

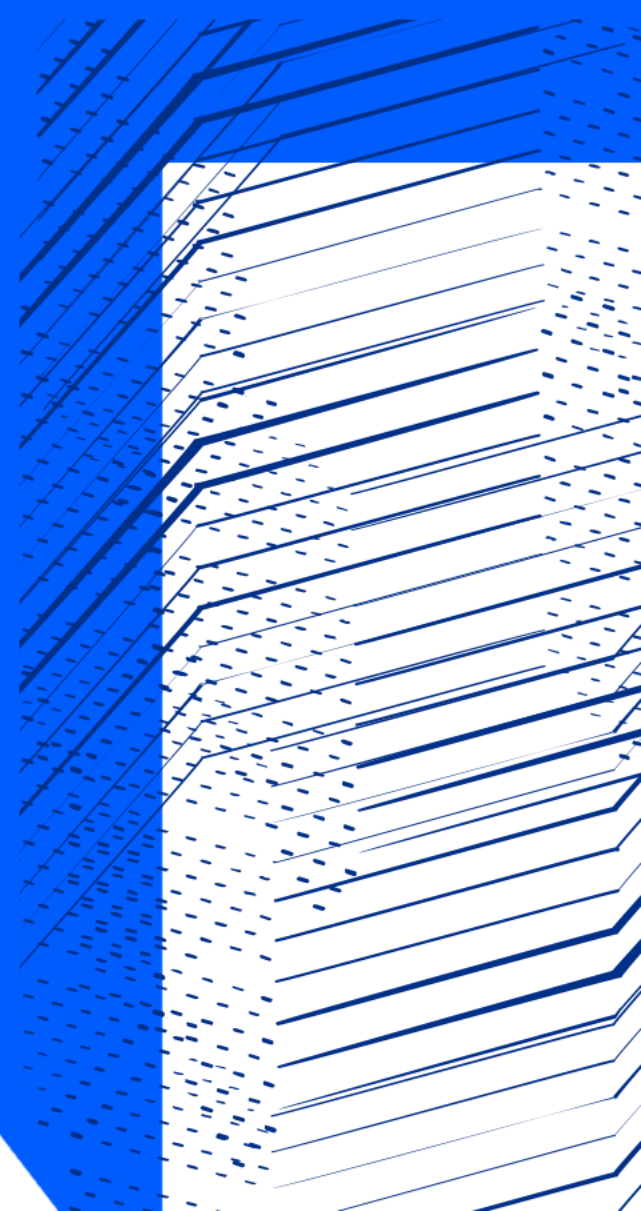


Science and
Technology
Facilities Council

PVD Deposition Of Nb_3Sn From An Alloy Target On Copper

Reza Valizadeh
on behalf of the team

Nb_3Sn Workshop 9th - 13th November 2020 Cornell University



MOTIVATION

❑ Bulk niobium (Nb) has been for the past three decades: the material of choice for SRF applications:

- It has the highest T_c (9.25K) for pure metal
- It has highest lower magnetic field H_{c1}
- Easy fabrication

But it has achieved the magnetic field limitation so further improvement of cavity RF performance dictate to turn to other superconducting materials.

❑ Nb₃Sn alloy is type II superconductor with ideal T_c of 18 K and superheating field of 400 mT. Hence can offer improvement

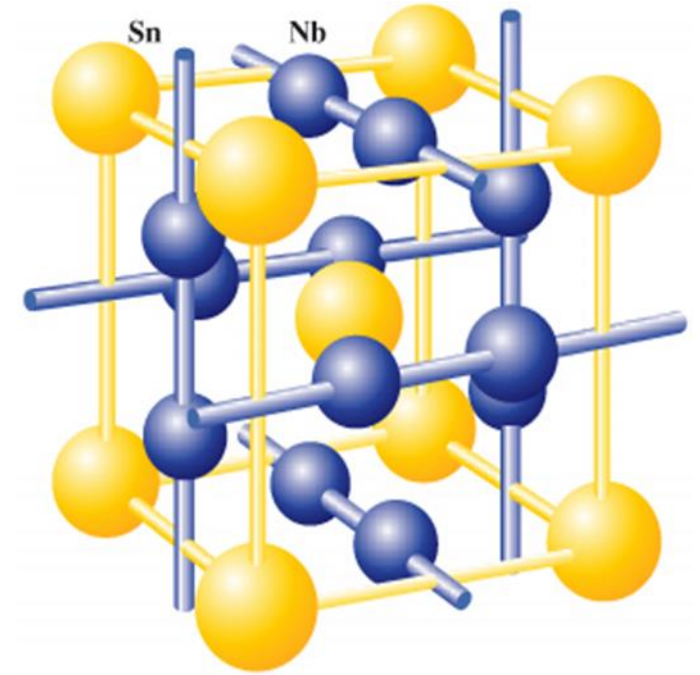
- Cryogenic efficiency
- Higher accelerating field.
- Recently there has been positive progress in producing Nb cavities with Nb₃Sn coating.

❑ The material can be deposited as thin film either in:

- Single layer (Nb₃Sn on Cu or Nb)
- Double layer (Nb /Nb₃Sn on Cu)
- Multilayer (SIS): Nb /Insulator/ Nb₃Sn, on Cu

