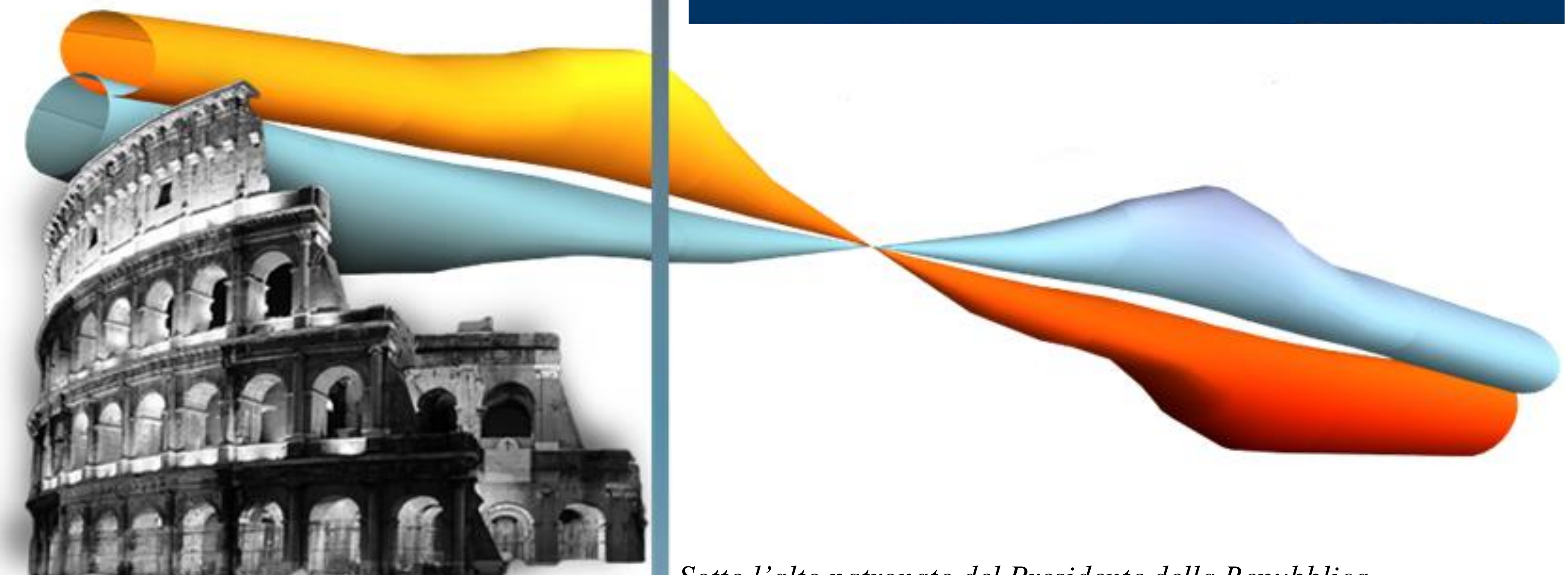
Innovative Nb_3Sn Thin Film Approaches and their Potential for Research and Applications, FNAL

FCC Week 2016
ROME 11-15 April 2016

POLITECNICO DI MILANO

Fermilab



Sotto l'alto patronato del Presidente della Repubblica

Introduction

❖ This proposal capitalizes in part on the 15-year effort at Fermilab in Nb3Sn research for high field accelerator magnets, which built the first reproducible series in the world of 10 to 12 T accelerator-quality dipoles and quadrupoles made of Nb3Sn.

❖ Originally based on a 2001 phenomenological model by J. McDonald and E. Barzi, it started as an ANL User Program in 2012 via a graduate student from the Politecnico di Milano (POLIMI) jointly mentored by a group leader at ANL and myself:

- Andrea Pisoni – 2012, Milan Polytechnics: "*Implementation of Nano Scale Formations in A15 Brittle Superconductors*", Prof. Riccardo Bertacco, E. Barzi, Tijana Rajh Advisors).

