Potential for Improvement in PIT and RRP® Strand

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Nb_3Sn still the superconductor of choice

- Relatively low cost compared to HTS
- Available in good enough quality for the building of test magnets
- But . . .
  - Current performance is insufficient to meet FCC goals of critical current densities (≥ 1500 A/mm^2 at 16 T) and filament diameters ≤ 20 μm

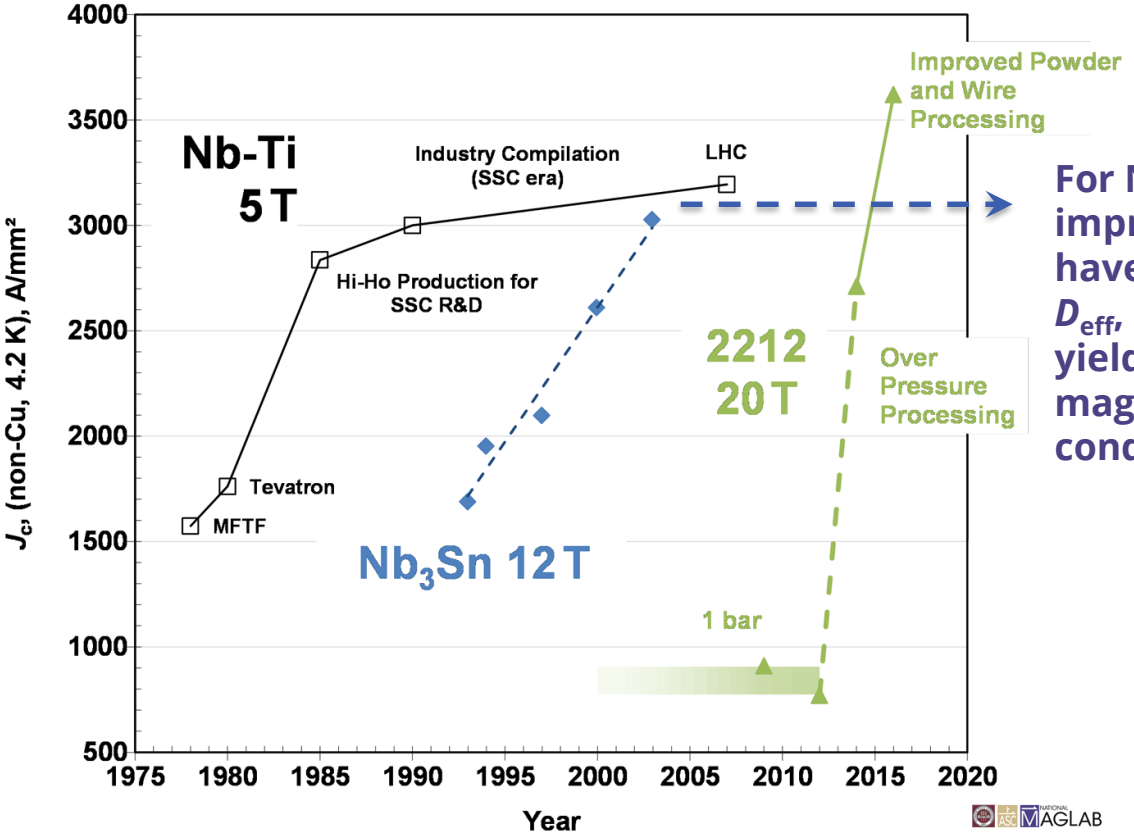
Progress in Round Wire J_c (4.2 K, non-stabilizer)

- **It has been over 10 years since there has been a significant increase in Nb_3Sn J_c**

Notes:

Nb_3Sn data from Parrell *et al.* 2004 http://dx.doi.org/10.1063/1.1774590

Bi-2212 strand manufactured by OST



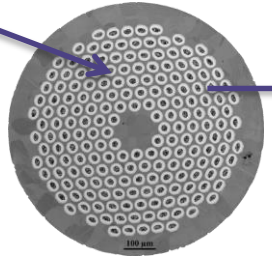
For Nb_3Sn the improvements have been in D_{eff}, RRR and yield = Better magnet conductor

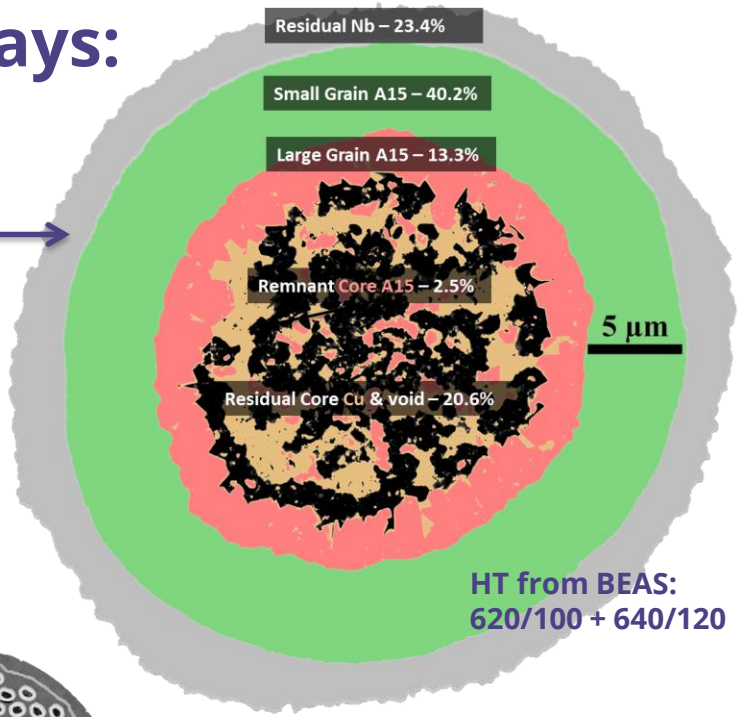
But much recent progress . . .

1. **PIT** - the **bundled barrier** has enabled **higher Sn** that allows **more A15. New HT** encourages small grains. **For the first time J_c >2600 A/mm² at 12 T**. However, tubes have broken and effects seem to be long range, suggesting that real J_c would be much higher - **need mechanical analyses**.
2. **RRP** (internal Sn) - **Nausite control HT**: significant improvement for small sub-elements, **enables high J_c *and* reduction in D_{eff}.** Is the FCC D_{eff} target in sight with Ta dividers? J_c (FCC) is not far off – perhaps it will be achieved with uniform H_{c2} distribution across the layer.
3. **APC** - detailed study of the Motowidlo conductor is underpinning our APC design effort - we clearly see both **refined grains AND a point pinning contribution**. The challenge now is to get H_{c2} up too.

PIT is held back in three major ways:

1. **About 1/4 of the A15 formed is LG A15 (13% of 53% area)**
   - This observation is nearly independent of reaction temperature (630°C-670°C) for full-length reactions.
   - **BUT**: We found that, ***early in the reaction***, **LG A15 formation IS temperature dependent**!
   - We try to **optimize the early hours of the HT to maximize SG A15 growth before LG A15 starts forming**. (Multistage HT)

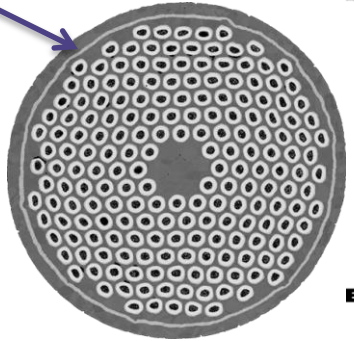




HT from BEAS: 620/100 + 640/120

2. **Much tube remains unreacted (21-24%)**
   - RRP is more effective with only ~10% un-reacted Nb
   - BEAS and CERN developed a new wire (bundle barrier) to drive up J_C without loss of RRR – previous talk
   - **Bundle barrier allows more tin, more reaction**
   - We apply multistage HT to bundle barrier wires

3. **There are filament breaks and locations of tin breach**
   - Filament breaches are often observed
   - These are **quite long, up to centimeters in length**, occurring more in **outer filaments**
     - Do inner filaments as a group have higher J_c?
       - Etch wire to measure I_c

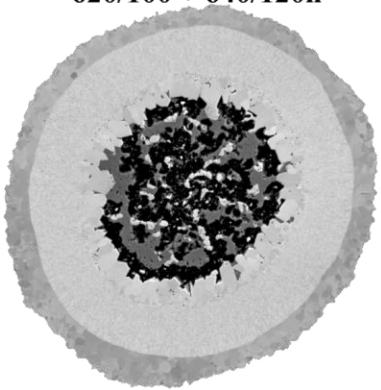


- Only **small grain A15** carries current
- Large and core grains of A15 do not carry any current

LG A15 formation is temperature dependent early in reaction

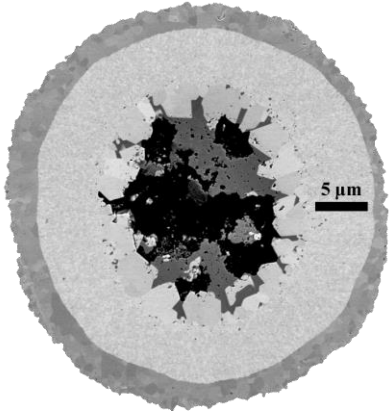
Full reaction in 620°C-670°C range

620/100 + 640/120h



SG 40.2%
LG 13.3%
J_c=2380 A/mm²
(12T,4.2K)

670/75



SG 42.3%
LG 13.7%
J_c=2404 A/mm²
(12T,4.2K)

