OXFORD INSTRUMENTS

OPERANDO NANOINDENTATION

iX05

Product Brochure

NANOINDENTATION

IN OPERANDO CONDITIONS

The iX05 is an operando nanoindenter. It is designed to measure local mechanical properties in real-world conditions such as high temperatures, low temperatures, high strain rates, or in liquid environments.
Traditional nanoindenters are optimized to measure materials in laboratory conditions. However, materials in practical applications often experience extreme environments that strongly influence their mechanical behavior. The iX05 is optimized to test materials in conditions representative of their targeted application. For example, gas turbine materials operate at high temperature, therefore they need to be tested in those conditions. Satellite components operate in vacuum with large temperature gradients ranging from -150°C to +150°C. Consequently, they need to be tested under those conditions. In automotive, materials used in the crumple zone must be evaluated under high strain rates to simulate crash conditions.
Over the last decade, nanoindentation technology has evolved from simply measuring the local mechanical properties of materials to mapping material properties over large areas by performing hundreds of thousands of indents in a well-spaced grid. This technique, known as mechanical microscopy, helps materials scientists bridge the gap between microstructure, features, and bulk performance of materials.
The iX05 combines the high resolution and speed offered by MEMS-based technology with fast control electronics and environment control, making it the ultimate tool for measuring and visualizing mechanical properties of materials in a wide range of operando conditions.

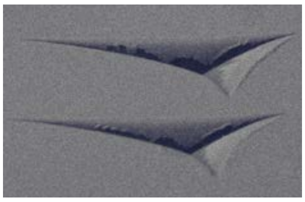
FEATURES

- Ultra-high resolution nanoindentation with unmatched repeatability to detect even the smallest variations in hardness and modulus
- Ultra-fast nanoindentation at speeds up to 30 indentations per second
- High-temperature nanoindentation with active tip heating for isothermal testing at temperatures up to +800°C
- Cryogenic nanoindentation at temperatures down to -150°C
- Motorized chamber with environment control: inert gas atmosphere or high vacuum to minimize oxidation and contamination of the loading tip and sample surface
- Displacement sensing with noise floor below 5 pm over the range of 20 μm, spanning 7 orders of magnitude
- Interchangeable MEMS sensors for SI-traceable force metrology from 100 pN (noise floor) to 2 N, covering 10 orders of magnitude
- Unmatched performance at ultrahigh speeds: FemtoTools MEMS force sensor (resonance at up to 100 kHz) combined with ultra-fast electronics (2 MHz sampling rate, 500 kHz feedback frequency)
- Intrinsic, position-controlled measurements (strain-rate and force-controlled measurements also available)
- Automated, high-precision calibration procedures for seamless use

TESTING MODES

NANOINDENTATION

Nanoindentation

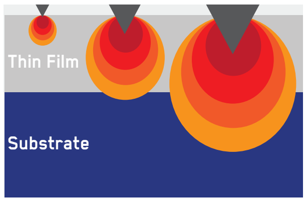


- Simple indentation
- CSM indentation
- Load range to 2 N

Scratch Testing

- Scratch testing
- Scanning Probe Microscopy (SPM)
- Tribological measurement

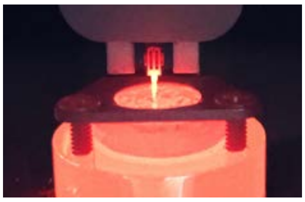
Thin Film Testing



- Ultra-thin film property measurement
- Bond-strengh measurement

OPERANDO INDENTATION

High-Temperature Testing

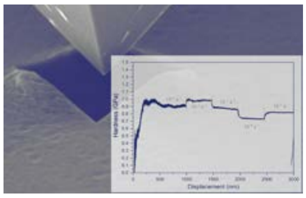


- Up to 800˚C (with tip heating)
- High vacuum
- Testing 3 samples in parallel

Cryogenic Testing

- Down to -150˚C
- High vacuum

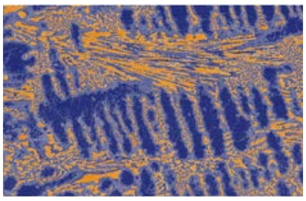
High-Strain Rate Testing



- Constant strain rate
- Impact testing
- Strain-rate jump testing

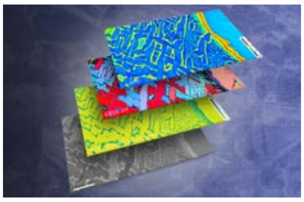
MECHANICAL MICROSCOPY

Mechanical Microscopy (Nanoindentation Mapping)



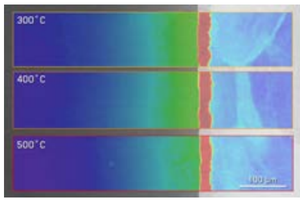
- High resolution (<200 nm)
- Ultra-high speed (>30 indents/s)
- High-sensitivity

