Science and Technology Facilities Council

Deposition and Characterisation of Alternative Superconducting thin films to Niobium for SRF applications

Reza Valizadeh

on behalf of the team

9th International Workshop on Thin Film JLAB

MOTIVATION

- Bulk niobium (Nb) for the past three decades has been the material of choice for SRF applications:
  - It has the highest Tc (9.25K) for pure metal
  - It has highest lower magnetic field H_{c1}
  - Can be Easily fabricated
- But it has achieved the magnetic field limitation so further improvement of cavity RF performance dictate to turn to other superconducting materials.
- Nb_3Sn and NbTiN alloy are type II superconductor with ideal Tc of 18 K and superheating field about 400 mT. Hence can offer improvement in
  - Cryogenic efficiency
  - Performing at higher accelerating field.
  - Recently there has been positive progress in producing Nb cavities with Nb_3Sn coating.

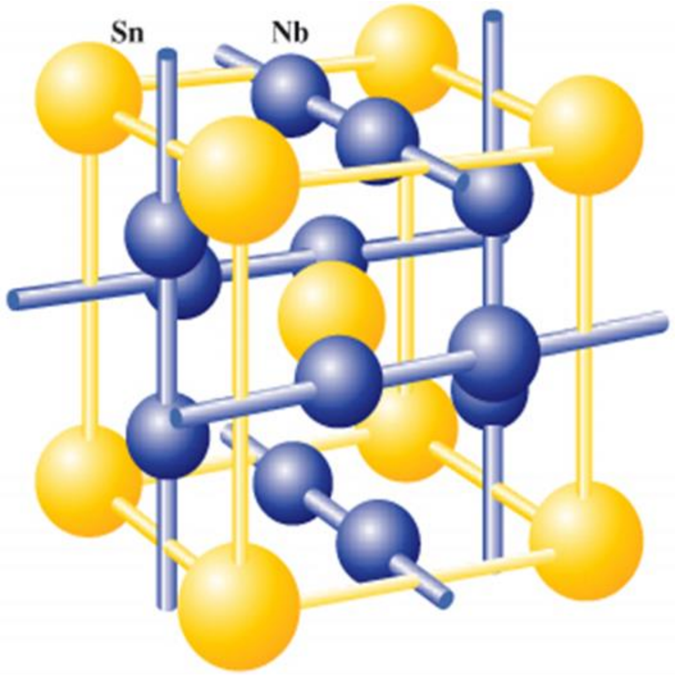
- The materials can be deposited as thin film either in:
  - Single layer (Nb_3Sn / NbTiN on Cu or Nb)
  - Double layer (Nb /Nb_3Sn or Nb / NbTiN on Cu)
  - Multilayer (SIS): Nb /Insulator/ Nb_3Sn, or Nb / insulator / NbTiN on Cu

Science and Technology Facilities Council

Nb_3Sn unit cell Structure

Material	T_c (K)	ρ_n(μΩcm)	$H_c(0)$ [T]	$H_{c1}(0)$ [T]	$H_{c2}(0)$ [T]	λ(0) [nm]	Δ [meV]	ξ [nm]
Nb_3Sn	18	20	0.54	0.05	30	80-100	3.1	4

- in Nb_3Sn unit cell the Sn atoms forms a bcc lattice and each cube face is bisected by orthogonal Nb chains.
- In bcc Nb the shortest distance between the atoms is 2.860 A starting from a lattice parameters of a= 3.300 A
- In Nb_3Sn the lattice parameters is about a = 5.290 A for stoichiometric composition and the distance between the Nb atoms is 2.650 A
- The reduction of distance between the Nb chains is responsible for the high Tc in comparison to bcc Nb.
- Sn deficiency may cause the Nb to occupy the site and effect the long range order



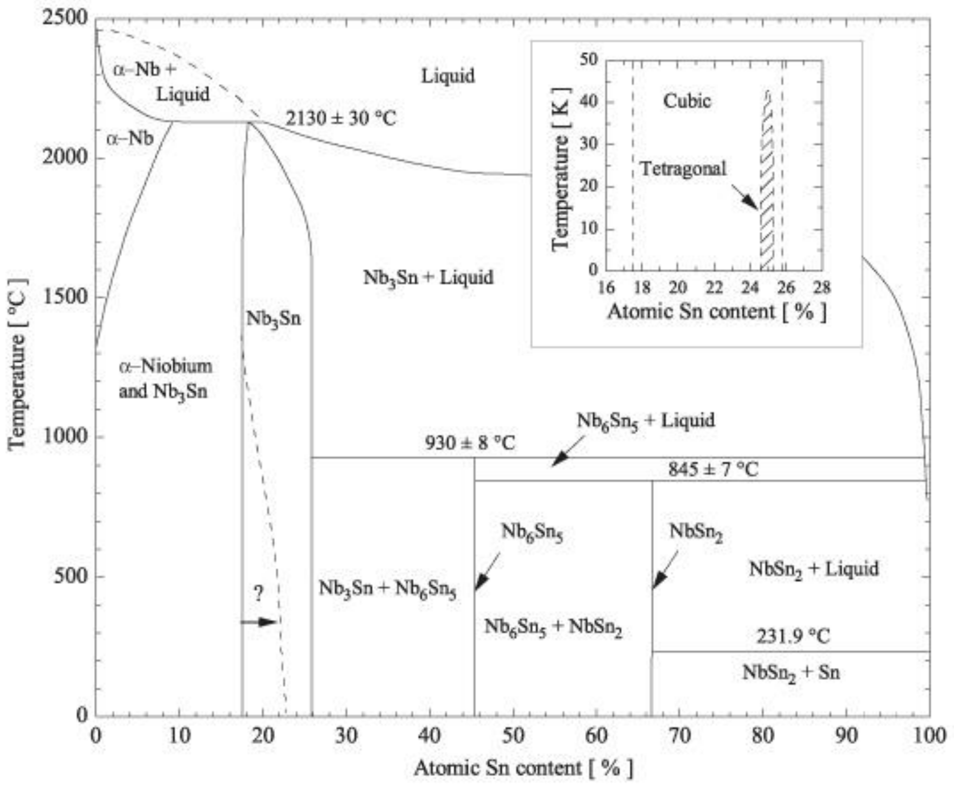
Science and Technology Facilities Council

Nb_3Sn binary phase diagram

- Intermetallic niobium–tin is based on the superconductor Nb, which exists in a bcc Nb structure or a metastable Nb_3Nb A15 structure

- When alloyed with Sn and in thermodynamic equilibrium, it can form either Nb1−βSnβ (about 0.18≤β≤0.25) or the line compounds Nb6Sn5 and NbSn2.

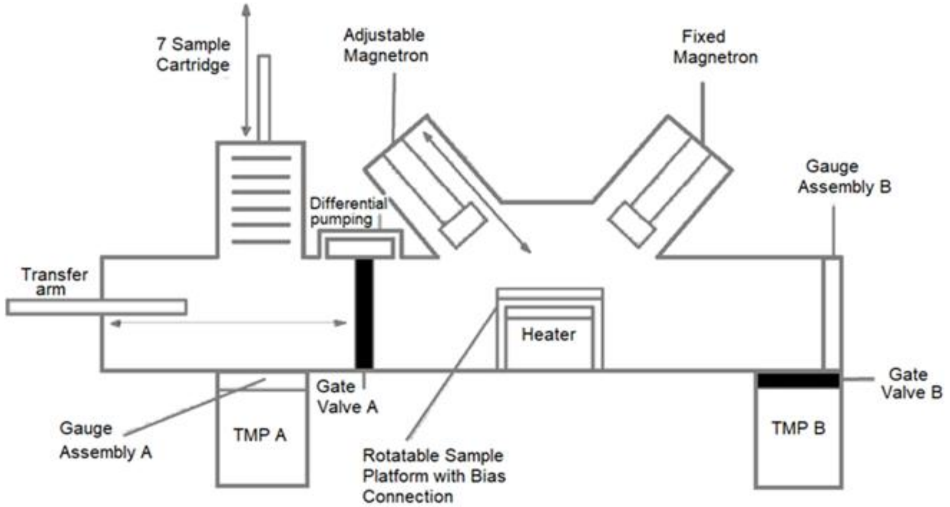
- Both the line compounds at β = 0.45 and 0.67 are superconducting, with
  - Tc<2.8 K for Nb6Sn5
  - Tc<2.68 K for NbSn2



Science and Technology Facilities Council

Nb_3Sn 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.

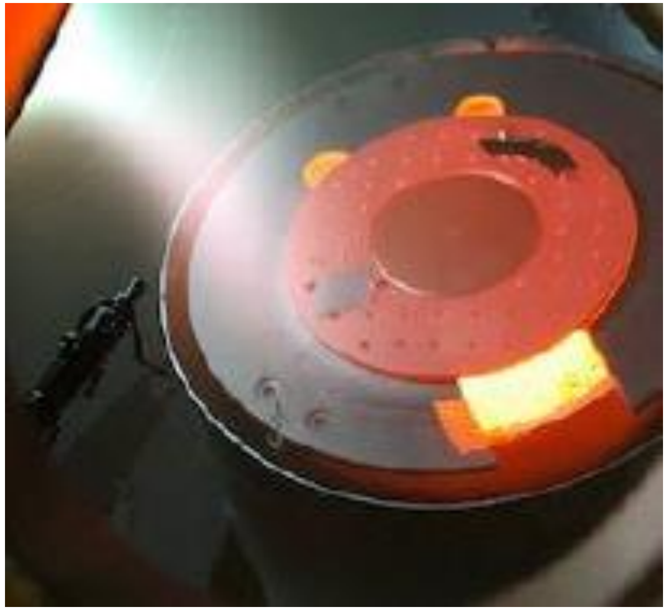


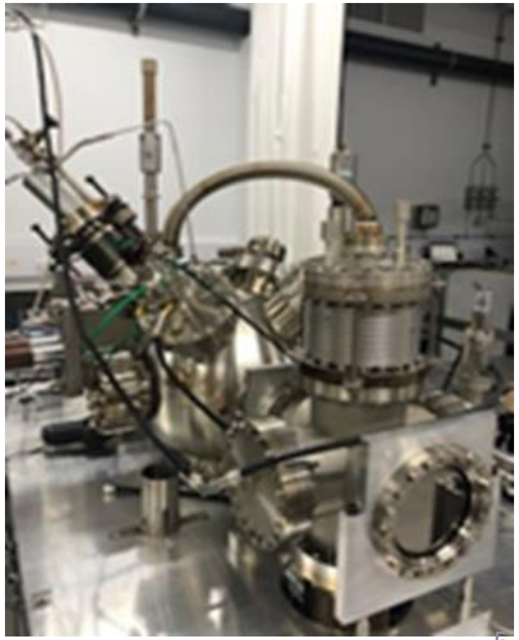
Nb deposition:

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

Nb3Sn deposition:

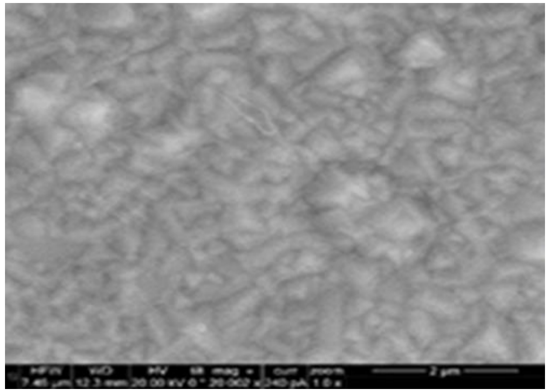
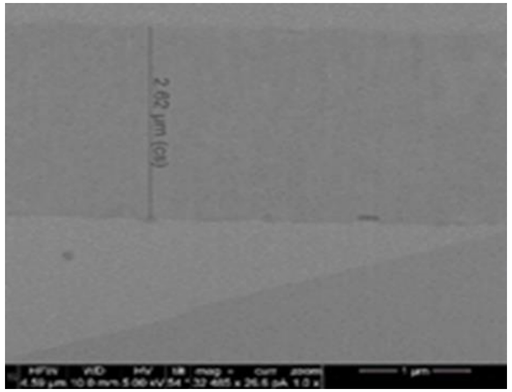
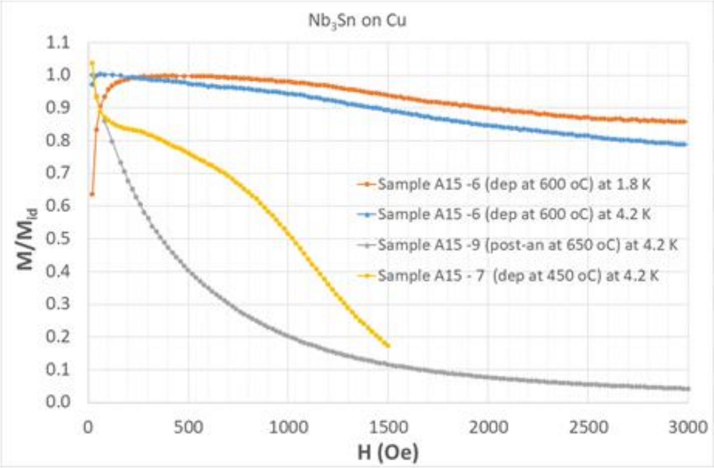
- 200 W, 489 V, 0.41 A
- 2 Hours deposition
- DC sputtering

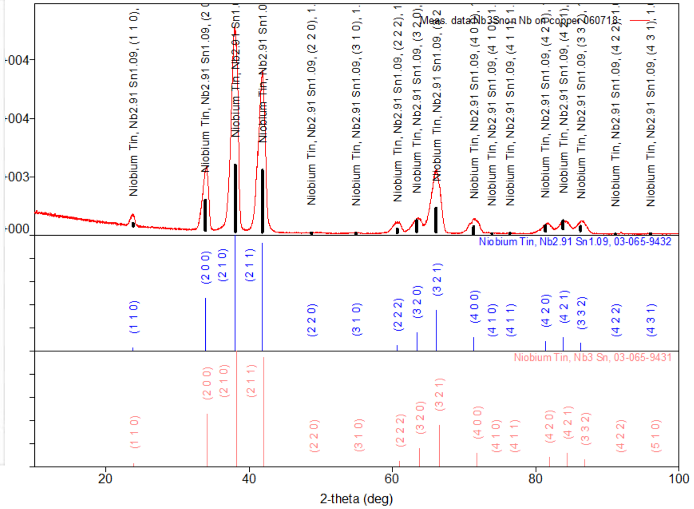




Science and Technology Facilities Council

Cu/Nb_3Sn deposition (single layer) at various Temperature

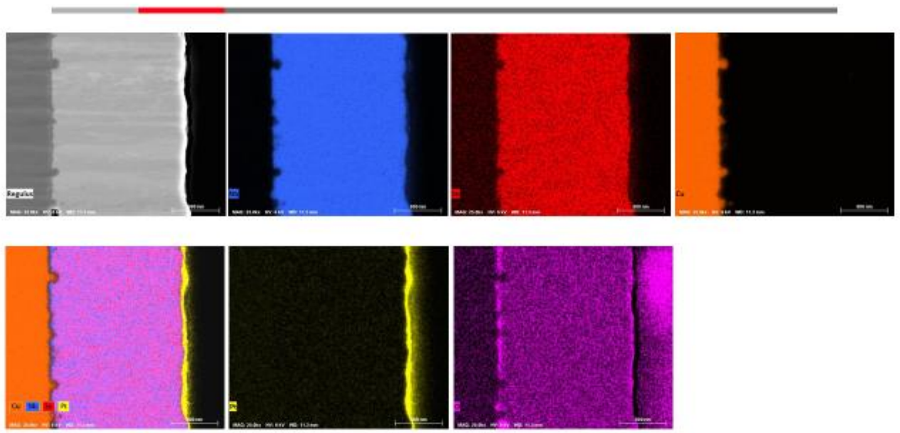


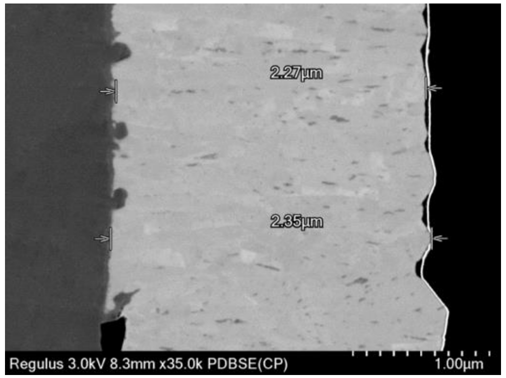
- Nb_3Sn were deposited on copper with no prior chemical cleaning such (EP or SUBU5) at various Temperature of RT, 450°C and 600°C. The RT deposition showed no sign of superconductivity.
- Best performance in terms of superconducting properties is achieved by the film deposited at 600°C (A15-6) with a Tc of 15.7 K, a lattice parameters of 0.529 nm and grain size in order of 8 to10 nm.
- The film deposited at moderate temperature of 450°C has its performance much reduced with Tc 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/Mi drop sharply at very low field of about 10 mT.

Science and Technology Facilities Council

Cu (EP)LNL / Nb_3Sn (single layer) deposited at 650C

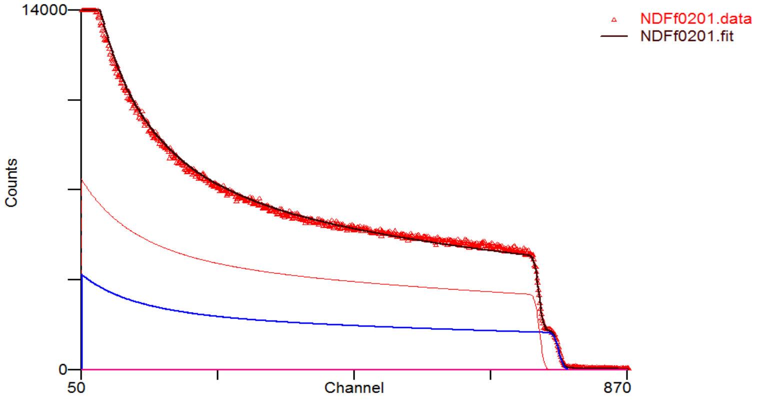
- There is some diffusion of copper at the interface
- There is a clear oxide layer at the interface despite high temperature treatment prior deposition
- There are area that seems to be Sn deficient where there are dark contrast spots.
- The Tc was determined to be between 17.75K (on sapphire) and 17.5 on copper
- First Ben for 50mT and 140mT deposited on Cu and Sapphire, and H_{C2} above 16 T.
- An interesting results which gives the opportunity of direct deposition of copper cavity rather than Niobium cavity.