Increasing Temperature

630°C 10h

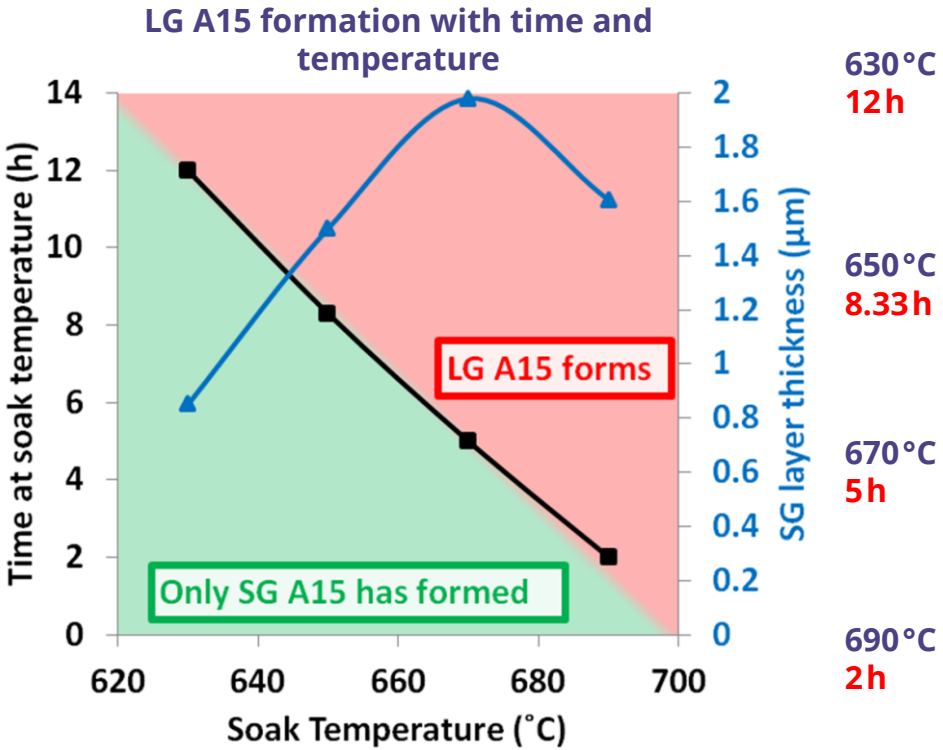
650°C 4.75h

670°C 4.5h

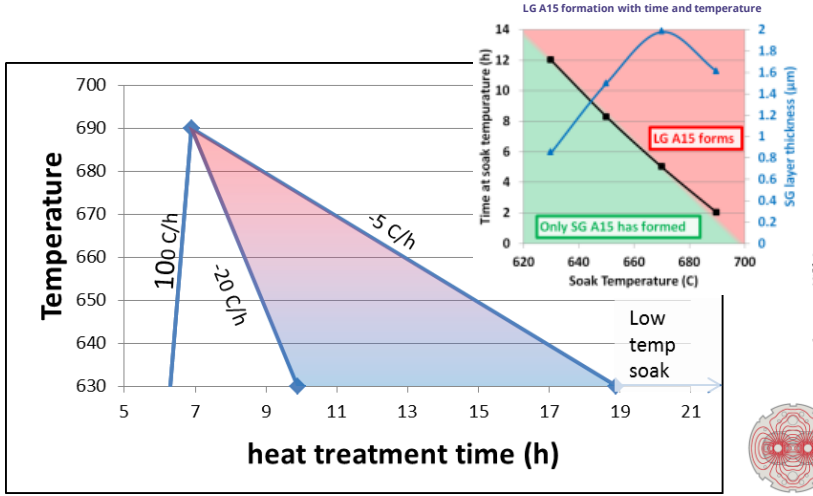
690°C 1.66h

Only SG A15 has formed

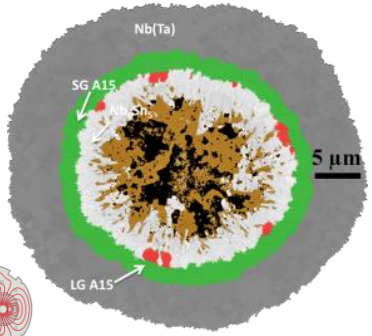
LG A15 has nucleated with Cu

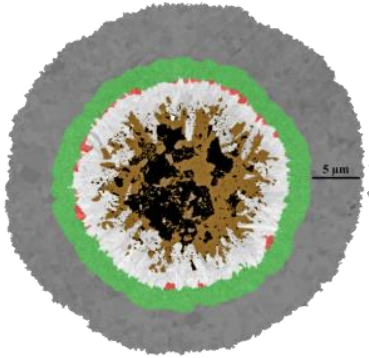


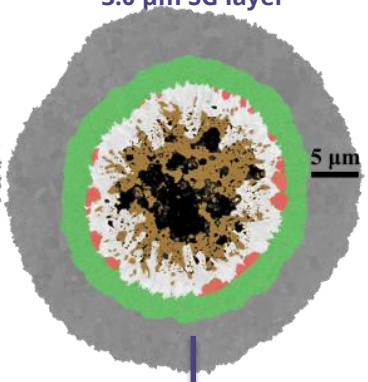
Multistage heat treatments further delay LG A15 formation while growing SG A15



Best single reaction stage (dwell)	~ 10% improvement with first HT.	50% improvement
670 °C 5 h – 2.0 µm SG layer	Stage A + 630 °C 13 h – 2.2 µm SG layer	Stage C + 630 °C 10 h – 3.0 µm SG layer

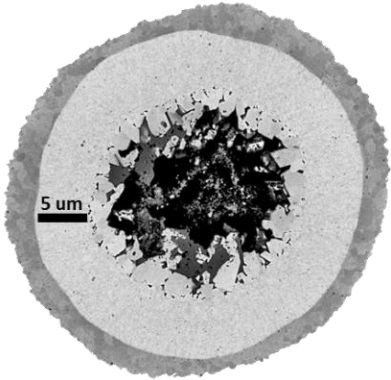






+ 200 h

Stage C + 630 ˚C 210h



LARP

Multistage heat treatments improve all phases and morphologies, but 12 T J_c decreases

Heat treatment description	Heat treatment (temp/dwell time)	I_c (A)	J_c (A/mm²)	J_c SG-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%
Multistage C	Stage C +630/210	491	2192	4926	24.0%	57.3%	1.2%	11.6%	44.5%
% change from recommended HT		-2%	-2%	-11.5%	+4.3%	2.3%	-52%	-12.8%	+10.7%

Transport properties went down

Microstructure improved SG/LG ratio improved from 3.0 -> 3.8

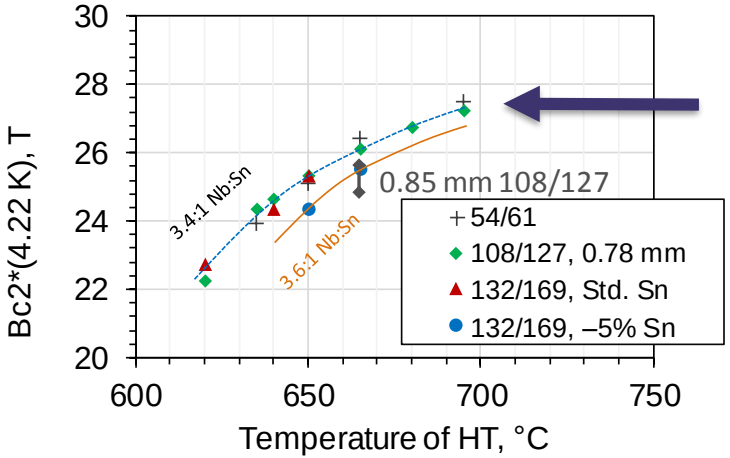
PIT summary

- HT optimization can reduce LG volume
- External barrier successfully implemented
  - Can this be fully exploited with Sn leakage?
- HT should be optimized for 16 T

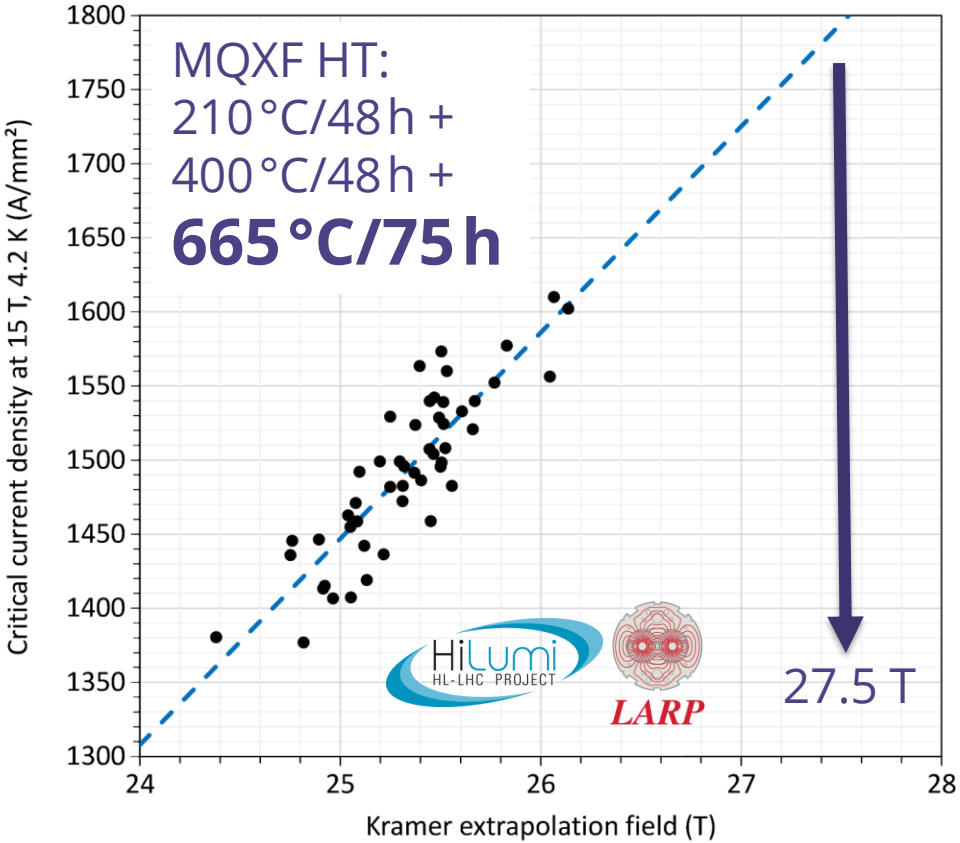
Potential for RRP Nb_3Sn

- Indications that HT needs to be reoptimized for 16 T
- Nausite-control HT provides breakthrough in J_c for small D_{eff}
- Quaternary doping may help achieve FCC targets

Conductor HT should target >680 °C for 15-16 T optimization B_{c2}* of 27.5 T



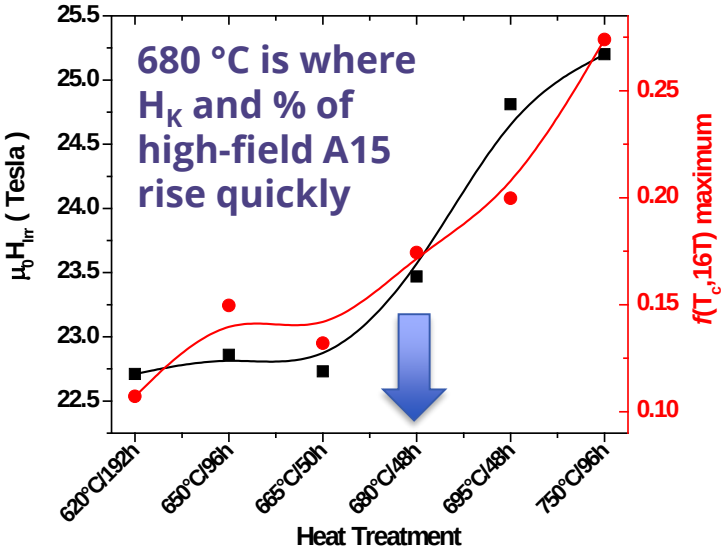
27.5 T



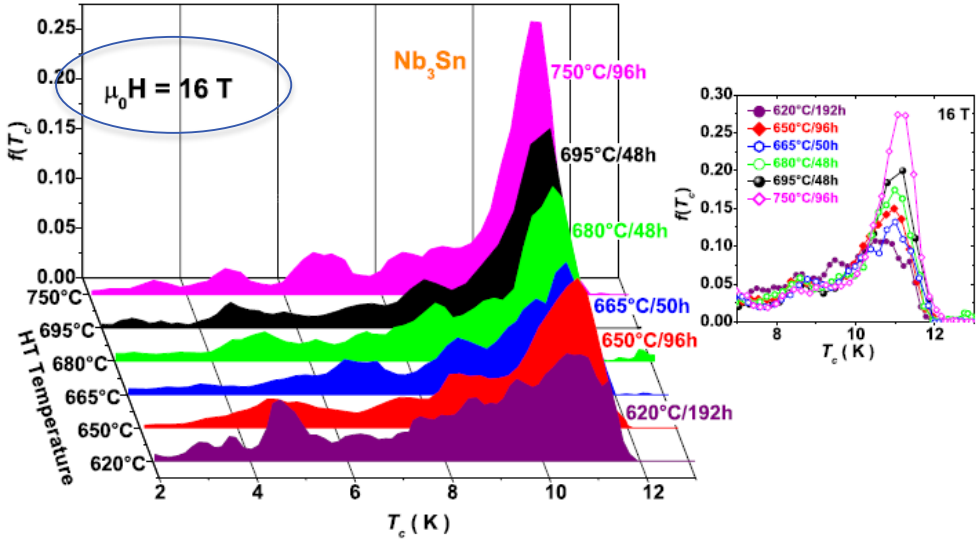
Trends suggest opportunity to achieve FCC J_c target by **reacting at 675-700 °C**

Adapted from L. Cooley – LTSW'17

High-field properties improve at > 680 °C



Extrapolated irreversibility field H_{Irr} at 4.2 K and the maximum of $f(T_c,16\ T)$ as a function of HT.