Correlative Mechanical Microscopy



- Correlate with EDS
- Correlate with EBSD
- Correlate with AFM

Temperature-Dependent Nanoindentation Mapping

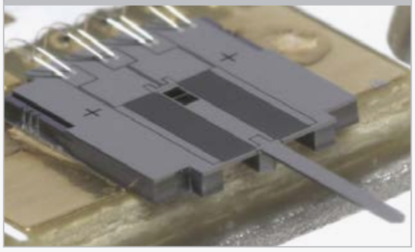


- Quantify changes in mechanical properties and microstructures from -150˚C to +800˚C

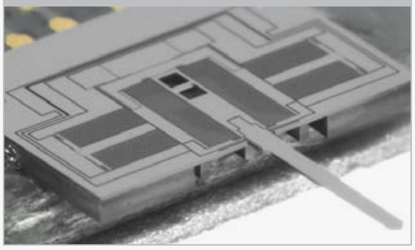
SYSTEM BUILDUP

MEMS-BASED NANOINDENTATION

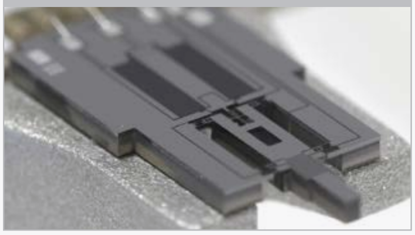
1-Axis Microforce Sensing Probe



2-Axis Microforce Sensing Probe



1-Axis Microforce Sensing Probe with integrated tip heater



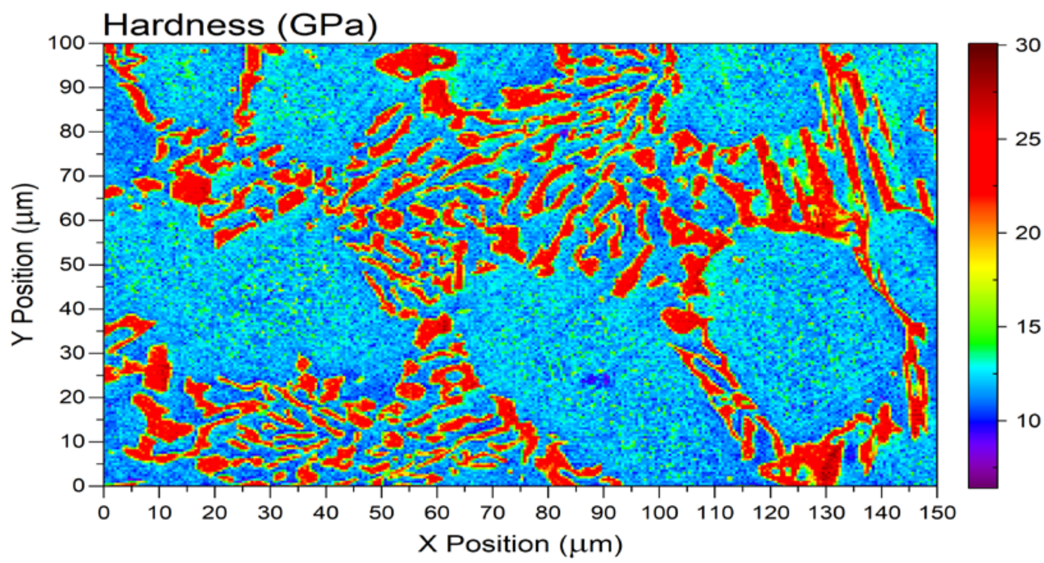
The iX05 is an operando nanoindenter that measures and visualizes the mechanical properties of materials at the nano- and micro-scale under different testing conditions.

It uses MEMS technology and a newly developed FPGA-based controller (sampling rate of 2 MHz and feedback-loop frequency of 500 kHz) to achieve high resolution over a large dynamic range. This allows for the quick generation of mechanical microscopy maps. In less than an hour, it can generate large maps of 100'000 indents, making it an efficient and data-rich metallographic technique.

Unlike conventional nanoindenters, the iX05 has an integrated vacuum chamber for conducting measurements in a high-vacuum environment. This significantly reduces oxygen content compared to inert gas shielding and eliminates oxidation issues during high-temperature experiments, as well as condensation problems during low-temperature testing.

With advanced software control, the iX05 provides a versatile platform for analyzing the mechanical properties of materials at the micro- and nano-scale.

