In-situ Observation and Multiscale Three-dimensional Characterization Ta Tensile Samples

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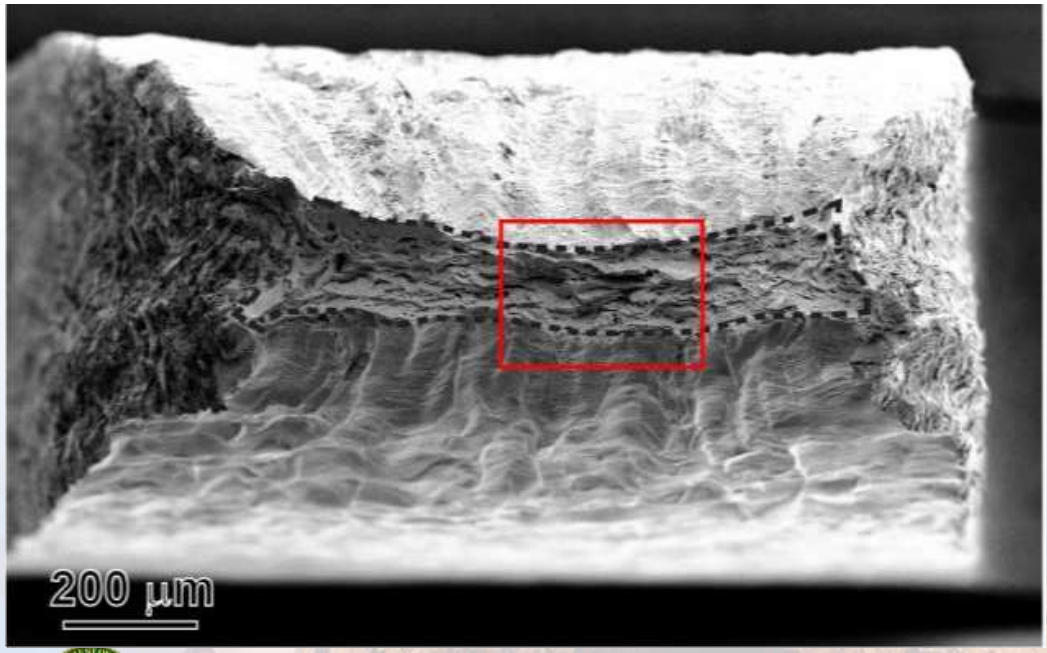

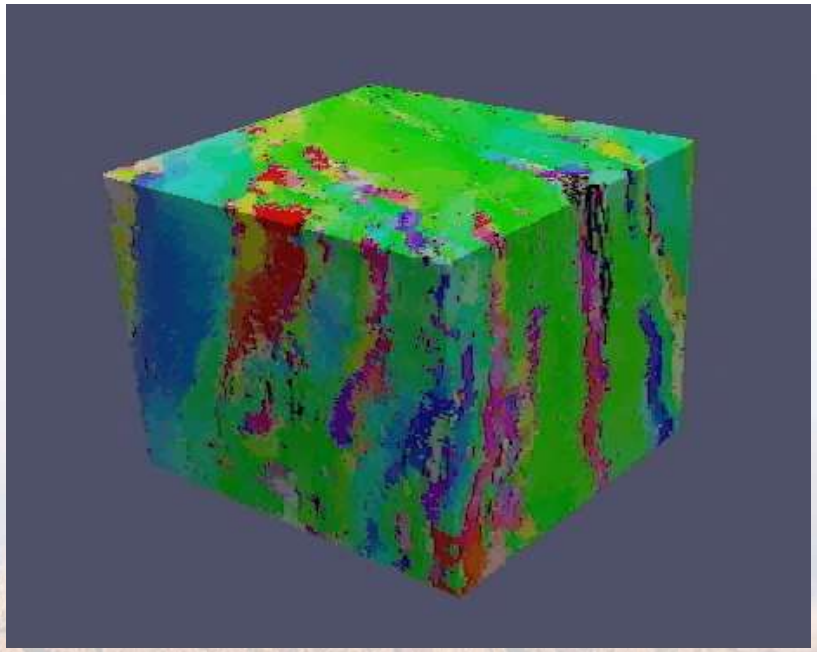

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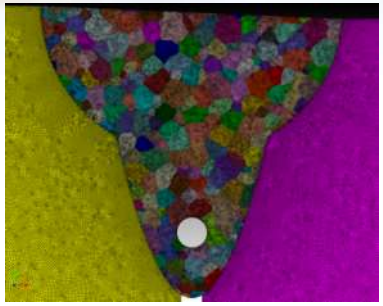
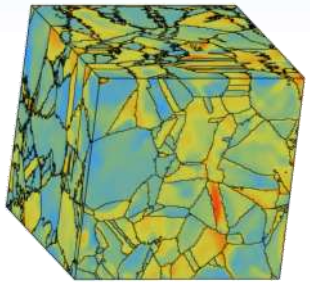
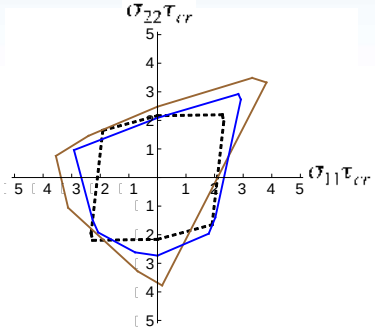
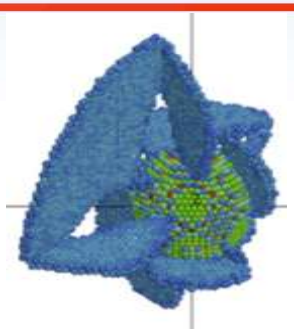
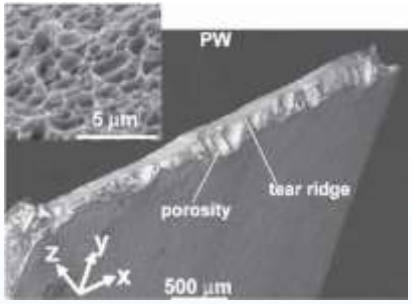
Understanding Stochastic Deformation and Failure Requires Multiscale Experiments and Models

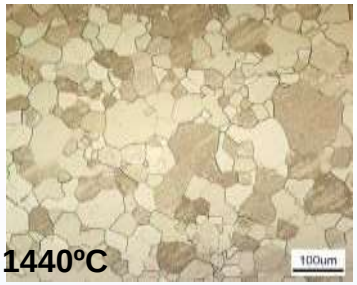


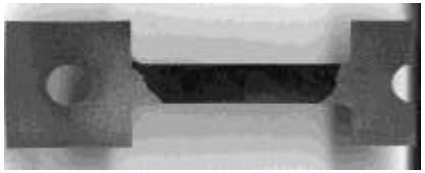


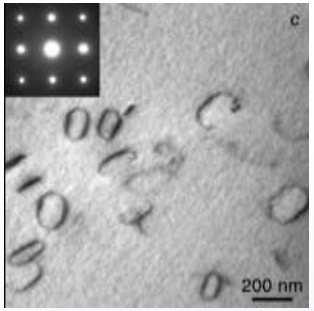












Material performance	Microstructural effects	Single crystal behavior	Atomic scale phenomena
10^0 m 10^6 s	10^{-3} m 10^3 s	10^{-6} m 10^0 s	10^{-9} m 10^{-9} s

Atoms-up: Develop physics-based models to provide scientific insight

Continuum-down: Augment engineering-scale models to provide customer value

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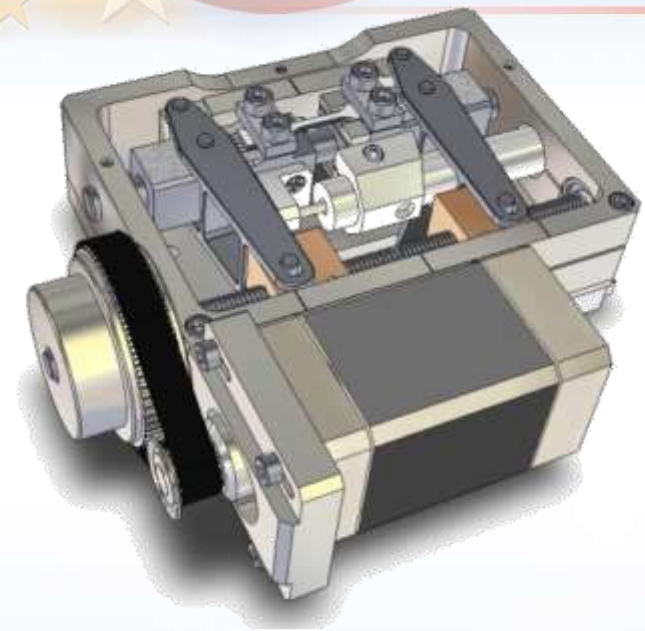
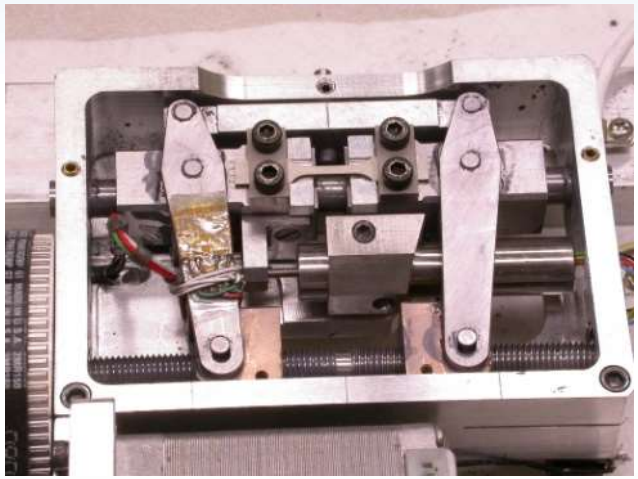
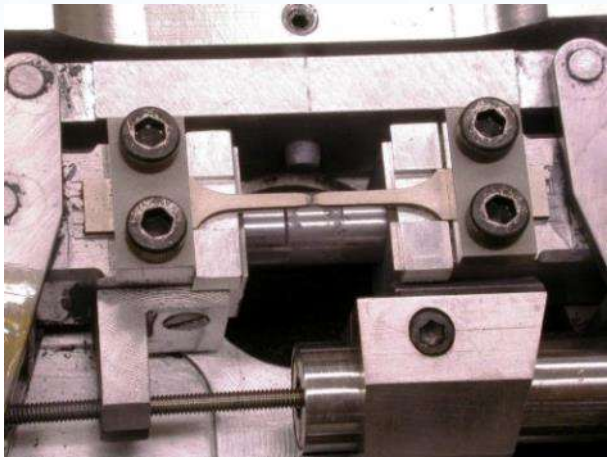
Custom Compact In-situ SEM / EBSD Tensile Stage



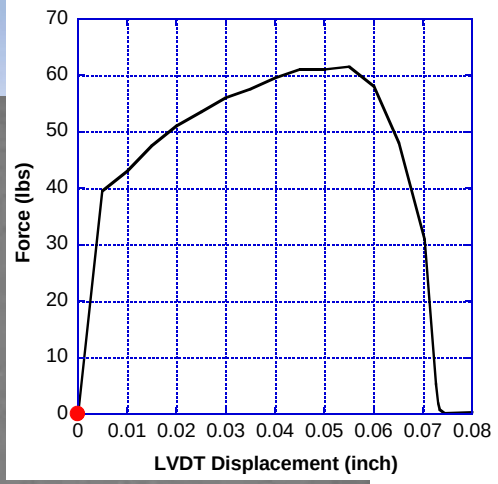




- Tensile stage with 2 kN capacity permits reasonable cross-section tensile bars (1 x 2 mm)
- Compact size (~deck of cards) enables high-angle tilting for EBSD measurements.
- Speckling of tensile bars (~300 nm copper particles) allows simultaneous DIC measurements.
- Symmetric loading from both grips keeps the electrons on the center of the column.
- Suitable for SEM or back-reflection x-ray

This capability permits real time characterization of the Ta sample during tensile deformation and failure

Observing Deformation in Tantalum

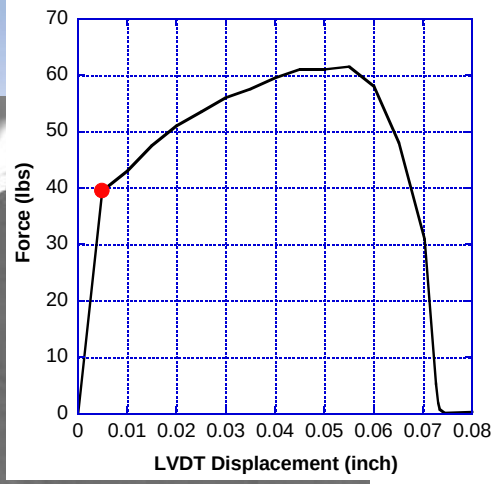


20 µm

EHT = 10.00 kV WD = 20.9 mm Signal A = SE2 File Name = Ta-N1_d-553mV_02.tif

atories

Observing Deformation in Tantalum



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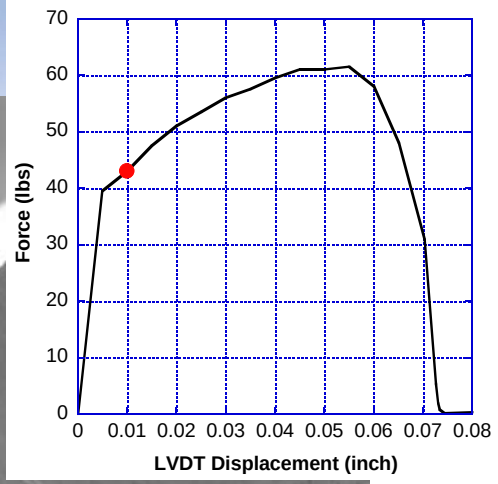
Observing Deformation in Tantalum



20 µm

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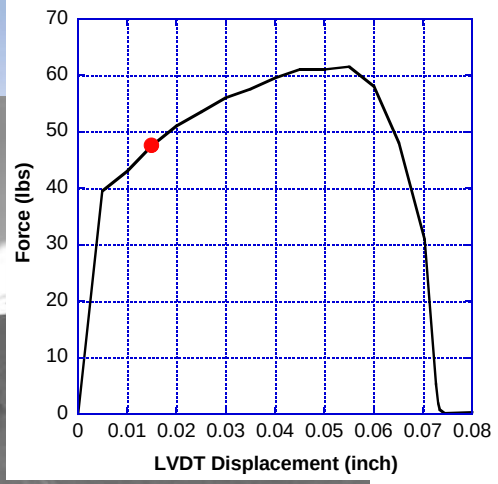
Observing Deformation in Tantalum



