SAND2006-5962C

FIB sample preparation for EBSD: Is it worth the effort?

Joseph R. Michael
Sandia National Laboratories
Albuquerque, NM 87185-0886
jrmicha@sandia.gov

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

Sandia National Laboratories

Outline

Introduction

Introduction to EBSD

In situ sample preparation – problems with milling geometry

Creeping Crud

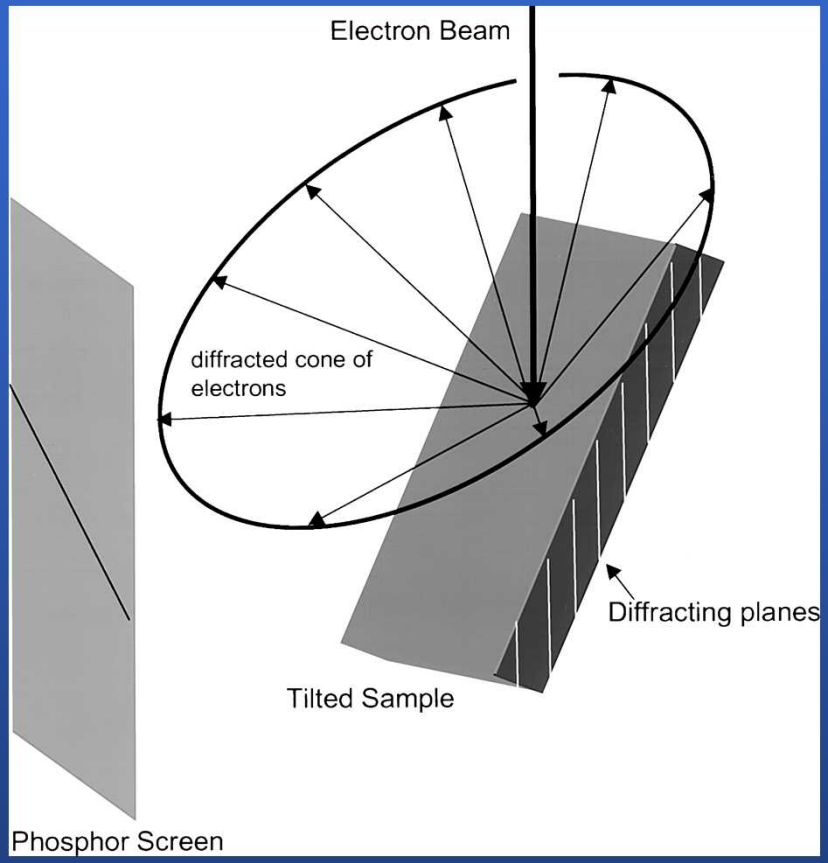
Lift-out sample preparation

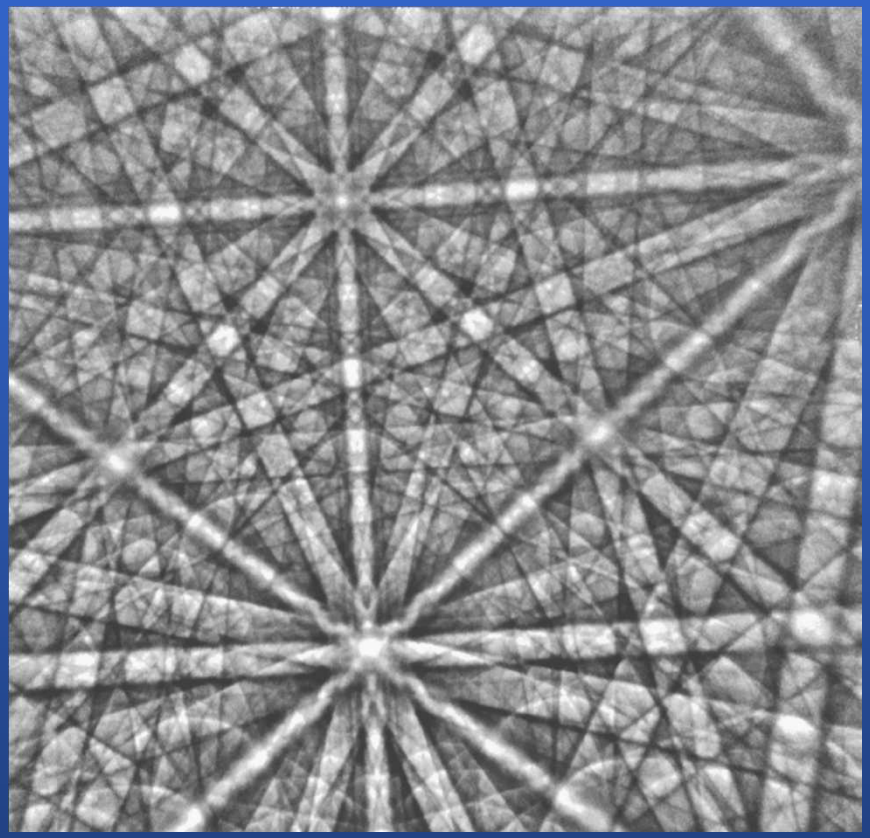
Examples

Examples of Successful FIB preparation for EBSD metals alloys, semiconductors and oxides.

Summary

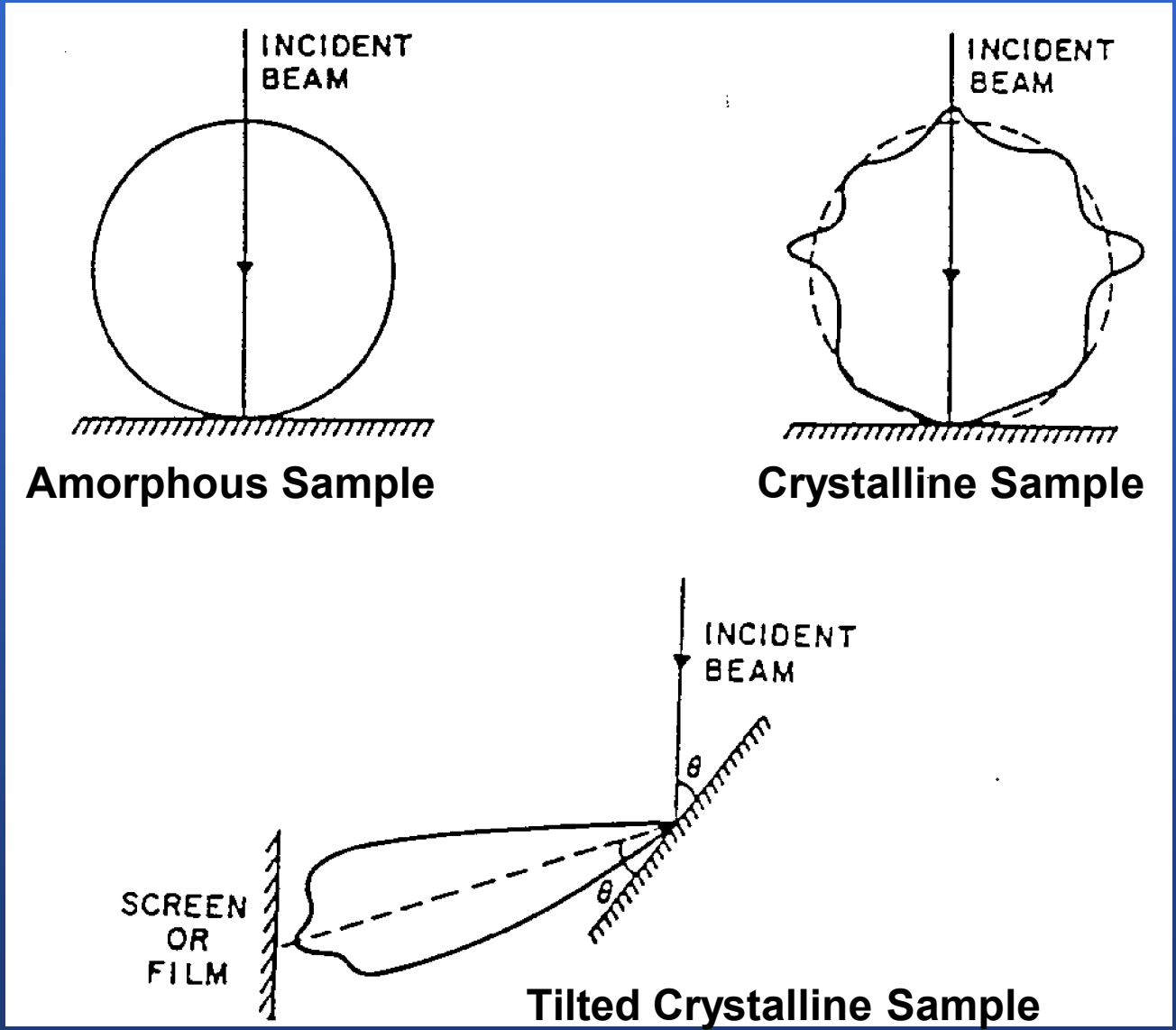
Electron Backscatter Diffraction (EBSD) in the SEM



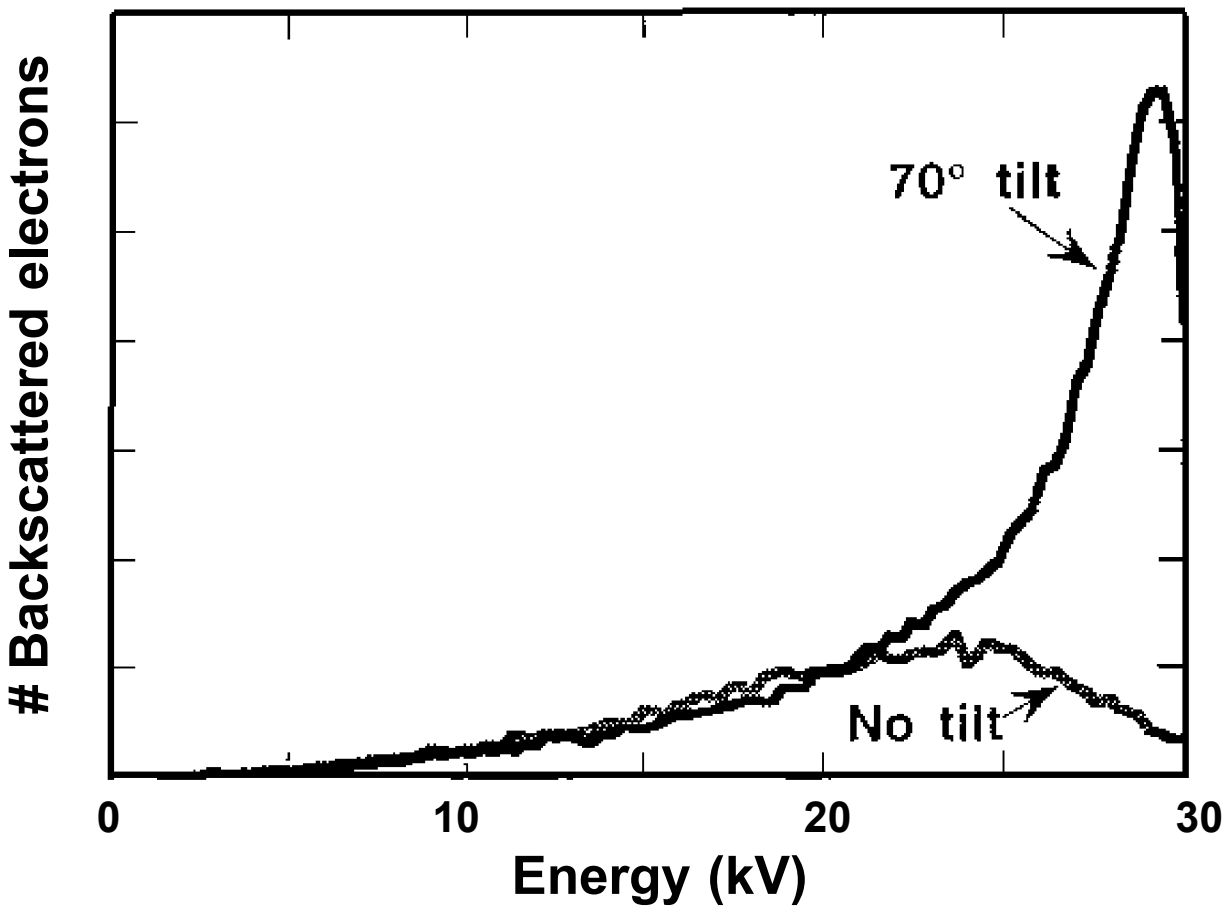


The EBSD experimental configuration

Origin of EBSP (Backscattered electron distributions)



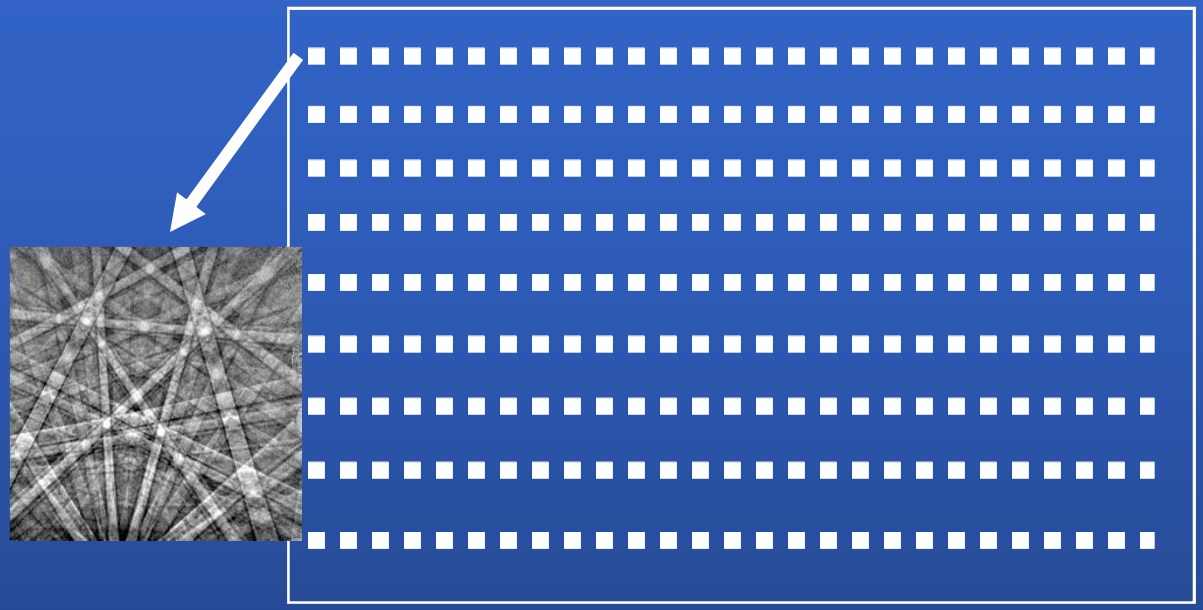
Effect of Sample Tilt on BS Electron Yield



Tilted sample has higher BS electron yield

Sample tilt results in sharp peak in BS electron energy distribution. Better defined energy of BS electrons results in sharper Kikuchi lines.