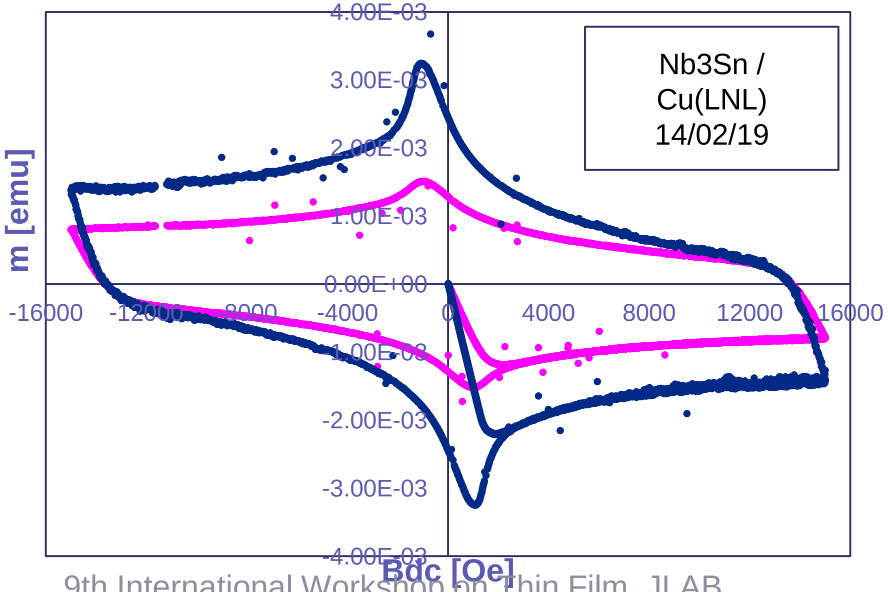


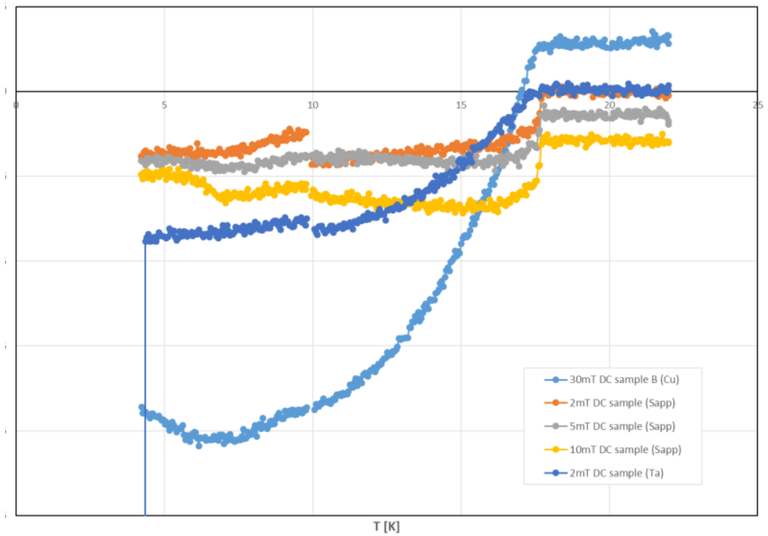


Film thickness of 2.5 Microns

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	(



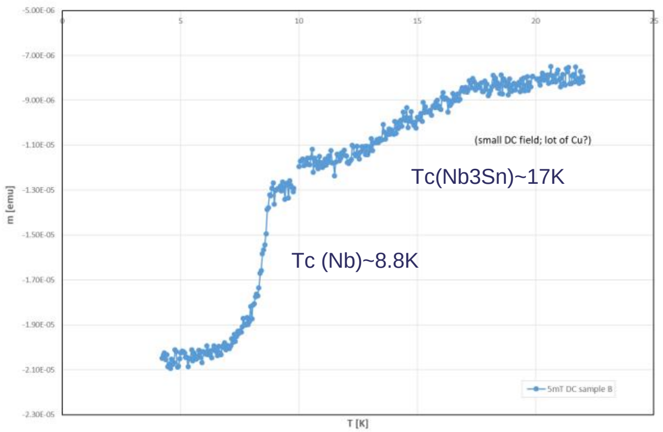


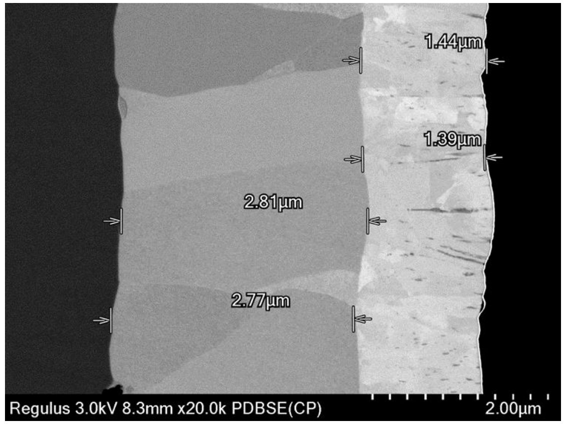


Science and Technology Facilities Council

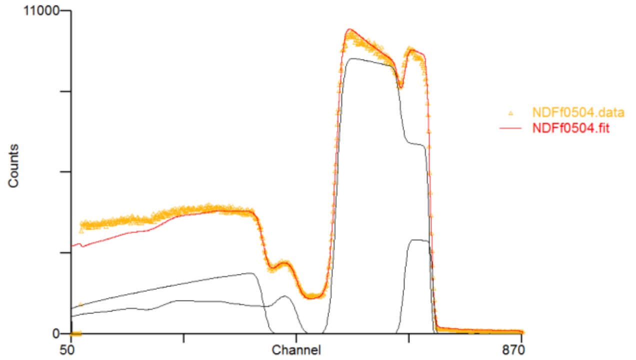
Cu /Nb/Nb_3Sn (double layer)

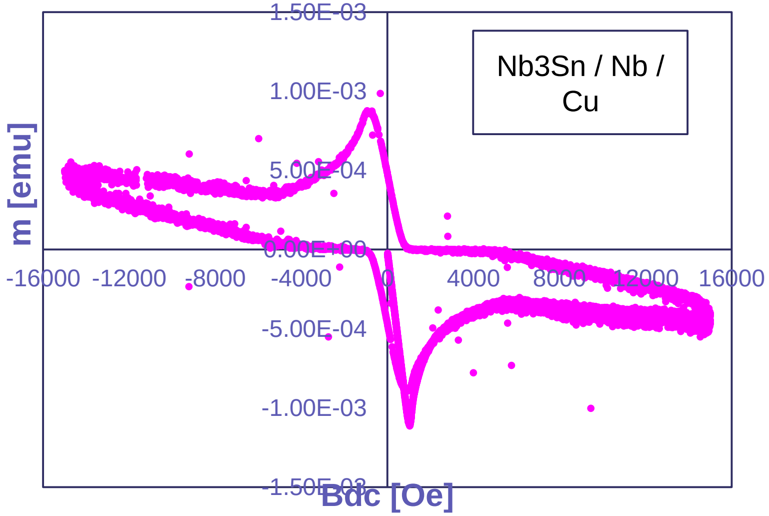
- The interfaces both at Cu/Nb and Nb/Nb3Sn is well define
- Nb layer is grown in large grain and in a perpendicular direction to the substrate surface
- No intermixing of elements is observed
- Some area of Sn deficiency and rich Sn in Nb3Sn layer can be observed
- First B_{en} is estimated at 95mT.

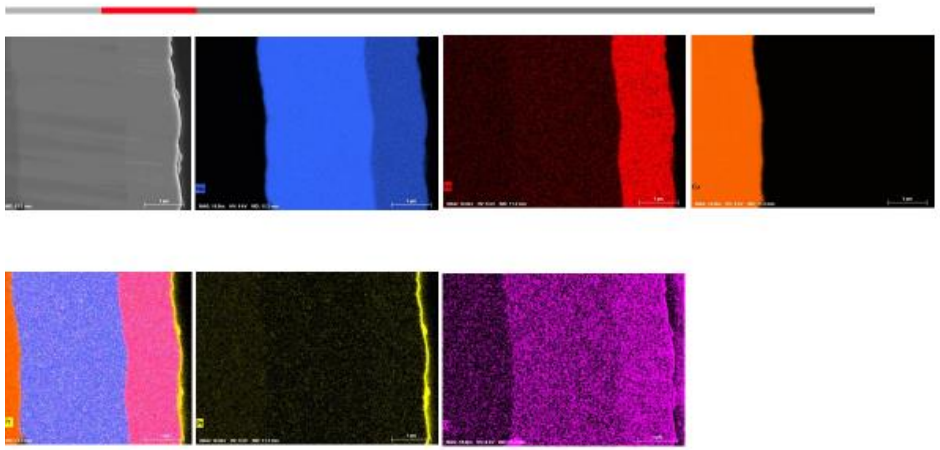




Layer	t (1e15at/cm2)	t (nm)	r(1e22at/cm3)	Nb	Sn	O	Al
1	7644.333	1497.201	5.106	75.0000	25.0000	0.0000	0.0000
2	15920.986	2855.270	5.576	100.0000	0.0000	0.0000	0.0000
3	898207.563	76119.281	11.800	0.0000	0.0000	60.0000	40.0000

