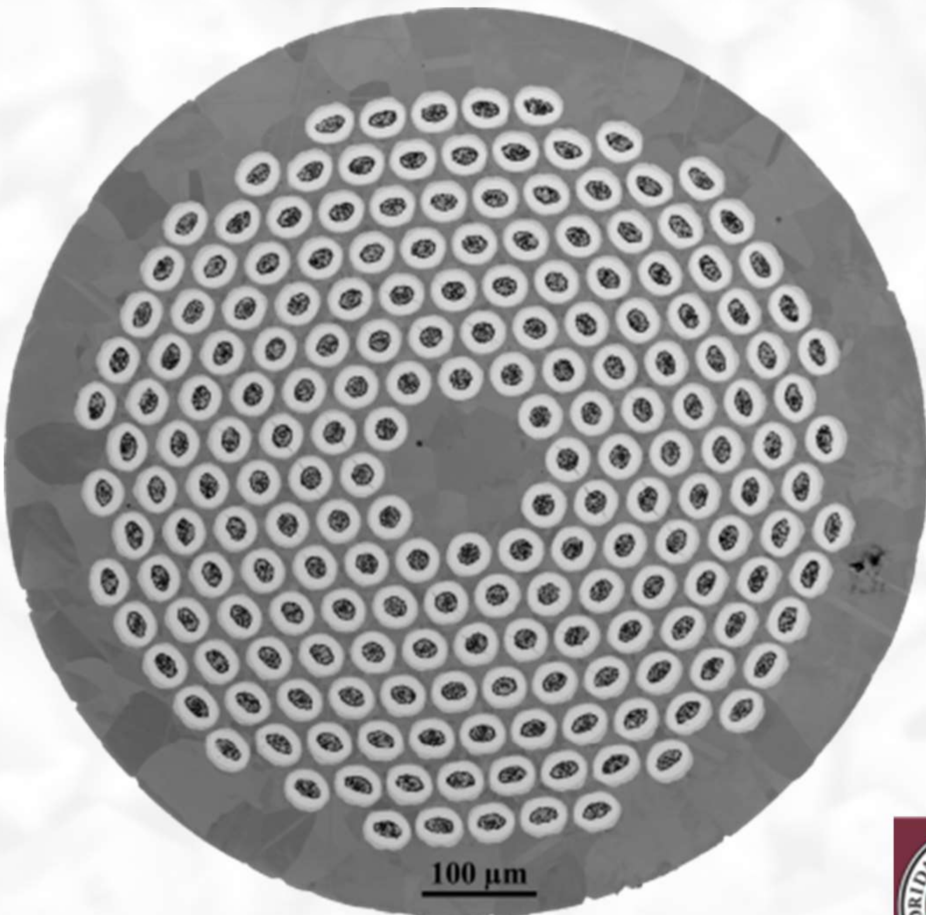
IEEE/CSC & ESAS SUPERCONDUCTIVITY NEWS FORUM (global edition), October 2016.
ASC 2016 paper 2MPo1C-07. ASC Victor Keilin Memorial Prize Winner.

Root cause of large grain A15 formation in Powder In Tube Nb_3Sn conductors



Chris Segal

September 5th, 2016

Florida State University 1851
ASC Applied Superconductivity Center
National High Magnetic Field Laboratory

Applied Superconductivity Conference, Denver, CO **Student Paper Competition**

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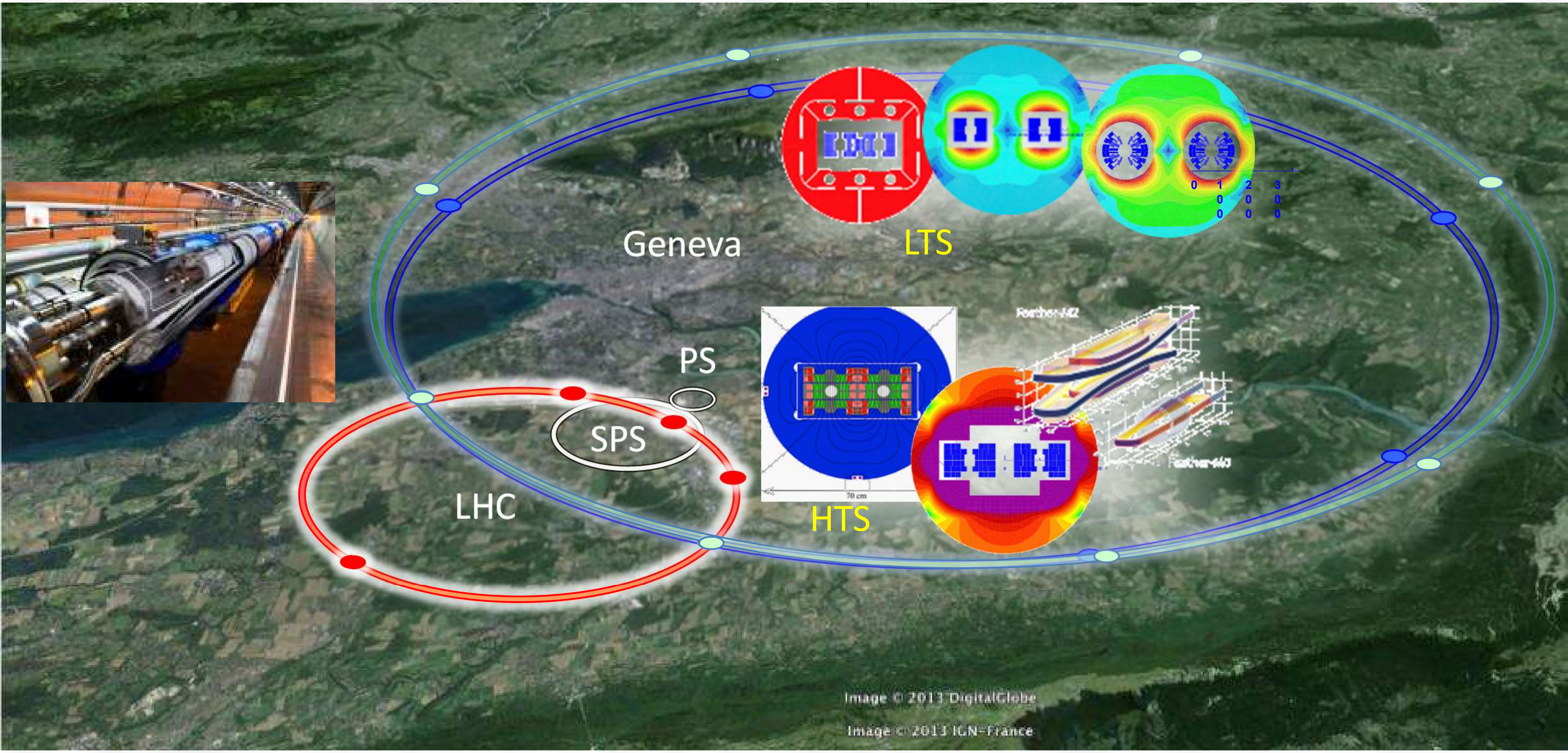
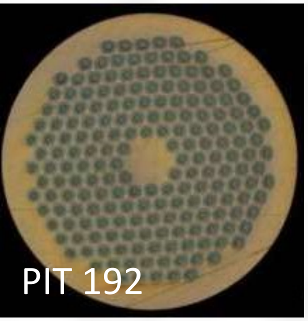
L. Bottura 2015

The FCC playground



LHC	HE-LHC	FCC-hh	FCC-hh
27 km, 8.33 T	27 km, **20 T**	80 km, **20 T**	100 km, **16 T**
14 TeV (c.o.m.)	33 TeV (c.o.m.)	100 TeV (c.o.m.)	100 TeV (c.o.m.)
1300 tons NbTi	3000 tons LTS	9000 tons LTS	6000 tons Nb_3Sn
	700 tons HTS	2000 tons HTS	3000 tons Nb-Ti

The practical HiLumi Nb_3Sn wire is in production!

