Micro Materials
Excellence in Nanomechanics

Multi-scale mechanical property mapping

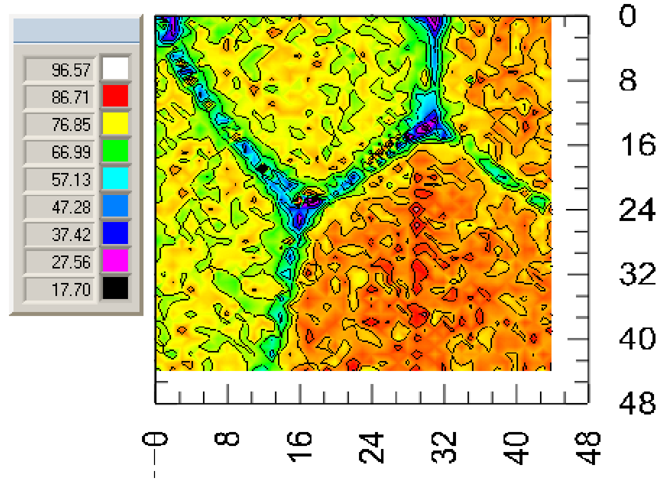
Introduction

Multi-scale mechanical property mapping is proving a useful tool for studying the links between microstructure and mechanical properties in highly heterogeneous and multi-phase materials systems such as biomaterials, or cementitious composites. Biological samples are complex hierarchical materials which can display multi-scale toughening mechanisms.

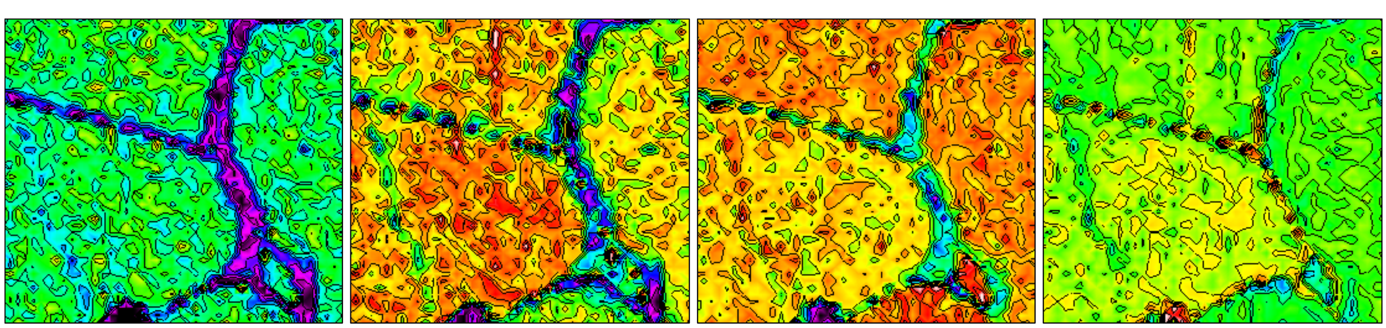
The rate of data acquisition in the NanoTest Vantage is optimised for high speed throughput without compromising data quality through dynamic artifacts or surface detection accuracy.

Full load-displacement curves are always acquired so that the influence of any surface roughness or porosity on the data can be determined. To achieve a sufficiently smooth surface for nanomechanical mapping the sample preparation can be challenging and at low load it can be difficult to deconvolute the influence of surface topography from mechanical properties, so, for data assurance it is critical that the load-displacement data be available.

In this Technical Note we show examples from sectors such as (i) geomaterials (ii) biomaterials (iii) alloy development and (iv) additive manufacturing, to illustrate the possibilities of multi-scale mechanical property mapping. Fine-scale high resolution mapping utilises the NanoTest's 3D-SPM nanopositioning stage (2 nm repositioning accuracy).



