Characterization of coated conductors in the ultra-high field regime.

D. Abramov$^1$, F. F. Balakirev$^2$, and D.C. Larbalestier$^1$

$^1$ASC, NHMFL, Tallahassee
$^2$NHMFL Pulsed Field Facility, Los Alamos National Laboratory

SuperPower Inc. 7.5% and 15% Zr doped tapes produced for CORC application were used for $I_c$ measurements

Plan

1. Motivation
2. Preparation ultra narrow bridges
3. Setup for measuring for ultra fast I-V measurements in pulsed magnetic fields
4. Testing bridges: $I_c(T, SF)$ and $R(T, B)$
5. Measuring $V(I, B, T)$ curves in pulsed fields up to 65T
6. Calculation of $I_c$ from $V(I, B)$ dependencies
7. Summary
8. Future plans

This work was supported in part by the U.S. National Science Foundation under Grant No. DMR-1157490 and the State of Florida.
Mo*va*on

ReBCO superconductors become a choice material for developing all superconducting magnets capable generating ultra-high fields due to very high critical current densities in the presence of magnetic fields and high irreversibility fields.

Therefore, it is important to develop instrumentation suitable for testing $I_c$, $J_c$, $f_p$ of ReBCO conductors in such regime.

Recent progress in introducing high concentration artificial pinning centers (APC) in ReBCO conductors makes them optimized for low-temperature high-field applications.

The understanding performance of APC at ultra-high fields is fascinating vortex dynamics problem and important application task.

For now pulsed magnets is the only available choice for generating fields above 45T.
Tapes from different manufacturers have different slope $\alpha$

$I_{\downarrow c} \sim B_{\uparrow} - \alpha$

<table>
<thead>
<tr>
<th>Manufacturer</th>
<th>Material</th>
<th>$\alpha$ at BIIc</th>
<th>$\alpha$ at 18 °</th>
</tr>
</thead>
<tbody>
<tr>
<td>SuperPower</td>
<td>ReBCO</td>
<td>0.67-0.76</td>
<td>0.7-0.9</td>
</tr>
<tr>
<td>SuNAM</td>
<td>ReBCO</td>
<td>0.6; 0.6</td>
<td>0.69; 0.68</td>
</tr>
<tr>
<td>SuperOx</td>
<td>ReBCO</td>
<td>0.54; 0.54</td>
<td>0.588; 0.59</td>
</tr>
<tr>
<td>Fujikura</td>
<td>ReBCO</td>
<td>0.641; 0.69</td>
<td>0.70</td>
</tr>
<tr>
<td>AMSC</td>
<td>ReBCO</td>
<td>0.74</td>
<td></td>
</tr>
<tr>
<td>Bruker</td>
<td>ReBCO</td>
<td>0.88-0.89</td>
<td></td>
</tr>
</tbody>
</table>

Transport $I_c(B,4K)$ were measured by D. Abraimov, A. Fransis, M. Santos, and J. Jaroszynski
Comparison $J_c$ from different ReBCO samples (0.9\,μm – 2\,μm thick)

Transport $I_c(B,4.2K)$ were measured by D. Abraimov, Aixia Xu, J. Jaroszynski

Uni Houston data taken from:
<<Strongly enhanced vortex pinning in a broad temperature and magnetic field range of Zr-added MOCVD coated conductors>>
Aixia Xu, et al.
Can we trust extrapolations?

Transport $I_c(B,4.2K)$ were measured by D. Abraimov, Aixia Xu, J. Jaroszynski

Uni Houston data taken from:
<<Strongly enhanced vortex pinning in a broad temperature and magnetic field range of Zr-added MOCVD coated conductors>>
Aixia Xu, et al.
Different tape architecture for magnet and cable applications
In-field transport properties at 4K, BIIc orientation for **SuperPower Inc.** 4 mm wide tapes