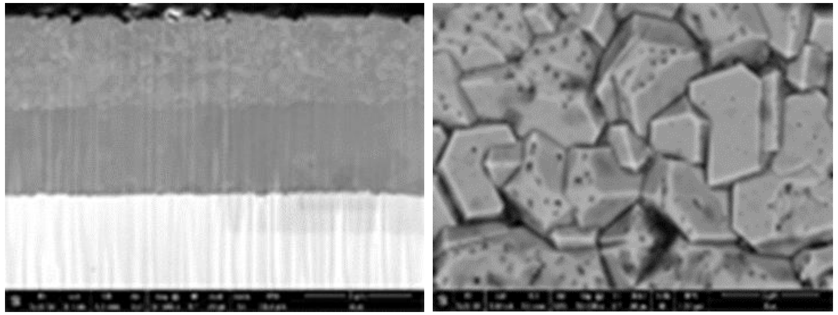


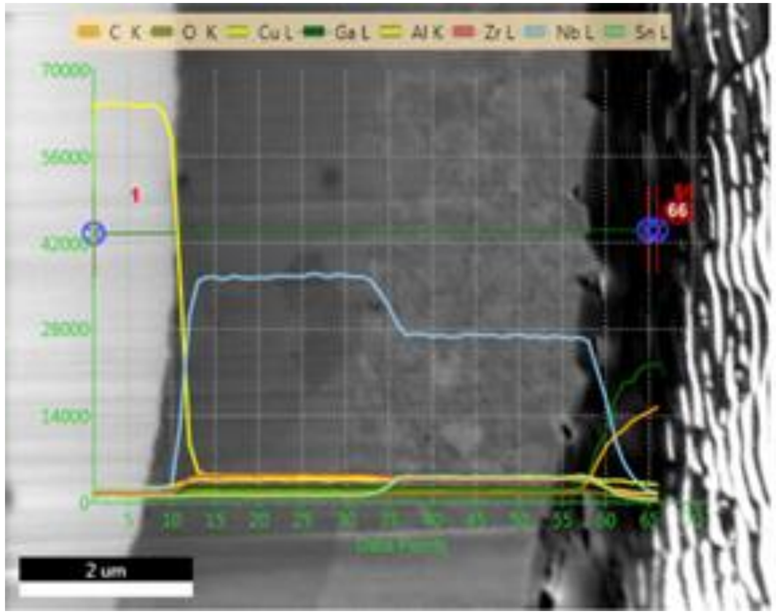


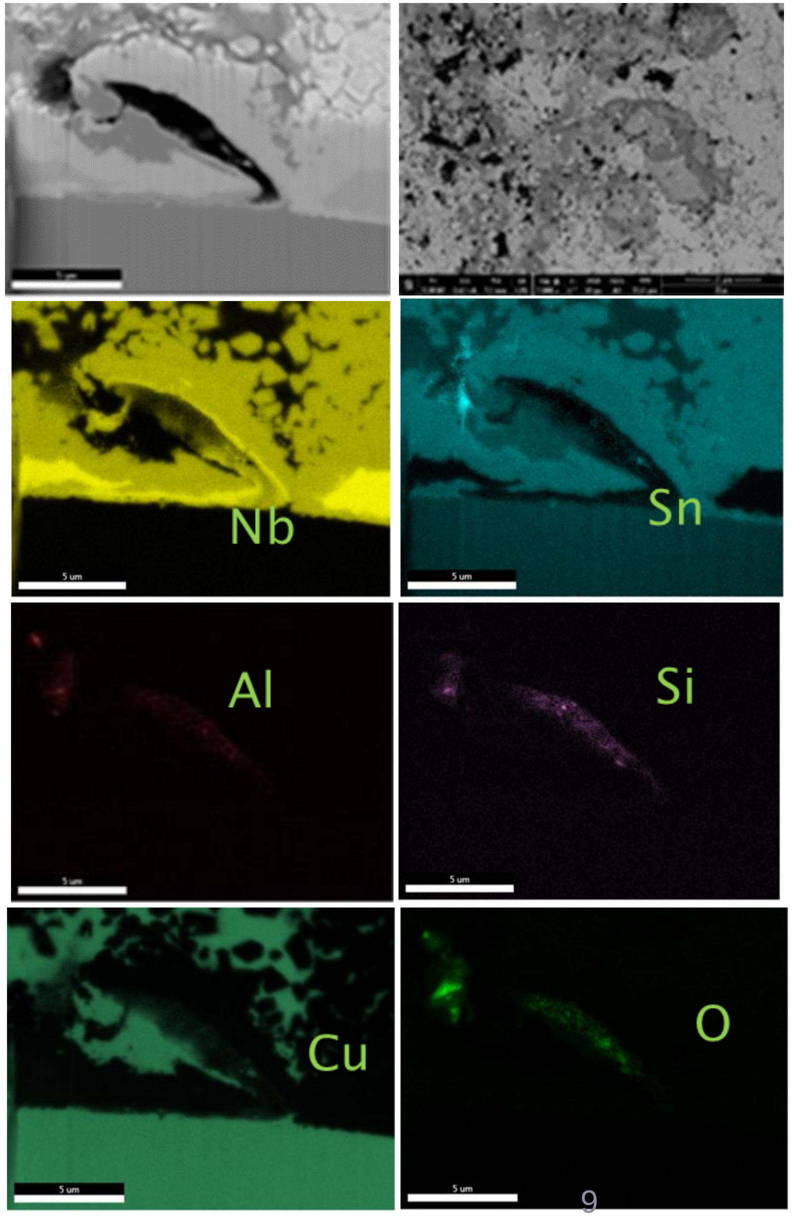


Cu/Nb/Nb_3Sn (double layer)

- 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 Nb_3Sn 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.





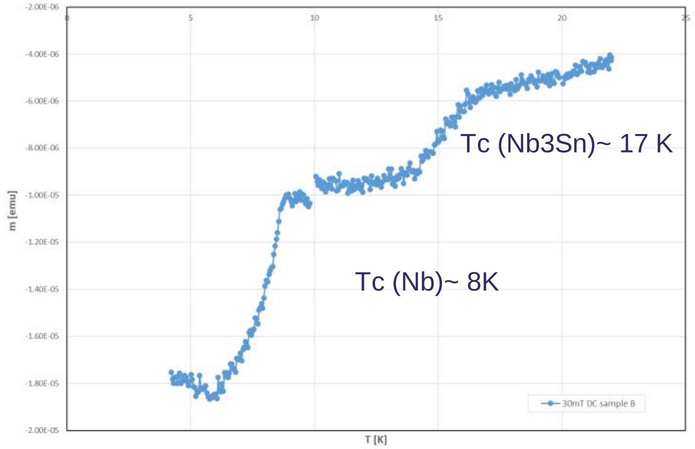


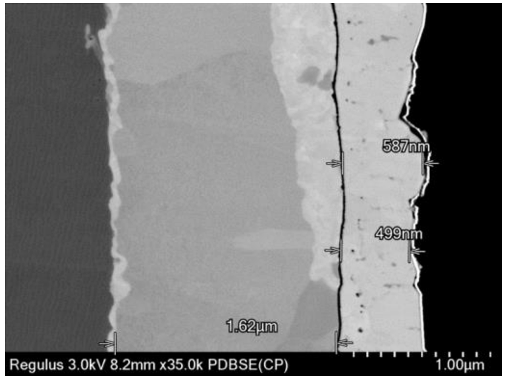
Science and Technology Facilities Council

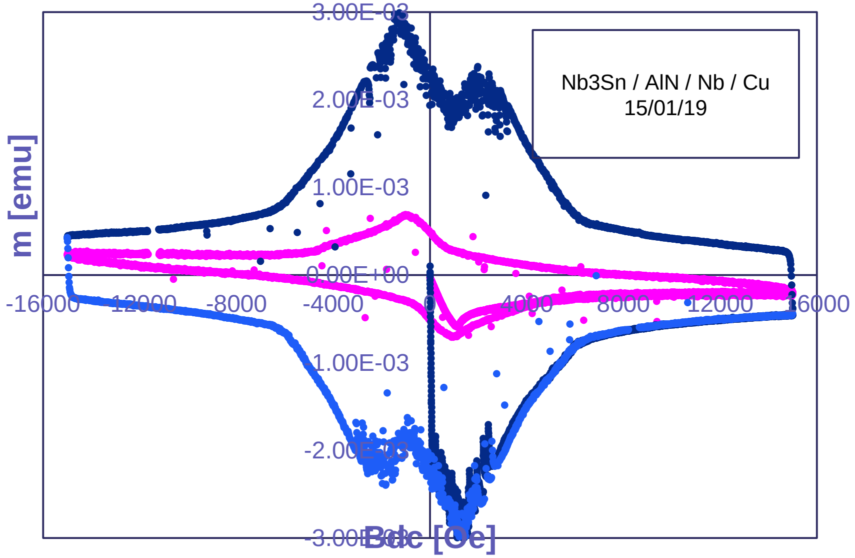
SIS Structure of thick Nb_3Sn/AlN/Nb multilayer on copper

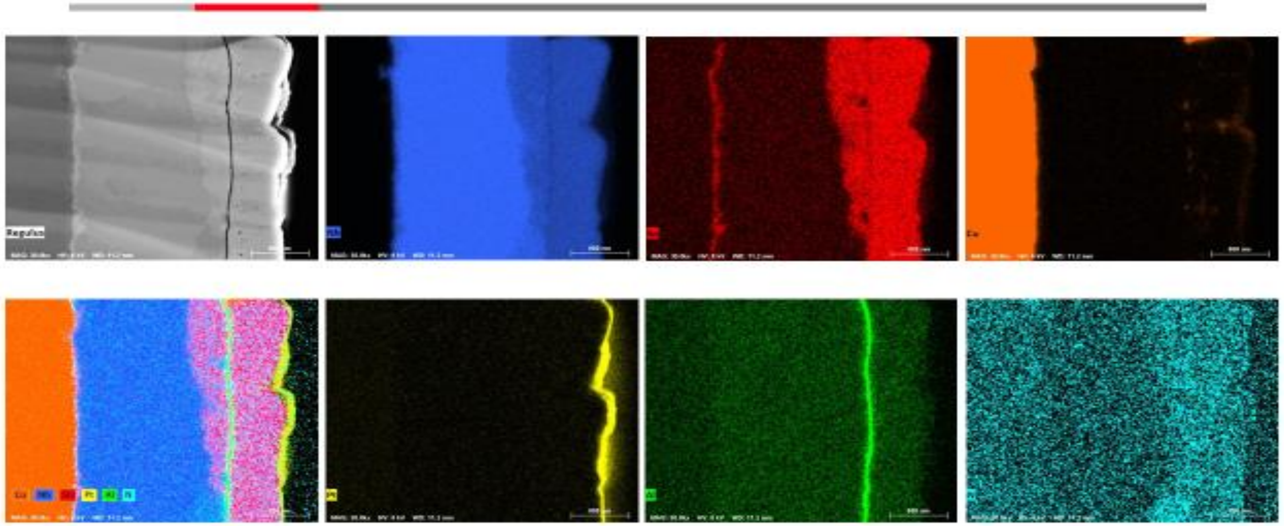
- Although the layers are well identified however there is again some degree of mixing can be observed.

1. Sn segregation at Cu/Nb interface
2. Nb_3Sn into Nb layer
3. Copper diffusion on to the surface
4. Some level of Nitrogen diffusion into all the layers.
5. The Ben in parallel external field is estimated to be 61mT and 4mt for Cu and Sapphire Substrate

