Fermilab

Optimization of Superconducting Nb_3Sn Electrochemical Film Technologies

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Premise

An electro-chemical deposition technique, under US Patent Application, to produce Nb_3Sn coatings was developed in the last few years by FNAL in collaboration with Politecnico di Milano.
[Synthesis of superconducting Nb_3Sn *coatings on* Nb *substrates, E.Barzi, M.Bestetti, F.Reginato, D.Turrioni, S.Franz, Supercond. Sci. Technol. 29015009]*

Premise

Targets:

- Reproduce and optimize the original recipe
- SEM/EDX Analysis
- Check superconducting properties

The experimental apparatus was procured and commissioned before my arrival, in order to synthesize thin films in-house. I was trained by E.Barzi, D.Turrioni, D.Hicks, P.Li.

REALIZATION OF THE SAMPLE

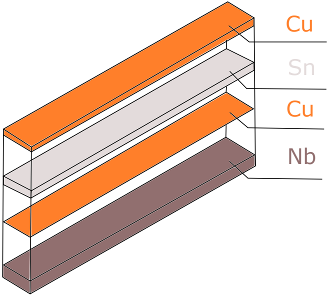
Realization of the sample

Nb substrate of dimensions 1cm x 10cm x 300μm

Electro-deposition processes:

- Cu seed layer onto the Nb substrate
- Sn layer onto the Cu seed layer
- Cu barrier layer onto Sn

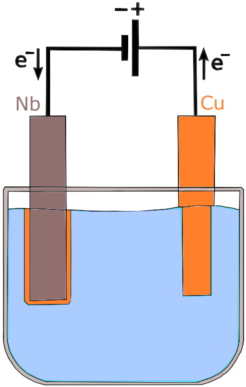
followed by heat treatment



First electro-deposition step

Bath:

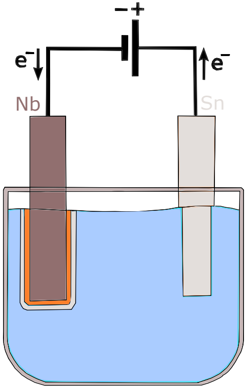
- HCl: 67.8ml
- H_2SO_4: 217.4ml
- $CuSO_4$: 120g
- H_2O: as needed to achieve 2L



Second electro-deposition step

Bath:

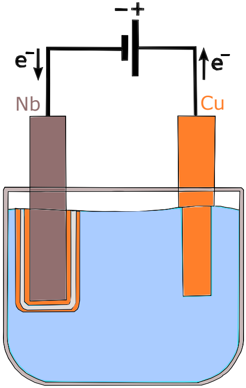
- Solderon Acid HC: $430\mathrm{ml}$
- Solderon Tin HS-300 Concentrate: $333\mathrm{ml}$
- Solderon MHS-W Primary: $200\mathrm{ml}$
- H_2O: as needed to achieve $2\mathrm{L}$



Third electro-deposition step

Bath:

- $Cu_2P_2O_7$: 52g
- $NaNO_3$: 10g
- $Na_4P_2O_7$: 360g
- H_2O: as needed to achieve 2L



DC electrodeposition

Parameters:

- Current density
- Time
- Temperature
- PH
- Stirring rate

Properties:

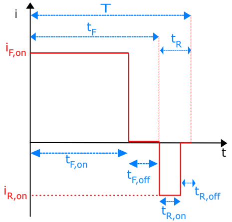
- Thickness
- Uniformity
- Adhesion

Too many nucleation sites on the boundaries !

