Development of Nb$_3$Sn for FCC at BRUKER

27.06.2019 / Brussels / C. Bühler
This work is supported by CERN under FCC-GOV-CC-0157 KE4187/TE/FCC
Key Topics of the Nb$_3$Sn Development Program

**Conductor Design**
- PIT/RRP® Hybrid
- **Internal Oxidation**
- Other APC Approaches

**Metal Forming**
- Powder Rheology
- Filament Roundness
- Heat Management

**Cost Savings**
- Pre-Materials
- High-Speed Processing
- Material Efficiency

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Start Date
Nov. 2018

Kickoff
Feb. 2019

End Date
Nov. 2020

Brussels, 27.06.2019
Four monofilament layouts that include an oxygen source (red) at different locations
Each with different diffusion pathways for Sn and oxygen
Each with different core compositions

**Broad spectrum of compositions**

Presentation will focus on -core concepts

**intOx-core**
Sn and O source central + mixed

**intOx-ring**
Sn and O source central + separated

**intOx-shell**
Sn and O source fully separated

**intOx-split**
Sn and O source fully separated
Results: Screening Conductors Morphology

- The filament type is intOx-core where O- and Sn-source are mixed centrally
- Six core-compositions were tested in Nb1Zr and Nb2.5Ti each
- Huge changes in morphology can be observed
  - Dark, Nb-rich oxide ring around bronze droplet
  - Suppressed coarse grain and core A15 fraction
  - Surprisingly large fine grain areas

Nb1Zr monofilaments after heat treatment for 300 h at 640°C
**Results: Screening Conductors**

**Morphology O-high**

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**Area Analysis**

- Oxidation of Nb-Sn intermetallics prior to Nb₃Sn formation
- This releases otherwise bound Sn
- High fine grain fractions ~ 41% of filament area and low residual Nb (24-26%)

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**EDX**

- Migration of alloying element into core. Low solubility in Nb₃Sn
- Bronze core can deplete in Sn despite the oxide ring
- Probable correlation between oxygen and Sn content

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*Strictly Confidential*

Hanau, 15.02.2019
Brussels, 27.06.2019
Results: Screening Conductors
Grain Size

- No grain refinement by internal oxidation in intOx-core concepts
- Probable cause is formation of oxygen-impermeable NbSn₂ / Nb₆Sn₅ layer at low temperatures
  - No shift in the Nb-\( T_c \) can be observed by VSM
  - No Oxidation of Nb-alloy prior to Nb₃Sn phase formation

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Results: Screening Conductors Precipitates

- Precipitates on grain boundary surfaces
  - Equivalent precipitate Diameter: 7 nm
  - Average neighbor distance: 55 nm
- No intracrystalline precipitates observed
- Assumed composition TiO$_2$
- Precipitates also observed for Nb1Zr
- TEM and APT analysis will show composition, interface and distribution
- Probably result of oxidation of Zr/Ti after Nb$_3$Sn formation
  - Therefore no effect on grain size

Do these precipitates act as artificial pinning centers?
**$I_c(B)$ Measurements**

**intOx-core**

- **Heat treatments**
  - A: 300 h 640 °C 30 K/h
  - B: 300 h 640 °C 300 K/h

- Kramer $B_{c2}$ is reduced due to lack of Ta dopant
  - PIT A: 27.0 T
  - intOx-core A: 22.3 T

- No correlation between oxygen content in core and $B_{c2}$

- Below 10.75 T higher non-Cu $J_c$ in oxidized sample*

- Plotted against $B/B_{c2} + 38\%$ increase in non-Cu $J_c$ at $b=0.5$ (vs. PIT reference at same HT)*

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* $J_c$ determined from filament area after heat treatment
**$I_c(B)$ Measurements**

intOx-core

- Normalized pinning curves indicate higher point pinning component
- Location on grain boundaries probably a hindrance for effective precipitate spacing
- No significant shift of $F_{p,max}$ to higher fields due to the lack of grain refinement
- At the same reduced field of $b=0.5$, $J_{c,layer}$ is increased by $\sim 40\%$
- Performance increase due to higher $J_{c,layer}$ and $\sim 10\%$ higher fine grain fraction

Ternary alloys are indispensible

WIP

Brussels, 27.06.2019
Summary and Outlook for internal oxidation

- Fine grain fraction increased by 10%
  - at $B/B_{c2}=0.5$
    - $J_{c,\text{nonCu}}+38\%$
    - $J_{c,\text{layer}}+40\%$

- Slight changes in pinning properties

- No grain refinement
- No shift of pinning curve
- Probably hindered $O_2$ diffusion due to intermetallics

- Already significant potential to improve state of the art PIT with intOx-core concepts
- w/o grain refinement, the potential of internal oxidation is not fully used

- intOx-ring/shell/split concepts separate the Sn and O source
- They aim at an uninhibited oxygen transfer before Nb-Sn intermetallics start to hinder the internal oxidation of the Nb alloy

- Designs with separated Sn and O sources are currently being manufactured
- Ternary alloys are needed to achieve appropriate $B_{c2}$

Brussels, 27.06.2019
Internal Oxidation is only one part of the R&D agreement

More information on:
- Conductor Homogeneity
- Hybrid Conductors
- Other APC Approaches
- Internal Oxidation w/ Ternary Alloys
- High Speed Processing

In the Future
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Oxidation of Nb-Sn intermetallics can lead to a more efficient release of Sn from a Sn-rich core:

- Example:

\[ \text{NbSn}_2 \rightarrow \text{Nb}_3\text{Sn} + 3\text{Sn} \quad / \quad 39,8 \, \frac{\text{mmol}}{\text{cm}^3} \]

Only releases 80% of all contained Sn

\[ \text{NbSn}_2 + \text{SnO}_2 \rightarrow \text{NbO}_2 + 3\text{Sn} \quad / \quad 47,5 \, \frac{\text{mmol}}{\text{cm}^3} \]

Releases 100% of all contained Sn
**Additional: intOx-core Phase Formation**

- **230°C**: intOx core
- **415°C**: Bronze mixing
- **500°C**: Nausite formation
- **625°C**: Nb$_6$Sn$_5$ Oxidation + Sn release
- **640°C**: Coarse grain Nb$_3$Sn

Reference and PIT identical. No signs for oxidation.

Deviating reaction path for *O-high-monos*.

* After adding 40 h at indicated temperature each
** After 300 h hold

Brussels, 27.06.2019
Additional: intOx-core VSM
## Additional: intOx-core

### Grain Sizes

<table>
<thead>
<tr>
<th></th>
<th>O-free</th>
<th>O-low</th>
<th>O-med</th>
<th>O-high</th>
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</thead>
<tbody>
<tr>
<td>Nb1Zr</td>
<td><img src="image1.jpg" alt="Image" /></td>
<td><img src="image2.jpg" alt="Image" /></td>
<td><img src="image3.jpg" alt="Image" /></td>
<td><img src="image4.jpg" alt="Image" /></td>
</tr>
<tr>
<td>Nb2.5Ti</td>
<td><img src="image5.jpg" alt="Image" /></td>
<td><img src="image6.jpg" alt="Image" /></td>
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400 nm