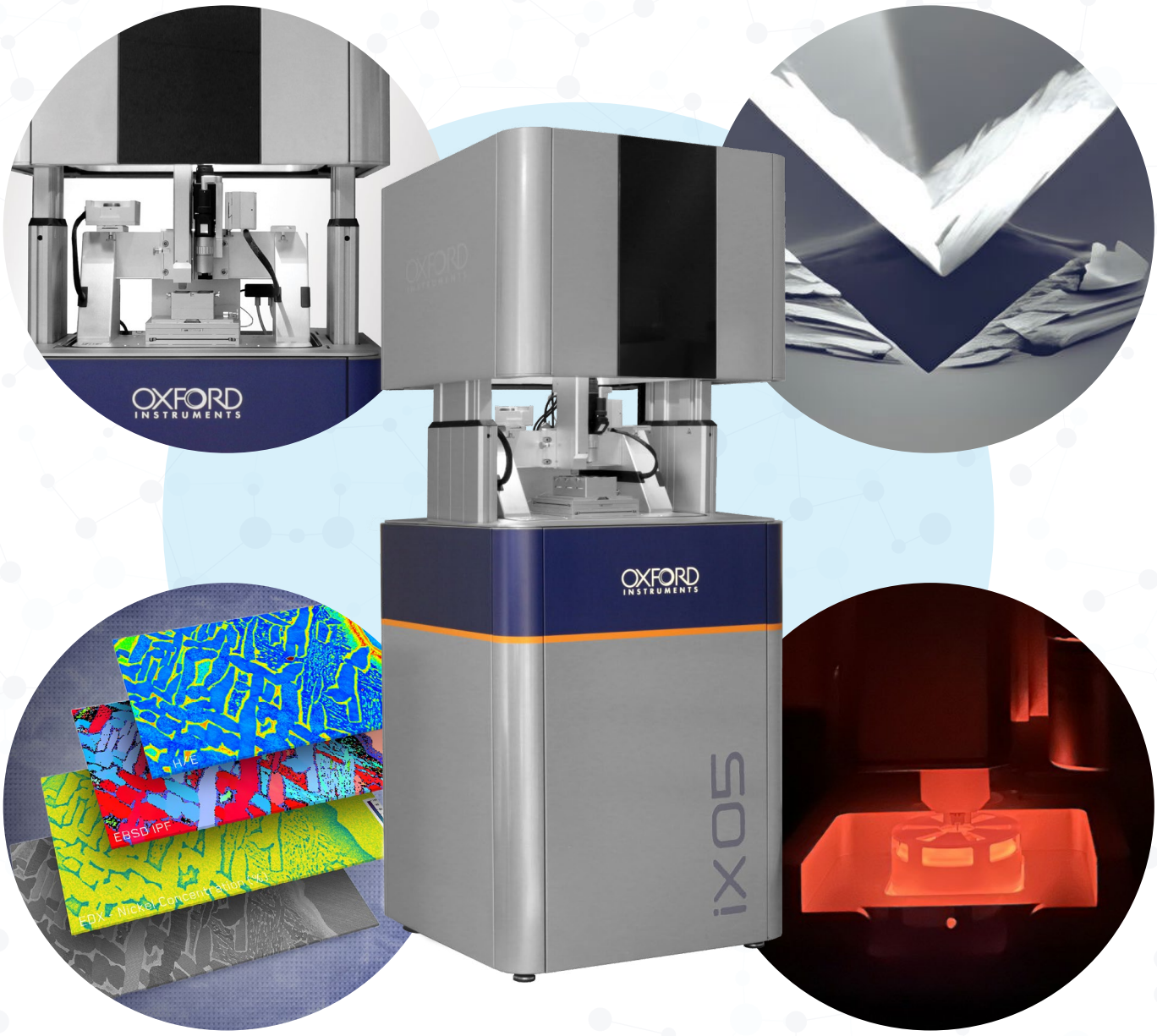
OXFORD INSTRUMENTS

iX05

Nanoindentation in operando conditions

Nanoindentation | Operando indentation | Mechanical microscopy



www.oxinst.com

iX05

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 high-vacuum environments.

