20 Years of Quantitative Microscopy:
Highlights from digital analysis of superconducting strand and cable

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# 1996 vs. 2016

<table>
<thead>
<tr>
<th>1996</th>
<th>2016</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>US Headlines</strong></td>
<td><strong>US Headlines</strong></td>
</tr>
<tr>
<td>US eases trade sanctions with Cuba</td>
<td>US eases trade sanctions with Cuba</td>
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<tr>
<td>Clinton runs for Democratic party nomination</td>
<td>Clinton runs for Democratic party nomination</td>
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<tr>
<td>Major snowstorm paralyses Midwest</td>
<td>Major snowstorm paralises Northeast</td>
</tr>
<tr>
<td>Cost of Gallon of Gas: $1.22</td>
<td>$1.93</td>
</tr>
<tr>
<td>Microsoft Shares (March): $4.37</td>
<td>$53.07</td>
</tr>
<tr>
<td>Apple Shares (March): $0.81</td>
<td>$102.26</td>
</tr>
<tr>
<td>1 year before multiplatform ImageJ introduced (NIH Image available for Mac since 1987)</td>
<td></td>
</tr>
</tbody>
</table>
Why Digital Image Analysis?

- Quantitative analysis of material microstructures provides us with direct information about the impact of processing e.g.:
  - Heat treatment
  - Deformation
  - Chemistry

And the more accurate the image analysis the more subtle the changes that can be observed.

The software and hardware has been getting more powerful and more easily accessible.
Non-digital analysis

- Analysis by comparison with standard microstructures is fast and works well in industrial application.

  Standard light microscope charts for grain size measurement according to ASTM and DIN. The image of the polished section appears on the ground glass screen in the central section of the plate.

*W. Lang, “Instrumentation of Quantitative Microstructural Analysis.” Practical Metallography, 9 (1972) 208*
Non-digital errors

- Optical illusions
  - Inner circle appears smaller because of environment

Digital Analysis: Circles shaded according to area (color + dark-small to light-large)

Pixel Areas: Center circle is actually bigger

Digital Analysis

- Provides us with much more information:
  - Inhomogeneity, anisotropy, position sensitivity
  - Feature analysis
    - Basic 2d: Area fractions, object sizes
    - Complex 2d descriptions: shape
    - Complex 3d descriptions: volume fractions, spatial distribution
  - Statistical distributions

<table>
<thead>
<tr>
<th></th>
<th>Area pixel²</th>
<th>Filled Area pixel²</th>
<th>Equiv. Diam. pixel</th>
<th>Length pixel</th>
<th>Breadth pixel</th>
<th>Aspect Ratio</th>
<th>Nearest Nbor Dist pixel</th>
<th>Min. Separation Dist. pixel</th>
<th>Mean Fiber Width pixel</th>
<th>Std. Dev., Width pixel</th>
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<tbody>
<tr>
<td><strong>Mean</strong></td>
<td>3320.5</td>
<td>3320.5</td>
<td>59.59</td>
<td>60.7</td>
<td>59.5</td>
<td>1.02</td>
<td>92.1</td>
<td>26.85</td>
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<td><strong>Min</strong></td>
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<td>18.07</td>
<td>28.52</td>
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<td><strong>Max</strong></td>
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<td>6138.0</td>
<td>88.4</td>
<td>89.5</td>
<td>88.7</td>
<td>1.04</td>
<td>108.7</td>
<td>34.30</td>
<td>87.50</td>
<td>1.10</td>
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<tr>
<td><strong>Std Dev</strong></td>
<td>2560.1</td>
<td>2560.1</td>
<td>27.00</td>
<td>27.2</td>
<td>26.9</td>
<td>0.01</td>
<td>16.0</td>
<td>6.87</td>
<td>27.20</td>
<td>0.35</td>
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<tr>
<td><strong>Coeff. Var. %</strong></td>
<td>77.10</td>
<td>77.10</td>
<td>45.32</td>
<td>44.76</td>
<td>45.23</td>
<td>0.87</td>
<td>17.4</td>
<td>25.57</td>
<td>47.28</td>
<td>81.65</td>
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</table>
Anisotropy

- One orientation of cross-sectional analysis is typically not sufficient to characterize a material.

- All superconducting wires and tapes are anisotropic to some extent.

