SPM Imaging

Micro Materials
Excellence in Nanomechanics

The NanoTest™

Bringing nanomechanical measurements into the real world

NANOPOSITIONING AND SPM IMAGING MODULE

The NanoTest advantage for nanopositioning and piezoscanning

- Closed loop piezo-scanning with SPM Imaging
- Positioning to within 2 nm
- 200 x 200 μm sample area
- Determination of fracture toughness
- ISO 14577 displacement calibration
- ISO 14577 surface roughness determination
- Imaging of wear scars
- Imaging of nano-scratch tracks
- Integration to TaiCaan surface analysis software
- In situ determination of the area of indentation (to include pile-up)

NANOPOSITIONING AND SPM IMAGING MODULE

NANOPOSITIONING KEY FEATURES

- **Closed loop piezo-scanning**
- **Precise positioning to 2 nm**
- **200 x 200 µm sample area**
- **Integration to TaiCaan surface analysis software**

INTRODUCTION

The NanoTest nanopositioning and SPM imaging module represents a unique collaboration between three companies – Physik Instrumente GmbH, Micro Materials and TaiCaan Technologies. The Physik Instrumente manufactured high-precision piezo-scanning stage is fully integrated and controlled from within the NanoTest NTX software, and advanced image post-processing/surface analysis is performed with the aid of TaiCaan's integrated BODDIES software.

Imaging with the closed loop piezoscanning stage avoids any issues with the compliance and thermal drift of open-loop AFM-hybrid nanoindentation systems. The large 200 x 200 µm scan area enables imaging of nano-scratches and micro-indentations beyond the capability of instruments with smaller scan ranges. This is important for

- Nanoscratch testing, assigning critical loads in long scratches.
- Nano and microindentation, ensuring that the entire crack lengths are visible to determine the fracture toughness reliably.

Figure 1

The nanopositioning stage in situ on a dual loading head (NanoTest and MicroTest high load head) system

APPLICATIONS

Beyond its use as an SPM for imaging, the applications of the nanopositioning stage include:-

- Accurate hardness measurements on soft samples
- Fracture toughness determination
- Imaging of nano-scratches and wear scars
- Imaging of indentation-induced fracture
- Nanopositioning for precise indent placement
- Displacement calibration for compliance with ISO-14577

MORE ACCURATE HARDNESS MEASUREMENTS ON SOFT SAMPLES

Standard methods of nanoindentation analysis which use the unloading curve (Oliver and Pharr, Doerner and Nix) assume Sneddon-based contact mechanics where the contact is assumed to be slightly sinking-in. The assumption is fine for glasses, ceramics and hard coatings but is less accurate for some plastics and most soft metallic and alloy samples where piling up around the indenter occurs. This "piled-up" material can increase the real area of contact leading to erroneously high measurements of hardness when the unloading curve is used, as shown by Lim and Chaudhri, (see Philos. Mag. A 79 (1999) 2979-3000). Imaging of indentations with the nanopositioning stage in combination with accurate determination of the indentation projected contact area (including pile-up) using the TaiCaan surface analysis software allows more reliable hardness measurements to be performed on soft samples.

To illustrate the procedure on work-hardened copper (fig.2).

- Perform indentation to 200 mN with a Berkovich indenter.
- Subsequent image levelling (avoiding the deformed central region).
- Draw around the piled up region.
- Automatic calculation of pile-up area.

The apparent hardness determined from the unloading slope analysis was 1.4 GPa. However, the image shows that the real area of contact was greater. A comparison of the areas from the corners of the indentation and the real area enables a factor (A imaging/A unloading curve) of 1.55 to be determined i.e. that the hardness in this case is over-estimated by over 50% when the unloading analysis is used (in agreement with previous determinations by Lim and Chaudhri). A more accurate measurement for the hardness of this work-hardened copper sample is 0.88 GPa.

In contrast annealed copper shows the opposite behaviour with actual area of contact lower than that estimated from the unloading curve.