Traditional nanoindenters are optimised to measure materials in laboratory conditions. However, materials in practical applications often experience extreme environments that strongly influence their mechanical behaviour. The iX05 is optimised 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 single measurements at selected locations to large-area mapping of mechanical properties. This is achieved by performing hundreds of thousands of indents in a densely spaced grid. This technique, known as mechanical microscopy, helps materials scientists establish direct links between fine microstructural features and component performance.

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 visualising mechanical properties of materials in a wide range of operando conditions.

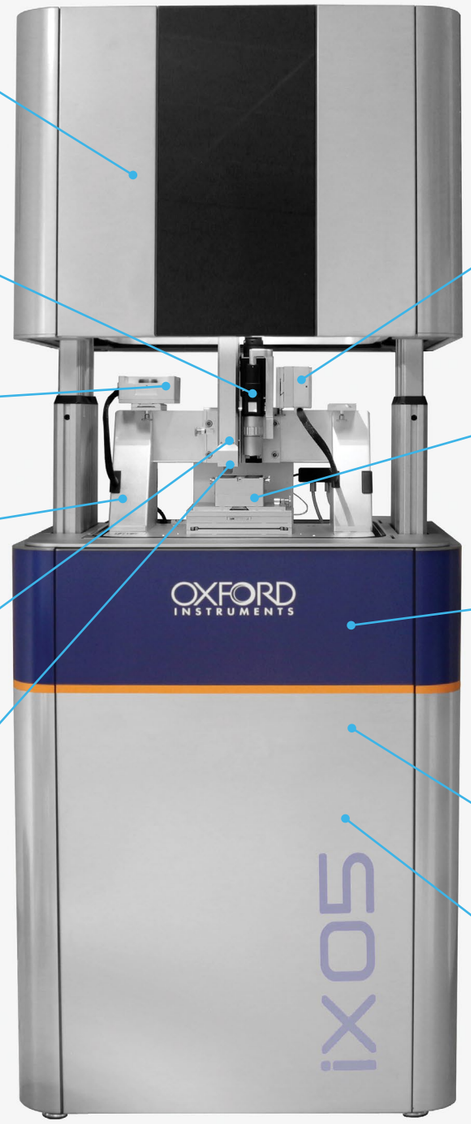
Features

- 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
- Motorised chamber with environment control: inert gas atmosphere or high vacuum to minimise 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 7orders 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 measurement and calibration procedures for high-throughput testing.



iX05

System buildup



1 VERTICALLY-MOTORISED HIGH-VACUUM CHAMBER

2 TOP-DOWN OPTICAL MICROSCOPE WITH COAXIAL ILLUMINATION

3 HIGH-TEMPERATURE MODULE (UP TO 800°C)

4 HIGH-STIFFNESS MEASUREMENT FRAME

5 FEMTOTOOLS HIGH-RESOLUTION PIEZO-SCANNER

6 FEMTOTOOLS MEMS-BASED FORCE SENSOR

7 CRYOGENIC TESTING MODULE (DOWN TO -150°C)

8 SAMPLE HOLDER

9 INTEGRATED ACTIVE ANTIVIBRATION SYSTEM

10 VACUUM PUMPING SYSTEM (TURBOMOLECULAR PUMP)

11 ULTRAFAST CONTROL ELECTRONICS (2 MHZ SAMPLING RATE, 500 KHZ FEEDBACK FREQUENCY)

Testing modes

Nanoindentation

Nanoindentation

- Standard nanoindentation (ISO 14577)
- CSM and DMA modes
- Load range to 2 N

Scratch Testing

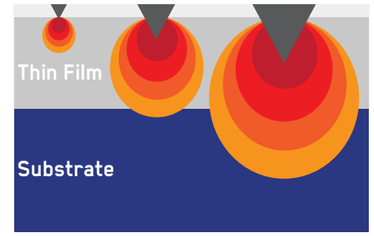
- Nanoscratch testing
- Scanning Probe Microscopy (SPM)
- Friction-force measurement

Thin Film Testing

- Ultra-thin film property measurement
- Bond-strength measurement







Operando nanoindentation

High-Temperature Testing

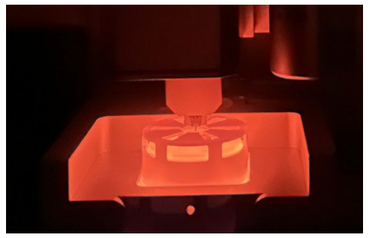
- Up to 800°C
- High vacuum
- Active tip heating