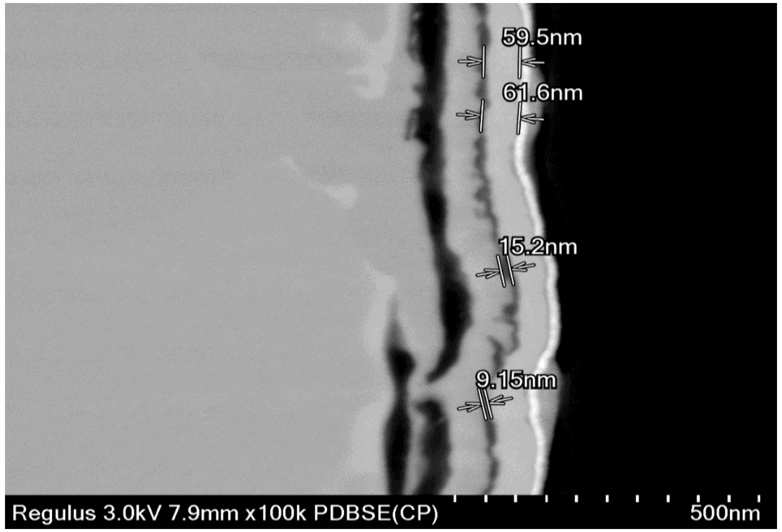


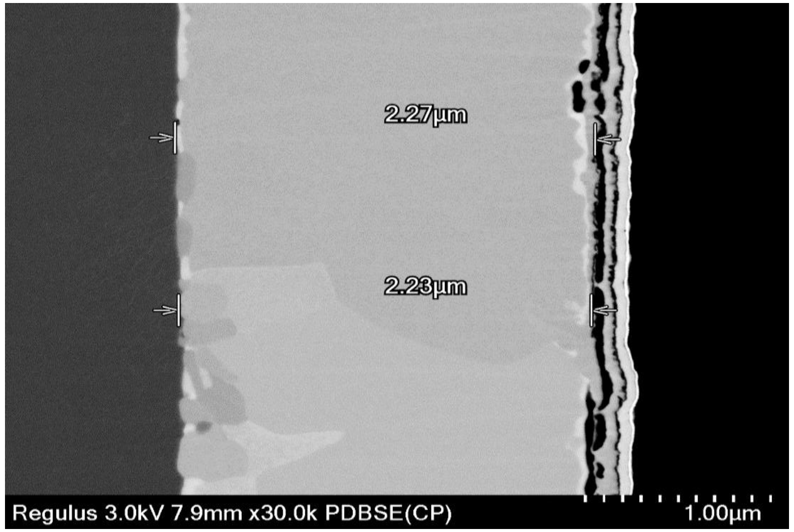


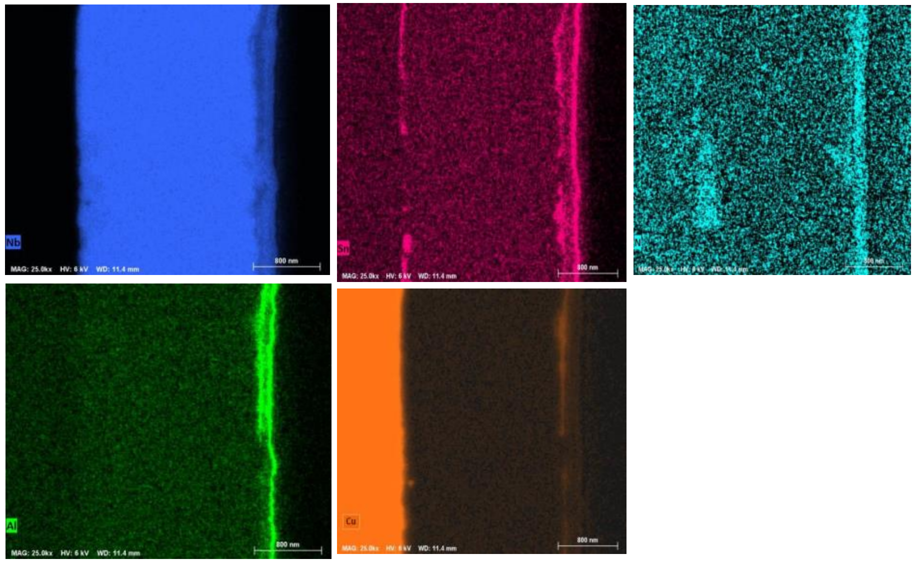




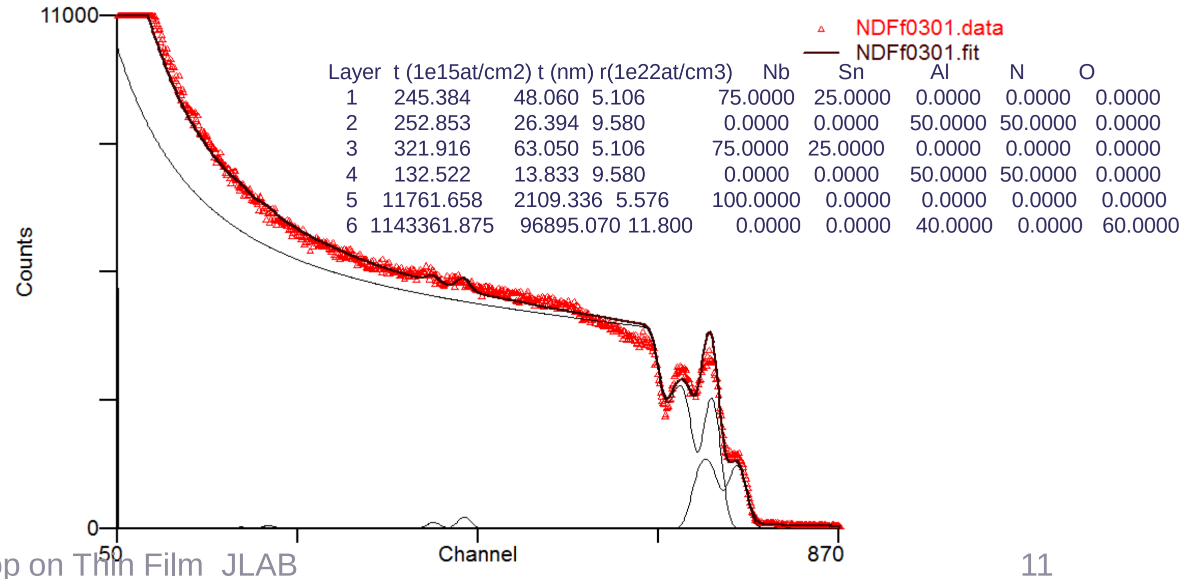
SIS Structure of thin layer $(Nb_3Sn/AlN)_2/Nb$ multilayer on Cu





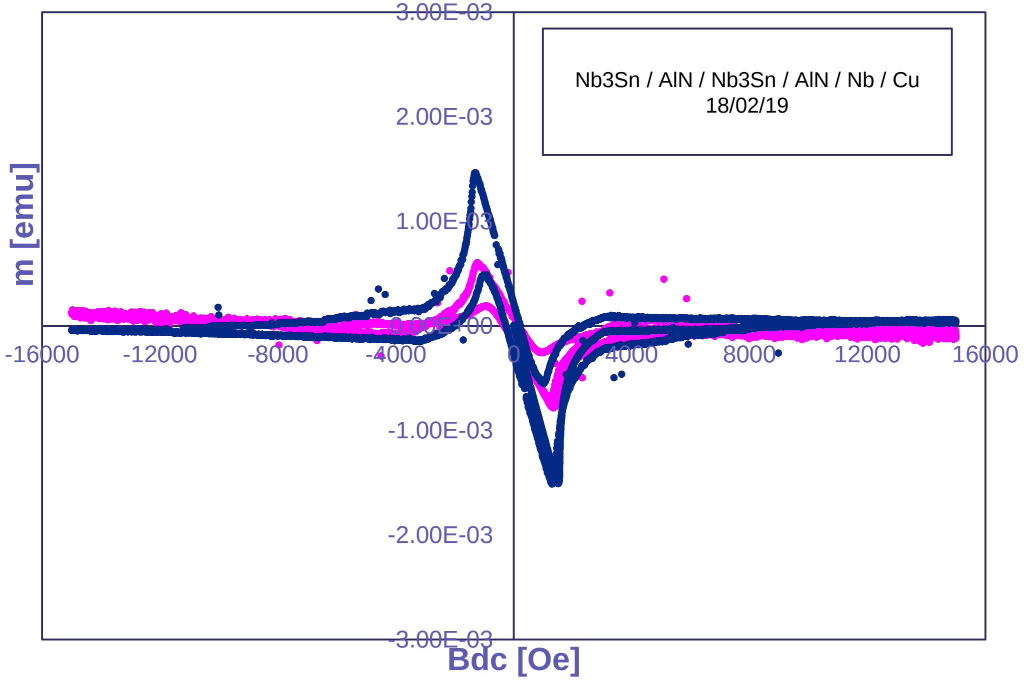


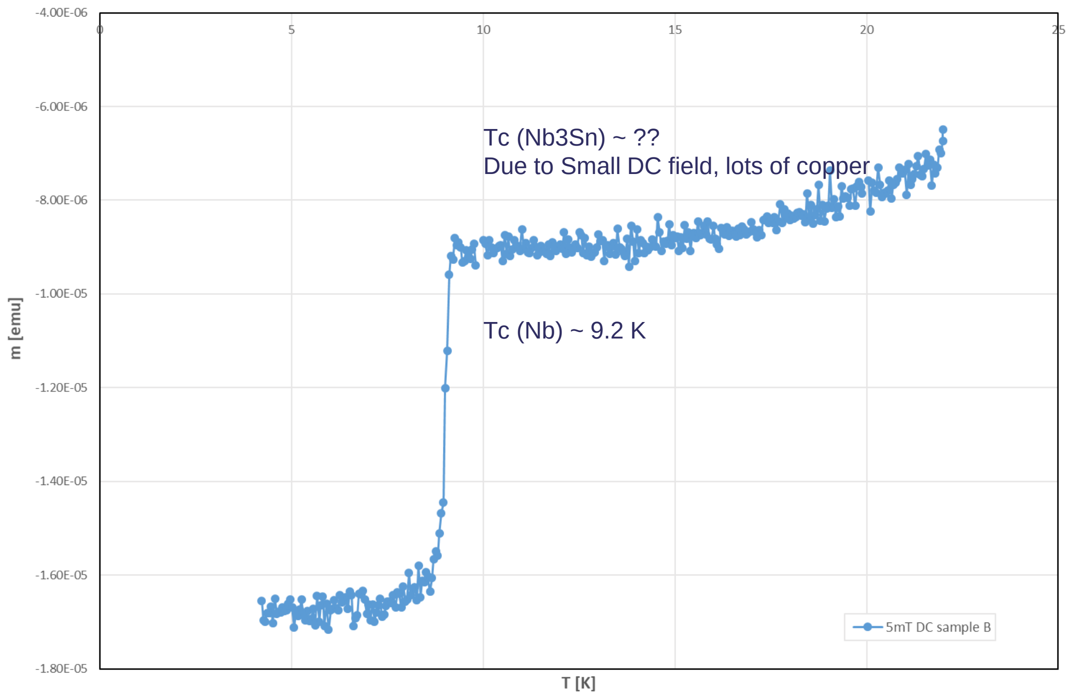
- The observed distortion of the Nb_3Sn / AlN bilayer is most probably due to mechanical polishing prior to ion beam milling, since such distortion is not present in the RBS spectra
- The RBS analysis predicts stoichiometric layers with the second AlN layer almost being double the thickness of the first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



