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Understanding microstructural changes in metals induced by Gallium ion beam irradiation

Joseph R. Michael
Materials Science Center
Sandia National Laboratories
Albuquerque, NM 87185-0886

U.S. DEPARTMENT OF ENERGY

NNSA

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The Problem – dark imaging areas appear with Ga+ exposure

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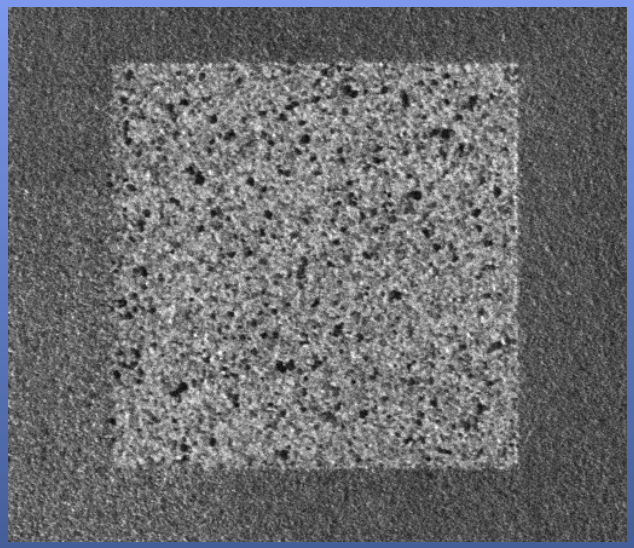
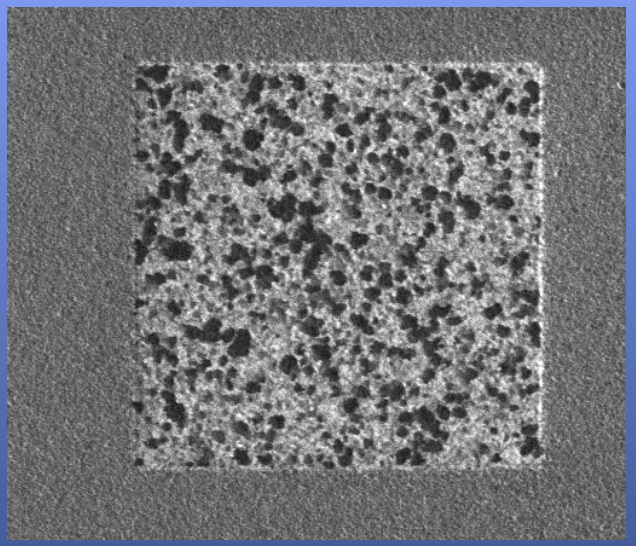
1 min

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



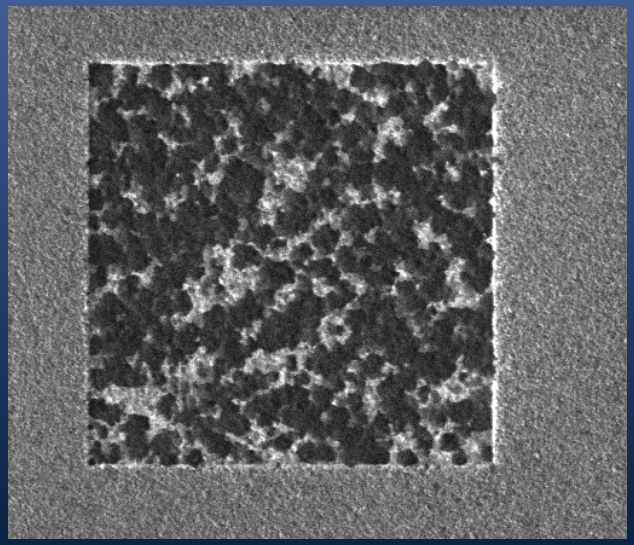


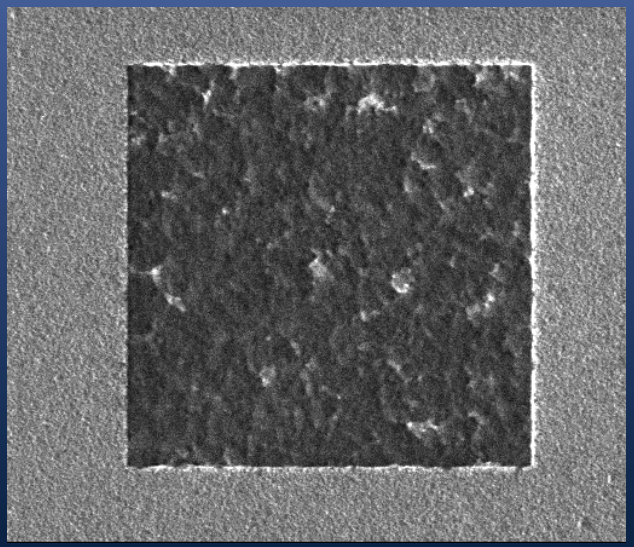
3 min

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

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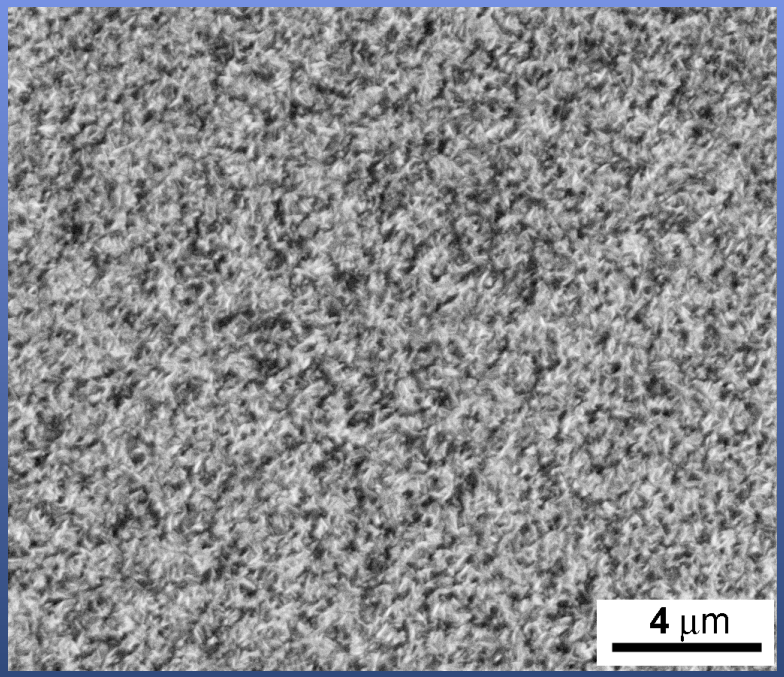
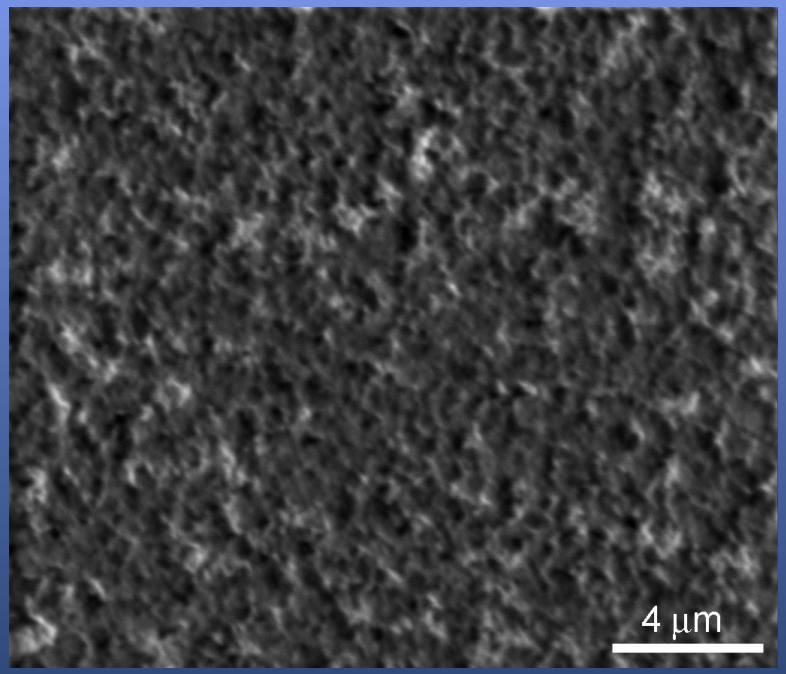