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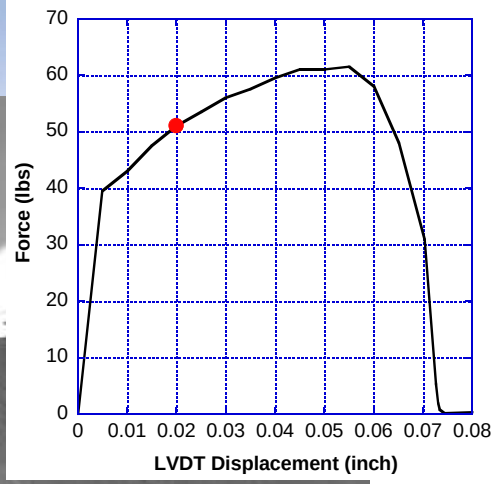

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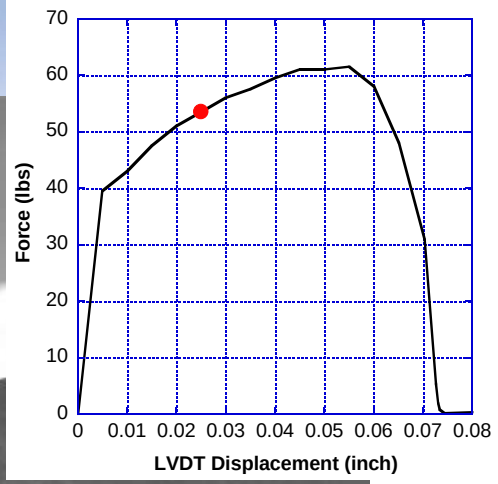

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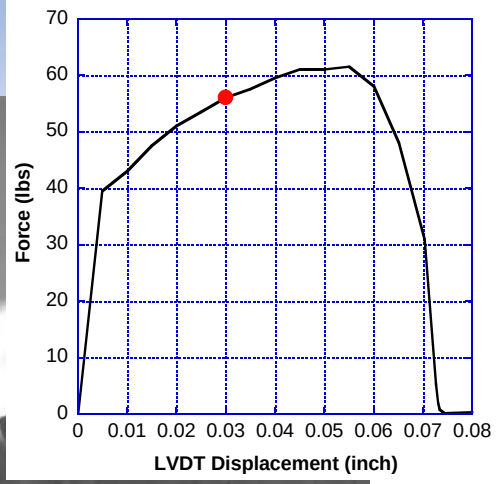

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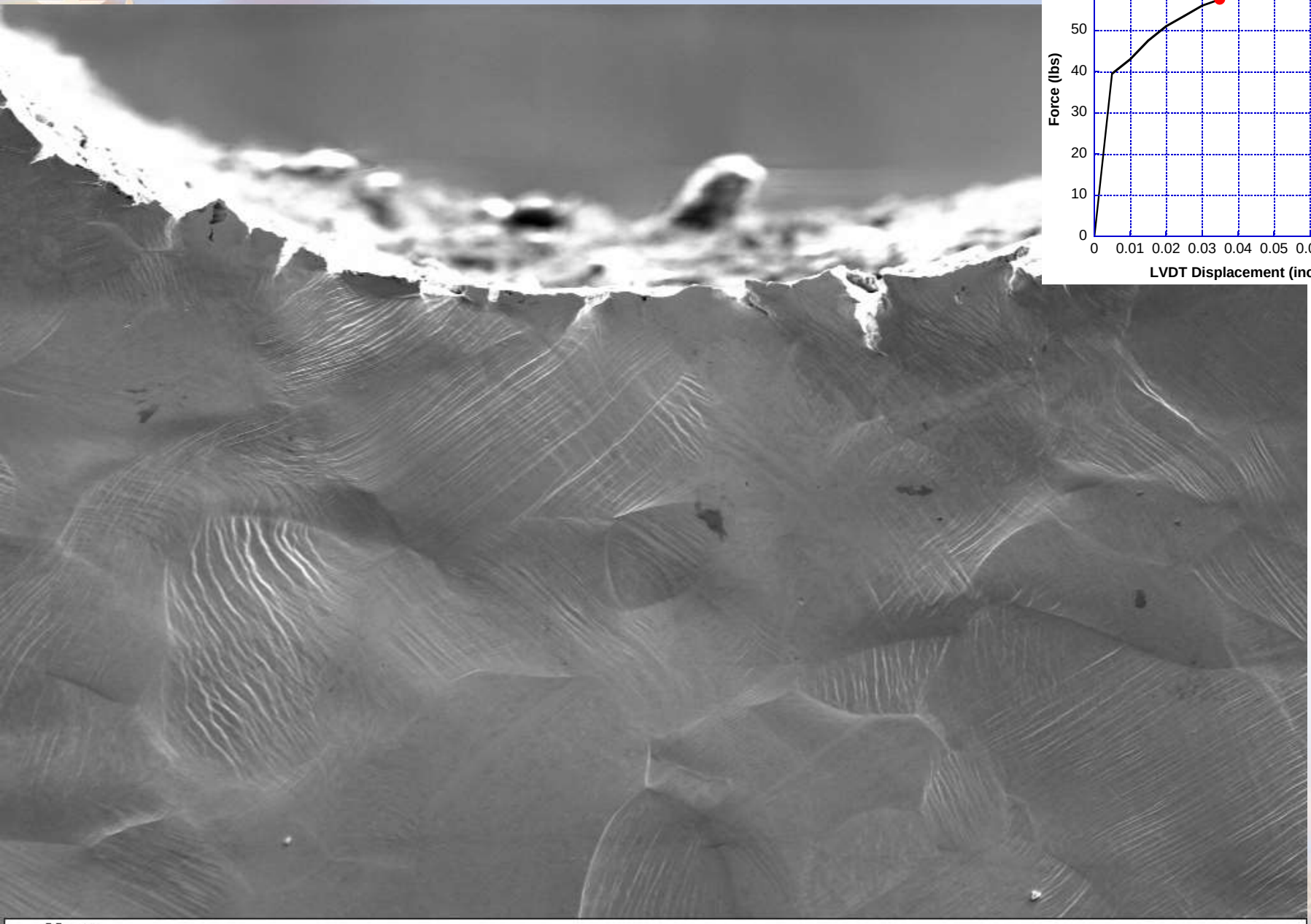

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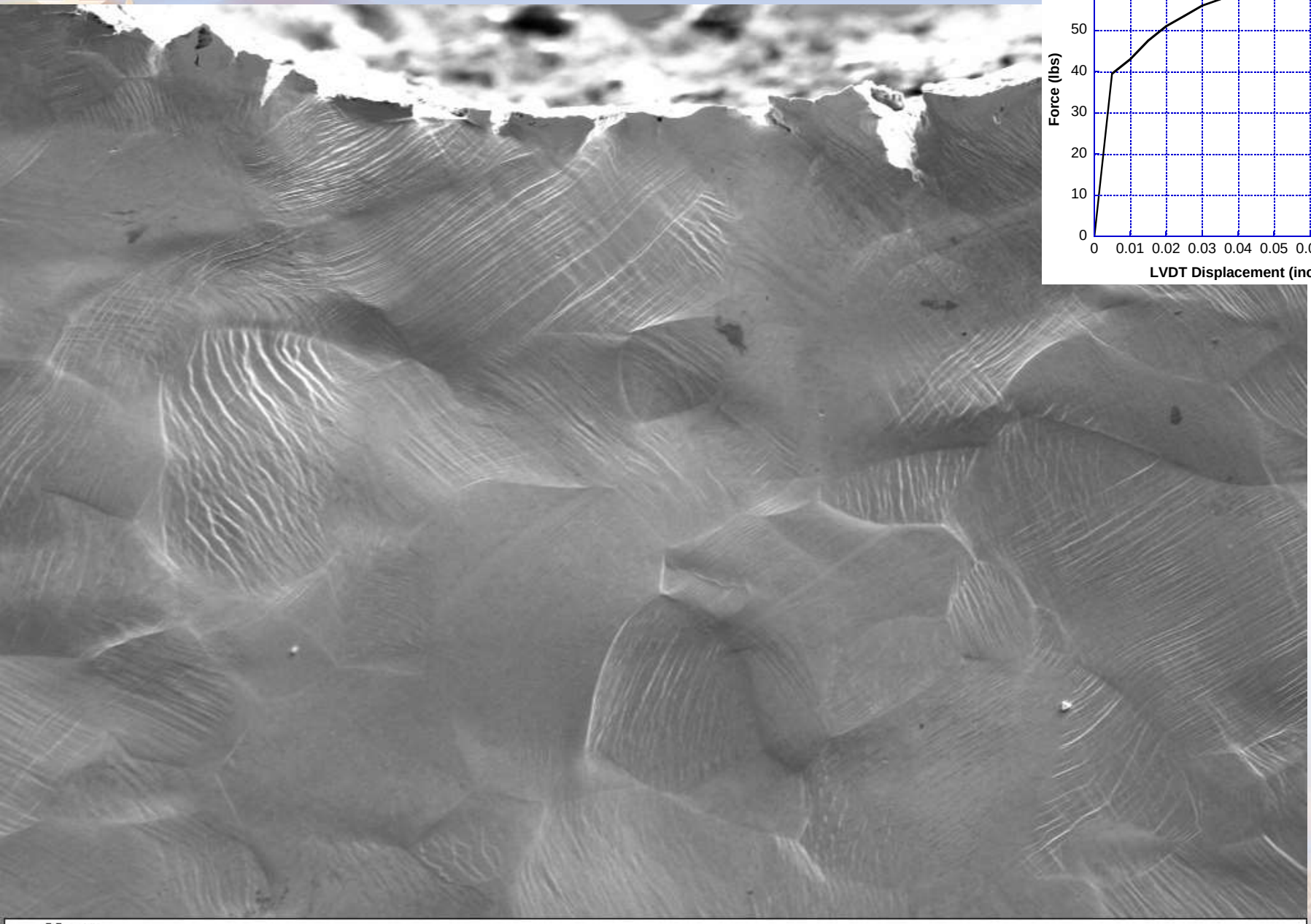

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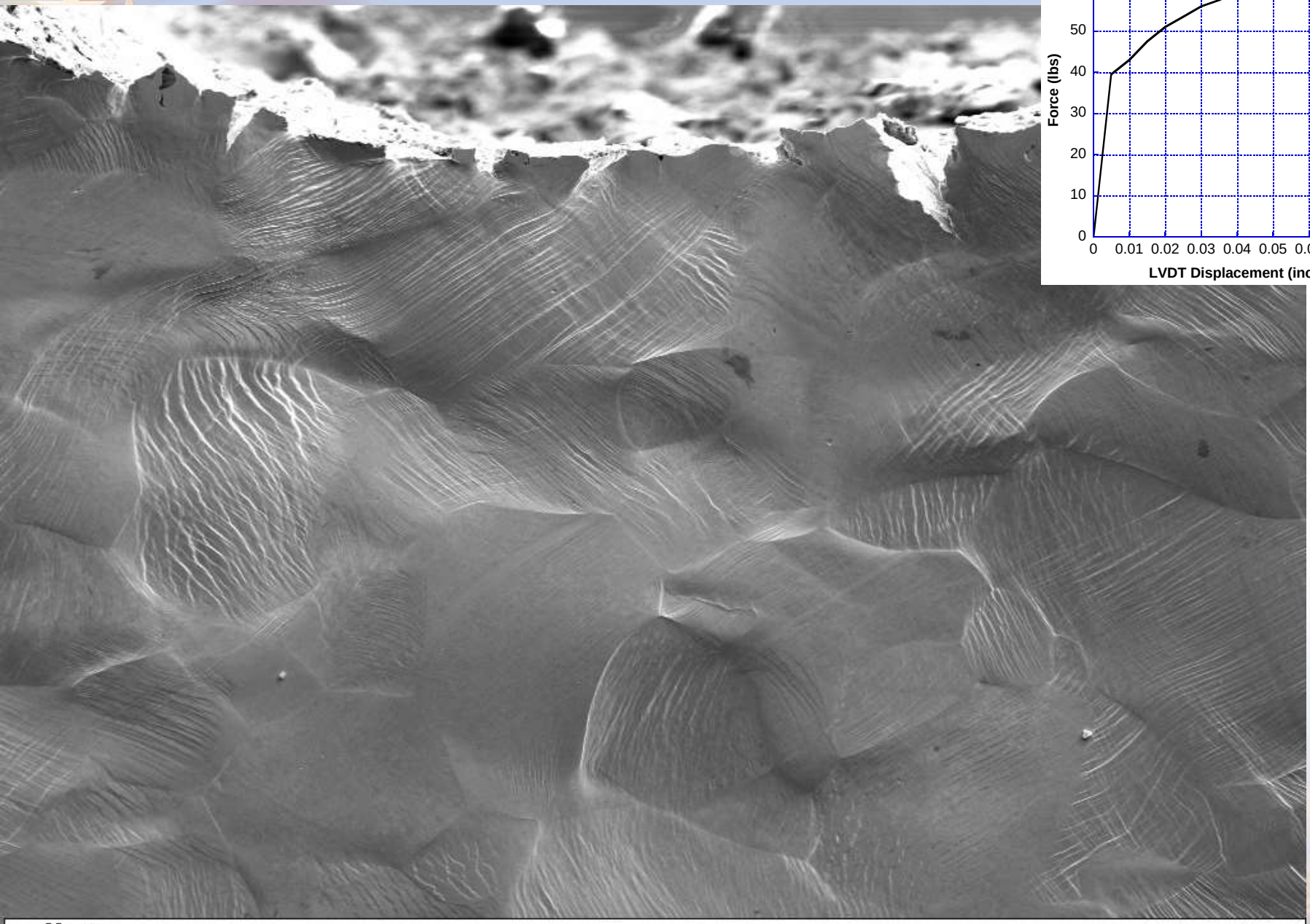