2025 indents (45 x 45 grid) show differences in stiffness



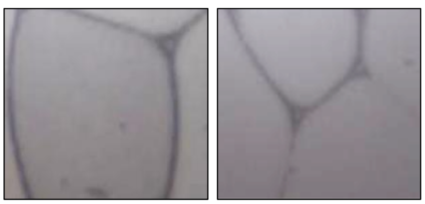
Maps of (from L-R) hardness, modulus, H/E and contact stiffness of calcite crystals in Pinna Nobilis shell

Case studies

1. Twin-based toughening mechanisms in protective armour of molluscs

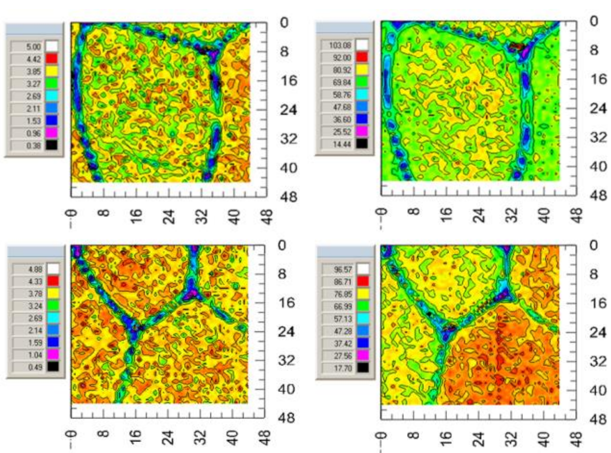
Materials researchers are increasingly looking to nature for inspiration. For example, Pinna Nobilis shells are both stronger and tougher than would be expected from literature calcite values. Understanding why is potentially key to intelligent design of new engineering materials with improved properties.

The Pinna Nobilis shell comprises calcite crystals of size a few 10s of microns distributed across a range of crystallographic orientations. For this study samples were sectioned such that the crystal structure was accessible to nanoindentation in the normal direction (ND). EBSD was used to create a crystal orientation map of the polished surface to identify suitable region with grains on three orientations of interest. The orientation alters the relative ease of different deformation mechanisms (slip, twinning and cleavage) resulting in mechanical anisotropy in the calcite crystals.



Microscopic images of selected regions for mapping

2025 x 1.5 mN nanoindentations were performed over the selected areas, spaced 1 µm apart.



Hardness (L) and elastic modulus (R) maps

Top images:- The grain in centre of top image is in orientation 1.

Bottom images:- grains in orientation 2 (top + left region of this image) and 3 (bottom + right))

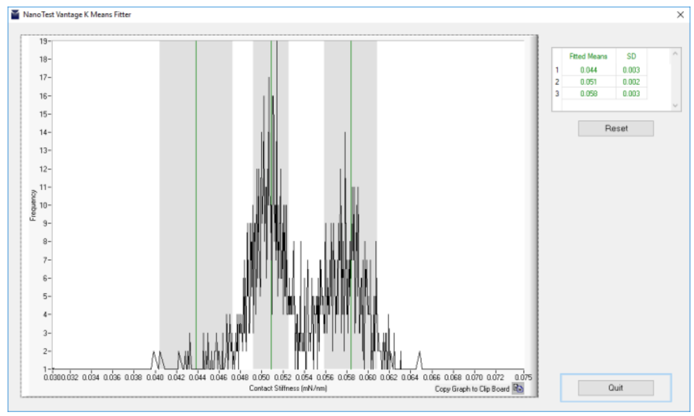
Grain 1 shows the lowest hardness
Grain 2 has higher elastic modulus
Grain 3 exhibits higher hardness than surrounding grains

The NanoTest mapping shows orientation dependent mechanical properties in the calcite crystals consistent with predictions from crystallography.

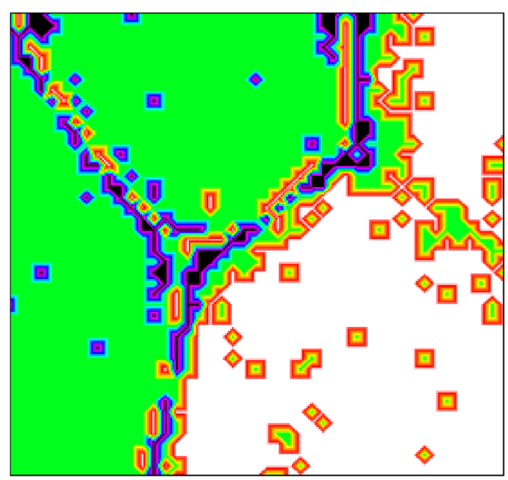
	Hardness (GPa)	Elastic modulus (GPa)
Grain 1	3.00 ± 0.22	69.6 ± 3.6
Grain 2	3.20 ± 0.21	76.2 ± 4.0
Grain 3	3.77 ± 0.23	65.5 ± 3.6

Statistical analysis

k-means distribution analysis allows results to be sorted in to statistically similar groups. The *k*-means method determines the mean and SD whilst retaining positional information.



