Comparison of Hydrogen Introduction Techniques for In-situ TEM Straining Mechanisms

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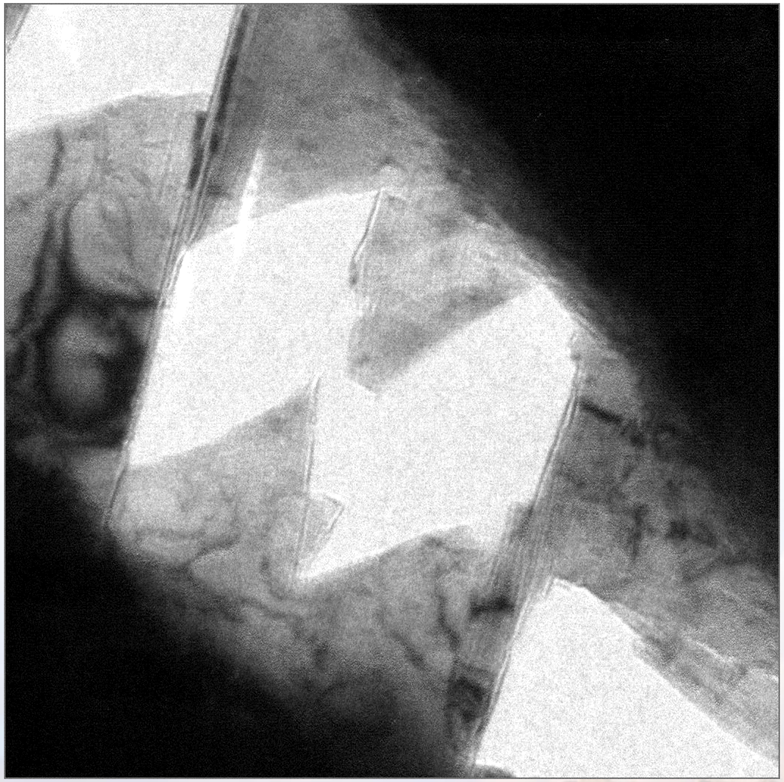
Sandia National Laboratories & Stanford University

March 13, 2018



Exploring the various methods to study hydrogen embrittlement in the TEM

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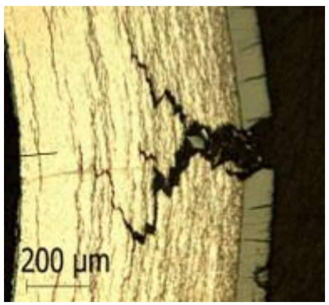
Fracture is a Costly Problem Outside of Sandia That Remains Unsolved

R81

FRACTURE COSTS US $119 BILLION A YEAR, SAYS STUDY BY BATTELLE/NBS*

J.J. Duga, W.H. Fisher, R.W. Buxbaum, A.R. Rosenfield, A.R. Buhr, E.J. Honton, and S.C. McMillan



Even if the probability of cladding failure from this cause during the lifetime of a fuel element is low (~ 10^{-5}), the consequences are not (~$1M/day if shutdown of the reactor is required and replacement power must be purchased) [Olander, JNM, 2009].

Hydrogen tank leak evacuates area near Pixar in Emeryville

Emeryville hydrogen tank leak

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By Isabel Angell

The Seattle Times
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State declares Tesoro blast was preventable

The Washington state Department of Labor & Industries on Monday fined Tesoro a record $2.39 million after a deadly explosion in April at the oil refinery in Anacortes.

By Susan Gilmore and Craig Welch
Seattle Times staff reporters

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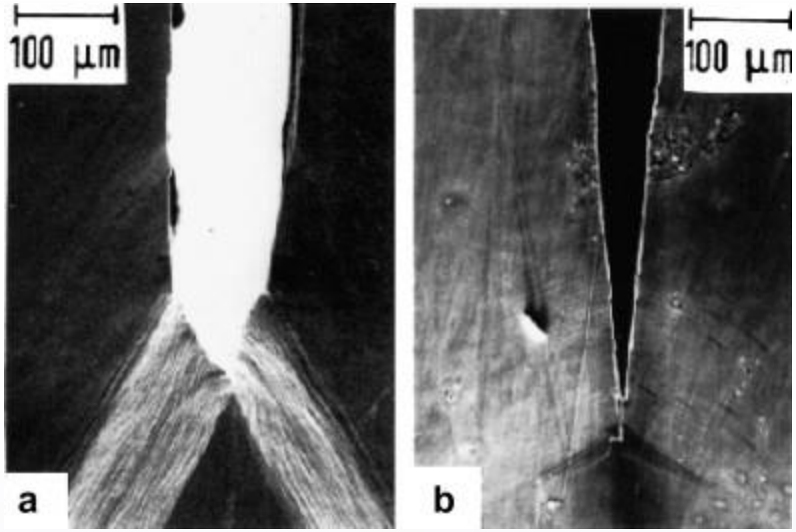
MOUNT VERNON — As the state Monday assessed a record $2.39 million fine against Tesoro for last

Hydrogen Embrittlement of Metals

Collaborator: for work on this slide

Hydrogen content degrades mechanical properties of metal systems

- **Loss in ductility, strength, and toughness**
- **Proposed Mechanisms**
  - Hydrogen-enhanced decohesion (HEDE)
  - Hydrogen-enhanced localized plasticity (HELP)
  - Hydride formation



Crack tip opening angle in Ni single crystal decreases as H pressure increases

Accumulation at microstructural interfaces

- **H induced cracks seen in steels, nickel and nickel based alloys**
  - Doesn't require stress or elevated temperature
- **H-dislocation interaction observed to be prelude to embrittltment**

Louthan Jr., M.R. Savannah River National Laboratory
Barnoush, A. et al. (2008). *Corrosion Science.*
Matusiewicz, G. (1985). *Acta Metallurgica.*

In-situ TEM techniques provide insight to mechanism of failure due to H embrittlement

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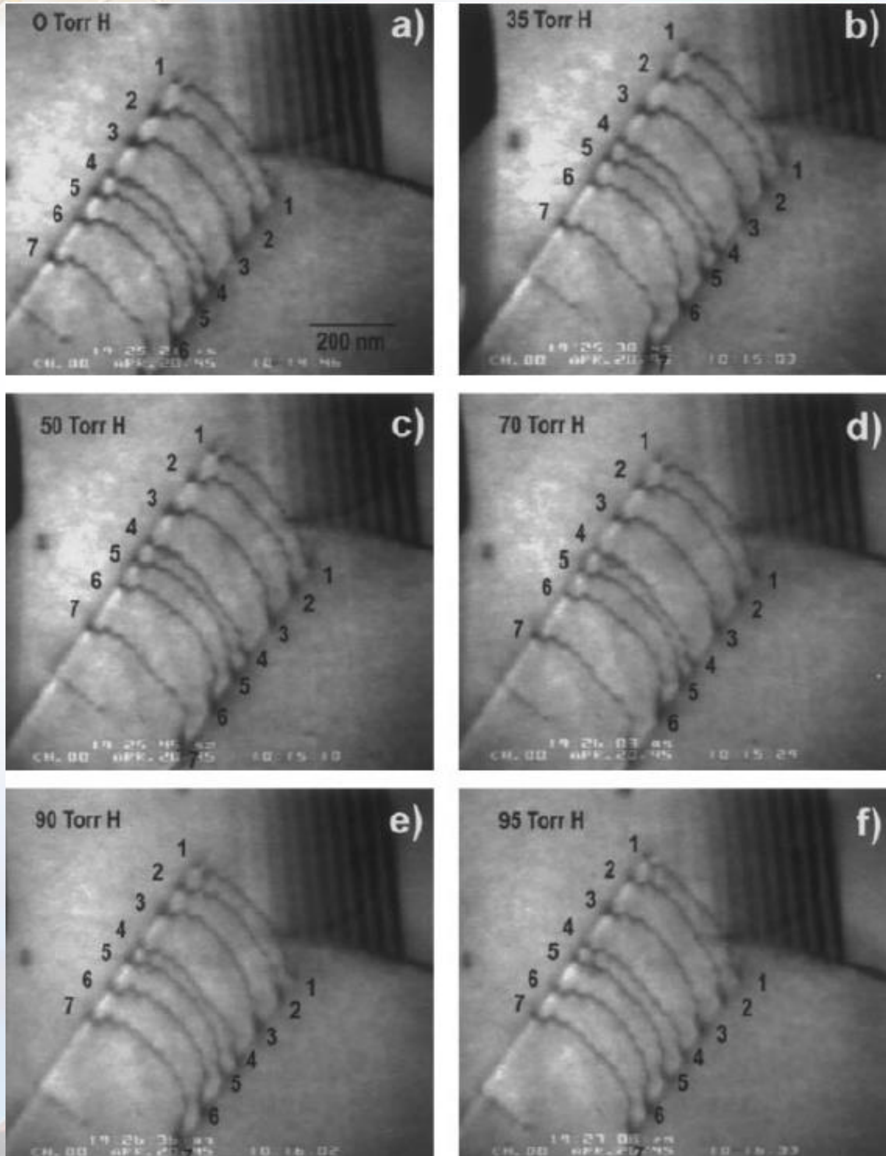
20 years of *In situ* TEM Hydrogen Studies

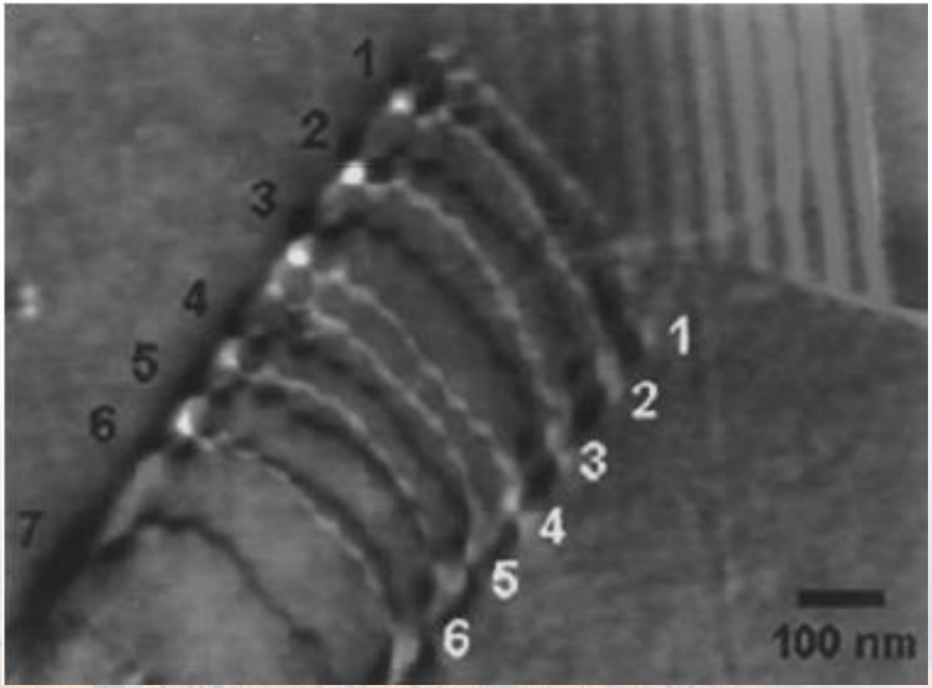


HYDROGEN EFFECTS ON THE INTERACTION BETWEEN DISLOCATIONS

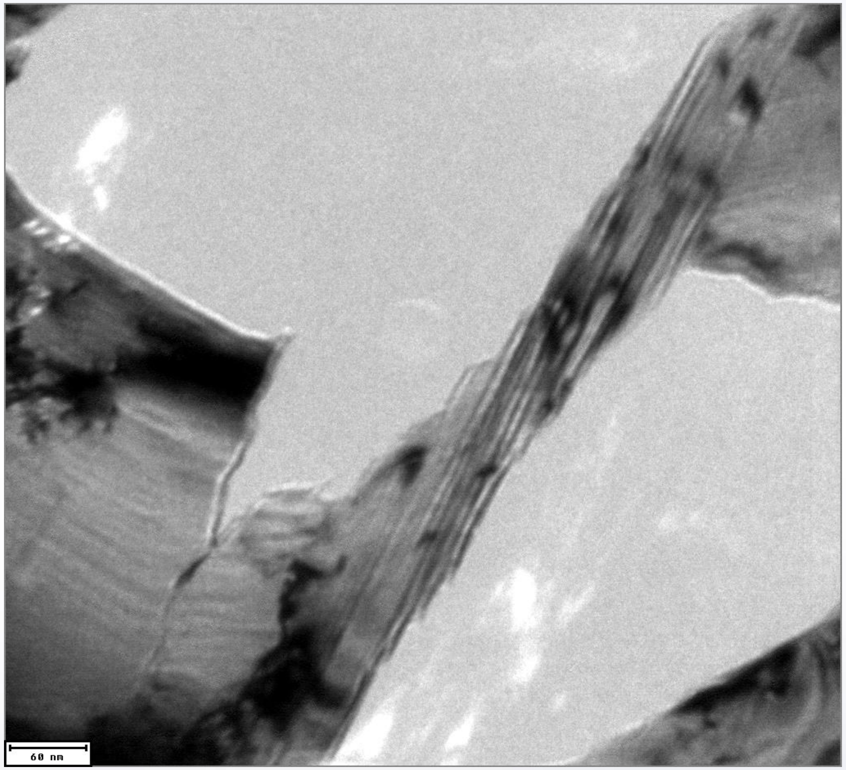
P. J. FERREIRA†, I. M. ROBERTSON and H. K. BIRNBAUM

University of Illinois, Department of Materials Science and Engineering, and Frederick Seitz Materials Research Laboratory, Urbana, IL 61801, U.S.A.

(Received 29 August 1997)

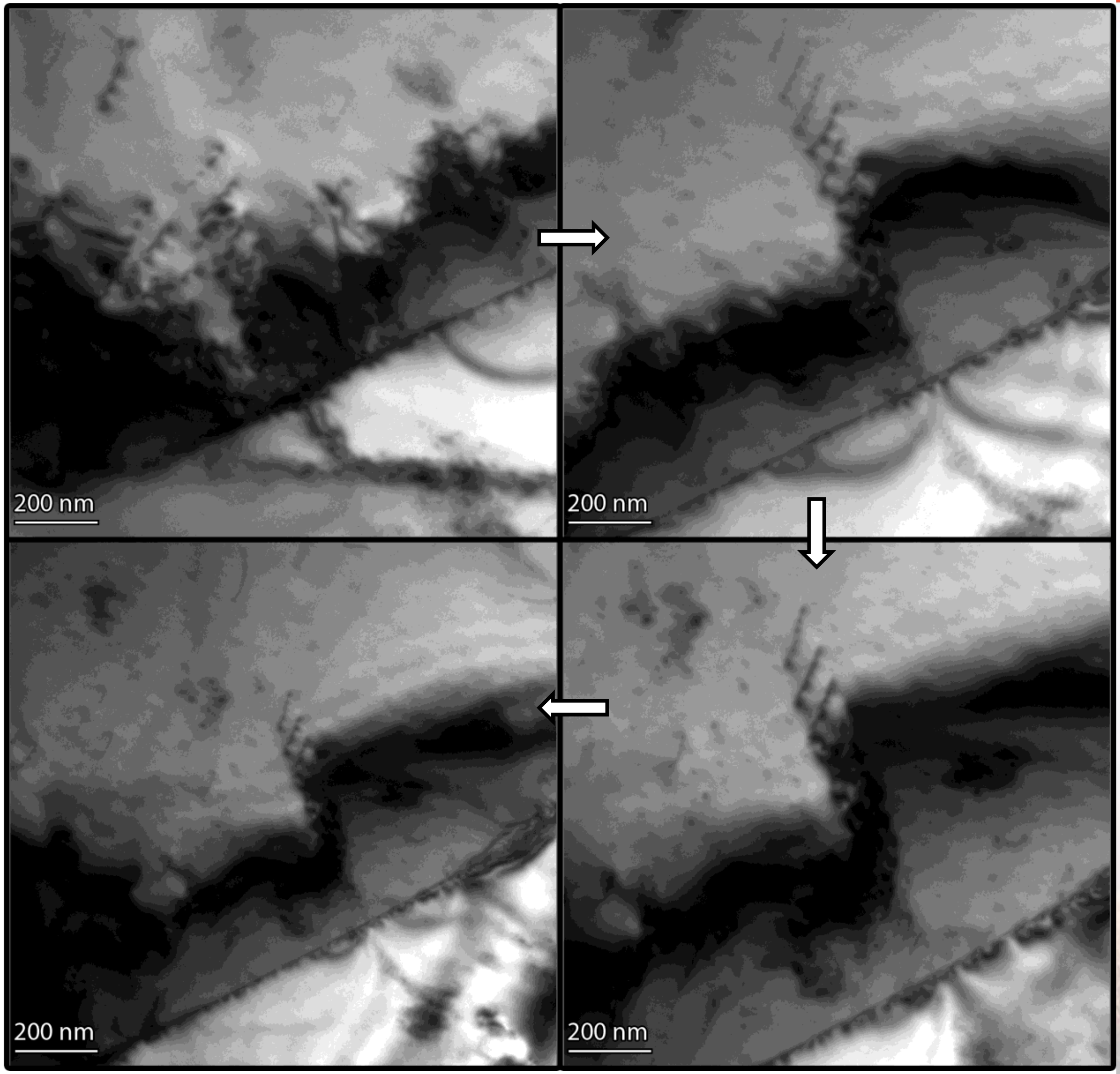


Control Experiments: Failure of pure Ni



In-situ TEM techniques provide insight to mechanism of failure due to H embrittlement