ULTRA-FAST NANOINDENTATION MAPPING

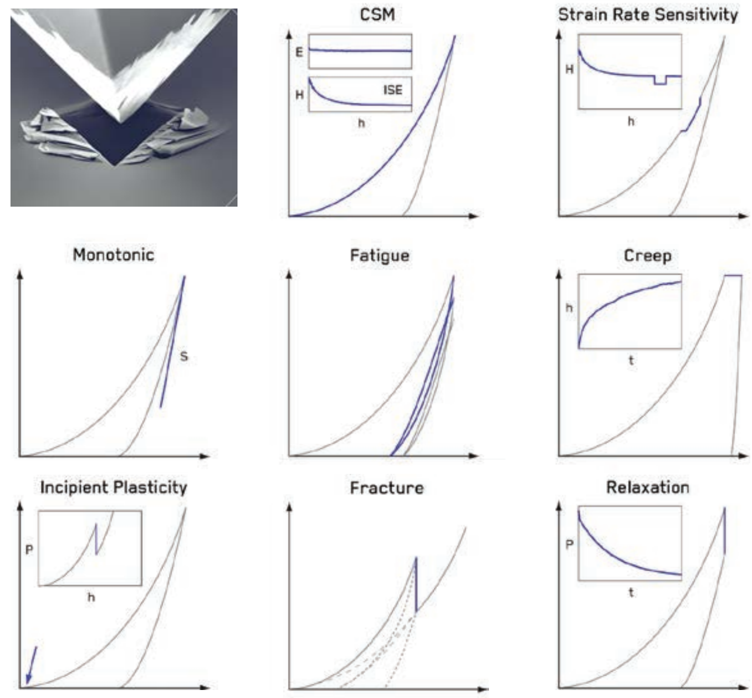


Sample Material: High-chromium Cast Iron
Curtesy of: Prof. M. Guitar, Saarland University
Indentation Depth: 50 nm
Inter-indent Spacing: 500 nm
Number of Indents: 60'000
Indentation Speed: 30 indents/s

APPLICATION EXAMPLES

NANOINDENTATION

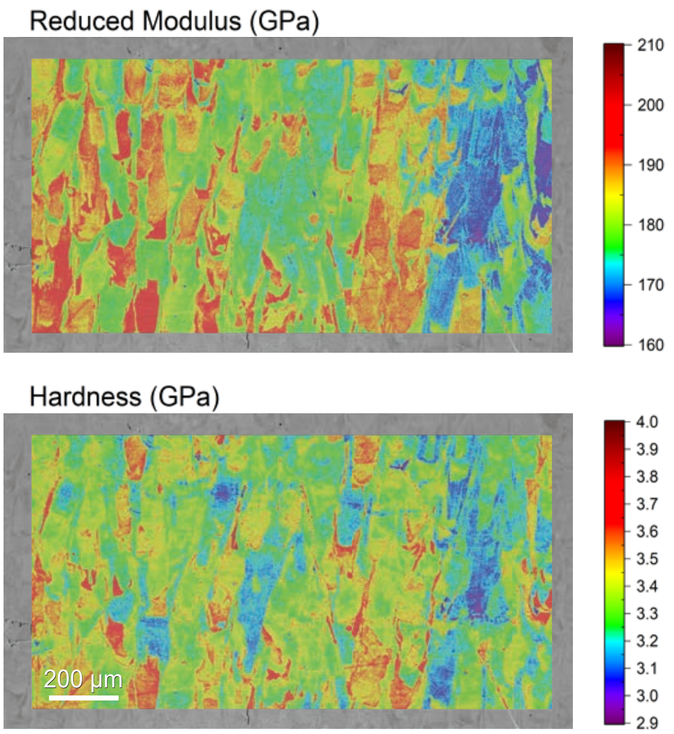
The iX05 enables the measurement of hardness, modulus, creep, fatigue, and fracture toughness of a wide range of materials from bulk metals and ceramics to soft polymers, thin films, and hard coatings. This is done by instrumented indentation. A sharp tip of known or calibrated shape is pressed into a material, while recording the force required to penetrate to a given depth. The iX05 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 less than 5 pm.



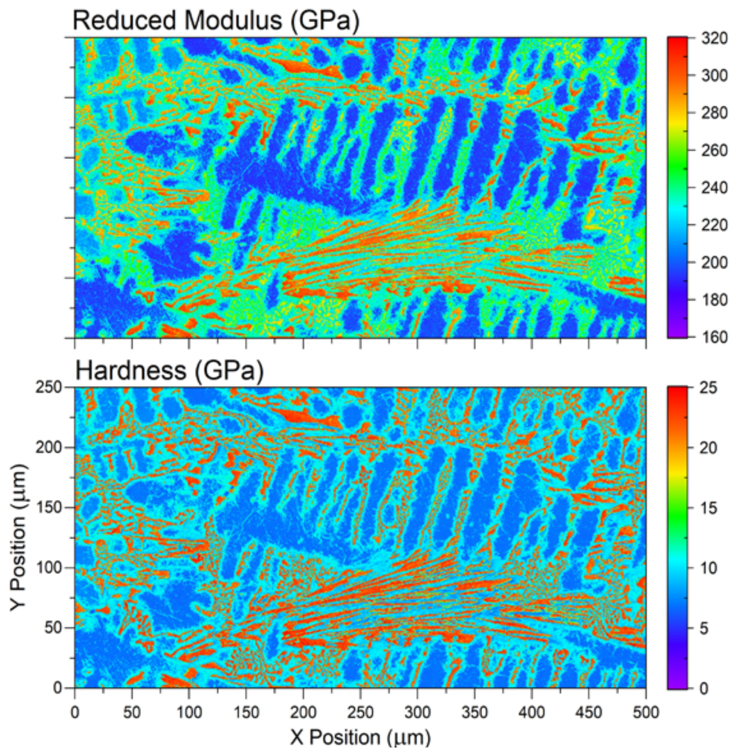
NANOINDENTATION MAPPING

Mechanical microscopy is a recent evolution of the nanoindentation technique, transforming it from a local 1D probe into a rapid 2D imaging technique capable of resolving variations in mechanical properties across a microstructure. By performing a closely-spaced grid of indents in displacement control, it is possible to generate a detailed map of these material properties while maintaining a consistent interaction volume.

