Evolution of nano-particles doping in Nb$_3$Sn wires

1Mattia Ortino
2S. Pfeiffer, 1T. Baumgartner, 3M. Sumption,
4X. Xu, 5X. Peng, 2J. Bernardi, 1M. Eisterer

1Atominsttitut, TU Wien (Vienna, AT)
2USTEM, TU Wien (Vienna, AT)
3The Ohio State University (Columbus, USA)
4Fermilab (Batavia, USA)
5Hyper Tech Research Inc. (Columbus, USA)

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Outline

• Introduction
  o $\text{Nb}_3\text{Sn}$ for FCC-hh
  o Internal oxidation method
  o Pros and cons

• Experimental results
  o $B_{c2}$
  o $J_c$
  o Microstructure
  o Pinning
  o Local properties: radial inhomogeneities

• Conclusions
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Introduction: Nb$_3$Sn for FCC-hh

The h(adron)-h(adron) Future Circular Collider (FCC)- CERN, Geneva (CH)

![Future Circular Collider diagram](image)

Cos$\theta$ – configuration
16T dipole example

Nb$_3$Sn: the best conductor candidate

<table>
<thead>
<tr>
<th>RRP</th>
<th>Nb$_3$Sn: the best conductor candidate</th>
</tr>
</thead>
<tbody>
<tr>
<td>PBT</td>
<td>RRP</td>
</tr>
<tr>
<td>A1775 outer ring</td>
<td>Nb$_3$Sn</td>
</tr>
<tr>
<td>Ferrimagnetic Iron</td>
<td>Cu wedges</td>
</tr>
<tr>
<td>Austenite Steel (316 LN) pad</td>
<td>Austenite steel keys</td>
</tr>
<tr>
<td>Ti6Al4V poles</td>
<td></td>
</tr>
</tbody>
</table>

FCC-Goal:
non-Cu $J_c=1.5$ kA/mm$^2$ (16 T, 4.2K)

State-of-the-art Nb$_3$Sn wires performances insufficient: a final boost is needed

<table>
<thead>
<tr>
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</tr>
</thead>
<tbody>
<tr>
<td>0.7 mm RRP</td>
<td>2676, 68</td>
<td>1410, 58</td>
<td>24.5, 0.39</td>
<td>1298, 55</td>
<td>610, 47</td>
<td>&gt;100</td>
</tr>
<tr>
<td>0.85 mm RRP</td>
<td>2835, 44</td>
<td>1601, 33</td>
<td>25.9, 0.19</td>
<td>1289, 36</td>
<td>785, 25</td>
<td>&gt;100</td>
</tr>
<tr>
<td>0.85 mm Bundle Barrier PIT</td>
<td>2323, 83</td>
<td>1342, 49</td>
<td>26.7, 0.1</td>
<td>1093, 40</td>
<td>688, 26</td>
<td>&gt;150</td>
</tr>
</tbody>
</table>

Courtesy of M. Boscolo, INFN (IT)

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1) O-source \[ \text{SnO}_2 \text{ powder (located between the Cu/Sn core and Nb–1Zr)} \]
2) Nb–1wt% Zr alloy \[ \text{Zr has much stronger affinity to O than Nb: oxidation of the alloy is possible} \]
3) Reaction temperature = 620°-700°

Present-day PIT sub-element:

APC-PIT sub-element:

End-2018: From binary to ternary compounds

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Introduction: pros and cons

Pros:
• Nanoparticles catalysing A-15 grain size refinement, hereby increasing $J_c\ (J_c = f(1/d_{\text{grain}}))$;
• ZrO$_2$ nanoparticles to become as well additional pinning centres (intra-granular);
• Ta addition to raise $B_{\text{irr}}$ and $B_{c2}$ of the superconducting phase;

But...Nb$_3$Sn formed by diffusion reaction: Sn diffuses outwards into a Nb tube (PIT) or -future possibility- in a region containing densely stacked Nb filaments (RRP)

Cons:
• Radial gradient in stoichiometry always present$^2$, specially in compounds doped with Ta$^3$
• Other types of inhomogeneities - sub-element sausaging or barrier breakage - may also occur

Inhomogeneities affect the superconducting performance: $B_{c2}, J_c$ and local properties need to be assessed, relating them to the microstructure


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Experimental results: $B_{c2}$

**Binary**-samples $B_{c2}$ were measured in a 17 T cryostat via resistive method (values extrapolated to low temperatures).

