CSM Technical Features

//// MICRO INDENTATION TESTER (MHT)

csm Instruments

www.csm-instruments.com

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//// Introduction

For the past twenty years, CSM Instruments and its mother company CSEM (Swiss Center for Electronics and Microtechnology) have been an active part of the development of nano and micro mechanical testing instruments for surface and coatings characterization. The CSM micro indentation tools are now at the forefront of the technology for characterization of materials at the nano- and micrometer scale. Most significantly, CSM's combination of high-performance depth-instrumented indentation in an integrated Platform configuration along with an AFM or confocal microscope commonly makes it the most useful indentation testing tool available for a laboratory. The instrument's unique referencing design allows for a number of beneficial features, including an open and accessible configuration, a continuous differential depth measurement, a greater thermal stability, and a fast indentation cycle. Combined with the automation abilities of the Open and Compact Platforms, the unique referencing design of the CSM Micro Indentation Tester provides great improvements to laboratory productivity and throughput.

The CSM Micro Indentation Tester (Micro Hardness Tester: MHT) has been used for a very wide range of applications. It represents a very efficient automated method of determining the hardness and elastic modulus values on precise positions on a sample surface. A variety of sample configurations and types of materials can be analyzed. The instrument has been used to quantitatively characterize the properties of bulk materials (soft and hard materials), polymer film systems, various layers and multi-phase alloys. Ceramics, metals and polymers have been largely investigated.

The MHT is equipped with a thorough, easy-to-use software package that allows the user to perform Micro Indentation tests in a wide variety of testing modes, including simple loading, multi-cycle loading (increasing or constant loads), automated matrices, and location-pinpointed testing.

An interesting recent development is the localized characterization of the mechanical properties of microsystems and MEMS devices and structures. Conventional indentation techniques provide a highly powerful method for measuring the load and depth response of bulk and coated materials, but can also be used to measure the mechanical properties of very small micro-machined silicon structures. Beam structures, such as accelerometers, need to be characterized in terms of the number of cycles to failure, the spring constant or the energy required to bend the beam by a required amount. Such localized testing needs to be adapted to work at various distances from the origin of the beam with a micrometer positioning accuracy. Initial studies have proved to be highly repeatable. This includes a variety of application areas, such as accelerometer beam structures, micro-switches and printer head components. In addition, localized testing of friction and wear in MEMS devices have been performed with the Scratch testing head (optional).

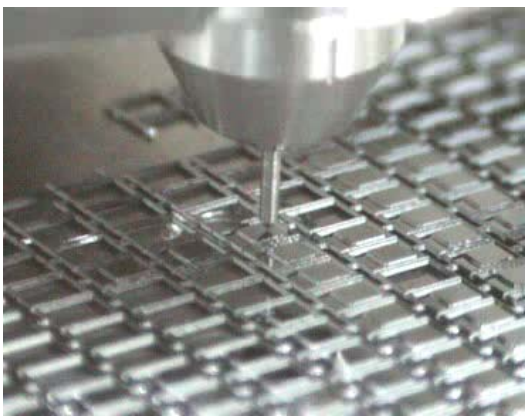


Figure 1. Functional indentation testing of accelerometer switch

//// Key Features

> Constant surface referencing

The design feature that truly sets the CSM Instruments Micro Indentation Tester (Micro Hardness Tester: MHT) apart from other instruments is its unique referencing system. As shown in the Figure 2 below, the design of the Micro Indentation Tester incorporates a reference point that encompasses the indenter and provides a constant reference for the amount of penetration made into the material.

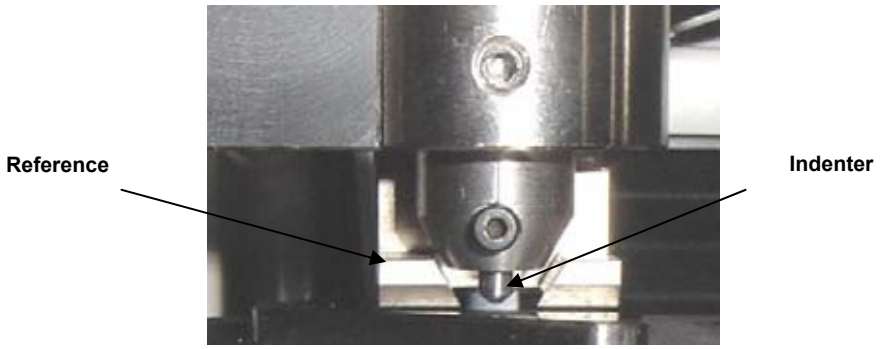


Figure 2. Surface referencing for the Micro Indentation Tester

Frame compliance refers to the amount of material composing the structure of an in instrument that can thermally contribute to geometric variations in shape or size of various components of a highly sophisticated instrument. These variations are negligible for most measurements but are detrimental on micro- and nano-scale measurements.

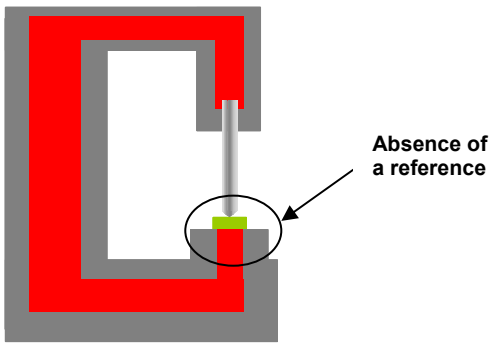


Figure 3. Conventional depth-instrumented indentation system frame compliance

The CSM Micro Indentation Tester takes the sample surface as its reference for measuring the penetration depth of the tip. The length of the system frame compliance, which corresponds to the distance from the sample surface to the depth sensor, is strongly reduced to a minimum.

The very small distance between the indenter tip and the depth sensor reduces the length of the system frame and provides low frame compliance for a high precision micro indentation instrument. The sample surface is referenced continuously throughout the measurement. The CSM Micro Indentation Tester (MHT) is the only system available which compensates for the sample compliance during testing.

> Instrumented Micro Indentation measurement

The Micro Indentation Tester uses the instrumented indentation measurement for the determination of the mechanical properties (Hardness, Elastic Modulus, Creep, …etc.).
During an indentation, the force and depth are continuously monitored. Therefore, a curve of force vs. depth can be displayed.

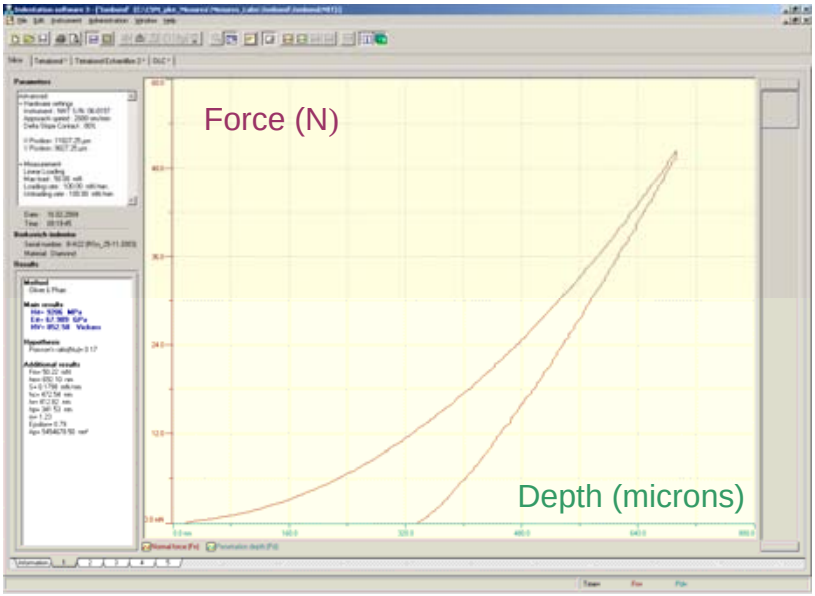


Figure 5. Indentation curve: force vs. depth

The dynamic measurement provides two important materials properties:

- **Hardness (H_{IT}, Hv)**
- **Elastic modulus (E_{IT})**

Other conventional micro hardness testers measure only the hardness with the recording of the diagonal! This method is highly users dependent and unusable on thin coating (the residual imprint is too small).