Cryogenic Testing

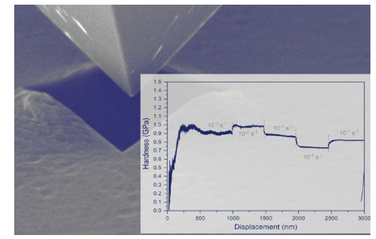
- Down to -150°C
- High vacuum
- Testing 3 samples in parallel

High-Strain Rate Testing

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







Mechanical microscopy

Mechanical Microscopy (Nanoindentation Mapping)

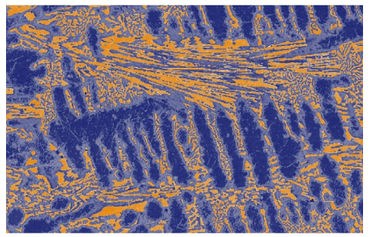
- High resolution (<200 nm)
- Ultra-high speed (>30 indents/s)
- High-sensitivity

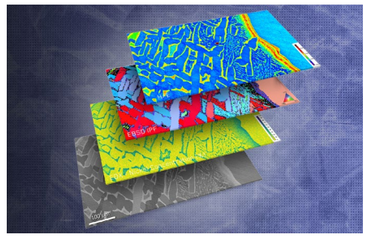
Correlative Mechanical Microscopy

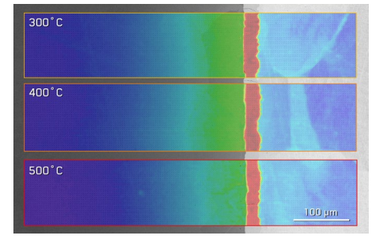
- Correlate with EDS
- Correlate with EBSD
- Correlate with AFM

Temperature-Dependent Nanoindentation Mapping

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







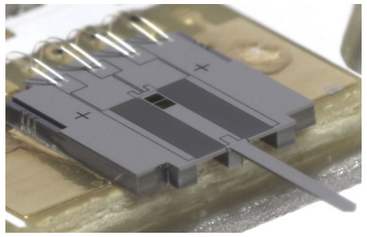
MEMS-based nanoindentation

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

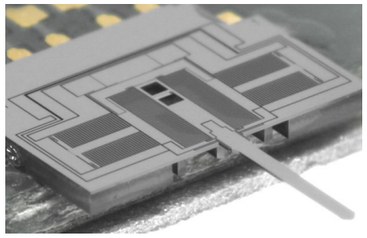
It uses MEMS technology and a newly developed FPGA-based controller (sampling rate of 2 MHz and feedback-loop frequency of 500 kHz) to achieve high resolution over a large dynamic range. This allows for the quick generation of mechanical microscopy maps. In less than an hour, it can generate large maps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

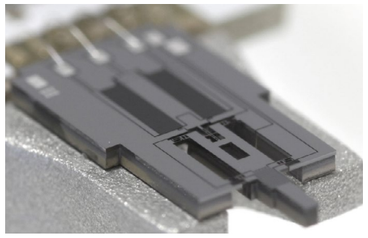
With advanced software control, the iX05 provides a versatile platform for analysing the mechanical properties of materials at the micro- and nanoscale.



