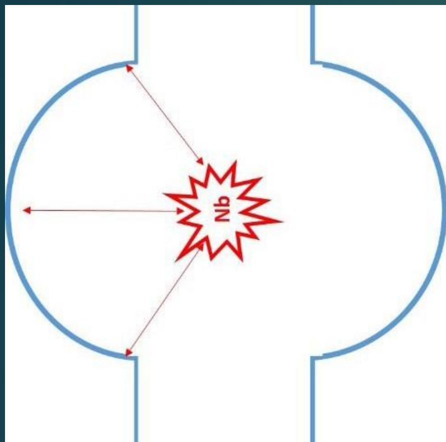
EBSD analysis of Nb layer, deposited on Cu substrate

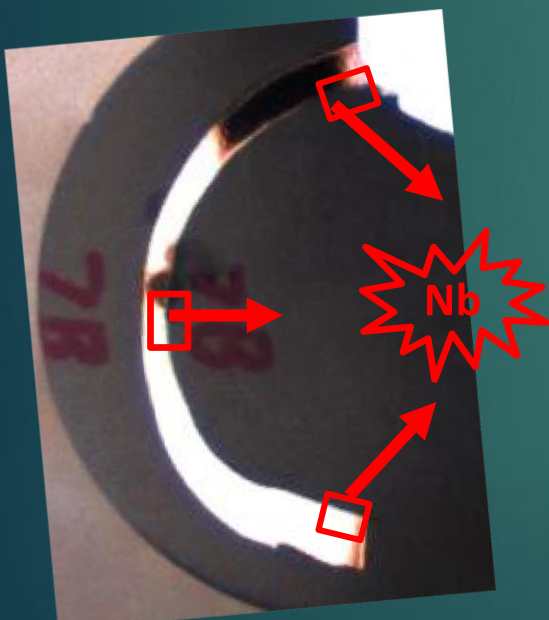
Sample Preparation Techniques

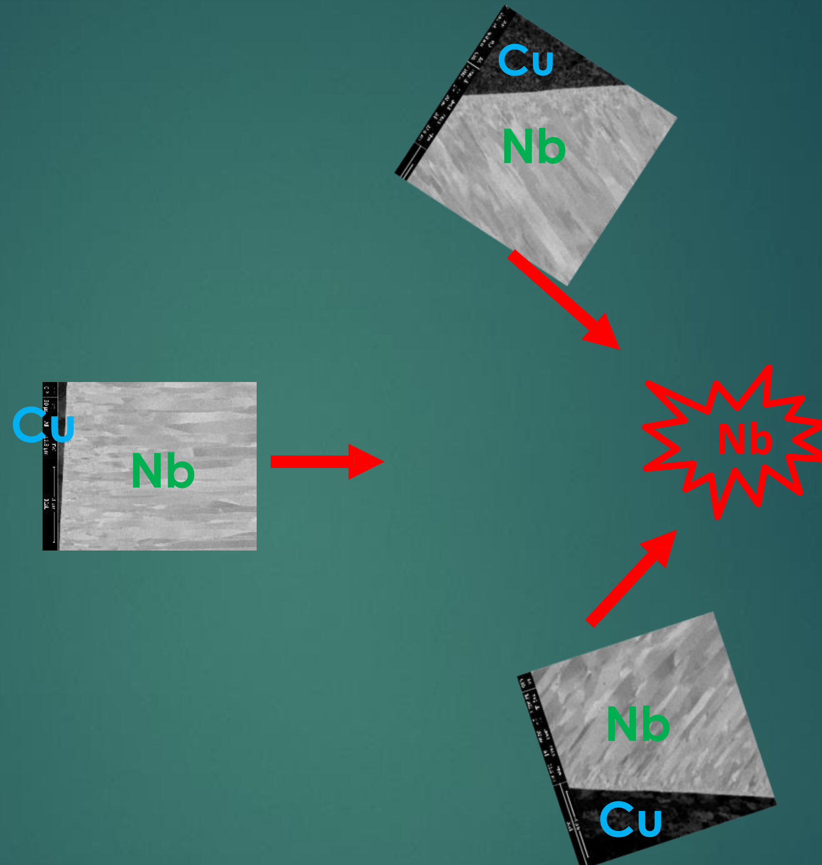
- Conventional metallurgical specimen preparation: Grinding (SiC papers), followed by 1 hour 40nm colloidal silica polish
- Hitachi IM4000 Plus, Broad Argon Ion Beam: Cross-section milling under vacuum, 5kV Ar beam energy, 2 hour mill time

SEM – Layer morphology



Consistent with previous observations the growth of the columnar Nb grains is influenced by the positioning of the Nb source

