Root cause of large grain A15 formation in Powder In Tube Nb\textsubscript{3}Sn conductors

Chris Segal
September 5th, 2016
The FCC playground

LHC
27 km, 8.33 T
14 TeV (c.o.m.)
1300 tons NbTi

HE-LHC
27 km, 20 T
33 TeV (c.o.m.)
3000 tons LTS
700 tons HTS

FCC-hh
80 km, 20 T
100 TeV (c.o.m.)
9000 tons LTS
2000 tons HTS

FCC-hh
100 km, 16 T
100 TeV (c.o.m.)
6000 tons Nb₃Sn
3000 tons Nb²Ti
The practical HiLumi Nb$_3$Sn wire is in production!

**Practical wire**

\[ J_C(12T) > 2450 \text{ A/mm}^2 \]

\[ D_{\text{fil}} < 55 \mu\text{m} \]

\[ \text{RRR} > 150 \]
Practical wire

$J_C(16T, 4.2K) > 1500 \text{ A/mm}^2$

$D_{fil} < 20 \ \mu\text{m}$

$RRR > 150$
The Fractional Real Estate of PIT – only 40% is valuable

- Wire diameter: 0.78mm
- Filament diameter: 39 μm

- HT from BEAS:
  - 620/100 + 640/120

- $I_c = 501 \text{ A}$
- $J_{c\text{ non-Cu}} = 2237 \text{ A/mm}^2$
- $J_{c\text{-layer}} = 5564 \text{ A/mm}^2$

- Only **small grain A15** carries current
- **Large and core grains of A15** do not carry any current (seen on next slide)
Can **large grains** really carry current?

Why does the LG form?

Can we manage it’s formation?

Here we look at the phase evolution

**Cu-rich** phases penetrate between LG’s

LG A15 appears very disconnected

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Furnace set up for sample quenching

- Samples are pulled out of the furnace and quenched in cold water, indicated by vertical lines in the plot to the right.

- This rapid cooling ‘freezes’ the microstructure at the reaction temperature, allowing examination of phases and chemical compositions which are present.
Sn is rapidly absorbed by Cu sleeve to form \( \eta \) early in the reaction.

Original Nb tube is inscribed by a Cu sleeve, and the core filled with a Sn rich powder.
η transforms to ε at 408°C, Nausite grows as a continuous layer

- Thin, continuous ring of Nausite between Nb and the layer of η/ε phase.
- Since η decomposes at 415°C, ε begins forming

- η has entirely transformed into ε phase
- Nausite ring rows
Nausite decomposes above 560 °C and by 610 °C the Sn rich layer is entirely NbSn₂

520°C → 560°C

610°C

• Nausite ring grows

• Full layer of NbSn₂ forms from Nausite expelling Cu
At 630°C NbSn$_2$ rapidly transforms into Nb$_6$Sn$_5$

- The transformation occurs quickly; about 45 minutes at 630°C
- The Nb$_6$Sn$_5$ contains a few at% Cu
Continuous layer of small grain Nb$_3$Sn is formed by 5 hours

**630°C - 5hr**

- Nb$_6$Sn$_5$ draws Sn from core to Nb barrier
- Nb$_3$Sn
- Core

**630°C - 10hr**

- Nb$_6$Sn$_5$
- SG Nb$_3$Sn
- Nb$_3$Sn layer has grown
- Core
- Nb
LG A15 has formed with the ejection of Cu by Nb$_6$Sn$_5$

630°C - 12hr

- What role does Cu play in the reaction?
LG A15 grows with time, a Cu-rich phase separates the A15 layer from the core

630°C - 30hr

- Large grains of A15 are a continuous layer.
- All of the large grains are surrounded by a Cu-rich phase

630°C - 58hr

- There is a continuous layer of large grain A15 and penetrating Cu. A small amount of Nb₆Sn₅ remains
The Fractional Real Estate of PIT

- Wire diameter: 0.78mm
- Filament diameter: 39 μm
- Optimal HT from BEAS: 620/100 + 640/120

• \( I_c = 501 \) A
• \( J_{c \text{ non-Cu}} = 2237 \) A/mm\(^2\)
• \( J_{c \text{-layer}} = 5564 \) A/mm\(^2\)

- Only small grain A15 carries current
- Large and core grains of A15 do not carry any current.
Can we convert the poorly connected large grains and core grains of A15 into current carrying small grain A15 phase by only altering the heat treatment?
LG A15 formation is temperature dependent

Only SG A15 has formed

LG A15 has nucleated with Cu

Increasing Temperature

630C 10h

650C 4.75h

670C 4.5h

690C 1.66h

630C 12h

650C 8.33h

670C 5h

670C 2h

LG A15 formation with time and temperature
SG A15 forms *first* by $\text{Nb}_6\text{Sn}_5$ reacting with the tube. LG A15 forms by decomposition of $\text{Nb}_6\text{Sn}_5$.

