Linking Materials Performance to Microstructure

In Situ Lab for ZEISS FE-SEM

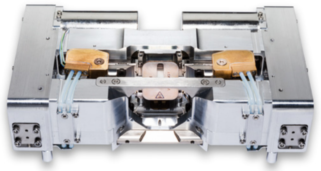
zeiss.com/fesem-insitu

Seeing beyond

In Situ Lab for ZEISS FE-SEM

Your Integrated Solution for Multi-modal *In Situ* Experiments

When you need to link materials performance to microstructure, ZEISS puts an automated *in situ* heating and tension experimental lab at your disposal. Observe materials under heat and tension automatically while plotting stress-strain curves on the fly. Extend your ZEISS FE-SEM* (field emission scanning electron microscope) with an *in situ* solution for heating and tensile experiments. What's more, all ZEISS FE-SEMs are plugged into the ZEN core ecosystem. This gives you access to connected microscopy, AI-based processing or analytics when using the ZEN core toolkits Connect, AI or Materials Apps.



Combine a mechanical tensile or compression stage, a heating unit, dedicated high-temperature SE (secondary electron), and BSE (backscatter electron) detectors with EDS (energy dispersive spectroscopy), EBSD (electron backscattered diffraction) analytics or both.

Thanks to the design of ZEISS Gemini electron optics, the integration of *in situ* hardware is very straightforward.

Control all system components from a single PC with a unified software environment that enables unattended automated materials testing in a ZEISS FE-SEM*. The automatic feature tracking provides you with a new standard for automatic series imaging and analytics such as EDS or EBSD mapping while multiple regions of interest are monitored. The collection of EDS or EBSD data is automated by the unified software. Investigate materials such as metals, alloys, polymers, plastics, composites, and ceramics with this fully integrated *in situ* lab.

Take advantage of:

- Automated *in situ* workflows for highly reproducible, precise, and reliable data collection
- High throughput data acquisition with high resolution. This rapidly creates statistically representative results
- High quality data for reliable post processing such as strain mapping using digital image correlation (DIC), powered by GOM
- Easy data post processing, and management.

Gain Deeper Insights into Material Properties with an Integrated *In Situ* Workflow

In situ materials testing in the SEM delivers precise measurement of the dynamic response of microstructures to mechanical load under defined temperature conditions. This enables materials researchers to understand the link between macroscopic mechanical properties and microstructure. Gain a deep understanding of the relationship between a materials' properties and its microstructure, which is essential for developing novel materials in a highly efficient way by adding information such as local chemical composition or crystallographic orientation using combined analytical techniques (e.g. EDS and EBSD).

Collect Meaningful Data with Automatic Feature Tracking and Autofocus

Start your *in situ* experiment by defining multiple regions of interest (ROIs), center them automatically, perform autofocus and use different imaging magnifications, scan rates or detectors. Subsequently, investigate each ROI during the automatic workflow at each deformation step. Different imaging conditions such as scan methods, dwell time and image resolution can be chosen individually for each ROI. Automatically trigger EDS and EBSD maps for selected ROIs as a function of applied tension at a set temperature.