Science and Technology Facilities Council

SIS Structure of thin layer $(Nb_3Sn/AlN)_2/Nb$ multilayer on Cu



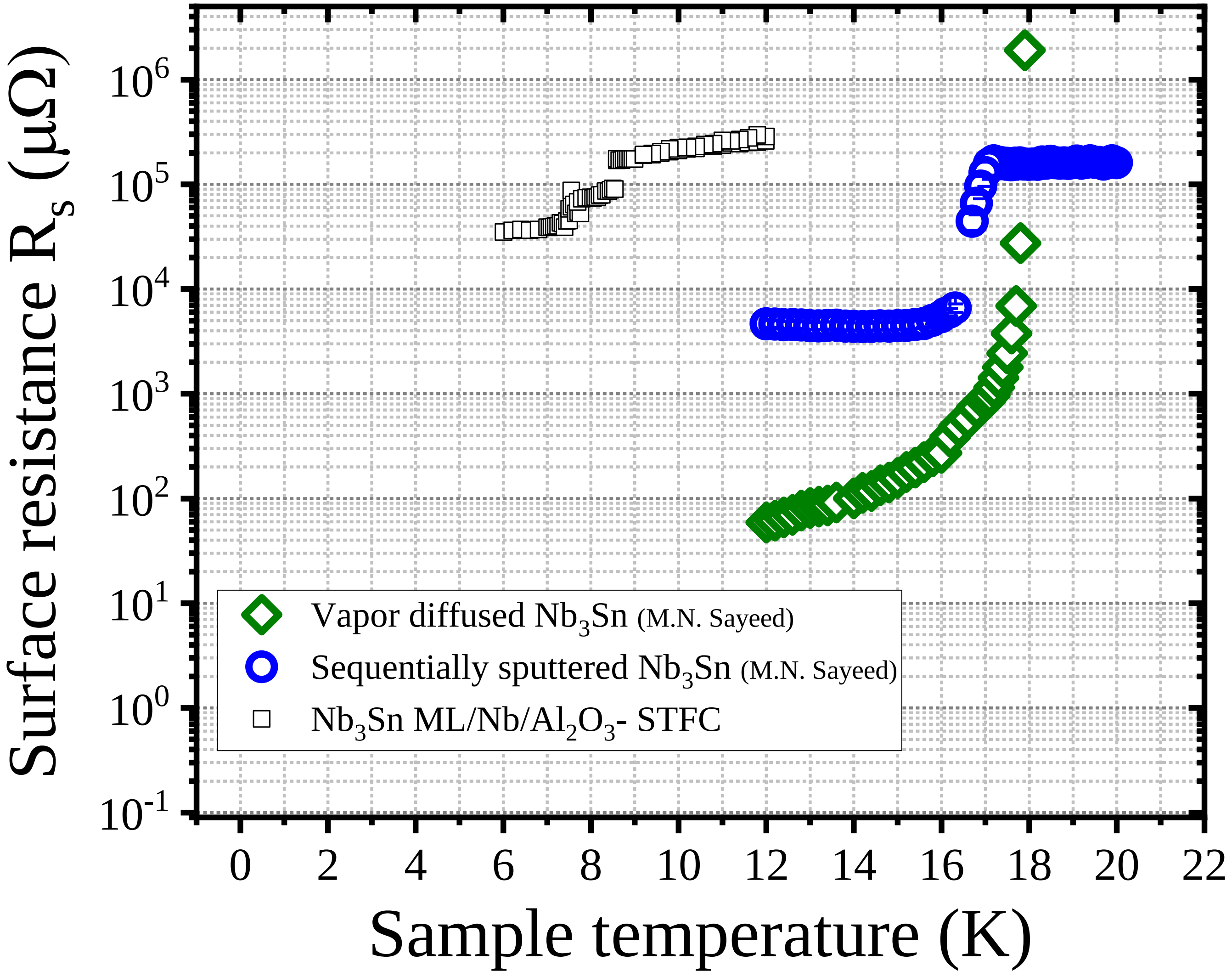


- ❑ The Ben in parallel external field is estimated to be 108 mT and 130 mT for Cu and Sapphire Substrate
- ❑ There is nearly no hysteresis in the thin double SIS structure
- ❑ This can be due to the protective effect of multilayers which reduces the sensitivity to pinning effect.

Science and Technology Facilities Council

Surface Resistance as function of Temperature

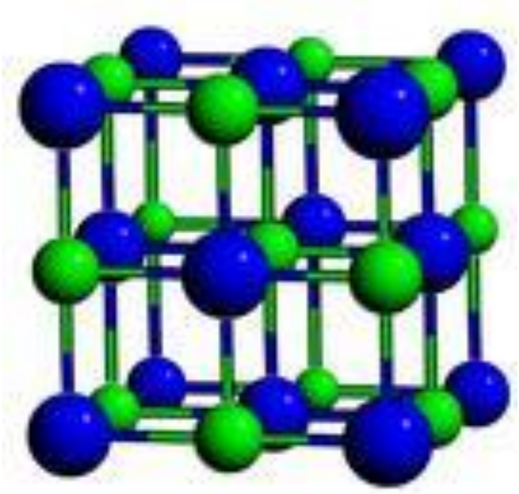
Multilayer Nb3Sn in SIS structure measured in SIC Cavity at JLAB courtesy Dr Sayeed.
The Tc of Nb3Sn is cannot be measured due to its thin layer or low field
The comparison between the ML layer structure and Self supporting Nb3Sn synthesised with different method at JLAB shows an increase of surface resistance by up to a factor of 10 which at 6 K is about 3.5 mΩ.



B1 compounds -NbTiN

Material	T_c (K)	ρ_n(μΩcm)	$H_c(0)$ [T]	$H_{c1}(0)$ [T]	$H_{c2}(0)$ [T]	λ(0) [nm]	Δ [meV]	ξ [nm]
NbTiN	17.3	35	-	0.03	15	150-200	2.8	5

- NaCl structure
- Ti and Nb form fcc lattice and N occupy all the octahedral interstices
- High Hardness,
- Not as brittle as Nb_3SN
- Good adhesion,
- Very good corrosion and erosion resistance
- Reasonably good conductor
- Moderately easy to synthesis
- High sublimation temperature
- Tc depends both on stoichiometry and deposition temperature



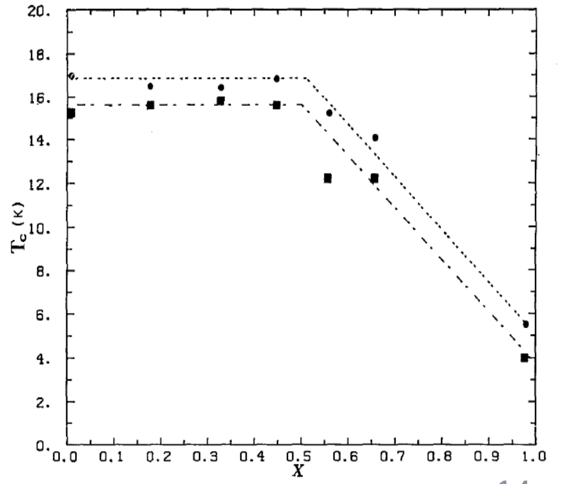
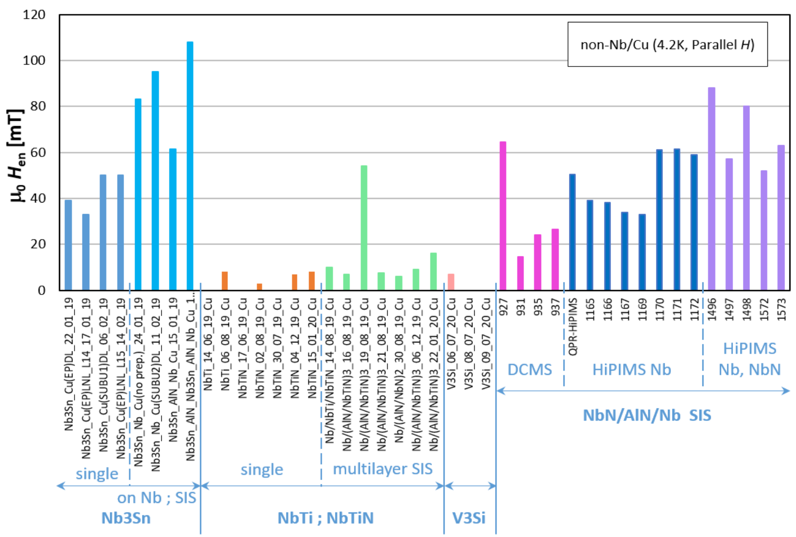
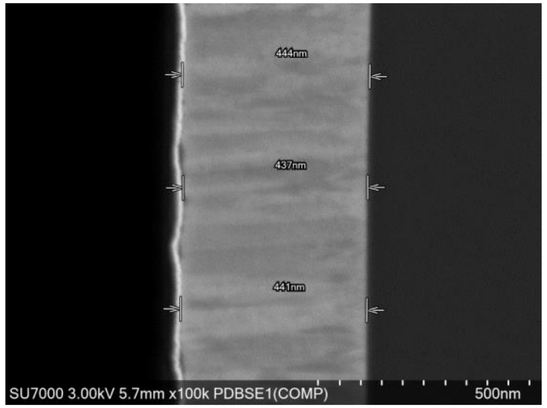


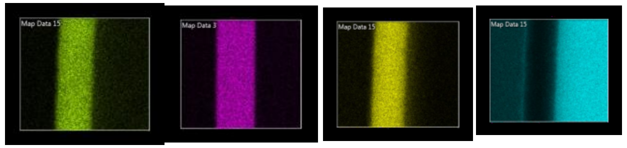
Fig. 1. Superconducting critical temperature T_c as a function of the titanium composition (x) for the ($Nb_{1-x}Ti_x$)N films deposited at T_s = 600°C (circles) and at T_s = 200°C (squares).

