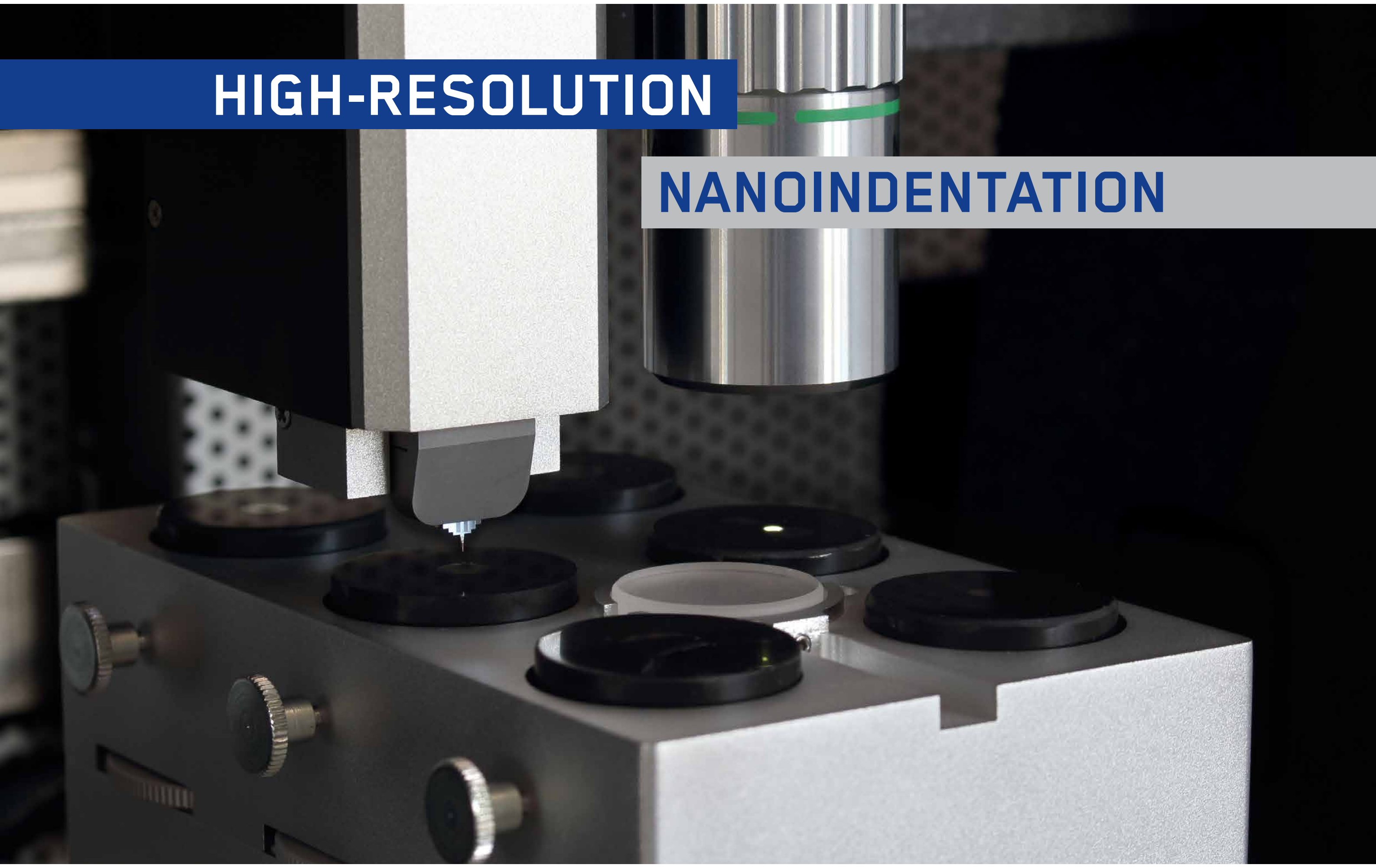
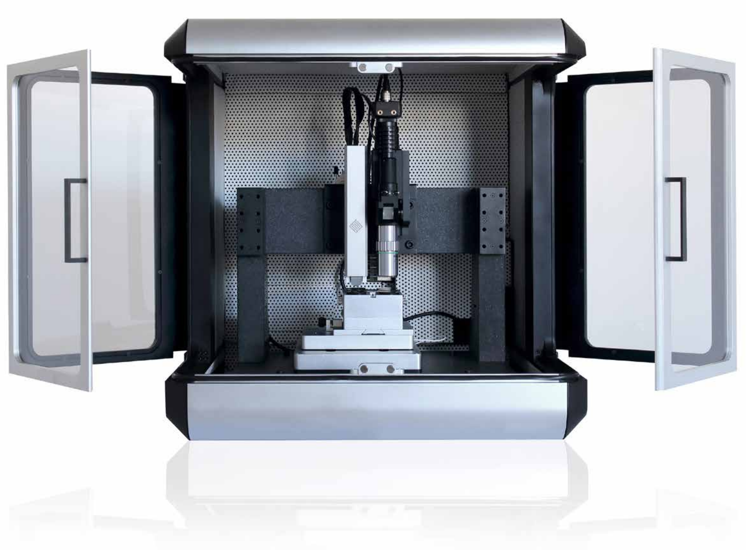
FEMTO TOOLS



HIGH-RESOLUTION NANOINDENTATION



FT-I04
FEMTO-INDENTER

Product Brochure

FT-I04 FEMTO-INDENTER

The FT-I04 Femto-Indenter is a high-resolution nanoindenter capable of accurately quantifying the mechanical and tribological properties of materials at the micro- and nanoscale.

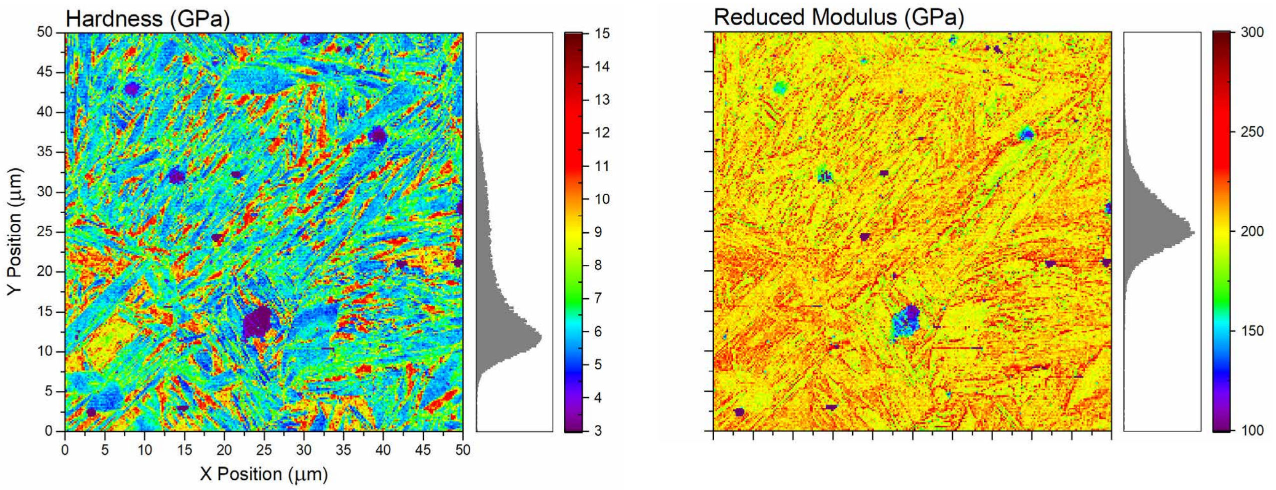
As the world's first MEMS-based nanoindenter, the FT-I04 uses the patented FemtoTools Micro-Electro-Mechanical System (MEMS) technology. Leveraging over two decades of innovations, this nanoindenter features unmatched resolution, repeatability, and dynamic stability.

The FT-I04 Femto-Indenter is optimized for the mechanical testing of metals, ceramics, thin films and coatings, as well as more compliant micro-scale structures such as metamaterials or polymers. Furthermore, the FT-I04 is very modular with several options that extend its capabilities to accommodate the requirements of various research fields.