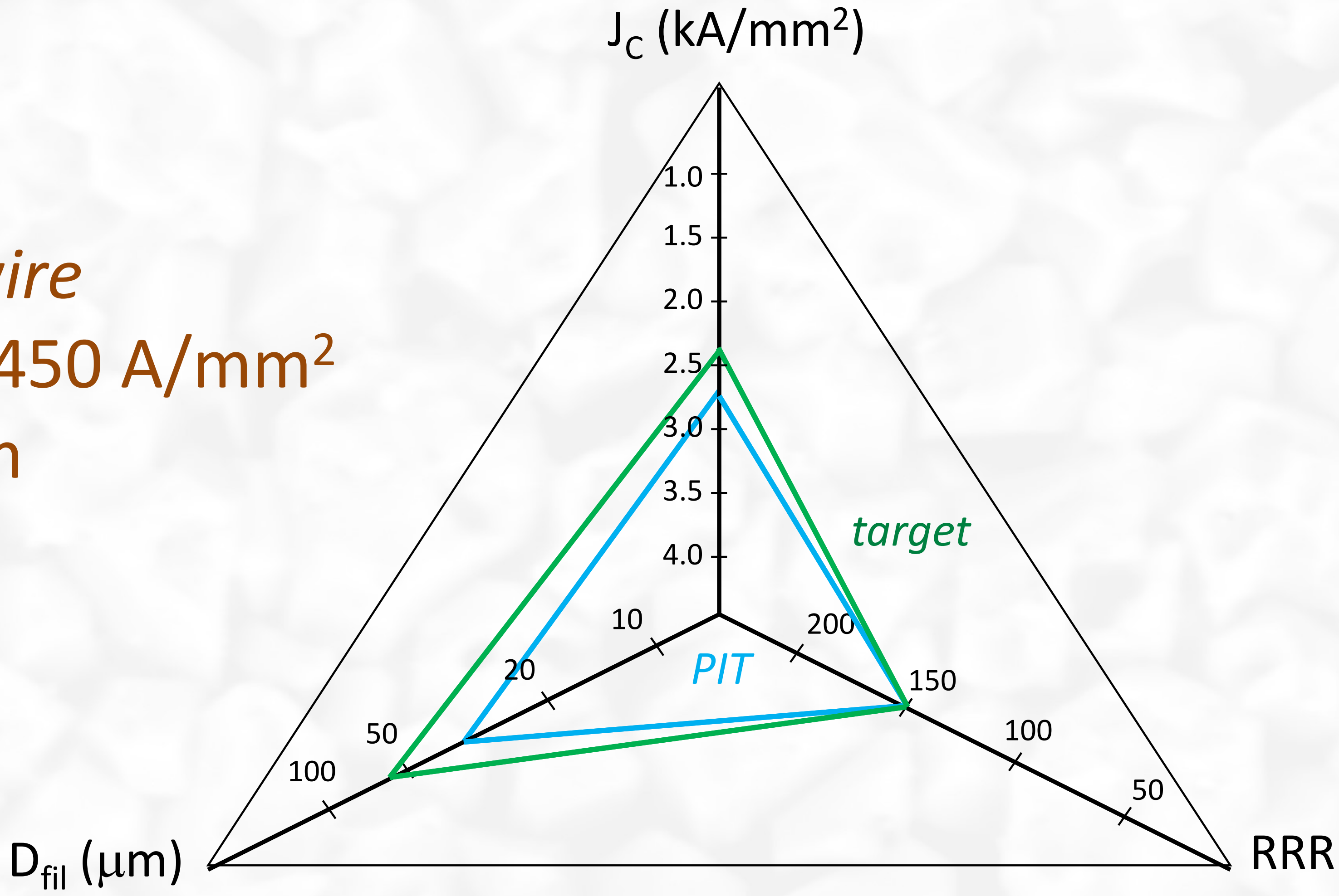


Practical wire

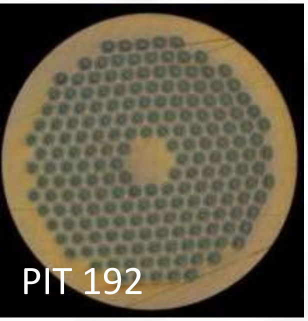
$J_C(12T) > 2450$ A/mm^2

$D_{fil} < 55$ μm

RRR > 150



Looking to the future FCC wire

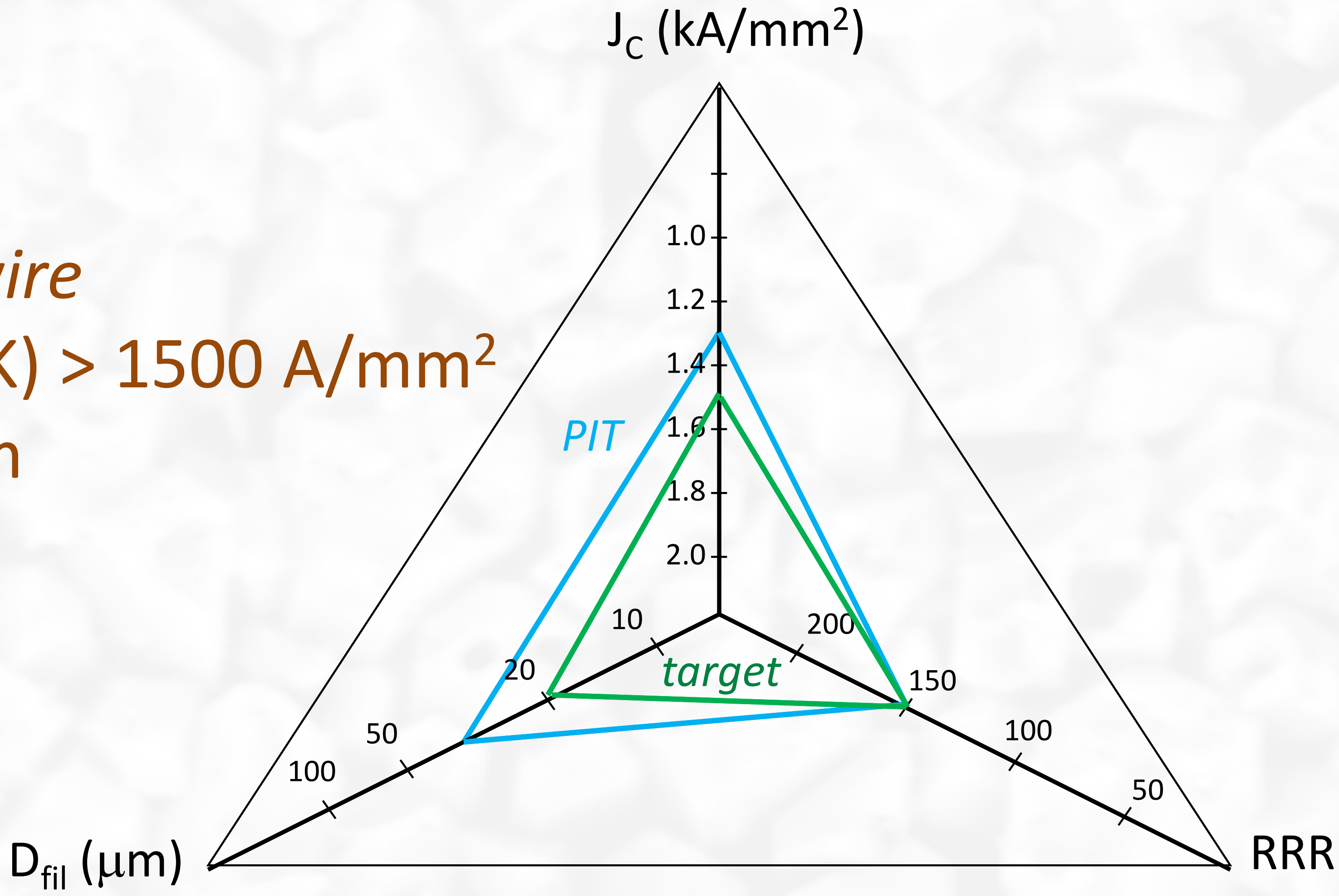


Practical wire

J_C(16T, 4.2K) > 1500 A/mm^2

D_{fil} < 20 μm

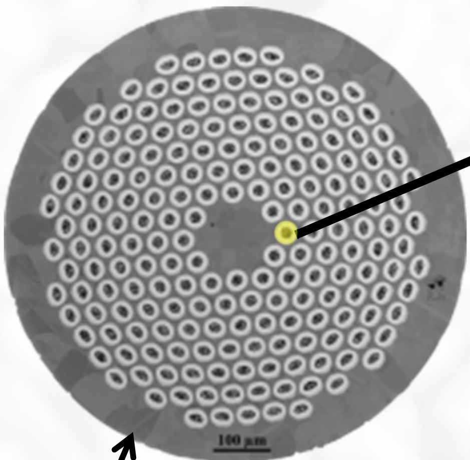
RRR > 150

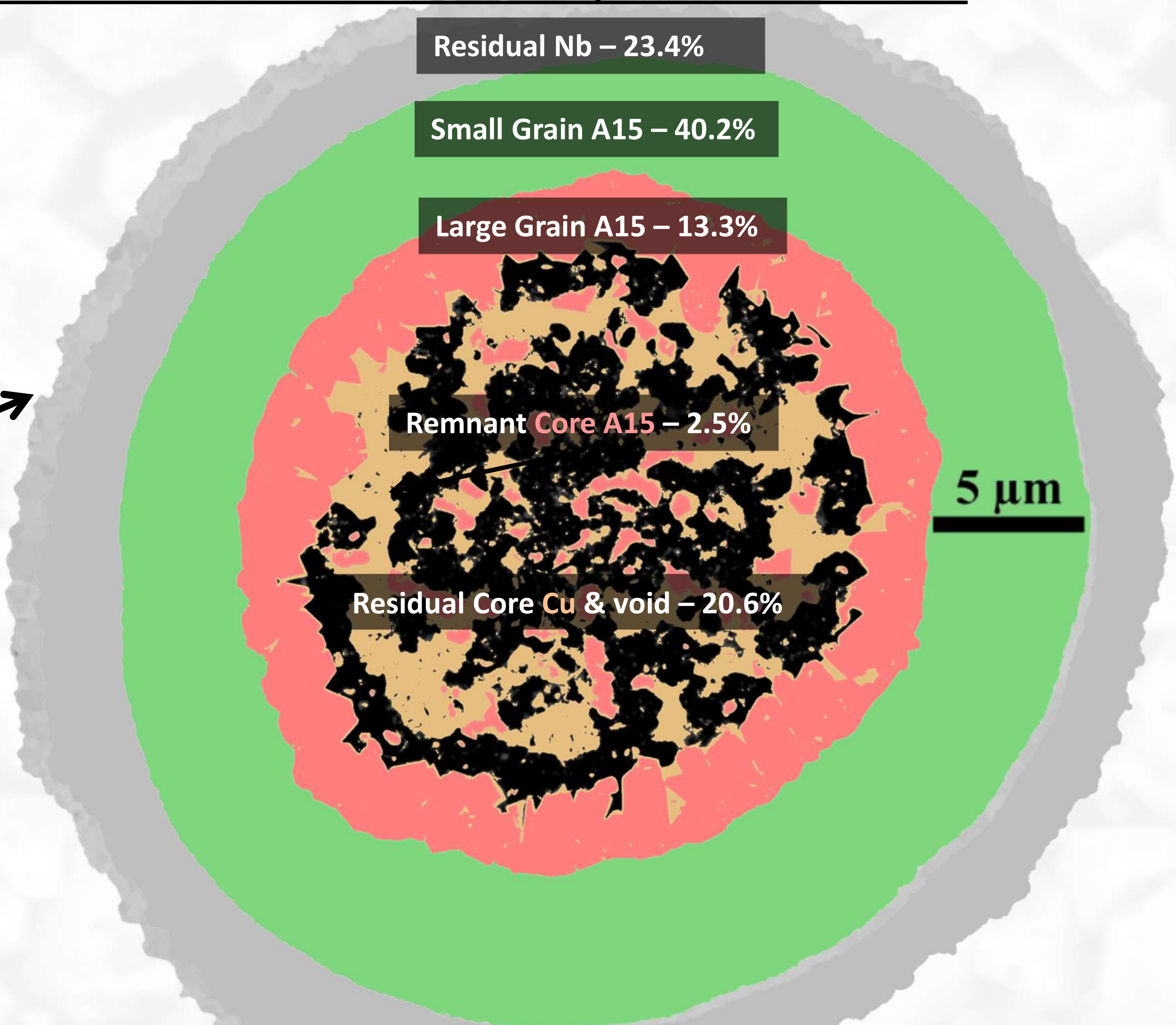


The Fractional Real Estate of PIT – only 40% is valuable

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

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





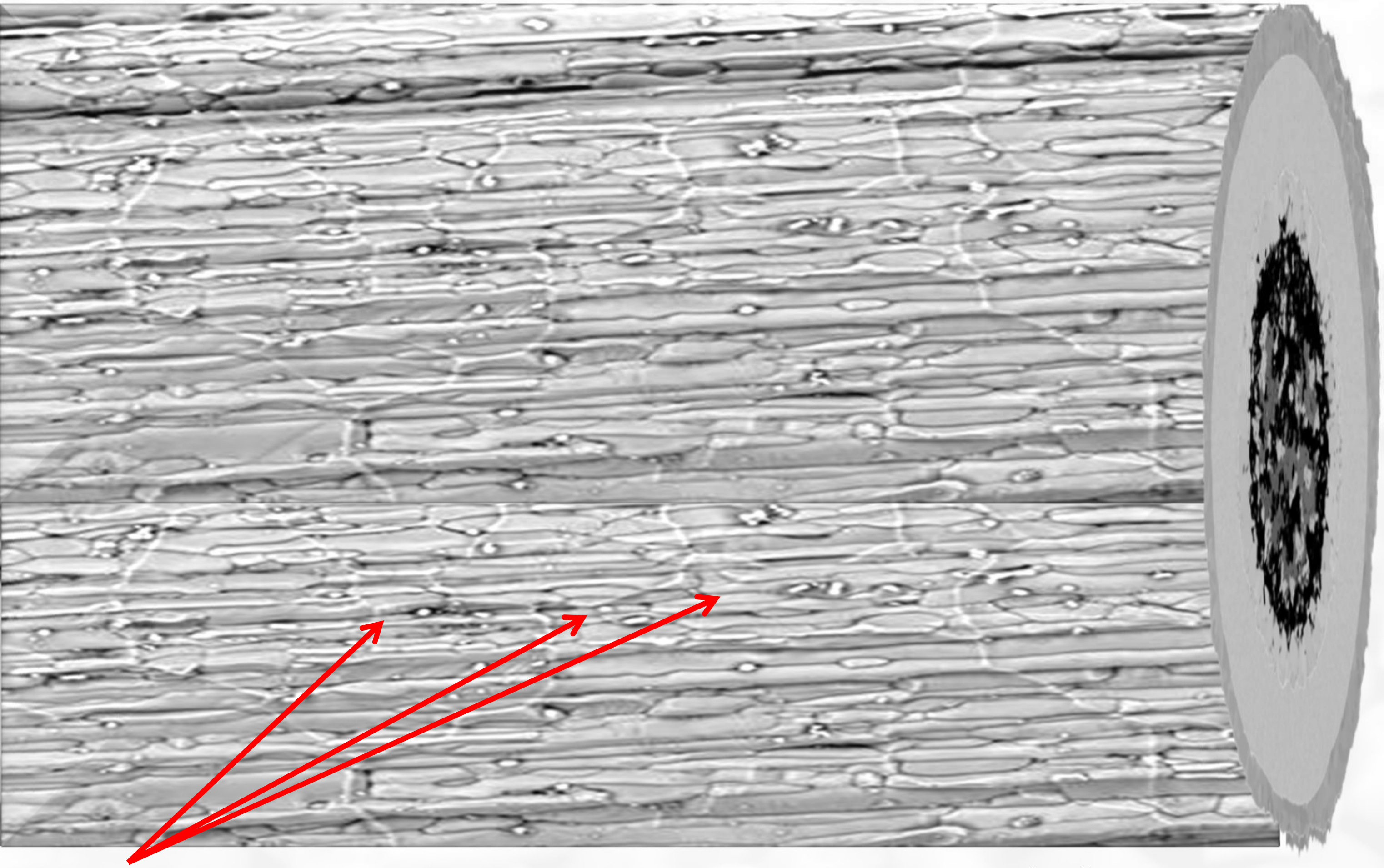
- I_c = 501 A
- $J_{c\ non\text{-}Cu}$ = 2237 A/mm^2
- $J_{c\text{-}layer}$ = 5564 A/mm^2

- Only **small grain A15** carries current
- Large and core grains of A15 do not carry any current (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



LG A15 appears very disconnected

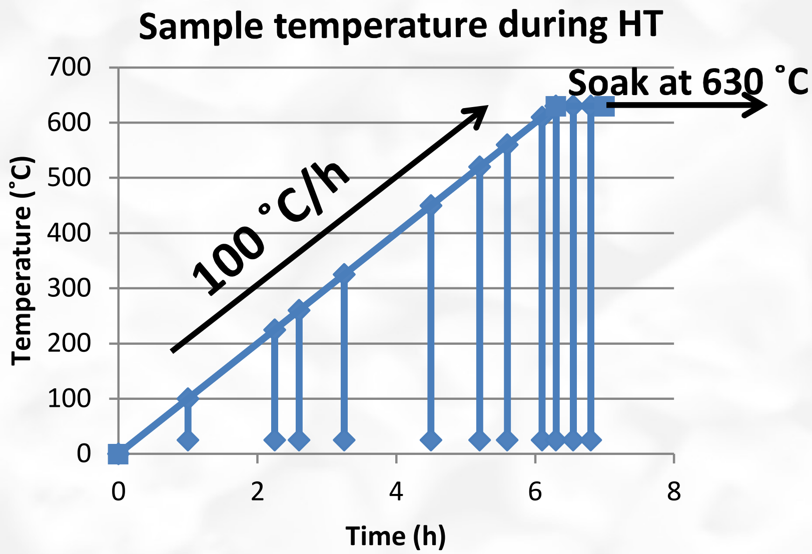
Cu-rich phases penetrate between LG's

630C200h Billet B29992
Longitudinal Cross Section

[1] C. Tarantini, C. Segal, Z. H. Sung, P. J. Lee, L. Oberli, A. Ballarino, L. Bottura, and D. C. Larbalestier, "Composition and connectivity variability of the A15 phase in PIT Nb3Sn wires," *Supercond. Sci. Technol.*, vol. 28, no. 9, p. 95001, Sep. 2015.