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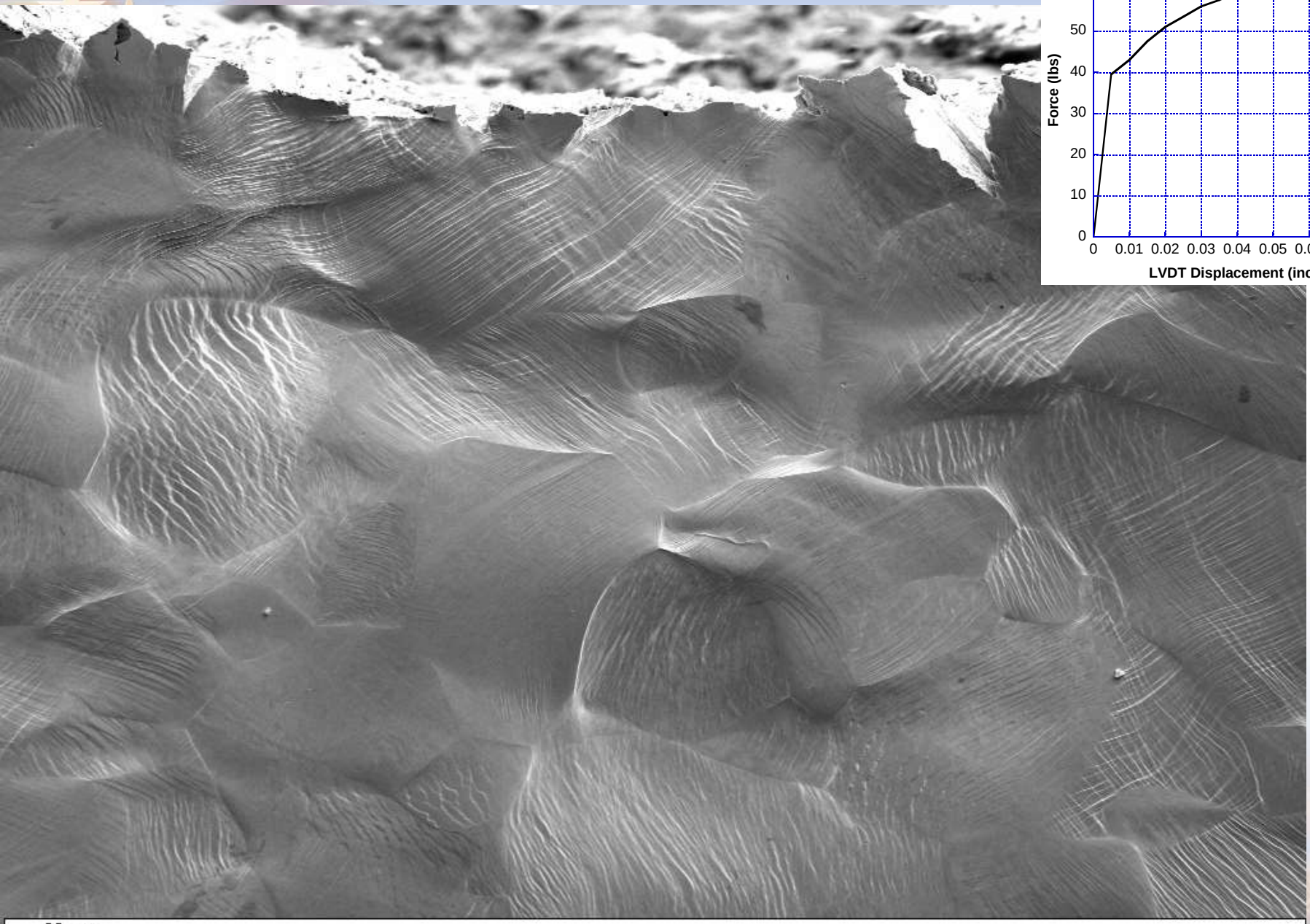

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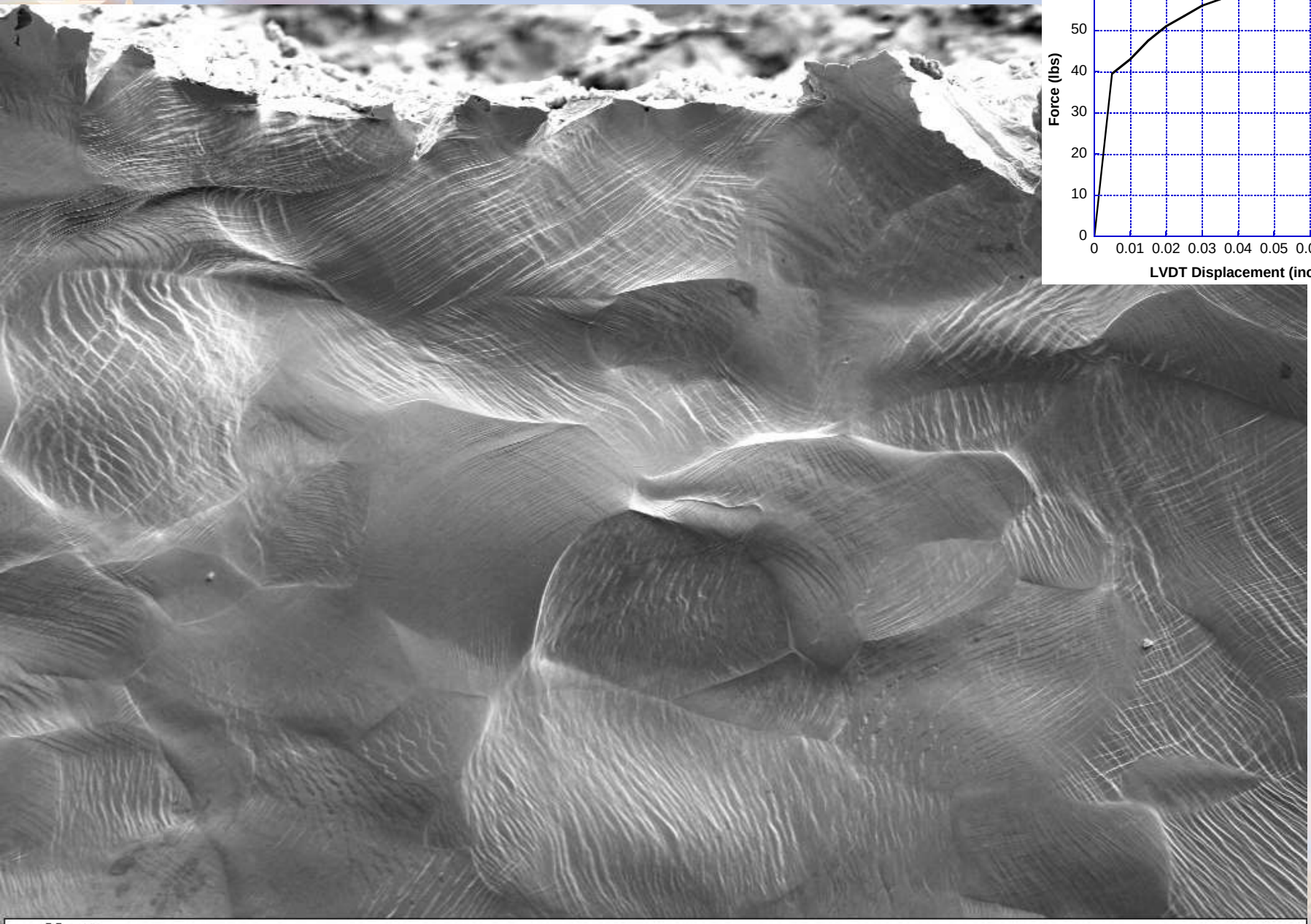

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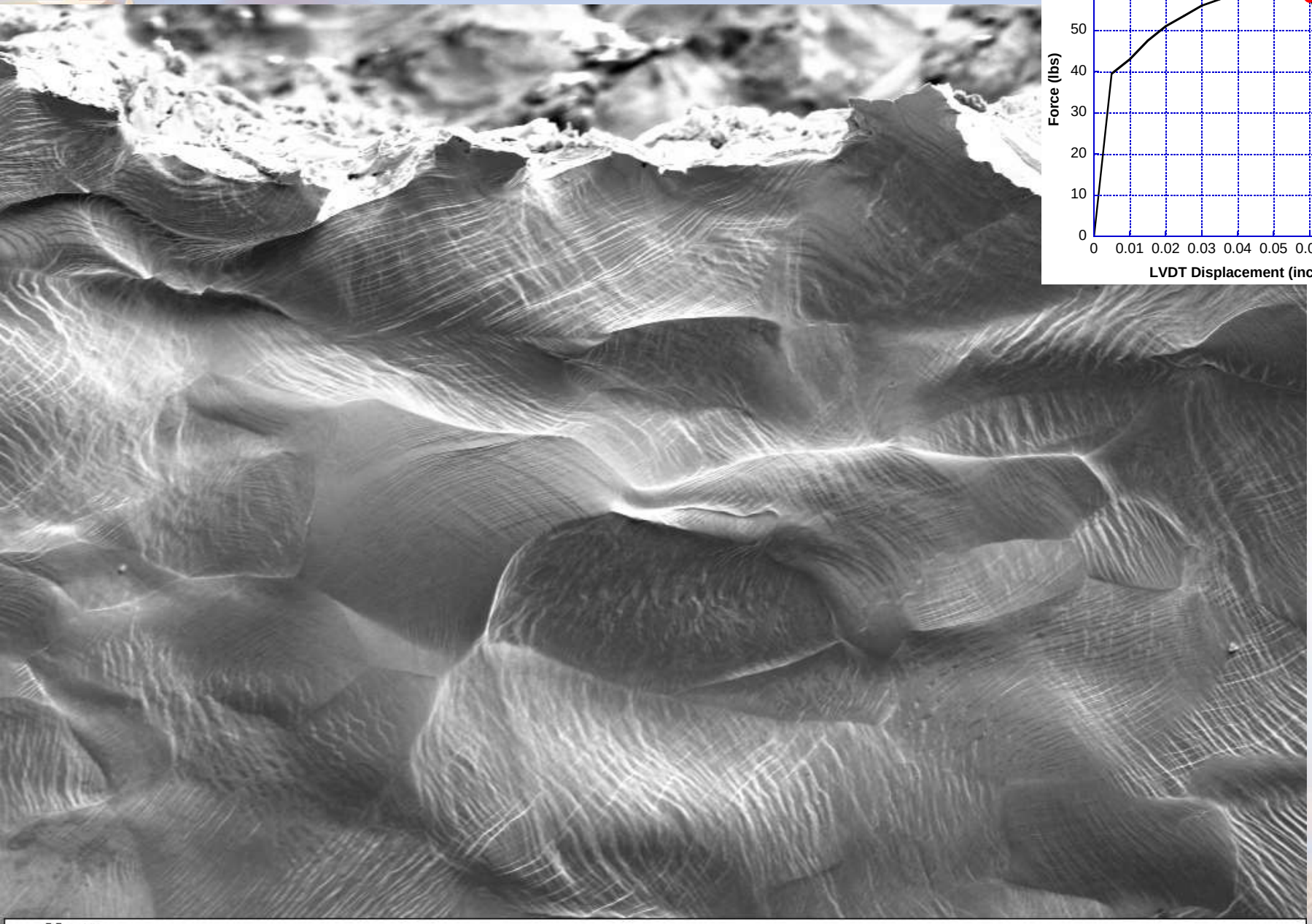

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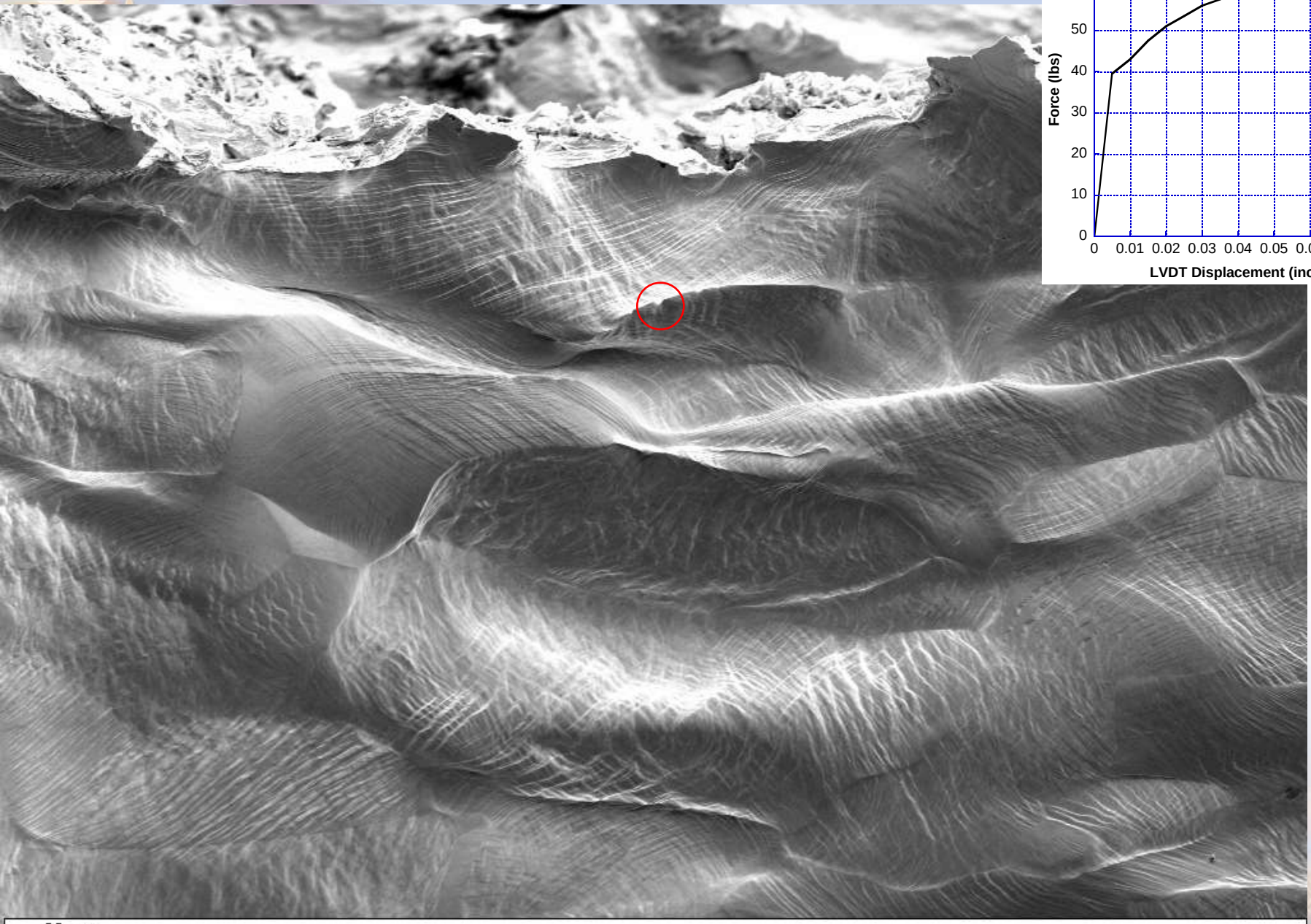