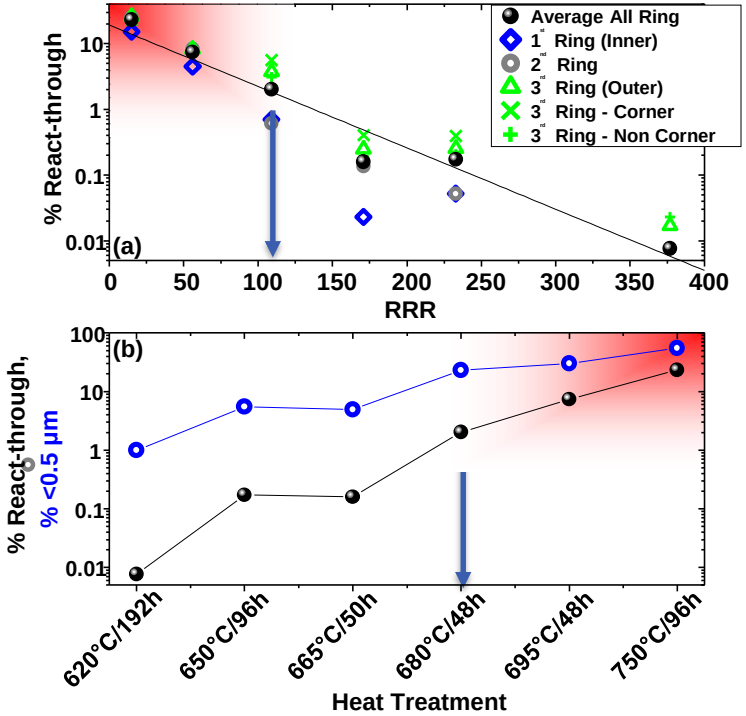


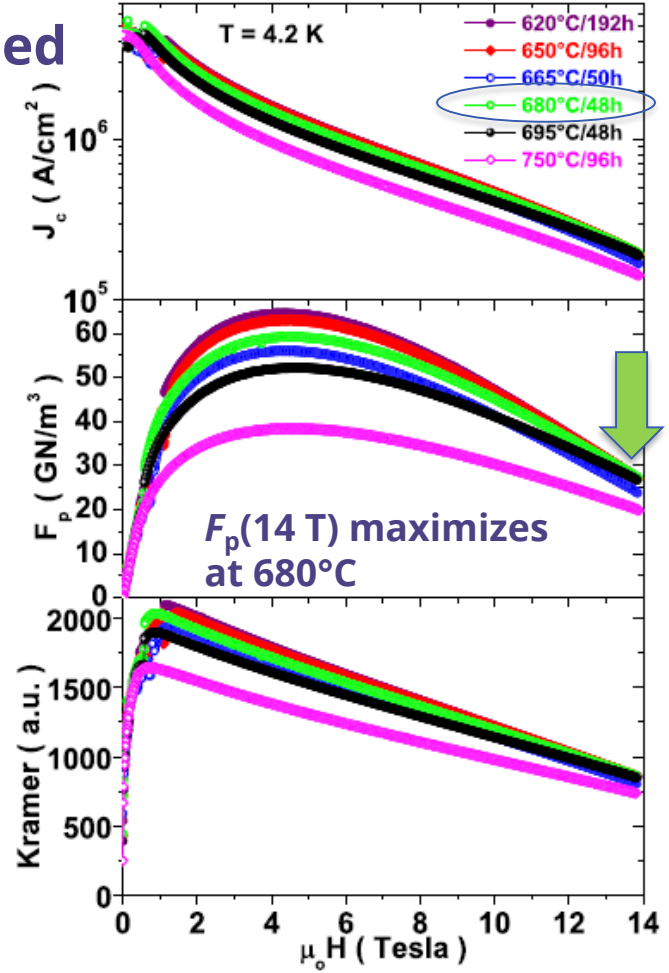
Ø= 0.7 mm, 54/61 stack

Tarantini *et al.* Supercond. Sci. Technol. 27 (2014) 065013 doi:10.1088/0953-2048/27/6/065013

But at >680°C, F_p & RRR may be compromised

1% of filaments react through at 680°C → RRR = ~100 or less





Ø= 0.7 mm, 54/61 stack

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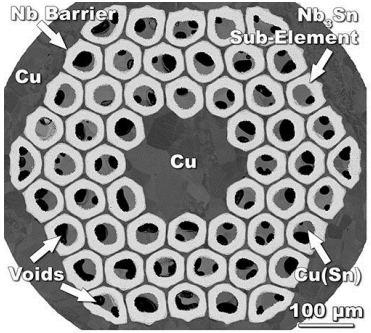
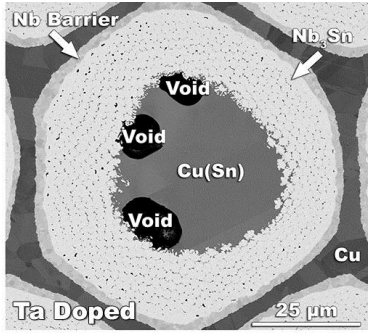
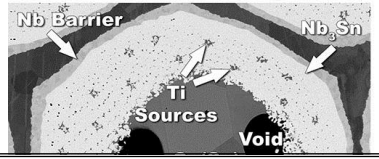
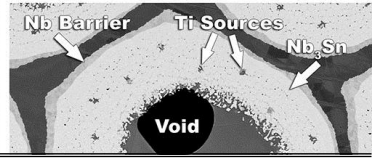
Reconsider Ta+Ti doping for 16 T optimization

- 54/61 high J_c strands heat treated at 640 °C/40 h with three different dopants :
  - Only Ta
  - **Ta+Ti**
  - Only Ti





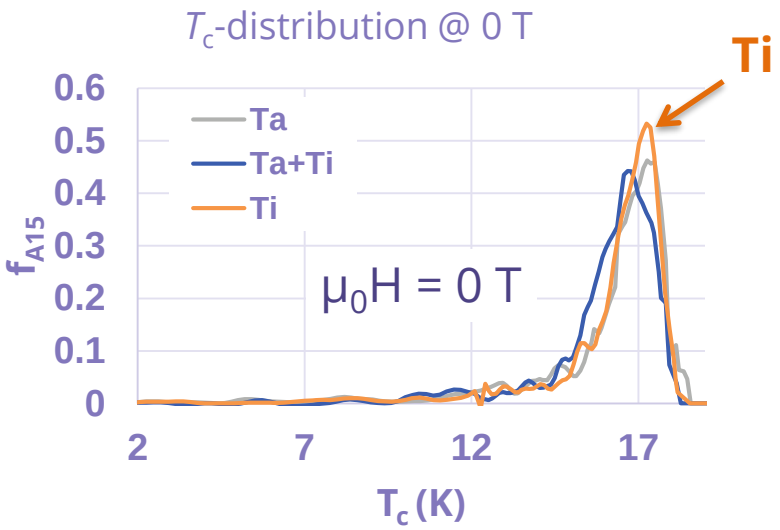


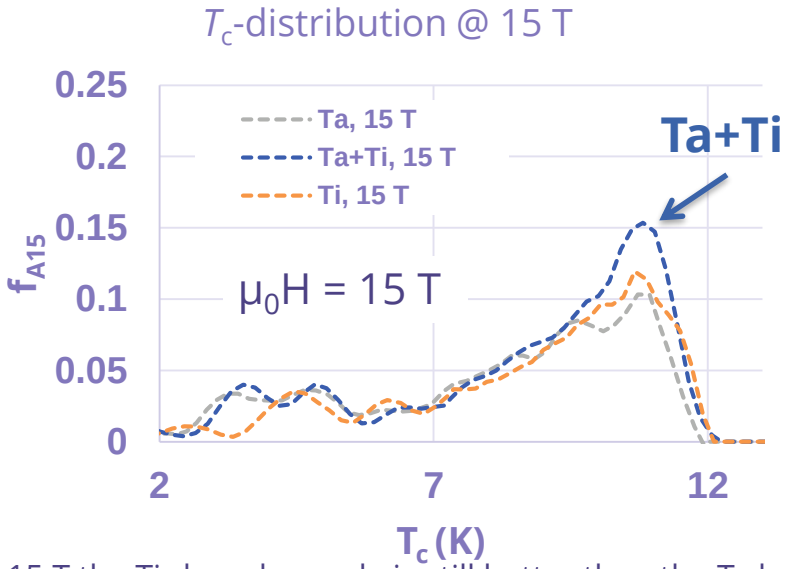


Ta+Ti Doping: Lower J_c(12 T) but Higher Kramer Field

Dopant	Dopant	Billet ID	Subs/ Stack	Final HT	Nb at %	Sn at %	Ta at %	Ti at %	$T_{c,Onset}$ K	$\mu_0 H_k$ T	Non-Cu J_c(12 T,4.2 K) A/mm²	A15 layer J_c(12 T,4.2 K) A/mm²
Ta	4 at. %Ta	8781	54/61	640 °C/40 h	72.46	25.17	2.37		18.4	22.66	2712	4860
Ta+Ti	4 at. %Ta +1 at%Ti	9362-5	54/61	640 °C/40 h	71.41	24.64	2.60	1.37	17.9	24.75	2622	4528
Ti	2 at%Ti	9415-BE	54/61	640 °C/40 h	74.87	23.40		1.73	18.1	23.75	2872	5065

Reconsidering Ta+Ti doping for 16 T optimization





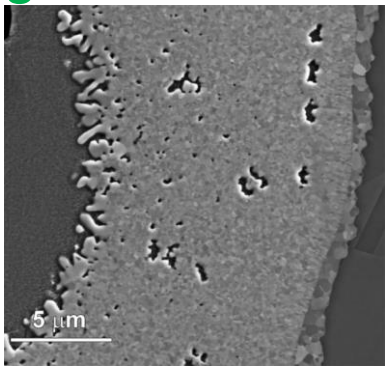
- At 0 T the larger homogeneity of the Ti sample is obvious
  - more intense peak, narrower distribution (just slightly lower T_c onset).
- Ta+Ti is the worst one:
  - the distribution peaks below 17 K and the peak is wider.

Analysis by Chiara Tarantini

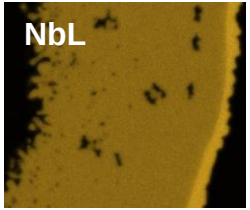
- At 15 T the Ti-doped sample is still better than the Ta but Ta+Ti is better than both (the volume fraction still in SC state at 15 T goes from 42-43% in the Ta or Ti samples to 50% in the Ta+Ti) .
- This suggests better performance of Ta+Ti at larger fields despite the inferior performance at 12 T.

