CHARACTERISATION OF NIOBIUM THIN FILM DEPOSITED ON 6 GHZ SRF CAVITY

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Motivation

- STFC is interested in acquiring the Nb/Cu cavity coating know-how and to further improve its analysis capabilities related to thin superconducting films deposited on Cavity and RF Tested.
- Enhance our network of collaboration with leading centres in cavity production, preparation and thin film superconducting deposition.

ACTIONS

- Microscopy and surface characterization of samples produced by INFN to determine:
  - Nature of grain boundary
  - Film density
  - Microstructure at Cu/Nb interface
  - Residual stress
  - Level impurities at grain boundaries, surface, Cu/Nb interface and the substrate.
  - Film adhesion
- Development of in-house 3D Nb/Cu coating capabilities.

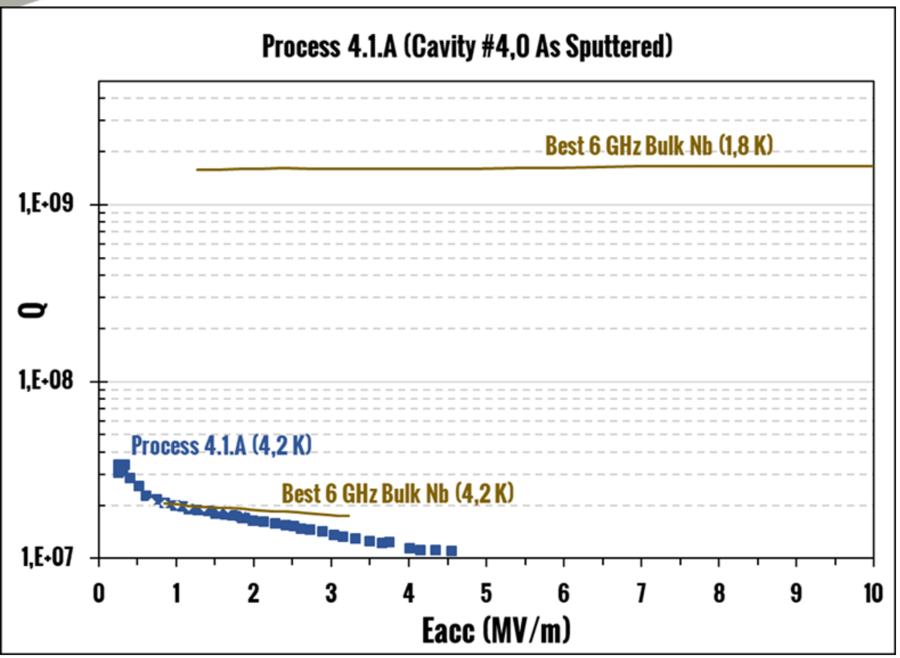
INFN 6 GHz Cavity and deposition Parameters

Coating parameters

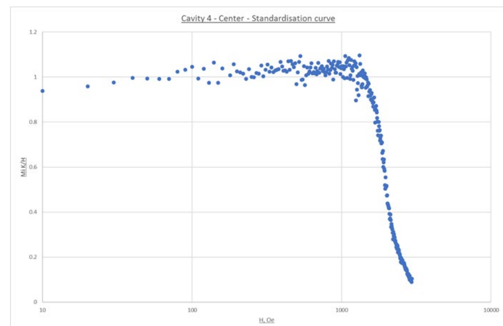
- cavity temperature: 550 °C (600 °C during the preliminary baking process);
- argon pressure: from 7♠10^{-3} to 5♠10^{-2} mbar;
- cathode current: 1 A (0,017 A/cm^2);
- standard thickness: from 20 µm to 70 µm (on the cell);
- deposition time: 4-9 hours;
- deposition mode: one shot or multilayer;
- venting mode: standard or high temperature.

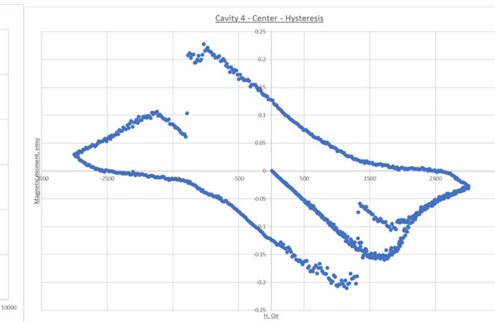
RF and DC Evaluation of Cavity 4

C	V (V)	P (mbar)	B (G)	T (˚C)	d (µm)	t (h)	deposition mode	venting mode
1	≈330	$7 \cdot 10^{-3}$	830	550	≈30	3	one-shot	High temp.

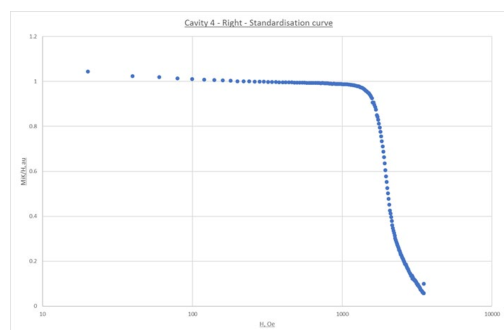


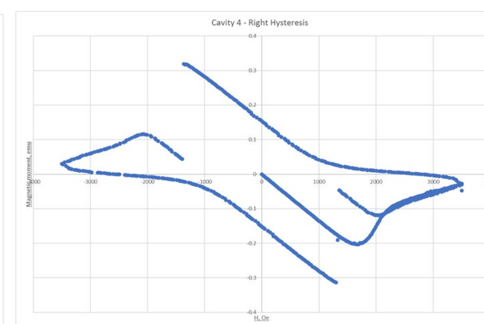
stripped Nb film on Kapton tape centre position





stripped Nb film on Kapton tape right position

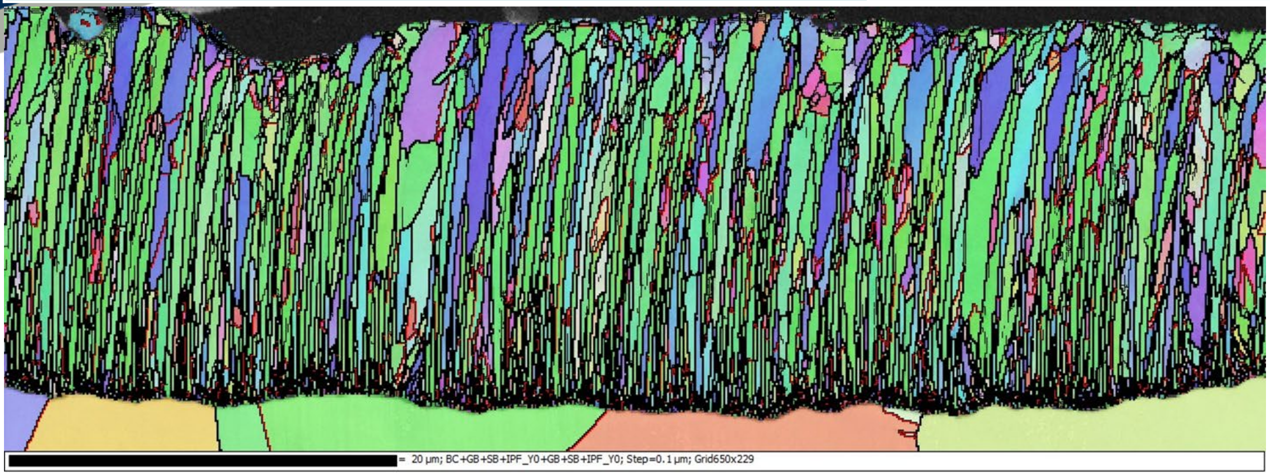


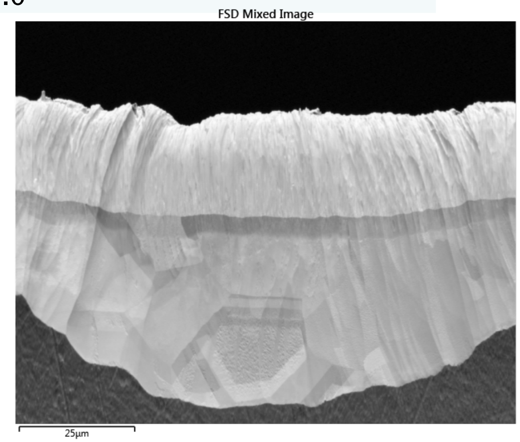


H_{fp} for centre position 155 mT at 5%
Flux jumping can be observed
H_{fp} for right position 148 mT at 5%
No flux jumping is observed
H_{c2} not reached potentially 400 mT

EDSB of Cross-Section Centre position of Cavity 4

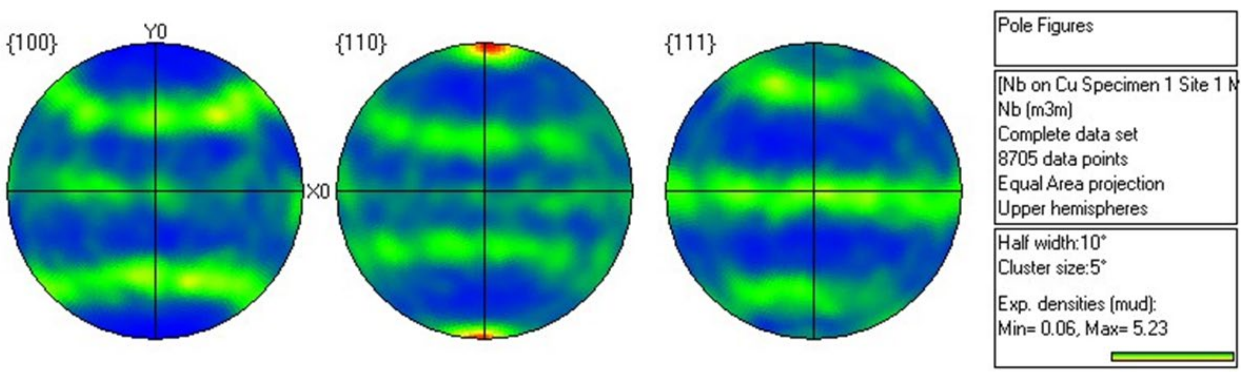
Phase (phase colour map)	Average grain size (mm)
Cu (yellow)	9.5
Nb (green)	1.0





- the average film thickness is about 30 microns with very sharp Cu/Nb interface.
- The deposited film is very dense and it consist of elongated grain structure aligned in the perpendicular direction to the substrate surface.
- At the interface the grains are much smaller than the the grains further away from the interface.