In this example, a 316L steel sample made by laser powder bed fusion (L-PBF) is viewed in cross-section. This mechanical microscopy map reveals the columnar fish-scale microstructure formed by adjacent melt pools in the build direction, as well as defects like pores and hardening at melt-pool boundaries.



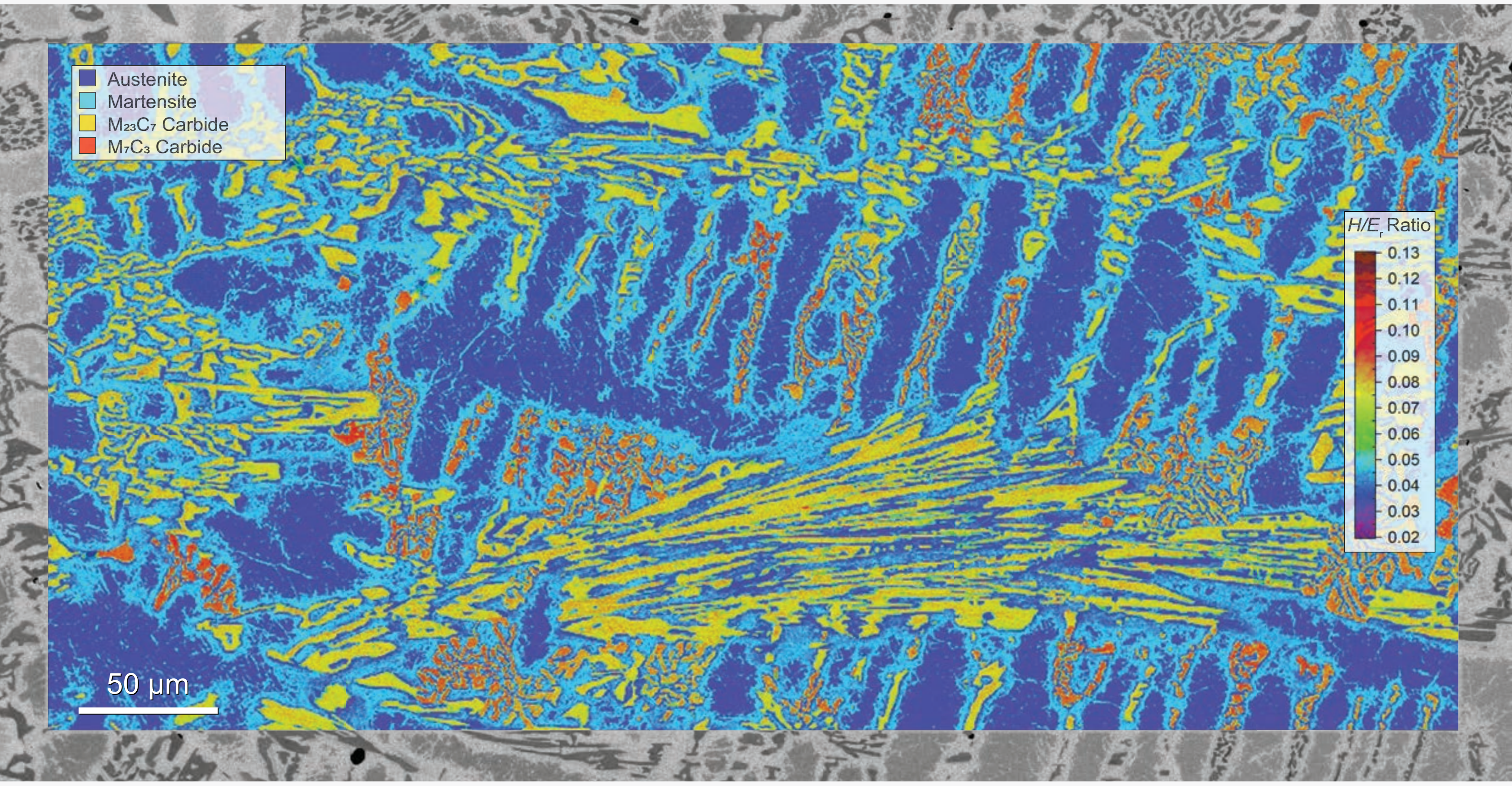
Sample Material: 316L Steel L-PBF
Curtesy of: Prof. M. Bambach, ETH Zurich
Indentation Depth: 200 nm
Inter-indent Spacing: 2 µm
Number of Indents: 320'000
Indentation Speed: 5 indents/s



Sample Material: High-Chromium Cast Iron
Curtesy of: Prof. M. Guitar, Saarland University
Indentation Depth: 50 nm
Inter-indent Spacing: 500 nm
Number of Indents: 500'000
Indentation Speed: 10 indents/s

Quantitative nanoindentation measurements are often slow, typically taking minutes or seconds per indent. The iX05, however, can perform quantitative measurements at speeds of up to 30 indents/s. For applications requiring higher sensitivity, the system can switch to high sensitivity, mapping at 10 indents/s. This mode enables the resolution of finer differences and features in nanoindentation maps.