Traditional Charging and In-situ Straining

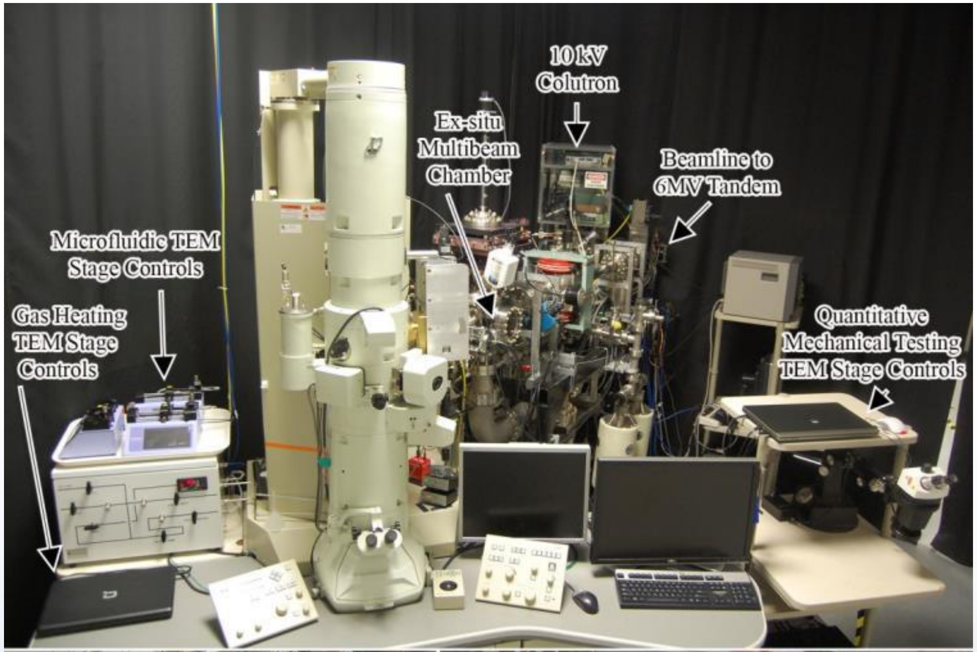


Dislocations appear to move toward and be absorbed into boundary

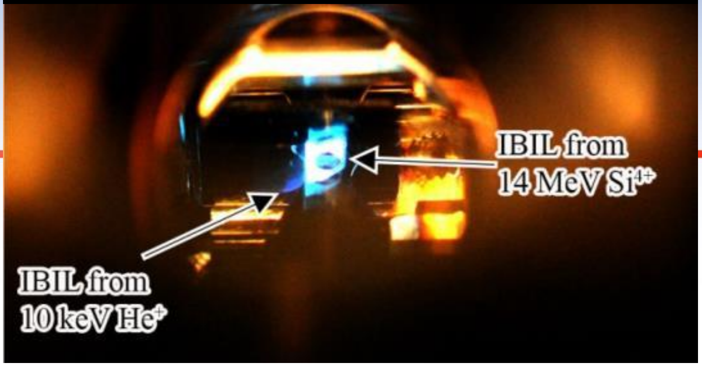
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

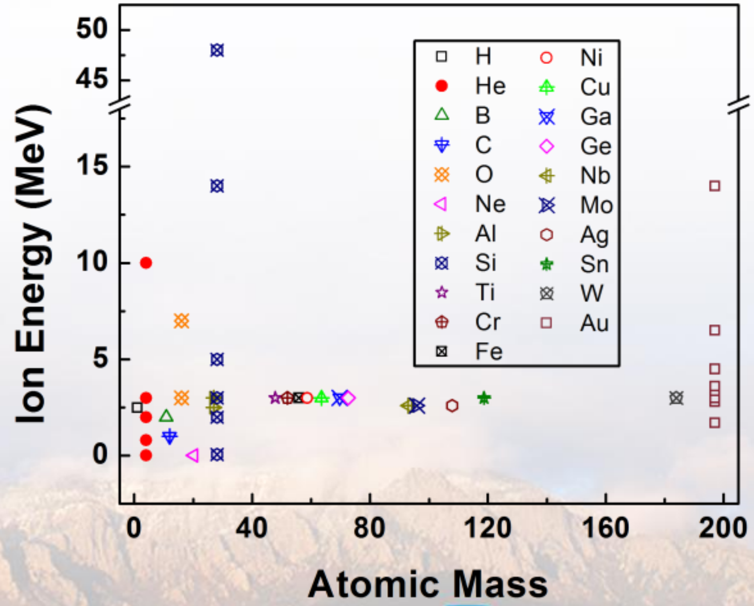


IBIL from a quartz stage inside the TEM

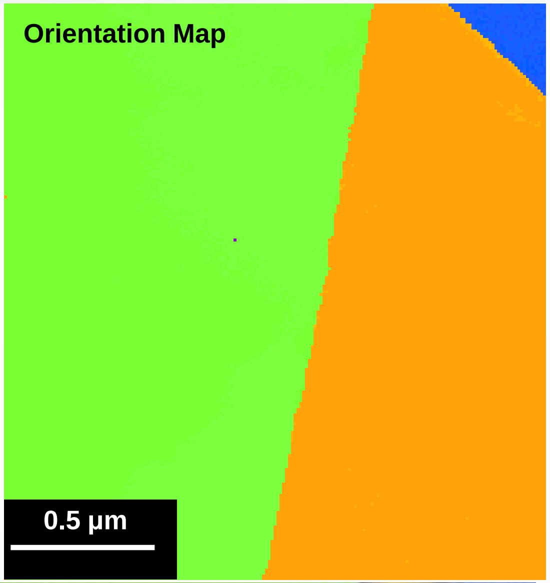


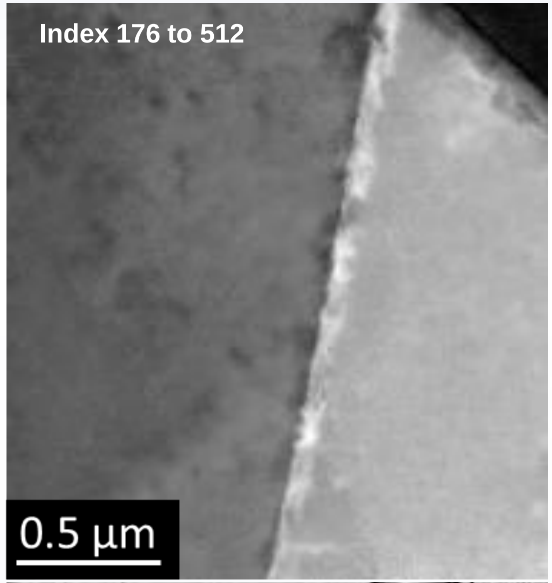
Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

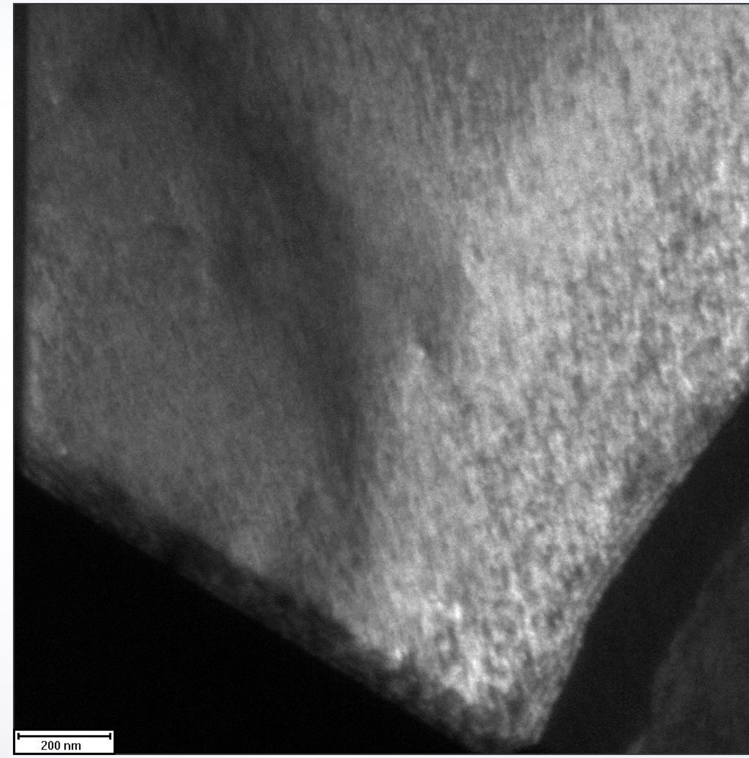
Ion species & energy introduced into the TEM

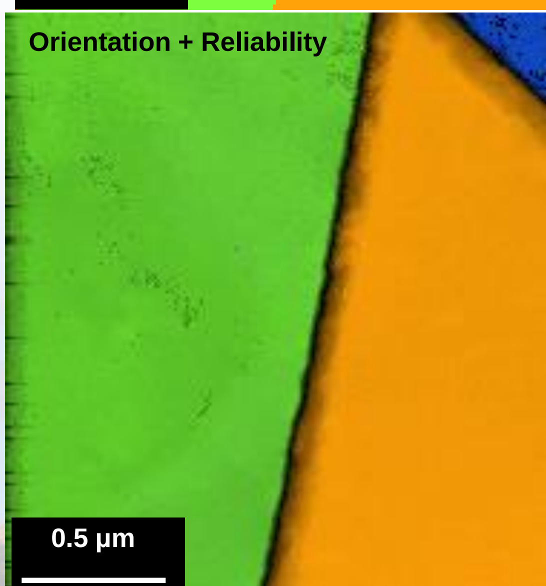


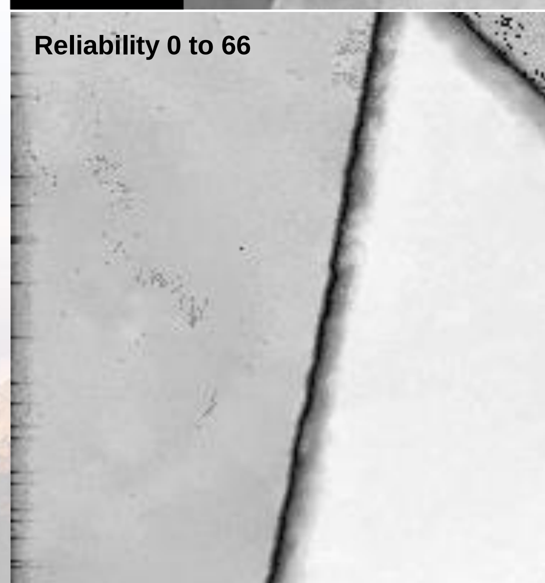
Characterization of Orientation, GB structure and Displacement Defects

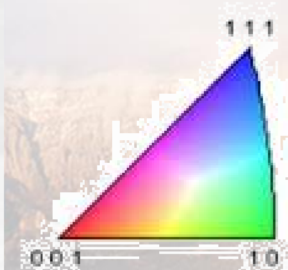








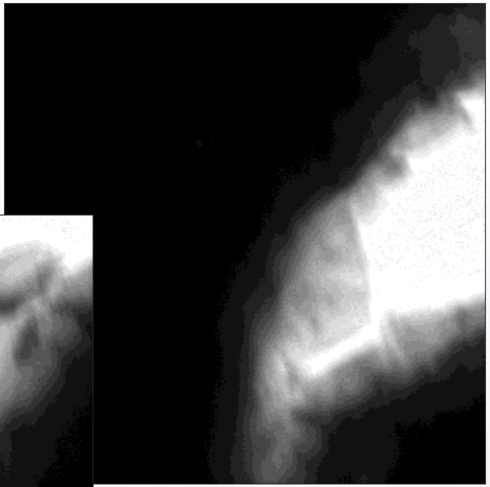


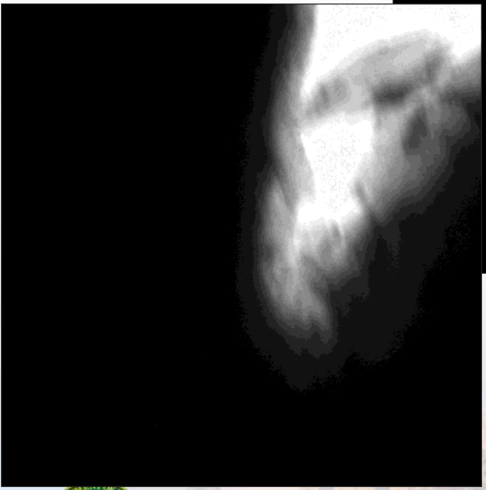


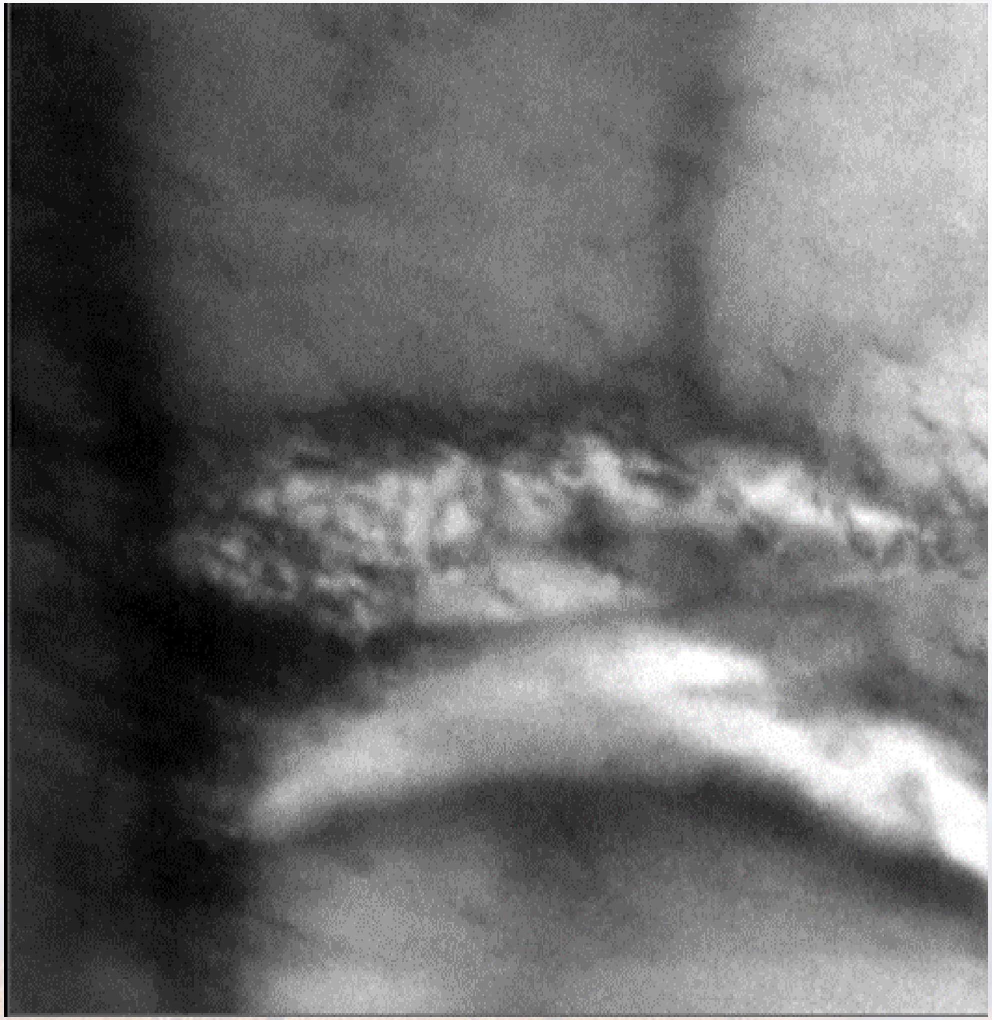
In-situ D Irradiation

<u>Pure Ni before and after Ni and D+ irradiation</u>

- **Mode of mechanical failure unchanged**
- **Dislocation movement**





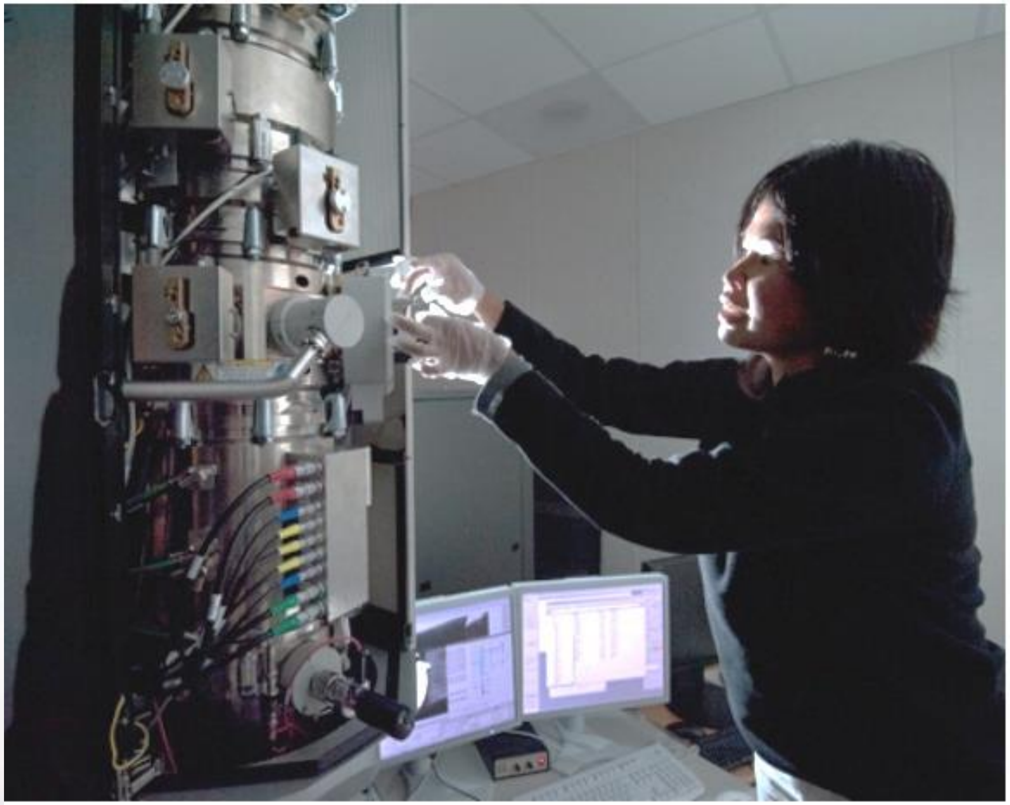


In-situ TEM techniques provide insight to mechanism of failure due to H embrittlement