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.



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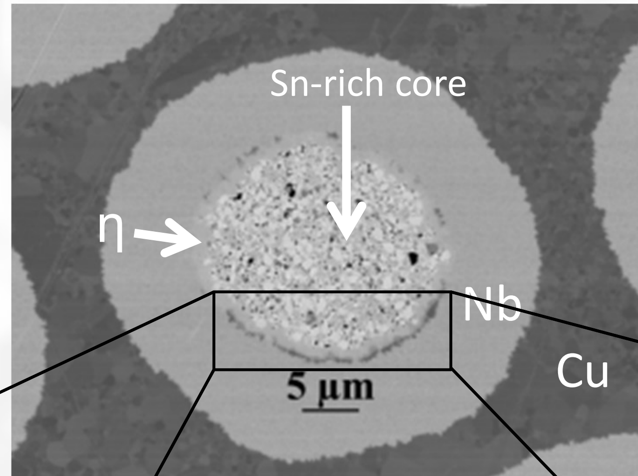
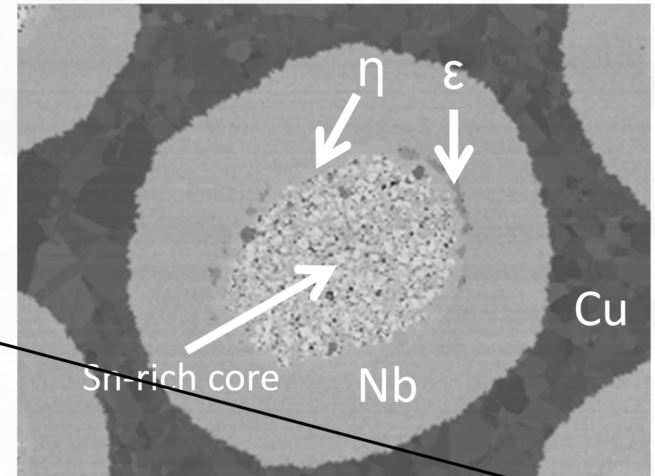
Sn is rapidly absorbed by Cu sleeve to form η early in the reaction

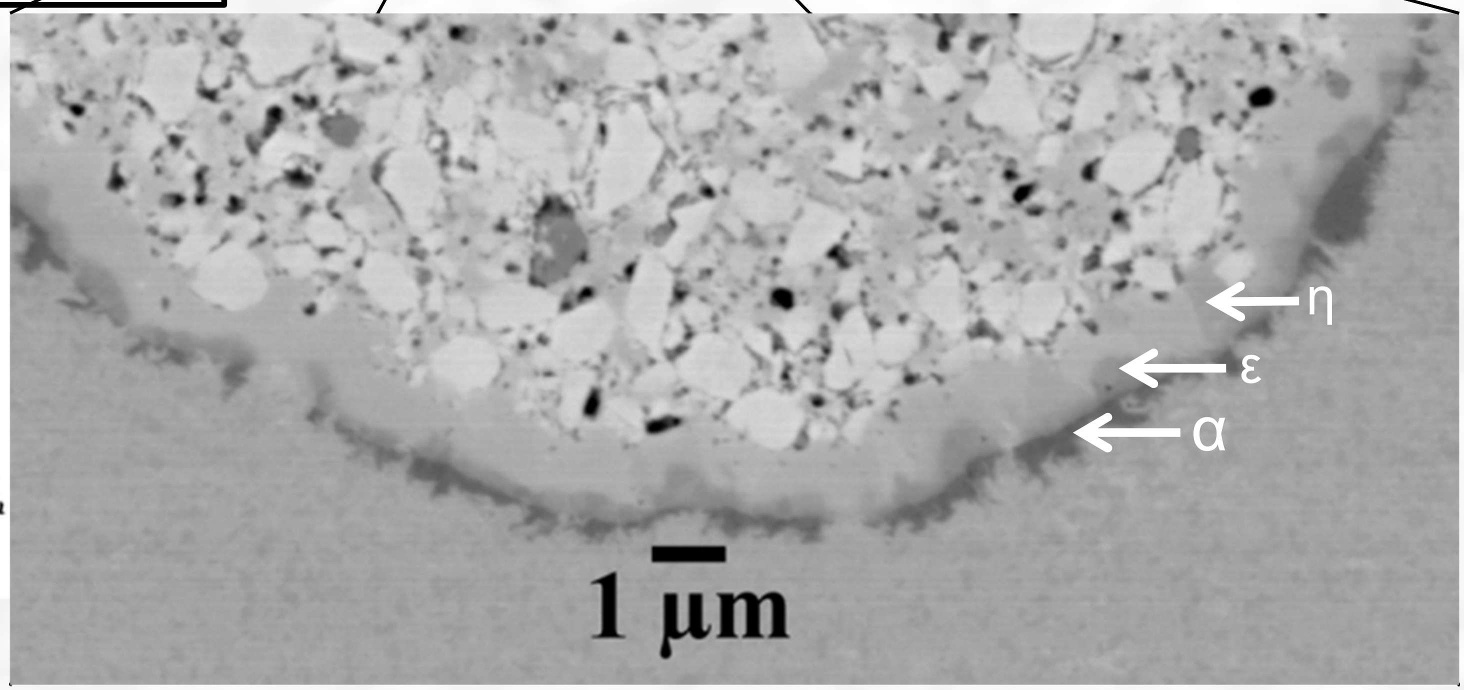
Original Nb tube is inscribed by a Cu sleeve, and the core filled with a Sn rich powder