Automated EBSD Pattern Indexing



SEM image with pixels for EBSD

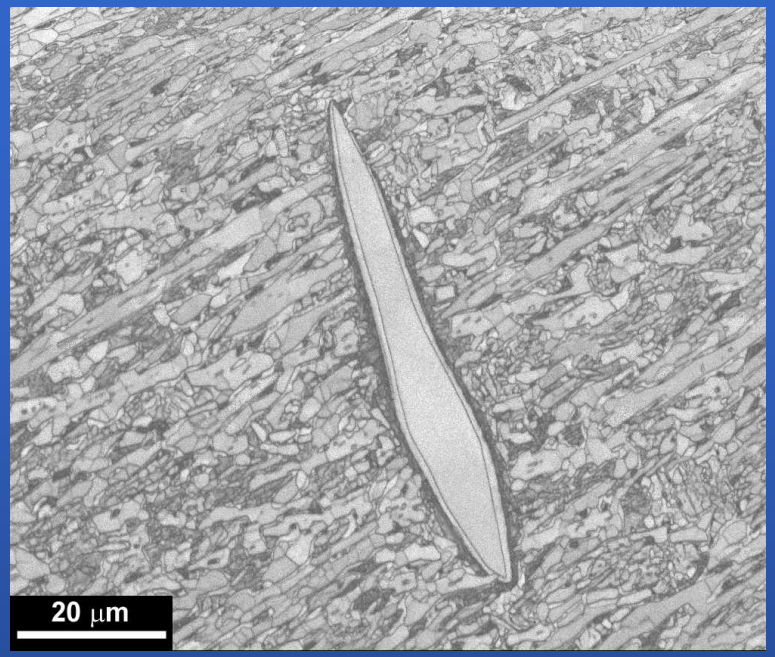
Step size dictated by microstructure and level of detail needed.

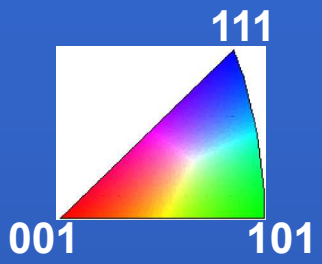
Minimum step size < 20nm!

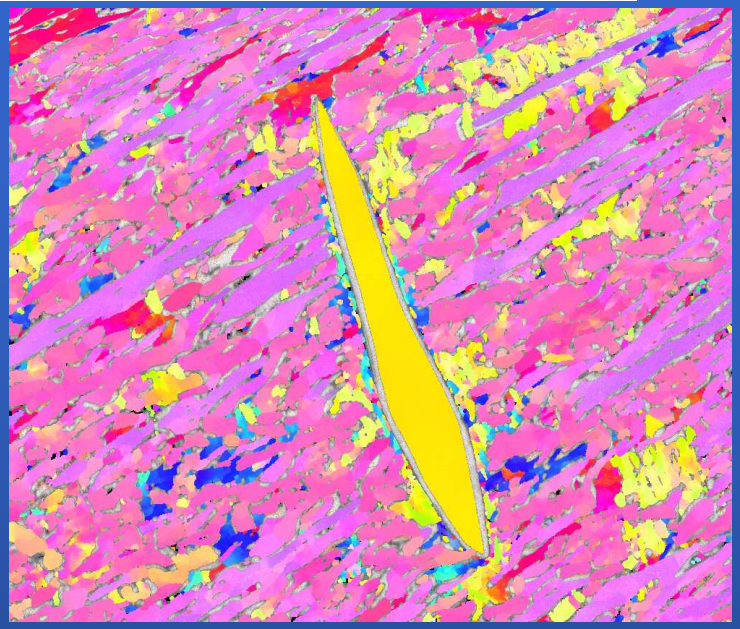
1. Scan area of interest pixel by pixel.
2. Collect EBSD pattern
3. Located 4 – 7 lines on pattern – Hough transform
4. Calculate angles between bands
5. Compare with known unit cells (short list)
6. Index pattern
7. Calculate orientation
8. Move to next pixel

Modern systems can do this up to 50 times per second!

Orientation mapping of iron meteorites





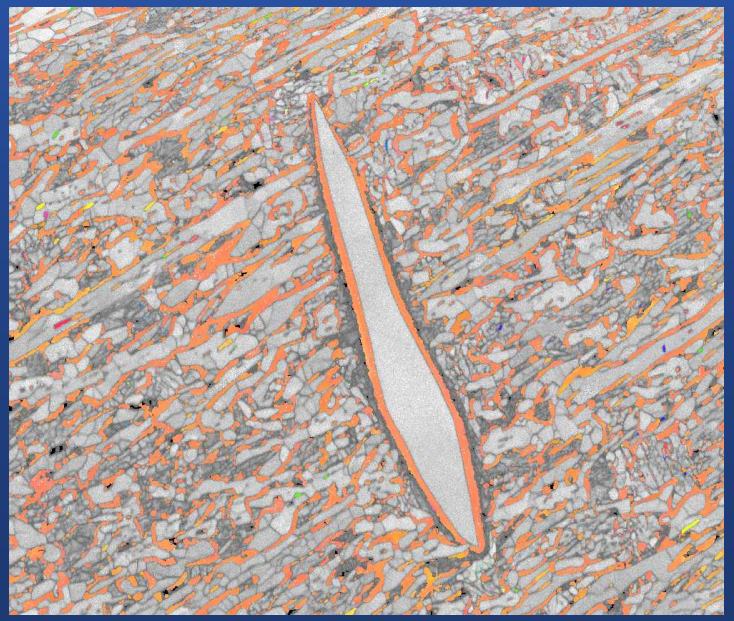


bcc

We can easily identify the fcc from the bcc phases of iron using EBSD.

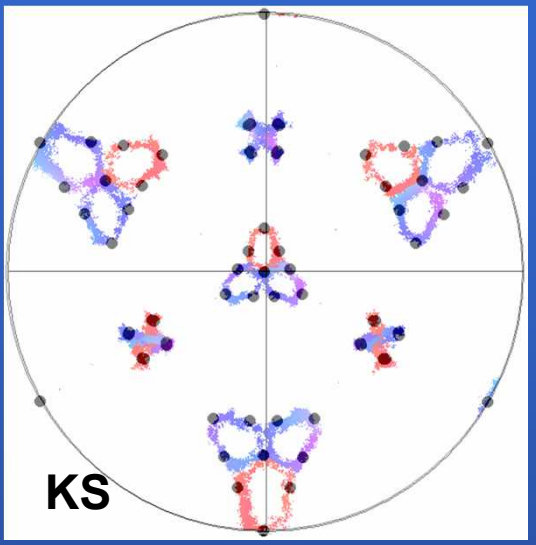
All fcc has the same orientation!

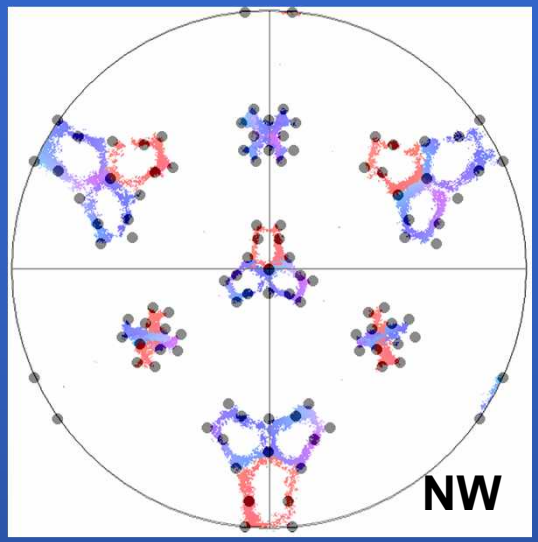
Appears to be retained austenite from a martensitic transformation

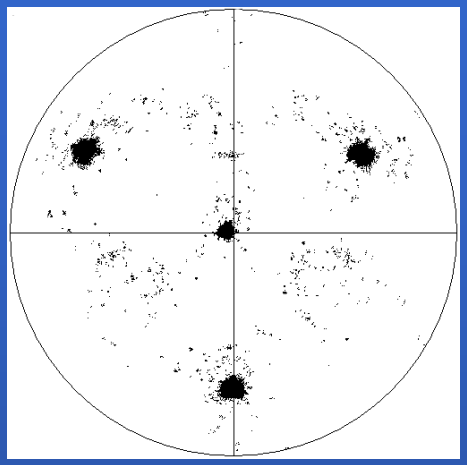


fcc

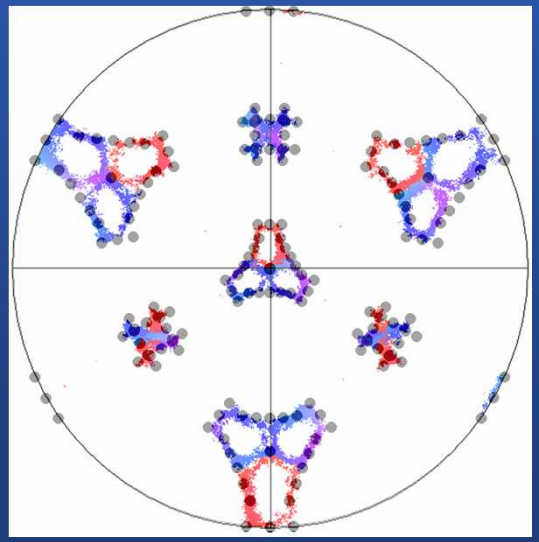
Orientation relationships between ferrite and austenite







111
fcc Pole figure



KS +NW
bcc <110>Pole figure

Complex <110>bcc pole figures indicate the orientation relationship between the fcc and the bcc phases is given by:

$<1\bar{1}0>_\gamma \parallel <1\bar{1}1>_\alpha$ or $<0\bar{1}1>_\gamma \parallel <001>_\alpha$

$(111)_\gamma \parallel (110)_\alpha$ $(111)_\gamma \parallel (110)_\alpha$

Kurdjumov-Sachs Nishiyama-Wasserman

Or some combination of KS + NW

Sample Preparation for EBSD

Main Sample Requirements

Clean surface with little surface deformation or damage

Some samples require no sample preparation:

fracture surface, crystal faces or facets, as grown layers etc.

Typical sample preparation may be as easy as mechanical polishing followed by a light chemical etch

Electropolishing works well

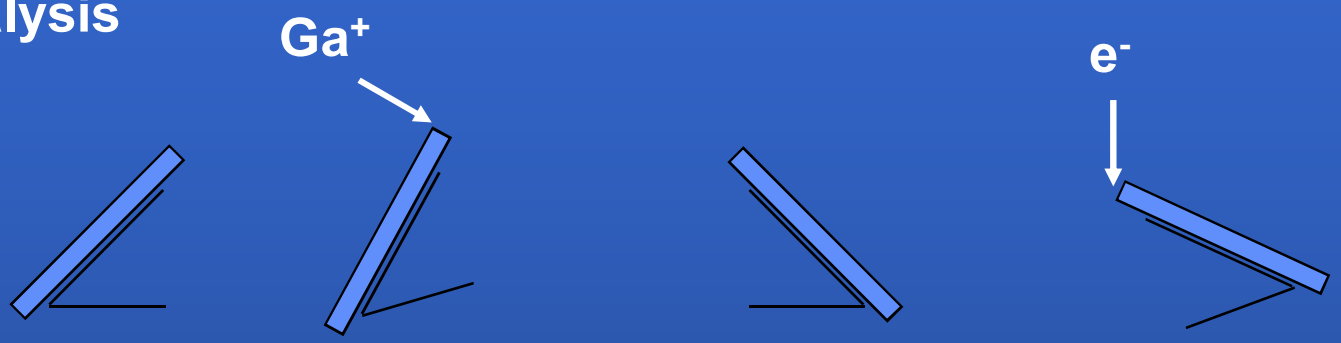
For ceramics - maybe more difficult, however fracture surfaces work quite well.

Alumina recipe - Mechanical polish, Etched with Phosphoric acid at 250°C, ion polish or thermal anneal at 1200°C (Mulvihill et al., Z. Metallkd.,89,(1998),p. 546.)