❖ The research was then continued with a group of experts from POLIMI led by Prof. M. Bestetti, and with the next student we were able to produce Nb_3Sn films 5 μm thick on a Nb substrate using electrochemical deposition:

- Federico Reginato – 2014, Milan Polytechnics: "*Electrochemical Synthesis of Nb-Sn Coatings for High Field Accelerator Magnets*", Prof. Silvia Franz, E. Barzi Advisors).

❖ While performing this work the recipe for direct deposition on Cu was also discovered:

- Luigia Glionna – 2015, Milan Polytechnics: "Electrochemical Deposition of Nb-Sn Films", Prof. Silvia Franz, E. Barzi Advisors.

❖ The best yet demonstration of superconducting RF properties of Nb3Sn has been its growth via high temperature vapor diffusion of Sn onto bulk Nb cavity structures followed by thermal reaction. This work was started in the 1980s by Univ. Wuppertal and has recently been replicated and further developed at Cornell University and at Thomas Jefferson National Accelerator Facility (JLab). High field performance is yet less than desired due to Nb rich regions. There is no precedence for forming Nb3Sn on Cu substrates.

Summary of work

Two different electro-chemical deposition techniques, both under US Patent Application No. 62/190, 199, to produce Nb_3Sn coatings have been developed in the last few years by FNAL in collaboration with Politecnico di Milano.

- **In the first technique, the Nb_3Sn phase is obtained by electrodeposition from aqueous solutions of Sn layers and Cu intermediate layers onto Nb substrates followed by high temperature diffusion in inert atmosphere. Subsequent thermal treatments are realized at 700°C to obtain pure Nb_3Sn superconducting phase, so far between 5.7 and 8.0 μm in thickness. All samples showed superconducting transport behavior, with a maximum obtained T_c of 17.68 K and B_{c20} ranging between 22.5 T and 23.8 T.**

"Synthesis of Superconducting Nb_3Sn Coatings on Nb Substrates", E. Barzi, M. Bestetti, F. Reginato, D. Turrioni and S. Franz, Supercond. Sci. Technol. 29 015009.

- **In the second technique, the synthesis of Nb-Sn coatings was carried out on Cu substrates by direct electrodeposition from 1-Butyl-3-methylimidazolium chloride (BMIC) ionic liquids at 130°C containing $SnCl_2$ and $NbCl_5$. No heat treatment whatsoever is necessary in this technique. The electrodeposited coatings showed a cubic Nb_3Sn phase with (211) preferred orientation, a disordered orthorhombic $NbSn_2$ phase and Sn-Cu phases. Film thickness was 200 to 750 nm in direct current mode and 500 to 1600 nm in pulsed current mode.**

"Electrochemical Synthesis of Nb3Sn Coatings on Cu Substrates", S. Franz, E. Barzi, D. Turrioni, L. Glionna, M. Bestetti, online on Sep. 11, 2015. Materials Letters, Volume 161, 15 December 2015, Pages 613–615.

Literature on Deposition of Nb_3Sn Coatings from Molten Salts

Kolosov, V.N. and Matychenko, E.S., Evaluation of High Frequency Superconductivity of Niobium Coatings Prepared by Electrodeposition Process in Molten Salts, in Refractory Metals in Molten Salts , Dordrecht: Kluwer, 1998, pp. 231–238;

V. N. Kolosov and V. Yu. Novichkov, Zero-Current Deposition of Superconducting Nb3Sn Coatings from Molten Salts, Inorganic Materials, Vol. 39, No. 5, 2003, pp. 485–491. Translated from Neorganicheskie Materialy, Vol. 39, No. 5, 2003, pp. 583–590

V. N. Kolosov and A. A. Shevyrev, Effect of Heat Treatment on the Structure and Properties of Superconducting Nb and Nb3Sn Electrodeposits, Inorganic Materials, Vol. 40, No. 3, 2004, pp. 235–240. Translated from Neorganicheskie Materialy, Vol. 40, No. 3, 2004, pp. 286–291.

Presence of superconductive Nb3Sn phase.