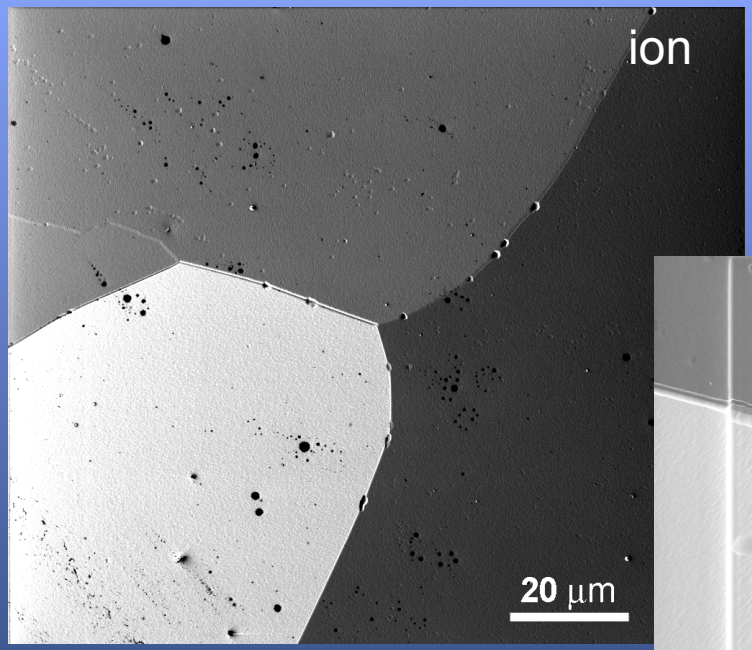
1 frame at 1 nA



20 frames at 1 nA

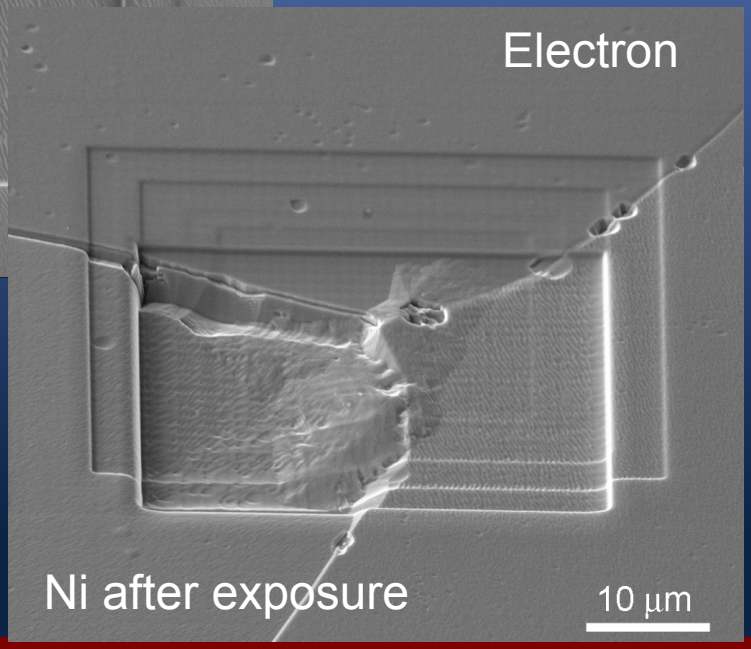
Evaporated W sample irradiated with 30 kV Ga+

Dark grains occur in coarse grained materials also



Ni before exposure

Ni after exposure



Coarse grained requires higher ion dose for orientation change

The Problem – dark imaging areas appear with Ga+ exposure

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

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

Some indication that this happens in tin(tetragonal)

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?

What are the dark areas after Ga^+ ion irradiation?

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Possible explanation from the literature:

Differential sputter yield from channeling orientations is much lower than random orientations – thus non-channeling orientations are removed leaving channeling oriented grains.

Differential damage model – the accumulation of ion damage is less in channeling orientations leading to movement of grain boundaries into the more damaged regions resulting in growth of grains with oreintations favorable to channeling.

Calculated channeling directions in Au, Cu and W

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Direction	Au X_0	Cu X_0
[110]	0.150	0.132
[100]	0.252	0.222
[112]	0.342	0.300
[130]	0.502	0.441
[111]	0.575	0.510

Direction	W X_0
[111]	0.146
[100]	0.181
[110]	0.303
[112]	0.695

X_0 = unchanneled fraction of ions in that direction

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

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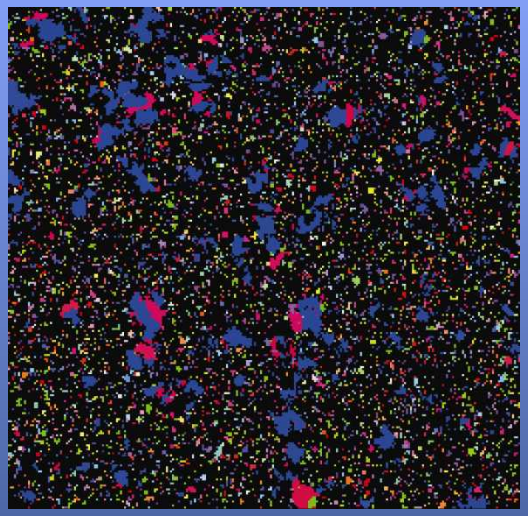
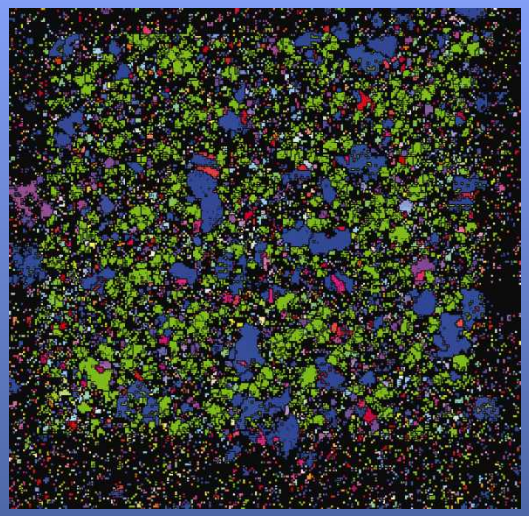
1 min

