

## Introduction

- In-situ observation of materials evolution in extreme environments allows for increased insight into the active mechanisms and temporal dependencies governing materials response and transformation
- Sandia National Laboratories (SNL) has previously developed the In-Situ Ion Irradiation Transmission Electron Microscope (I<sup>3</sup>TEM) facility capable of studying the effects of radiation damage, high temperatures, mechanical stresses and other environmental variables on microstructural transformation in thin-film specimens<sup>1</sup>
- However, the I<sup>3</sup>TEM fails to capture mesoscale and surface transformation effects, and in-situ TEM experiments in general are known to be subject to various thin-film effects
- To this end, SNL has developed the In-Situ Ion Irradiation Scanning Electron Microscope (I<sup>3</sup>SEM) facility to complement the I<sup>3</sup>TEM capabilities and enabling in-situ materials studies at larger length scales.

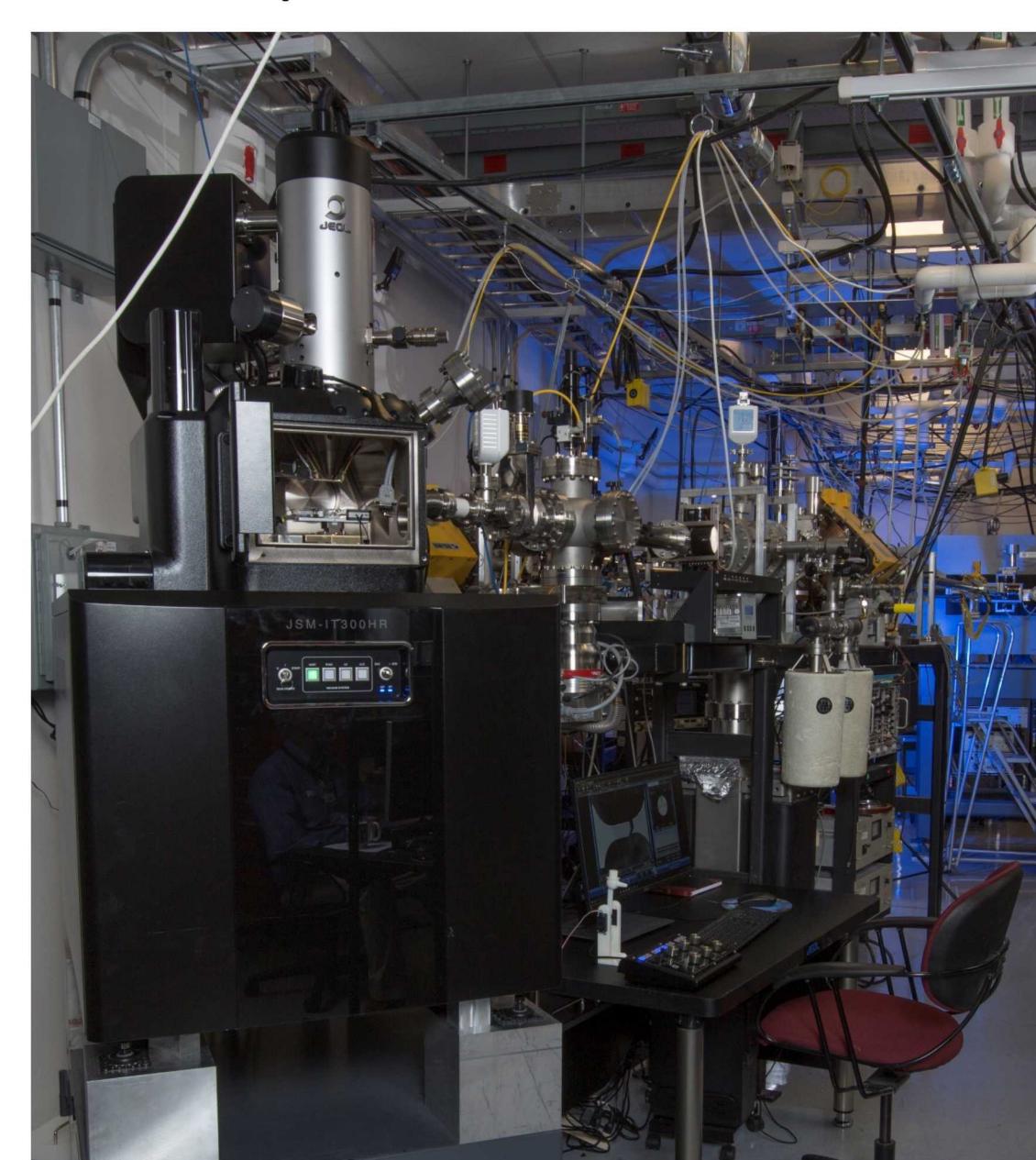


Figure 1: Image of I<sup>3</sup>SEM facility as installed in the SNL Ion Beam Laboratory

