Understanding In-Field Performance of REBCO Conductor with Artificial Pinning Centers by Scanning Raman Spectroscopy and 2D-XRD

Goran Majkic

Department of Mechanical Engineering
Advanced Manufacturing Institute
Texas Center for Superconductivity
University of Houston, Houston, TX, USA
Collaborators:

Venkat Selvamanickam, Chirag Goel, Nathaly Castaneda - University of Houston

Andre Mkhoyan - University of Minnesota

Huikai Cheng, Lee Pullan, Dan Gostovic - Thermo-Fisher Scientific

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Background

Strong in-field performance can be achieved by combining thick (2-4 μm) REBCO films with optimized BMO pinning centers.

- Pinning force plateau of 2 TN/m³ at 4.2 K B>5T, B||c
- Near-identical performance between 15% Zr and 15% Hf
- At B||ab, near-linear increase in Fp up to 31.2 T

What determines equilibrium nanorod diameter and density?
How to quickly evaluate pinning center density over long lengths?
Performance at (77K, 0T) is not indicative of in-field performance at low temperatures and high fields.

What influences the large scatter observed, regardless of deposition technique?

Can we determine performance at (B,T) of interest quickly and non-destructively?
TEM – Nanorod Self-Assembly:
Scenario 1

- Hole: N unit cells
- Nanorod: N unit cells
- Compress nanorod
- Insert nanorod and relax
- Coherent
TEM – Nanorod Self-Assembly:
Scenario 2

Hole: N unit cells

Nanorod: N-1 unit cells

Insert nanorod and relax:
Minimal compression to match hole
Minimized strain + misfit dislocations

Semi-Coherent
Plane view TEM:

BZO Nanorod in REBCO

Coherent in Y direction
(8 unit cells BZO, 8 unit cells REBCO)

Semi-Coherent in X direction
(7 unit cells BZO, 8 unit cells REBCO)
Yellow – peak pair (Ba-RE REBCO columns)
Cyan – peak pair (Cu REBCO columns)
White – peak pair (Ba BZO columns)

Classify as I-0 type:
(number of antiphase lines or missing unit cells in x and y directions)
Classify as I-I type:

(number of antiphase lines or missing unit cells in x and y directions)

NOTE: I-I size larger than I-0
Minimization of Mismatch strain:

\[ \epsilon = \frac{M}{M - P} k - 1 \]

- \( a_M \) and \( a_N \) - lattice parameters (matrix and nanorod)
- \( k = a_M/a_N \)
- Matrix hole \( M \) unit cells
- Nanorod \( N = M - P \) unit cells

Deep minima in strain for ~14 and 28 unit cells.
Where is the II-II type?

Bottleshape modulation in BZO diameter: from I-I to II-II back to I-I
Nanorods assume discrete sizes:
Bimodal Distribution of Nanorod Diameter

a.) nominal 25% Zr
b.) nominal 11% Zr

Amount of dopant alone does not determine nanorod density.

Nanorod type is important.
Dopant amount alone does not control amount of nanorods (#2):

RE elements dissolve in BaZrO$_3$!

This forms BaZr$_{1-x}$RE$_x$O$_3$

For every RE dissolved, we increase amount of Ba(M,RE)O$_3$.

$1$ BaZrO$_3$ + $y$ Ba + $y$ RE = $(1+y)$ Ba(Zr,RE)O$_3$

Where do excess RE and Ba come from?
Dopant amount alone does not control amount of nanorods

Where do excess RE and Ba come from?

1.) Ba non-stoichiometry.

Variable $\text{BZC} = \frac{Ba+Zr}{Cu}$ metric ($Zr=\text{constant}$):

Increase in BZC results in depletion of REO precipitates
Dopant amount alone does not control amount of nanorods

Where do excess RE and Ba come from?