In situ H_2 experiment at Stanford

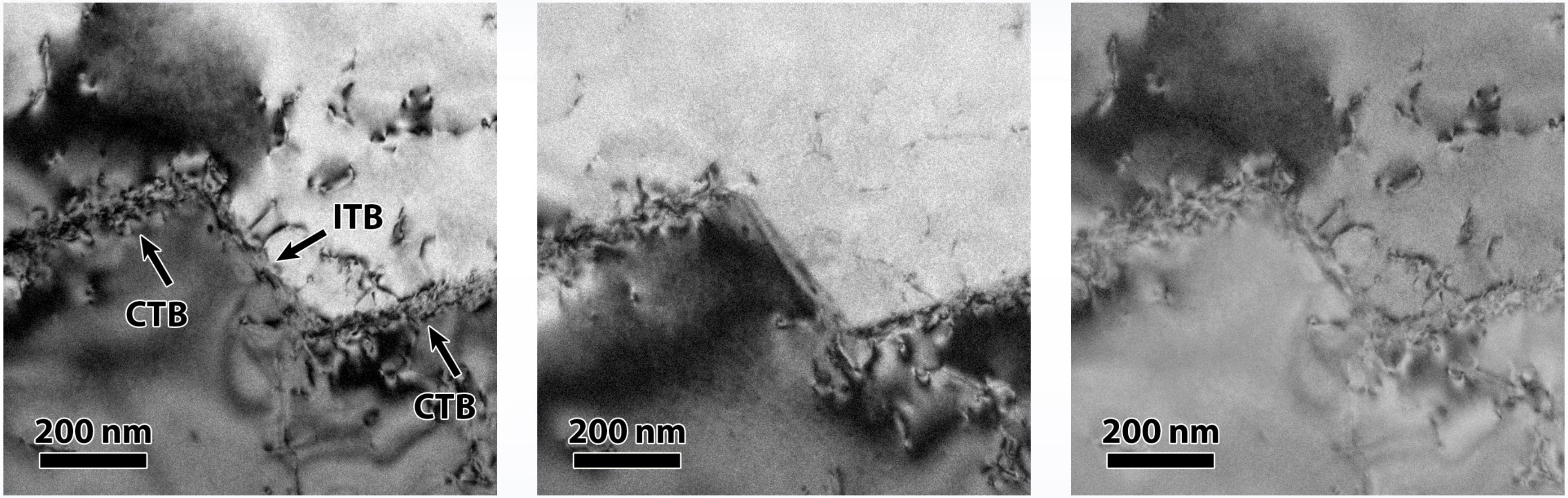
- Imaging in H_2 gas using double-tilt heating holder
- Max pressure is ~10 mBar.
- Limited to lower pressure for hot-stage work due to thermal losses to gas (e.g, at 500° , P_{H2} max ~2-3 mBar).

• Aim: investigate predicted defaceting transition with H_2 exposure for Σ=3 boundaries in Nickel and the change in fracture mechanisms.



Environmental TEM at the Stanford Nano Shared Facility (SNSF). (http://snsf.stanford.edu/equipment/eim/titan.html)

In situ H_2 and Annealing Experiment

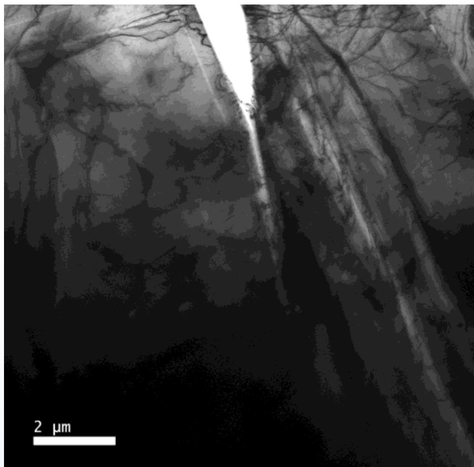


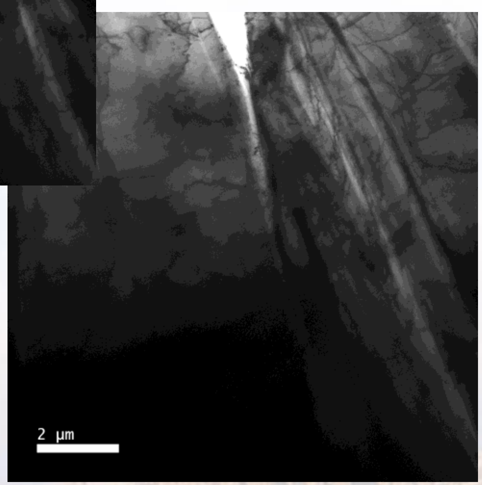
- Left to right: before, after, difference image
- Most notable changes: cleaning of ITB
- CTB/ITB corners may have rounded, but some of that may be to tilt. Disappearance of dislocations in upper portion is due to change in imaging condition.

In situ H_2 and Straining Experiment

Ni behavior during H flow

- **Dislocation behavior during H introduction**
  - Between 10^{-3} and 9 mbar





Pre-existing crack in pure Ni at 10^{-3}mbar (left) and in a 9mbar H environment (right)

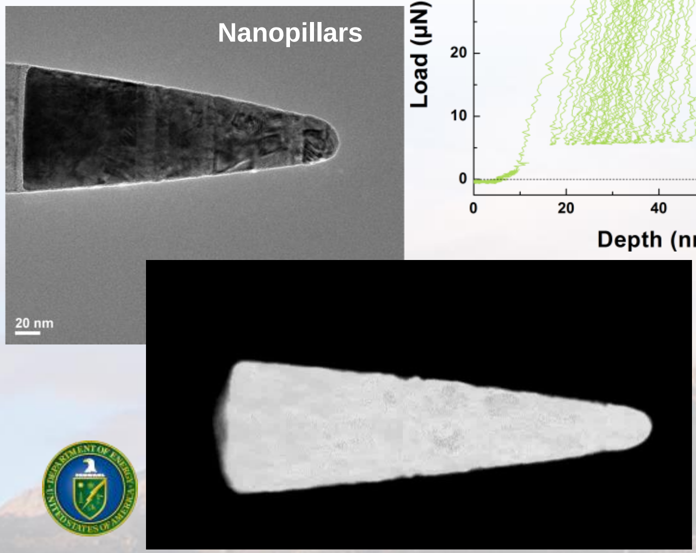
In-situ TEM techniques provide insight to mechanism of failure due to H embrittlement

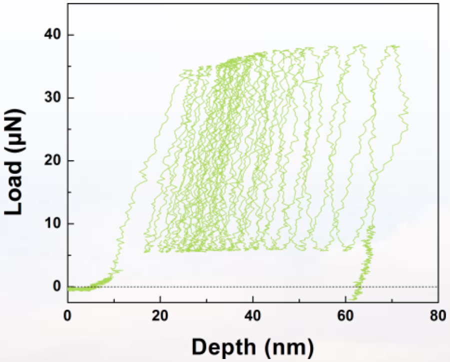
Future Directions

In situ Quantitative Mechanical Testing

Hysitron PI95 *In Situ* Nanoindentation TEM Holder

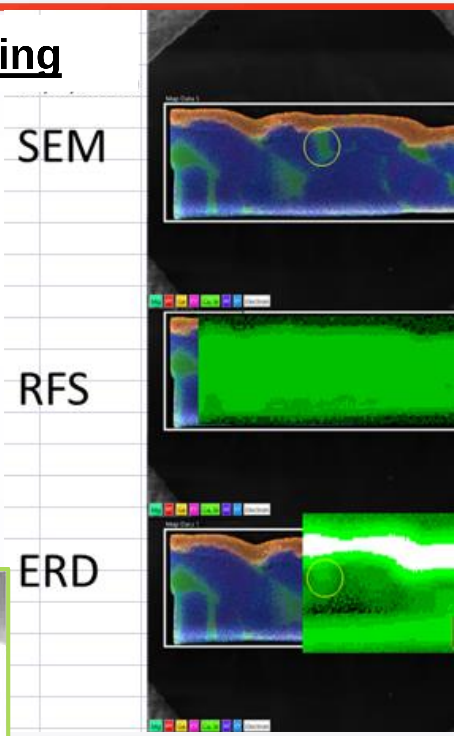
- Sub nanometer displacement resolution
- Quantitative force information with µN resolution
- **Concurrent real-time imaging by TEM**

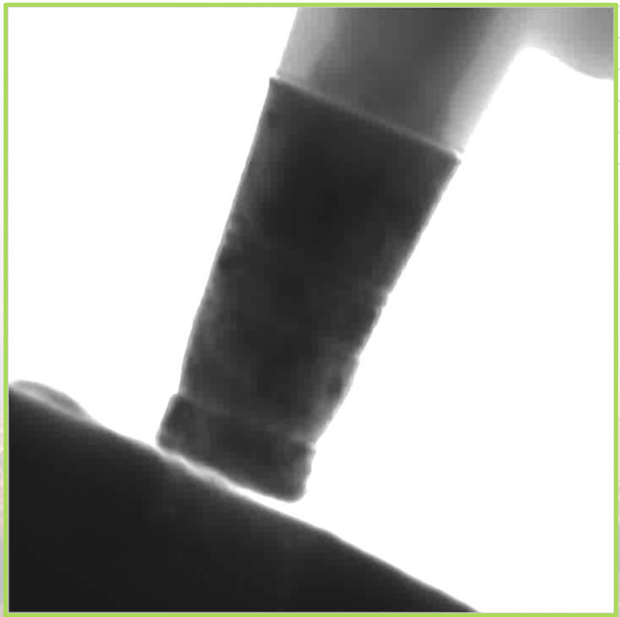




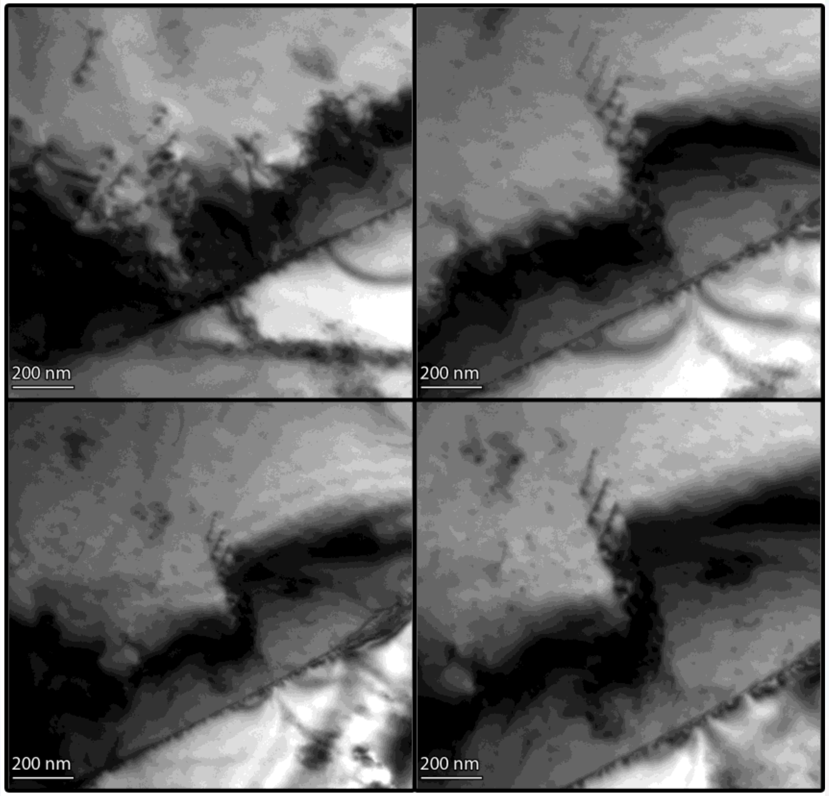
Hydrogen Isotope Mapping

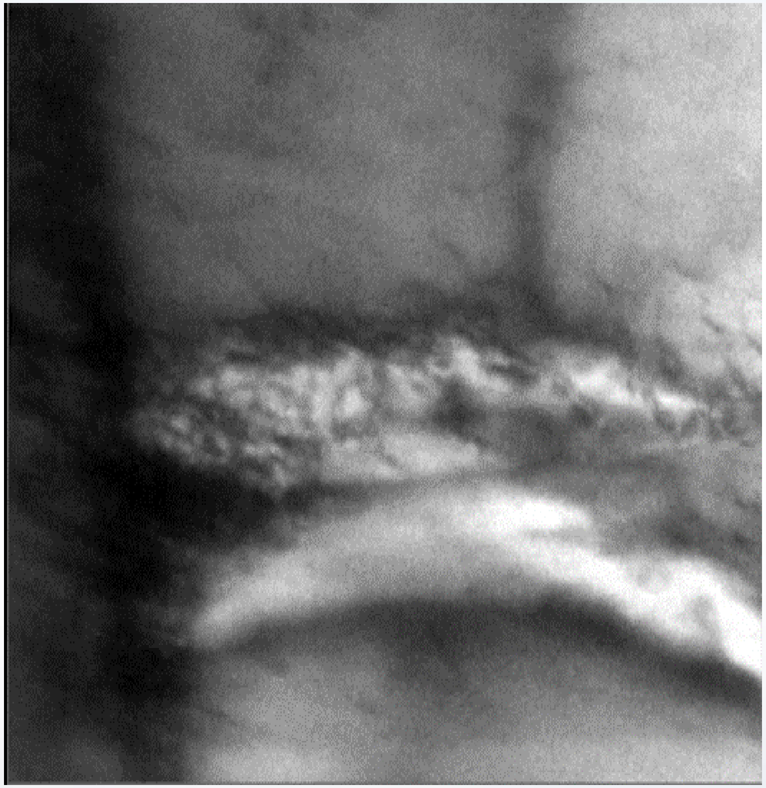
Development of methods to chemical map out hydrogen and helium isotopes using transmission electron elastic recoil detection





Summary





In situ hydrogen gas exposure appears to be the best path to study defect response to hydrogen environment

External access and collaborations available through CINT and NSUF user proposals

Please contact Khalid Hattar khattar@sandia.gov for more information

This work was supported by the US Department of Energy, Office of Basic Energy Sciences. Or other funding source.