## Facility Description

### Base Instrument – JEOL JSM-IT300HR/LV

- 30 kV FEG SEM w/ low vacuum capabilities
- Largest available sample chamber

### Irradiation Capabilities

- 6 MV Pelletron EN Tandem Accelerator
  - SNICS Source
  - Alphatross He Source
  - Duoplasmatron Proton Source
  - Hiconex 834 Sputter Source
- KRI KDC10 1.2 keV Plasma Ion Source



Figure 2: Ion beam induced luminescence (IBIL) on a quartz target in the SEM

### Mechanical Testing Capabilities

- Hysitron PI-85 SEM Picoindenter
- MTI Fullam Tensile Stage
- Custom Piezo Fatigue Stage<sup>2,3</sup>

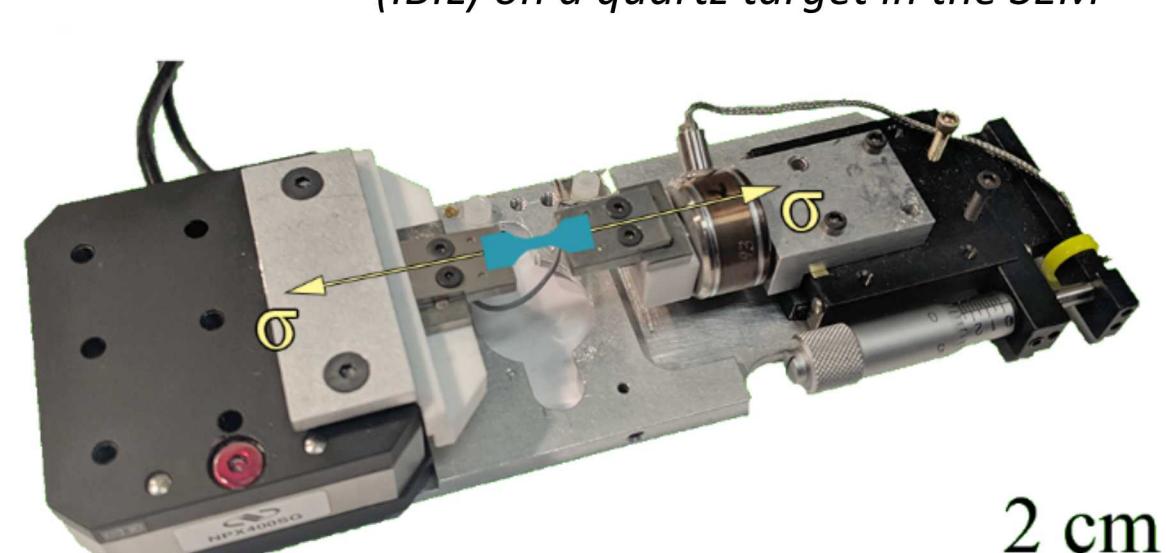


Figure 3: Custom piezo fatigue testing stage

### Analytical Capabilities

- EDAX Octane Super Elite EDS Detector
  - 70 mm<sup>2</sup> detector, up to 400,000 cps
- EDAX Velocity Super EBSD Detector
  - Up to 4,500 indexed points per second

