### Microstructure -Property Correlations in Superconducting Wires

Peter J. Lee

With contributions from:
David Larbalestier,
Shreyas Balachandran,
Chiara Tarantini,
Anatolii Polyanskii,
Fumitake Kametani,
Bill Starch, and Yi-Feng Su

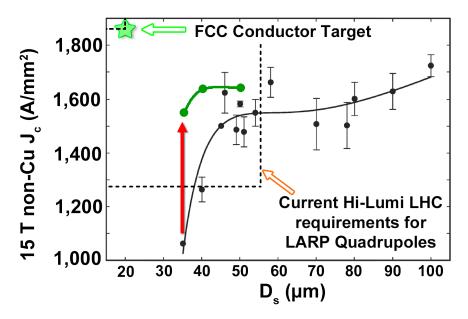




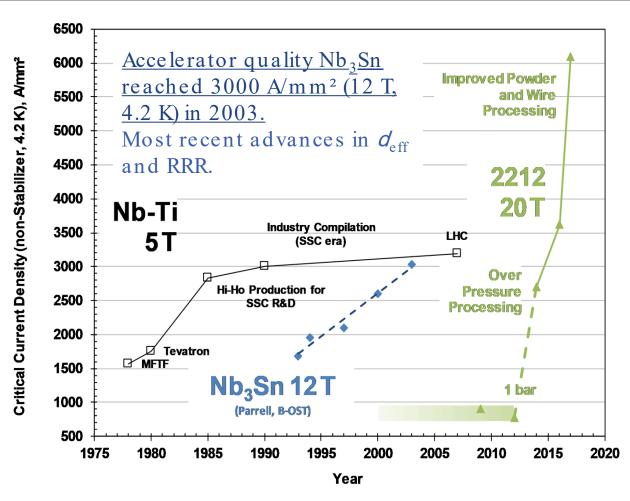
And past ASC graduate students: Jim McKinnell, Jeff Parrell,
Christoph Meingast,
Matt Jewell,
Charlie Sanabria,
Chris Segal and undergraduates
Jonathon Cooper and
Benjamin Walker

### Production Nb - Ti and Nb 3 Sn 4s have plateaued

• But significant increases in Nb<sub>3</sub>Sn critical current density are required to reach FCC targets



Charlie Sanabria et al 2018 Supercond. Sci. Technol. 31 064001

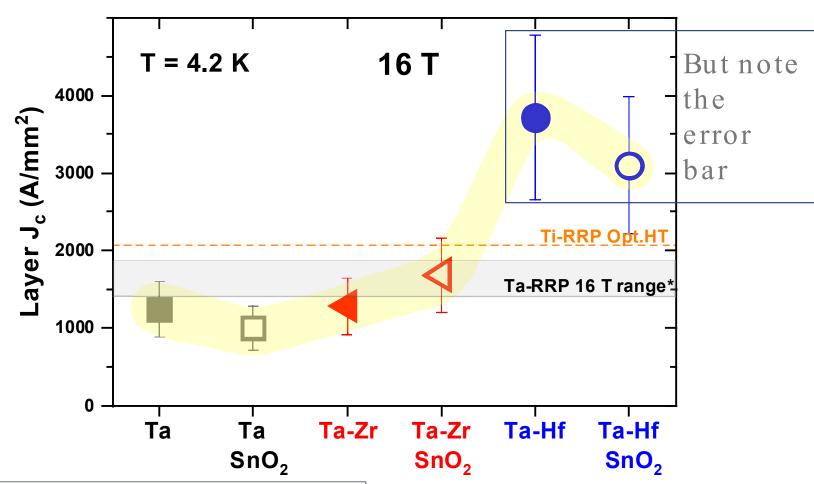


Nb<sub>3</sub>Sn data: J. A. Parrell, Adv. Cryo. Eng. 50, pp. 369–375, 2004: <a href="http://dx.doi.org/10.1063/1.1774590">http://dx.doi.org/10.1063/1.1774590</a> 2212 data compilation: courtesy Jianyi Jiang, Applied Superconductivity Center - NHMFL



#### New Hf results suggest that major improvements are still possible

- Xingchen Xu (1MOr2B-05) and co-workers at OSU,FNAL and HyperTech have shown GE's Nb-1Zr APC approach for Nb<sub>3</sub>Sn can be adapted to wires using Nb1-Zr + SnO<sub>2</sub> producing high pin densities and high critical currents.
- On Monday we reported that  $SnO_2$  may not be needed if Hf is used instead of Zr (Shreyas Balachandran's talk on Monday: 1MOr2B-06)
  - We see the potential to meet or even exceed FCC Nb<sub>3</sub>Sn targets with or without SnO<sub>2</sub>!



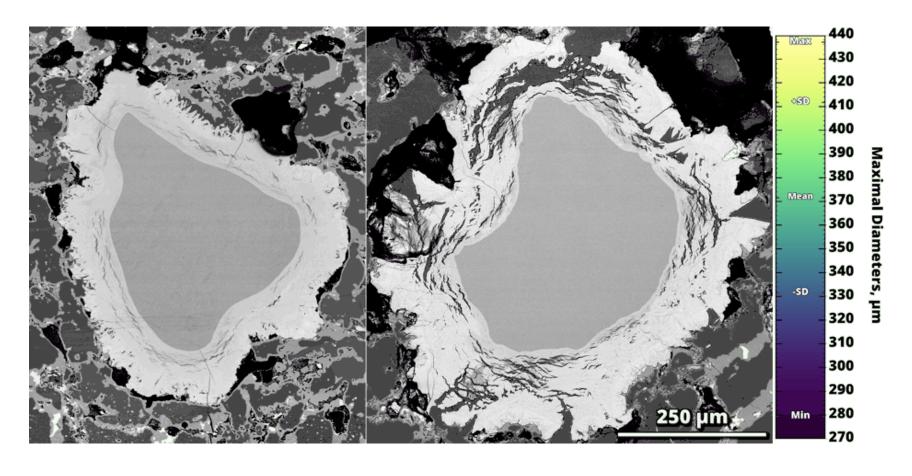
http://arxiv.org/abs/1811.08867 S. Balachandran et al., "Beneficial influence of Hf and Zr additions to Nb4at.%Ta on the vortex pinning of Nb<sub>2</sub>Sn with and without an O source," arXiv:1811.08867[cond-mat], Nov. 2018.





### Error bar originates from inhomogeneity of prototype

- Variations in core diameters along the length and rod asymmetry illustrated here.
- Variations in Nb<sub>3</sub>Sn layer thickness



http://arxiv.org/abs/1811.08867 S. Balachandran et al., "Beneficial influence of Hf and Zr additions to Nb4at. "Ta on the vortex pinning of Nb<sub>3</sub>Sn with and without an O source," arXiv:1811.08867 [cond-mat], Nov. 2018.

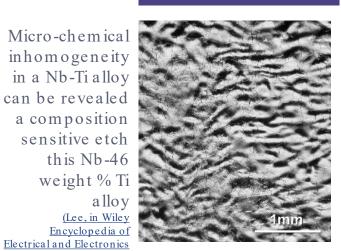




# Addressing homogeneity was a key factor in developing our understanding of Nb-47Ti

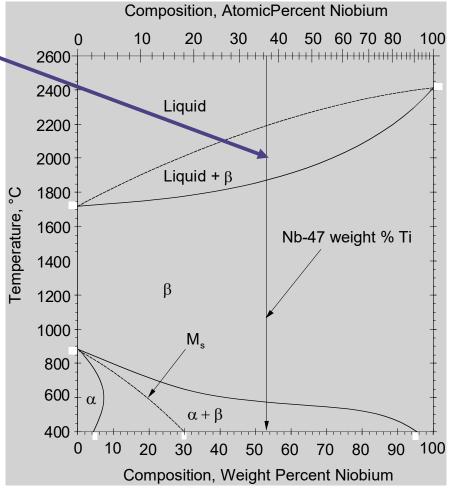
- Microscopy techniques only ever sample small fractions of the wire volume.
  - If the wire is inhomogeneous it is hard to extract quantitative data that can be compared to the properties.
  - This was a major issue for Nb-Ti because it was hard to manufacture a homogeneous alloy.

Large phase separation produces coring in alloy



Moffat, D. L. and Kattner, U. R. "The Stable and Metastable Ti -Nb Phase Diagrams" *Metallurgical Transactions A*19, no. 10 (1988): 2389–2397. doi:10.1007/BF02645466

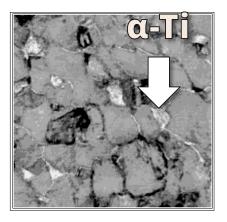
Engineering, 1999).