An example of k-means fitting of 3 components

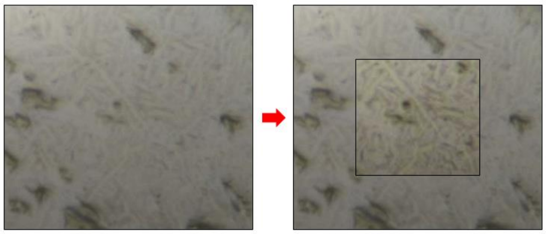


Assigning a colour to each fitted set allows individual crystal orientations to be differentiated mechanically (example of 2 phases).

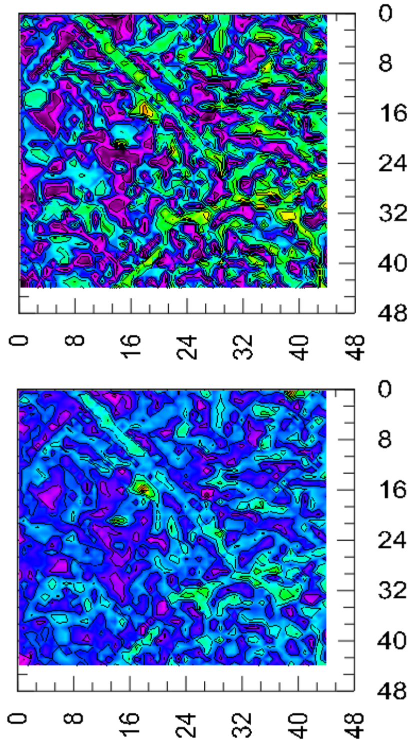
The *k*-means statistical treatment can also be applied to nano-impact maps.

2. Additive Manufacturing – Ti-Nb composites

The processing involved in additively manufactured components results in complex microstructures that differ from conventionally case alloys.



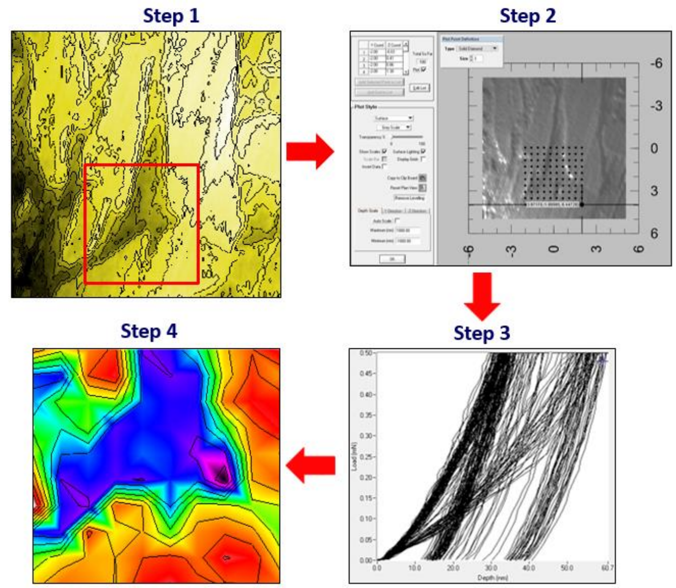
A region was selected using the NanoTest optical microscope and a grid of 2025 nanoindentations to 1 mN performed in a Ti-20at.%Nb composite revealing a bi-phase structure with a strong indentation size effect in hardness. The hardness and elastic modulus maps correspond well with the individual phases seen optically.



Hardness (top) and modulus (bottom) 1 mN mapping of the region highlighted in the optical image above

For finer-scale mechanical property mapping a simple multi-step process can be performed:-

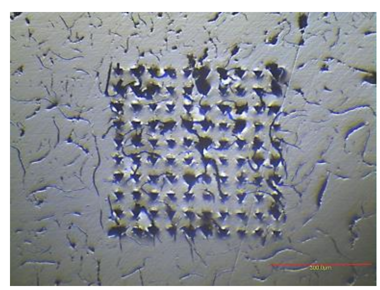
1. SPM Nanopositioner image created and location for indentation chosen
2. Indentation locations defined
3. Indentations performed
4. Map created in NanoTest software



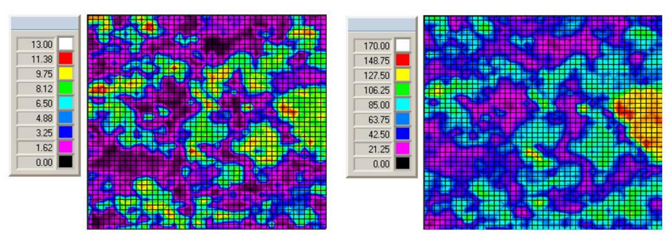
In the example above the indentation force was reduced to 0.5 mN enabling the indentations to be spaced more closely together.