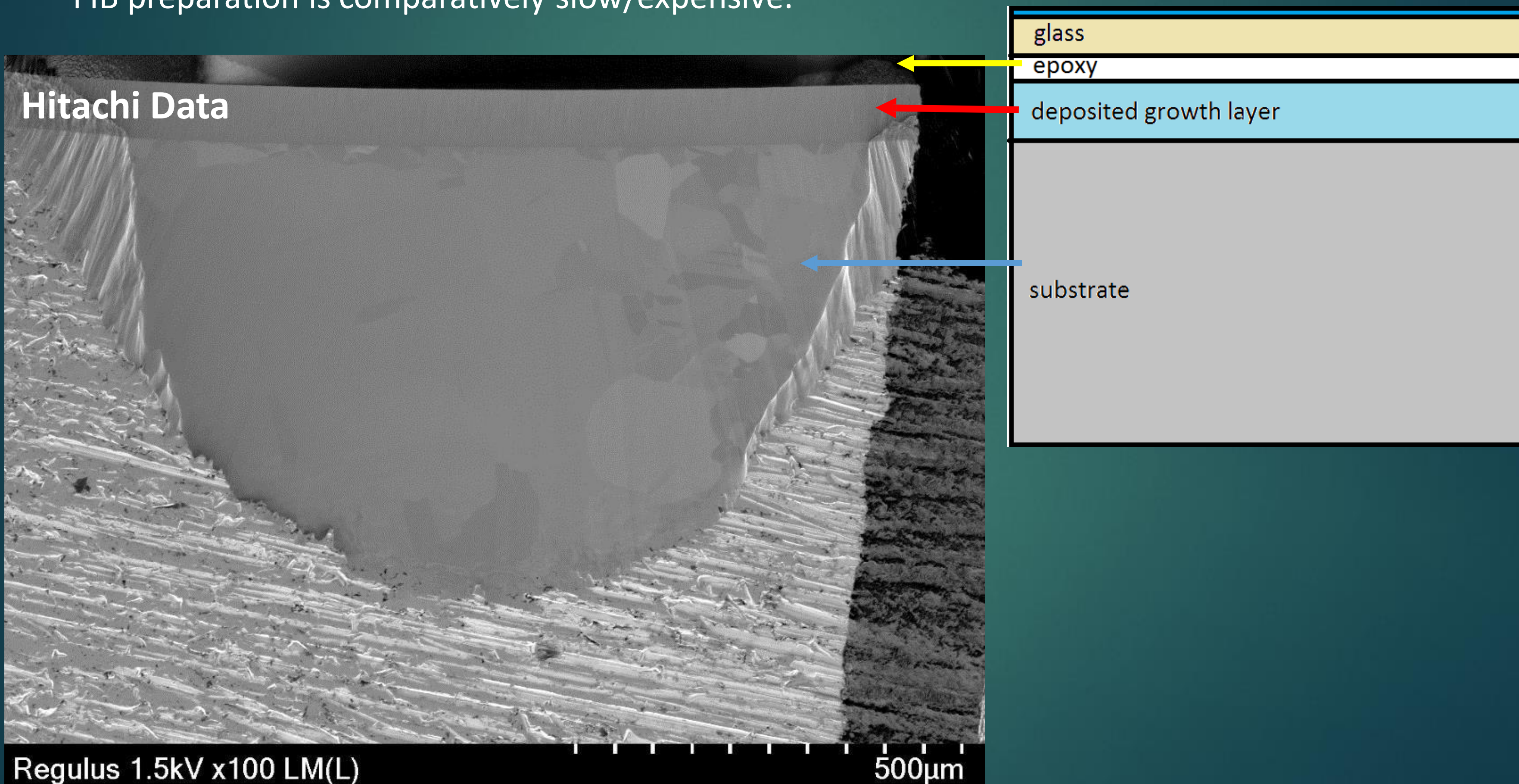




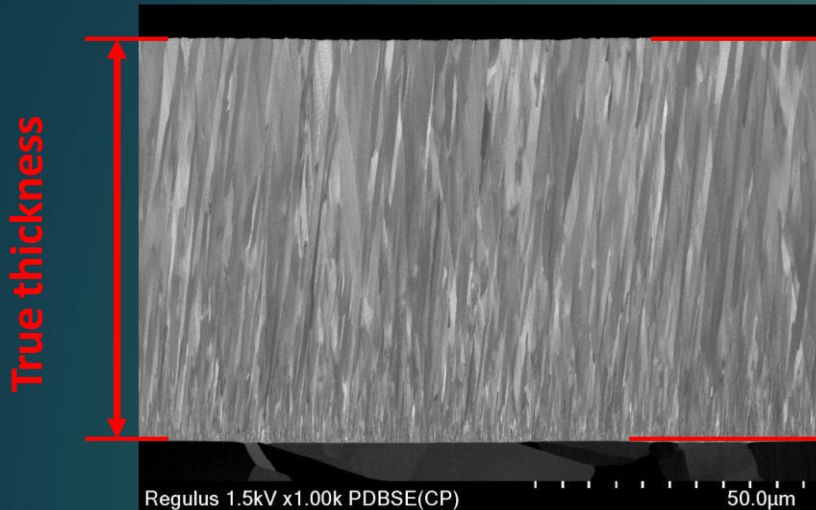
In general, columnar grains grow toward the source. Therefore, grains located close to the curve midpoint tend to orient perpendicular to the substrate/Nb interface, whereas grains at either end of curve tilt toward the

Hitachi IM4000 – Ar ion beam

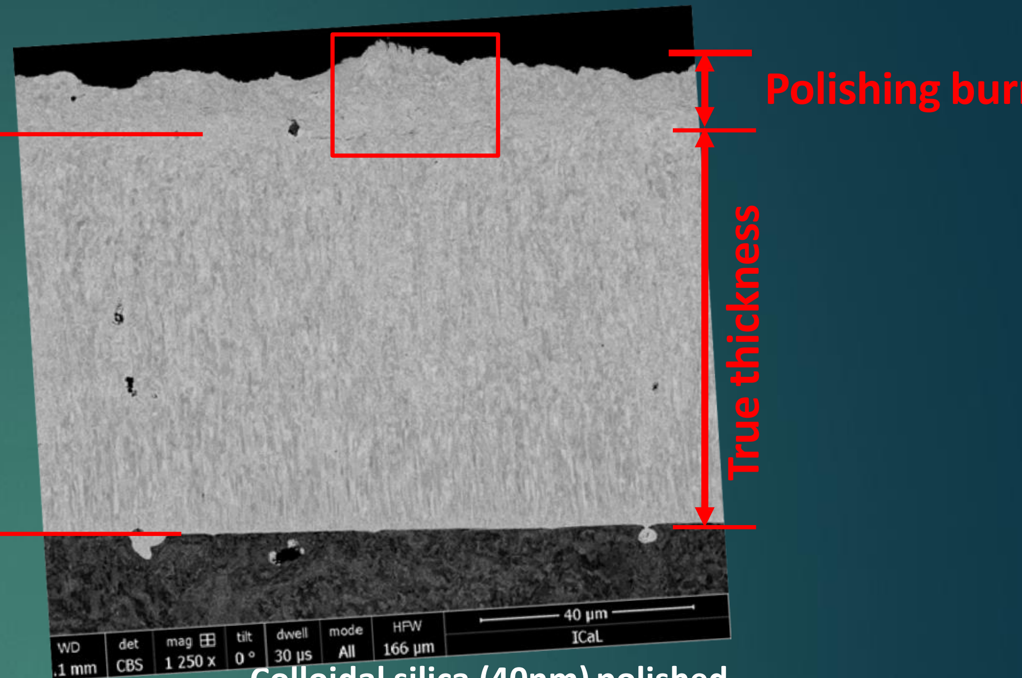
- Technique capable of preparing large area (1mm^2) compared to Ga ion FIB (20 x 100 µm)
- Low kV setting reduces surface damage (also available using FIB)
- Inert species largely avoids problems associated with FIB Ga implantation
- Liverpool’s Helios FIB has a maximum (realistic) cut depth of approx. 20 µm, hence would not be able to prepare the full thickness of the Nb layer here.
- FIB preparation is comparatively slow/expensive.



SEM – Preparation Artefacts

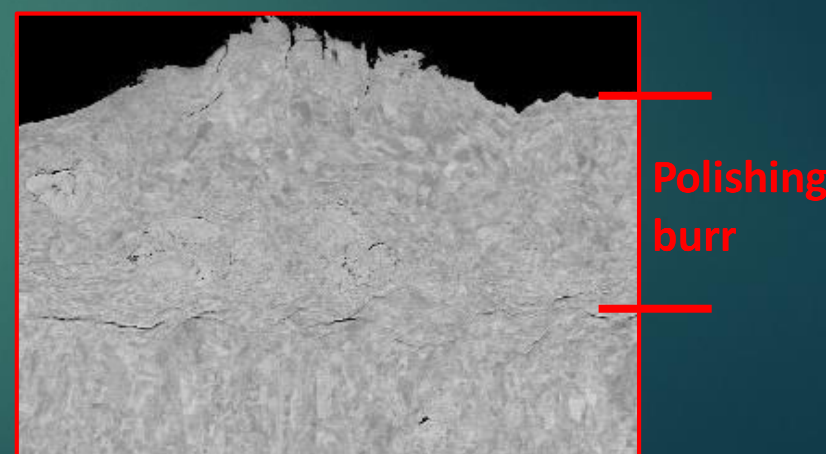


Hitachi IM4000 Plus prepared



Colloidal silica (40nm) polished

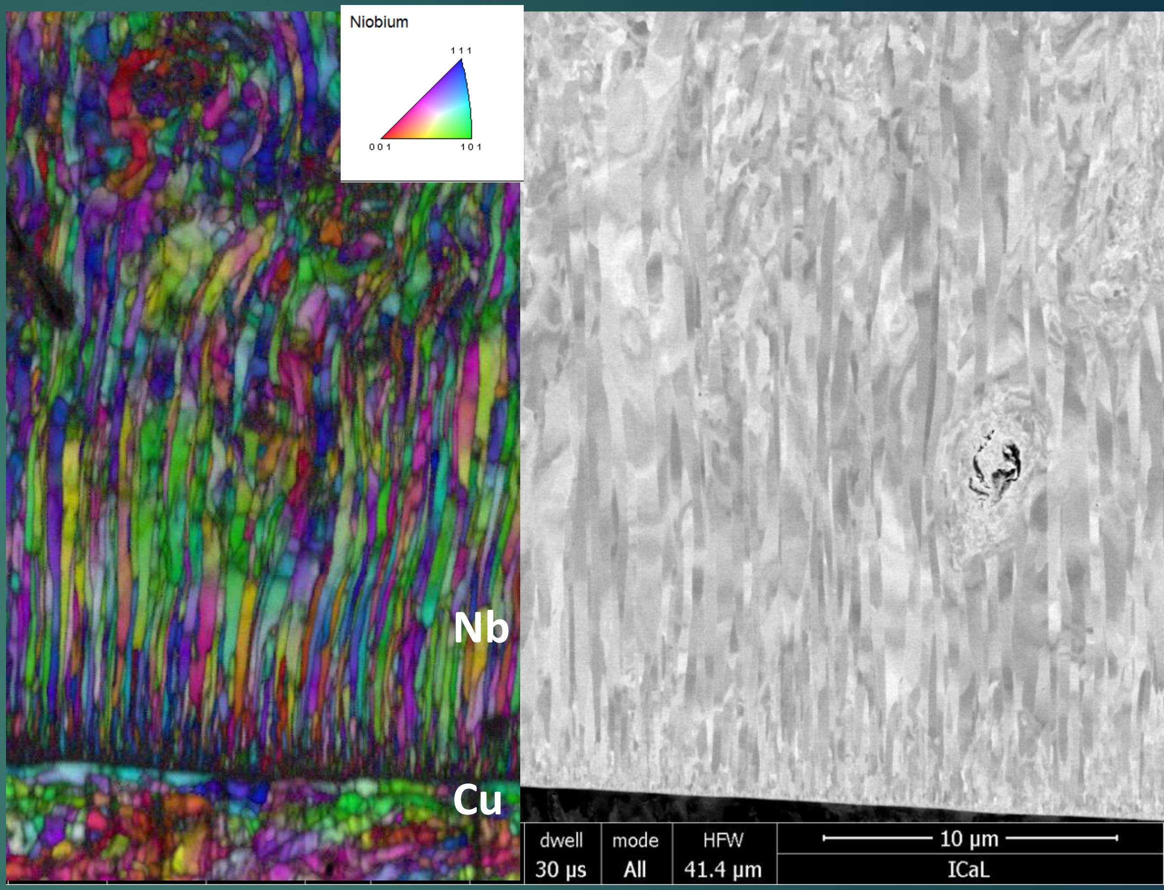
Manual polishing can cause artefacts. Burrs induced by mechanical polishing will promote inaccuracies in the determination of layer thickness.
Clearly the columnar microstructure is also compromised by the effects of mechanical polishing. Material transfer was observed, including Nb to the Cu substrate and silicon carbide polishing aggregates engrained in both the Nb and substrate areas.