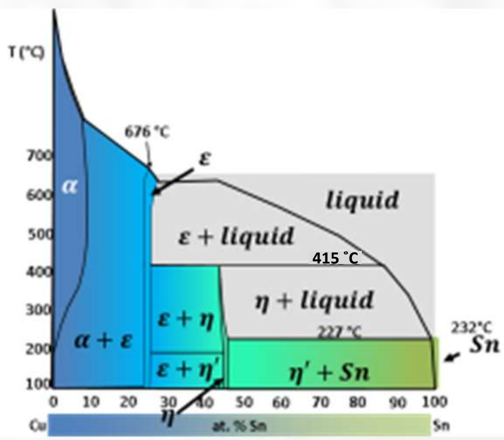
225 ˚C



260 ˚C

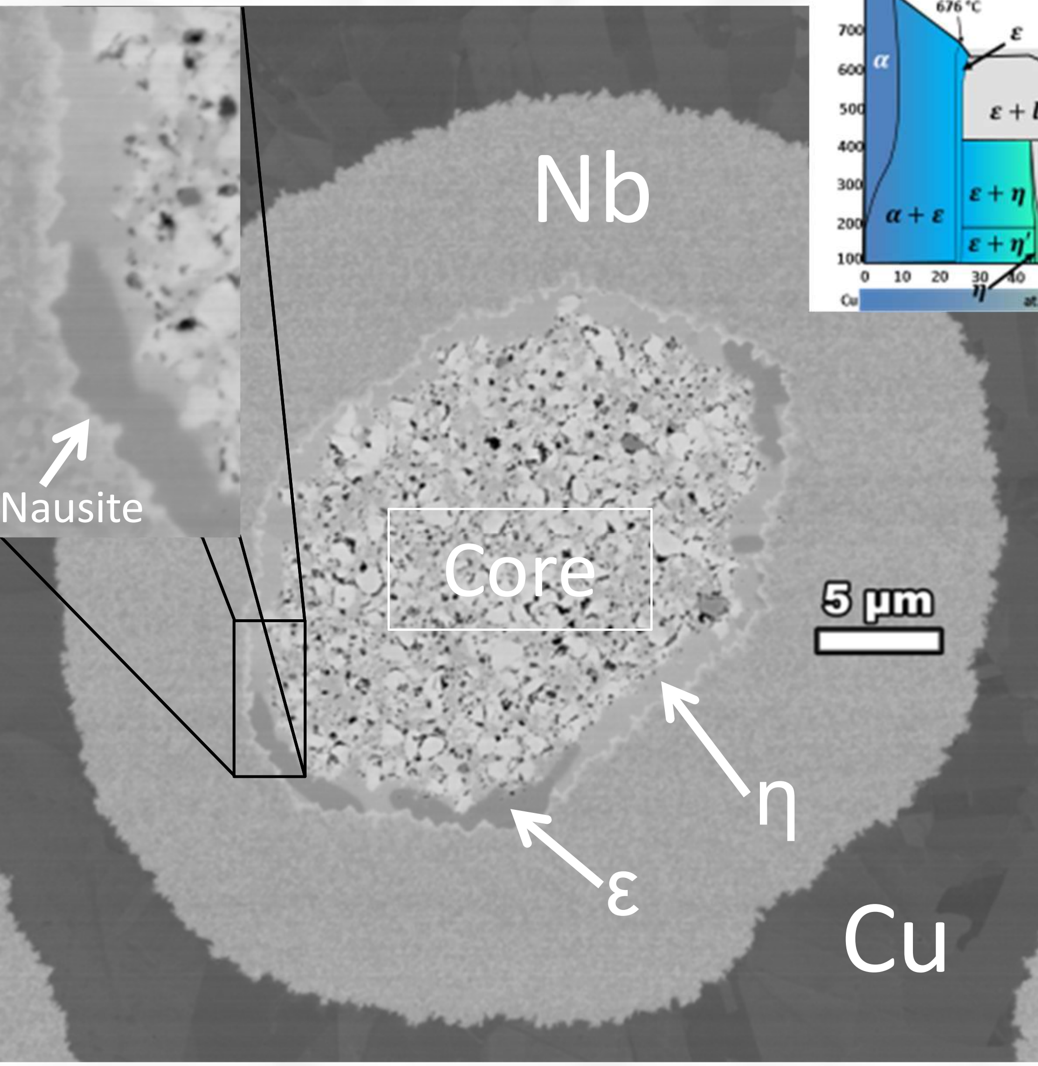


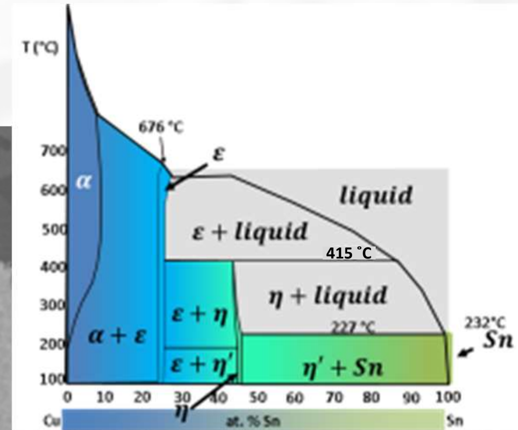




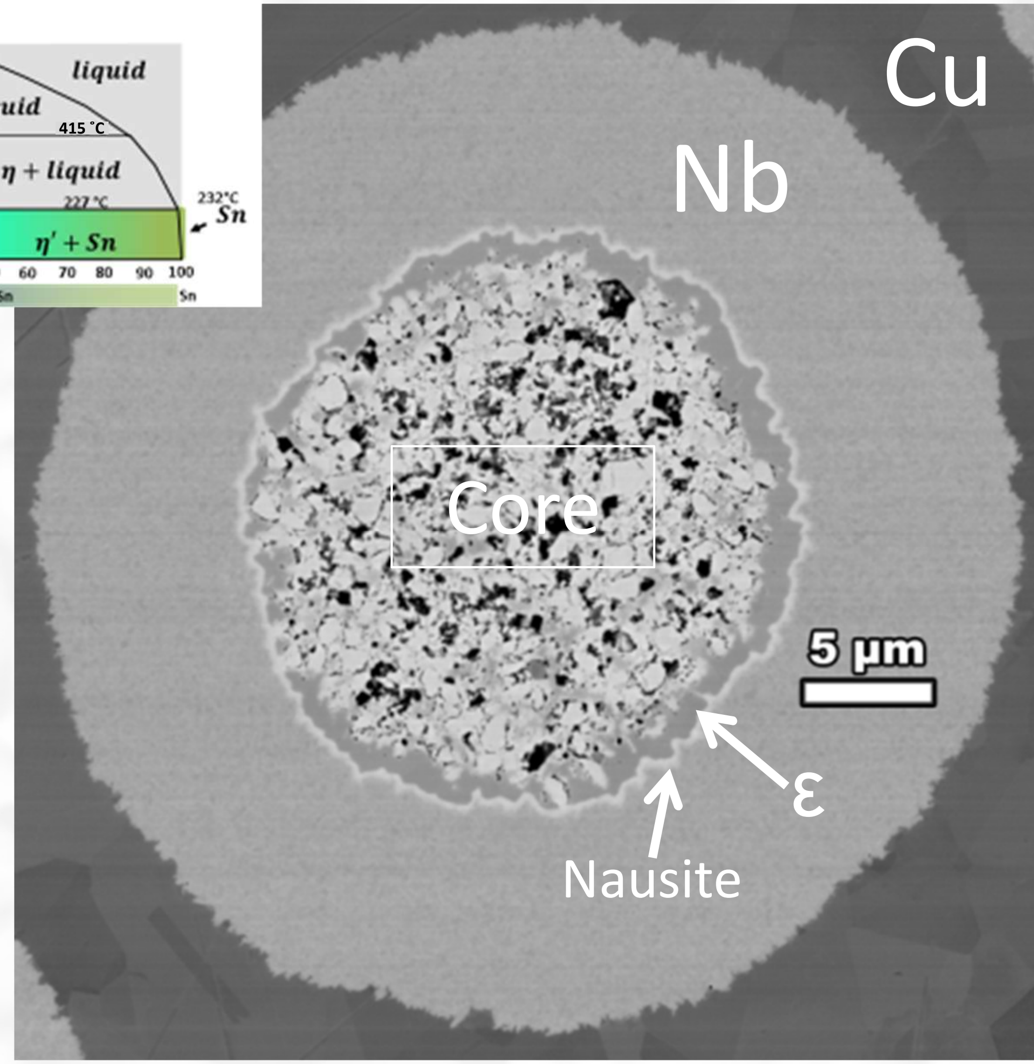
η transforms to ε at 408˚C, Nausite grows as a continuous layer

400˚C





450˚C



- 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

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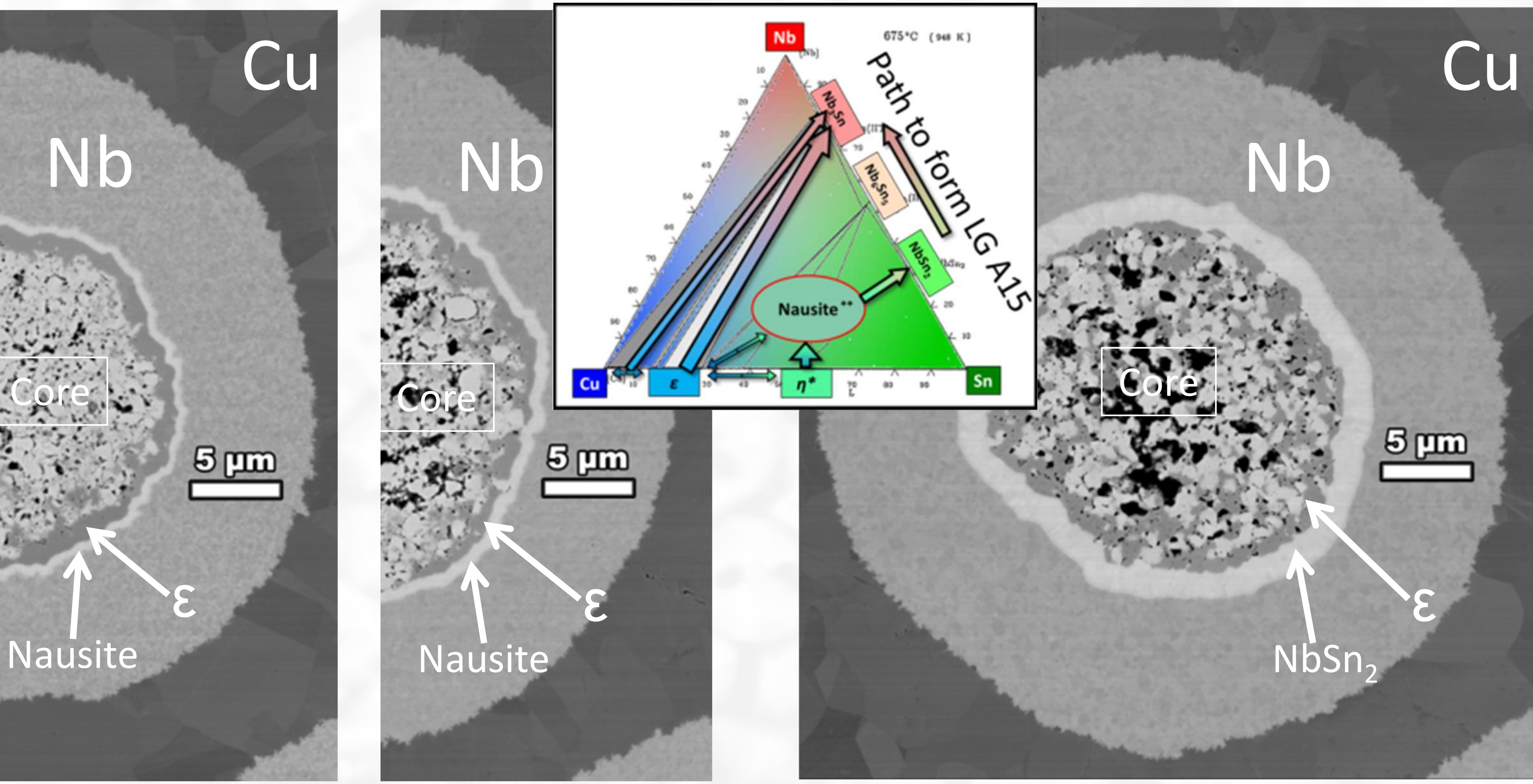
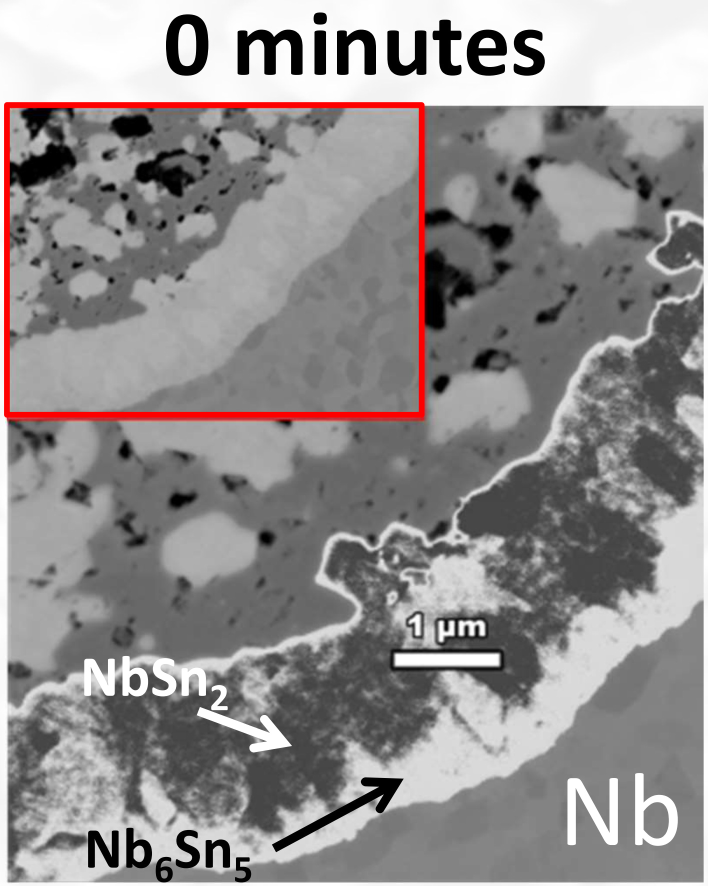
Nausite decomposes above 560 ˚C and by 610 ˚C the Sn rich layer is entirely $NbSn_2$

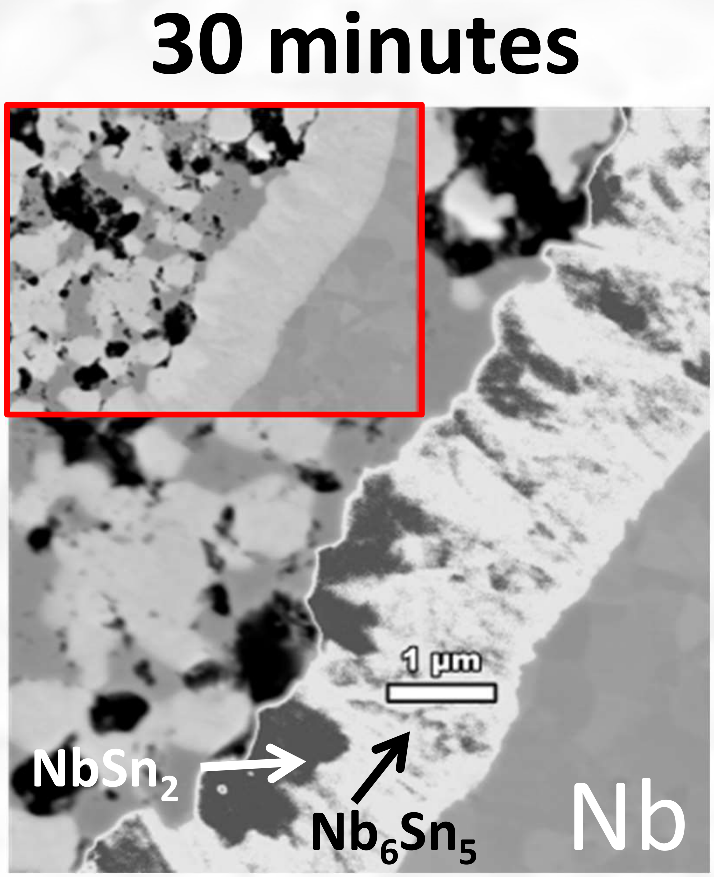
520˚C → 560˚C **610˚C**



- Nausite ring grows
- Full layer of $NbSn_2$ forms from Nausite expelling Cu

At 630˚C $NbSn_2$ rapidly transforms into Nb_6Sn_5

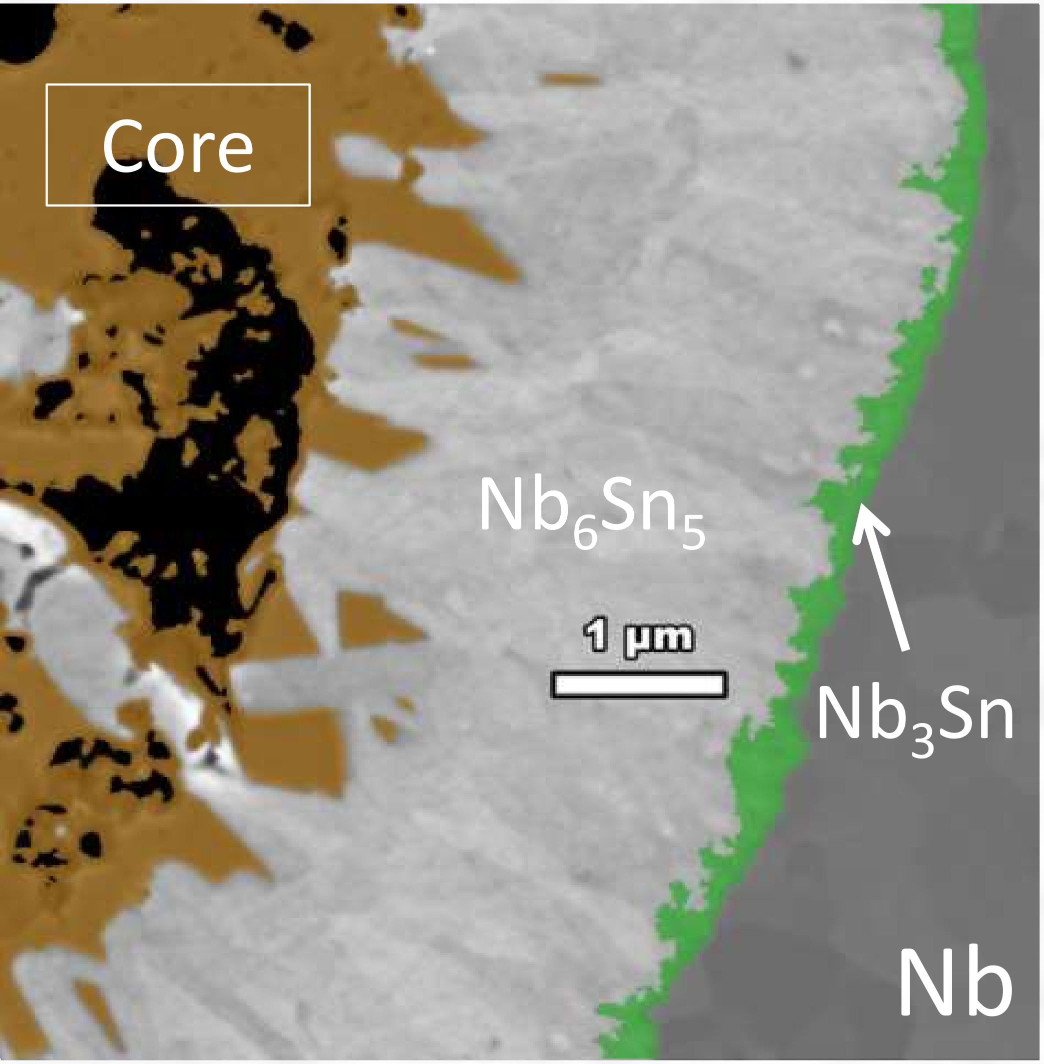




- The transformation occurs quickly; about 45 minutes at 630˚C
- The Nb_6Sn_5 contains a few at% Cu