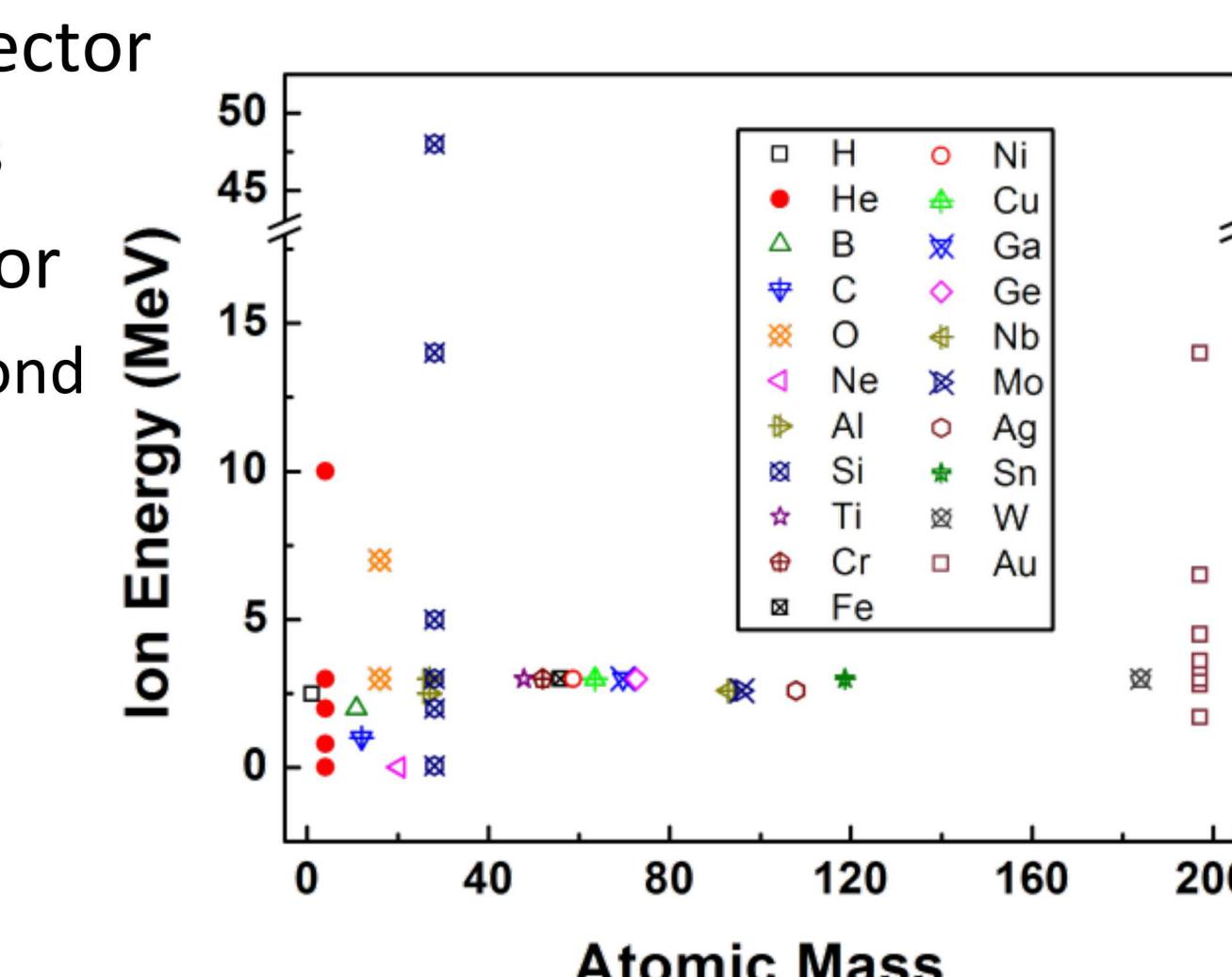


Figure 4: Ion species and energies run in beamline to date

### Future Capabilities

- High-power Specimen Drive Laser
  - 355, 532, 1064 nm wavelengths
  - 8 ns pulses – continuous operation
  - Up to 100 mJ power
- 3-axis straining stage
- Gatan SEM Cryostage

## Facility Configuration

- Microscope is raised significantly above ground level to allow 69" tandem beamline to enter the chamber via a custom WDS port adapter
- Beamline gate valves are interlocked with SEM vacuum system, preventing accidental venting of accelerator with routine sample exchanges
- Custom WDS port adapter allows for tandem beam, KDC10 ion beam, and specimen drive laser to be simultaneously incident on sample