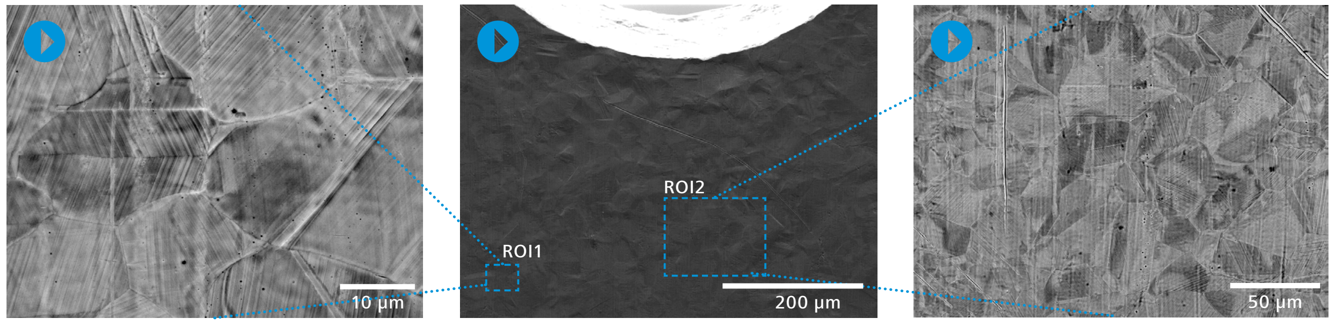


Figure 1: In situ *tensile experiment on a stainless-steel sample using different magnifications and various detectors. Left: ROI 1, 2000× magnification (referenced to Polaroid format), 1k (768 * 1024 pixels) image, BSE detector. Images have extremely high channeling contrast and capture the slip bands' formation during* in situ *loading. Center: Overview, 200×, SE, 4k (3072 * 4096 pixels) images. Right: ROI 2, 500×, BSE, 1k images).*

Next, perform long-term unattended tensile testing experiments. When investigating e.g. deformation of a specimen, different ROIs can be set, tracked and imaged automatically for several hundred deformation steps until the sample breaks. The software then detects the sample breaking and safely stops the experiment. Although the specimen's deformation might have reached several mm, the ROI is tracked successfully and always placed in the center of the SEM image. These experimental measurements are taken overnight without user interaction routinely.

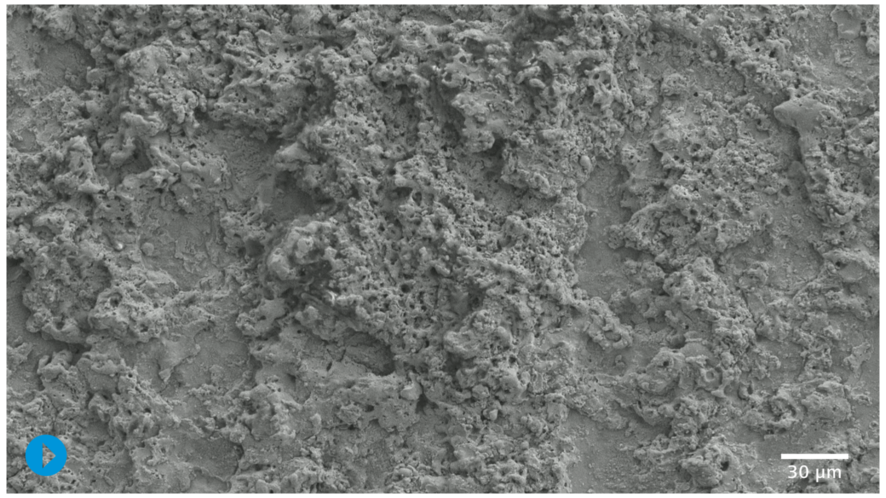


Figure 2: *Tensile* in situ *experiment on a 3D printed high temperature Ni-based superalloy. Three different ROIs are tracked and imaged with the SE detector automatically for more than 200 deformation steps until the sample breaks. Example images on one ROI are shown.*

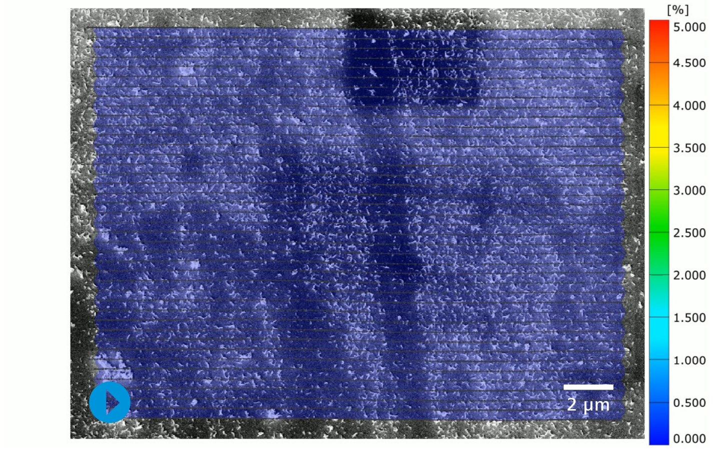


Figure 3: A mild steel sample (S235JRC) is polished and small particles on the specimen surface are used as markers for DIC. The automated feature tracking is used during deformation. The amplitude and direction of major strain can be displayed in the image using a color code for the strain amplitude and arrows for strain direction.

