BRUKER NANO ANALYTICS

In-situ SEM nanoindentation combined with 3D EBSD

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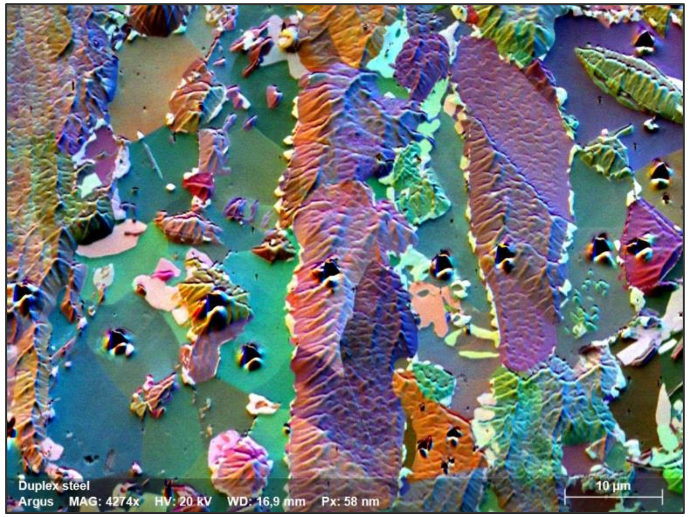
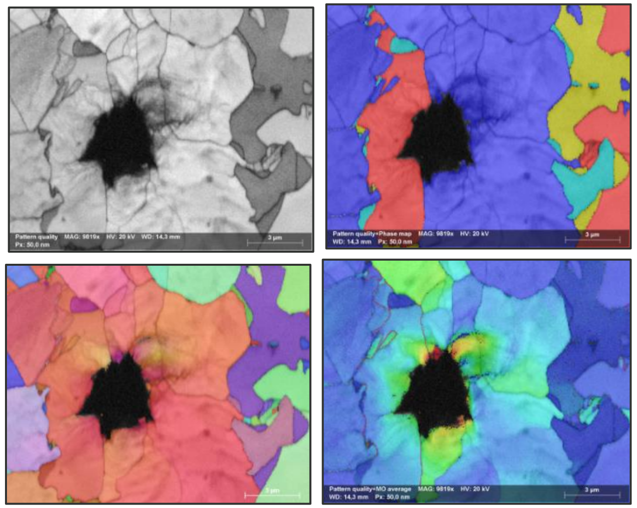
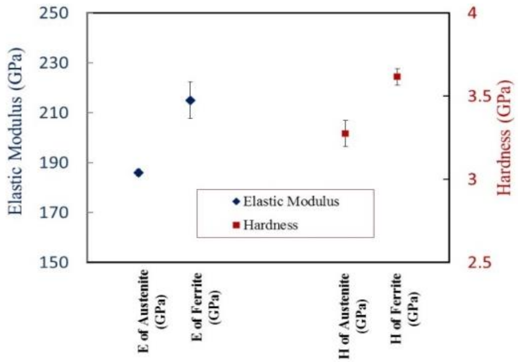
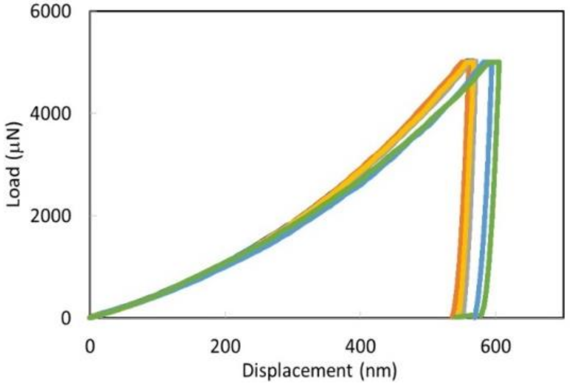
In-situ SEM nanoindentation combined with EBSD

- Correlating mechanical properties with microstructural features
- Study of plastic deformation mechanisms, e.g. EBSD measurement of the deformed volume below the indent
- Investigation of residual stresses, e.g. with HR-EBSD measurement
- Tensile testing on thin lamella









In-situ SEM nanoindentation combined with 3D EBSD

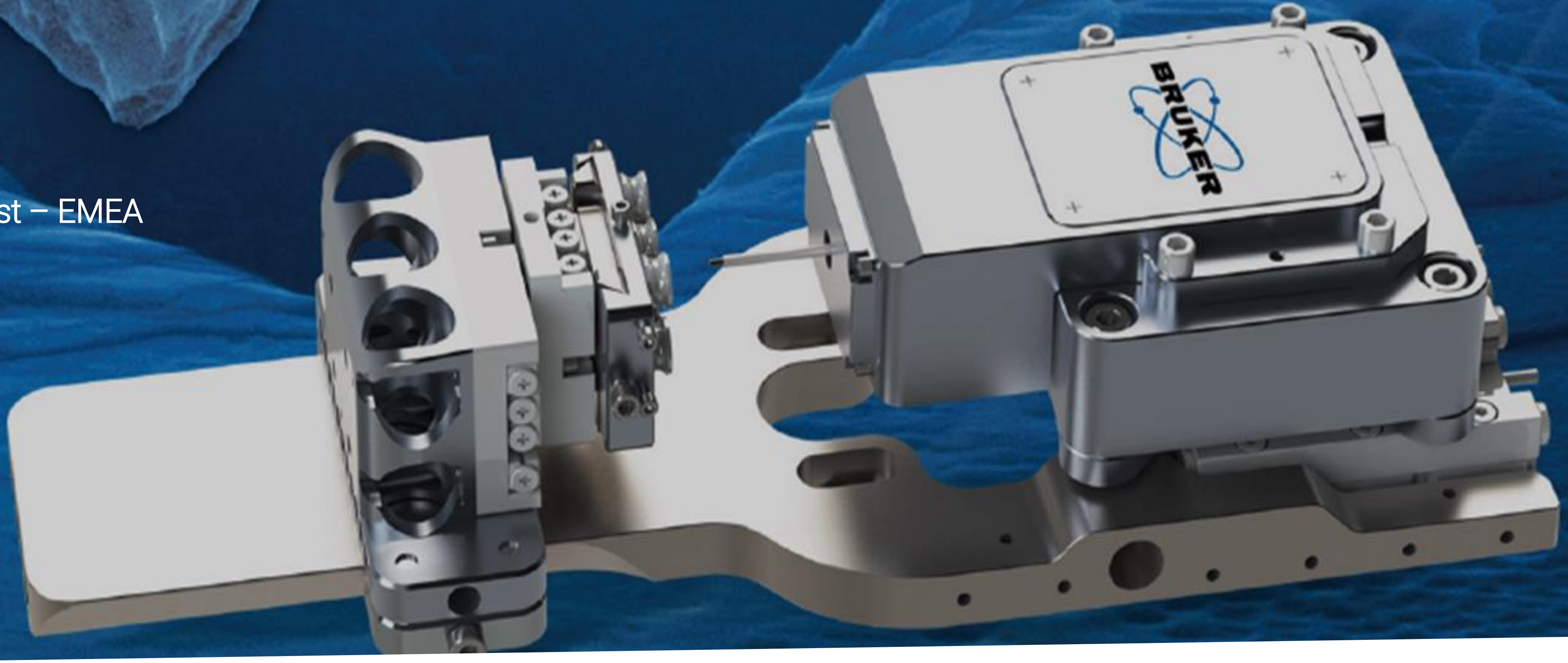
BRUKER NANO ANALYTICS WEBINAR

Nanomechanical testing setup PI89 SEM PicoIndenter

BRUKER BNSM DIVISION

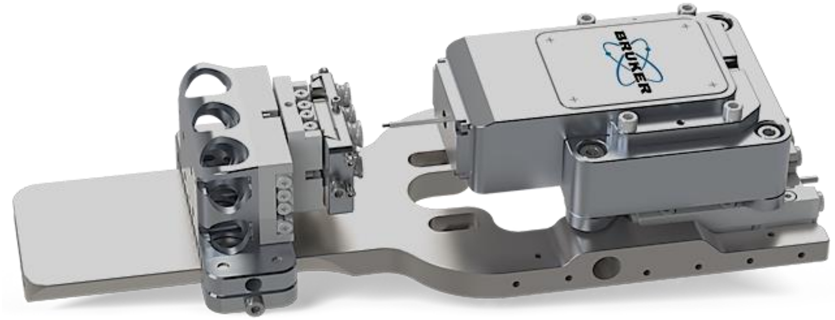
In-situ SEM Nanoindentation

Jaroslav Lukeš, Ph.D.
Nanoindentation Applications Scientist – EMEA



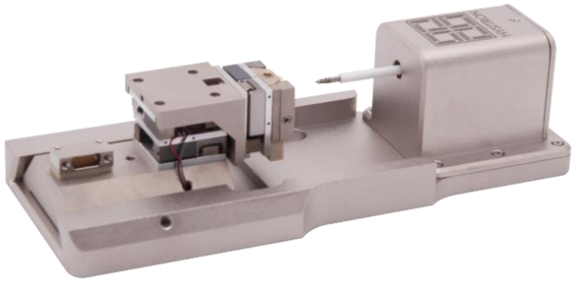
Nanomechanical Instruments for Microscopes

PI 89 SEM PicoIndenter®



PI 95 TEM PicoIndenter®

PI 85L SEM PicoIndenter®



BioSoft®

Intraspect360®

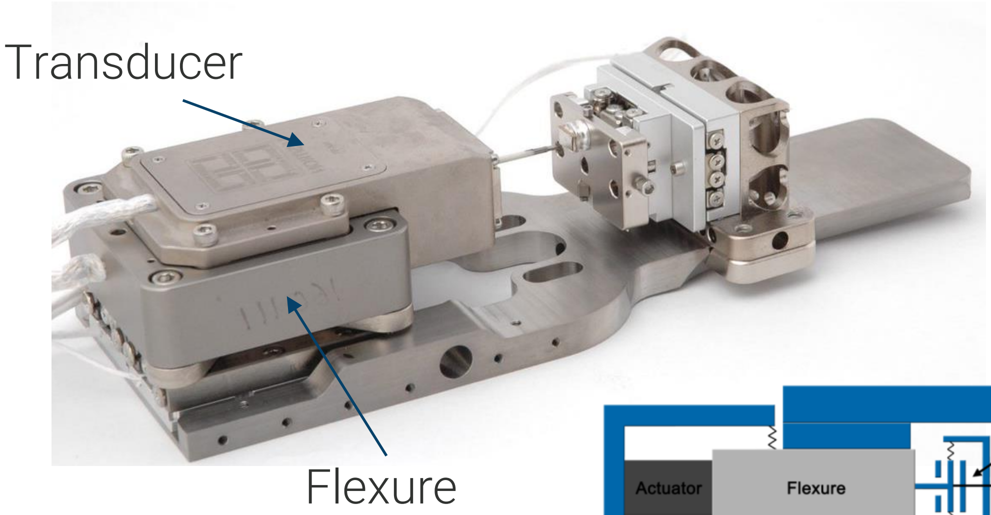
for X-Ray Microscopes (XRM) Beamline/Synchrotron Sources

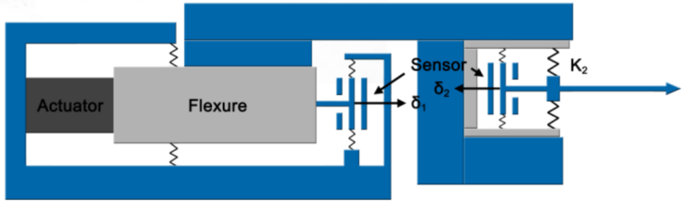
Hysitron PI 89 SEM PicoIndenter

B. xR Transducer with higher load – 3.5 N

- xR" Transducer- 10 mN, 500 mN and 3.5 N force and 150 mm displacement
- Max load 3.5 N and max displacement 150 um
- Displacement actuation by intrinsically displacement-controlled piezo actuator
- In load control mode, electrostatic actuation is used