The grain boundaries above 10° are shown as black lines and twin boundaries as red lines. There are twin boundaries both in the Nb



Nb layer is highly textured with (110) parallel to Y-direction

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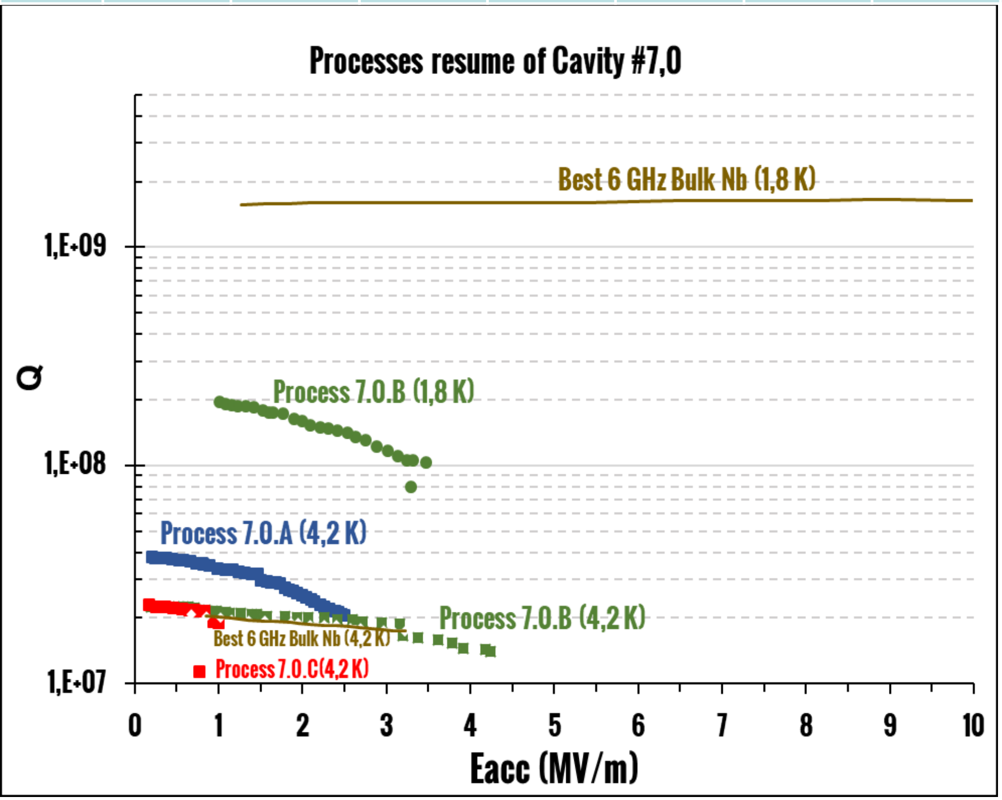
RF and DC Evaluation of Cavity 7

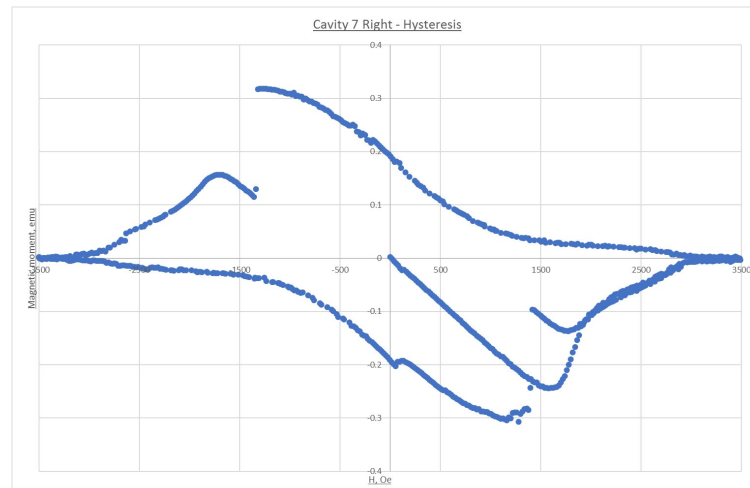
I (A)	V (V)	P (mbar)	B (G)	T (˚C)	d (µm)	t (h)	deposition mode	venting mode
1	≈480	$7 \cdot 10^{-3}$	830	550	≈70	7	one-shot	High temp.

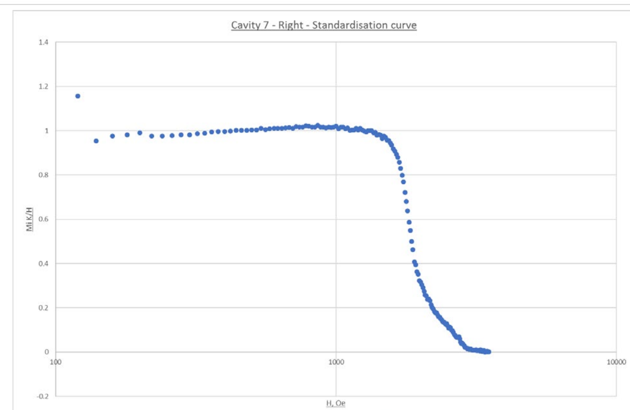
Solution	concentration (ratio)	time (s)	Etching thickness (µm)
$HF:HNO_3:H_3PO_4$	1:1:3	120	5,5