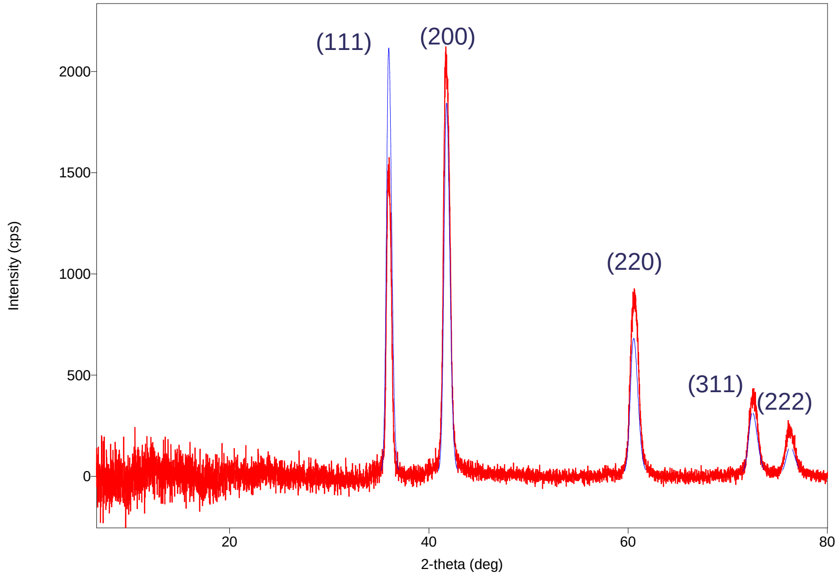
NbTi and NbTiN Single layer

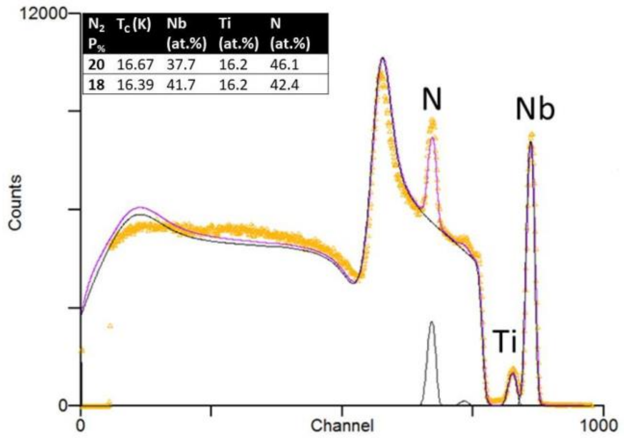
- For both system the superconducting phase only materialises at high Temperature.
- The higher the temperature the higher Tc ranging from 14 K to 17 K.
- The best Tc and hysteresis was found to be on Cu substrate.
- Can be synthesised quite easily with both alloy target and concurrent single elemental target.
- Both RBS and EDS showed good compositional uniformity for all the film.
- The XRD matched well with the published data with lattice parameter of 4.326 A and grain size of 28.5 nm.











Multilayer SIS deposition of B1 SC

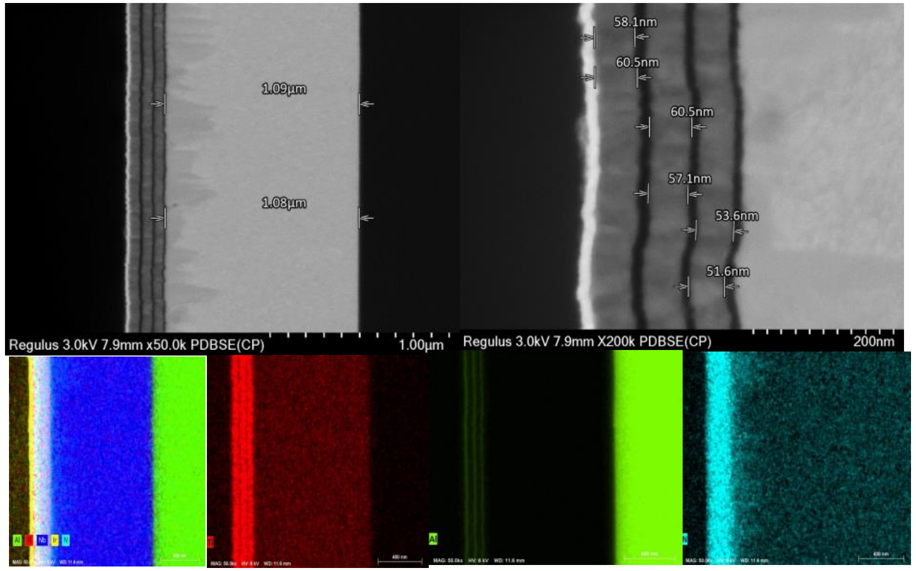
- Substrate heated 20 h prior deposition deposition temperature 650C
- Deposition SIS:
- Nb/AlN/NbTiN/AlN/NbTiN/AlN/ NbTiN
- 5 h or 2h (400 W DC) for Nb, 7 min (200 W Pulsed DC) for AlN (10 - 15 nm) and 10 min (300W DC) for NbTiN (30 nm)

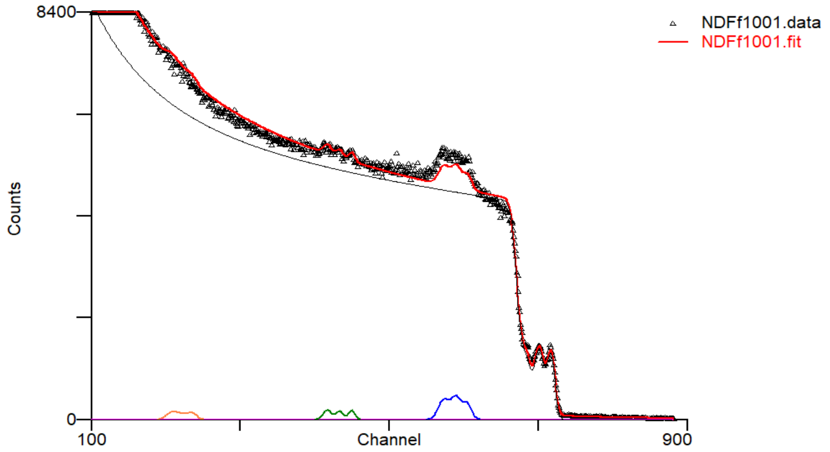
- Nb/AlN/NbN/AlN/NbN
- 2h (400 W DC) for Nb, 7 min (200 W Pulsed DC) for AlN (10 - 15 nm) and 10 min (300W DC) for NbN (35 nm)

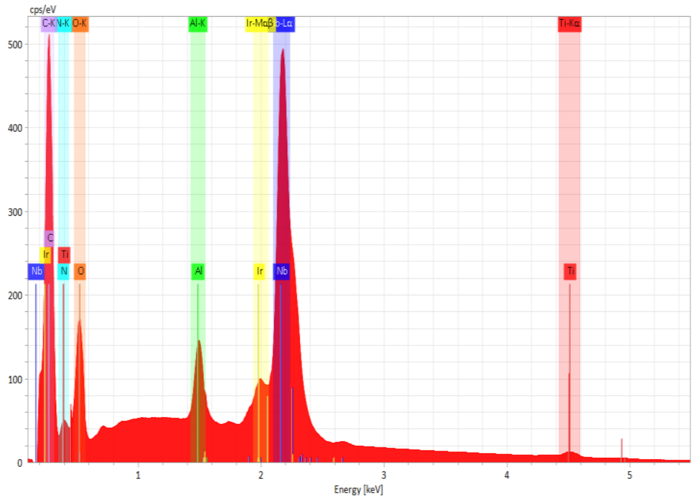
Science and Technology Facilities Council

Multilayer SIS deposition

Layer	Layer thickness (1e15at/cm²)	Layer thickness (nm)	Nb	Ti	N	Al	O
1	227.7	52.6	20.1876	19.33	60.47	0	0
2	109.3	11.4	0	0	50	50	0
3	263.95	58.7	20.8	26.05	53.1	0	0
4	105.68	11.03	0	0	50	50	0
5	285.32	67.11	15.2	20.51	64.27	0	0
6	141.06	14.7	0	0	50	50	0
7	13510.8	2423.05	100	0	0	0	0



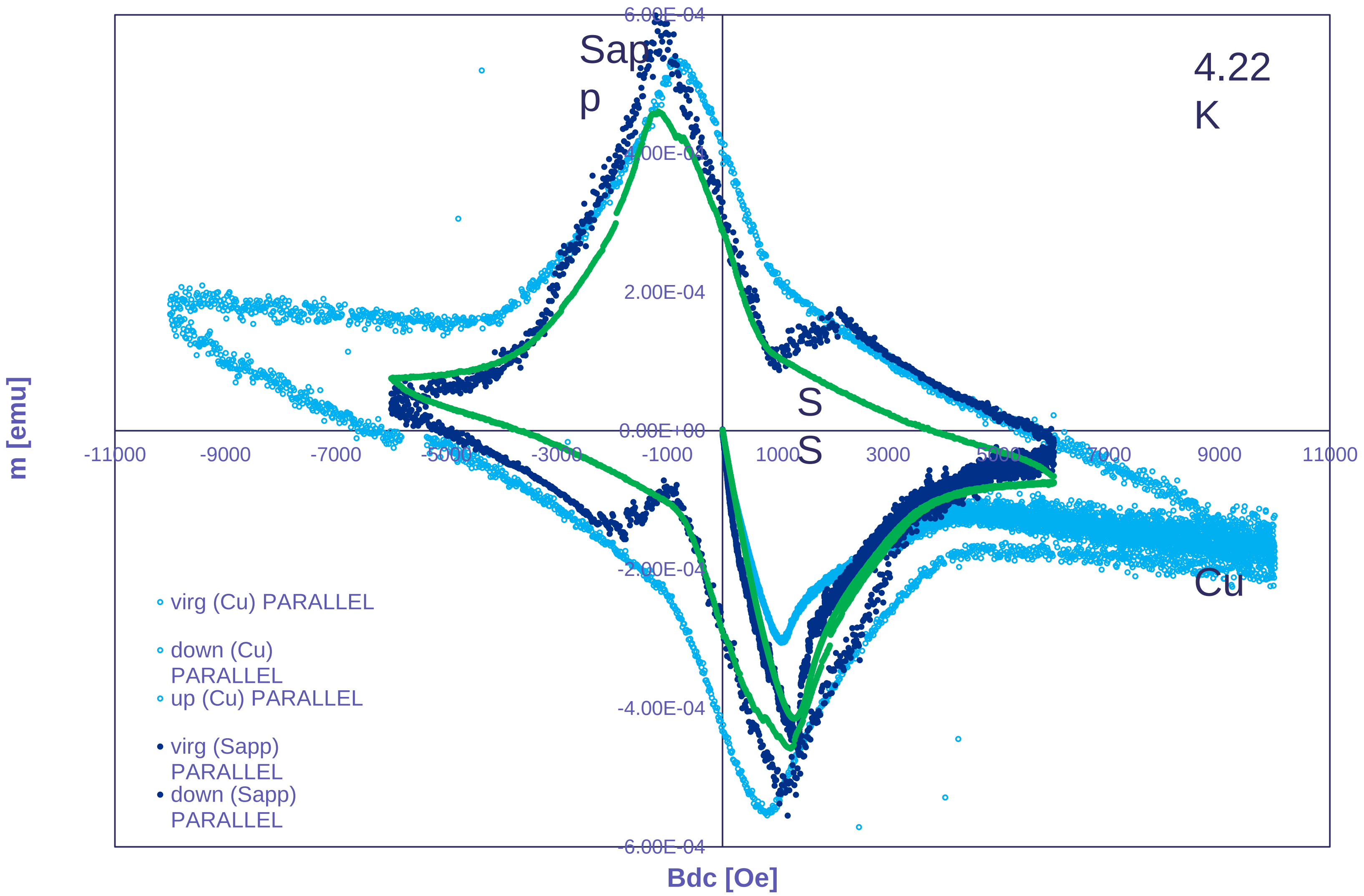




21/08/19 – Nb/[AlN/NbTiN]3

(Nb 2h, AlN 7min, NbTiN 10min)