Pulsed electro-deposition

Parameters:

- Temperature, PH, stirring rate, time
- Waveform parameters

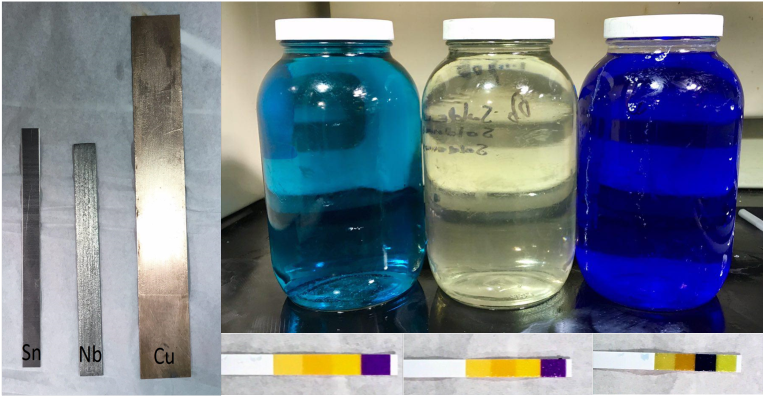


Properties:

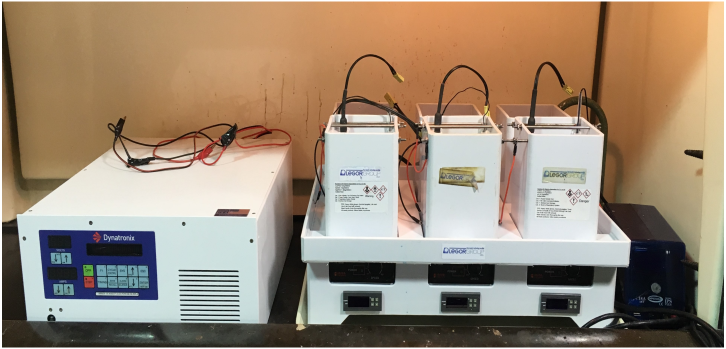
- Thickness
- Uniformity
- Adhesion

Depositions are much more uniform !

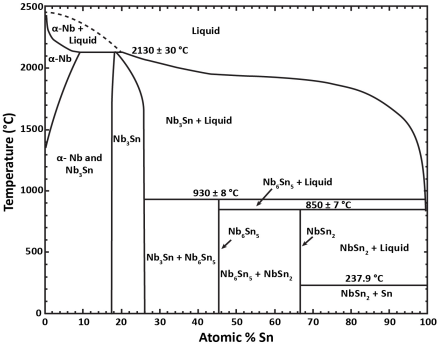
Electro-deposition: experimental setup



Electro-deposition: experimental setup

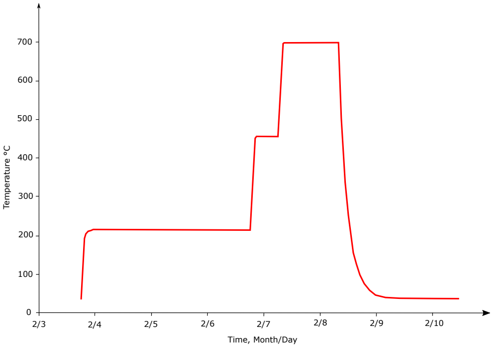


Phase Diagram of Nb-Sn



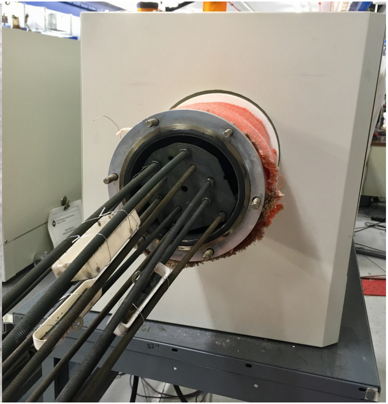
Minimize the presence of other phases would require a temperature above 930°C !

Heat treatment for Nb/Cu/Sn/Cu samples



The presence of Cu makes the Nb_3Sn phase stable at lower temperatures!