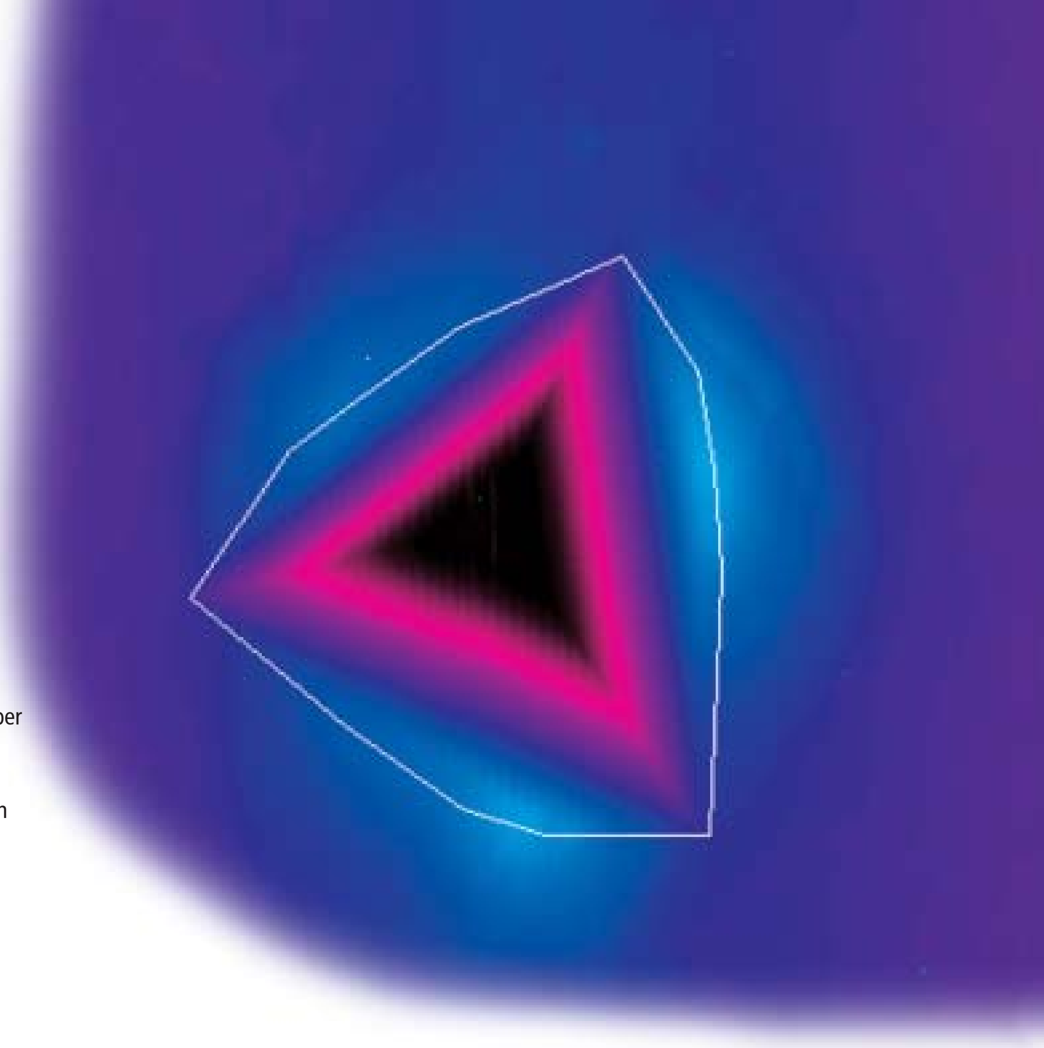

Figure 2

Levelled image of an indentation into work-hardened copper. The pile-up area calculation is automatic in the TaiCaan software.