<table>
<thead>
<tr>
<th>Project</th>
<th>SP machine</th>
<th>ReBCO thickness, μm</th>
<th>Zr doping, %</th>
<th>Substrate thickness, μm</th>
<th>Cu layer thickness, μm</th>
<th>$\alpha$</th>
<th>$&lt;I_c(15T, 4K)&gt;, \text{ A}$</th>
<th>$J_e(15T, 4K), \text{ kA/cm}^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>32T</td>
<td>M3</td>
<td>0.9-1</td>
<td>7.5</td>
<td>50</td>
<td>2x50</td>
<td>0.746 j</td>
<td>183 j</td>
<td>45.5 j</td>
</tr>
<tr>
<td>32T</td>
<td>M4</td>
<td>1.5-1.6</td>
<td>7.5</td>
<td>50</td>
<td>2x50</td>
<td>0.776*</td>
<td>283*</td>
<td>70.4*</td>
</tr>
<tr>
<td>CORC®</td>
<td>M3</td>
<td>≈1</td>
<td>7.5</td>
<td>38</td>
<td>2x5</td>
<td>0.754</td>
<td>241</td>
<td>108</td>
</tr>
<tr>
<td>CORC®</td>
<td>M4</td>
<td>≈1.5-1.6</td>
<td>7.5</td>
<td>30</td>
<td>2x5</td>
<td>0.77</td>
<td>302</td>
<td>164</td>
</tr>
<tr>
<td>CORC®</td>
<td>M4</td>
<td>≈1.5-1.6</td>
<td>15</td>
<td>50</td>
<td>2x5</td>
<td>0.864</td>
<td>320</td>
<td>117</td>
</tr>
</tbody>
</table>

\[ I_c \sim B^{-\alpha} \]

Slightly lower $\alpha$ values for M3 tapes suggest that those tapes may have smaller Zr doping, than M4 tapes. Tapes with nominal 15% Zr doping have larger $\alpha$, but not as high, as for tapes produced in Houston Uni. lab.

The first two rows are representative M3 and M4 tapes purchased for 32T magnet project. Other rows were tested for CORC® project.

* Transport $I_c(B, 4K)$ were measured by D. Abraimov, A. Francis, M. Santos, and J. Jaroszynski

* Average out of 15 representative tapes

$^j$ Average out of 12 representative tapes
What is shape of pinning force density at higher fields?

Transport $I_c(B,4K)$ were measured by D. Abraimov, Aixia Xu, J. Jaroszynski
1. Technical: find procedure of measuring $I_c(B,T,\theta)$ in pulsed field up to 65T for commercially available high $J_c$ ReBCO;

2. For magnet application:
   - Transport current property - explore $I_c$ limiting factors at critical magnetic field orientations – near $18^\circ$-$20^\circ$;
   - Transport current and mechanical property - find experimentally maximum in field shear stress for ReBCO layer in B_∥ tape orientation;

3. Physics/Material science and assistance to tape manufacturers:
   - calculate and compare vortex pinning force dependence $f_p(B, T)$ for two SuperPower Inc. tapes containing different Zr doping: 7.5% and 15%;
Since pulse durations are <0.1 sec., measurements of high $I_c$ are a tough technical task.

Major problems:

1. High induction voltages in the sample,
2. The very limited timeframe to
   - ramp current,
   - detect voltage,
   - compare it with defined threshold, and
   - decide to stop current ramp just above $I_c$;
3. Possible Joule heating in the sample due to induced eddy currents;
4. Forces on the sample and leads due to magnetic interaction with the applied field.
Sample preparation for $I_c$ measurements in ultra-high magnetic fields

*Ultra-narrow bridges*

- To compare the effect of different BZO pinning concentrations we used SuperPower Inc. tapes with 7.5% Zr and 15% Zr doping.
- Tapes with 30 µm, 38 µm, and 50 µm thick substrates and 5 µm thick Cu layer were used.
- Two stage photolithography process was used to pattern 4mm x 4mm samples.
- We used chemical etching to remove Cu, Ag, and ReBCO.
- To get uniform width and reduce $I_c$ all bridges were subsequently trimmed with a focused ion beam (FIB).
- Using NHMFL facilities we prepared 14 bridges ranging 47µm- 7.7µm in length and 4.8µm – 0.49µm in width.
Sample preparation

Sample: 4X4 mm;
Bridge: 50 um X 200 um
Current pads: 820 um X 830 um

Used photolithography to pattern structures

Used chemical etching

Bridges were trimmed with focused ion beam in SEM

Mask pattern
For ultra narrow bridges we choose region free from a-axis grains and CuO particles.
Thanks to Bob Goddard and Fumetake Kametani we have very negligible stage drift and therefore can make routinely bridges about ~0.5 µm wide and about 8 µm long.
SEM image of bridge N10
SEM image of bridge N10

V 1 = 8.118 µm

H 1 = 498.1 nm
• To prevent overheating and generating forces/torques due magnetization currents minimize volumes of conductive materials in sample;

• To avoid using massive current leads reduce critical current values by reducing bridge cross section;

• To prevent breaking a bridge by Lorentz force choose sample orientation in which Lorentz force is directed toward substrate;

• To avert large Eddy currents in current and voltage leads use twisted pairs with a small pitch. Place connections between probe leads and sample leads away from field center.