EBSD - Colloidal silica polished surface

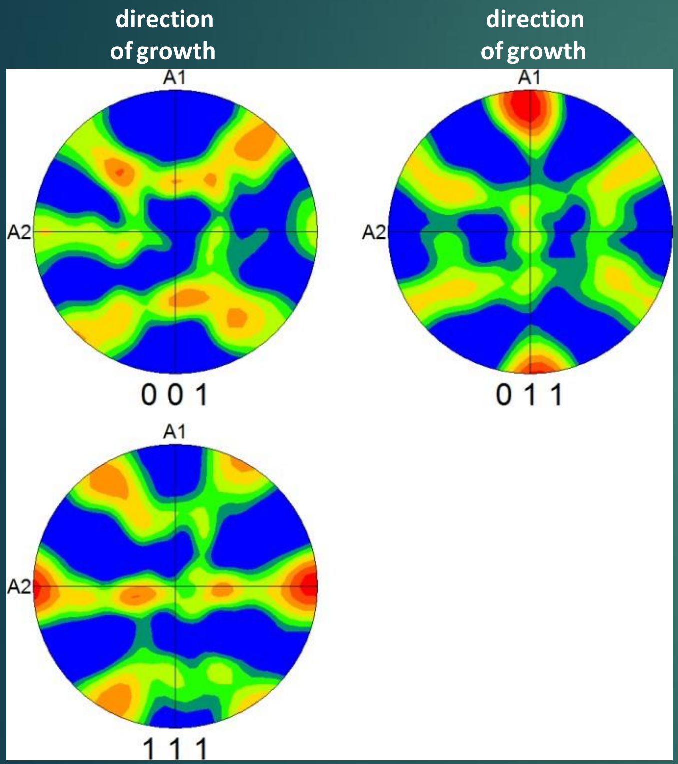
Distortion of columnar crystal growth also evident in EBSD patterns. Crystallographic texture has been modified by the polishing process.

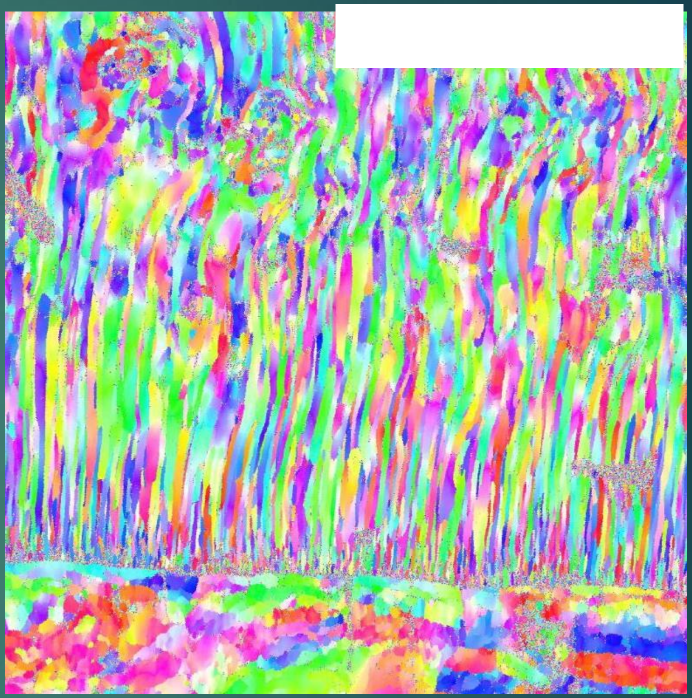
Initial fine grain Nb formation in the early stages of deposition gives way to the formation of columnar grains. However, distortion of the microstructure is evident at distances greater than 10 µm from the interface.

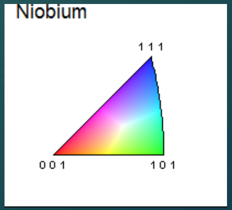


Inverse Pole Figure (IPF) orientation map

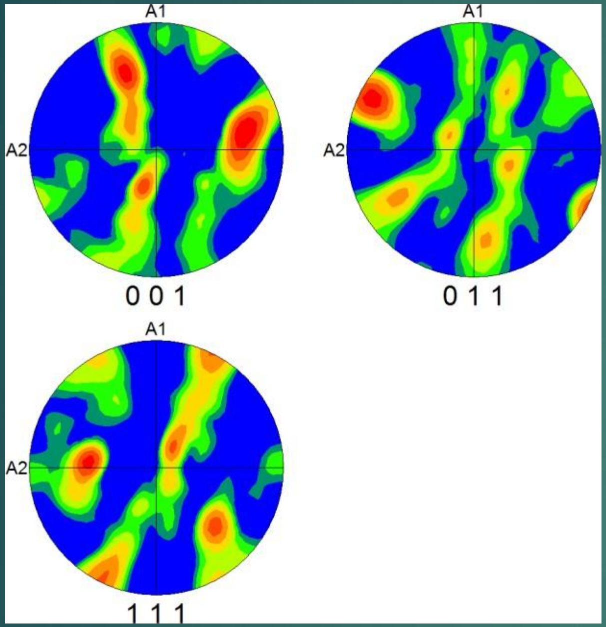
Niobium Texture plots indicate a preferred 011 crystal orientation parallel to the columnar grain growth direction.

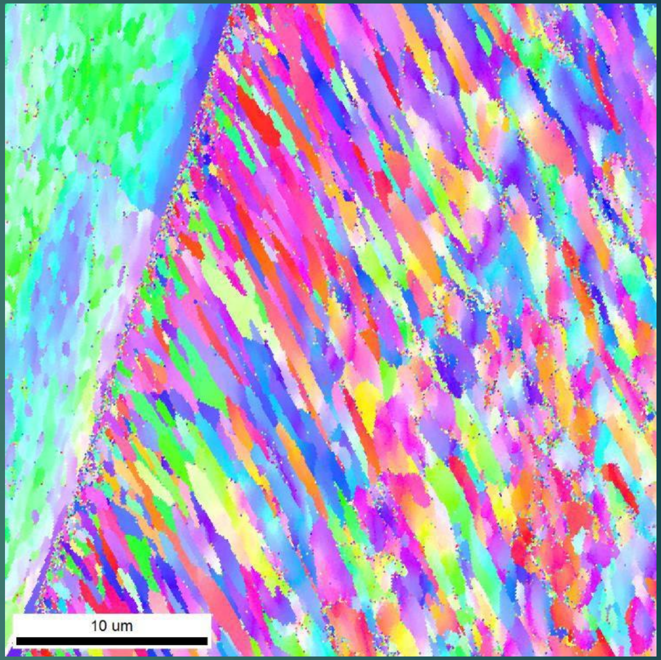




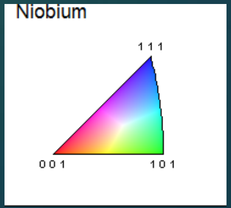


EBSD Texture

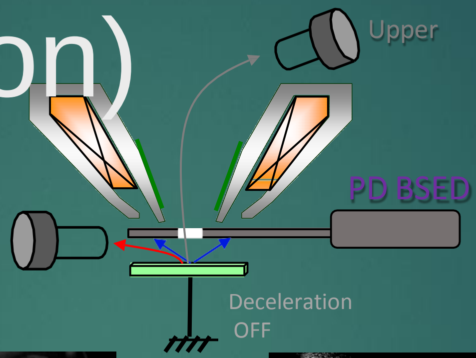




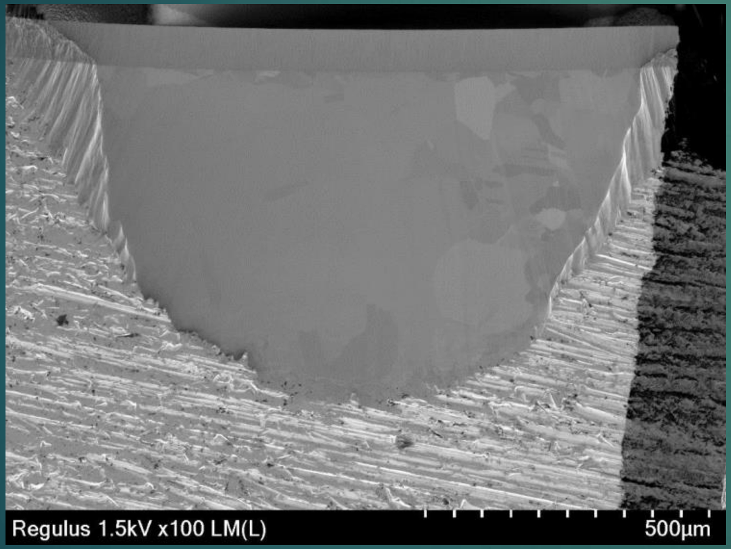
A preferred 011 crystallographic texture, parallel to the columnar grain long axis, is also observed where growth is not normal to the Cu surface.



