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Progress on A15 Thin films at Daresbury Laboratory

11th International Workshop on Thin Films and New Ideas for Pushing the Limits of Superconductivity

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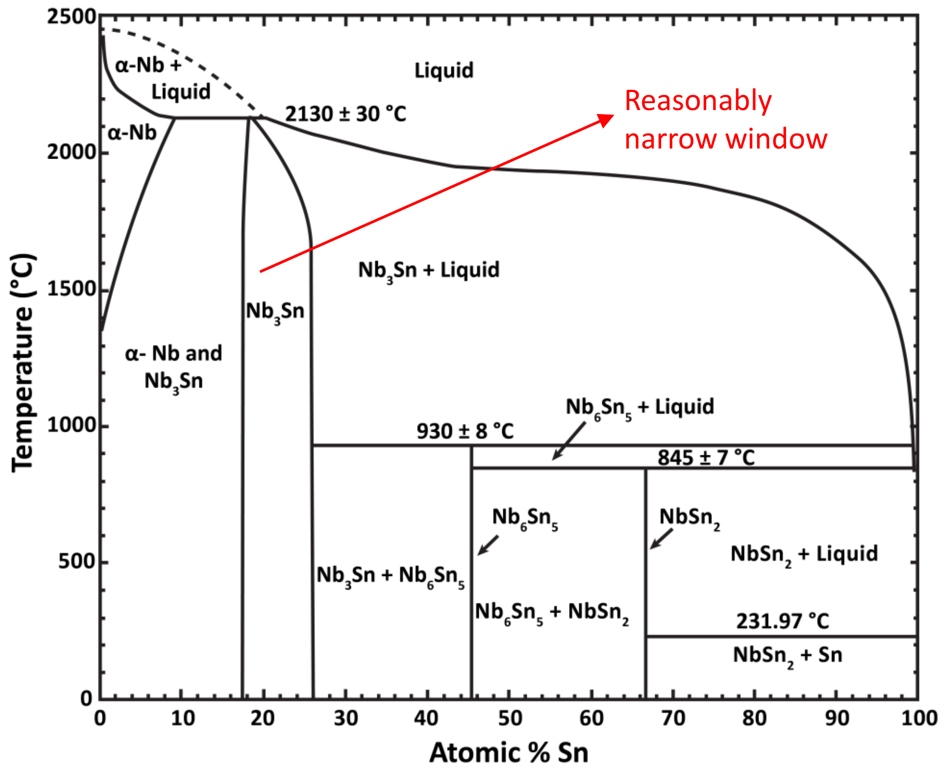
Motivation

- To optimise deposition parameters to synthesise A15 thin film with optimum SRF properties such as Tc and RF surface Resistance.
  - Optimum Target for optimum stoichiometry
  - Deposition power
  - Deposition temperature
  - Substrate dependence

Target

- Since the stoichiometry for the superconducting phase is reasonably narrow 19 > Sn < 25 %
- For this reason a high purity stoichiometric alloy target was used.
- Deposition parameters was chosen to conserve the instantaneous target stoichiometry.
  - To reduce Sn evaporation deposition power was set between 50 W to 200 W.
  - Deposition Temperature set between 550 C to 650 C.
  - To synthesis thin films with low level of impurity, the residual gas was limited to below 10^{-11} mbar, by baking the system and samples were introduced via a baked load lock.



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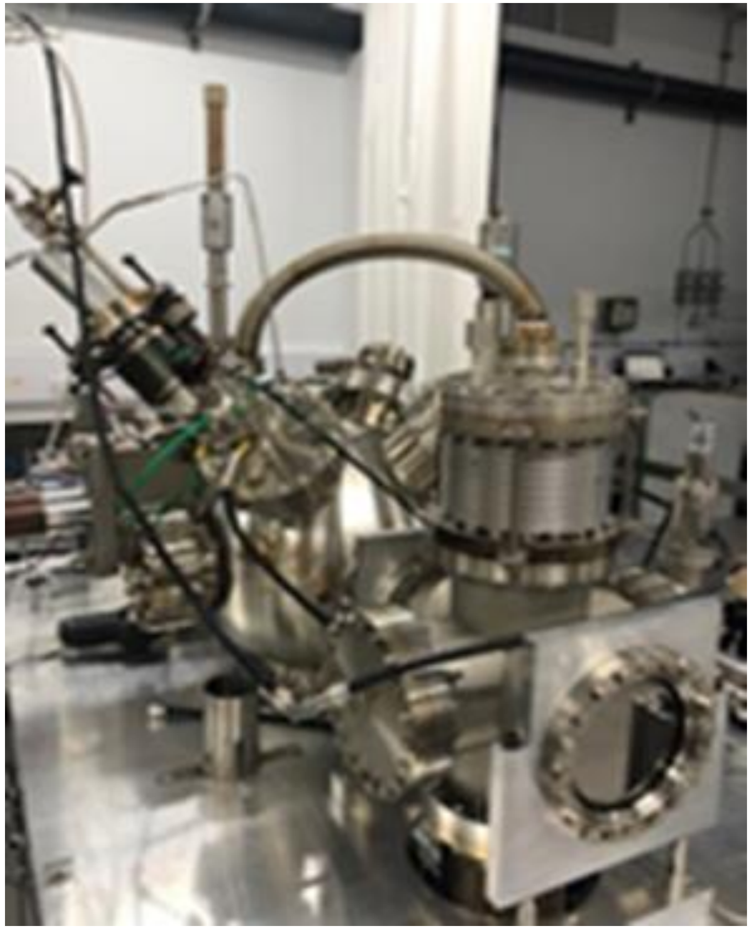
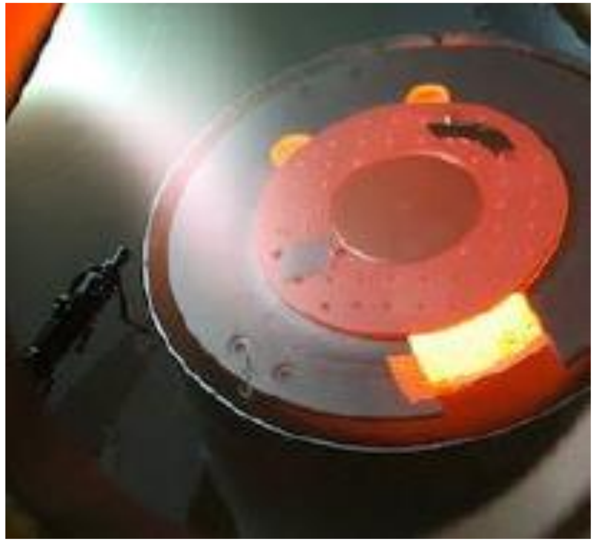
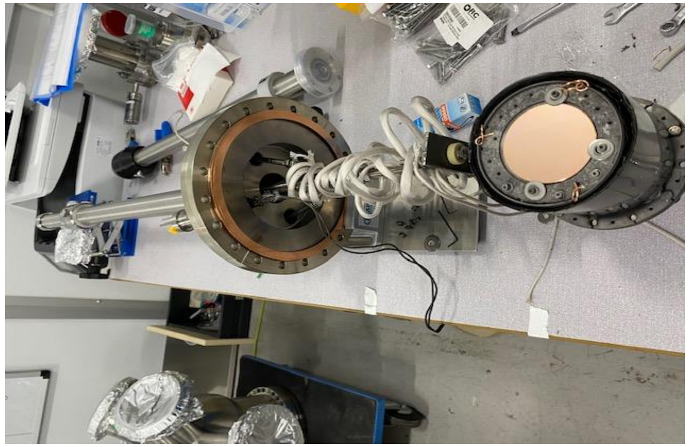
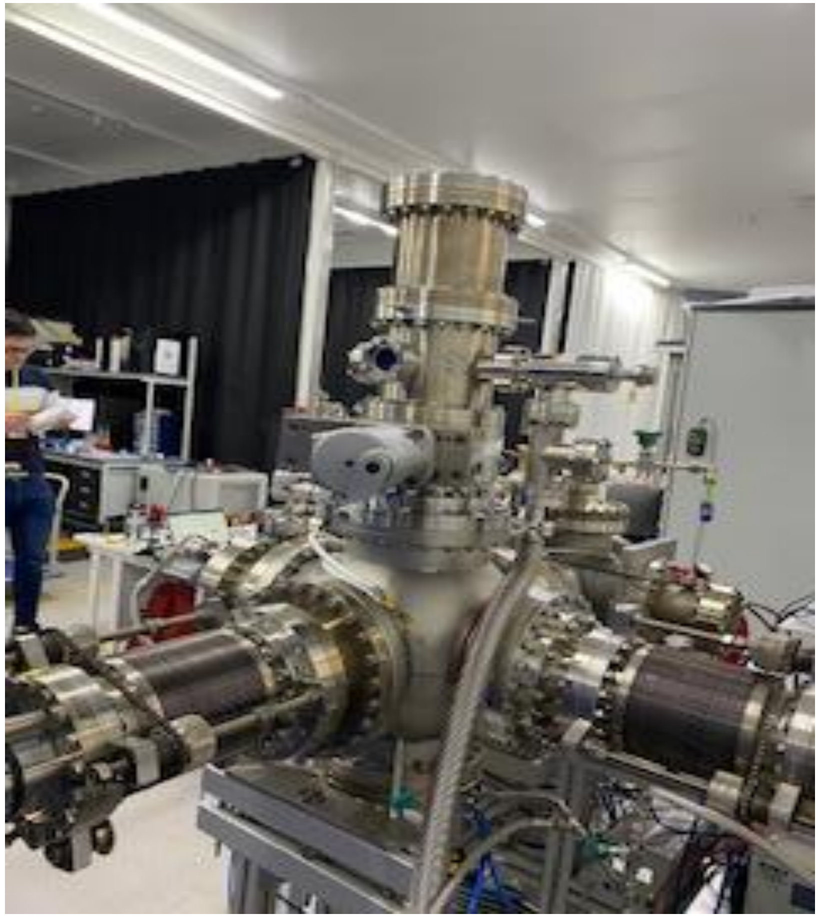
QPR & Planar deposition facility









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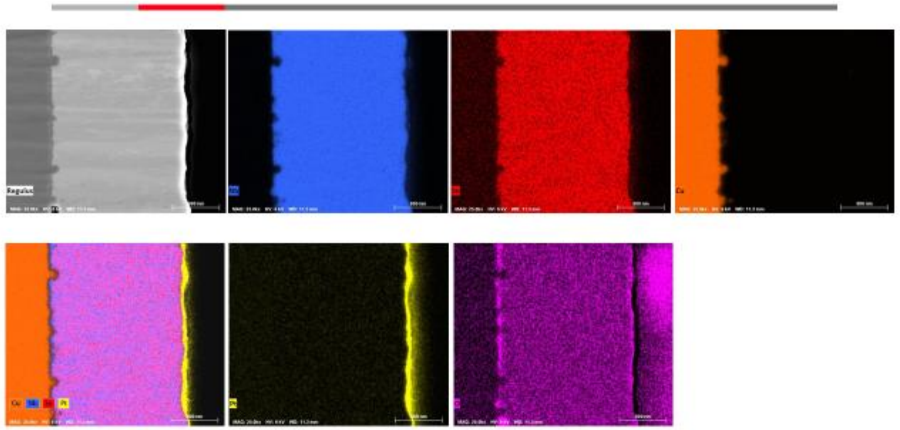
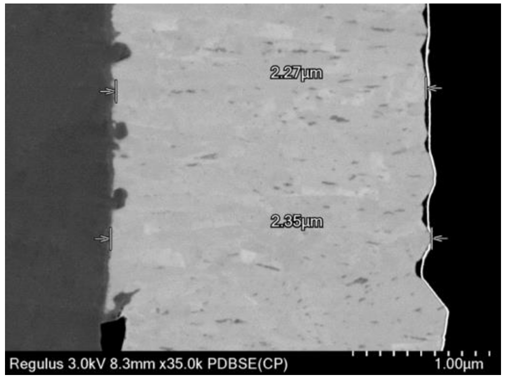
Cu (EP)LNL / Nb_3Sn (single layer) deposited at 650C and 200 W DC

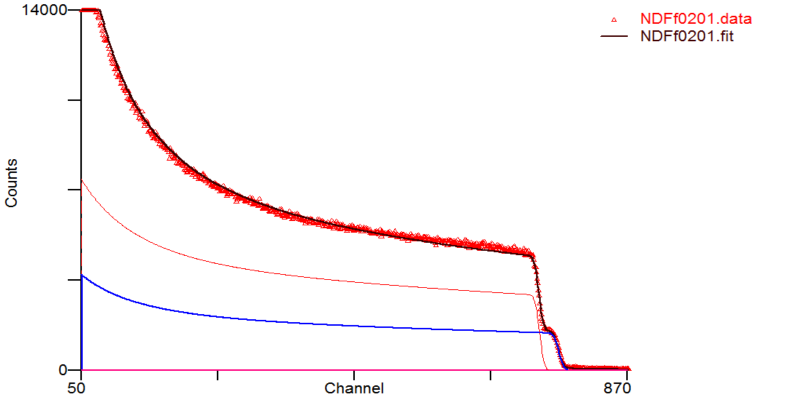
- There are area that seems to be Sn deficient where there are dark contrast spots.
- The Tc was determined to be between 17.75K (on sapphire) and 17.5 on copper
- First Ben is estimated to be 50mT and 140mT deposited on Cu and Sapphire respectively, and H_{C2} above 16 T.
- An interesting results which gives the opportunity of direct deposition of copper cavity rather than Niobium cavity.