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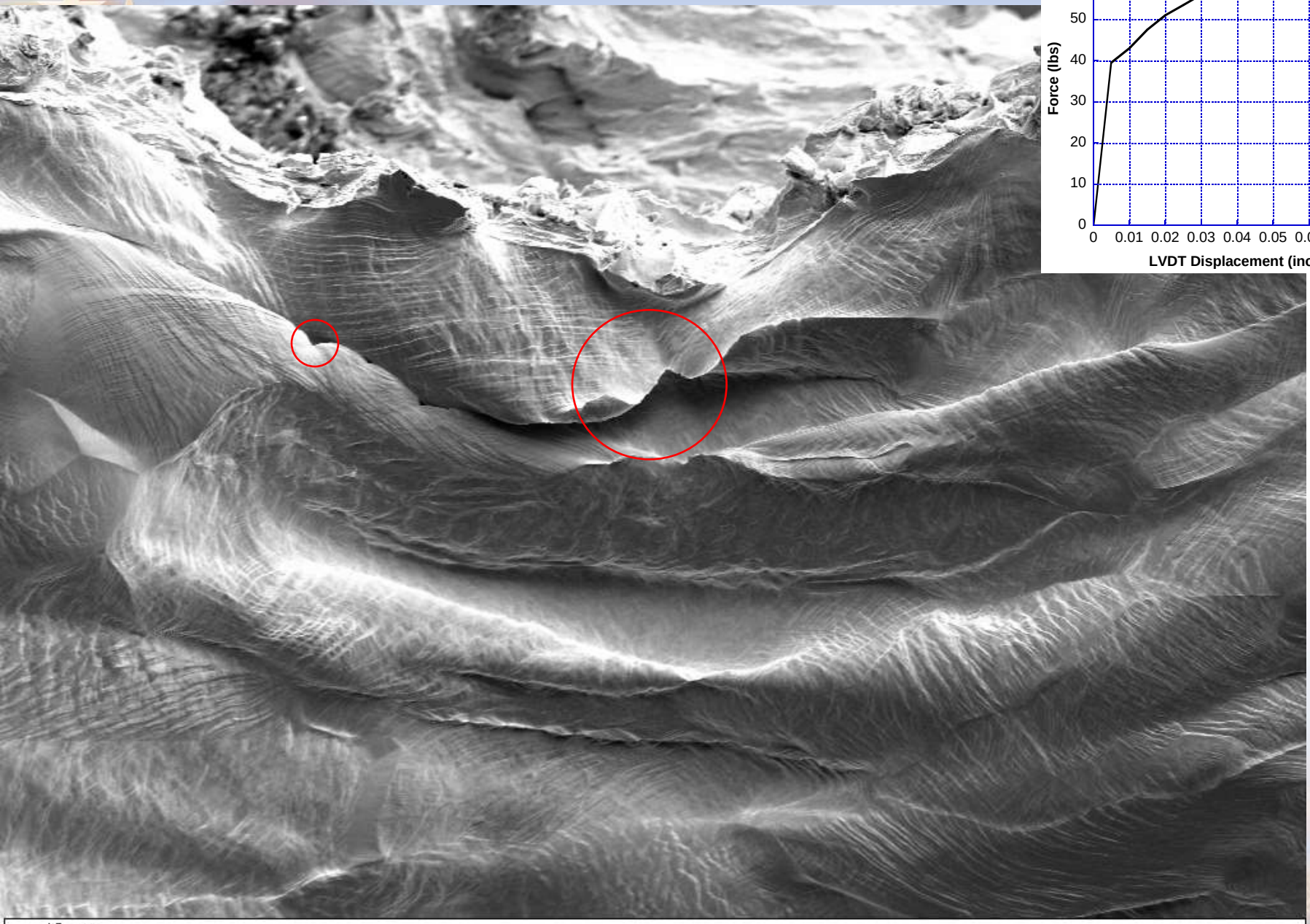

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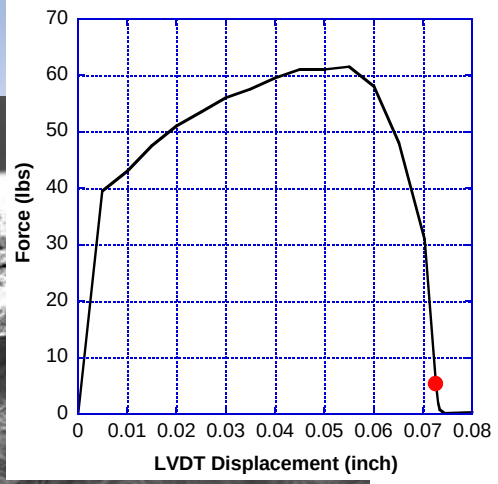

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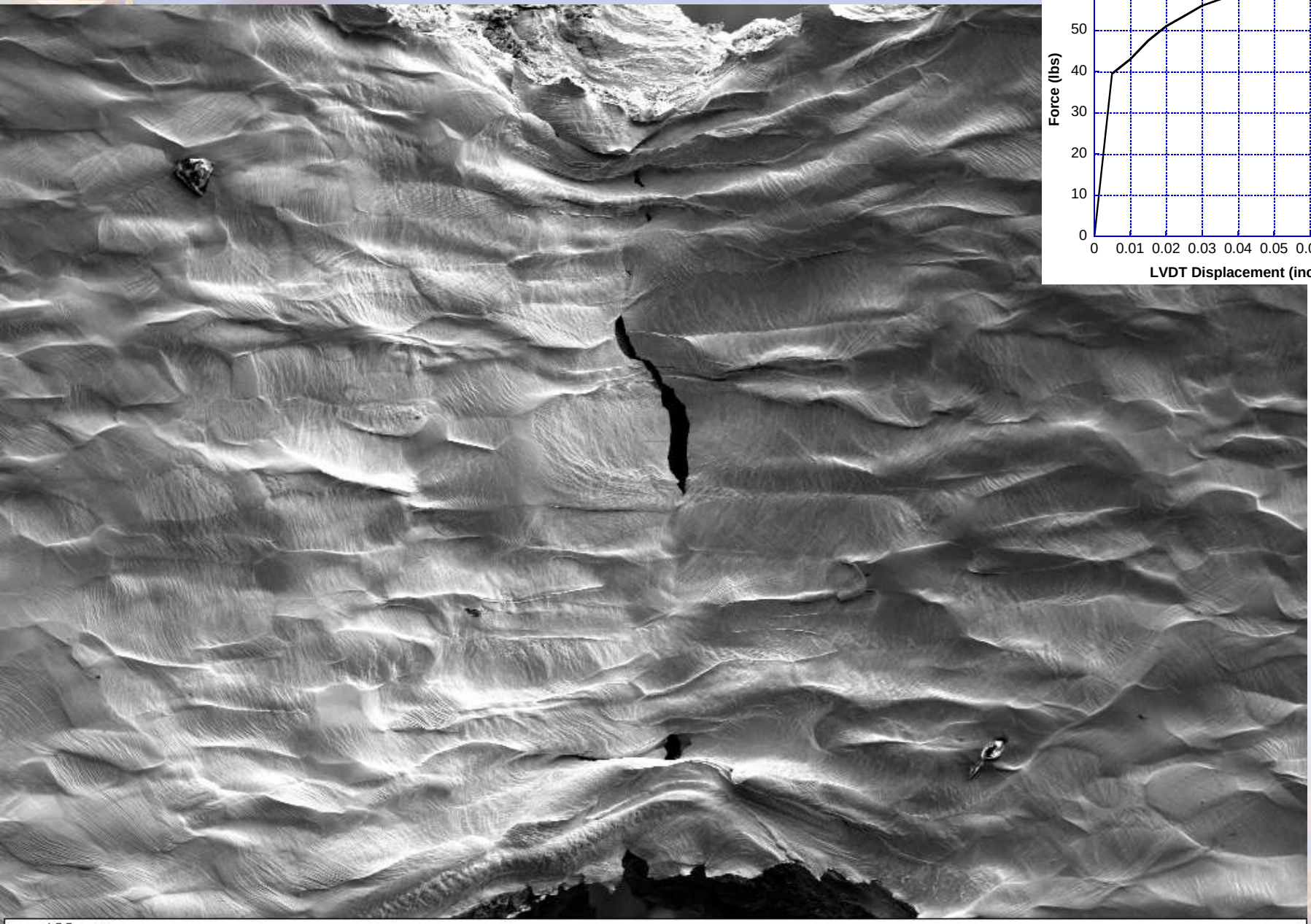

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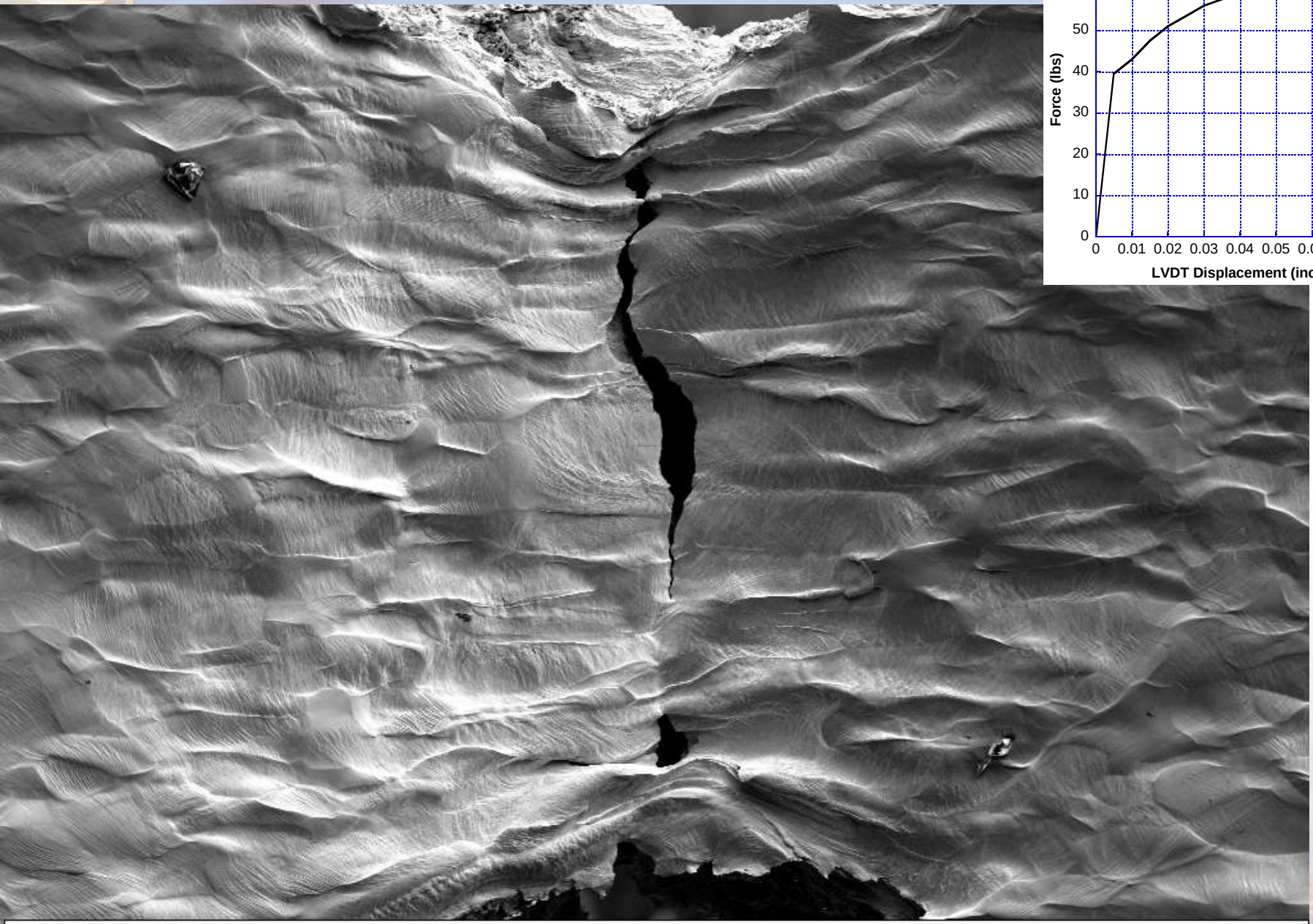