Use Digital Image Correlation (DIC) for Strain Mapping

When the experiment's objective is to obtain microscopic strain distribution, a high resolution SEM image series is your tool of choice: feature tracking takes place during deformation and is eventually used for digital image correlation (DIC). Export your resulting images to the 2D DIC GOM Correlate software** to analyze strain distribution and overlay with your SEM images. The high-resolution SEM images can be transferred to the correlated DIC software, which calculates the strain distributions.

***2D DIC software GOM Correlate*
https://www.gom.com/3d-software/gom-correlate.html

Perform Automatic Serial Imaging During Mechanical Testing and Combine it with EDS Analysis

Understand mechanical properties in metals and alloys and how inclusions influence fracture initiation and migration. Perform automatic serial SEM imaging and EDS mapping of the inclusions during mechanical testing to comprehensively investigate cracking behavior. Combining imaging and analysis enables you to characterize mechanical properties influenced by a combination of the following inclusion parameters: shape, size, quantity, interspacing, distribution, orientation and interfacial strength.

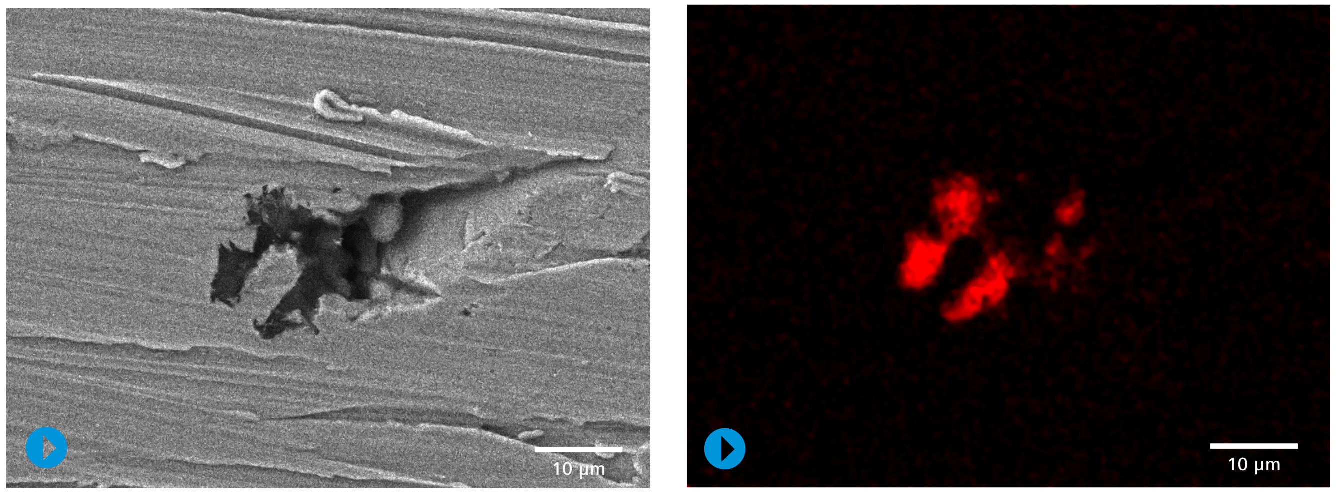


Figure 4: In situ *tensile experiment on a brass sample imaged at 20 kV using 500 pA beam current illustrates serial imaging and analysis (left: SE image, right: EDS map). The sample is imaged at each deformation step and corresponding EDS maps are automatically acquired. The maps show the elemental distribution of Si on the inclusion, where the crack has initiated.*

Complement Your Imaging with Analysis – EBSD Mapping of a Load Series at High Temperature

Mere imaging is not enough when you want to characterize your specimen comprehensively. Couple EBSD analysis to *in situ* mechanical tensile tests to observe e.g. misorientations or defects in grains that occur during mechanical loading at high temperature. Derive a stress-strain curve of an automatic *in situ* experiment and perform a series of EBSD maps when using the *in situ* heating stage.