Ion Beam Preparation works well on many materials

Routes to in-situ sample preparation for EBSD

Mill parallel to an edge – then orient edge for EBSD analysis



Mount sample at 45º pre-tilt

Tilt +7º and mill sample on edge

Rotate sample

Tilt +25º and perform EBSD on edge

Mill normal to a surface and then tilt sample for EBSD analysis

OR:

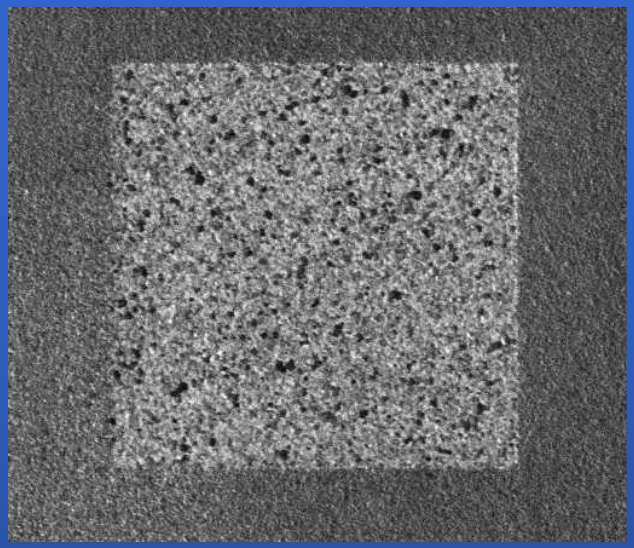
~~Tilt sample to 52º and mill with ion beam normal to surface.~~

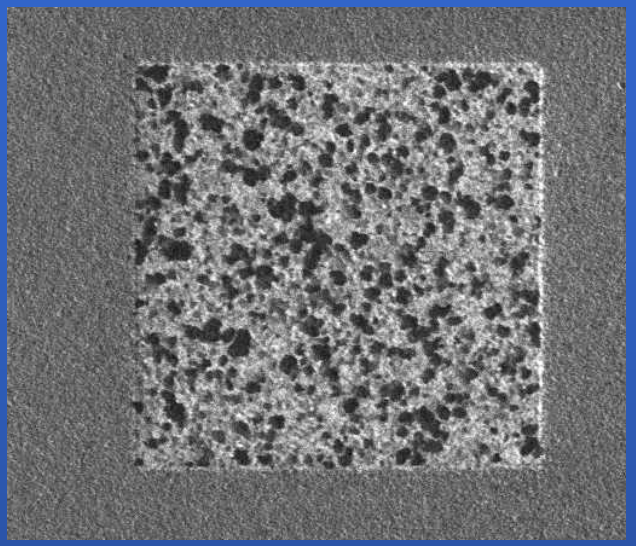
~~Tilt sample to about 60-70º and perform EBSD~~

The Problem – dark imaging areas appear with Ga+ exposure

1 min

1.1 x 10^{16} $Ga^{+}/\mu m^{2}$



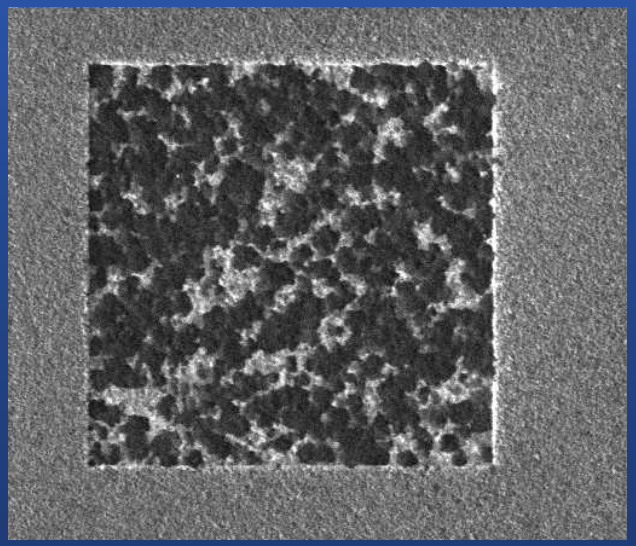


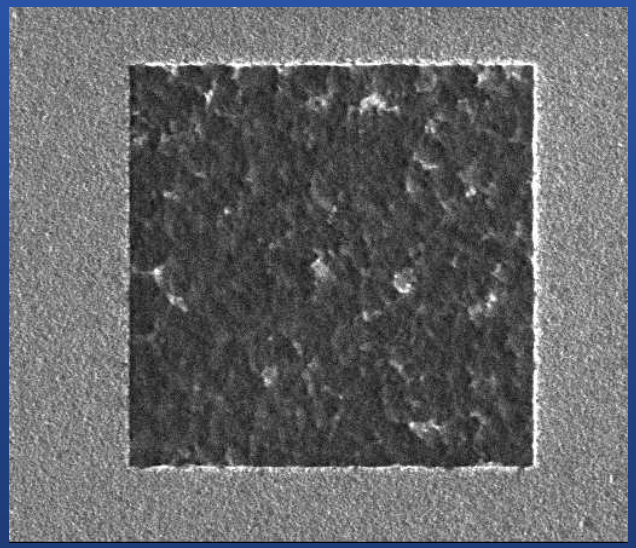
3 min

3.4 x 10^{16} $Ga+/\mu m^{2}$

6 min

6.8 x 10^{16} $Ga^{+}/\mu m^{2}$





10 min

11 x 10^{16} $Ga/\mu m^{2}$

Evaporated Cu sample irradiated at 30 pA in 100 μm^{2} area

The Problem – dark imaging areas appear with Ga+ exposure

Similar behavior noted in Cu, Ni, Au (and other FCC metals)

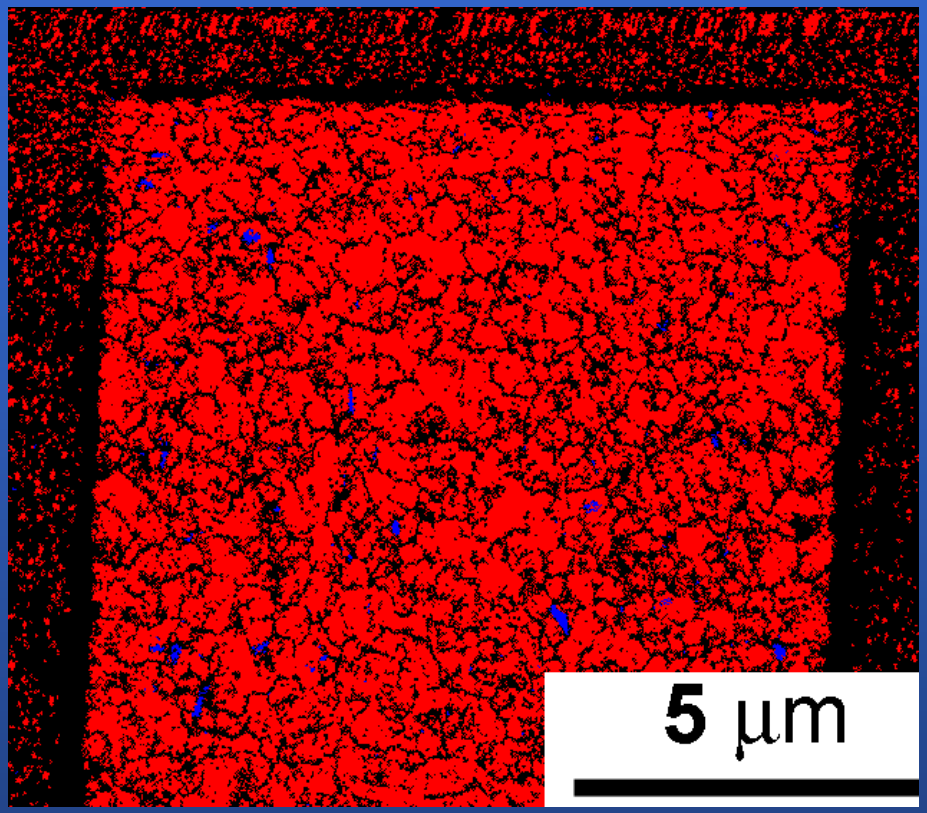
Similar behavior noted in W and Ta (and other BCC metals)

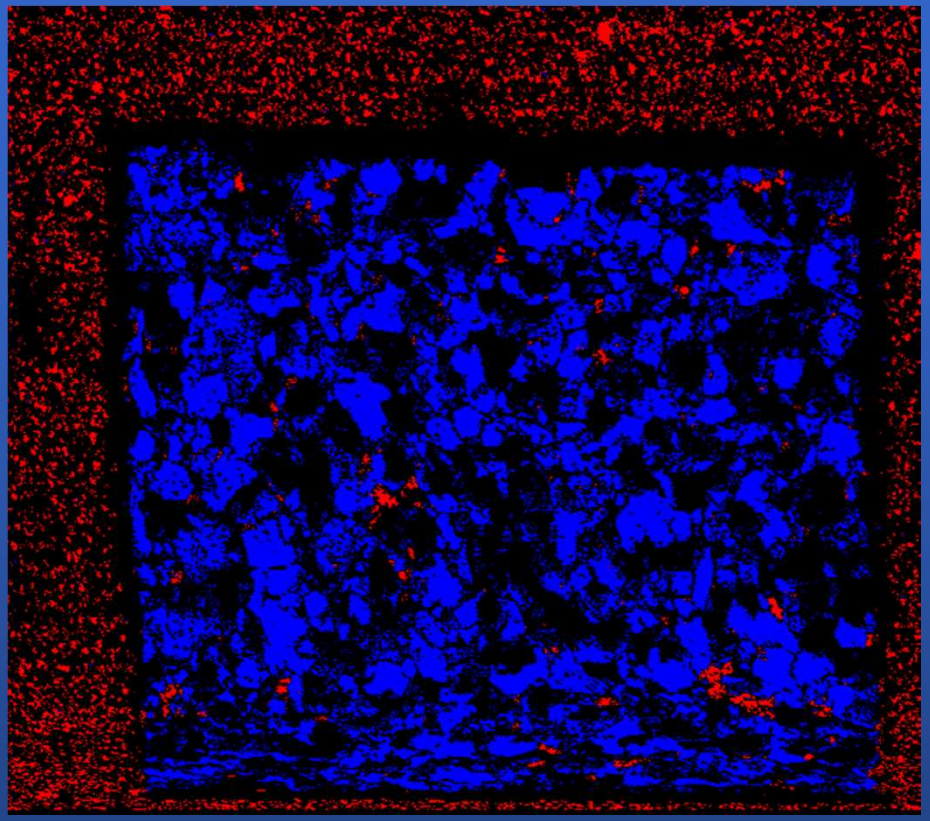
All show the development of dark imaging regions

Growth of dark imaging regions occurs more slowly in coarse grained materials

Is this recrystallization, texture development or Ga intermetallic formation in the ion beam exposed regions?

Phase Distributions in ion milled regions of fine-grained Cu





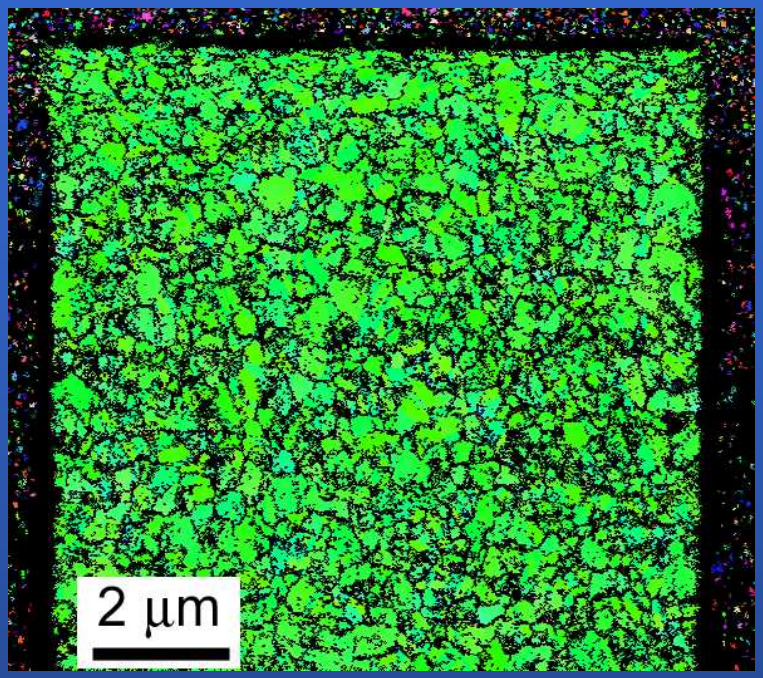
2 min 330 pA - 25 x 10^{16} $Ga^{+}/\mu m^{2}$

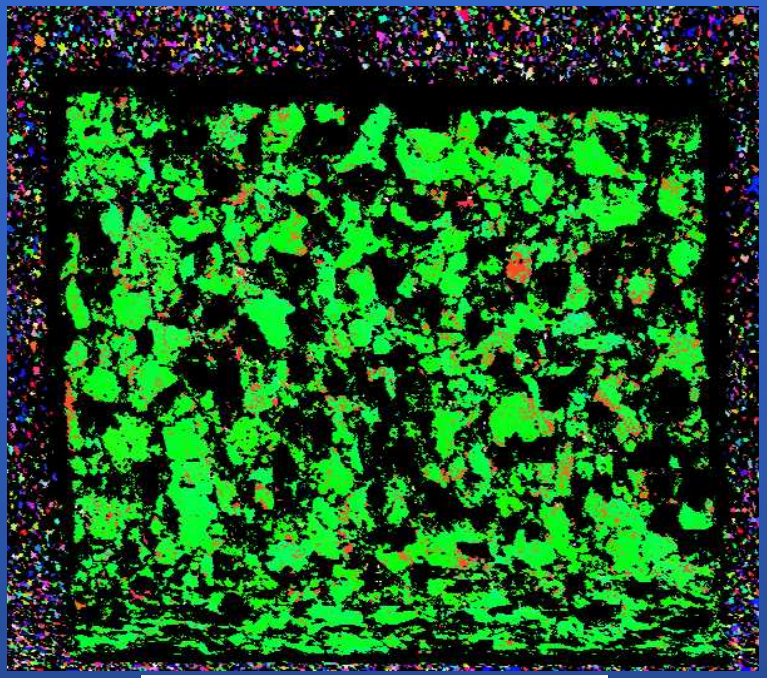
3 min 330 pA - 37 x 10^{16} $Ga^{+}/\mu m^{2}$

Red = Cubic phase (Cu)

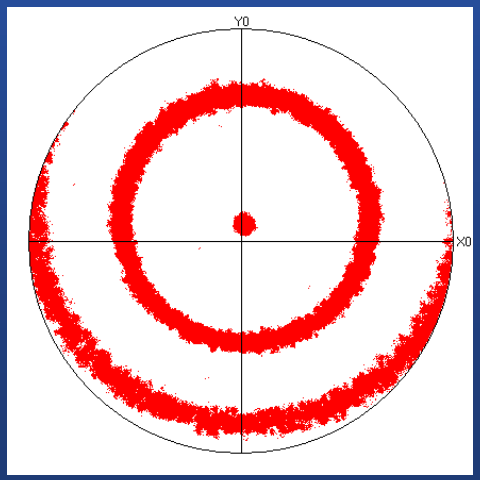
Blue = Cu_3Ga (hexagonal)

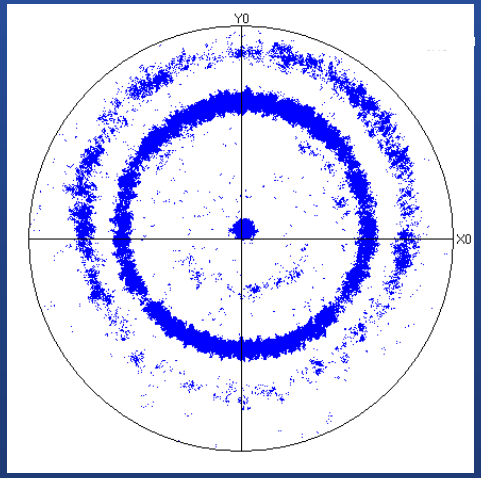
Orientation changes in ion milled regions of fine-grained Cu