Heat treatment: experimental setup



SEM/EDX ANALYSIS

SEM/EDX

Scanning Electron Microscopy (SEM)

- Thickness of the three electro-deposited layers
- Thickness of the Nb_3Sn phase formed

Energy-dispersive X-ray spectroscopy (EDX)

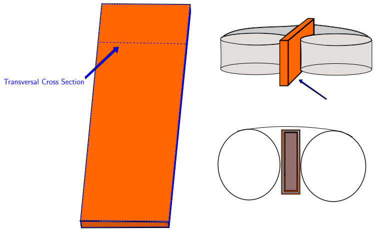
- Chemical characterization of the sample

! The sample has to be prepared properly!

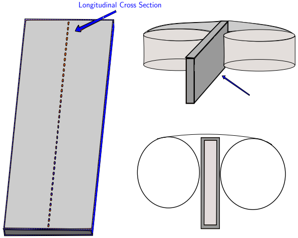
Cutting of the sample

The dimensions of the sample must fulfil the ones required by SEM!

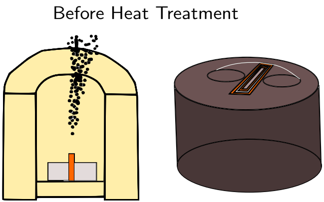
Before Heat Treatment

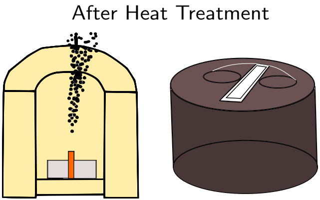


After Heat Treatment



Epoxy process





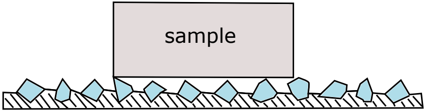
The bakelite has to be conductive to avoid charge localization during the SEM analysis

Epoxy process: experimental setup

Grinding and Polishing

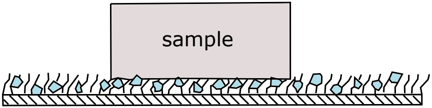
Get a flat a smooth and flat surface:

- Grinding process
  - Low grit: 320, 400, 600
  - High grit: 800, 1200



Grinding

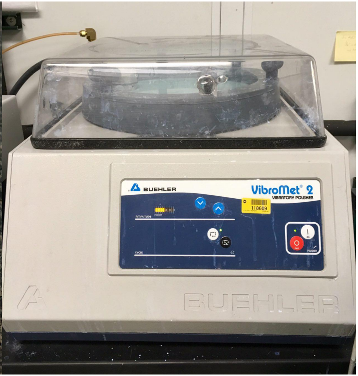
- Polishing process



Polishing

Grinding process: experimental setup

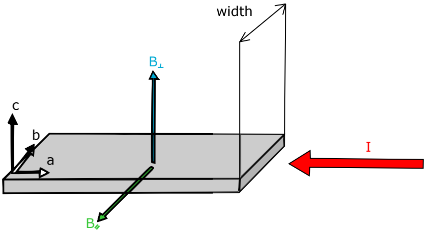
Polishing process: experimental setup



SUPERCONDUCTING PROPERTIES

SC experiment

- I_c: critical current
- B: magnetic field
- B_{c2}: upper critical magnetic field
- B_{c20}: upper critical magnetic field at zero temperature



Phase diagram I_c - B at $T = 4.2K$

Critical current density

$$I_c(B,T)B = h_0 \left(\frac{B_{c2}(T)}{B_{c20}}\right)^{\eta} \left[1 - \frac{B}{B_{c2(T)}}\right]^2 \left(\frac{B}{B_{c2}(T)}\right)^{0.5}$$

$$\frac{B_{c2}(T)}{B_{c20}} = \left[1 - \left(\frac{T}{17.5K}\right)^{1.5}\right]$$

h_0, B_{c20}, η are free parameters for the fit

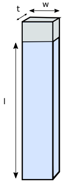
! These equations are valid at fixed strain!

