

EXPLORING M

D.C.

Probing grain

MICROSTRUCTURAL STABILITY

C. Bufford, B.R. Muntife

boundary stability in nanocrystalline

STABILITY OF METALS

ering, W.M. Mook, F. A.

line metals at elevated temperature

S VIA *IN SITU* TRANS

odeljawad, D.P. Adams,

es under mechanical stress and u

SMISSION ELECTRO

S.M. Foiles, and K. Ha

under irradiation using advanced T

N MICROSCOPY

ttar

EM methods

Capabil-

Sandia's *In Situ* Ion Irradiation TEM (I³TEM) Facility

- ◆ 200 kV JEOL 2100 TEM with two attached ion sources providing a large range of *in situ* sample manipulation

Schematic of the TEM, accelerators, and beamline hardware.

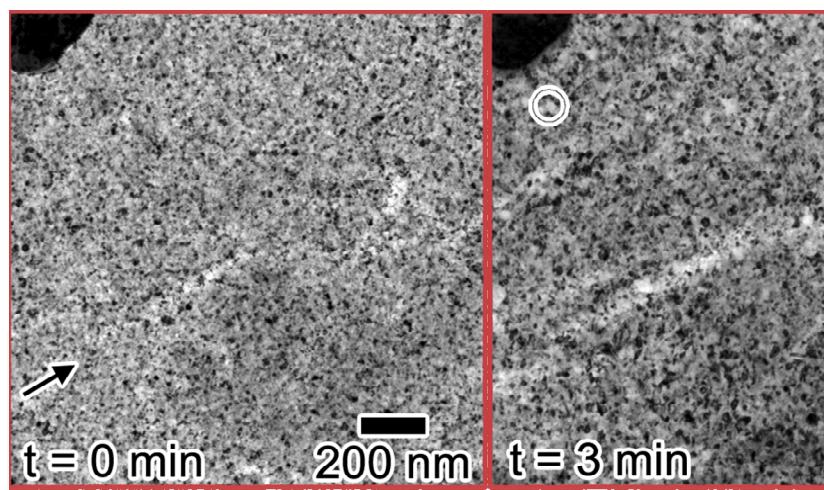


Boundary stability in nanocrystallites radiation

/
ed ion accelerators and a
on capabilities

Annealing

- ◆ Microstructure evolution
- ◆ Quantifiable grain size

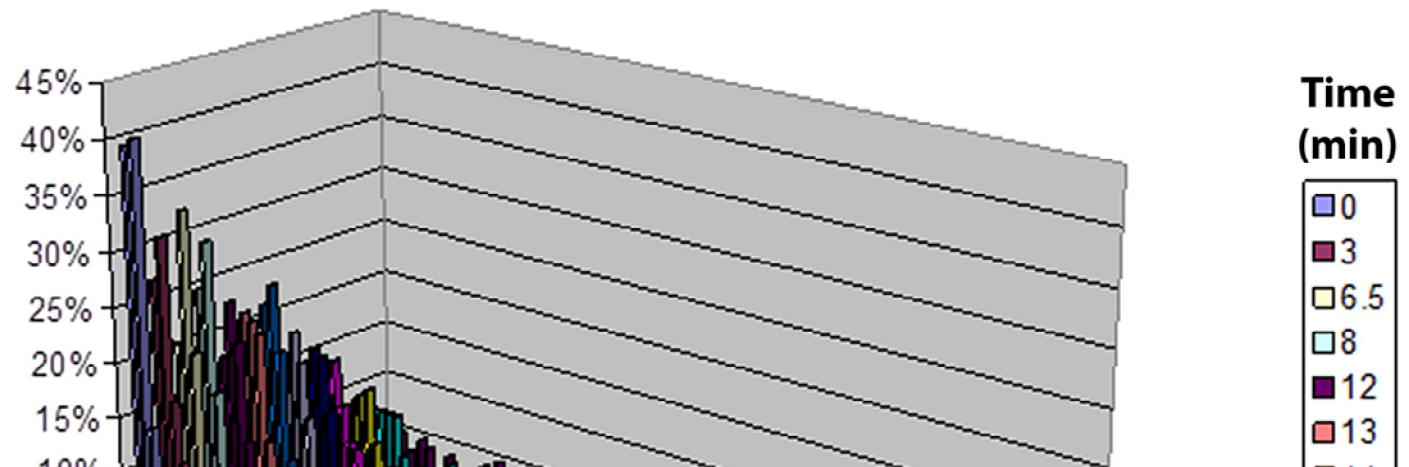
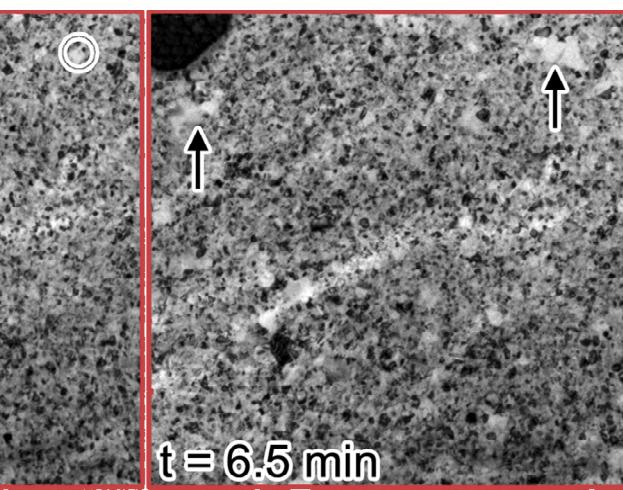


line metals at elevated temperature

Thermal g and Grain Growth

olution in the TEM at temperatures up to 1000 °C

n size and crystallographic orientation changes



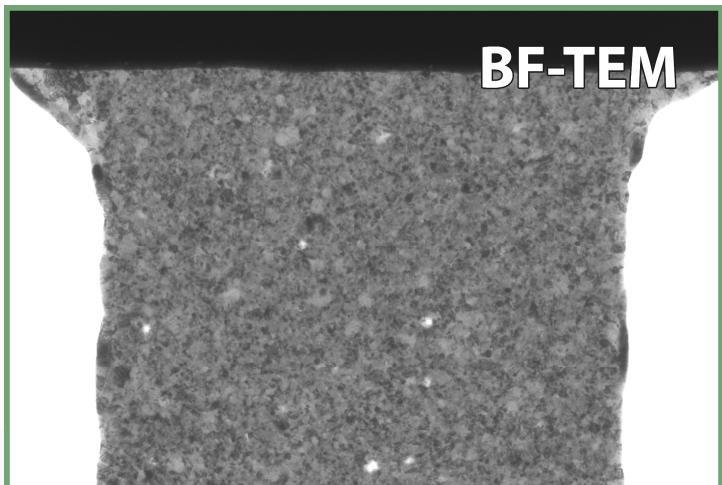
es, under mechanical stress, and un

Mechanical

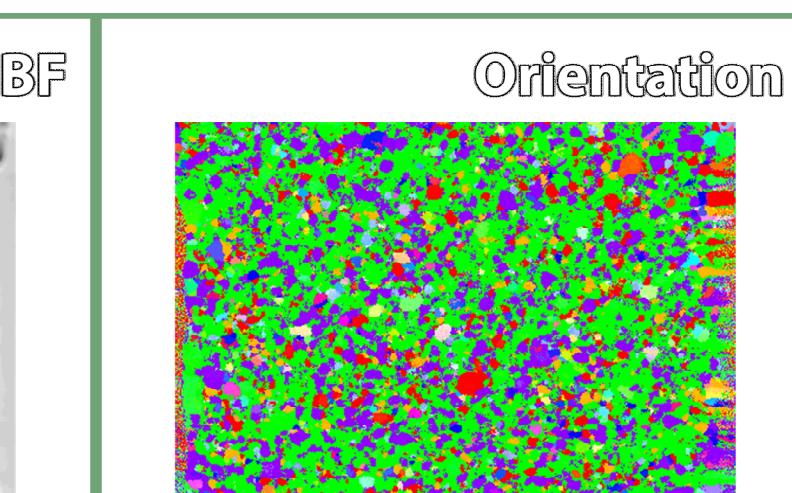
Cyclic Loading and Fracture

- ◆ Quantitative tension and compression testing of nanocrystalline Cu
- ◆ Texture and structure changes tracked by orientation mapping

Nanocrystalline Cu tension specimen shown by conventional TEM and orientation mapping before and after cyclic loading.



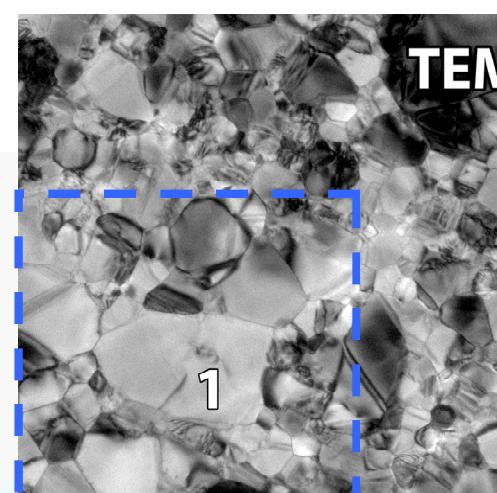
Radiation-Induced Phase Transitions in Nanoscale Specimens



Radiation-Induced Phase Transitions

- ◆ Characterizing the same induced phase transitions
- ◆ Coordinated phase field modeling

The same area of a nanocrystalline Au sample shown by bright-field TEM micrographs and orientation



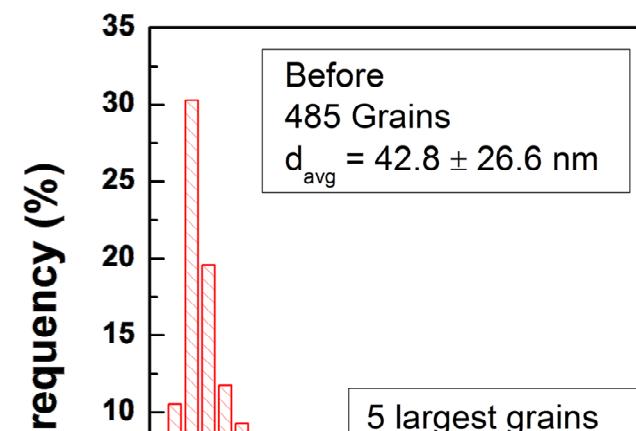
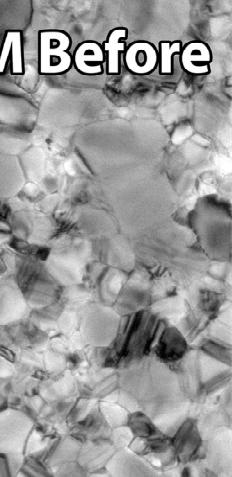
EMI methods.