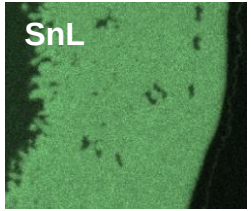
Micro-chemistry: Ti doped more homogeneous

Ti-doping Standard Sn



Z-H. Sung's analysis

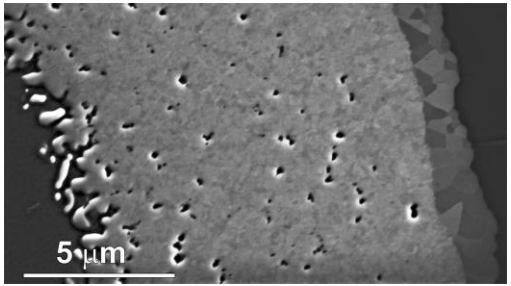


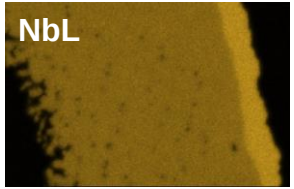


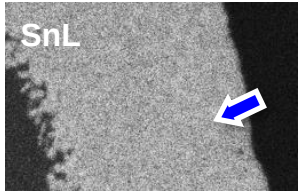
Sn very uniform

Ti lower between original filaments

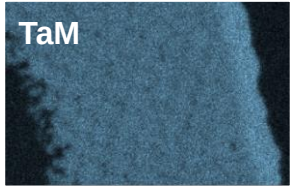
Ta-doping Standard Sn







Sn variation most noticeable toward barrier

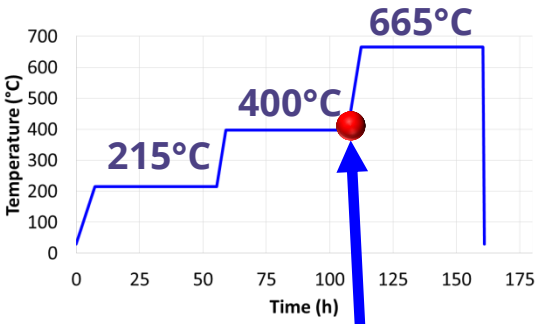


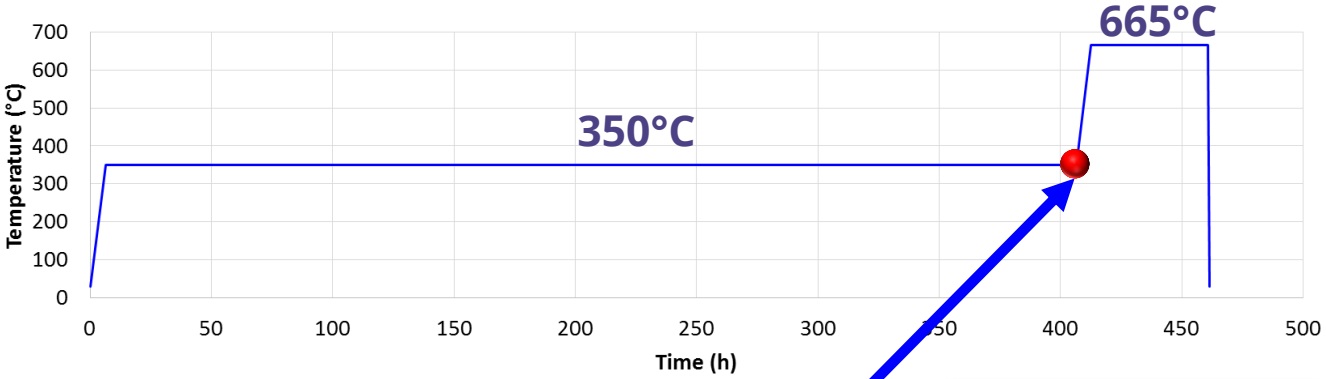
Clear honeycomb structure in the Ta map

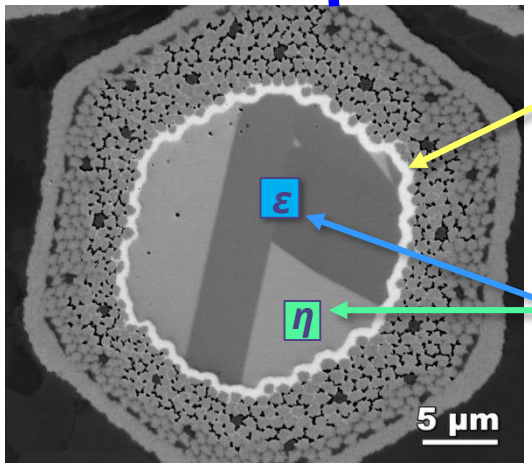
Doping: If Ti is close to max – Try Ti+Ta

- 7.5 Ta + 2 Ti is regarded as overdoped for RRP but is this the best for 16 T?
- Do we need custom Ta + Ti alloy?
- Can Nausite control gain us even more?
- Do we need doped APC Nb_3Sn . . .

Nausite Control: Use single 350 °C HT to optimize Nausite ring and increase the Cu diffusion

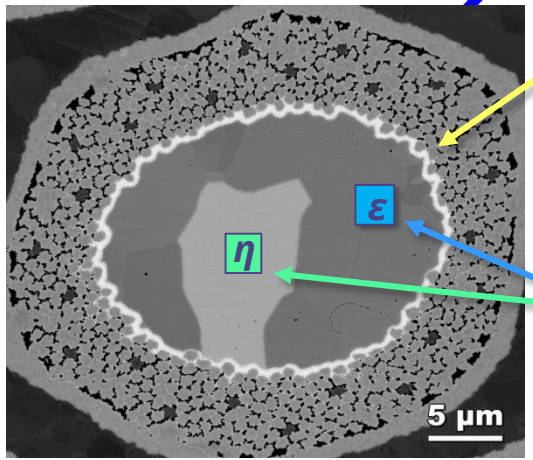






Nausite Membrane thickness 0.65 µm

31.3 at. % Sn

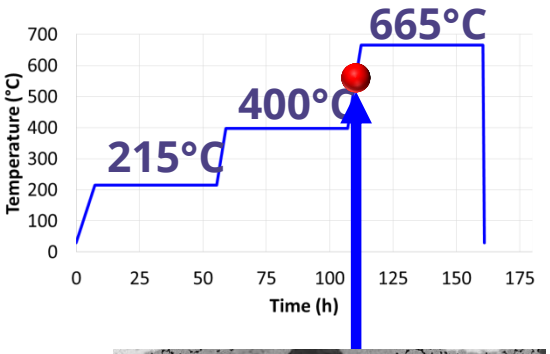


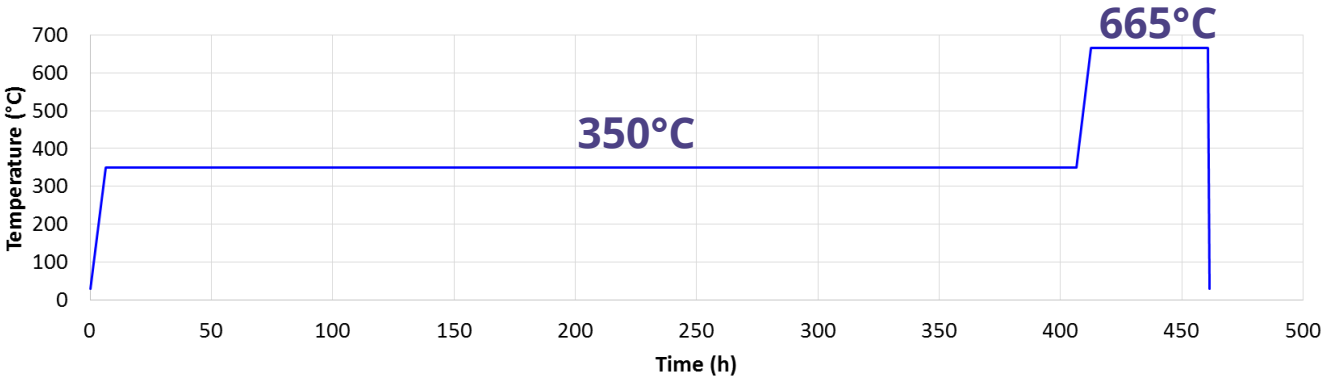
Nausite Membrane thickness 0.29 µm

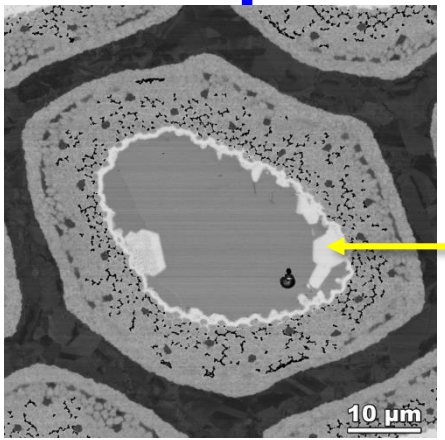
28.3 at. % Sn

Images and values are from a wire with D_s = 35 µm

Conventional 3-step HT lacks Nausite control

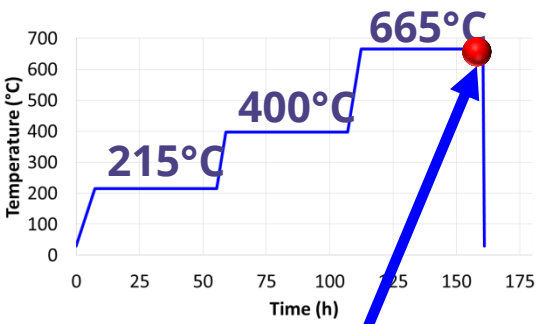


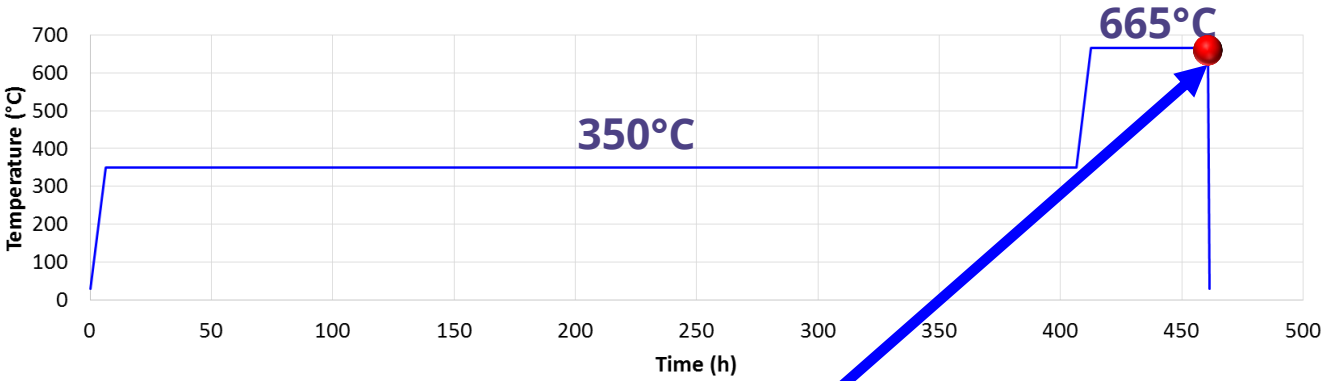


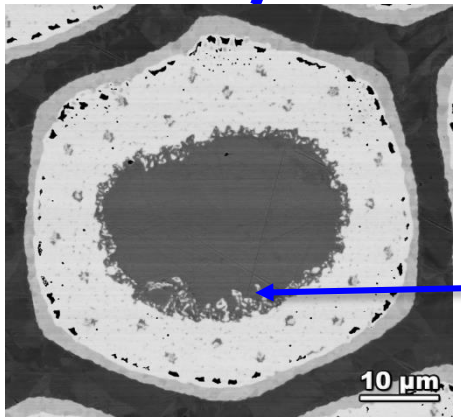


Remaining η produces liquid and more Nausite

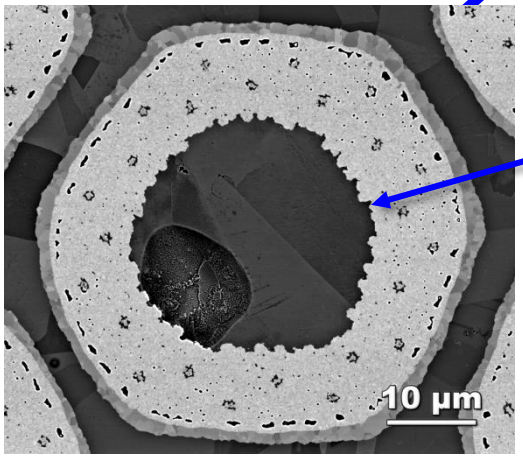
2-stage Nausite control HT reduces wasted Nb_3Sn