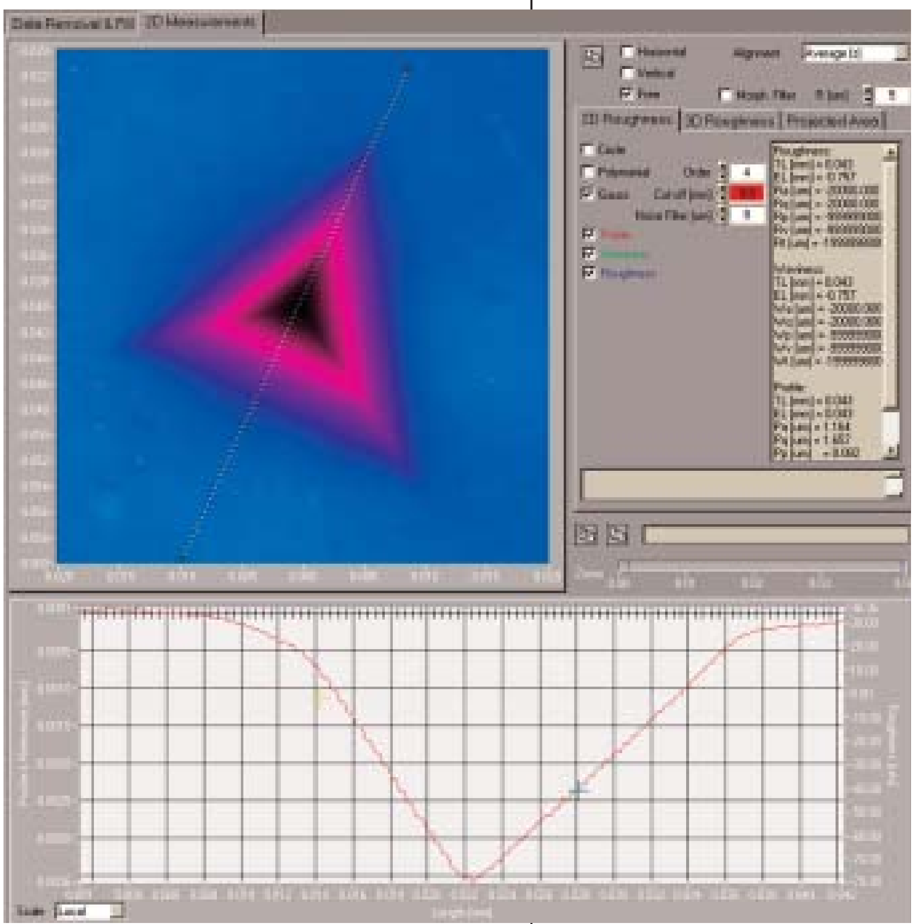

Figure 3

Typical indentation behaviour (sink-in) on annealed copper.

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FRACTURE TOUGHNESS DETERMINATION

Fracture toughness of glasses and ceramics can be easily determined using the NanoPositioner stage. The large load range (0-500 mN NanoTest head; 0-20 N high load head) possible in the NanoTest allows production of fully developed radial/median crack systems with a wide range of indenter geometries (e.g. cube corner, Berkovich, Vickers).

The standard fracture mechanics equation [Eqn. 1] is used to determine fracture toughness quantitatively over a wide range of loads for the widest choice of samples.

$K_c = a(E/H)^{1/2}(P/c^{3/2})$ Eqn .1

Where P is the peak load, a = 0.032 for a cube corner (0.016 for a Berkovich) and c is the average measured crack length for the radial/median crack system. The procedure is illustrated in the example below:-

- A sharp cube corner indenter has been used to indent fused silica to a peak force of 400 mN.
- The NanoTest software controls the PI stage and produces a high-resolution image of the indentation.
- Image levelling (excluded deformed region) in the TaiCaan surface analysis software
- Crack length measurement followed by the application of Equation 1 produces the value of fracture toughness.

The value of K_c obtained, 0.58 MPa m^{-2}, is in close agreement with standard literature values (0.6 MPa m^{-2}). Values over a wide load range produce K_c values that are invariant with load confirming the accuracy of the method.

Small-scale fracture toughness measurements of this type are also finding applications in the pharmaceutical industry. For example, L.J. Taylor and co-workers have shown (Org. Process. Res. Dev. 8 (2004) 674-679) that a brittleness index (H/K_c) determined from measurements on single crystals using Berkovich indenters can be used in the effective prediction of the milling of pharmaceutical materials. Excellent correlation between the NanoTest nanoindentation data on single crystals and large pilot scale results have now been obtained for a wide range of materials.

