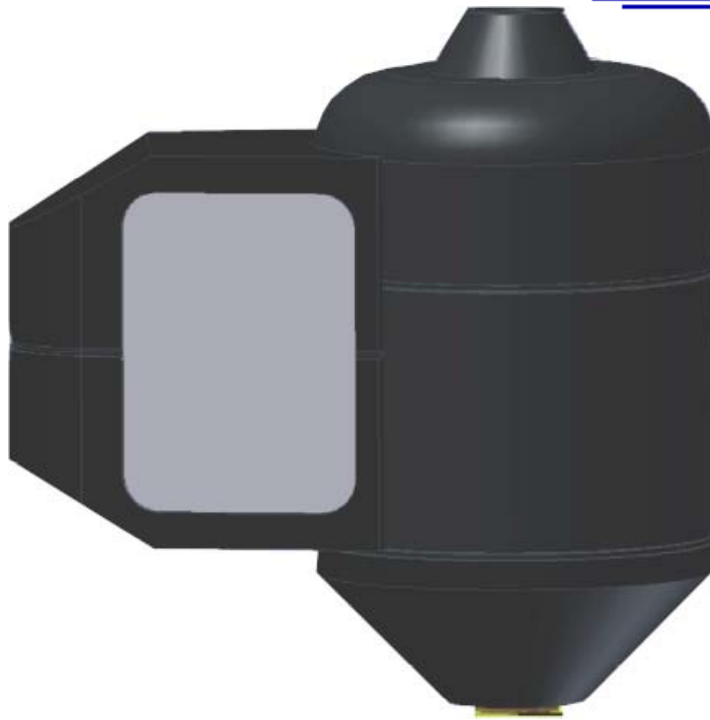


UMIS
Nanoindentation System



**AFM OBJECTIVE
PRODUCT INFORMATION
DS95-Series**

Fischer-Cripps Laboratories Pty. Limited

ABN 26 000 551 459

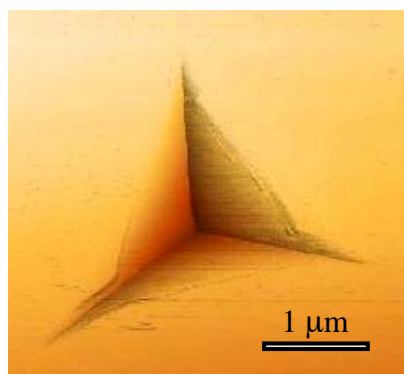
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The DME Dualscope 95-200

Normally, the size of the residual impression of an indentation made during a nanoindentation experiment is too small to be measured optically with an acceptable accuracy, hence, it is the depth of penetration that is measured and the size is calculated from the known geometry of the indenter. It is of interest in some applications to image the indentation so that features of interest such as: cracking, delamination, slip bands and other forms of damage may be observed. The AFM option provides a method of viewing these features at a detailed level.

Fused silica



The DualScope™ DS 95-200 scanner has an SPM measurement volume of 200×200×15 micron with the 15 micron in the Z-direction, i.e. the direction of the centre line of the optical microscope.

Dualscope DS95-200 probe head specifications.

- Tip radius: 10 nm
- 200 x 200 µm scan size
- 15 (up to 18) µm z range
- Z resolution: Typical < 200 pm (rough sample)
< 1 pm for flat samples with no physical slope.
- XY resolution: 16-bit resolution on all axis < 300 pm
- >1.5 mm approach
- Optical view 1 x 0.75 mm (x15 objective)

The DME Dualscope® DS 95-200 series is a high end SPM instrument offering a wide variety of operating modes:.