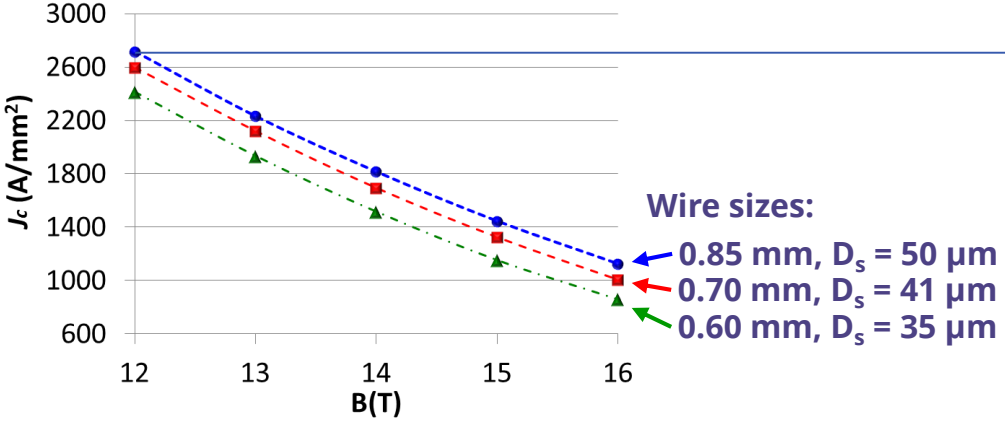
Excess Nausite ends up as disconnected Nb_3Sn



New Nausite control HT can eliminate wasted Nb_3Sn.

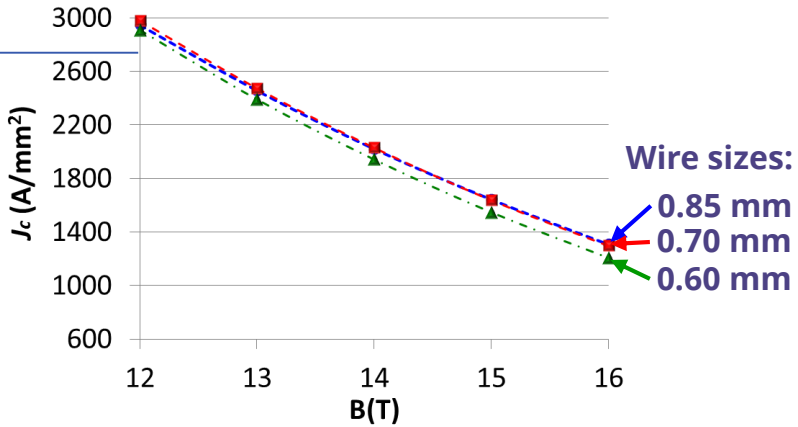
Nausite control HT: Minimizes liquefaction and increasing the critical current appreciably

Standard HT
(215 °C/48 h + 400 °C/48 h + 665 °C/50 h)



Wire Size	J_c (12 T)	J_c (16 T)	H_k (self field)
0.85	2719	1126	24.56
0.70	2598	1006	23.77
0.60	2415	859	22.89

New HT
(350 °C/400 h + 665 °C/50 h)



New data last week thanks to HanPing Miao and colleagues at Bruker-OST

Wire Size	J_c (12 T)	J_c (16 T)	H_k (self field)	RRR
0.85	2946	**1307**	**25.58**	304
0.70	2979	**1296**	**25.34**	244
0.60	2910	**1207**	**24.68**	140

Compare with Table from Bernardo Bordini this morning

B. Bordini and A. Ballarino Presentations at FCC Week 2017

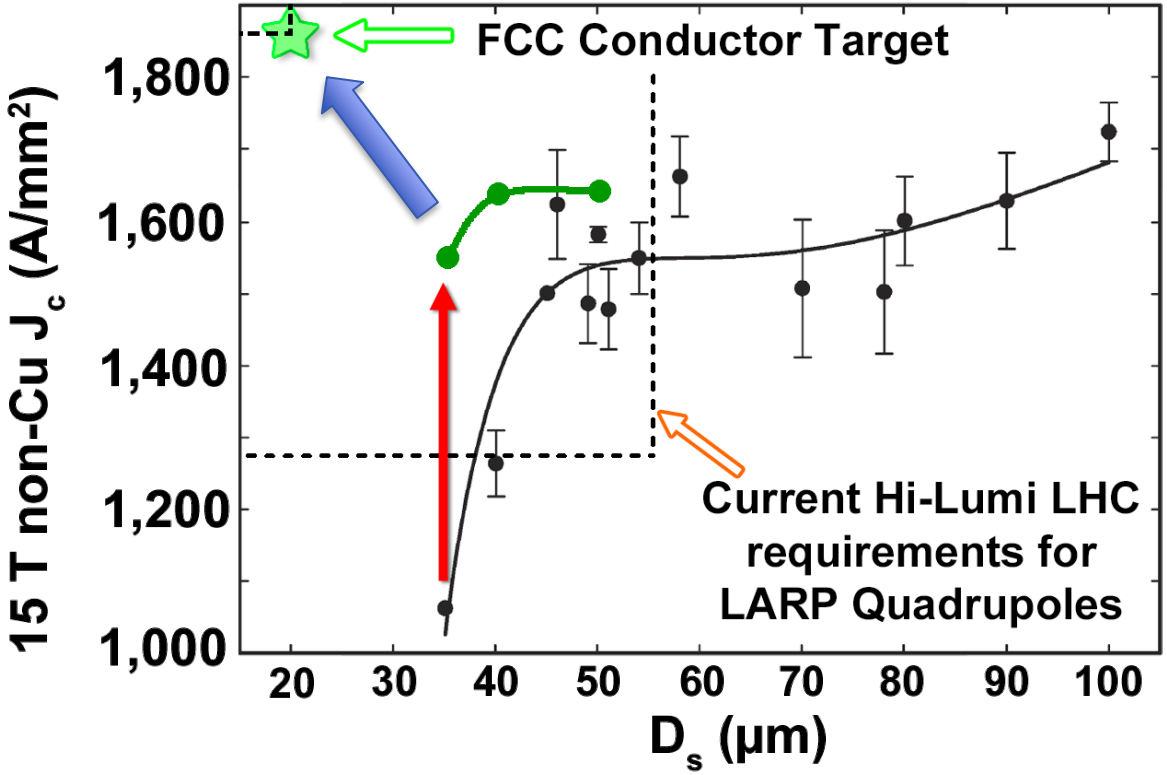
	Layout	Sub-Element size	J_c (12 T), *RMS* [A/mm²]	J_c (15 T), *RMS* [A/mm²]	B_{c2}(4.3 K), *RMS* [T]	J_c (16 T), *RMS* [A/mm²]	J_c (18 T), *RMS* [A/mm²]	Degradation J_c (15% rolling) [%]	Minimum RRR (15% rolling) -
0.7 mm RRP	108/127	46 µm	**2676**, *68*	**1410**, *58*	**24.5**, *0.39*	**1098**, *55*	**610**, *47*	0	>100
0.85 mm RRP	108/127	55 µm	**2835**, *44*	**1601**, *33*	**25.9**, *0.19*	**1289**, *30*	**785**, *25*	0	>100
0.85 mm Bundle Barrier PIT	192	39 µm	**2323**, *83*	**1342**, *49*	**26.7**, *0.1*	**1093**, *40*	**688**, *26*	5.5 %	>150

Wire Size	Billet	Stack	Sub-Element size	J_c (12 T)	J_c (16 T)	H_k (self field)	RRR
0.85	16630-2C	132	50	2946	**1307**	**25.58**	304
0.70	16630-2B	132	41	2979	**1296**	**25.34**	244
0.60	16630-2A	132	35	2910	**1207**	**24.68**	140

New Nausite Control HT

A significant advance for small D_{eff}

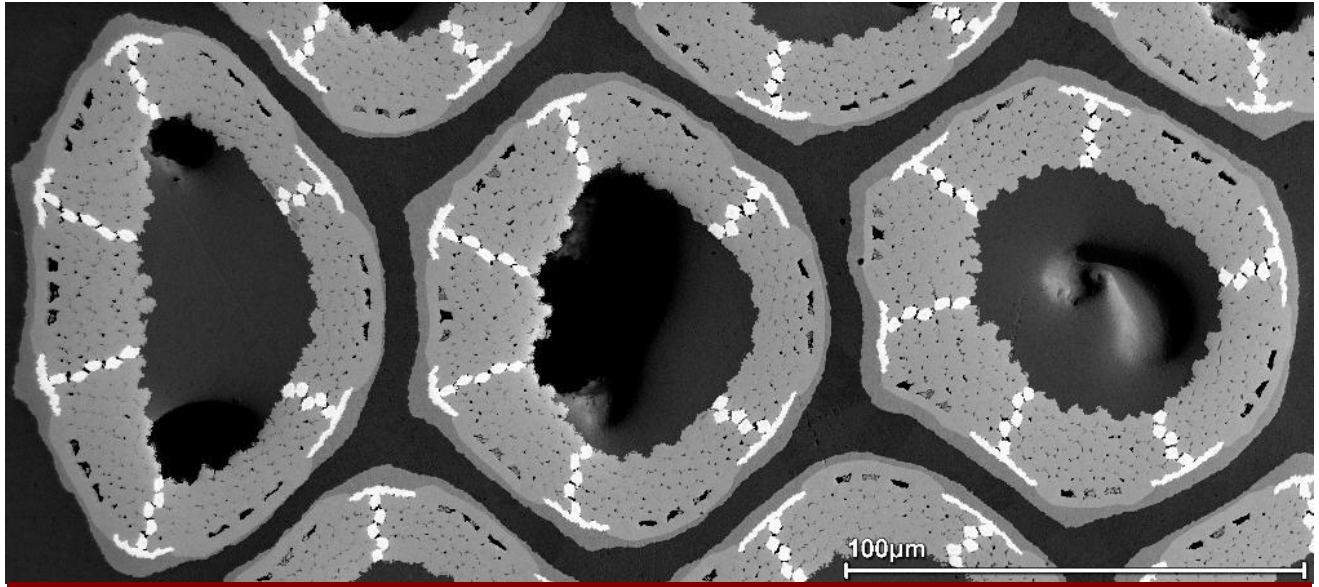
- Nausite control HT significantly increases J_c
- But more is needed!