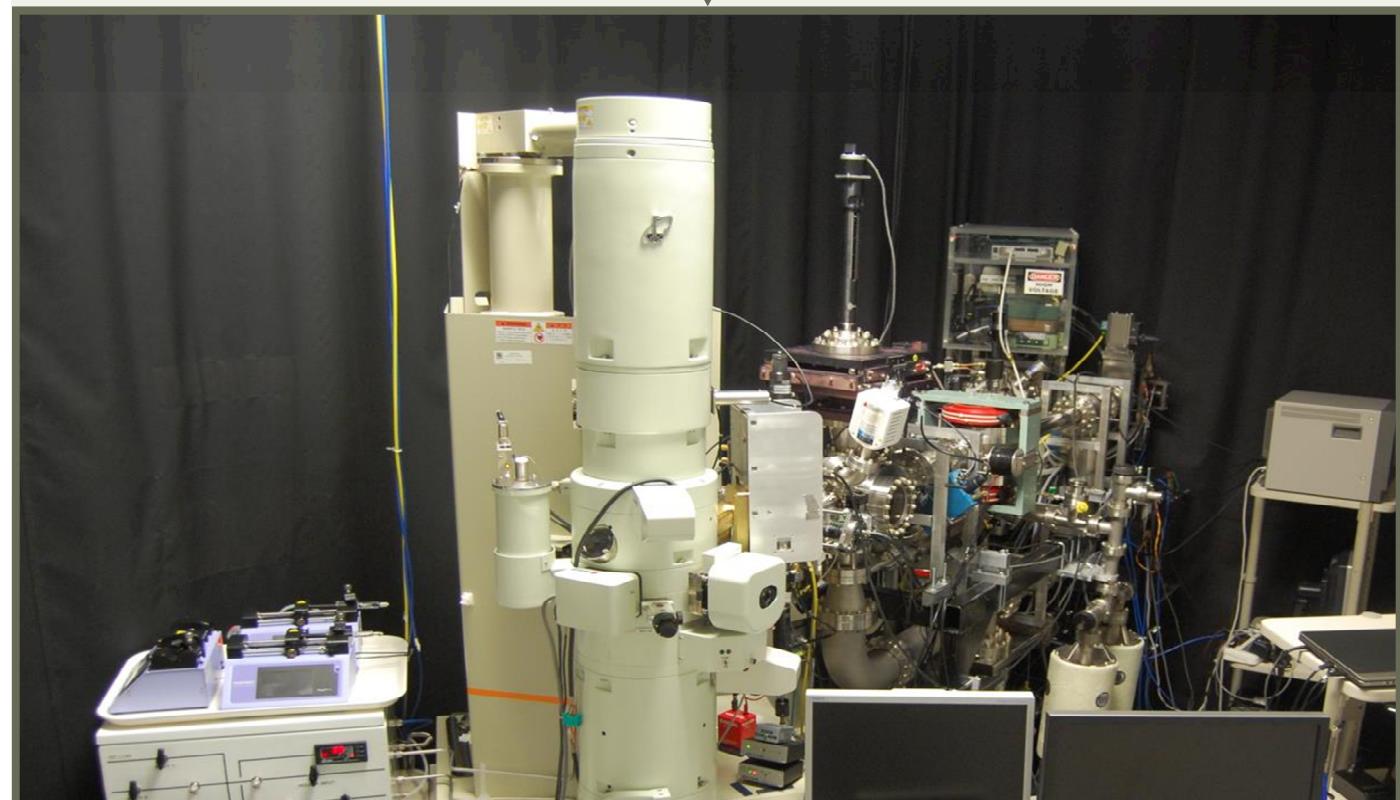
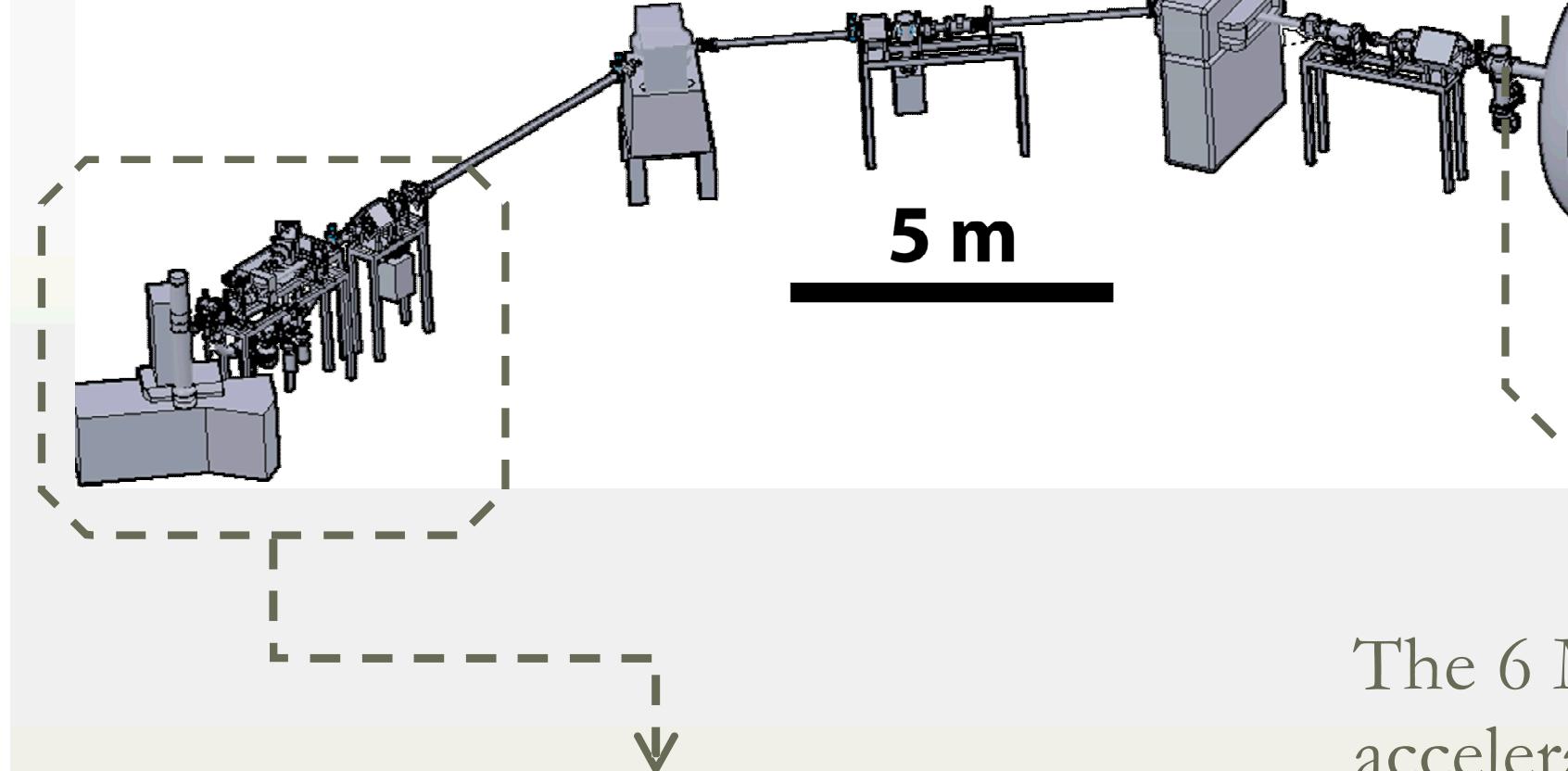
radiation

uced Grain Growth

lividual grains after increasing ion doses

odeling





The 6 L
accelera
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highly
periodi

| 1 | 2 |
|----|------------|
| H | Boron |
| Li | 4 |
| Na | Magnesium |
| K | 12 |
| Rb | Sodium |
| Cs | 20 |
| Fr | 38 |
| | 57-70 |
| | * |
| | 57-102 |
| | ** |
| | 174 |
| | Lawrencium |
| | Lutetium |

MV EN Tandem
ator with ions deliv-
the TEM to date
hted green in the
c table below.

| Element Name | Atomic number | Symbol |
|--------------|---------------|--------|
| Hydrogen | 1 | H |

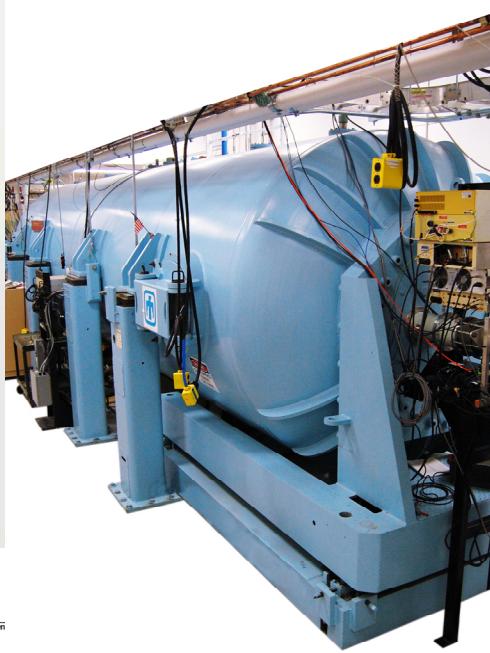
| Element Name | Atomic number | Symbol |
|--------------|---------------|--------|
| Helium | 2 | He |

| Element Name | Atomic number | Symbol |
|--------------|---------------|--------|
| Lithium | 3 | Li |

| Element Name | Atomic number | Symbol |
|--------------|---------------|--------|
| Thorium | 4 | Th |