Temperature (˚C)	Time (hours)
120	24



stripped Nb film on Kapton tape right position





H_{fp} for right position 154 mT at 5% drop

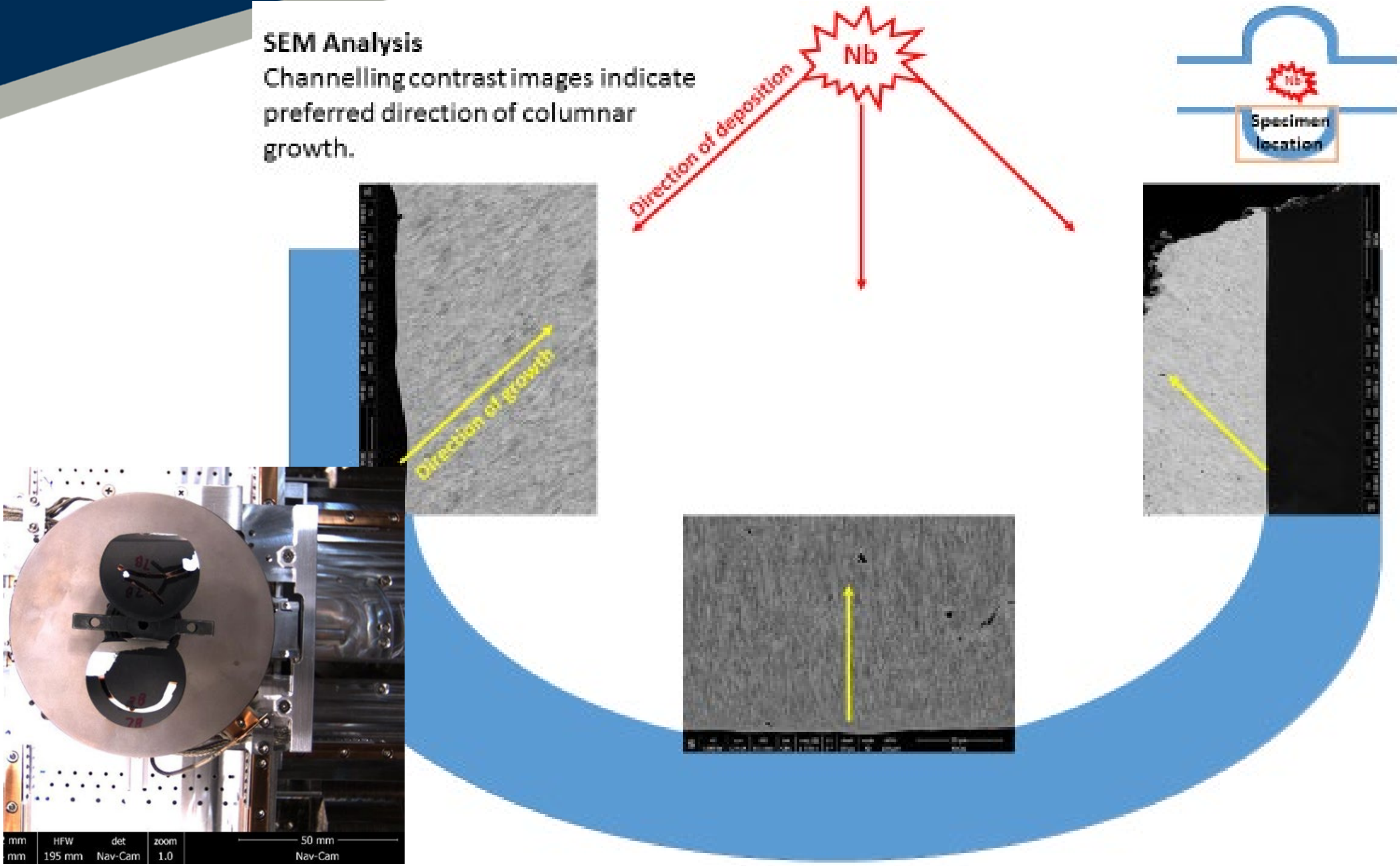
No flux jumping is observed

H_{c2} has reached at 320 mT

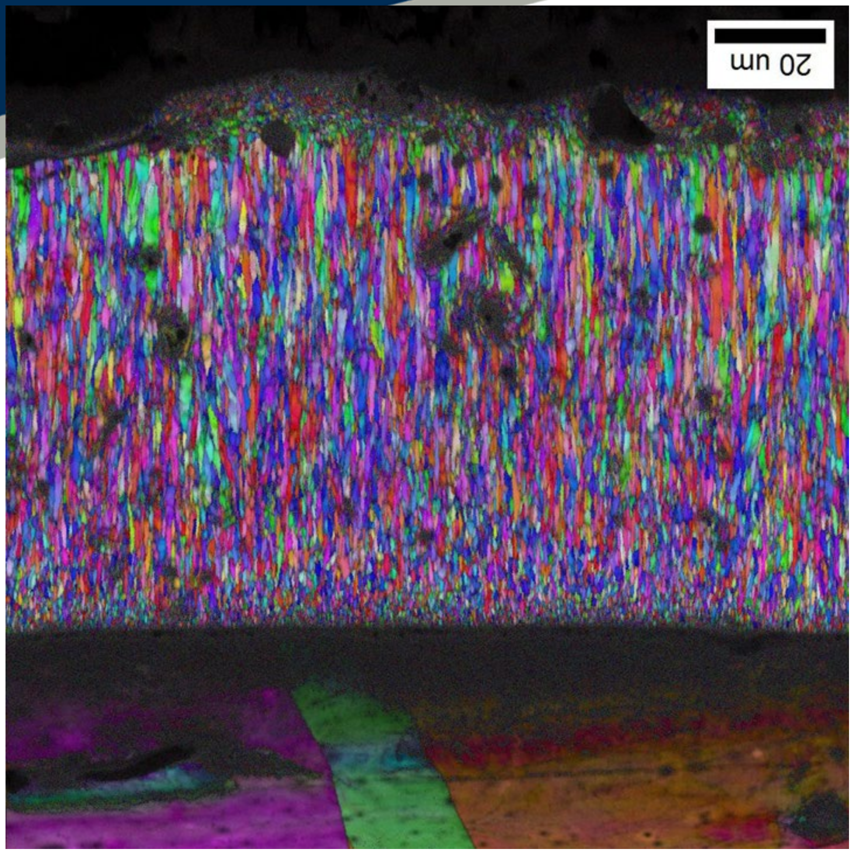
Cross section of cell and Iris

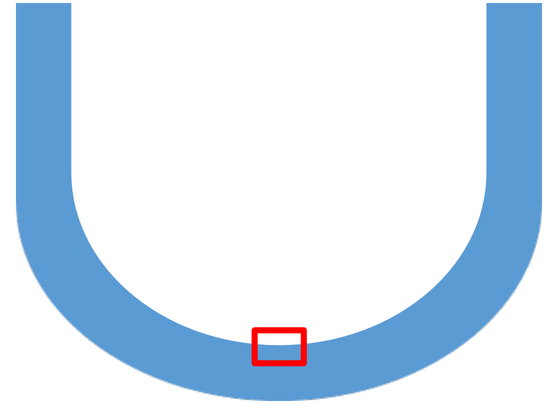
SEM Analysis

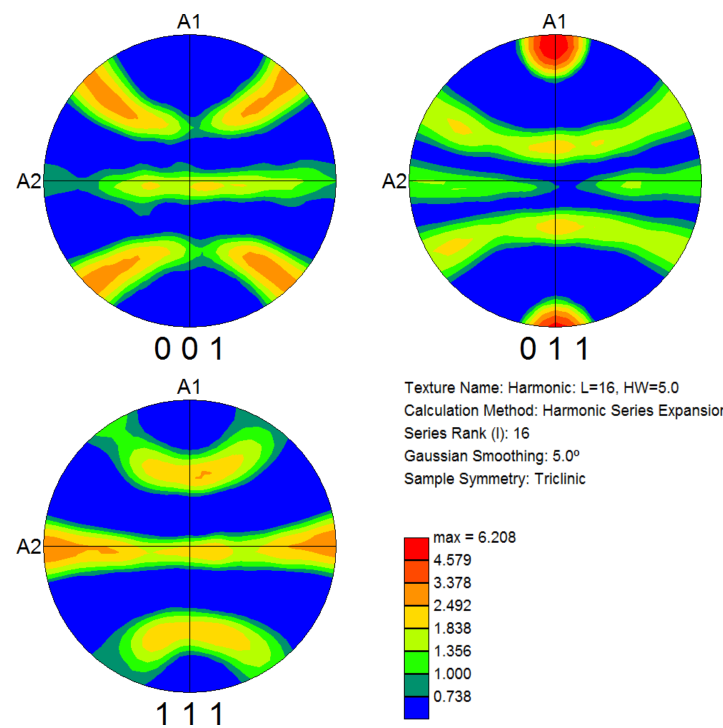
Channelling contrast images indicate preferred direction of columnar growth.



EBSD measurement of the cell section



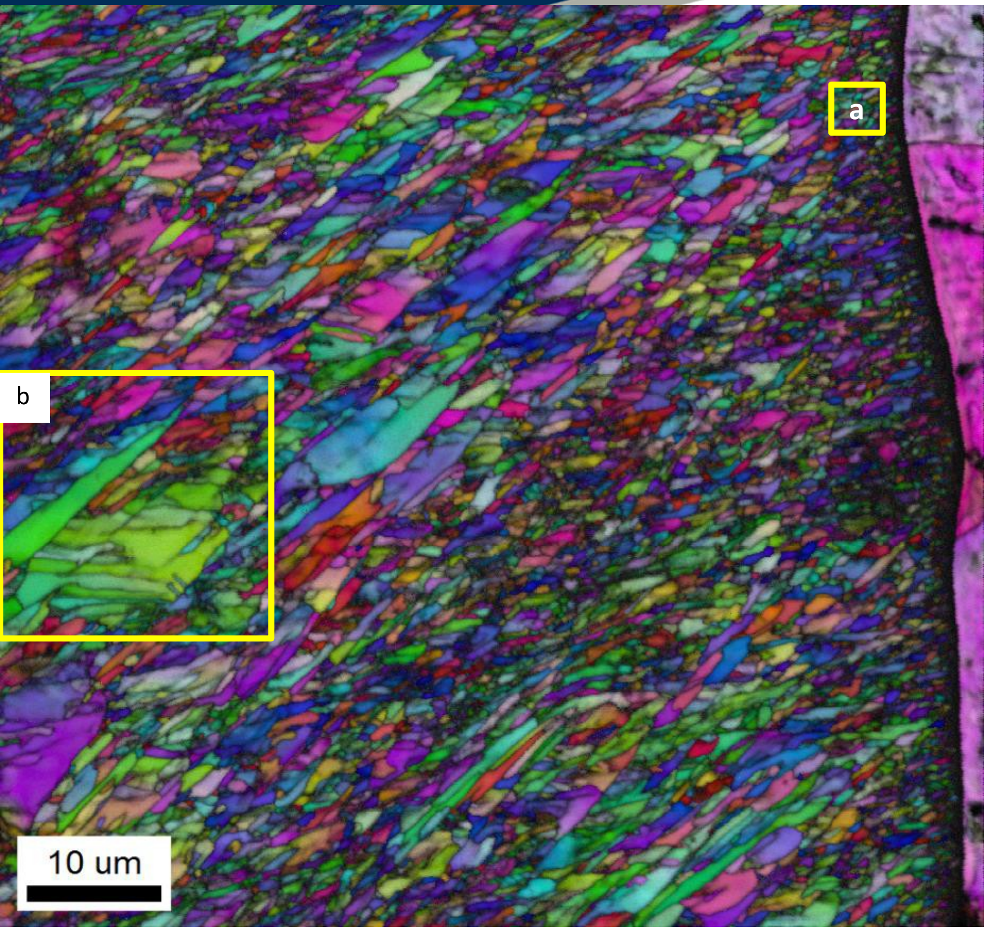


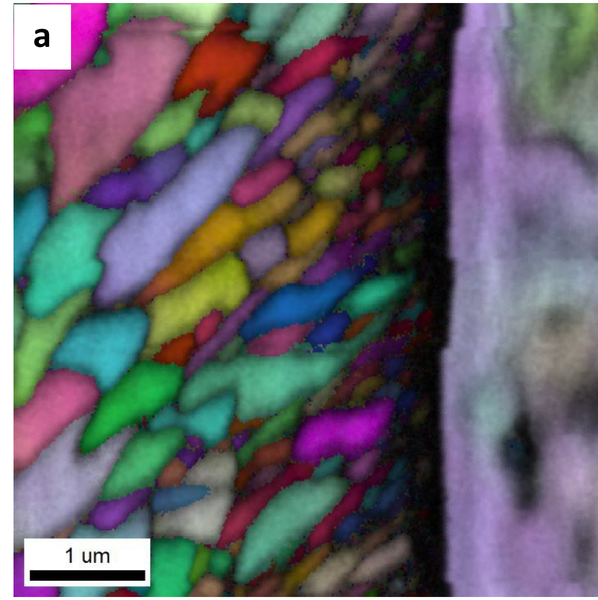


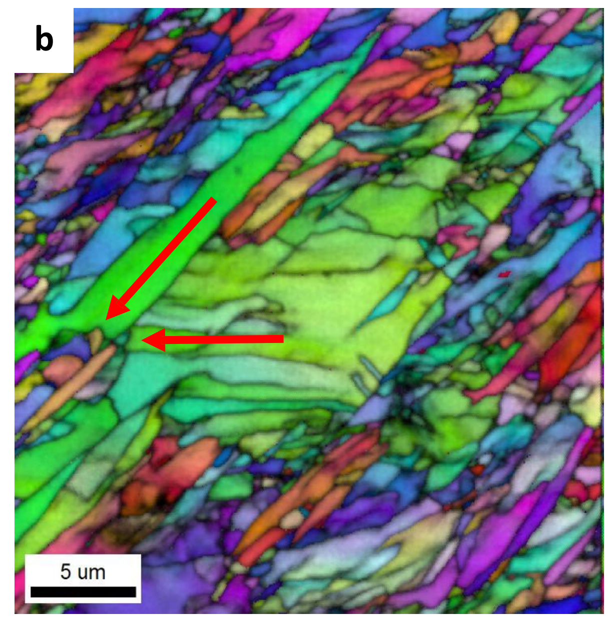
Nb [110] growth texture identified at the mid point of x-section.