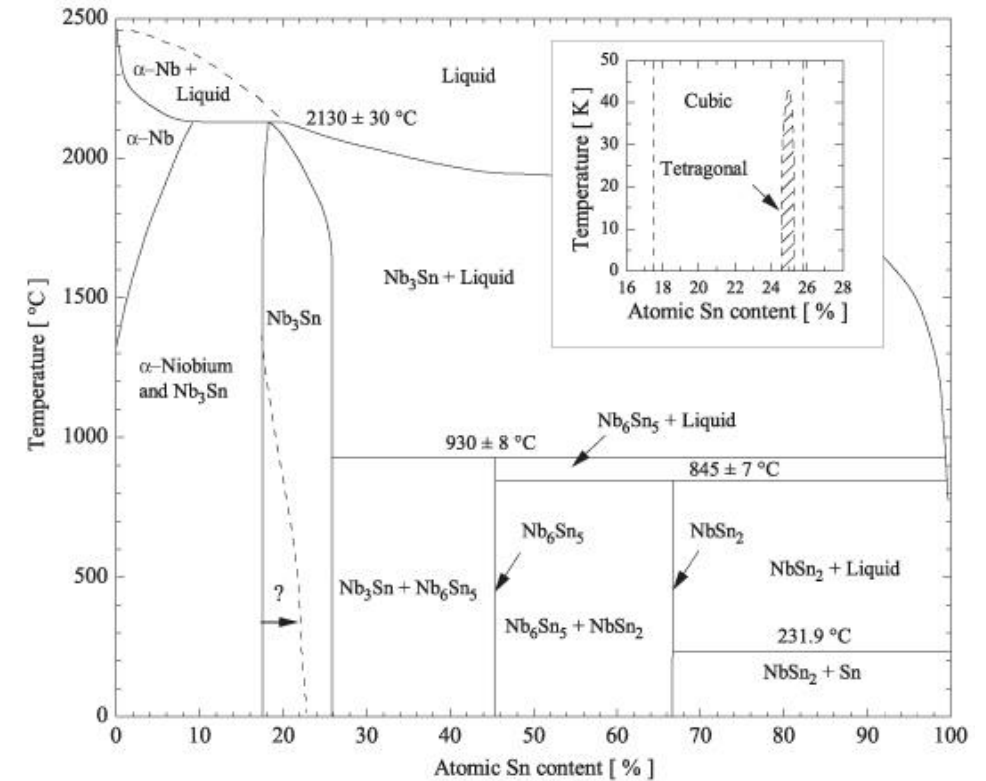
Nb₃Sn unit cell Structure

- The Sn atoms form a bcc lattice and each cube face is bisected by orthogonal Nb chains.
- In bcc Nb the shortest distance between the atoms is 0.286 nm starting from a lattice parameter of $a = 0.330$ nm
- In Nb₃Sn the lattice parameter is about $a = 0.529$ nm for stoichiometric composition and the distance between the Nb atoms is 0.265 nm
- The reduction of distance between the Nb chains is responsible for the high T_c in comparison to bcc Nb.
- Sn deficiency may cause the Nb to occupy the site and affect the long range order



Nb₃Sn binary phase diagram

- ❑ Intermetallic niobium–tin is based on the superconductor Nb, which exists in a bcc Nb structure or a metastable Nb₃Nb A15 structure
- ❑ When alloyed with Sn and in thermodynamic equilibrium, it can form either Nb_{1-β}Sn_β (about $0.18 \leq \beta \leq 0.25$) or the line compounds Nb₆Sn₅ and NbSn₂.
- ❑ Both the line compounds at $\beta = 0.45$ and 0.67 are superconducting, with
 - ❖ $T_c < 2.8$ K for Nb₆Sn₅
 - ❖ $T_c < 2.68$ K for NbSn₂



Nb₃Sn deposition system and parameters

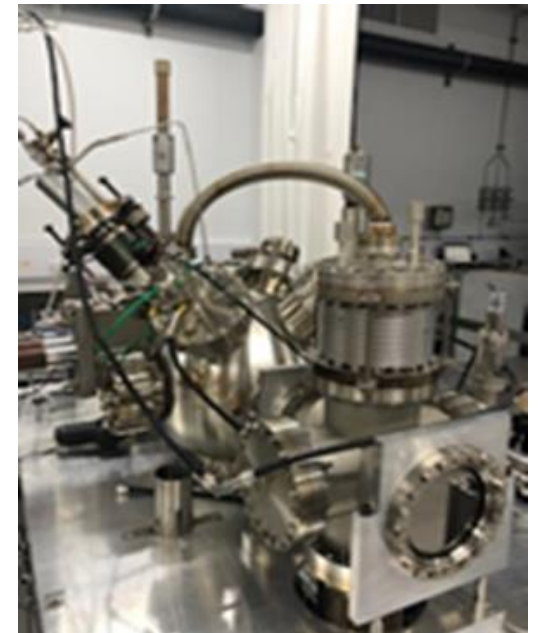
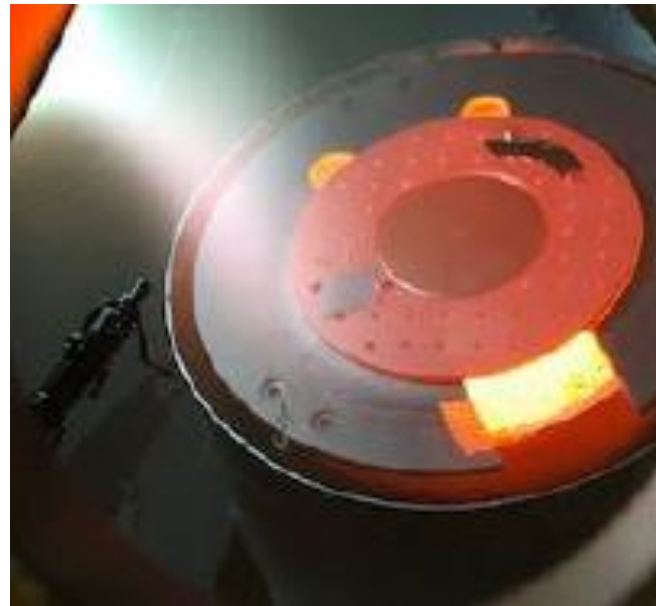
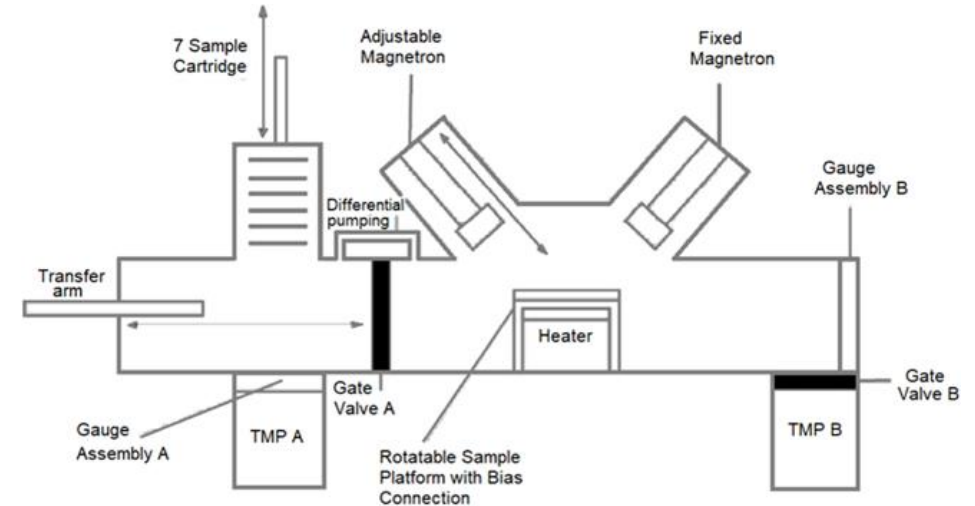
- ❑ Magnetron sputtering from a RRR 300 Nb target
- ❑ Substrate Temperature, Deposition Rate, Deposition Thickness, Substrate Bias, Concurrent Ion Bombardment can be varied independently.
- ❑ Substrates are loaded into the load lock and system fully Baked.
- ❑ Base pressure 2×10^{-9} mbar is routinely achieved

Nb deposition:

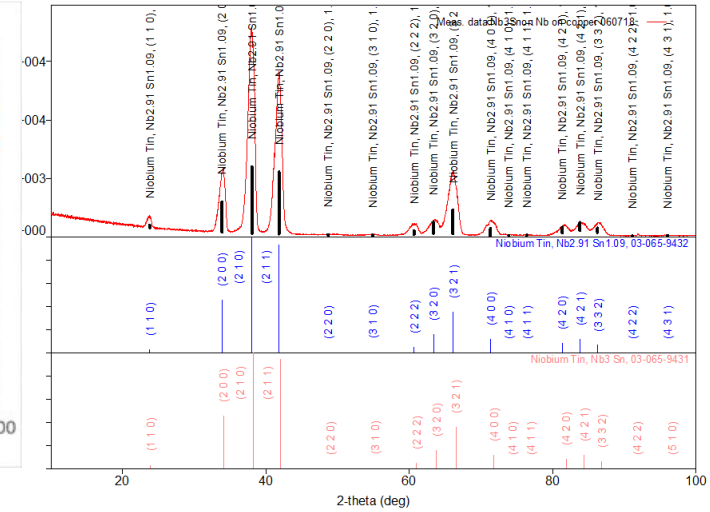
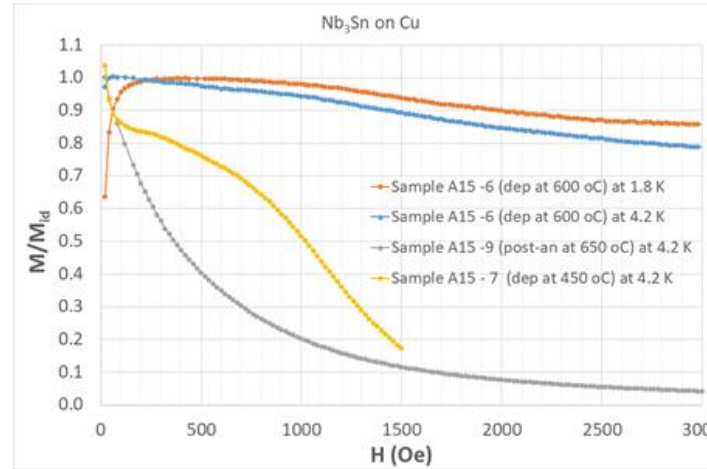
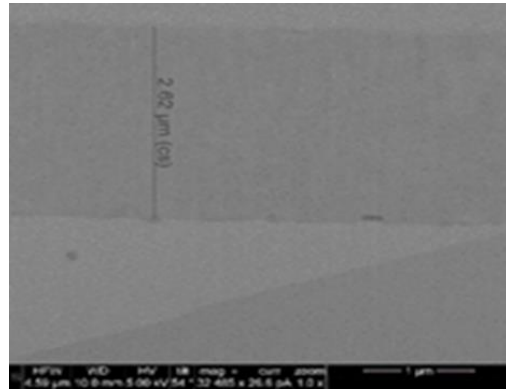
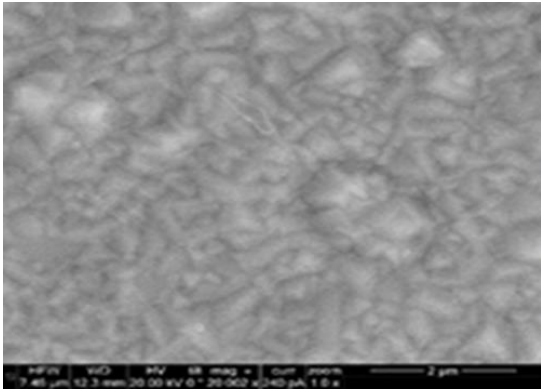
- 400 W, 470v, 0.85A
- 4 hours deposition
- DC sputtering

Nb₃Sn deposition:

- 200 W, 489 V, 0.41 A
- 2 Hours deposition
- DC sputtering



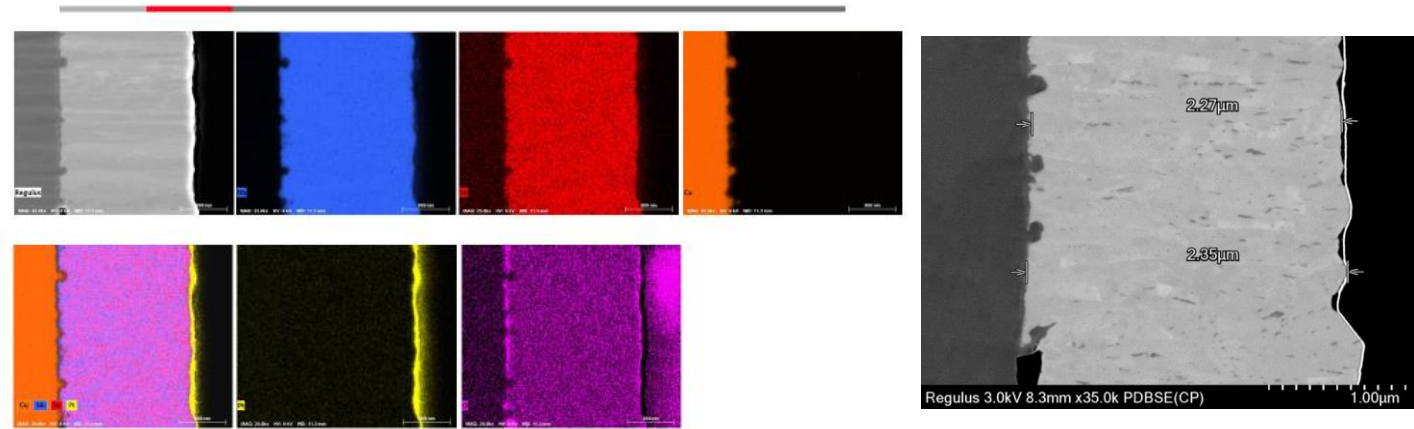
Cu/Nb₃Sn deposition (single layer) at various Temperature