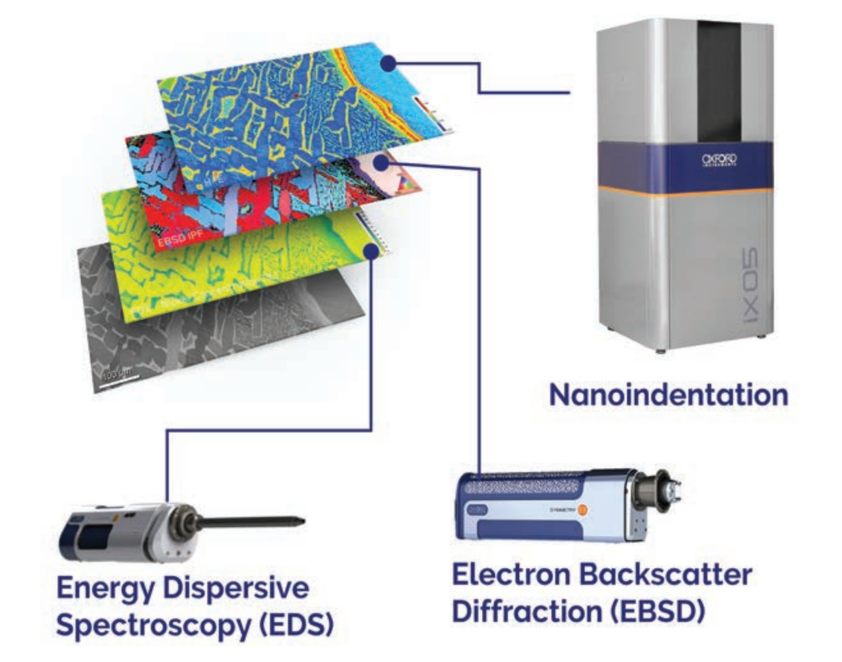
In this example, a sample of high-chromium cast iron (white cast iron) was mapped at different indentation speeds. Using the Ultra-Fast Mapping module, a map of 60'000 indents was completed in only 33 minutes. The eutectic carbides were clearly distinguishable from the austenitic matrix. Switching to high-sensitivity mode enabled further differentiation between the $M_{23}C_7$ and the M_7C_3 carbides, as well as the identification of a thin peripheral martensitic layer surrounding the carbides. These features require a sensitivity of less than 1% in H/E_r ratio to be identified!