Sub micron (100nm) fine grain structure formed during the initial stages of growth. Within 2 µm of growth, from the Nb/Cu interface, a well defined columnar structure is produced.

EBSD measurement of the Iris section

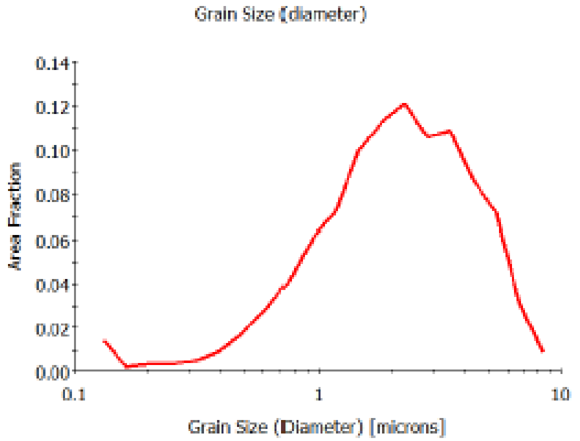


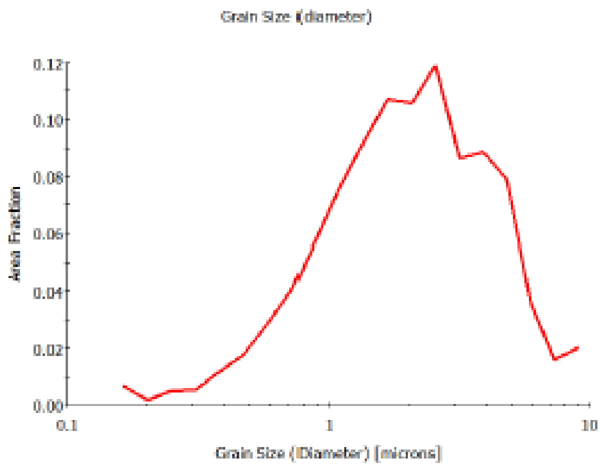


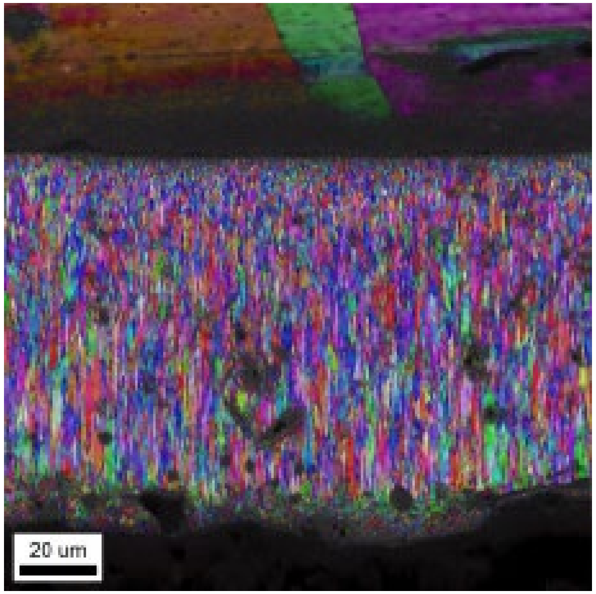


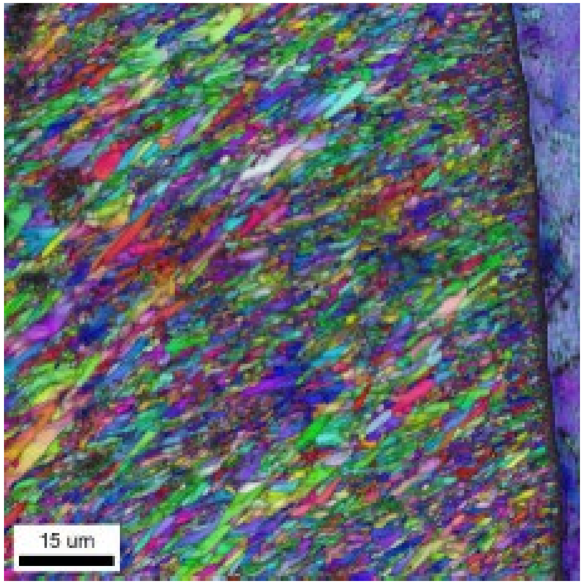
EBSD measurements show (a) ultra fine grain Nb at the Nb/Cu interface. (b) Mixed orientations of columnar growth, made up of columnar grain growth perpendicular to the interface and other grains elongated approximately 45° to the interface.

Grain Size Distribution









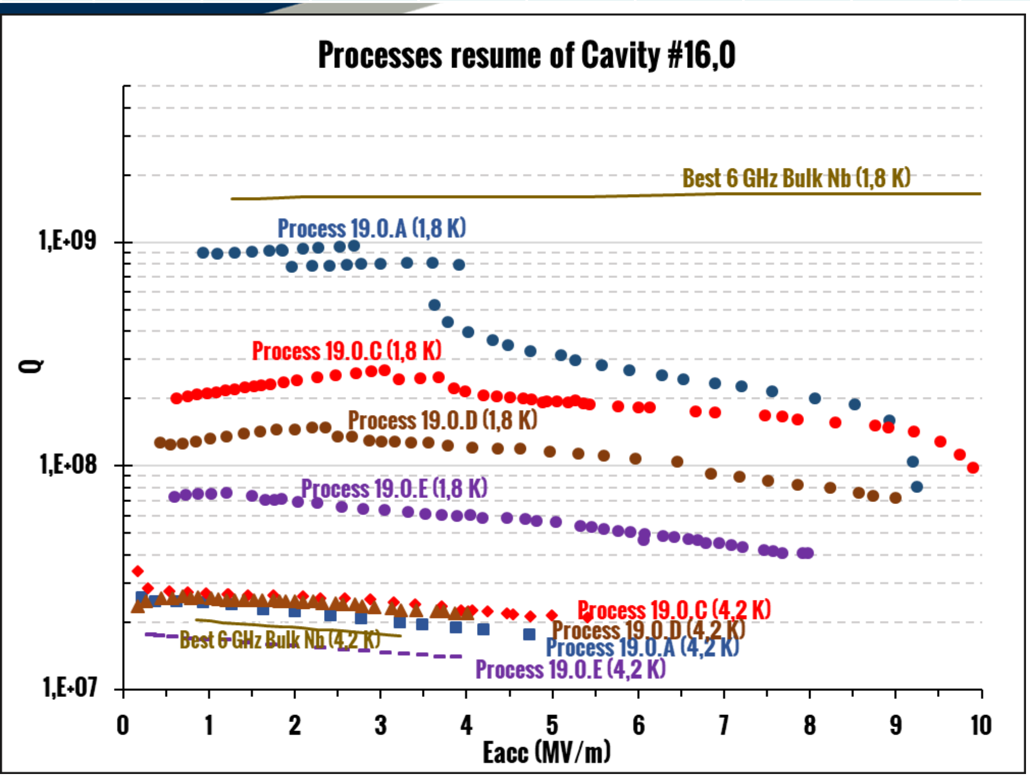
Grain size distributions are similar along the length of the sample. However, grain diameter and aspect ratio differs through the Nb layer thickness.

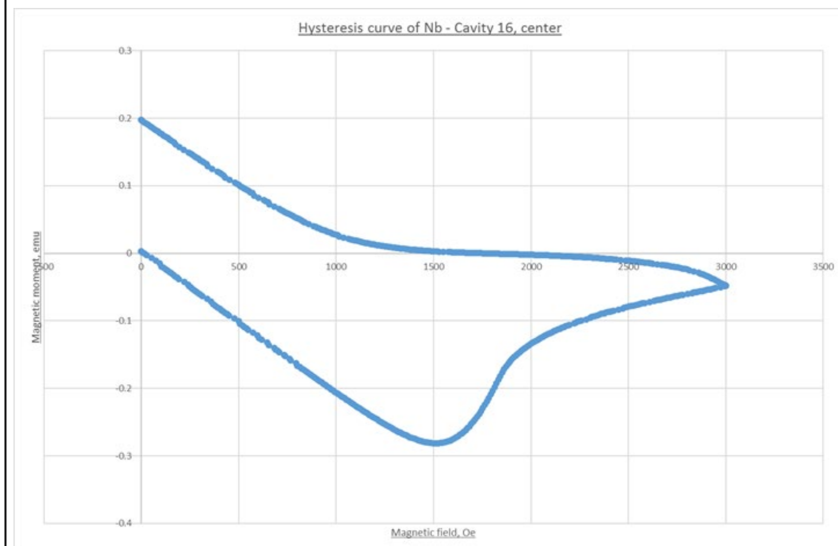
RF and DC Evaluation of Cavity 16

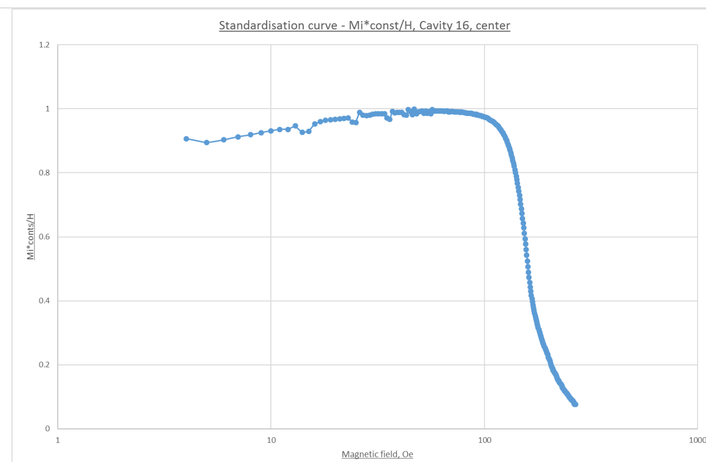
I (A)	V (V)	P (mbar)	B (G)	T (°C)	d (μm)	dep. mode	d_{sl} (nm)	n_{sl}	D	venting mode
1	≈320	$5{\cdot}10^{-2}$	830	550	≈70	multilayer	500	140	0.7	standard