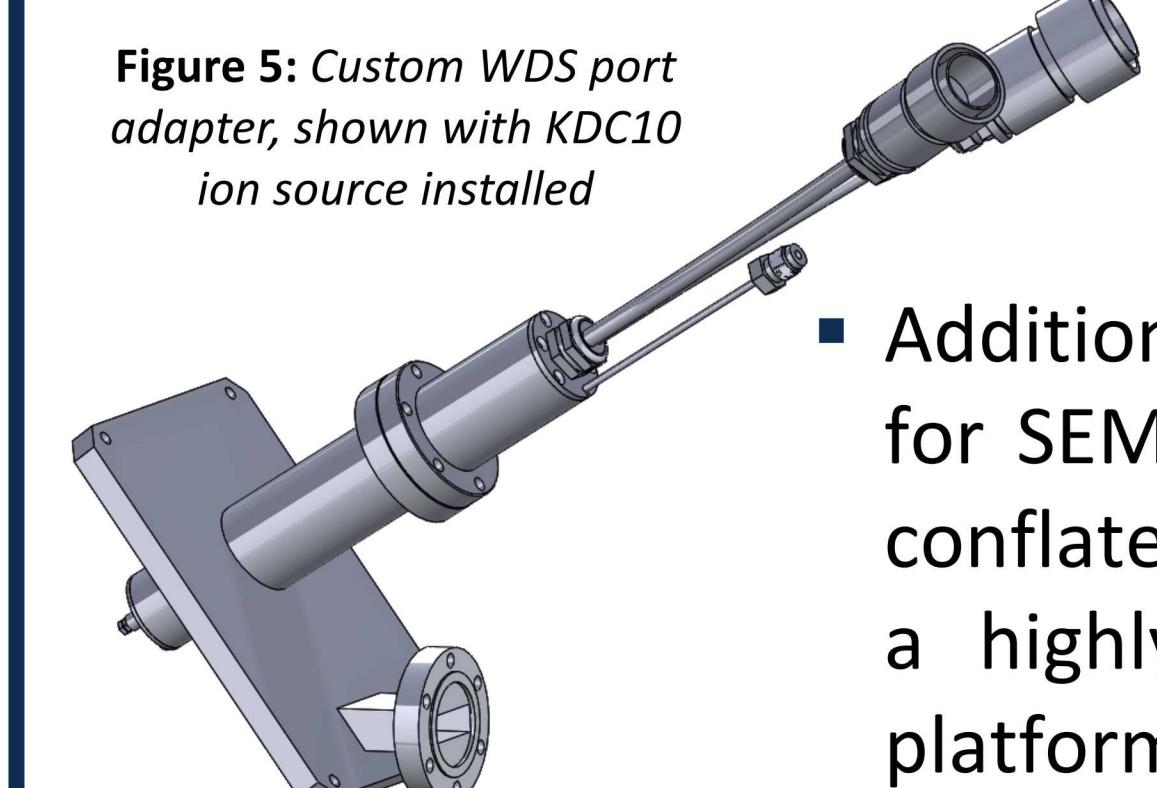


Figure 5: Custom WDS port adapter, shown with KDC10 ion source installed

- Additional custom feedthroughs for SEM stage instrumentation, conflat adapters, etc. result in a highly flexible experimental platform

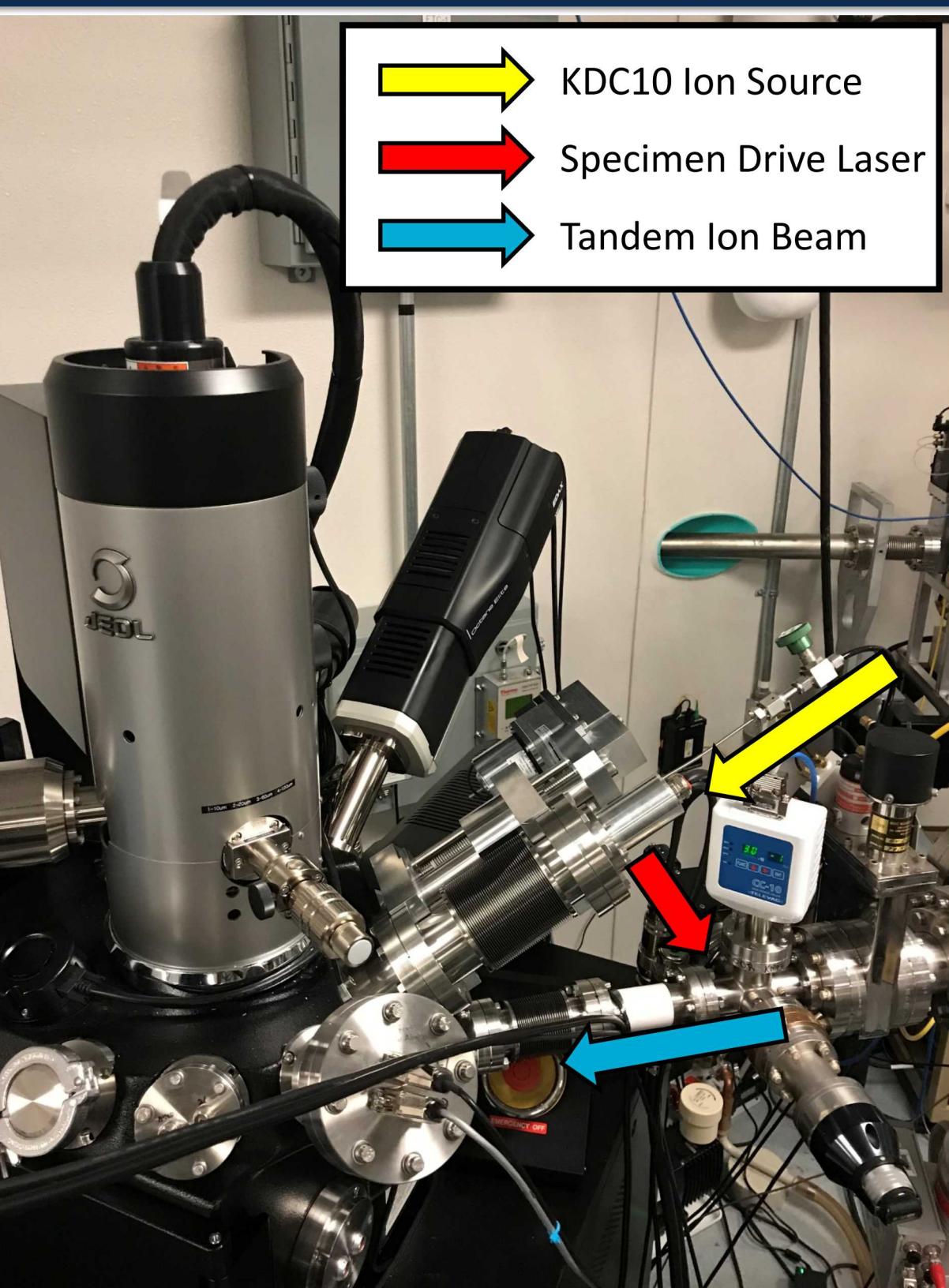


Figure 6: Schematic demonstrating simultaneous heavy and light ion irradiation with laser heating enabled via a custom adapter utilizing the stock WDS port