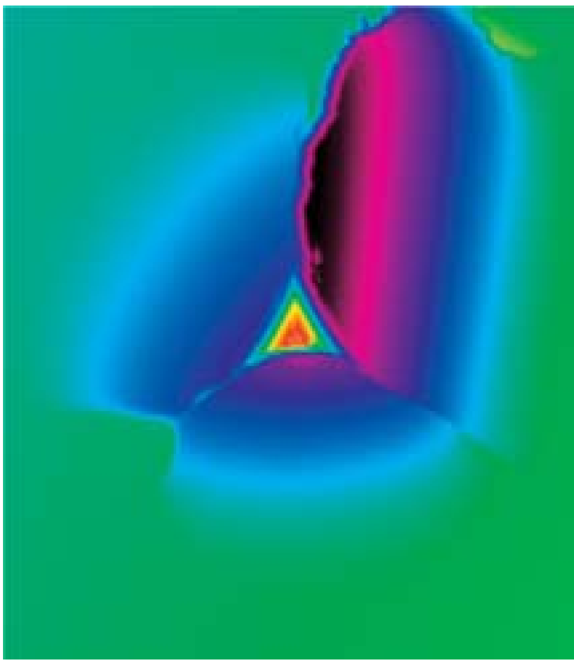
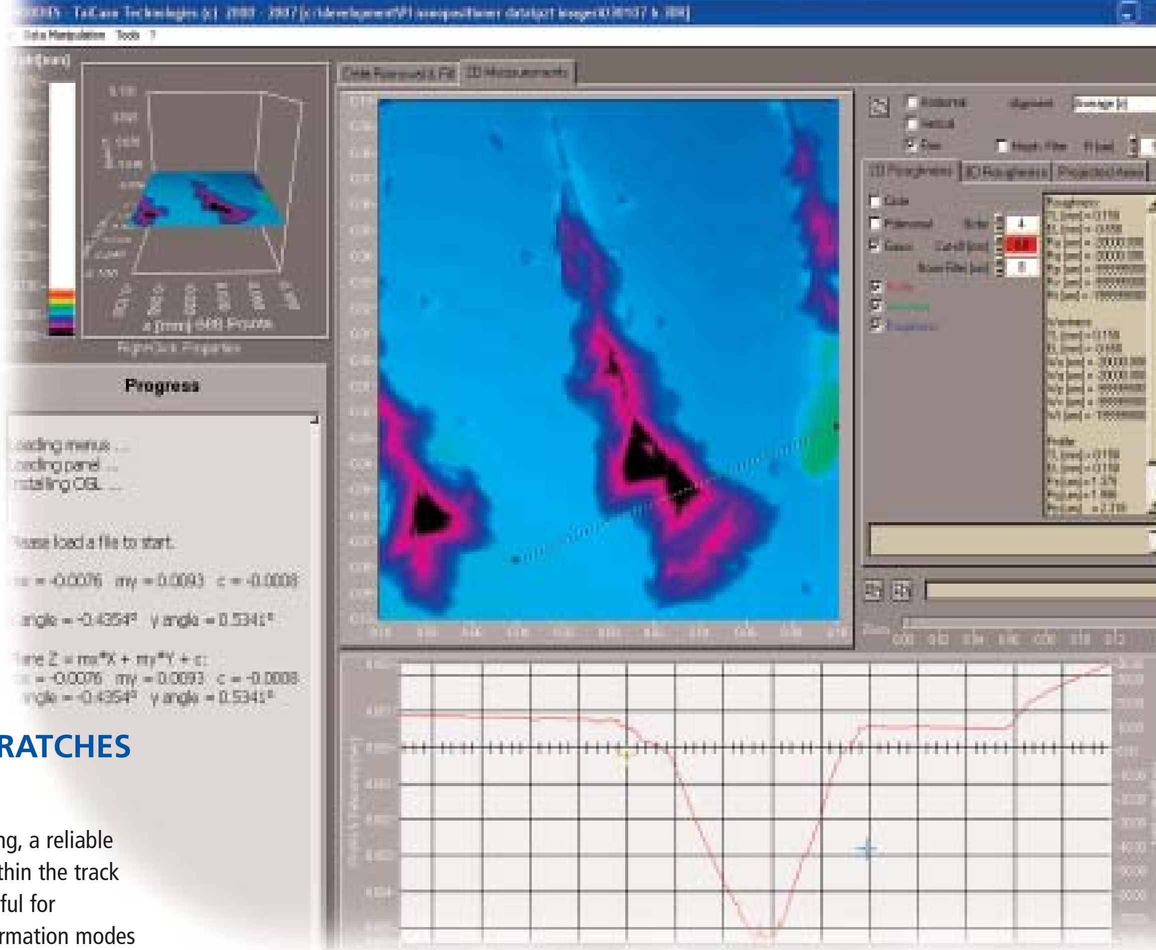


Figure 4

Extensive cracking at the corners of a 400 mN indentation into fused silica.

APPLICATIONS

- **SPM Imaging**
- **Determination of fracture toughness**
- **ISO 14577 displacement calibration**
- **ISO 14577 surface roughness determination**
- **Determination of brittleness index**
- **Imaging of wear scars**
- **Imaging of nano-scratch wear tracks**
- **Accurate determination of indentation projected areas (to include pile-up)**



IMAGING OF NANO-SCRATCHES AND WEAR SCARS

For nano-scratch and nano-wear testing, a reliable 3D profile of the deformation both within the track itself and adjacent to the tracks is useful for mechanistic interpretation of the deformation modes and can e.g. confirm blistering events.