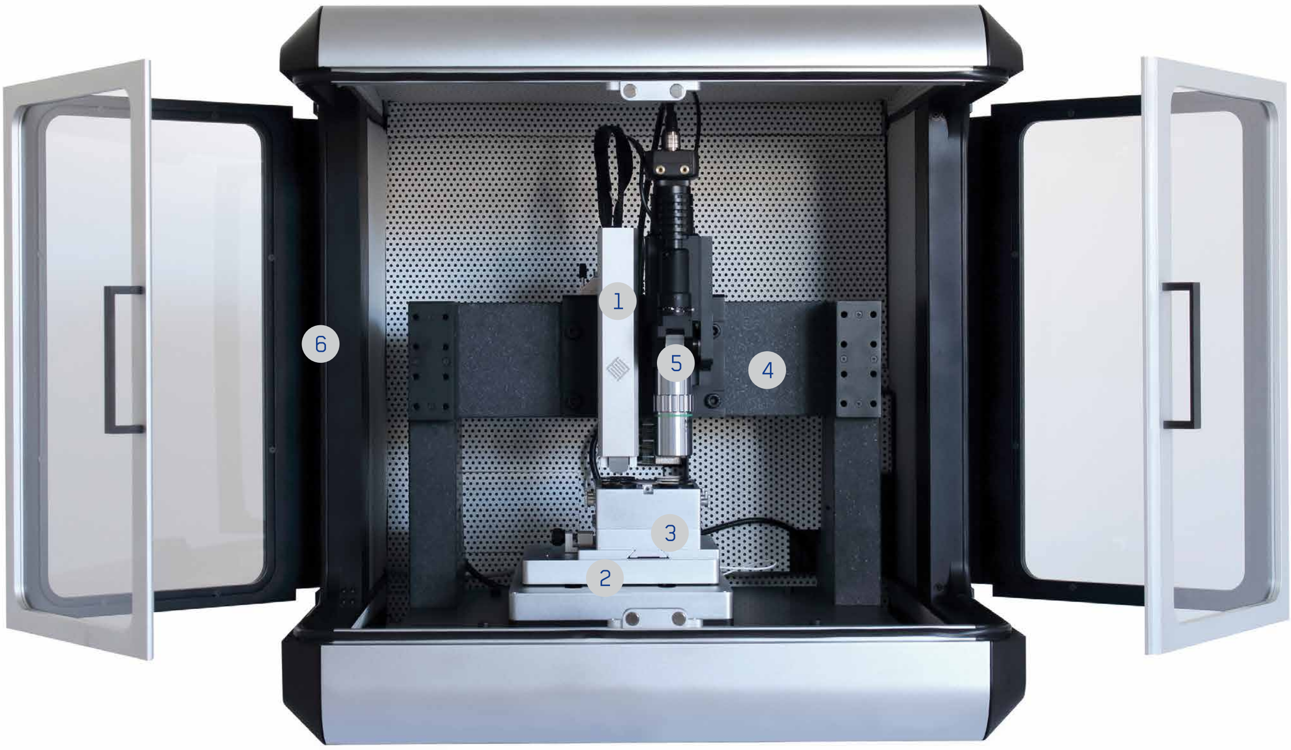
Typical applications include the quantification of hardness and elastic modulus as a function of indentation depth using the integrated continuous stiffness measurement (CSM) mode, as well as the high-resolution mapping of mechanical properties. Furthermore, optional modules enable scratch and wear testing, high-resolution scanning probe microscopy (SPM), and high temperature testing (coming soon).

With an unmatched noise floor of only 500 pN in force (guaranteed real world values) and 50 pm in displacement (guaranteed real world values) and comparatively large ranges of 200 mN and 20 µm, the Femto-Indenter enables the comprehensive study of mechanical behavior of materials over many length scales with an unprecedented accuracy and repeatability.

UNMATCHED SPATIAL RESOLUTION



- Quenching and Partioning (Q&P) steel
- 250 x 250 indents (62'500 indents)
- 20 nm indentation depth
- 200 nm spatial resolution



SYSTEM COMPONENTS

① Measurement head (interchangeable) consisting of:

- Long-range positioner for the fast and accurate approach of the nanoindentation tip to the sample surface over a range of 40 mm with a position-sensing resolution of 1 nm
- Piezo-scanner for high-resolution, position-controlled nanomechanical testing of material. It features a continuous movement range of 20 μm and a guaranteed noise floor of under 50 pm
- FT-S Micro-Force Sensing Probe (interchangeable) with various tip geometry and materials options, enabling the measurement of forces up to 200 mN (FT-S200'000) and down to 500 pN (guaranteed noise floor of the FT-S200)

② 2-axis sample stage for the accurate positioning of the sample under either the microscope or the nanoindenter head. The stage provides a travel range of 130 x 130 mm and features a closed-loop position control with a noise floor of 1 nm

③ Interchangeable sample tray for up to 6 samples and 1 fused silica reference specimen

④ High-stiffness granite measurement frame

⑤ High-resolution, top-down optical microscope with coaxial illumination for sample observation and easy selection of the indentation locations with 5, 10, 20, and 50× magnification lenses available

⑥ Enclosure for acoustic shielding and environmental control

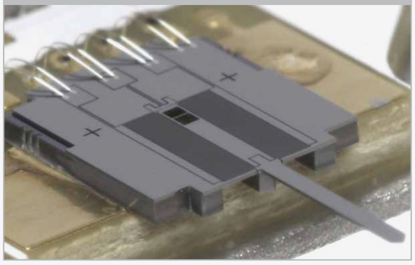
FT-I04 FEMTO-INDENTER

FEATURES

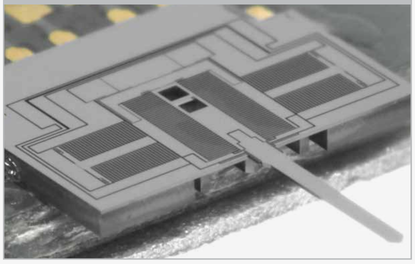
- High-resolution nanoindentation with unmatched repeatability to detect even the smallest variations in hardness and modulus
- Intrinsic, position-controlled measurements enabling the direct recording and quantification of fast plasticity and fracture events (optionally, force-controlled measurements are possible, as well)
- Displacement sensing range of 20 µm with a guaranteed noise floor of less than 50 pm (in fine mode) and up to 40 mm with a 1 nm noise floor (in coarse mode), spanning 9 orders of magnitude
- Interchangeable force sensing probes with various indenter tip geometries and materials enabling force-sensing from 500 pN to 200 mN in both tension and compression, covering 9 orders of magnitude
- Highest stiffness nanoindenter on the market with the highest dynamic range, featuring load cells with a resonant frequency of up to 50 kHz and a data acquisition rate of 96 kHz
- Integrated procedures for the fast and accurate calibration of the nanoindenter tip geometry (area function calibration) from ultra-shallow depths to microns
- SPM imaging of surfaces, scratches, and residual indents enabling the quantification of surface deformation and topology such as slip steps, pile-up, or sink-in
- High-resolution nano-scratch, nano-wear, and nanofriction measurement using the optional Scratch Testing Module in combination with the 2-axis Microforce Sensing Probe
- Extensive software tools for data analysis and visualization with customizable fits and functions for the extraction and visualization of test parameters and material properties