1-Axis Microforce Sensing Probe

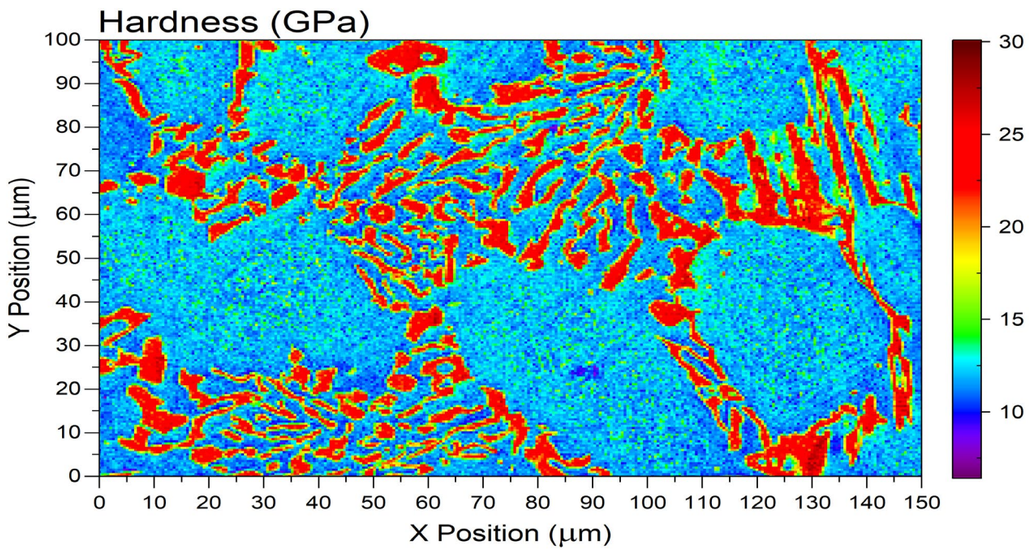


2-Axis Microforce Sensing Probe



1-Axis Microforce Sensing Probe with integrated tip heater

Ultra-fast nanoindentation mapping



Sample Material: High-chromium Cast Iron, courtesy 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.

Meet our product range

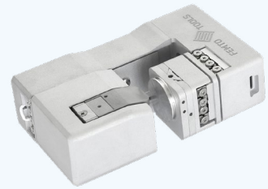
iX05

Operando nanoindenter for ultrafast nanoindentation and measurement of local mechanical properties in real-world conditions: from cryogenic to high temperatures, high strain rates, high vacuum environment.



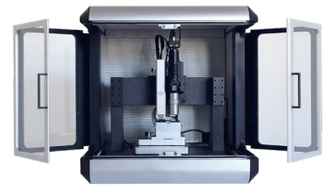
NMT04

In-situ FIB/SEM nanoindenter for compression, tensile and fracture testing of micro- and nanoscale samples with simultaneous high-resolution imaging of the tested sample using SEM.



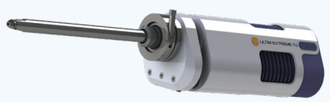
i04

Standalone nanoindenter for high-resolution high-speed measurement of local mechanical properties of materials in air, liquid or inert gas environment.



EDS Detectors

Ultim® Max ∞ (“Infinity”) SDD detectors unlock the infinite potential of EDS for characterising materials in the SEM. They combine unparalleled accuracy, sensitivity and speed to power AZtecLive software to solve the most complex or difficult analysis challenges.



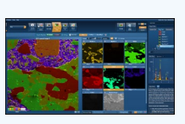
CMOS EBSD Detectors

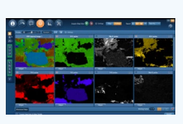
Our CMOS range of detectors, including the market-leading Symmetry S3, the C-Nano+ and the C-Swift+, all deliver exceptional performance for every type of EBSD analysis.



Software Solutions

AZtec software solutions for electron microscopy. AZtecLive Platform is designed for users who require more advanced functionality and flexibility. AZtecOne platform has a simplified interface, designed for users with little or no EDS knowledge and for those who require a system to deliver quick results.