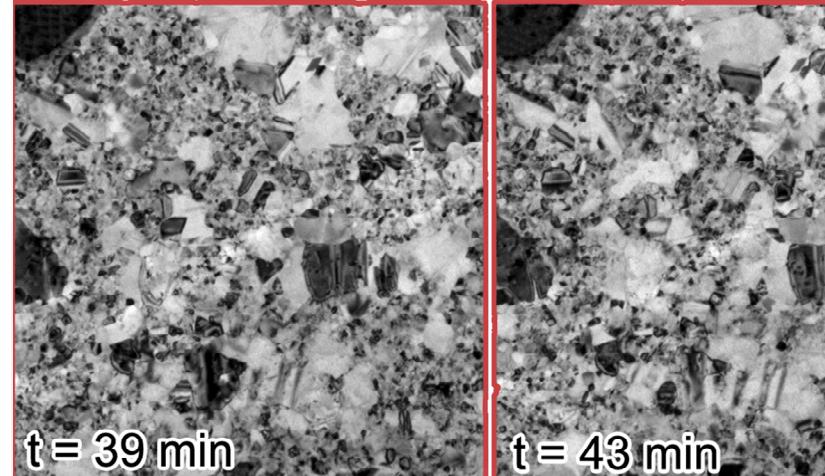
| Element Name | Atomic number | Symbol |
|--------------|---------------|--------|
| Neon | 5 | Ne |

| Element Name | Atomic number | Symbol |
|--------------|---------------|--------|
| Hydrogen | 1 | H |



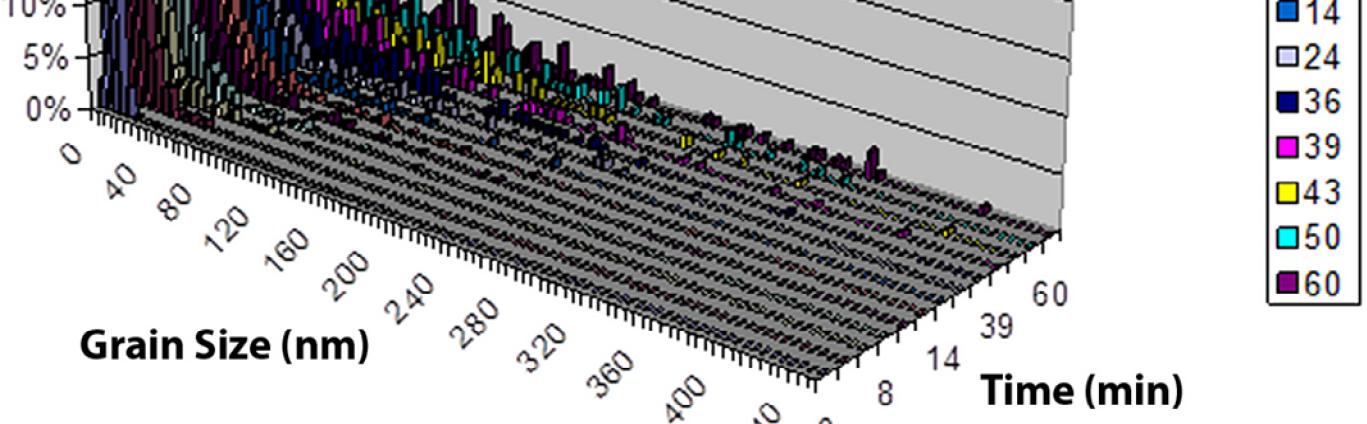
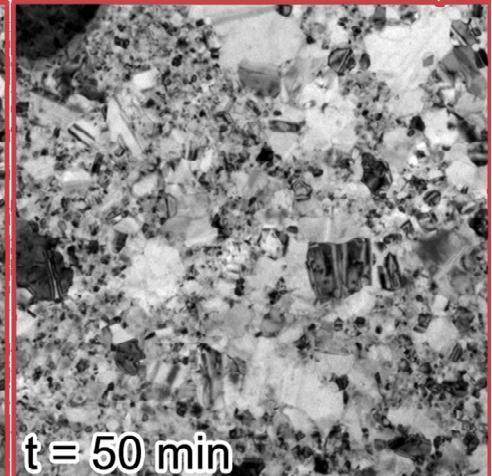
| Element Name | Atomic number | Symbol |
|--------------|---------------|--------|
| Hydrogen | 1 | H |

3 4 5 6 7 8 9 10 11 12 13 14 15

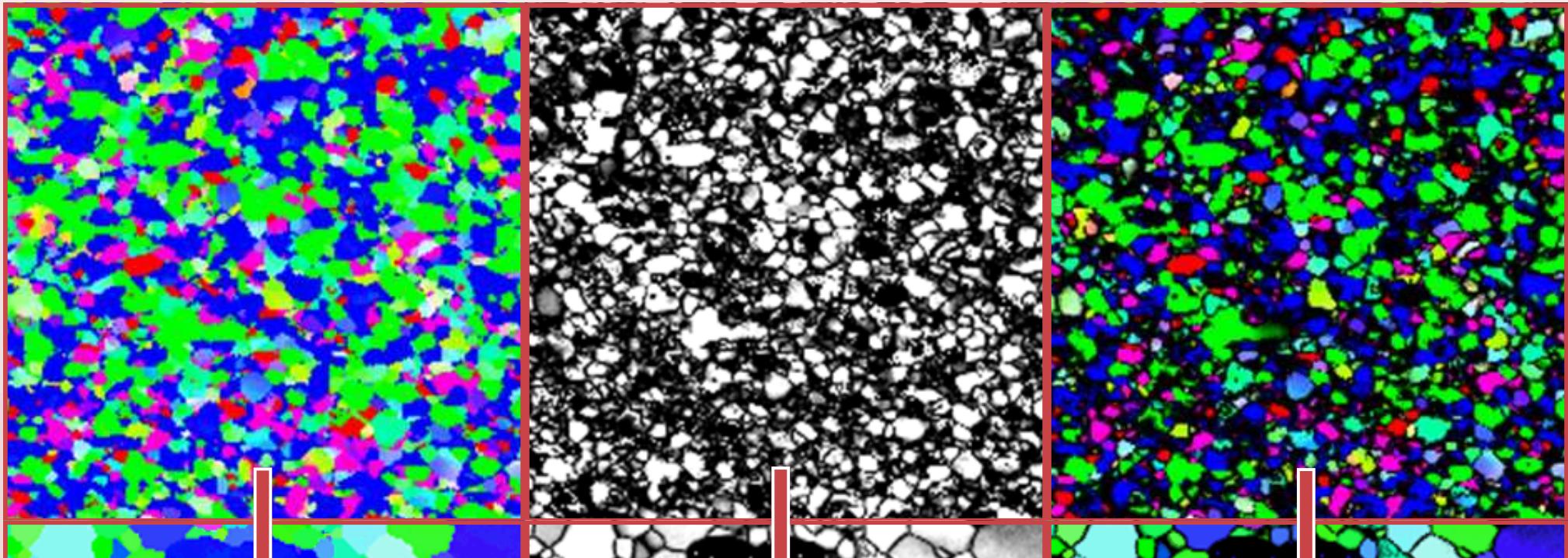


(Above) Nanocrystalline Thesis, 2009). There were counted over 13 time steps.