DC "contact mode", non oscillating cantilever

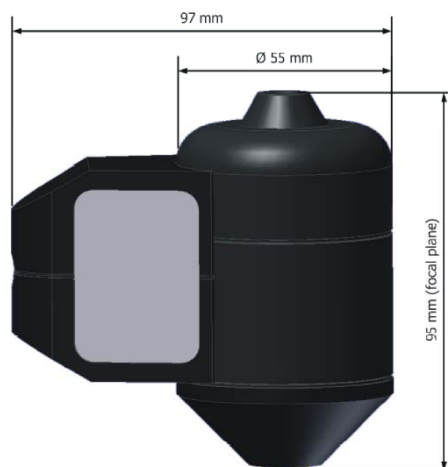
AC "non-contact mode", oscillating cantilever. Also called DME SenseMode™ = detection of oscillation amplitude and phase.

LFM Lateral Force Measurement mode. Measurement of the twisting of the probe in the longitudinal direction by means of quadrant detectors (friction force mode)

EFM Electrostatic Force Measurement. Measuring of the force interaction with the lever created by locally applying a charge to the surface. During scanning, the SPM probe will interact with the force created by the applied charge.

PCM Phase Contrast Mode. Measurement of phase shift signal as "contrast Mechanism". Very sensitive to small details in the surface (contrast function).

MFM Magnetic Force Measurement. Measurement of the magnetic active deflection of the AFM probe as a result of the interaction with magnetic layers on the SPM object.

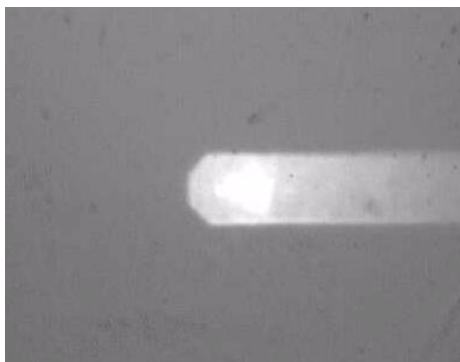


Dualscope is a trademark of Danish Micro Engineering

Cantilever Alignment

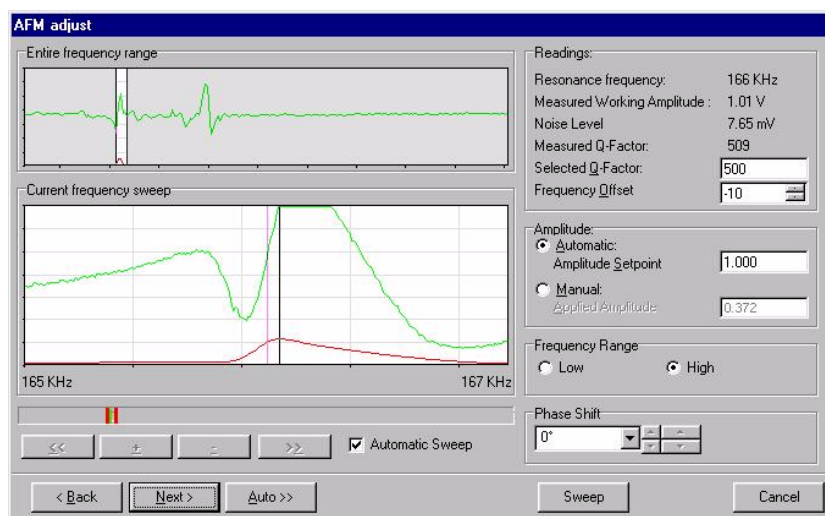
The Dualscope consists of an optical objective and AFM scanner. This enables the user to view the back side of the cantilever for precise alignment of the laser spot. The cantilever tip is supplied pre-mounted on a magnetic holder. The magnetic holder is positioned using a special tool to allow for spot positioning. Positioning the spot typically takes about 1 minute.

The figure shown here is a view of the laser spot on the back of the cantilever. The actual tip (not visible in this figure) extends downwards into the page at the left hand edge of the cantilever.



In the DS95-200, the pre-mounted cantilevers are automatically aligned. Simply snap into position with the magnetic tool and go.

The figure shows one of the sequences for initialising the AFM sensor. The software automatically scans for a resonant frequency. Setup of the sensor typically takes about 2 minutes.