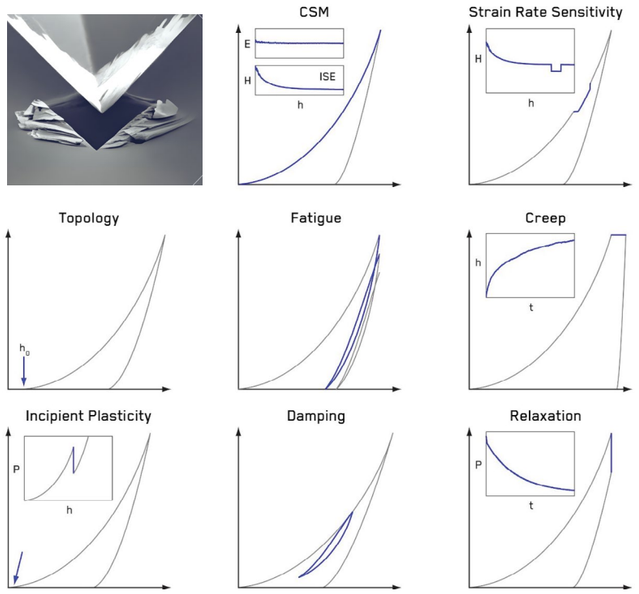




Applications

Nanoindentation

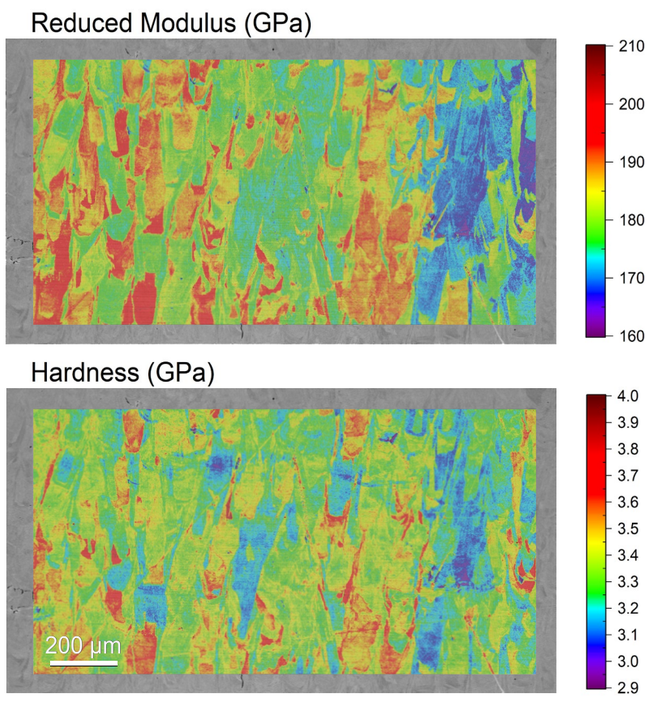
The iX05 enables the measurement of hardness, modulus, creep, fatigue, and fracture toughness of a wide range of materials from bulk metals and ceramics to soft polymers, thin films, and hard coatings. This is done by instrumented indentation. A sharp tip of known or calibrated shape is pressed into 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.

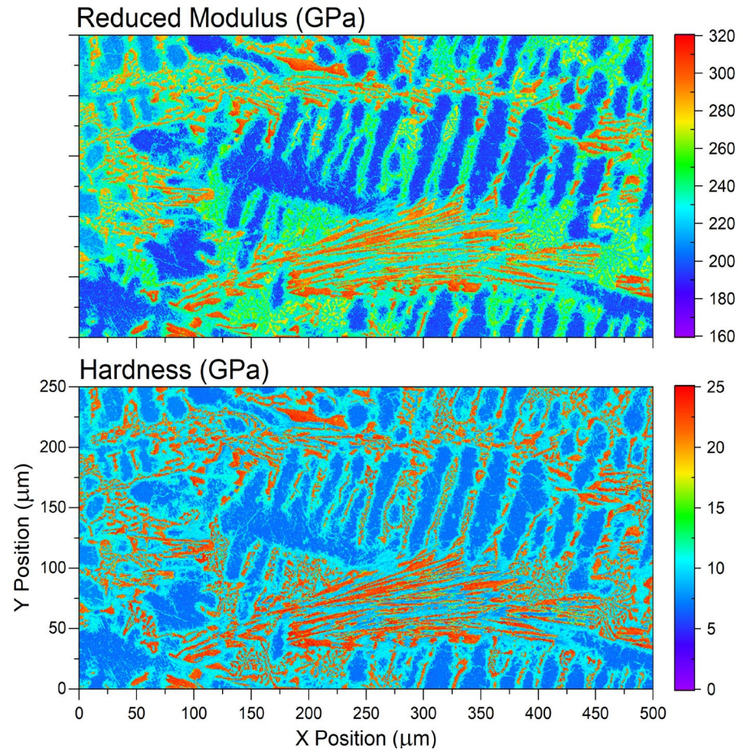


Sample Material: 316L Steel L-PBF courtesy of: Prof. M. Bambach, ETH Zurich. Indentation Depth: 200 nm. Inter-indent Spacing: 2 µm. Number of Indents: 320,000. Indentation Speed: 5 indents/s