## Initial Results

### Demonstration of Irradiation Capabilities

- Beams aligned using quartz targets and optical windows/camera
- Irradiation of zirconia waste form using 10 MeV Au<sup>4+</sup> ions
  - Ion irradiation causes initially rough, porous surface to evolve into plate-like structures

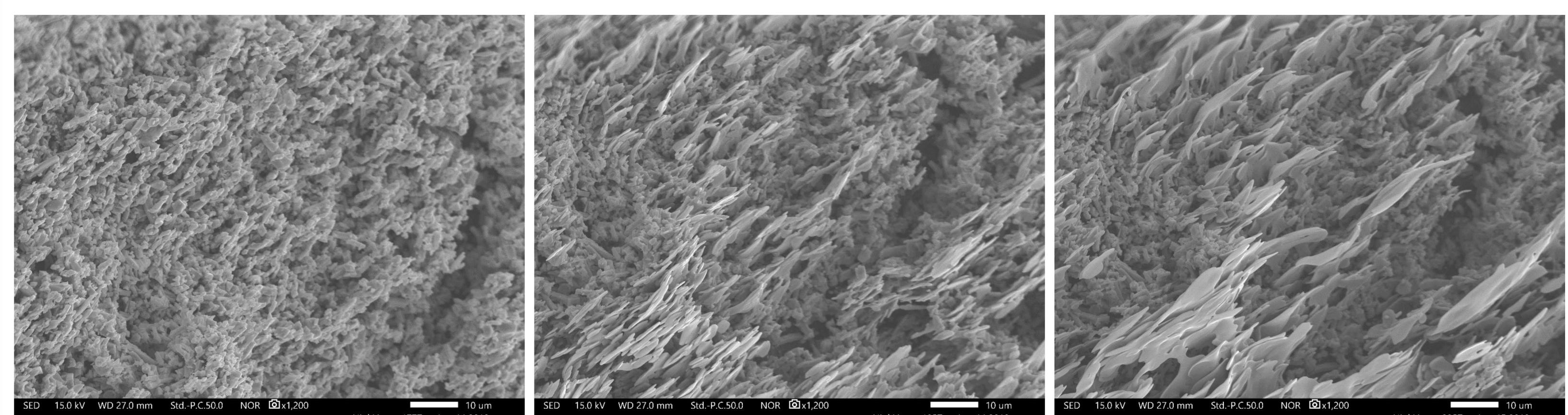


Figure 7: Surface structure evolution of zirconia waste form during in-situ ion irradiation

### In-situ Micro-Compression of Small Particles

- Kovar (Fe-Ni-Co) microparticles on Kovar substrate deposited from laser ablation
- Allows for analysis of elastic deformation and eventual yield and failure by shearing