With the CSM MHT, there are multiple measurement modes, which provide important information on materials properties:

- Hardness and elastic modulus as a function of depth
- Fracture toughness, cracks formation
- Creep behavior, evaluation of strain-rate sensitive materials
- Better understanding of coating/substrate interactions

> Continuous multicycles (CMC) mode

The indentation software offers many measurement capabilities, including the unique Continuous Multi Cycles (CMC) mode.

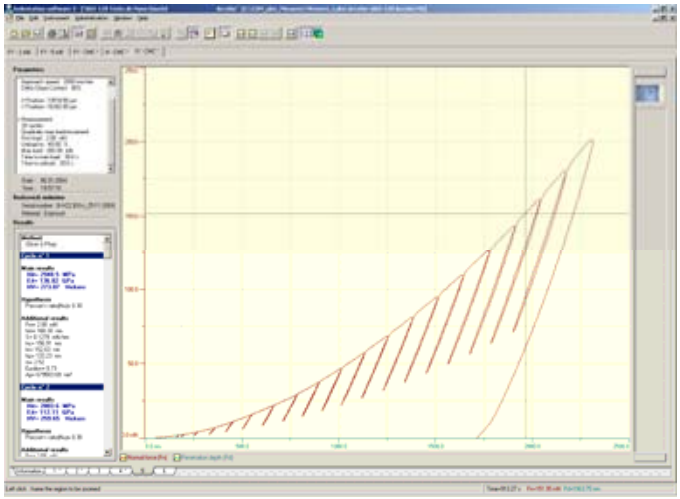
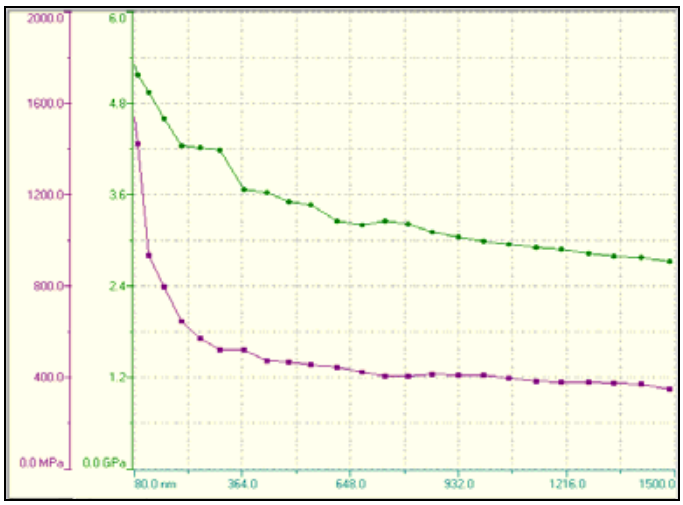


Figure 6. Continuous Multicycles Modes (CMC) method

Different cycles of micro indentation curves can be performed at increasing loads and depths.
The partial unloading curves allow a direct calculation of the hardness and elastic modulus.

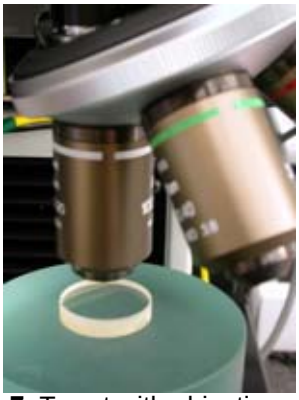
The indentation hardness, H_{IT}, the elastic modulus, E_{IT}, and the stiffness, S, can be displayed as a function of the penetration depth.

The variations of the mechanical properties (hardness and Elastic's modulus) with depth are easily observed.



Hardness and Elastic Modulus vs depth

> High quality optical imaging



Our integrated microscope system consists of a turret having high-quality Nikon objectives and a USB 2.0 video camera rather than a single-objective microscope of lesser quality. When imaging an indent, the ease in changing from a magnification from x200 to x2000 (objectives x5 to x50 or higher) becomes ever-more important for testing throughput. Our combination of automated sample platform, ease in changing tips, and flexible microscope makes the system the most favourable instrument available for a laboratory with different testing needs.

Figure 7. Turret with objectives

> Positioning synchronization and visual indentation

The modules (testing and imaging options) are “positionally synchronized” to each other, meaning that the position of each tip and focus point is precisely calibrated to the locations of the others. This method of configuration provides you with an unmatched level of flexibility and throughput from the system, as a single sample (or multiple samples) can be tested, imaged, and analyzed very quickly.

With a single click, the location of an indent can be exactly positioned under the optical microscope, ConScan or AFM for subsequent imaging. The CSM method of module integration also allows you to pre-select very specific locations for testing. For example, you can center an area of interest under the crosshairs of the video microscope, click a button in the software, and that location will be moved under the testing module of choice, here the Nanoindentation head.

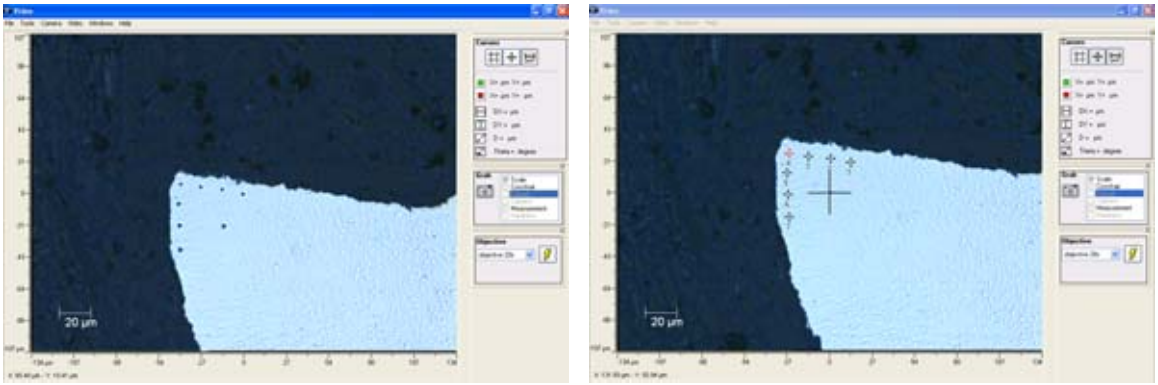


Figure 8. Sample with indents before and after tests observed under the optical microscope

> Easy testing with the mapping mode

The MHT can be used in a mapping mode to automatically take data from a variety of locations on your sample. A matrix of measurements can be set up so that each indentation is performed at a specific location with different conditions. The instrument can also be equipped with various sample holders such as a 300-mm wafer chuck to fit your specific needs.

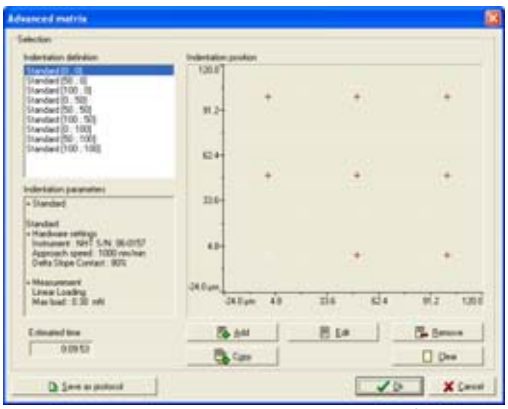


Figure 9. Matrix of measurements with mapping model

> Modular system

The modular system of the Compact and Open Platforms can include different measuring heads, which can be installed together with the Micro Indentation head or at a later stage. Additional possible measuring heads are as follows:

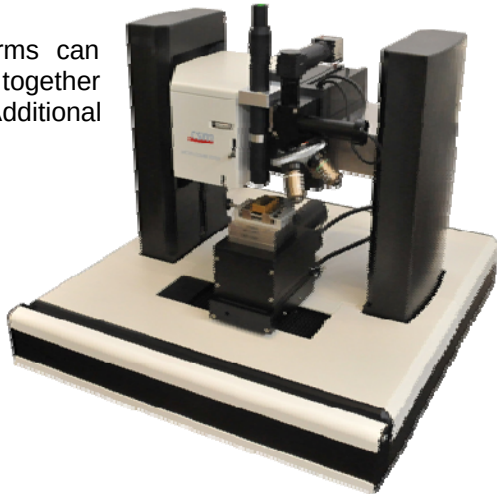
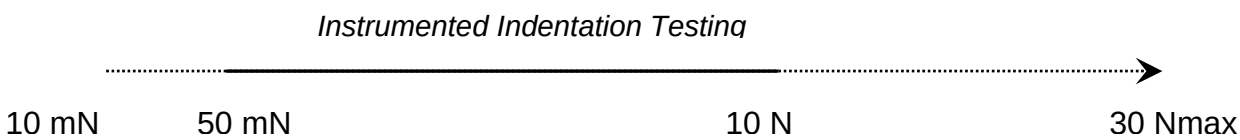


Figure 10. The CSM modular platform

- Ultra Nanoindentation
- Nanoindentation
- Micro Scratch
- Micro Combi (Micro Scratch + Micro Hardness) with an additional 3D imaging head:
- AFM objective
- ConScan objective (confocal-based objective)

> Large range of forces: from nano to macro-indentation

The MHT easily applies forces from 10 mN to 10 N with a load resolution of 0.3 mN. An indentation with a maximum load of 30 or 50 mN can be performed. This force range largely covers a great part of Nanoindentation experiments. Indentations can also be performed up to 10 N in depth-instrumented indentation testing (or to 30 N in maximum load with optical hardness measurements). This 30 N force range covers very well the range of Macro indentation experiments.



The CSM MHT is therefore a multipurpose indenter system that would provide useful information for all kinds of materials.

> Easy and ultra fast change of tips

In Micro Indentation experiments, the different studies on materials properties or MEMS devices testing may require different tip geometries: Vickers indenter for micro hardness tests or fracture toughness, spherical indenters for stress-strain studies, flat punch indenter for MEMS testing in fatigue and failure analysis or particles crushing, among other things.

The Swiss design of the Micro Indentation head brings great reliability and robustness to the instrument, as well as an unmatched ease-of-use. Indenters are directly changed on the head and calibrations of new indenters are directly entered. The change of the indenter tip is quick and easy, requiring only the loosening of a set screw, as shown in the figure below. Tests can be performed with Vickers, Berkovich, Cube-Corner, Knoop and spherical indenters on the same sample.

Figure 11. Easy change of indenter tip with CSM MHT

The change of tips on the MHT is straightforward and elementary. The CSM indenter holder provides efficiency and rapidity.

> Application flexibility

In terms of overall use of the system, a CSM instrument provides an open, easy-to-use environment for testing. The system is not concealed within a cumbersome chamber; so many different sample geometries can be tested. We have a number of multi-sample holders available, and can assist you in developing a custom holder. A variety of scratch/indentation tips can be installed very easily on the system in less than three minutes. It is a very straightforward procedure, requiring little more that the loosening of a set-screw to change the tip. We offer more than 20 different tips, and can work with you to make custom tips as your needs require. The instrumental control and data acquisition is fully integrated within our intuitive and thorough software package. An in-depth summary of CSM's Xpert Indentation Software follows.

> Indentation testing and software normalized

The indentation software from CSM Instruments is normalized according the new international norm ISO 14577 of depth-instrumented indentation testing, where results are given in Indentation Hardness, H_{IT}, Vickers Hardness, HV, Martens Hardness or Universal Hardness, H_M, and Elastic Modulus, E_{IT}.

//// General Information

> Complete Micro Indentation Tester system includes

- CSM platform: Compact Platform or Open Platform configuration
- Micro Indentation head assembly with referencing system
- Vickers diamond indenter (premounted and calibrated)
- Optical imaging system with microscope, Nikon objectives and CCD USB2.0 camera
- Computer workstation (Dell Computer) with dual 17” monitors
- Data acquisition card
- Electronic control module
- Motorized sample manipulation (x, y, and z tables), sample stage, holder, accessories
- CSM Xpert Indentation software
- (with automated matrices and test protocols - see Indentation software functionalities below)
- CSM Video software
- Industrial joystick controller
- Integrated table

> Options

- Peltier heating module (110 °C)
- Heating module (450 °C)
- AFM Objective
- ConScan Confocal Objective
- Micro, Nano Scratch
- Nanoindentation

> Consumables

- Vickers indenter
- Berkovich indenter
- Cube-corner indenter
- Knoop indenter
- Spherical indenter (multiple radii available)

> Recommendations for use of the Micro Indentation Tester

- The Micro Indentation Tester should be placed in an environment isolated from mechanical vibrations. The optional anti-vibration table can be activated on the Compact Platform.

- Temperature-controlled room (preferable)

//// Micro Indentation Technical Specifications

> Normal load

Normal maximum load range *usable load range of depth-instrumented indentation*	**0.03 to 10 N**
Load resolution	**0.3 mN**
Maximum load	**30 N**
Minimum load	**10 mN**
Loading rate	**Up to 300 N/min**
Contact force hold time	**Unlimited**

> Penetration depth

	Standard	*High range * (optional)*
Maximum indentation depth	**200 µm**	1 mm
Depth resolution	**0.3 nm**	1.5 nm
Maximum indenter range	**1 mm**	

*** High range option: to be specified at time of order**

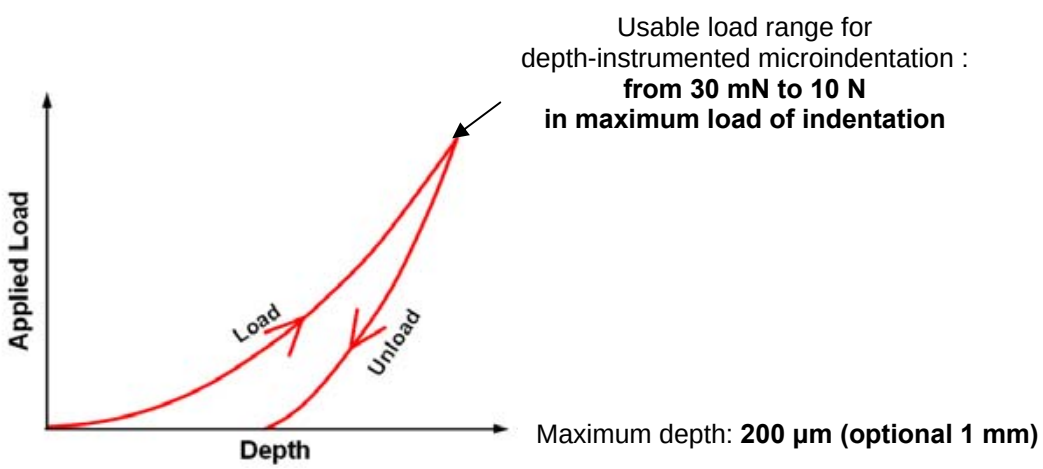


Figure 12. Schematic of a typical depth-instrumented indentation curve.

//// CSM Instruments Platform

CSM Instruments provides excellent configuration flexibility to grow with all customer's needs. Multiple testing and imaging modules could be installed together on the 2 CSM platform: "Compact Platform" or "Open Platform".

All the measurement and imaging modules are "Positionally Synchronized" to each other, the optical microscope being included as a standard module on the platform.

These platforms include a video microscope with at least 2 optical objectives and a video camera (USB2.0 camera).

(a) Compact Platform

(b) Open Platform

Figure 13. Indentation and/or Scratch CSM modules can be mounted on one of two platforms, depending on how many modules will be required. The Compact Platform (a) can accommodate 2 modules, whereas the Open Platform (b) can accommodate 3 modules total. A full video microscope comes standard on either platform.

//// Platform Specifications

CSM Instruments proposes two platforms of measurements: Compact Platform (CPX) and Open Platform (OPX) (please see Figure 1 for pictures of the platforms). The descriptions of the platforms are also available on a separate brochure.