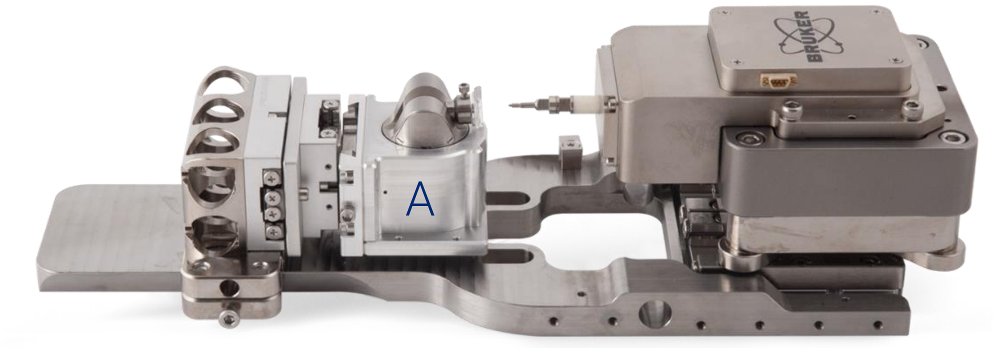
Replaceable "xR" Transducer:
10 mN, 500mN and 3500 mN, 150 µm (flexure)

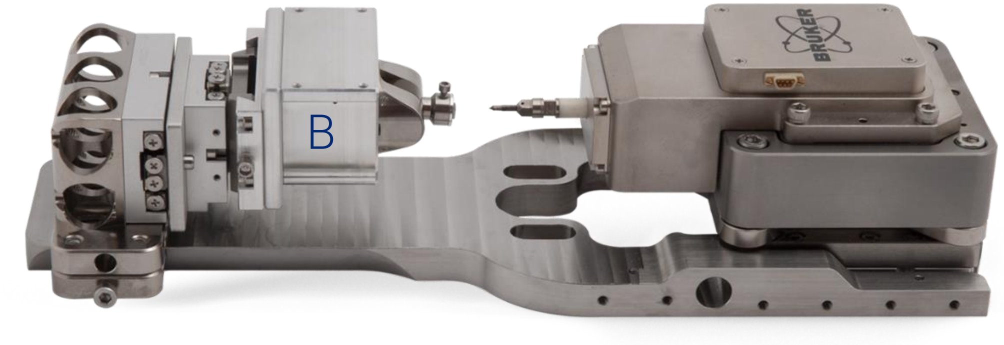




Variable Sample Stage

Dual configuration rotation and tilt stage

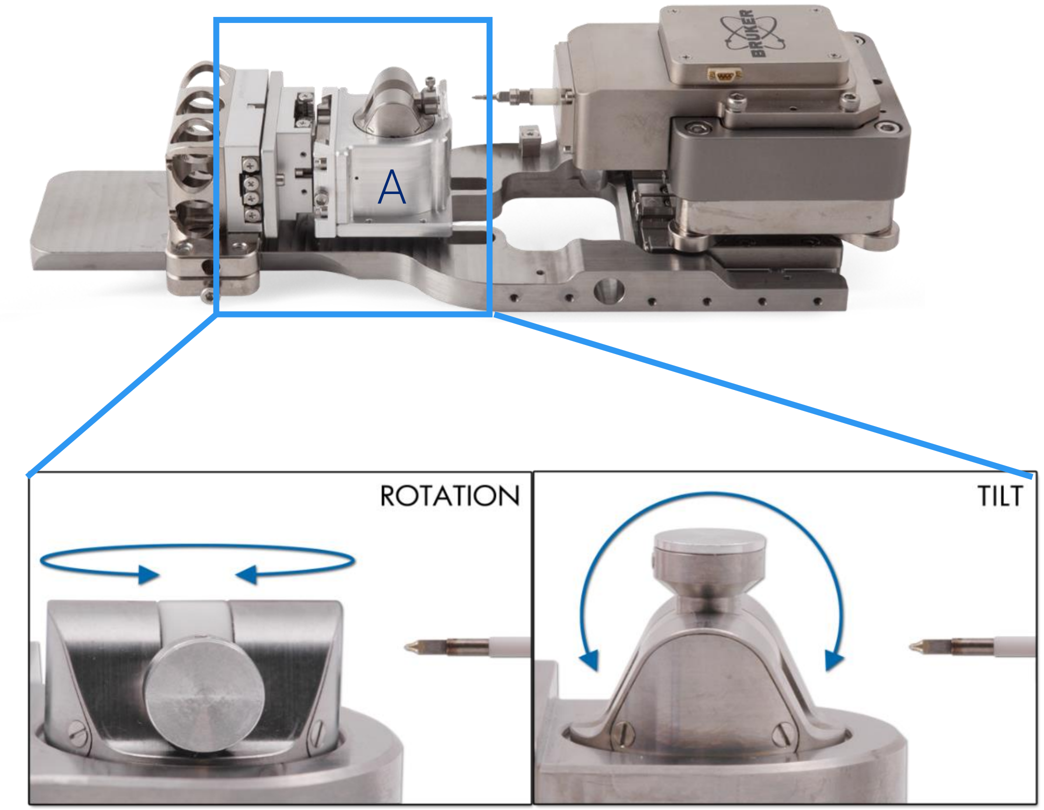


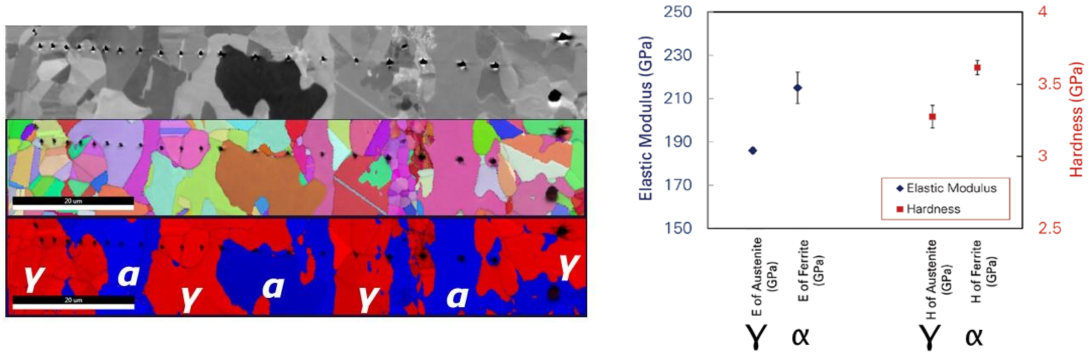


Patented in Japan and USA

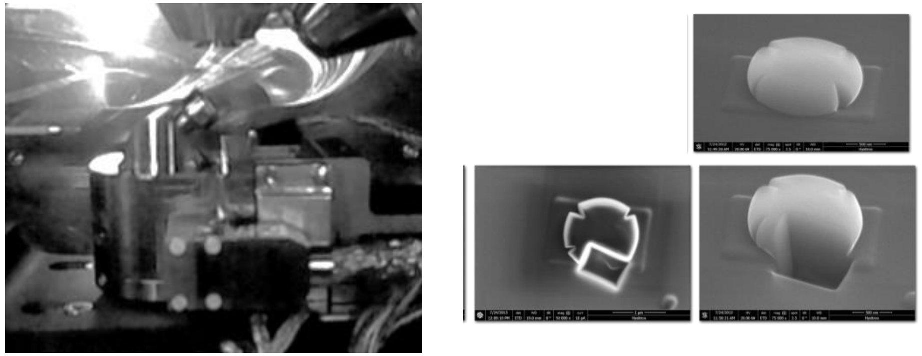
Rotation and Tilt Stage – Configuration A

- ***5 Degrees of Freedom* for Sample Positioning**





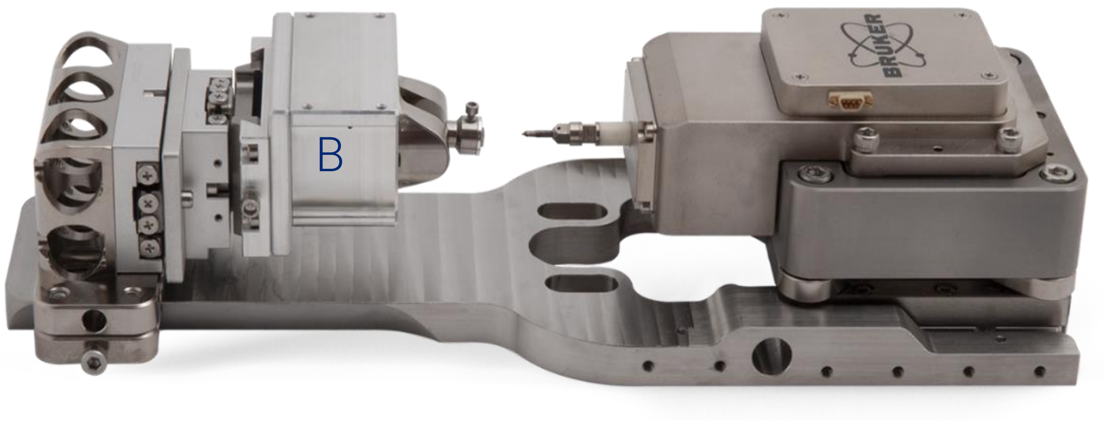
Indentation and EBSD mapping of phases of duplex steel (lower)



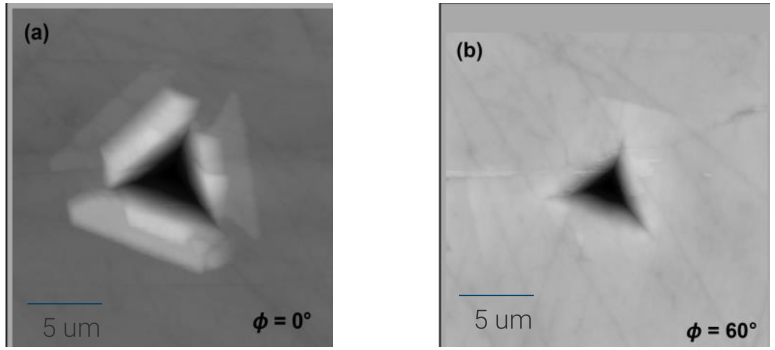
Particle compression and in-situ FIBing

Sample positioning towards **electron column for imaging**, FIB column for **milling**, and detectors such as EDS, BSE, EBSD, for **analytical data and imaging**.

Rotation and Tilt Stage – Configuration B



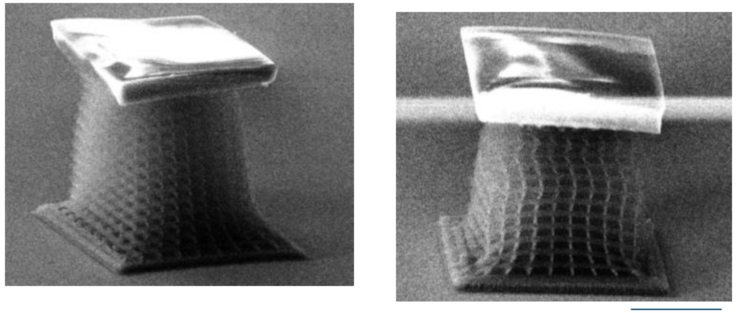
- ***5 Degrees of Freedom* for Sample Positioning**



Example: Anisotropic indentation behavior in calcite
(Courtesy; Prof. Shefford P. Baker, Cornell))

Tilt-Rotation of a sample – Video

Spindle-Rotation of a sample – Video

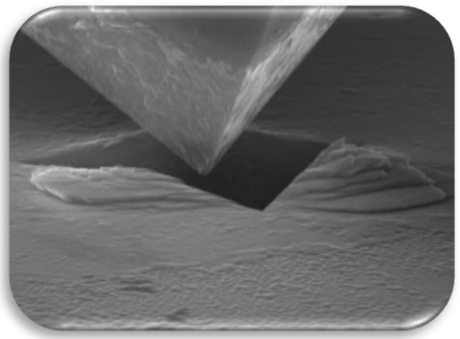


5 um

Spindle rotation of a pillar to observe other sides

Sample positioning towards **electron column for imaging**, detectors such as EDS, BSE, EBSD, for **analytical data and imaging, spindle-mode rotation of sample.**

In-Situ Testing Techniques and Applications



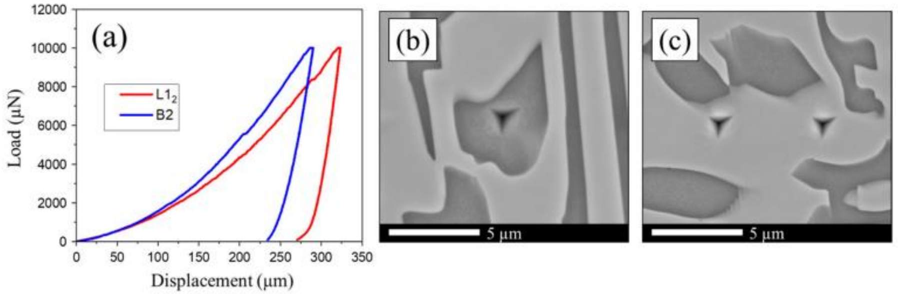
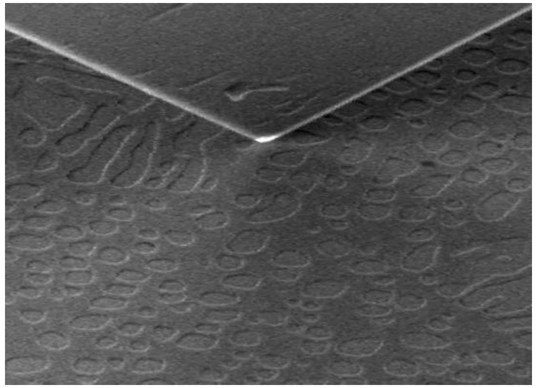