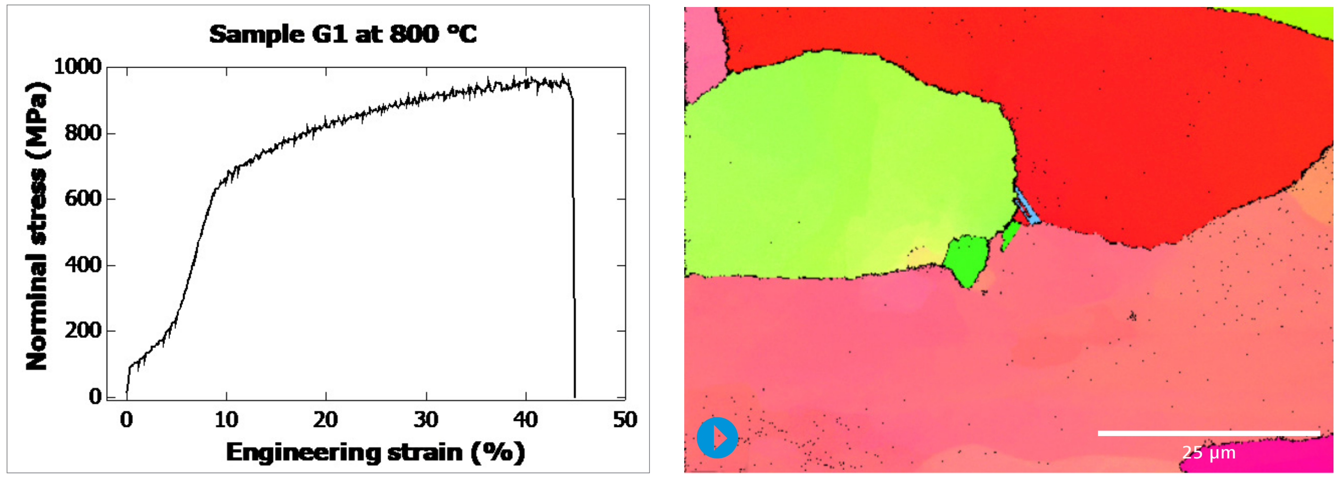


Figure 5: In situ *mechanical tensile tests and EBSD analysis using specimens of Haynes 282, a wrought, γ′ strengthened superalloy for high temperature structural applications resulting in grain misorientations and defects. A stress-strain curve is shown on the left. A series of EBSD maps on one of the ROIs is shown as an example using the* in situ *heating stage. The slip bands can be seen clearly. A cavity has formed close to the small green grain (EBSD) in the middle generating a concave area in the SEM image.*

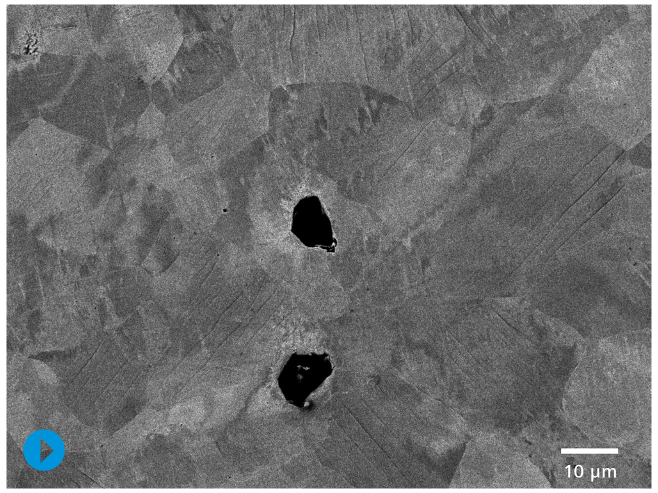


Figure 6: A steel sample at 600°C imaged under in situ *tensile load using the BSD detector that is based on a scintillator with cooling. Images show extremely high channeling contrast and capture the slip bands' formation during* in situ *loading as shown in the video.*