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



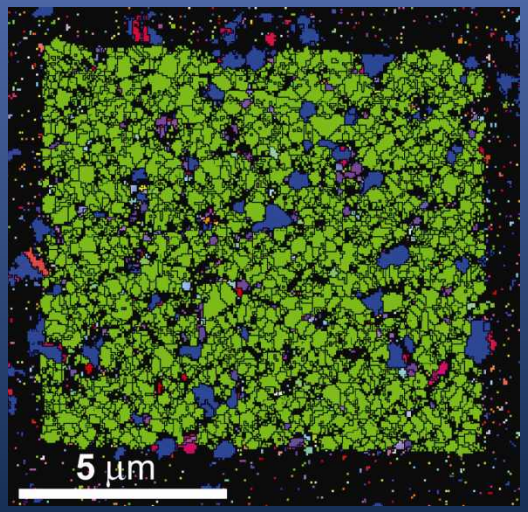


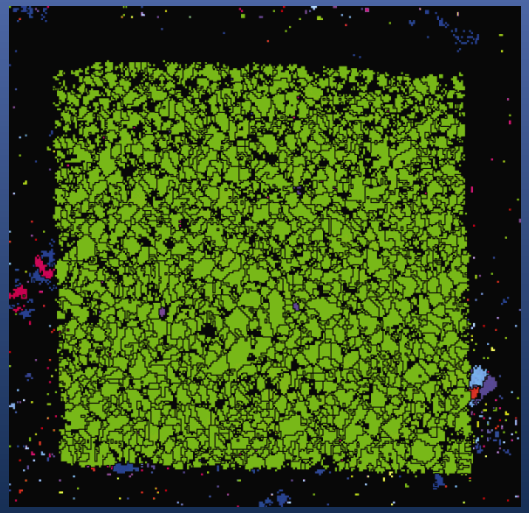
3 min

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

6 min

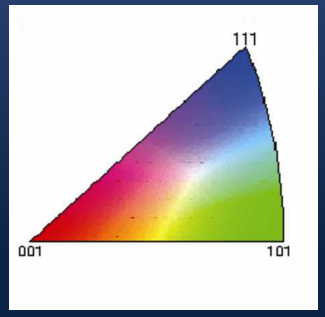
6.8 x 10^{16} $Ga^+/\mu m^2$





10 min

1.1 x 10^{17} $Ga/\mu m^2$

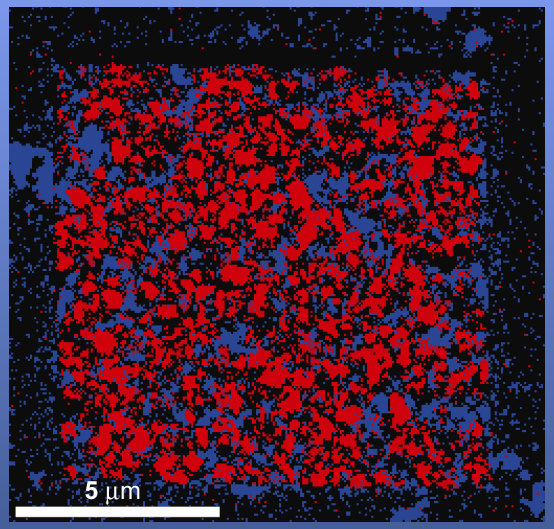


Strongest channeling direction in Cu is <110>

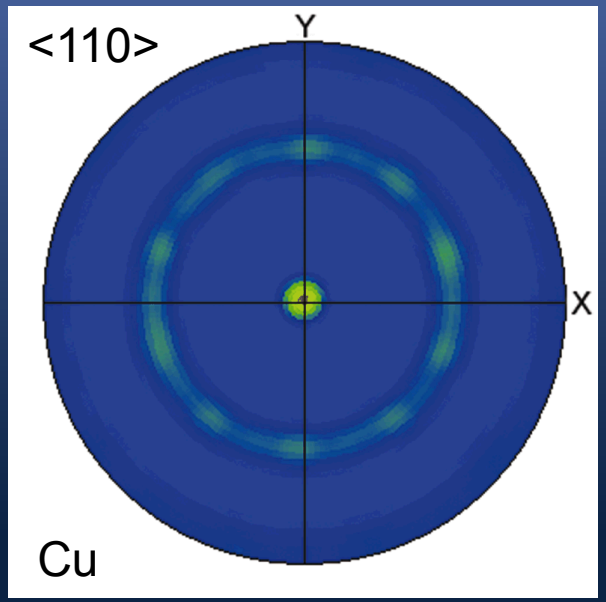
Phase Distributions in ion milled regions of fine-grained Cu

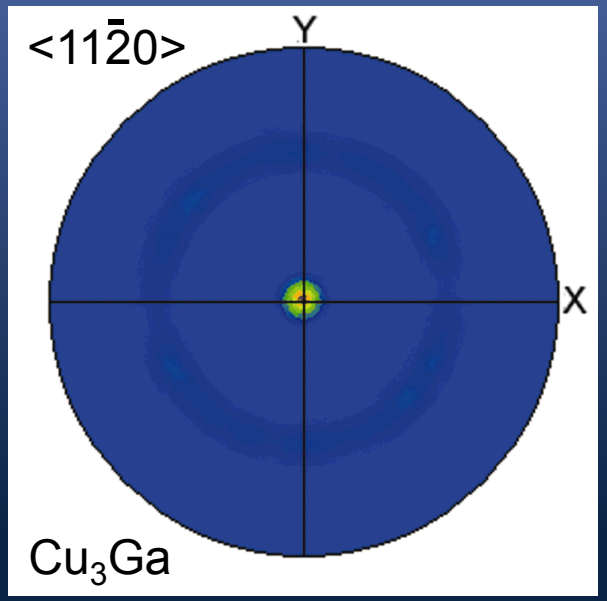
Red = Cubic phase (Cu)

Blue = Cu_3Ga (hexagonal)

