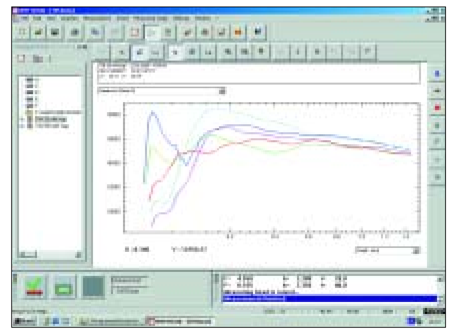
- Martens hardness HM
- Indentation hardness H_{IT} (convertible to HV)
- Modulus of indentation E_{IT}
- Indentation creep C_{IT}
- Percent elastic portion η_{IT} of the indentation work W_{elast}/W_{total}
- Additional characteristic material quantities according to standard
- Quantities such as the Martens hardness at a certain test load, portion of plastic deformation, etc.
- Characteristic quantities computed from the measurement plot according to the previous DIN 50359.

Vickers hardness

In addition to the conversion of the indentation hardness (H_{IT}) into a Vickers value according to ISO 14577-1, a direct measurement of the hardness penetration according to the "classic" Vickers method is possible as well (applies only to measurement systems with positioning device).

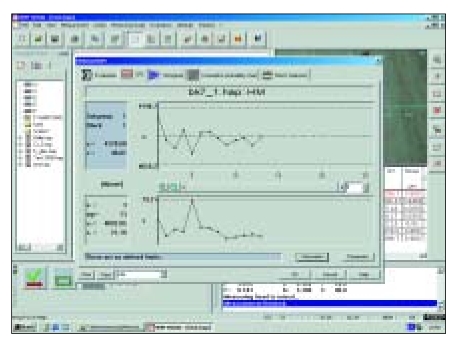
Evaluation

Graphical representation of all measuring results on a color screen, indentation depth-load, hardness-load, load-time diagrams with selectable definition and scaling of the diagram axes. Computation of the mean value plots and adding of measurement plots from other measurement series for comparison purposes.



Measurement plots with mean value plots of hard material coatings in the evaluation window.

The material characteristic values determined, can be represented in tabular form, as well as in a process control chart, in order to check the quality of, for example, a coating process. Presentation of the measurement data as a histogram or in the normal probability chart is also possible.



SPC chart representation.

Data export

Test load, indentation depth and the statistical values can be exported either into Microsoft® EXCEL® spreadsheets or as ASCII files.

FISCHERSCOPE® HM2000

FISCHERSCOPE® HM2000, measurement systems for the determination of the hardness, the elasticity or the viscoelastic behavior of materials.

Applications

- Paint and lacquer coatings, polymeric materials, e.g., in automobile manufacturing
- Electroplated coatings, e.g., thin gold or other electroplated coatings on components of the electrical and electronics industry
- Hard material coatings (PVD, CVD)
- Anodic coatings
- Implants, e.g., artificial heart valves, hip joints, etc.
- Synthetics and rubber, e.g., to determinate the influences of radiation or the effects of filler and hardener proportions
- Materials research, e.g., the matrix of fiber-reinforced synthetic materials. Determination of properties of metals.
- Anodic coatings on aluminum.

Technical data

Measuring head

- Maximum test load: 2000 mN
- Maximum indentation depth: 150 µm
- Load resolution: ≤ 0.04 mN
- Distance resolution: ≤ 100 pm
- Approach speed of the indenter: ≤ 2 µm/s

Positioning device

- Video microscope with 40x, 200x and 400x magnification
- Travel:
  Manual measuring stage: 25 x 25 mm
  Prog. measuring stage: 100 x 100 mm

Measurement system versions

Depending on the measurement system version, the control and evaluation unit is a correspondingly configured PC with the WIN-HCU® software installed. In connection with a positioning device, it also assumes control of the latter. Easy calibration and maintenance-free operation make the FISCHERSCOPE® HM2000 a very economical measurement system.

Three different instrument models enable the user to select the version that is most suited to his needs and budget:

FISCHERSCOPE® HM2000 S

Support stand design. The standard version includes support stand, a measuring head and a PC with the pre-installed WIN-HCU® software.