Deposition T in the range 400-1000 K

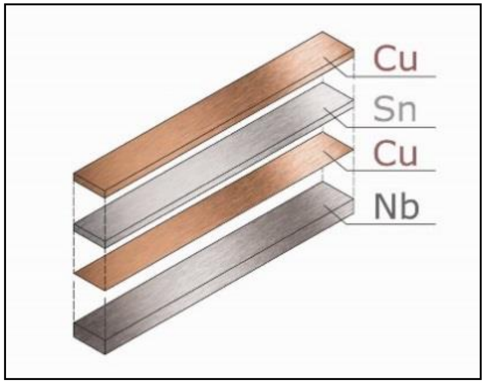
Residual pressures no higher than 2.5 Pa

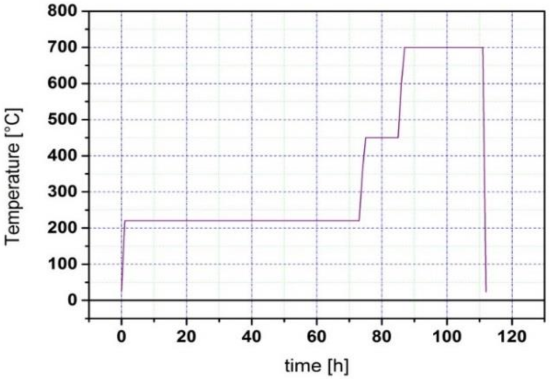
Most of the common advantages of electroplating (i.e. operation at room or near-room temperatures and atmospheric pressure, use of several substrate materials) are lost.

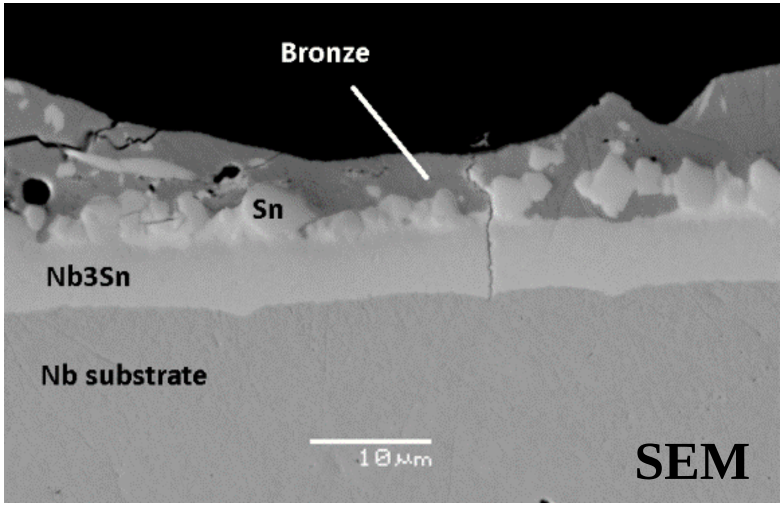
A GOOD ELECTROCHEMICAL PROCESS FOR THE SYNTHESIS OF Nb3Sn COATINGS IS STILL MISSING

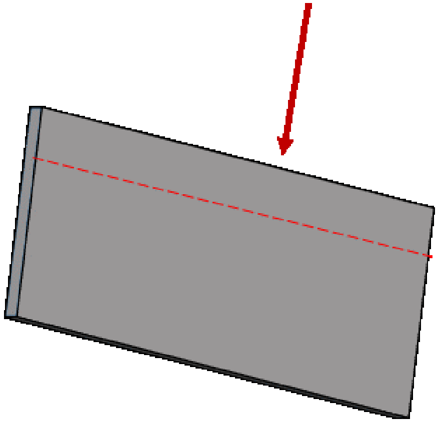
Nb$_3$Sn Thin Films on Nb

The Nb$_3$Sn phase is obtained by electrodeposition of Sn layers and Cu intermediate layers onto Nb substrates followed by high temperature diffusion in inert atmosphere. Electrodeposition was performed from aqueous solutions at current densities in the 20 to 50 mA/cm^2 range and at temperatures between 40 and 50°C. Subsequent thermal treatments were realized to obtain the Nb$_3$Sn superconductive phase.