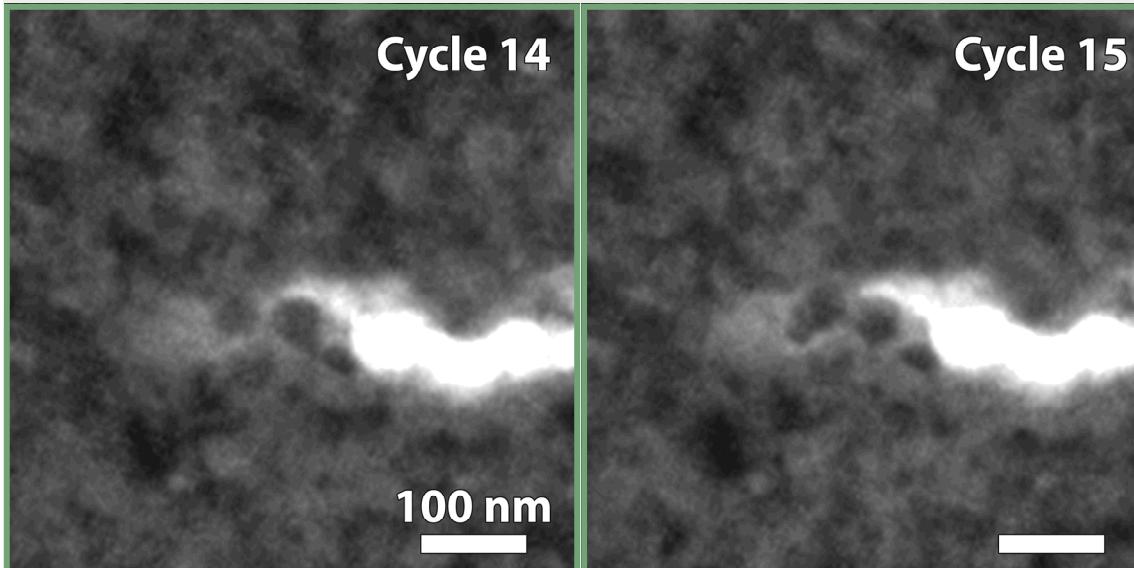
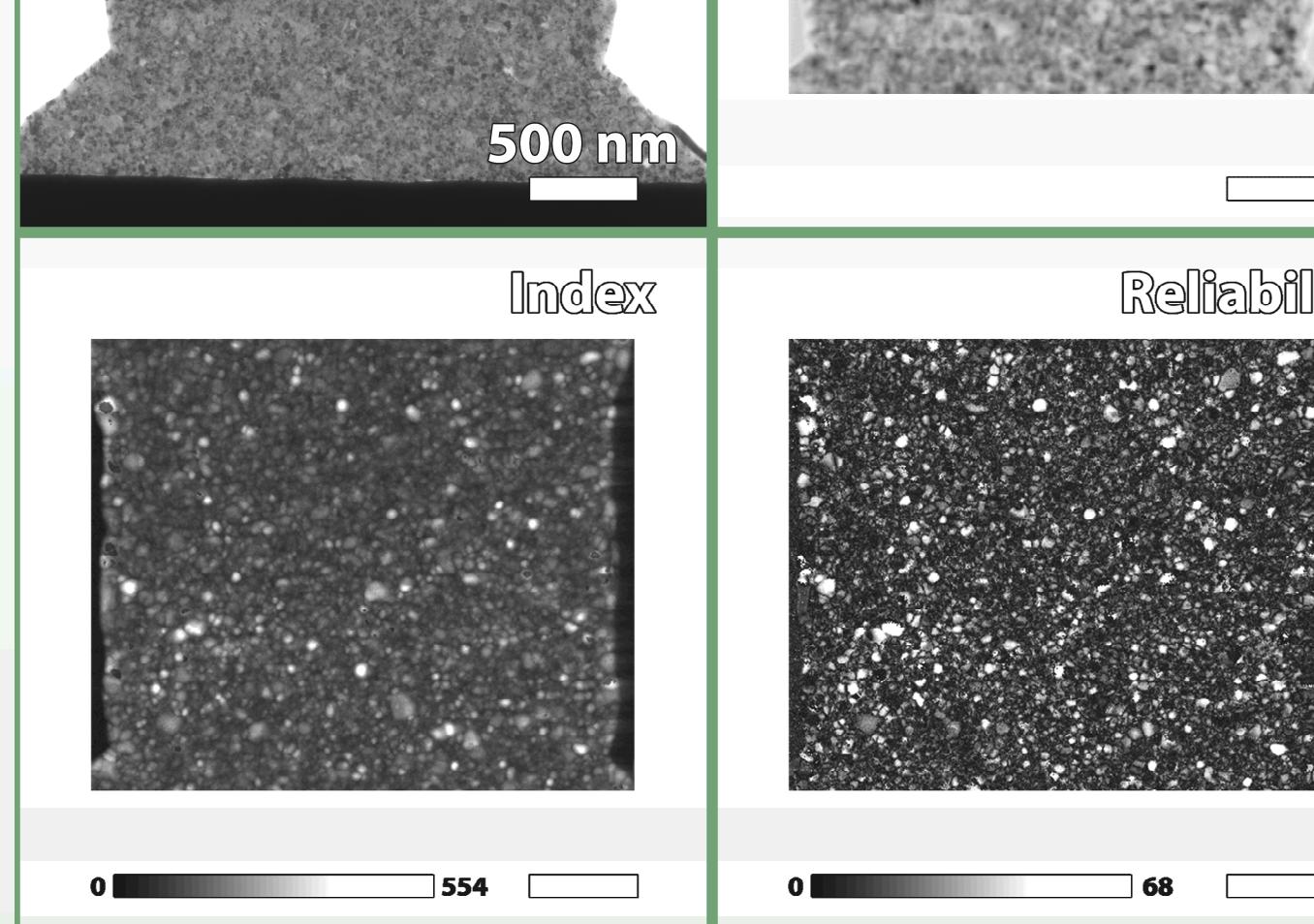
(Right) Nanocrystalline Fe before and after *in situ* annealing to 550 °C shown by orientation maps. This analysis of 1097 initial grains in a 1 μm × 1 μm area was performed in minutes.



Ni annealed *in situ*, and analyzed by hand (from Hattar Ph.D.). There 435 grains counted initially, and a total of 3408 grains now. This experiment and analysis represent weeks of work.

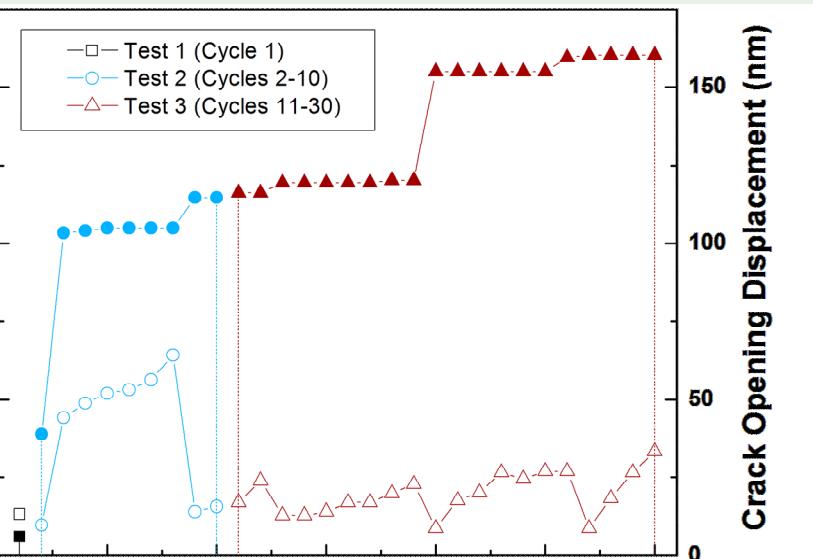
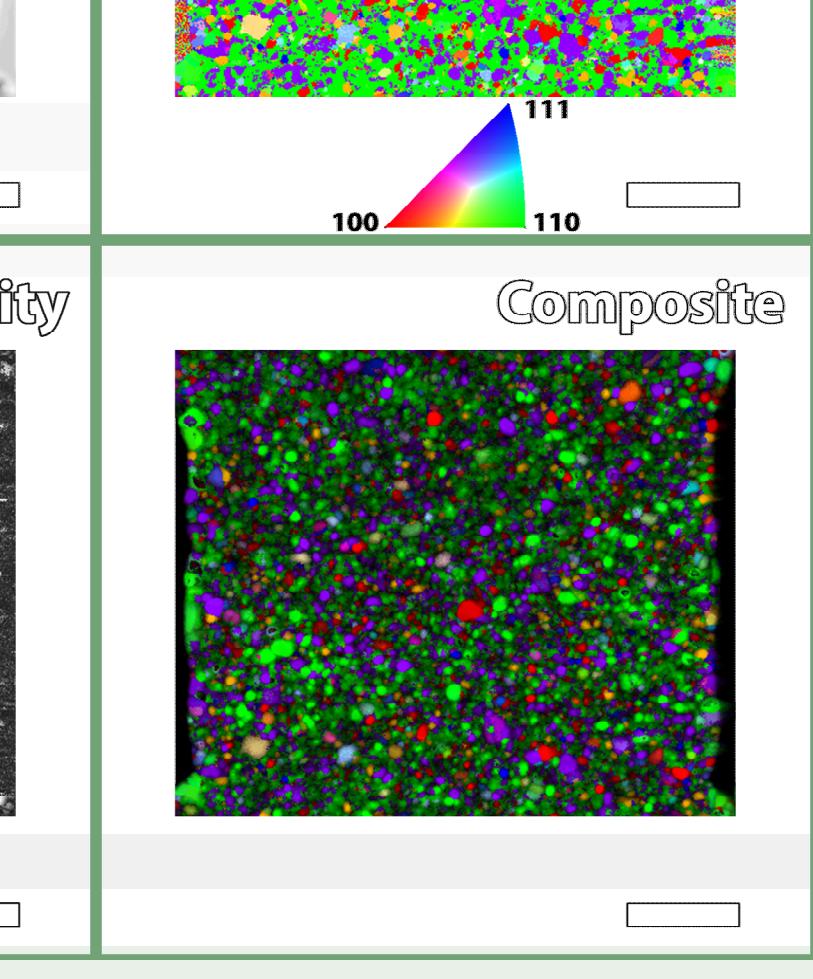


before deformation. These include a virtual STEM image, orientation mapped to the inverse pole figure, correlation and reliability indices, and a composite image of orientation and correlation index information.