MEMS-BASED NANOINDENTATION

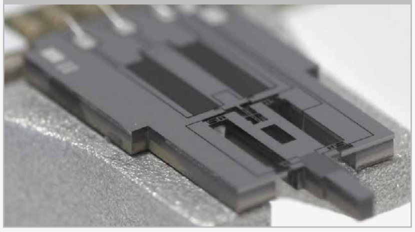
1-Axis Microforce Sensing Probe



2-Axis Microforce Sensing Probe



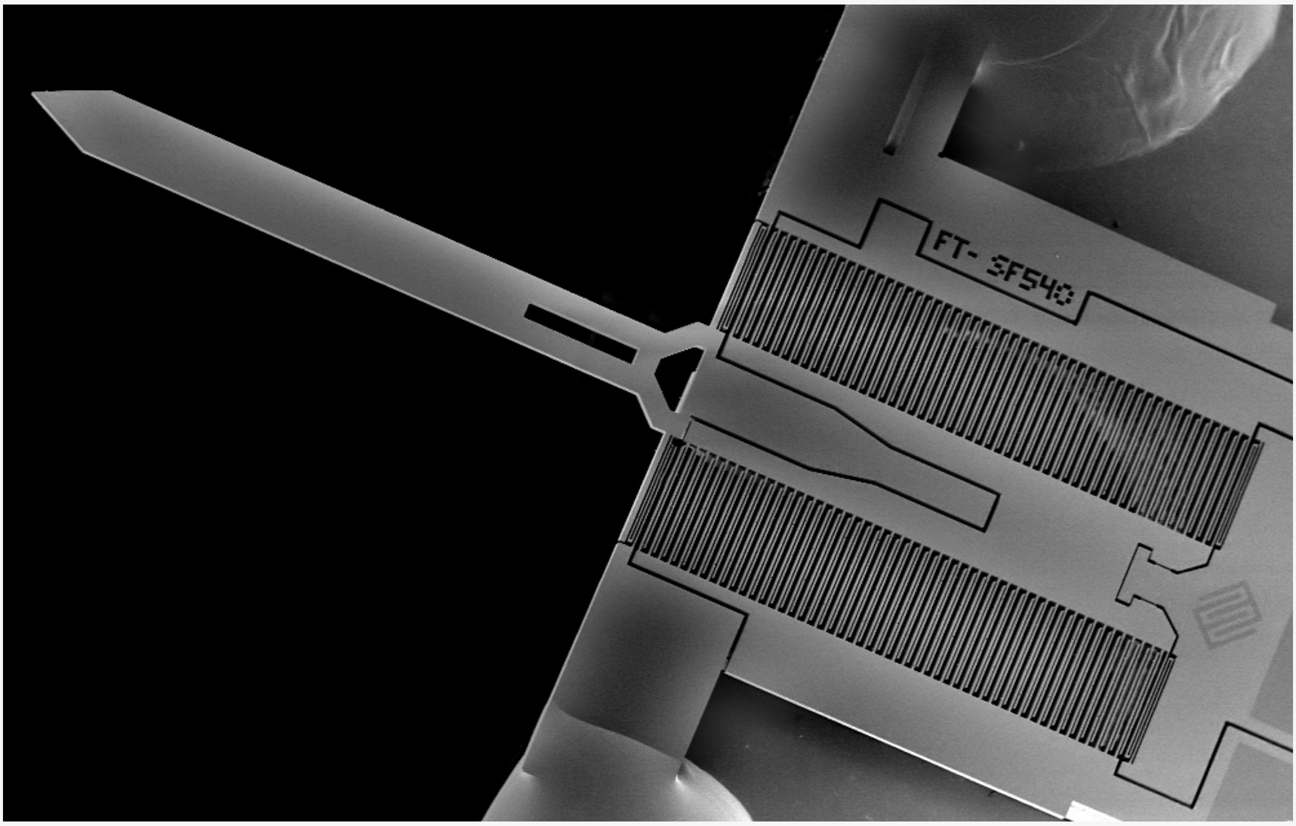
1-Axis Microforce Sensing Probe with integrated tip heater



The FT-I04 Femto-Indenter is a high-resolution nanoindenter based on Micro- Electro- Mechanical Systems (MEMS) technology.

While typical nanoindentation systems feature force-sensing technologies based on precision-machined and assembled components, FemtoTools is using semiconductor fabrication technology to machine the entire force sensor out of single crystal silicon wafers. This approach enables the fabrication of much smaller structures, making load cells with high sensitivity, resolution, and repeatability therefore overcome the limitations oftraditional technologies. Furthermore, the small size of the MEMS sensing element results in a mass orders of magnitude lower than conventional load cells. In combination with the high stiffness of silicon flexures, the FemtoTools FT-S Microforce Sensing Probes provide a high natural frequency (up to 50 kHz) and the related ability to measure fast events or to conduct fatigue or cyclic tests at high frequencies.

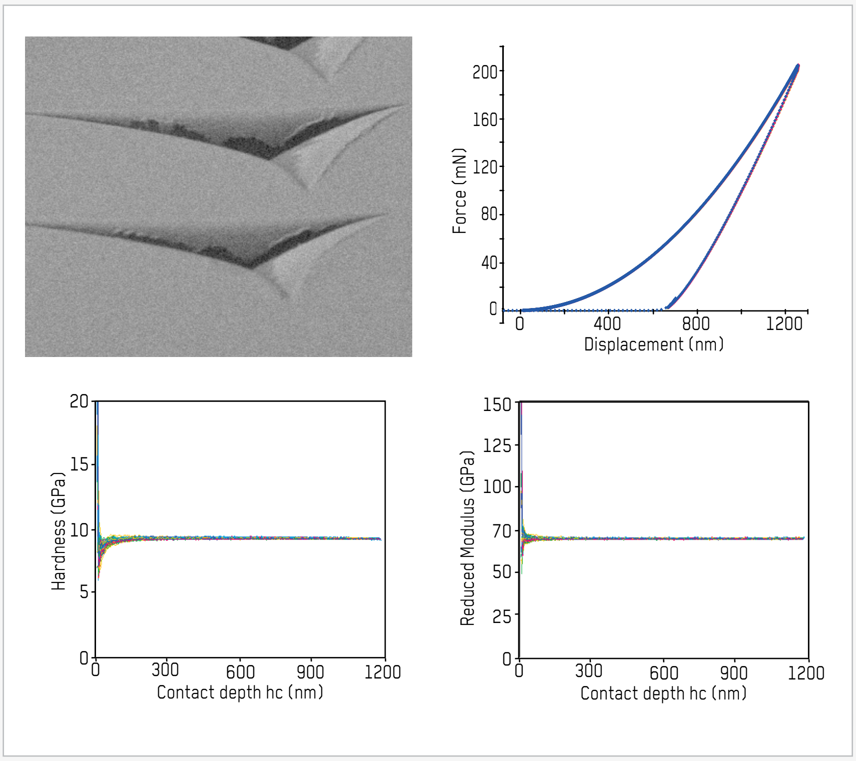
MEMS-BASED MICROFORCE-SENSING PROBE



APPLICATION EXAMPLES

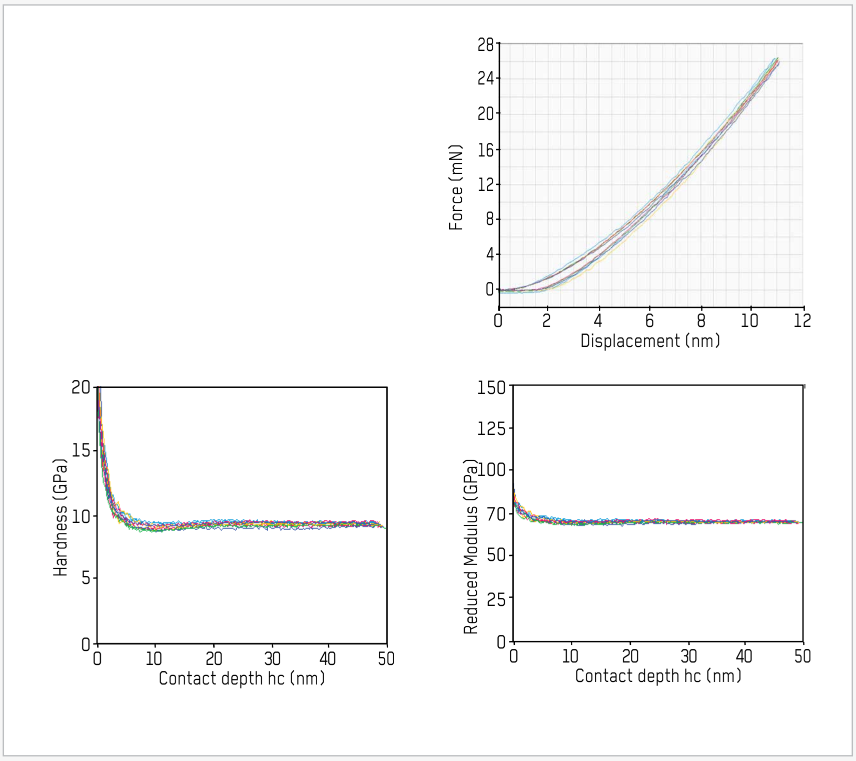
NANOINDENTATION

The Femto-Indenter enables the measurement of hardness, modulus, creep, stress relaxation, and fracture toughness of a wide range of materials from bulk metals and ceramics to soft polymers, thin films, and hard coatings. This is done by instrumented indentation of a sharp tip with a known or calibrated shape into a material, while recording the force required to penetrate to a given depth. The Femto-Indenter features standard indentation (ISO 14577) and automated procedures for quick tip shape calibration. The integrated continuous stiffness measurement (CSM) mode enables the measurement of mechanical properties as a function of the indentation depth. This benefits from a depth-sensing noise floor of 50 pm. As an example of the outstanding data quality and stability, the results from 100 indents into quartz glass are shown on the right.
Featuring intrinsic position control, the Femto-Indenter enables the quantification of fast stress drops, transients and high speed events with a quality that is not possible with force-controlled or softer systems.