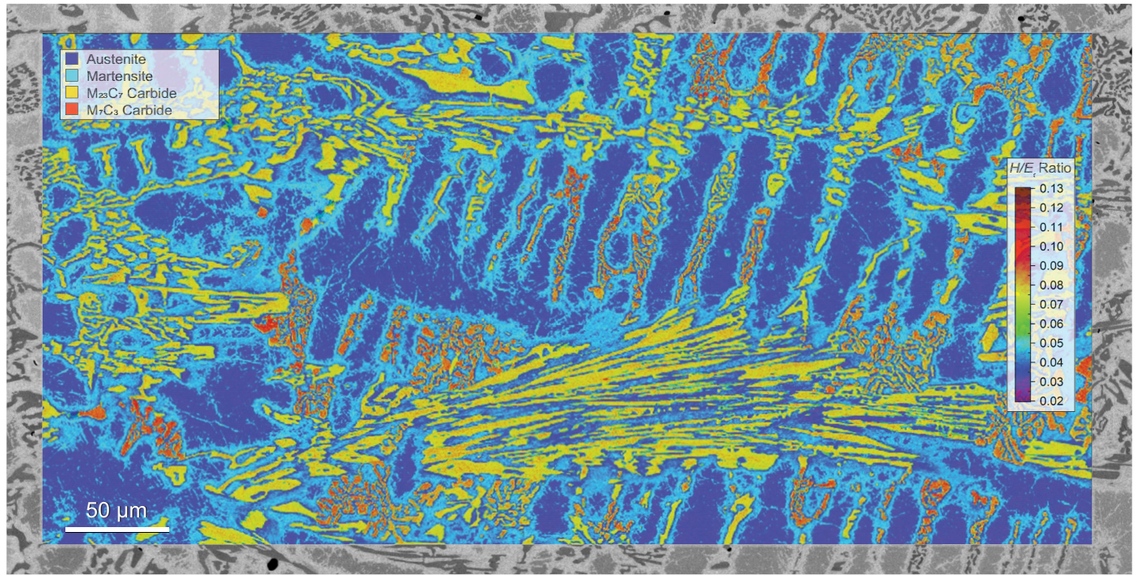
High sensitivity nanoindentation mapping

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!



Sample Material: High-Chromium Cast Iron, courtesy 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.



Applications

Correlative mechanical microscopy

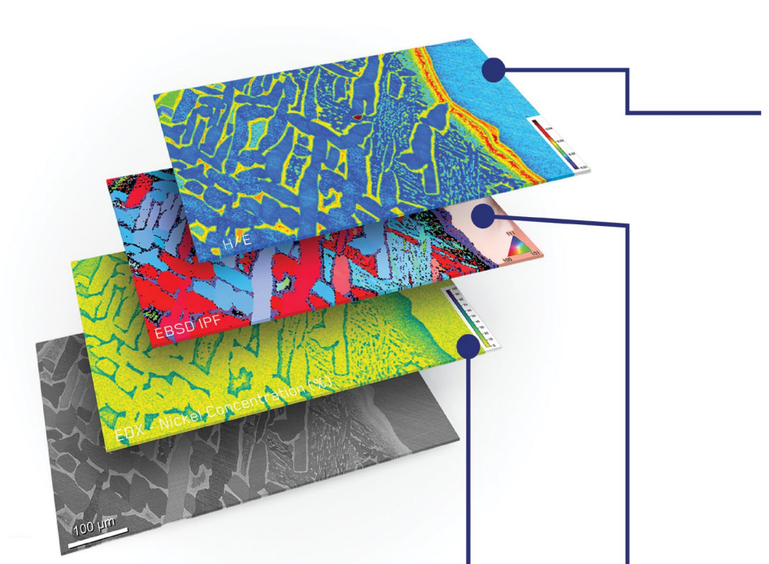
A core theme of materials science is the relationship between microstructure and mechanical properties. While local composition and crystal structure can be obtained with techniques such as EDS (Energy-Dispersive X-ray Spectroscopy) and EBSD (Electron Backscatter Diffraction), the mechanical properties 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 analysed 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 behaviour.