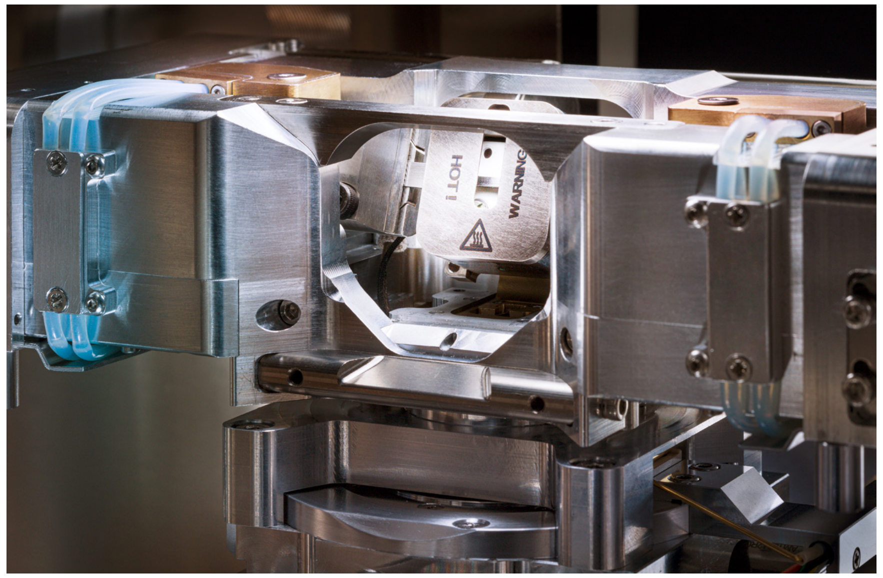
Achieve Excellent Channelling Contrast

For heating experiments, take advantage of the high temperature BSD detector which delivers excellent channeling contrast to gain even more information out of your specimen. When investigating e.g. a stainless-steel sample in an *in situ* tensile load test, the BSD images show channeling contrast that enables you to capture the slip bands' formation. Benefit from the tailored high temperature BSD detector equipped with cooling for long-time series imaging.

In Situ Lab for ZEISS FE-SEM

Your Integrated Solution for Multi-modal *In Situ* Experiments

__Figure 7:__ Tensile stage. The sample is tilted to 70° for EBSD analysis. The heater is mounted beneath the sample for high temperature testing.