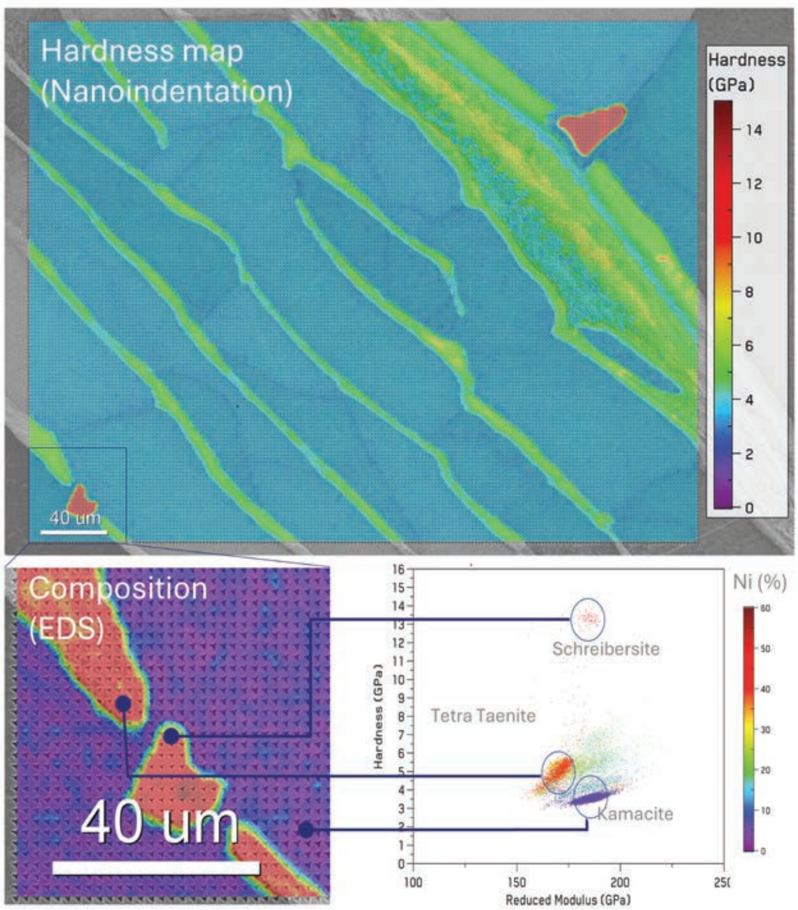


APPLICATION EXAMPLES

CORRELATIVE MECHANICAL MICROSCOPY

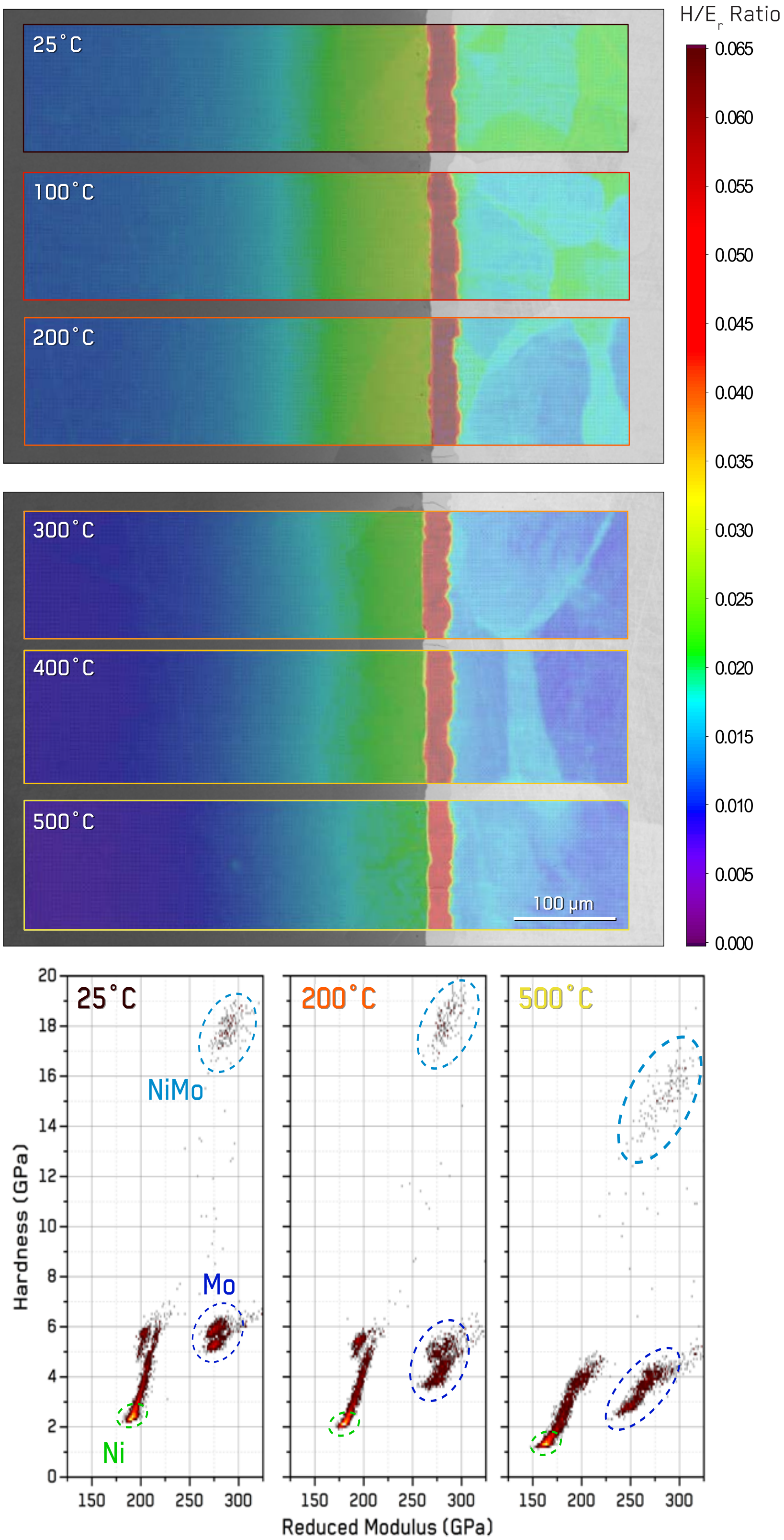
A core theme of materials science is the relationship between microstructure and mechanical properties.
While local composition and crystal structure can be obtained with techniques such as EDS (Energy-Dispersive X-ray Spectroscopy) and EBSD (Electron Backscatter Diffraction), the mechanical properties are often measured only at a macroscopic scale or with insufficient data points to be fully representative.
With the iX05, it is possible to perform a large number of nano-indents quickly while retaining a high data precision. By probing the local mechanical properties of the material in the same region of interest as the one analysed by EBSD or EDS, it is possible to easily link microstructure, chemical composition and mechanical properties of the material.
In this example, the microstructures of a fragment from the Seymchan meteorite were analyzed using correlative mechanical microscopy. The microstructures present in this meteorite are a result of extremely slow cooling over millions of years in the vacuum of space, making them exceptionally rare and impossible to be replicated on Earth.
By combining the compositional data obtained through EDS with the local mechanical data collected through nanoindentation, it is possible to distinguish and characterise all the nickel-iron crystal structures present in the specimen.
Correlative mechanical microscopy allows to distinguish the harder Schreibersite phase from the softer Tetrataenite phase, even though they have similar nickel content. This level of synergy between the chemical, structural and mechanical data is instrumental to further our understanding of materials behavior.