Cu, Ta, Sapphire subst., Self-Supported film (SS)

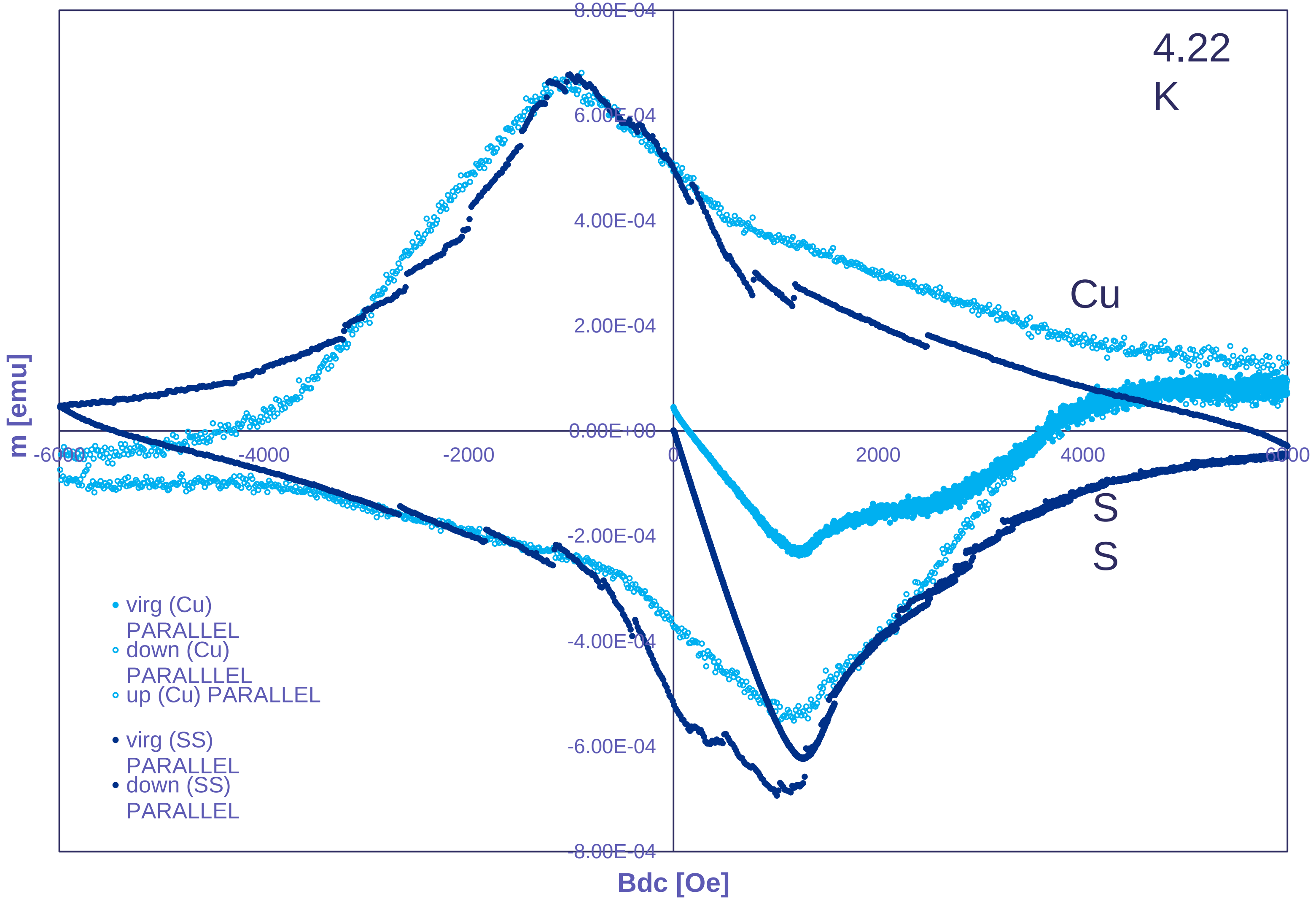


Science Technology Facilities Council

16/08/19 – Nb/[AlN/NbTiN]3

(Nb 2h, AlN 7min, NbTiN 5min
)

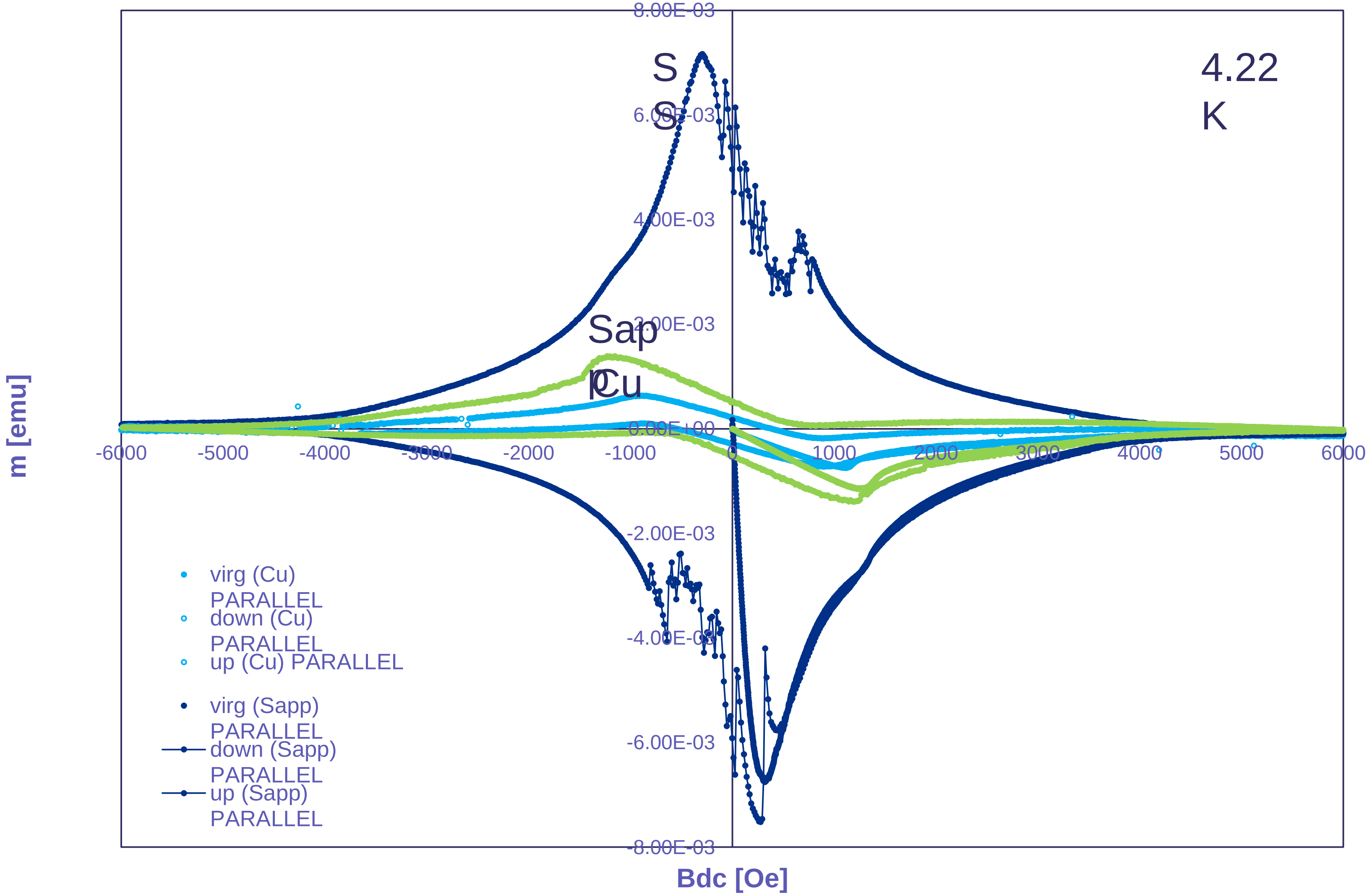
Cu, Ta, subst.



Scie
Tech
Facil

19/08/19 – Nb/[AlN/NbTiN]3 (Nb **5h**, AlN 7min, NbTiN 5min)

Cu, Ta, Sapphire subst., Self-Supported film (SS)



Science Technology Facilities

Summary

- Nb3Sn 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 Nb3Sn 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 extend is been shown :
- Reducing sensitivity to pinning defects
- Multilayer structure even without insulating layer reduced the hysteresis loop
- Defects are still present in individual layers but 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.
- NbTiN can be considered as a practical alternative material due to its ease of synthesis and reproducibility.
- It has good adhesion, wide range of SC composition window, has high hardness and good resistivity to corrosion and erosion with moderate normal surface resistance. Can be deposited at lower temperature with good SC performance.

Science and Technology Facilities Council

Special Thanks to all the team member STFC and international Partners

STFC

- Adrian Hannah
- Gavin Stenning
- Francis Lockwood Estrin
- Felix Walk
- Daniel Turner
- Yukari Dan (Hitachi)
- Karl Dawson
- Sepideh Aliasghari
- Stuart Wilde
- James Conlon
- Oleg Malyshev
- Tobias Junginger
- James Bradley
- Graeme Burt
- Vinod Dannak

IEE

- Eugen Seiler
- Rastislav Ries

University of Siegen

- Michael Vogel
- Stewart Leith

INFN

- Cristian Pira
- Eduard Chyhyrynets

CAE

- C.Z.Antoine

JLAB

- Anne Marie Valente
- M N Sayeed

HZB

- O. Kugeler
- D. Tikhonov

RTU

- A Medvids

Science and Technology Facilities Council

Thank you

Science and Technology Facilities Council

@STFC_matters

Science and Technology Facilities Council