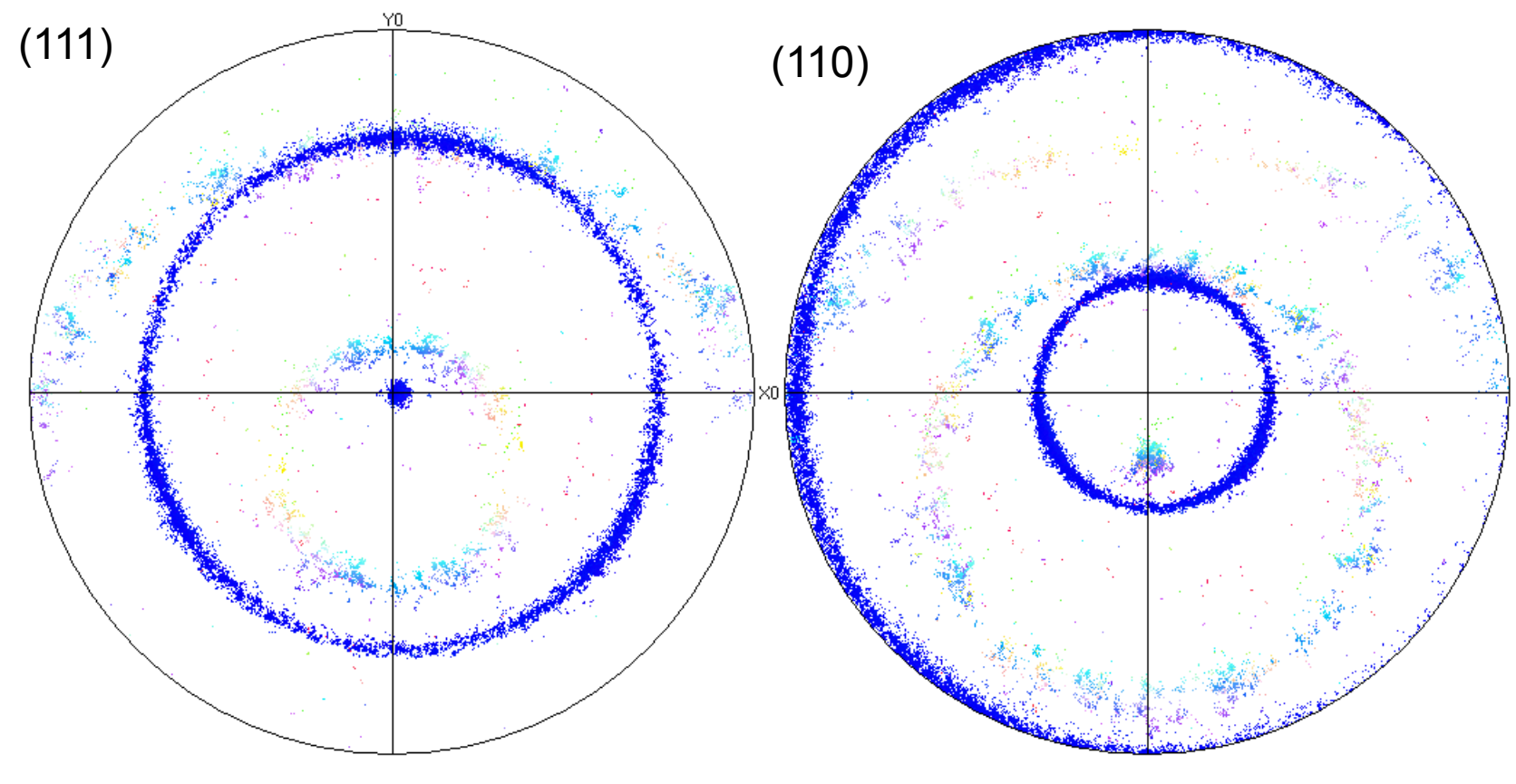


Both the Cu and the Cu_3Ga are highly textured





New grains form with respect to the ion beam direction



Ga+ ion beam tilted 23˚ with respect to normal

New (110) texture develops along beam direction

STEM imaging and microanalysis of Ga+ into Cu

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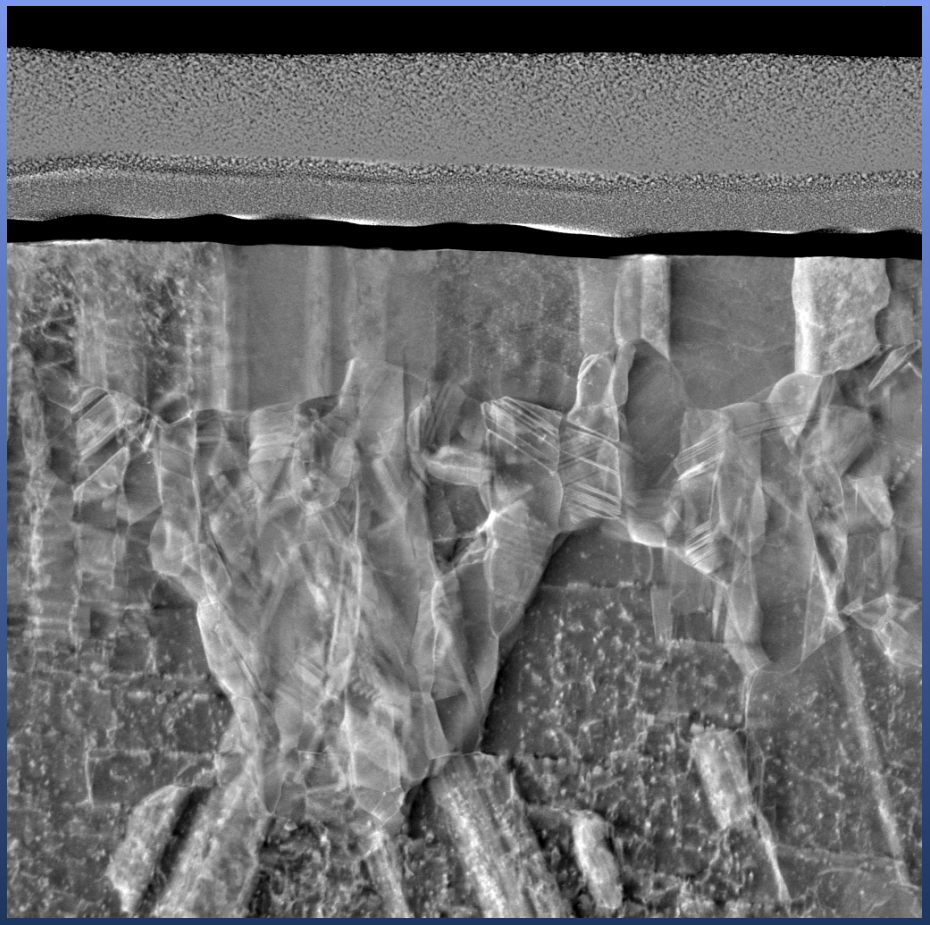
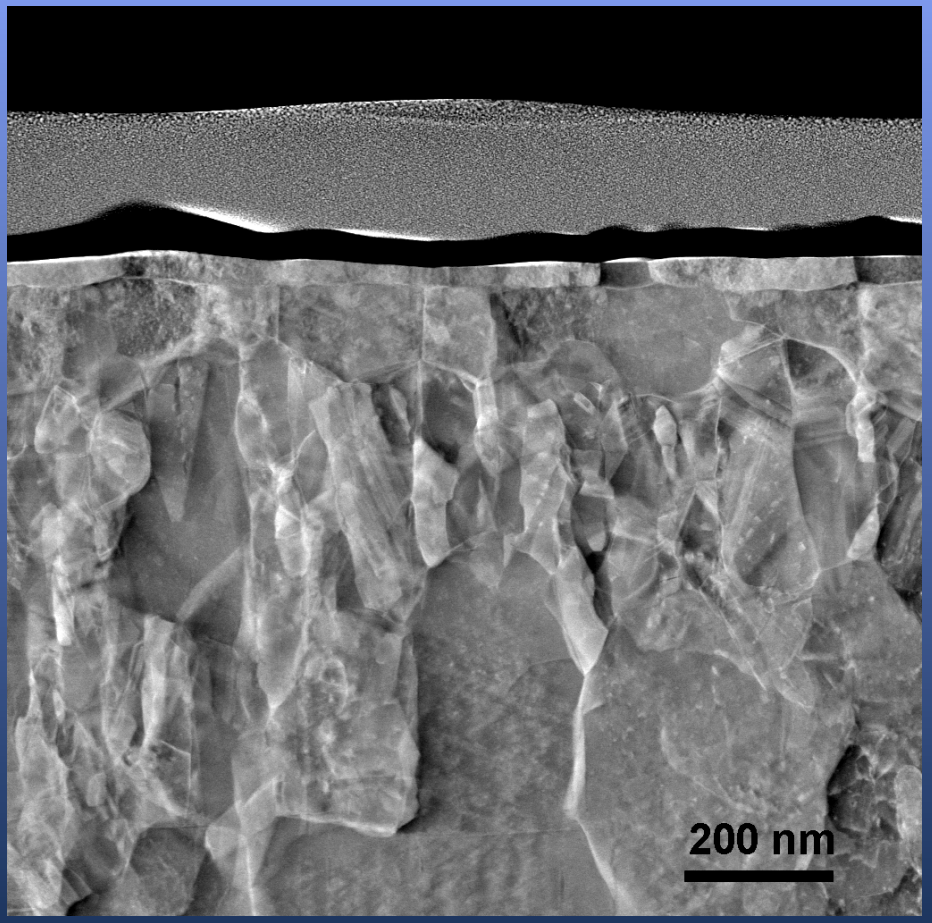