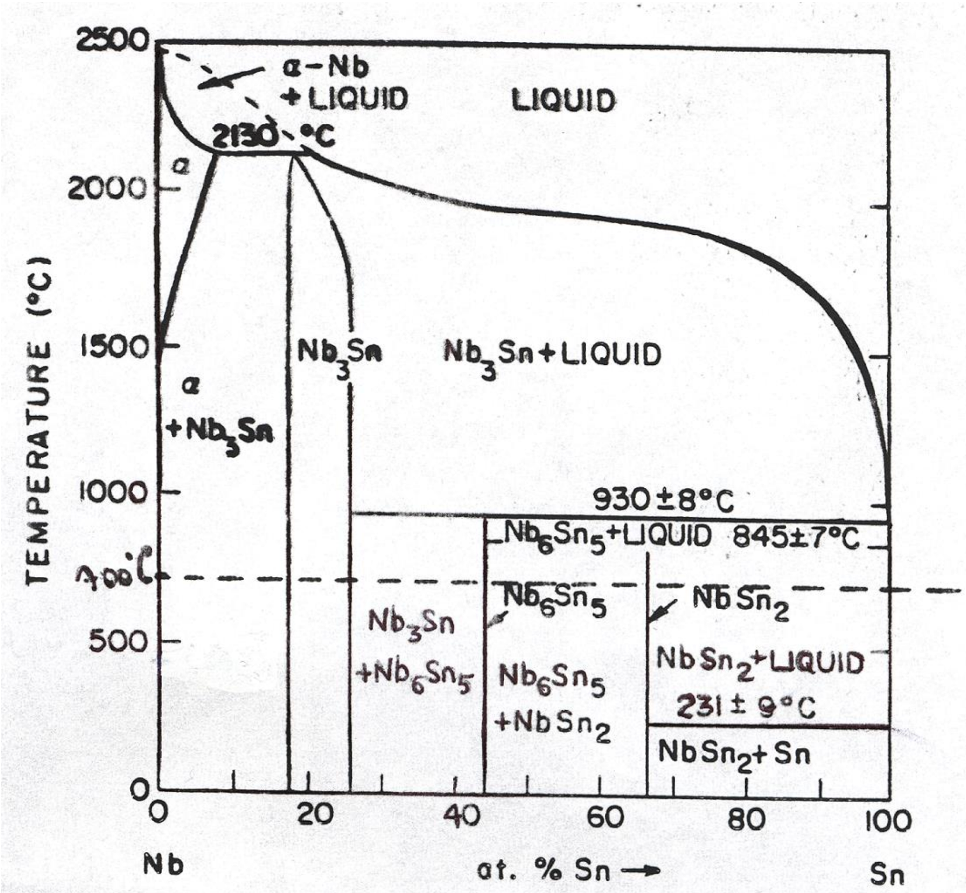






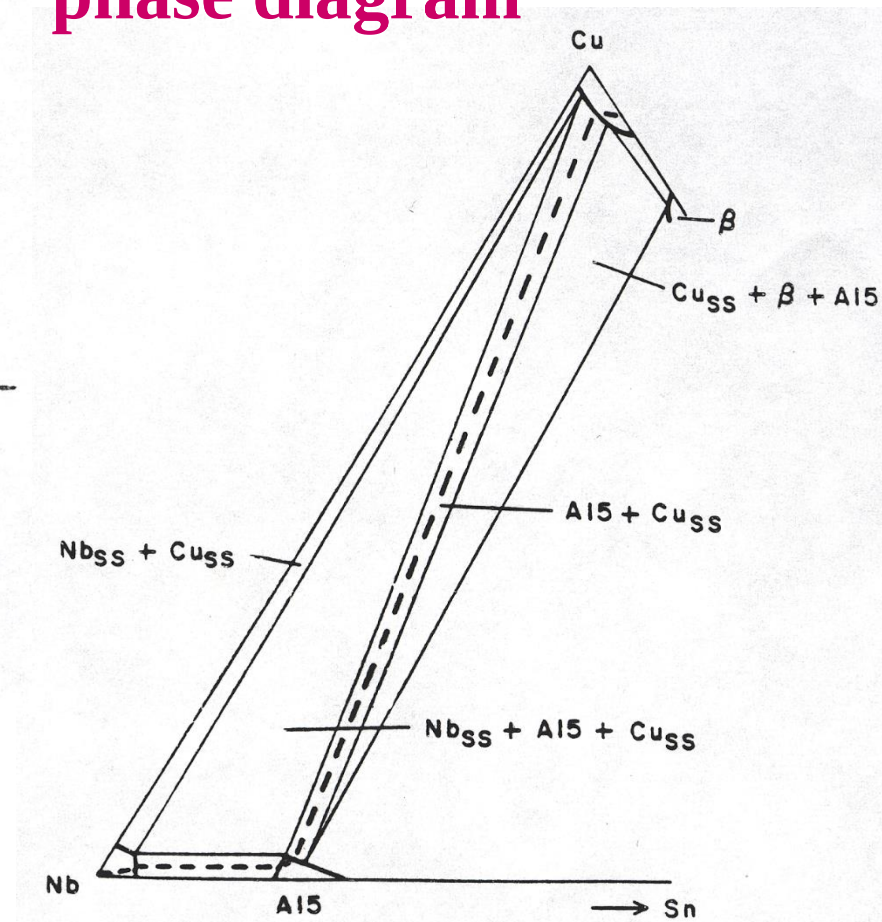


Nb_3Sn Phase Diagram

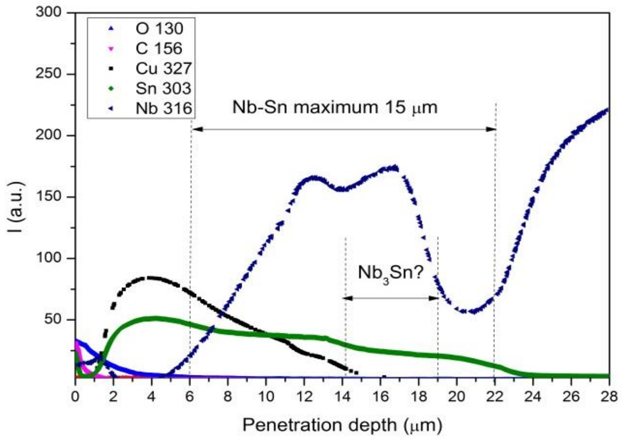


Nb-Sn binary phase diagram

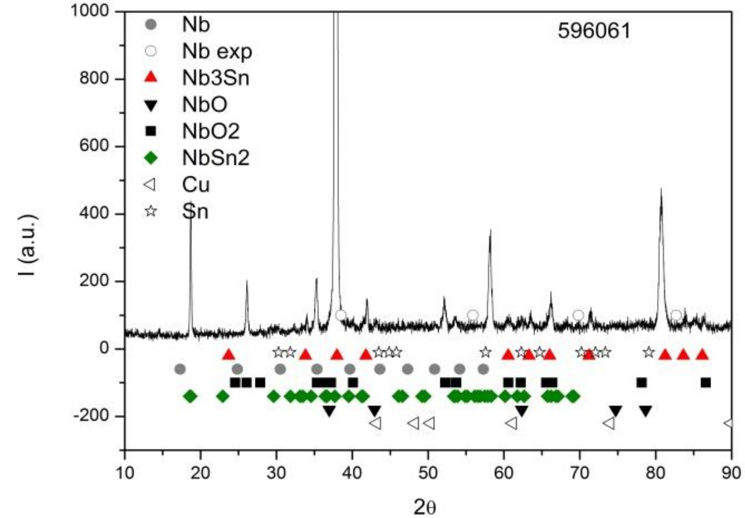
Nb-Cu-Sn ternary phase diagram



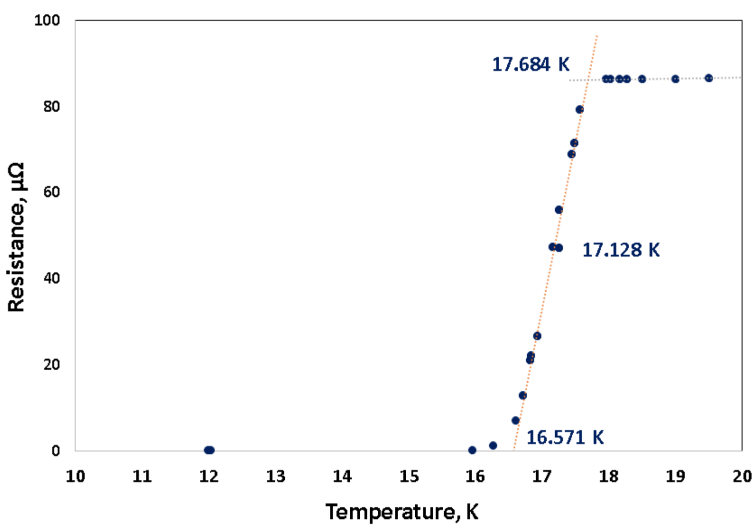
Nb_3Sn Thin Films on Nb - Results

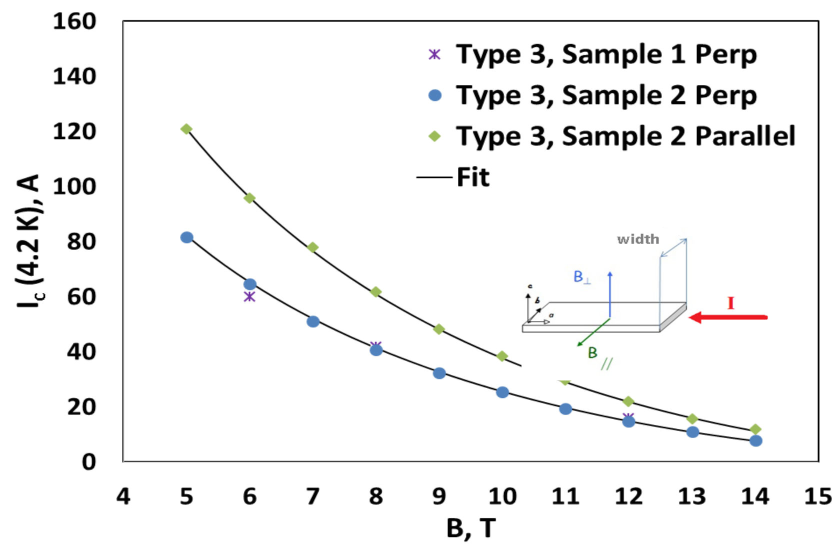


Glow Discharge Optical Emission Spectrometry



X-ray Diffraction





The maximum obtained T_c was 17.68 K and the B_{c20} ranged between 22.5 T and 23.8 T

Literature on Electrodeposition of Nb_3Sn Coatings from Ionic Liquids

Ito H. Koura N., Ling G. Electrodeposition of Nb-Sn alloy from ambient temperature molten salt electrolytes by pulse electrolysis, Hyoumen Gijutsu, 48 (4), 454 – 459 (1997)

N.Koura, T Umebayashi, Y Idemoto and Gouping Ling, Electrodeposition of Nb-Sn Alloy from SnCl2-NbCl5-EMIC Ambient Temperature Molten Salts, Electrochemistry, 67(6), 689(1999).

N.KOURA, K.SHIBANO, F.MATSUMOTO, H.MATSUZAWA, T.KATOU, Y. IDEMOTO and G. LING, Electrodeposition of Nb3Sn Alloy Superconductor from NbC15-SnC12-EMIC Room Temperature Molten Salt, Hyoumen Gijutsu 52 (9) 645-646 (2001).

Nb content from 33at% to 74at%, presence of superconductive Nb3Sn phase.