SCRATCHES ON NANOCOMPOSITE TiN/Si_3N_4 COATINGS

These coatings have attracted much interest recently due to the possibility to make them "super-hard". As research into producing coatings with an optimum combination of hardness and toughness in tribological situations, nano-scratches several hundred µm long have been imaged (fig. 5). Even the smallest underlying sample slope can be revealed in these large area scans and so post-processing is necessary to remove the ~0.5˚ slope in this example. After acquiring the images they have been levelled using the TaiCaan BODDIES surface analysis software integrated within the NanoTest control software. The TaiCaan software enables the image to be levelled using the non-scratched part of the surface only to ensure accuracy.

In the example below the image has also been inverted to pick out detail after the total film failure.

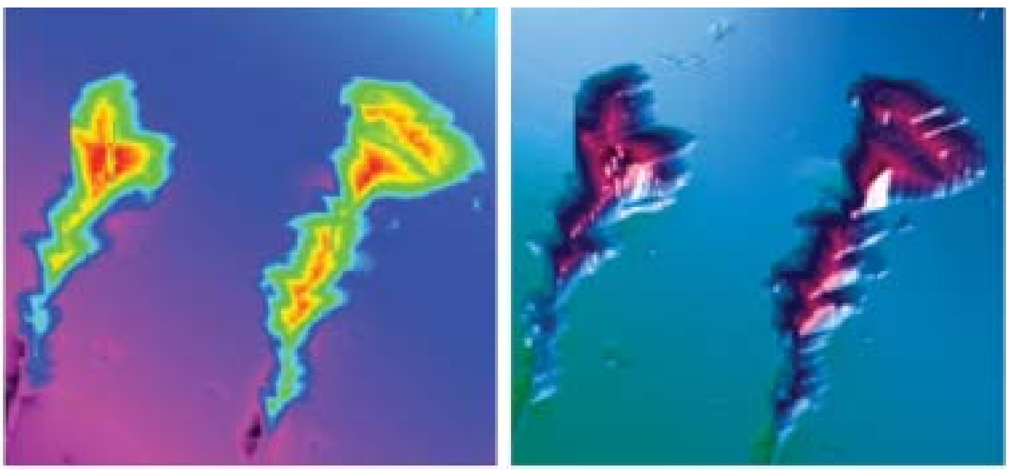


Figure 5

200 x 200 µm images of scratches on hard nanocomposite TiN/Si_3N_4 coatings on Si.
Left = as acquired;
Right = after levelling.

NANOPOSITIONING AND SPM IMAGING MODULE

INDENTATION-INDUCED DEFORMATION

The nanopositioning stage has been used to investigate the indentation behaviour of Silica films on Si. Depending on the PECVD deposition conditions these silica films show discontinuities during loading (at ~50 mN in figure 6 (a)) that the nanopositioning stage confirmed were related to indentation-induced fracture and delamination of the films (fig. 6(b). However, using the NanoTest, the researchers at CIVEN have been able to develop improved silica films that exhibit greatly enhanced plasticity thus eliminating the delamination failures seen in figure 6.