### The precipitate morphology Nb -47Ti is very sensitive to composition

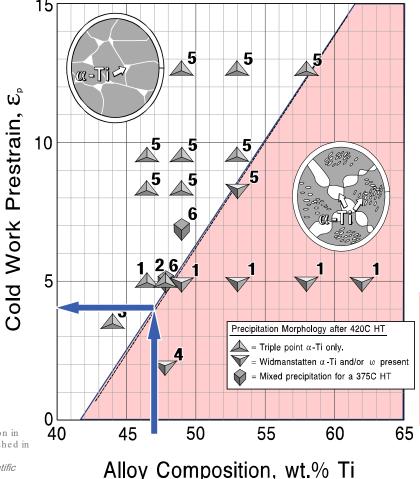
- The higher the Ti content the higher the prestrain required to avoid Widmanstätten precipitation
- If the cold work prestrain is high enough and the alloy is homogeneous the microstructure will be homogeneous.

#### Good

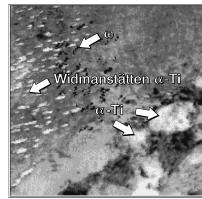


α-Ti only at grain boundary intersections – uniform size and distribution.

P. J. Lee, J. C. McKinnell, and D. C. Larbalestier, "Progress in the Understanding and Manipulation in High & Nb-Ti Alloy Composites," Originally published in Proc. of New Developments in Applied Superconductivity, ed. Y. Murakami, World Scientific Presspp. 357-362, 1989.



**Bad** 



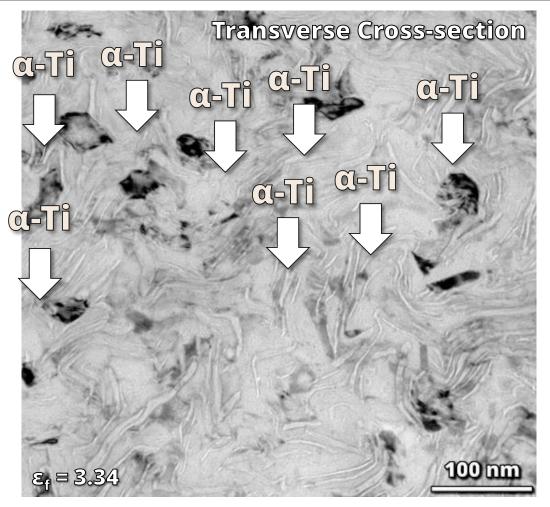
Precipitate is very inhomogeneous in size and intragranular Widmanstätten  $\alpha$ -Ti causes severe local work hardening.



### Nb-Ti: Anisotropy and Interpretation

- Before Focused Ion Beam TEM sample preparation, it was much easier to make cross-section of longitudinal lengths of Nb-Ti/Cu composites.
  - In LCS the importance of  $\alpha$ -Ti is not apparent

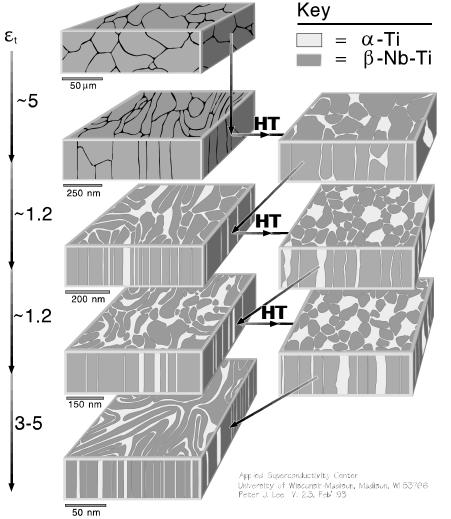
• By developing new plating and electropolishing techniques it was possible to view transverse cross-sections of any size.



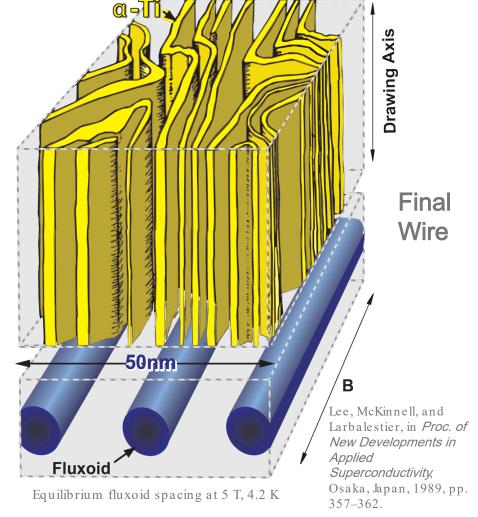
Lee and Larbalestier, Acta Met 1987 6x10h@405°C



### Multiple HT and Drawing Cycles: Folded Sheets of α -Ti

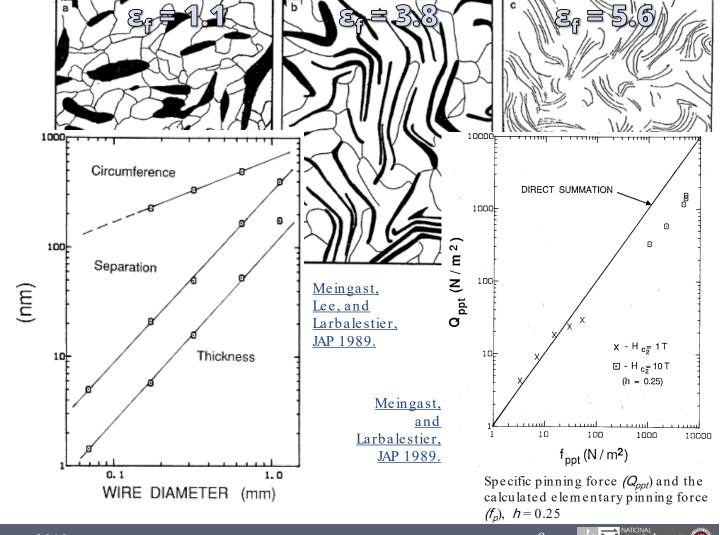


• The multiple heat treatment and drawing cycles produce a highly aspected microstructure



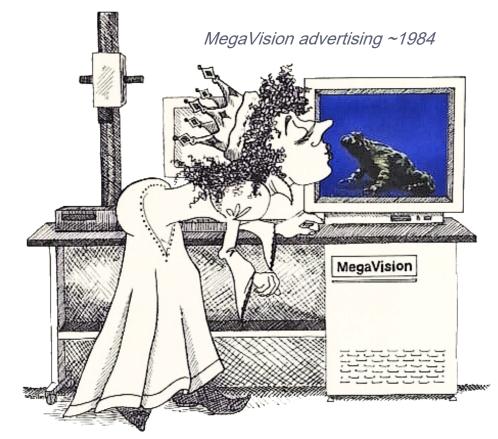
### But Even with high quality TEM images manual quantification was required

- The complexity of the TEM contrast (diffraction contrast + atomic number contrast + foil thickness) still meant that manual measurement of the microstructures was necessary.
- However, with the excellent homogeneity of the microstructure the trends were clear and reliable correlations could be made between properties and microstructure



### The digital revolution: Mega Vision 1024XM – introduced 1984

- Our first digital IA system used for all Phase II R&D SSC Nb-47Ti strand analysis
- The 1024XM had 1 Megapixel resolution, up 32 MB of real-time internal image memory and 350 Mbyte/second processing speed which was remarkable for the time ... so was the original MSRP for a complete system: \$200,000.
- Output: Area, centroid, boundary length, form factor.



You've got to see it to believe it!

Many thanks to Dave Sutter at DOE-HEP: perhaps the first digital IA instrument for superconductivity



### We the Mega Vision we could combine TEM images at different tilts to remove diffraction contrast and leave mostly Z-contrast

Nb-47Ti  $\varepsilon_{\rm f} = 3$ 

Mega Vision 1024XM processing 1994



TEM images aligned and then combined by "max intensity" – All now possible in Image J/Fiji for free!