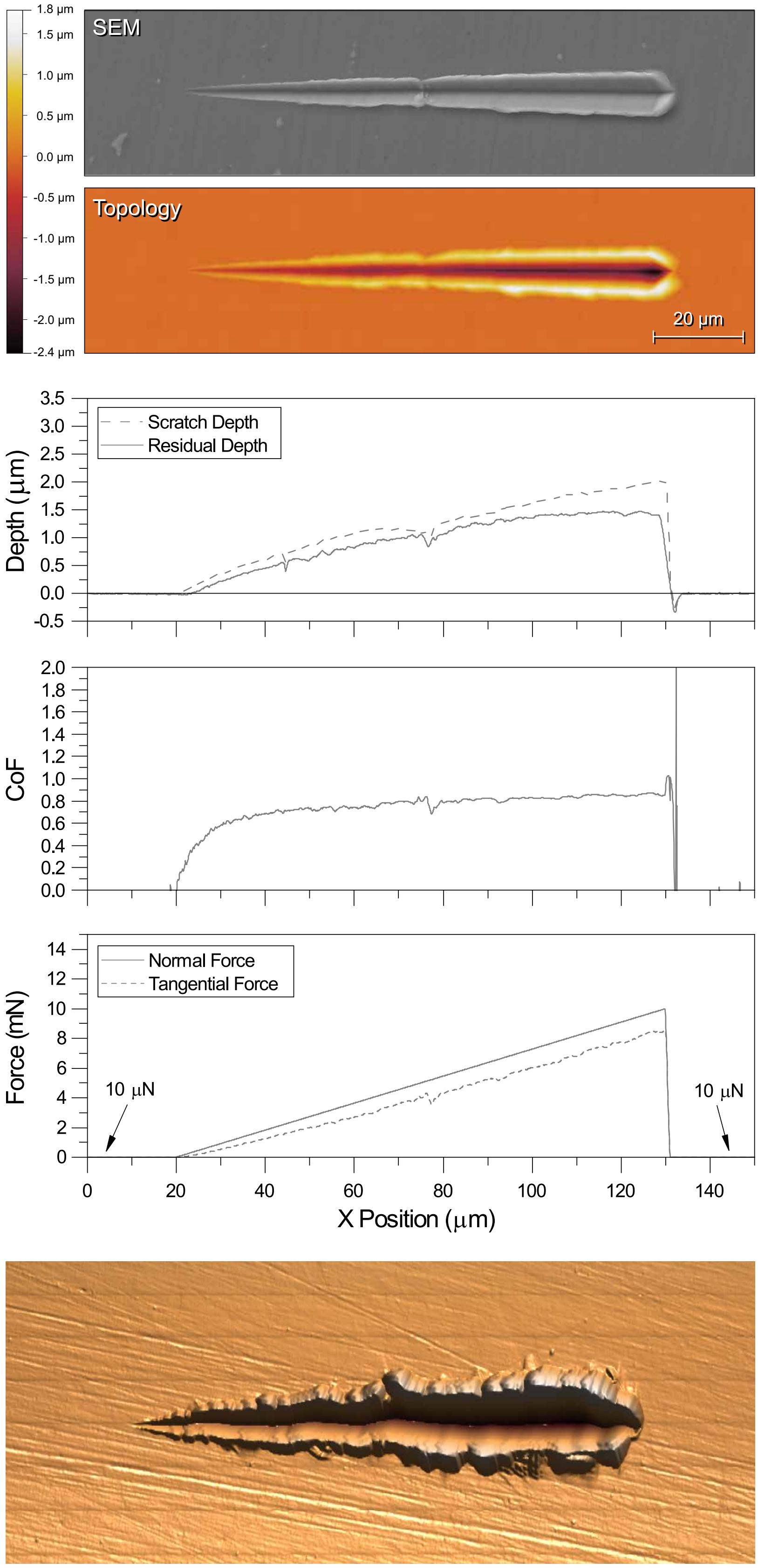


ULTRA-SHALLOW NANOINDENTATION

The Femto-Indenter is the ultimate nanoindenter for repeatable, high-resolution indentations at low loads and shallow depths. It enables the quantitative characterization of properties at the nanoscale, with applications in thin films, nanocomposites and nanostructures. Moreover, its intrinsic position control enables the characterization of low load discontinuities linked to dislocation events. Typically, indentation load-displacement behavior follows a Hertzian curve at low loads until the nucleation of dislocations and their subsequent interactions trigger fast stress drop events. The direct quantification of stress drop amplitudes enables the measurement of activation volumes and energies relevant to nanoscale plastic deformation mechanisms. Furthermore, the capacity for quantitative measurements at ultra-shallow depths is required for high-resolution mapping of properties.



SCRATCH AND TOPOLOGY MEASUREMENT



Sample Material: 5052 Aluminum Alloy

Speed: 1 µm/s

Maximum Load: 10 mN

Scratch testing is the oldest way to measure the hardness of materials. It was first quantified in 1812 by Friedrich Mohs, who developed a comparative scale which ranks common minerals in terms of their abrasion resistance or their Mohs Hardness number. Since then, scratch testing has developed into a modern technique. Modern scratch tests are typically performed using 3 passes: an initial topology scan performed at low force, a scratch performed with a progressively increasing load, and a final topology scan to determine the residual depths. A margin around the scratch is recommended to allow pile-ups in front and behind the scratch trace to be measured.

The Scratch Testing Module of the FT-I04 can quantify the abrasive resistance of a material, even at the nanoscale, by measuring the normal and tangential forces, the coefficient of friction (CoF), and the active and residual penetration depths of a scratch. This provides a wealth of information for determining relative scratch resistance and measuring the scratch hardness.

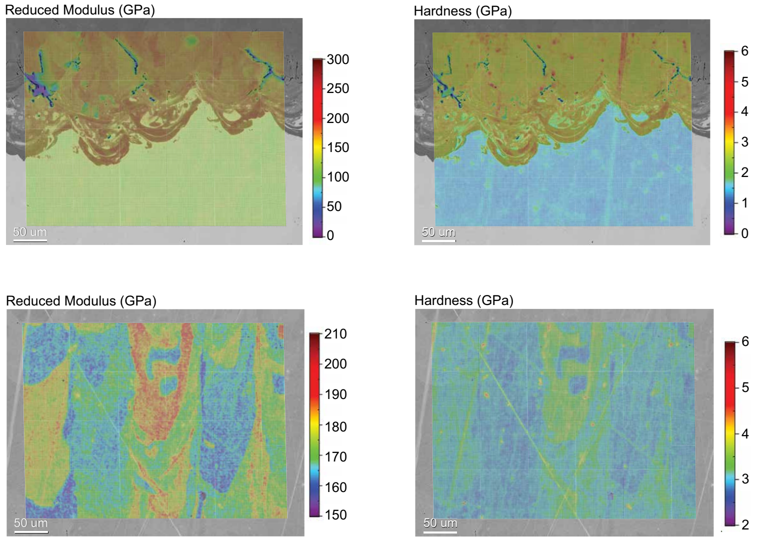
However, sometimes further information is also required, because the material around a scratch is also deformed and frequently forms pile-ups. To fully understand the scratch behavior, the full topology of the surface is needed. This is why we developed the topology scanning mode for the Scratch Testing Module of the FT-I04, which enables the full characterization of the surface topology after scratch or indentation testing using the same indenter tip.

In the figure to the left, topology and secondary electron (SEM) micrographs are shown for a scratch performed with a progressively increasing load to a maximum of 10 mN on a 5052 Aluminum alloy. Excellent correlation is seen between the electron micrographs and topology scans with a decrease in pile-up corresponding to an inclusion in the alloy clearly visible in the middle of the scratch. The effects of this inclusion can also be seen in the depth, coefficient of friction, and tangential load measurements.

APPLICATION EXAMPLES

MICROSTRUCTURAL CHARACTERIZATION OF ADDITIVELY-MANUFACTURED ALLOYS

Laser powder bed fusion (L-PBF) is a prominent method for additive manufacturing of metals. This technique uses a laser to locally melt powder and form solid voxels within a layer in a metal powder bed. As these voxels solidify, these melt zones crystallize into different crystal orientations depending on neighboring material and many other factors. Mechanical Microscopy allows characterization of these complex features due to their elastic and plastic anisotropy. In these results, the hardness and modulus maps are overlaid on back-scattered electron (BSE) micrographs, showing the influence of the melt zones and recrystallization on the mechanical properties of the material in both multi-material (Cu and 316L steel) and 316L steel deposits shown in cross-section.