Displacement tables	**OPX Range**	**CPX Range**	**Standard Resolution**	**High Resolution** (optional)
X	245 mm	120 mm	0.25 µm	0.1 µm
Y	120 mm	20 mm	0.25 µm	0.1 µm
Z	30 mm	30 mm	10 nm	-

Maximum sample size **X, Y and Z**	Compact Platform: 230 x 580 x 120 mm Open Platform: 250 x 480 x 120 mm
Usable areas of analysis **X and Y**	Compact Platform : 70 x 20 mm Open Platform : 195 x 120 mm
System Dimensions (with anti-vibration table)	Compact Platform: 600 x 600 mm, 1200 mm height Open Platform : 900 x 600 mm, 1200 mm height Total weight: ~ 150 kg

The usable areas of analysis indicate the areas of possible automated analysis in the X&Y directions. This area will also vary with the other modules installed (Scratch, AFM and/or ConScan).

> Positioning accuracy

- Standard resolution of X&Y tables: 0.25 µm per axis
- Repositioning accuracy of standard tables: 1 µm per axis

For the high resolution tables, please ask CSM Instruments for more details.
All table movements (X, Y, Z) can be controlled with Joystick.

> Platform table

The MHT is delivered on the Compact Platform table (see figure attached) with the optional activation of the anti-vibration table. The Open Platform already integrates the anti-vibration table.

Compressor for the standard active air table can also be ordered.

Optional environmental enclosure is not required under normal operation due to the top reference system design.

Figure 13. Compact Platform table

//// Indentation Software V4.0

The new software, V4, for Scratch and Indentation has been developed with completely new features in order to help our customers use our instruments more efficiently and accurately.
(The enhanced application standards and the obvious traceability of those are an entire part of our new development.)

> Full software package for data acquisition and analysis including:

> Complete measurements modes

- Complete control of indentation parameters
  (loading/unloading rate, maximum load, pauses, loading modes, …)
- Real time display of force and depth data during acquisition, with the possibility to initiate an instantaneous user controlled unloading during the experiment
- Fully user definable indentation modes (single/multi cycles, linear and square root loading ramps, unlimited hold time, user-defined load profiles)
- Powerful indentation modes including CMC™ (Continuous Multi Cycle) and large area mapping
- System setting programmable for every single indent in a multi-indent experiment
- Fully customized user access rights management
- Integration of the indenter tip calibration curve
- Automated positioning correlation between indentation and imaging analysis
- Full integration of AFM and Video imaging into the indenter control software
  (with AFM option only)
- MultiFocus Imaging: MultiFocus produce a pictures with a perfect depth of field

> Data analysis

- Powerful and fully integrated statistical module (data and graphical tools)
- Automatic measurement report generator, unlimited templates and test protocols
- Logging of all operations executed on the instrument
- Multi language support
- Data export in ASCII format top open files in Excel and Text software

> Mechanical properties results

- Automatic calculation of Hardness(H_{IT}), Elastic Modulus (E_{IT}) and Creep (C_{IT}), Recalculated Vickers (Hv) and Relaxation (R_{IT})
- Plastic and elastic parts of the indentation work
- Display of all indentation data results : maximum depth, contact depth, residual depth, projected contact area, stiffness (according to depth-instrumented indentation theories)
- Display of Hardness and Elastic Modulus vs. Depth
- Multiple models for hardness and elastic modulus evaluation
- Standard evaluation according to ISO 14577 and ASTM E2546

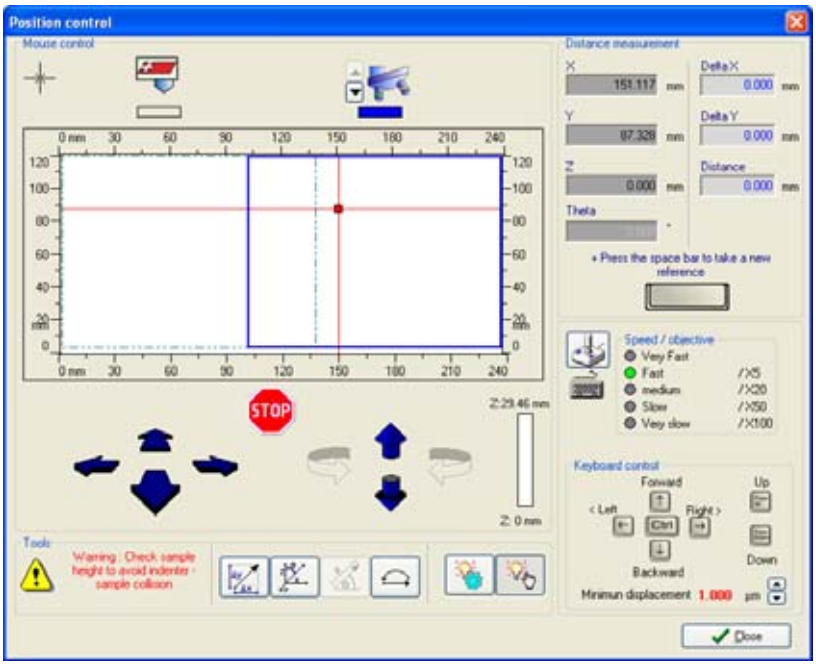
> Possible additional analysis for specific material properties

- Analysis of fracture toughness, creep and stress-strain behavior
- Analysis of plastic-elastic energy work
- ALSO INCLUDED: Material deformation modeling software
  (additional modeling software based on elastic contact mode: Hertz theory)

> General features (Windows Software)

> Positioning control

X-Y-Z Position control for sample positioning and motion between measurements

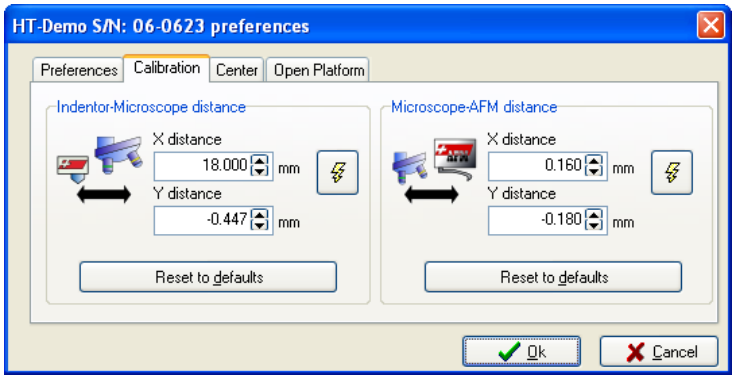


Sample positioning with industrial joystick, keyboard, mouse, table crosshairs, or vectors.

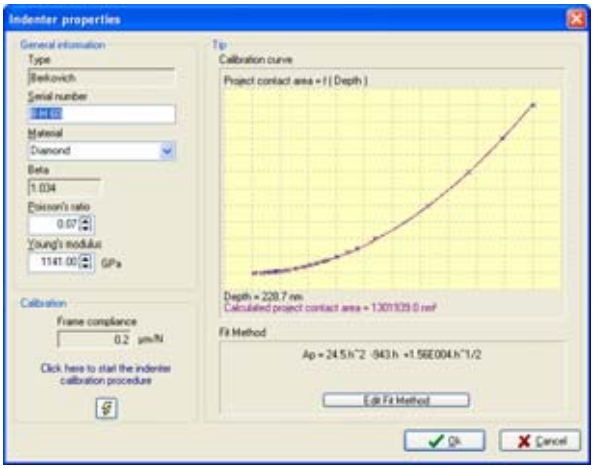
> Automated calibration procedures

Calibrating procedure:

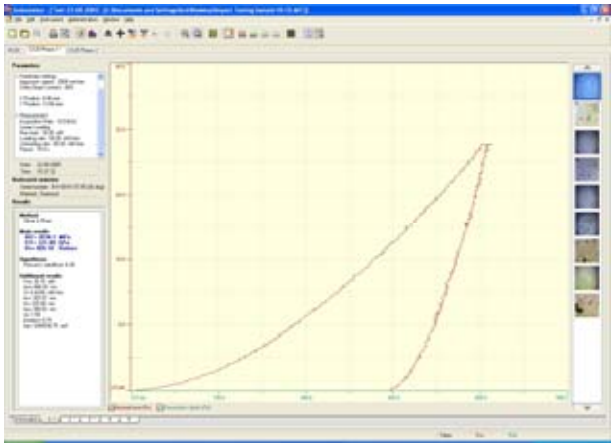
- MHT – Optical distance
- Optical – AFM distance (or Optical - ConScan objective)



Data of the calibrated indenter (with frame compliance included)



All the data are presented in a clear and very understandable format on the screen



> Data acquisition

Continuous real time data acquisition of load (F_N) and displacement (D_Z) signals during an indentation.