Nb-47Ti – Anisotropy through strain

- Nb-47Ti: Linear orientation anisotropy developed by wire drawing and extrusion cycles.

Cube assembled from one TCS TEM and one LCS TEM image using "MegaVision XM1024" 1990
MegaVision 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.

MegaVision advertising ~1984
We can also combine TEM images at different tilts to remove diffraction contrast and leave Z-contrast

\[ \text{Nb-47Ti} \quad \varepsilon_f = 3 \]

MegaVision 1024XM processing 1994

Bright Field (SAD) TEM Images taken at different tilt angles (changing diffraction contrast)TE

TEM images aligned and then combined by “max intensity” – All now possible in ImageJ/Fiji for free!
Crude but effective . . .

- The microstructure/property relationships for Nb-Ti were quantified with these relatively primitive (and expensive) digital tools.

- Now we can use FESEM-BSE images at most relevant magnifications.
Advanced IA techniques now readily available . . . and free!

- 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 “ImageJ” 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:


  Extensive tutorials are available for Fiji:


Much more extensive list of object measurements typically available

- **Object classifications from BioVoxxel plugin for ImageJ/Fiji**

Mathematical formulas:

- **Circularity** = \( 4 \pi \frac{\text{Area}}{\text{(Perimeter)}^2} \)
- **Roundness** = \( \frac{4 \times \text{Area}}{\pi \times \text{max diameter}^2} \)
- **Solidity** = \( \frac{\text{Area}}{\text{Convex Area}} \)
- **Compactness** = \( \sqrt{\frac{4 \times \text{Area}}{\pi}} \div \text{max diameter} \)

- **FeretMax** and **FeretMin**

- **StdDev** and **Mean**
Next most important tool: The Spreadsheet

- Once the data is digital we can perform full statistical analysis
- Spreadsheet macros allow us to perform additional functions
  - Most important for us is the analysis of location:
    - Composition variation with location
      - Interpreted from backscattered electron images
    - Morphological variations:
      - e.g. Aspect ratio variation with distance from Sn core in Nb₃Sn
    - Barrier thickness variations
Positional Normalization to Interface

Here we have a high resolution atomic number (z) sensitive FESEM BSE image of a Nb$_3$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 coordinates.
3. We use a simple Microsoft Excel macro to calculate the distance of each pixel (z value) from the interface.
Now have sub-μm composition information

Plateau 1  Plateau 2

Very sharp < 50 nm interface
Compare interface with: Free Simulation Software

Casino

Simulates a large numbers of electron trajectories in a solid of your choice. The main idea is to simulate enough electron trajectory to represent the condition used to image structures in a scanning electron microscope (SEM).

And "Win-Ray" or "MAC-Ray"

Obtains:
- Backscattered electron distributions
- Absorbed electron distributions
- Energy loss distributions
- Characteristic X-rays
- Bremsstrahlung X-rays
- X-ray spectrum

Raynald Gauvin’s group at the Université de Sherbrooke, Québec, Canada

http://montecarlomodeling.mcgill.ca/
BSE Simulation for Nb₃Sn:Cu Interface

We have a good match with the measured interface.
Sn content gradient in diffusion barrier wrap

- Cu layer between barrier wraps provides excellent bonding and allows flux penetration.
- Z-contrast analysis shows that Sn diffuses along entire length as shown by Z-contrast gradient.
- Gradient inside barrier suggests depletion gradient towards wrap.
Intergranular Fracture of Nb₃Sn Yields Grain Boundary Contrast in SEM

“Fortunate” the Nb₃Sn fractures so easily at grain boundaries!
We can also interface-normalize microstructural features

- Size
- Morphology
- Location
- Angular Orientation

Each filament contains ~ 1000 grains

Features:

MJR Internal Sn Nb$_3$Sn
Large Magnification Range: But beware on non-planar surface

Use stereo tilt.

3D Necessary to show that some areas are not really a planar cross-section

PIT-Nb₃Sn Superconducting Strand
High $J_c$ Nb$_3$Sn: Barrier and Individual filament – interface normalization

Billet ID: TWC 1912 MJR

Was Nb barrier

Was Sn core
Interface normalized image analysis (1999)

Apart from the columnar grains at the barrier there is a continuous but small morphological variation across the layer in grain boundary density.
IA Normalized to Edge of Individual Filament

Billet ID: TWC 1912

Much stronger variations across individual filaments
Chemical and Microstructural Changes (Equiaxed to Columnar) At the Same Scale (VAC bronze-process Nb₃Sn)

Detail from a BSE traverse: Level backscatter intensity up to 300 nm followed by a continuous drop until approximately 900 nm.