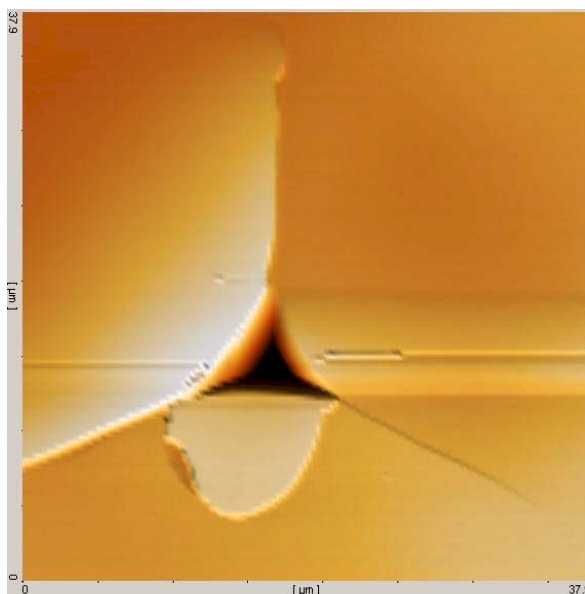


Unlike traditional AFM instruments, the Dualscope is very much wizard-driven for setup operations. The only mechanical adjustment required is the initial positioning of the last spot, and this is made easy due to being clearly visible in the optical microscope view. After setup, the AFM tip retracts to a stand-by distance and scanning can then begin on the sample surface.

Image Acquisition

To obtain an image of the residual impression from the indentation, the tip-to-scope distance with UMIS can be precisely calibrated so that the indentation is brought underneath the AFM head position with an accuracy of less than a micron and typically about 200 nm. Local positioning of the AFM scan area is possible from within the Dualscope software.

Once the indentation is positioned underneath the AFM head, it is useful to do a wide scan at low resolution to check for gross features on the specimen surface. The largest scan size for the DME Dualscope DS95-200 is 200 x 200 microns. The picture shows a low resolution scan of a residual impression in fused silica made with a corner cube indenter for the purposes of crack length measurement for fracture toughness determination.



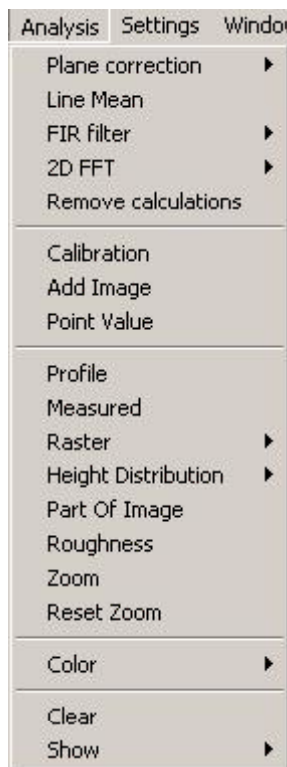
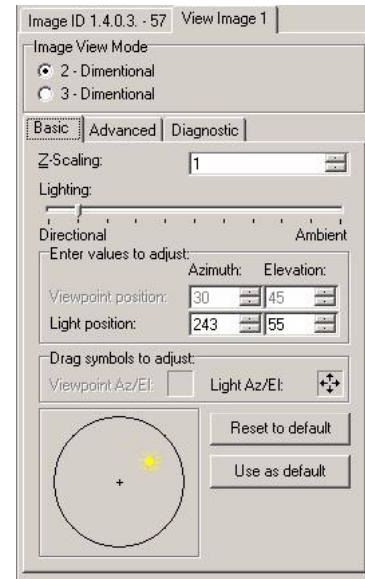
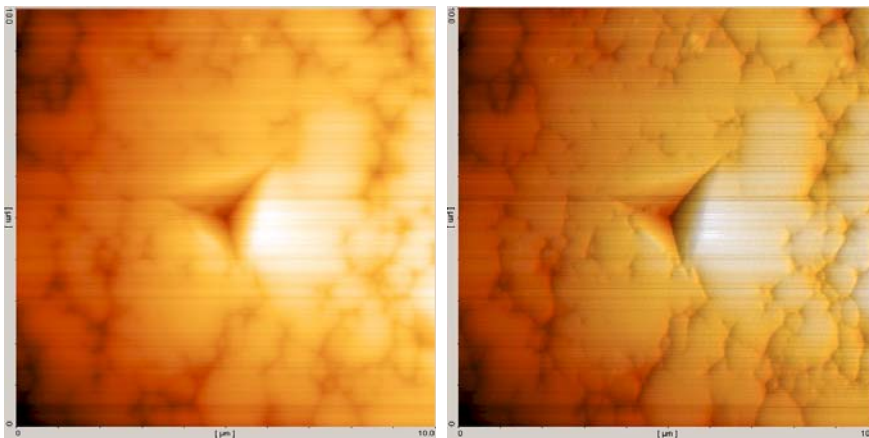
Within the larger scan range, it is possible to interactively select a region for closer, more finer resolution scanning. This is done using a point and click operation with the mouse. The number of scan lines can also be set, as well as the scanning speed. The adjustment of these scan parameters is a trade-off between time taken to acquire the scan and resolution of the final image.