- Normal force FN sampling rate : > 5000 points/s
- Displacement DZ sampling rate :> 5000 points/s
- DZ and FN filtering : 10 points/s

> Data analysis

Analysis of MHT measurement files using modified W.C. Oliver and G.M. Pharr calculation method

Reference:
- *Journal of materials research. Vol. 7, No. 3, Mar 1992*
- *Journal of materials research. Vol. 7, No. 6, Mar 1992*
- *Thin Solid Films, 308-309 (1997) 297-303*
- *Journal of materials research Vol. 19, No. 1, Jan 2004*

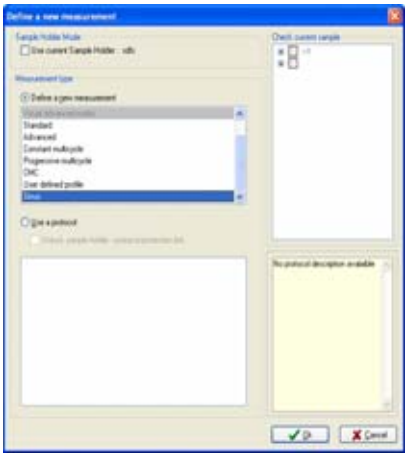
Other methods: Tangent method, Martens Hardness are available

> Measurement report

In the same file, you can put all the indentation curves corresponding to a single sample. Pictures and comments can also be included to provide a more comprehensive analysis

A complete synthetic report for a set of measurements can be automatically generated

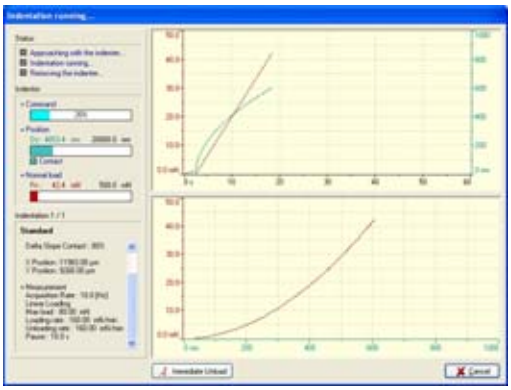
> Measurement modes



New measurement type
- Standard
- Advanced
- Constant Multicycle
- Progressive Multicycle
- CMC (Continuous Multicycle)
- Sinus (with option installed)
- Line
- Simple matrix
- Advanced matrix
- User defined profile
-

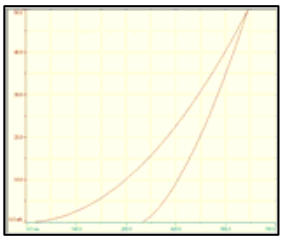
Use of a protocol
A previous measurement type can be launched.

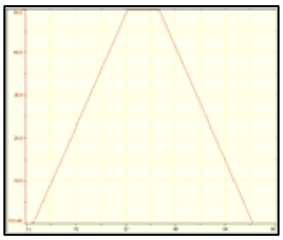
During the measurement process, an on screen real time monitoring of the indentation curves is displayed



> Simple indentation

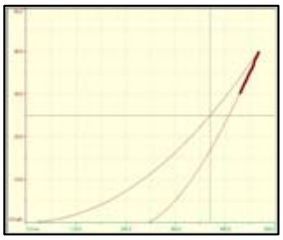
- Maximum depth or maximal load
- Loading rate
- Pause
- Unloading rate
- Linear or quadratic loading

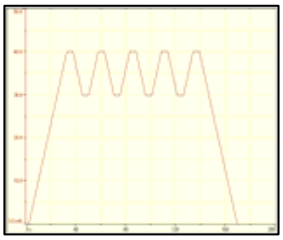




> Multicycles with constant load

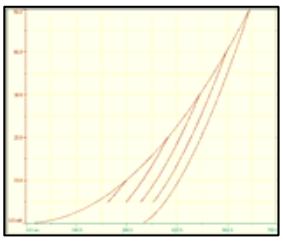
- Maximum loading depth or force
- Minimum unloading depth or force
- Number of cycles
- Loading rate
- Pause
- Unloading rate

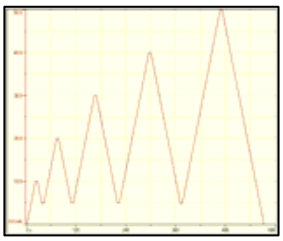




> Multicycles with progressive load

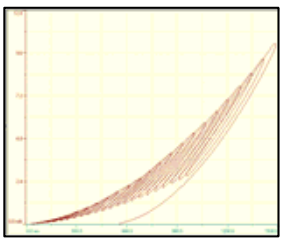
- First maximum loading depth or force
- Minimum unloading depth or force
- Number of cycles
- Loading rate
- Pause
- Unloading rate

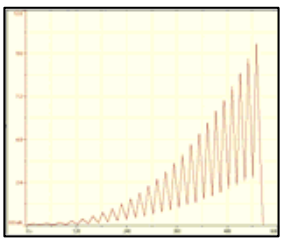




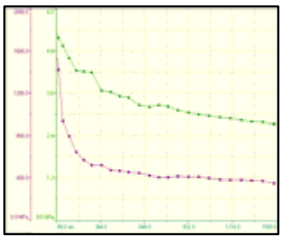
> Continuous Multy Cycle (CMC™)

- First minimum load
- Maximum load
- Choose the unloading %
- Time to max load
- Pause
- Time to unload
- Number of cycles



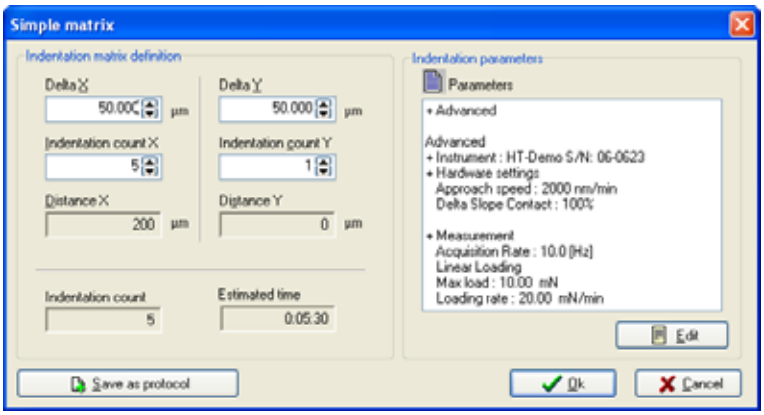


After the CMC measurements, the graphs of Hardness and Elastic Modulus vs penetration depth can be directly analyzed.