Bright Field (SAD)

at different tilt

diffraction

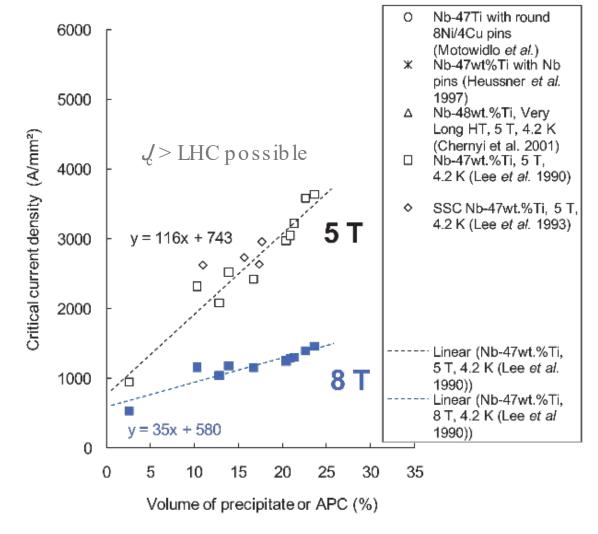
contrast)TE

angles (changing

TEM Images taken

#### Tedious but effective . . .

- The microstructure/property relationships for Nb-Ti were quantified with these relatively primitive (and expensive) digital tools combined with painstaking TEM.
- Now we can use FESEM-BSE images at most relevant magnifications.
- These later results show that critical current densities beyond the LHC are possible with new alloys and APCs



#### The Field Emission SEM revolution

- When we acquired our first Field Emission Scanning Electron Microscope in 1997 it made most TEM work obsolete!
  - 1 nm spatial resolution
  - High-signal lownoise Z-contrast Backscattered electron images
  - EDS and subsequently EBSD

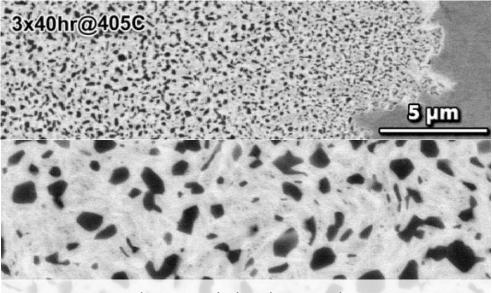
IGC Processed Nb44-17Ta cross-section for the Fermilab IR-Quad R&D program



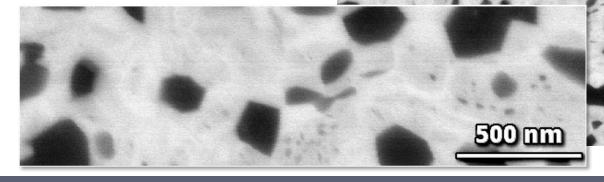


Now have intensity peaks for each phase for auto thresholding . . . But why are they not distinct?





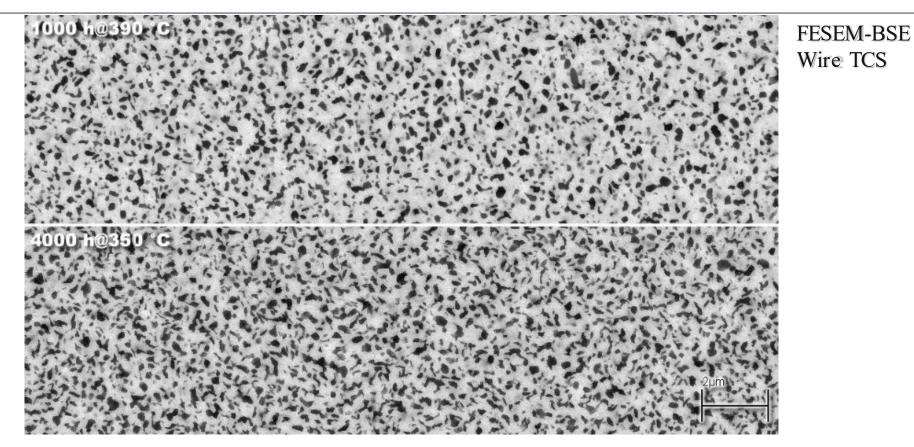
We can see the precipitation and compositional variation with great detail



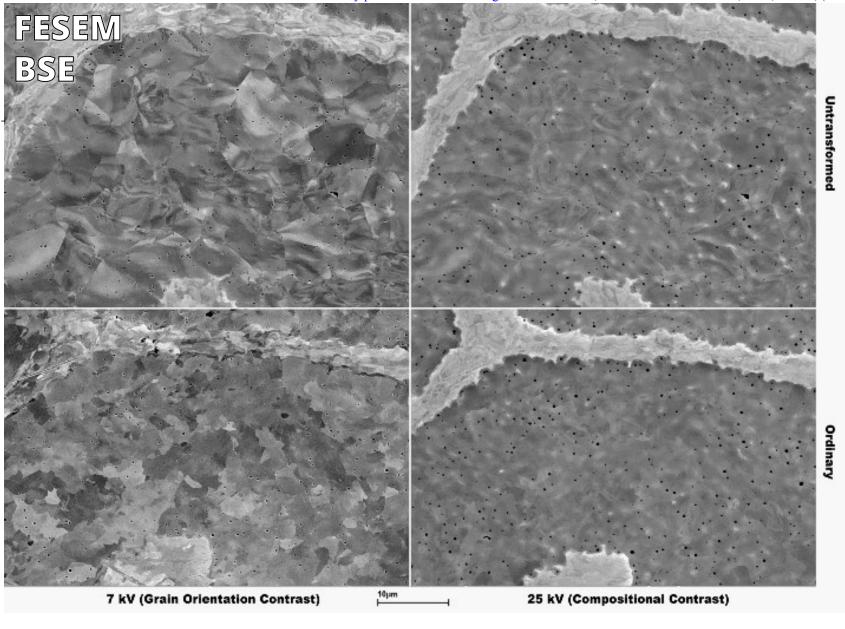




### FESEM: High resolution *and* uniform image over very large areas – perfect for IA



Extended low temperature HTs produce increased a-Ti (black) volume while retaining fine scale producing world record  $\frac{1}{2} > 4000 \text{ A/mm}^2$  at 5 T, 4.2 K (Kharkov CRDF collaboration/Oleg Chernyi)



# Can control BSE contrast mechanism with kV

Residual chemical inhomogeneity and large grains sizes in Nb<sub>3</sub>Al

At 25 kV light areas=high Nb

Nb<sub>3</sub>Al strand courtesy Takao Takeuchi, NIMS

Lee *et al.*, IEEE Transactions on Applied Superconductivity, vol. 13, no. 2, pp. 3398–3401, Jun. 2003.

IEEE CSC & ESAS SUPERCONDUCTIVITY NEWS FORUM (global edition), February 2019.

#### Imaging at all scales possible

By using both scanning laser confocal microscopy and FESEM we have access to the full range of scale critical for Nb<sub>3</sub>Sn cable







Sultan-tested cable courtesy of courtesy of Pierluigi Bruzzone (Plasma Physics Research Center) with agreement from the Japan Atomic Energy Agency (JAEA) and US ITER.

C. Sanabria, P. J. Lee, W. Starch, A. Devred, and D. C. Larbalestier, Superconductor Science and Technology, vol. 28, no. 12, p. 125003, Oct. 2015. http://dx.doi.org/10.1088/0953-

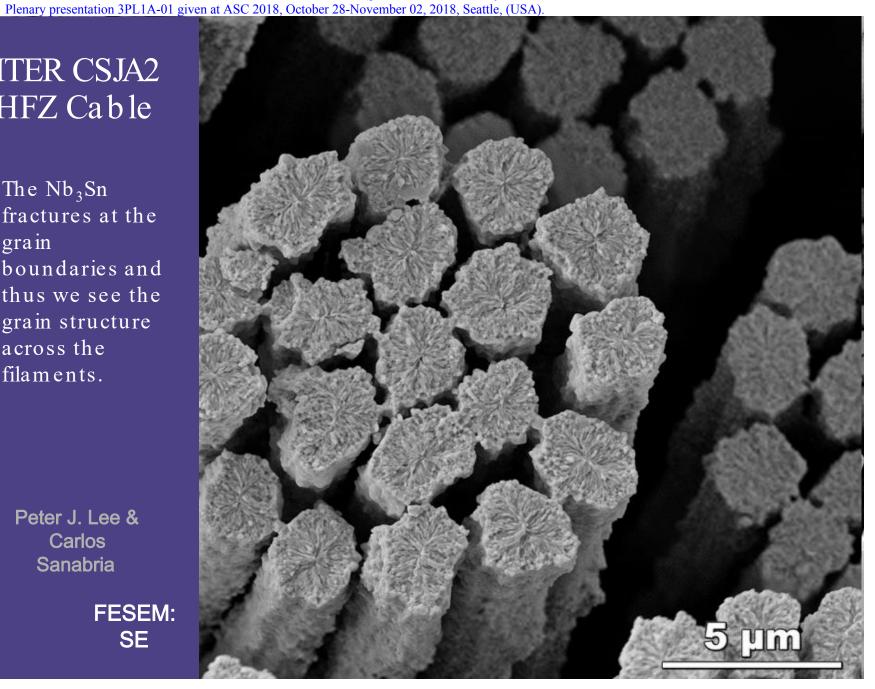
2048/28/12/125003

#### ITER CSJA2 HFZ Cable

The Nb<sub>3</sub>Sn fractures at the grain boundaries and thus we see the grain structure across the filaments.