(110)



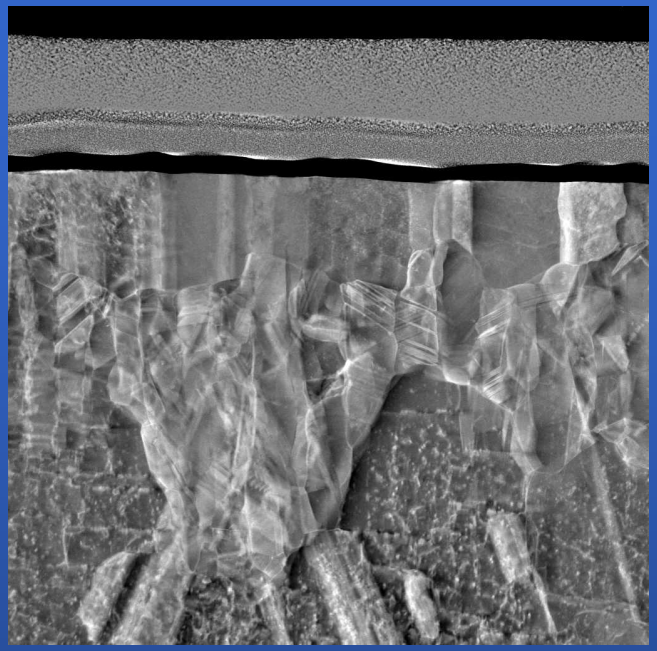


(11$\bar{2}$0)

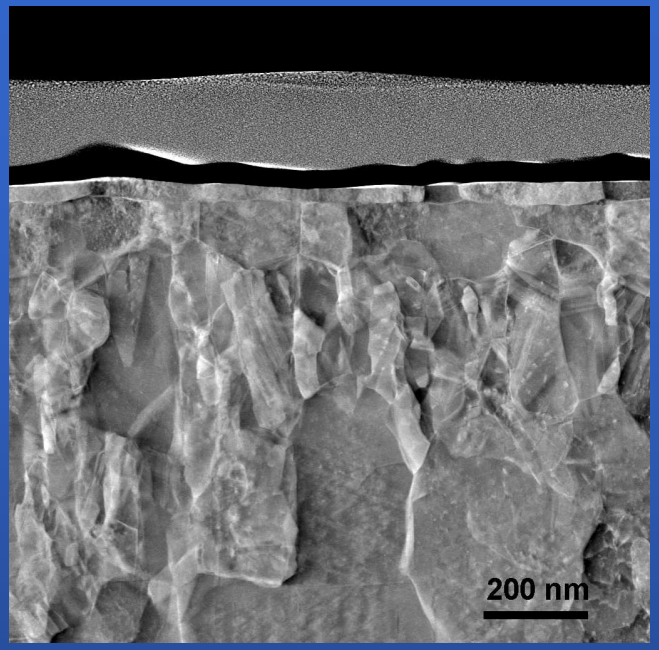
2 min 330 pA - 2.5 x 10^{16} $Ga^{+}/\mu m^{2}$

3 min 330 pA - 3.7 x 10^{16} $Ga^{+}/\mu m^{2}$

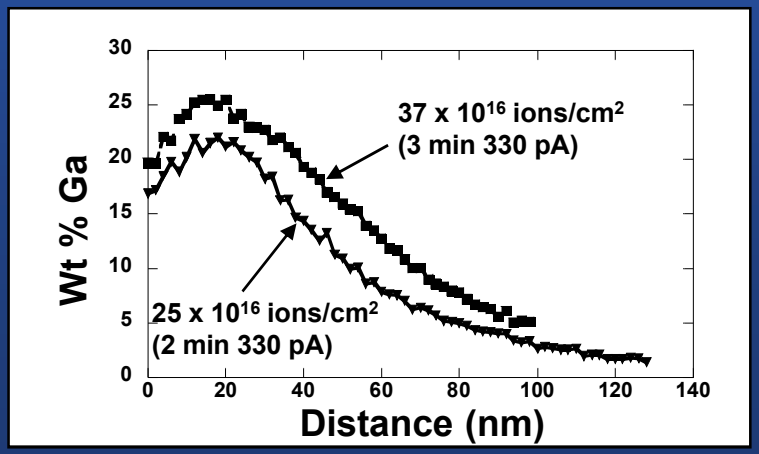
STEM imaging and microanalysis of Ga+ into Cu



2 min 330 pA - 25 x 10^{16} $Ga^{+}/\mu m^{2}$

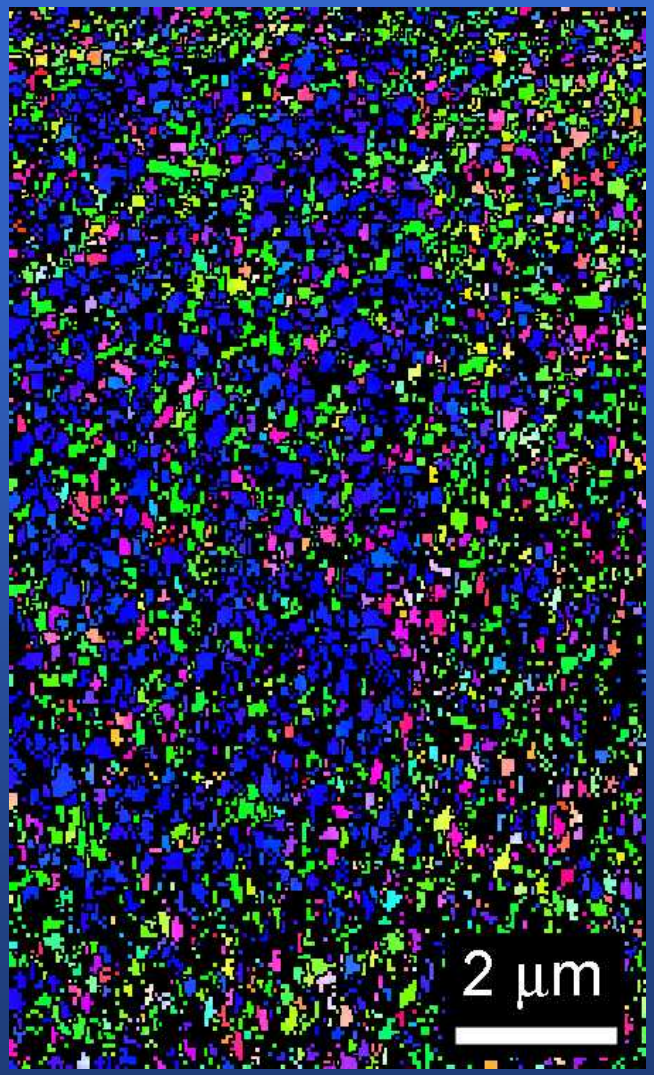


3 min 330 pA - 37 x 10^{16} $Ga^{+}/\mu m^{2}$

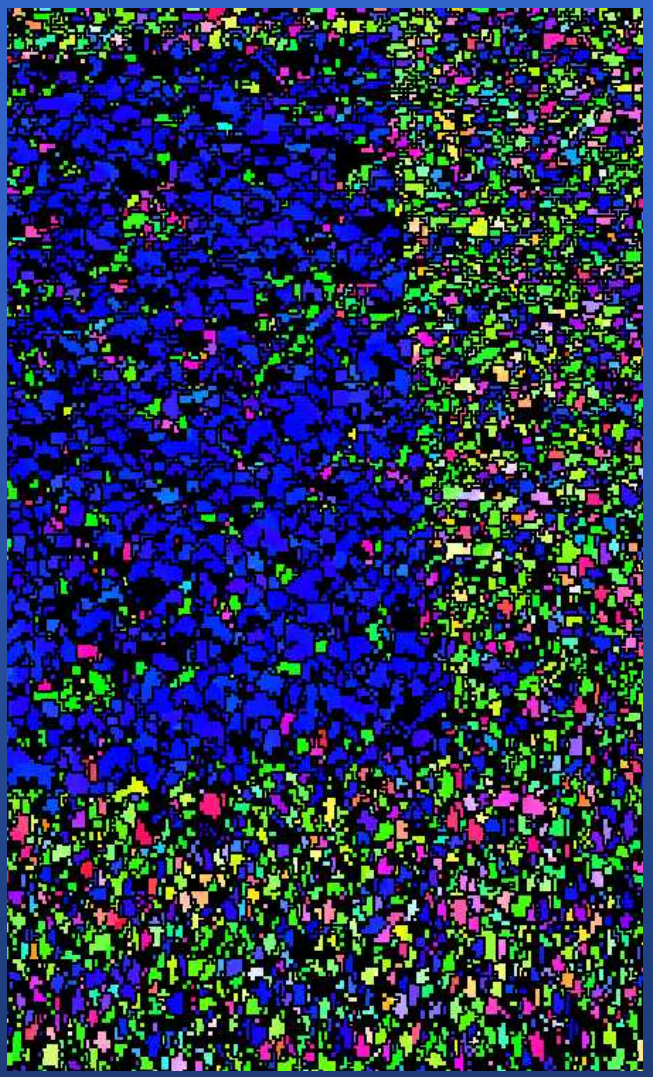


SRIM estimates the range of 30 Ga+ in Cu to be 10 nm

Orientation changes in ion milled regions of fine-grained W



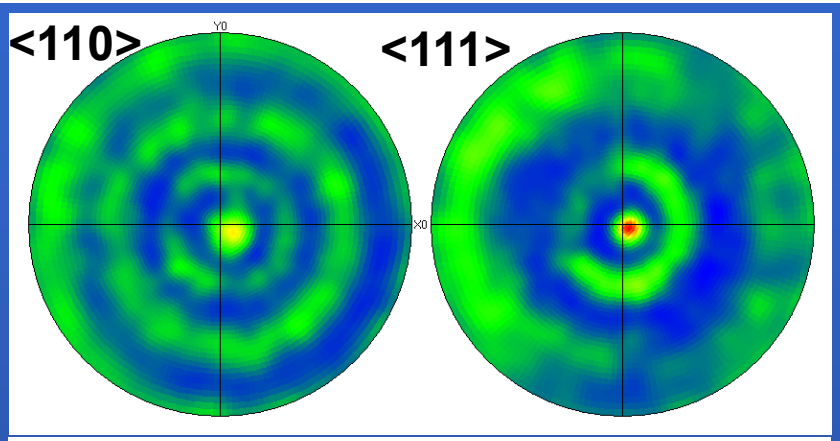
7.5×10^{16} $Ga^{+}/\mu m^{2}$
4 min at 50 pA in 100 μm^{2}



18.7×10^{16} $Ga^{+}/\mu m^{2}$
10 min at 50 pA in 100 μm^{2}

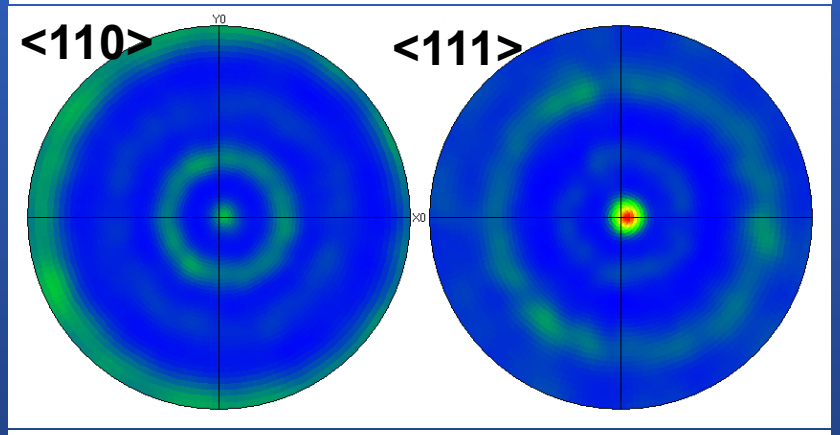
Orientation changes in ion milled regions of fine-grained W

As-deposited



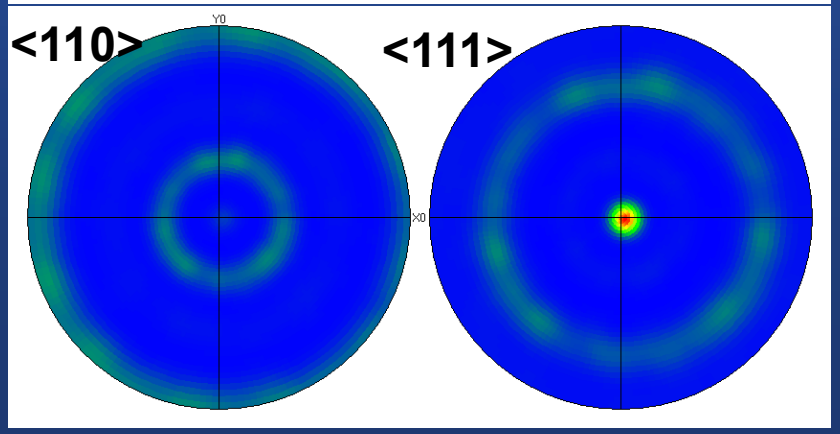
3 times random

7.5×10^{16} $Ga^{+}/\mu m^{2}$
4 min at 50 pA in
100 μm^{2}



12 times random

18.7×10^{16} $Ga^{+}/\mu m^{2}$
10 min at 50 pA in
100 μm^{2}



21 times random

Summary: Do not attempt to mill normal to surface for EBSD

FCC metals:

Dark regions in Ni and Au are growth of surface grains with <110> fiber texture. This is a strong channeling direction in FCC.

Dark regions in Cu are initially <110> fiber textured FCC grains followed by <11$\bar{2}$0> Cu_3Ga (hexagonal). Phase change is due to Ga+ implantation and low difference in sputter yield.

BCC metals:

Dark regions in W and Ta are due to growth of surface grains with <111> fiber texture. No change with continued exposure.