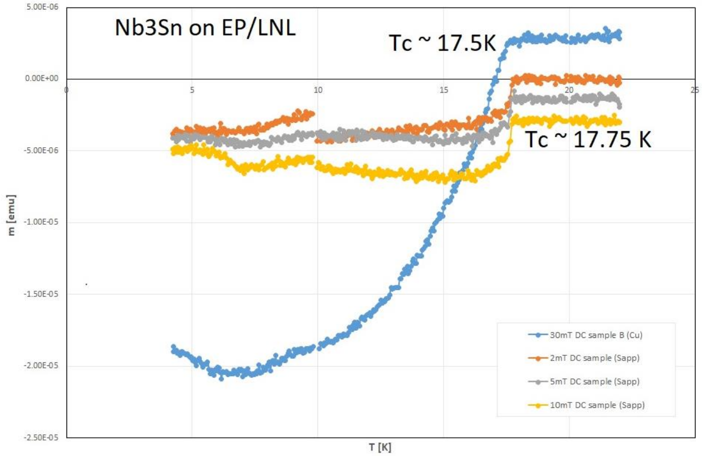


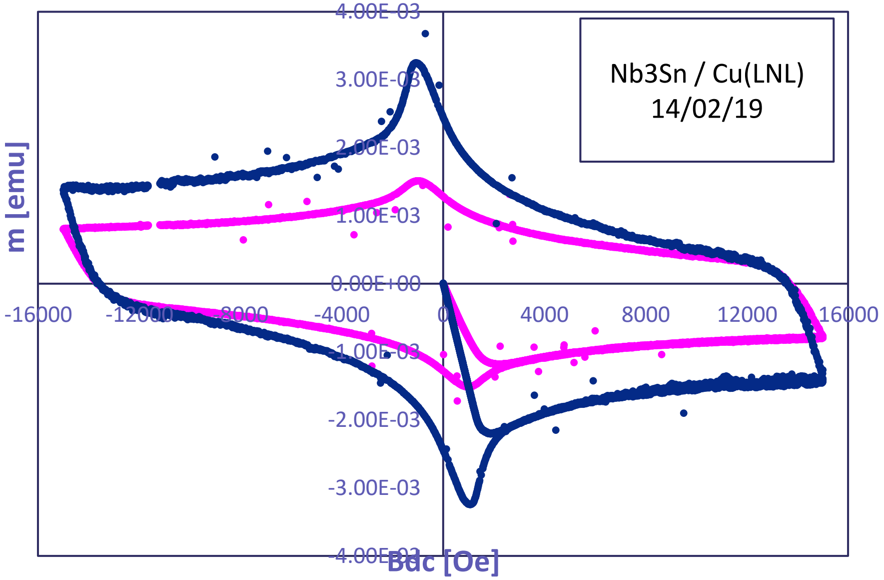


Film thickness of 2.5 Microns

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	60.0000







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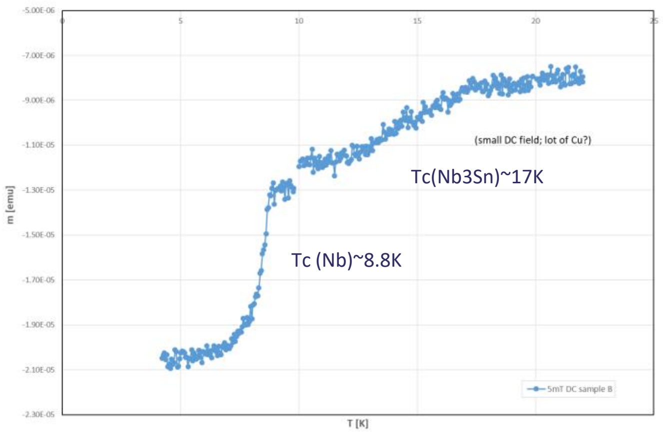
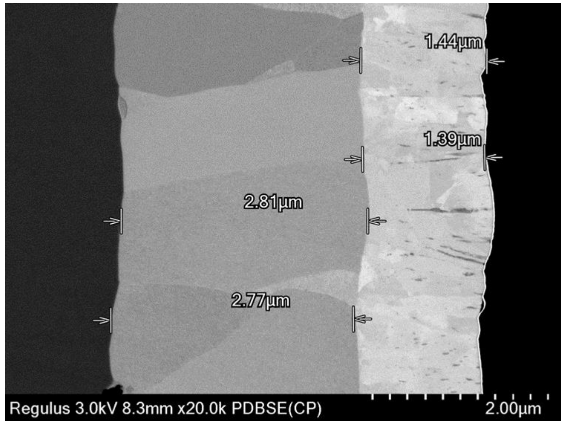
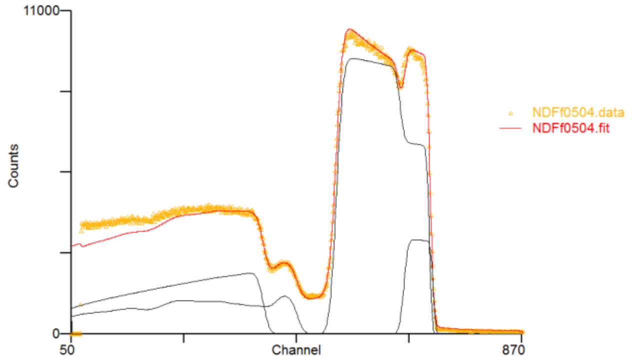
Cu /Nb/Nb_3Sn (double layer) Nb 400W and Nb3Sn 200W

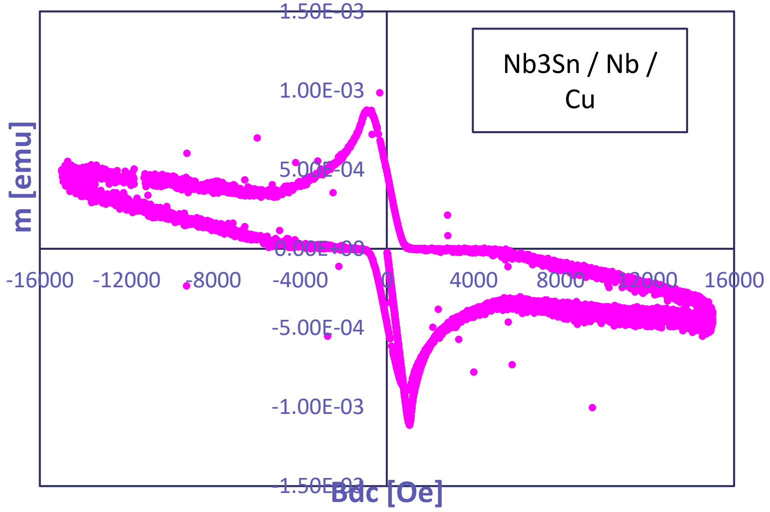
- The interfaces both at Cu/Nb and Nb/Nb3Sn is well define
- Nb layer is grown in large grain and in a perpendicular direction to the substrate surface
- No intermixing of elements is observed
- However some area of Sn deficiency and rich Sn in Nb3Sn layer can be observed
- First B_{en} is estimated to be at 95mT.
- RBS analysis shows a uniform film with good stoichiometry which matches the SEM results

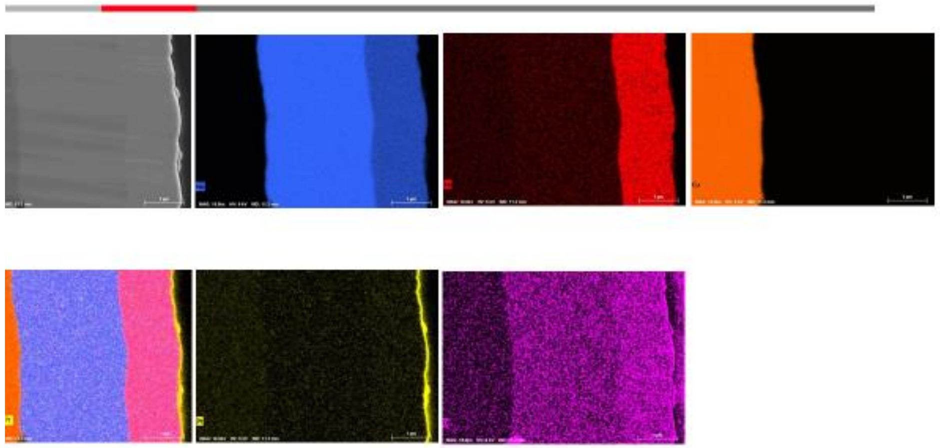




Layer	t (1e15at/cm2)	t (nm)	r(1e22at/cm3)	Nb	Sn	O	Al
1	7644.333	1497.201	5.106	75.0000	25.0000	0.0000	0.0000
2	15920.986	2855.270	5.576	100.0000	0.0000	0.0000	0.0000
3	898207.563	76119.281	11.800	0.0000	0.0000	60.0000	40.0000







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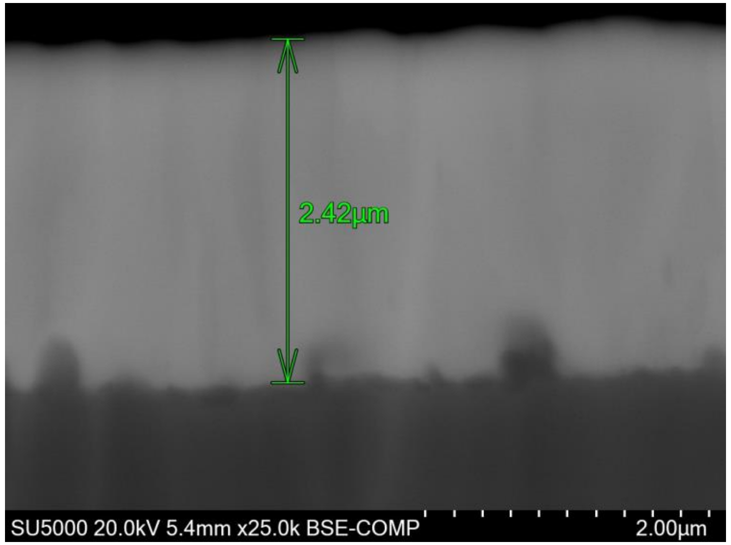
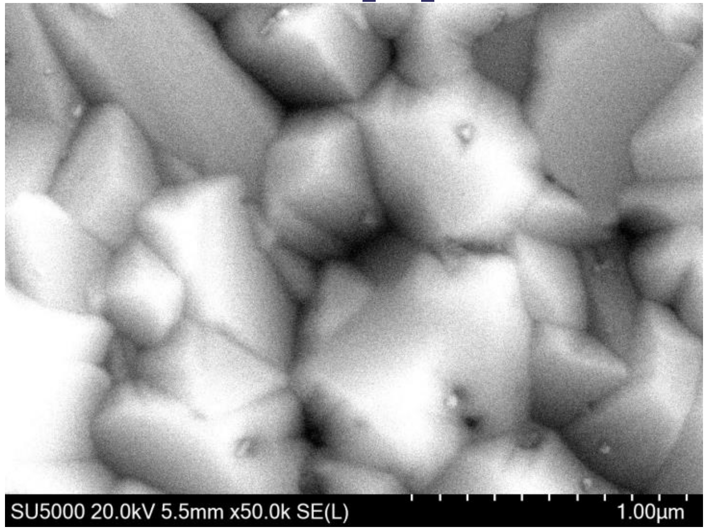
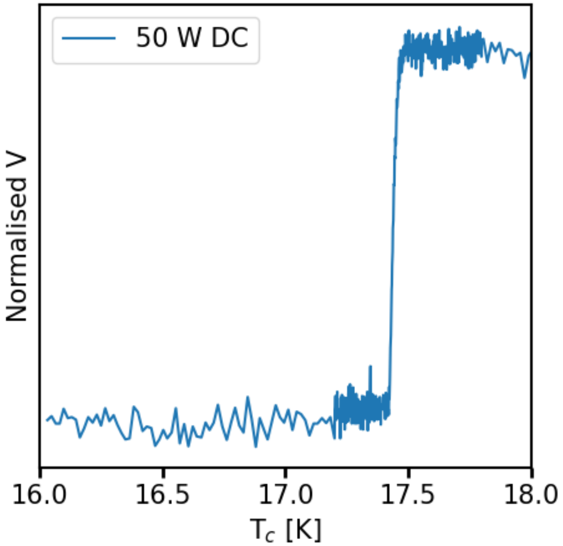
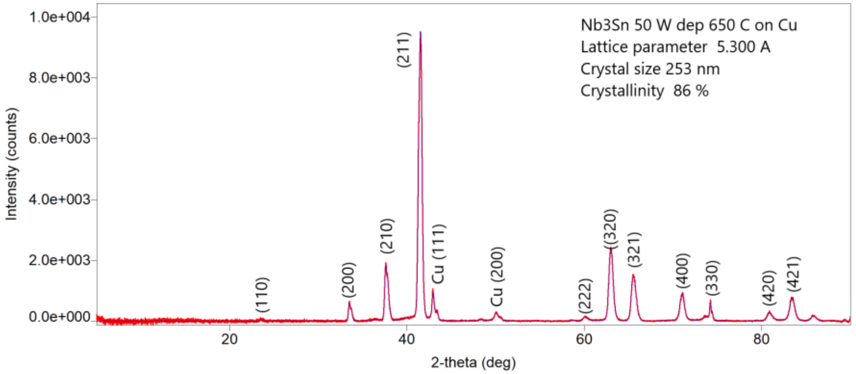
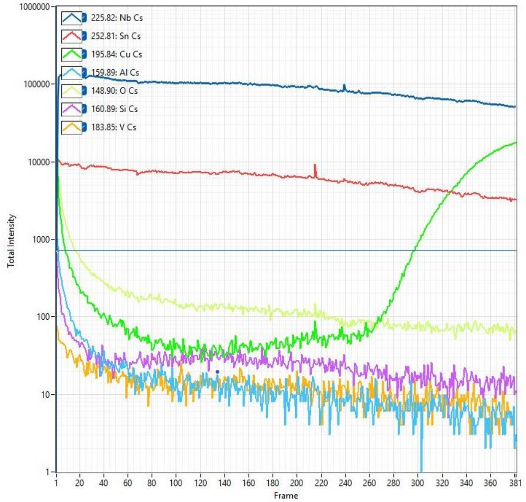
50 W DC Nb3Sn on 50 & 100 mm diamond turned copper