D_{eff}

- With Nausite control can we can get to RRP J_c Plateau with 40 μm subelements
- Is 20 μm now possible with a simple sub-division?
- Will dividers interfere with the Nausite ring/membrane?

RRP Example using Ta Filaments to subdivide each sub-element

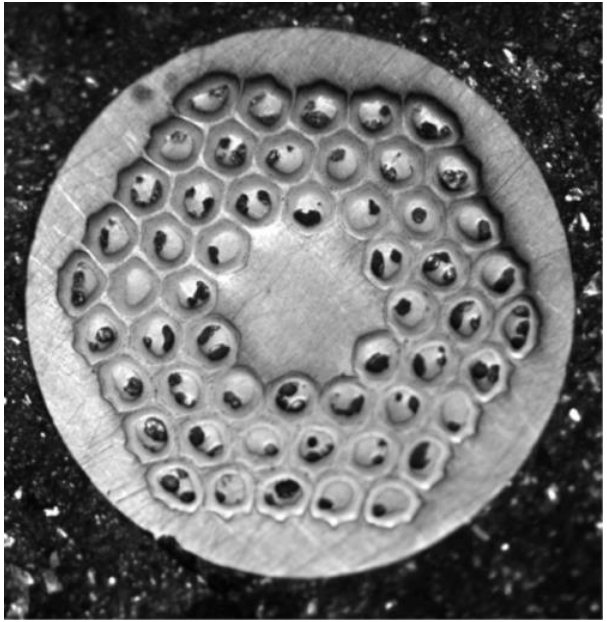


	Sub1 (outer)	Sub2 (center)	Sub3 (inner)
% of Non-Cu is Ta	4.8	5.4	5.1
% of Nb_3Sn Area lost to Dividers	9.1	10.6	9.9
% of Non-Cu is Nb_3Sn	52.7	51.3	51.6

- Previous attempts assumed far more sub-division would be necessary:
  - 6 dividers = ~10% loss in Nb_3Sn area

Sn core size was deliberately reduced in this composite

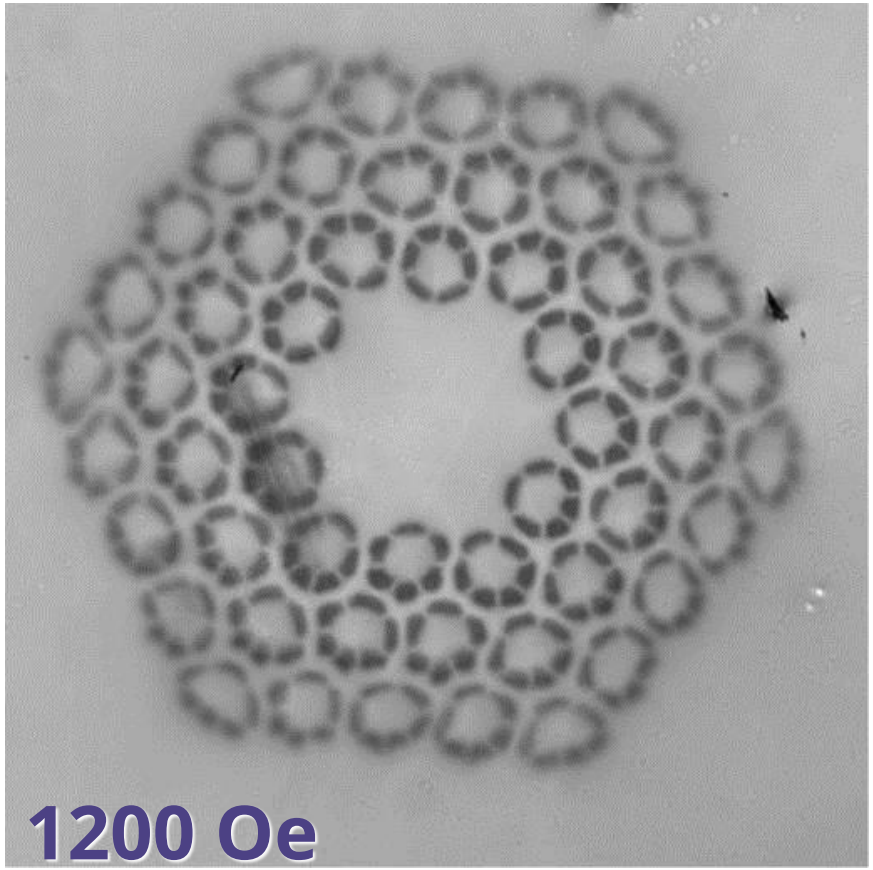
MO imaging demonstrates successful subdivision



Zero Field Cooled, T=6 K

All sub-elements divided *and* cores penetrated

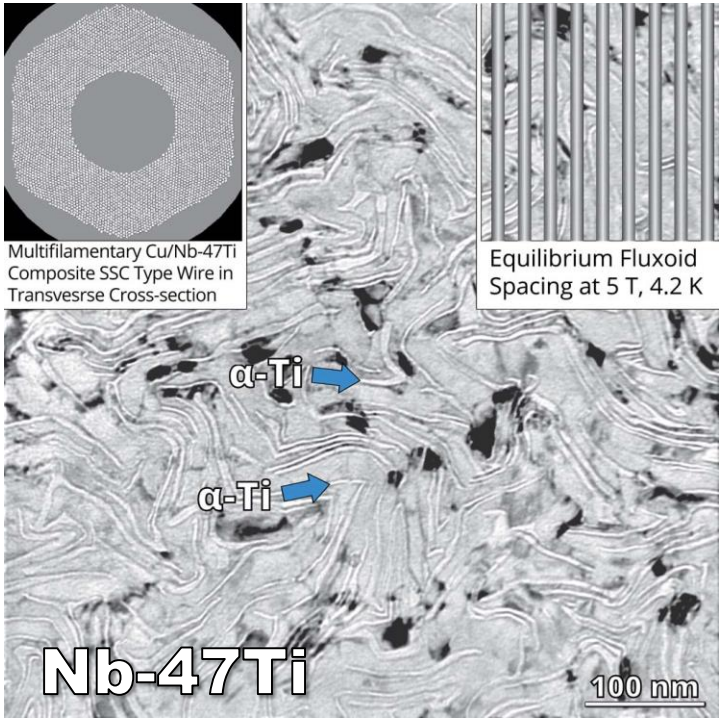
Increasing Field

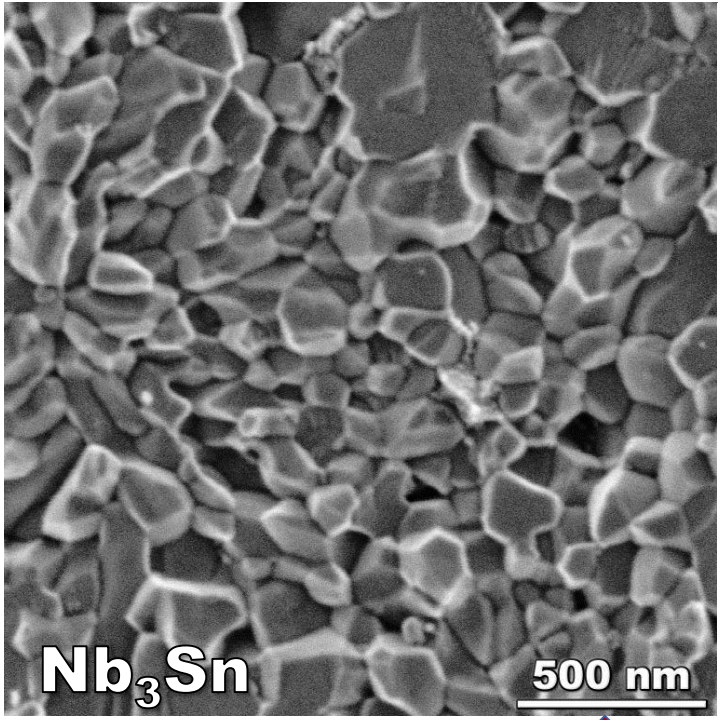


MO imaging by Anatolii Polyanskii

Pinning Nb-Ti vs Nb_3Sn

- Grain boundary pinning in Nb_3Sn is not as efficient as α-Ti pinning in Nb-47Ti

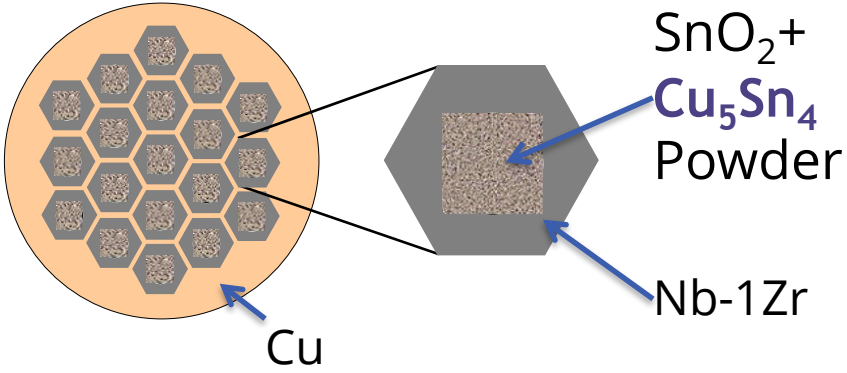




APC: PIT Approach

OSU (see next talk) has shown that the internal oxidation of Zr in Nb-1Zr developed at GE for tape can be applied to wire . . . .