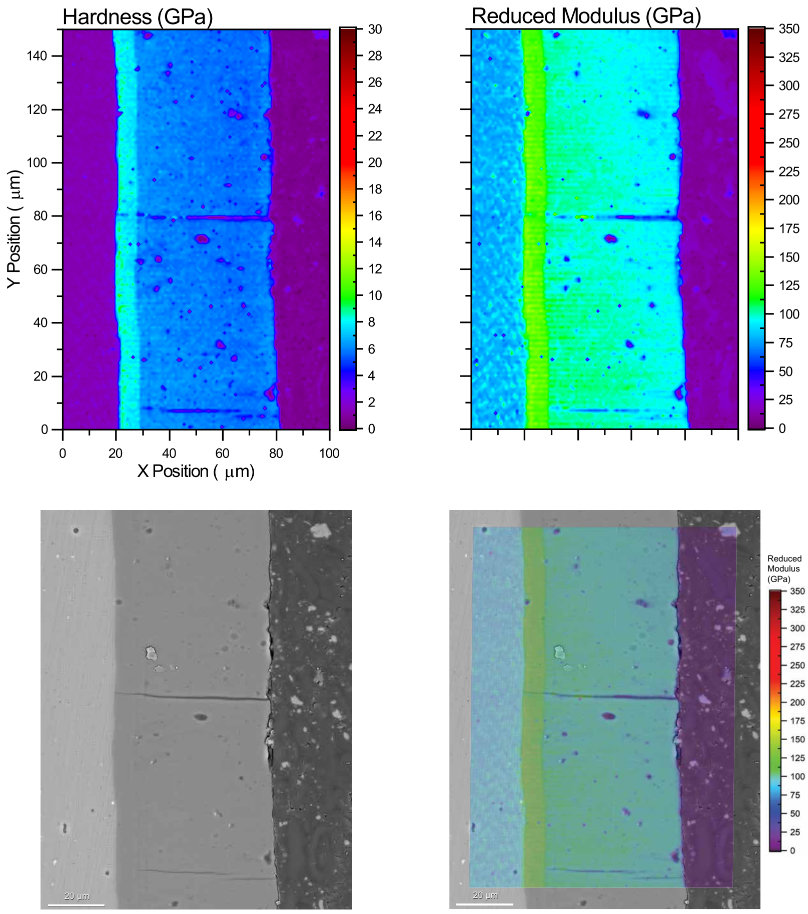
Sample Material: L-PBF 316L Steel & Cu
Origin: Prof. Markus Bambach of ETH Zurich, Switzerland

Indentation Depth: 200 nm
Inter-indent Spacing: 2 µm
Grid size: 400 × 300 µm

COATING CHARACTERIZATION

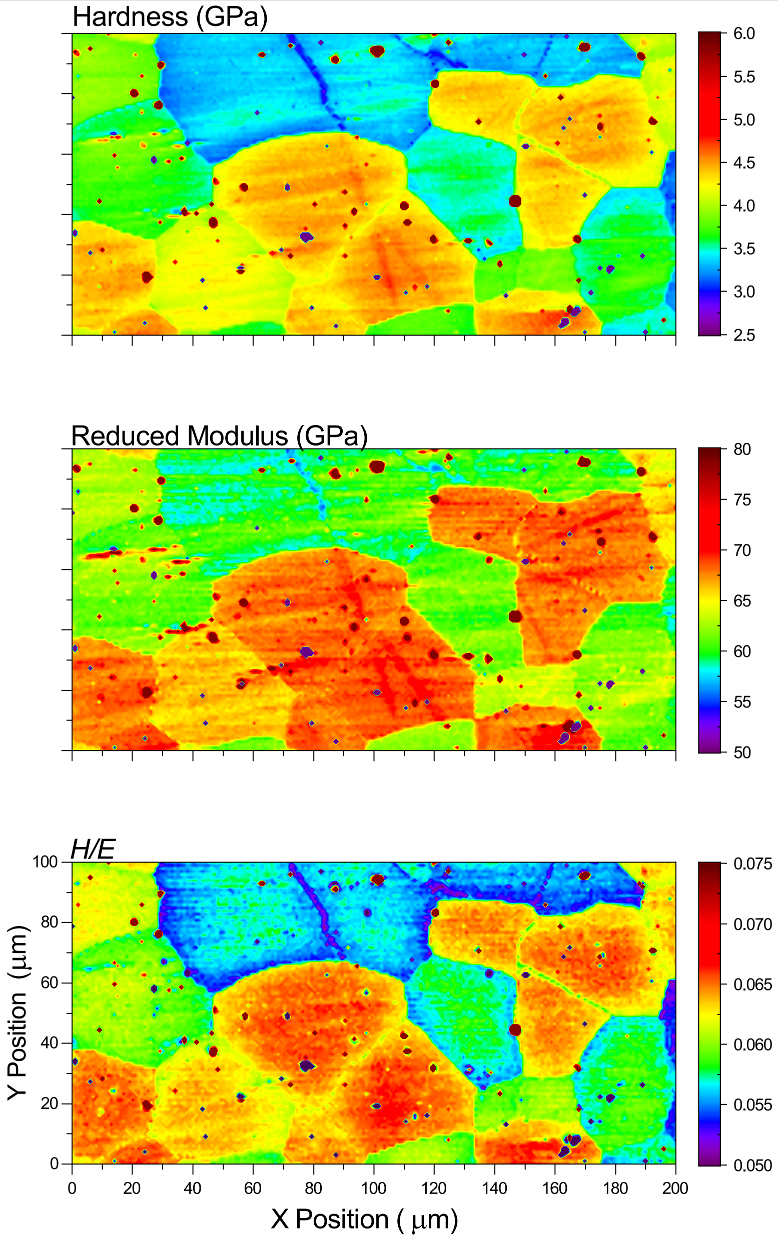
Nanoindentation has long been the method of choice for the characterization of coatings and surface modifications. For thin films, this is usually approached with top-down indentation. For thicker coatings, cross-sectional indentation can provide far more information. On the right, hardness and reduced modulus maps are shown of a metallurgically mounted and prepared cross-section of a hard anodized coating on a 5052 aluminum alloy. A backscattered electron (BSE) micrograph and an overlay of both techniques are also shown. The aluminum alloy substrate is to the left side, and the bakelite mounting material is on the right. Pores and cracks are clearly observable within the coating, with excellent correspondence between property maps and the electron micrograph.

However, even more information can seen in the indentation maps than in the electron micrographs! A higher density adhesion layer with higher hardness and modulus is seen at the aluminum interface, and a gradient in hardness and modulus can be seen towards the coating surface. This is valuable information for understanding mechanical behavior of coatings in abrasive environments.



Sample Material: Hard Anodized Aluminum
Origin: Dr. Suman Shrestha of Keronite, Ltd.
Indentation Depth: 100 nm

Inter-indent Spacing: 1 µm
Grid size: 150 × 100 µm



Sample Material: NiTi Shape Memory Alloy
Indentation Depth: 100 nm
Inter-indent Spacing: 1 µm
Grid size: 100 × 200 µm

To demonstrate the capability of the Femto-Indenter, results from CSM nanoindentation measurements on a Ni-Ti alloy are presented here. Ni-Ti alloys are a family of metallic alloys designed with near-equal atomic percentages of nickel and titanium that display two unique temperature-dependent properties: the shape memory effect and super-elasticity. Shape memory relates to their capacity to undergo plastic deformation and retain the deformation upon unloading until heating up to a critical temperature. Upon reaching this critical temperature, the alloy undergoes phase transformation and recovers from plastic deformation back to its original state. At temperatures higher than this critical temperature, Ni-Ti alloys exhibit super-elasticity where they undergo large deformations via reversible phase transformation and immediately recover on unloading. Since they combine high biocompatibility, corrosion resistance, and wear resistance, Ni-Ti alloys are amongst the most well-known shape memory alloys (SMA). They are used in a wide range of commercial applications from medical implants to intelligent reinforced concrete and damping components. Many research efforts are devoted to developing a full mechanistic understanding of the shape memory effect and its limitations, particularly, the relationship between crystal orientation and elastic modulus appears to be critical in explaining local strain accommodation and localization which leads to irreversible deformation and the progressive loss of the shape memory effect. Here, the FT-I04 Femto-Indenter enables the quantification of hardness and modulus variations with crystal orientation. Remarkably, it also enables quantification of the narrow and soft gradients of properties (<3%) near grain boundaries. Featuring a high spatial resolution over large areas and maintaining high precision even at high mapping speeds, FT-I04 results can be directly compared to EBSD and EDX maps (shown later) for a full understanding of the link between microstructures and materials properties.