Cu_24_07_23 50 W Tc = 17
Cu_24_07_23_recentre Tc = 16.1

Sapp_24_07_23 50 W Tc = 17.5
Sapp_24_07_23_recentre Tc = 17.6

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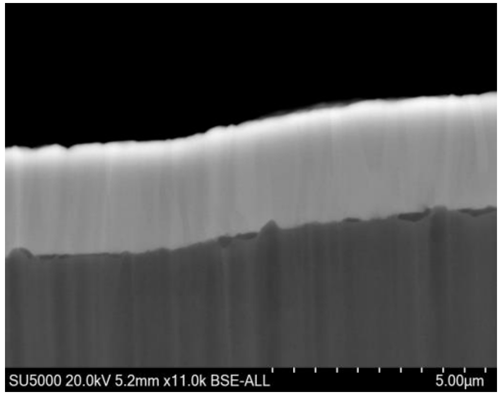
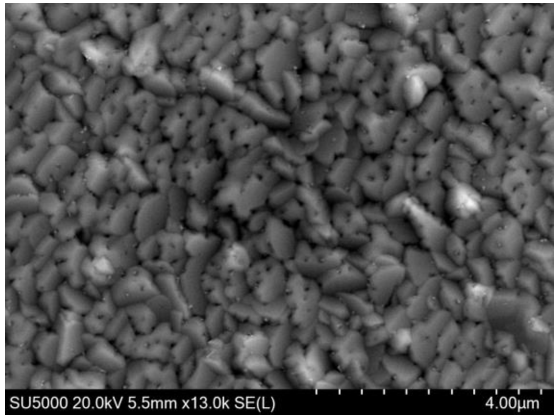
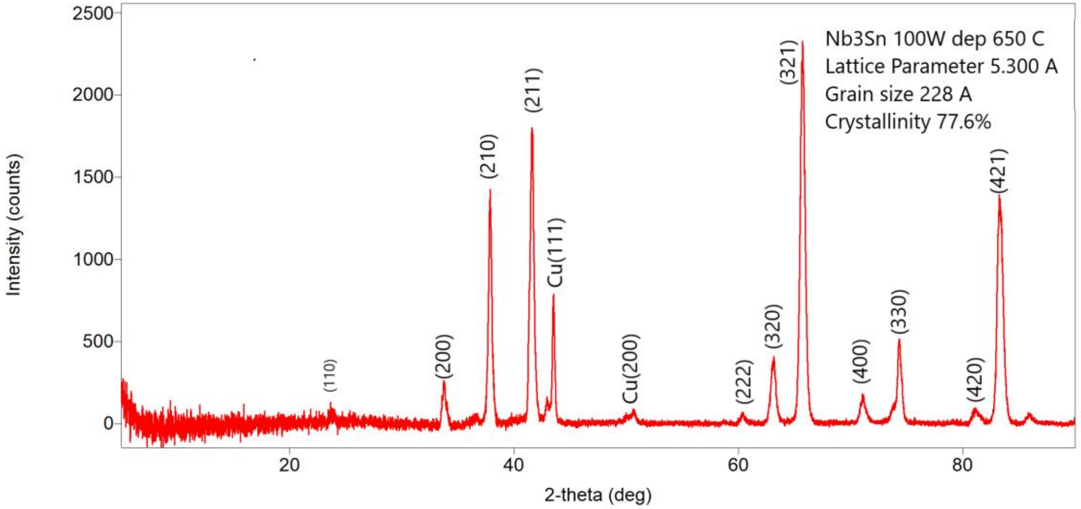
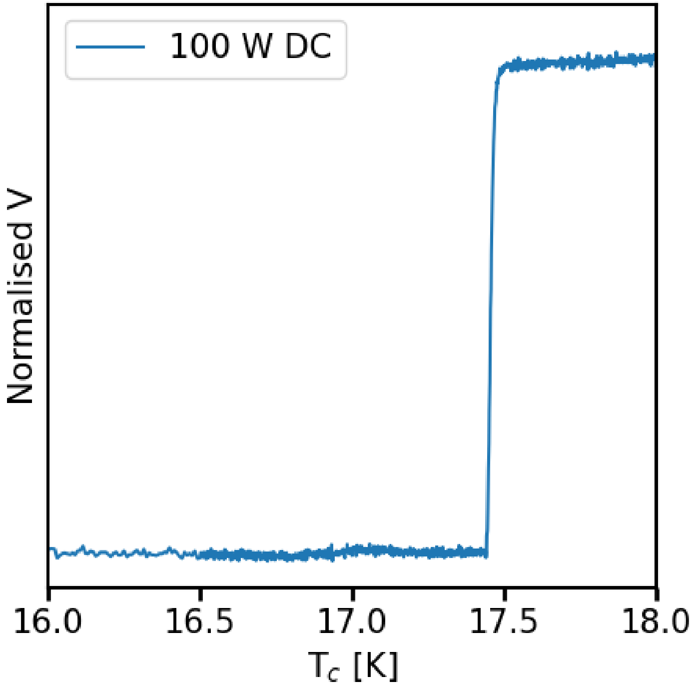
100 W DC Nb_3Sn on 50 diamond turned copper









Cu_28_06_23 100W Tc = 17

Sapp_28_06_23 100W Tc = 17.5
Sapp_28_06_23_recentre Tc=17.6

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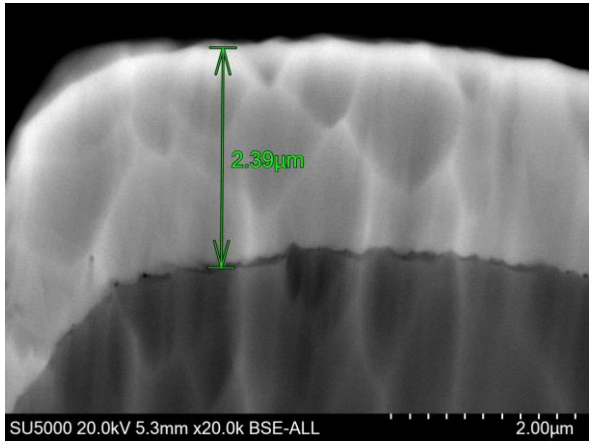
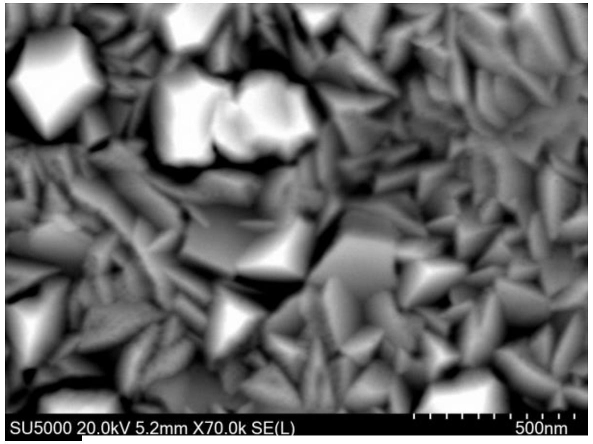
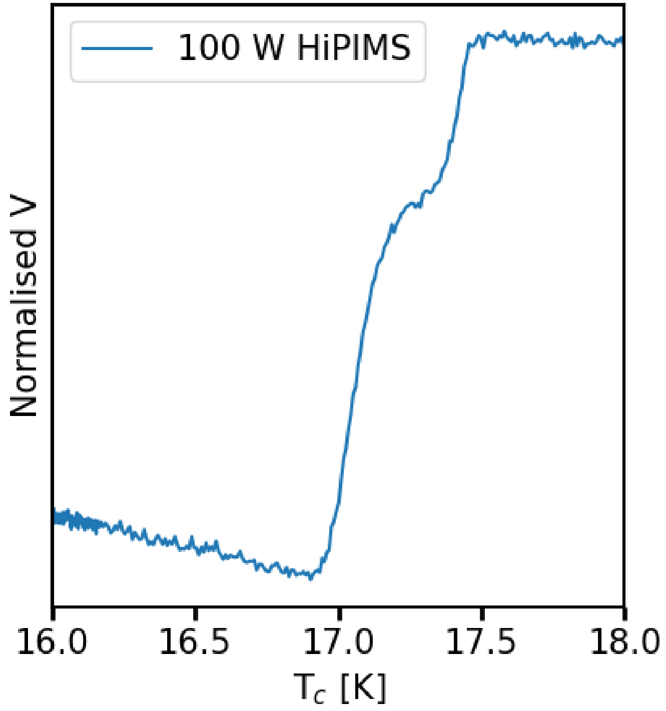
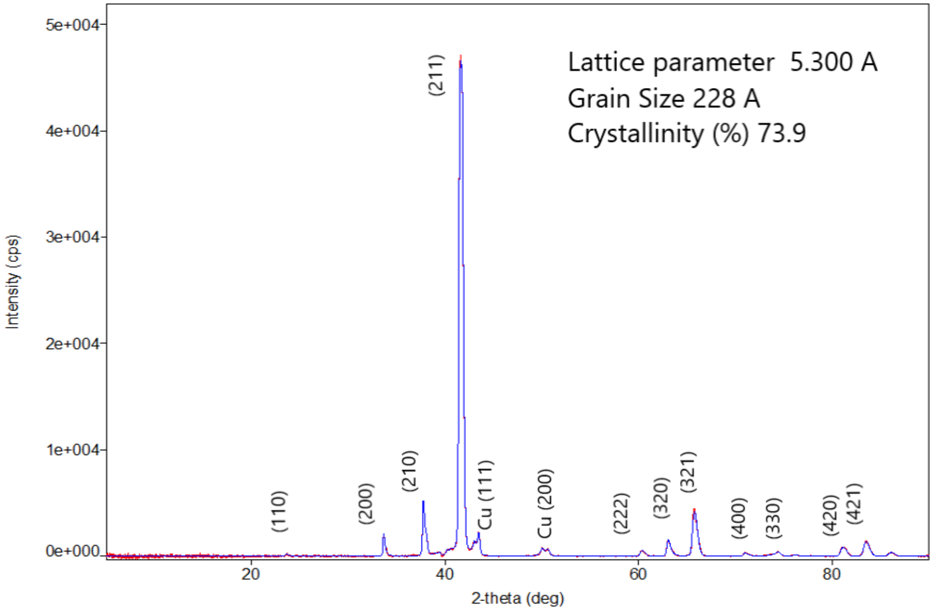
100 W HIPIMS Nb3Sn on 50 and 100 mm diamond turned copper