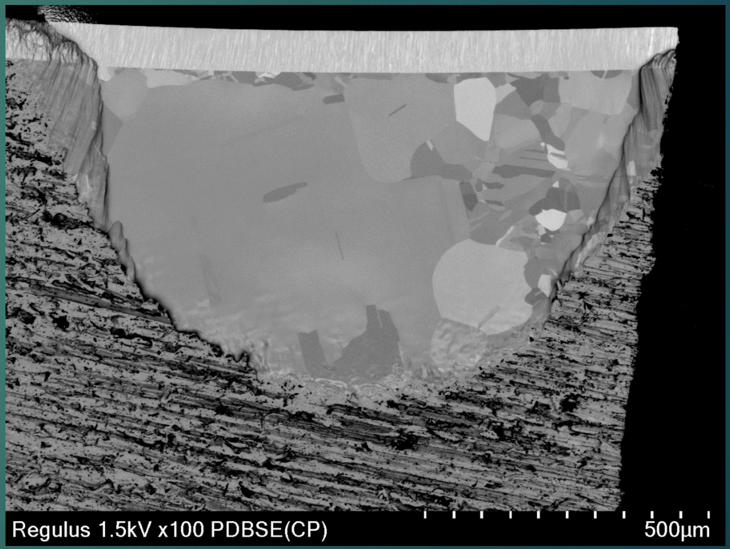
2. Nb layer on Cu substrate (Cross section)



Vacc.	1.5 kV
Mag.	100x
Signal	LM(L), PDBSE(CP)



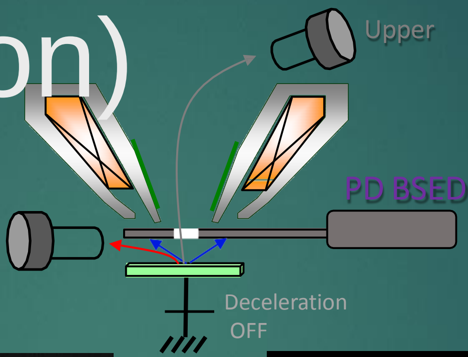
LM(L) : Topographic information



PDBSE(CP) : Compositional contrast

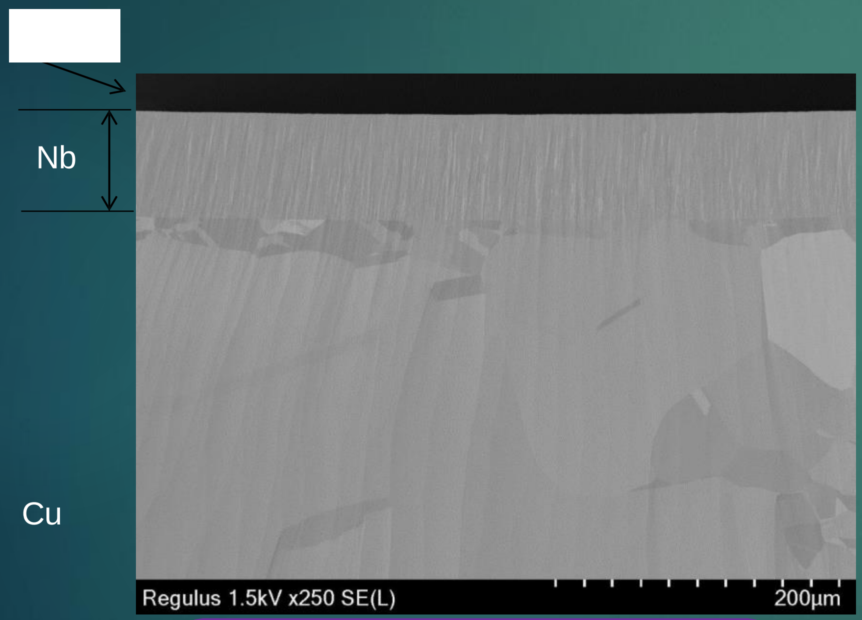
2. Nb layer on Cu substrate

(Cross section)

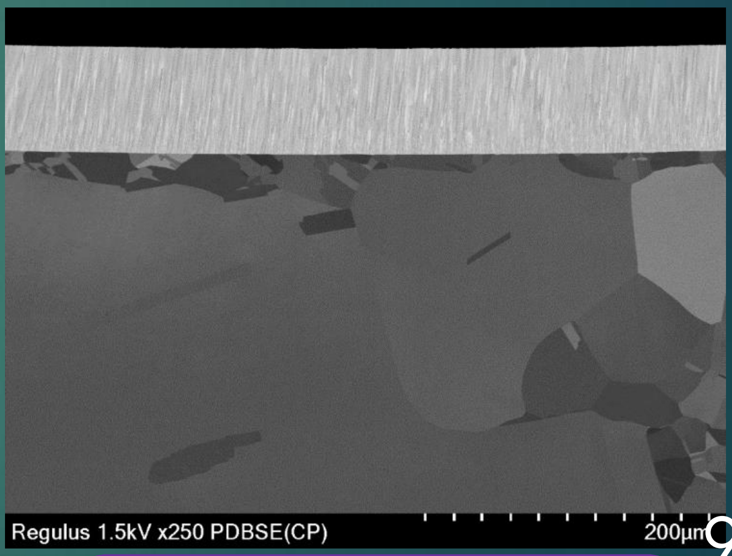


- SE
- BSE
- Conversion Signal

Vacc.	1.5 kV
Mag.	250x
Signal	SE(L), PDBSE(CP)