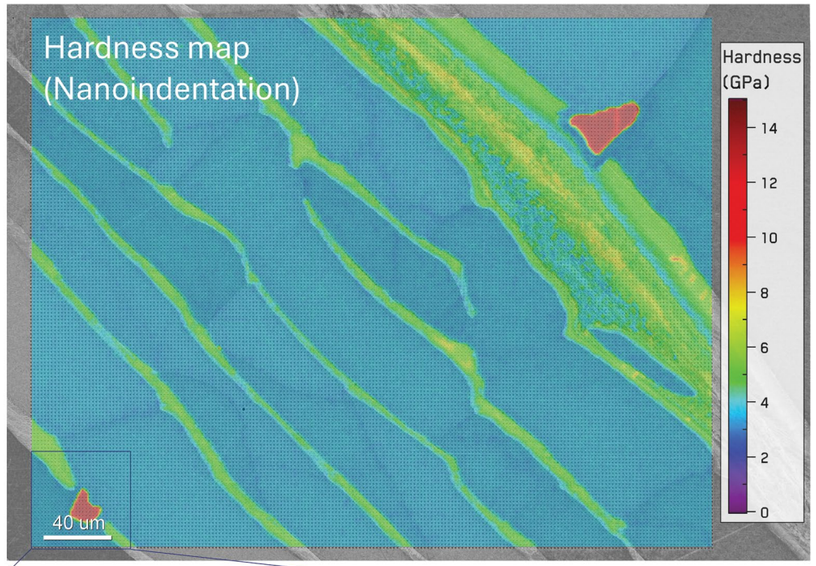
Nanoindentation



EDS



EBSD



Sample Material: Meteorite (Fe-9Ni-0.5Co) courtesy of: Prof. S. Korte-Kerzel, RWTH Aachen
Indentation Depth: 190 nm
Inter-indent Spacing: 2 µm
Number of Indents: 30,000
Indentation Speed: 0.25 indents/s*
*Data recorded with the i04 Femto-Indenter. Similar data can be obtained with the iX05 nanoindenter 10x faster

Applications

High temperature nanoindentation mapping

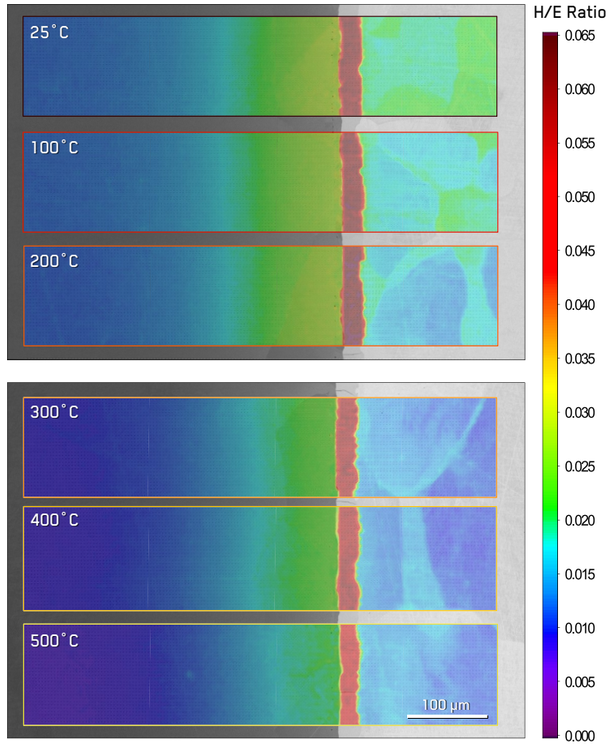
Materials in engineering applications are often used at temperatures that deviate from standard laboratory conditions (room temperatue, ambient pressure). The iX05 allows testing the mechanical performance of materials in conditions that match those experienced in service.

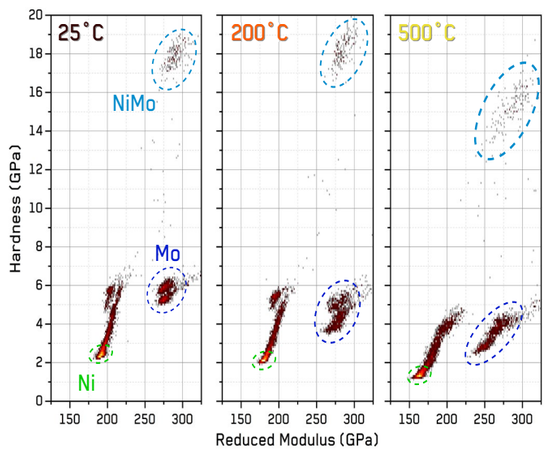
Using high-speed, displacement-controlled nanoindentation, the iX05 can visualise, quantify, and statistically evaluate microstructures under different temperature conditions. This method ensures a uniform indentation size, regardless of local composition, hardness, or thermal softening, enabling consistent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





Sample Material: Ni-Mo Diffusion Couple, courtesy 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 NMT04 In-Situ SEM Nanoindenter. Similar data can be obtained with the iX05 nanoindenter 10x faster.