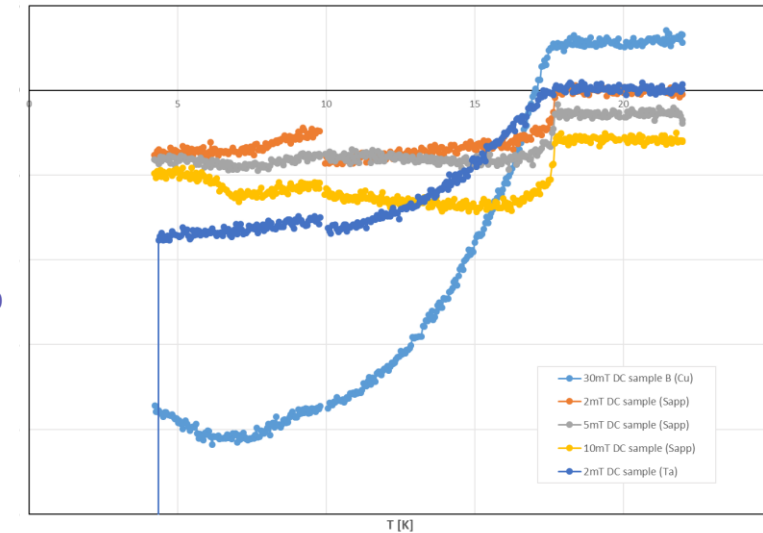
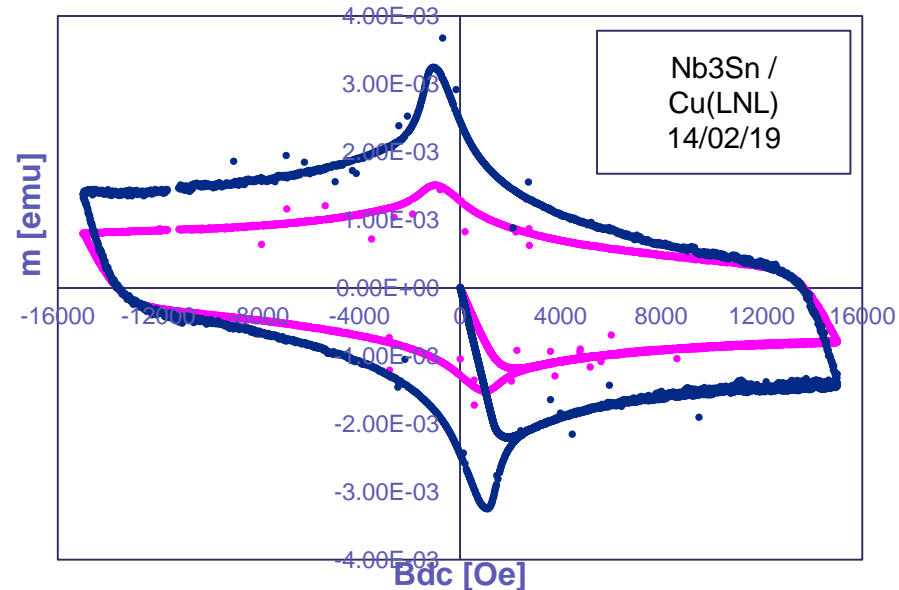
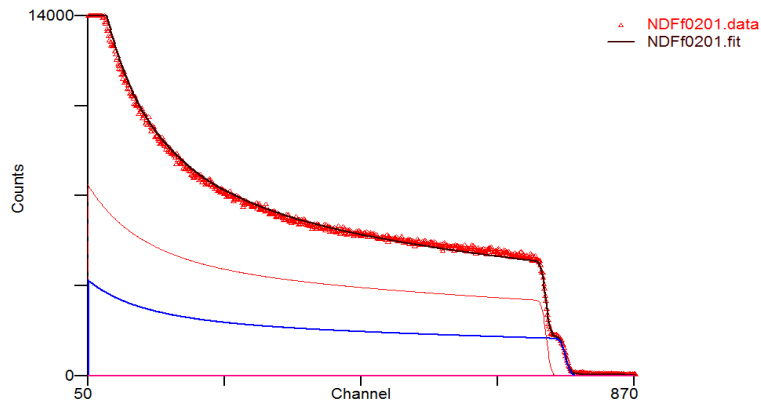
- ❑ Nb₃Sn were deposited on copper with no prior chemical cleaning such (EP or SUBU5) at various Temperature of RT, 450°C and 600°C. The RT deposition showed no sign of superconductivity.
- ❑ Best performance in terms of superconducting properties is achieved by the film deposited at 600°C (A15-6) with a T_c of 15.7 K, a lattice parameters of 0.529 nm and grain size in order of 8 to 10 nm.
- ❑ The film deposited at moderate temperature of 450°C was superconducting but its performance is much reduced with T_c of 14.6 K
- ❑ The film deposited at room temperature and then post annealed at 650°C (A15-9) has the worst performance since M/M_i drop sharply at very low field of about 10 mT.

Cu (EP)LNL / Nb₃Sn (single layer)

- There is some diffusion of copper at the interface
- There is a clear oxide layer at the interface despite high temperature treatment prior deposition (no prior chemical processing)
- There are area that it is Sn deficient.
- The T_c was determined to be between 17.75K (on sapphire) and 17.5 on copper
- First B_{en} estimated to be 50mT and 140mT deposited on Cu and Sapphire.

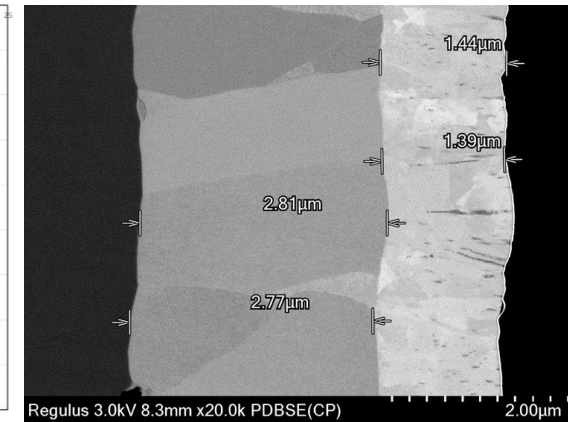
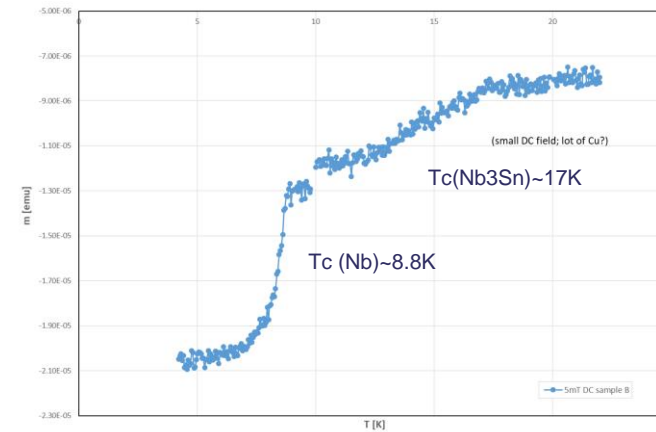


Layer	t (1e15at/cm2)	t (nm)	r(1e22at/cm3)	Nb	Sn	Al	O
1	7606.631	1489.817	5.106	75.0000	25.0000	0.0000	0.0000
2	821647.625	69631.156	11.800	0.0000	0.0000	40.0000	60.0000