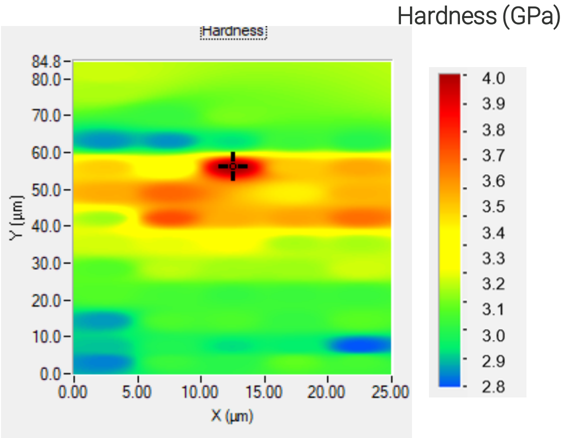
Fig. 6 **a** Load-displacement curves obtained from nano-indentation; SEM image of an indent: **b** on the B2 phase and **c** on the L12 phase

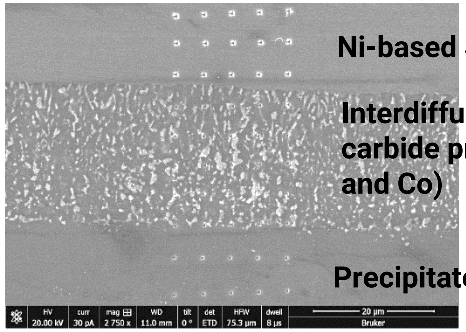
Eutectic high entropy alloy, Vahid Hasannaeimi, Aditya V. Ayyagari, Saideep Muskeri, Riyadh Salloom and Sundeep Mukherjee, 2019, npj materials degradation, **3** (2019) 1886

Integrated Accelerated Property Mapping (XPM) with PI 89



XPM– Video





XPM with PI 89 : Available at High Temperature

XPM with PI 89 : RT Stage and EBSD

Nanoindentation Accelerated Property Mapping (XPM)

Compression

Direct Tension PTP

Bending

DMA/Fatigue

Scanning Probe Microscopy

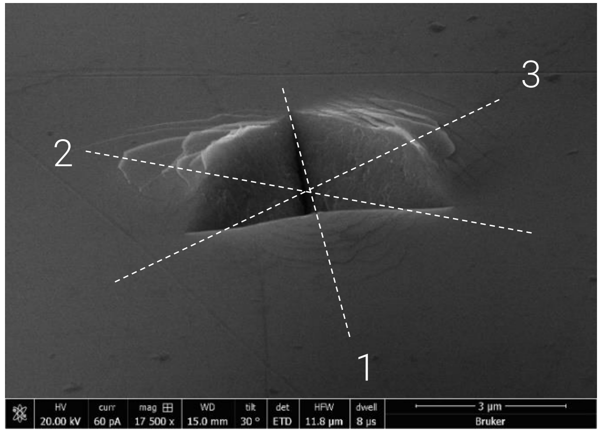
TKD

NanoTribology

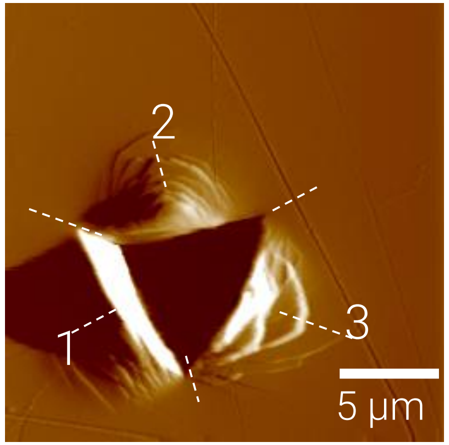
High Temperature

In-Situ SEM Nanomechanics with SPM Imaging

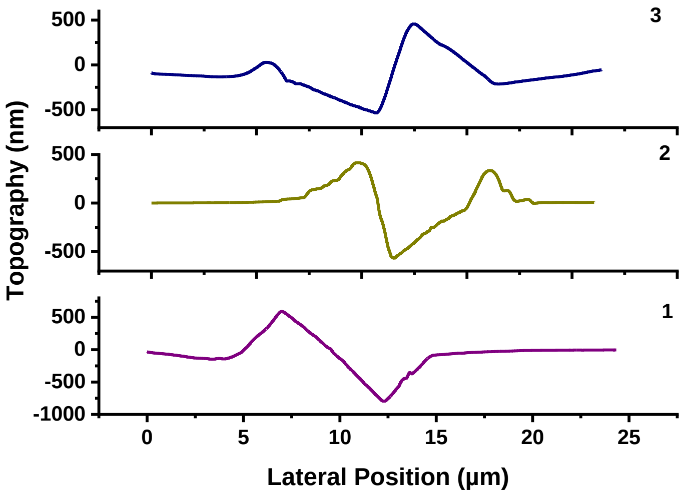
SPM imaging and profile of an *in situ* SEM nanoindentation on bulk metallic glass



SEM image



SPM image

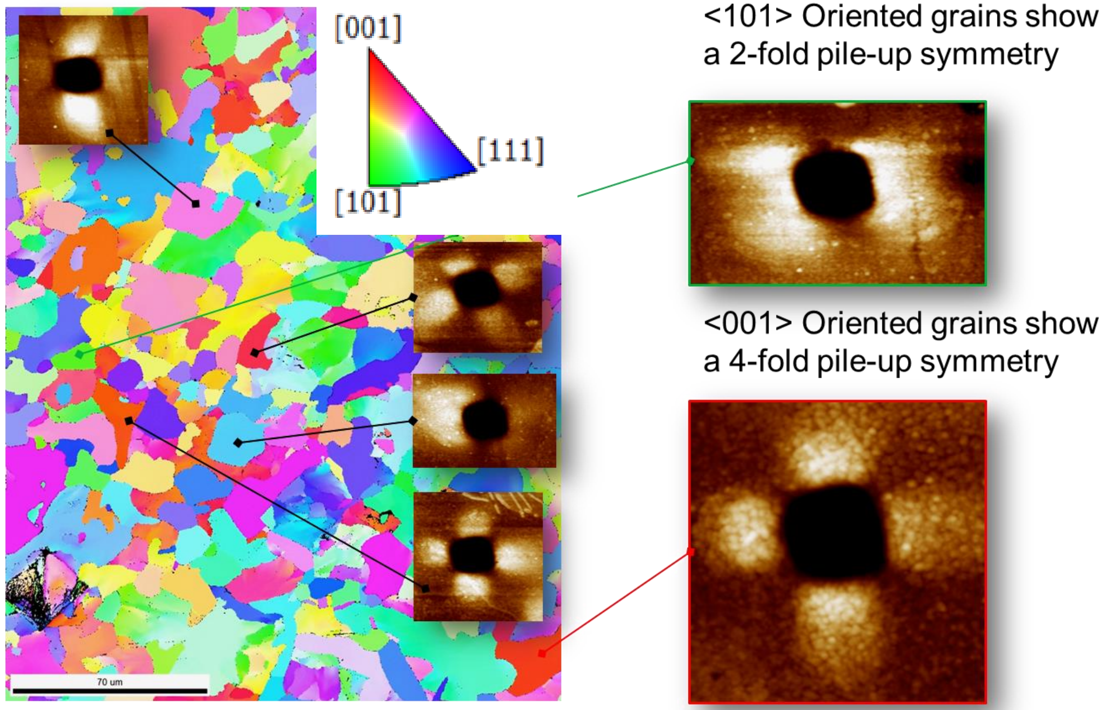


Profile across the indentation can quantify pile up volume and steps formed by slip bands.

- Topographical features can be viewed in high-resolution with secondary electron imaging in the SEM. However, quantitative topographical data is challenging.
- Accurate height information of sample features, surface roughness, pile-up or sink-in from nanoindentation.