2.) Consume stacking faults:

$3 \text{REBCO}(124) = 4 \text{REBCO} (123) + 1 \text{RE} + 2 \text{Ba}$

124 stacking faults accommodate lattice mismatch along c direction.

Depletion of 124 will affect c-axis mismatch strain
Result: wide range of nanorod densities and in-field performances for same nominal amount of M dopant

All samples have 15% Zr addition (nominal).

Vary BZC content =>

Nanorods vary from <5% to ~23% (u.c. fraction)

Quick, nondestructive characterization of nanorod density and in-field performance is vital for both:

1.) process control
2.) conductor utilization
Nondestructive Evaluation:

1.) 2D-XRD
1.) Streak angle of BZO 101:
- Along $2\theta$ left
- Perpendicular to c-axis right

2.) REO 222
- Strong left
- Absent right

**Consistent with TEM:**
- Streak angle right = sinc broadening
- REO – consumed at high nanorod density
15% Zr – low nanorod density

1.) REBCO 006 2θ stretch
   - Increased (right)

2.) BZO 002 Intensity
   - Increased (right)

3.) REO 004
   - Decreased (right)

Consistent with TEM:
   - Loss of 124 s.f.
   - More BZO domains
   - REO consumed

15% Zr – high nanorod density

BZO 002: Intensity

REBCO 006: 2θ Stretch

REO 004
Inline Implementation: 2D-XRD

Streak angle correlates well with Jc at lower temperatures:
- At 4.2, 20 K from 2 T
- At 30, 40 K from 5 T

Pearson Correlation – BZO streak angle vs Jc(B,T)

2D-XRD integrated inline with A-MOCVD

X-ray Source
Tape path in A-MOCVD

2D Detector
Inline Implementation: 2D-XRD

Continuous inline measurement at feature extraction
Nondestructive Evaluation:

2.) Scanning Raman
Scanning Raman Capabilities Demonstrated for Defect Detection

Raman Integrated Spectra

$x = 4-6$ mm

Intensity (a.u.)

$y = 8.5-10.5$ mm

Cu2, O2+/O3-, O2+/O3+, O4, C-dis

$y = 5-7$ mm

$y = 0.5-2.5$ mm

Raman Shift [1/cm]

O2+/O3- Intensity

2D-XRD

NEXT: Can we characterize pinning center density?

Can we characterize pinning center density?

Strain: Tape Curvature vs O2+/O3- Raman Shift:

Strong linear correlation between measured curvature and Raman shift.

Higher nanorod density = higher residual strain = induced curvature.

Can be detected via O2/O3- peak shift:

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Comparison with 2D-XRD on same samples

- REBCO c-axis lattice parameter increase with curvature and BZO(002) intensity.
- BZO c-axis bimodal, (4.23 and 4.17 Å) – more 4.17 at higher curvature.
- REO intensity decrease with curvature
- Strong linear trend between BZO(002) intensity and Raman O2/O3- shift.

Increasing Curvature

Clear linear trend between BZO (002) integral and O2/O3- Raman shift:

Use Raman to evaluate nanorod density
Can Raman O2/O3- Predict Jc(B,T) and Retention $\rho(B,T)$?

Critical Current Density $J_c(B,T)$

Retention Factor $\rho(B,T) = \frac{J_c(0,T)}{J_c(B,T)}$

Both $J_c(B,T)$ and $\rho(B,T)$ vary widely over the selected samples.
Can Raman O2/O3- Predict Jc(B,T) and Retention $\rho(B, T)$?

For Jc(B,T), no strong correlation

However, for Retention $\rho(B,T)$:

strong negative correlation
(4.2-40K)
(~2 -14 T)

Significance Level: $p<0.01$
Cross Validation over a Large Data Set

Set of 53 samples.

A mixture of 0, 5 and 15% Zr or Hf.

Also, 0.025 and 0.05 M molarity

Correlation:

$$C = -0.81$$

($p < 0.01$)

Raman can measure retention factor

(proportional to pinning center density)
Thank you