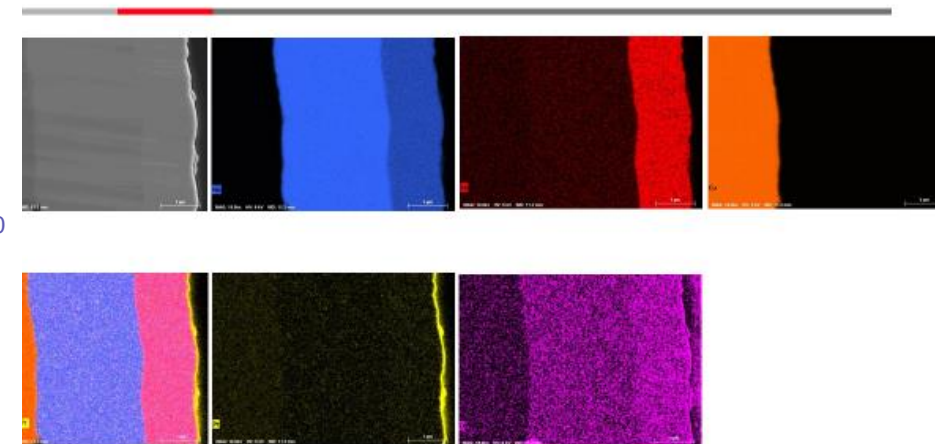
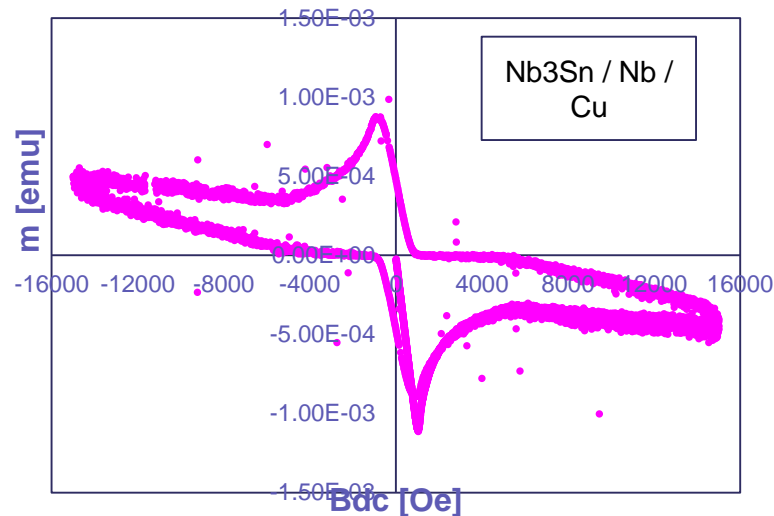
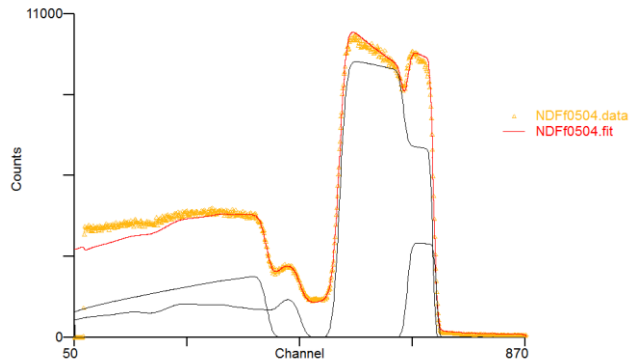


Cu /Nb/Nb₃Sn (double layer)

- The interfaces both at Cu/Nb and Nb/Nb₃Sn is well define
- Nb layer is grown is large grain and in a perpendicular direction to the substrate surface
- No intermixing of elements is observed
- Some area of Sn deficiency and rich Sn in Nb₃Sn layer can be observed
- First B_{en} is estimated at 95mT.

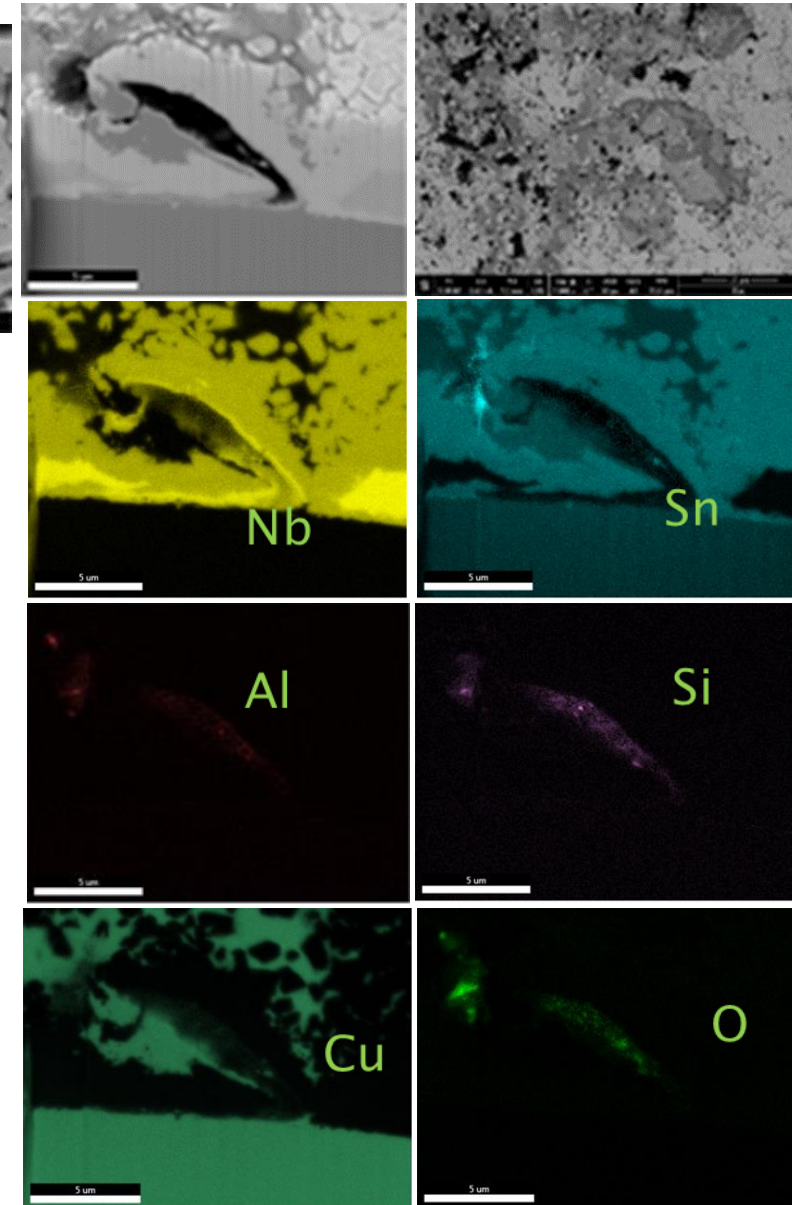
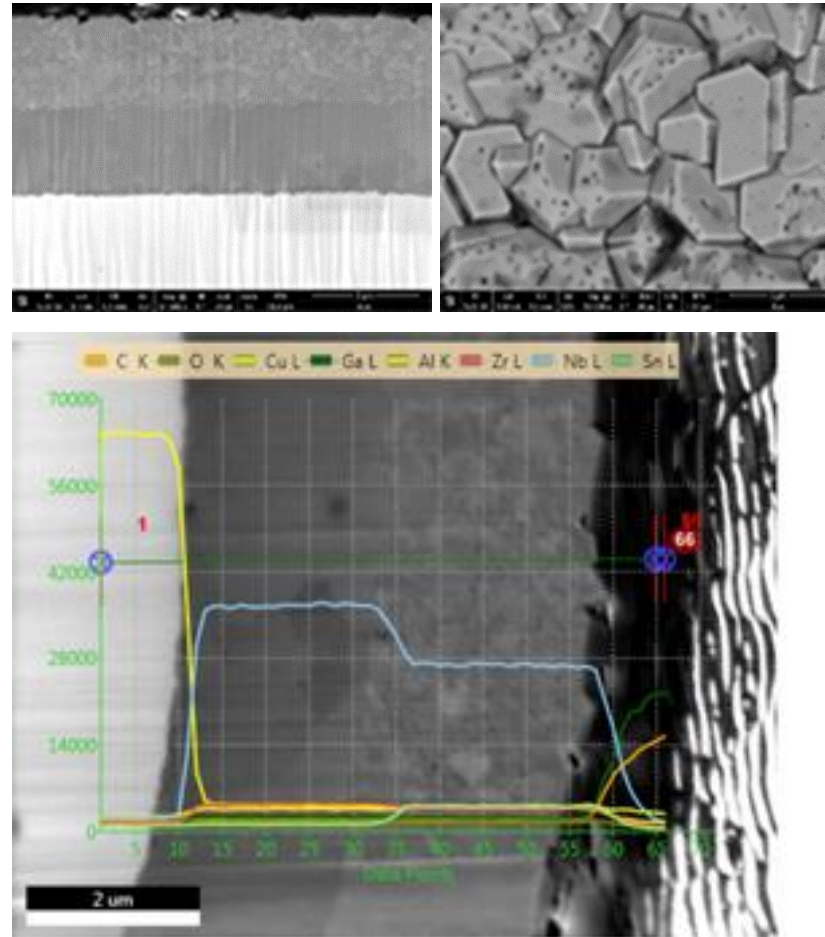