3. Multi-scale composites

The wide load range in the NanoTest Vantage conveniently enables size effects to be studied and heterogeneity to be investigated on different length-scales. In the example below micro-indentations into graphite-rich and iron-rich regions produce a statistical variation. Properties of the individual phases were then determined with 10 mN indentations.



450 µm x 450 µm grid of indentations to 1 N in Compacted Graphite Iron (CGI).



45 x 45 array of 1 mN indentations spaced 1 µm apart on a shale sample (L = hardness, R = modulus)

The elastic modulus distribution reveals the presence of three distinct major phases in the sample.

NanoTest mapping specifications

Maximum map size:	100 µm x 100 µm
Max. no. indentations:	100 x 100 (10,000 @1 µm spacing)
Maximum indent rate:	100 indentations in 10 minutes

Modes of operation

1. Grid indentation of test point spacing ≥ 1 µm. Locations chosen from optical microscope image.
2. Grid indentation of test point spacing ≥ 250 nm. Locations chosen from 3D-Nanopositioner image. This is option is particularly useful when features to be tested are at the limit or even beyond the limit of optical resolution.

Additional functionality

- Statistical *K*-means distribution analysis.
- *K*-means mapping
- Fully compatible with environmental control options (high temp, low temp, humidity etc).

Benefits of NanoTest approach

- Retains full load-displacement curves for subsequent correlation.
- Advantages of a wide load range, high lateral rigidity and high precision stage motion are that any surface roughness/polishing effects minimised
- Advanced data analysis software for statistical analysis – e.g. k-means distributions

Acknowledgements

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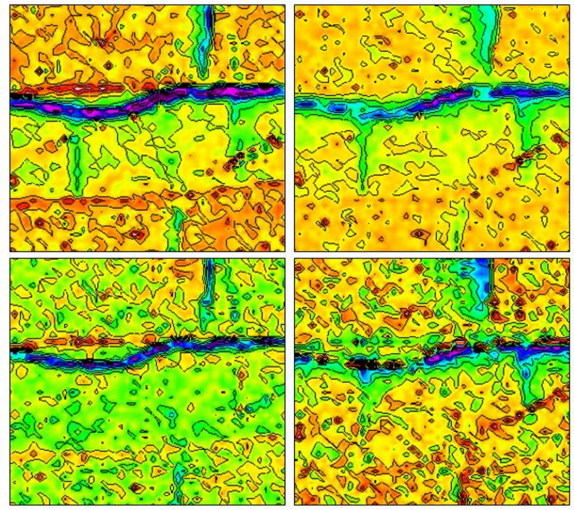
At the forefront of nanomechanics since 1988:-

- First commercial high-temperature nanoindentation stage
- First commercial nano-impact stage
- First commercial liquid cell
- First commercial instrument for high-vacuum, high-temperature nanomechanics

Mapping in the vicinity of cracks

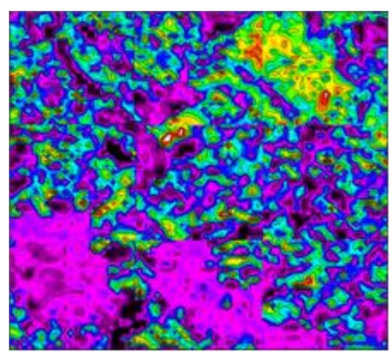
The high lateral rigidity of the NanoTest loading head enables indentations to be placed in the vicinity of cracks. In the example below, a grid of 2025 indents have been placed in a biomaterial with a brick-work structure, and a crack running through these blocks.



Maps of hardness, modulus, H/E and contact stiffness of calcite crystals in Cymbiola Nobilis shell

Multiple grids

The results from several individual grids can be combined as in the example below.



Nine 50 x 50 µm grids (total area = 150 µm x 150 µm)

Although the relative proportions of the different phases varied across the sample surface statistical analysis returned the sample mean values for the major components.

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