Aspect ratio and GB density also show change in behavior at 300 nm.
Nb₃Sn Filaments Point - Tail Comparison: Reveals that the billet was improperly cropped (1996)

Representative images – always an issue: This is the same strand just at different ends.

How homogenous is the sample? How many samples do you need?

Distribution of mean minimum filament spacing, in microns (radial axis) for the 12 outside sub-elements of TWC 1721 point and tail.

MJR internal Sn Nb₃Sn
Routine ITER Strand Analysis

- We provided an analysis of all production strand designs for the ITER TF coils.
- ITER needed to know how variable the strands were and what the variables are and the impact if any on cable performance.
Anatomy of an ITER Cable-in-Conduit

- We use IA to measure features at all scales.
- CICC Void %
- Strand Distribution
  - Movement
  - Spacing
- Cr thickness
- Barrier uniformity
- Filament and void size, spacing and cracks
- Grain size
The $\text{Nb}_3\text{Sn}$ fractures at the grain boundaries and thus we see the grain structure across the filaments.
High Resolution SLCM Images

Digital stage control and stitching software allow is to cover large areas at high resolution.

This 290 Megapixel image uses 475 SLCM frames.

Each frame is a pixel-by-pixel focused SLC image combined with a color image.
Iter EUTF5
CICC after testing

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).
ITER CSIO1: SC vs SC+Cu

CSIO1-1
without copper strands
(reduce current/strand
also 20% more SC)

CSIO1-2
with copper strands
(typical design).

SULTAN-tested High Field Zone

Nearest Neighbor analysis

Lorentz Force

Strand Movement

Sanabria et al. SuST 2015
ITER CSIO1-1: Extracted Strand

Locations of cracks are shown in blue.

(b) cold worked copper at a contact point with a neighboring strand or jacket
(c) cracks induced by contact stresses
(d) strain free copper grains
(e) cracks induced by tensile stresses (bending).

CSIO1-1 vs. 2: Lower number of e) cracks (associated with bending). But compressive-like filament fractures c)
Cu strands appear to absorb compressive loads.
Filament Geometry and Instability

<table>
<thead>
<tr>
<th></th>
<th>Area (µm²)</th>
<th>Diameter (µm)</th>
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</thead>
<tbody>
<tr>
<td>Mean</td>
<td>25.3</td>
<td>5.7</td>
</tr>
<tr>
<td>Min</td>
<td>0.1</td>
<td>0.3</td>
</tr>
<tr>
<td>Max</td>
<td>46.7</td>
<td>7.7</td>
</tr>
<tr>
<td>Std Dev</td>
<td>2.9</td>
<td>0.4</td>
</tr>
<tr>
<td>Coeff. Var. %</td>
<td>11</td>
<td>7</td>
</tr>
<tr>
<td>Count</td>
<td>3125</td>
<td>3125</td>
</tr>
</tbody>
</table>

ITER internal Sn Nb₃Sn

ASC’12 5MB-06 35

Images of the same unreacted sub-element of an internal Sn strand imaged from multiple cross-sections over a ~ 5 – 15 mm length
Void: Nb$_3$Sn Interface Density

It might seem obvious that voids in contact with filaments may weaken the composite.

The distribution varies significantly from composite to composite.

". . . despite the fact that porosity in the WST wire was twice as high as in the Luvata wires, $\varepsilon_{\text{irr}}$, of the WST strand was larger, thus indicating no obvious connection between 2D porosity and $\varepsilon_{\text{irr}}$, 0 in the wires studied." Cheggour et al. SuST 2014

Strands electromechanically tested at NIST-Boulder by Najib Cheggour.
Detailed ITER fatigue crack study (2012): Hitachi TF 1.14% strain (center) 1000 cycles

- 56 examples from Hitachi 1000 cycles@1.14% center section

- But this section has >300 cracks

- A crack is more likely to be found where the filament is locally necked.