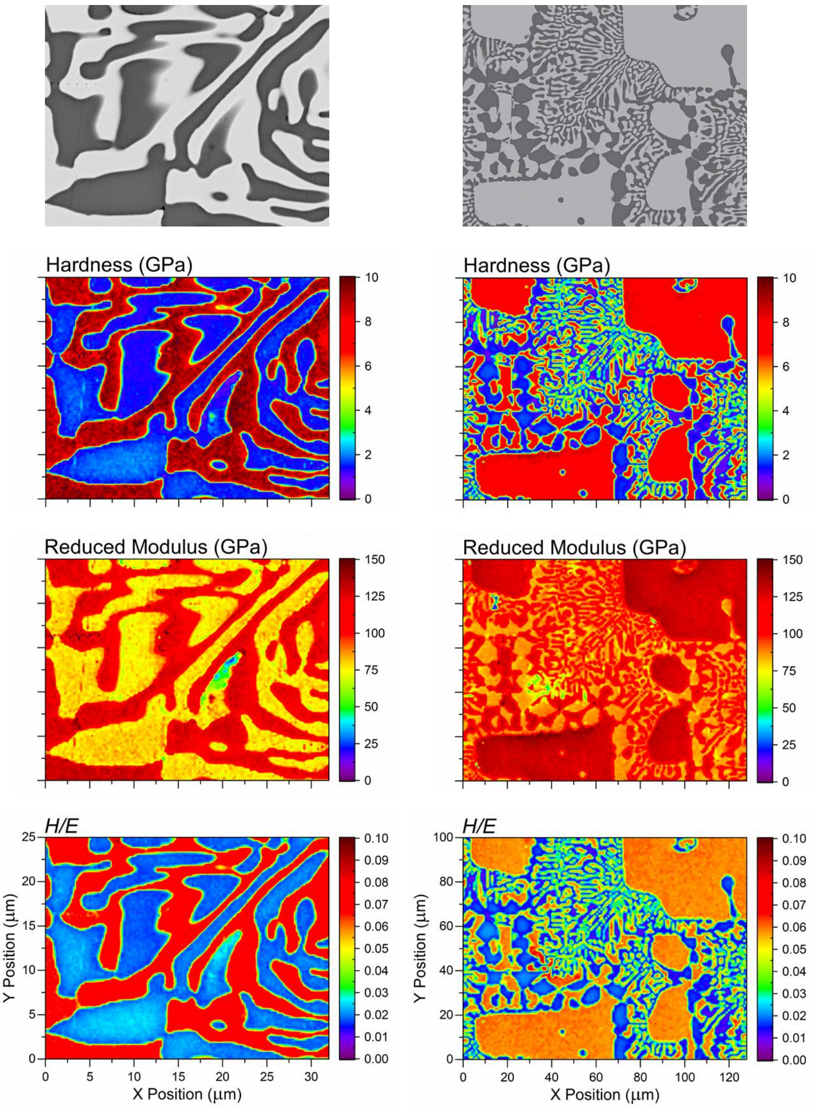
APPLICATION EXAMPLES

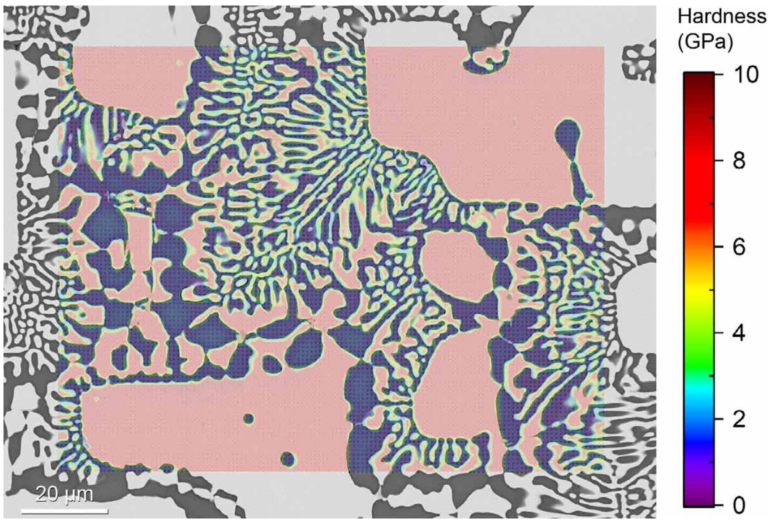
MULTI-SCALE MICROSTRUCTURAL MAPPING

In microstructures featuring phases with different properties, displacement-controlled indentation is essential for mapping, as a load-controlled indentation would produce indentations with large variations in size, sacrificing both accuracy and lateral resolution. Since indentation size effects can have a large effect on sample hardness, it is essential that indentations are all performed to similar depths to ensure accurate comparisons are possible. By performing indentations to targeted depths, indentation maps with high precision can be produced, even on very dissimilar materials. This allows high resolution indentation maps to be performed with great confidence.

For the indentation size and spacing it is important to consider the sample's microstructure. Indentations should be spaced such that more indentations are contained within a single phase than indentations which straddle phase boundaries. Indentations which are performed on a phase boundary measure a mixture of the properties of both phases. This means that the indentation spacing should be a fraction of the smallest microstructural feature to be resolved in the map. This can be challenging for fine microstructures like eutectic dendrites.

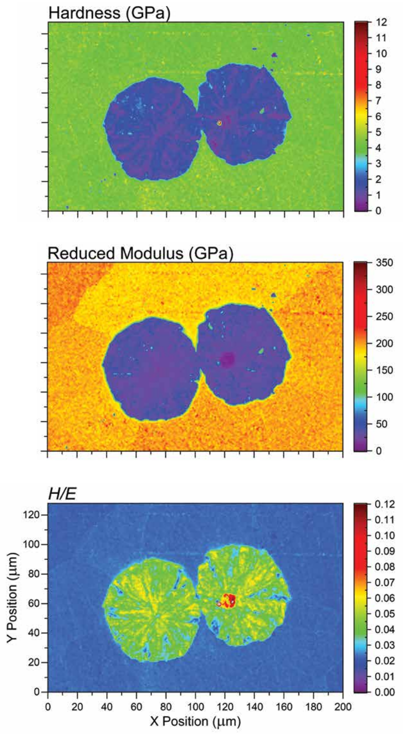
On the right, a backscattered electron (BSE) and mechanical micrographs of an Al-33 wt.% Cu eutectic alloy can be seen at two different length scales. At the bottom, an overlay of a hardness map on an electron micrograph highlights the excellent correspondence between both imaging techniques. The maps to the right were performed with a 100nm depth and 1 µm spacing, while the maps to the left were performed with 4× greater resolution at a 25nm depth and 0.25 µm spacing. In the larger scale map, many datapoints can be seen to be intermediate values (green coloring), which indicates the indentations straddle both phases. In the higher resolution hardness map, this is not observed. Nearly all indentations are red or blue, occurring within single phases. Even at depths of only tens of nanometers, the Femto-Indenter can deliver accurate, beautiful maps.

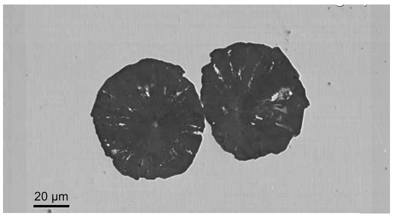


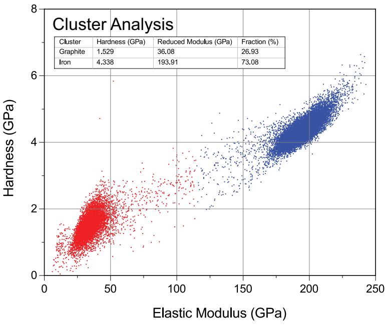


Sample Material: Al-33 wt% Cu Eutectic Alloy
Origin: Dr. Bin Gan of CISRI, Beijing
Indentation Depth: 25 & 100 nm
Inter-indent Spacing: 0.25 & 1 µm
Grid size: 128 × 100

CLUSTERING ANALYSIS OF MICROSTRUCTURAL PHASES







Cluster	Hardness (GPa)	Reduced Modulus (GPa)	Fraction (%)
Graphite	1.529	36.08	26.93
Iron	4.338	193.91	73.08