The same authors say that THE PROCESS IS NOT STABLE AND NOT REPRODUCIBLE

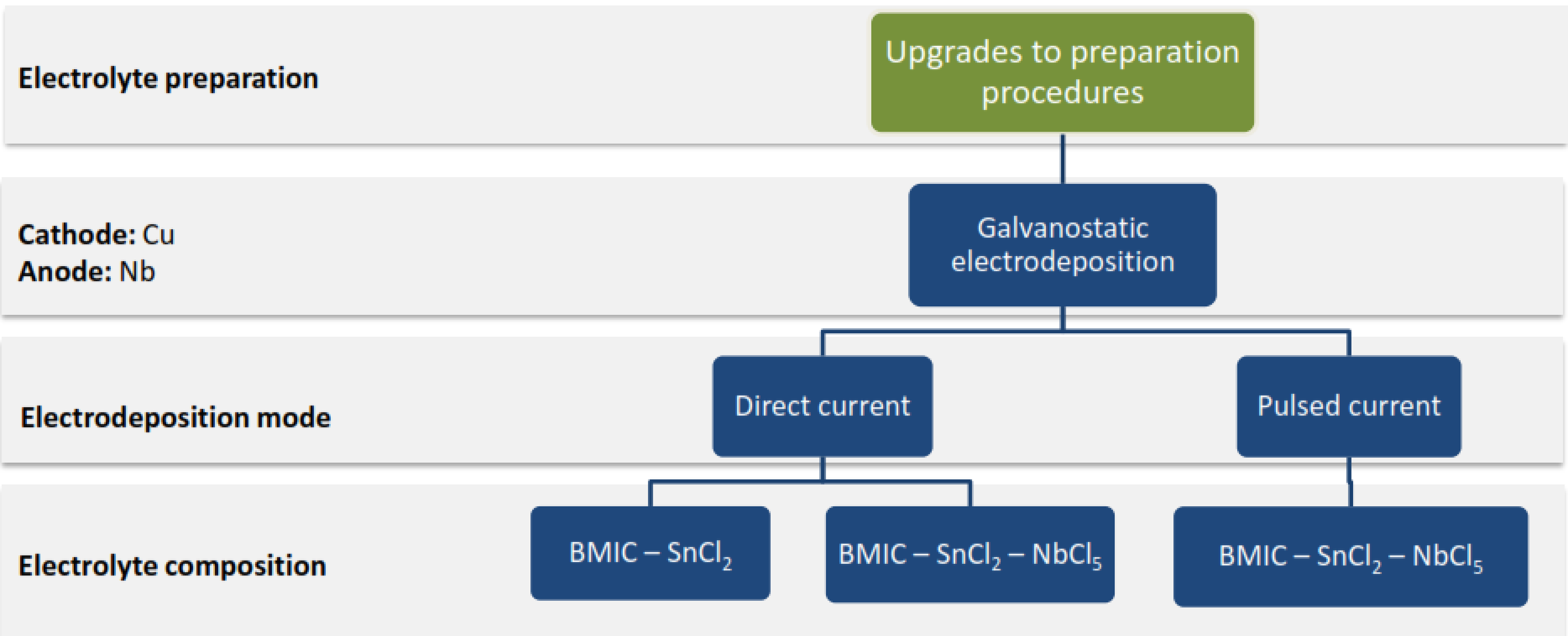
Koichi Ui, Sakai H., Takeuchi K., Ling G., Koura N., Electrodeposition of Nb3Sn Alloy Film from Lewis basic SnCl2-NbCl5-EMIC melt, Electrochemistry, 77 (9) 798-800 (2009).

Presence of Nb3Sn phase by XRD analysys.