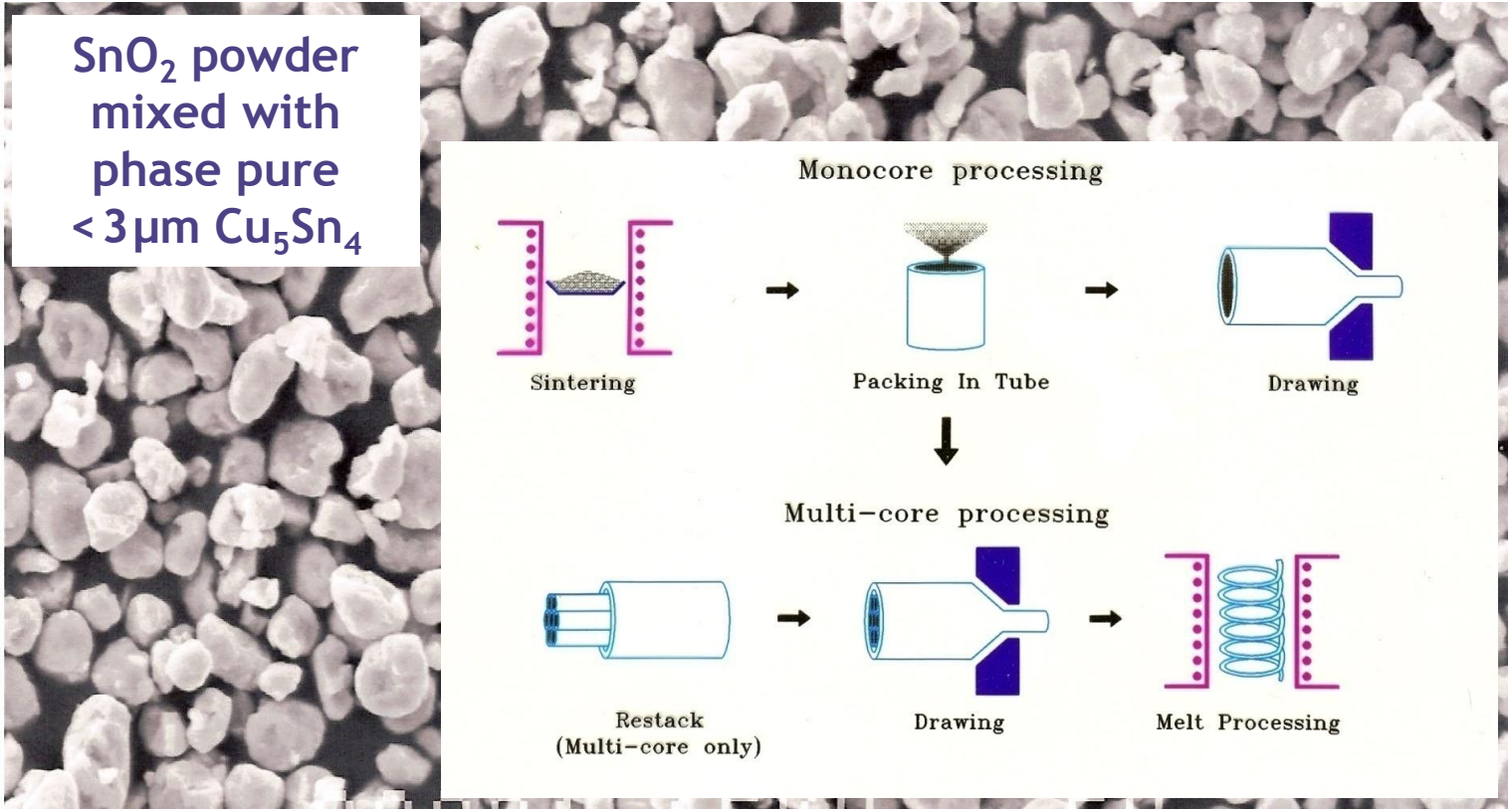
IF there is no Cu to block Oxygen diffusion



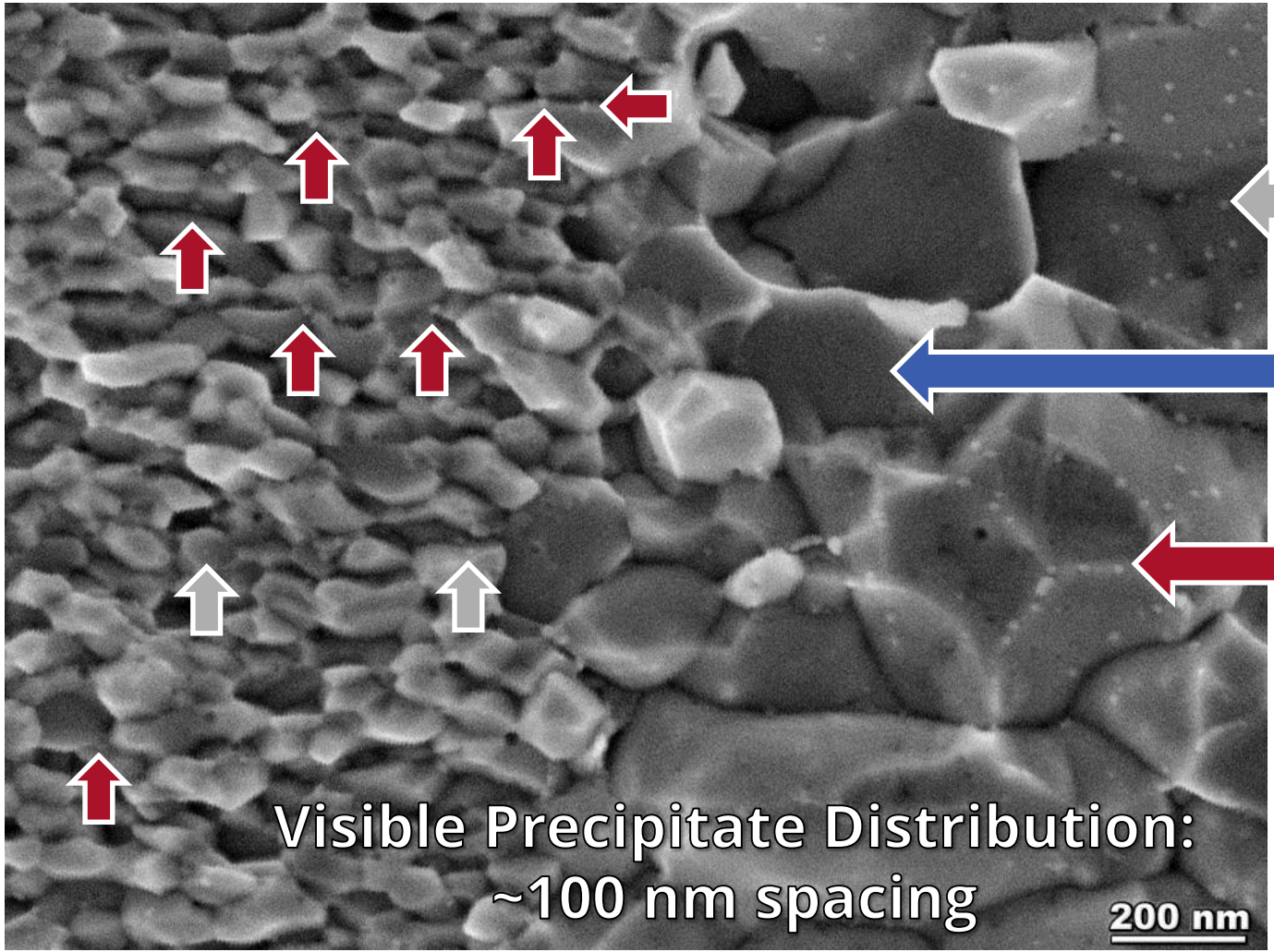
SupraMagnetics PIT APC

SupraMagnetics (Cu_5Sn_4+SnO_2) PIT Process

SnO_2 powder mixed with phase pure < 3 µm Cu_5Sn_4



Precipitates prefer GB intersections



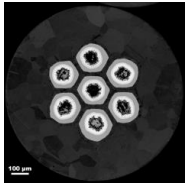
Precipitates at intergranular fracture surface

Transgranular fracture: no precipitates

Precipitates along GB intersection

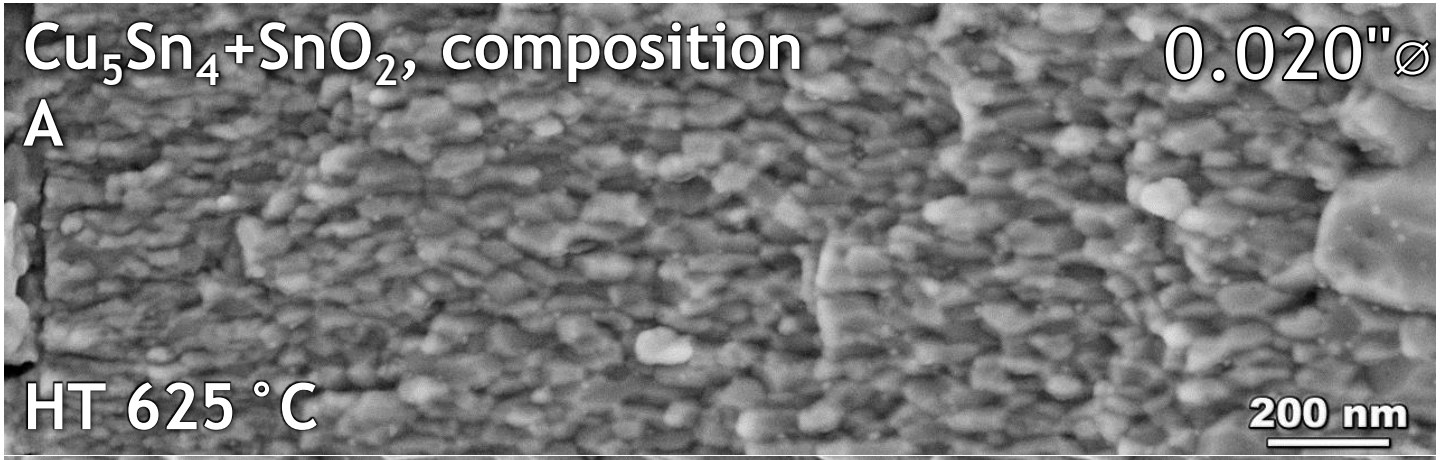
But not all precipitates may be within FESEM resolution, and fractographs reveal only intergranular surfaces.