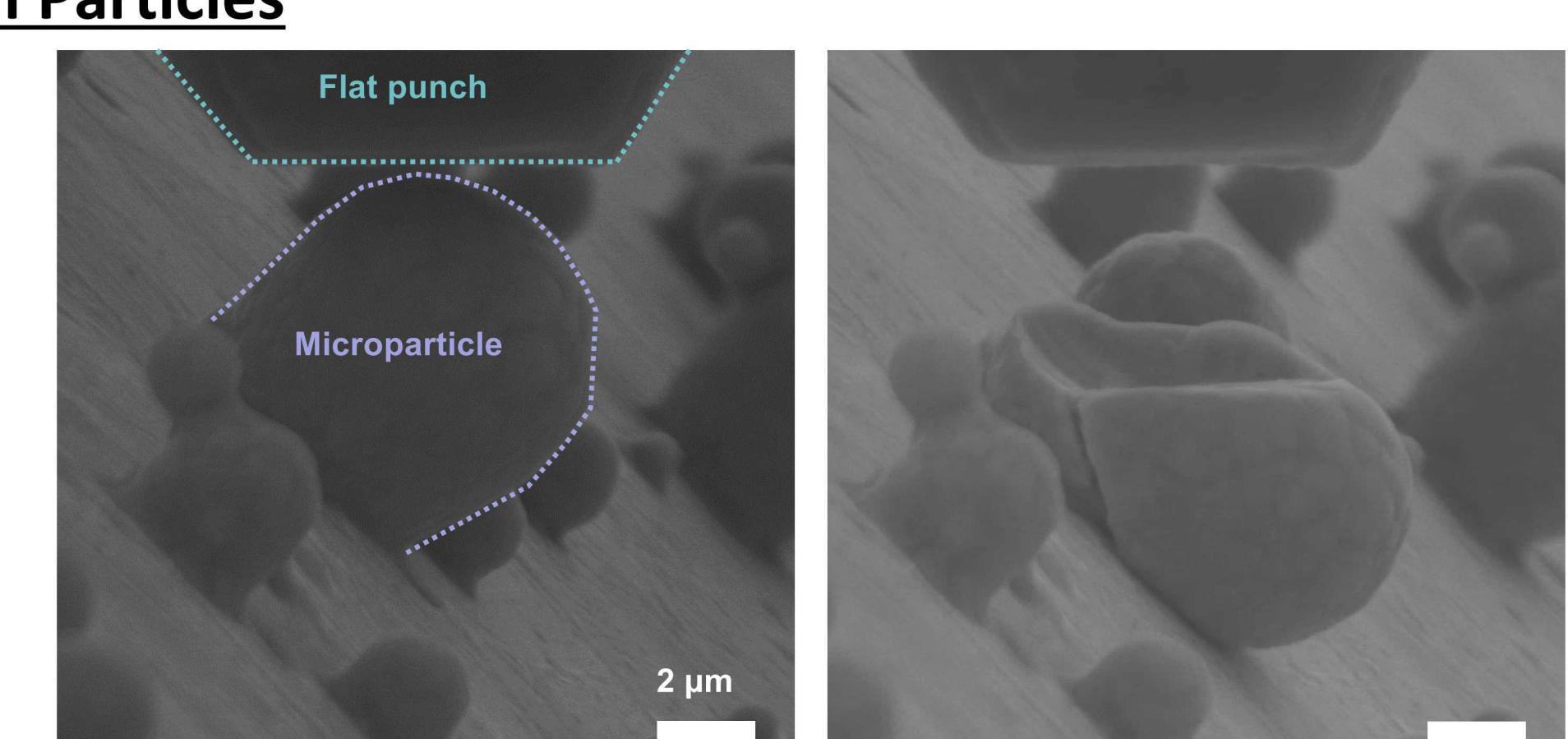


Figure 8: Before (left) and after (right) images of Kovar microparticle compression tests