Support stand design. The FISCHERSCOPE® HM2000 S constitutes the most price conscientious measurement system design.

An optional vibration damper system is available for all three versions.

FISCHERSCOPE® HM2000 XYm

Model with positioning device. It is equipped with a video microscope and an XY stage with manual travel. The microscopic image of the specimen surface appears in the video image on the PC monitor allowing for a precise travel to the desired measurement position. To make measurements, the stage travels automatically under the measuring head such that the hardness indentation occurs exactly at the desired position. In addition to the positioning device, the standard shipment of this instrument version also includes the measuring head and a specially configured PC with the pre-installed WIN-HCU® software.

Positioning device with XY stage with manual travel on the vibration damper system available as an option.

FISCHERSCOPE® HM2000 XYp, total measurement system with PC, keyboard and positioning device with a programmable measuring stage on the optionally available vibration damping system.

FISCHERSCOPE® HM2000 XYp

Model as FISCHERSCOPE® HM2000 XYm, however, with a programmable XY stage; it is particularly suited for fully automatic measurements at pre-programmed measurement spots on the specimen. The programmable measuring stage offers many options for travel to the measurement spot(s):

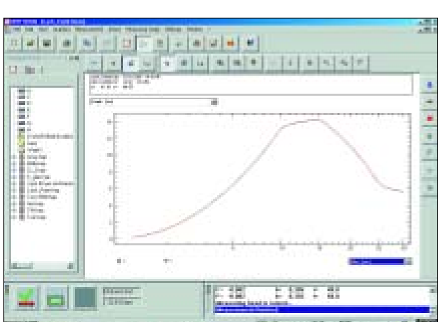
- Direct approach of the measurement position with the click of the mouse in the video image, the so-called "Point & Shoot" function.
- With the joystick of the keyboard.
- Programming of several measurement spots: Distributed as desired, in a straight line or in a rectangular field (array).

FISCHERSCOPE® HM2000, Applications

Paint and lacquer coatings

Various painted surfaces from the automotive field.

In order to obtain a durable protective action, paint coats must be sufficiently thick and must also have specific mechanical properties. In the lacquer and polymer industries, increasingly material parameters such as surface hardness, degree of crosslinking, reflow behavior or modulus of elasticity are considered important quality features. In reality, however, most of these characteristic quantities can be determined only through very elaborate and time-consuming trials. With the FISCHERSCOPE® HM2000, these values or similar ones of the same kind that allow for drawing conclusions about the desired characteristic material quantities are often available after only one minute of measuring time. In this manner, a comparison of several chemical process parameters can be presented quickly and effectively. Hardness measurements using the FISCHERSCOPE® HM2000 provide information about the degree of polymerisation, changes in hardness due to temperature influences, brittleness arising due to UV radiation, the alternation of viscoelastic properties due to weather influences, scratch resistance and other characteristics of coatings.

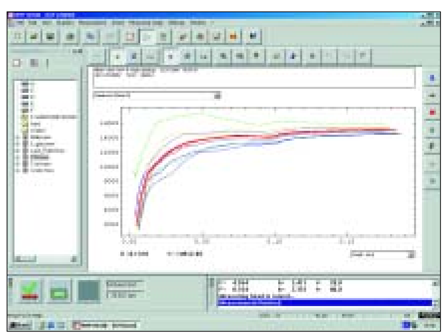


The pattern of the indentation depth over time is indicated. In order to determine the viscoelastic properties (creeping) of the paint, the test load was held at a constant level for 5 seconds at maximum as well as minimum load.

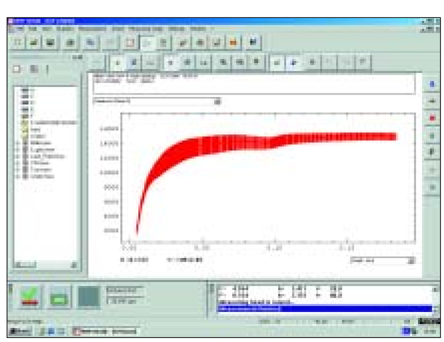
Electroplating

Electroplated parts are used in many areas. The figure shows a small selection of electroplated parts.