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)

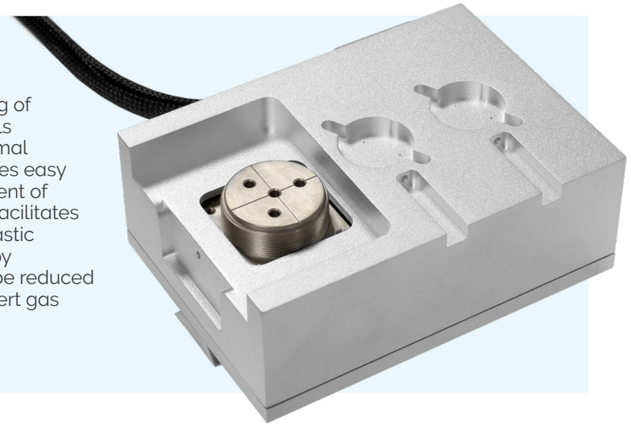
Microscope	
Camera	5 megapixel CMOS sensor
Objective lens options	5x, 10x, 20x
Focus	Motorised
Coaxial illumination	LED, Adjustable



Accessories

High Temperature Module

The FT-X-HT05 High-Temperature Module enables heating of specimens up to 800°C. In combination with the FemtoTools Microforce 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.



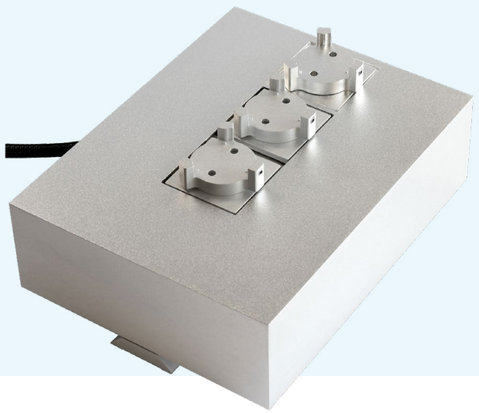


Low Temperature Module

The FT-X-LT05 Low-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 FT-X-ST05 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.

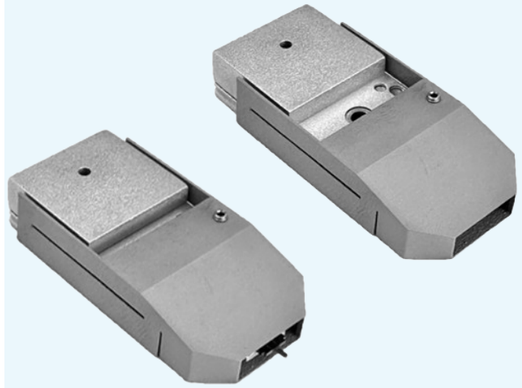


Ultra-Fast Mapping Module

The FT-X-UFM05 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 highresolution maps with over 100,000 indents in under an hour.

The specialised sample stage is essential for rapidly accelerating, stopping, stabilising, performing the nanoindentation measurement, and then accelerating to the next indent position in under 33 milliseconds, all while maintaining exceptional accuracy.





Microforce Sensing Probes

The FemtoTools Microforce Sensing Probes are MEMS 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. Specialised 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.

Model	Range	Noise Floor (10Hz)
FT-X200	+/- 200 µN	0.1 nN
FT-X2'000	+/- 2,000 µN	0.5 nN
FT-X20'000	+/- 20,000 µN	5 nN
FT-X200'000	+/- 200,000 µN	50 nN
FT-X2'000'000	+/- 2,000,000 µN	500 nN
FT-X20'000-2Axis	+/- 20,000 µN (normal)	10 nN
	+/- 20,000 µN (tangential)	10 nN
FT-X200'000-HT	+/- 200,000 µN	100 nN

Order Today

Need more information? We are committed to finding the correct solution for you. With a dedicated team of technical advisors, we are able to offer you one-to-one guidance and technical support on all products.

To request more information please see:

nanoindentation.oxinst.com/contact-us

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