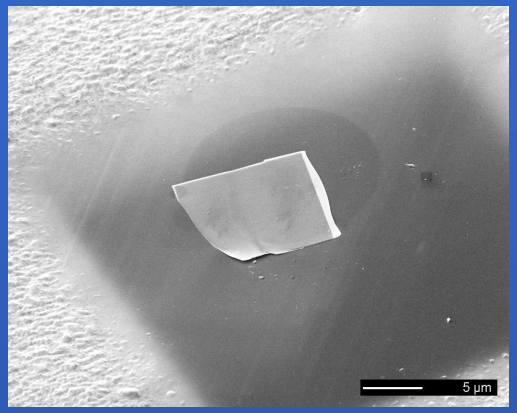
In either case, the fiber texture develops along ion beam direction.

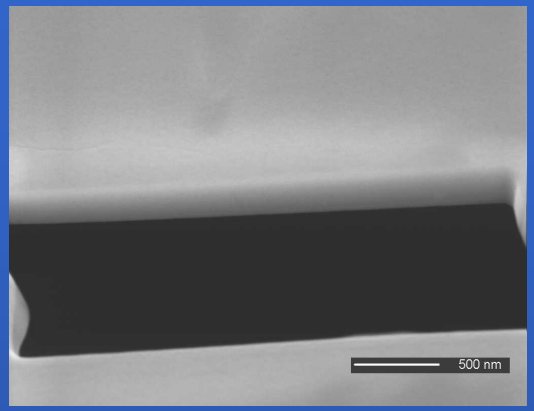
Kinetics faster in fine grained metals. Role of grain boundaries?

Rate of orientation change maybe related to sputter yield.

Higher sputter yields result in lower surface compositions. Ability to exceed solubility limits.

Contrast from thin FIB samples (thin Al sample)



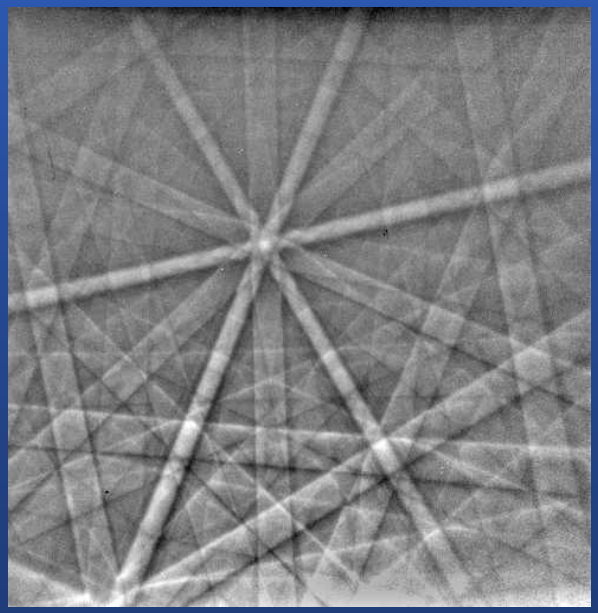


Sample thickness:

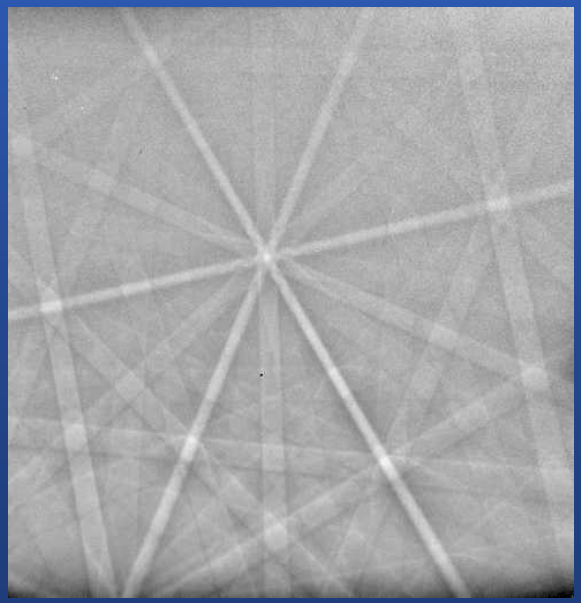
CBED - 240. nm

EELS - 190 nm

Direct measurement - 220 nm



20kV



40kV

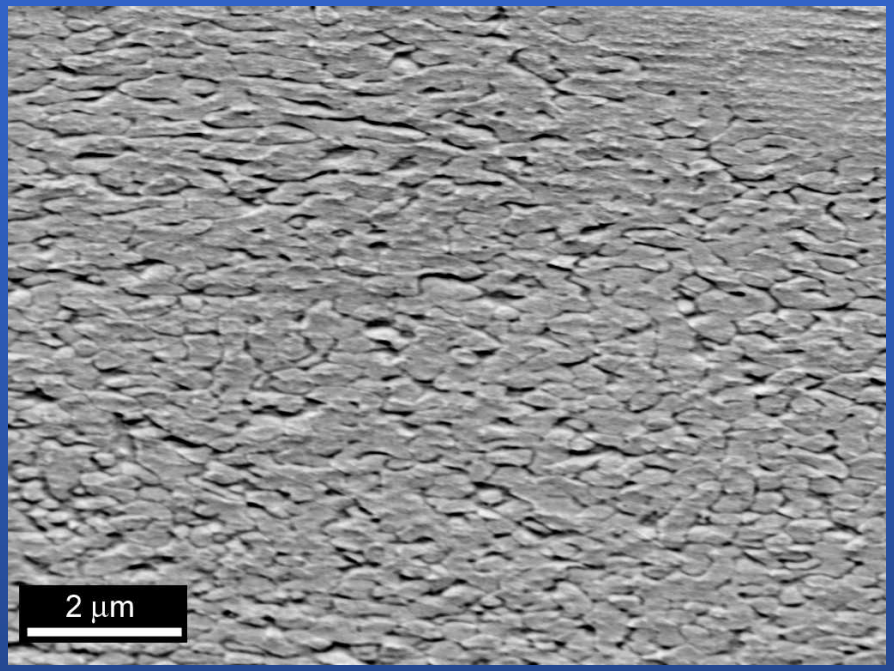
Monte Carlo Backscatter yields

	20 kV	40 kV
Bulk	0.5	0.5
Thin	0.3	0.08

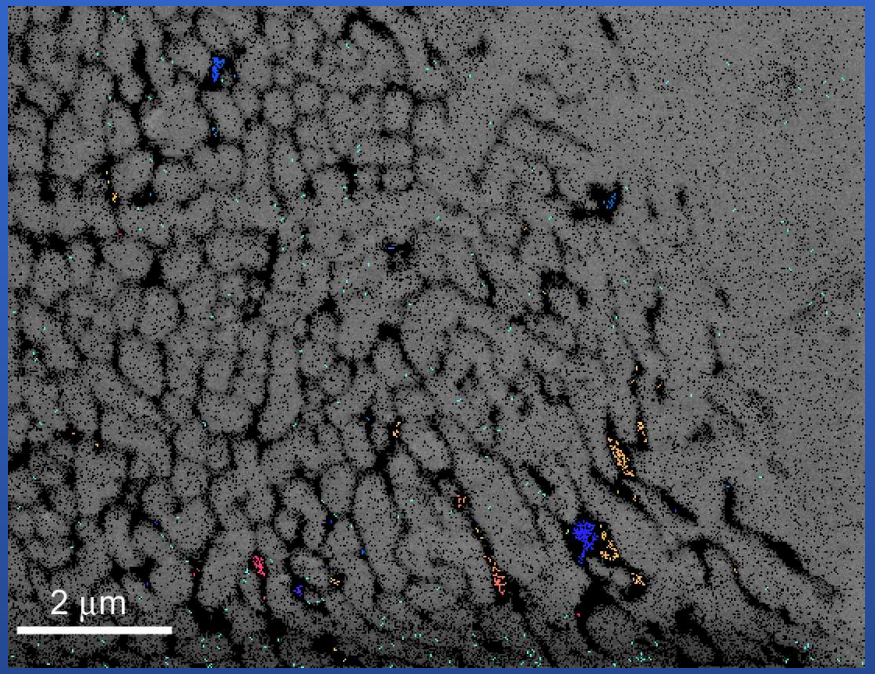
Remainder of electrons in thin sample are transmitted.

Improving spatial resolution of EBSD – thin samples

FSE Image



Orientation map

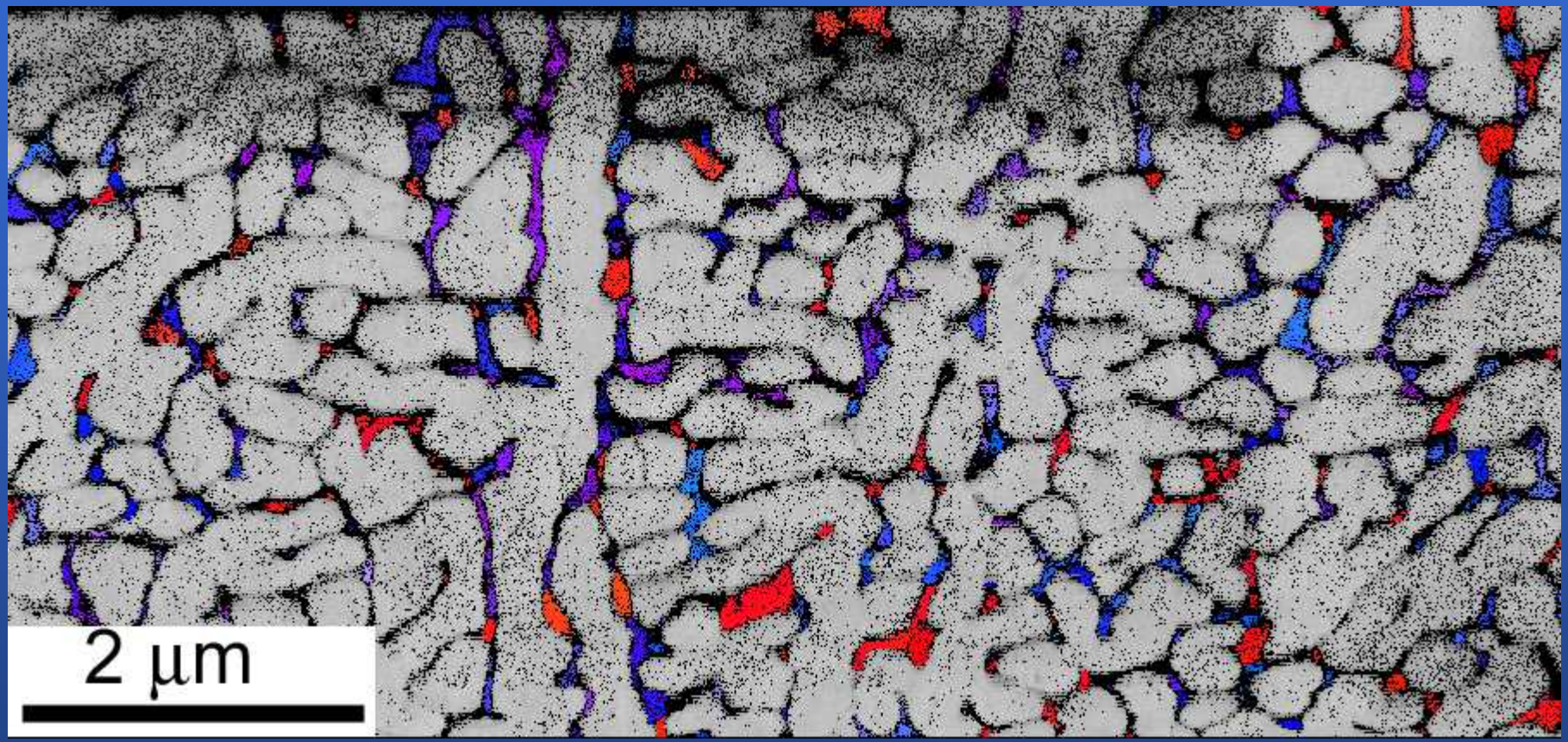


15 kV, 10 nm step size,
10 patterns/sec

Best result from careful metallographic polish of sample

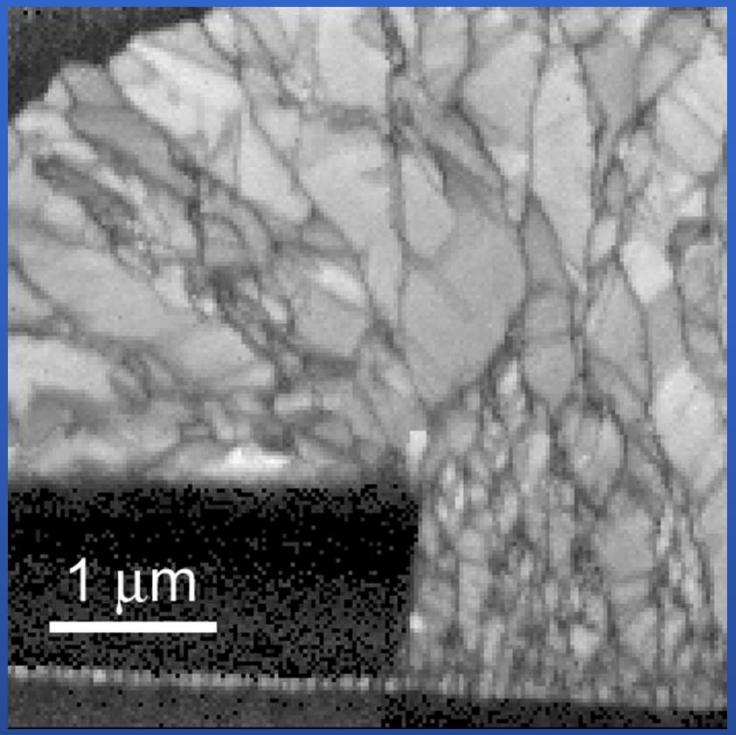
Not very good!