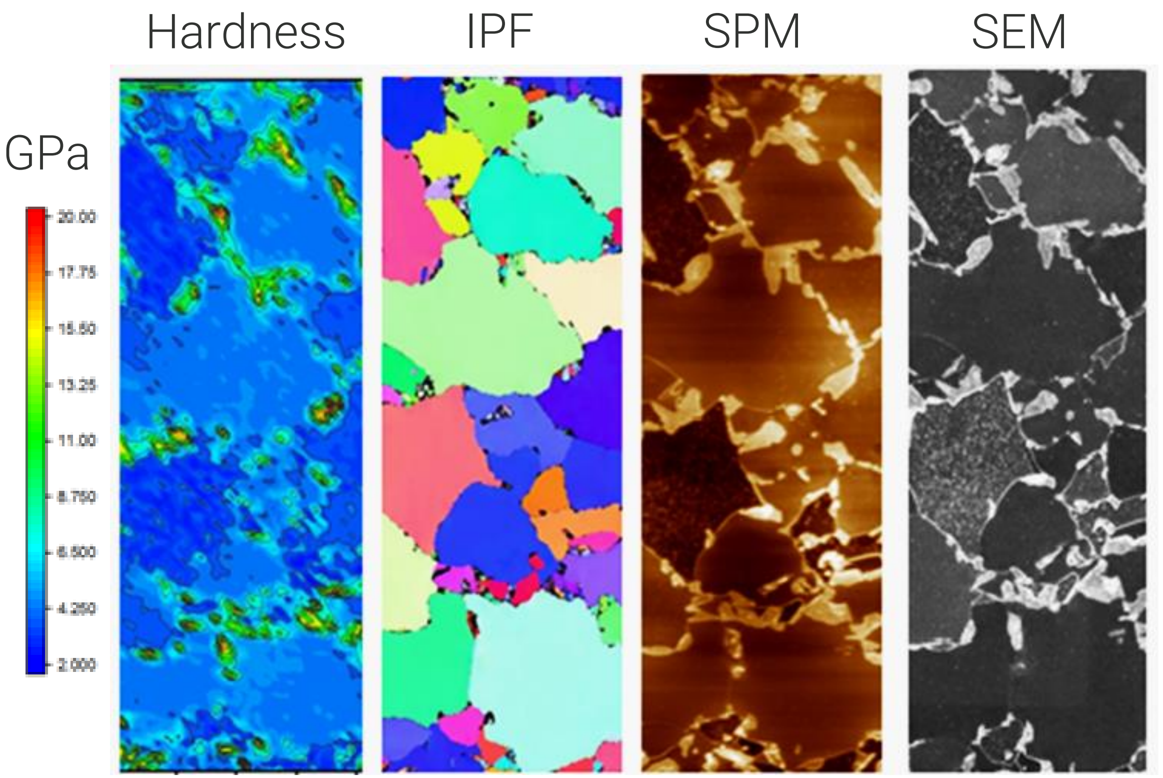
Correlated XPM and EBSD

Pile-up symmetry depends on crystallographic orientation



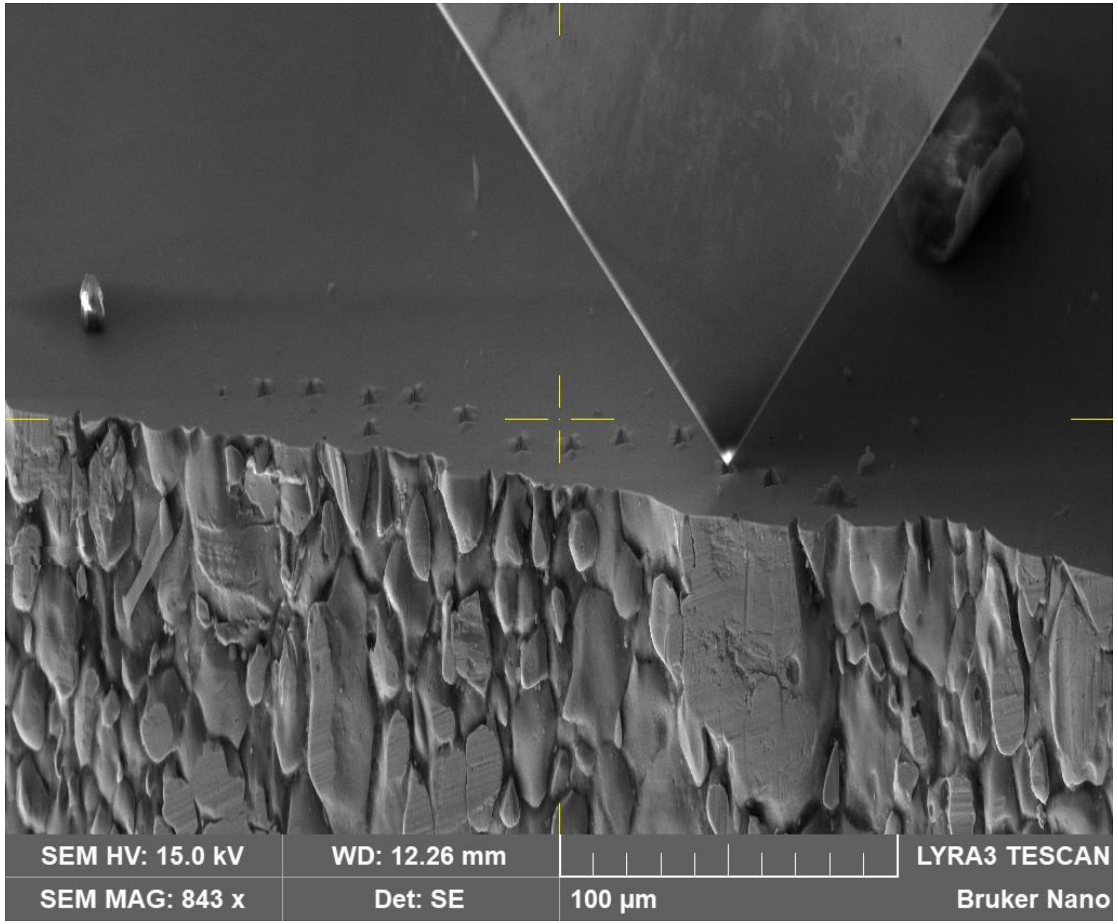
IEHK STEEL INSTITUTE

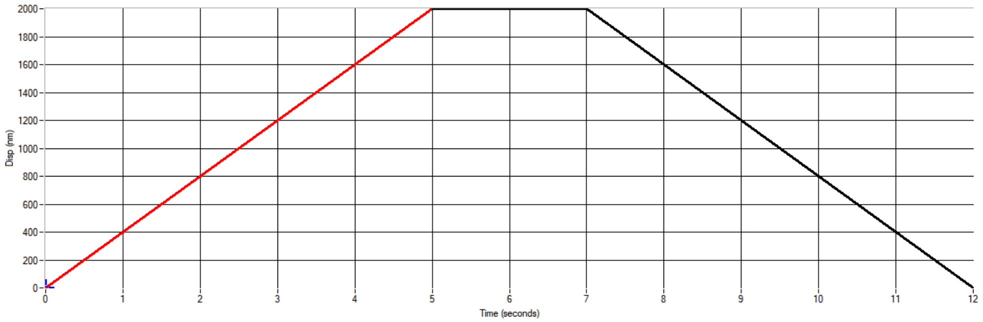
ICAMS
INTERDISCIPLINARY CENTRE FOR ADVANCED MATERIALS SIMULATION



Inverse pole figure (IPF) orientation component uses a basic RGB colouring scheme, fit to an inverse pole figure. For cubic phases, full red, green, and blue are assigned to grains whose <100>, <110> or <111> axes, respectively, are parallel to the projection direction of the IPF.

Indent Positioning and Load Function

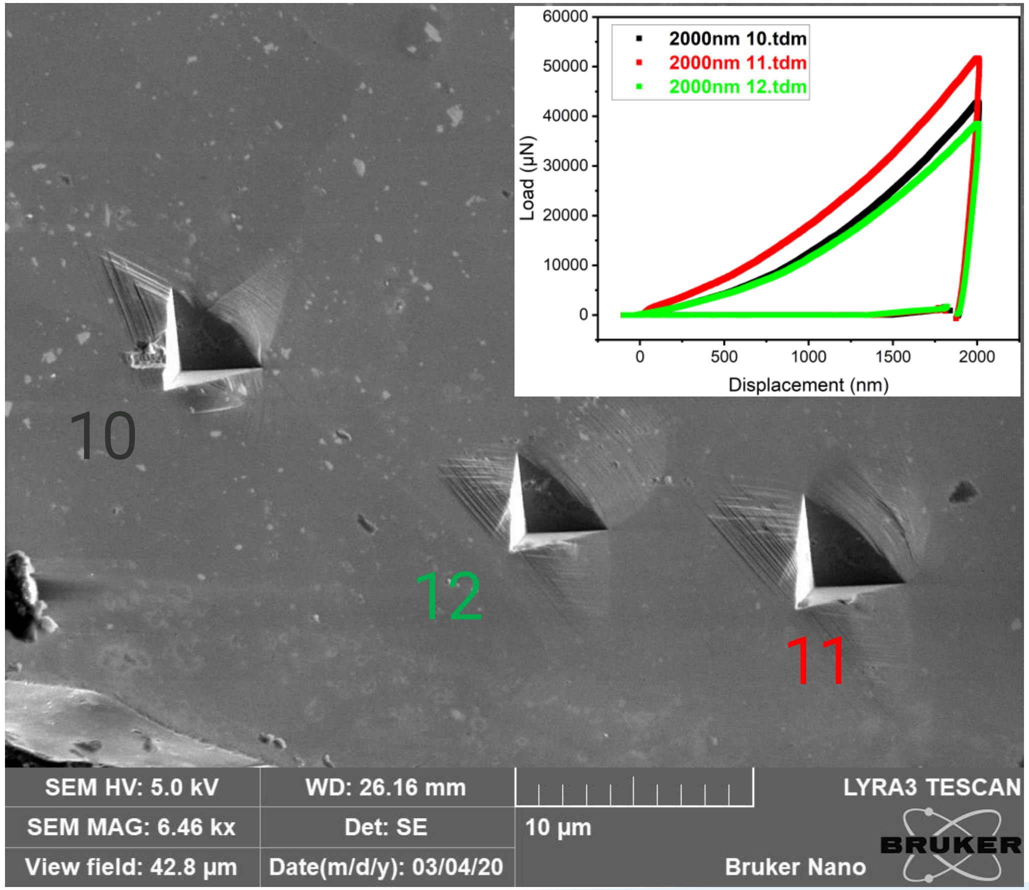


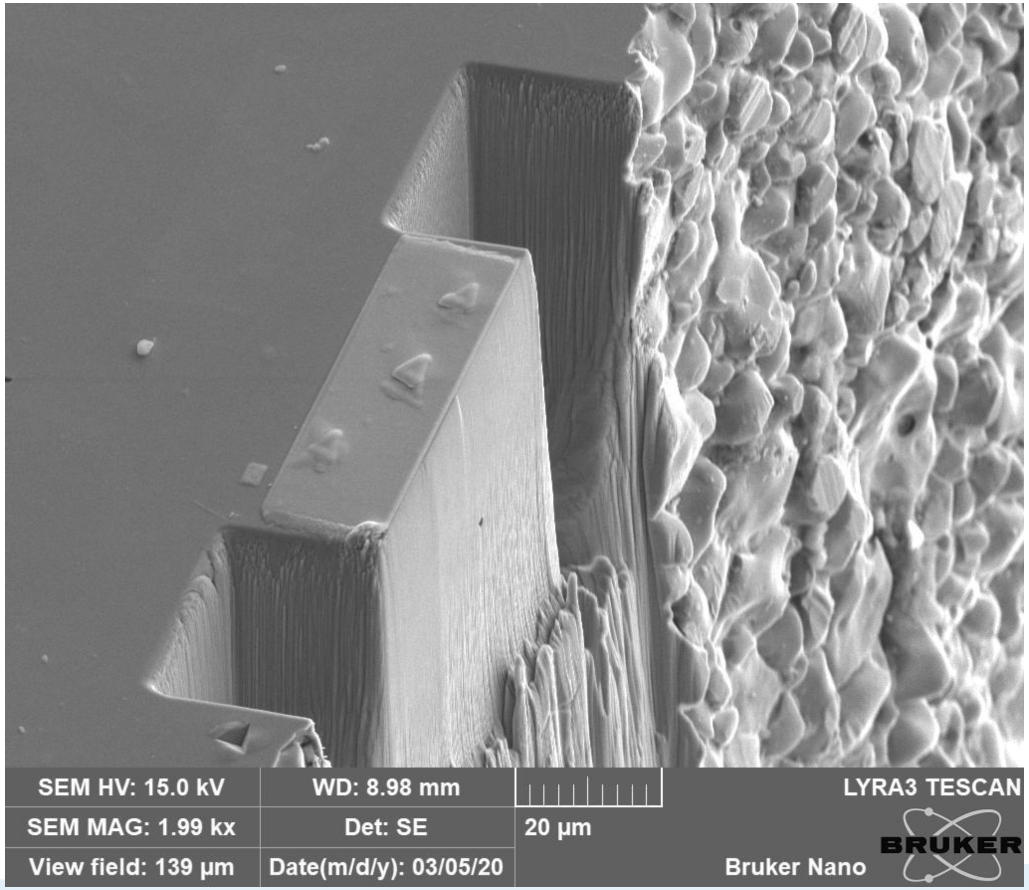


- Displacement Control Feedback Loop
- Peak displacement 2μm
- 5-2-5s of Load-Hold-Unload segments
- Cube Corner tip
- Indentation along the edge of sample
- ROI was defined by EBSD map before indentation

Residual Indents for 3D EBSD

- indents 12&11 made in the same grain.
- indent 11 requires greater applied force to reach the same penetration depth.





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EBSD technique overview

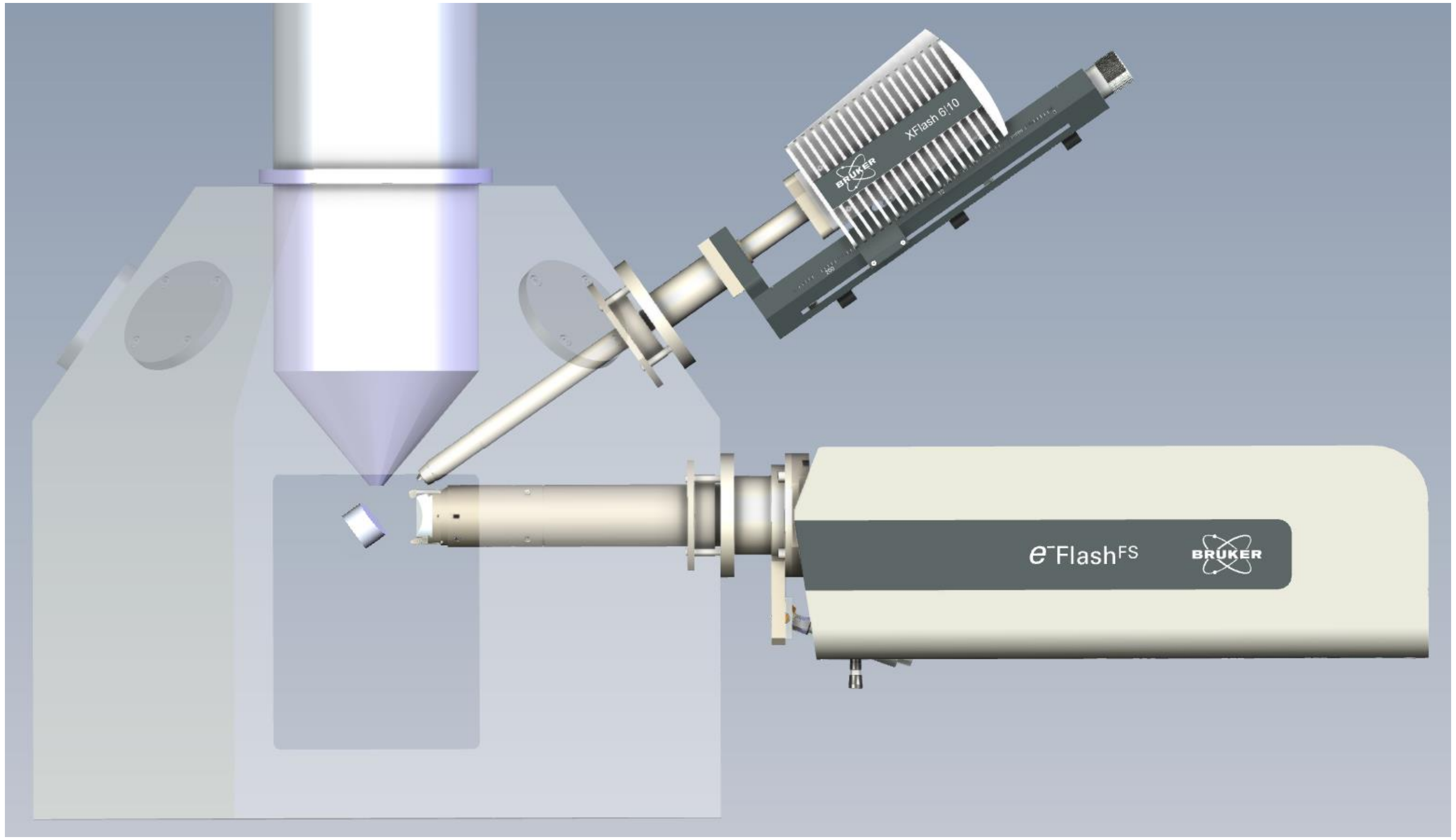
EBSD: What for?