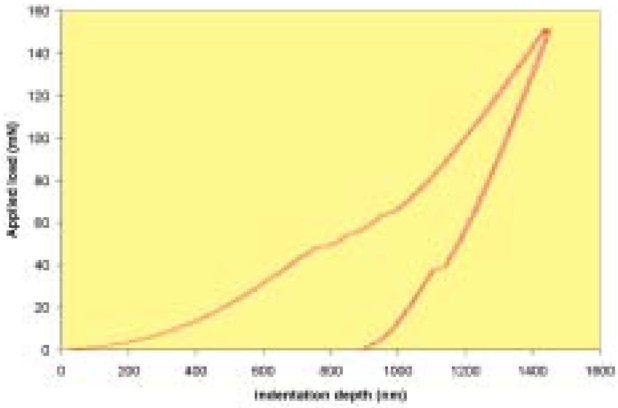
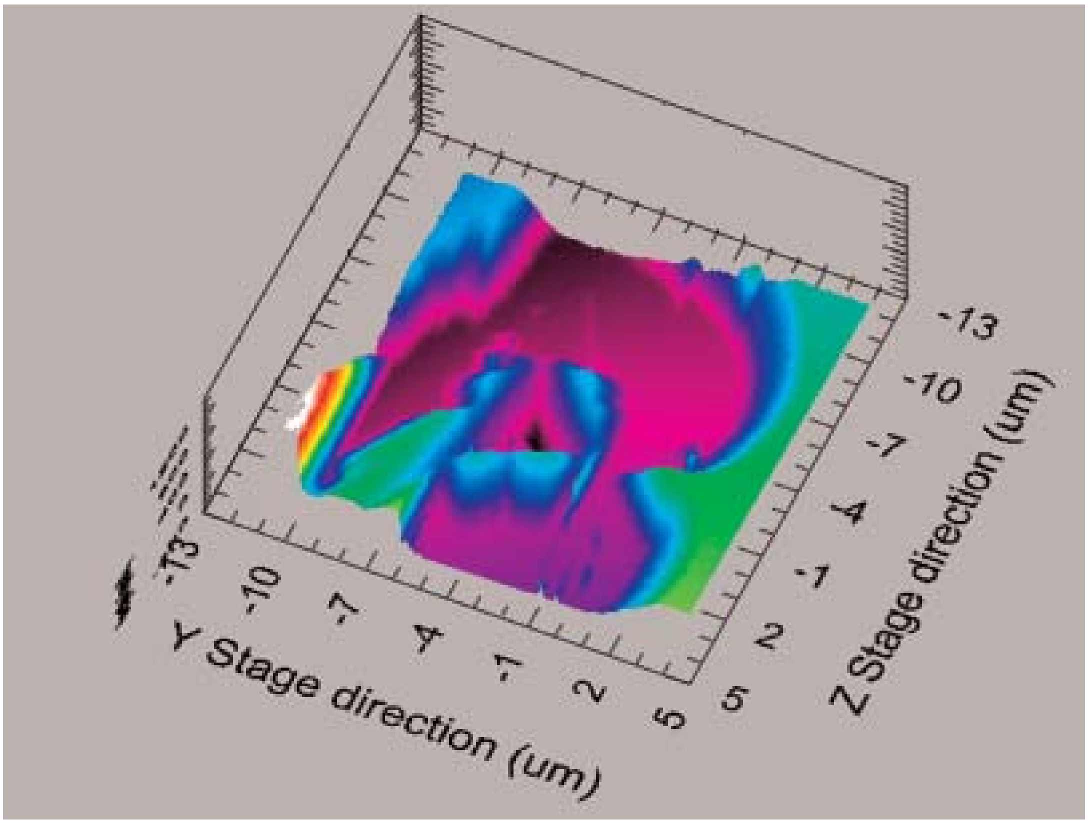


Figure 6

(a) indentation behaviour on PECVD silica film on Si showing fracture and delamination on loading and Si pop-out on unloading (b) subsequent imaging with the nanopositioning stage. Data courtesy of Dr Alessandro Patelli, CIVEN, Italy.