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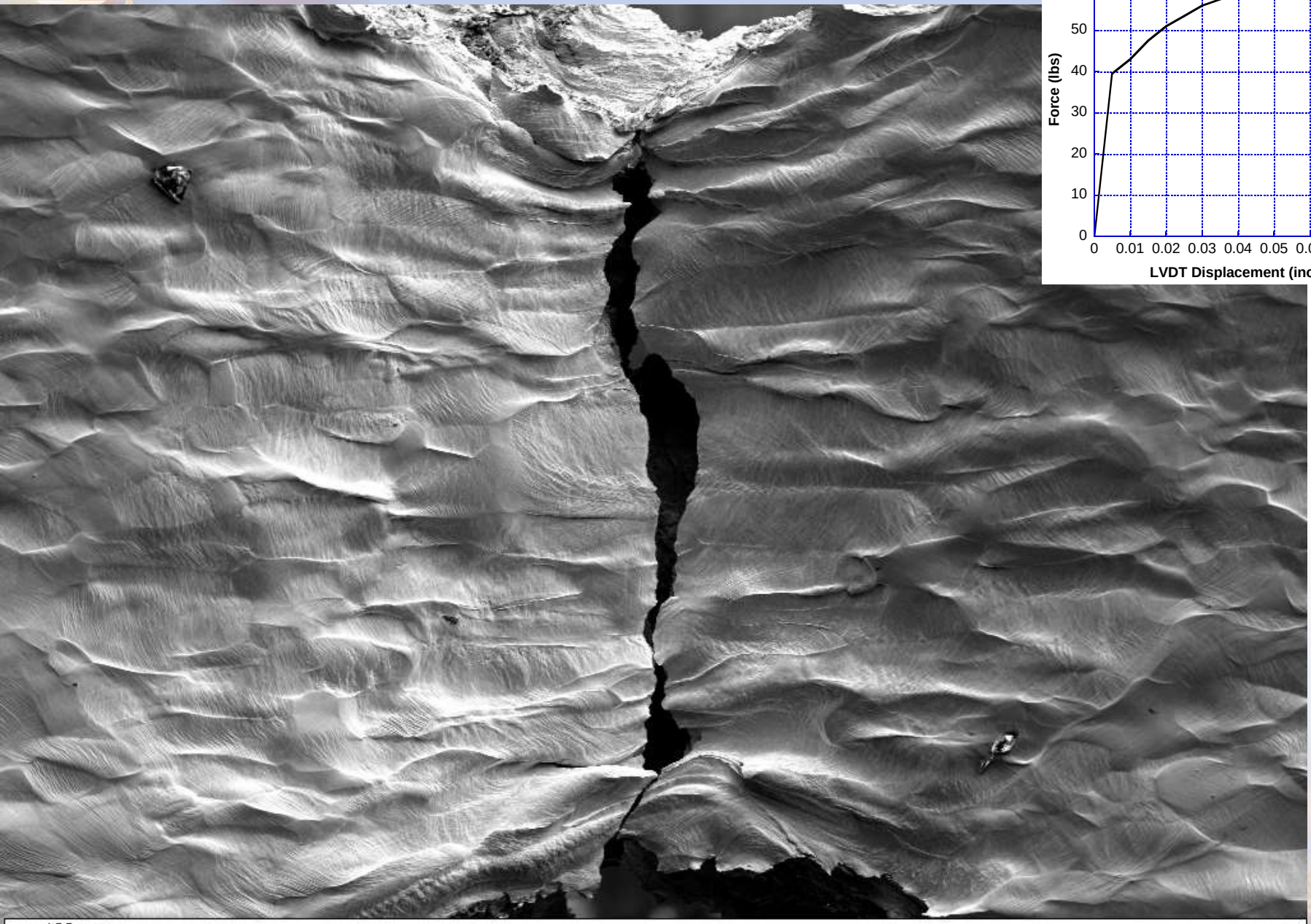

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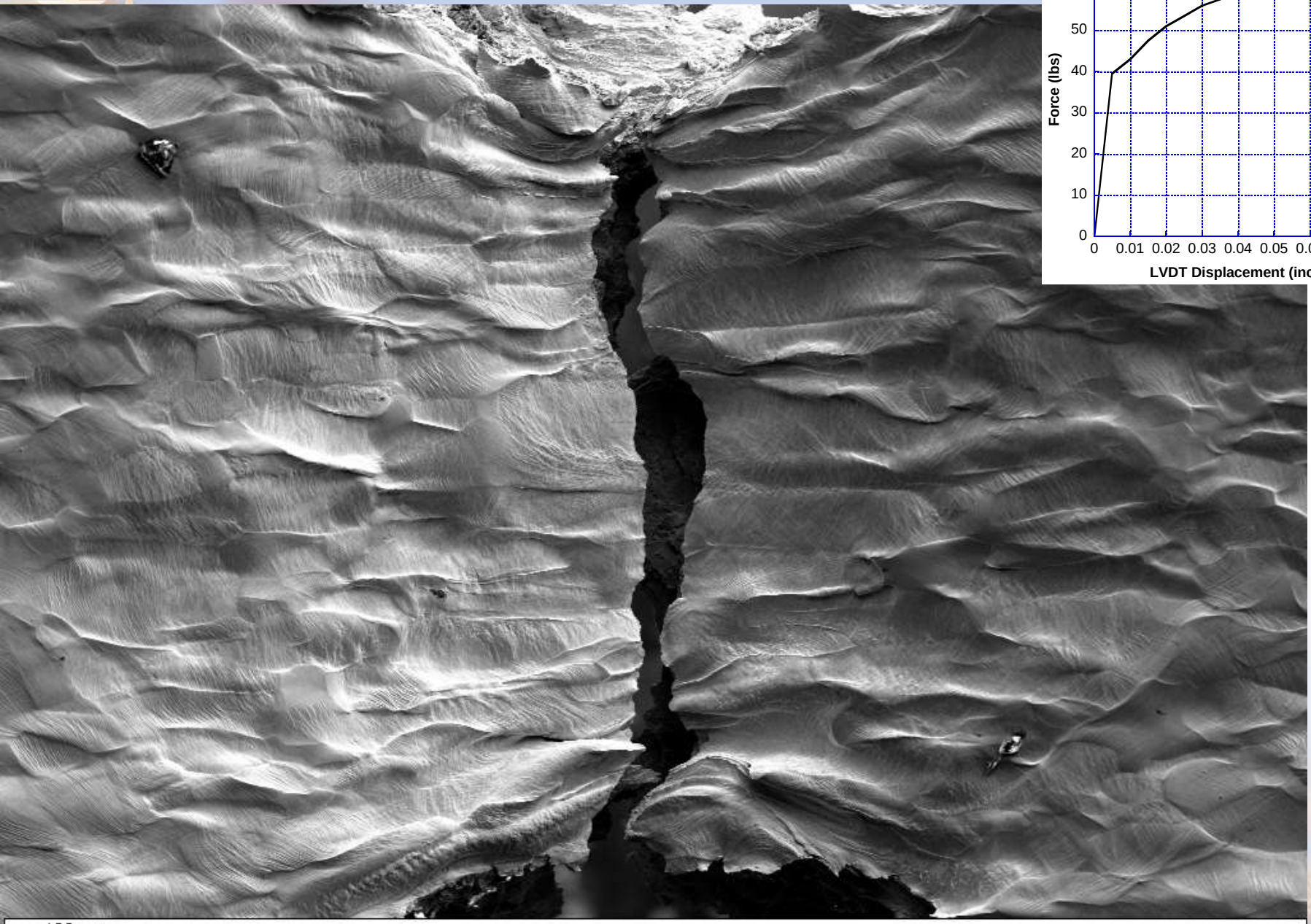

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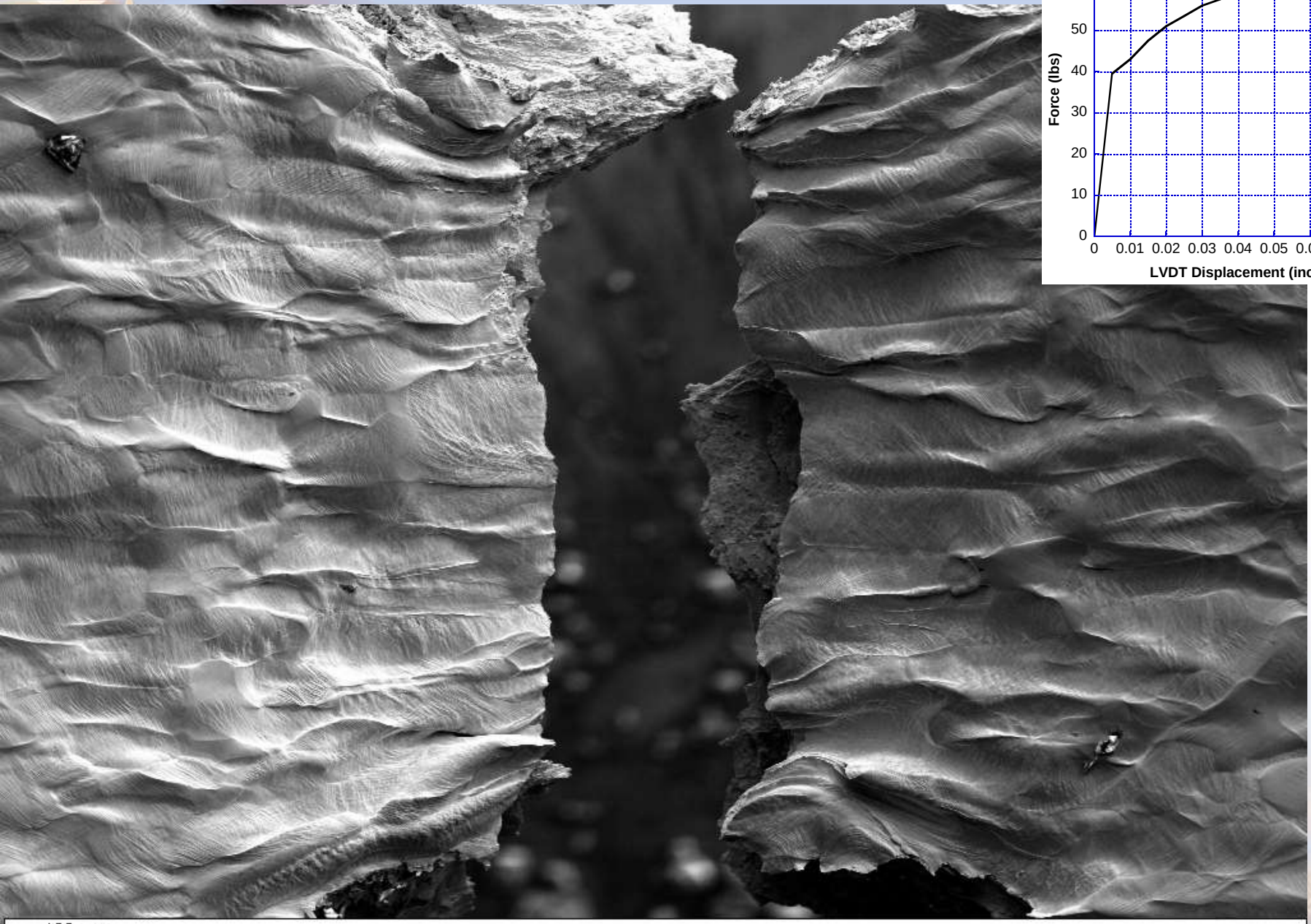

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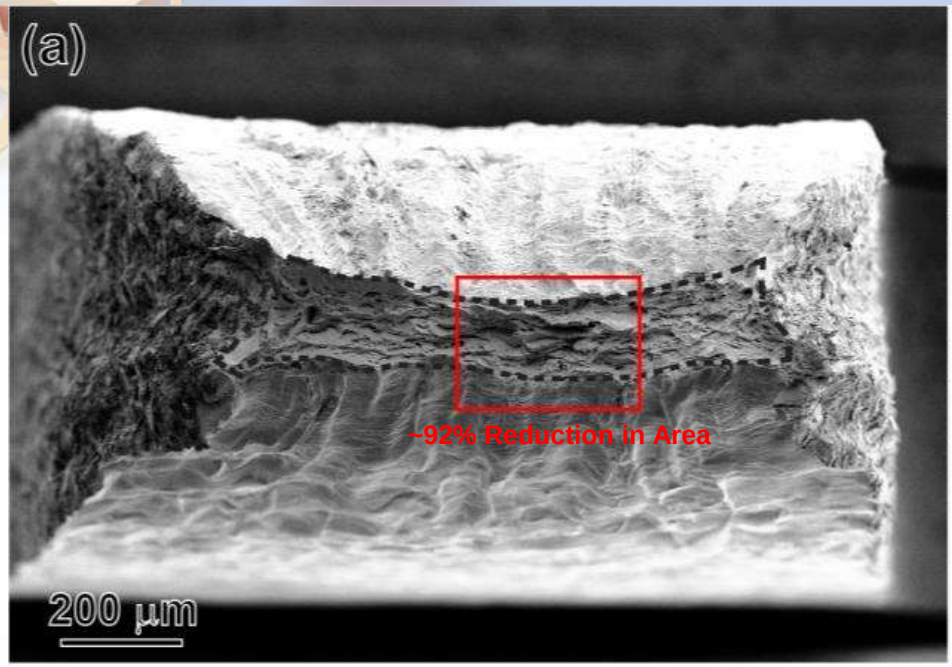
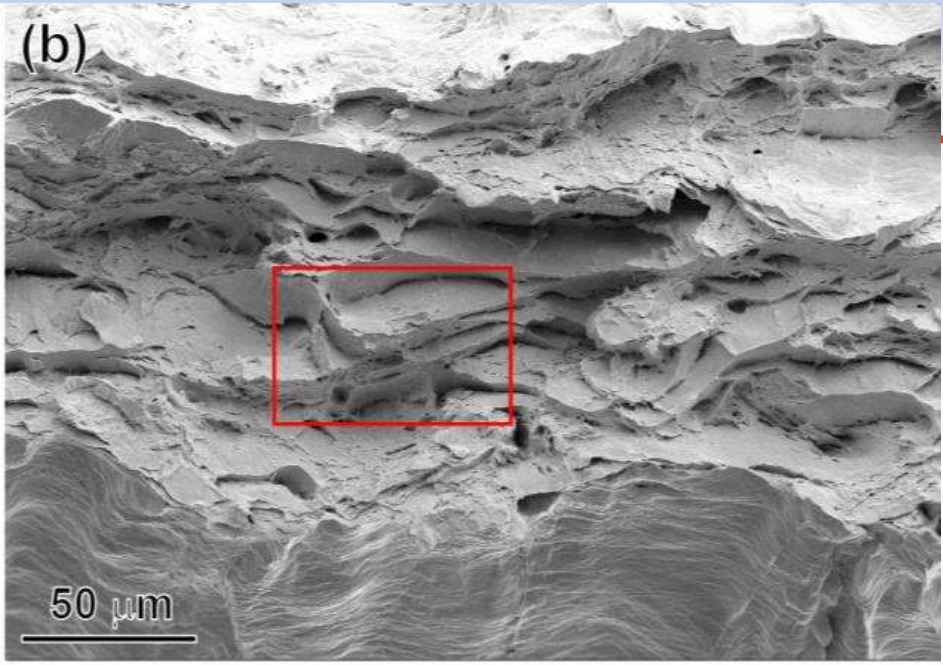
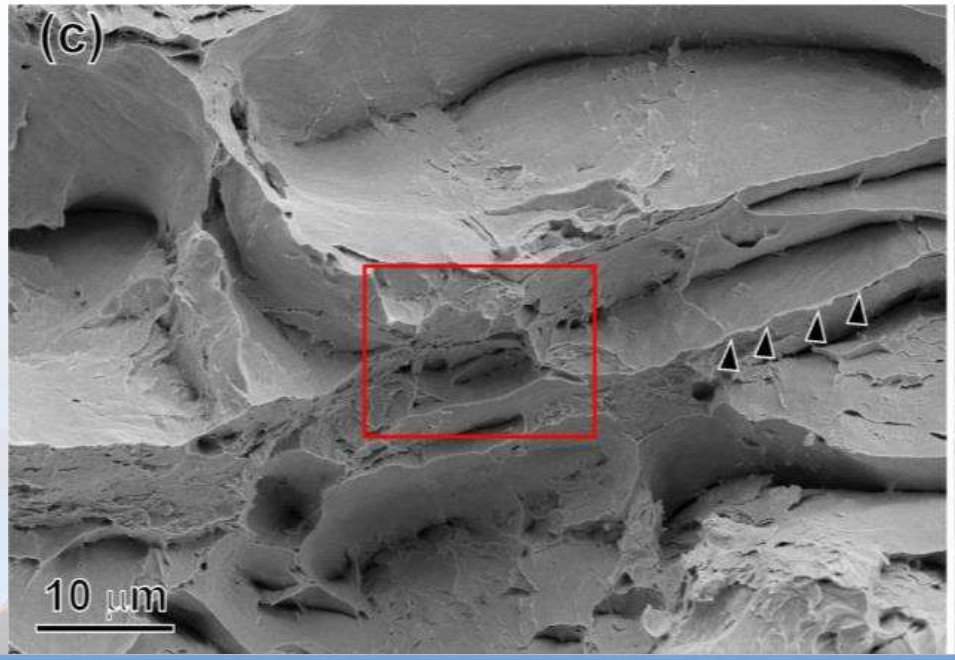
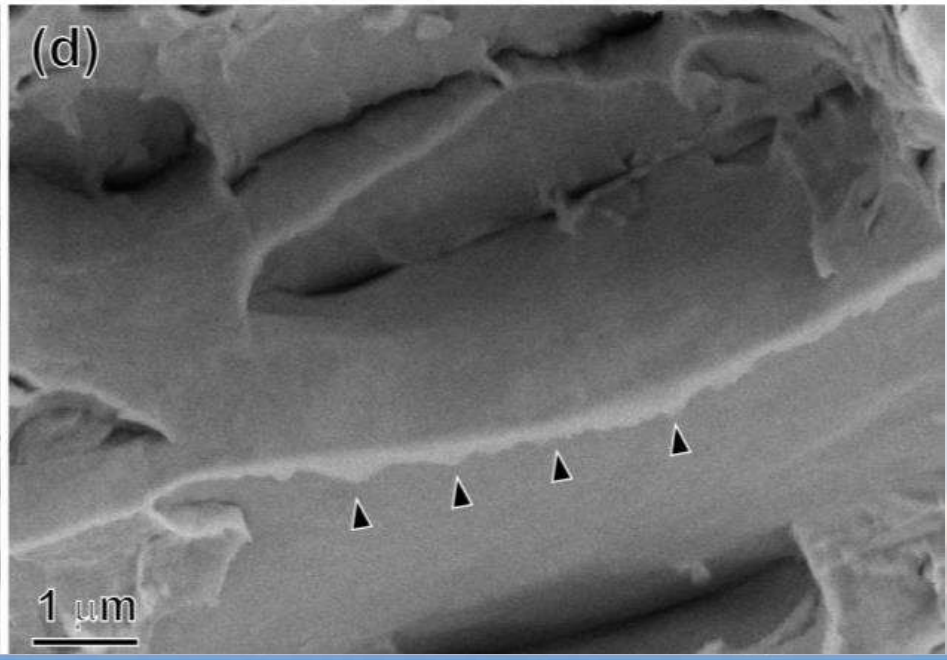
Lack of Classic Ductile Dimple Morphology