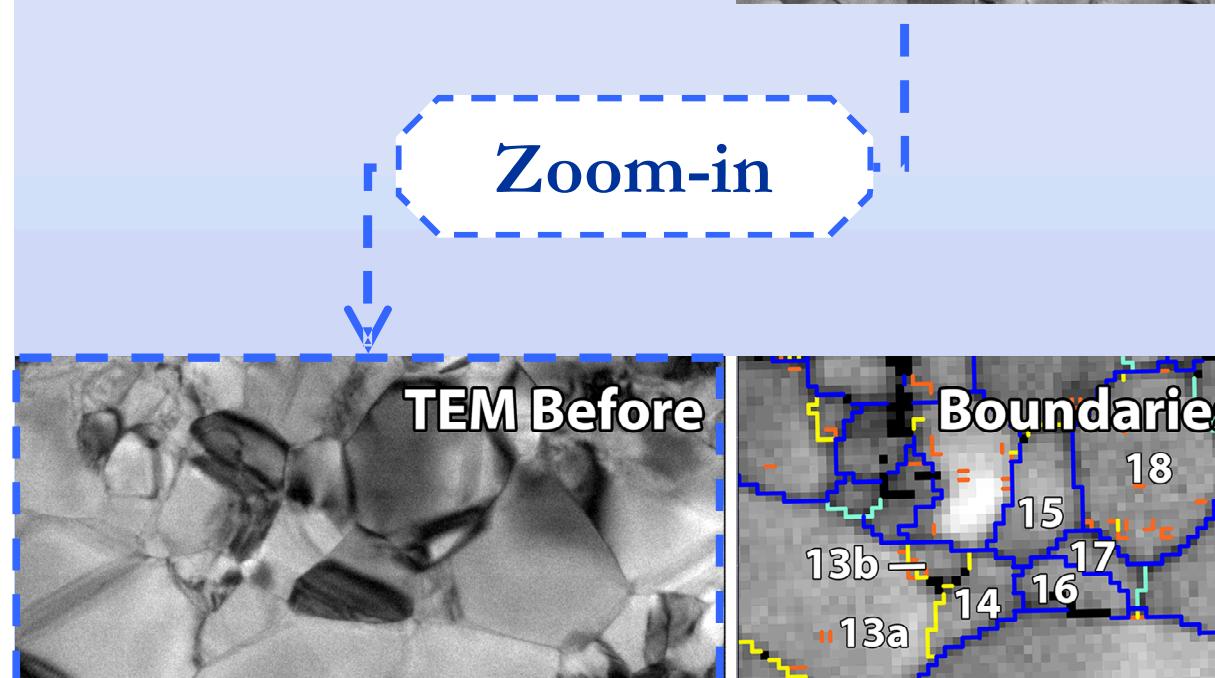


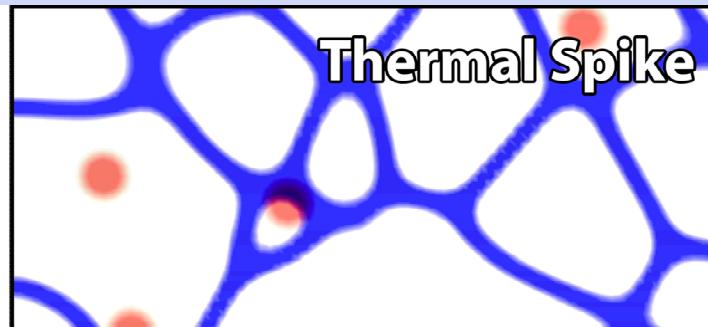
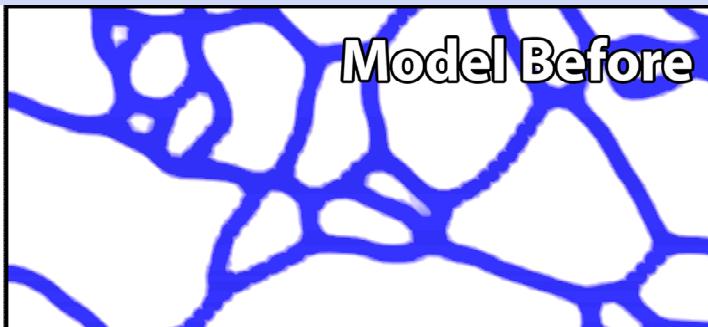
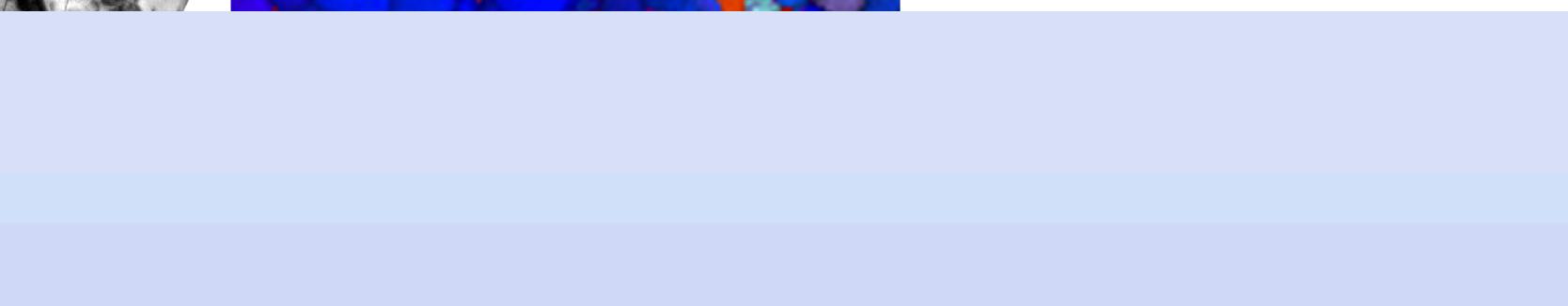
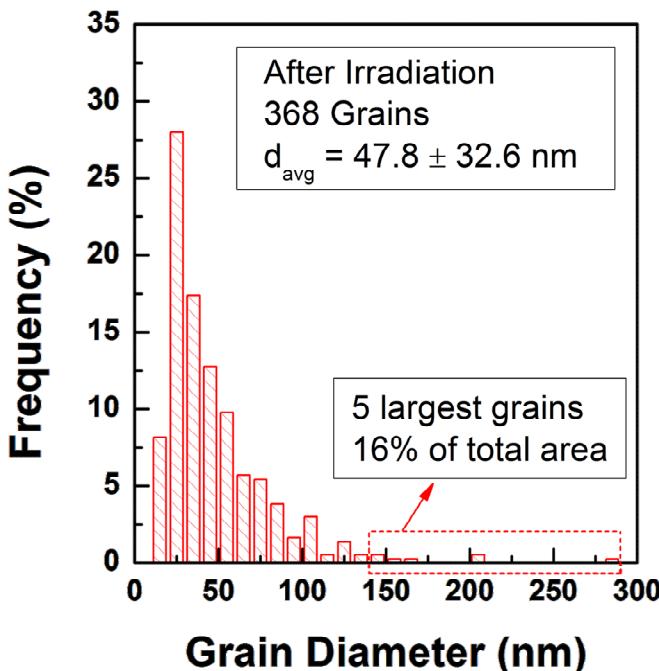
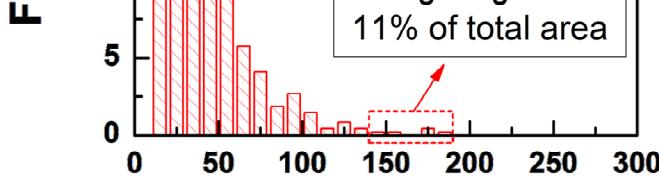
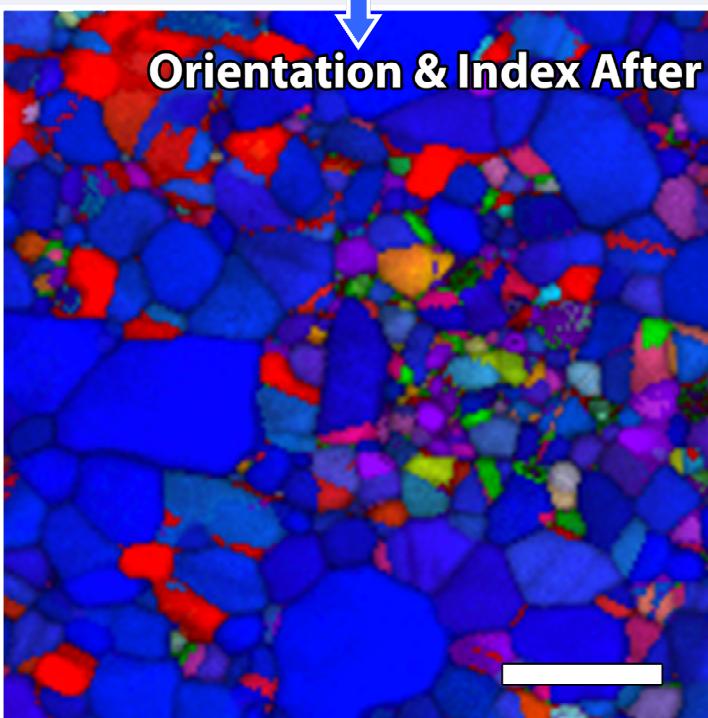
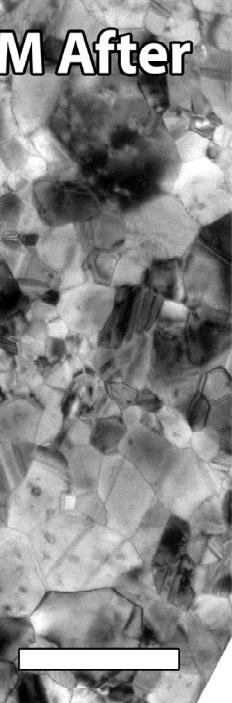
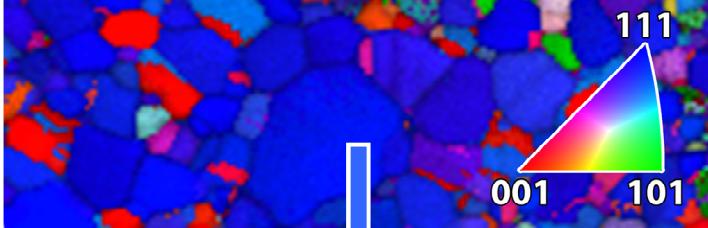
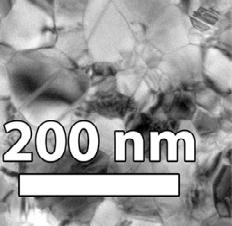
(Left) Increasing crack tip opening displacement as seen *in situ* in the TEM during low-





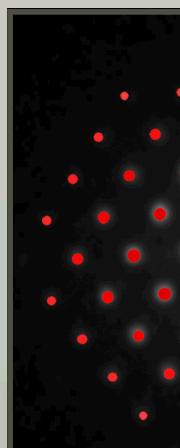
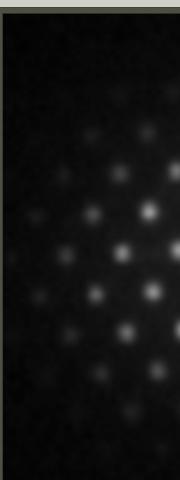
Orientation and index maps collected before and after *in situ* TEM ion irradiation with 10 MeV Si³⁺ to a dose of 2×10^{15} cm⁻². The maps display combined orientation and index data. Corresponding grain size histograms.