The microhardness provides information about the functional effectiveness of electroplating layers. For example, connectors have contact surfaces selectively plated with gold (the HM is between 1200 and 6000 N/mm^2), for cost reasons, layer thicknesses of up to 0.8 µm are utilized. A microhardness measurement allows one to draw conclusions about the abrasion resistance and the bonding capability.



The results of the hardness measurement on a 20 µm chromium layer on steel are shown graphically. The red curve represents the mean value from eight individual measuring series. There is a relatively wide dispersion on the measurement values for small indentation depths, due to the rough surface of the test sample, however the mean value is very stable beyond 0.1 µm.

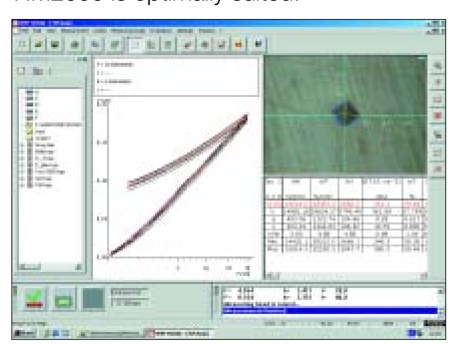


Other presentation of the measurement results. Shown is the mean value plot with the display of the 95 % confidence interval. The mean value plot has been generated from the 8 individual measurement series.

Hard material coatings

Milling, cutting and drilling tools with differing hard material coatings.

It is well-known that hard material coating layers improve the performance of deformation and metal removing tools. The wear resistance arises due to the high level of hardness in the layers that are applied, for example, 20,000 to 25,000 N/mm^2 for TiN. This corresponds to a Vickers hardness of about 2000 to 3000 HV0.05. Quality assurance in the case of tools require reliable measurement of microhardness. Conventional hardness testing devices are only partially suitable for this purpose, since they use excessively high testing forces. The testing bodies pass through the layers, and measure a mixed hardness consisting of the protective layer and the substrate. In order to reliable determine the hardness of the layer, the indentation depth must not exceed 1/10th of the layer thickness – a range for which the FISCHERSCOPE® HM2000 is optimally suited.



Measuring results of a 3 µm thick hard material layer applied to steel. It can be seen that the curve turns upward with rising load (bottom part) at around 0.2 – 0.3 µm. This indicates an incipient effect on the softer substrate material. The high values, typical for hard material layers, of indentation hardness of almost 29,000 N/mm^2 as well as the indentation modulus of 475 GPa (only around 200 GPa for conventional steels) must also be considered.

An additional interesting characteristic material quantity is the elastic deformation portion of the indentation work (W_{elast}/W_{total}).

PICODENTOR® HM500

The PICODENTOR® HM500 can be used to determine the Martens hardness HM, characteristic elastic quantities and additional material parameters, even in the nanorange, using the instrumented indentation test according to ISO 14577-1.
The instrument distinguishes itself by its simple handling and its excellent price/performance ratio. The achieved resolution and accuracy for the load and distance measurement is in the same range as that of instruments with a much higher purchase price. Through its practical design with programmable XY stage and clear presentation of the measurement results, in comparison to other instruments, the PICODENTOR® HM500 is not only suitable for lab applications but also for the production area.

Applications

- Hard material coatings, general
- Ultra thin DLC coatings
- Protective coatings on glass
- Soil-resistant coatings (sol-gel coatings)
- Coatings of PC hard disks and CDs
- Very thin paint coatings
- Ion implanted surfaces
- Nanocoatings for sensors
- Medical technology (Implants, etc.)
- Matrix effects in alloys
- Biological materials
- Ceramic materials

Technical data

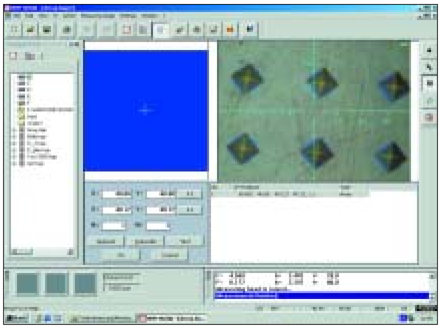
- Maximum test load: 500 mN
- Maximum indentation depth: 150 µm
- Load resolution: ≤ 100 nN
- Distance resolution: ≤ 40 pm
- Approach speed of the indenter towards the specimen surface: ≤ 100 nm/s
- Video microscope with 40x, 200x and 400x magnification
- Programmable xy measuring stage with a travel of 100 mm x 100 mm

The measurement system

PICODENTOR® HM500, complete measurement system with PC, keyboard and positioning device with a programmable measuring stage on the active anti-vibration table in the measurement chamber.