Cu_06_07_23 HiPIMS Tc = 16.5
Cu_06_07_23_recentre Tc = 16.5



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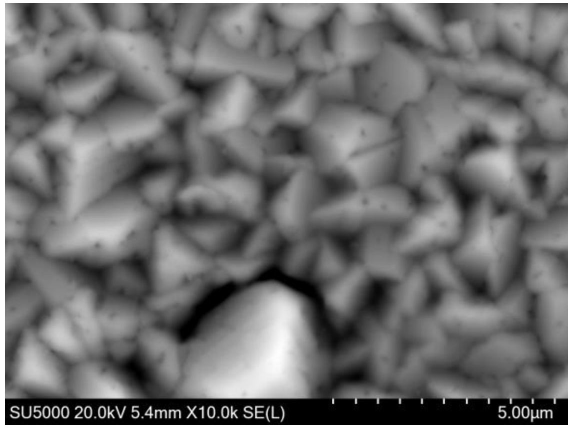
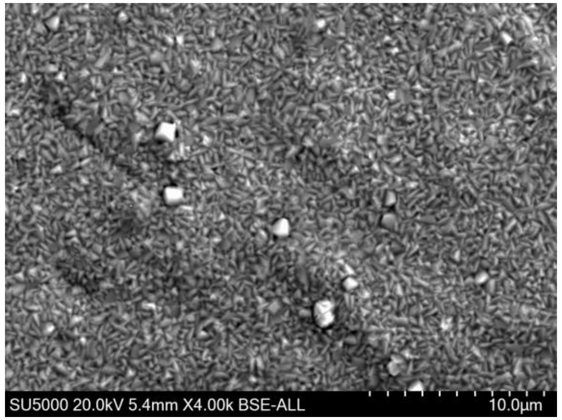
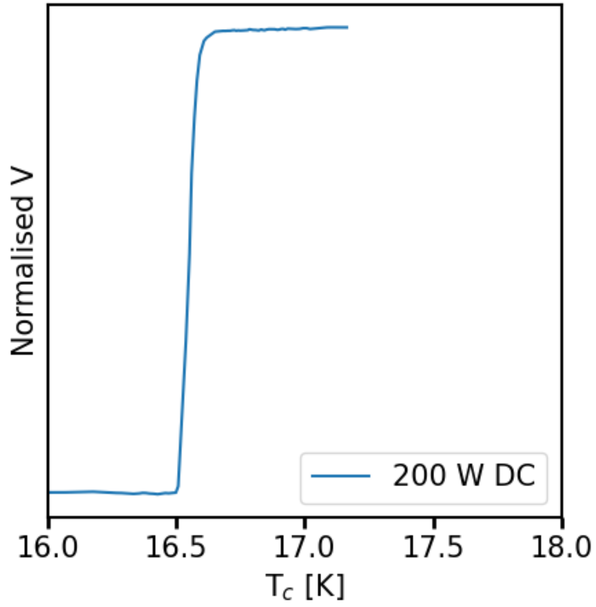
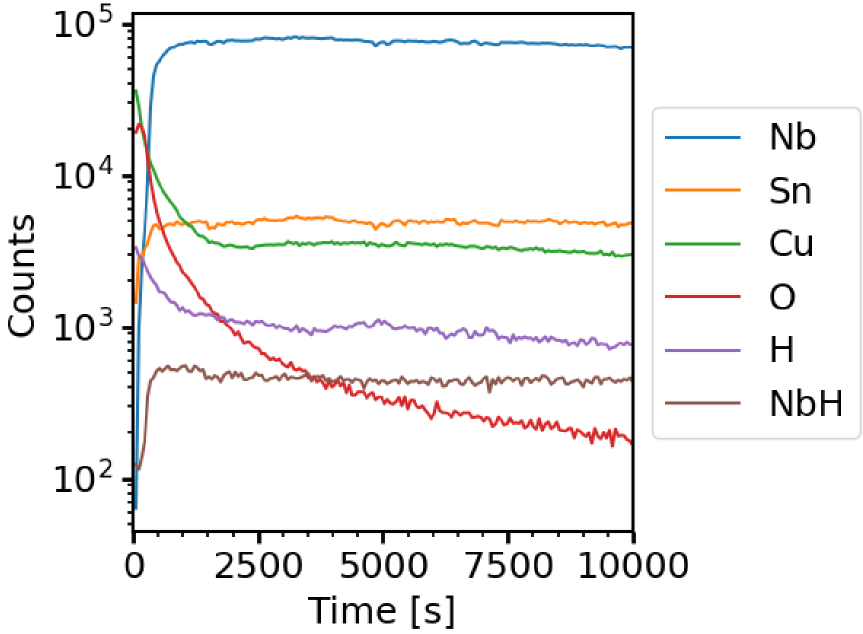
200 W DC Nb3Sn on 50 & 100 diamond turned copper

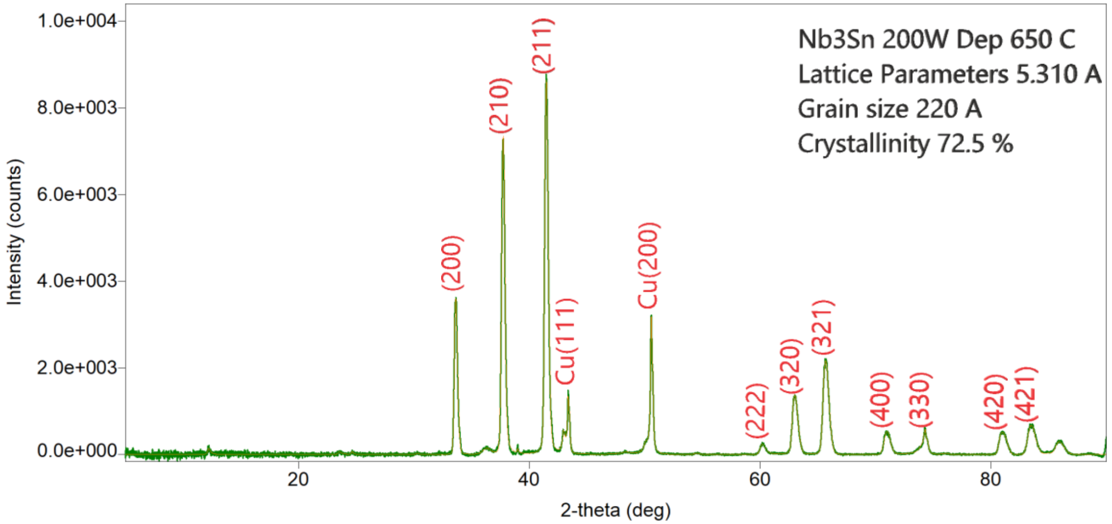




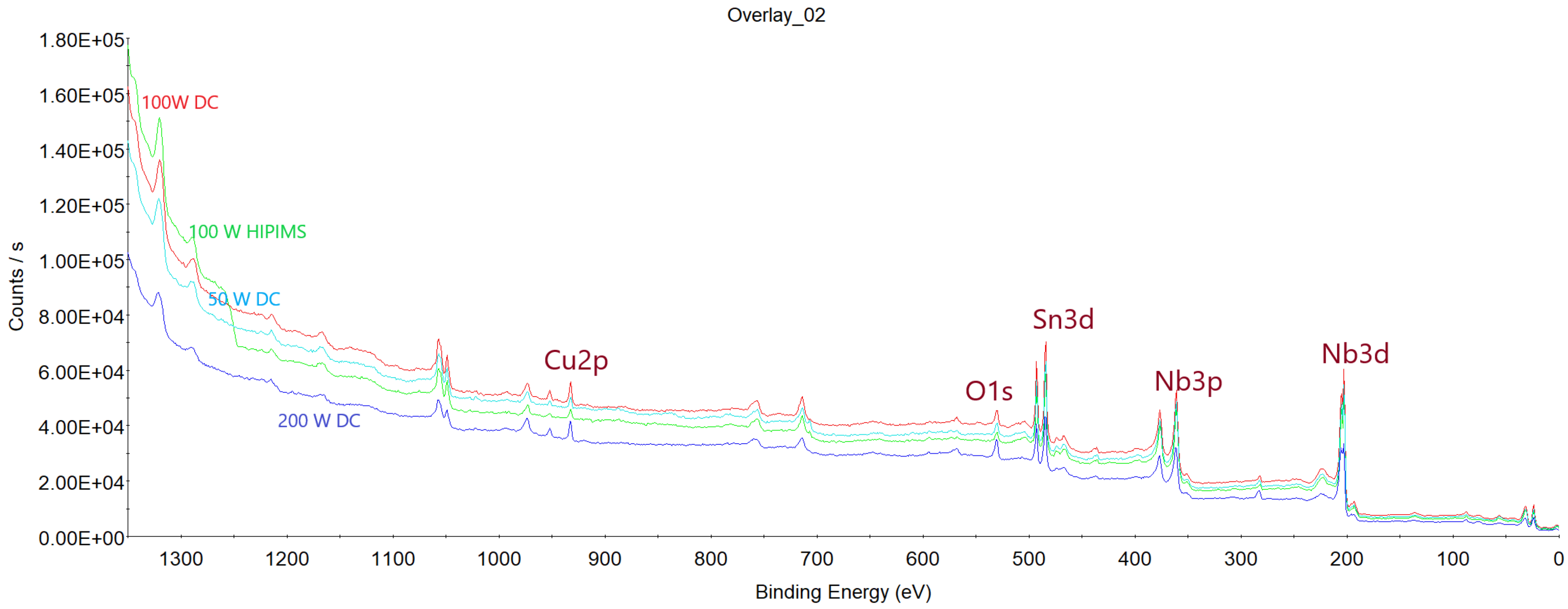
Cu_08_12_22 Tc = 16.8
Cu_08_12_22_recent Tc = 16







XPS analysis of Nb3Sn on copper



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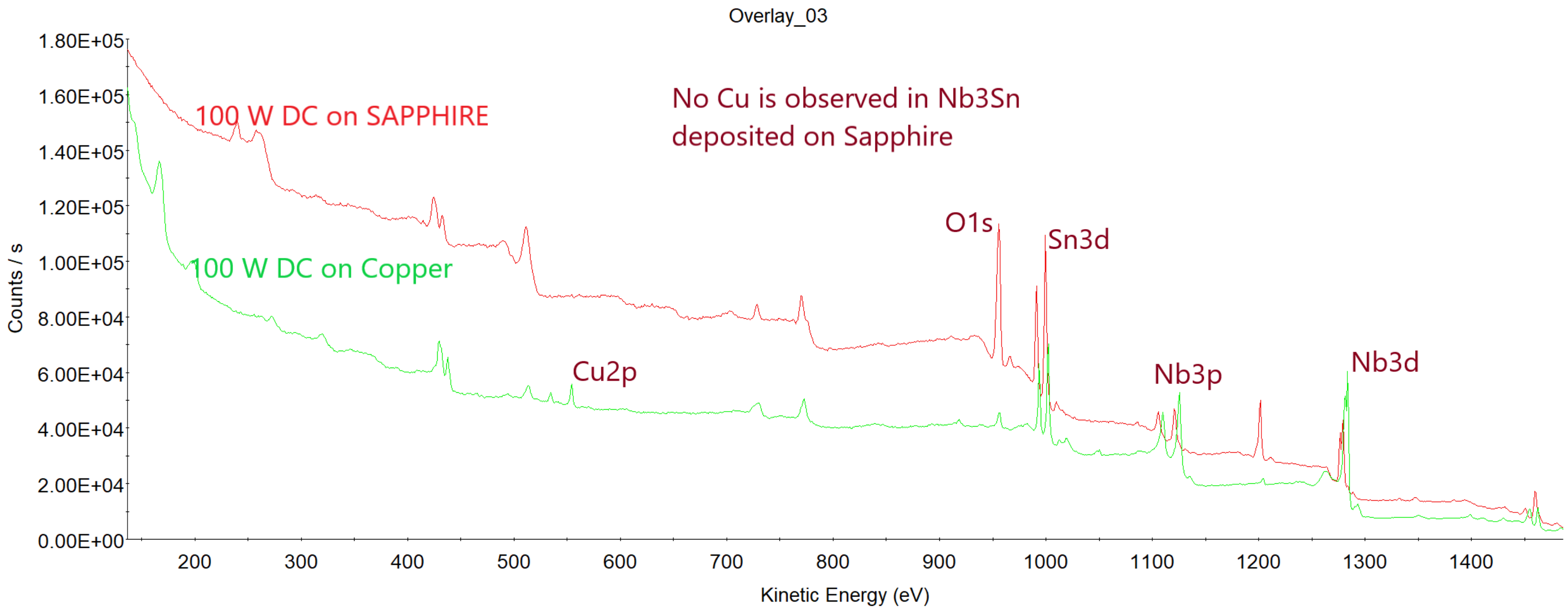
XPS analysis of Nb3Sn on copper and Sapphire



- In both case the surface is dominated with Carbon and Oxygen
- There is no traces of Cu for Nb3Sn deposited on Sapphire
- Cu can only be seen on Nb3Sn deposited on copper
- Both Nb and Sn are at oxide state with Nb both in Nb2O5 and NbO2 , and Sn in SnO