• To avoid voltage and bias current noise fix voltage and current leads to probe;
Electronics used for ultrafast $I_c (B,T)$ measurements:

- For data acquisition we use RedPitaya development board with
  - RF input and output: DAC, ADC - Sample rate **125 Msps**
  - CPU: Dual core ARM Cortex A9 / FPGA: Xilinx Zynq 7010 SoC
  - Used to generate bias current signal, detect sample voltage and voltage proportional to sample current
  - RedPitaya was programmed in Verilog
  - Board was connected with PC via TCP protocol (Internet)
- Current source: Valhalla Scientific 2500
- Voltage amplifiers:
  - Home built X100,
  - Stanford Research SR560 with low noise preamplifier
- Cryonon 22C temperature controller
Width and length of FIB trimmed ReBCO bridges

SuperPower 15% Zr doped tapes

$W, \mu m$

$L, \mu m$
Estimation of bias current values by extrapolation

$I_c$ measured in 15T SC magnet rescaled from full width tape to bridge width

We need to measure currents up to 10 mA - 200 mA
Expected Lorentz force at 60T for prepared bridges
Geometry of FIB trimmed ReBCO bridges from SuperPower tapes and estimated transport /mechanical properties

<table>
<thead>
<tr>
<th>Bridge</th>
<th>SuperPower</th>
<th>Zr</th>
<th>IcMin@77K</th>
<th>Length</th>
<th>Width FL</th>
<th>ReBCO</th>
<th>Vc at &lt;E&gt;</th>
<th>Ic at 60T</th>
<th>F Lorentza</th>
<th>Shear Stre</th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>tape I.D.</td>
<td>%</td>
<td>A</td>
<td>um</td>
<td>um</td>
<td>um</td>
<td>nV</td>
<td>mA</td>
<td>Extrapolate</td>
<td>mgf</td>
</tr>
<tr>
<td>1 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>46.7</td>
<td>3.3</td>
<td>1.55</td>
<td>4.67</td>
<td>82</td>
<td>23.43</td>
<td>44.92</td>
<td>problem with voltage leads</td>
</tr>
<tr>
<td>2 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>45</td>
<td>4.8</td>
<td>1.52</td>
<td>4.5</td>
<td>116.7</td>
<td>32.13</td>
<td>43.19</td>
<td>problem with voltage leads</td>
</tr>
<tr>
<td>3 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>25.2</td>
<td>2.1</td>
<td>1.53</td>
<td>2.52</td>
<td>52</td>
<td>8.02</td>
<td>24.47</td>
<td>problem with voltage leads; problem with ReBCO etching</td>
</tr>
<tr>
<td>4 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>20.1</td>
<td>1.9</td>
<td>1.52</td>
<td>2.01</td>
<td>68</td>
<td>8.36</td>
<td>28.40</td>
<td>problem with voltage leads; problem with ReBCO etching</td>
</tr>
<tr>
<td>5 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>15</td>
<td>2.48</td>
<td>1.523</td>
<td>1.5</td>
<td>63</td>
<td>5.78</td>
<td>15.01</td>
<td>problem with voltage leads</td>
</tr>
<tr>
<td>6 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>9.7</td>
<td>1.21</td>
<td>1.53</td>
<td>0.97</td>
<td>29.4</td>
<td>1.74</td>
<td>9.24</td>
<td>problem with voltage leads</td>
</tr>
<tr>
<td>7 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>10</td>
<td>1.2</td>
<td>1.52</td>
<td>1</td>
<td>29.4</td>
<td>1.80</td>
<td>9.87</td>
<td>problem with big contact pads</td>
</tr>
<tr>
<td>8 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>14.8</td>
<td>1.44</td>
<td>1.55</td>
<td>1.48</td>
<td>35.8</td>
<td>3.24</td>
<td>14.24</td>
<td>problem with big contact pads; ReBCO etched too much - problem</td>
</tr>
<tr>
<td>9 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>7.7</td>
<td>0.76</td>
<td>1.47</td>
<td>0.77</td>
<td>18.4</td>
<td>0.87</td>
<td>7.81</td>
<td>problem with big contact pads; ReBCO etched too much - problem</td>
</tr>
<tr>
<td>10 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>8.1</td>
<td>0.49</td>
<td>1.513</td>
<td>0.81</td>
<td>11.9</td>
<td>0.59</td>
<td>7.80</td>
<td>problem with big contact pads</td>
</tr>
<tr>
<td>11 M4-290-8</td>
<td>15</td>
<td>134</td>
<td>9.9</td>
<td>0.697</td>
<td>1.538</td>
<td>0.99</td>
<td>16.8</td>
<td>1.01</td>
<td>9.20</td>
<td>problem with big contact pads</td>
</tr>
<tr>
<td>12 M3-1113-16</td>
<td>7.5</td>
<td>116</td>
<td>15.11</td>
<td>0.66</td>
<td>1.49</td>
<td>1.511</td>
<td>14.8</td>
<td>1.37</td>
<td>13.64</td>
<td>problem with big contact pads</td>
</tr>
<tr>
<td>13 M4-337-2</td>
<td>7.5</td>
<td>99</td>
<td>12.4</td>
<td>0.577</td>
<td>1.538</td>
<td>1.24</td>
<td>15.8</td>
<td>1.20</td>
<td>13.26</td>
<td>problem with big contact pads</td>
</tr>
<tr>
<td>14 M4-337-2</td>
<td>7.5</td>
<td>99</td>
<td>15.7</td>
<td>0.771</td>
<td>1.55</td>
<td>1.57</td>
<td>21.1</td>
<td>2.03</td>
<td>16.83</td>
<td>problem with big contact pads</td>
</tr>
</tbody>
</table>