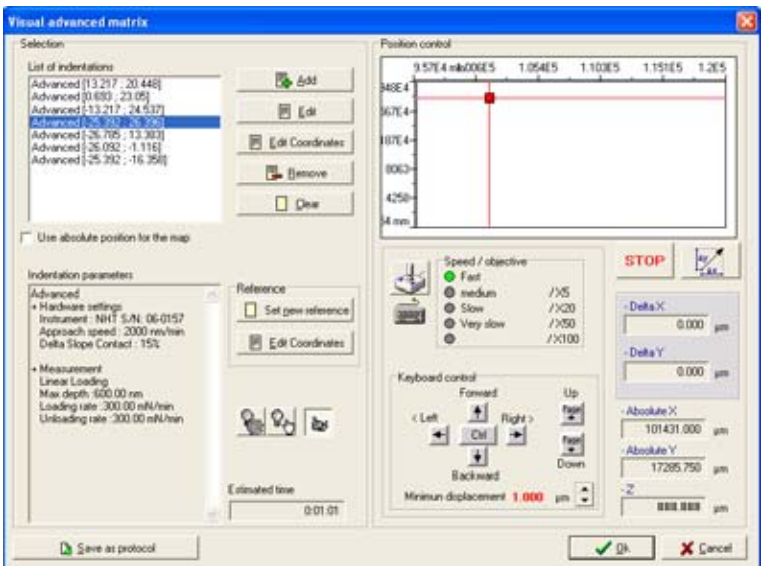
> **Matrix (all indentations with settings)**

- Series of indentations with defined X and Y distances between each indentation
- One indentation setting
- Possibility to store the matrix setup for future use

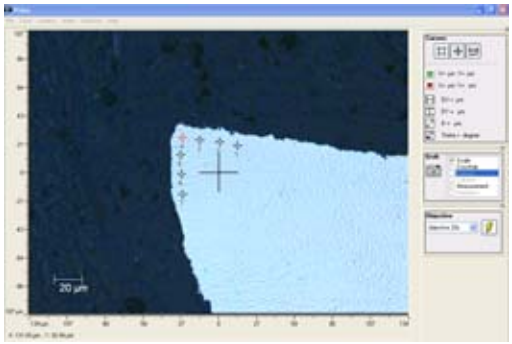


> **Visual Matrix (all indentations with free settings)**

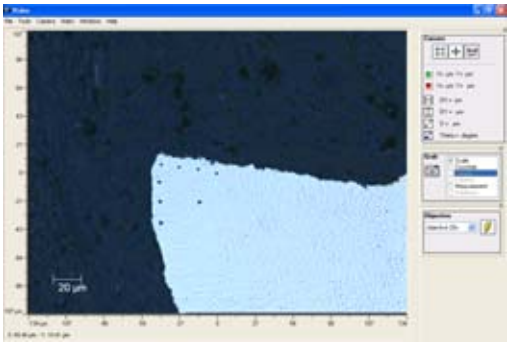
- Series of indentations selected optically,
- You select the Indentation positioning with the computer mouse directly on the PC screen.
- User defined indentation settings for each indentation



Before indentation testing

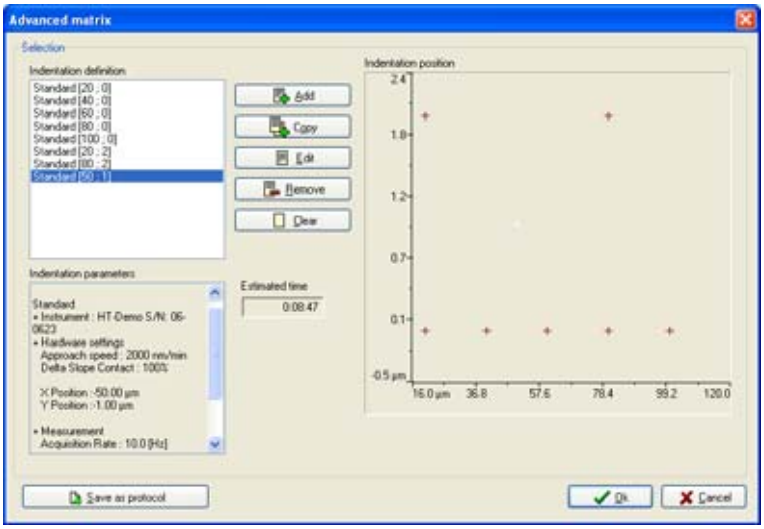


After indentation testing



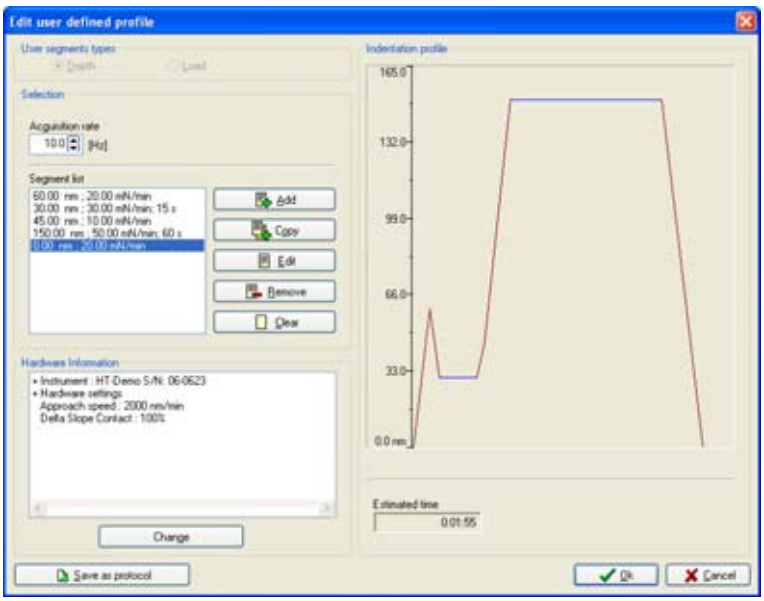
> User defined matrix (all indentations with free settings)

- Series of indentations with user defined X and Y distances between each indentation
- User defined indentation settings for each measurement
- Possibility to store the user defined matrix setup for future use



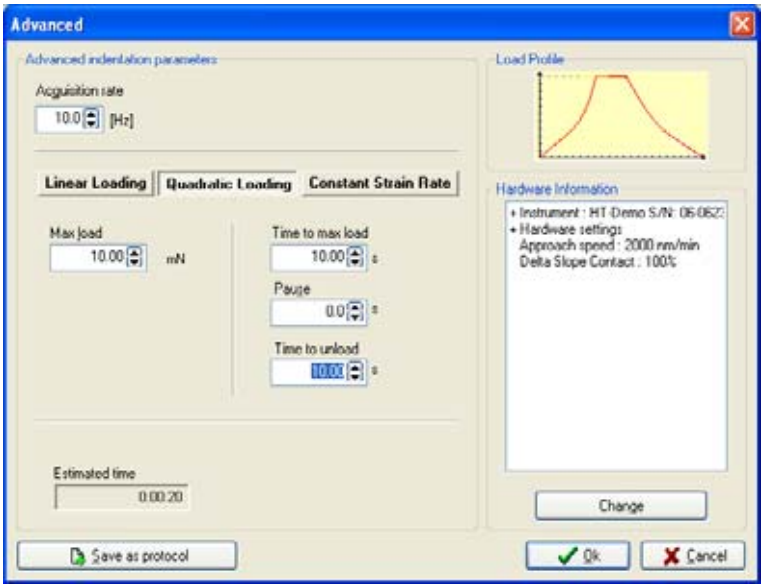
> User defined profile

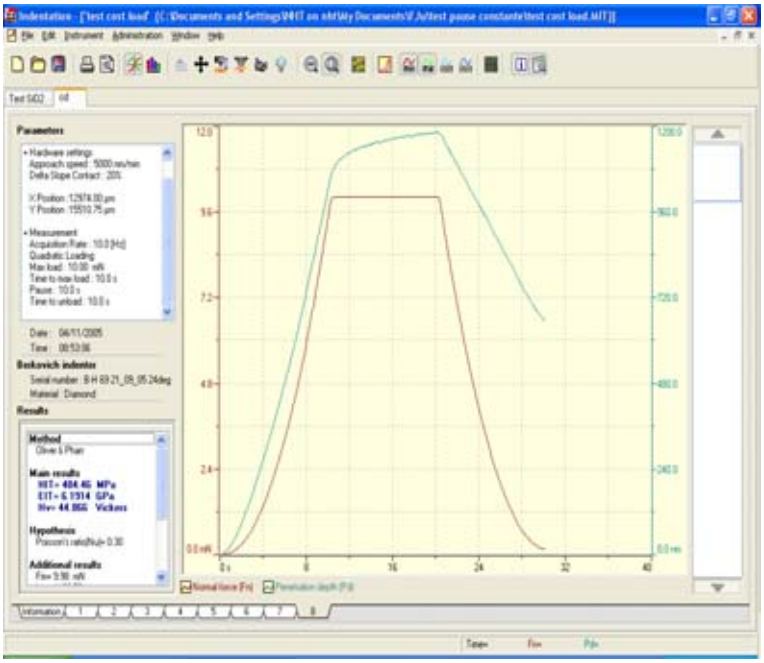
- Indentations with user defined profile
- Possibility to store the user defined profile setup for future use
- quadratic or linear loading