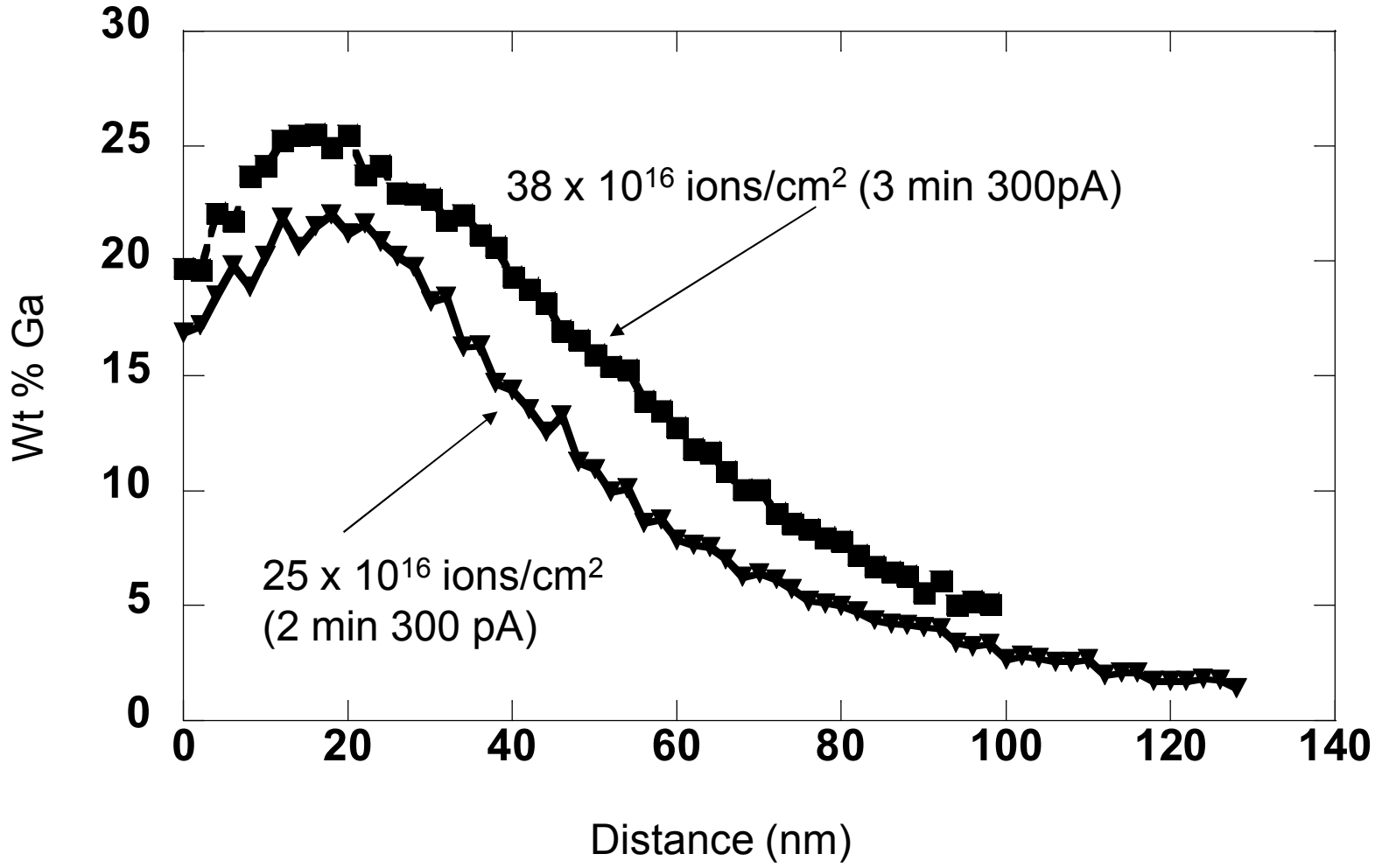


2 min 330 pA - 2.5 x 10^{16} $Ga^{+}/\mu m^{2}$

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

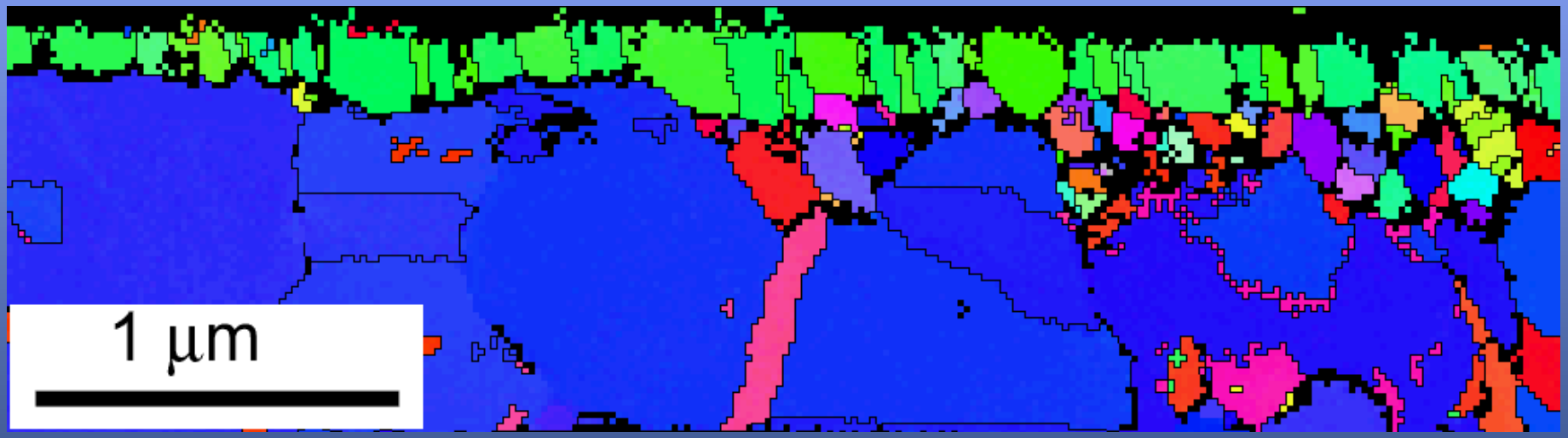
SRIM estimates the range of 30 Ga+ in Cu to be 10 nm

STEM imaging and microanalysis of Ga+ into Cu

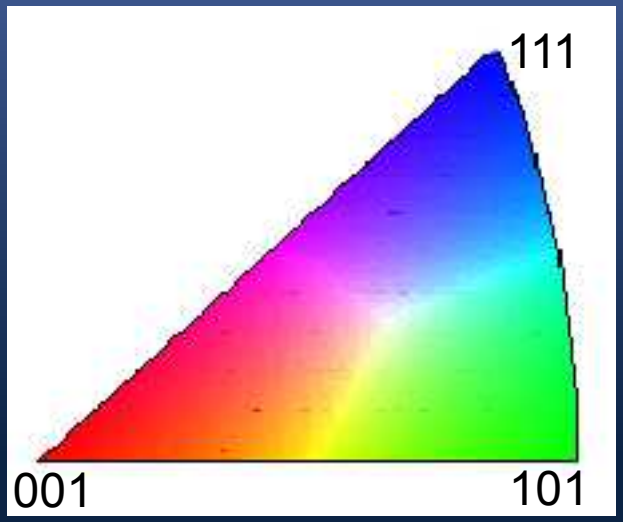


Ga+ implantation is not insignificant!

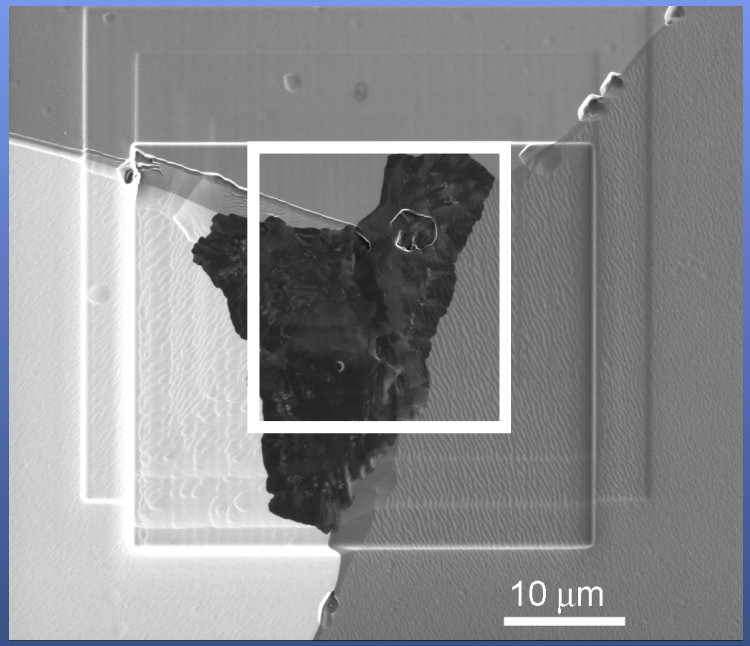
EBSD orientation mapping of Ga+ milled Cu