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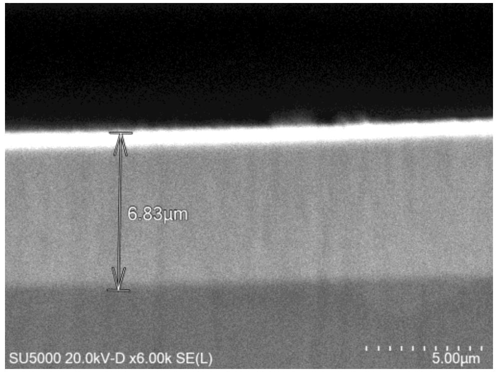
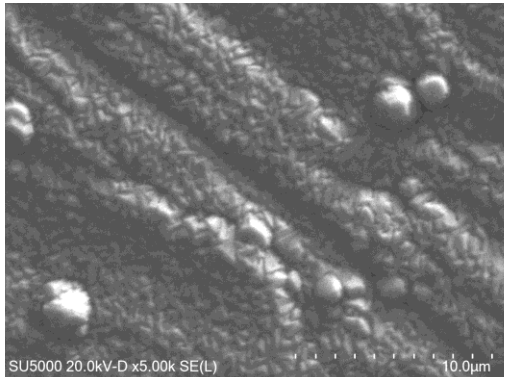
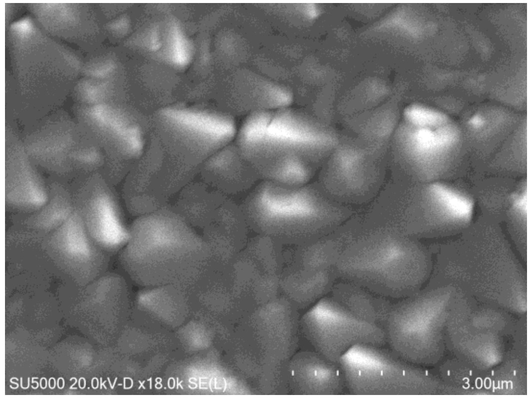
Deposition Temperature Optimisation

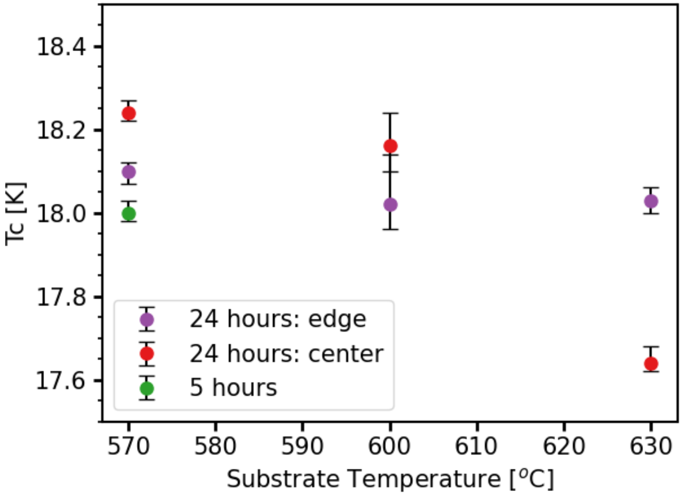
- The deposition power was set at optimum power of 50 W established in power optimisation
- The base pressure was in 5 x10^{-10} mbar and at deposition temperature 2 x10^{-8} mbar. The deposition pressure was set at 5 x10^{-3} mbar and deposition temperature of 570 C to 630 C.

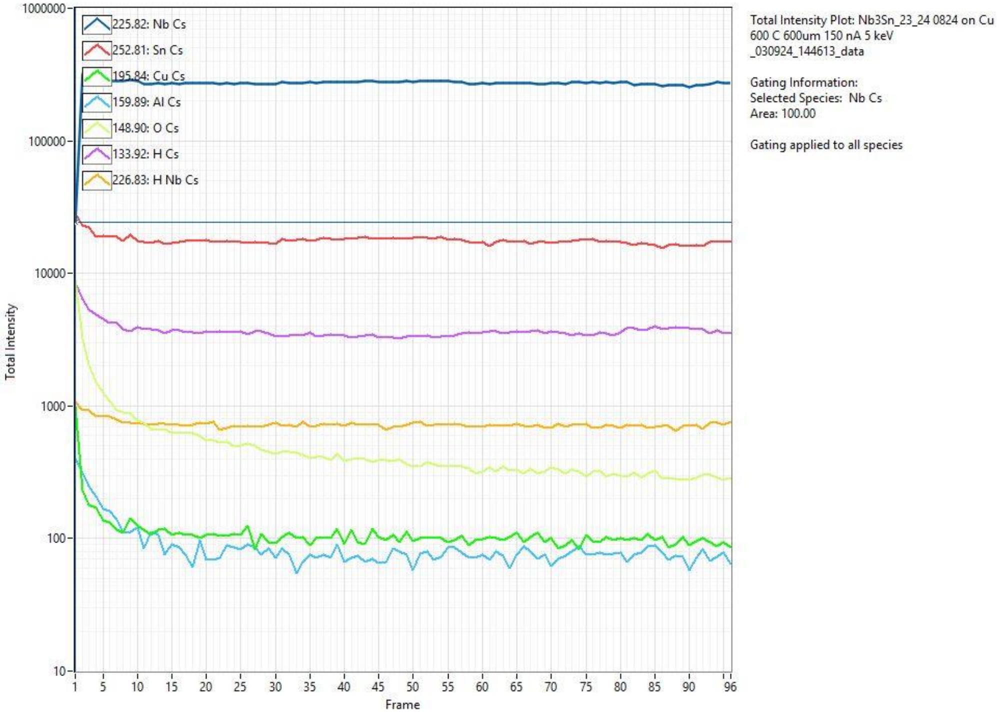






- The optimum Tc of 18.26 K for Nb3Sn deposited on Sapphire at 570 C.
- Thinner film gave a Tc of 18 K.
- Increasing the deposition range reduces the Tc to 18.1 K and 17.6 K for dep temperature of 600C and 630C respectively.





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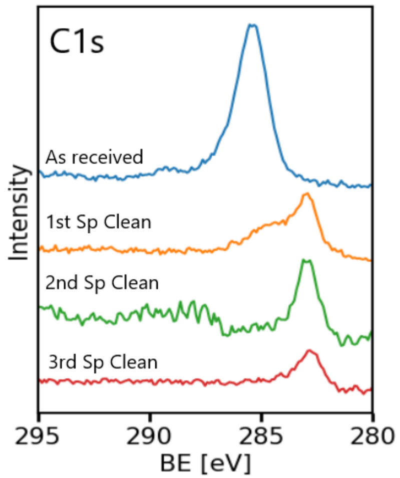
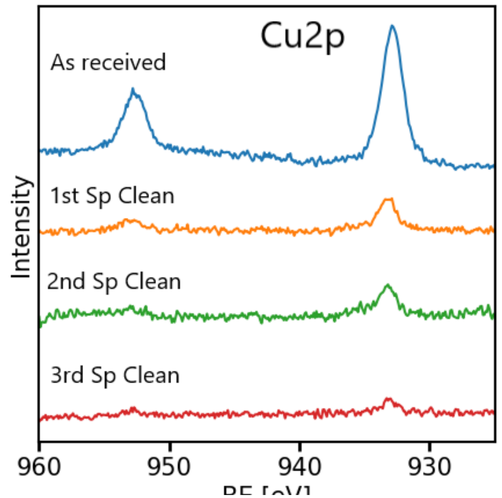
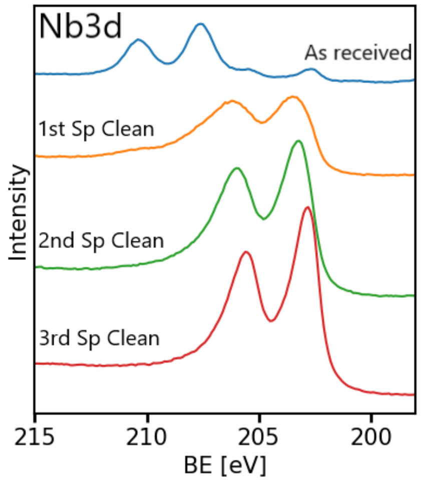
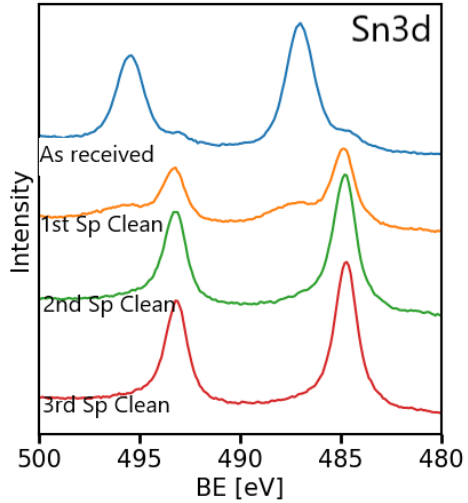
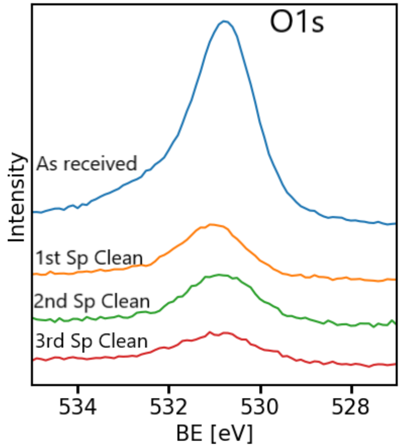
XPS analysis of Nb3Sn on Cu at 570C

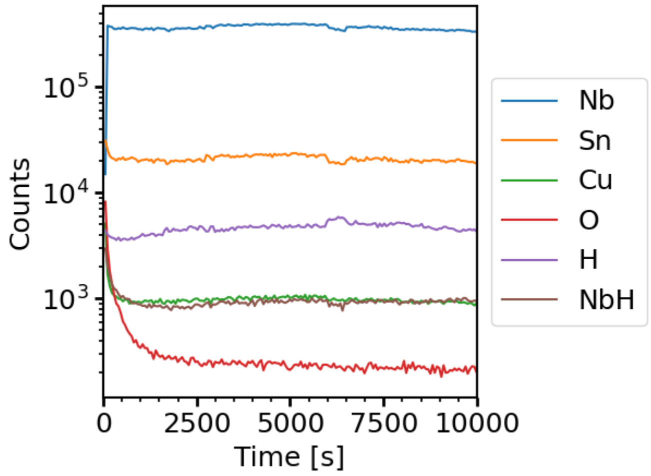












	As received	As received	1st Sput clean	1st Sput clean	2nd Sput clean	2nd Sput clean	3rd Sput clean	3rd Sput clean
Nb	47.74	6.09	65.61	13.6	75.05	22.23	75.92	22.47
Sn	52.26	3.22	34.39	7.14	24.95	7.39	24.06	7.12
Cu		2.61		1.69		1.41		1.2
O		37.16		46.33		23.71		25.55
C		49.92		31.23		22.63		21.83
H		?		?		?		?

As seem in SIMS depth profile the surface is dominated by oxygen, carbon as well as being Sn rich and presence of Cu on the surface.

Both Nb and Sn are in oxide state of Nb2O5 and SnO respectively.