Sample Material: Nodular Cast Iron

Origin: Dr. Carl Dahlberg of KTH, Sweden

Indentation Depth: 100 nm

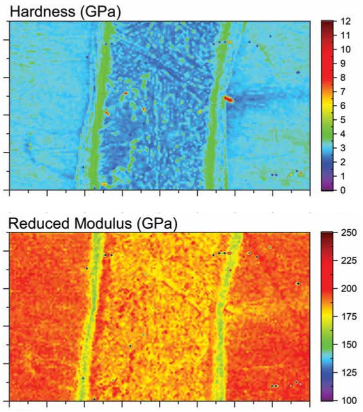
Inter-indent Spacing: 1 µm

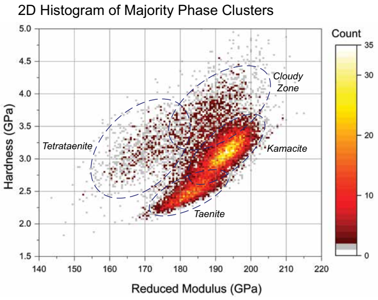
Grid size: 128 × 200 µm

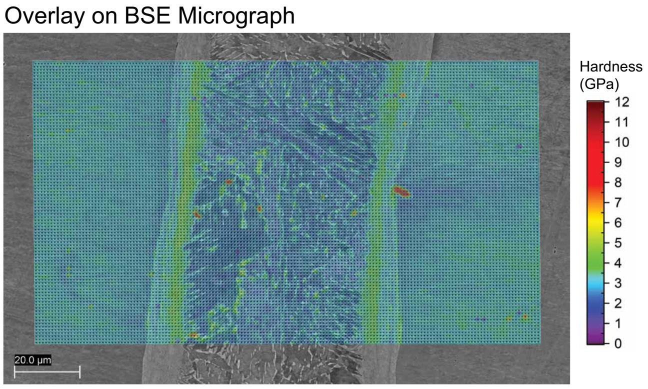
In microstructures with phases with very different properties, displacement-controlled indentation is essential for mapping, as a load-controlled indentation would produce indentations with large variations in size. This sacrifices both accuracy and lateral resolution. By performing indentations to targeted depths, indentation maps with regular sizes can be produced with high precision, even on very dissimilar materials like the Nodular Cast Iron sample shown in this example. Both iron and graphite phases are clearly resolved, despite their difference in elastic modulus of nearly an order of magnitude.

This difference makes statistical analysis of the phase's properties simple, as the K-means clustering algorithm implemented in the FemtoTools analysis software can easily separate the two phases and determine their average properties and phase fractions.

OUT OF THIS WORLD SENSITIVITY







Sample Material: Muoniunalusta meteorite fragment

Origin: The Meteorite Exchange

Indentation Depth: 100 nm

Inter-indent Spacing: 1 µm

Grid size: 160 × 90 µm

Meteorites are one of the most tangible objects of outer space. They are a piece of another world that you can hold. To the left, you can see property maps taken from the Muoniunalusta meteorite. The mapped region consists of a single Plessite band between two Kamacite laths with a border of Tetrataenite and Cloudy Zone phases. The high sensitivity of the FT-I04 FemtoIndenter allows clear observation of the different phases in the spinodally-decomposed Plessite band. However, since the properties of these phases are convoluted, K-means clustering is difficult for these materials. Instead, 2D Histograms are a simple statistical method to show the datapoint concentration within a bin range. These allow us to visualize the relative concentrations of properties for different phases. Advanced statistical methods or Correlative methods using EDX are required to deconvolute the various phase properties with high confidence.

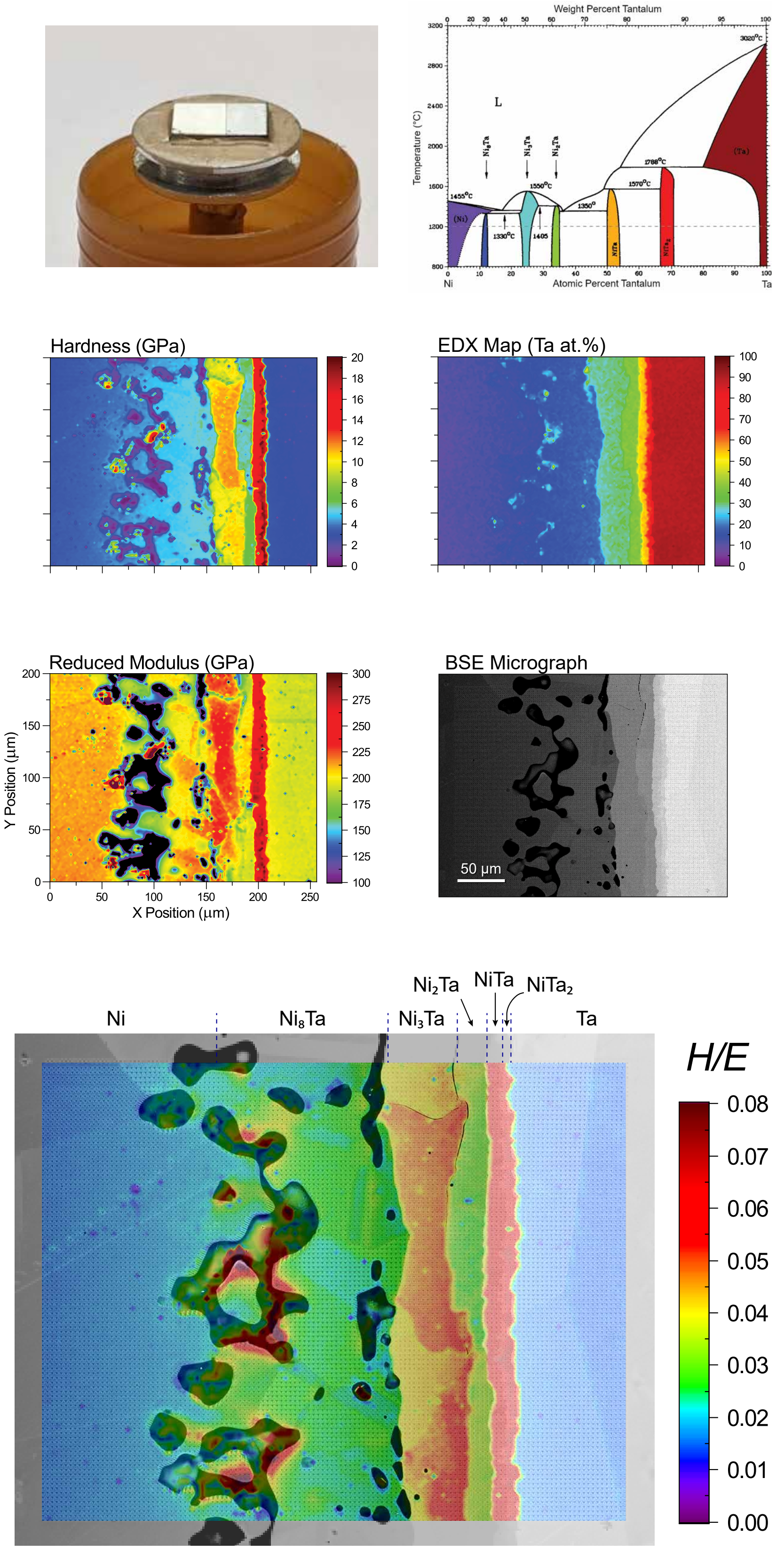
APPLICATION EXAMPLES

CORRELATIVE NANOINDENTATION AND EDX MAPPING

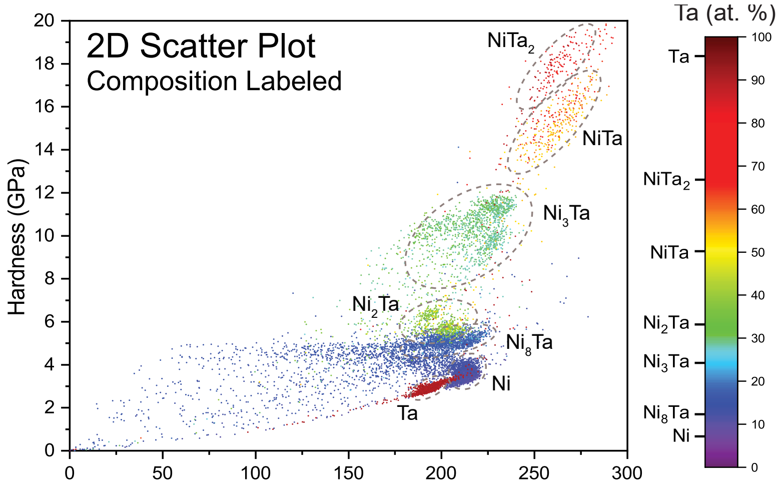
Nanoindentation and energy-dispersive X-ray spectroscopy (EDX) have long been complementary techniques. However, in order to correlate between the two techniques, you always had to either analyse each location manually (0D) or fit some linear relationship of properties along a line (1D). This can limiting for investigations on complex microstructures.