Peter J. Lee & Carlos Sanabria

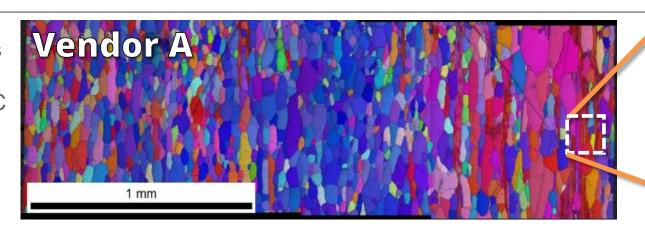
> FESEM: SE

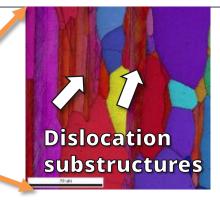


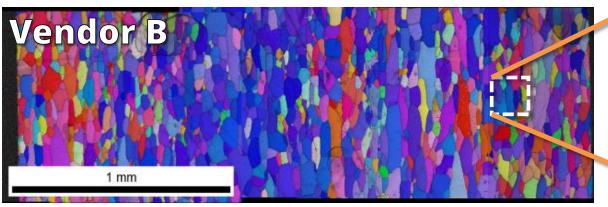
### EBSD in FESEM: Deformation structures present in as sheets before any cavity level deformation

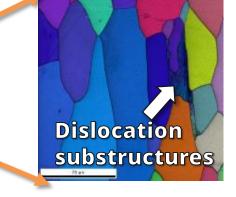
#### -received LCLS -II SRF Nb

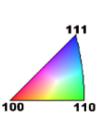
Shreyas Balachandran: 1LOr1A-06- ASC 2018, Seattle, Oct 28- Nov 2











High resolution EBSD. Inverse pole figure (IPF), showing crystallographic orientations in Nb sheet material

Fine scans reveal micro - misorientations typically due to dislocation substructures





### Electron channeling contrast imaging (ECCI) of SRF Nb

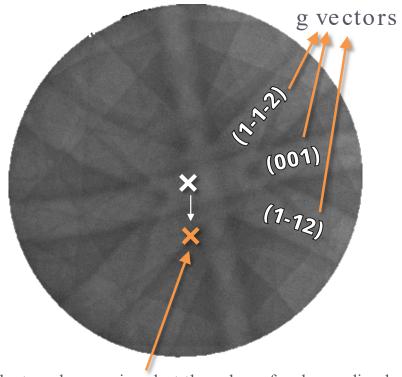
Dislocations observation in bulk samples in large areas, with non-destructive sample preparation. For SRF Nb it is really important to be able to image samples that have received exactly the same processing history as SRF cavities.

- ECCI im a ging:
  - Sample tilted and rotated to reach channeling condition (similar to TEM 'two-beam' condition)
  - Dislocations appear as channeling contrast when sample is at channeling condition
- Identify dislocation (Burgers vector, slip plane) using g
   b analysis

For more details about ECCI please refer to these publications: <sup>1</sup>M. A. Crimp, Microscopy Research and Technique **69** (5) (2006). <sup>2</sup>S. Zaefferer and N.-N. Elhami, Acta Materialia **75**, 20 (2014).

MICHIGAN STATE Mingmin Wang
UNIVERSITY MS&T 2018

Slide courtesy of Mingmin Wang (MSU) not to be redistributed without request Selected area channeling patterns (SACP)



Electron beam aimed at the edge of a channeling band of g=(1-1-2) to reach a specific imaging condition



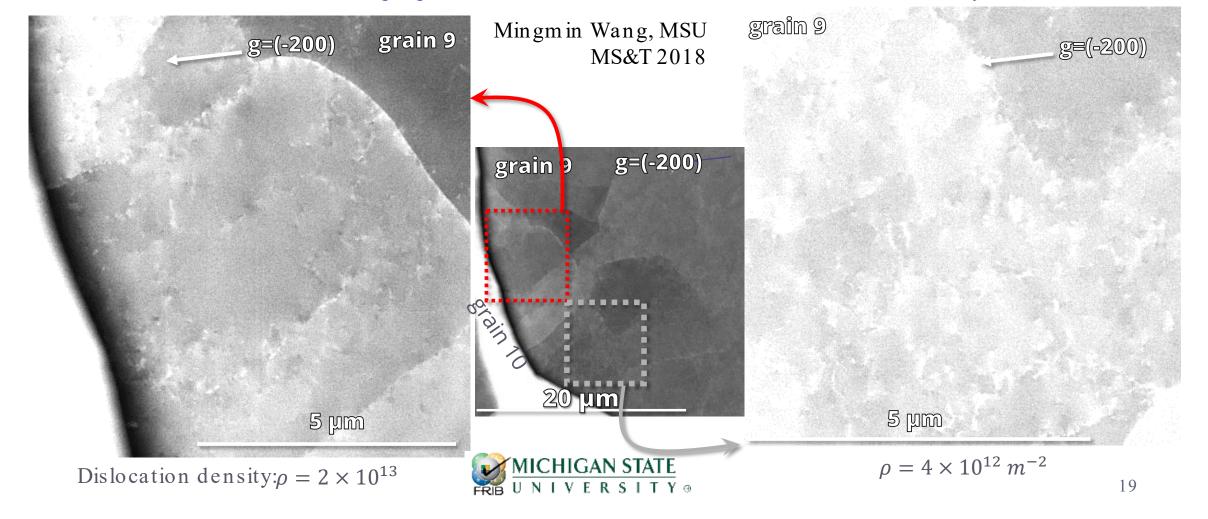


### Dislocation density can be estimated by manually counting number of dislocations per area from an ECCI images ( $\rho \sim 10^{12}$ to $10^{13}~m^{-2}$ )

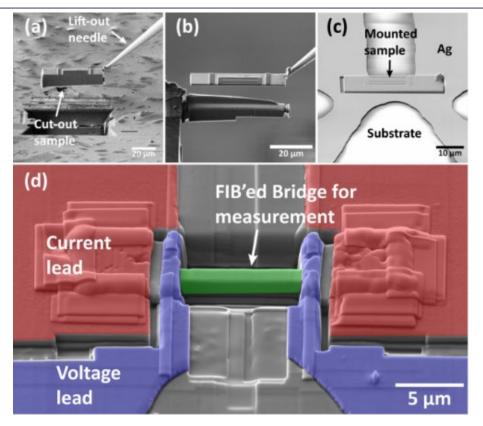
This is a lower bound of dislocation density estimation since:

- Not all dislocations are visible in a single ECCI image due to invisibility criteria
- Dislocation lines that are not perpendicular lead to underestimate of dislocation density

Slide courtesy of Mingmin Wang (MSU) not to be redistributed without request



### Micro-circuit fabrication by using Focused Ion Beam

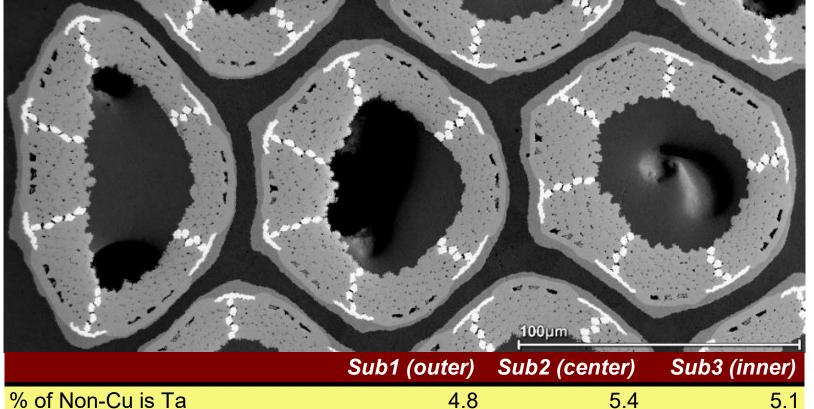


- Single crystal or bicrystal lamellae can be made from any form of tiny single crystal or polycrystal, even those possessing stoichiometric non-uniformity.
- Narrow FIB'ed micro-bridges drastically improve S/N
- Accurate current injection along particular crystallographic axes enables study of anisotropic behavior in any inorganic materials

- a) Cutting and lift-out.
- b) Sculpting to desired shape.
- c) Mounting on a Ag-patterned substrate.
- d) Sculpture and deposition of metal contacts by GIS. Here the final bridge width is  $\sim 1 \mu m$ .