Temperature (°C)	Time (hours)
120	24

Temperature (°C)	Time (hours)	Temperature (°C)	Time (min)
600	10	730	40

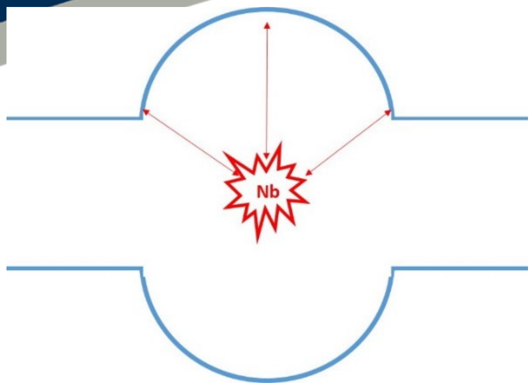


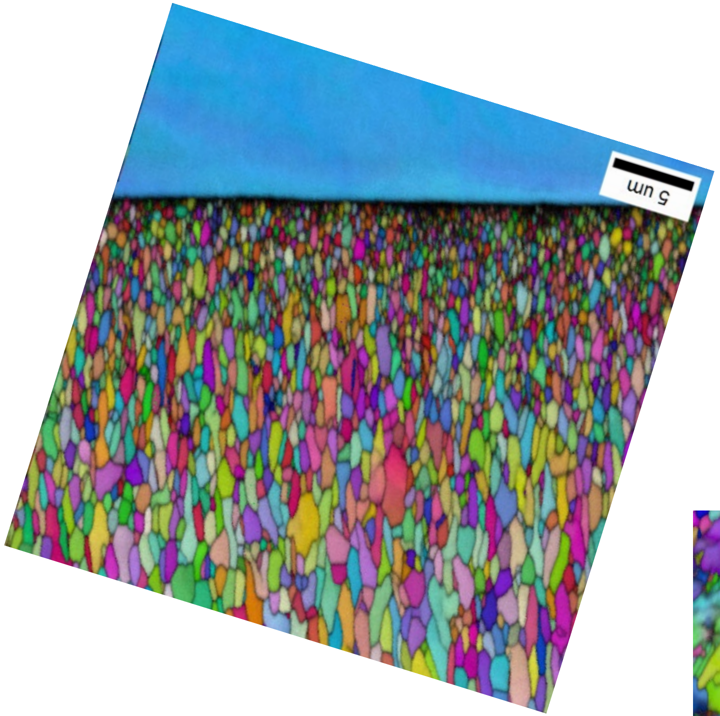




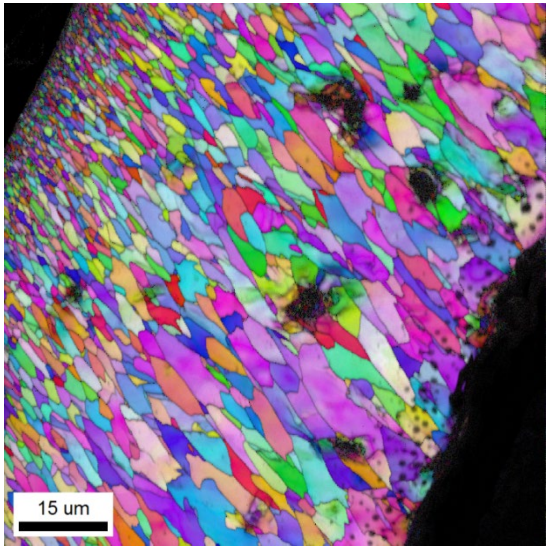
H_{fp} for right position 114 mT at 5% drop
No flux jumping is observed
H_{c2} not reached potentially above 350 mT

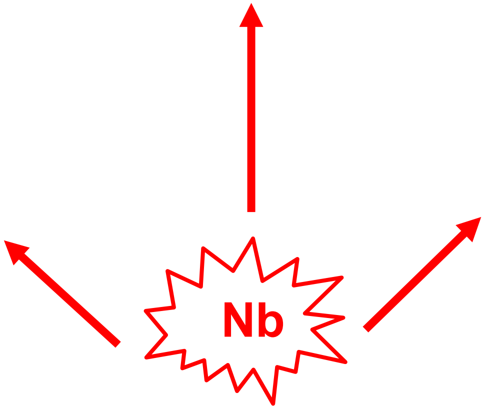
EBSD Inverse Pole Figure Of Cavity 16

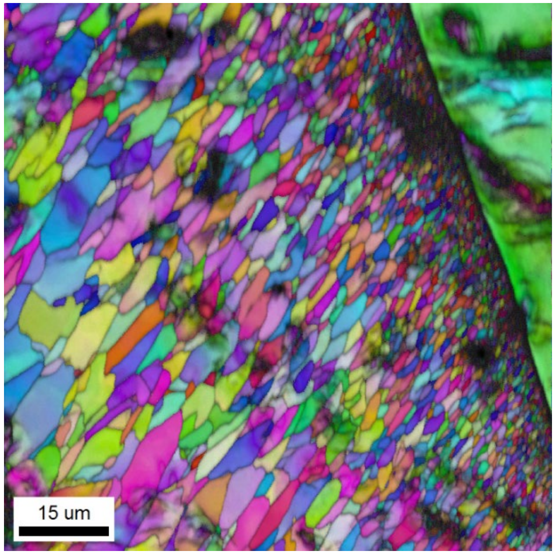




EBSD IPF maps show sub-μm, near equiaxed grains, at the Cu/Nb interface. Columnar structures were observed in all other regions and there was a general increase in grain size as the distance from Cu increased

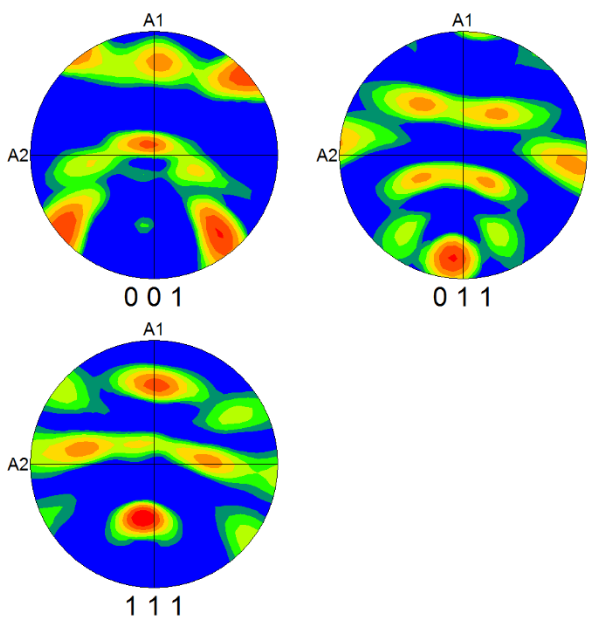


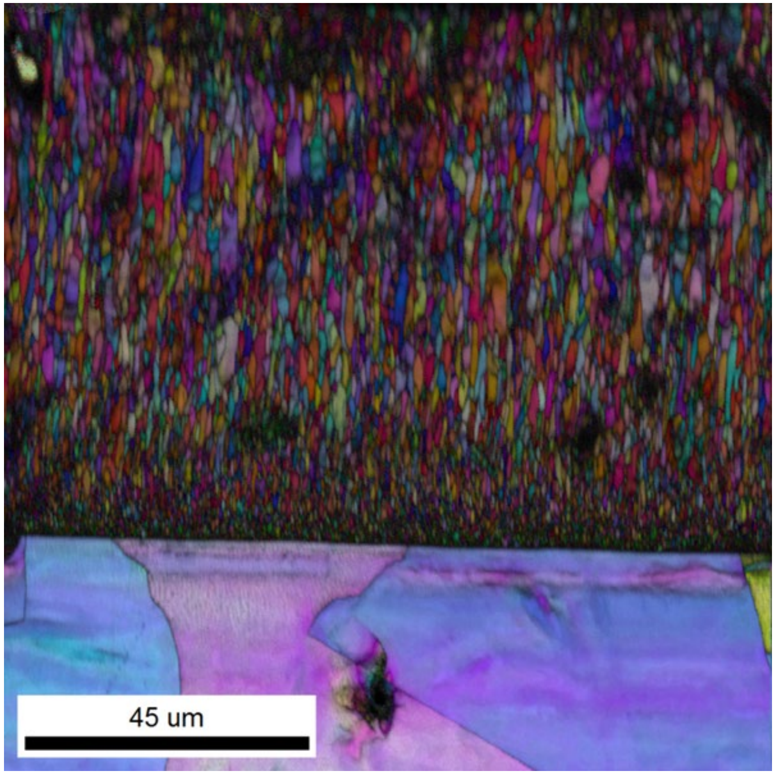




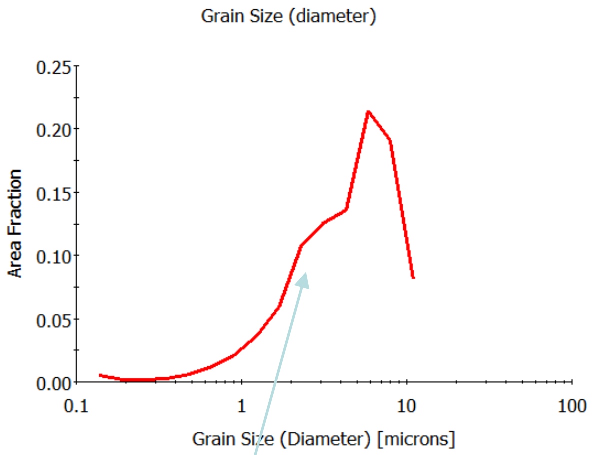
EBSD- Texture of Centre position

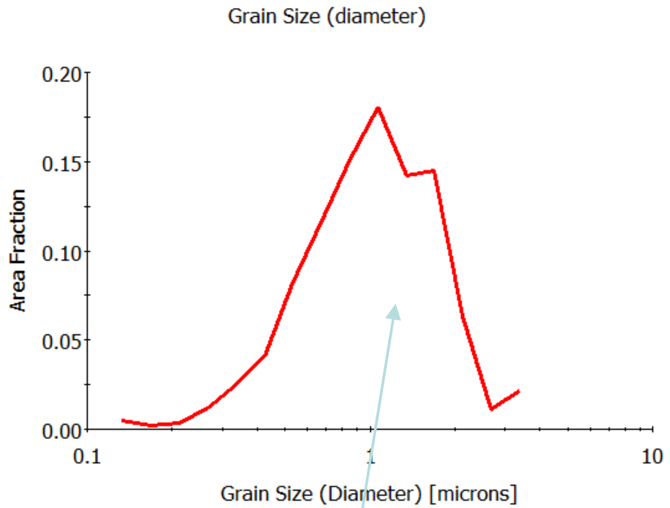
Niobium texture plot indicates a preferred orientation of growth, direction close to [011] but rotated approx. 10° into the page. Similar to the irregularities with the thickness measurements, this is possibly caused by tilting of the specimen in the sample mount.