APPLICATIONS

- **SPM Imaging**
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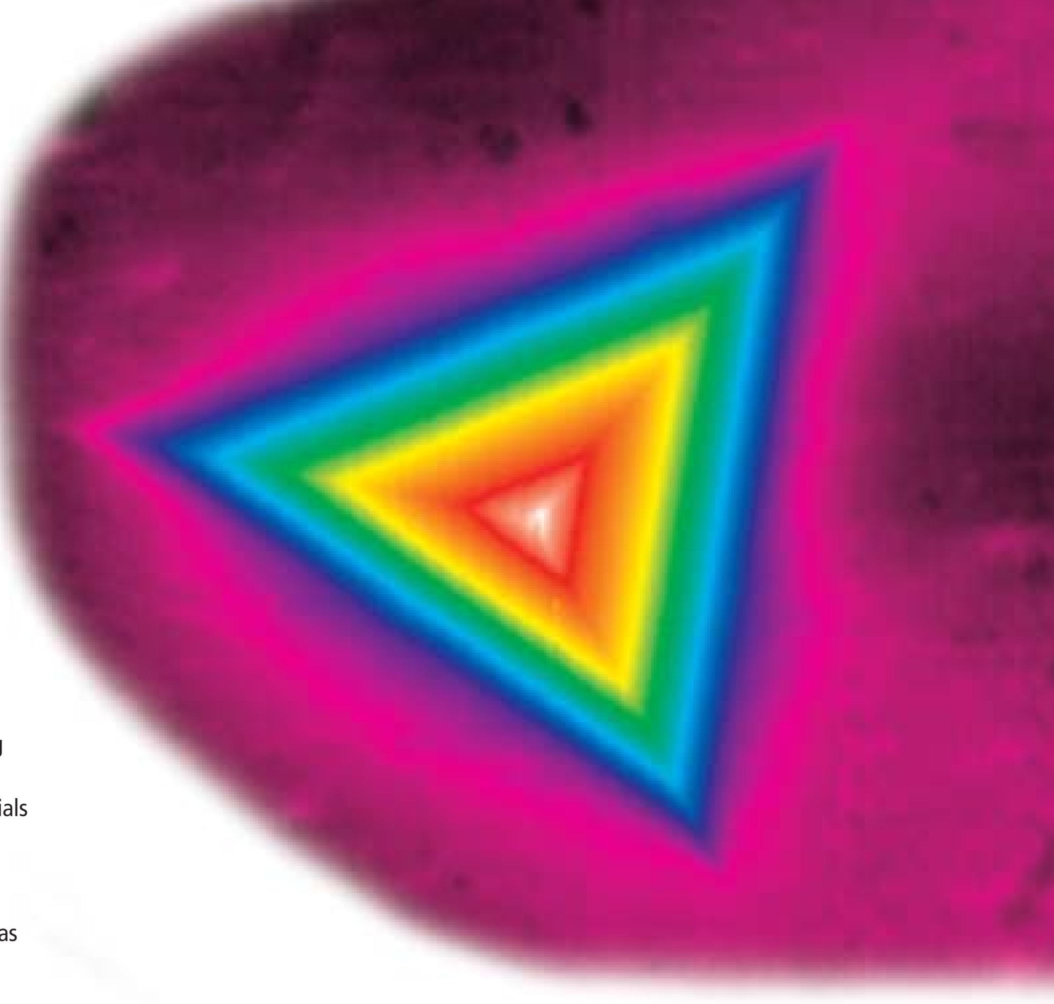
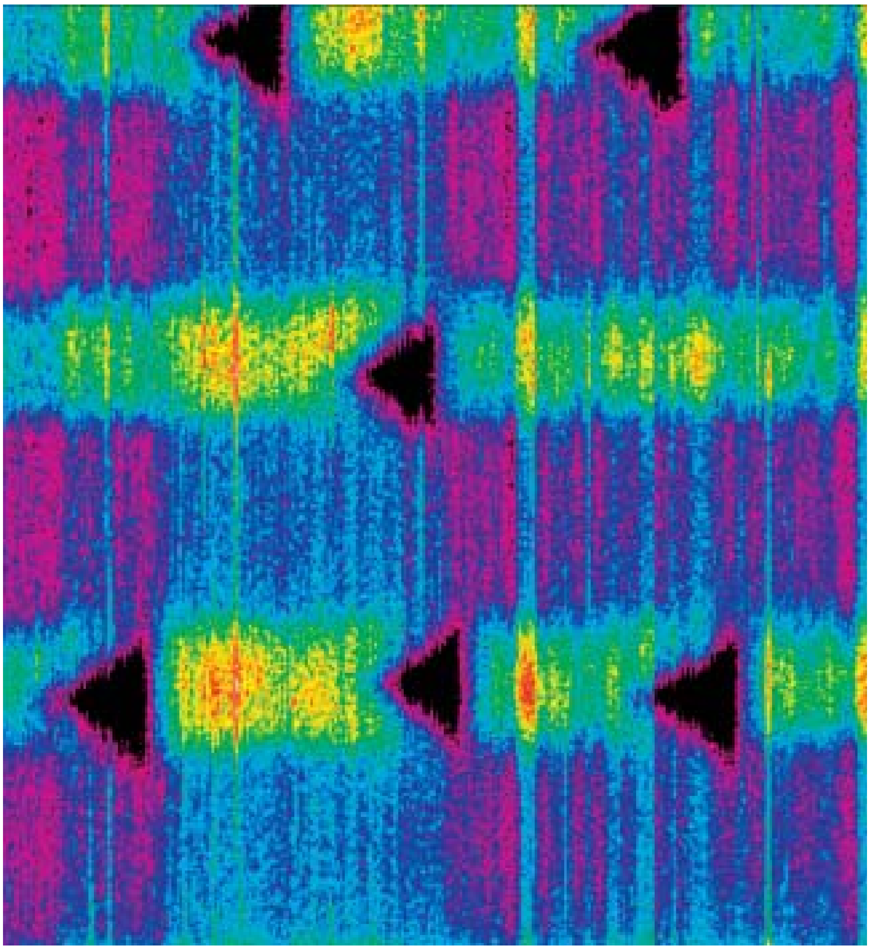


NANOPOSITIONING FOR PRECISE INDENT PLACEMENT

Precise positioning can be necessary for targeting nanoindentations in specific regions of heterogeneous surfaces, particularly for biomaterials such as bone. The procedure is illustrated by the example shown in figure 7:-

- A quick scan with the nanopositioning stage has been acquired.
- The height contrast in this image is used to precisely position the area of interest under the indenter (in this case several 60 nm deep indentations in 400 nm wide tracks running from left to right which are ~ 10 nm higher).