With Mechanical Microscopy, both hardness and elastic modulus can now be characterized over an area (2D) with high spatial resolution. This generates a large volume of statistical data, which can be used for determining phase-level properties. However, segmenting the data between phases can be really challenging if the statistical distributions of the properties overlap. To solve this, we developed a simple method to correlate EDX and nanoindentation data, so that we have composition and mechanical properties data for every point in the maps. This involves performing a medium sized map, approximately 100 by 100 indents, with aspect ratio dimensions that match your EDX system: e.g. 128 by 100 for EDAX/TEAM software. This facilitates sampling alignment for the two techniques, so that the location of each indentation and EDX measurement perfectly coincide. This effectively combines the high-resolution mechanical property mapping capability of the FTI-04 Femto-Indenter with the compositional mapping ability of electron microscopy.

To demonstrate the power of this correlative technique for combinatorial investigations, in collaboration with Prof. Ralph Spolenak at ETH Zürich and Dr. Bin Gan at the China Iron & Steel Research Institute Group (CISRI), we demonstrated this method on the Ni-Ta system using a diffusion couple as a combinatorial library. All seven phases in the system are clearly resolved in the resulting maps, and the mechanical properties and composition ranges for each phase are determined. This allows easy data segmentation without requiring complicated statistical deconvolution methods.



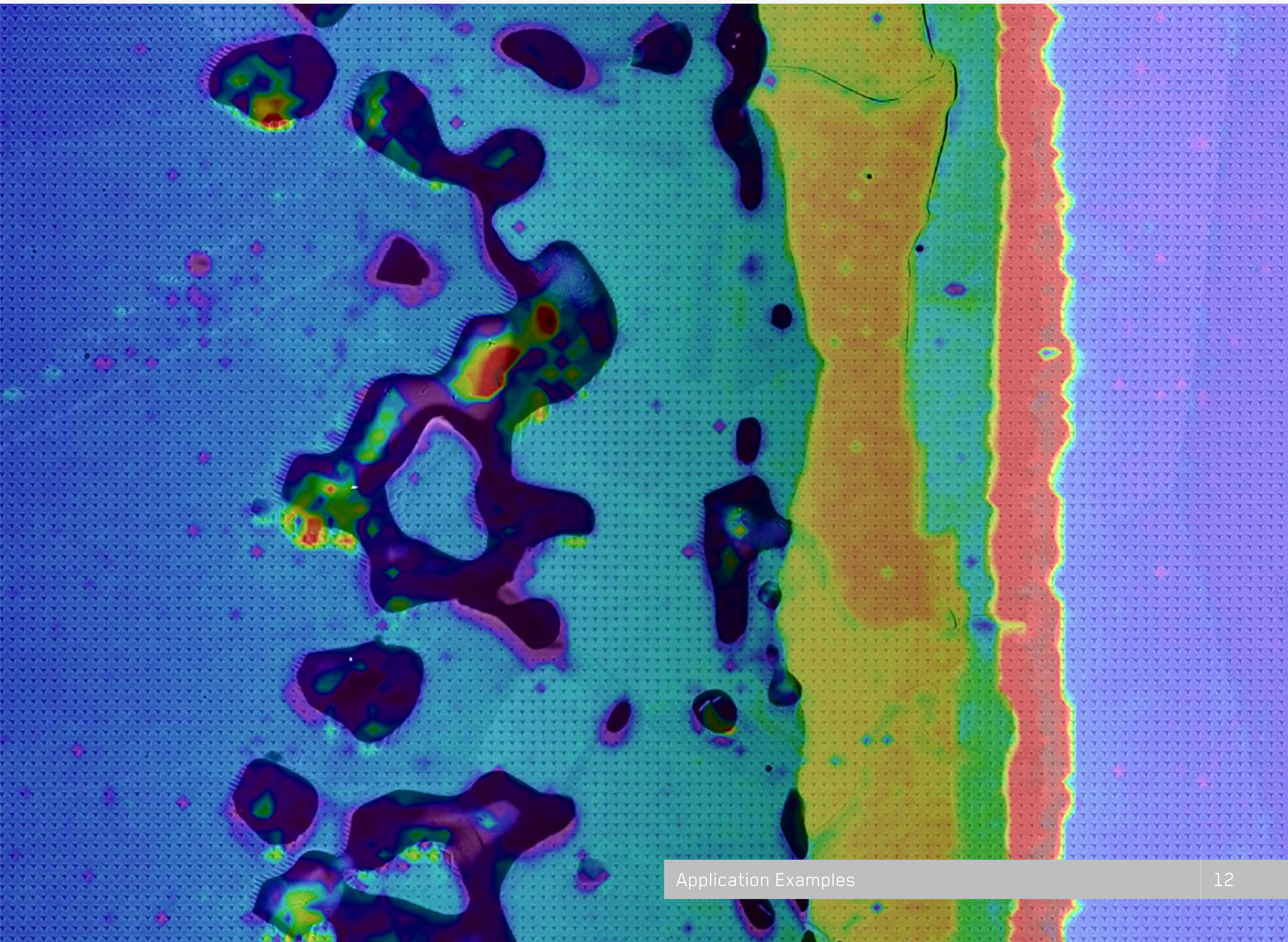
Wheeler, J. M., Gan, B., Spolenak, R., Combinatorial Investigation of the Ni-Ta System via Correlated High-Speed Nanoindentation and EDX Mapping. Small Methods 2021, 2101084. https://doi.org/10.1002/smtd.202101084



Sample Material: Ni-Ta Diffusion Couple
Origin: Dr. Bin Gan of CISRI, Beijing
Indentation Depth: 100 nm
Inter-indent Spacing: 2 µm
Grid size: 256 × 200 µm

Without this correlative method, phase deconvolution of the Ni–Ta system would be challenging due to several phases possessing adjacent compositions and mechanical properties. This demonstrates the potential of this new correlative approach for future investigations, particularly those involving complex microstructures and/or compositional variation.

OVERLAY OF HARDNESS MAP ON BSE MICROGRAPH



ACCESSORIES

FT-S MICROFORCE SENSING PROBES

The FemtoTools FT-S Microforce Sensing Probes are sensors capable of measuring forces from sub-nanonewtons to 200 millinewtons along the sensor's probe axis. Both compression and tension forces can be measured. Single SI-traceable pre-calibration for each probe in combination with an outstanding long-term stability guarantees significantly higher measurement accuracy than other force sensing systems in this force range. Specialized versions are also available, including 2-Axis Microforce Sensing Probes or Tip Heating.

The FT-S Microforce Sensing Probes are available with a wide variety of tip materials and geometries including diamond Berkovich, cube corner, flat punch, wedge, conical and more.

Model	Range	Noise Floor(10Hz)
FT-S200	+/- 200 µN	0.5 nN
FT-S2'000	+/- 2'000 µN	5 nN
FT-S20'000	+/- 20'000 µN	50 nN
FT-S200'000	+/- 200'000 µN	500 nN
FT-S20'000-2Axis	+/- 20'000 µN (normal)	100 nN
	+/- 20'000 µN (tangential)	100 nN

FT-ST04 SCRATCH TESTING MODULE

The Scratch Testing Module consists of an exchangeable sample stage with an integrated piezoscanner. This enables the in-plane movement of the sample while simultaneously applying a normal force. Combined with the FemtoTools 2-Axis Microforce Sensing Probe, this module enables nanoindentation, nanoscratch and nano-wear testing, as well as SPM imaging of surface roughness, high-aspect ratio features and residual scratches or indents.

HIGH TEMPERATURE MODULE

The High Temperature Module (coming soon) enables heating of specimens. In combination with the FemtoTools Tip Heating Sensors, it makes isotherm nanomechanical testing of samples at various temperatures possible. It enables not only the measurement of materials properties evolution as a function of temperature, but also the quantitative study of thermally-induced changes in plastic deformation and fracture mechanisms at the nanoscale.

TECHNICAL SPECIFICATIONS

MEASUREMENT HEAD

Measurement principle	**position controlled**

Force	
Range	up to 200 mN
Noise floor (10 Hz)	down to 500 pN
Digital resolution	down to 0.5 pN
Sampling frequency	96 kHz

Position	
Coarse range	40 mm
Coarse noise floor (10 Hz)	1 nm
Fine range	20 µm
Fine noise floor (10 Hz)	50 pm
Digital resolution	0.05 pm
Sampling frequency	96 kHz

SAMPLE STAGE

Range	130 x 130 mm
Noise floor (10 Hz)	1 nm

MICROSCOPE

Camera	5 megapixel CMOS sensor
Objective lens options	5x, 10x, 20x, 50x
Focus	motorized
Coaxial illumination	LED, Adjustable

FemtoTools AG
Furtbachstrasse 4
8107 Buchs / ZH
Switzerland

T +41 44 844 44 25
F +41 44 844 44 27

info@femtotools.com
www.femtotools.com