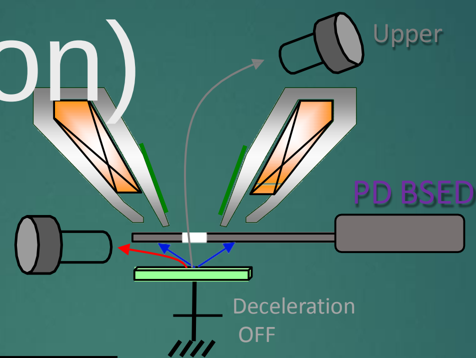
SE(L) : Topographic information



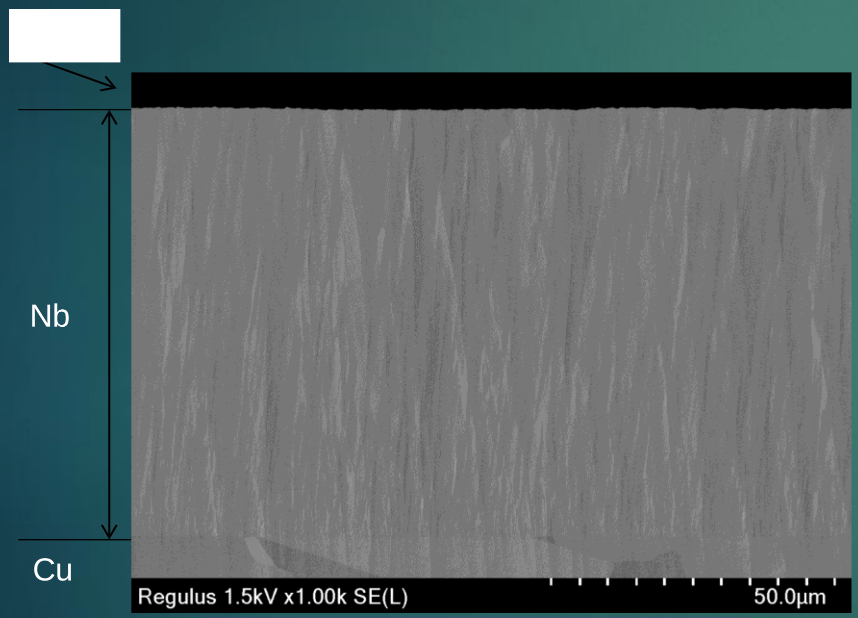
PDBSE(CP) : Compositional contrast

2. Nb layer on Cu substrate

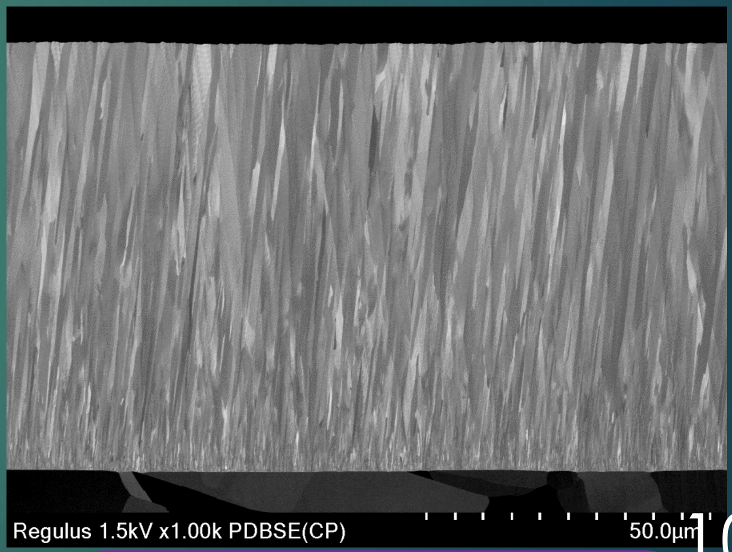
(Cross section)



Vacc.	1.5 kV
Mag.	700x
Signal	SE(L), PDBSE(CP)



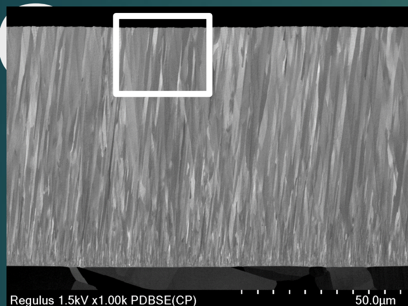
SE(L) : Topographic information

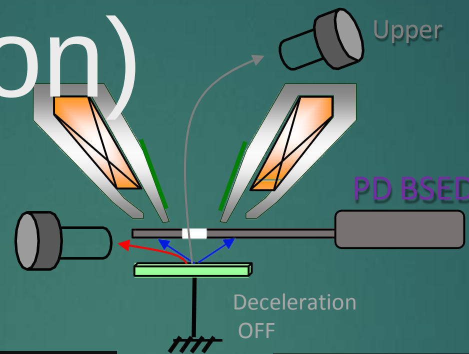


PDBSE(CP) : Compositional contrast

2. Nb layer on Cu substrate

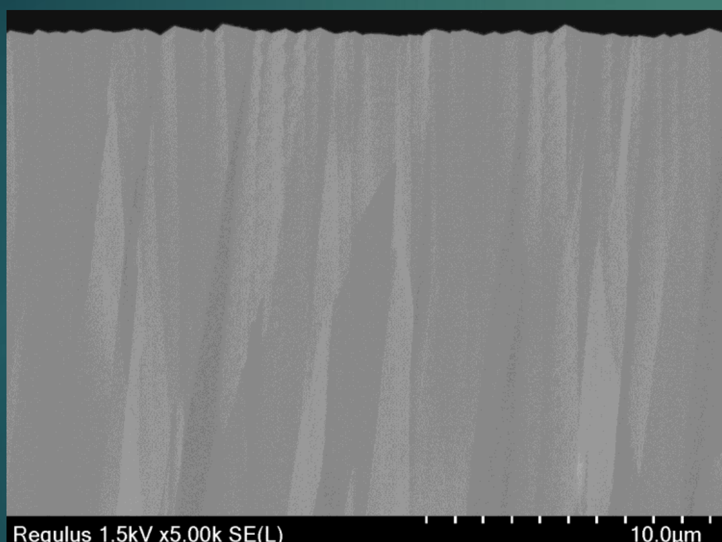
(C ection)



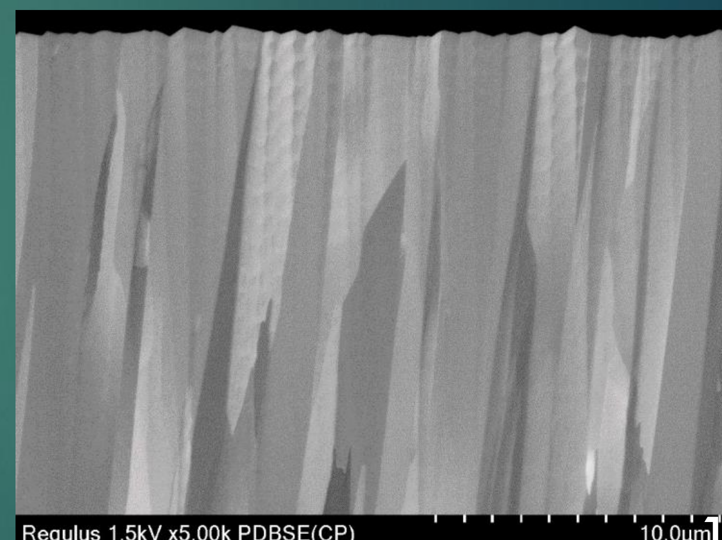


SE
BSE
Conversion Signal

Vacc.	1.5 kV
Mag.	5kx
Signal	SE(L), PDBSE(CP)



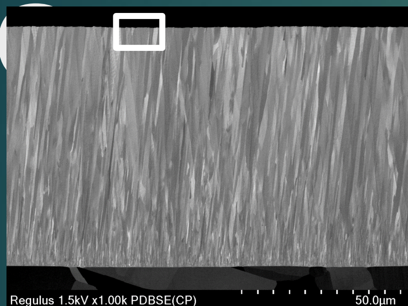
SE(L) : Topographic information

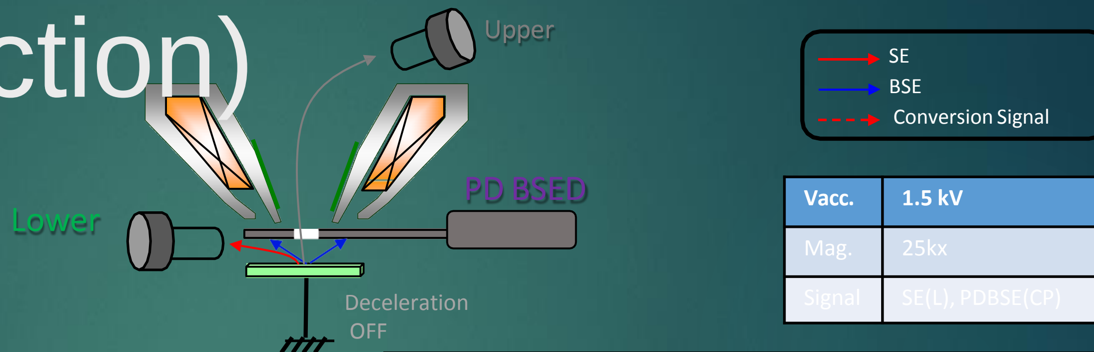


PDBSE(CP) : Compositional contrast

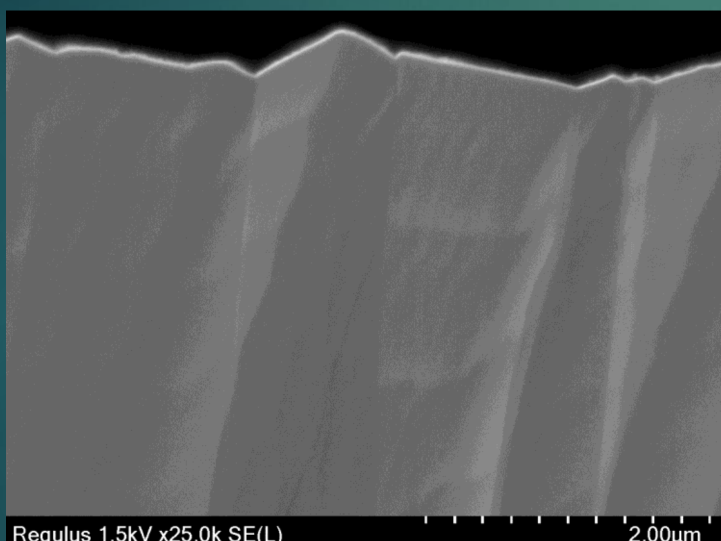
2. Nb layer on Cu substrate

(…ection)