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NSUF Nuclear Science User Facilities

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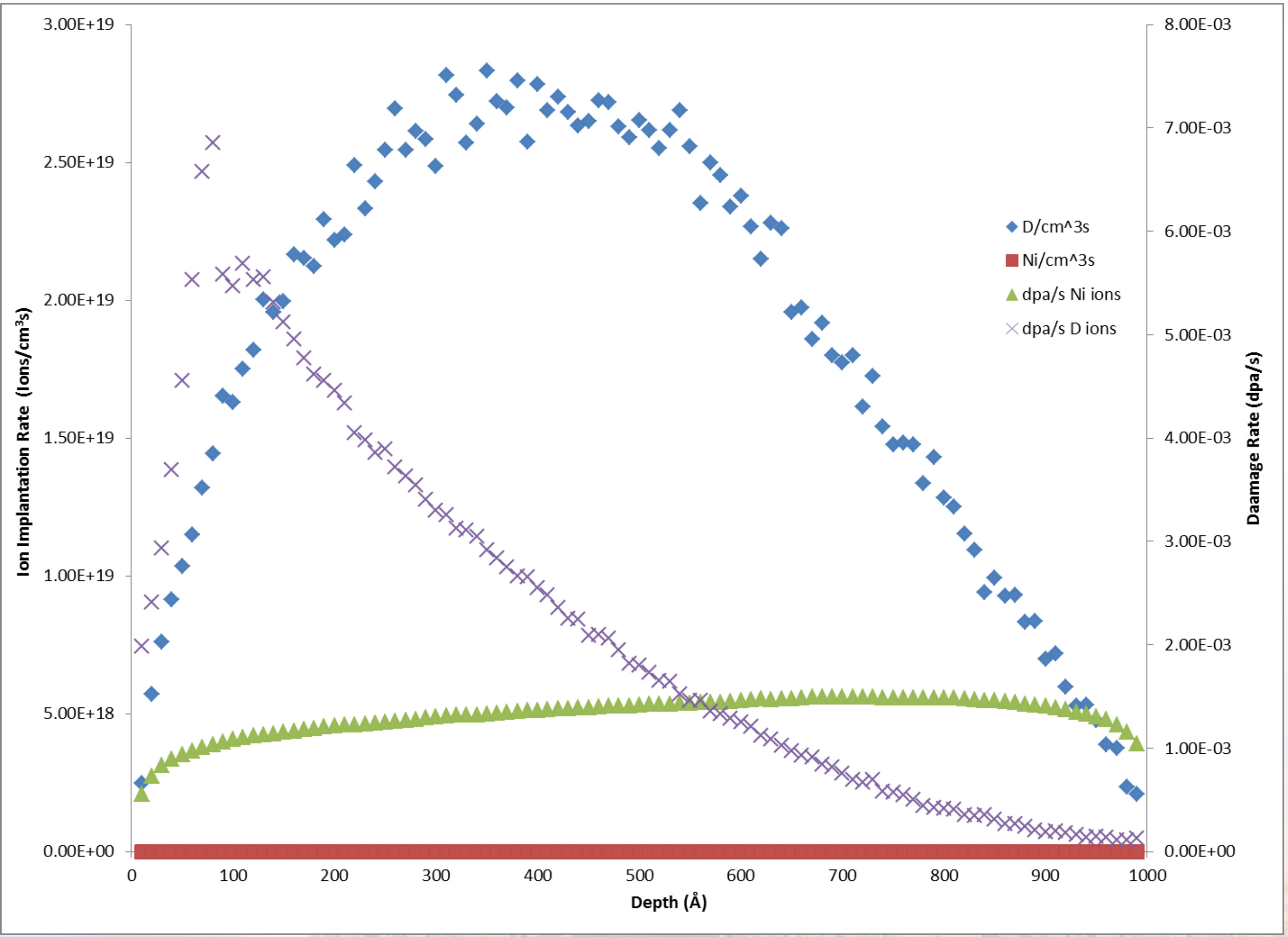
D^+ and Ni^{3+} into large grained Jet polished Nickel



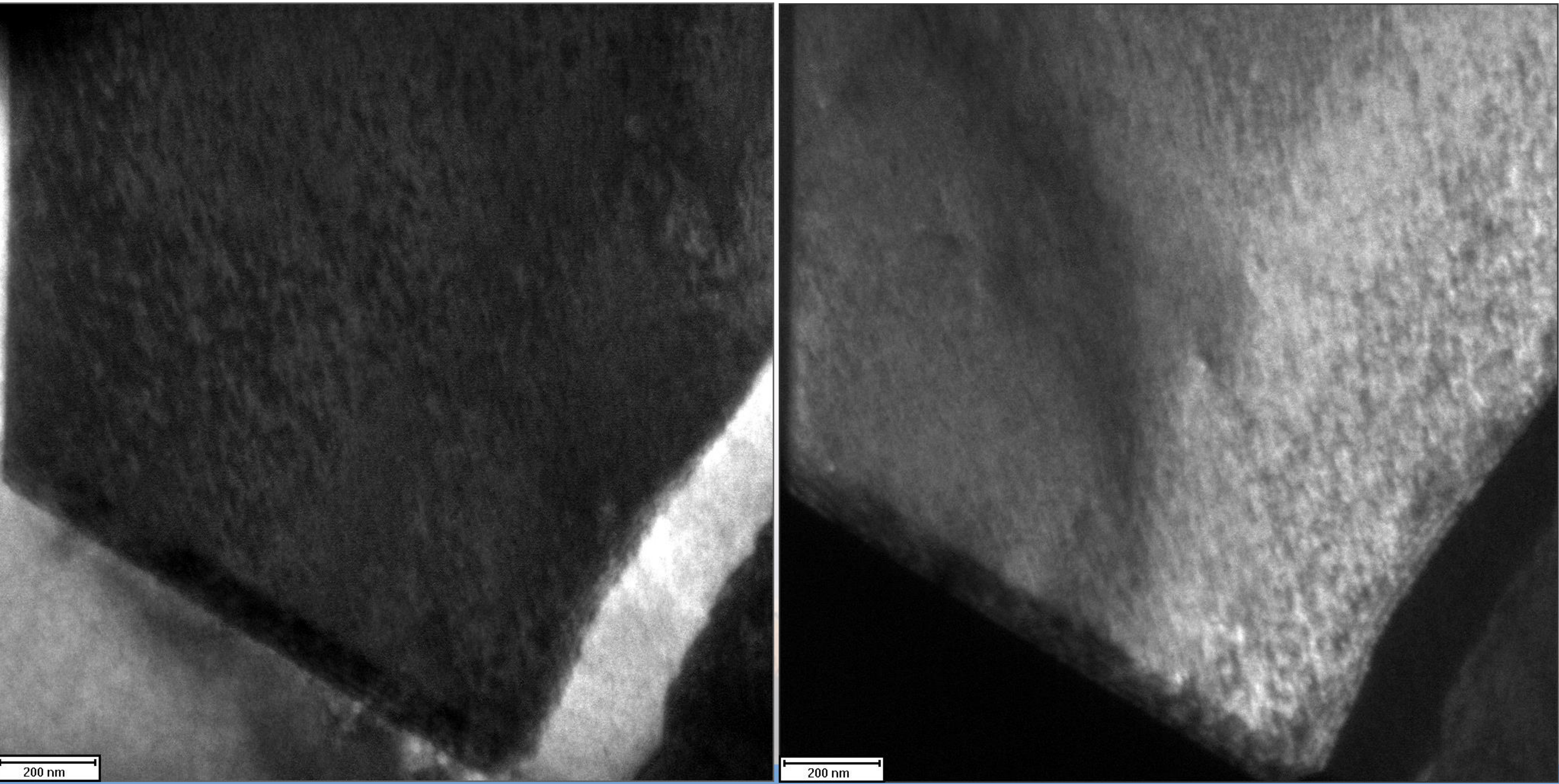
Irradiation Rates:

2.6 E14 D^+/cm^2s

4 E11 Ni^{3+}/cm^2s

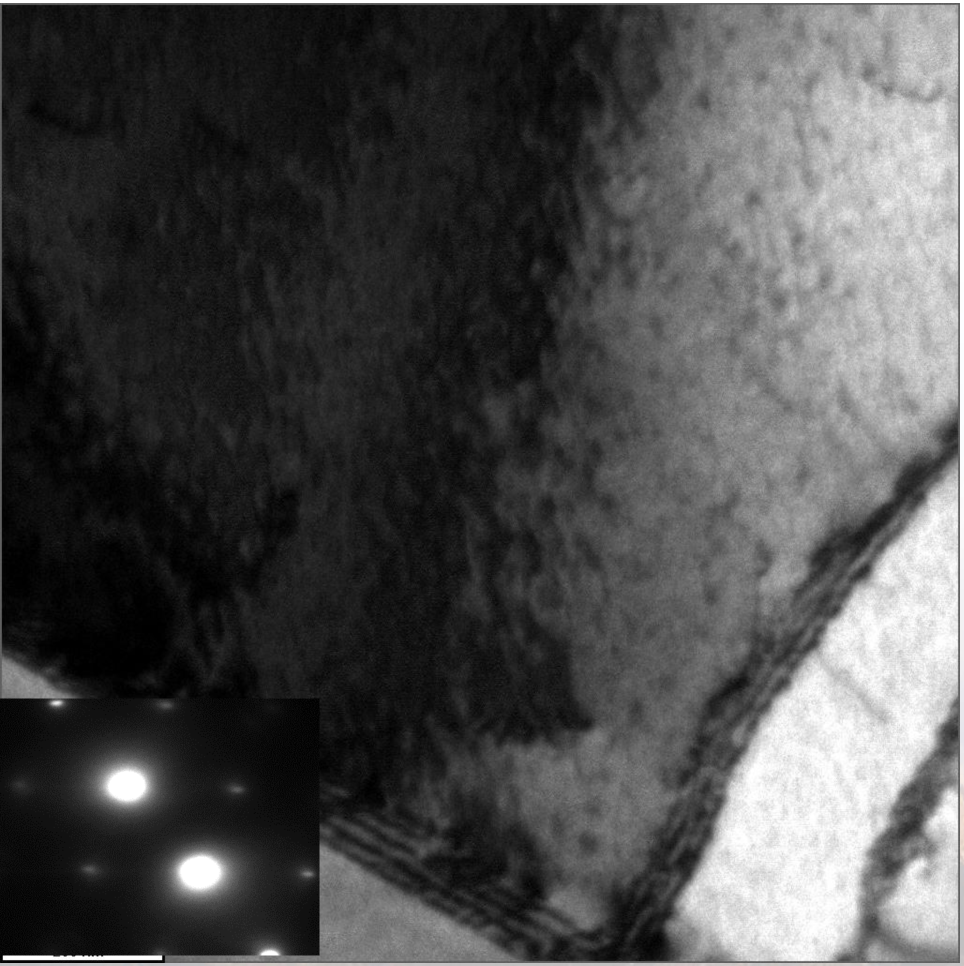
Irradiated to 2 dpa from D^+ (approximately 19 at % D^+) and 2 dpa from Ni^{3+} for a total of 4 dpa