The control and evaluation unit is a specially configured PC with the installed WIN-HCU® software. In addition, the standard shipment of the measurement system contains the measuring head, the positioning device with the programmable measuring stage, the active anti-vibration table, the measuring box and the HCU keyboard. The microscopic image of the specimen surface appears in the video image on the PC monitor allowing for a precise travel to the desired measurement position. The programmable measuring stage offers many options for travel to the measurement spot(s):

- Direct approach of the measurement position with the click of the mouse in the video image, the so-called "Point & Shoot" function.
- With the joystick of the keyboard.
- Programming of several measurement spots: Distributed as desired, in a straight line or in a rectangular field (array).



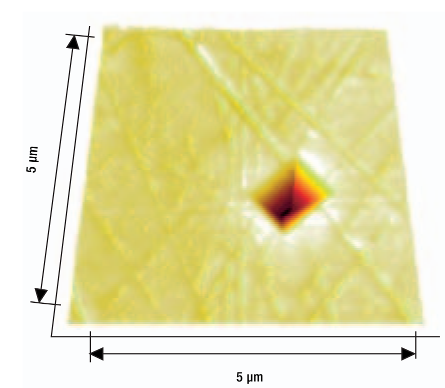
Program window for programming several measurement spots. Shown here is an array programming. The indentation after the measurement can be seen in the video image.

Coated wafer as the specimen under the video microscope for the accurate determination of the measurement position(s).

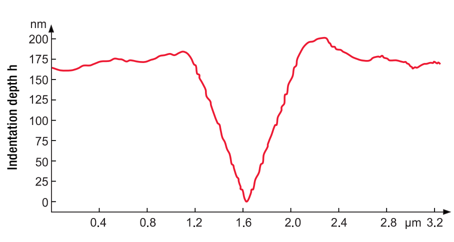
To make measurements, the measuring stage travels automatically under the measuring head such that the hardness indentations occur exactly at the selected measurement positions.

PICODENTOR® HM500, Applications

Coatings with thicknesses of only a few micrometers or even only a few tens or hundreds of nanometers are gaining in importance due to their excellent properties. Hard material coatings of TiN, TiC or diamond-like carbon with thicknesses of 1 to 4 micrometers are already common for tools and engine components. Highly complex coating systems in the nanometer range have been developed over the past years to achieve scratch-resistant, soil-resistant, antistatic, reflecting or storage-capable surfaces. The determination of the technological properties of such coatings is essential for their optimization.
The PICODENTOR® HM500 is the ideal instrument for these measurements. Through high-precision distance measurements in the picometer range and load generation down to a few micronewtons, the PICODENTOR® HM500 can be used – according to the load/indentation depth method – to characterize ultra-thin coatings or surface areas with regard to their mechanical properties.

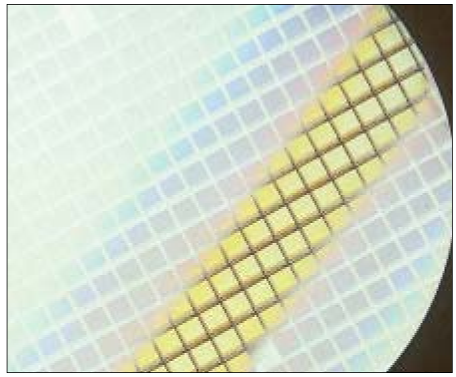


Penetration of a Vickers diamond in a silicon disc using a test load of 5 mN.



Profile of the diamond penetration in the material after the measurement.