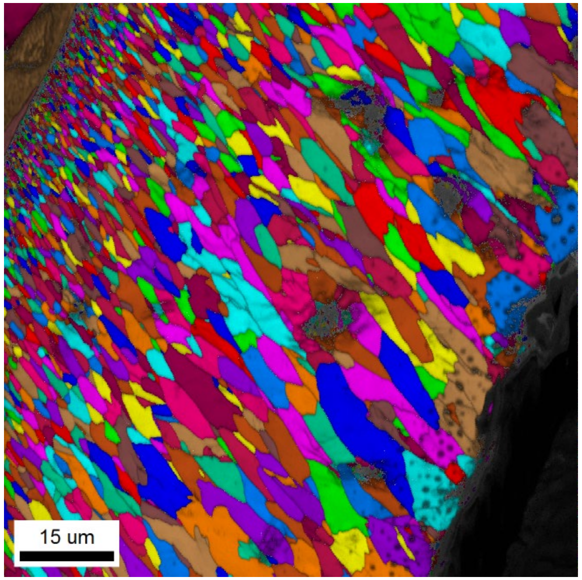




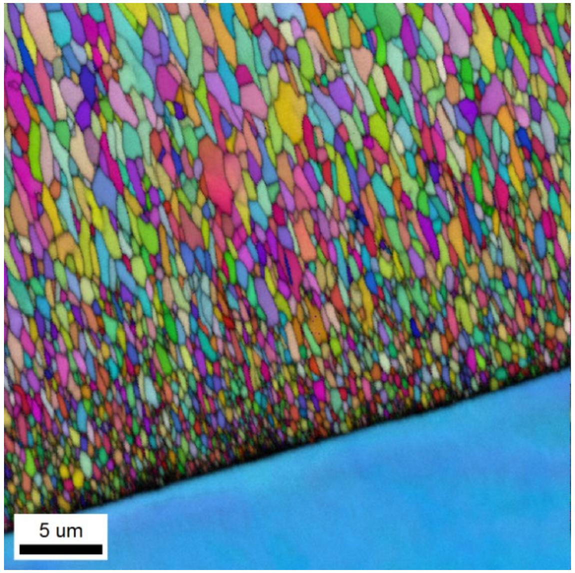
EBSD – Grain Size Measurement at Iris and Cell





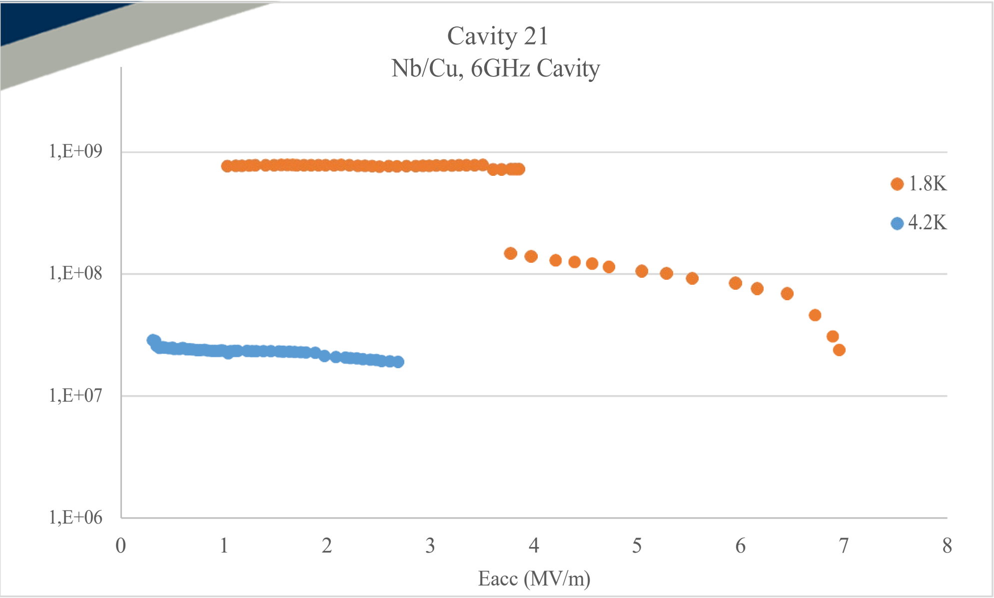


Measurement influenced by position of analysis: finer grains measured towards Cu interface (note scale on x-axis).

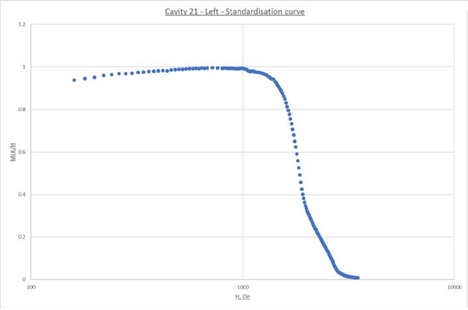


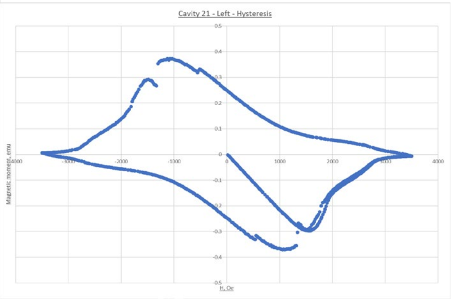
RF and DC Evaluation of Cavity 21

I (A)	V (V)	P (mbar)	B (G)	T (˚C)	d (μm)	dep. mode	d_{sl} (nm)	n_{sl}	D	venting mode
1	≈320	$5\cdot10^{-2}$	830	550	≈70	multilayer	500	130	0.7	standard

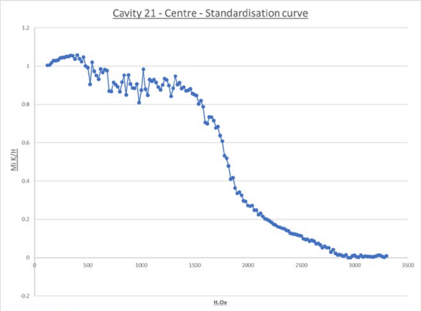


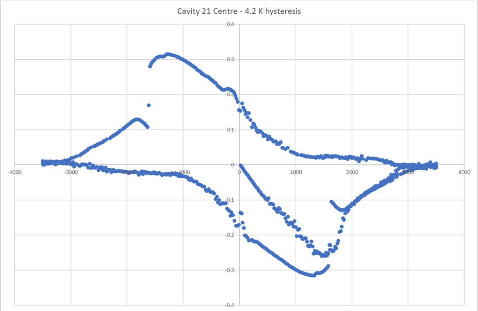
stripped Nb film on Kapton tape left position



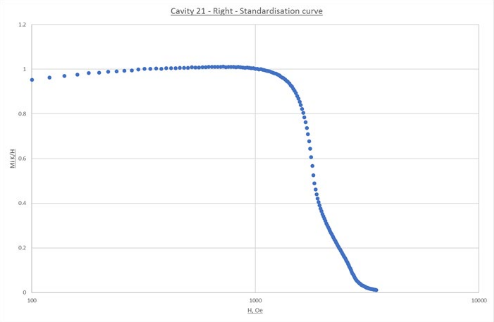


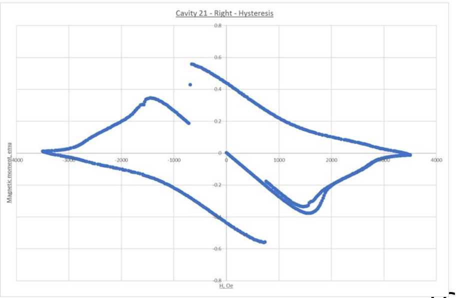
stripped Nb film on Kapton tape centre position