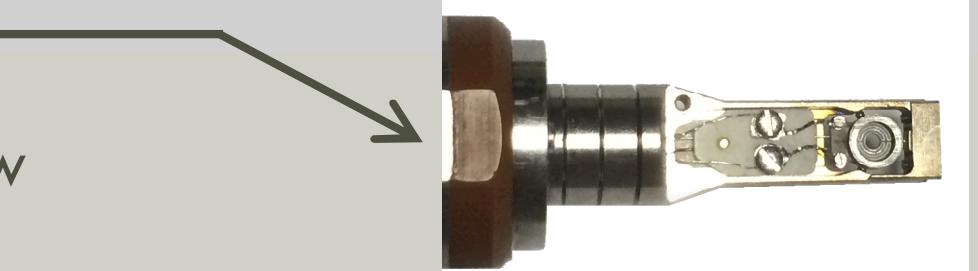


Or
♦ P
b



In Situ Manipulation

- ♦ Heating
- ♦ Liquid flow
- ♦ Gas flow + heating



- ♦ Double tilt/rotate
- ♦ Single tilt tomography

- ♦ Straining

- ♦ Quantitative
nanoindentation

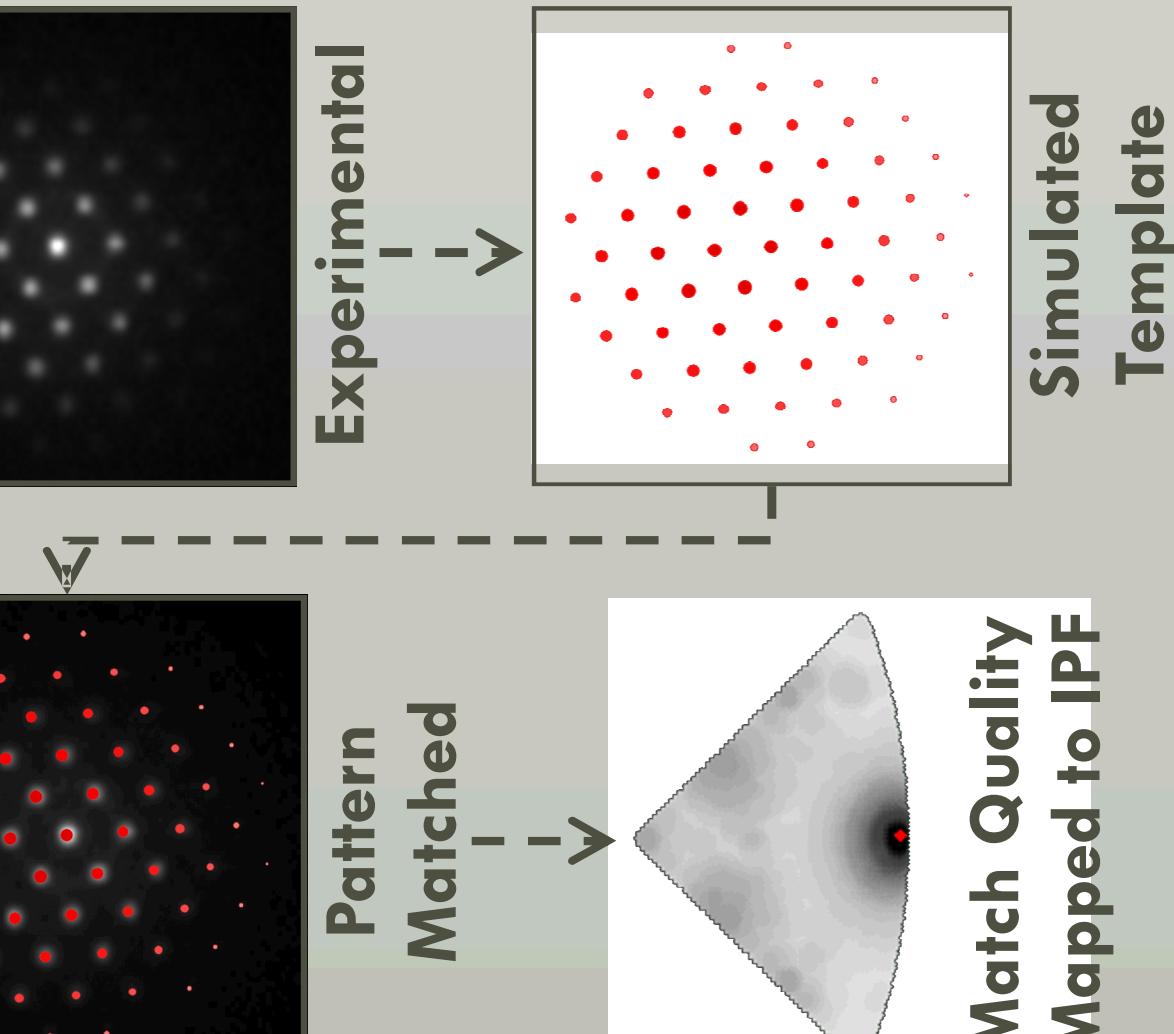


- ♦ Custom electrical feedthrough

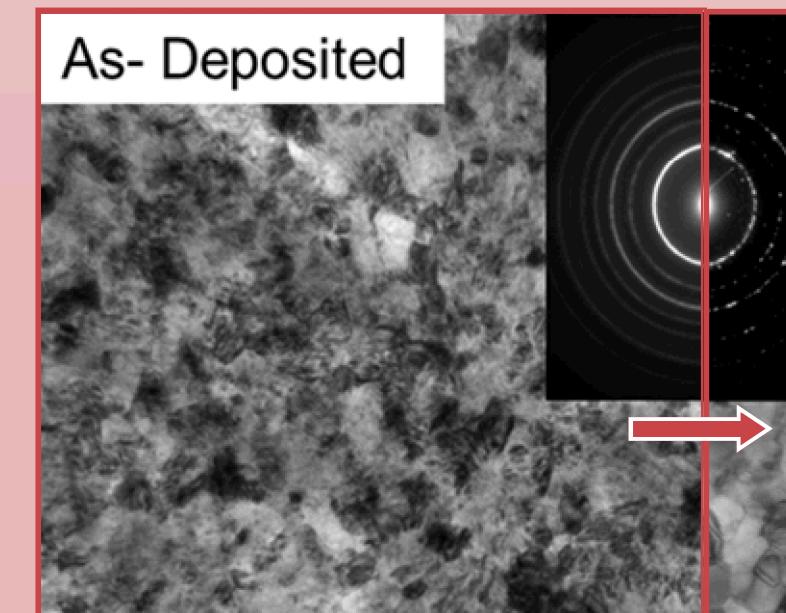
| | | | | |
|--------------|-----------|----|--------------|----|
| Yttrium | 173.05(4) | Yb | Ytterbium | 70 |
| Erbium | 167.25(9) | Er | Erbium | 68 |
| Thulium | 169.02(4) | Tm | Thulium | 69 |
| Holmium | 162.50(1) | Ho | Holmium | 67 |
| Dysprosium | 159.25(2) | Dy | Dysprosium | 66 |
| Gadolinium | 157.25(2) | Gd | Gadolinium | 64 |
| Europium | 150.36(2) | Eu | Europium | 63 |
| Samarium | 144.24(3) | Sm | Samarium | 62 |
| Promethium | 140.11(6) | Pm | Promethium | 61 |
| Neodymium | 130.07(6) | Nd | Neodymium | 60 |
| Praseodymium | 124.24(3) | Pr | Praseodymium | 59 |
| Cerium | 58 | Ce | Cerium | 57 |
| Yttrium | 173.05(4) | | | |