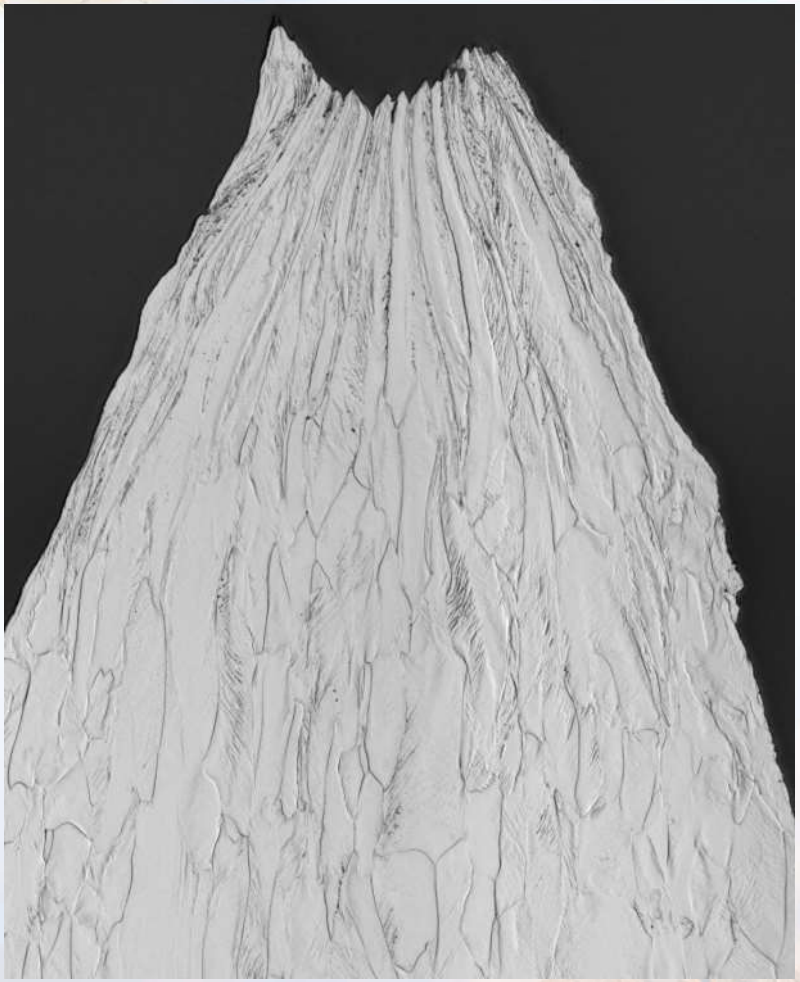


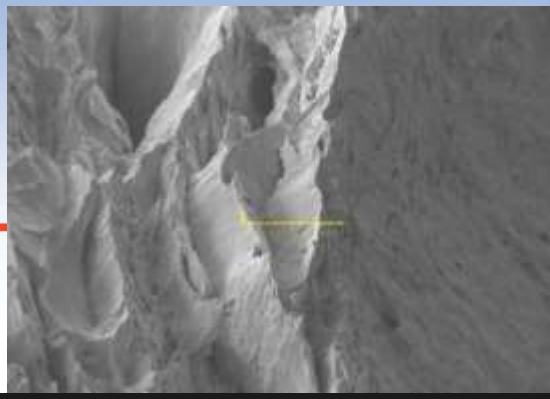


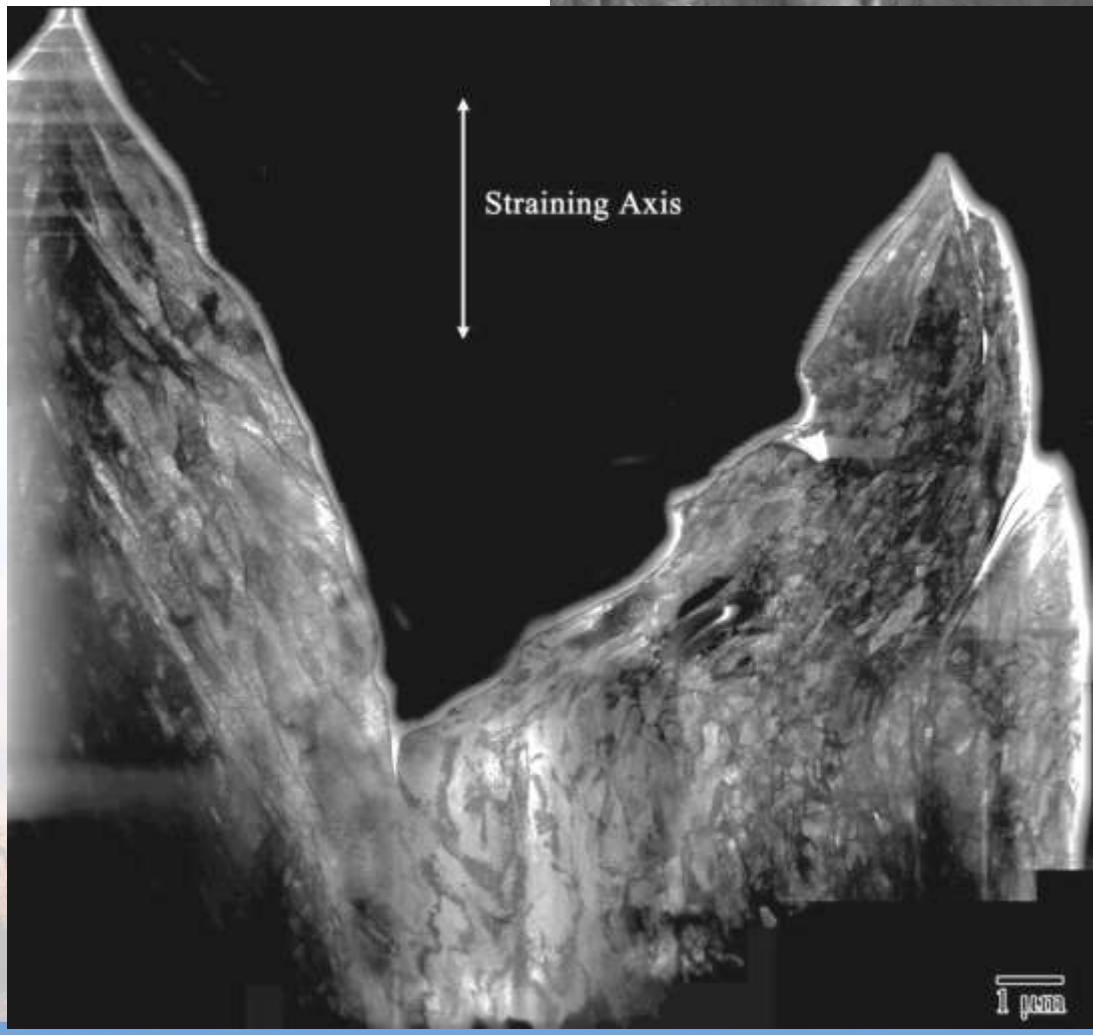




SEM and TEM Cross-section of Fractured Neck in Tantalum

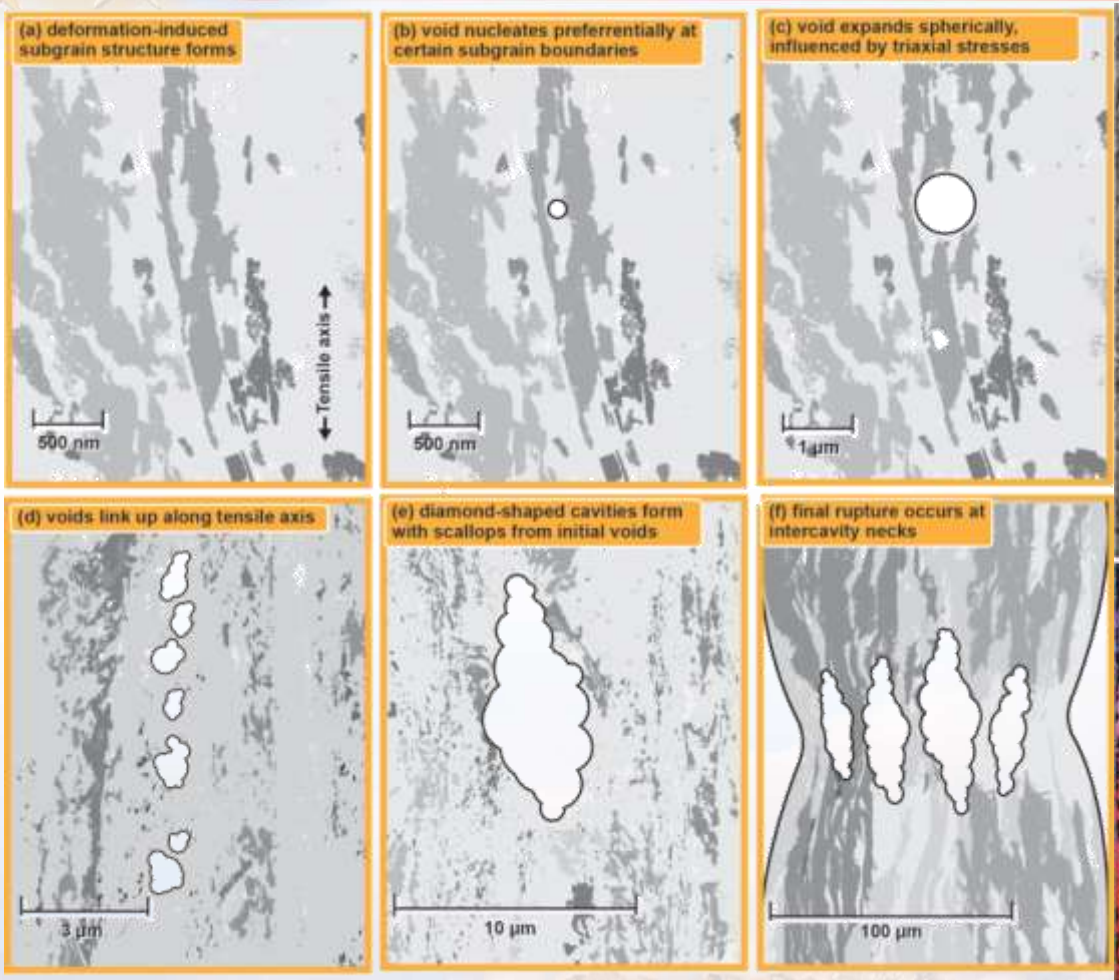


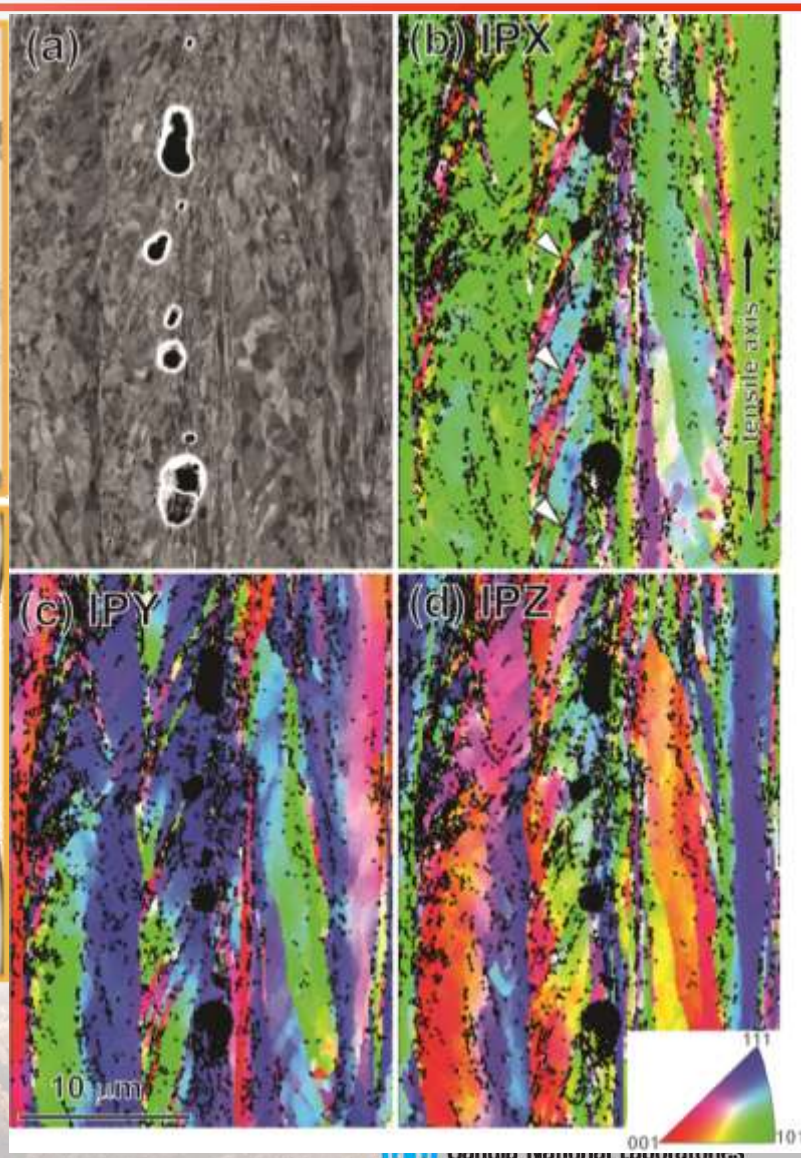




Cross-sectional characterization reveals a complex grain and sub-grain structure in the necked region

A Schematic of Damage Progression Leading to Catastrophic Fracture

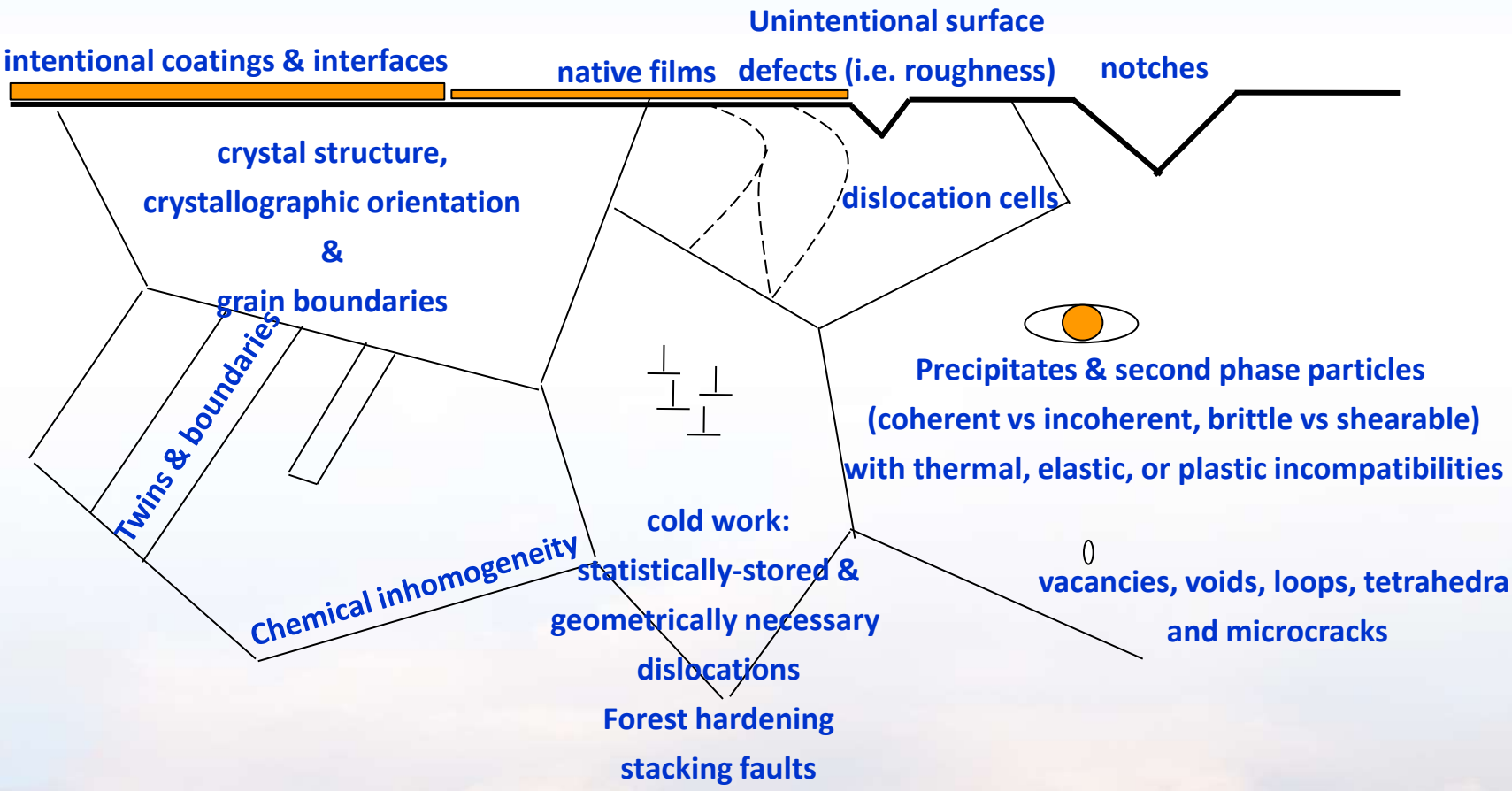




Thus far we've only provide surface information

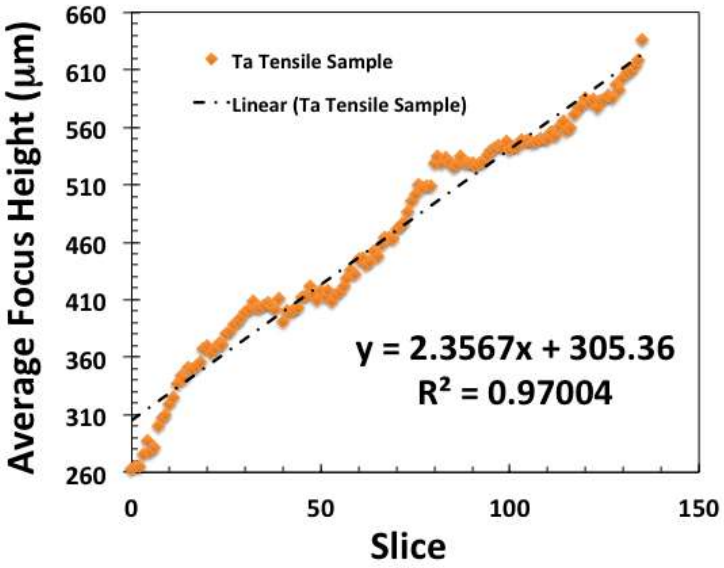
Can we gain any further insight with 3D characterization to confirm or refute the proposed schematics?

Real Materials Have Complex Features that Influence Failure



The relative importance of each of these factors varies from material to material

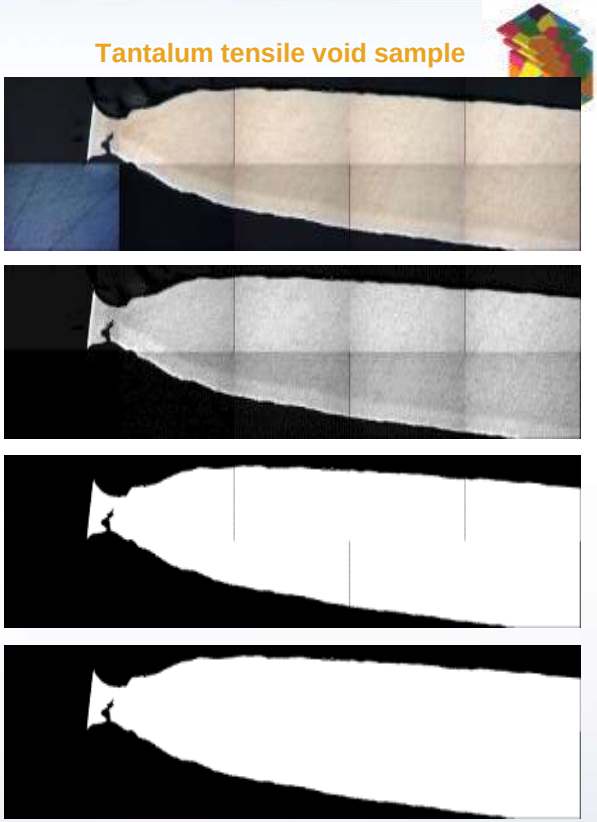
3D RoboMet Polishing Provides Significant Insight



montage: 5 x 2