Layer	t (1e15at/cm2)	t (nm)	r(1e22at/cm3)	Nb	Sn	O	Al
1	7644.333	1497.201	5.106	75.0000	25.0000	0.0000	0.0000
2	15920.986	2855.270	5.576	100.0000	0.0000	0.0000	0.0000
3	898207.563	76119.281	11.800	0.0000	0.0000	60.0000	40.0000



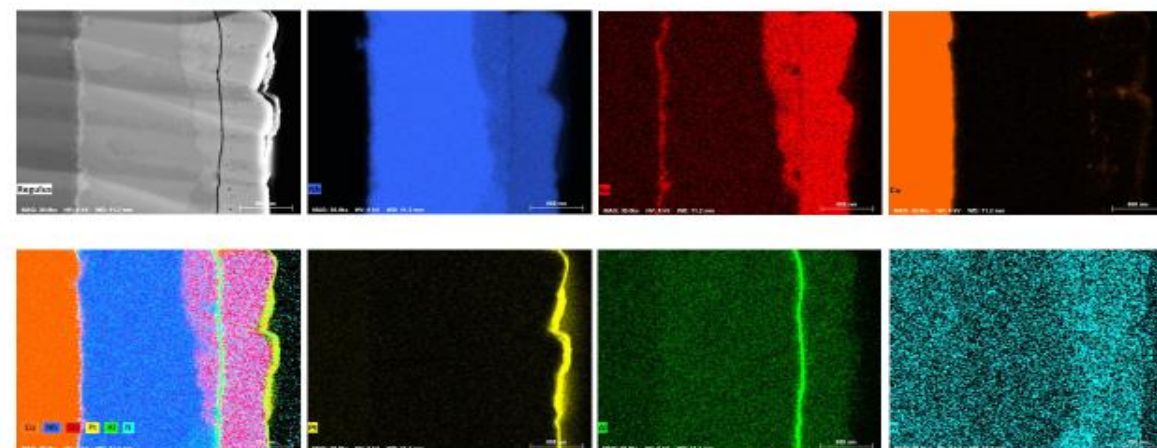
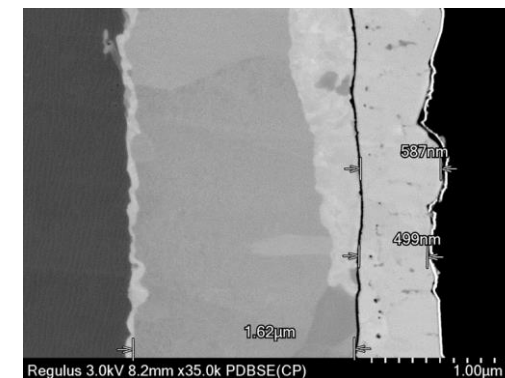
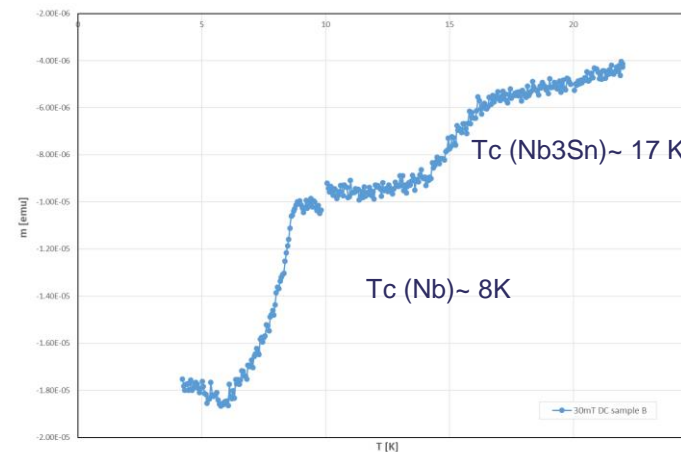
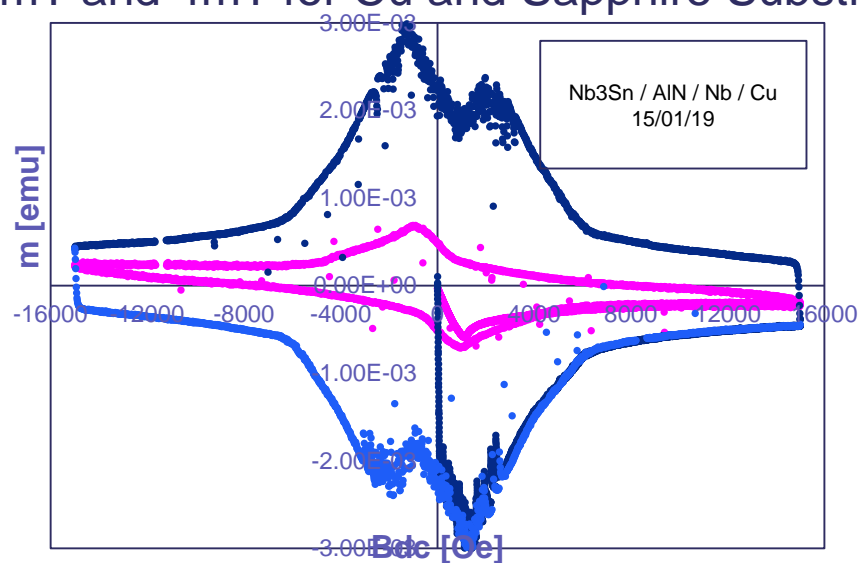
Cu/Nb/Nb₃Sn (double layer)

- Two distinct area can be observed:
 - Perfect area with sharp interface with correct stoichiometry for Nb₃Sn layer
 - Copper diffusion from the interface to top surface.
- Nb and Nb₃Sn layers are completely intermixed and there is a substantial volume of copper substrate is present throughout the depth of the layer and at the surface
- Inside the cavity there are trace silicon and aluminium oxide.