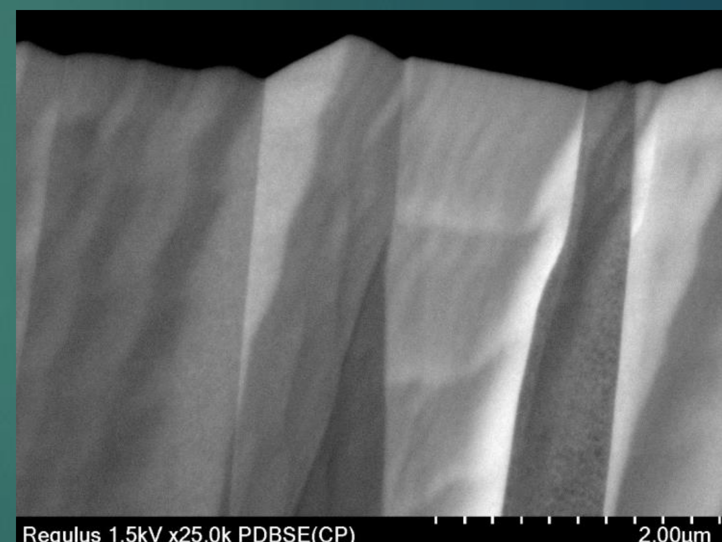




Vacc.	1.5 kV
Mag.	25kx
Signal	SE(L), PDBSE(CP)



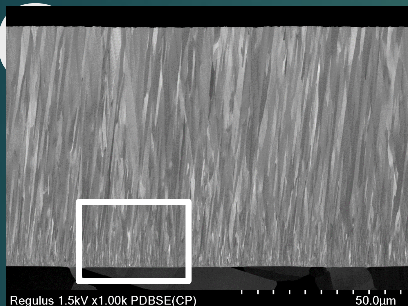
SE(L) : Topographic information

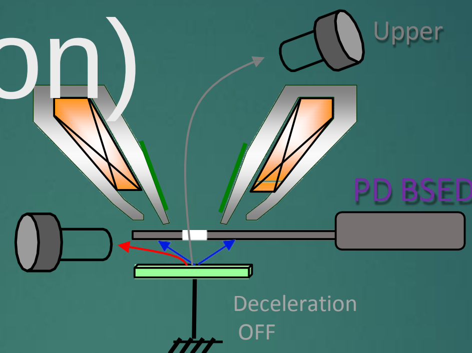


PDBSE(CP) : Compositional contrast

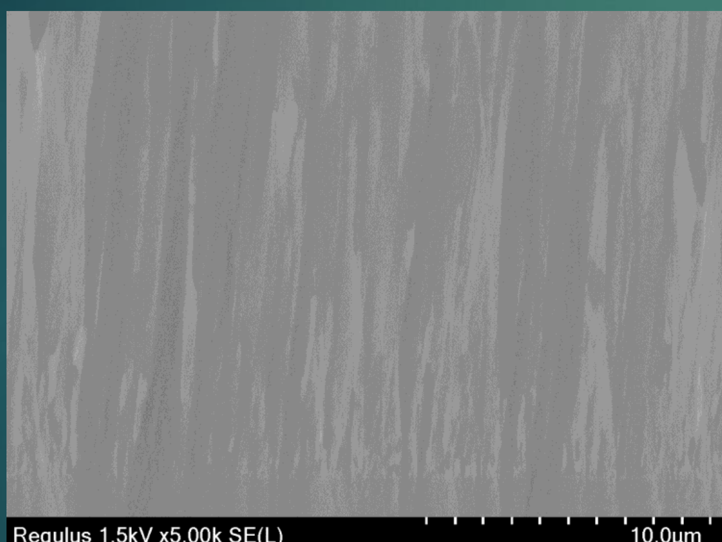
2. Nb layer on Cu substrate

(C ection)

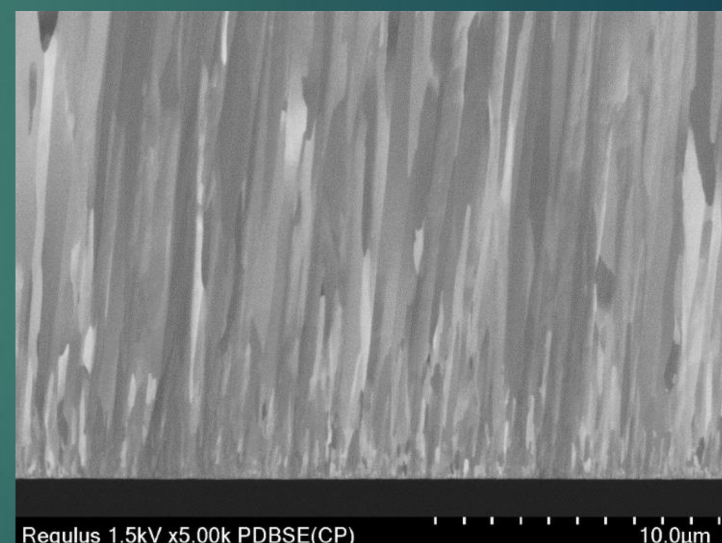




Vacc.	1.5 kV
Mag.	5kx
Signal	SE(L), PDBSE(CP)



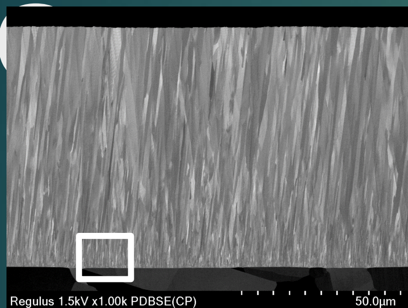
SE(L)： Topographic information

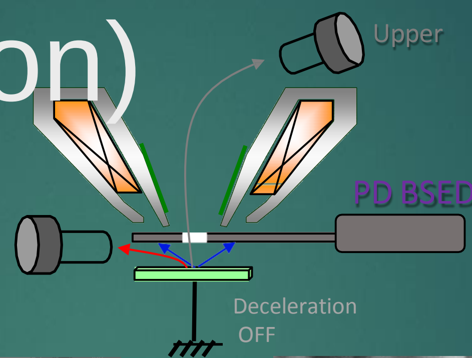


PDBSE(CP)： Compositional contrast

2. Nb layer on Cu substrate

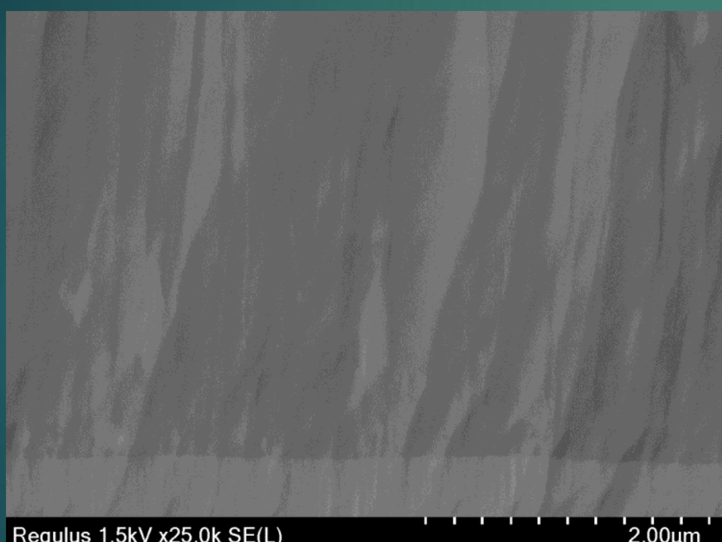
(C…ection)