magnification : 20X

Tantalum tensile void sample



ROBO-MET.3D™

Image processing

raw optical image

8 bit grayscale

image cleaning

binarize for reconstruction

Ta Tensile Voids Serial Sectioning Experiment – June/July 2013

- Completed 135 serial sections at ~ 2.5 microns per slice (total sectioning depth of ~ 340 microns)
- Sample reveals voids at initial cross-section which quickly link to a full crack across the gauge cross-section
- Crack appears to close at further distances into the gauge section (see end of animation)
- Developed an image segmentation process to binarize the image set for reconstruction and keep fidelity of voids and cracks in gauge section
- Should have full 3d reconstruction by week's end

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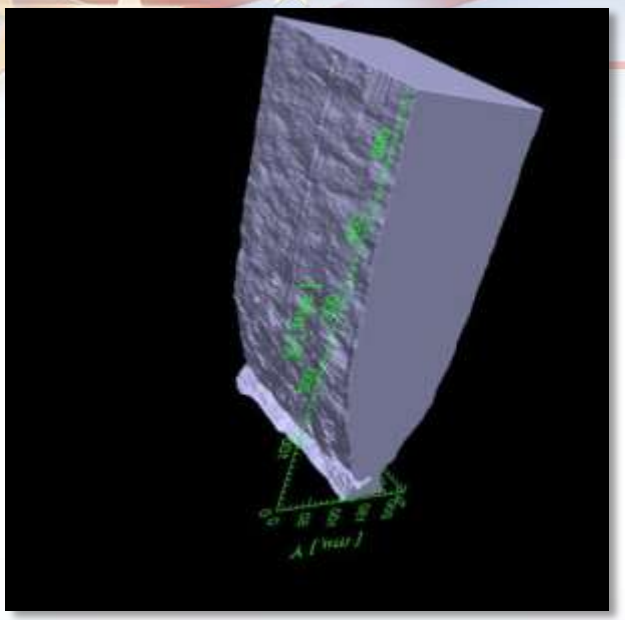
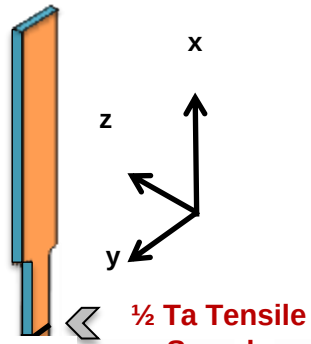
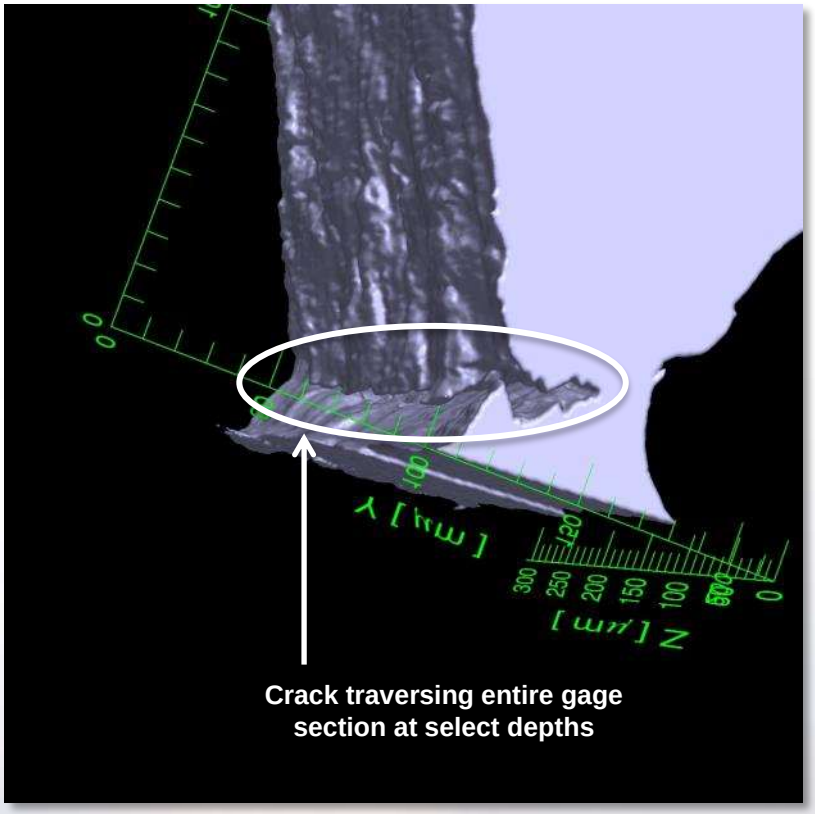
3D Reconstruction Shows Asymmetric Failure