Courtesy Fumitake Kametani

### Reducing d<sub>eff</sub> in Nb<sub>3</sub>Sn for FCC: Example using Ta Filaments to subdivide each sub-element



52.7

- Probably more dividers than would be necessary:
  - 6 dividers = ~10% loss in Nb<sub>3</sub>Sn area

OST Fabricated Strand courtesy Seung Hong and Jeff Parrell

9.9

51.6

10.6

51.3

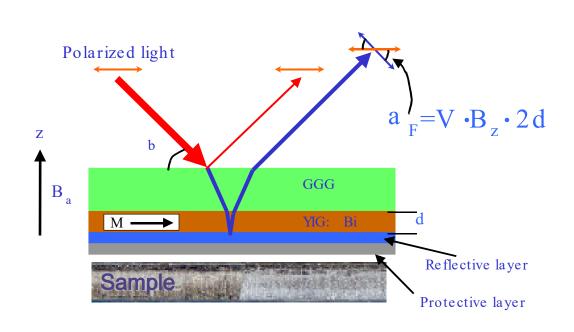
Sn core size was deliberately reduced in this composite

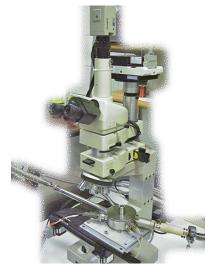
% of Nb<sub>3</sub>Sn Area lost to Dividers

% of Non-Cu is Nb<sub>3</sub>Sn

# Magneto Optical Imaging: Allows Direct Imaging of B<sub>z</sub> in Plane Above Sample

Double Faraday effect occurs in reflective mode using Bi-doped YIG indicator film with in-plane magnetization





Conventional polarizing light microscope using long working distance lenses

Cryostat

Courtesy Anatolii Polyanskii



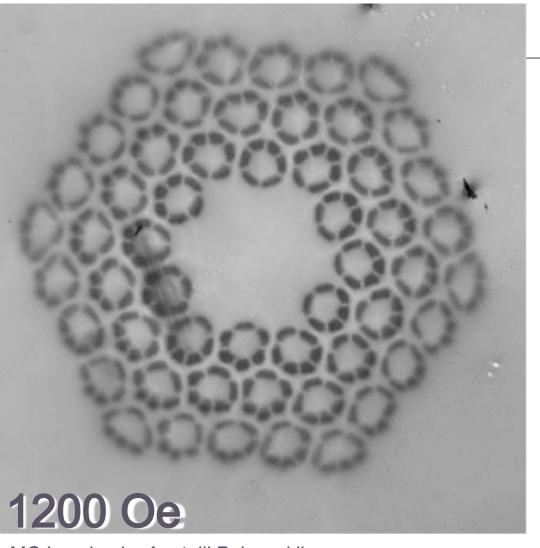


# MO imaging demonstrates successful subdivision

All subelements divided and cores penetrated

Zero Field Cooled, T=6 K

### **Increasing Field**



MO imaging by Anatolii Polyanskii



### The computing revolution

• Needless to say the availability of increasingly powerful and more affordably computing has greatly aided the process of quantifying images.



ASC'18 Chair: Matt Jewell ~2002



Paul Allen (28) and Bill Gates (26) in Microsoft publicity photo from 1981

#### NIH has supported the development of free and powerful IA software

- Image J/Fiji is free, open source and widely supported by a very large user community!
- NIH has supported the development of a multi-platform (Linux, Mac OS X and Windows, in both 32-bit and 64-bit modes) IA tool 'Image J' for many years to assist research in life sciences but the same tools can be used for materials science.
- A standardized open source package is now available:

"Fiji" http://fiji.sc/wiki/index.php/Fiji

Extensive tutorials are available for Fiji:

http://fiji.sc/wiki/index.php/Category:Tutorials

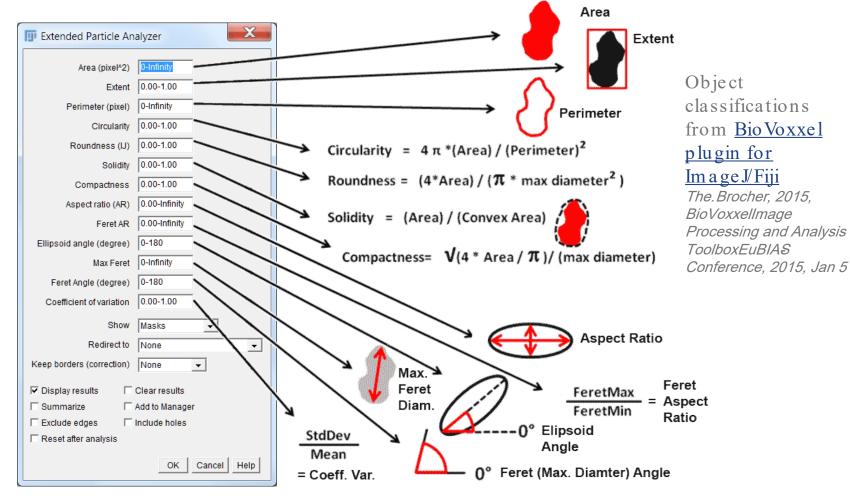


And the standard ImageJ: http://rsbweb.nih.gov/ij/docs/examples/index.html

Most importantly, the macro scripting language is designed to be easy to use and almost all actions can be recorded directly into an editable macro/script.

### Very extensive list of object measurements already available and easily extendable

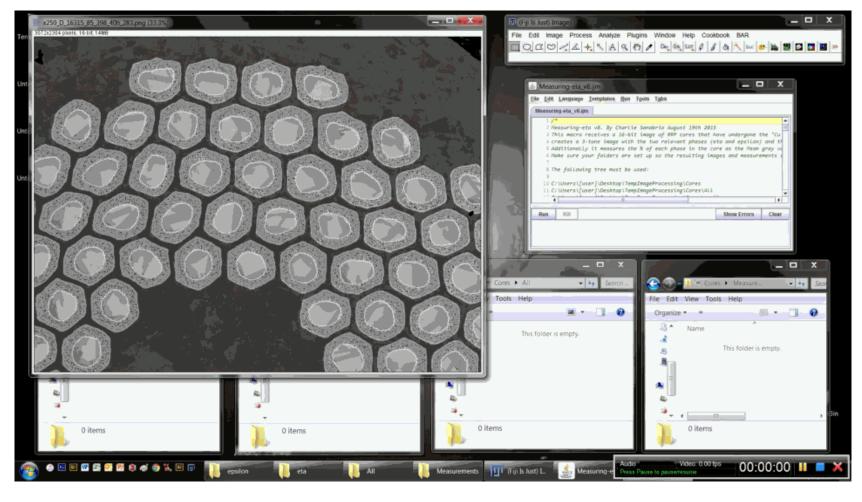
- The very large user community means that there are already a wide variety of feature characterizations already available.
- These measurements can be used filter and classify features.



### The Power of ImageJ/Fiji Macros

By using macros
you can apply a
uniform and
complex
processing and
analysis process
to multiple
images.

This macro by <u>Charlie</u> <u>Sanabria</u> analyses the ε to η ratio in RRP internal Sn Nb<sub>3</sub>Sn strand.



### ASC/NHMFL developed ImageJ macros available

http://fs.magnet.fsu.edu/~lee/asc/ImageJUtilities/ASC ImageJ Utils.html



The variation in thickness in the Nb diffusion barrier along its length is measured for outward diffusion along its length.

The darker the line the shorter the outward diffusion path (note, however the large grains - for grain boundary diffusion we would need a different analysis).



### Minimum (and Maximum) — Distance Measurements



- Many microstructural features relevant to superconducting properties change in size, distribution, morphology and chemistry with distance from an interface (e.g. relative to a diffusion source or a diffusion barrier).
- This macro adds the minimum distance between the centroid of a set of analyzed objects and a reference set of points to the ImageJresults table.
  - This data can then used to provide position normalized quantification of microstructures, microchemistry
  - And even images (Next Slide)
- Examples:
  - Minimum and Maximum distance to reference set.
     Minimum distance to center (based on mean of coordinates) of reference.
     Angular orientation of minimum distance direction (degrees)





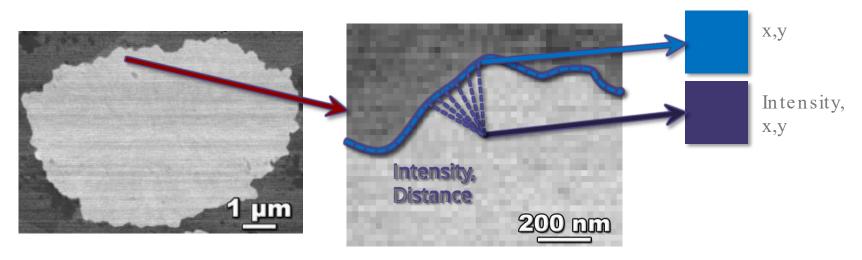
#### Positional Normalization to Interface/Feature

Here we have a high resolution atomic number (z) sensitive FESEM BSE image of a Nb<sub>3</sub>Sn filament.