- After 5 hours at 670°C, there is nearly a 2 μm thick layer of SG A15 as the LG forms

- We do not believe that LG A15 forms directly from the tube material
Can a multistep heat treatment suppress LG formation?
Microstructure is improved using multistep HT’s!

Previous HT’s

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Layer Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>630/12</td>
<td>0.8 μm</td>
</tr>
<tr>
<td>670/5</td>
<td>2.0 μm</td>
</tr>
<tr>
<td>A+630/13</td>
<td>2.2 μm</td>
</tr>
</tbody>
</table>

~ 10% improvement with first HT.

Step A + 630 °C 13h – 2.2 μm SG layer thickness!
Refining multistep HT’s increased SG layer thickness early in reaction

Previous HT’s
630/12 0.8 μm
670/5 2.0 μm
A+630/13 2.2 μm
C+630/10 3.0 μm

*Steps are at higher temperatures

50% improvement!

Step C + 630 °C 10h – 3.0 μm SG layer thickness!
Multistep Heat Treatments do well converting core and LG A15 to more SG A15

- Sample B31284, 0.78mm wire
- Heat treatments done at ASC
- Critical current measurements on short samples (~ 4cm) at 12T, 4.2K

<table>
<thead>
<tr>
<th>Heat treatment description</th>
<th>Heat treatment (temp/soak time)</th>
<th>$I_c$ (A)</th>
<th>$J_c$ (A/mm²)</th>
<th>$J_c$ SG-layer (A/mm²)</th>
<th>Nb</th>
<th>Total A15</th>
<th>core A15</th>
<th>LG A15</th>
<th>SG A15</th>
</tr>
</thead>
<tbody>
<tr>
<td>BEAS recommended</td>
<td>620/100+640/120</td>
<td>501</td>
<td>2237</td>
<td>5564</td>
<td>23.4%</td>
<td>56.0%</td>
<td>2.5%</td>
<td>13.3%</td>
<td>40.2%</td>
</tr>
</tbody>
</table>

Why?
- The steps before soaking at 630°C are higher temperature– producing larger grains of SG A15 than BEAS recommended HT
Summary of LG formation experiments

• Initial low temp HT at 630°C creates a thin layer of \textit{SG A15} before \textit{LG's} nucleate from the \textit{Nb}_6\textit{Sn}_5. (0.85\mu m)

• At higher temperatures (670°C), this \textit{SG} layer is 2x its low temp counterpart. (1.98 \mu m)

• Using multistep HT (630°C- 690°C), we can get 3x the layer thickness without \textit{LG} forming (3.0 \mu m)
Findings for discussion

The two main A15 morphologies form by different reaction paths

- Small grains of A15 form by Nb$_6$Sn$_5$ feeding Sn into the Nb(Ta) diffusion barrier.
  - SG A15 ALWAYS forms first.
- Large grains of A15 form mostly by decomposition of Nb$_6$Sn$_5$ in the core after some critical exhaustion point in the reaction.

More SG A15 forms with higher temperature reactions

- High temperature reactions tend to reduce core grains by allowing more of the Sn source in the core to react with the Nb-7.5wt.%Ta tube
  - Quench experiments show this happens in the early hours of the reaction
    - A short, high temperature spike early on gives the same effect even when soaking at low temperature for most of the reaction

We believe under these novel multistep heat treatments, the filaments can utilize more Sn efficiently, and by changing the ratio of Nb/Sn/Cu in the core, it may be possible to prevent the core Nb from nucleating large grains of A15 and drive up $J_c$. 

![Image](image.png)
Acknowledgements

David Larbalestier
Peter Lee
Chiara Tarantini
Amalia Ballarino
Luca Bottura
Christian Scheuerlein
Bernardo Bordini
Klaus Schlenga
Bernd Sailer
Manfred Thoener

This work was supported by the National High Magnetic Field Laboratory (which is supported by the National Science Foundation under NSF/DMR-1157490), the State of Florida, and CERN under grants KE1920/TE and RF02226
End of Presentation
675°C reacted for 4 hours – a much thicker SG layer has formed

This SG A15 layer looks much thicker than mine at 630 °C 12 hours...does the LG A15 form differently as a function of reaction temperature?