Sample Material: Meteorite (Fe-9Ni-0.5Co) | Inter-indent Spacing: 2 µm
Curtesy of: Prof. S. Korte-Kerzel, RWTH Aachen | Number of Indents: 30'000
Indentation Depth: 190 nm | Indentation Speed: 0.25 indents/s*

*Data recorded with the FT-I04 Femtoindenter. Similar data can be obtained with the iX05 nanoindenter 10x faster.



Sample Material: Ni-Mo Diffusion Couple
Curtesy of: Prof. Bin Gan, NWPU, Xi'an
Indentation Depth: 200 nm

Inter-indent Spacing: 4 µm
Number of Indents: 6 × 4'500
Indentation Speed: 0.4 indents/s*

*Data recorded with the FT-NMT04 In-Situ SEM Nanoindenter. Similar data can be obtained with the iX05 nanoindenter 10x faster.

Materials in engineering applications are often used at temperatures that deviate from standard laboratory conditions (room temperatue, ambient pressure). The iX05 allows testing the mechanical performance of materials in conditions that match those experienced in service.

Using high-speed, displacement-controlled nanoindentation, the iX05 can visualize, quantify, and statistically evaluate microstructures under different temperature conditions. This method ensures a uniform indentation size, regardless of local composition, hardness, or thermal softening, enabling consistent microstructural analysis. Performing measurements at high speed minimizes thermal drift and enhances statistical sampling.

A combinatorial study of the Ni-Mo system exemplifies this approach. A diffusion couple was prepared using hot isostatic pressing and annealing. Nanoindentation mapping has been performed at high temperatures using a diamond Berkovich indenter at testing temperatures ranging from ambient to 500°C in 100°C increments.

The key technical advancements offered by the iX05 ensure high thermal stability. The low thermal mass of the MEMS heater allows rapid alignment of the indenter and sample surface temperatures via an automated procedure. This alignment compensates for thermal expansion and maintains low thermal drift, guaranteeing reliable high-temperature measurements.

Mechanical property maps of the Ni-Mo diffusion couple closely correlated with backscattered electron micrographs, confirming consistent indentation depth and position across temperatures. Cluster analysis using a Mixed Gaussian approach revealed hardness and reduced modulus values consistent with literature for primary phases.

ACCESSORIES

HIGH TEMPERATURE MODULE

The High Temperature Module enables heating of specimens up to 800°C. In combination with the FemtoTools Microforce sensing Probes with active tip-heating, it makes isothermal nanomechanical testing of samples at various temperatures easy to perform. This module not only allows for the measurement of material properties as a function of temperature but also facilitates the quantitative study of thermally-induced changes in plastic deformation and fracture at the nanoscale. Furthermore, by performing tests in high vacuum, the oxygen content can be reduced by a factor of one million compared to systems that use inert gas preventing unwanted oxidation.

CRYOGENIC TESTING MODULE

The Cryogenic Temperature Module uses liquid nitrogen as a cooling agent to achieve temperatures as low as -150°C, allowing experiments to be conducted at extremely low temperatures. By operating under high-vacuum conditions, the iX05 effectively prevents condensation-related issues, ensuring smooth and reliable experimentation at cryogenic temperatures.

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.

ULTRA-FAST MAPPING MODULE

The ultra-fast mapping module boosts the speed of indentation from 3 indents per second to up to 30 indents per second, enabling the generation of high-resolution maps with over 100'000 indents in under an hour.
The specialized sample stage is essential for rapidly accelerating, stopping, stabilizing, performing the nanoindentation measurement, and then accelerating to the next indent position in under 33 milliseconds, all while maintaining exceptional accuracy.

MICROFORCE SENSING PROBES

Model	Range	Noise Floor (10Hz)
S200	+/- 200 µN	0.1 nN
S2'000	+/- 2'000 µN	0.5 nN
S20'000	+/- 20'000 µN	5 nN
S200'000	+/- 200'000 µN	50 nN
S2'000'000	+/- 2'000'000 µN	500 nN
S20'000-2Axis	+/- 20'000 µN (normal)	10 nN
	+/- 20'000 µN (tangential)	10 nN
S200'000-HT (800°C)	+/- 200'000 µN	100 nN

The FemtoTools Microforce Sensing Probes are sensors capable of measuring forces from sub-nano Newtons to 2 Newtons along the sensor's probe axis. Both compression and tension forces can be measured. SI-traceable pre-calibrations for each probe in combination with 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 High Temperature Probes (HT) featuring active tip heating up to 800°C.
The 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.