<table>
<thead>
<tr>
<th>Sample type</th>
<th>$B_{c2}$ [T]</th>
</tr>
</thead>
<tbody>
<tr>
<td>T3607-mono</td>
<td>22.1</td>
</tr>
<tr>
<td>T3657-multi</td>
<td>22.6</td>
</tr>
<tr>
<td>T3680-mono</td>
<td>23.4</td>
</tr>
<tr>
<td>T3682-mono</td>
<td>22.3</td>
</tr>
</tbody>
</table>

Still low $B_{c2}$ values (at 4.2 K)

**Ternary** (+ Ta)-samples $B_{c2}$ were measured in a 31T cryostat via resistive method at NHMFL in Jan 2019.

Ta-doping raised $B_{c2}$ of APC-samples (20% to 32%): high field performance of the high-$J_c$ binary samples is expected to improve.

<table>
<thead>
<tr>
<th>Diameter [mm]</th>
<th>Characteristics</th>
<th>$B_{c2}$ [T]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.71</td>
<td>Nb-4at.%Ta-1at.%Zr tube + SnO2 powders</td>
<td>27.3</td>
</tr>
<tr>
<td>0.71</td>
<td>Nb-4at.%Ta-1at.%Hf tube + SnO2 powders</td>
<td>26.7</td>
</tr>
<tr>
<td>0.84</td>
<td>Nb-4at.%Ta-1at.%Zr tube + SnO2 powders</td>
<td>27.8</td>
</tr>
</tbody>
</table>

$B_{c2}$ values used for $J_c$ extrapolation at TU Wien.

Courtesy of X. Xu, Fermilab
Experimental results: $J_c$

Low field data (up to 7 T) measured in a SQUID, then extrapolated to $B_{c2}$ ($F_p(B)$ dependence).

Resistive measurements at NHMFL match the extrapolation: the FCC target at 16 T is reached!

What has been improved?

1) Ta gives a different high field behaviour
2) Better Cu/non-Cu ratio: present wires have 1.3 (previous generation had between 2.5 and 3.3)
3) Microstructure? Was the grains size further refined?

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Experimental results: microstructure

**Binaries (TT) best grain size modal value: ~ 60 nm**

- Grain size refinement not affected by Ta-doping
- Likewise, nanoparticles do not show a size change

**Ternaries (PIT) best grain size modal value: ~ 50 nm**

- Still no preferential deposition

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Experimental results: Pinning

Single contributions of pure GB- pinning and point-pinning were investigated.

From literature (Bronze-route + PIT), the achieved grain-size refinement would be enough to explain the $F_{p,\text{max}}$ values obtained (Commercial-PIT values consistent).

Ternary samples exhibit a reduction of the $F_{p,\text{max}}$ but (as expected) a further peak-shift is visible.
Experimental results: Pinning

- Elementary pinning force approach:
  \[ f_{p,max} \cdot \rho_{defects} \]
  \[ F_{p,max \ (model)} = 51\% F_{p,max \ (exp.)} \]

How to weight GB and point pinning contribution still work in progress!

Maximum shift in ternaries:
\[ b(f_{\text{max}}) = 0.25 \ (\text{Hf-doped samples}) \]
\[ b(f_{\text{max}}) = 0.22 \text{ in binaries} \]

Peak shifts suggest a point pinning contribution (also saw in n-irradiation studies) but its evaluation is difficult

- **Dew Hughes approach:**
  \[ F_p = \eta L f_p = -\eta L \Delta W/x \]
  \[ F_{p,max \ (model)} = 16.8\% F_{p,max \ (exp.)} \]

- Parameters:
  - \( d_{avg} = 4.5 \text{ nm} \)
  - \( \rho_{defects} = 25.000 \mu m^{-3} \)
  - \( l_{avg} \approx 30 \text{ nm} \)


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Experimental results: radial inhomogeneities

Assessing $T_c$ distribution $\equiv$ radial A-15 inhomogeneities (coarse to fine grain size region). AC-susceptibility method (SQUID) was used to identify the Meissner shielding contours.