Danny Escobedo and Manan Sheth, Dustin McRae and Bob Walsh

ASC’12 5MB-06 37
These IA techniques have been demonstrated extensively in Michael Brown's talk earlier this meeting.
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 Charlie Sanabria analyses the $\epsilon$ to $\eta$ ratio in RRP internal Sn Nb$_3$Sn strand.
Thanks

- Powerful image analysis software is widely available (see links on the next slide) and perform an immense range of functions; try them out and have fun!
Image Analysis Links

**General Image Analysis and Processing:**

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 "ImageJ" 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:

- Extensive tutorials are available for Fiji:
- The ImageJ site is here:
- With the full (and very long) list of plugins here:

Commercial Image Analysis within Photoshop (by John and Chris Russ):

- [http://reindeergraphics.com/science.html](http://reindeergraphics.com/science.html)
- Some of the basic image processing components as well as tutorials and Photoshop actions are available for free from John Russ:
  - [http://drjohnruss.com/download.html](http://drjohnruss.com/download.html)
- Extensive tutorials here:
  - [http://reindeergraphics.com/foveaprotutorial.html](http://reindeergraphics.com/foveaprotutorial.html)
- The current version of Fovea Pro can be run (we do) on CSS and Windows 7 but not without difficulty.

**Electron Trajectory Simulation Software:**


**3D Reconstruction**

- There is a new variant "MC Xray" that we have not experimented with here: [http://www.memrg.com/#!programs-download/c143t](http://www.memrg.com/#!programs-download/c143t)

- Our example a 2005 vintage ImageJ plug in by Albert Cordona:
  - [http://www.mcdb.ucla.edu/Research/Hartenstein/software/imagej/](http://www.mcdb.ucla.edu/Research/Hartenstein/software/imagej/)
- This plugin has been superseded by "TrakEM2" included in the Fiji distribution
  - [http://www.ini.uzh.ch/~acardona/trakem2.html](http://www.ini.uzh.ch/~acardona/trakem2.html)
- Standalone GNU/Linux: Reconstruct by John C. Flala
  - [http://synapses.cim.utas.edu/tools/reconstruct/reconstruct.stm](http://synapses.cim.utas.edu/tools/reconstruct/reconstruct.stm)

**Extended Depth of Field/Height Mapping:**

- Fiji or download ImageJ plug in separately:
  - [http://fiji.scwww.epfl.ch/demo/edf/](http://fiji.scwww.epfl.ch/demo/edf/)

**Image Alignment**:

- Photoshop layer alignment or Fiji, ImageJ plug in can be downloaded separately:

**3D Flicker Animation**

- Save tilted image pair as animated GIF directly from Fiji
- or
- Use freeware software package such as StereoPhoto Maker:
  - [http://stereo.jpn.org/eng/stphmkr/](http://stereo.jpn.org/eng/stphmkr/)
- Many more examples of our tilt-pair images at:
  - [http://magnet.fsu.edu/~lee/images/image.htm](http://magnet.fsu.edu/~lee/images/image.htm)

**Image Stitching**

- Free/Open Source:
  - Included in Fiji (2d and 3d!) See link above
- Also Microsoft Image Composite Editor:
- Commercial (there are many but we use this one PanoramaStudioPro):
- You can also create huge 3D-like image mosaics for web use using Image Composite Editor
Acknowledgements

- Iter work funded by ITER Organization with additional funding from the State of Florida and US DOE Office of Fusion Energy Science Grant DE-FG02-06ER54881 and US-ITER contract 6400011187.
- SULTAN-tested cable was provided by courtesy of Pierluigi Bruzzone (Plasma Physics Research Center) with agreement from Fusion for Energy.
- HEP supported by the US Department of Energy under multiple awards including DE-SC0012083.
- RRP® strand provided under the DOE Conductor Development Program that is managed by D. Dietderich of the Lawrence Berkeley Laboratory.
- Metallographic assistance from Bill Starch, Timothy Blum and Jen Gavin (FSU)
Some recent publications


- C. Sanabria, P. J. Lee, A. Devred, and D. C. Larbalestier, “Metallographic autopsies of full-scale ITER prototype Cable-in-Conduit Conductors after full cyclic testing in SULTAN: III. The Importance of Strand Surface Roughness in Long Twist Pitch Conductors,” submitted *Superconductor Science and Technology*, MEM’16 Special Issue.