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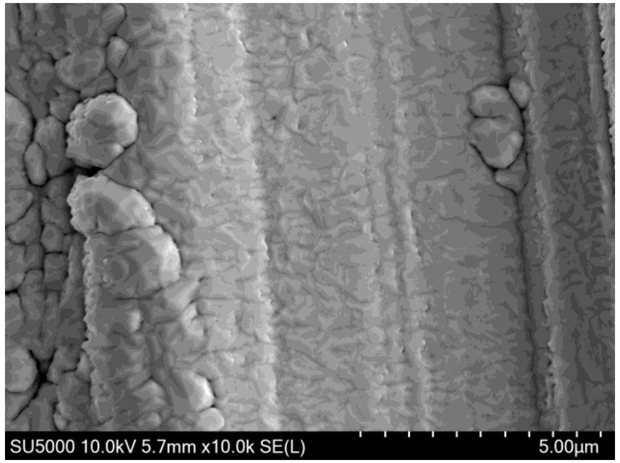
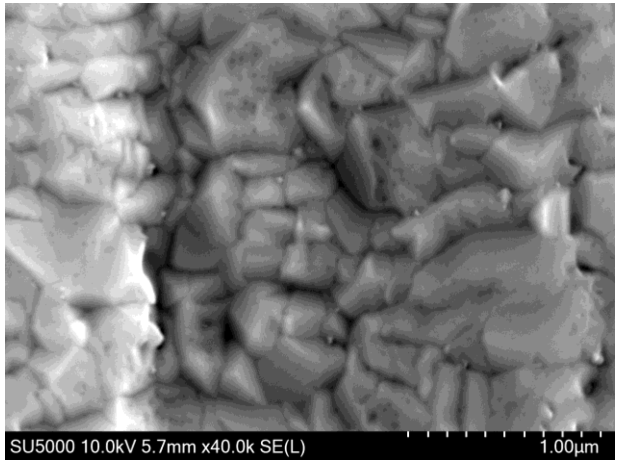
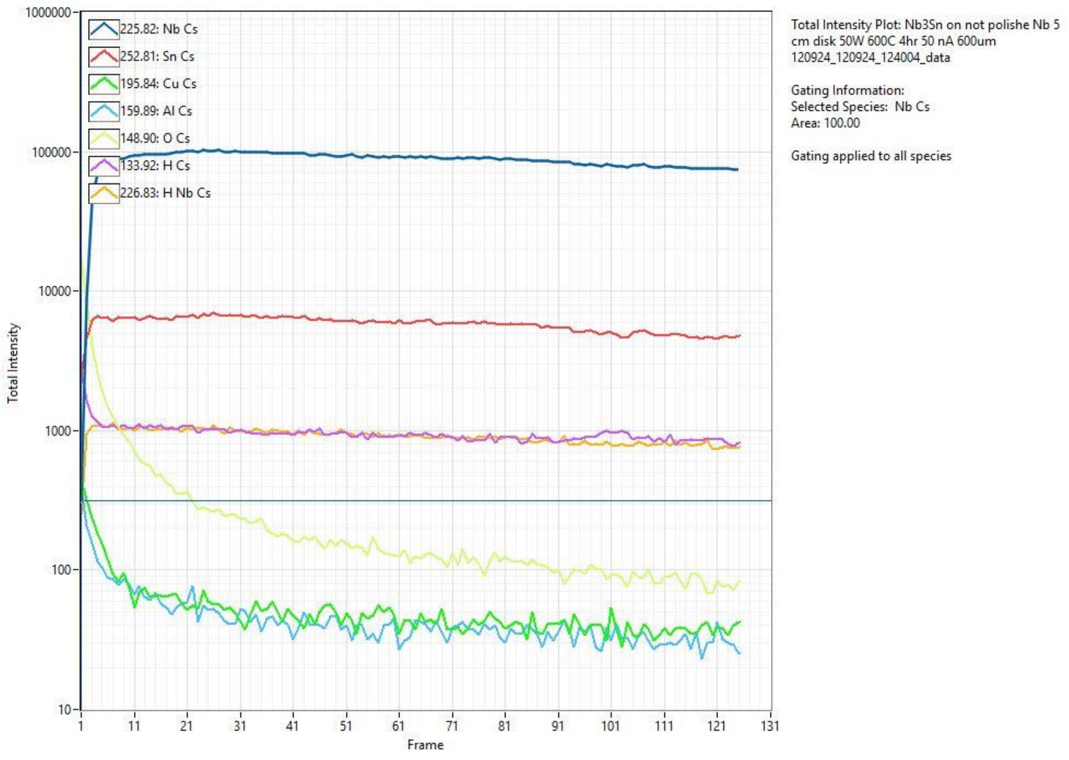
Nb3Sn deposited on Nb at 50 W and 650C

- The grain are much larger in comparison with films deposited on Cu and sapphire.
- Tc of 16 K was determined by penetration method.
- SIMS analyses showed the highest impurity present in the Nb3Sn is hydrogen
- More analysis of being done to stablish the low Tc as compared to both Cu and sapphire.







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Synthesis of V3Si

Motivation and Difficulties

V_3Si is a potential candidate for SRF thin films with similar superconducting parameters as other A15 Materials.

However, there are some difficulties:
- Stoichiometry control is very narrow window.

- Impurities can drastically affect T_c.

- Vanadium has a relatively high diffusivity when considering typical cavity substrates (Nb, Cu).

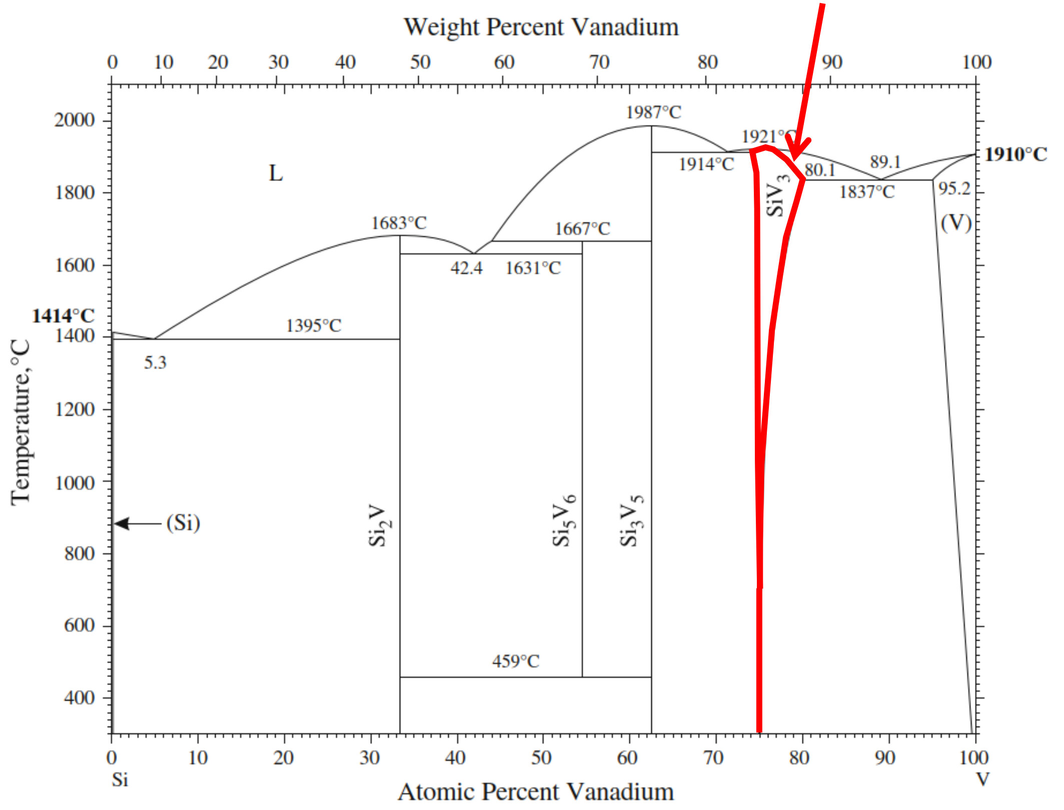
Table 1. Superconducting parameters for some candidate materials considered for SRF applications.

Material	T_c (K)	ρ_n ($\mu\Omega$ cm)	$H_c(0)$ (T)	$H_{c1}(0)$ (T)	$H_{c2}(0)$ (T)	λ (nm)	Δ (meV)	ξ (nm)
Nb	9.23	2	0.2	0.18	0.28	40	1.5	35
NbN	16.2	70	0.23	0.02	15	200–350	2.6	3–5
NbTiN	17.3	35		0.03	15	150–200	2.8	5
Nb_3Sn	18	8–20	0.54	0.05	28	80–100	3.1	4
V_3Si	17	4	0.72	0.072	24.5	179	2.5	3.5
Nb_3Al	18.7	54			33	210	3	
Mo_3Re	15	10–30	0.43	0.03	3.5	140		
MgB_2	40	0.1–10	0.43	0.03	3.5–60	140	2.3 /7.2	5
Pnictides	30–55		0.5–0.9	30	50–135	200	10–20	2

A15 materials

Table from: Anne-Marie Valente-Feliciano 2016 *Supercond. Sci. Technol.* **29** 113002

V_3Si



V-Si binary phase diagram [1]

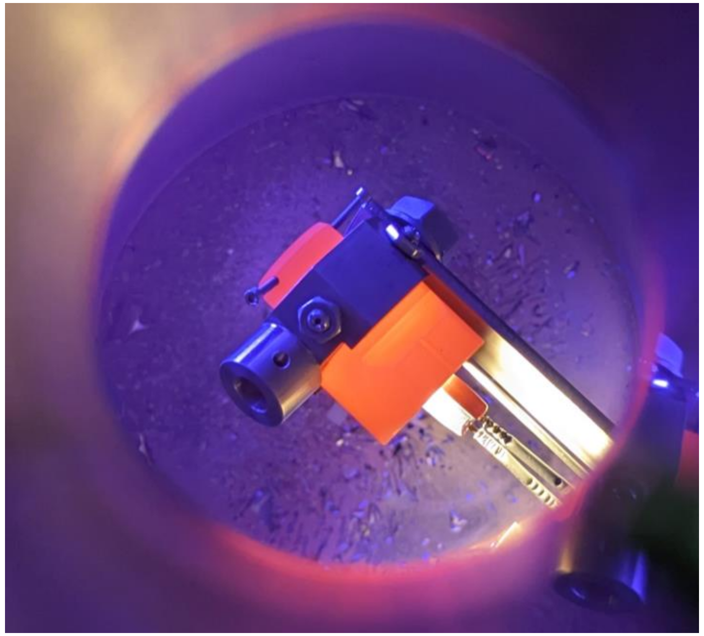
V_3Si Deposition System

- Deposition system has a load lock to maintain a clean V_3Si target with a base pressure <4x10^{-9} mbar.

- Sample stage reaches a max temperature of ~850 ˚C and can be biased during deposition.

- All samples were deposited using a single $V_{75\%}Si_{25\%}$ alloy target.

- Kr gas was used as the process gas.



Sample holder and heater stage.

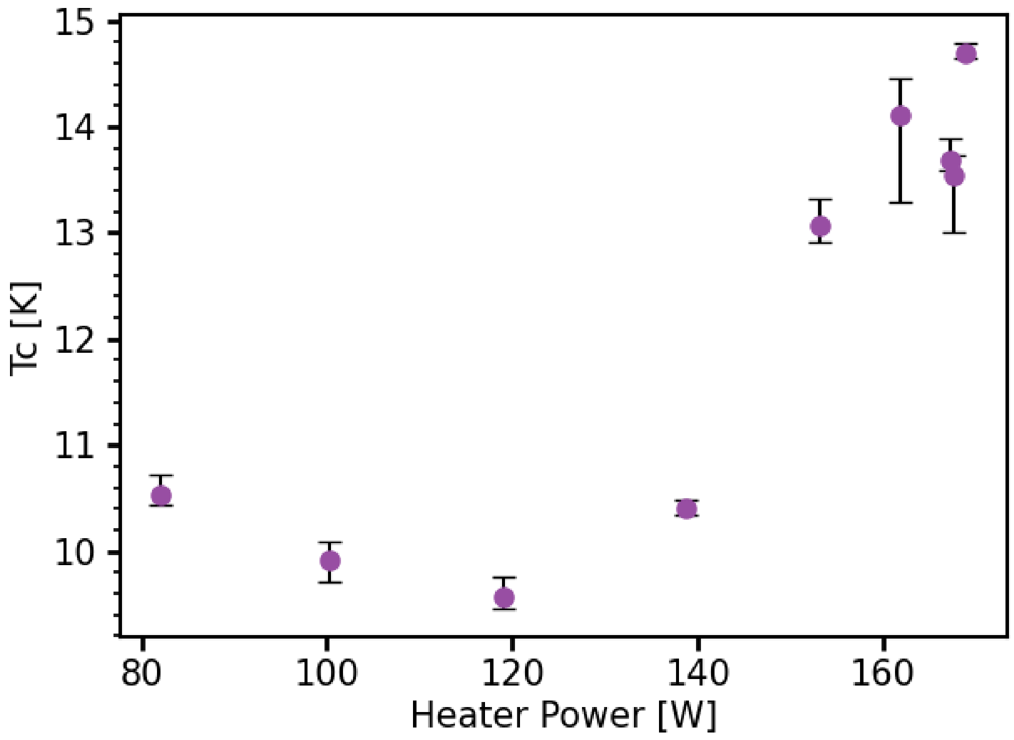
Pulsed DC Deposition parameters:

Frequency 350 kHz	
Pulse width	1.1 µs
Power	300 W
Pressure	5x10^{-3} mbar

HiPIMS Deposition parameters:

Frequency 1000 Hz	
Pulse width	100 µs
Power	250 W
Duty Cycle 10 %	
Pressure	5x10^{-3} mbar

Pulsed DC magnetron sputtering: Sapphire substrates

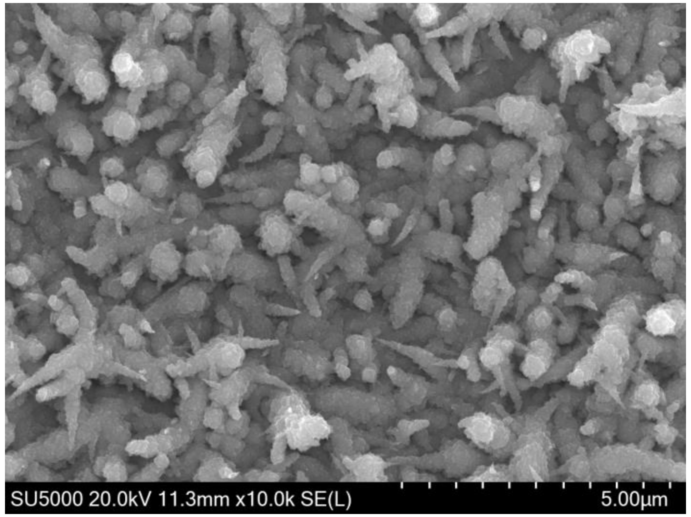


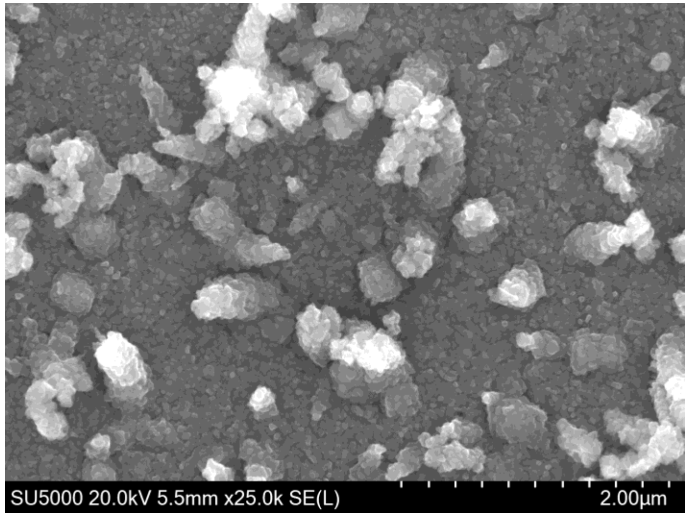
T_c measured by four-point probe [1 mA]

- Max Tc Achieved - 14.69 K ± 0.12 K at the maximum of the sample heater.