Understanding the material strength and deformation properties by:

- Measuring crystal orientation
- Analyzing the grain structure
- Distinguishing phases
- For quality control, phase transformation, fracture analysis, strain analysis, tectonophysics studies...

Hardware - Experimental setup

- Simultaneous 2D and 3D EDS/EBSD measurement
- Detectors in-situ tilt options to maximize measurement quality
- One software platform



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ESPRIT QUBE
3D EBSD/EDS data processing

3D EBSD overview

EBSD is an extremely versatile tool:

- microstructure, texture (phase discrimination)
- grain boundaries, dislocations (residual stress)

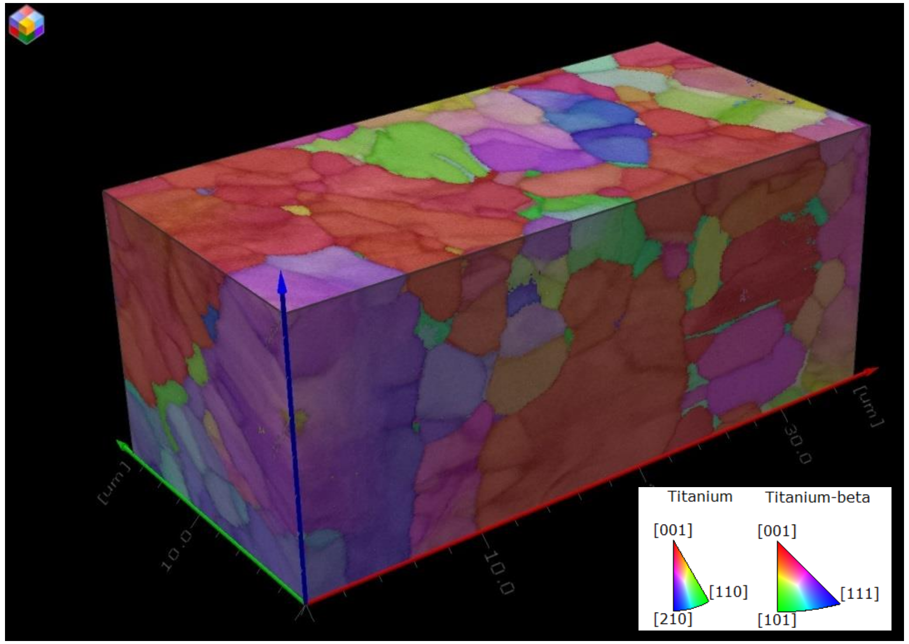
3D EBSD is a specialty tool, brings the most advanced 3D insights into the deformation mechanisms of materials:

- wide range of serial section techniques (destructive)
- slice registration /realignment
- growing list of applications : 5 parameters grain boundary characterization, microstructure characterization in 3D, Nye tensor / Geometrically Necessary Dislocation analysis, ….

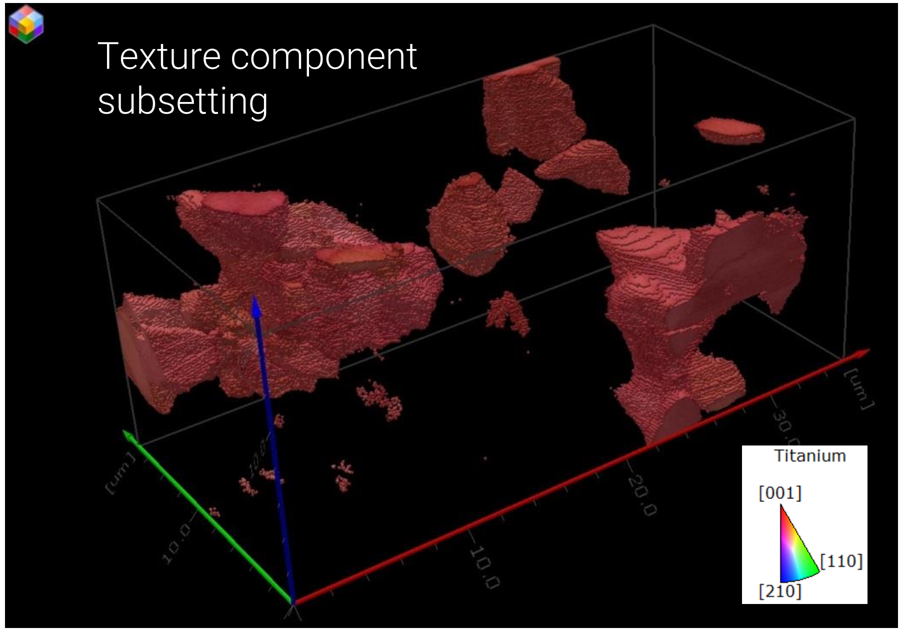
3D EBSD data processing with ESPRIT QUBE

ESPRIT QUBE:

- Advanced postprocessing capabilities for crystal plasticity studies, e.g. GND density distribution
- Multiple advanced slice realignment features
- Multiple subsetting

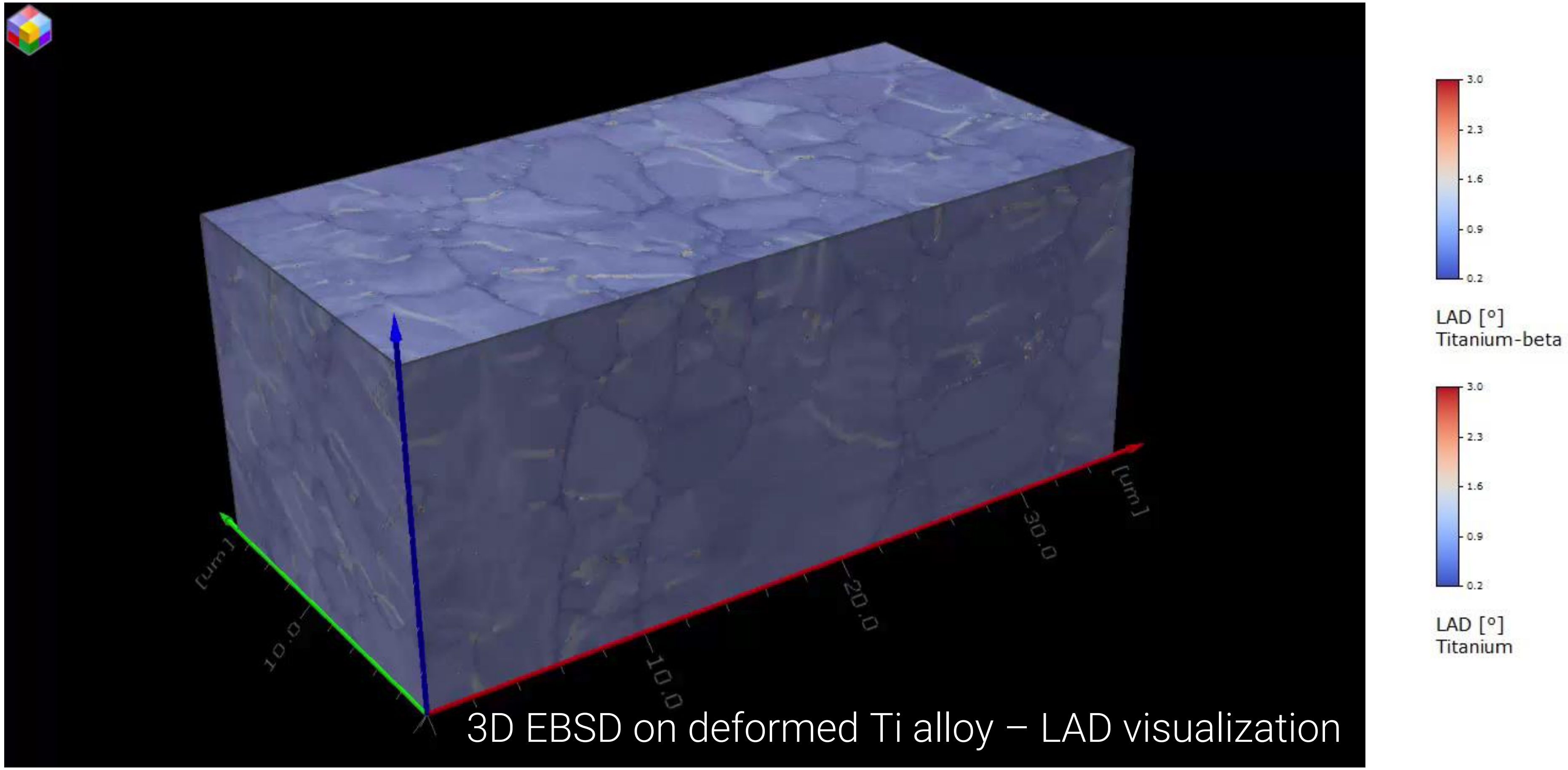


- sample: deformed Ti alloy (α and β phases)
- Large data cube – 245 slices (35x20x20μm³)
- Serial sectioning using a PFIB-SEM with non-static 3D
- Slice preparation time: ~2min/slice
- 3D EBSD data acquisition time: ~7min/slice



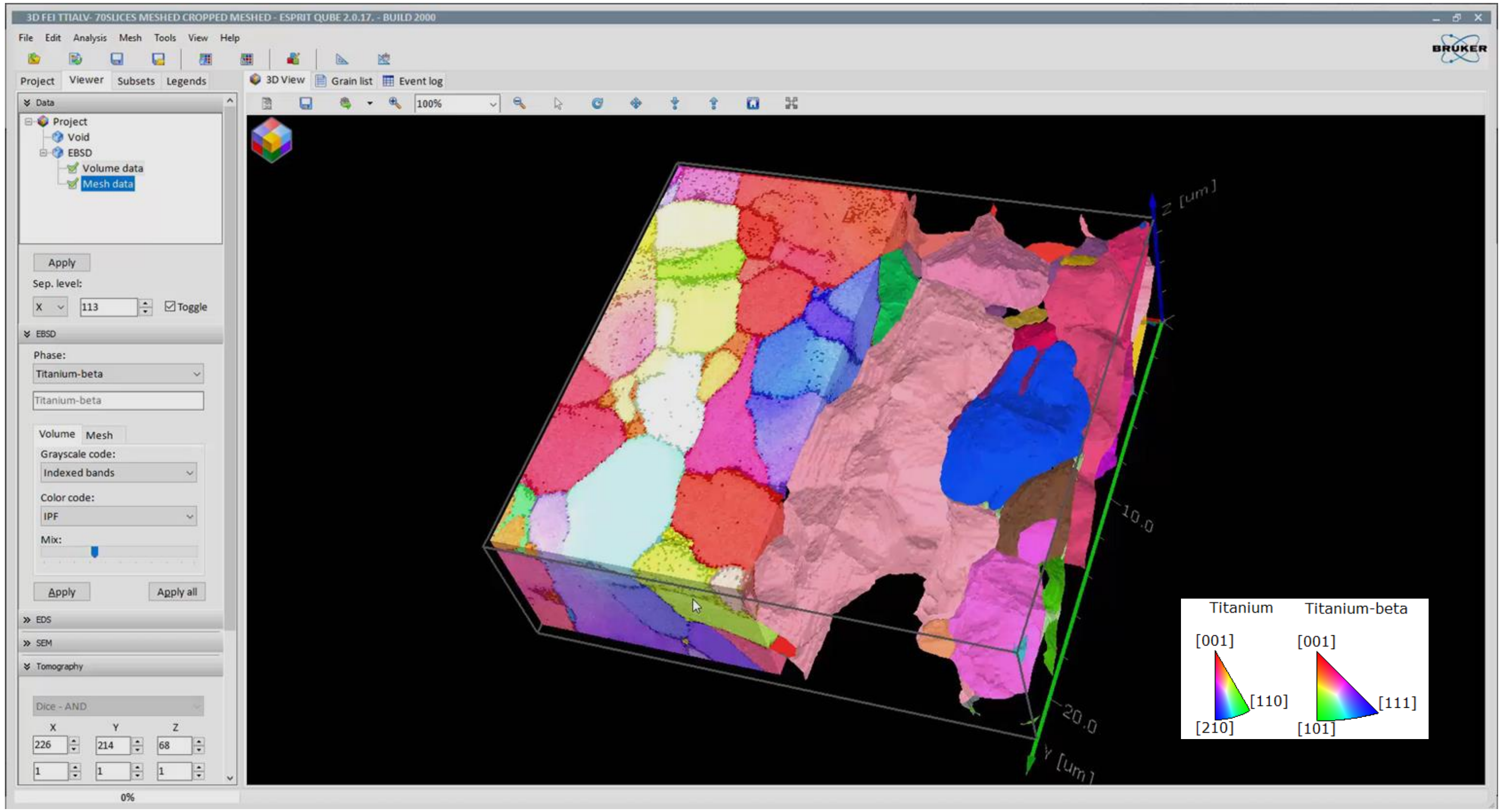
3D EBSD data processing with ESPRIT QUBE