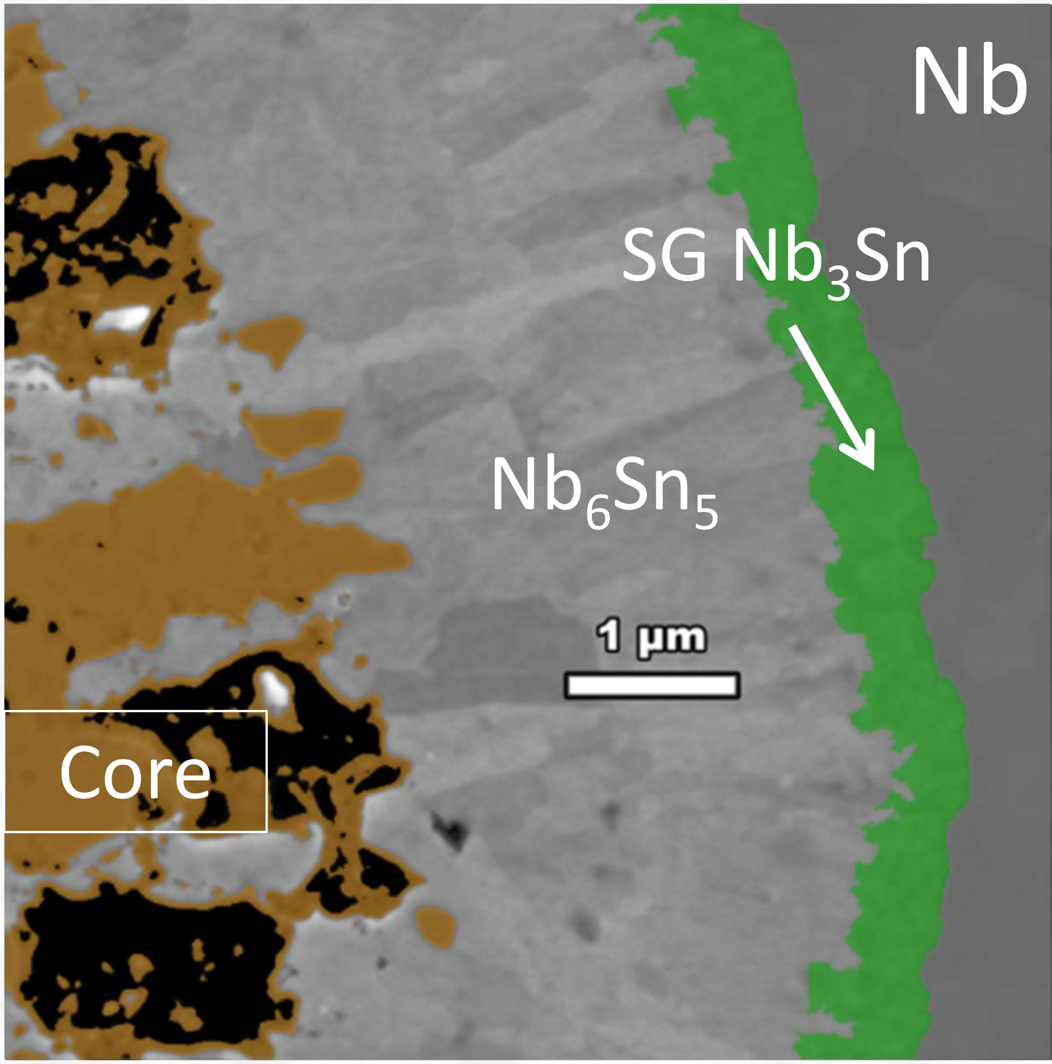
Continuous layer of small grain Nb_3Sn is formed by 5 hours

630˚C - 5hr



- Nb_6Sn_5 draws Sn from core to Nb barrier

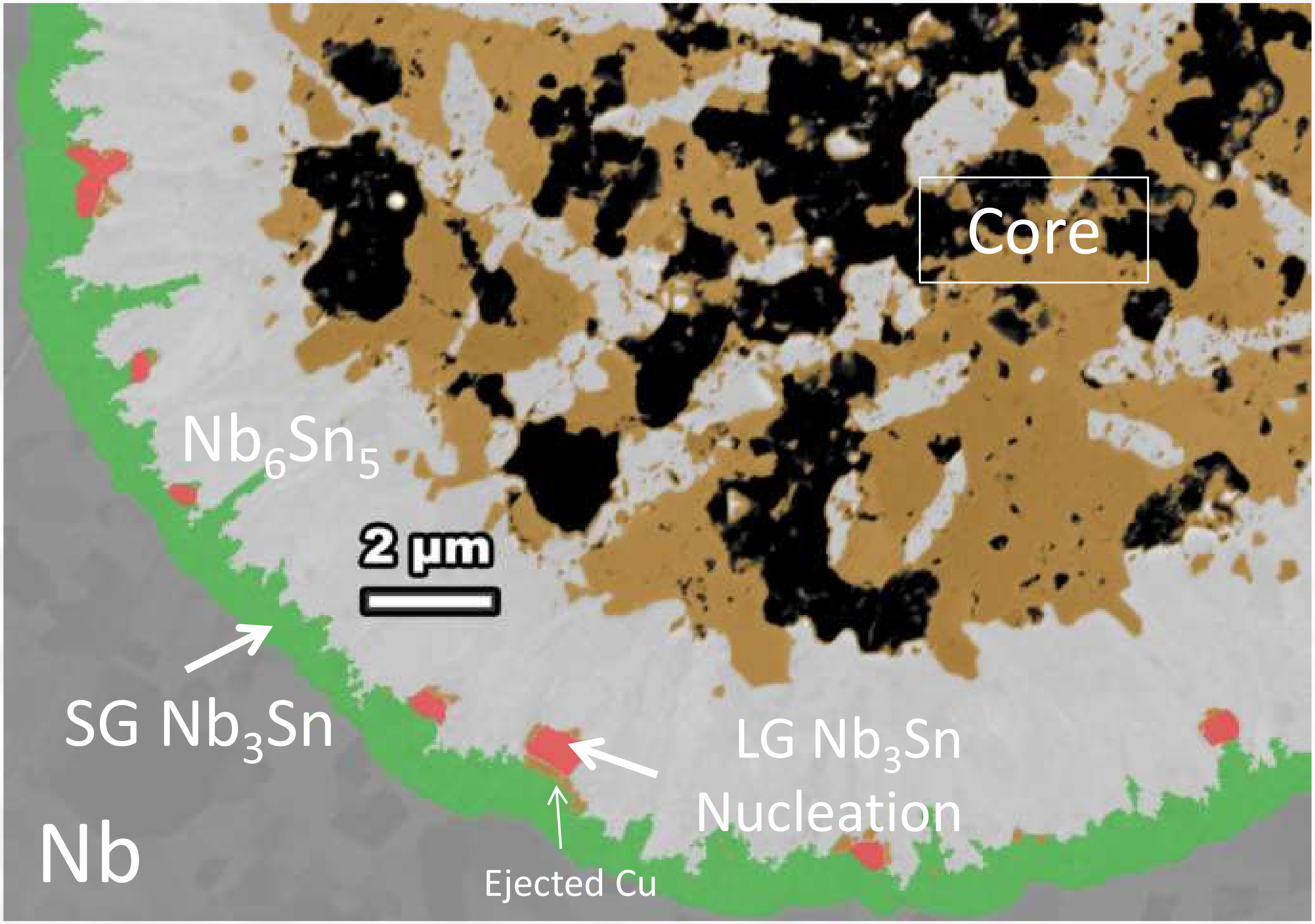
630˚C - 10hr



- Nb_3Sn layer has grown

LG A15 has formed with the ejection of Cu by Nb_6Sn_5

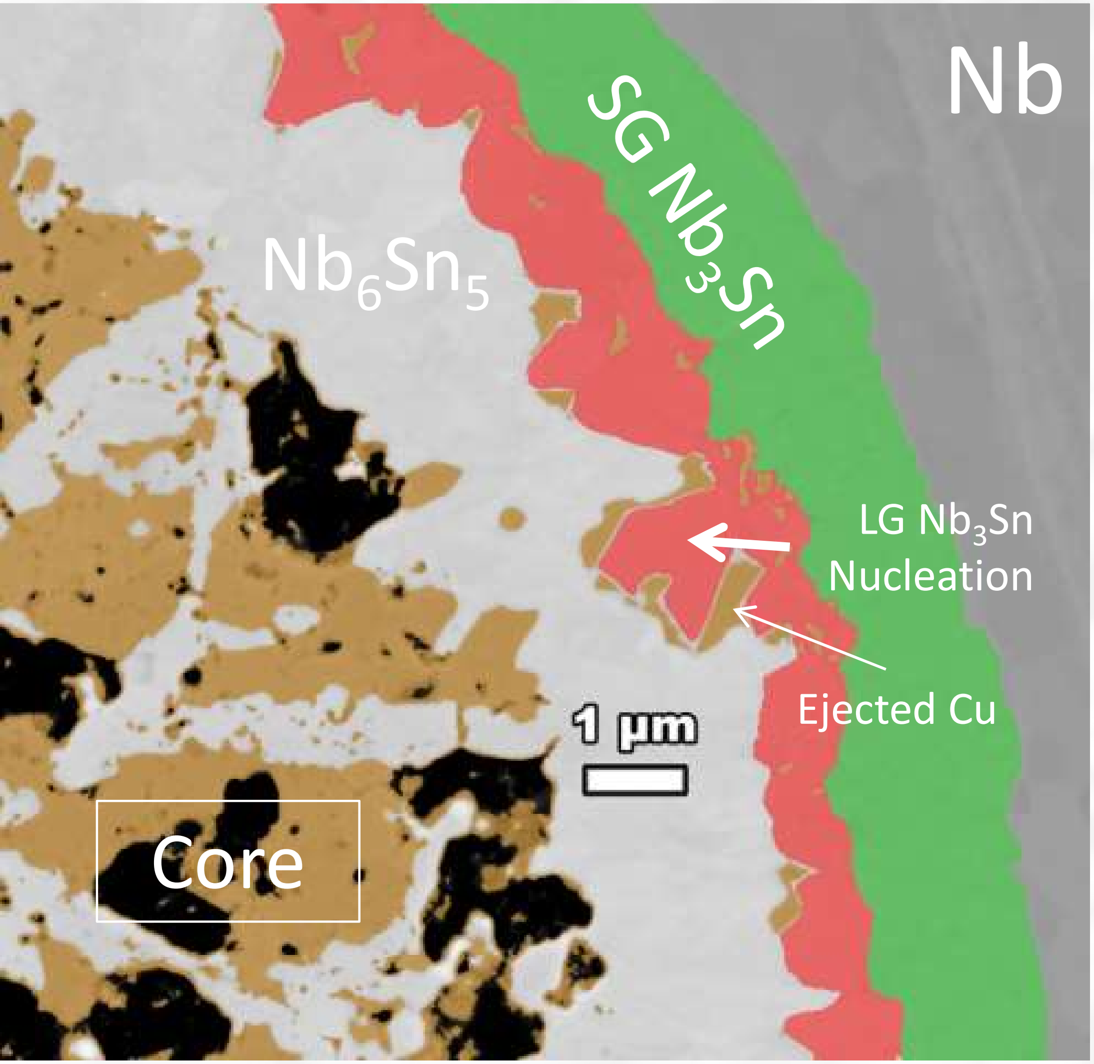
630˚C - 12hr



- What role does Cu play in the reaction?