**Ingredients:**
- SQUID or Scanning Hall probe microscopy (SHPM)
- $B_{app} < B_{c1}$ (Meissner state)
- No other magnetic signals
- Temperature sweep
- Thin and flat sample for SHPM

With $T_{sample}$ increasing, a shrink of the Meissner shielding volumes is expected.

Radii($T$) of the single sub-elements are then converted to relative position inside the inner and outer radius of the A-15 region.

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Experimental results: radial inhomogeneities

Evaluation model based on some assumptions:

1. Sub-elements inside the sample are parallel tubes with circular cross sections;

2. All sub-elements are identical (geometry/composition);

3. Each sub-element exhibits a monotonic radial Sn gradient with the highest value on the inside.

Simulation runs on a single sub-element by changing its radial $T_c$ distribution until the computed $m(T) \equiv m_{exp}(T)/N$.

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Experimental results: radial inhomogeneities

Radial inhomogeneities → radial Sn content:

For a more accurate analysis, the effective boundaries of the single sub-elements A-15 edges were evaluated by means of pixel counting.

$T_c(\beta) = \frac{T_{c, min} - T_{c, max}}{1 + e^{(\beta - \beta_0)}} + T_{c, max}$

EDX and magnetic evaluations show similar behaviour but different absolute values.

Conclusions

• APC-Nb$_3$Sn wires produced with 4at.%Ta additions confirm their high $J_c$ achievements (beyond FCC-goals), as well by means of magnetometry;

• 1at.%Hf+O doped sample shows similar performances if compared with the 1at.%Zr-doped ones (even better homogeneity);

• Peak shifts in $f_p$ show a possible point pinning contribution: a further investigation is needed;

• Microstructure has not changed in ternary compounds: same grain and nano-particle size as in the binary generation, still no preferential deposition;

• Inhomogeneities: more accurate investigation of the model (inter-granular gradient to be raised/lowered) or of the $T_c$-Sn% (still referring to binary compounds) is needed;

• Further $T_c$ distribution analysis with SHPM coming: difficult to perform but with less restrictions than AC-susceptibility.
Thanks for your attention!
$F_p(B) = F_{pmax} \times C \times \left( \frac{B}{B_{c2}} \right)^p \times \left( 1 - \left( \frac{B}{B_{c2}} \right) \right)^q$

<table>
<thead>
<tr>
<th>Sample</th>
<th>$B_{c2\text{avg}}$</th>
<th>$p_{\text{avg}}$</th>
<th>$q_{\text{avg}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>T3657 (binary)</td>
<td>22.6</td>
<td>0.71</td>
<td>2.19</td>
</tr>
<tr>
<td>T3682 (binary)</td>
<td>23.4</td>
<td>0.64</td>
<td>2.15</td>
</tr>
</tbody>
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<table>
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<tr>
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</thead>
<tbody>
<tr>
<td>T3912 $d=0.71$</td>
<td>27.3</td>
<td>0.68</td>
<td>2.28</td>
</tr>
<tr>
<td>T3914 (Hf-sample)</td>
<td>26.8</td>
<td>0.73</td>
<td>2.29</td>
</tr>
<tr>
<td>T3912 $d=0.84\text{mm}$</td>
<td>27.8</td>
<td>0.68</td>
<td>2.32</td>
</tr>
</tbody>
</table>
• Elementary pinning force approach:

$$f_{p, \text{max}} \cdot \rho_{\text{defects}}$$

$$f_{p, \text{max}} = \frac{U_{p, \text{max}}}{\xi} = \frac{\mu_0 H_c^2}{2} \frac{4}{3} \pi r_p^3$$

• Dew Hughes approach:

$$F_p = \eta L f_p = -\eta L \Delta W / x$$

$$F_p(B) = \frac{BV_f}{\Phi_0} \cdot \frac{\pi \xi^2 (H_{c2} - H)^2}{4.64k^2}$$

$$\frac{r_p}{2}$$