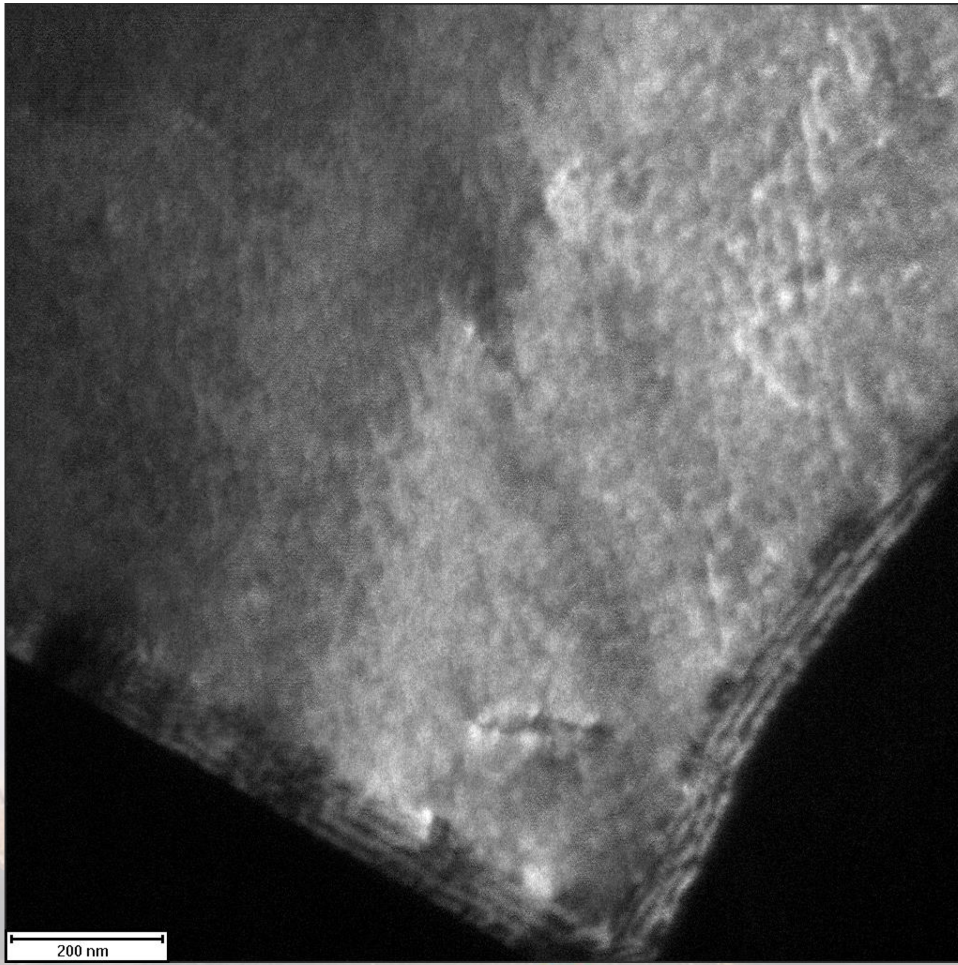
After D^+ implantation



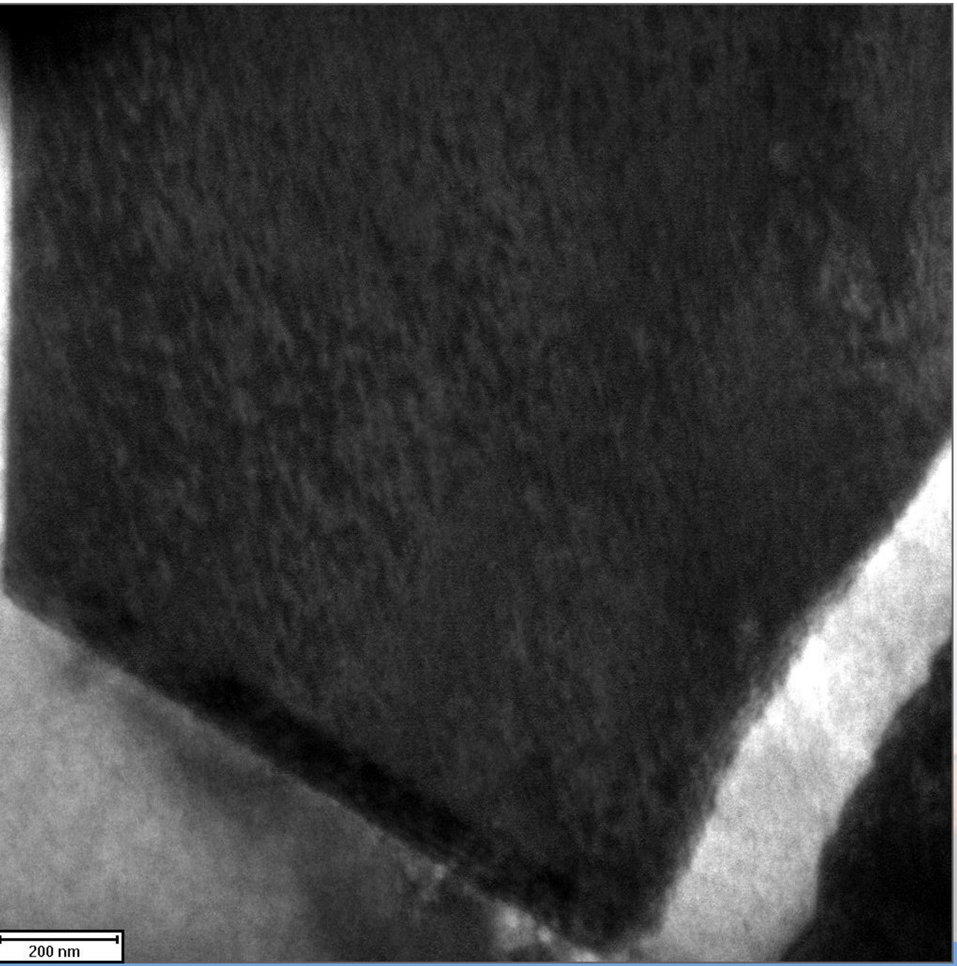
1: D^{+} then Ni^{3+}

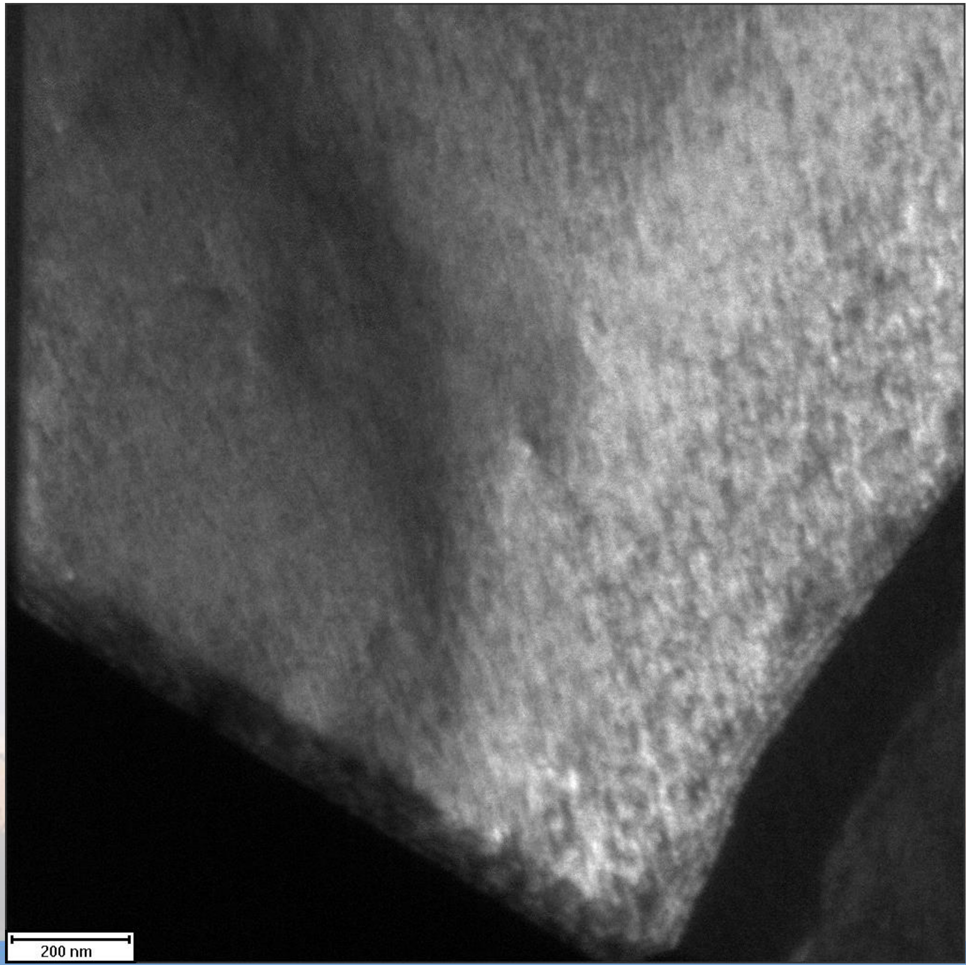
Before irradiation

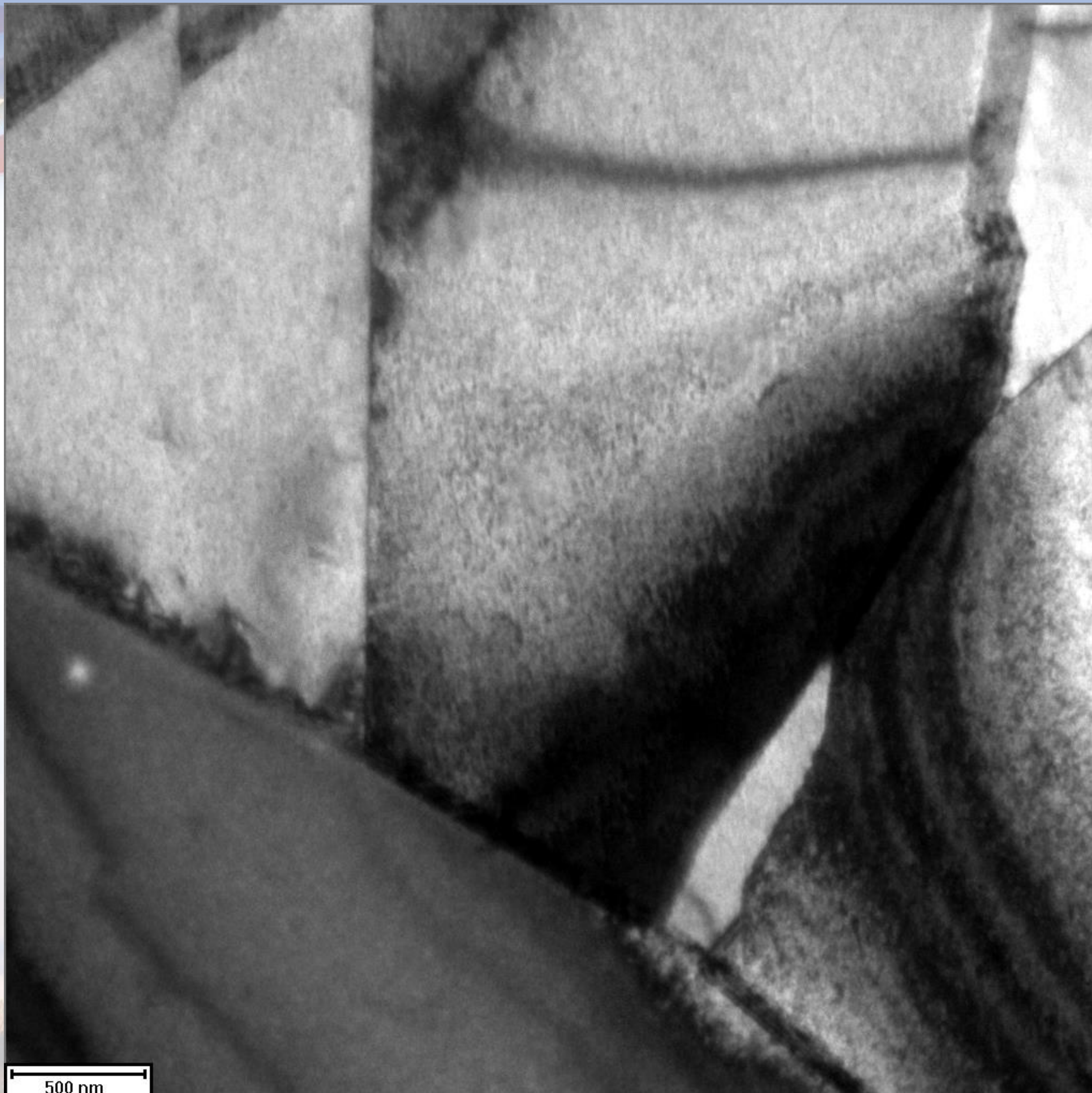




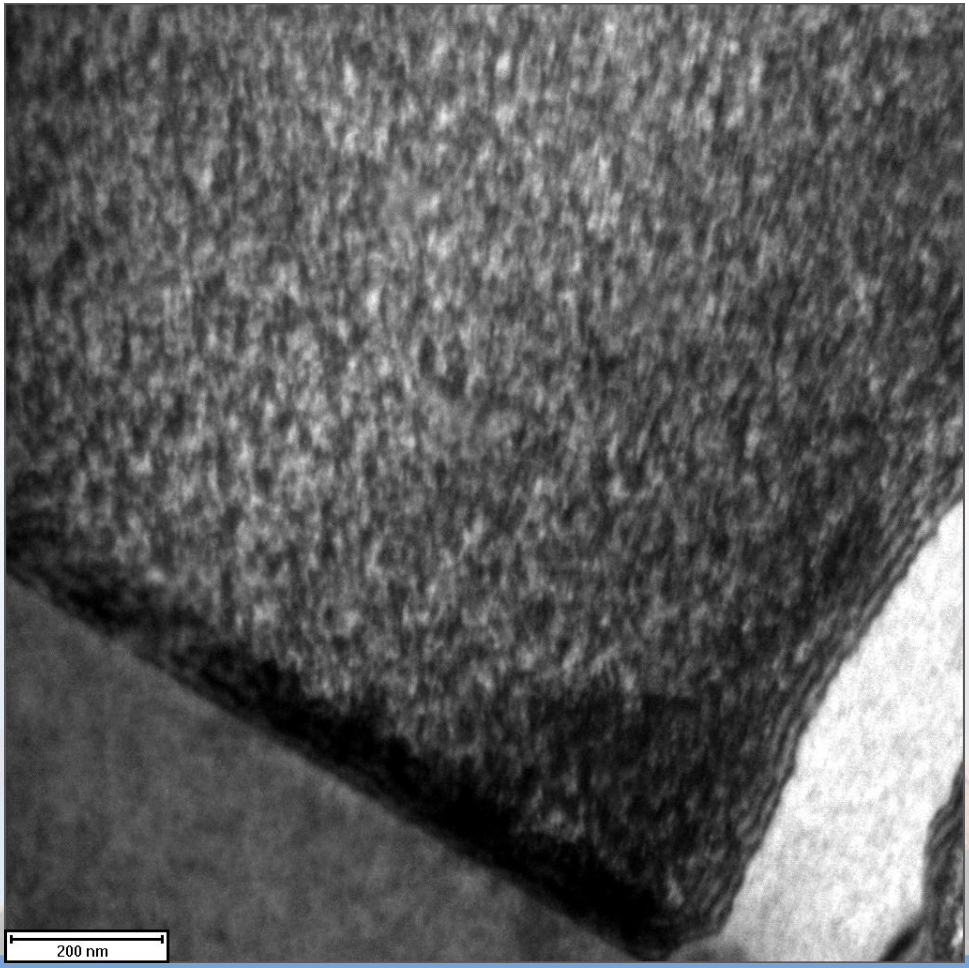
After D^+ implantation

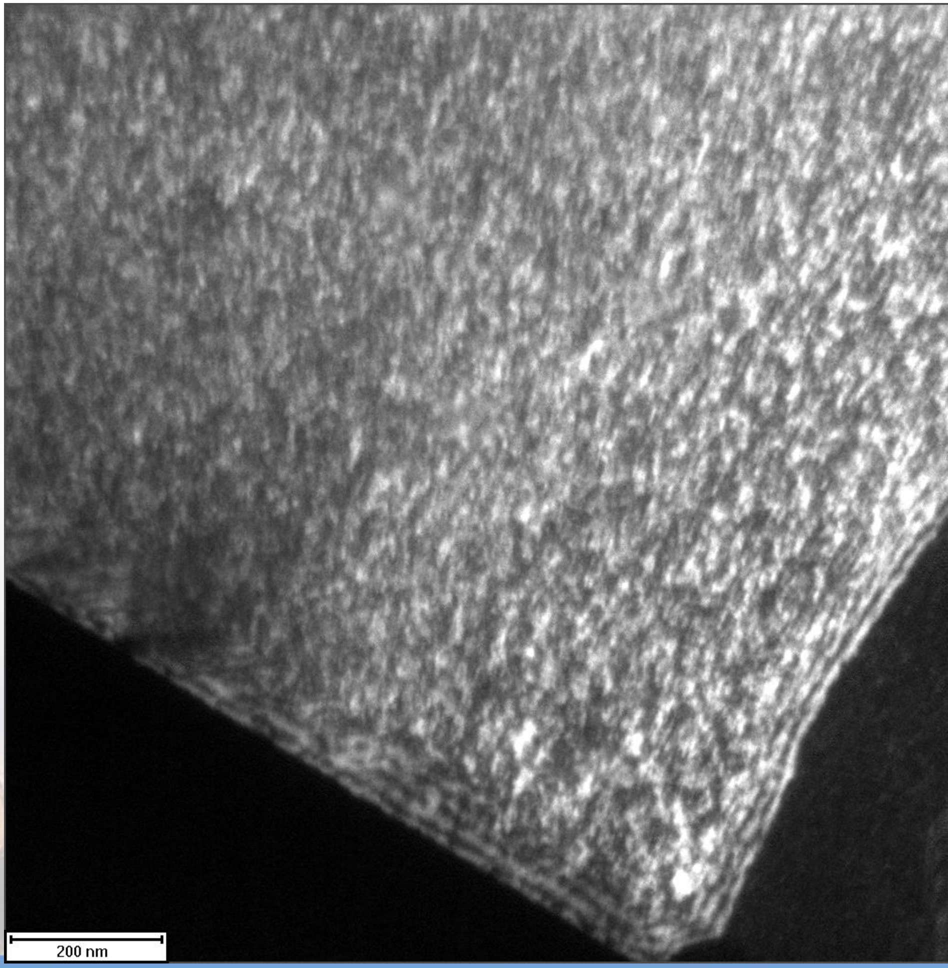