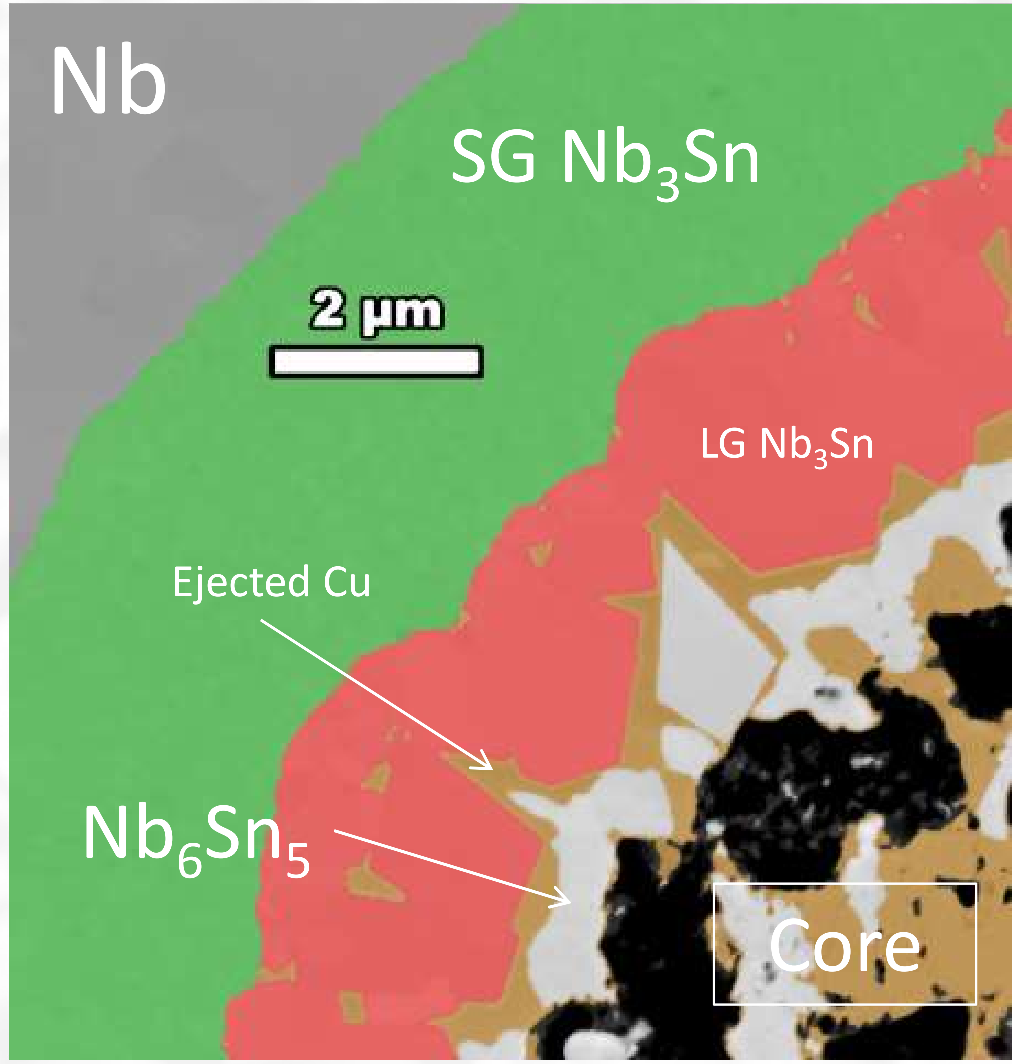
LG A15 grows with time, a Cu-rich phase separates the A15 layer from the core

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

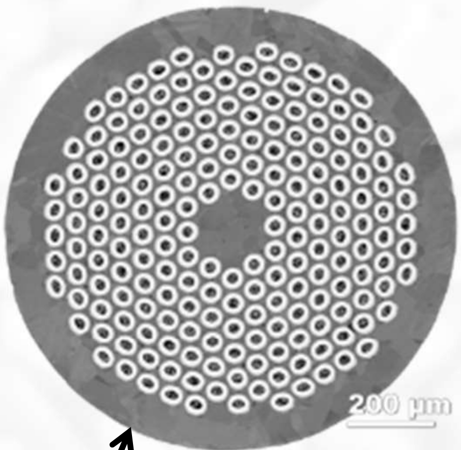


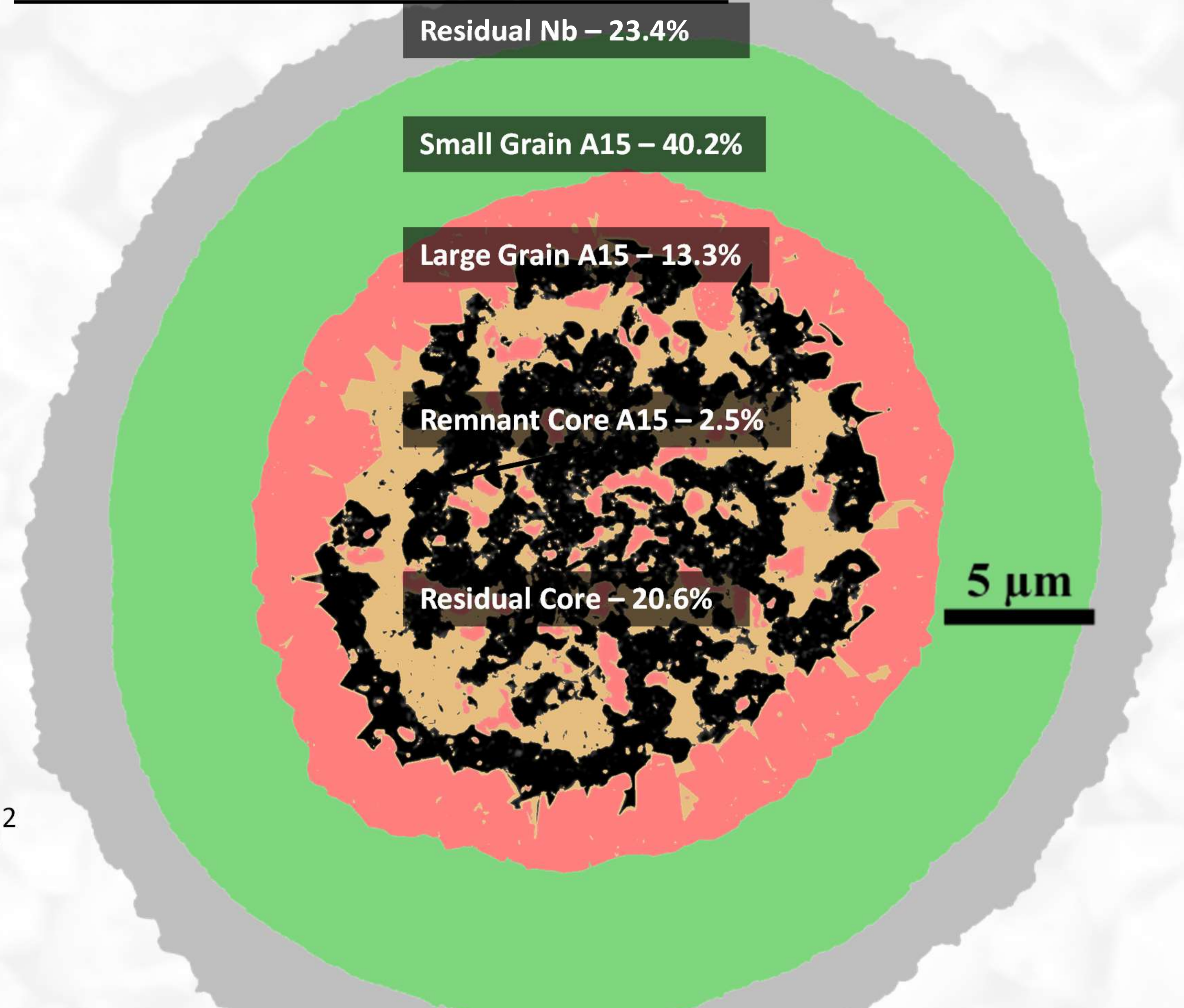
- There is a continuous layer of large grain A15 and penetrating Cu. A small amount of Nb_6Sn_5 remains

The Fractional Real Estate of PIT

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

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

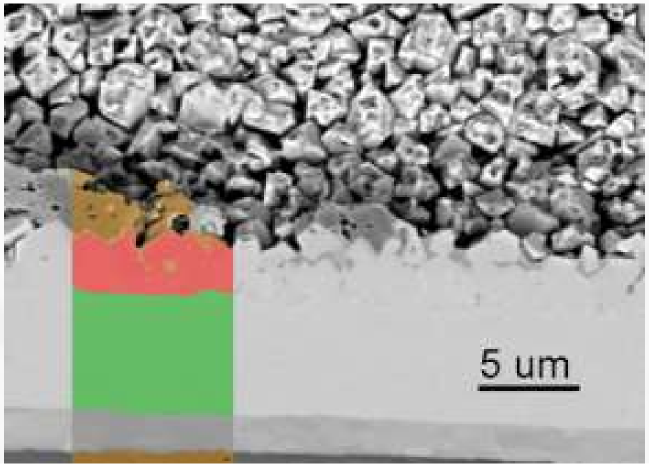




- I_c = 501 A
- $J_{c\ non\text{-}Cu}$ = 2237 A/mm^2
- $J_{c\text{-}layer}$ = 5564 A/mm^2

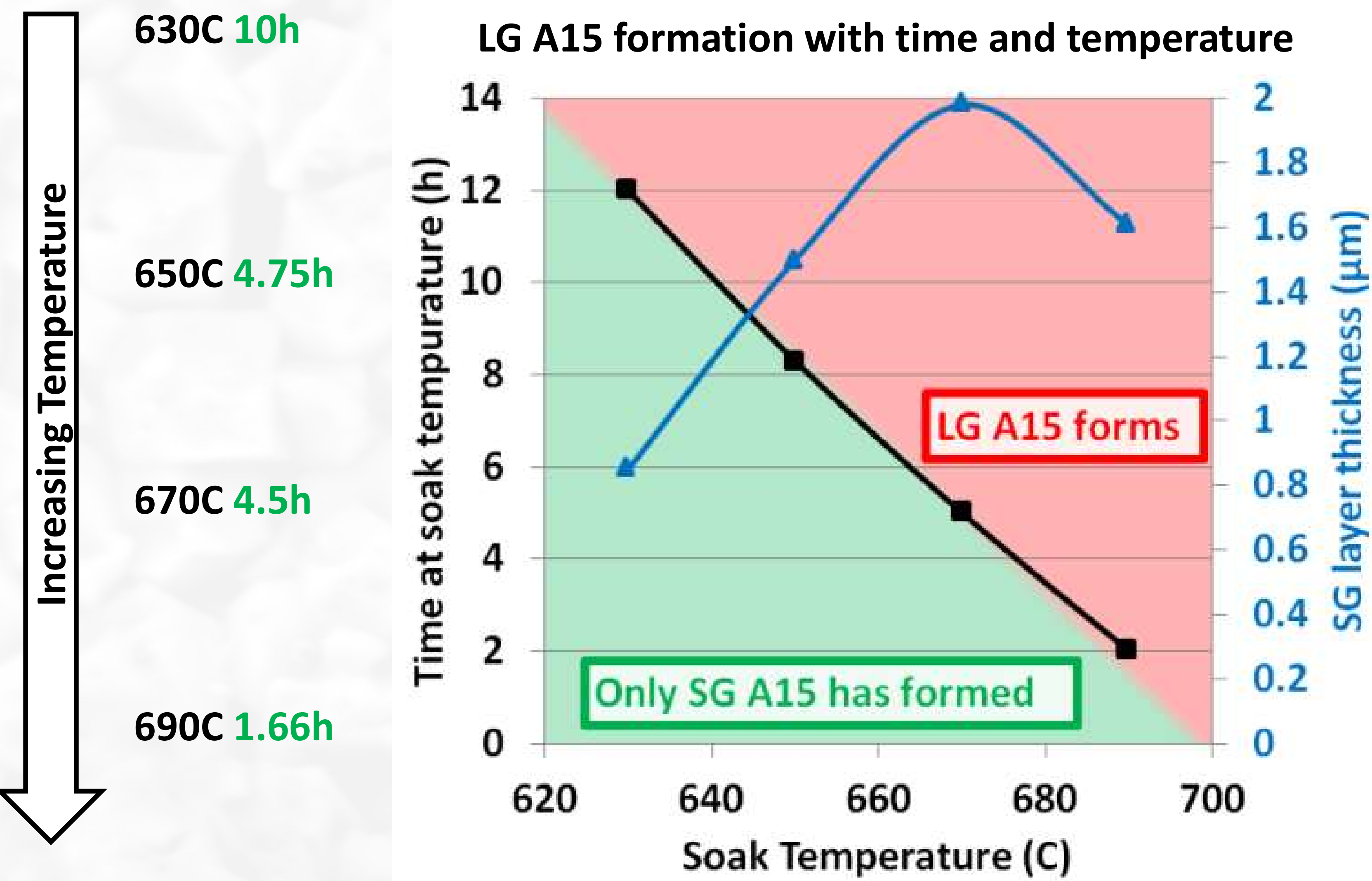
- Only **small grain A15** carries current
- Large and core grains of A15 do not carry any current.