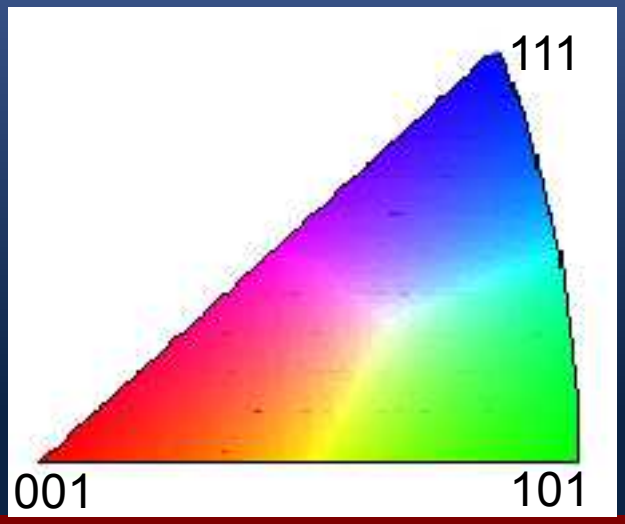


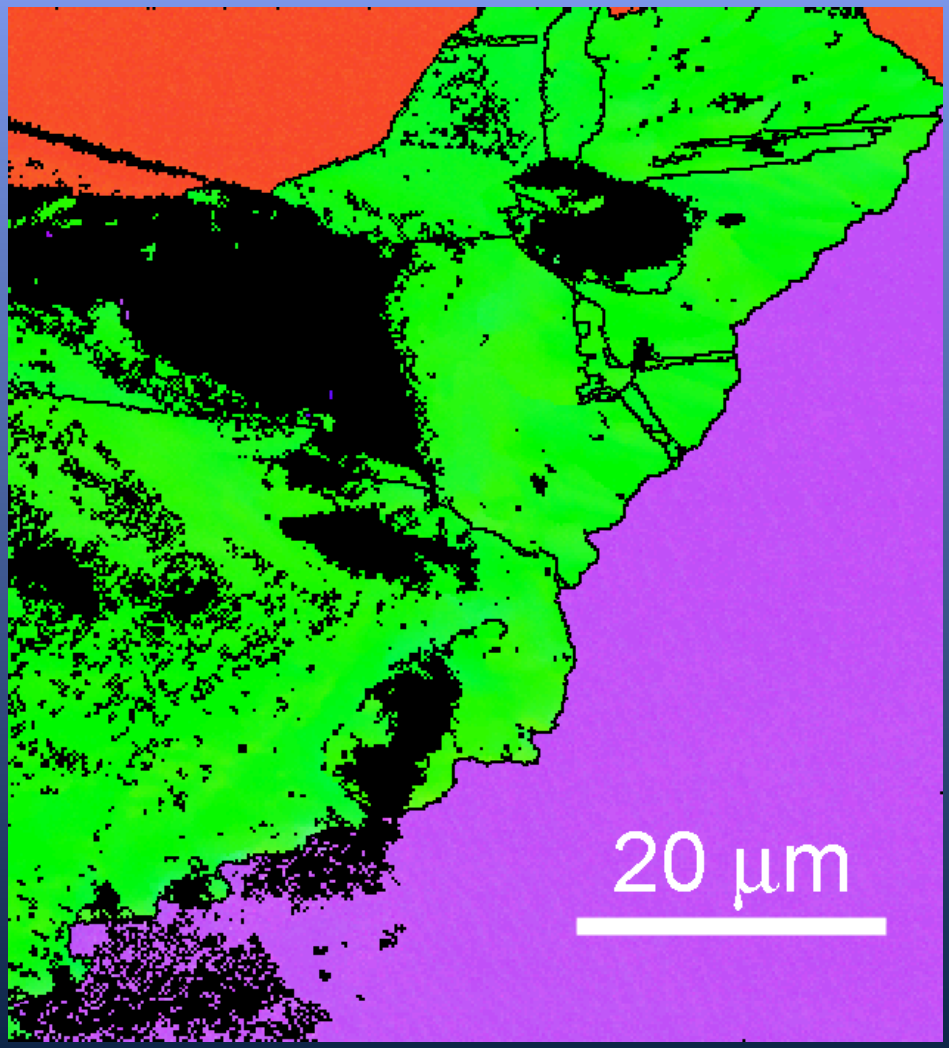
2 min 330 pA - 2.5 x 10^{16} $Ga^{+}/\mu m^{2}$



Reorientation occurs in coarse grained materials also (Ni)







Orientation changes in Ga^+ irradiated fine-grained Au

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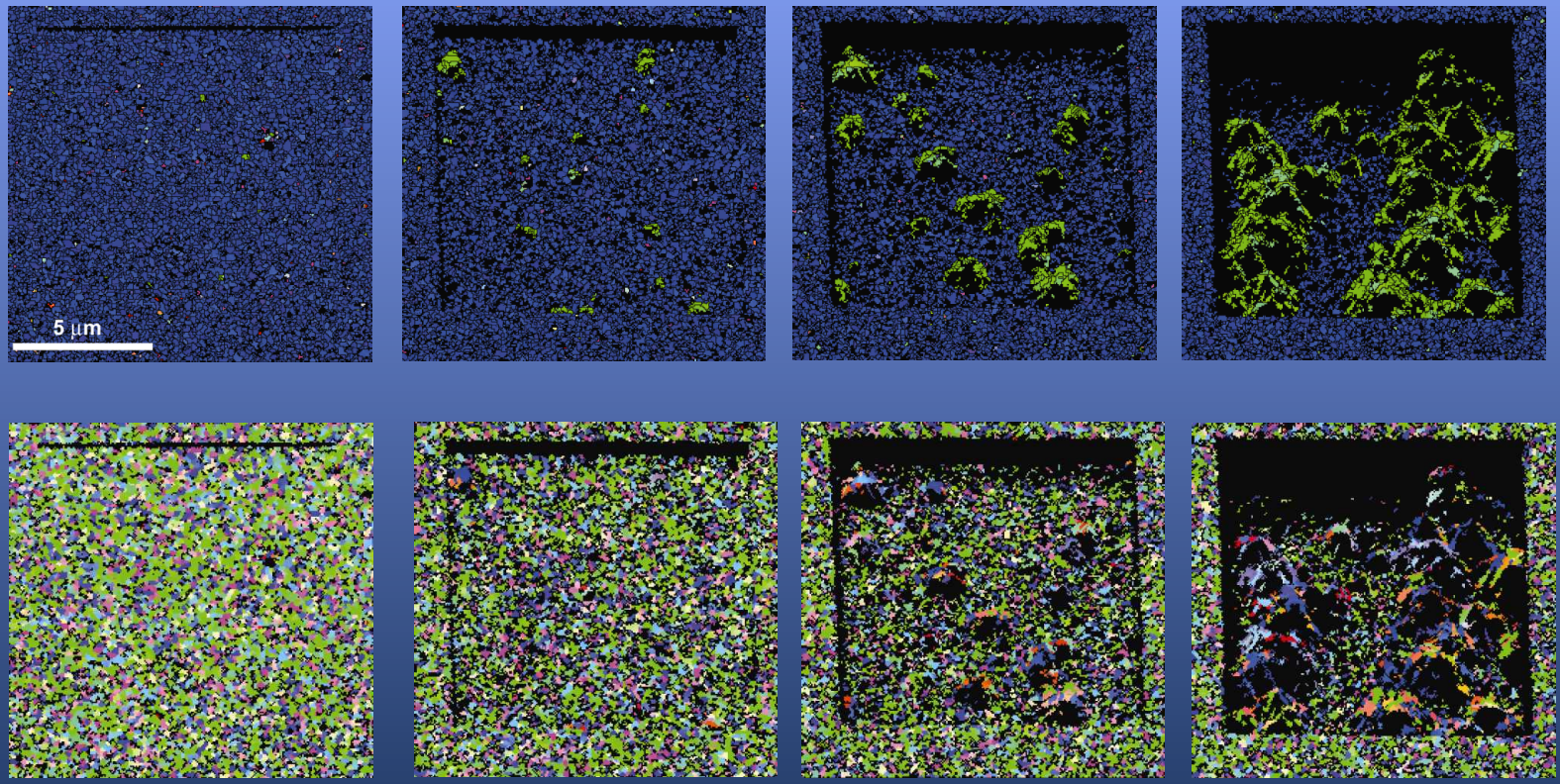
Surface Normal