The image is noisy, there is some dirt on the surface and there is some orientation contrast...

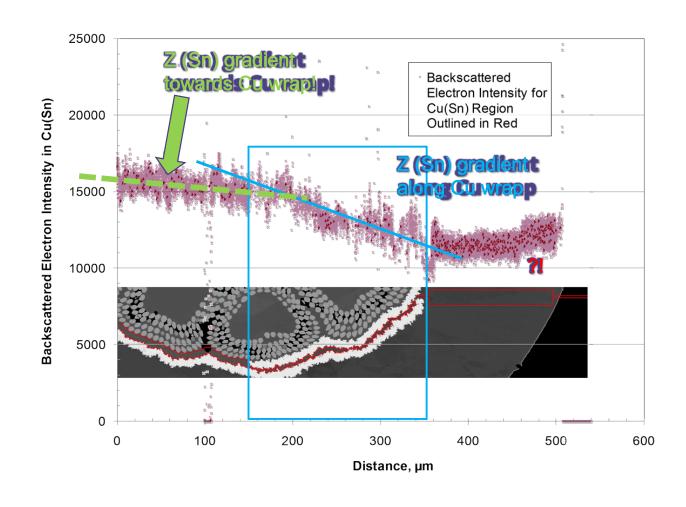
but it will do! *Mostly Intensity*  $\propto Z$ 

- 1. We know the x,y coordinates of each pixel in the filament.
- 2. In ImageJ we can generate an outline of the filament and output those x,y co-ordinates.
- 3. We can use the Image J/Fiji macro to calculate the distance of each pixel (z value) from the interface.



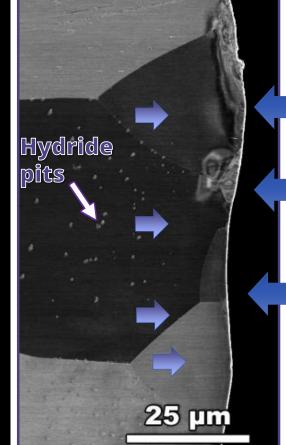
### Sn content gradient in diffusion barrier wrap

- Cu layer is added between barrier wraps to enhance bonding and to allow flux penetration and reduce hysteretic loss.
- Z-contrast analysis shows that Sn diffuses along the entire length as shown by Z-contrast gradient
- Gradient inside barrier suggests a Sn depletion gradient towards wrap.



### SRF Nb: Impact of N doping on hydride precipitation





See: P. Garg et al., "Revealing the role of nitrogen on hydride nucleation and stability in pure niobium using first-principles calculations," *Supercond. Sci. Technol.* vol. 31, no. 11, p. 115007, Oct. 2018.

N diffusion into Nb

From Shreyas Balachandran



#### Lower hydride density from the surface boundary in N doped Nb.

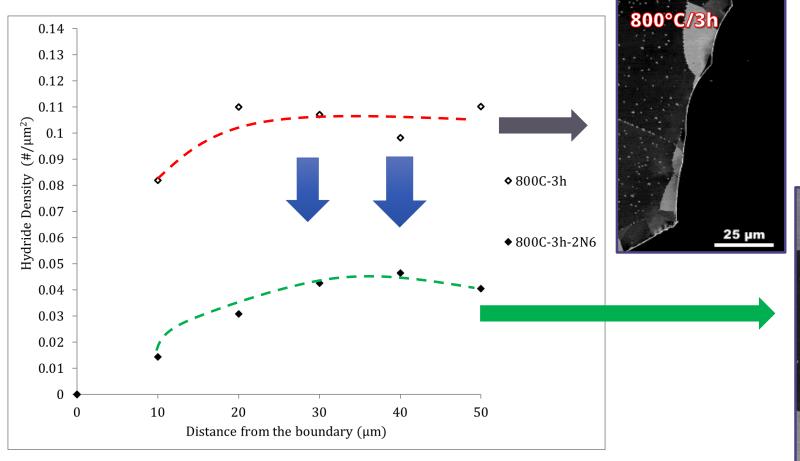


Image J/Fiji macro to calculate the distance between hydride pit and boundary



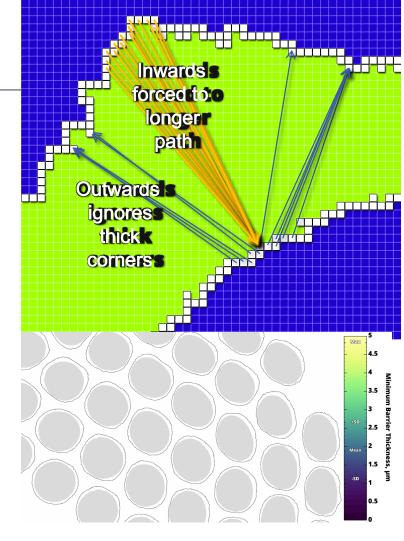
See: P. Garg et al., "Revealing the role of nitrogen on hydride nucleation and stability in pure niobium using first-principles calculations," <u>Supercond.</u> Sci. Technol. vol. 31, no. 11, p. 115007, Oct. 2018.

From Shreyas Balachandran



### Layer Thickness

- These macros measure the minimum wall thickness of open or closed objects from the perspective of each pixel on the interior or exterior of a wall.
  - Choosing the direction here is important
- If the wall is broken (incomplete) there is a version that uses an interior object to define the open and closed regions (this also allows a measurement of the percent of wall that is open).
- A detailed explanation of the two macros is provided at the web site.



Unreacted barrier thickness in Bruker PIT strand fabricated for CERN Image Analysis by Chris Segal (now at CERN).

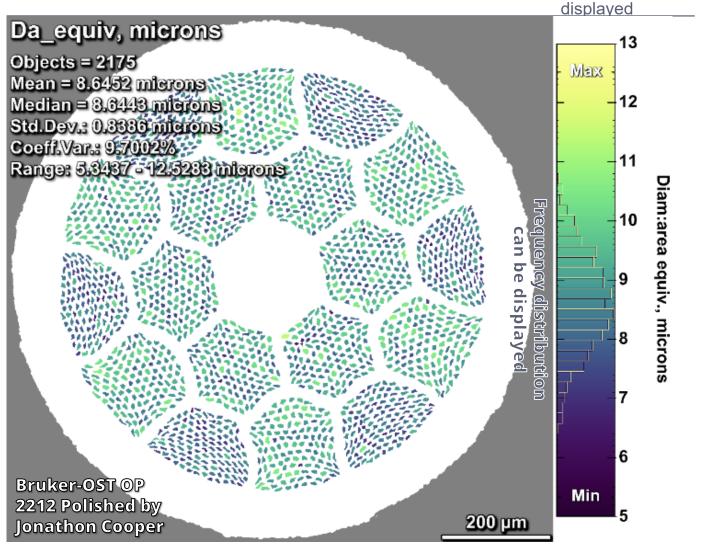






- The BAR ("Broadly Applicable Routines") collection maintained by Tiago Ferreira has numerous features but our favorite is the ROI (region of interest) color coder:
  - Colors each analyzed object with a color based on any set of feature values.
- We have extended the formatting options of this macro to provide more flexibility; the number of options might look intimidating but the defaults should be reasonable choices and scale with image size.

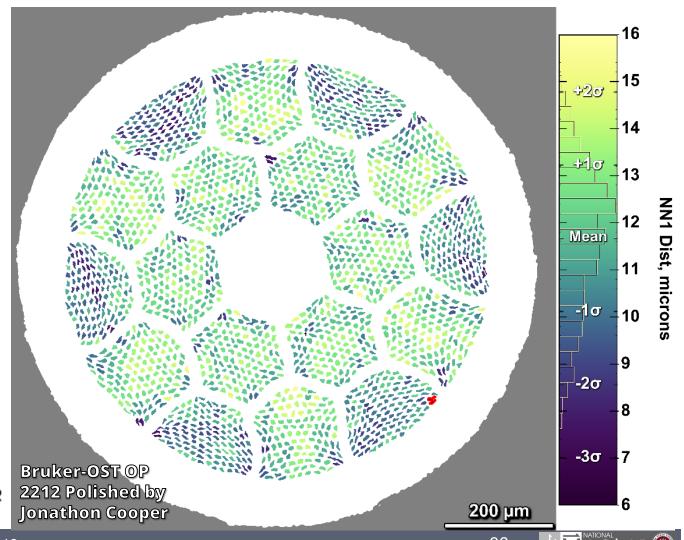
### Color By Feature



Linear or log stats can be

### **Touch Count and Proximity**

- The minimum separation of objects can be measured
  - The number of objects touching each other can be counted
  - The number of adjacent objects within a set distance can be counted
- The nearest neighbor distance can be calculated