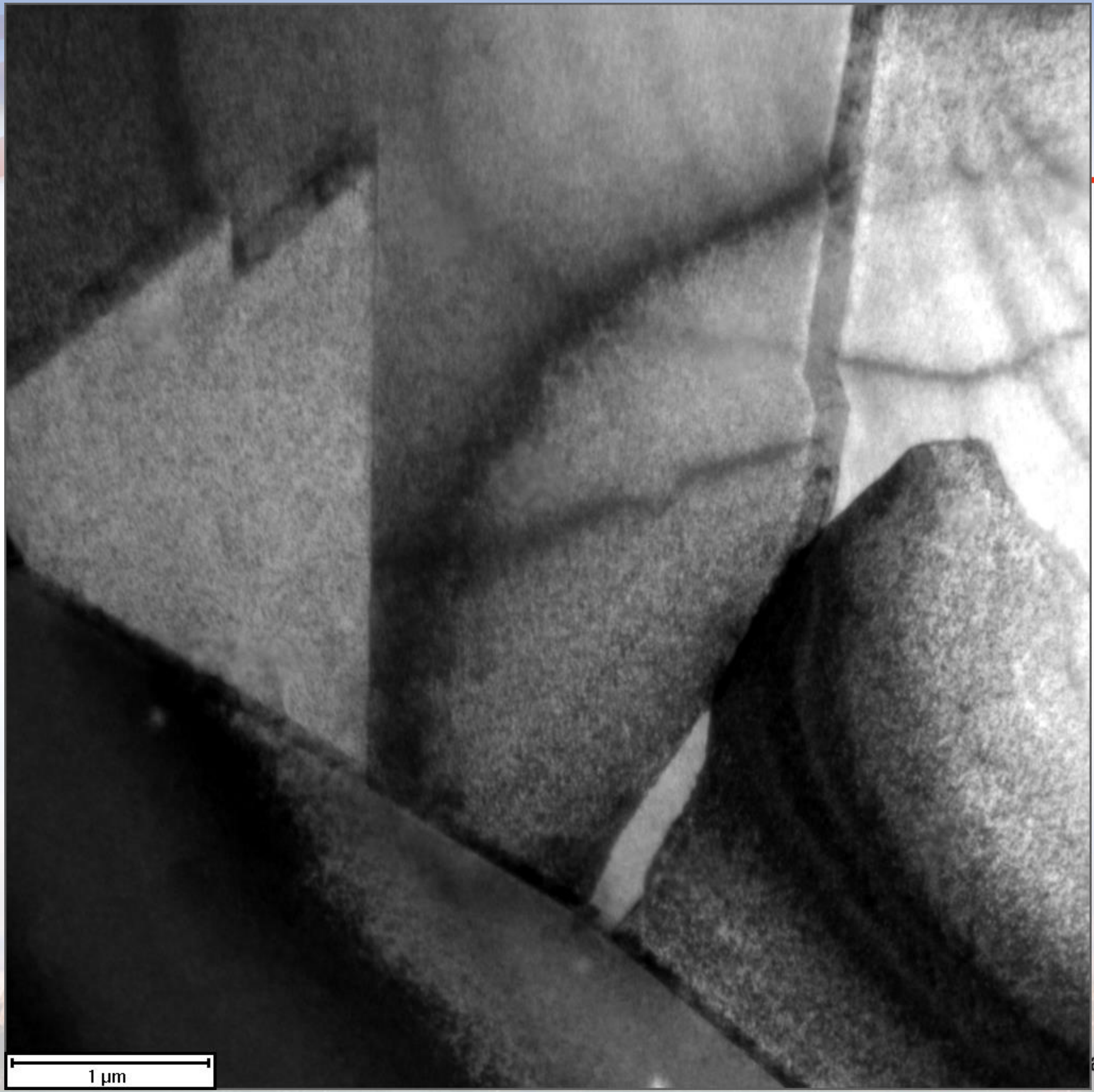


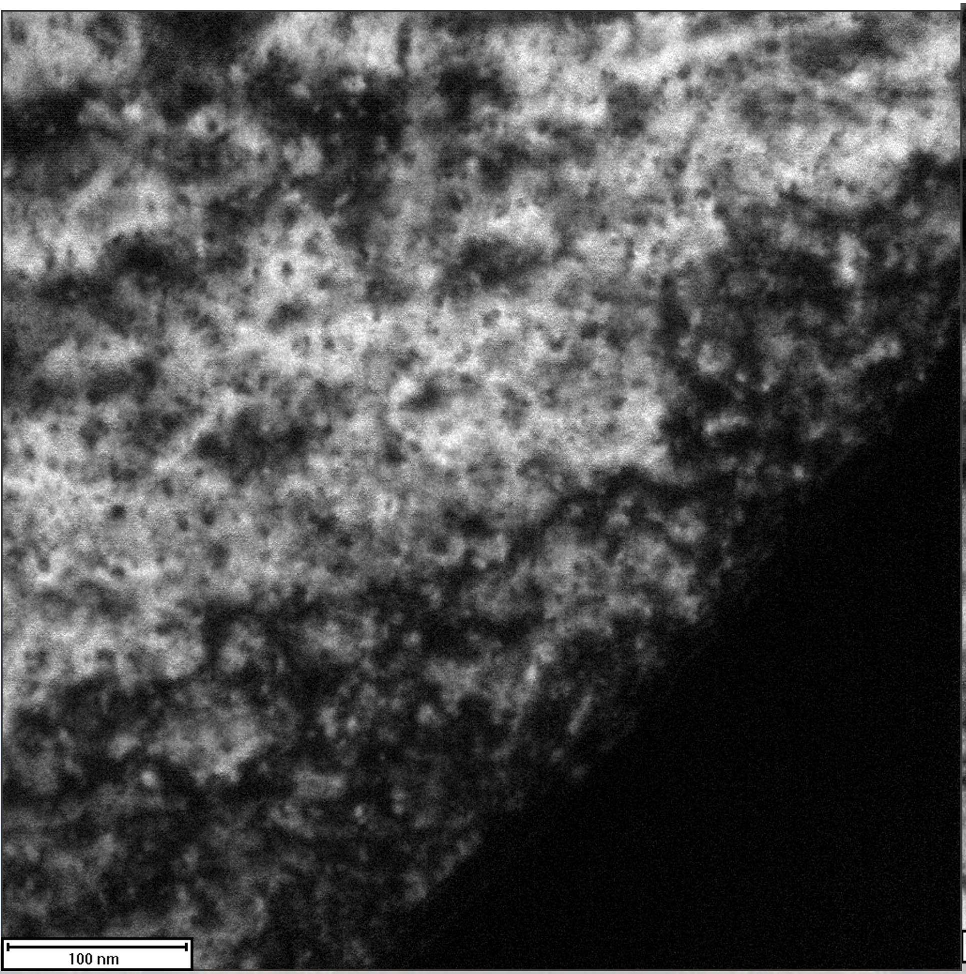

500 nm

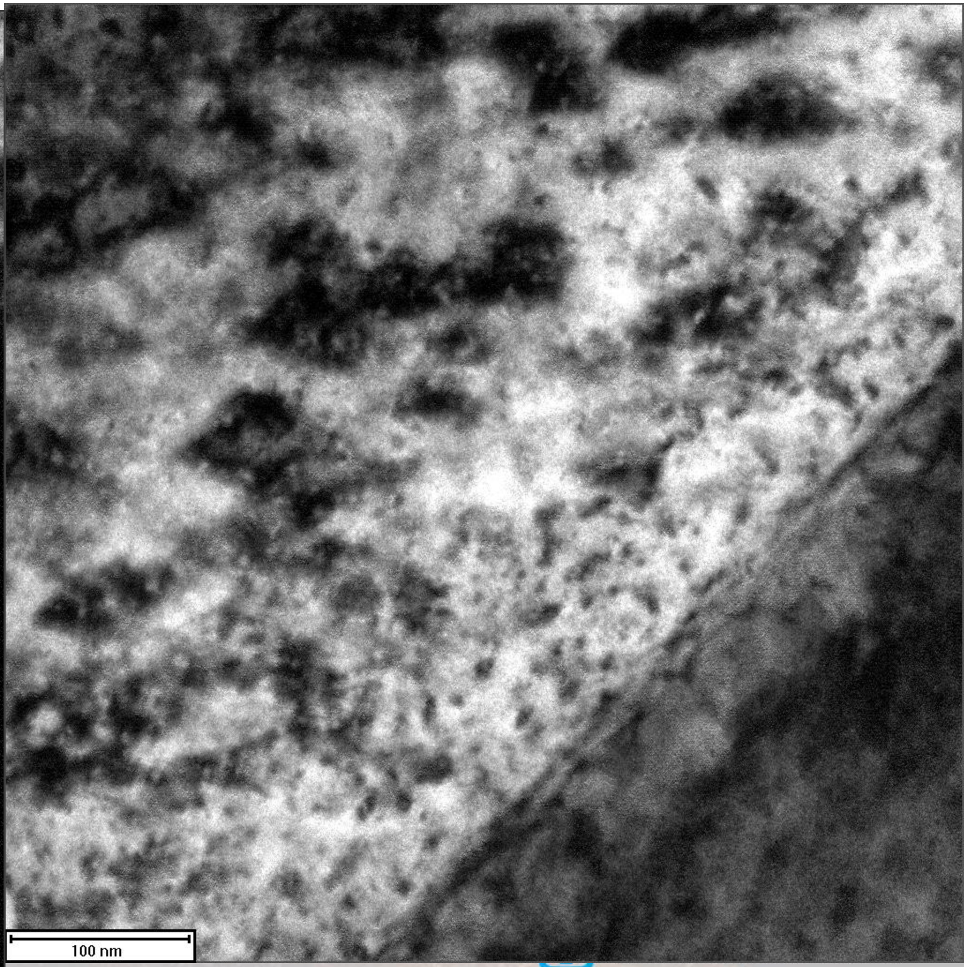
After Ni^{3+} additional irradiation





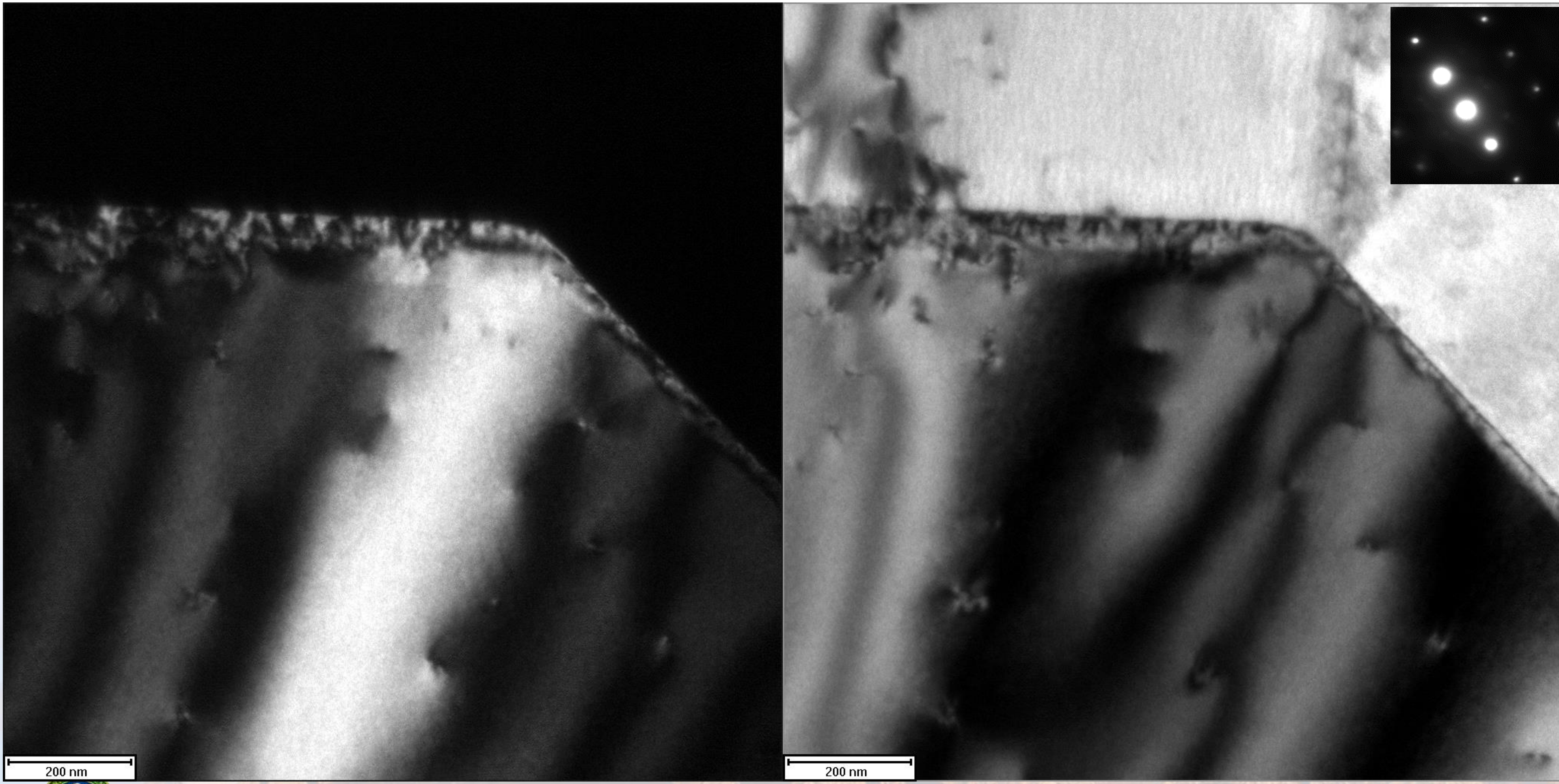

1 µm


100 nm

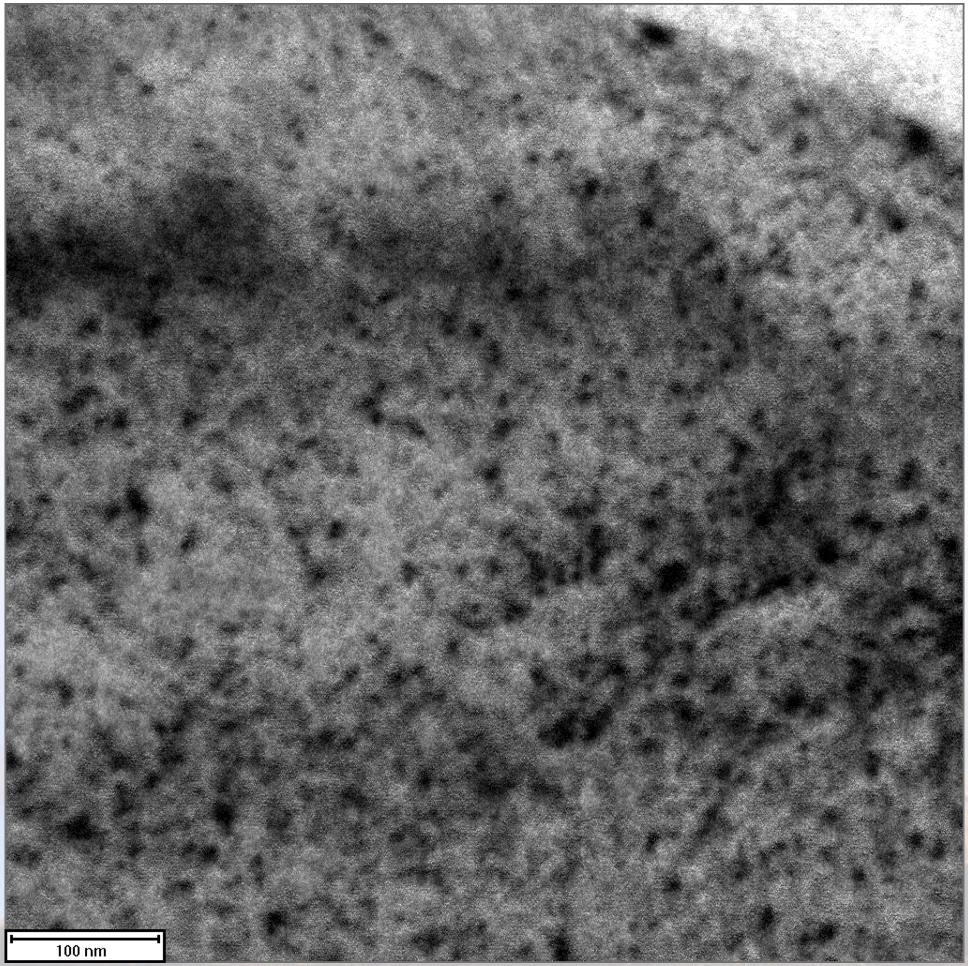

100 nm

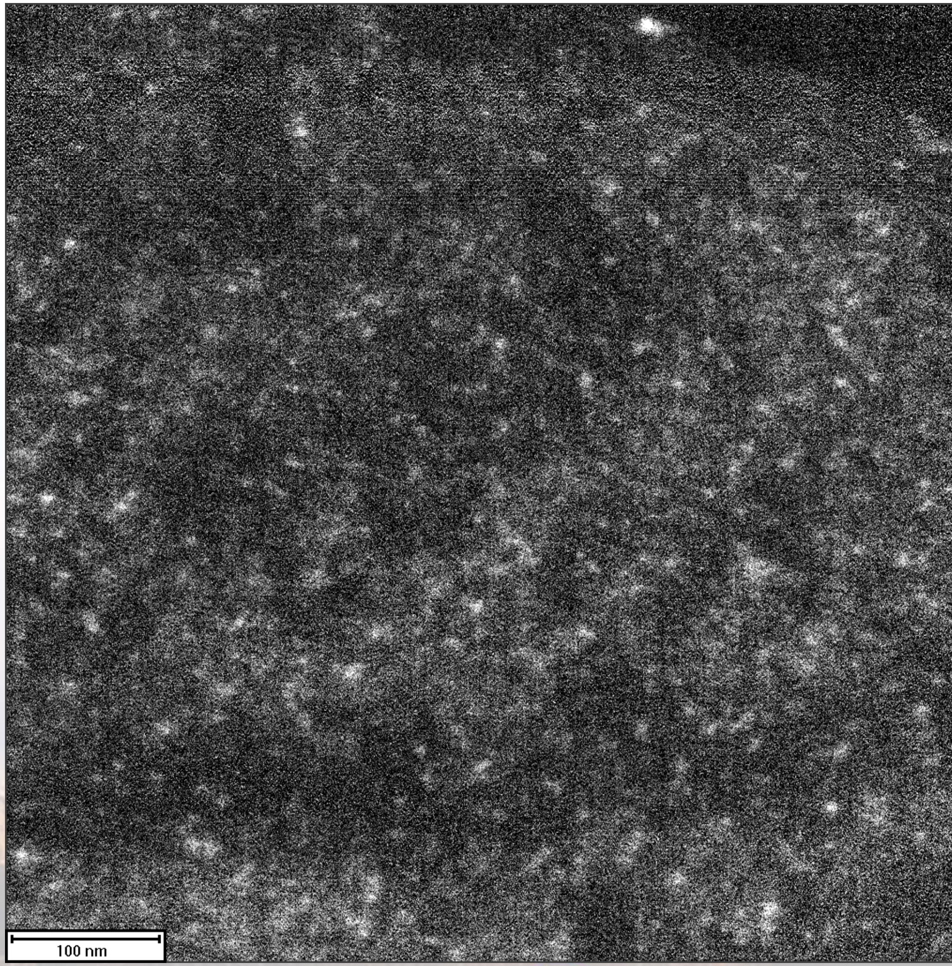