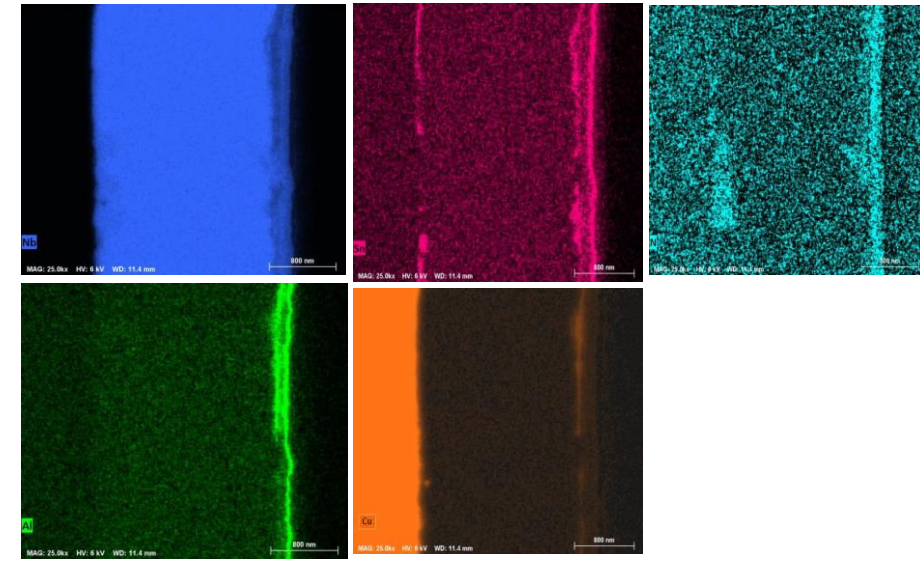
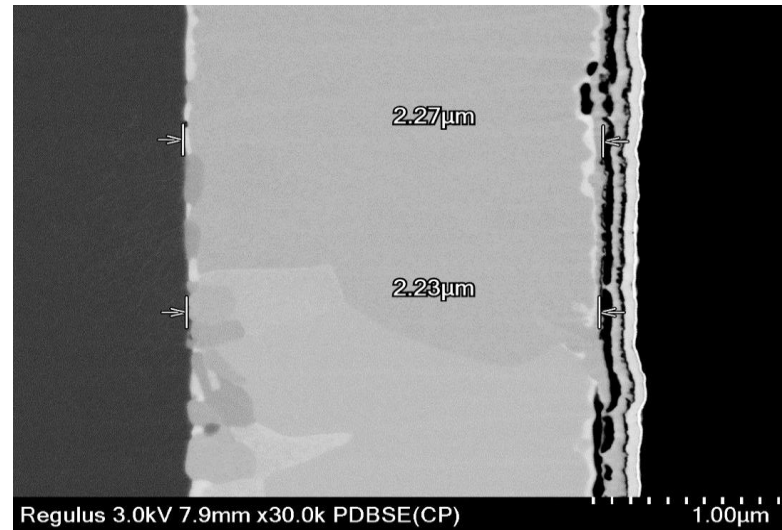
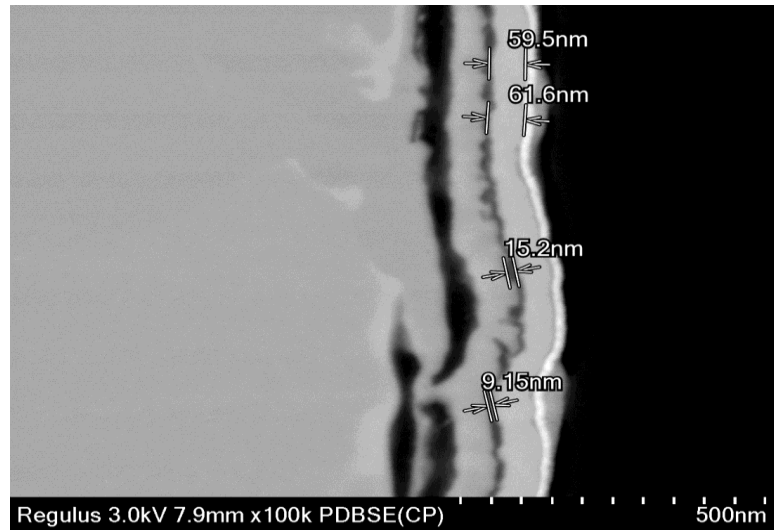


SIS Structure of thick Nb₃Sn/AlN/Nb multilayer on copper

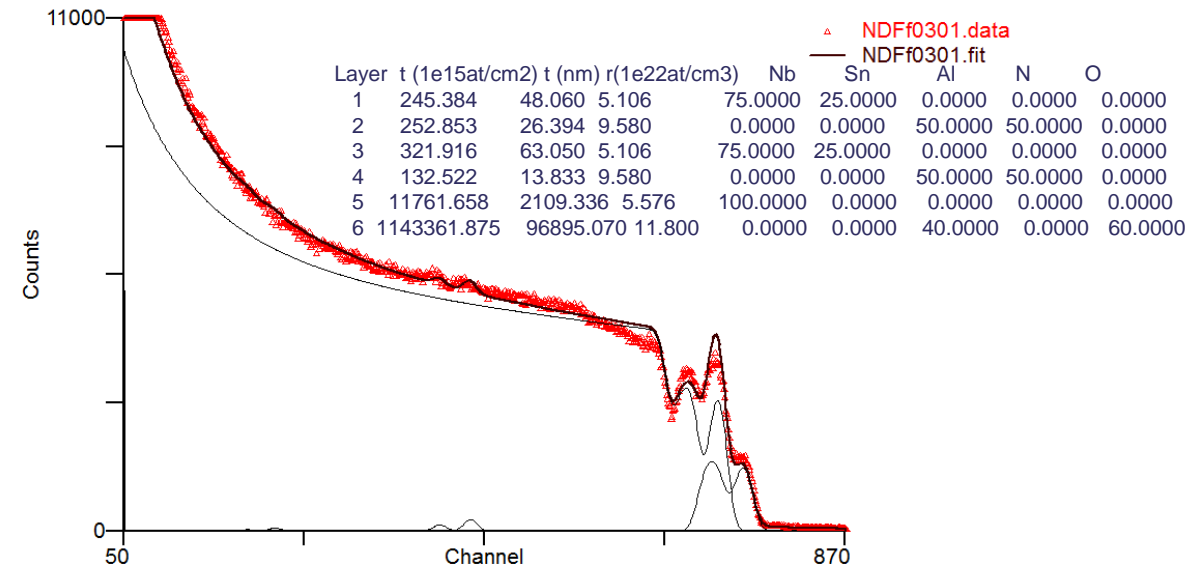
- Although the layers are well identified however there is again some degree of mixing can be observed.
- 1. Sn segregation at Cu/Nb interface
- 2. Nb₃Sn into Nb layer below the AlN layer at some places
- 3. Copper diffusion on to the surface
- 4. Some level of Nitrogen diffusion into all the layers.
- 5. The B_{en} in parallel external field is estimated to be 61mT and 4mT for Cu and Sapphire Substrate