Ultra-thin protective coatings



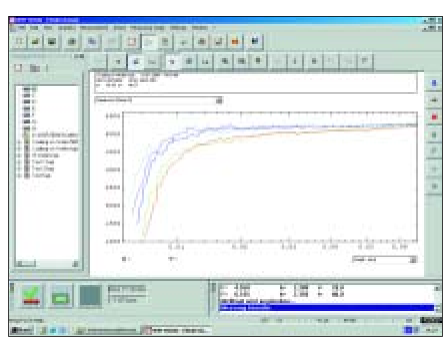
Coated wafer.

Microelectronics as well as the miniaturization of structures and components necessitate an instrumentation technology that adapts to these circumstances.

For example in the electronics industry, the printed conductors and their coating thickness are becoming ever finer and thinner.

Since the indentation depth of the indenter for hardness tests should only be at most 10 percent of the coating thickness, the test load must be reduced accordingly to a minimum.

Very thin, transparent nanometer coatings are applied to hard discs or CDs and DVDs to increase the wear protection and abrasion resistance.

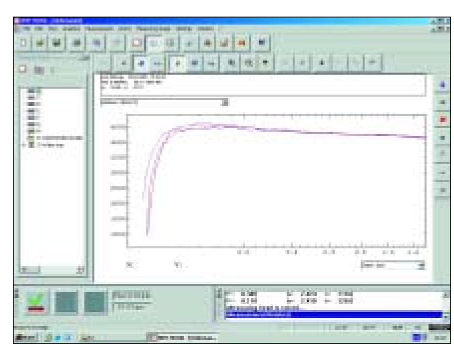


Here, the results of the hardness determination are shown on an about 400 nm thick silicon oxide coating on a wafer in the graphical evaluation mode.

Coated spectacle lenses

For spectacle lenses made of synthetic material, scratch-free protective coatings are indispensable.

As a rule, eyeglasses are used as visual aids and for occupational safety. Protective eyeglasses, for example, must exhibit a certain hardness to offer protection to the impact of chips of any material. When eyeglasses are used as visual aids, synthetic glasses are very popular due to their significantly lower weight and better breaking resistance in comparison to normal glass. Today's synthetic glasses receive several coatings of different thicknesses in the nanometer range to obtain a scratch resistant, soil resistant and anti-reflective surface. Testing the mechanical properties of such thin coatings requires a measurement system with a high-precision distance measurement in the picometer range and a load generation down to a few micronewtons.



Measurement on coated synthetic eyeglasses. The results are presented in the graphical evaluation menu. Measurements were made at the max. test load of 500 mN to demonstrate the influence of the carrier material. The evenly declining plot progression shows the elastic behavior of the carrier material during the penetration into the coating. During the measurement, the coating material presses elastically into the carrier material as if it were a pillow. Conclusion: For the carrier material to be without influence on the results, it is recommended to measure in this case with a test load of about 0.5 mN, which corresponds to a max. indentation depth of about 150 nm, to obtain meaningful results.

Active around the world

The Institute for Electronics and Measurement Technology HELMUT FISCHER in Sindelfingen/Germany is an innovative leader in the field of coating thickness measurement, material analysis, microhardness testing, electrical conductivity- and ferrite content measurement as well as for density and porosity testing. The company is able to recommend the best solution for any application. A comprehensive range of products is offered using X-ray fluorescence; Beta-backscatter; Magnetic; Magnetic induction; Electric resistance; Eddy current and Coulometric techniques. HELMUT FISCHER has 13 subsidiary companies and 33 marketing agencies strategically located around the globe.

The high quality standard of FISCHER instruments is the result of our efforts to provide the very best instrumentation to our customer. If needed, we adapt the Win-HCU® Software to customer-specific requirements.

FISCHER is a reliable and competent partner, offering expert advice, extensive service, and training seminars for the WIN-HCU® Software as well as our other FISCHER products.

Today, FISCHER instruments are used successfully in all technological fields of industry and research.

FISCHERSCOPE® X-RAY XDL®-B to measure the coating thickness according to the X-ray fluorescence method.

The measuring system FISCHERSCOPE® MMS® PC integrated multiple measuring methods for coating thickness measurements and general test procedures of materials.

The information in this brochure contains only general descriptions and performance features that do not always apply as written, or that may be changed due to continuous development of the products. The desired performance features are binding only if they are expressly agreed upon in the contract.

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