#2: Ni^{3+} then D^{+}

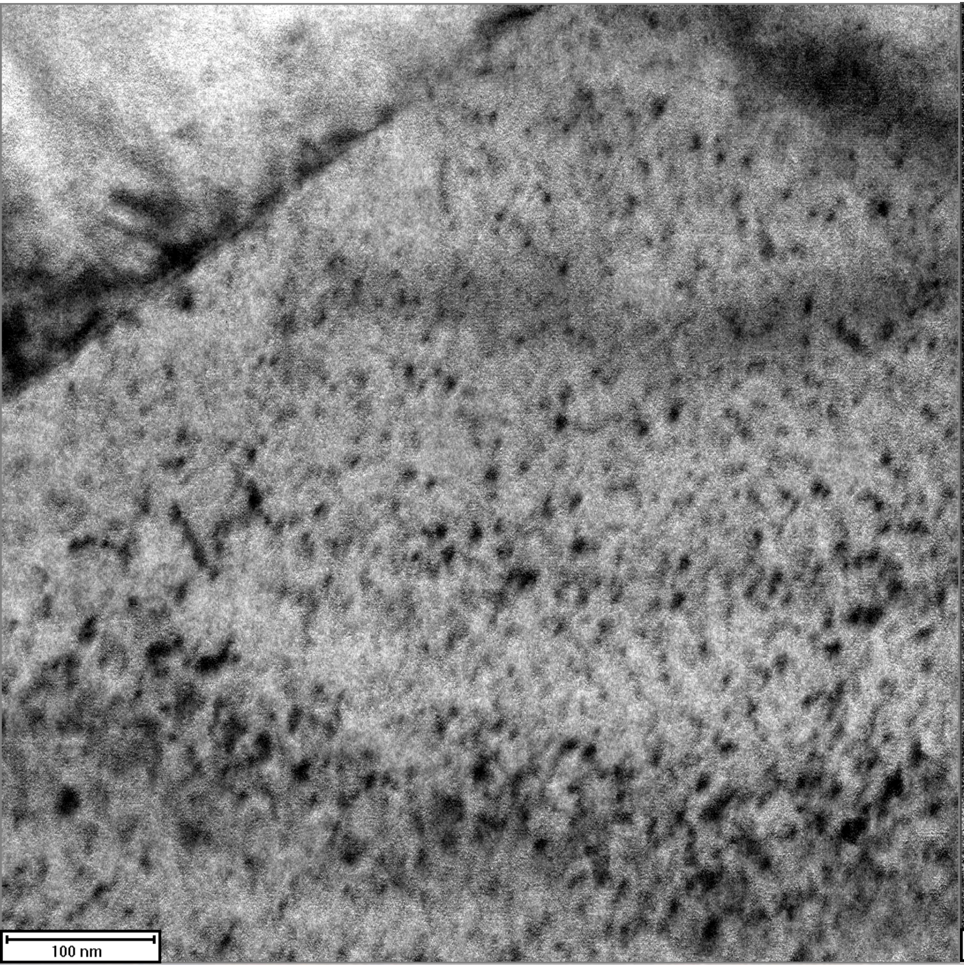
Before Irradiation



After Ni^{3+}



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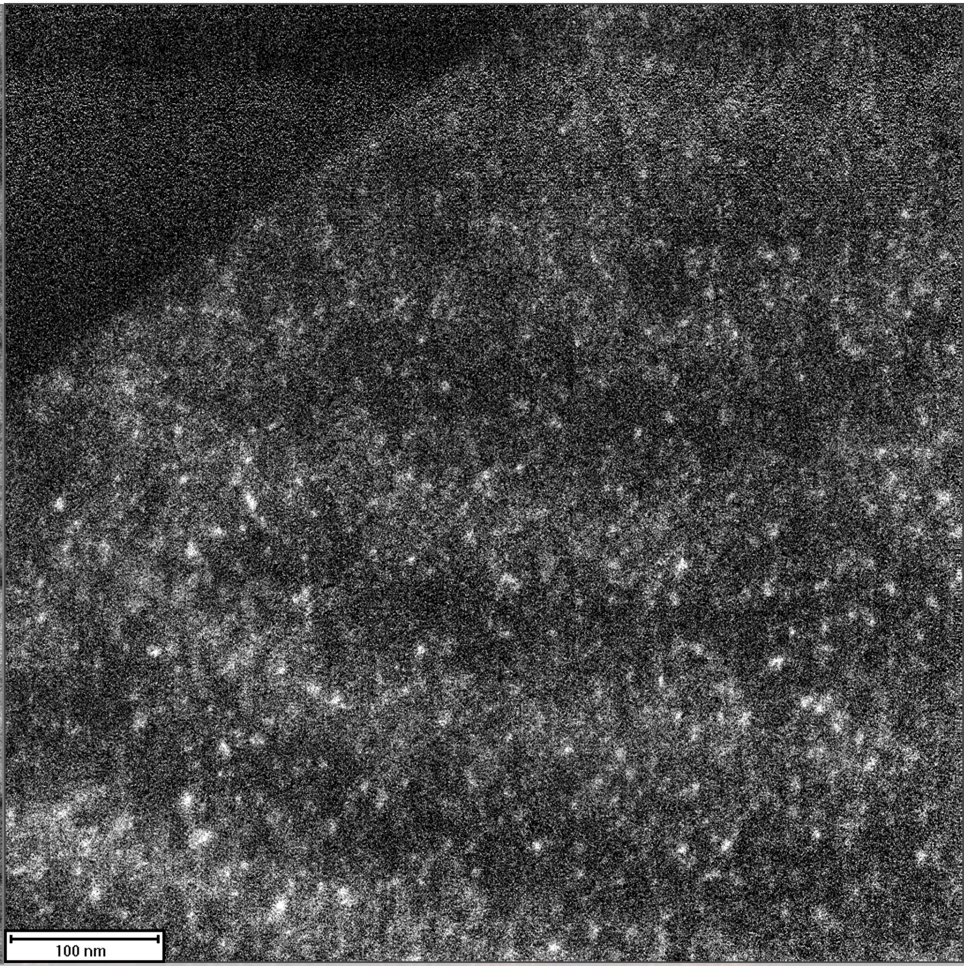
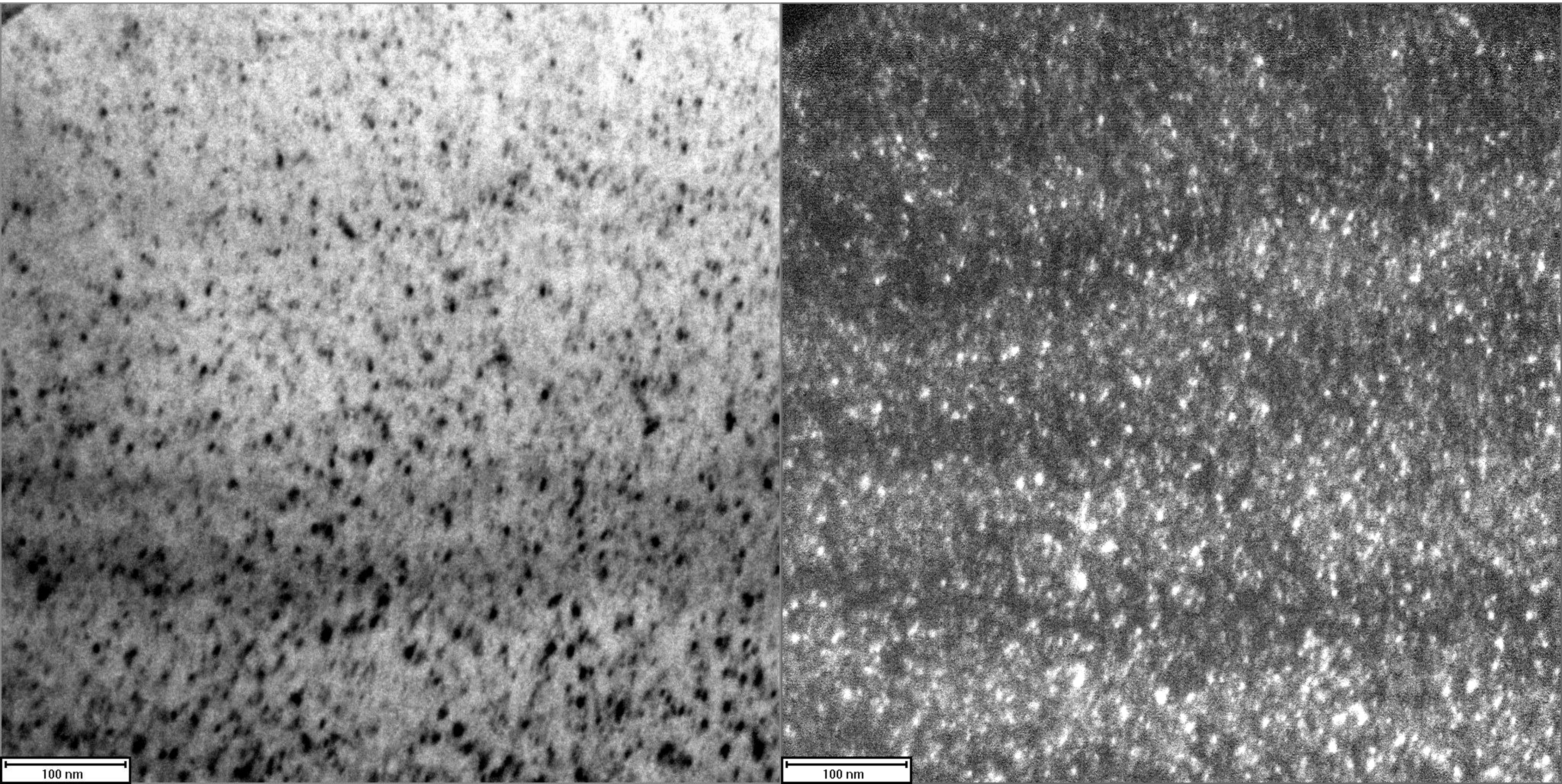
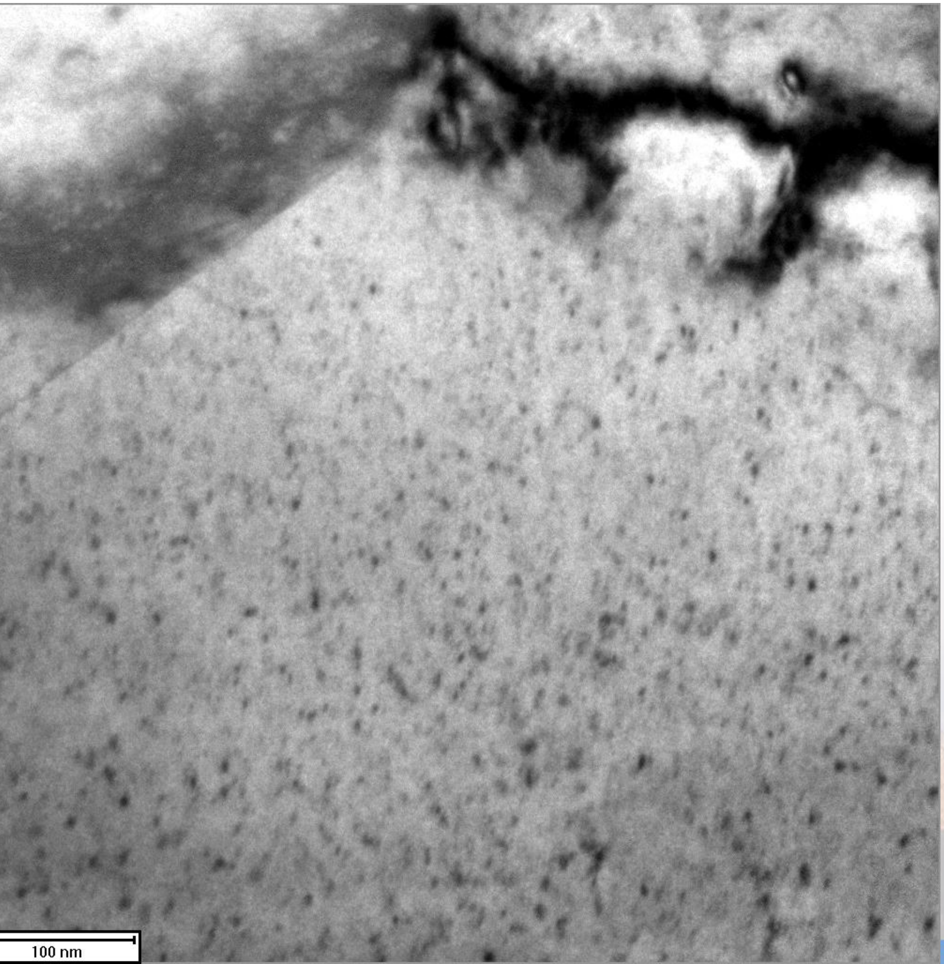
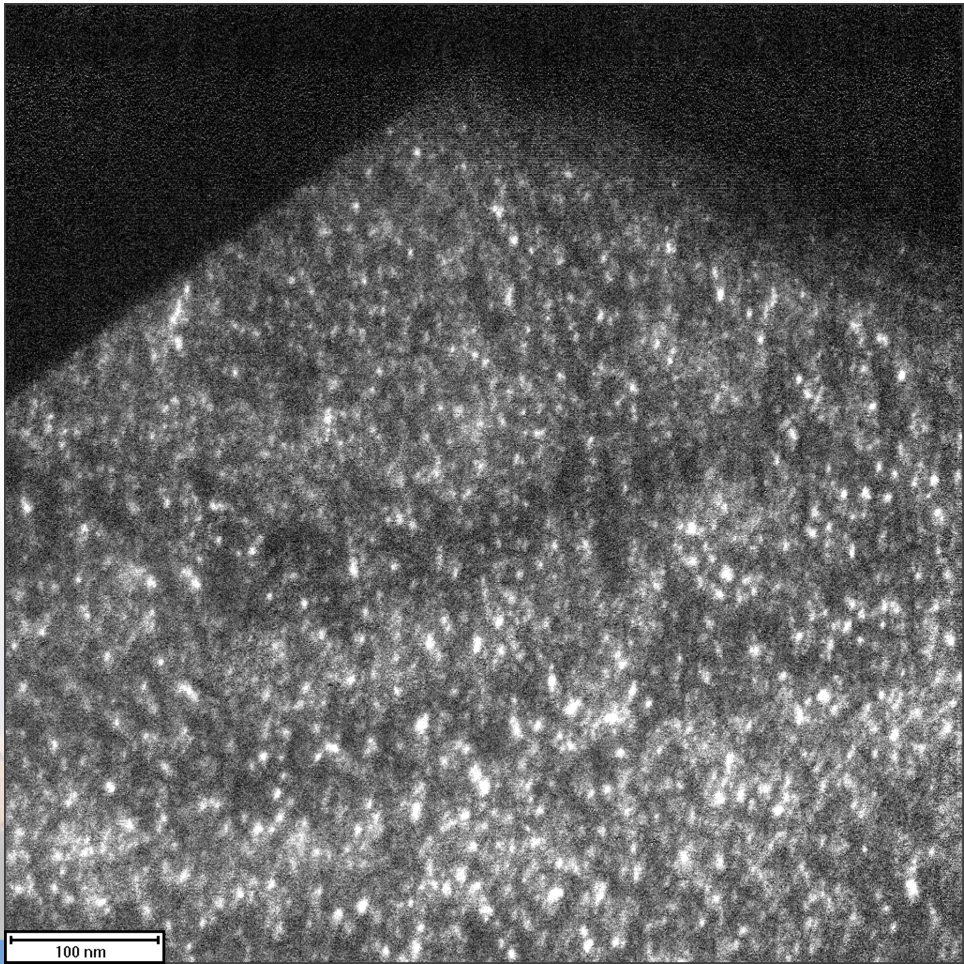