COMPLIANCE WITH ISO-14577

With its flexibility to allow easy and rapid automated user-calibrations of force, displacement, area function and instrument compliance the NanoTest user can ensure continuing compliance to indentation standards such as ISO 14577. Direct calibration of the displacement with the nanopositioning stage provides an alternative to the normal capacitive method. In addition, ISO-14577 requires the surface roughness to be noted for reporting purposes. Levelling and roughness analysis of the images of the surface (prior to indentation) is trivial with the TaiCaan software.

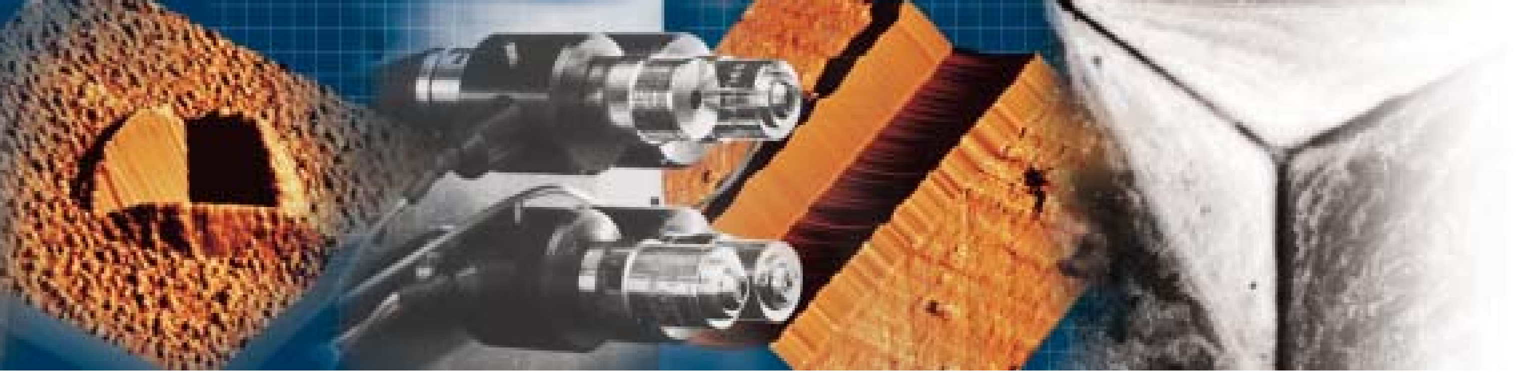
SPECIFICATIONS

- A XYZ piezo flexure stage for sample scanning
- Integrated software for driving this within the NanoTest
- Subsequent surface analysis

It enables in situ 3D imaging of indentation area and therefore a high accuracy in placement of probe for indentation site selection, so specific features can be found and their mechanical behaviour analysed, using click and point software in the NanoTest software environment.

Figure 7

Precise positioning of 60 nm indentations into features 10 nm tall.



NANOPOSITIONING AND SPM IMAGING MODULE

STAGE SPECIFICATIONS

- 200 µm x 200 µm x,y stage from PI
- 20 µm Z height range
- Capacitive feedback sensors
- X, Y stage resolution: 2 nm
- Z image resolution: < 0.1nm (dependent on noise floor)
- Closed loop linearity: 0.03%
- Includes mounting onto NanoTest stage

Note: 100 x 100 x 20 µm and 300 x 300 x 20 µm x,y,z stage are possible options

The nanopositioner stage is fully controlled from within the NanoTest software environment which provides options to export either to ASCII or to TaiCaan for post-processing and surface analysis.

POST-PROCESSING CAPABILITY

Advanced TaiCaan Technologies BODDIES surface analysis software including the following powerful features:-

- 2D and 3D surface imaging and representation
- Data manipulation including levelling and form removal
- Image levelling using only non-indented virgin surface
- Accurate determination of the projected area of indentation (to include pile-up)
- Accurate determination of the height and volume of pile up
- Volume, area and roughness analysis
- Wear analysis and surface comparison tools

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Authors details: Dr Ben Beake, Dr Stephen Goodes, Dr Ashley Gray and Adrian Harris, Micro Materials Ltd

Micro Materials
Excellence in Nanomechanics

For further information contact us at

Micro Materials Limited
Willow House
Yale Business Park
Ellice Way
Wrexham LL13 7YL UK
Tel: +44 1978 261615
Fax: +44 1978 356966
info@micromaterials.co.uk
www.micromaterials.co.uk

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