stripped Nb film on Kapton tape right position

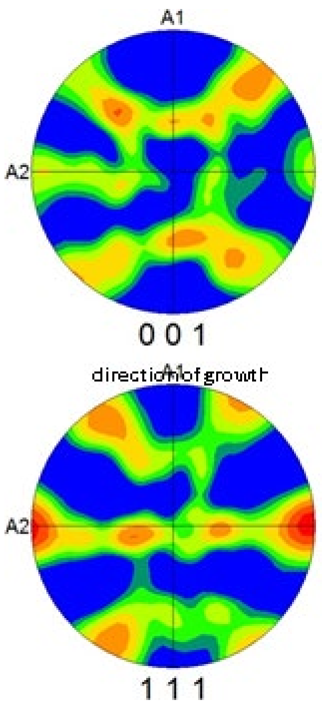


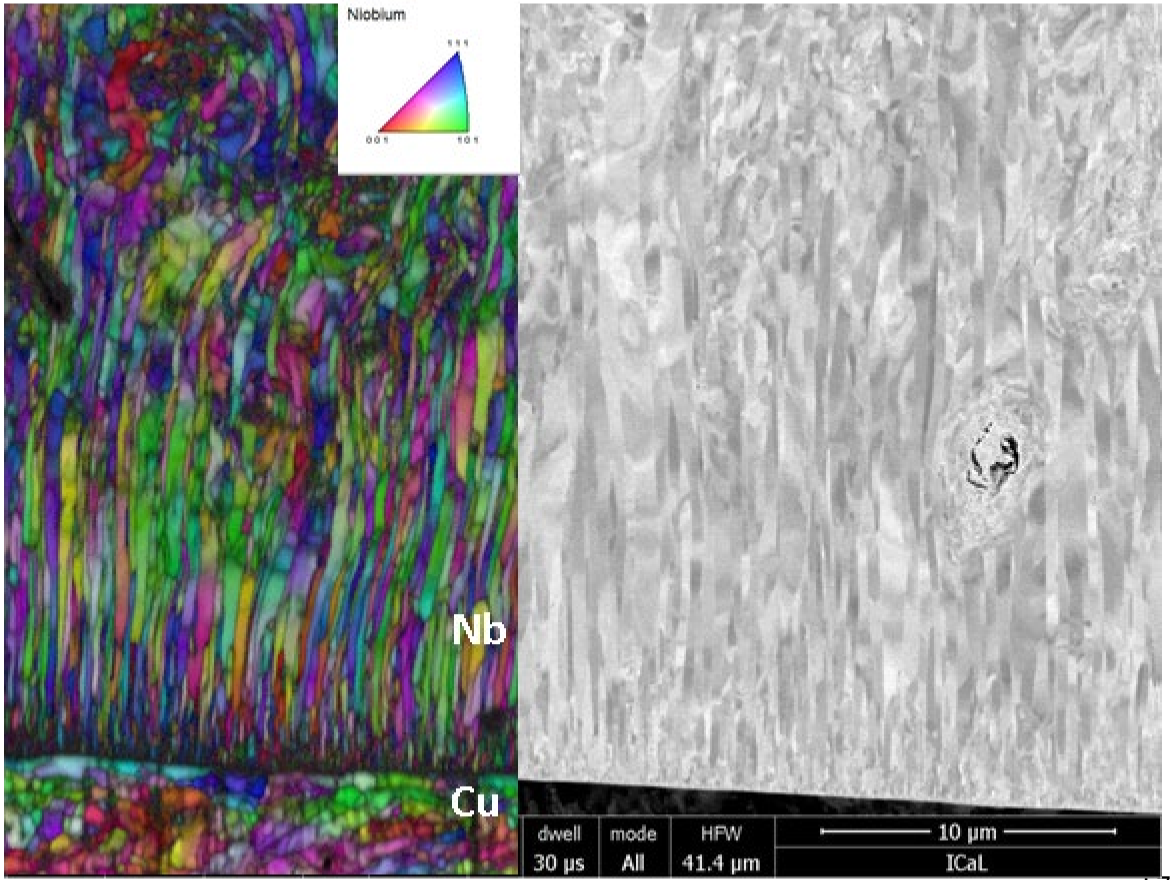


H_{fp} for centre position 148 mT at 5%
Flux jumping can be observed
H_{fp} for right position 134 mT at 5%
No flux jumping is observed
H_{c2} at 350 mT

EBSD - Colloidal silica polished surface

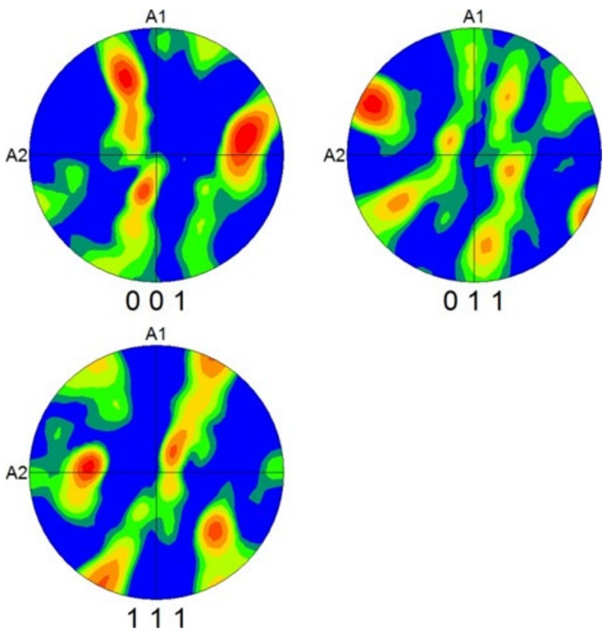
Distortion of columnar crystal growth also evident in EBSD patterns. Crystallographic texture has been modified by the polishing process. Initial fine grain Nb formation in the early stages of deposition gives way to the formation of columnar grains. However, distortion of the microstructure is evident at distances greater than 10 µm from the interface.

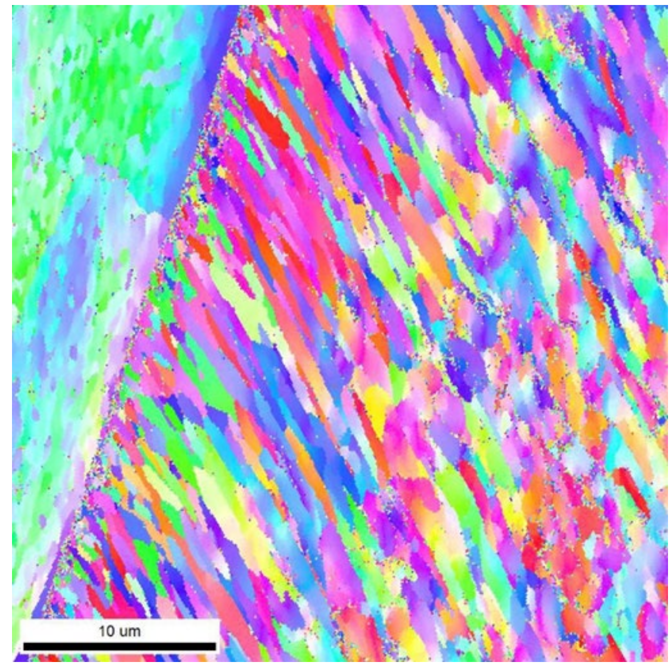


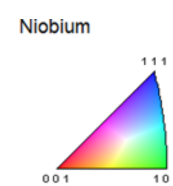


Niobium Texture plots indicate a preferred 011 crystal orientation parallel to the columnar grain growth direction

EBSD-Texture of IRIS position



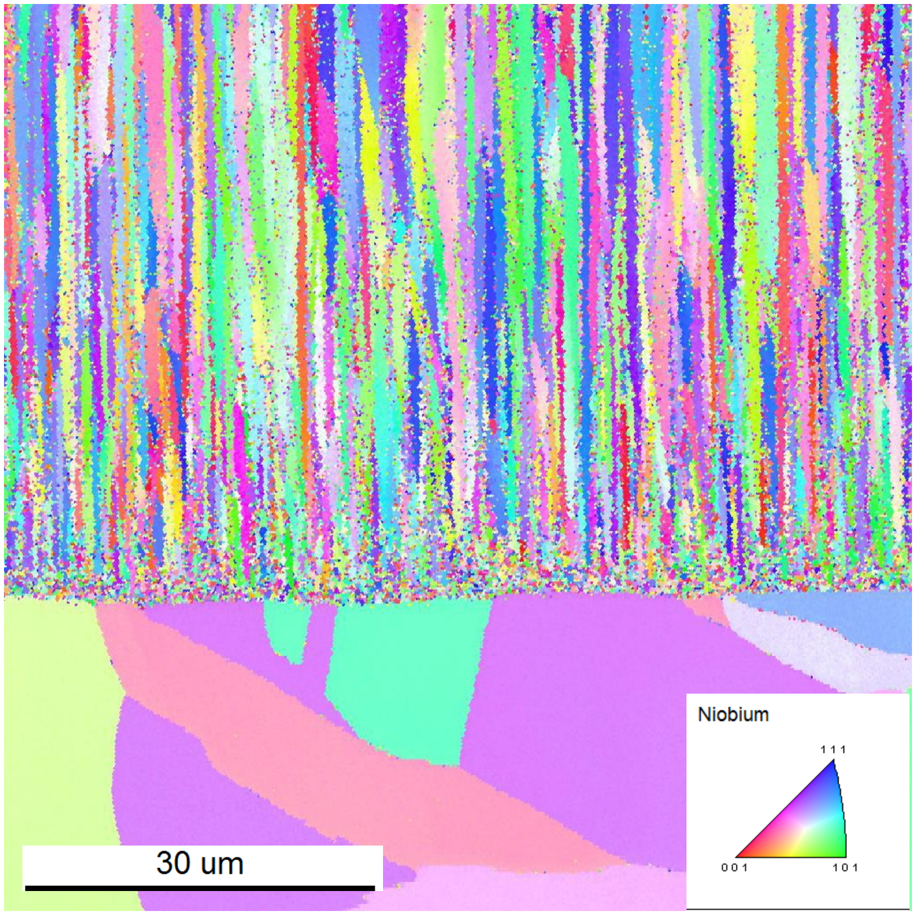


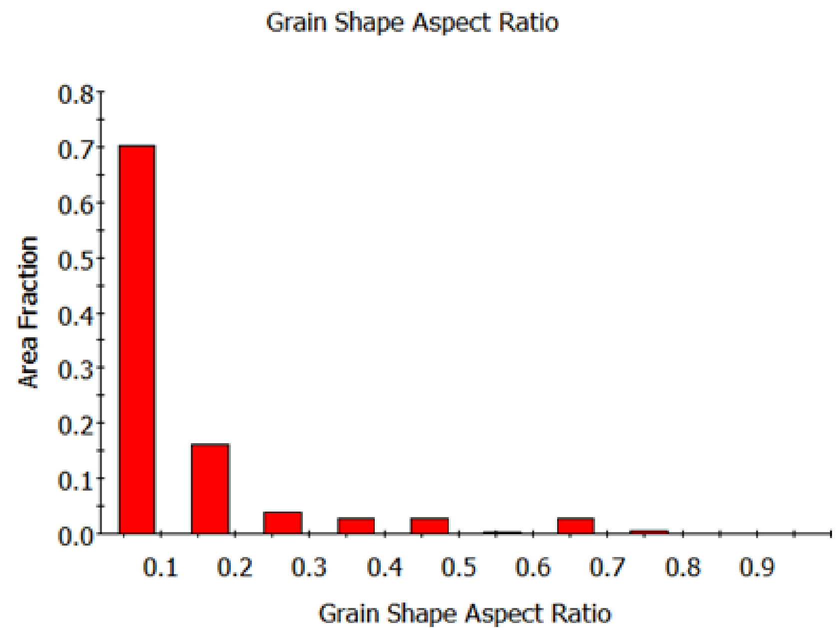


A preferred 011 crystallographic texture, parallel to the columnar grain long axis, is also observed where growth is not normal to the Cu surface.