Calculations		Z Scale	Palette
Scan setup		Scan Info	
Signal	Topography		
Retrace	No		
Point X	512		
Point Y	512		
Width X	10.0		µm
Width Y	10.0		µm
Offset X	-0.24		µm
Offset Y	2.15		µm
Direction	0		*
Speed	2.60		µm/s
Loop Gain	1.00		
Loop Filter	3 Hz		
Force	0.15		nN

Image Analysis

The Dualscope software provides extensive image analysis functions. Shading and z axis-enhancement, profilometry, and also plane correction are just a few among the many facilities available.

The picture shows before and after 10 x 10 um images of a residual impression in a TiNSiN superhard film with a Berkovich indenter. The image at the right was made by just altering the shading and light position using interactive controls.



Other more extensive functions are available from the analysis menu. Of particular usefulness is the interactive plane correction whereby the user can draw out a region within the scanned image and the software will apply a plane correction based on information in this image area. This is useful for "levelling" out a sloping specimen surface.

Contact Area Measurement

One of the most limiting aspects of nanoindentation testing is the inability of standard methods of analysis to account for piling-up of material around the edge of the indentation. For ceramics and glasses, piling-up is usually not such a problem. For metals, and other strain-hardening materials, piling-up can lead to significant over-estimates of the Elastic Modulus and Hardness as computed from the load-displacement curve. The limitation arises because the standard methods of analysis assume a certain level of sinking-in in accordance with the equations of contact that are used to calculate E and H. If the actual true area of contact were known, the the effects of piling-up can be accounted for. This is where an AFM image becomes very important.

For example consider the results below obtained with a Berkovich indenter on a super-hard TiN/BN thin film Using the usual methods of analysis, the Elastic Modulus was computed to be 454 GPa and the Hardness was computed to be 44.6 GPa. These results were obtained from a power law fitting to the unloading data in the load-displacement curve. The area of contact computed using this procedure was $2.227 \mu\text{m}^2$.

The image at the right was taken with the DME Dualscope® AFM probe head. Piling-up is in evidence around the straight edges of the impression as can be seen by the light colouring on the image. Using an image-analysis program, the projected area of contact was measured to be $2.62 \mu\text{m}^2$. Notice that the true area of contact, as measured using the AFM, is larger than that estimated from the load-displacement curve analysis. Using the true area of contact, the Elastic Modulus evaluated to 403 GPa and the Hardness was estimated to be 37.9 GPa.

As well as providing the most accurate determination of the contact area, high resolution AFM imaging close to, or within the indentation can provide valuable information about the mode of deformation in the material.

Further, by precisely aligning the AFM scan position with the indenter tip, very precise indenter positioning of the indentation prior to testing can be obtained.