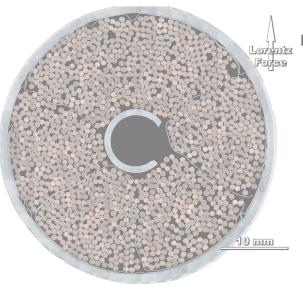




Iter EUTF5 CICC after testing in Sultan

### ITER CSIO1: SC vs SC+Cu

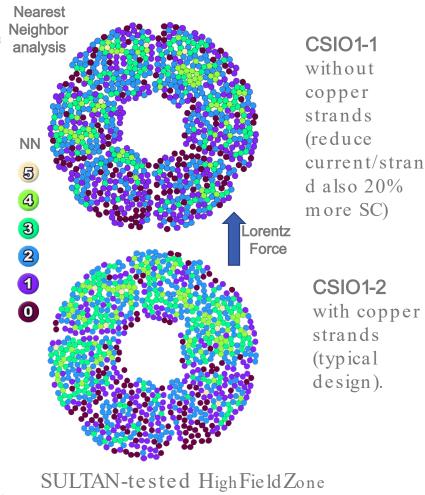
Sanabria et al. SuST 2015

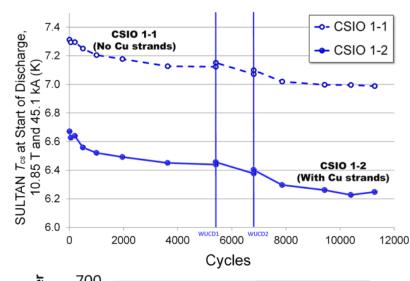


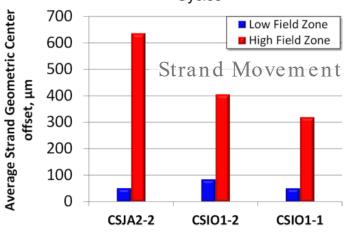
After cyclic testing in the SULTAN facility we analyze this cross section to show strand movement caused by the immense Lorentz force.

Strands are shaded according to the adjacent strand count, which ranges from zero to 5 (most densely packed ≤ 2.5 µm separation).

Sultan-tested cable courtesy of courtesy of Pierluigi Bruzzone (Plasma Physics Research Center) with agreement from US ITER.









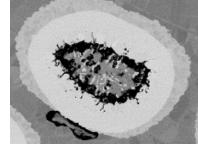


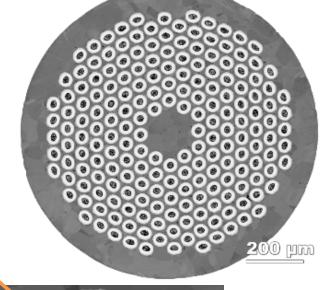
### Powder In Tube processed Nb 3Sn superconducting wires

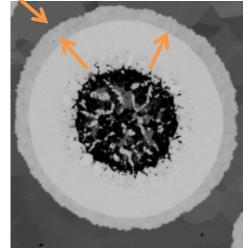
- For FCC application, PIT strands offer the potential of both high- $\mathcal{L}$  and small effective filament diameter
- Composite wires are 156 or 192 superconducting filaments stacked into a Cu stabilizer matrix.
  - Ideally filaments are perfectly circular with the Sn-rich core centered in the Nb-Ta reaction tube
    - The superconducting A15 phase is formed when the Sn diffuses radially outward into the Nb-Ta tube which houses it.
- Unfortunately, the filaments undergo nonuniform deformation during wire manufacture which leads to:
  - Ellipticity
  - Sn core drifting from center of Nb-Ta tube.
  - Reduced A15 volume
  - Reduced RRR











reacted

From Chris Segal



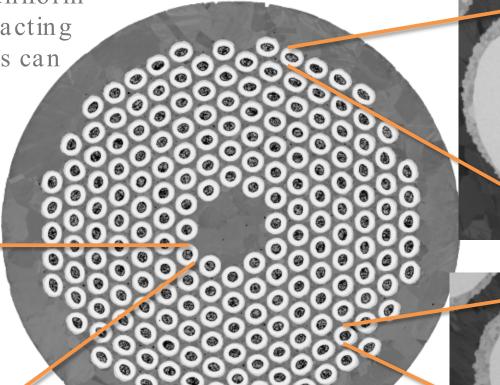
### What's keeping PIT...in the pit

Shape analysis shows non-uniform deformation negatively impacting electrical stability as Sn leaks can

• Reduce RRR

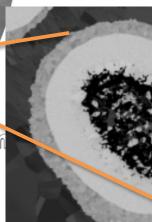
Reduce A15 volume

• lower 4



Sn increases resistivity of Cu





Thin barriers

Bad for RRR







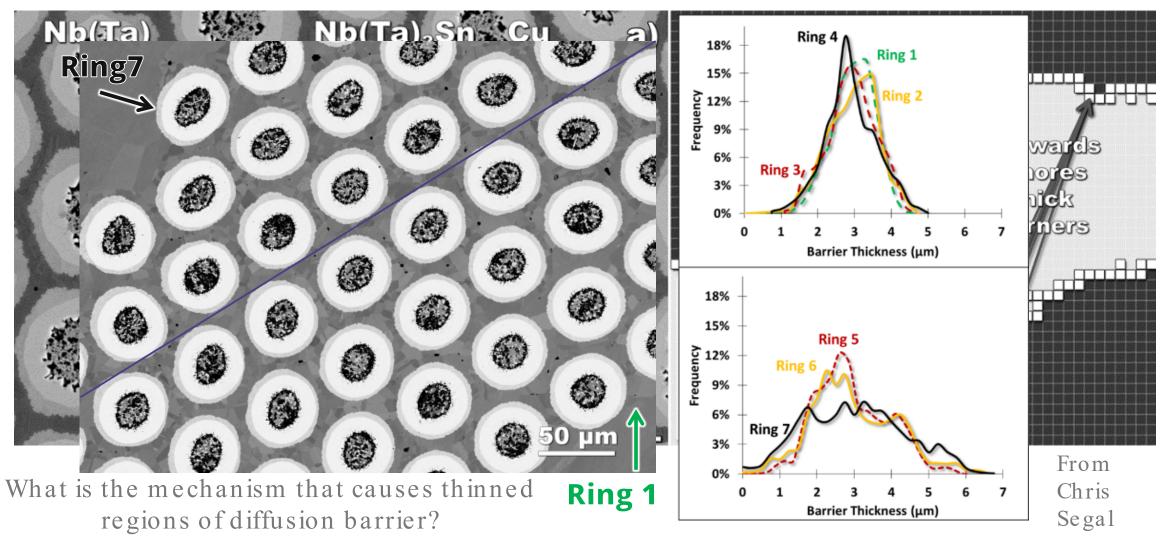
Good for RRR but bad for

From Chris Segal



#### Filaments show a wide distribution of barrier thickness

-Bad for RRR, and bad for A15 formation



### Centroid drift between Nb -Ta tube and powder core can cause leaks

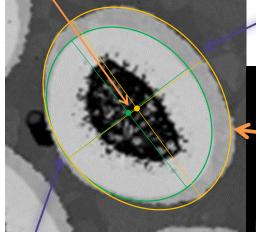
1.5 μm (4.2% of diameter)

#### Two main issues

- Filaments become more elliptical as the wire is processed to final size, outer filaments much more than inner [1][2][3].
- The powder core deforms at a different rate than the Nb-Ta tube housing it, causing the centers to drift from one another.
  - This creates an uneven reaction front.

#### How do we measure it

- The centroid drift is the distance between the centroid of the entire filament (orange) and the centroid of the A15 layer (green)
- To most accurately compare between filaments and wires, we normalize each filament's core offset to its own effective diameter, derived from its area.