In-plane

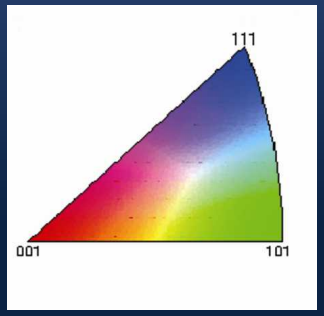


1.8×10^{16} $Ga^+/\mu m^2$

5.2×10^{16} $Ga^+/\mu m^2$

1.1×10^{17} $Ga^+/\mu m^2$

2.1×10^{17} $Ga^+/\mu m^2$



Strongest channeling direction in Au is <110>

Orientation changes in ion milled regions of fine-grained W

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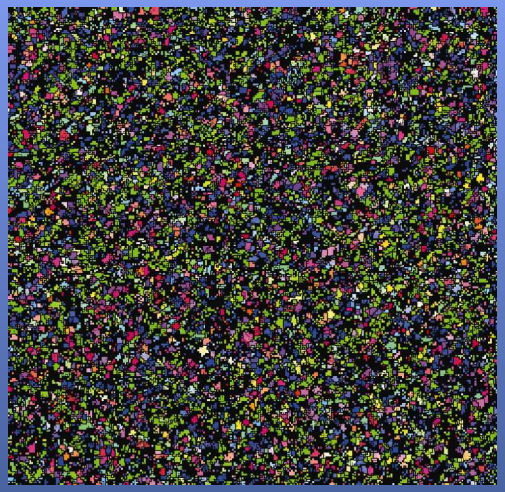
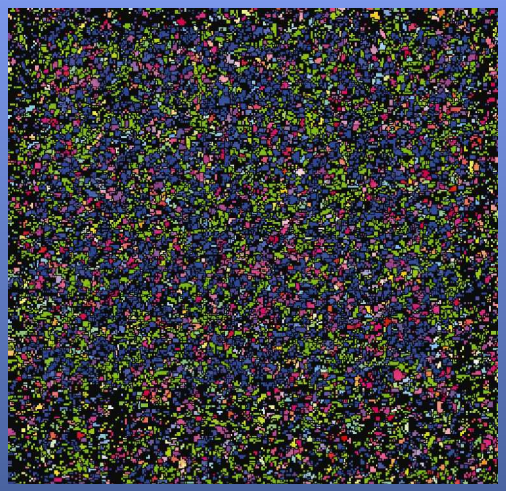
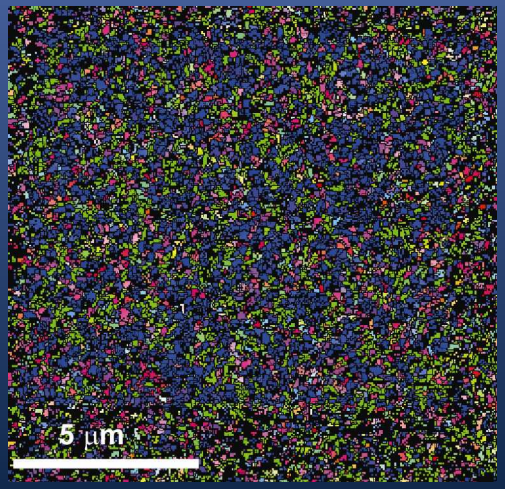
As-deposited

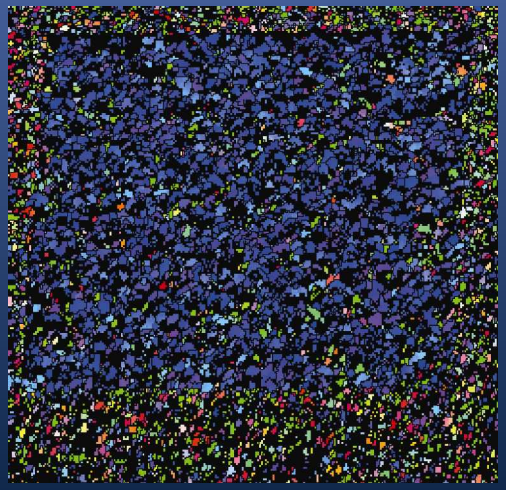




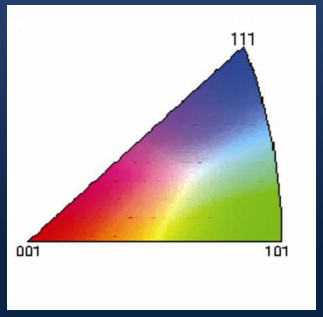
3.6×10^{16} $Ga^{+}/\mu m^{2}$

5.4×10^{16} $Ga^{+}/\mu m^{2}$





2.1×10^{17} $Ga/\mu m^{2}$



Strongest channeling direction in W is <111>

Orientation changes in ion milled regions of fine-grained W

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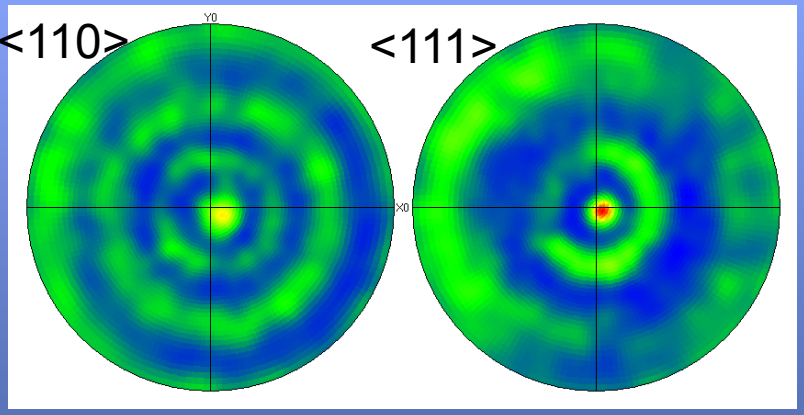
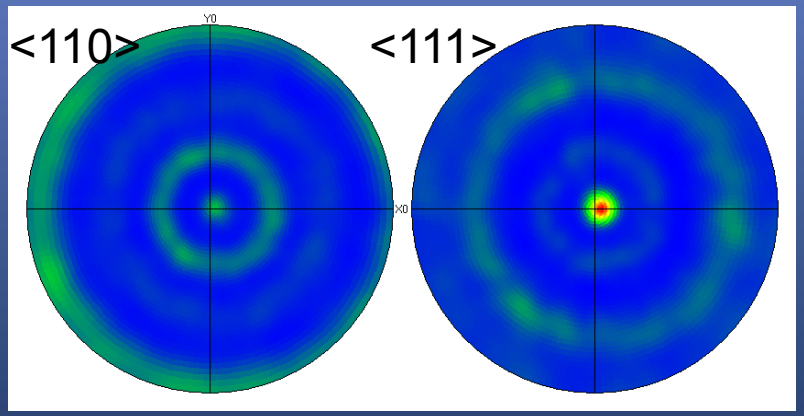
As-deposited