Comments:

- problem with voltage leads
- problem with ReBCO etching
- pattern with big contact pads
- ReBCO etched too much - problem
Temperature dependence of self field $I_c$ for Bridge N5

May 9, 2016
Bridge N5 measured with KE 6220 and KE2182A
Example of I-V curve for bridge N10 measured at 84.6 K; SF

I-V point measure during ~70 ms. with voltage noise ~25 nV (SD=9nV)
$R(B,T)$ measured in pulsed magnet - bridge N5

33.6° to magnetic field; maximum Lorentz force
$H_{\text{irr}}(T)$ measured in pulsed magnet - bridge N5

33.6° to magnetic field; maximum Lorentz force
$H_{irr}(T)$ for bridges N5 and N10

BN5 at 33.6° to magnetic field; BN10 at 41.6° to magnetic field; maximum Lorentz force orientation
Example of time dependencies of magnetic field, bias current and sample voltage for bridge N10 at 52K

Current step 200 µA

Voltage trace values are calculated by FPGA algorithm at the end of each current pulse as a difference between current on and current off portions of the pulse.

41.6 deg. to magnetic field; maximum Lorentz force orientation
Time dependencies of magnetic field, bias current and sample voltage for bridge N10 at 30K

Current step 200 μA

We detected one V-I-B point in about 0.33 ms. with voltage noise reaching up to about 2-3 μV. Field values were sampled at rate 2 μs. per point.

Duration of this process 36 ms.
Example of $V(I,B)$ curve measured at 30K in pulsed field
Seven $V(I,B)$ curves measured at 30K (maximum $B=50T$)
Time dependencies of magnetic field, bias current and sample voltage for bridge N10 at 10.4K

Current step 220 µA
Current offset 100 µA

Couple hundred megabytes of high resolution current and voltage traces are recorded by the same apparatus for each 30-65T magnet pulse, allowing careful off-line analysis of current-voltage response.