- Local Average Disorientation after slice realignment & data filtering



3D EBSD data processing with ESPRIT QUBE

- Grain Boundary network (Ti alloy) IPF and grain orientation mean (Euler coloring)



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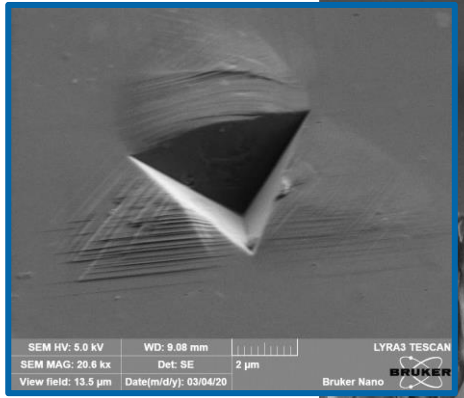
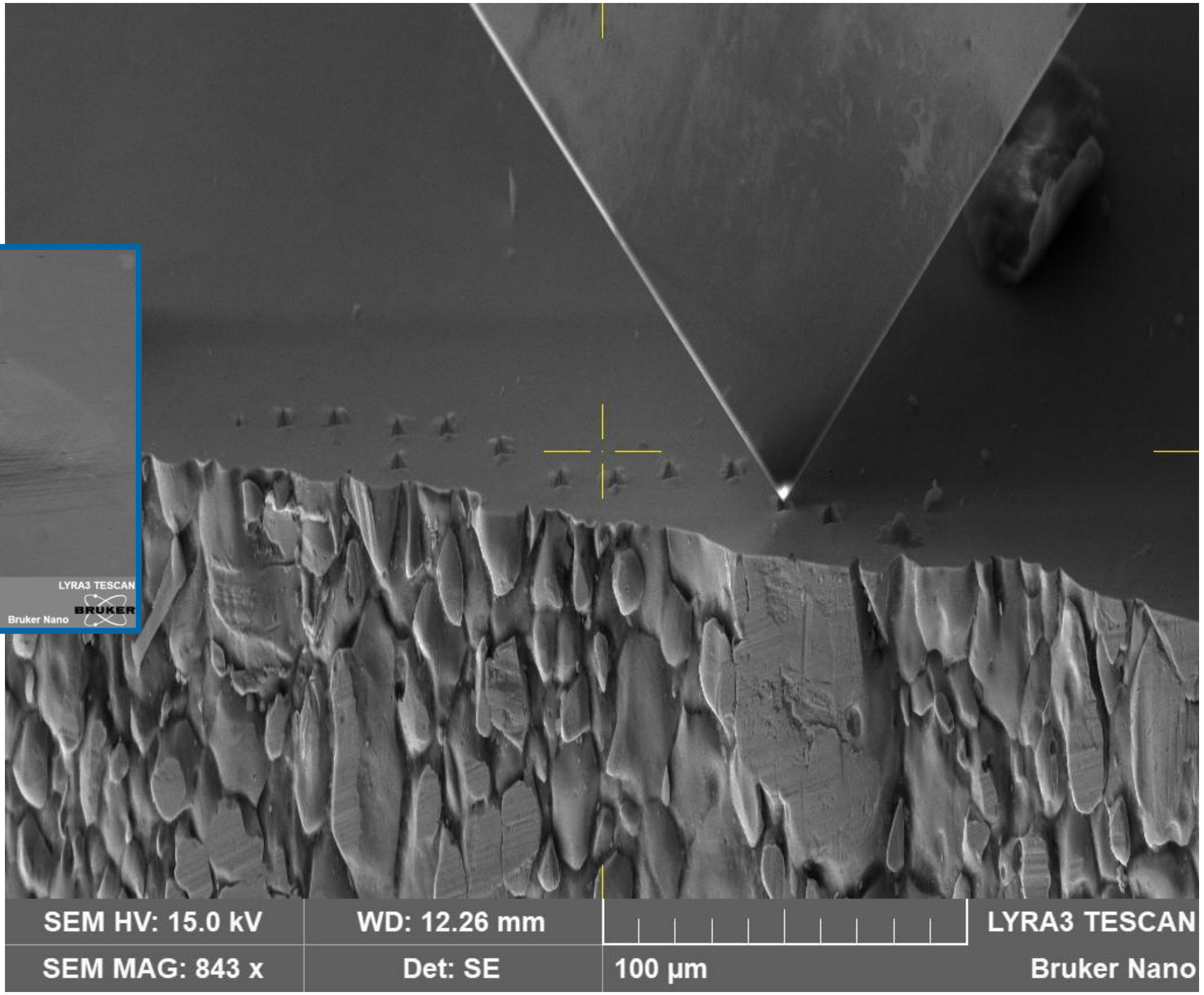
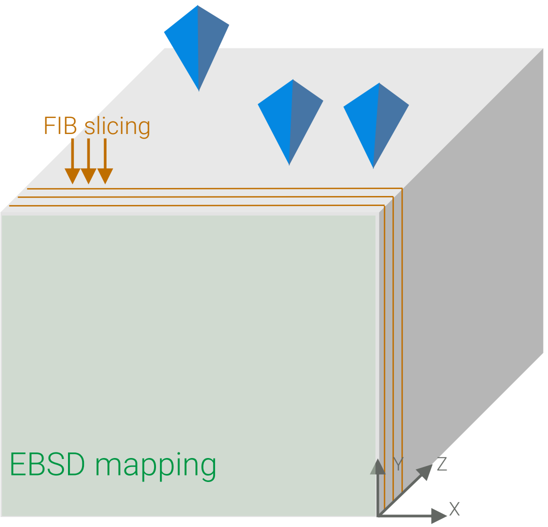
3D EBSD measurement and data processing

Experimental setup - indentation



Stacking faults
visible at the surface

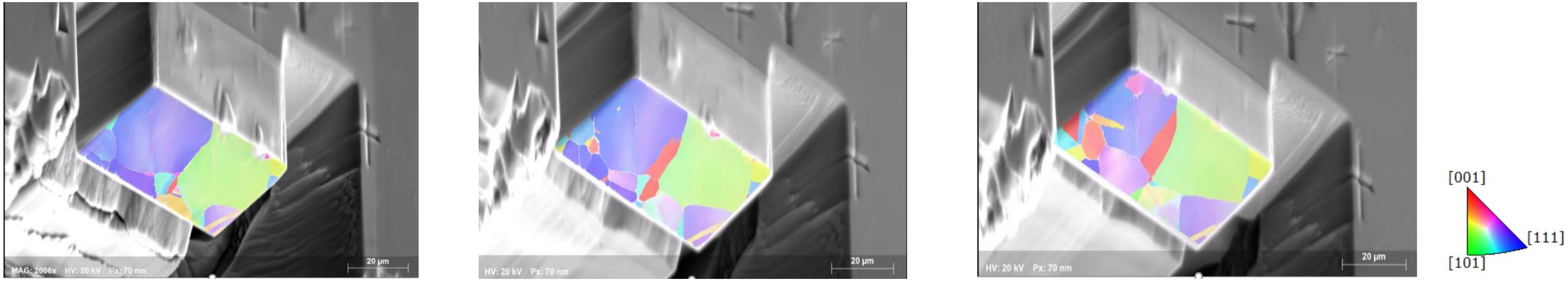




Voxel size is 70nm³
245 slices, 56(x)*35(y)*171(z) µm
160x120 pixels
2 ms

Experimental setup – 3D EBSD measurement

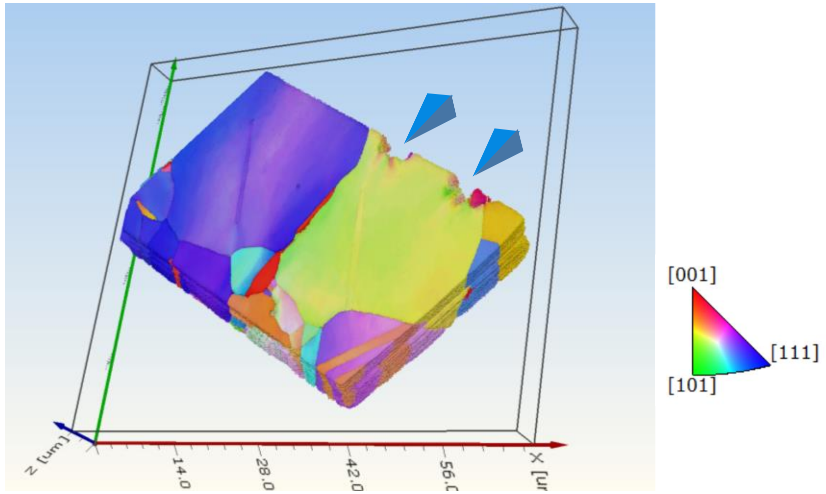
- Watch the recording to access the video of the 3D EBSD measurement



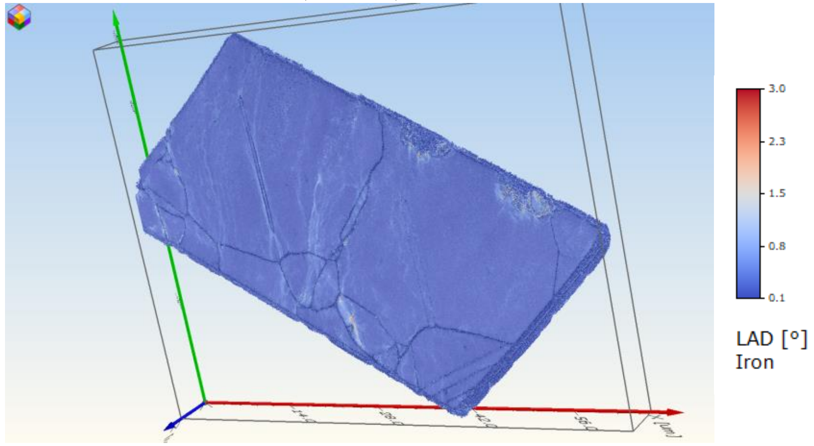
3D EBSD data processing

Rigid slice realignment and noise filtering applied on selected area below (~37 Millions of points)