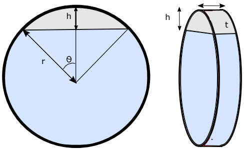
SC properties: experimental setup

SAMPLES PRODUCED AND RESULTS

Samples

Two shapes of the substrate



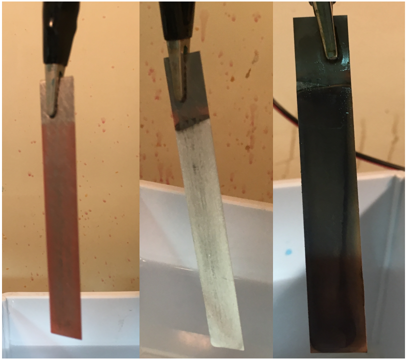


$$A_p = 2(l \cdot w + l \cdot t) + w \cdot t \qquad A_d = 2r^2 \left[\pi - \theta + \left(1 - \frac{h}{r} \tan(\theta) \right) \right] + 2rt(\pi - \theta)$$

Optimal current densities:

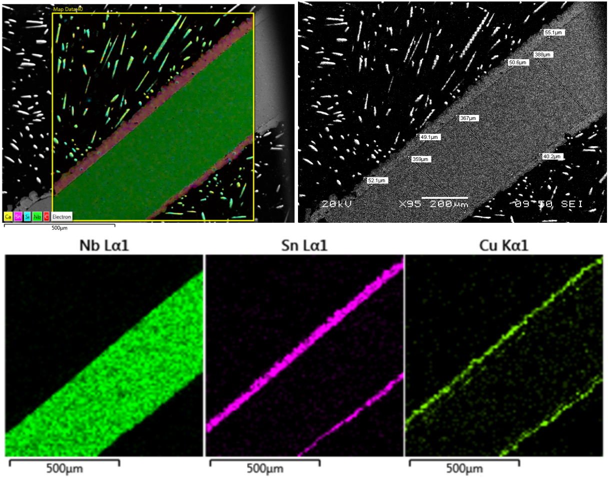
$$J_1^* = 20mA/cm^2 \quad J_2^* = 35mA/cm^2 \quad J_3^* = 15mA/cm^2$$