Duration of this process 39 ms.
Transport $I_c(B)$ at $3 \mu V \approx 3700 \mu V/cm$ of 15% Zr doped ReBCO oriented at $41.6^\circ$ to field measured in pulsed magnet. This sample survived 19 pulses $B_{max} = 5T - 65T$.
Transport $J_c(B)$ at $3\mu V$ ($\approx 3700 \mu V/cm$) of 15% Zr doped ReBCO oriented at $41.6^\circ$ to field measured in pulsed magnet

$T=30K$ data fit to $I\downarrow c \sim B\uparrow - \alpha$
- $14.3 T - 49.5 T \quad \alpha = -1.94$
- $14.3 T - 34 T \quad \alpha = -1.63$

$T=10.4 K$
$T=30 K; run 1$
$T=30 K; run 2$
Calculating $I_c$ defined at standard voltage criterion $V_o$ ($<E>=1 \mu V/cm$) from data measured at different voltage criterion using n-value

$$V\downarrow 1 = V\downarrow c (I\downarrow c1 / I\downarrow c )^n$$

$$I\downarrow c = I\downarrow c1 / \exp[\ln(V\downarrow 1 / V\downarrow c )/n]$$
Timescales related to this experiment

V-I-B point measured in pulsed mode $\sim 0.332$ ms.

Current pulse length $t_{\text{current}} = 0.166$ ms. (smooth $\sin(\omega t)$ shaped)

Magnet pulse duration about 100 ms.

$\frac{\text{dB}}{\text{dt}} = 2408 \text{T/s}$, which is about 9 time larger max. $\frac{\text{dB}}{\text{dt}}$ in AC modern loss setup

“Inverse attempt frequency of pinned fluxons usually to is assumed to be of order $10^{-10} - 10^{-13}$ s.”

$10^{-10} \ll t_{\text{current}}$

Therefore in spite of fast V-I-B detection we could expect observing flux creep regime and therefore power dependence $E \sim J^n$.

However, exposing sample to high $\frac{\text{dB}}{\text{dT}}$ will lead to high electric fields $10^2 - 10^3$ $\mu$V/cm much above $E_c = 1$ $\mu$V/cm. Therefore we expect to get flux flow regime.


Fitting $I_c$ data from $V(I,B)$ curves

To calculate $I_c$ in experiments with DC field we use **several I-V points** near region of resistive transition by fitting data points with dependence $V=V_{\downarrow c} \left( \frac{I}{I_{\downarrow c}} \right)^n$.

During pulsed measurements we measure $V(I,B)$ dependence, therefore fitting is more difficult:

\[
V(I,B)=V_{\downarrow c} \left( \frac{I}{I_{\downarrow c}} (B) \right)^n
\]

Within small field range

\[
I_{\downarrow c} (B)=I_{\downarrow c0} \ B^{\uparrow -\alpha}
\]

Combining above equations together:

\[
V(I)=V_{\downarrow c} \left( \frac{I}{I_{\downarrow c0}} (B_{\downarrow 0} + dB/dI (I-I_{\downarrow 0})) \right)^{\uparrow -\alpha} \uparrow^n
\]

For short periods of time:

\[
B(t)\approx B_{\downarrow 0} + dB/dt \ t
\]

\[
I(t)=I_{\downarrow 0} + dI/dt \ t
\]

\[
B(I)\approx B_{\downarrow 0} + dB/dI (I-I_{\downarrow 0})
\]

Near pulse peak we may add $d^2 B/dI^2$ term

Too many fitting parameters
Summary

• We have shown the possibility of detecting the $I_c$ of high $J_c$ ReBCO tapes in ultra-high pulsed fields;
• However $\alpha$ values ($I\downarrow c \sim B\uparrow -\alpha$) are unrealistically high.

Future plans:
• In future to understand the influence of I-V sampling rate, we plan to correlate $J_c$ measured with standard low noise electronics and FPGA-based electronics using a superconducting magnet;
• To get more $V(I,B,T)$ curves per magnet pulse, we plan to introduce time-dependent maximum ramping current synchronized with pulse profile $B$(time).
• With more $I_c$ points measured, we expect to get smooth pinning force density $f_p(B,T)$ dependencies in the ultra-high field region;
• We plan to correlate $J_c(B,T)$ and $f_p(B,T)$ measured in ultra-high fields at different orientations with images of pinning defects obtained on the state of the art sub-Angstrom scanning/transmission electron microscope (NHMFL-FSU). Such correlations can be used for improving understanding of pinning anisotropy in new coated conductor tapes and, therefore, stimulate engineering pinning precipitates optimized for magnet applications.