A15 Grain Size Sensitive to Composition



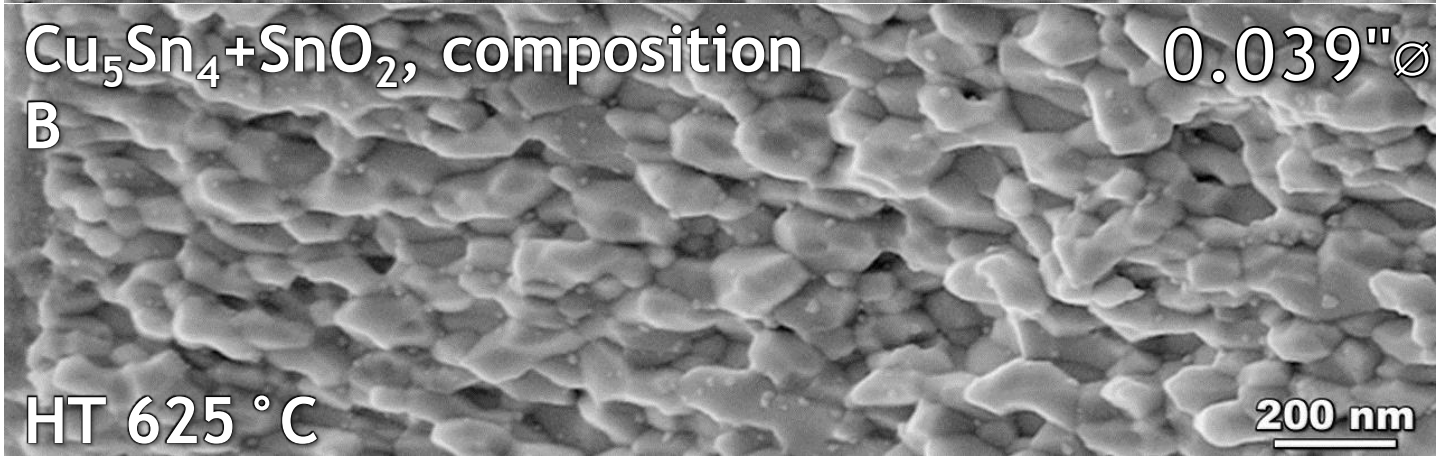
300 h/
625 °C

Nb



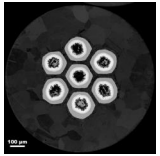
Core

Nb



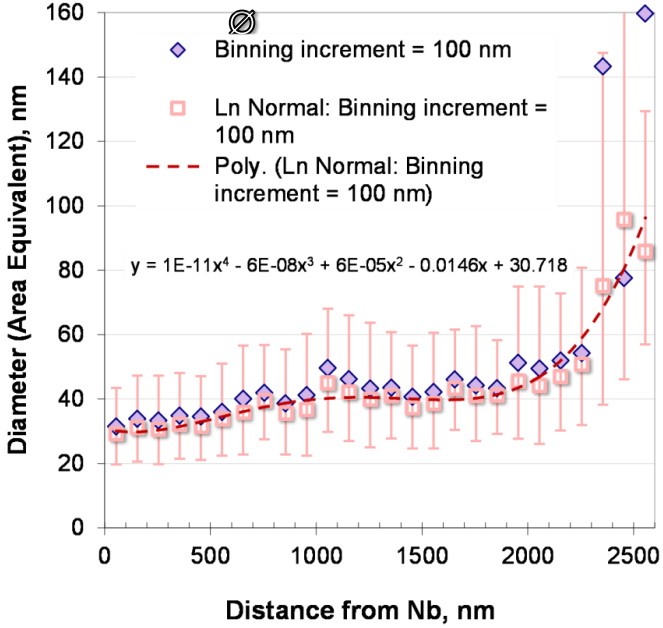
Core

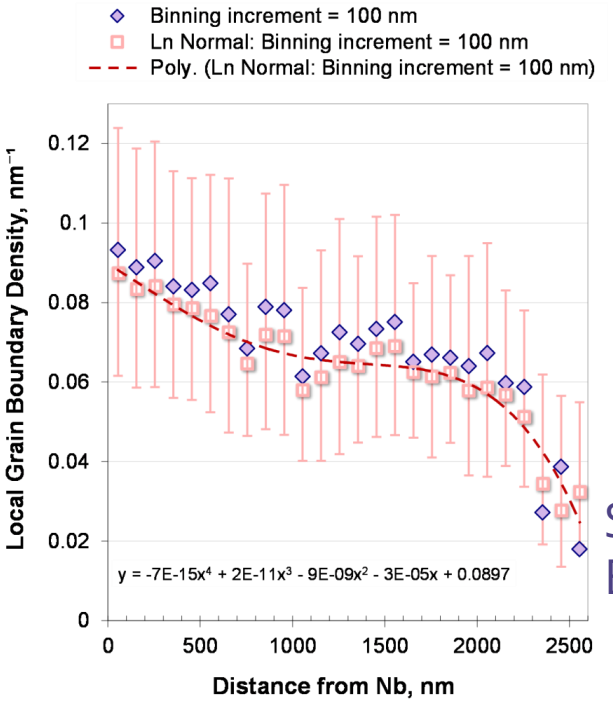
Microstructural Gradients: Suggest grain growth



300h/625 °C

0.020"



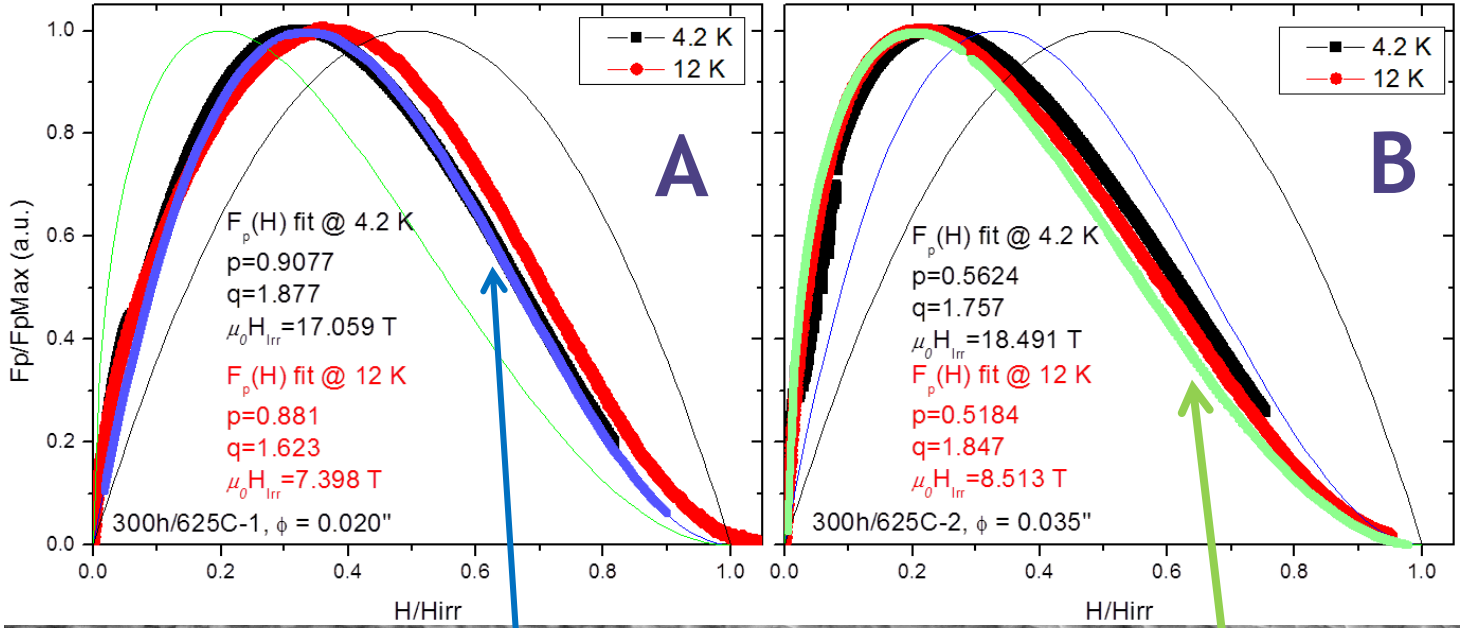


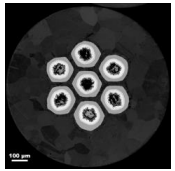
- Smallest grains ~ 30 nm diam. furthest from core (i.e. newest grains)
- Middle of layer ~ 40 nm

Steep gradient in Grain Boundary Density

Cu_5Sn_4+SnO_2, composition A

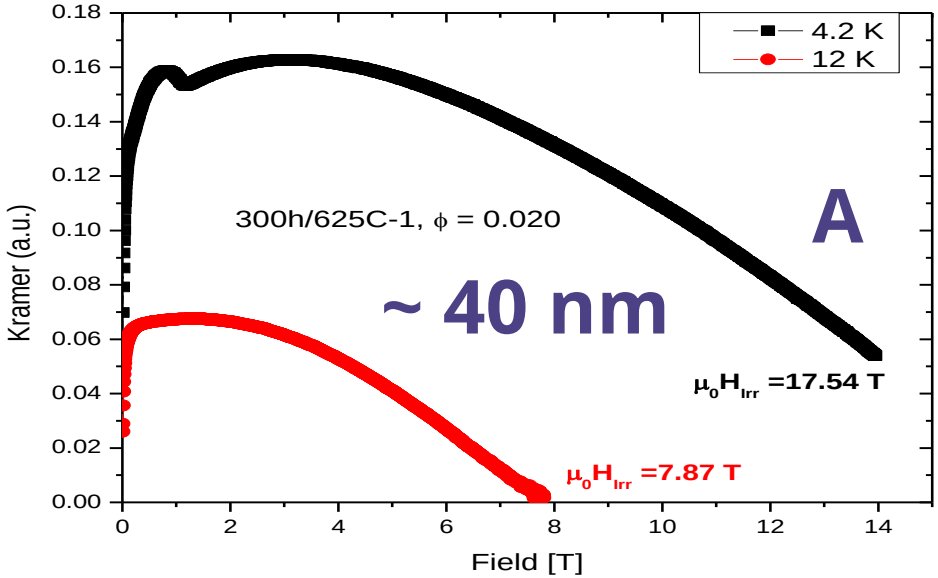
Magnetization: A shift in the peak pinning force curve for Compo A compared to Compo B.



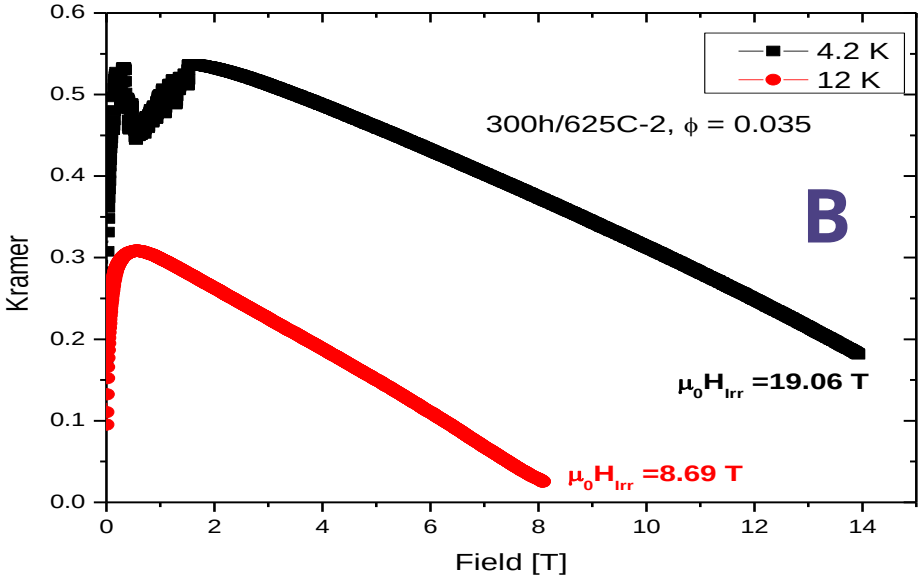


300h/625 °C

Kramer plot: Smallest grains = Less GB-like, less H_{irr}



Suppressed H_{irr}



More grain-boundary-like

PIT APC Summary

- Preliminary results show an average grain size as small as 30 nm.
- A shift in the peak pinning force curve toward higher magnetic field is demonstrated.
- The volume percent SnO_2 is an important parameter in the control of grain size and homogeneity of the A15 layer.
- Additional control is available through HT.
- These results provide strong evidence for a new and economical route to a very high current density Nb_3Sn strand.
- But there is evidence that the H_{irr} is suppressed for small grain sizes.
- A different approach may be required to meet FCC targets.

Recent progress . . .

1. **PIT** - the bundled barrier has enabled higher Sn that allows more A15 and new HT enable a more favorable SG/LG ratio - for the first time J_c >2600 A/mm² at 12 T.
   - However, tubes have broken and effects seem to be long range, suggesting that real J_c would be much higher - need for mechanical design.
2. **RRP** (internal Sn) - Nausite control HT: significant improvement – effective reduction in D_{eff}. With Ta subdivision the FCC defective target is in sight. J_c (FCC) is not far off - and future work not presented here is addressing a more uniform H_{c2} distribution across the layer.
3. **APC** - clearly see both a refined grain AND a point pinning contribution. The challenge now is to get H_{c2} up too.
   - The next talk will focus on new developments in APC conductors

Acknowledgments

- We would like to thank
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