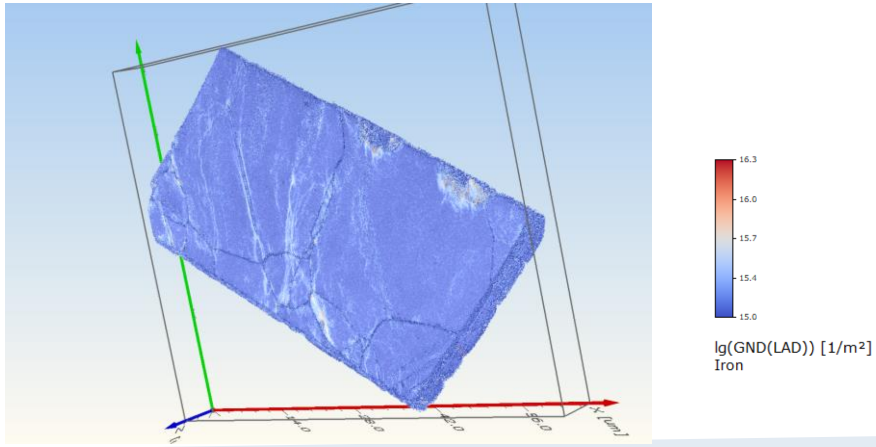
- IPF



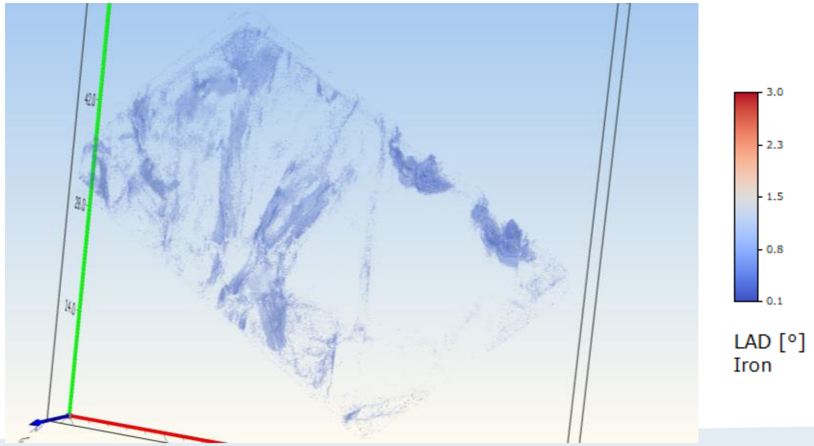
- Local Average Disorientation (LAD)



- GND

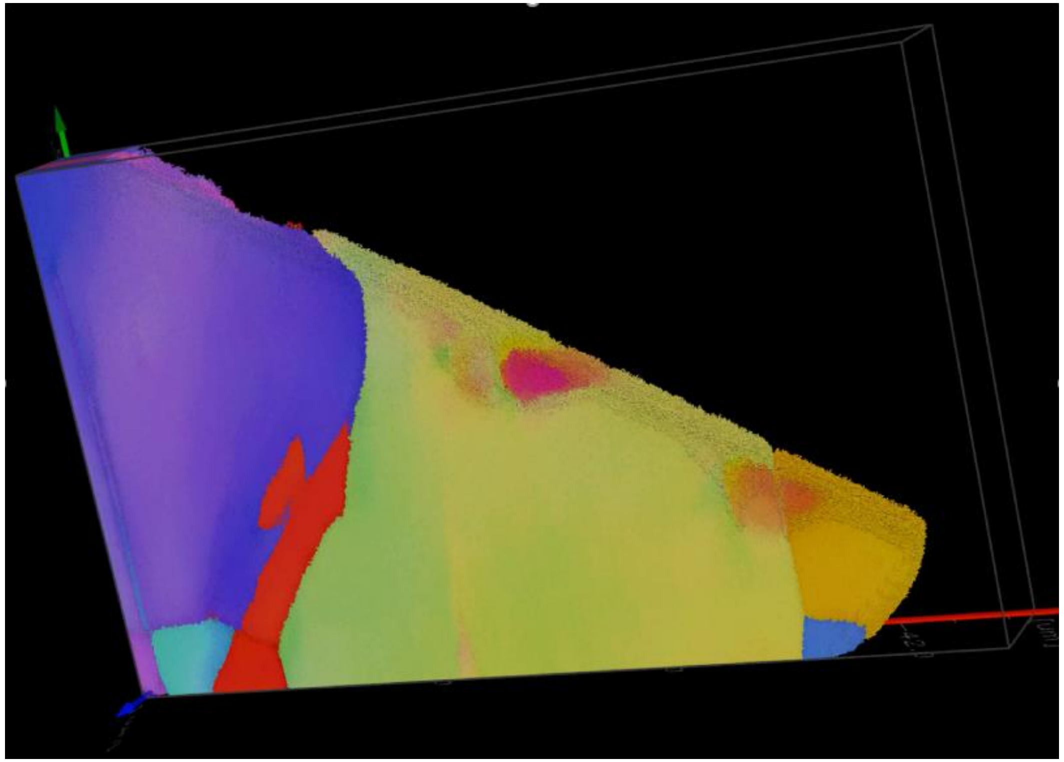


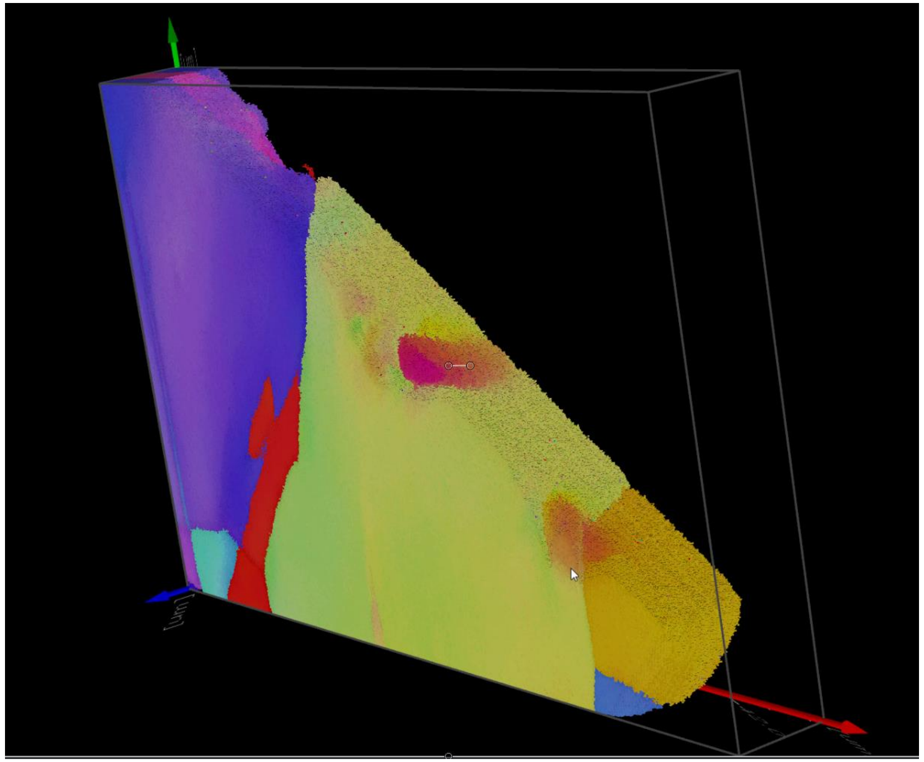
- LAD scalar color scheme

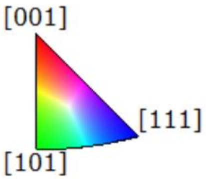


3D EBSD – Orientation Distribution (IPF)

- Close-up on the indents area – plastic deformation area clearly visible
- Indent 11 (right) has a smaller plastic deformation zone. It was made in the vicinity of a grain boundary.

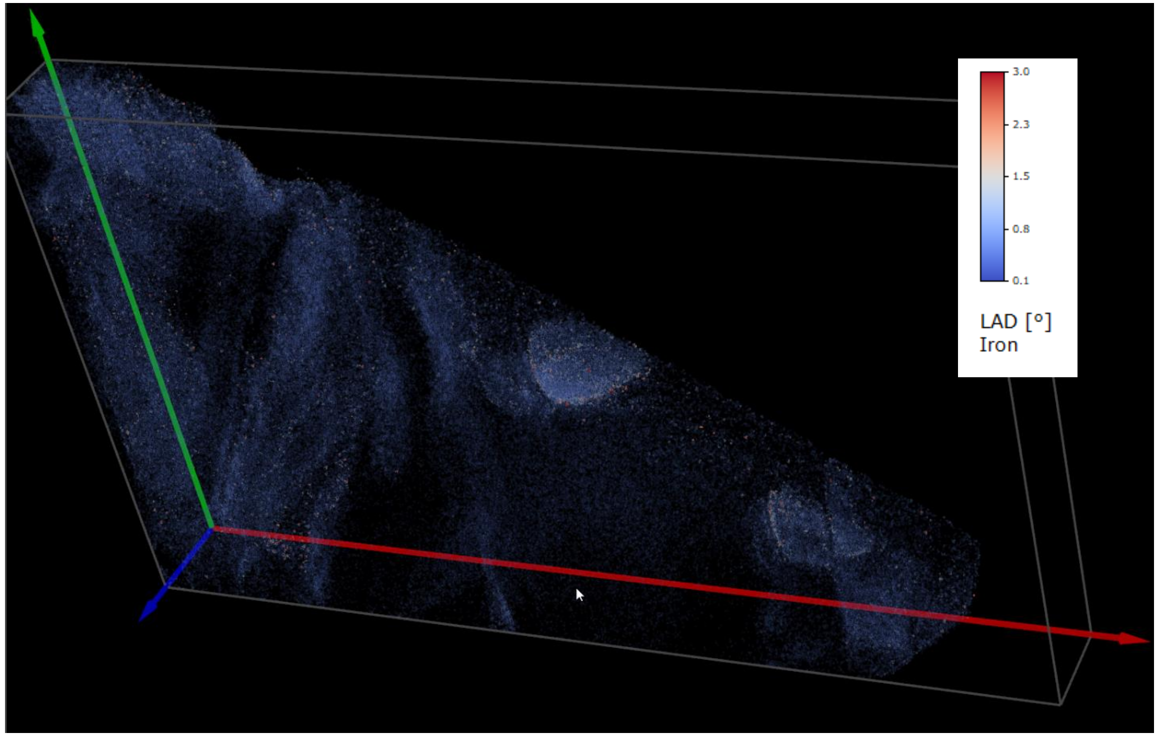


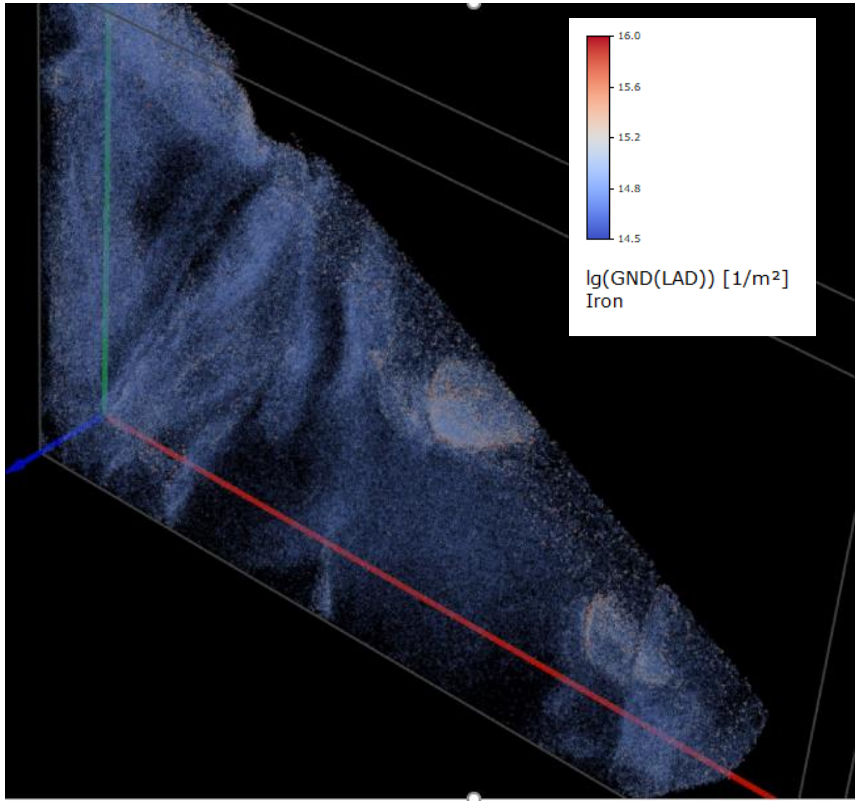




3D EBSD - Local Average Disorientation (LAD/KAM) and Geometrically Necessary Dislocation (GND)

- LAD : plastic deformation delimited by low angle boundaries
- GND: they are stacking up at low angle boundaries





Plastic deformation zones clearly delimited by LAD and GND distribution. Indent 11 (right) has a smaller plastic deformation zone and required more applied force because of the vicinity of a grain boundary which reduces the mobility of dislocations.