TECHNICAL SPECIFICATIONS

MEASUREMENT HEAD

Force	
Range	up to 2 N
Noise floor (10 Hz)	down to 100 pN
Digital resolution	down to 0.01 pN
Sampling frequency	2 MHz

Position	
Coarse range	30 mm
Coarse noise floor (10 Hz)	500 pm
Fine range	20 µm
Fine noise floor (10 Hz)	5 pm
Digital resolution	0.01 pm
Sampling frequency	2 MHz
Feedback-loop frequency	500 kHz

SAMPLE STAGE

Range	130 x 130 mm
Noise floor (10 Hz)	500 pm

VACUUM CHAMBER

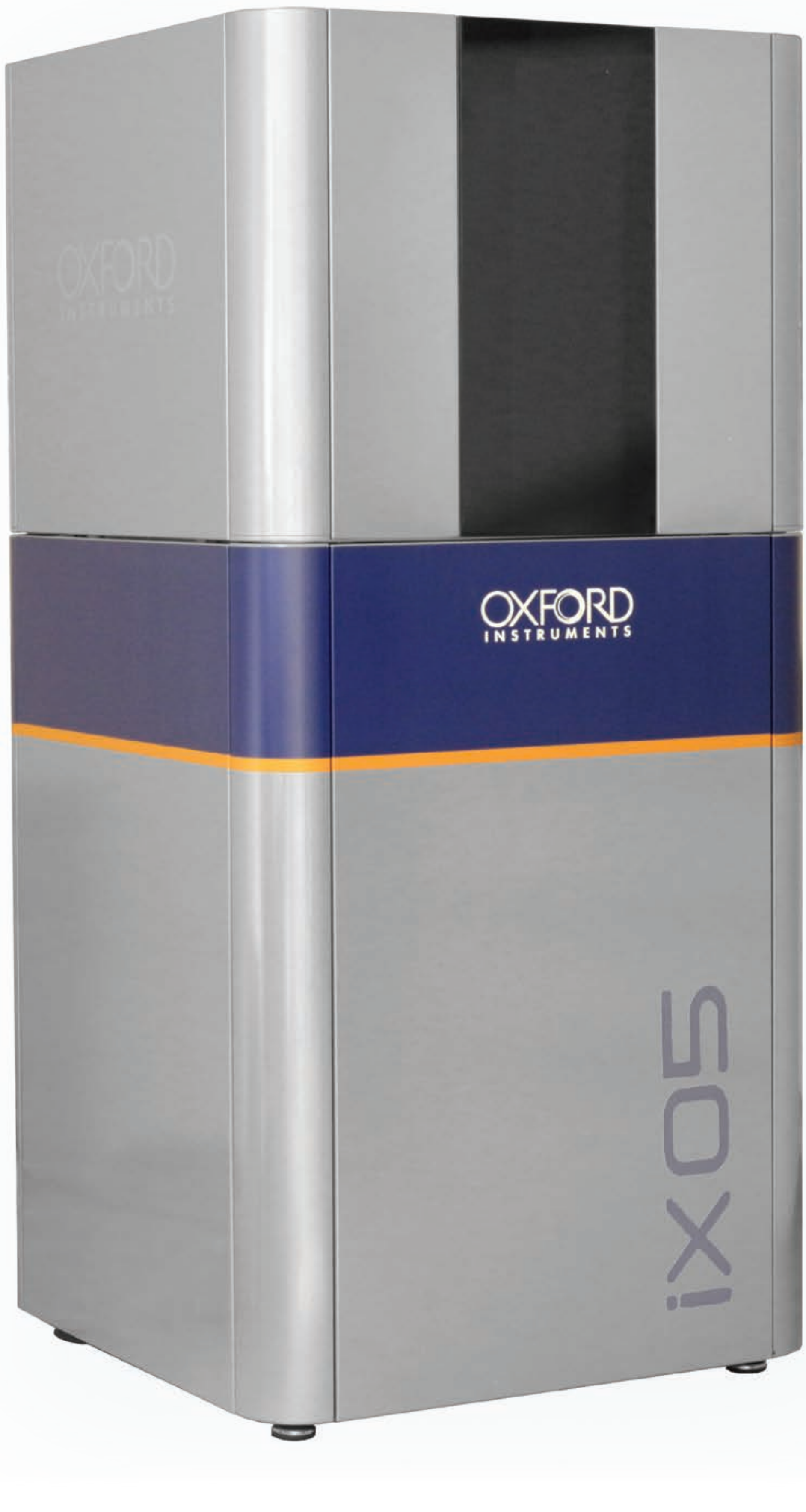
Pressure	less than $5 \cdot 10^{-4}$ mBar

VIBRATION ISOLATION

Active vibration isolation (integrated)

MICROSCOPE

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