EBSD – Ion Beam milling prepared centre specimen

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

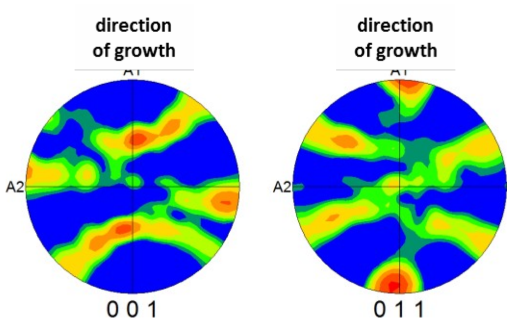


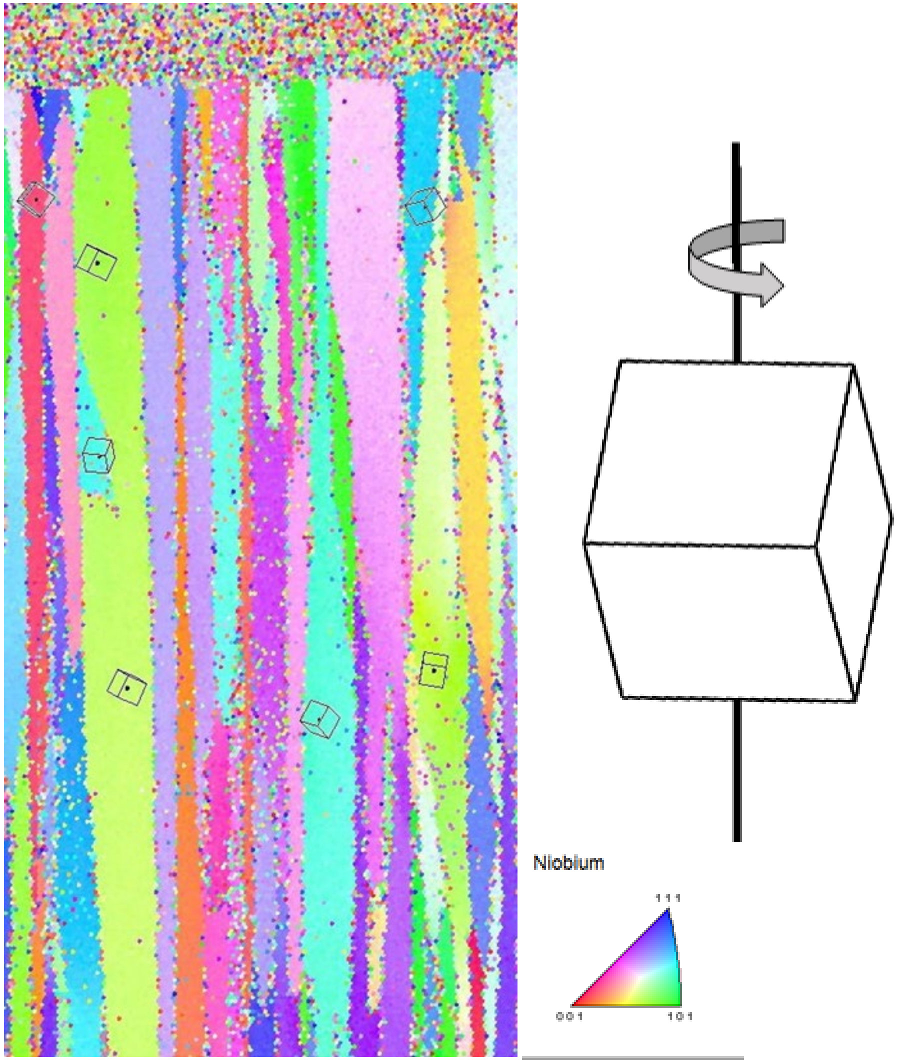


The fine equiaxed Nb grains, formed during the early stages of deposition are not easily resolved. Large twinned grains were observed in the Cu substrate.

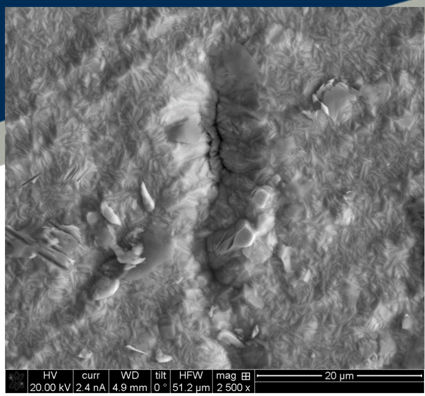
EBSD Texture For Ion Beam Milled Centre Sample

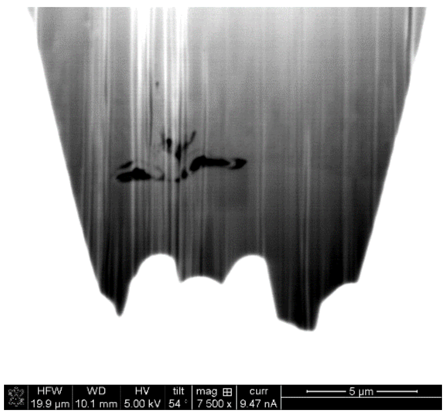
EBSD texture measurements performed on the Ar ion beam prepared niobium layer were consistent with those recorded from the colloidal silica prepared materials. A strong <011> texture was measured, parallel to the long axis of the columnar grains, which is approximately parallel to the growth direction.



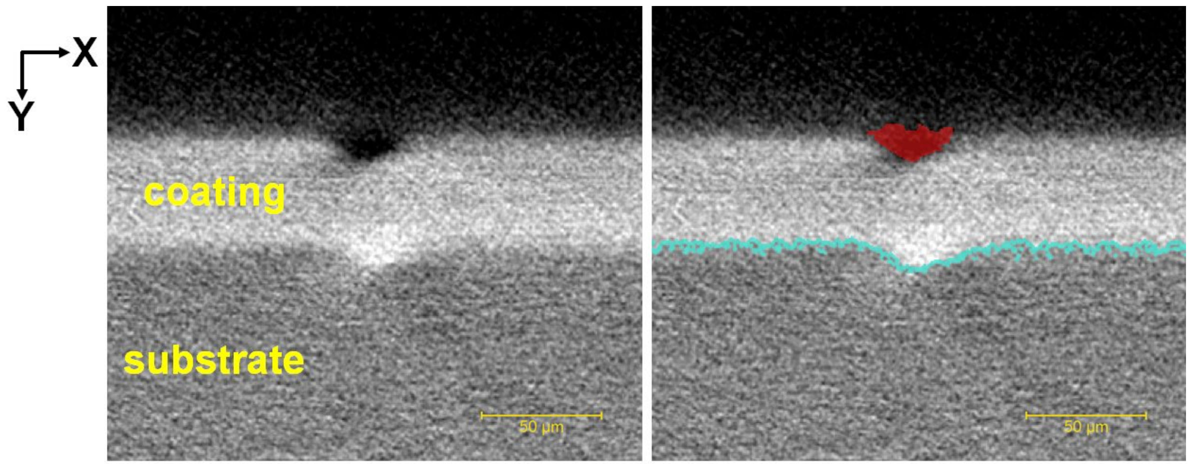


X-Ray CT Scan and FIB SEM Of Surface Defect

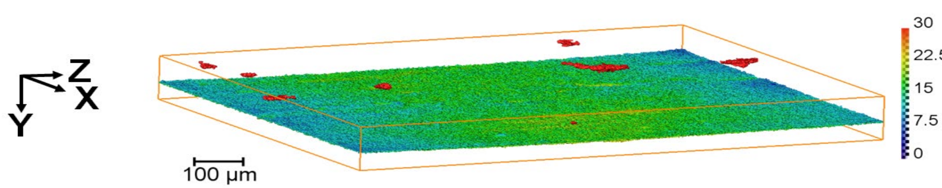




Virtual cross-section showing the coating and substrate. The interface is highlighted in cyan and the defects at the coating surface is highlighted in red.



3D view of the interface coloured using the y-coordinate of the interface. The defects on the coating surface is rendered in red.



SUMMURY

- In all cases the direction of growth at the cell is always perpendicular to the surface and is at 45 degree to the surface at the Iris.
- The microstructure and film preferred texturing has been consistent in all the four cavities. The film grows in small grain at the interface and after several microns then it grows with high aspect ration columnar grains through the near complete thickness of the deposited film
- All films has shown almost identical superconducting behaviour in DC magnetometer at 4.2 K achieving a reasonably high H_{fp} value between 140 to 155 mT which is in good agreement with RF results which was better or equal to Q value of best Niobium cavity.
- Mechanical disruption of the microstructure, when polishing using SiC papers and colloidal silica damages the columnar structure of the niobium.
- Argon ion beam polished surfaces were of a higher quality than those prepared by mechanical means.
- Straight, high aspect ratio, columnar niobium grains were observed through near full thickness of the layer prepared by Ar ion beam.
- X-Ray CT scan can be valuable tool to study large area of the sample for determining relatively large defects.