100 nm

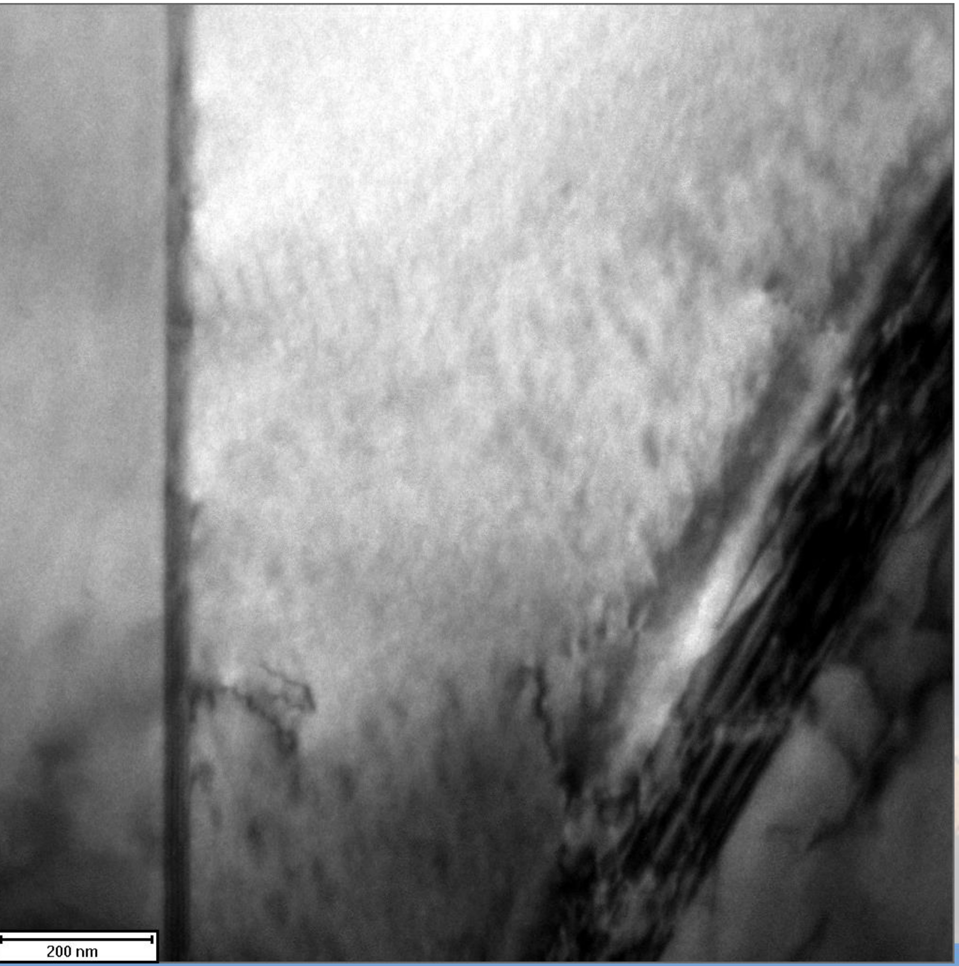
After addition of D^+



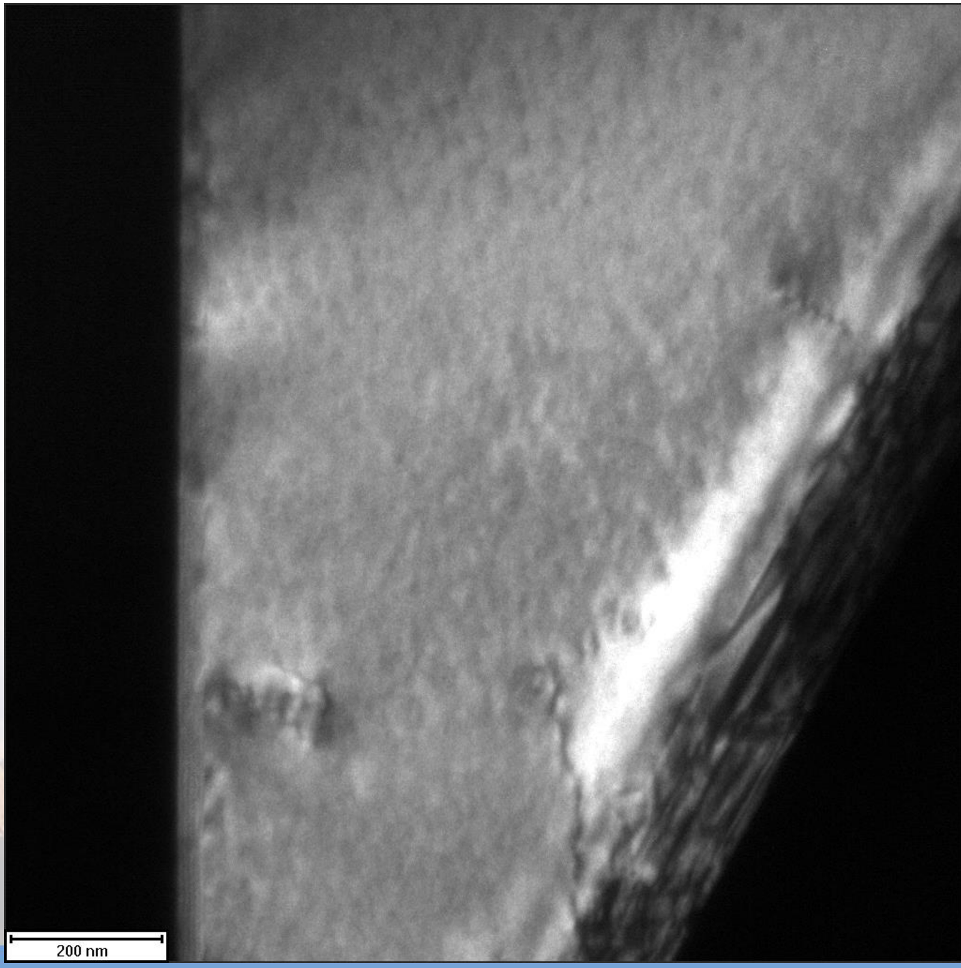

100 nm


100 nm

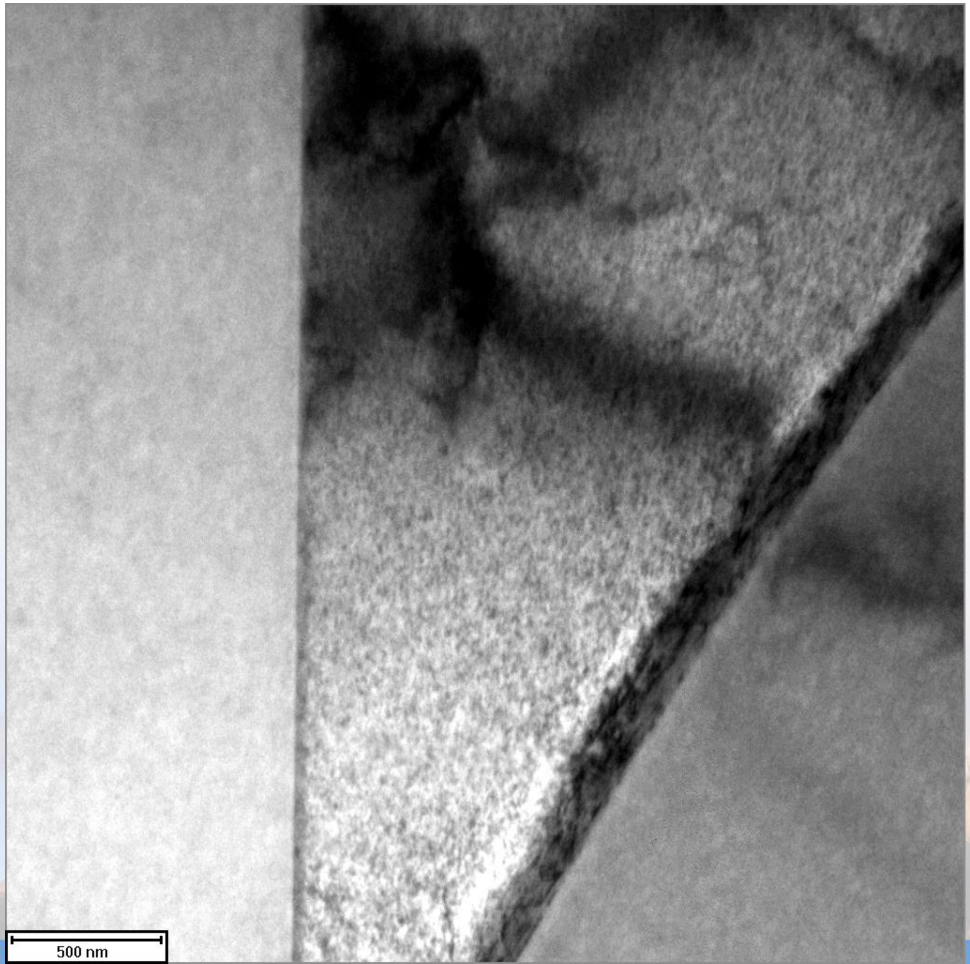
Ni^{3+} and D^{+} same time

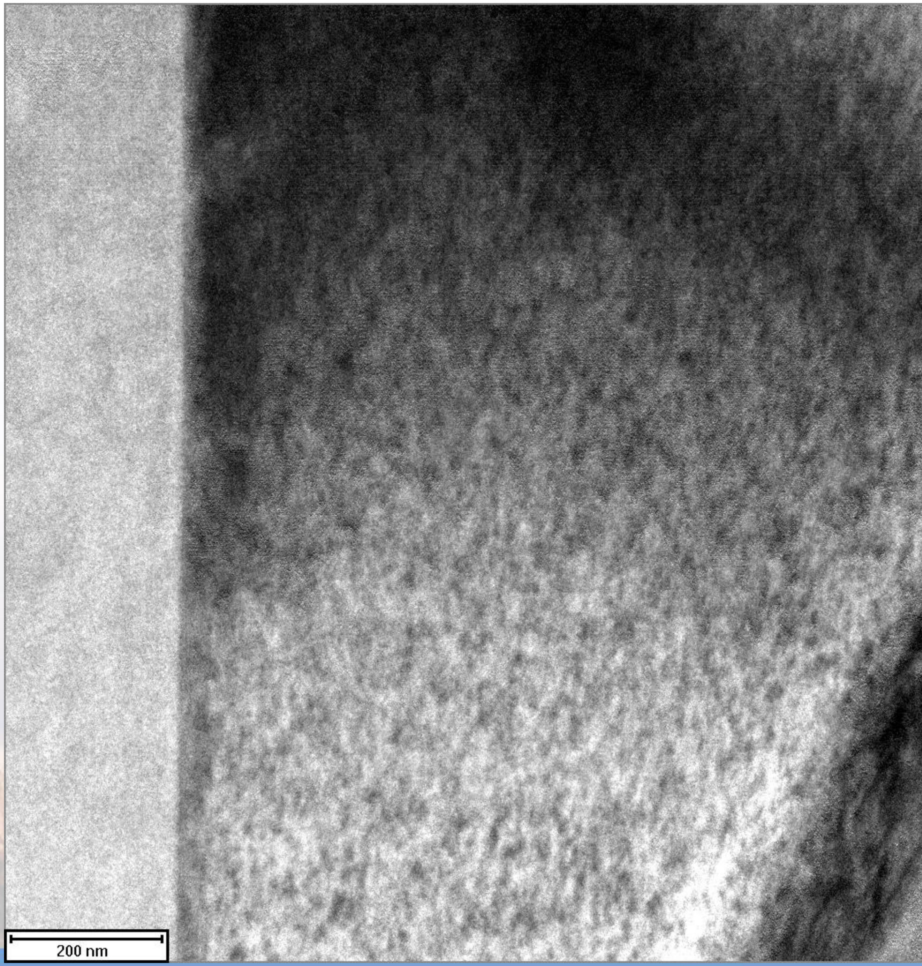


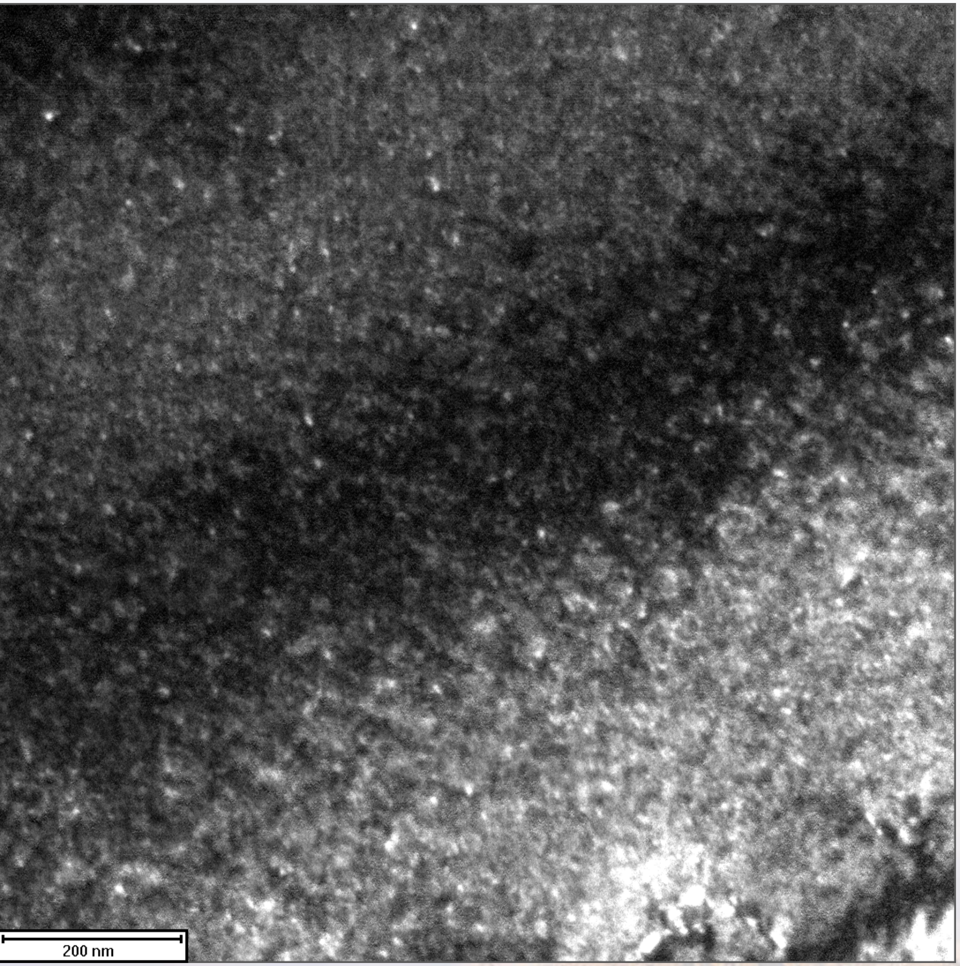
before

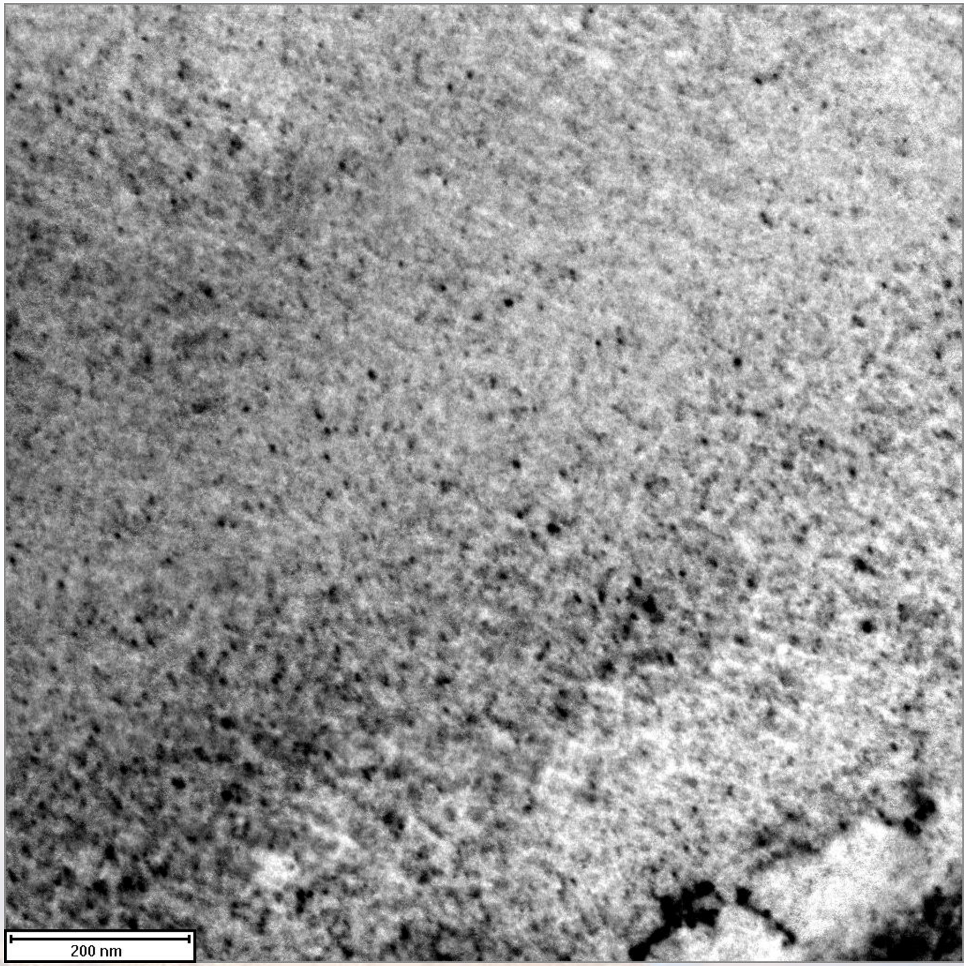


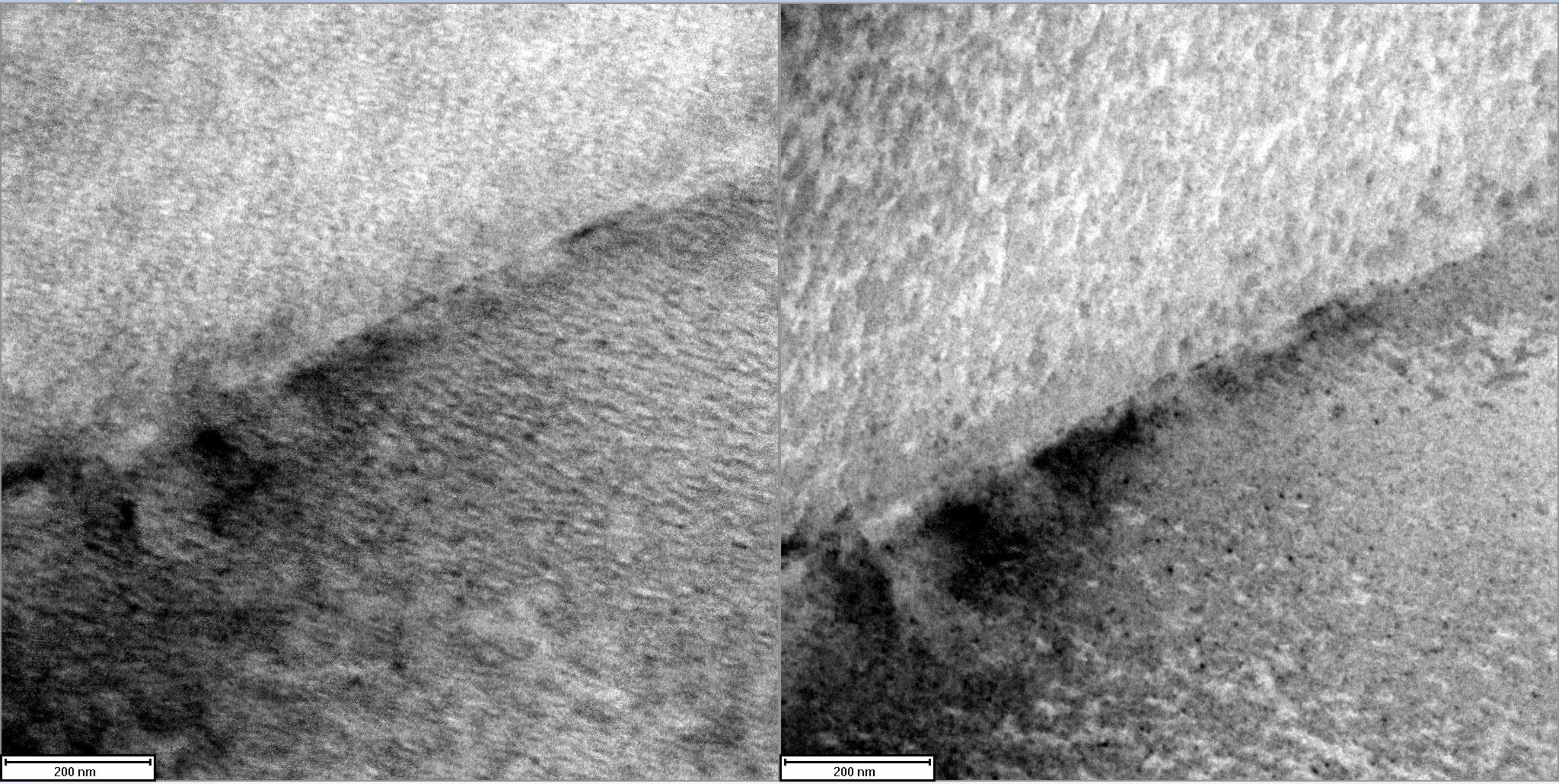
After Ni and D






200 nm


200 nm



Over and Under Focus

Straining Plan

- 1) Control
- 2) D-> Strain-> D + Strain
- 3) Ni-> Strain -> Ni + Strain
- 4) D + Ni -> Strain -> D + Ni + Strain