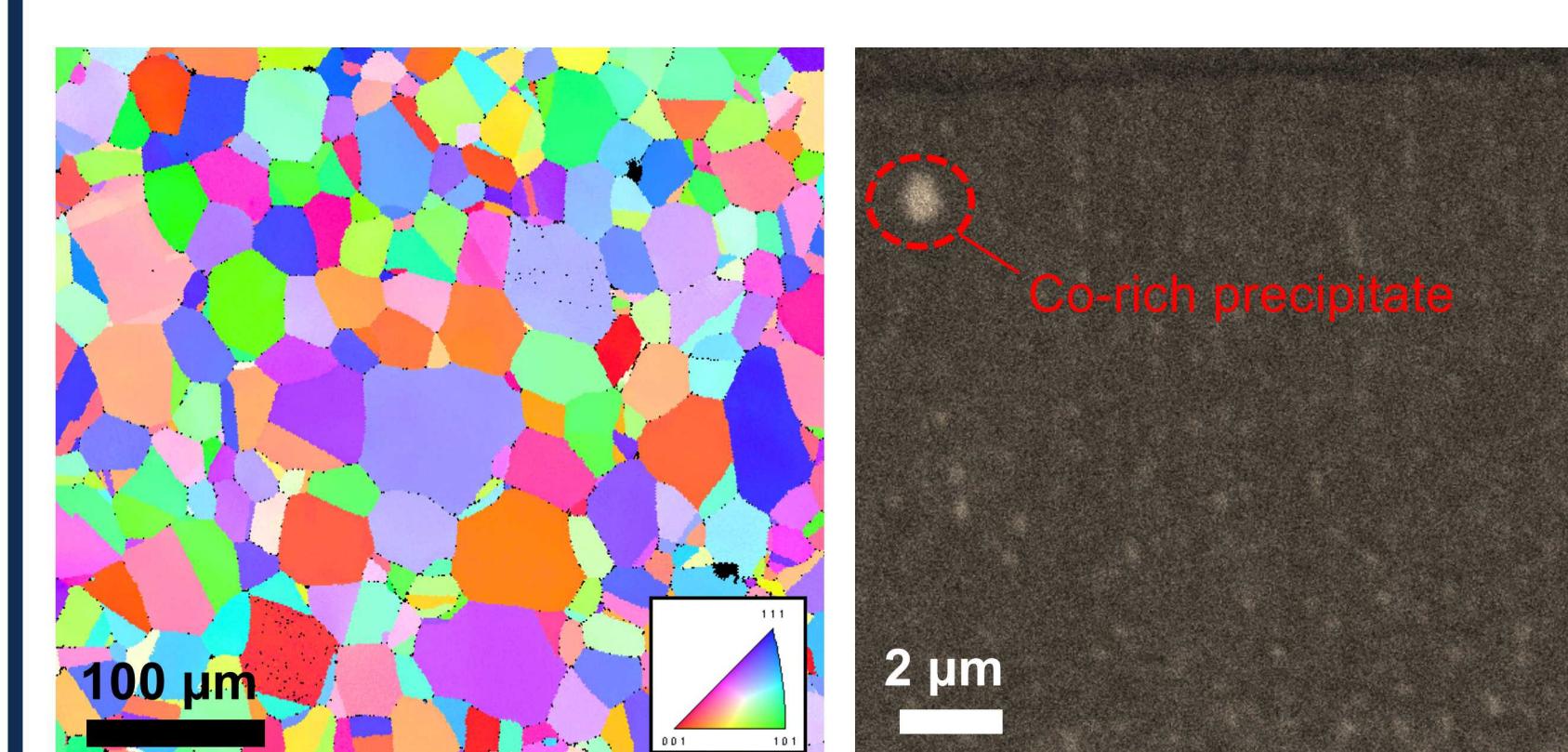


Figure 9: (left) EBSD inverse pole figure (IPF) of annealed Ni sample, acquired at 3000 indexed patterns/second. (right) EDS map showing Co-rich precipitates in a sputtered Inconel 725 alloy

## Initial Results (cont'd)

### Pt Fatigue testing

- High-cycle fatigue crack growth in a FIB-notched nanocrystalline Pt tensile specimen using custom piezo fatigue stage