Nb content not specified
No electrical measurements

Nb_3Sn Thin Films on Cu - Methods

Experimental plan



Nb_3Sn Thin Films on Cu – Latest Results

Pulse current mode

85 mol% BMIC – 10 mol% $NbCl_5$ – 5 mol% $SnCl_2$ electrolyte

SINGLE CURRENT STEP with T=50 ms

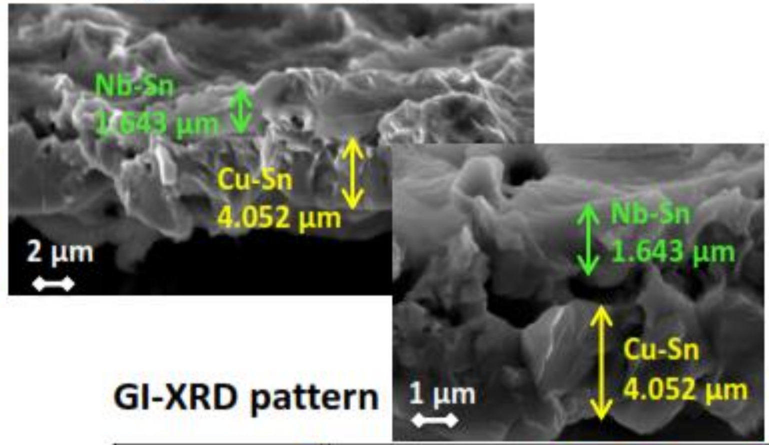
i_{on}=150 mA/cm²

i_{off}=15 mA/cm²

Q=340.2 C/cm²

$\delta = t_{on}/T = 0.2$

Reproducibility Verified

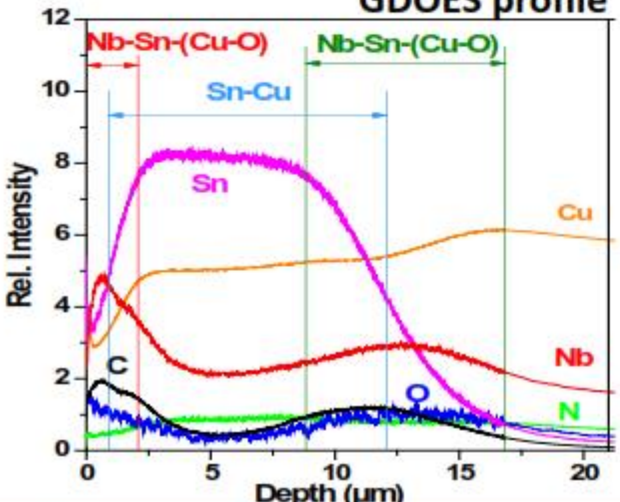


EDX: Cross section composition

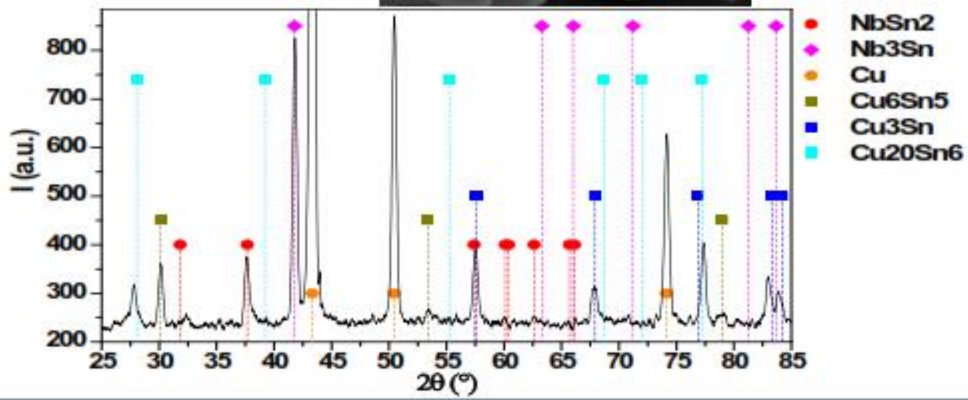
spt 2	Nb	Sn
at.%	87	13

spt 1	Cu	Sn
at.%	74	26

GDOES profile



GI-XRD pattern



Conclusions and Challenges

- Electrodeposition of Sn onto Nb substrates followed by thermal treatment produced Nb3Sn films between 5.7 and 8.0 μm in thickness, with a maximum obtained Tc of 17.68 K and Bc20 ranging between 22.5 T and 23.8 T. As electrochemical deposition is scalable to 3D irregular shapes, this allows for the first time to use superconductors as surface coatings as opposed to bulk, wires and cables.

- Direct electrodeposition of Nb_3Sn alloys on Cu can be carried out from ionic liquids. The coating of Cu with Nb_3Sn is unique in that it had been previously attempted unsuccessfully. Also, this is the first time that Nb3Sn is reproducibly formed in molecular form and at much lower temperatures than in any other known method, which is by solid diffusion. However, the Nb3Sn phase composition and homogeneity needs further improvement.

- These techniques could be attempted to produce high performance superconducting magnetic shields for accelerator magnets, MRI, MAGLEV and other applications and/or deliver high performance superconducting RF cavities for linear and circular accelerators.

- For applications, the main technological challenge to be solved is producing the superconducting coatings on 3D geometries. Electrochemical deposition is controllable