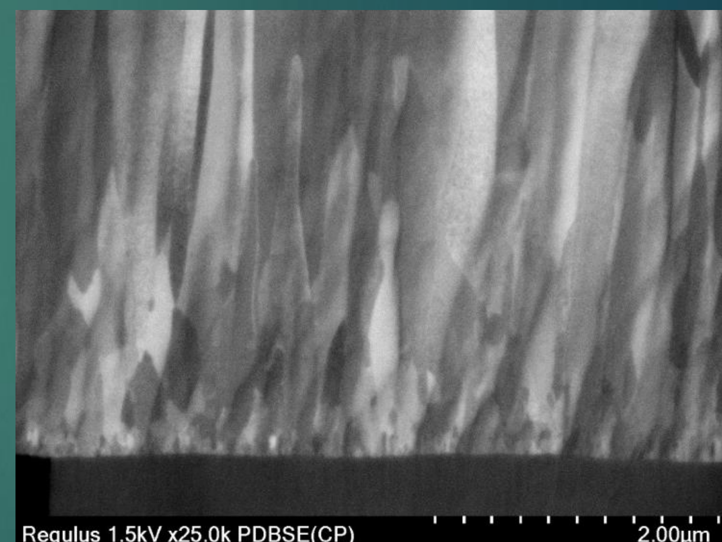


SE
BSE
Conversion Signal

Vacc.	1.5 kV
Mag.	25kx
Signal	SE(L), PDBSE(CP)

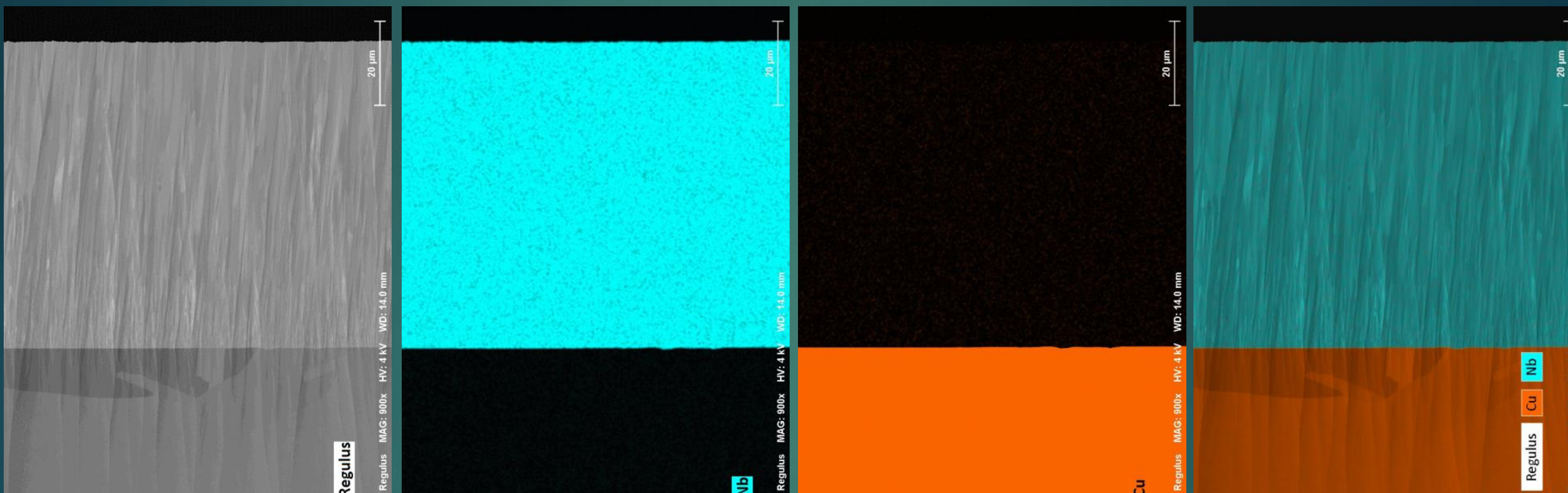


SE(L) : Topographic information

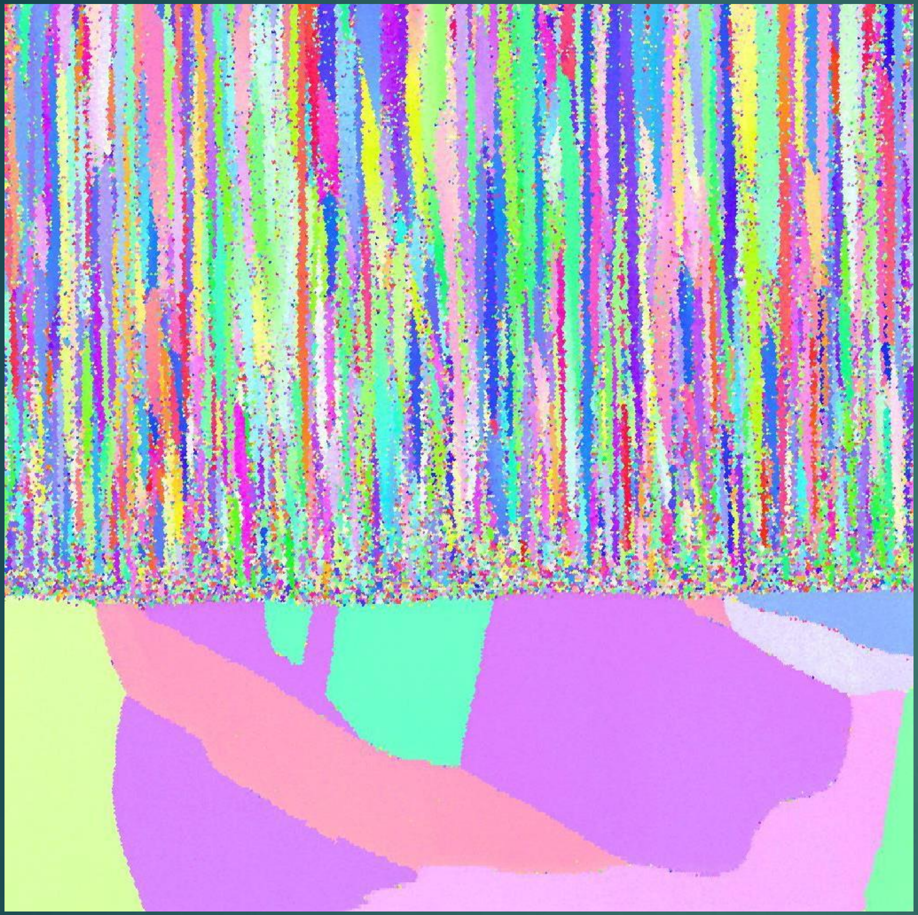


PDBSE(CP) : Compositional contrast

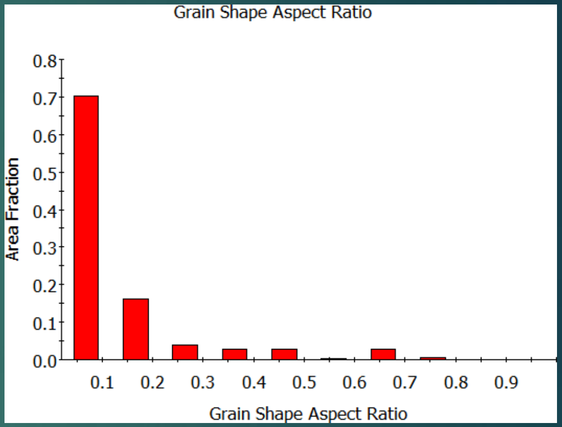
2. Nb layer on Cu substrate (Cross section/EDX analysis) 4kV, 900x, 389sec



EBSD – Hitachi IB prepared specimen



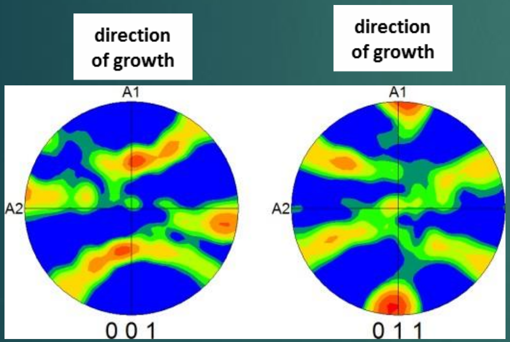
EBSD Orientation maps of the Hitachi Ar ion beam prepared sample revealed high aspect ration columnar grains through the near complet thickness of the deposited film.

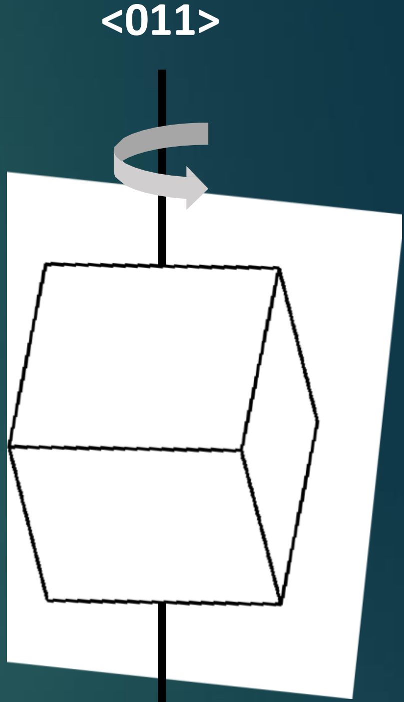


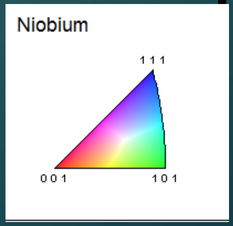
The fine equiaxed Nb grains, formed during the early stages of deposition are not easily resolved. Large twinned grains were observed in the Cu substrate.