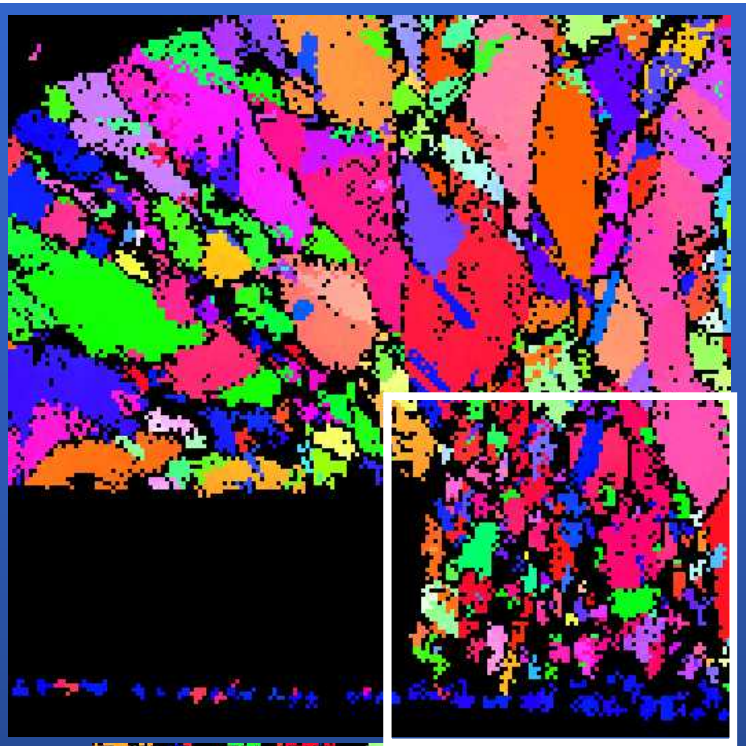
Improved spatial resolution with FIB prepared thin sample



15 kV, 10 nm step size,
20 patterns/sec

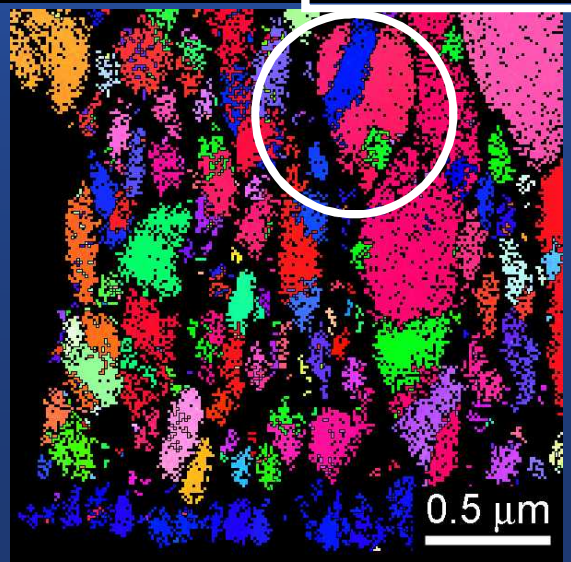
Growth of Electrodeposited Ni on Patterned Silicon (thin sample)





Pixel spacing of 25 nm

20 kV

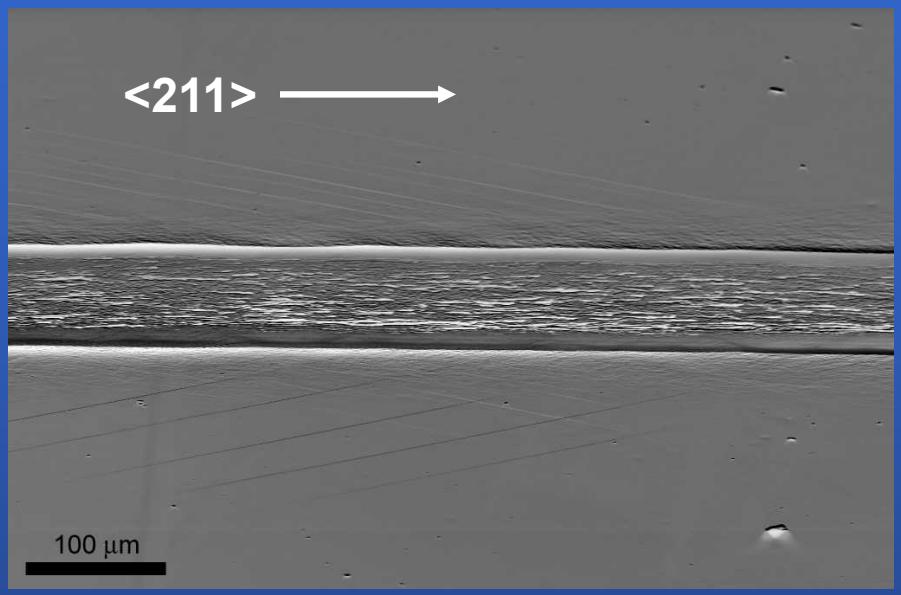


10 nm resolution

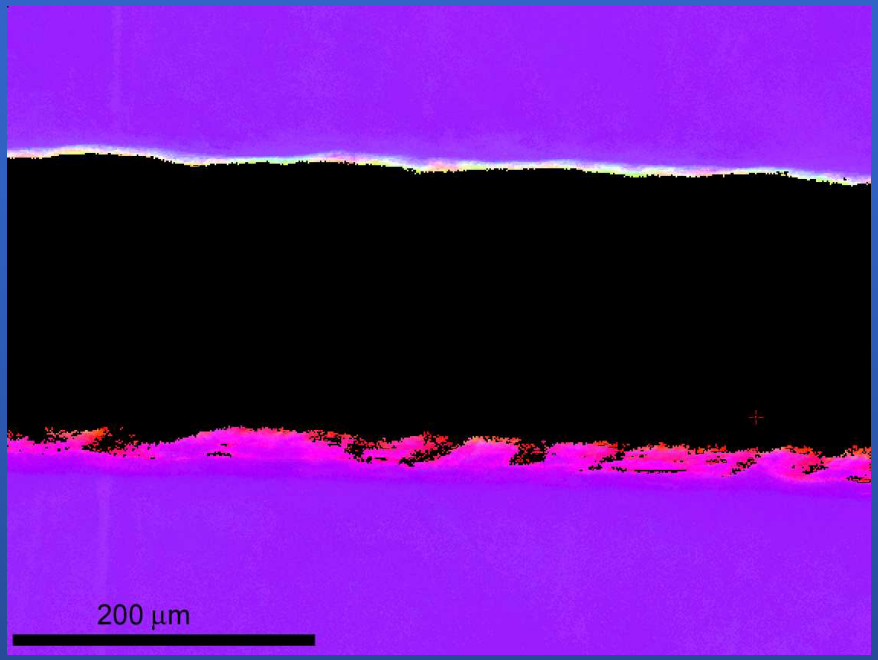
Pixel spacing of 10 nm

20 kV

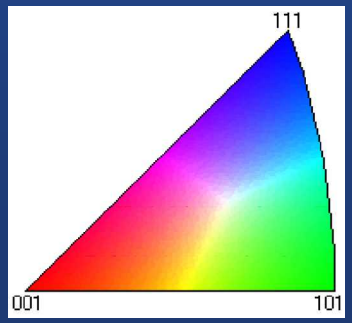
SEM imaging (FSE) and EBSD of wear scars in plan-view



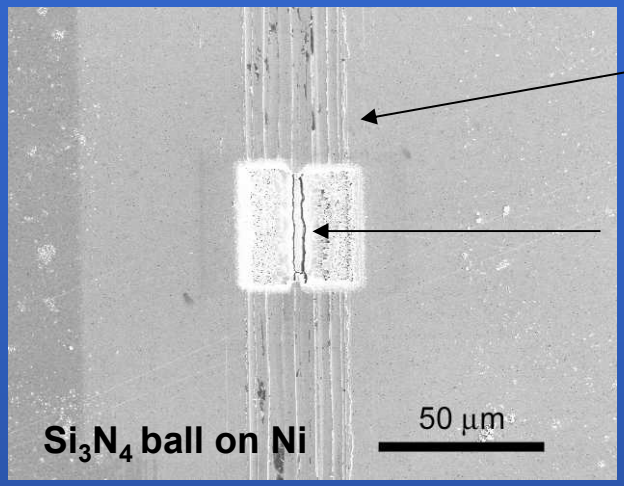
SEM image of forward scattered electrons (FSE). Note visibility of {111} slip traces. (Image not corrected for 70º tilt.)

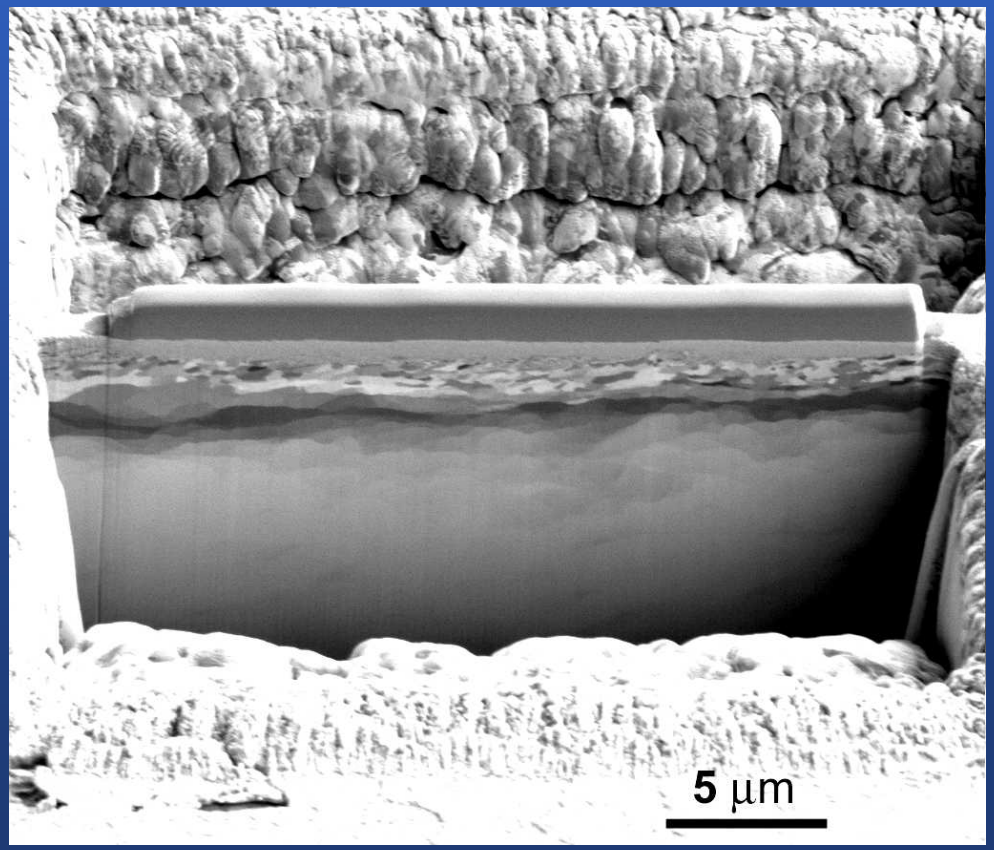


EBSD IPF map with respect to the sliding direction. Note dark region due to high plastic deformation in wear scar



FIB sample preparation for EBSD and TEM





Ion channeling contrast image of wear scar

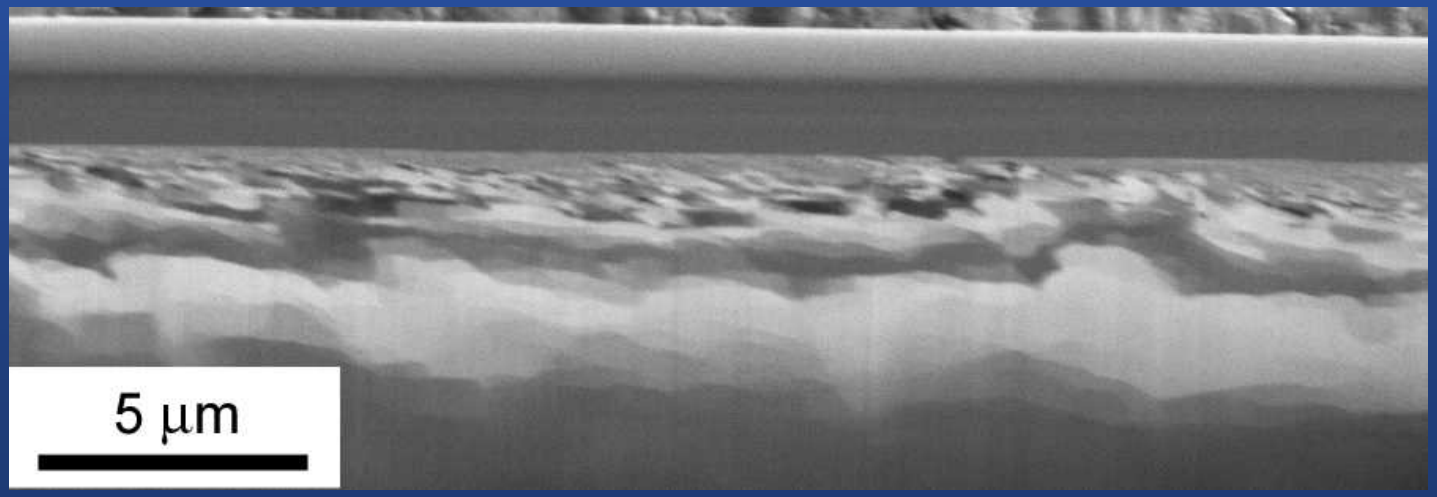
<211> on (111) Ni single crystal

Ion Channeling images provide qualitative measure of deformation

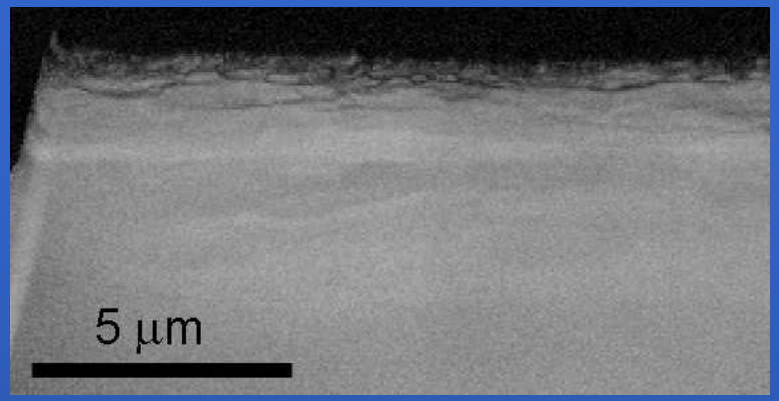
Cross sections of <110> on (111) Ni single crystals