Some suggested references…

- N. Zaafarani, D. Raabe, R.N. Singh, F. Roters, S. Zaefferer, Three-dimensional investigation of the texture and microstructure below a nanoindent in a Cu single crystal using 3D EBSD and crystal plasticity finite element simulations, Acta Materialia 54 (2006) 1863-1876
- M. Rester, C. Motz, R. Pippan, Microstructural investigation of the volume beneath nanoindentations in copper, Acta Materialia 55 (2007) 6427–6435
- "Assessment of geometrically necessary dislocation levels derived by 3D EBSD", P.J. Konijnenberg *et al.,* Acta Mat. 99 (2015) 402–414, doi: 10. 1016/j.actamat.2015.06.051
- " 3D HR-EBSD Characterization of the plastic zone around crack tips in tungsten single crystals at the micron scale " S. Kalácska *et al.,* Acta Mat. 200 (2020) 211-222
- "Investigation of geometrically necessary dislocation structures in compressed cu micropillars by 3-dimensional HR-EBSD" S. Kalácska *et al.,* Mat. Sci. Eng. A. 770 (2020) 138499, doi: 10.1016/j.msea.2019.138499 .

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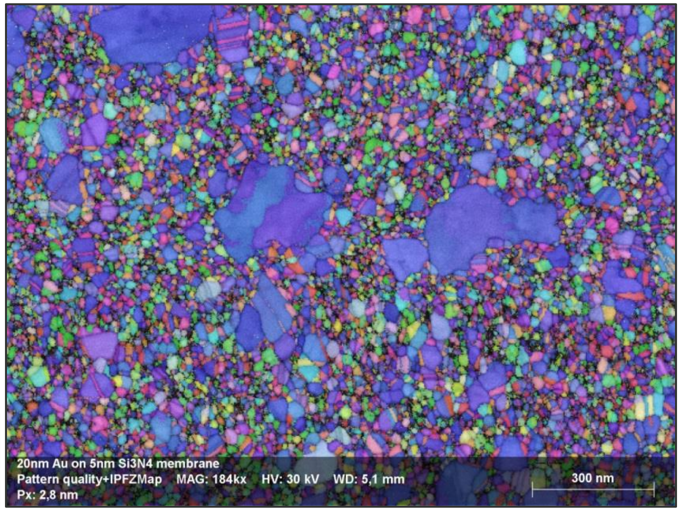
Advanced applications: combination with OPTIMUS TKD

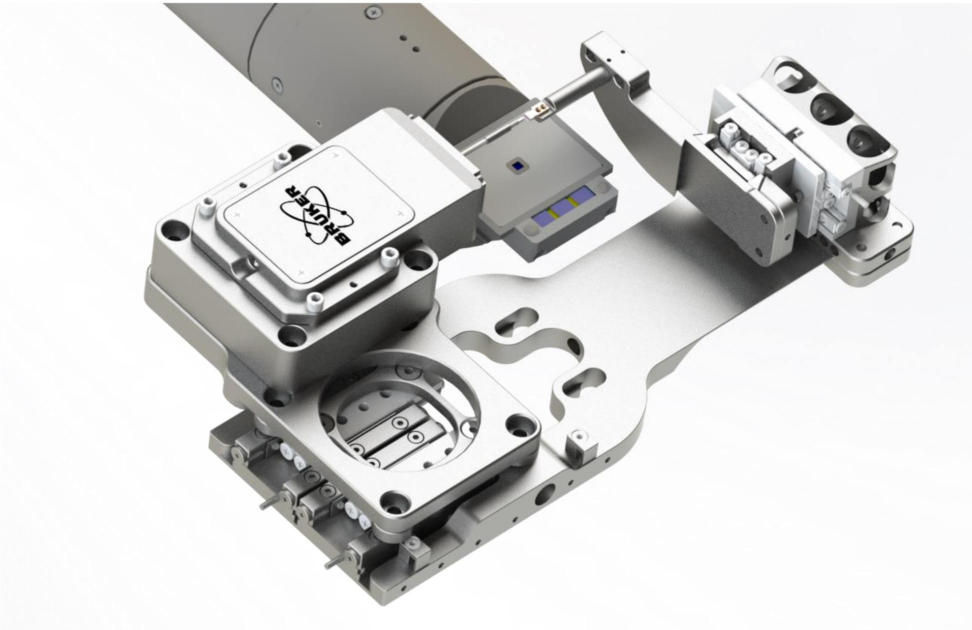
Tensile testing of nanowires or thin films

OPTIMUS 2 TKD Detector



- OPTIMUS 2 is an add-on option of the e-Flash EBSD detectors
- On-axis Transmission Kikuchi Diffraction
- Effective spatial resolution 1.5 nm
- ARGUS™ imaging system with near real-time visualization

SEM PicoIndenter with OPTIMUS 2 detector



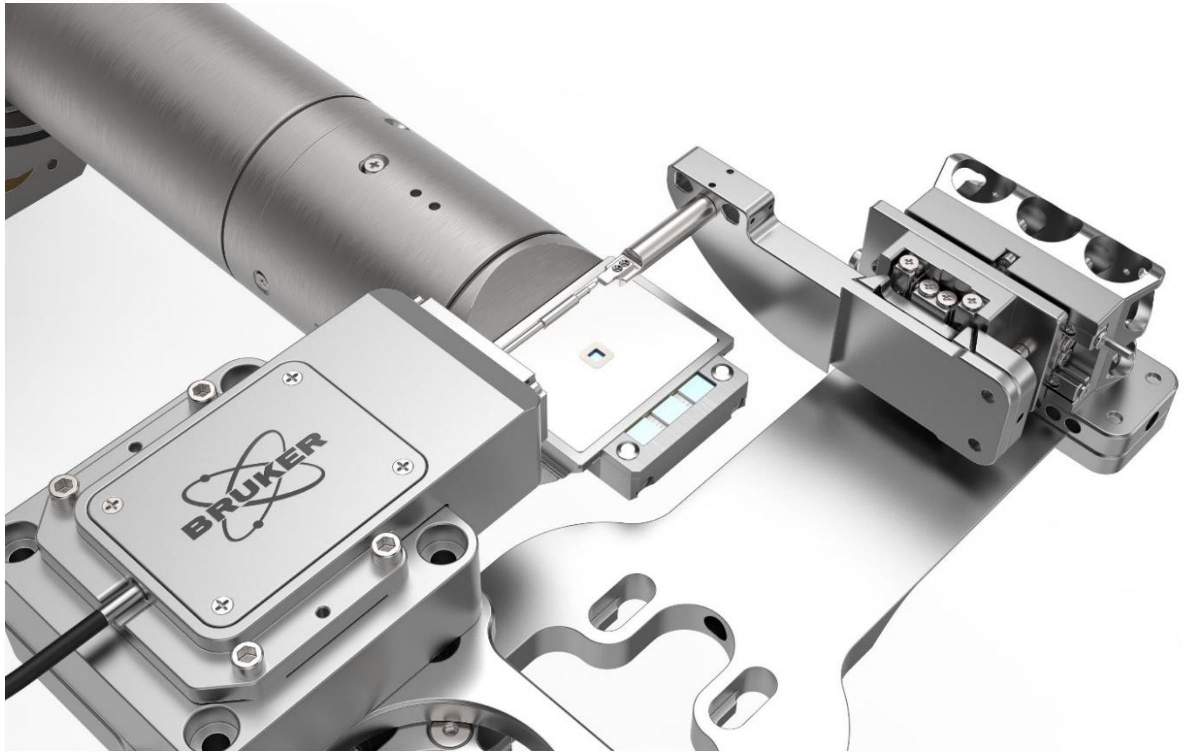
- Tensile testing of thin films
- Strain, grain structure and deformation mapping
- Suitable with Push-to-Pull device

Integration of multiple instruments
OPTIMUS 2 and PicoIndenter PI 89 with Push-to-Pull device

Time Resolved Measurements during in-situ experiments:

- *Near real-time visualization* at up to 125,000 pixels/second during in-situ tensile testing with Push-to-Pull device on PI 89
- Time resolved mapping at up to 600 points/second during in-situ tensile testing – quantify every microstructural change in the sample
- Same capabilities apply to in-situ heating and electrical biasing experiments

Sample and detectors setup for Time Resolved Measurements during in-situ tensile testing of nanomaterials



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Summary

Summary

Combined mechanical testing with 3D EBSD measurement allow :

- Correlating mechanical properties with the complete microstructural features (GB, twins, low angle boundary, GNDs…)
- Study of plastic deformation mechanisms, crystal plasticity
- Study the strength of heterogenous materials
- Fracture analysis (cantilever)
- Micropillar compression
- Investigation of residual stress with HR-EBSD, 3D shape of deformation zone, …
- Understanding the role of crystal orientation and different dislocations in the plastic deformation, …

- Further: Tensile/heating testing on thin lamella

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Q&A

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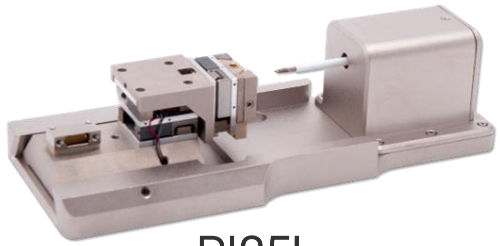
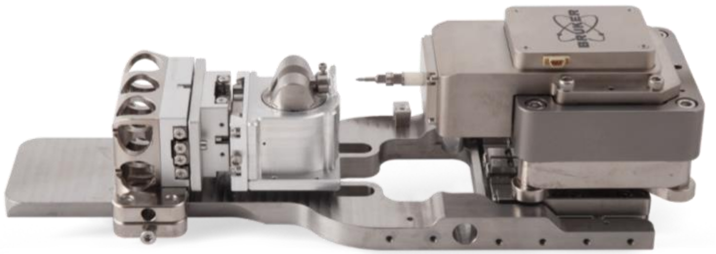
Appendix

Enabling Your Control Mode



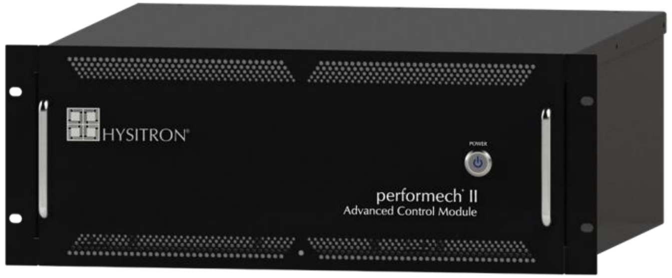
PI85L
„True Load Control"



PI89
„True Load Control"
„True Deplacement Control"

+

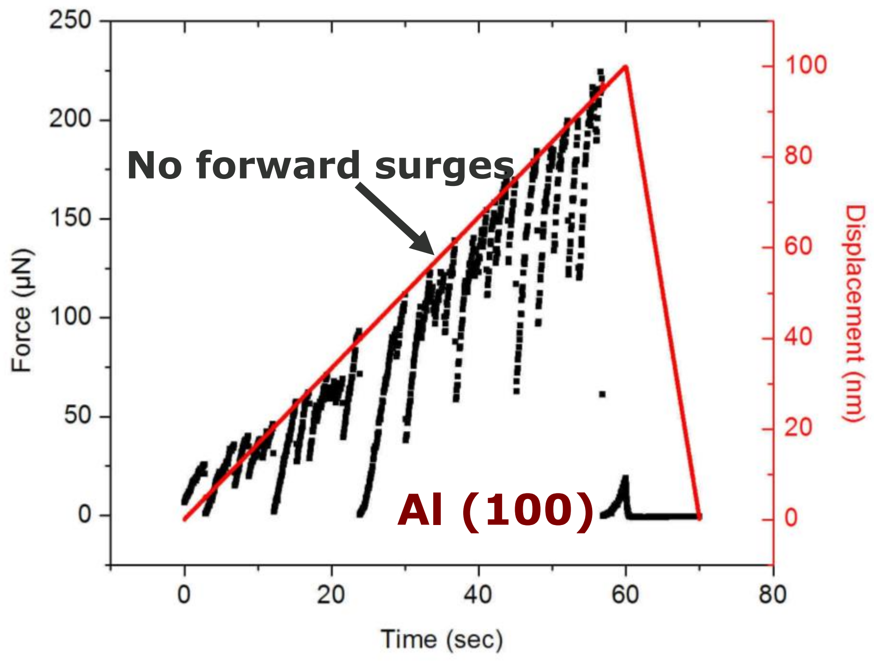
Performech II



- 1.2MHz Data Sample Rate
- 78kHz Feedback Control Loop
- 38kHz Data Acquisition Rate
- 20nN Force Noise Floor
- 0.1nm Displ. Noise Floor

=

Displacement Control



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Thank you!

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