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?



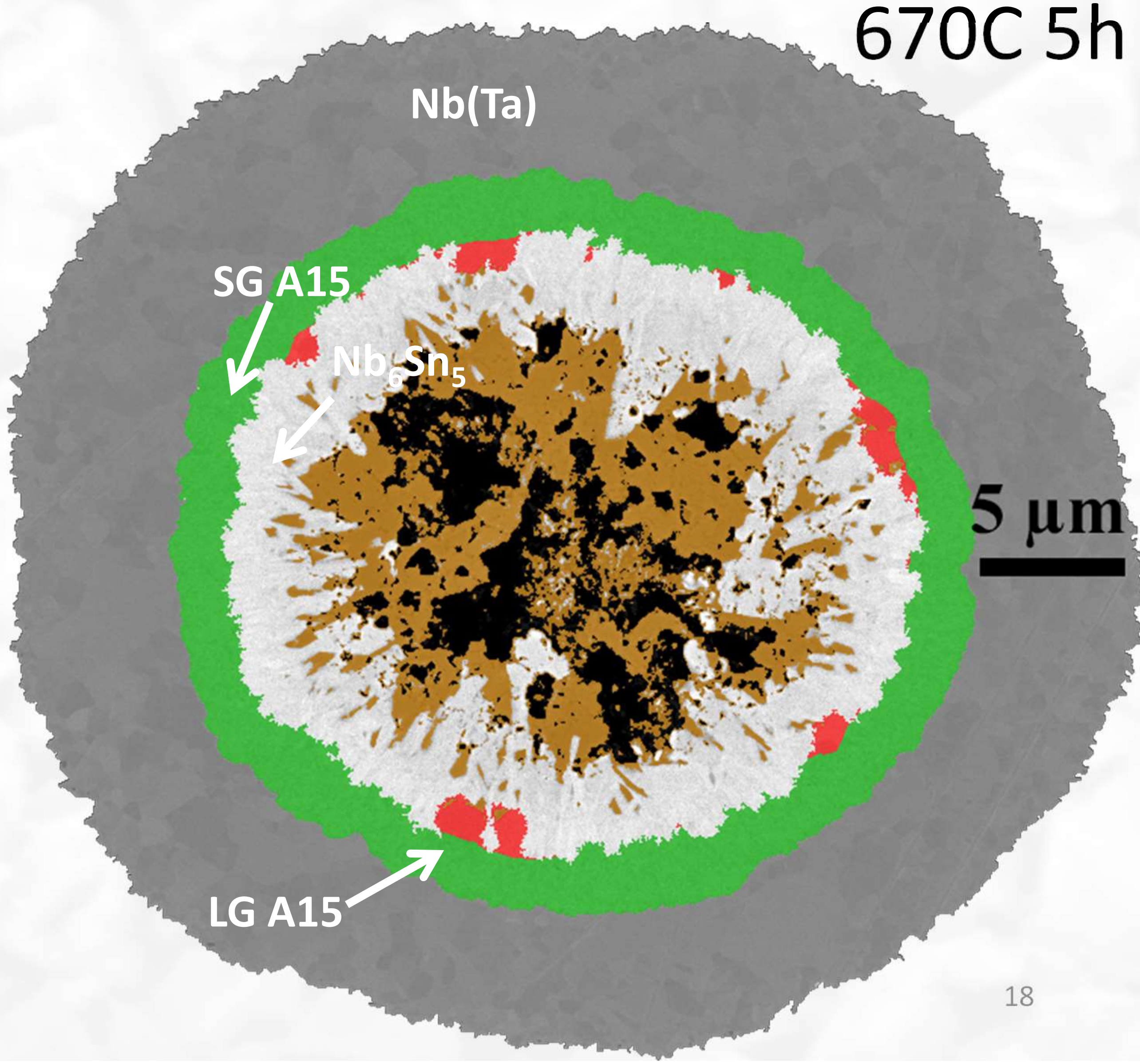
LG A15 formation is temperature dependent

Only SG A15 has formed	LG A15 has nucleated with Cu
630C 10h	630C 12h
650C 4.75h	650C 8.33h
670C 4.5h	670C 5h
690C 1.66h	670C 2h



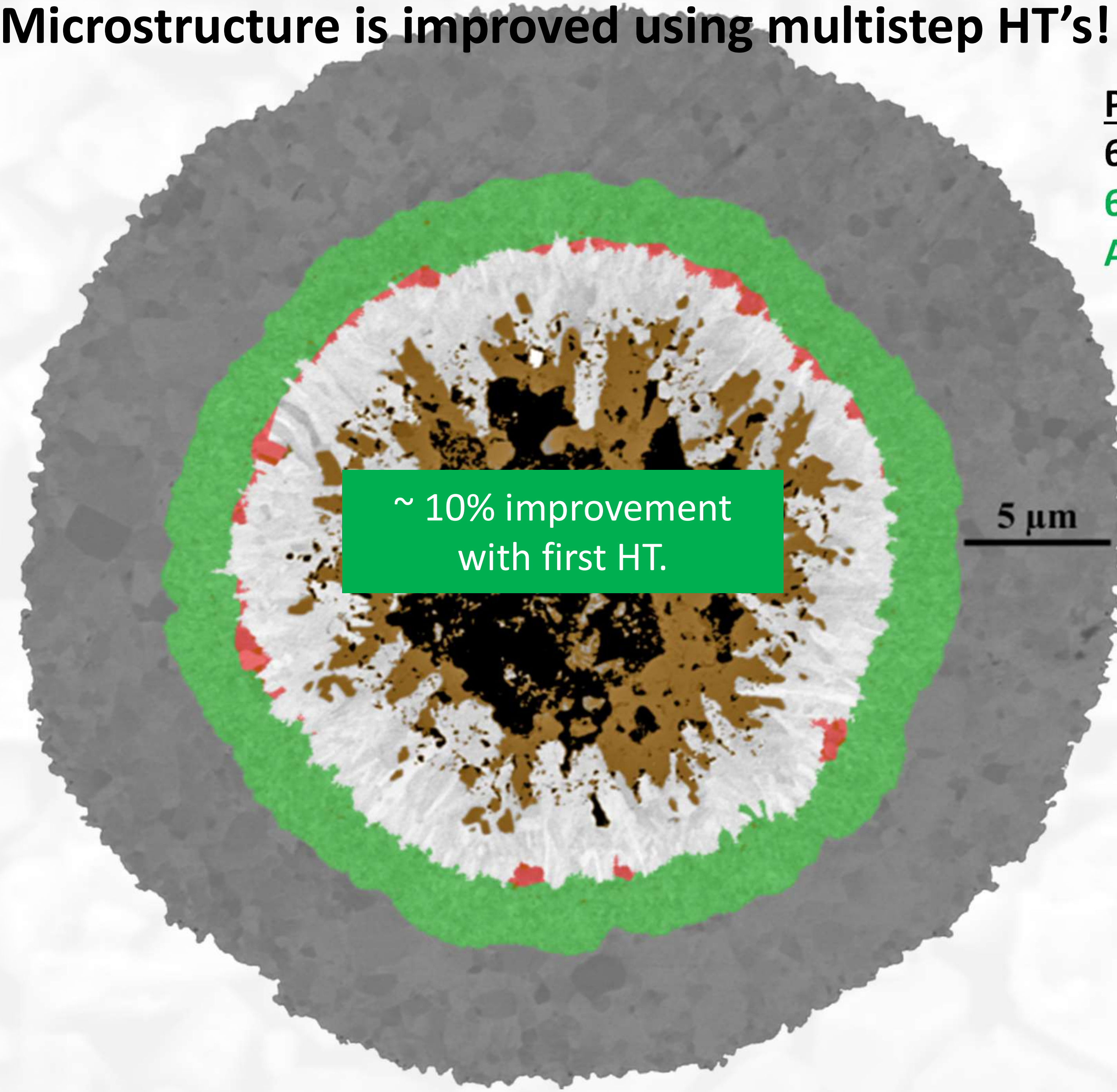
SG A15 forms *first* by Nb_6Sn_5 reacting with the tube. LG A15 forms by decomposition of Nb_6Sn_5.

- 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!



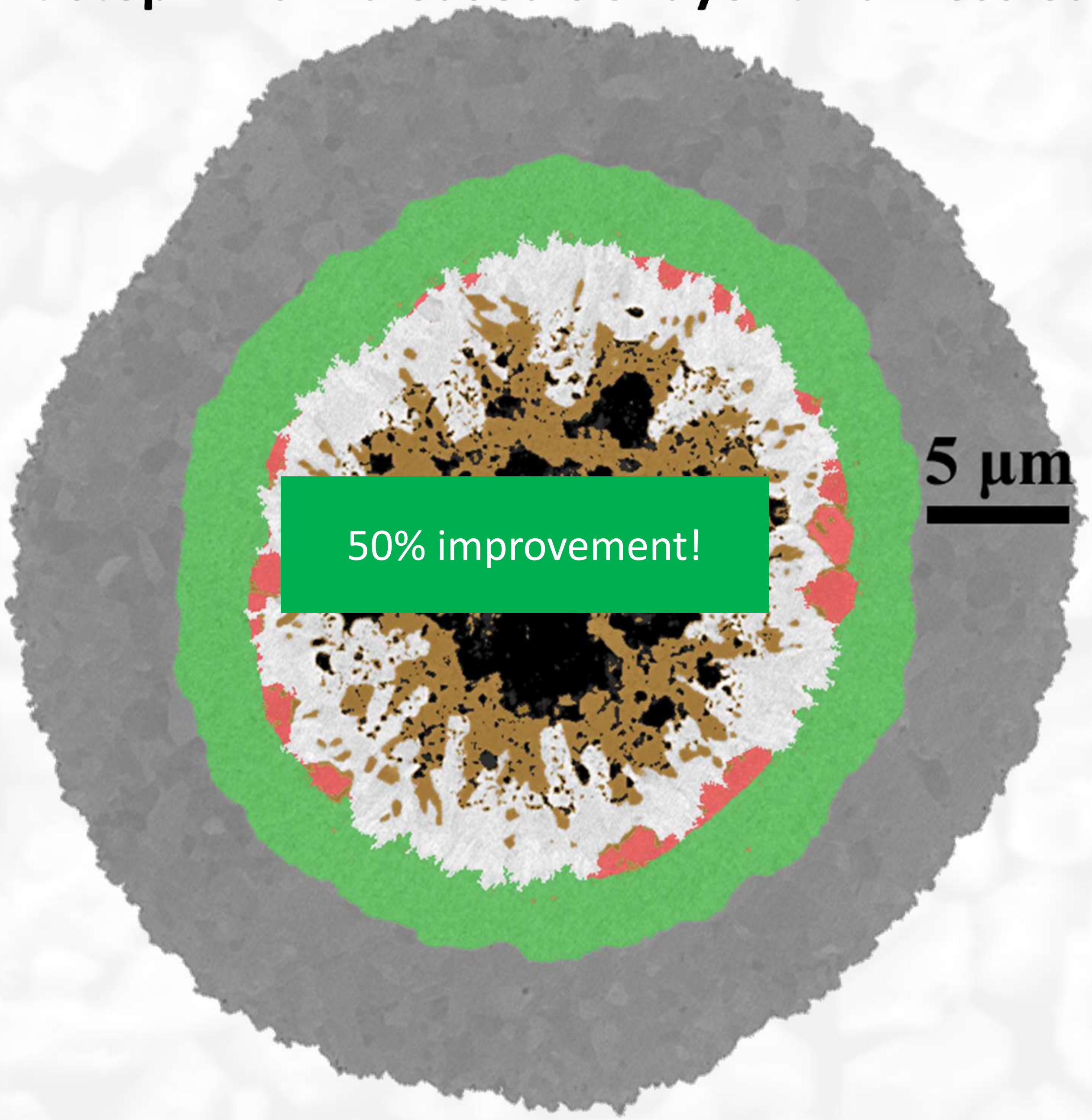
Previous HT's

630/12	0.8 µm
670/5	2.0 µm
A+630/13	2.2 µm

*Steps are at higher temperatures

Step A + 630 ˚C 13h – 2.2 µm SG layer thickness!