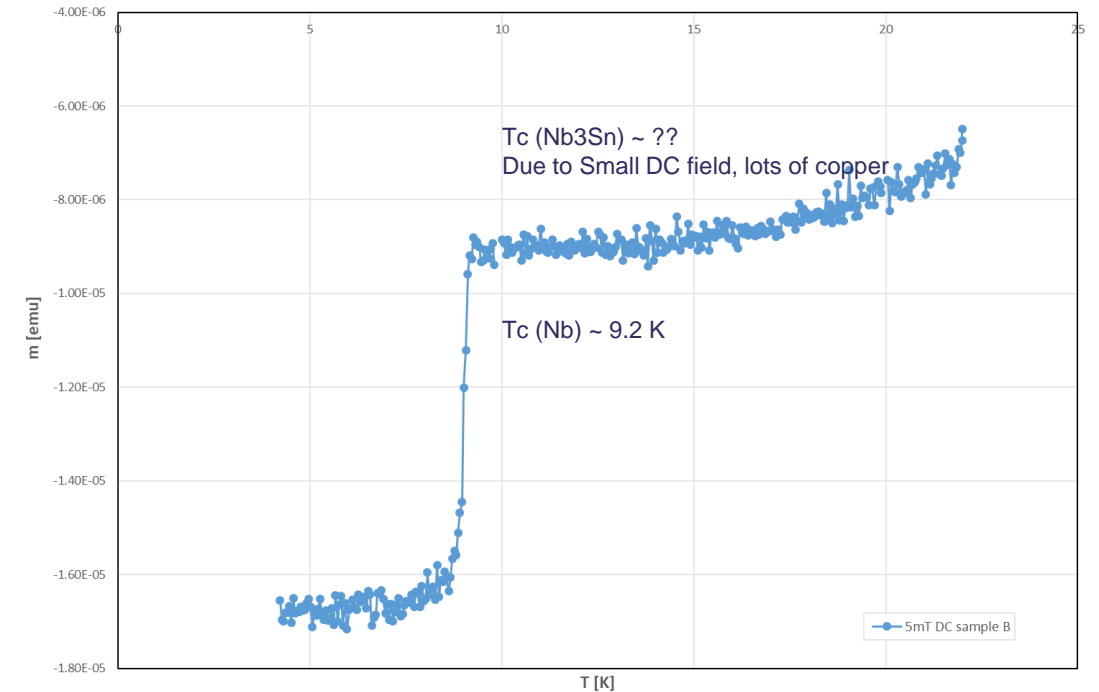
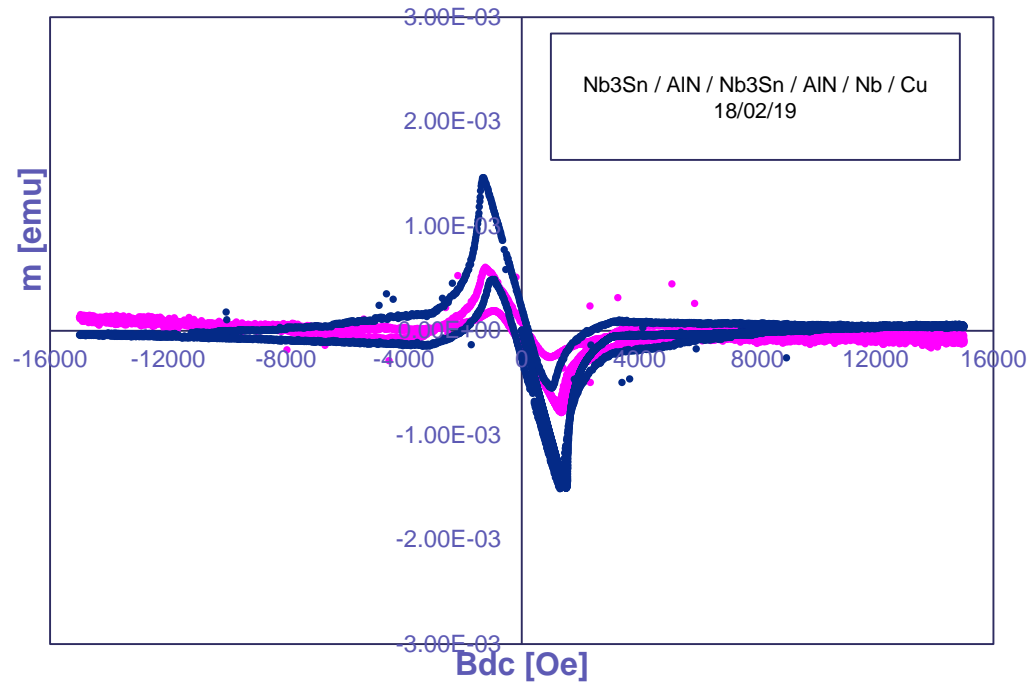
SIS Structure of thin layer $(\text{Nb}_3\text{Sn}/\text{AlN})_2/\text{Nb}$ multilayer on Cu



- The observed distortion of the Nb_3Sn / AlN bilayer is most probably due to mechanical polishing prior to ion beam milling, since such distortion is not present in the RBS spectra
- The RBS analysis predicts stoichiometric layers with the second AlN layer almost being double the thickness of the first AlN layer. Similarly the first Nb_3Sn layer is 25% thicker than the second Nb_3Sn layer.



SIS Structure of thin layer $(\text{Nb}_3\text{Sn}/\text{AlN})_2/\text{Nb}$ multilayer on Cu



- The B_{en} in parallel external field is estimated to be 108 mT and 130 mT for Cu and Sapphire Substrate
- There is nearly no hysteresis in the thin double SIS structure
- This can be due to the protective effect of multilayers which reduces the sensitivity to pinning effect.

Summary

- ❑ Nb_3Sn can be successfully deposited from an alloy target with satisfactory SC properties when it is deposited at high temperature (around 600-650 °C).
- ❑ Final Smooth surfaces and sharp interface between layers can be achieved by suitable surface preparation method/process of substrate
- ❑ Impurities such as silicon oxide may cause complex defects to be formed when Nb_3Sn is deposited in multilayer structure.
- ❑ Substrate preparation can influence the growth of the film and hence its SC properties
- ❑ Protective effect of multilayers to some extent is been shown:
 - Less sensitivity to pinning defects
 - Multilayer structure even without insulating layer reduced the hysteresis loop
 - Defects are still present in individual layers
 - Not detected anymore when SIS structure fully SC.
- ❑ Complementary non-destructive technique such as RBS proved to be a powerful technique to distinguish post sample preparation damage.

Special Thanks to all the team member

STFC

- Adrain Hannah
- Gavin Stenning
- Daniel Turner
- Yukari Dan (Hitachi)
- Karl Dawson
- Spideh Aliasghari
- Stuart Wilde
- Oleg Malyshev
- Tobias Junginger
- Graeme Burt
- Vinod Dannak

University of Siegen

- Michael Vogel
- Stewart Leith



IEE

- Eugen Seiler
- Rastislav Ries

INFN

- Cristian Pira
- Eduard Chyhyrynets

CEA

- C.Z Antoine

JLAB

- Anne Marie Valente

HZB

- O. Kugeler
- D. Tikhonov

RTU

- A Medvid



Science and
Technology
Facilities Council

Thank you



Science and Technology Facilities Council



@STFC_matters



Science and Technology Facilities Council