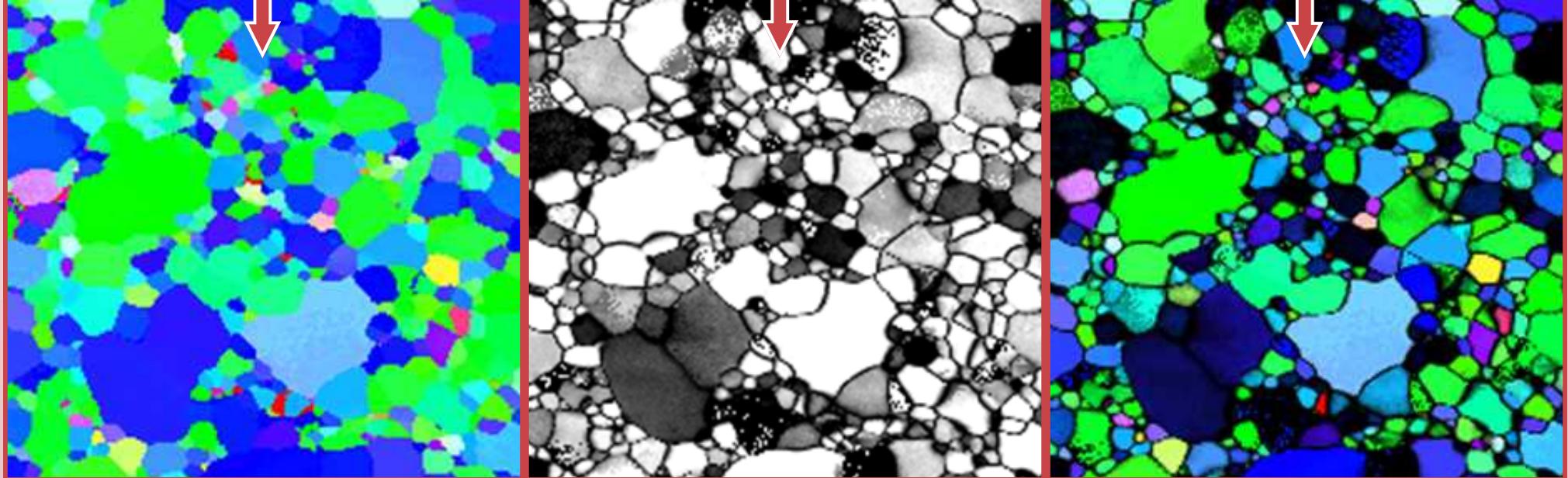
ientation Mapping

recession-enhanced electron diffraction based local orientation mapping

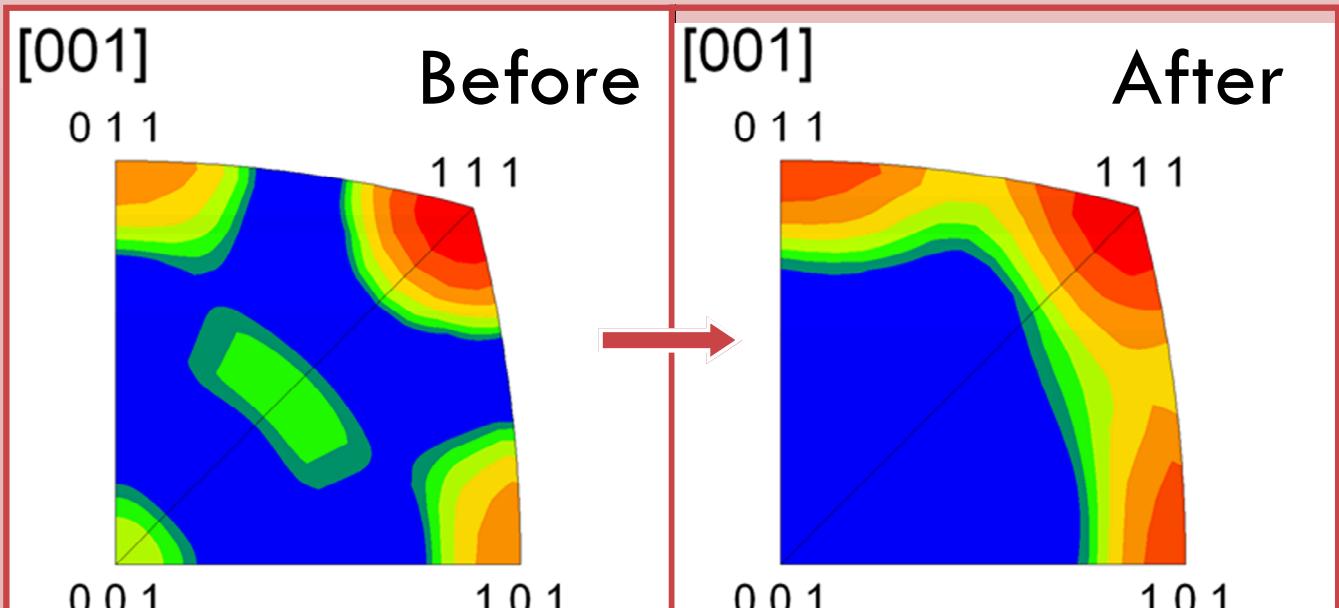
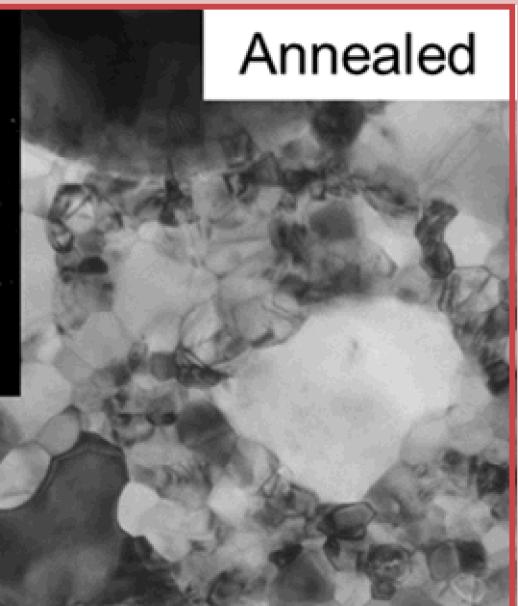


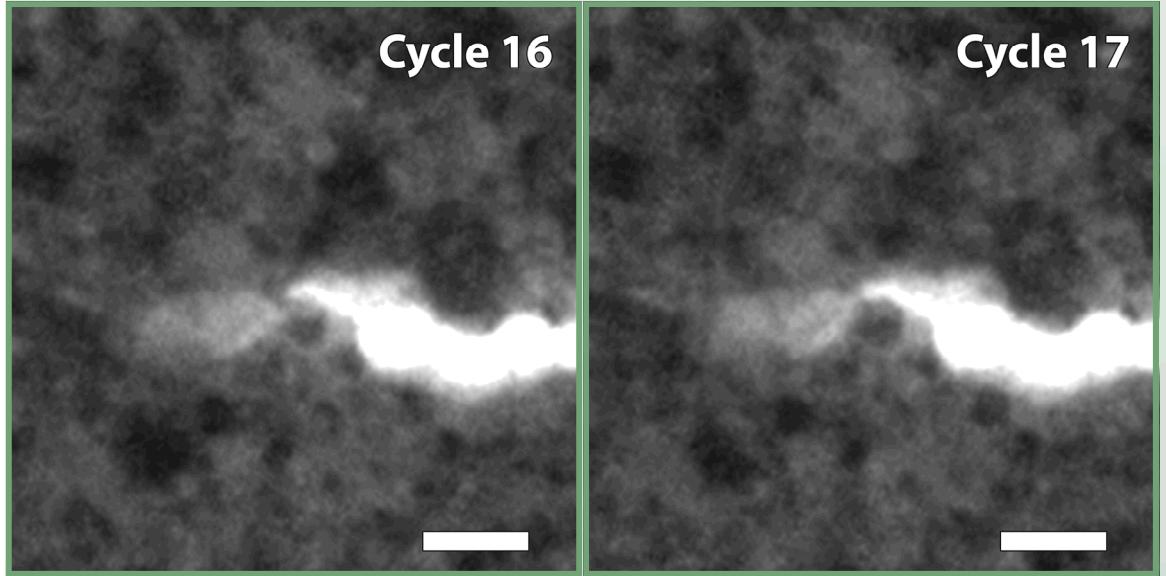
performed in minutes, and also includes orientation information. (Below, left) TEM micrographs with inset electron diffraction patterns. (Below, right) The corresponding orientation distribution function (011) and (111) textures in a suitable format. (Courtesy of





ns plotted on the [001] inverse pole figure show strengthening and weakening of the (001) texture in a more easily interpretable way (B.R. Munifering).



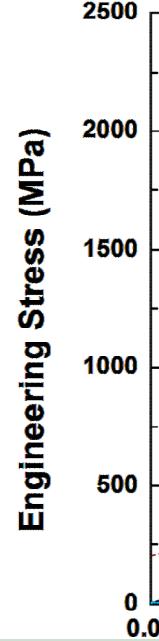
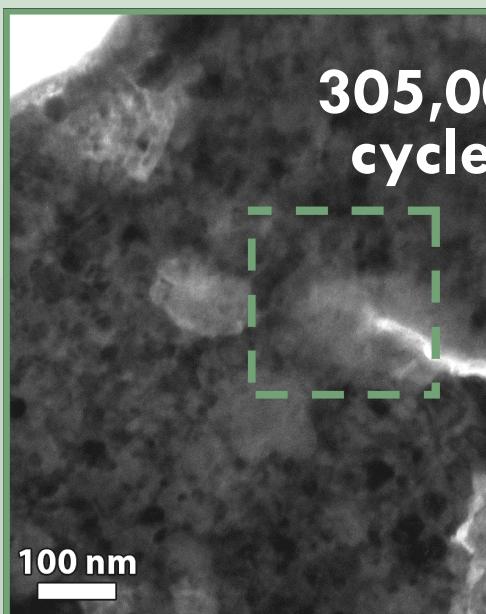


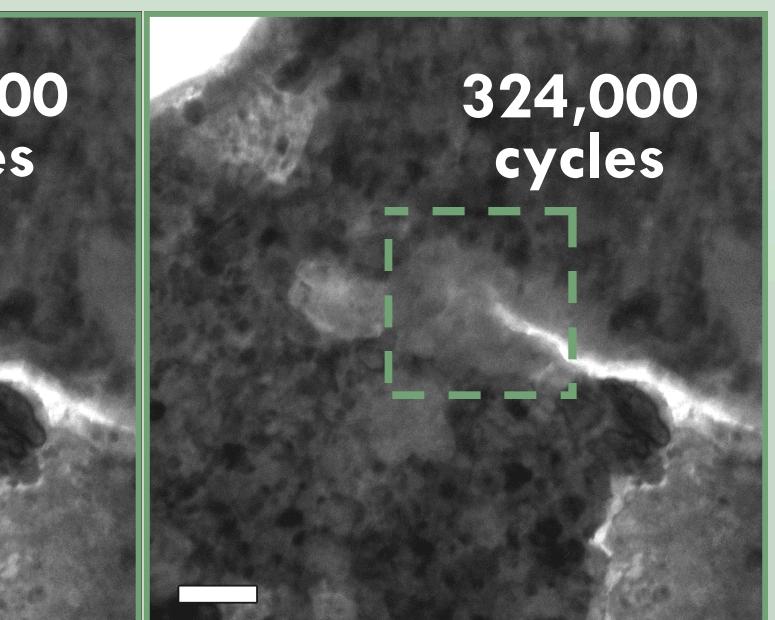
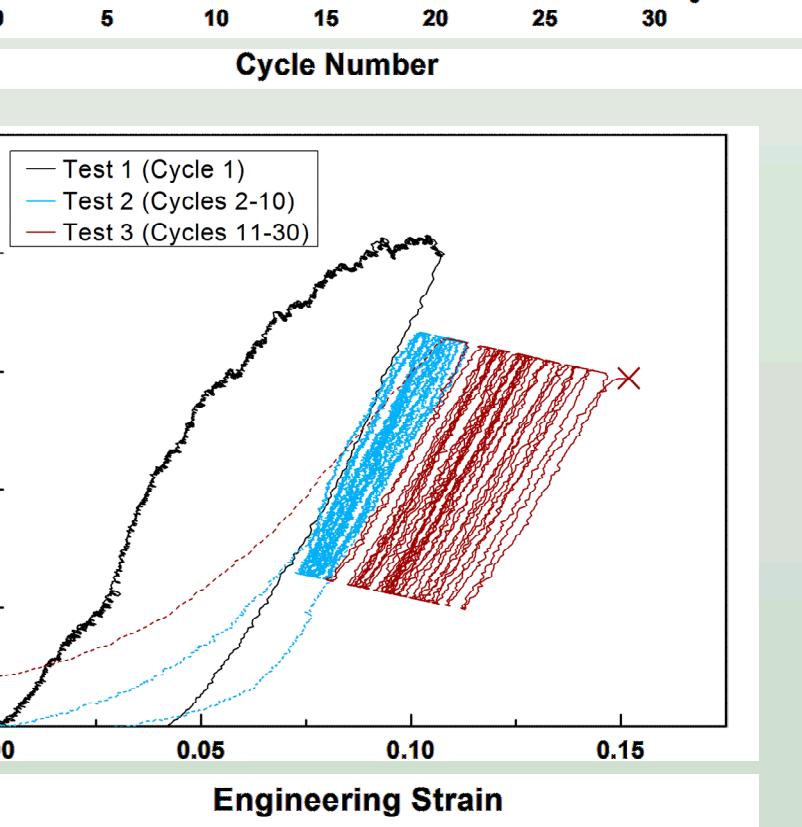
cycle testing.
(Right) Quantitative
crack measure-
ments and engi-
neering stress-strain
curves.