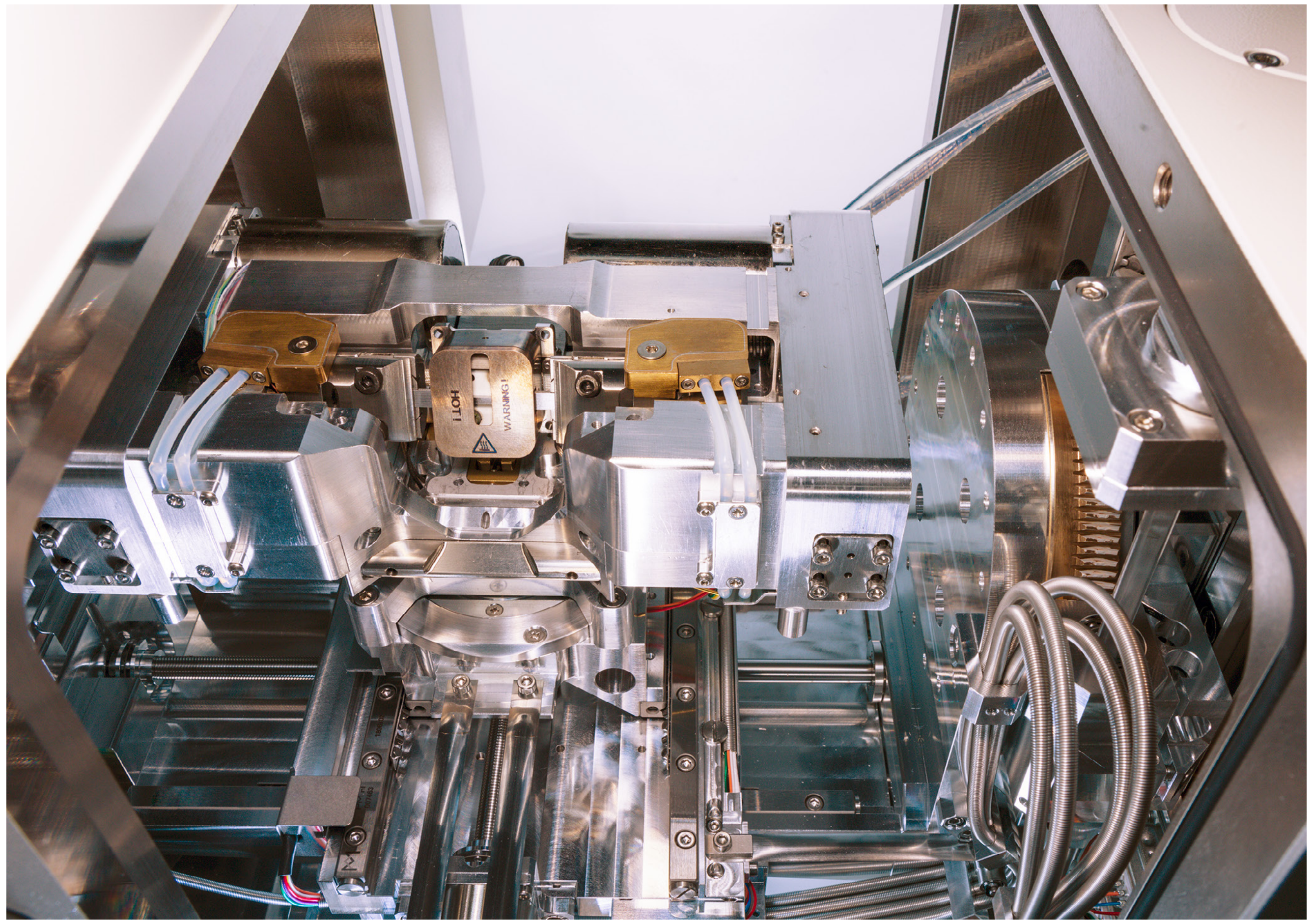


__Figure 8:__ The tensile stage is mounted on the SEM stage.

Technical Data

In Situ Lab for ZEISS FE-SEM

In Situ Lab for ZEISS FE-SEM offers:

Essential Specifications	Testing Stage and Software
Stage	Mechanical tensile/compression up to 5 kN, double-motor design
Loadcell	With built-in thermal compensation, exchangeable
	200 N (@Resolution 0.02 N); 1 kN (@Resolution 0.1 N); 5 kN (@Resolution 1 N)
Heater module (optional)	Removable, adjustable from room temperature to a maximum of 800°C (on the heater) with a resolution of 0.1°C
Tensile & compression speed	0.55 to 30 µm/sec
Jaws tilt	0° for imaging and EDS; 70° for EBSD mapping
Flat imaging working distance	Min. 10 mm
Software	Automated tensile and/or heating experiments
	Automatic feature tracking
	Configurable result graphs
	Sample exchange wizard
	Multiple regions of interest (ROIs) with arbitrary imaging or analytical parameters
	Digital Image Correlation using GOM software
Recommended software for digital image correlation	GOM Correlate
High-temperature BSD detector (optional)	High temperature BSD detector with cooling for long-term experiments (up to 800°C heater temperature)
***Compatibility**	*Available as option on the ZEISS FE-SEMs GeminiSEM 360, GeminiSEM 460 and Sigma 560 with eucentric stage.*
	Choose GeminiSEM 460 for your most demanding applications: based on the Gemini 2 design, it allows to expand your analytical capabilities with exceptionally high, tunable current density—even at low kV. Sigma 560 and GeminiSEM 360 combine imaging and analytics based on Gemini 1 optics.
	Contact your regional sales representative for more information.

Carl Zeiss Microscopy GmbH
07745 Jena, Germany
microscopy@zeiss.com
www.zeiss.com/fesem-insitu

EN_42_012_353 | CZ 05-2023 | Rel. 1.1 | Design, scope of delivery and technical progress subject to change without notice. | © Carl Zeiss Microscopy GmbH