EBSD – Hitachi IB prepared specimen

EBSD texture measurements performed on the Ar ion beam prepared niobium layer were consistent with those recorded from the colloidal silica prepared materials. A strong <011> texture was measured, parallel to the long axis of the columnar grains, which is approximately parallel to the growth direction.







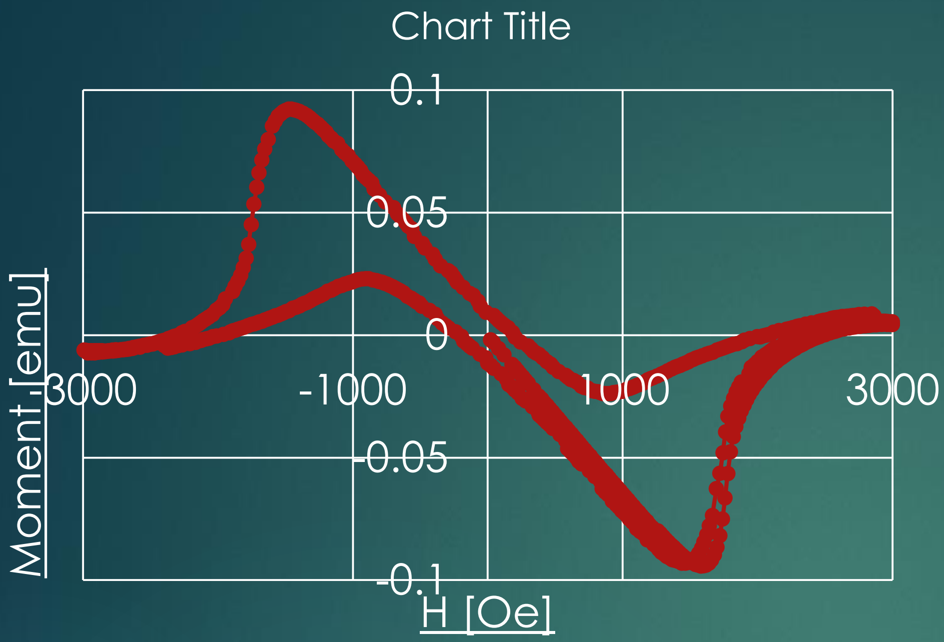


Chart Title
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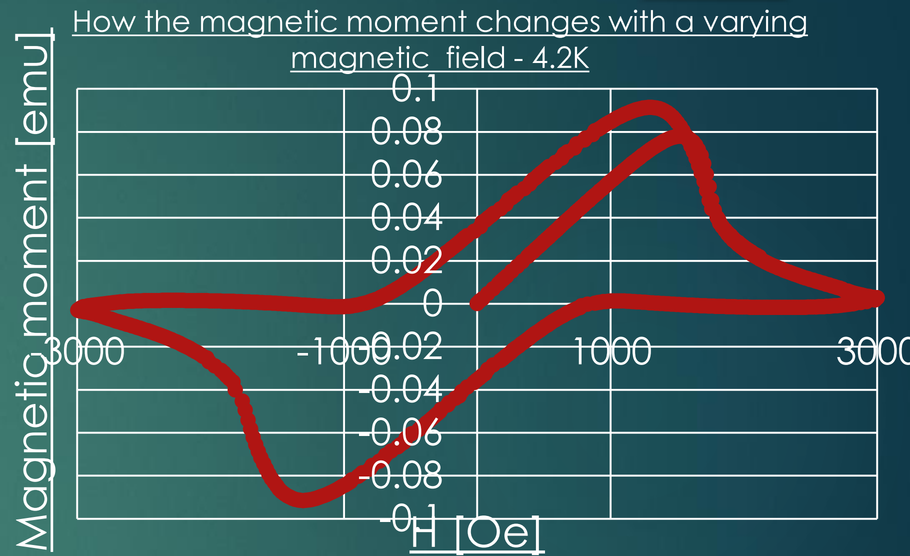
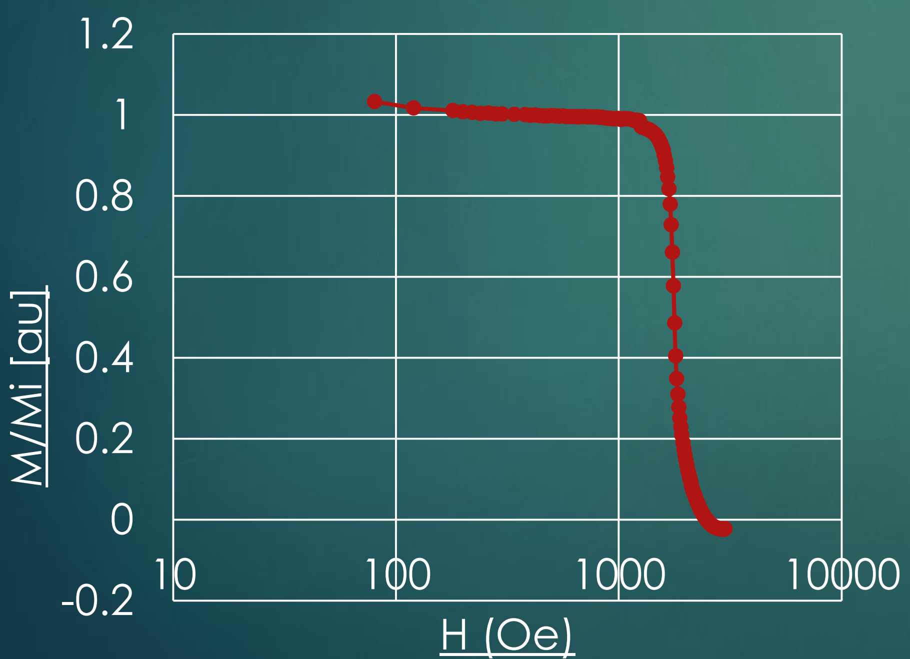
0.05
0
-0.05
-0.1
-3000
-1000
1000
3000
Moment [emu]
H [Oe]


How the magnetic moment changes with a varying magnetic field - 4.2K
0.1
0.08
0.06
0.04
0.02
0
-0.02
-0.04
-0.06
-0.08
-0.1
-3000
-1000
1000
3000
Magnetic moment [emu]
H [Oe]


1.2
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0.8
0.6
0.4
0.2
0
-0.2
10
100
1000
10000
M/Mi [au]
H (Oe)

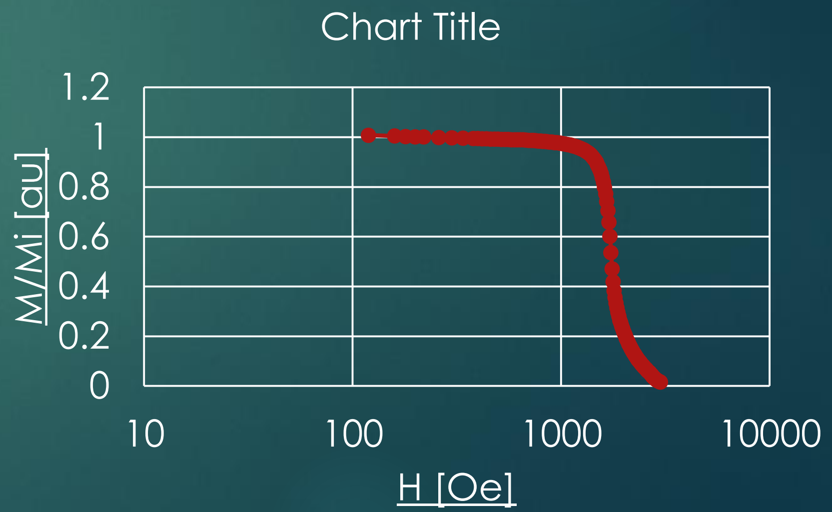
Chart Title
1.2
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0.8
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0.4
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10
100
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10000
M/Mi [au]
H [Oe]