In Situ Fatigue

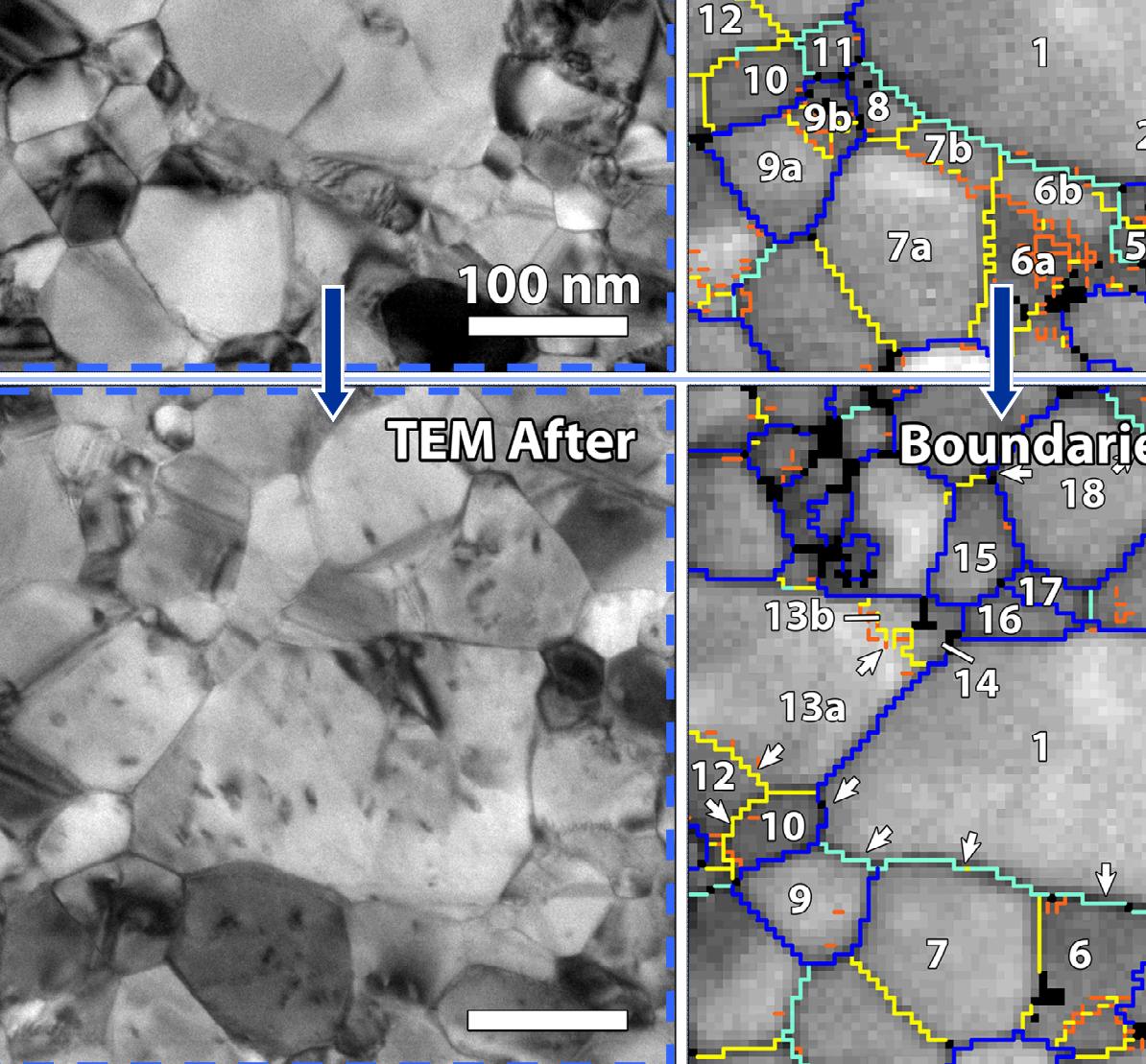
- ◆ First ever tests exceeding 10^5 cycles in the TEM
- ◆ Crack initiation, propagation, and associated grain growth

Cracked nanocrystalline Cu sample after approximately 305,000 loading cycles as seen *in situ* in the TEM during fatigue testing. Crack propagation and

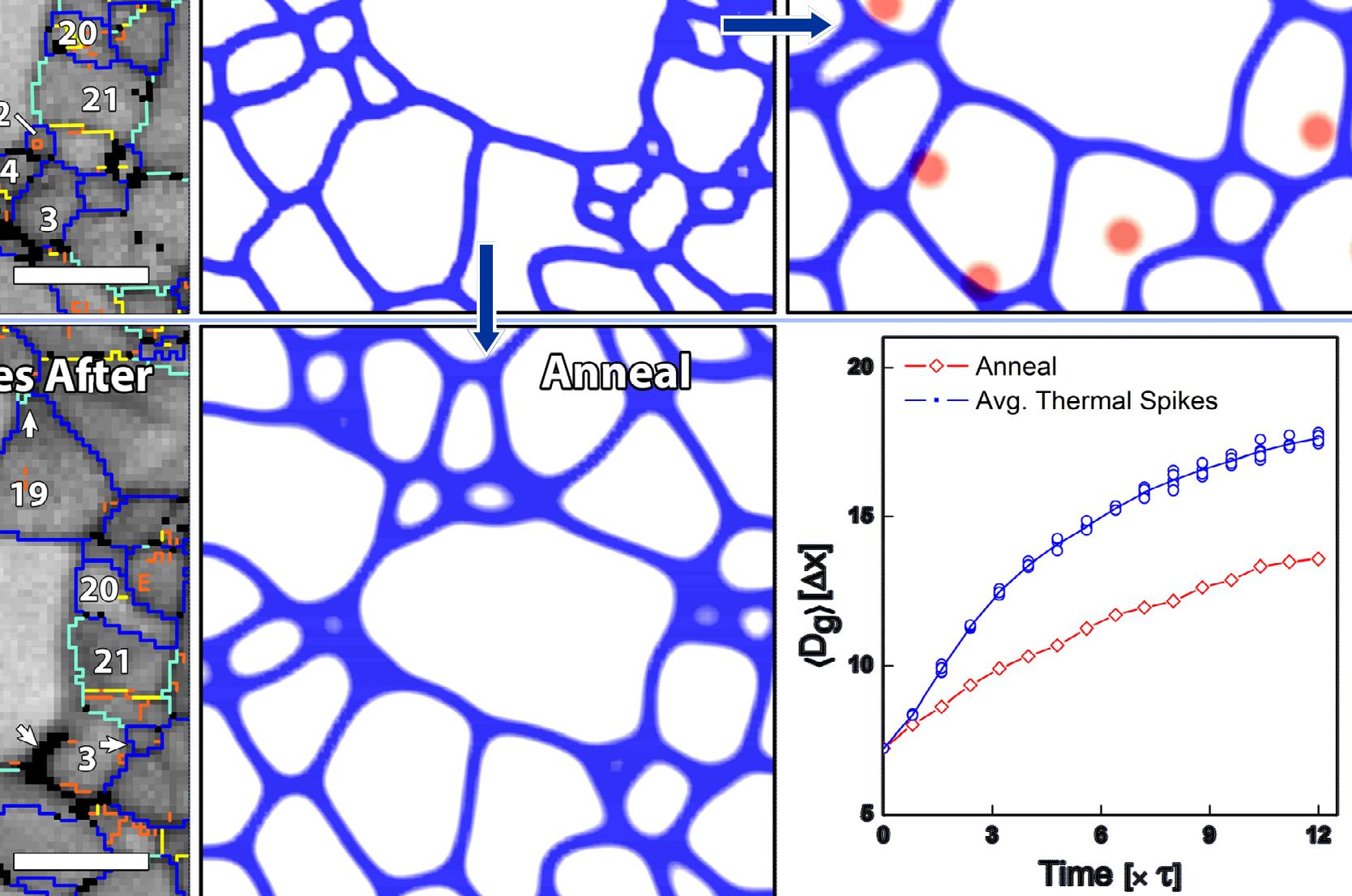




microstructural change



(Above, left) Bright-field TEM micrograph after *in situ* irradiation to a dose of 324,000 cycles. Initial structure displayed in the micrograph is shown after homogeneous annealing and thermal treatment. (Bottom) Average grain size over time, with boundary movement indicated by arrows.



micrographs and grain boundary maps before and after annealing at 600°C for $2 \times 10^{15} \text{ cm}^{-2}$ with 10 MeV Si^{3+} . (Above, right) Evolution of average grain size $\langle D_g \rangle$ versus time for the model. Evolved microstructures after simulated heating and cooling are shown in the bottom panels. The graph shows that the evolution of average grain size for the annealing and for the evolution due to thermal spikes (i.e. radiation damage). The graph shows that the evolution of average grain size for the annealing and for the evolution due to thermal spikes follows both scaling $\propto T^{1/n}$, where n is between 2 and 3.

- ◆ *In situ* heating, deformation, imaging
- ◆ TEM imaging and advanced data analysis

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U.S. DE
EN

on irradiation & more
diffraction capabilities

e and Engineering, Office of Basic Energy Sciences, U.S. De-
Nanotechnologies, an Office of Science User Facility operat-
14A0026. Sandia National Laboratories is a multi-program

DEPARTMENT OF
ENERGY

Office of
Science

40 nm

◆ Revealing re-
character and

In contradic-
chanical, and

lationships between grain boundary
and texture evolution during annealing.

ction to most thermodynamic and kinetic
d radiation stimuli is heterogeneous dep
portant phenomena such as

near the tip are evident after an additional 19,000 cycles.

- ◆ **First *in situ* TEM observations of grain ahead of a growing fatigue crack.**

: theories, grain boundary response to the bending on grain boundary character, and as abnormal grain growth.

growth

thermal, me-
and drives im-

- ◆ Differences between deviations from norm

The authors acknowledge expertise provided by A. Darbal (AppFive), S. Bhowmick, L. Kuhn, and D. Stauffer (Hysitron, Inc.), and thank C. Chisholm, P. Hosemann, A. Minor (University of California, Berkeley), D. Kaoumi (University of South Carolina), D. Buller, B. Clark, C. Gong, H. Lim, M.T. Marshall, C.J. O'conor, and others for their contributions, conversations, assistance with experiments, and facilities.



**Experiment and model reveal
small grain growth.**



Sandia National Laboratories

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