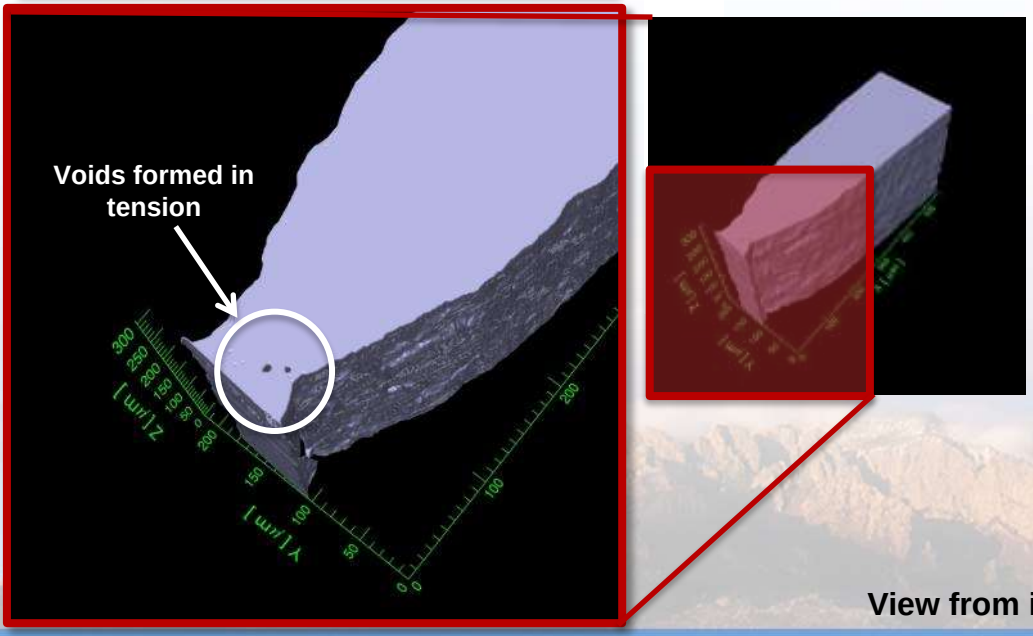




½ Ta Tensile Sample

View from final sectioning plane





3D fracture characterization shows a full length crack progressing through only half of the necked region

View from initial sectioning plane

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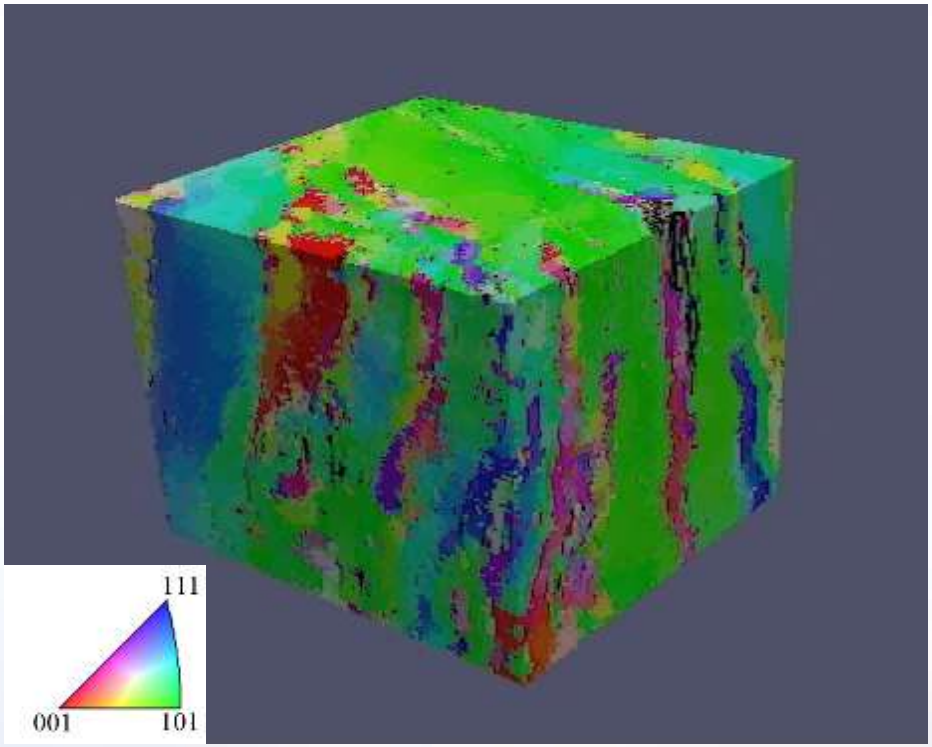
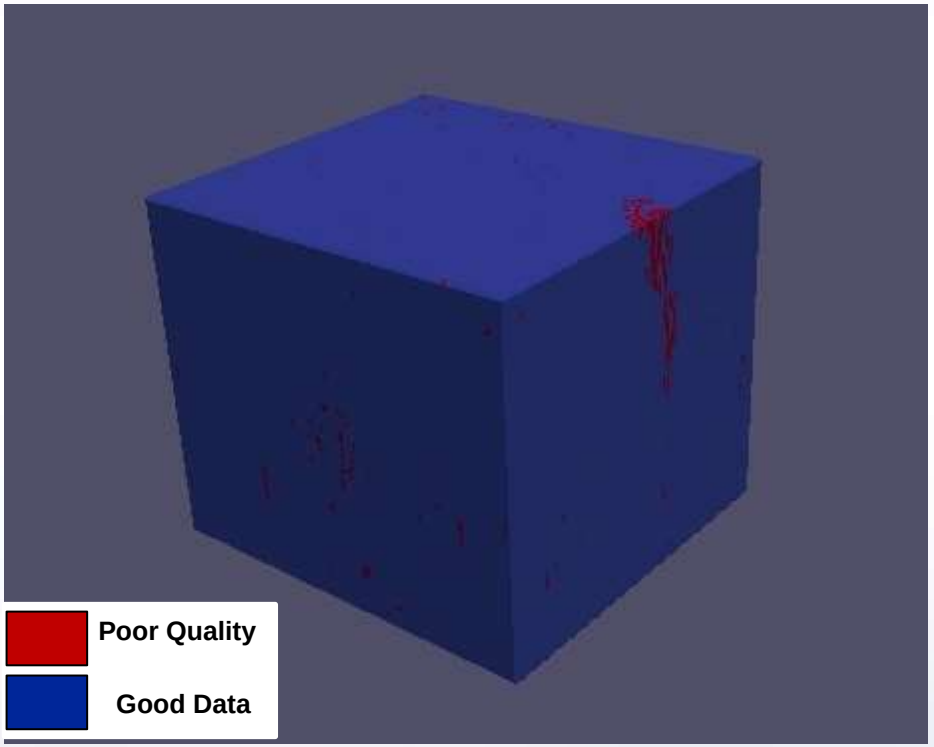
3D EBSD in a Single Subsurface Void

Video of 3-D EBSD reconstruction with IPF colors



Video of the registered errors in 3-D EBSD



127 slices **Step size: .05 µm**

3D EBSD done with Oxford Instruments & DREAM3D EBSD software

3D EBSD highlights the relationship between the local grain boundary location and texture with the voids formed

Filters and Parameters Used in Reconstruction

Multi Threshold (Cell Data): Error=0

Align Sections Misorientation tolerance: 5˚

Neighbor Orientation Comparison : 5˚ & 6 Nearest Neighbor

Rename Cell Array from MAD to Confidence Index

Neighbor Orientation Correlation

-Minimum Confidence Index: 0.05

-Cleanup Level: 0

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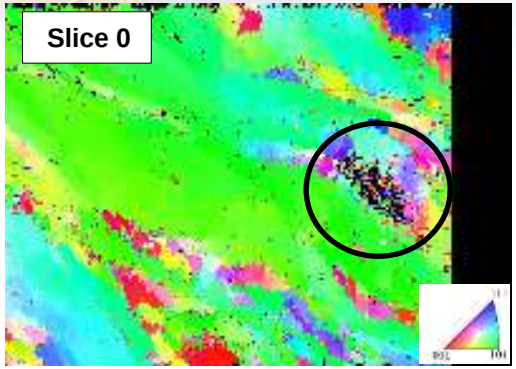
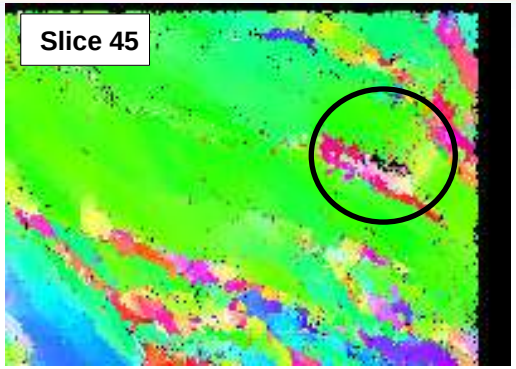
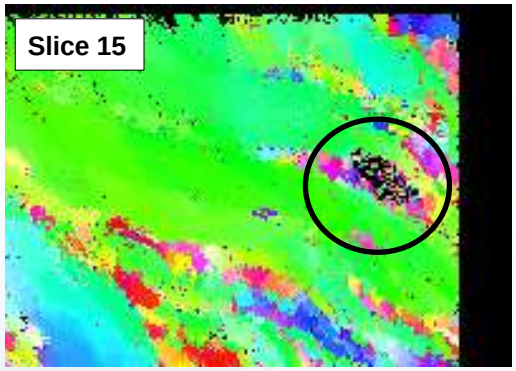
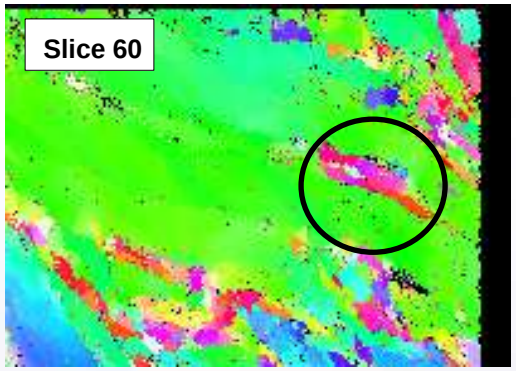
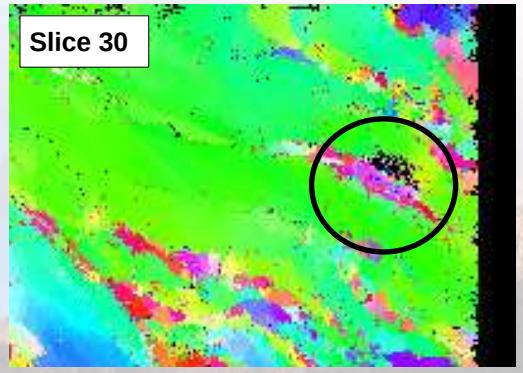
Frame by Frame Analysis of a Single Void

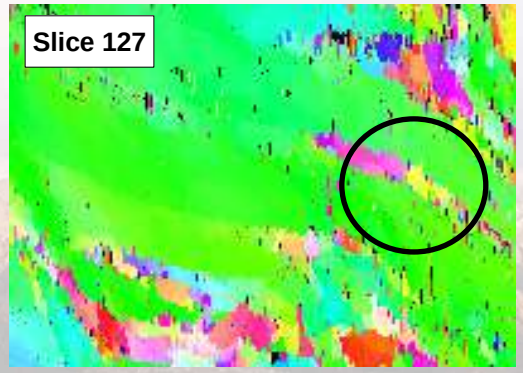












The void starts large, and gradually tapers off. ~50% of the way through the sample it disappears, revealing a sub-grain boundary

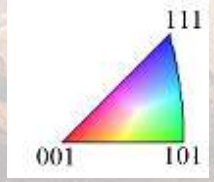
Benefits

- 3D visualization of the local region
- Grain orientation through thickness
- Clearer understanding of the depth and tapering of voids

Limits

- Rapid scans result in high error
- Processing software is still under development
- Full capabilities still being explored

The noise and shape of the void are suspected to be artifacts of the current data processing



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Summary

In-situ Ta straining experiments

- Ductile failure is a stochastic phenomenon
- Even average behavior for ductile fracture is difficult to predict
- Crystal plasticity models may provide a pathway to assess microstructural effects on ductile fracture. To that end, a BCC Crystal plasticity model is under development for Ta.
- In-situ mechanical testing provides a method to both validate crystal plasticity models and observe fracture processes for the development of practical microstrutural-scale failure models.

Current status on the 3D characterization

- Feasible at Optical Microscopy Level
  - Demonstrate asymmetric failure
- Feasible at EBSD level of a single void
  - Demonstrate the importance of sub-grain boundaries in void location

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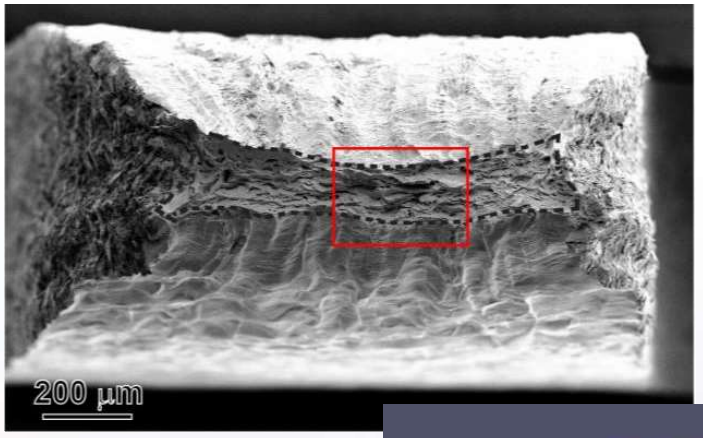

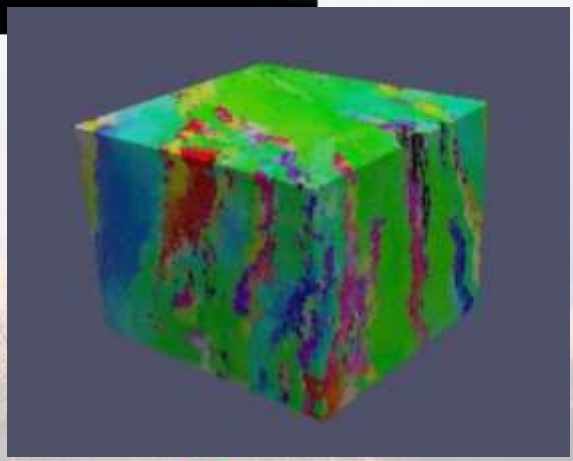

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