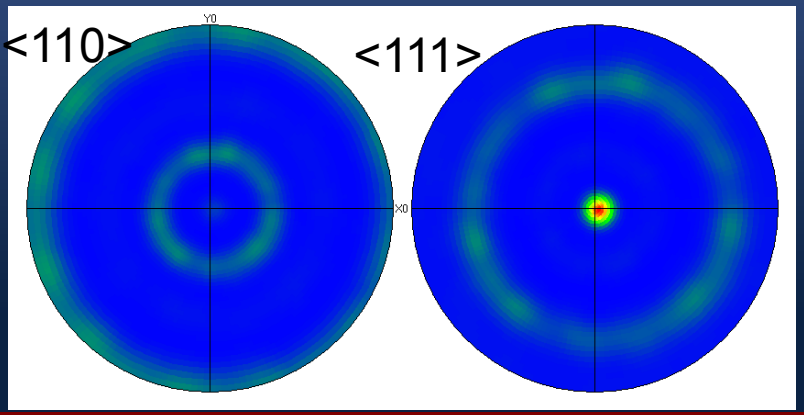
3 times random

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

The Problem – Dark grains do appear in many metals

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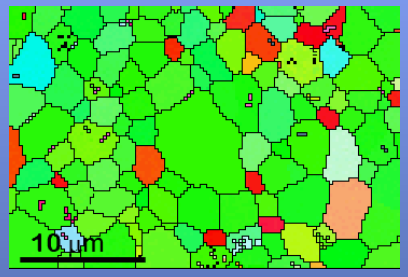
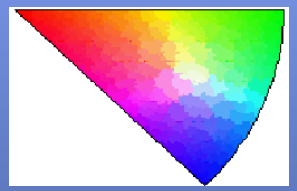
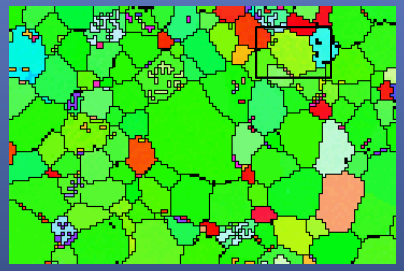
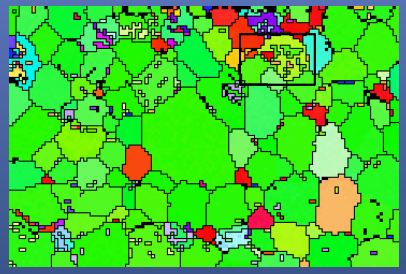


Tin
As-sputtered

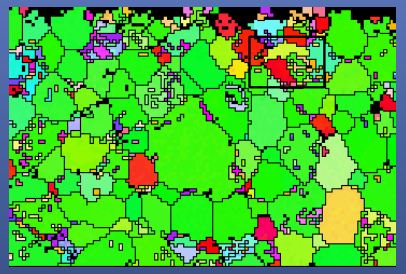




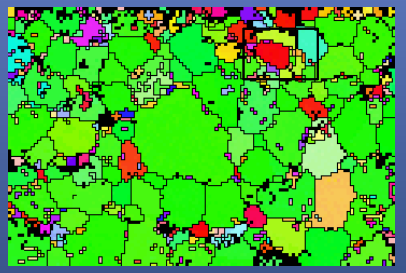
$3x10^{16}$ cm^{-2}



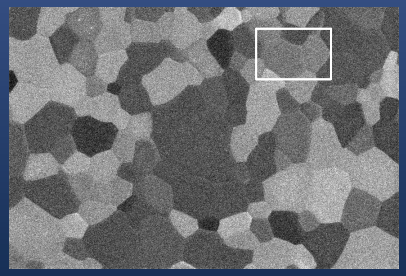
$6x10^{16}$ cm^{-2}



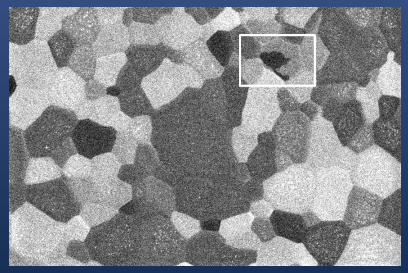
$9x10^{16}$ cm^{-2}



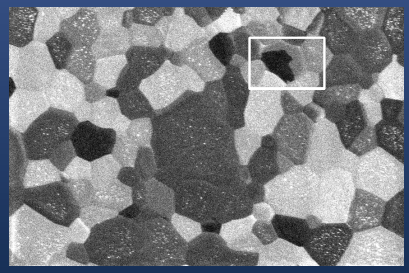
$1.8x10^{17}$ cm^{-2}



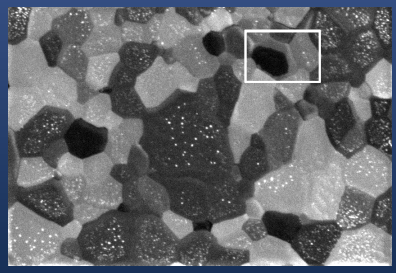
460 pA for 60 s



460 pA for 120 s

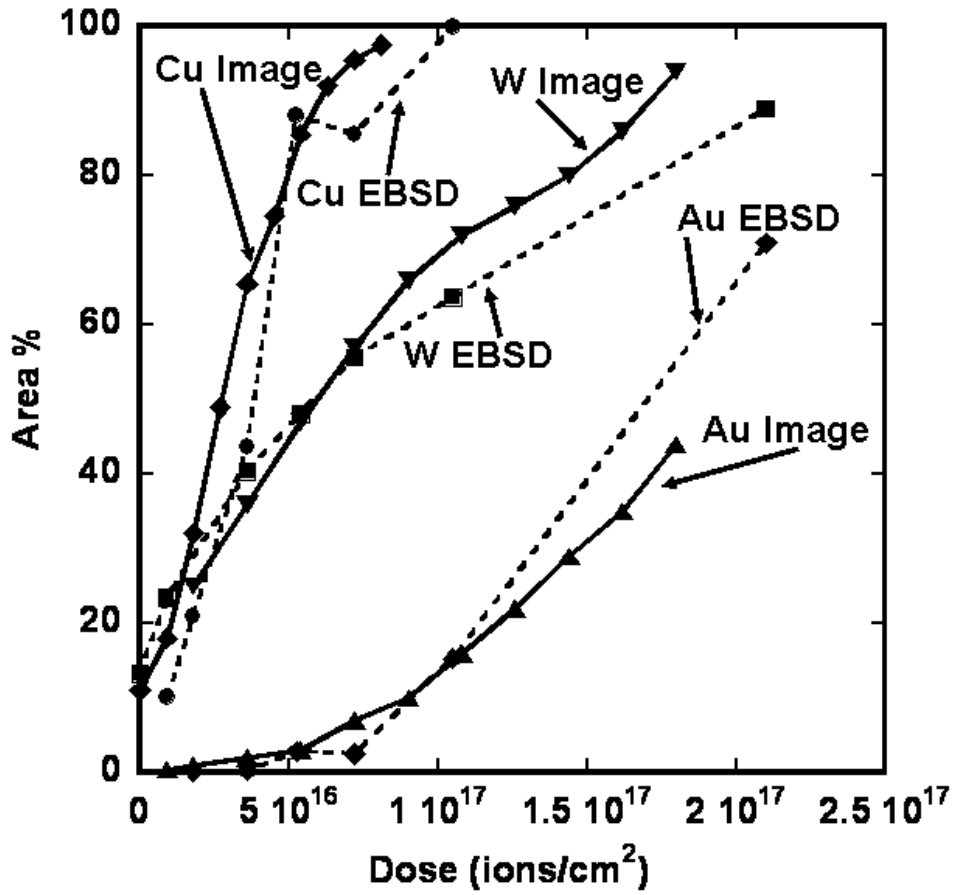


460 pA for 180 s



460 pA for 360 s

Texture development with ion dose



Sputter yield for 30 kV Ga

Cu =10

Au = 17

W = 7

Sn = 8

Summary of observations

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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).

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.

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

Possible Mechanisms

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Differential sputter yield from channeling orientations is much lower than random orientations – thus non-channeling orientations are removed leaving channeling oriented grains.

This does not seem to be reasonable given that the rate of texture evolution is not related to sputter yield.

Differential damage model – the accumulation of ion damage is less in channeling orientations leading to movement of grain boundaries into the more damaged regions resulting in growth of grains with oreintations favorable to channeling.

Possible- but we do not see any intermediate textures developing

Texture evolution must be a result of ion beam-assisted nucleation of new grains with the easiest channeling direction aligned with the ion beam direction.

Implications to FIB sample preparation

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No ion-induced microstructural changes have been noted in grazing incidence orientations.

During normal preparation of cross sections for SEM or TEM applications there should be no concerns provided that the ion beam is never used to directly image the sample!

Proper FIB practice of protecting the surface of the sample with Pt, W or C should eliminate any microstructural changes in the sample during imaging.

Additional care should be used when working with fine-grained or nano-crystalline samples as these are most susceptible to ion beam induced microstructural alterations.