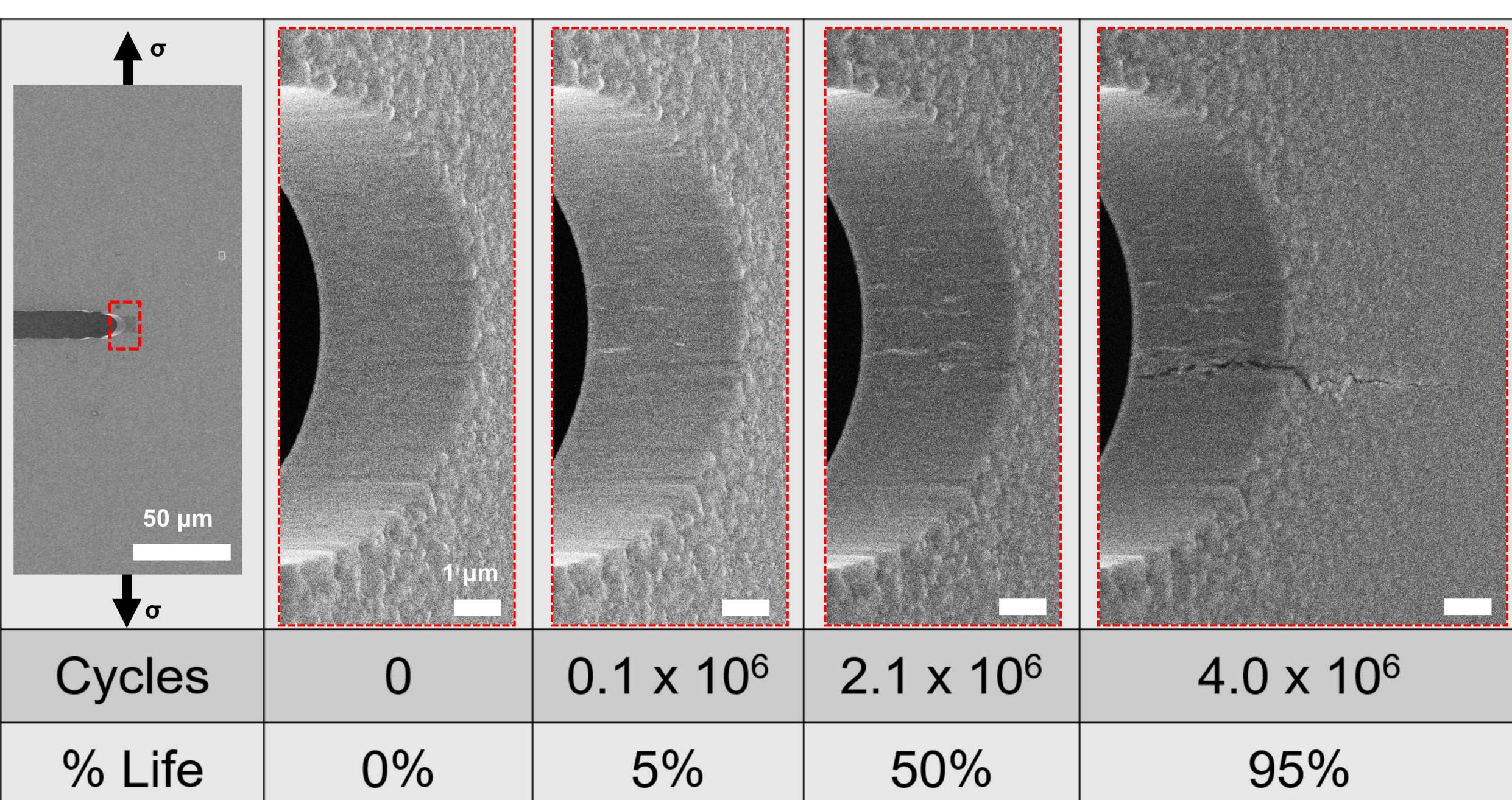


Figure 10: Automated image acquisition at notch for  $4.2 \times 10^6$  cycle fatigue test to failure

## Future Work

### He-Implantation & Pseudo Triple Beam Experiments

- Potential for simultaneous heavy ion irradiation with D<sub>2</sub>+He ion implantation

### Irradiation Creep & Fatigue

- Allows for insight into synergistic effects of irradiation, stress, and temperature in nuclear environments

### Tungsten Fuzz in Fusion Reactor Diverters

- Monitor evolution of tungsten-based material surfaces under low-energy, light-ion irradiation at high temperatures

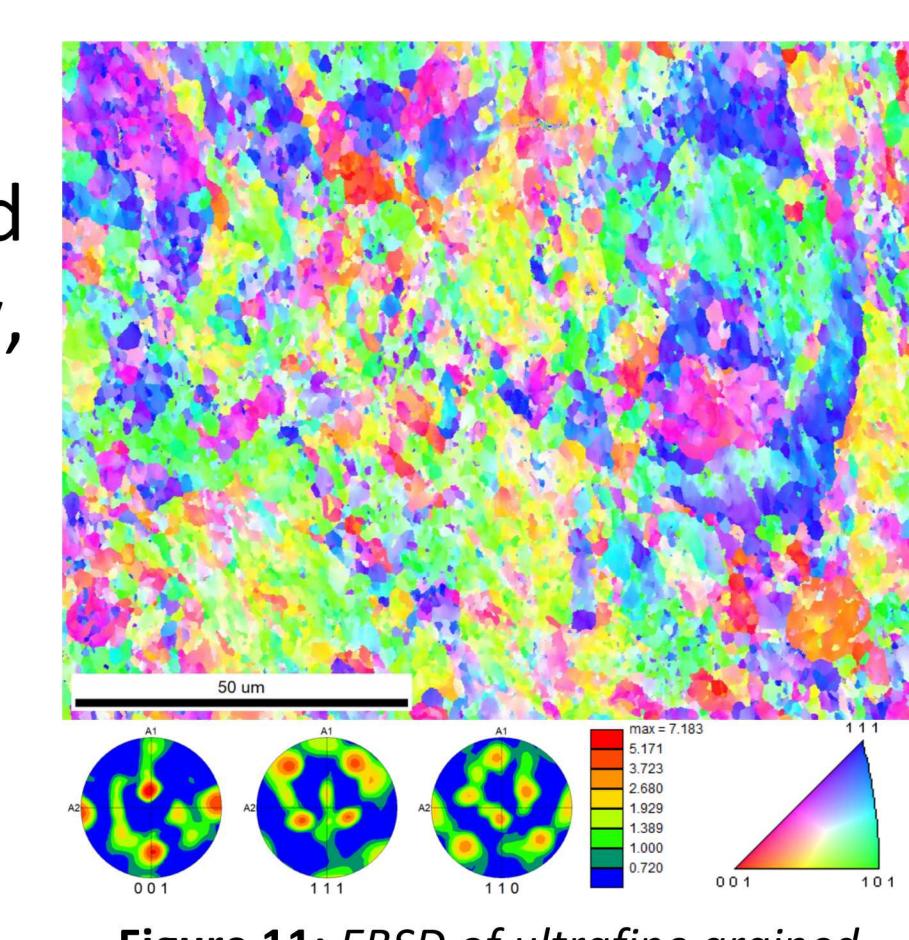


Figure 11: EBSD of ultrafine grained Tungsten with high indexing rate (3000/s), and high confidence rating

### Various User Experiments

- The I<sup>3</sup>SEM and I<sup>3</sup>TEM facilities are available to external users through both the Nuclear Science User Facilities and the Center for Integrated Nanotechnologies.

### Acknowledgements

This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

### References

- [1] K. Hattar, D. C. Bufford, & D. L. Buller. Nuclear Instruments and Methods in Physics Research, Section B: Beam Interactions with Materials and Atoms, 338 (2014), p. 56–65.
- [2] T.A. Furnish, et al. Journal of Materials Research, 31 (2016), p. 740–752.
- [3] T.A. Furnish, et al. Journal of Materials Science, 52 (2017), p. 46–59.