10 gram load
1000 cycles

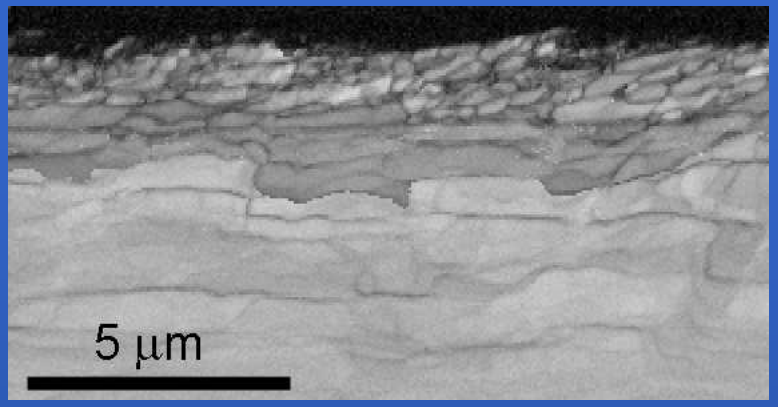
100 gram
load 1000
cycles

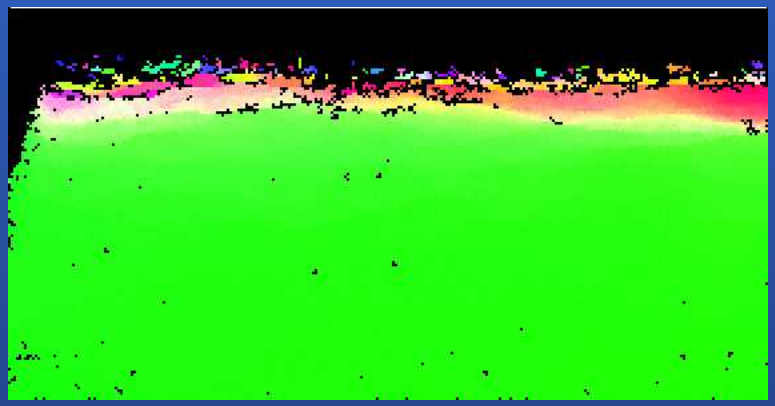


EBSD provides quantitative information (<110> on (111) Ni

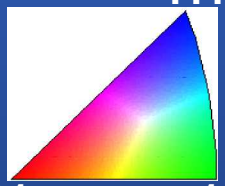


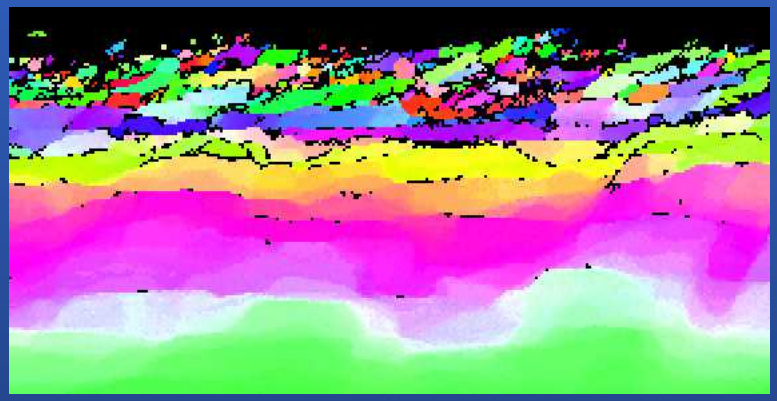
Band contrast

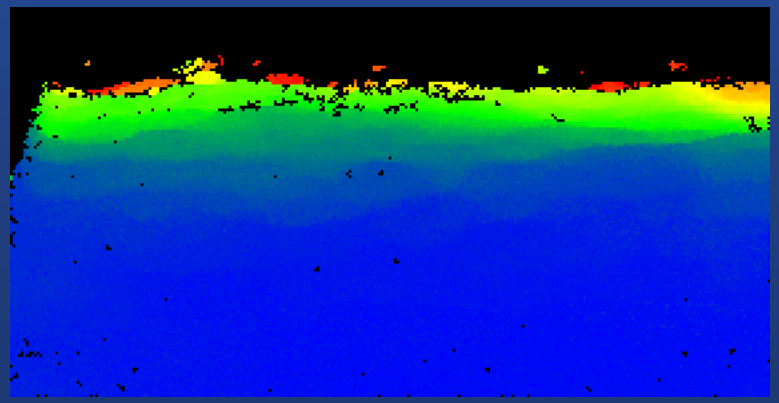




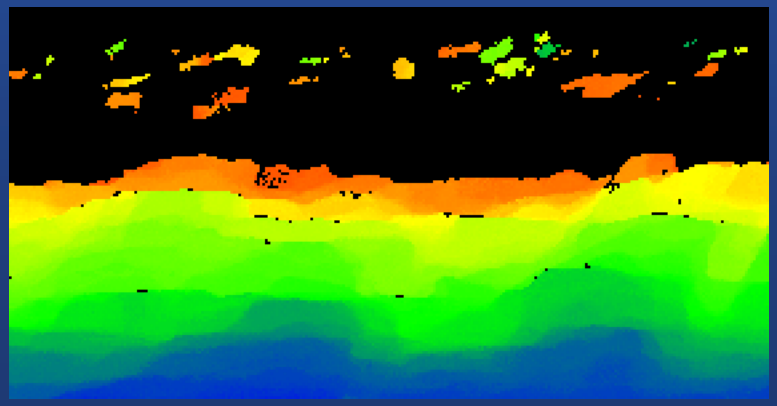
Sliding direction







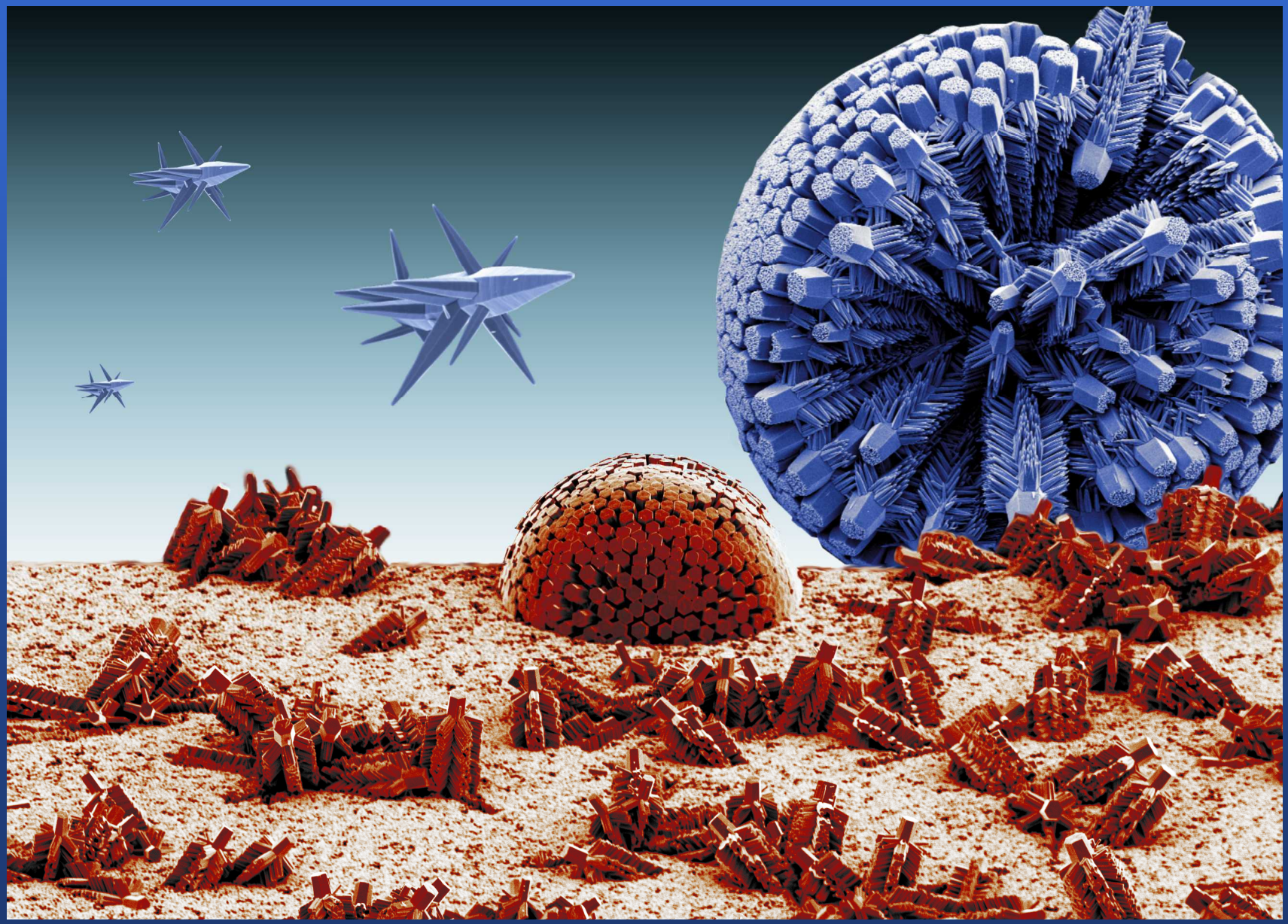
Orientation difference



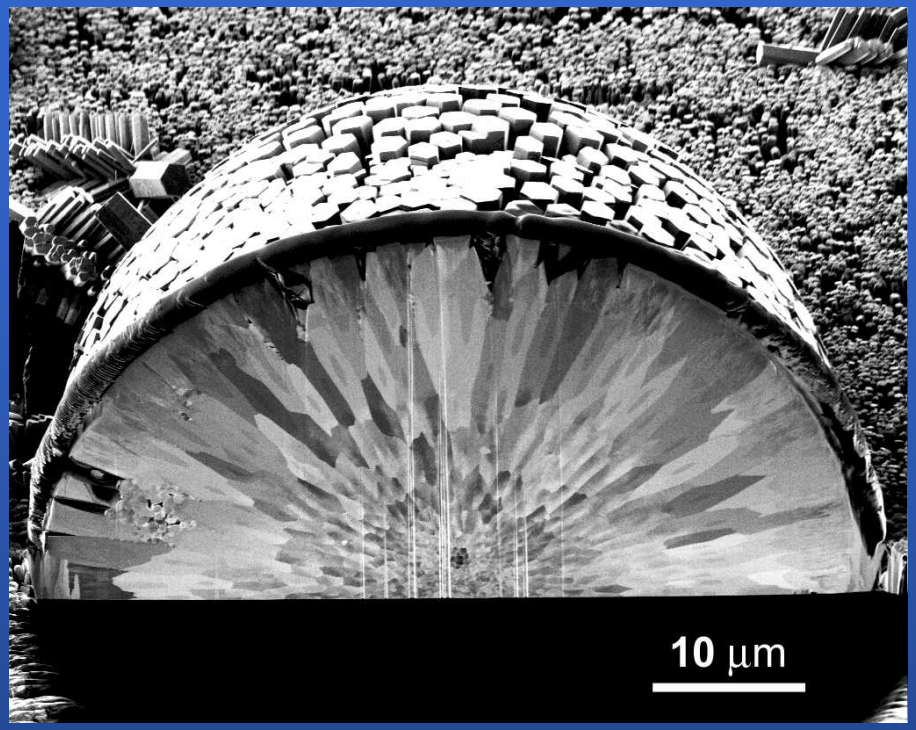
10 gram load for 1000 cycles

100 gram load for 1000 cycles

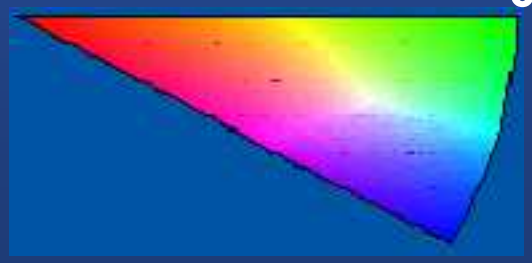
FIB and EBSD of ZnO Crystals



FIB and EBSD of ZnO Crystals

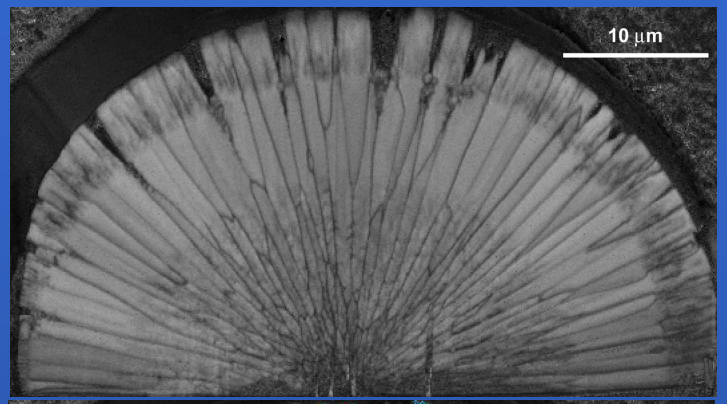


Ion induced SE image

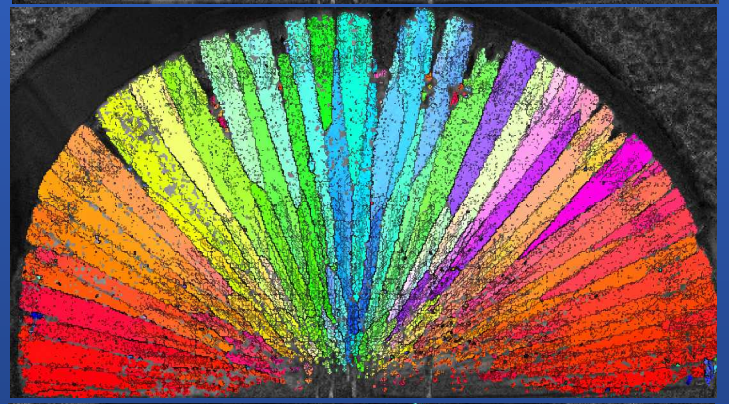


Color legend

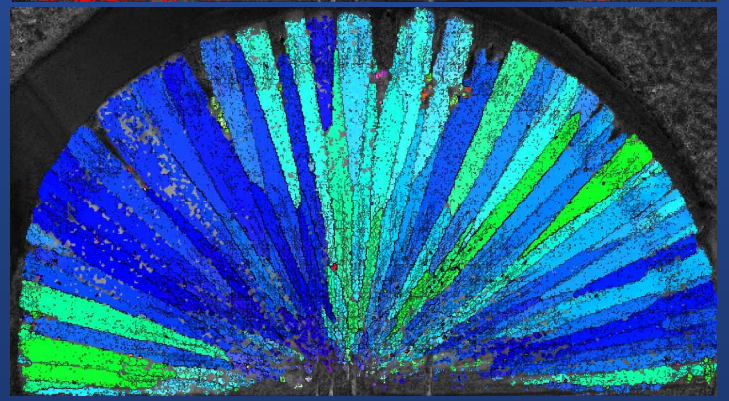
BC



IPF X

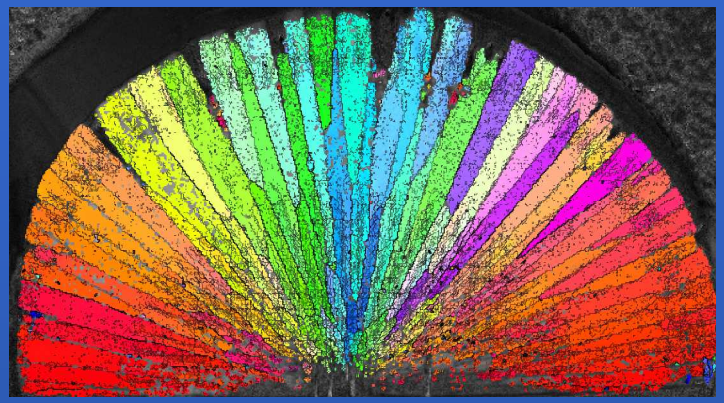


IPF Z

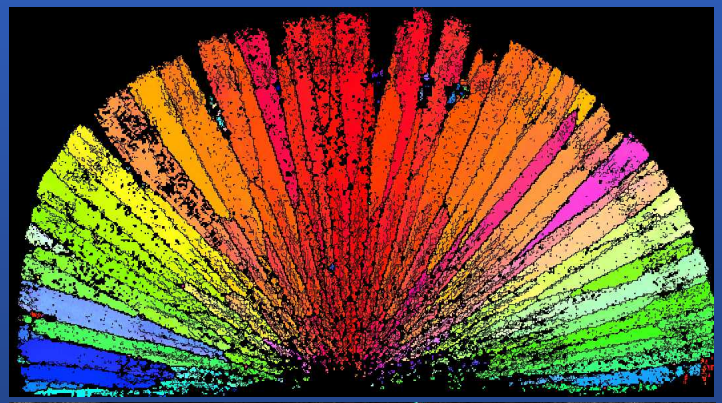


FIB and EBSD of ZnO Crystals

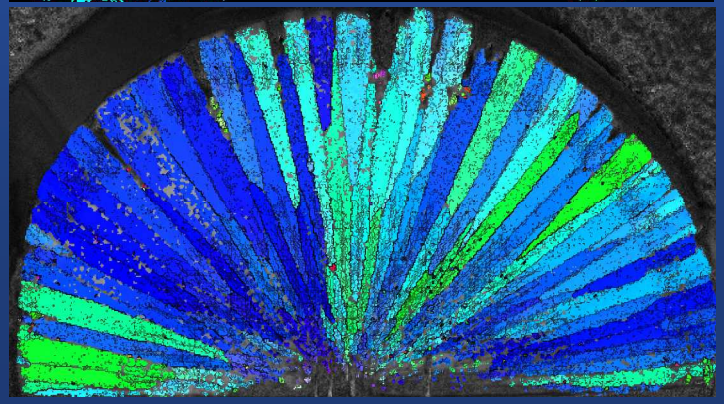
IPF X

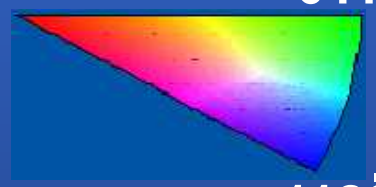


IPF Y

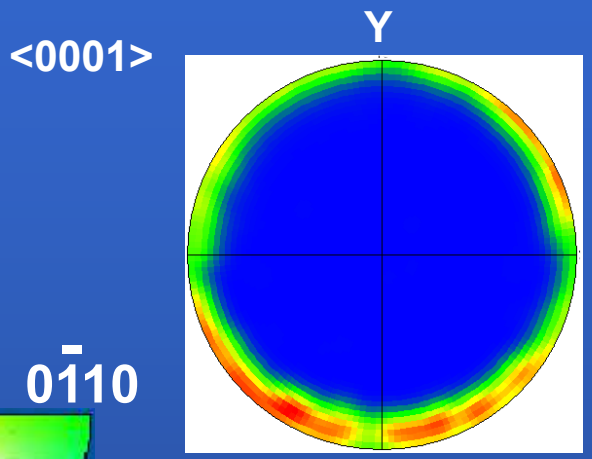


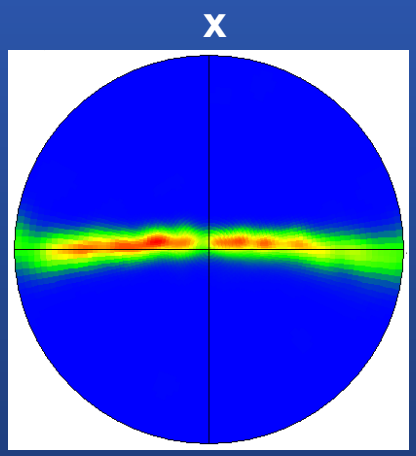