> Martens Hardness

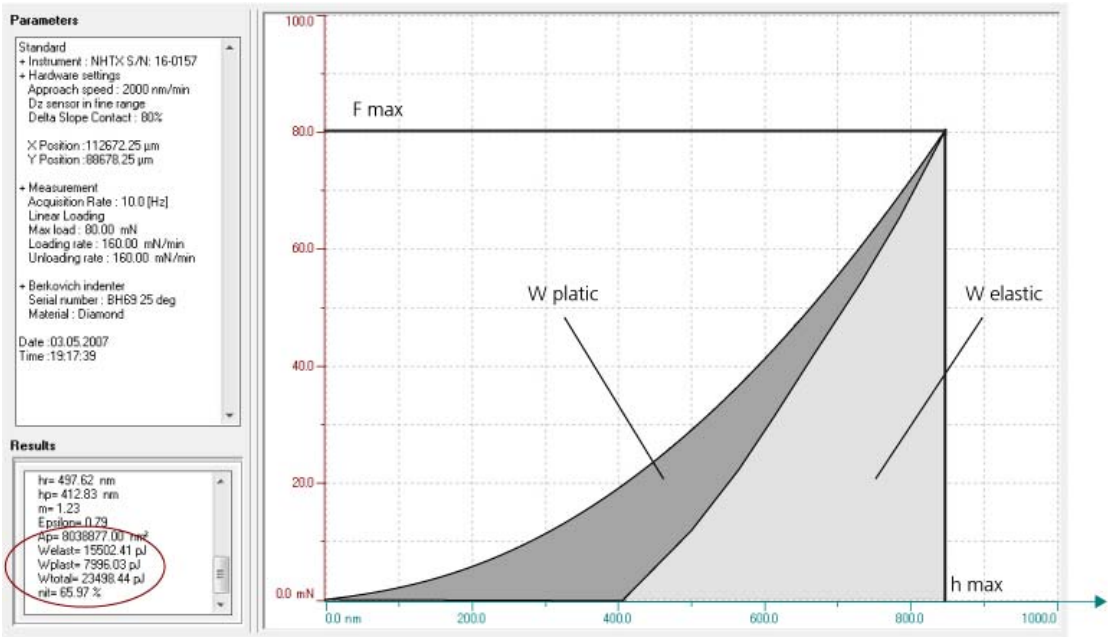
- Indentation using a quadratic loading profile (Norm DIN 50359-1)
- Possibility to store the user defined profile setup for future use





> Plastic and elastic parts of the indentation work

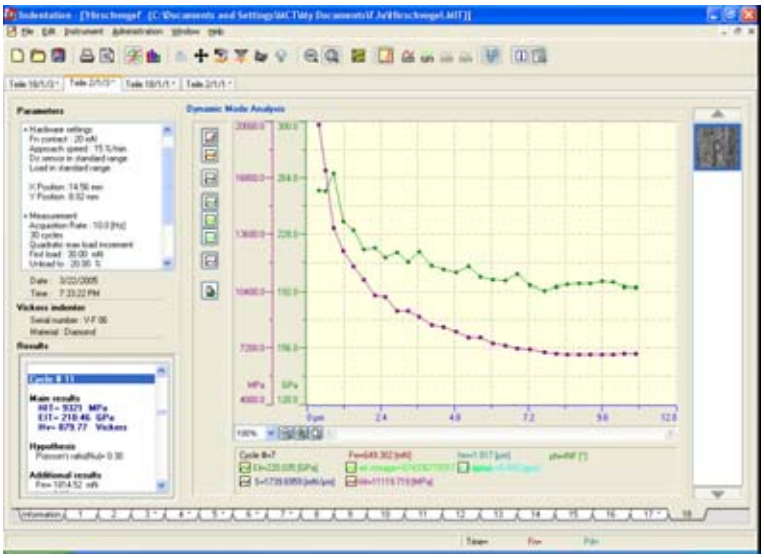
The mechanical work *W*total indicated during the indentation procedure is only partly consumed as plastic deformation work *W*plast. During the removal of the test force the remaining part is set free as work of the elastic reverse deformation *W*elast. According to the definition of the mechanical work as *W* = *Fdh* both parts appear as different areas in Figure bellow.



> Data and measurement analysis

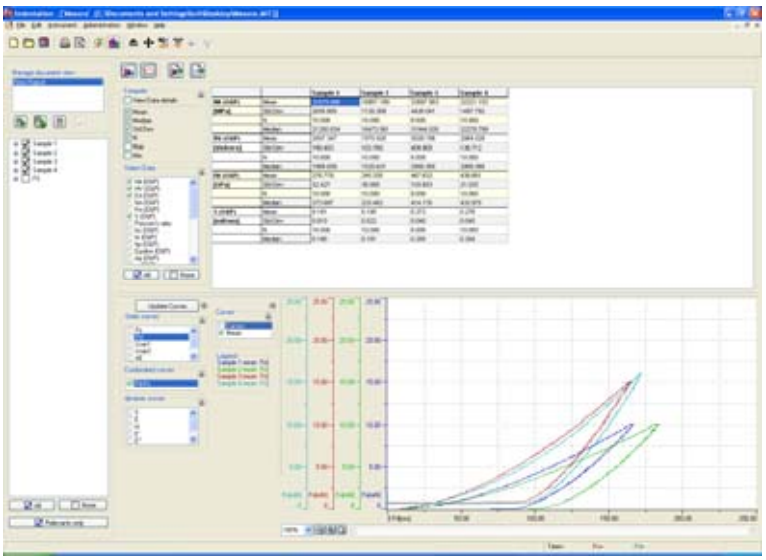
- Various analysis methods (according to norm ISO 14577): Oliver and Pharr, Tangent, Martens (Universal Hardness)

- Continuous determination of hardness and elastic modulus:
  Hardness, Elastic Modulus and Stiffness vs. Penetration Depth



> Statistical mode:

- average, standard deviation, min, max, median, number of measurements per sample



- Exportable data and statistics in text files (for Excel, …)