- Inconsistency at higher heater power may be attributed to poor thermal conductivity between sample and holder

- SEM show a preferential 'spiked' and porous film growth unsuitable for SRF applications.





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HiPIMS - Sapphire

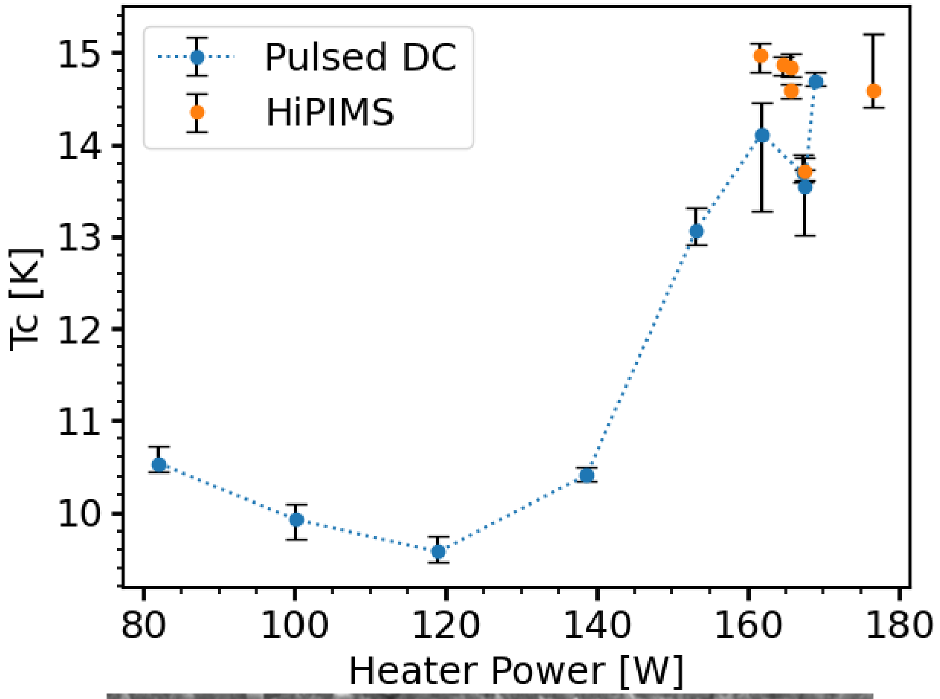
High-power pulsed magnetron sputtering (HiPIMS) was used to improve the density of the films and increase the quality of the films.

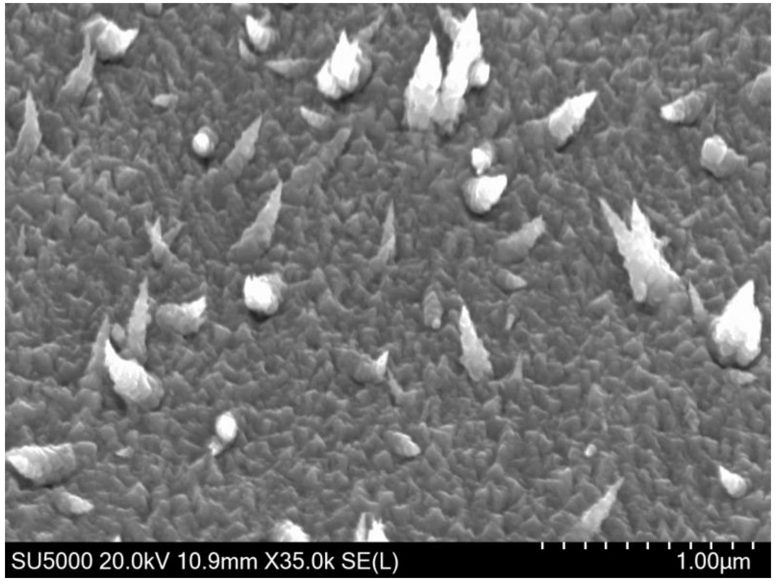
Initial study using a duty cycle of 10 % at high temperatures shows improved Tc with smaller ΔT.

Maximum Tc achieved is 14.98 K ± 0.2 K.

The SEM images show an improved film growth.

The ‘spikey’ features are still present but in fewer numbers on the surface of the film. Further work is required to investigate lower duty cycles.





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HiPIMS – Polished Nb

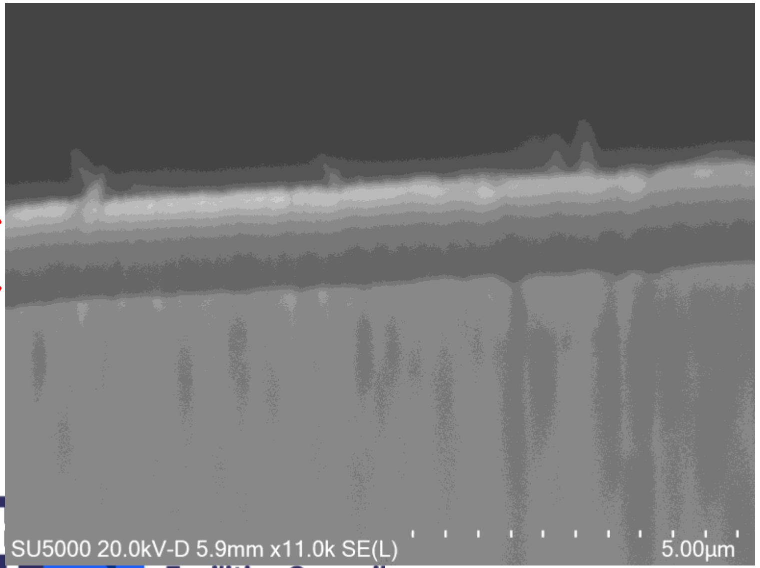
V_3Si films were also deposited on polished Nb substrates (R_{rms} = < 150 nm). Cu was not chosen due to the high temperatures required for deposition.

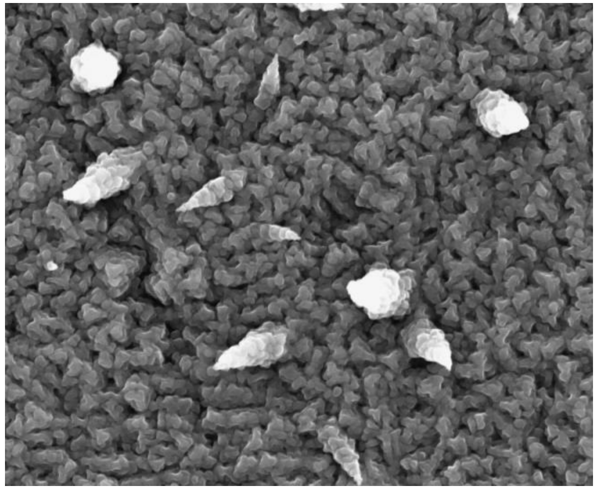
The SEM shows a slightly more open structure compared to the sapphire deposited films. The spiked features are still observed.

SQUID VSM was conducted at RAL, showing a transition at ~12.2 K.

