PVD Deposition Of Nb$_3$Sn From An Alloy Target On Copper

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on behalf of the team

Nb$_3$Sn Workshop 9$^{th}$ - 13$^{th}$ 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 Tc (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.

- Nb$_3$Sn alloy is type II superconductor with ideal Tc 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 Nb$_3$Sn coating.

- The material can be deposited as thin film either in:
  - Single layer (Nb$_3$Sn on Cu or Nb)
  - Double layer (Nb /Nb$_3$Sn on Cu)
  - Multilayer (SIS): Nb /Insulator/ Nb$_3$Sn, 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 parameters of \( a = 0.330 \) nm
- In Nb₃Sn the lattice parameters of 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 Tc in comparison to bcc Nb.
- Sn deficiency may cause the Nb to occupy the site and affect the long range order
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 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
**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 $2 \times 10^{-9}$ 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/Nb$_3$Sn deposition (single layer) at various Temperature

- Nb$_3$Sn 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 areas that it is Sn deficient.
- The Tc 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.

<table>
<thead>
<tr>
<th>Layer</th>
<th>$t_{1}$ (1e15at/cm²)</th>
<th>$t_{2}$ (nm)</th>
<th>$r$ (1e22at/cm³)</th>
<th>Nb</th>
<th>Sn</th>
<th>Al</th>
<th>O</th>
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<tbody>
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<td>1489.817</td>
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<td>75.000</td>
<td>25.000</td>
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<td>11.800</td>
<td>0.000</td>
<td>0.000</td>
<td>40.000</td>
<td>60.000</td>
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</table>
Cu /Nb/Nb$_3$Sn (double layer)

- The interfaces both at Cu/Nb and Nb/Nb$_3$Sn is well defined.
- 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 Nb$_3$Sn layer can be observed.
- First $B_{en}$ is estimated at 95mT.

<table>
<thead>
<tr>
<th>Layer</th>
<th>$t$ (1e15at/cm$^2$)</th>
<th>$t$ (nm)</th>
<th>$r$ (1e22at/cm$^3$)</th>
<th>Nb</th>
<th>Sn</th>
<th>O</th>
<th>Al</th>
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<tbody>
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<td>0.0000</td>
<td>0.0000</td>
<td>60.0000</td>
<td>40.0000</td>
</tr>
</tbody>
</table>

$T_c(Nb_3Sn) \sim 17K$  
$T_c(Nb) \sim 8.8K$
Cu/Nb/Nb$_3$Sn (double layer)

- Two distinct areas can be observed:
  - Perfect area with sharp interface with correct stoichiometry for Nb$_3$Sn layer
  - Copper diffusion from the interface to top surface.

- Nb and Nb$_3$Sn layers are completely intermixed and there is a substantial volume of copper substrate present throughout the depth of the layer and at the surface.

- Inside the cavity there are trace silicon and aluminium oxide.
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
The observed distortion of the Nb₃Sn / 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 Nb₃Sn layer is 25% thicker than the second Nb₃Sn 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$_3$Sn 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:
  - 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
Thank you