IPF Z





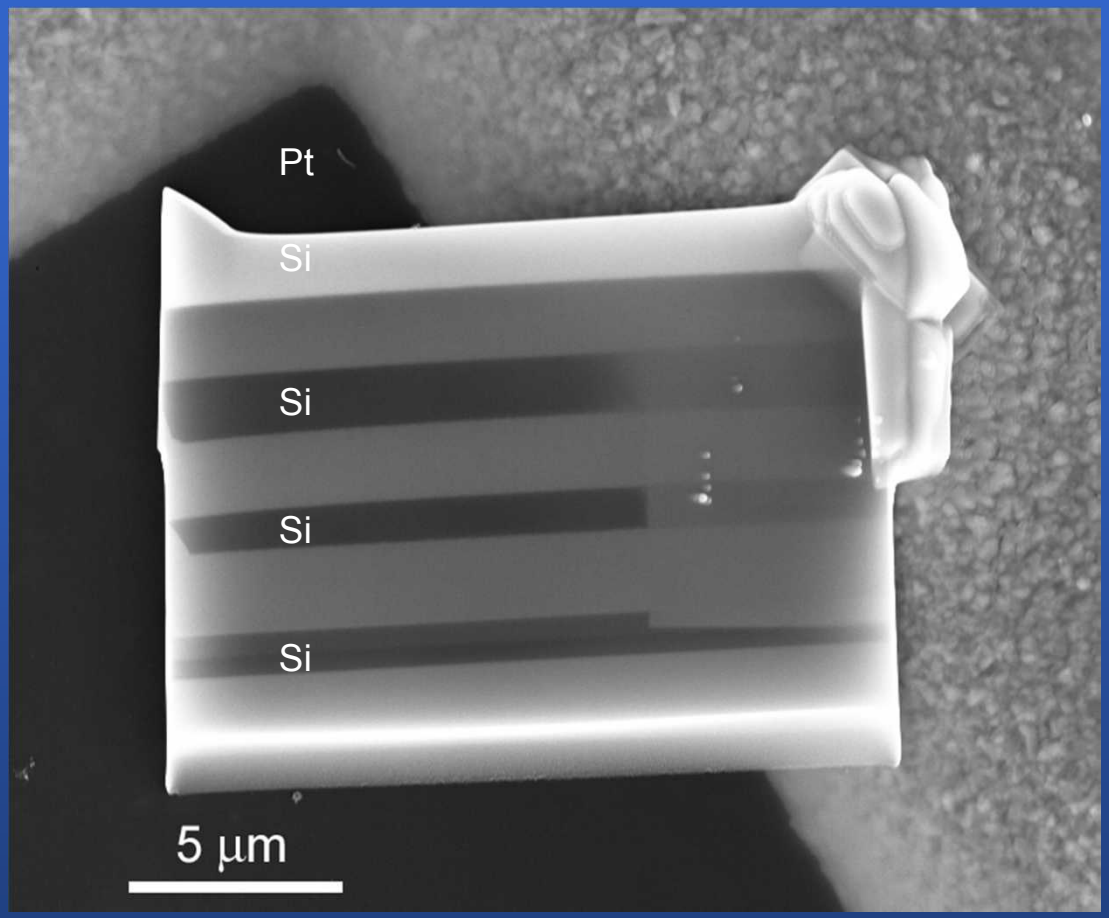
Color legend





All ZnO crystals grow with same crystallographic direction, geometry is a complicating factor.

FIB and EBSD of Silicon MEMS Structures



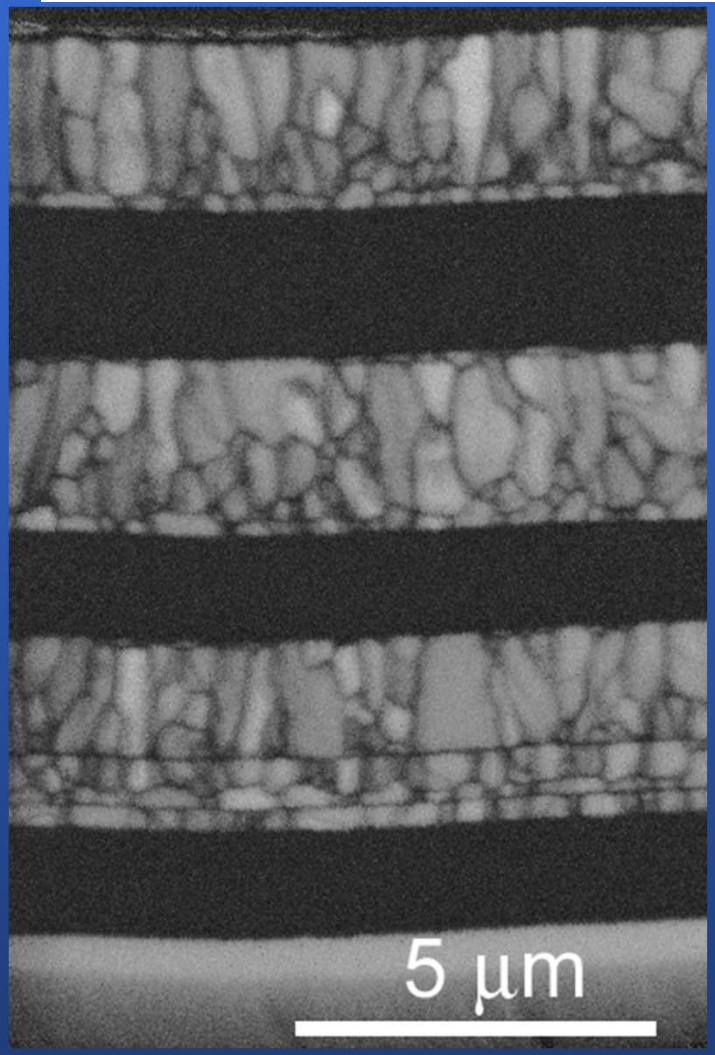
Many materials ca be imaged with EBSD after FIB sectioning.

Si requires low energy ion milling to produce surfaces suitable for EBSD measurements.

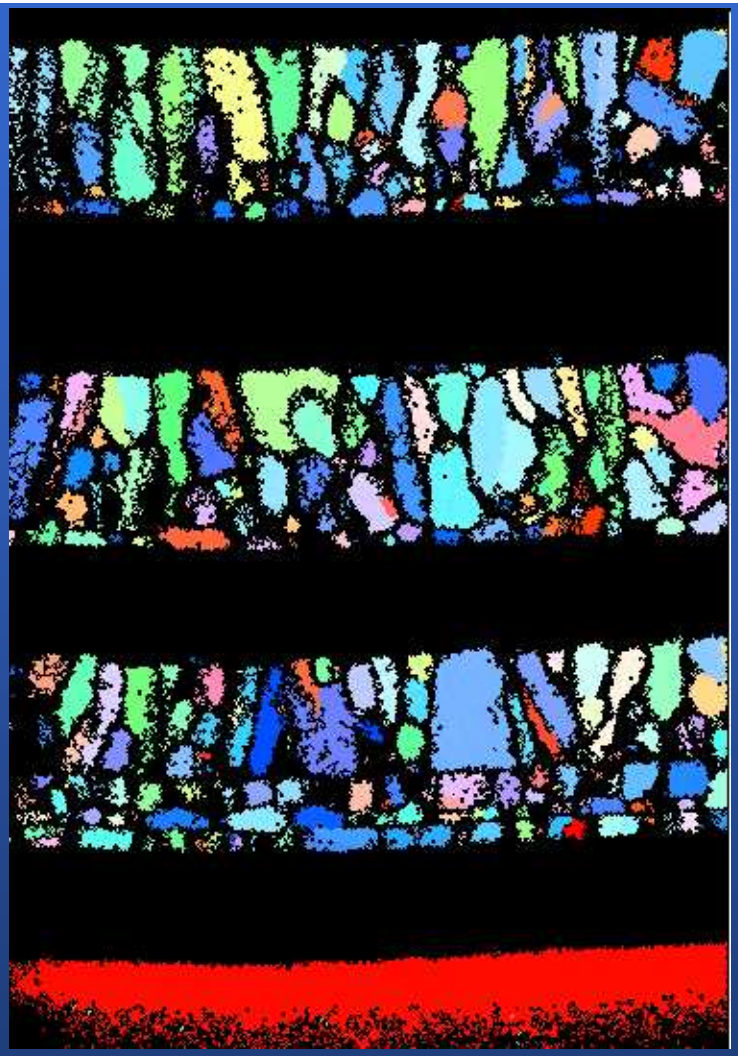
New low energy capable ion columns should make this even easier.

FIB section through unreleased multilayer MEMS device structure in polycrystalline silicon.

FIB and EBSD of Silicon MEMS Structures



Band Contrast Map



Orientation Map

EBSD maps are 600 X 500 pixels with a spacing of 25 nm

Summary

Ion milling normal to surface can result in damage, oreintation changes, recrystallization and Ga phase formation – not helpful for EBSD

Lift-out cross sections prepared by FIB are for most materials excellent for EBSD studies

Small features, like wear scars, are relatively easy to study using FIB and EBSD

FIB preparation of samples for EBSD is very useful for analysis of small features.