//// Video Software V4.0

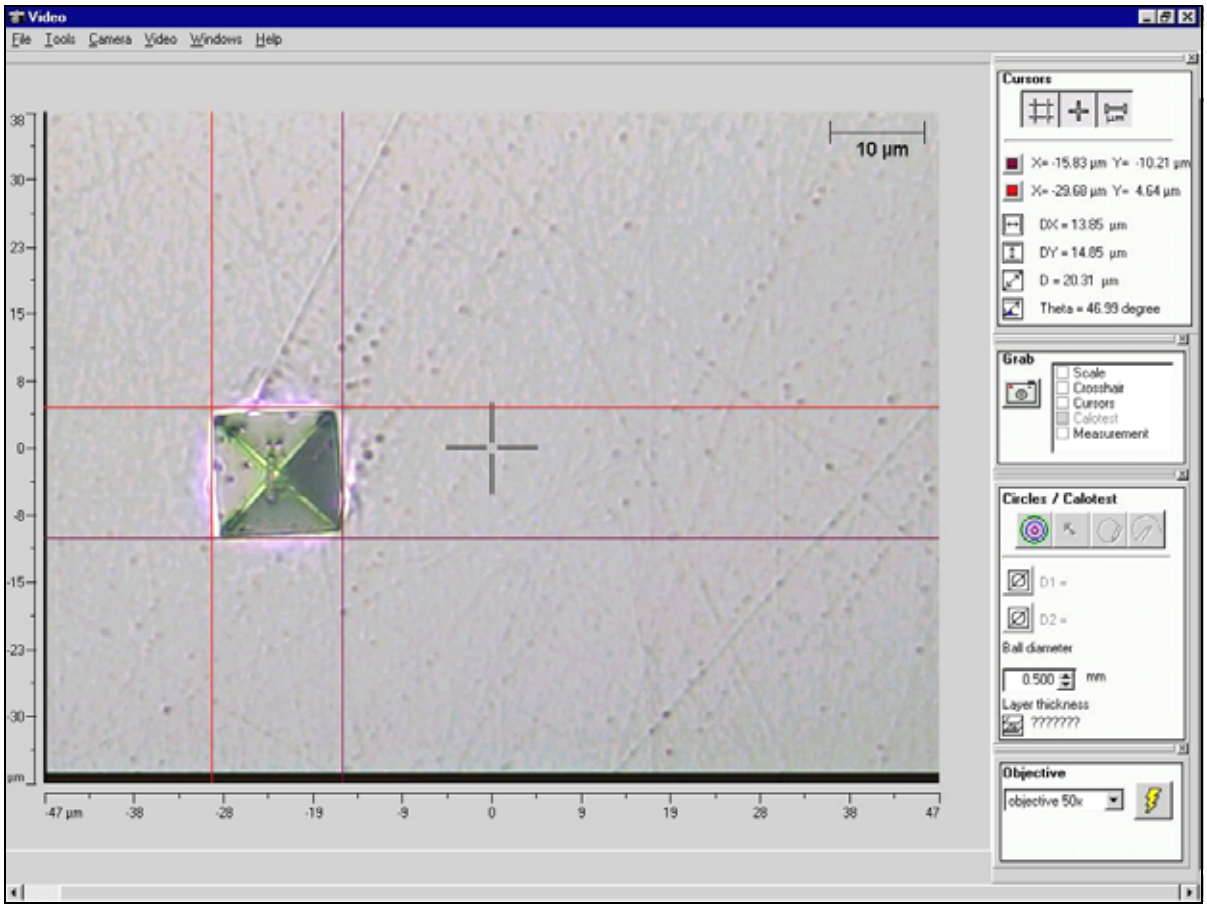
> General features

Instruments are delivered with a video microscope. Objectives are available from x5 to x100 magnification. An optional Nomarski mode is also available.

Two video cameras are available: the standard unit with a resolution of 768 x 582 or the optional high resolution (1280x1024) model using a progressive scan sensor with a very high sensitivity.

The CSM Video Software is the perfect companion of the CSM Indentation and Scratch Testers for the optimal use of video microscopy:

- Real-time video display
- Crosshair, scales, measurement tools
- Image capture, the picture will automatically be added to Scratch or Indentation image gallery
- Layer thickness measurement (Calotest)



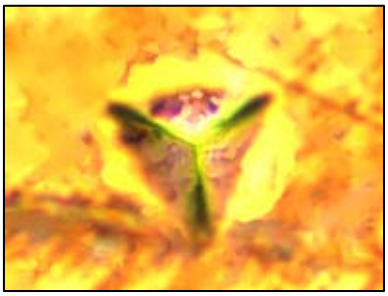
With this software, you can:

- Capture the picture of an indentation,
- Measure the distances with the cursors,
- Show the picture scale,
- Calculate the radius of a circle
  (if you have the Calotest, you can also calculate the thickness of the coating).

> Multi Focus Image (only with Z axis automation)

MultiFocus produce a picture with a perfect depth of field. During the video capturing, our platform will move in Z direction in order to take and combine different levels of focused depth into one image.

Examples: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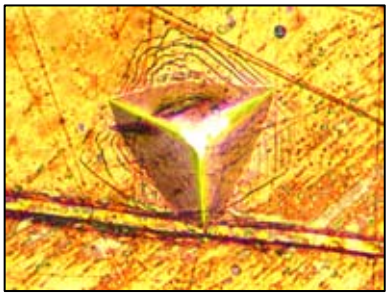
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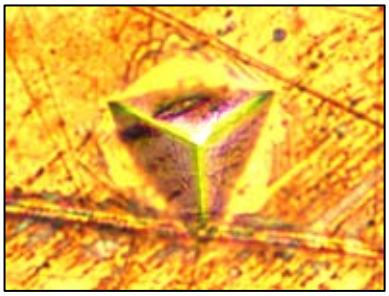

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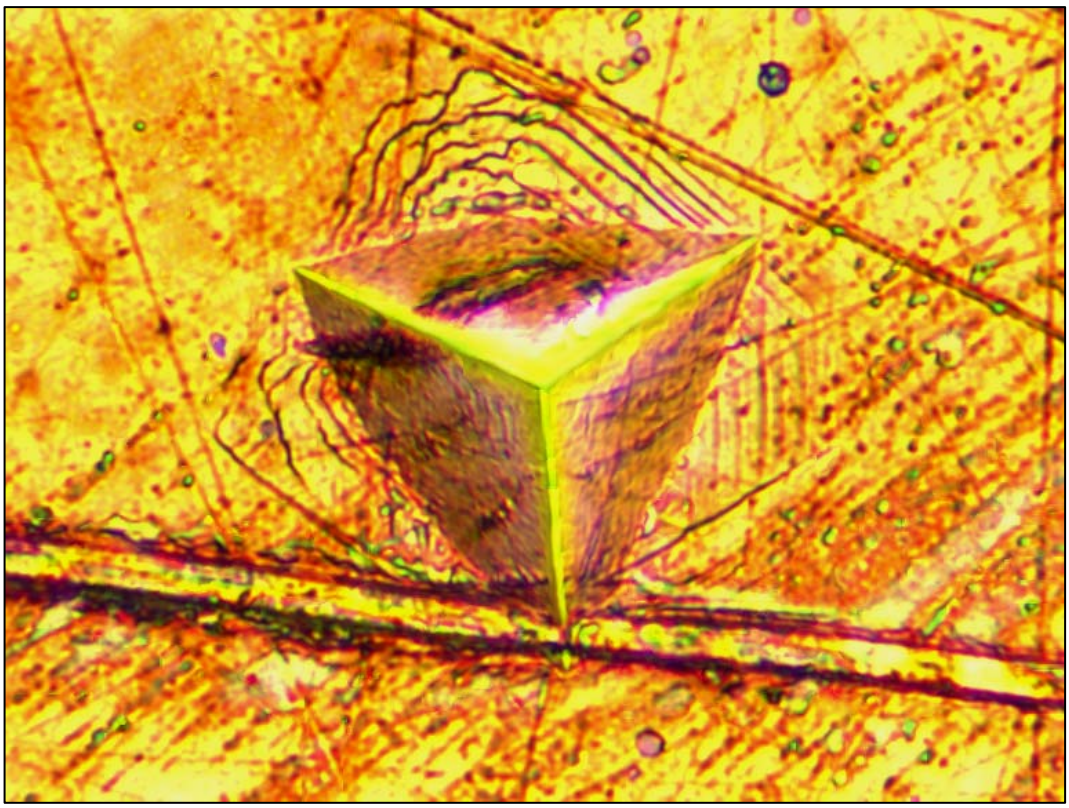

//// Microscope

- The measuring head and the microscope are mounted side-by-side and linked by the two X- and Y- translation tables
- Video microscope magnification: 200x, 2000x
- Allows a specific sample site to be selected
- Modular focusing unit

- Stroke	30 mm
- Coarse focusing	5.2 mm/rotation
- Fine focusing	0.1 mm/rotation
- Distance optical axis	141 mm
- Mounting surface	

- Power supply - Transformator 220 V or 110 V
- Objectives :

- 5x	Working distance	22.50 mm
- 20x	Working distance	3.10 mm
- 50x	Working distance	0.54 mm
- 100x	Working distance	0.30 mm

//// CCD Camera

Standard Resolution Camera

Resolution	768 x 582 (CCIR/PAL)
Sensor	High Quality 1/2" SONY CCD sensor with square pixels
Scanning	Progressive scan
Frame Rate	Up to 50 fps
Mount adapter	C-Mount
Interface	USB 2.0
Power supply	Via USB (<1.5W)
Size (HxWxD)	34 x 32 x 34.4 mm
Weight	75 g

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