Refining multistep HT's increased SG layer thickness early in reaction



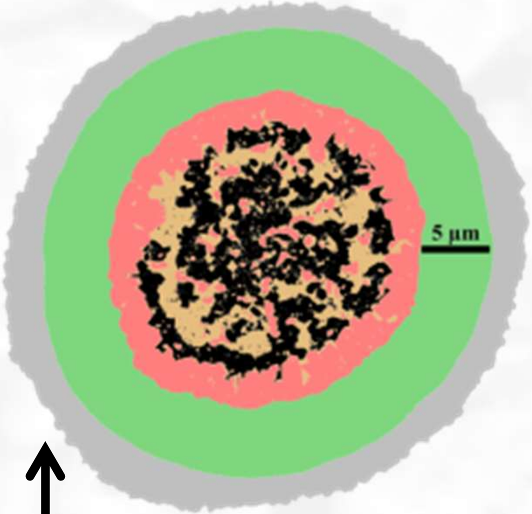
Previous HT's

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

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

Heat treatment description	Heat treatment (temp/soak time)	I_c (A)	J_c (A/mm²)	$J_{c\ SG\text{-}layer}$ (A/mm²)	Nb	Total A15	core A15	LG A15	SG A15
BEAS recommended	620/100+640/120	501	2237	5564	23.4%	56.0%	2.5%	13.3%	40.2%

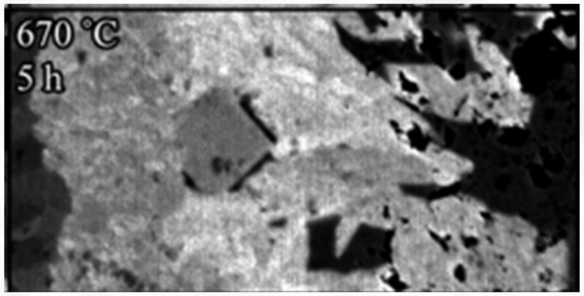
Why?

- The steps before soaking at 630˚C are higher temperature– producing larger grains of SG A15 than BEAS recommended HT

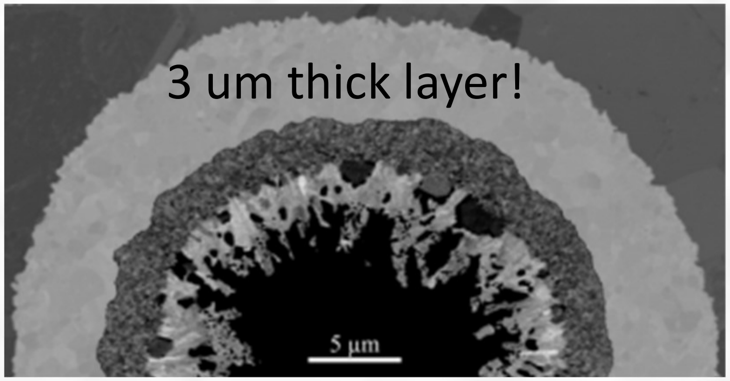
Summary of LG formation experiments

- Initial low temp HT at 630˚C creates a thin layer of SG A15 before LG's nucleate from the Nb_6Sn_5. (0.85µm)

- At higher temperatures (670˚C), this SG layer is 2x it's low temp counterpart. (1.98 µm)



- Using multistep HT (630˚C- 690˚C), we can get 3x the layer thickness without LG forming (3.0 µm)



Findings for discussion

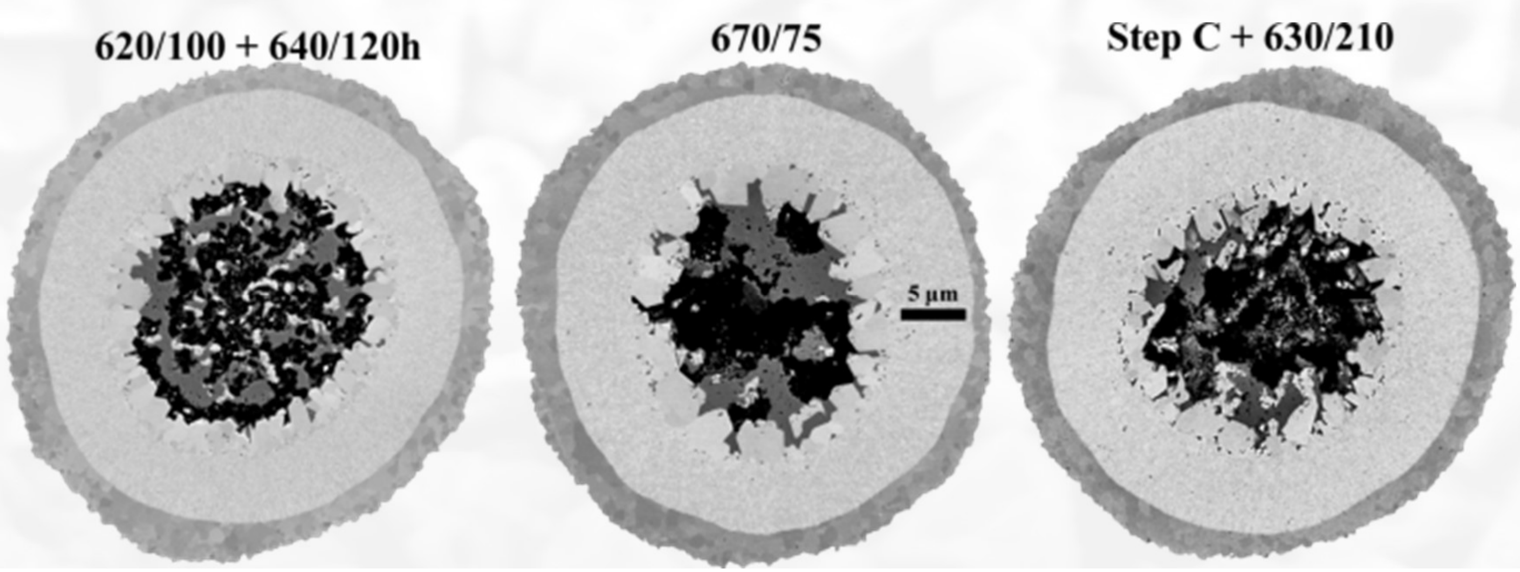
The two main A15 morphologies form by different reaction paths

- Small grains of A15 form by Nb_6Sn_5 feeding Sn into the Nb(Ta) diffusion barrier.
  - SG A15 ALWAYS forms first.
- Large grains of A15 form mostly by decomposition of Nb_6Sn_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.



Acknowledgements

David Larbalestier
Peter Lee
Chiara Tarantini

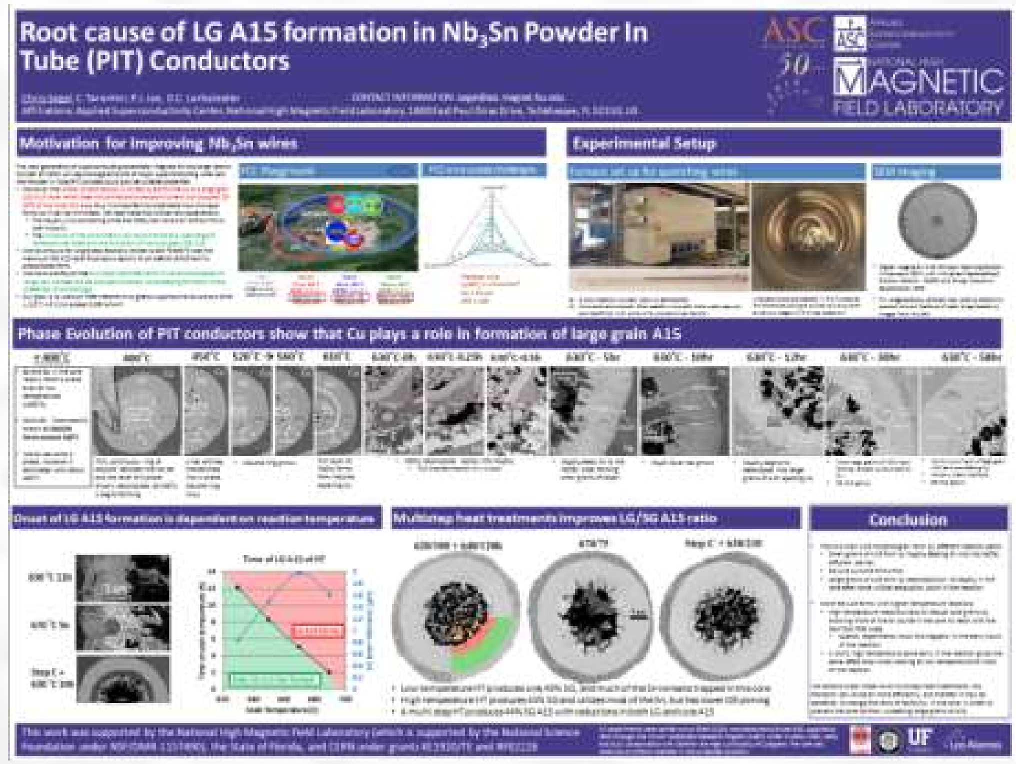
NATIONAL HIGH MAGNETIC FIELD LABORATORY

ASC APPLIED SUPERCONDUCTIVITY CENTER

2MPo1C-07[166]

Amalia Ballarino
Luca Bottura
Christian Scheuerlein
Bernardo Bordini

CERN

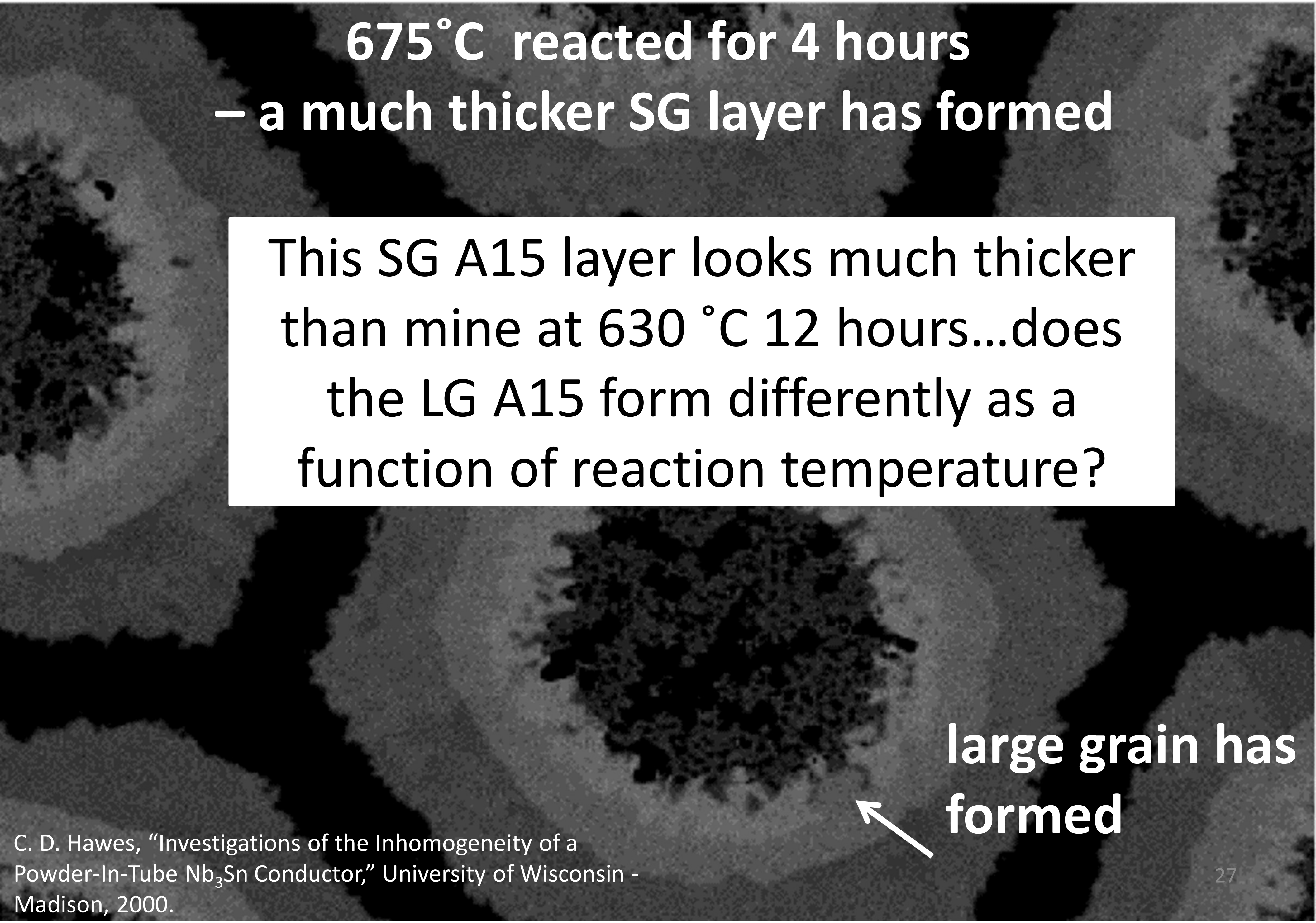


Klaus Schlenga
Bernd Sailer
Manfred Thoener

BRUKER

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?

large grain has formed

C. D. Hawes, “Investigations of the Inhomogeneity of a Powder-In-Tube Nb_3Sn Conductor,” University of Wisconsin - Madison, 2000.