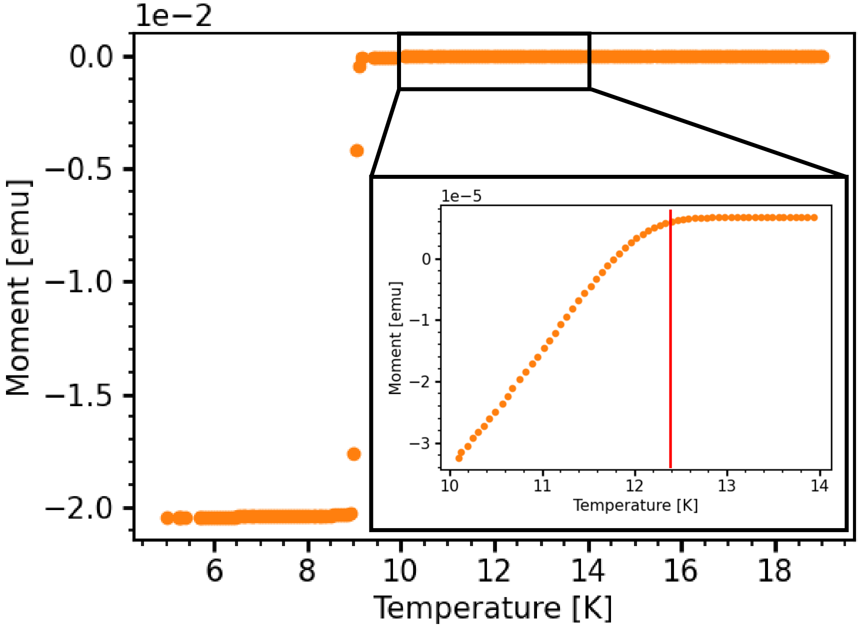
V$_3$Si
1.6 µm

Nb
Substrate



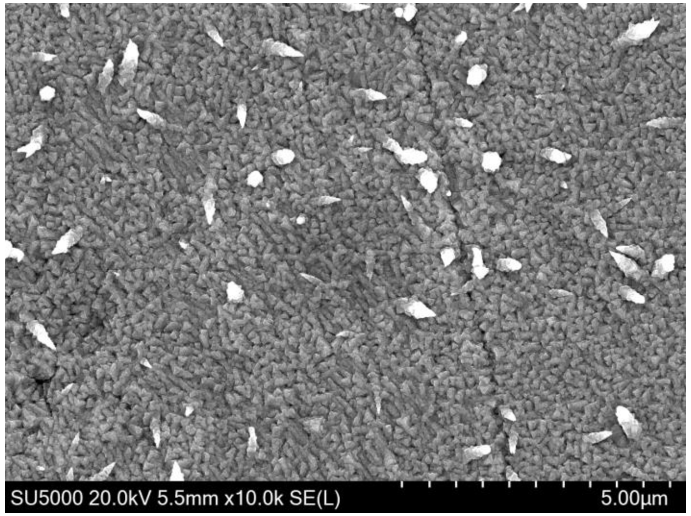


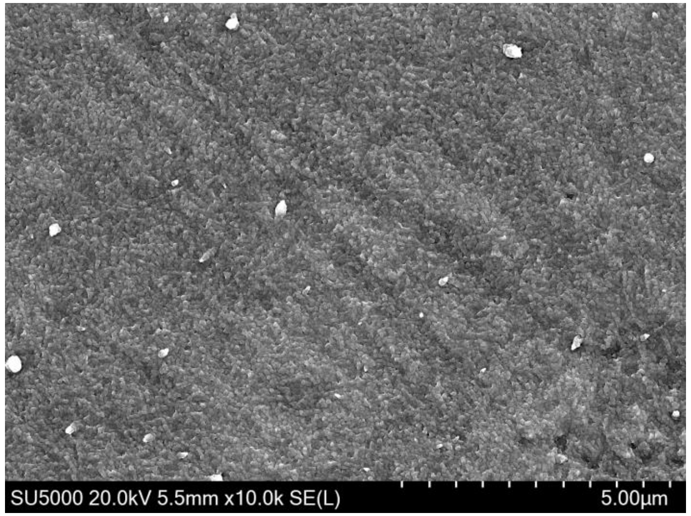
1µm

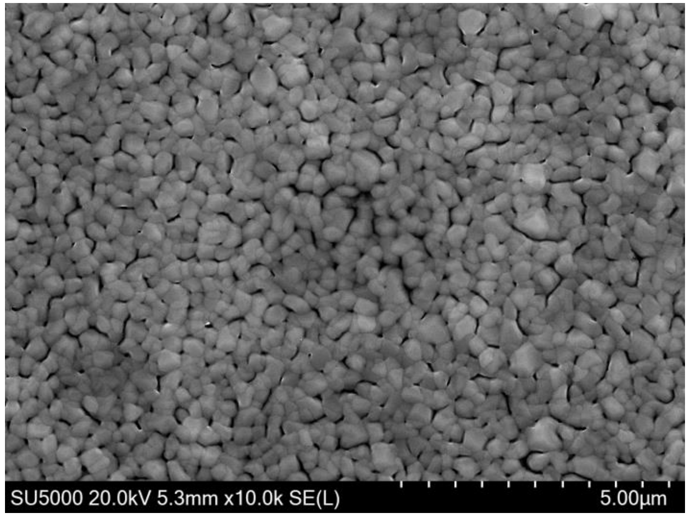


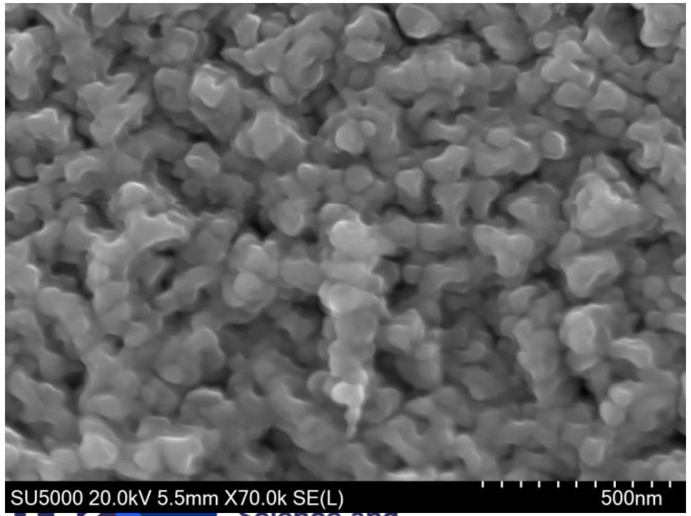
T_c measured by SQUID VSM at RAL

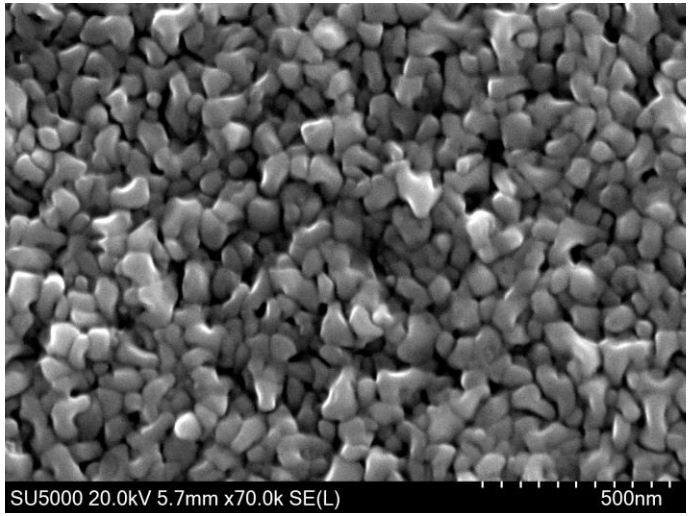
HiPIMS and sample bias

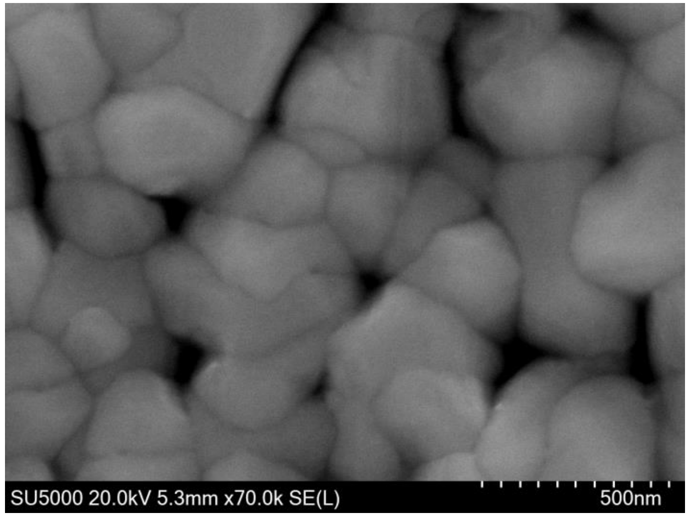












Sample Bias [V] 0 -50 -75

Increased Density, Larger crystallites

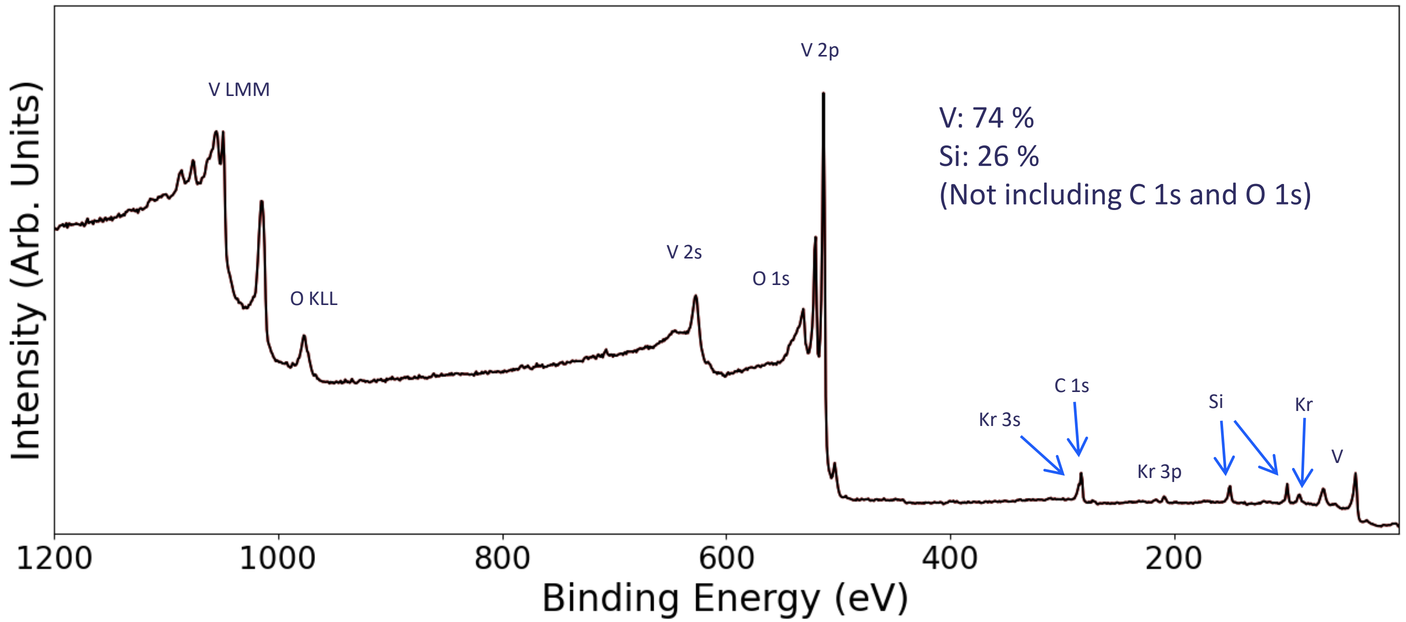
Biased HiPIMS

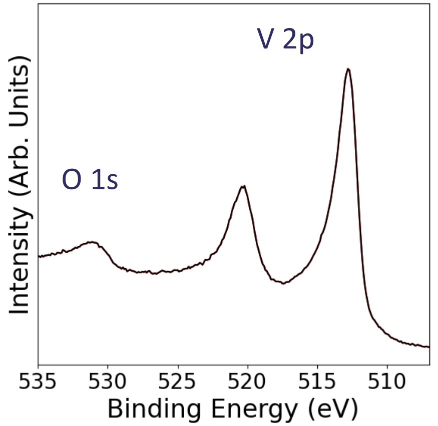
- SIMS shows a distinct V_3Si film with a consistent level of carbon throughout the film. Oxygen is mostly at the surface but is present in the bulk.

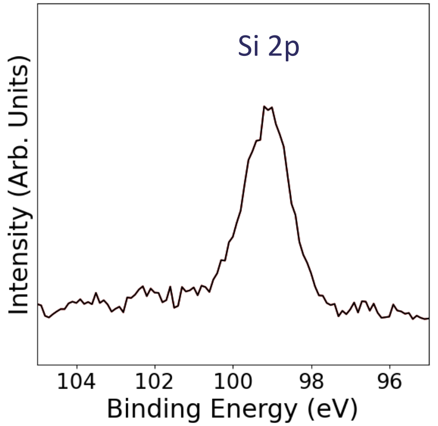
- XPS shows a V:Si ratio of 74 %: 26% and the presence of carbon and oxygen in the bulk of the film agreeing with SIMS.

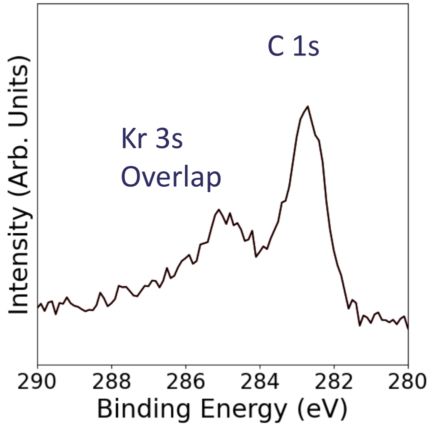
- Carbon is in a carbide bonding environment suggesting formation during deposition.

XPS after Kr^+ sputtering *in situ*

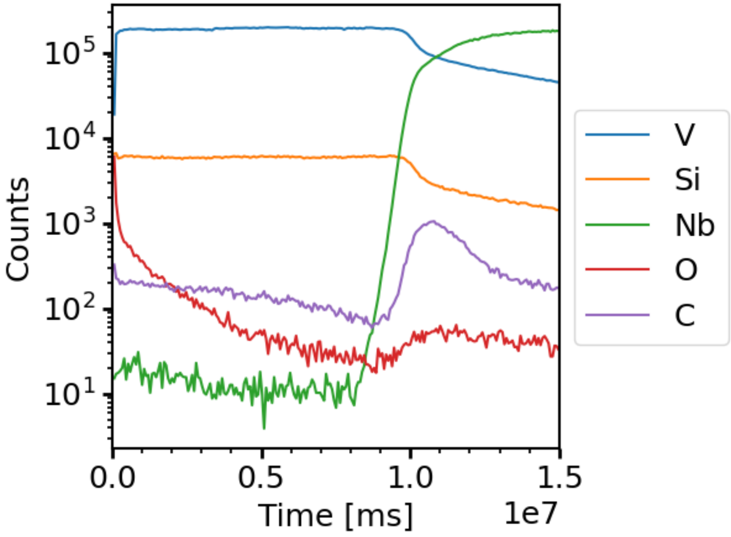








SIMS



Summary and Future Work

Significant progress has been made towards V_3Si films for SRF applications.

Further work:

- A study of duty cycle to improve film density and quality.

- Carbon contamination needs to be investigated (target or deposition system?).

- Samples need to be produced for surface resistance measurements and testing.

- Thin film growth on different substrates

Summary (Nb_3Sn)

- Synthesis Nb3Sn using an alloy target ensure the correct stoichiometry through out the depth of the film.
- The surface of the Nb3Sn is always terminated by Nb2O5, NbO2 and SnO.
- The surface is also Sn rich and Nb deficient.
- Optimum deposition parameters was stablished for Nb3Sn on sapphire with Tc very close to bulk value of 18.3 K
- The optimum deposition parameters were:
  - Deposition power 50W DC
  - Deposition temperature 570 C
  - Deposition pressure $5x10^{-3}$ mbar
- Nb3Sn can be deposited without any buffer layer on Cu substrate with good adhesion and surface resistance (results from Choke cavity, Dan Seal)
- The observed Cu concentration at the surface is estimated to be about 2 to 3 percent within the 150 nm top surface, the concentration gradually decrease below 1 % through the bulk.
- There is sharp interface between the copper substrate and Nb3Sn.
- Nb3Sn deposited on sapphire
- The Cu segregation to the surface does not seems to be the source of lower Tc as compared with Sapphire, no copper contamination was observed for Nb3Sn deposited on Nb which showed a lower Tc of 16 K.

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