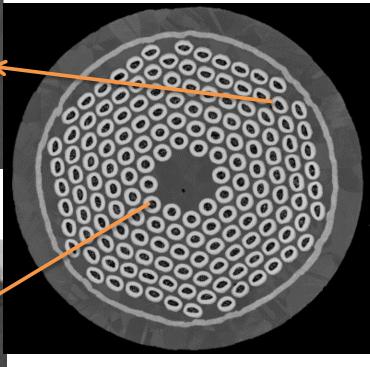


Thin side

72 nm (0.2% diameter)

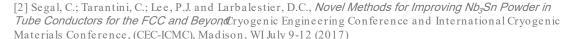


Thick side



0.7 mm wire diameter, 36 μm filament diameter

From Chris Segal



November 4-6 (2013)

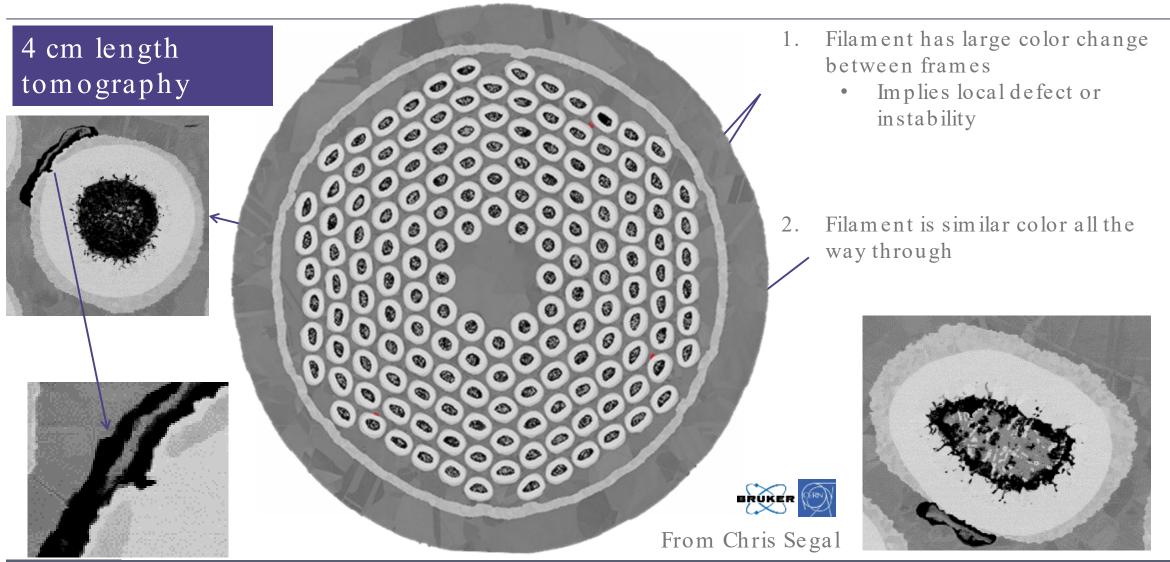
[3] C. Segal et al., "Evaluation of critical current density and residual resistance ratio limits in powder in tube Nb<sub>3</sub>Sn conductors," Supercond. Sci. Technol.vol. 29, no. 8, p. 85003, 2016.

and its Effect on RRR ow Temperature Superconductivity Workshop (LTSW), St. Petersburg, FL,





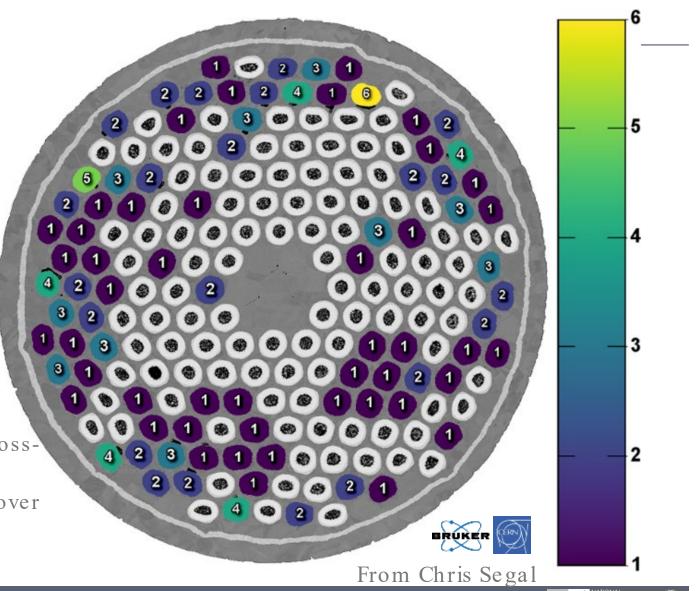
### How does centroid drift in a filament change along length?



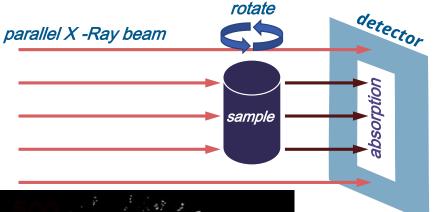
#### Serial section tomography: Leak distribution of each filament over 4 cm

Filaments with Kirkendall voids are plentiful along the entire 4 cm of wire, marked in black and integrated over 8 images

- Higher frequency in the external rings
- About half of the filaments leak enough Sn to produce a Kirkendall void in the 4 cm length.
  - In some cases the Kirkendall void may be centimeters long
- Bernd 0.7 mm wire, very high Sn
- 15-20 Kirkendall void leaks in any given crosssection
- 81 leaked filaments with Kirkendall voids over 4 cm
- (8 cross sections over 4 cm)



### X-ray microtomography: ESRF Grenoble





- 360° rotation of the sample
- 30000 projections
- 2560 x 2160 pixels
- 0.57 µm/pixel resolution





Quantitative correlation between the void morphology of niobium-tin wires and their irreversible critical current degradation upon mechanical loading

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Slide courtesy of Carmine Senatore (U-Geneva) not to be redistributed without request





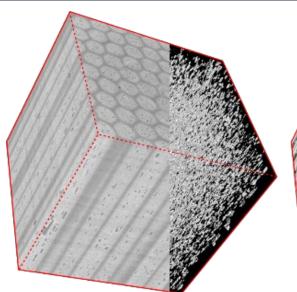
Nondestructive 3D volume reconstruction with separation of internal features



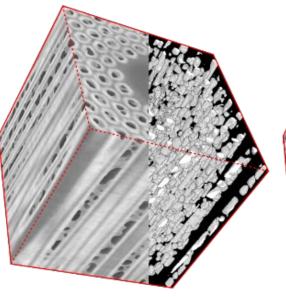


# Reconstruction of the voids inside Nb X-ray microtomography

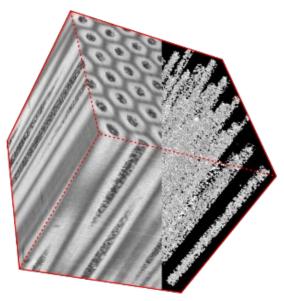
### <sub>3</sub>Sn sub-elements by ASC'18 2MOr0102







Internal Sn 132/169 sub-elements



PIT
192 filaments

Statistical analysis of void size, shape and orientation

> Slide courtesy of Carmine Senatore (U-Geneva) not to be redistributed without request

Scaling behavior of the critical current under transverse stress in RRP and PIT



Carmine SENATOR EChristian BARTH, Luc GAMPERLE

Department of Quantum Matter Physics, University of Geneva, Switzerland



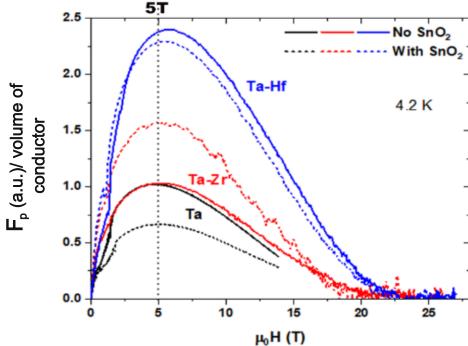
Jose FERRADAS, Bernardo BORDINI, Davide TOMMASINI
CERN, Switzerland



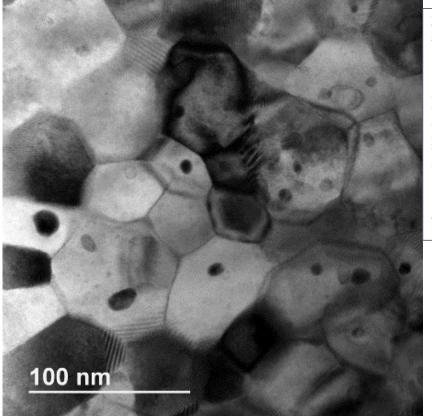


# TEM has also advanced with field emission sources and aberration correction . . . No more dark rooms/wet developers!

1MOr2B-06: Shreyas Balachandran et al.: Ta-Hf doped Nb<sub>3</sub>Sn for very high  $\mathcal{L}$ 







http://arxiv.org/abs/1811.08867
S. Balachandran et al., "Bene ficial influence of Hf and Zr additions to Nb4at.%Ta on the vortex pinning of Nb<sub>3</sub>Sn with and without an O source," arXiv:1811.08867 [cond-mat], Nov. 2018.





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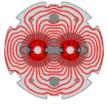
• Preliminary TEM-EELS microanalysis suggests that these precipitates are Tarich, and Sn deficient.

### We have come a very long way

- But we still hope to see more surprises like Ta-Hf doped Nb<sub>3</sub>Sn
- And perhaps even explain them using more advances in microscopy techniques.





















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