Sample 10

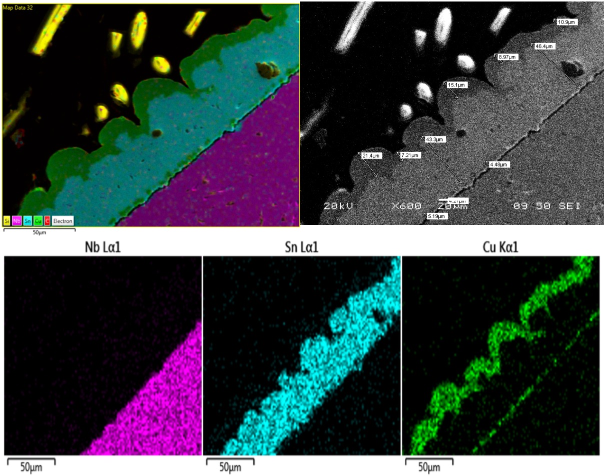


Heat Treatment

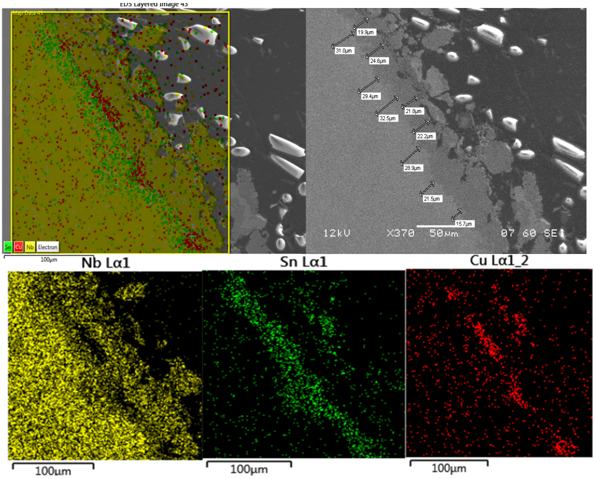
Sample 10 - SEM before heat treatment



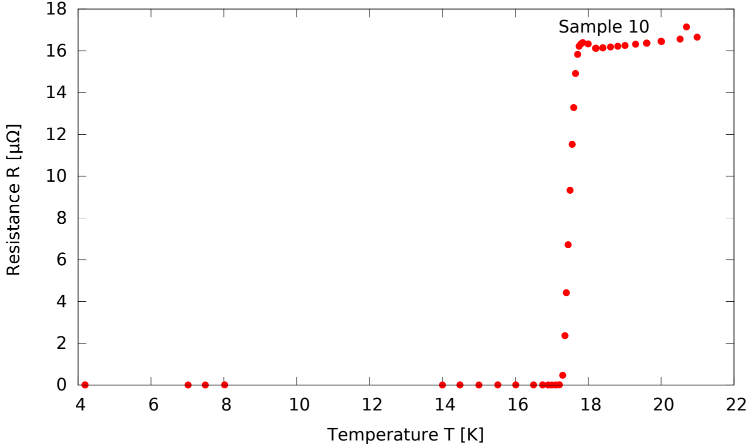
Sample 10 - SEM before heat treatment



Sample 10 - SEM after heat treatment



Sample 10: Resistance vs Temperature



Phase Diagram I_c - B

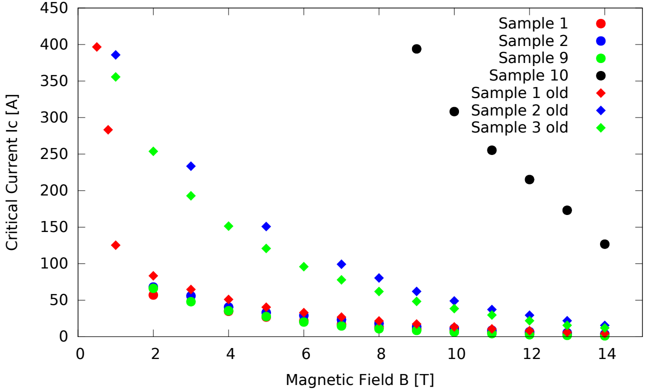
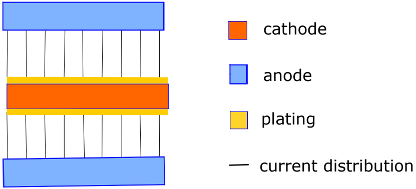


Figure: Critical current $\mathrm{I_c}$ vs magnetic field B at $4.2\mathrm{K}$. $\mathrm{I_c}$ is expressed in A; B is expressed in T. Data collected by D. Turrioni.

POTENTIAL FOR RESEARCH AND APPLICATIONS

Further Possible Improvements

- Use a double anode geometry cell
- Estimation of deposition rates
- Optimize further the parameters



Superconducting wires

- Use Nb_3Sn films to research and investigate nanotechnology methods to enhance the transverse component of pinning.
- If a technique proves to be successful, work with billet manufacturing company to transfer and implement it in wires.

Superconducting magnetic shields

- Shielding capabilities depend on the total superconducting thickness of the material. For stability, each superconducting shield layer cannot be thicker than a given value.
- A multi-layer shield can be obtained by replicating the $\mathrm{Cu/Sn}$ deposition method on sputtered Nb until the desired total thickness is achieved.

SRF cavities (High Q, High gradient and low cost)

- Produce samples of appropriate geometry (2 inch discs) for surface resistance measurements at JLab. Surface resistance relates to Q. A good Q is necessary condition for implementation to SRF cavities.
- Based on results achieved, scale up the technique to 3D surfaces, using the appropriate design of the anode and of customized galvanic cells as well as the appropriate software.

THANK YOU !!!

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