IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), October, 2023. Presentation given at CEC-ICMC, July 11, 2023, Honolulu, Hawaii, USA

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

Tuesday, Jul 11, 2023
10:40-11:00 AM (20 min)
Room 318

M2Or1B: Latest Development in Flux Pinning & Critical Current - REBCO I
CEC-ICMC Conference, Jul 9 – 13, 2023, Honolulu, HI

M2Or1B-04

Collaborators:

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

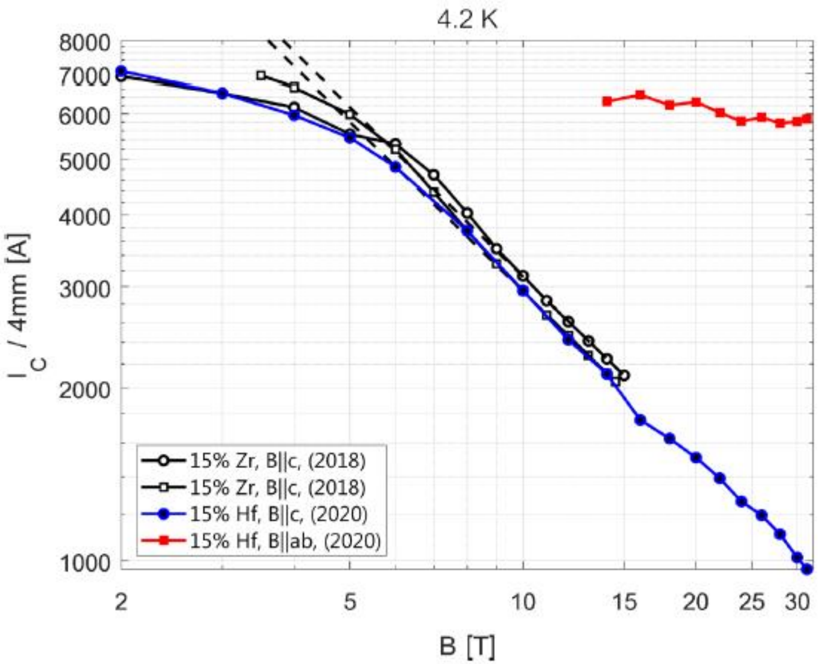
Andre Mkhoyan - **University of Minnesota**

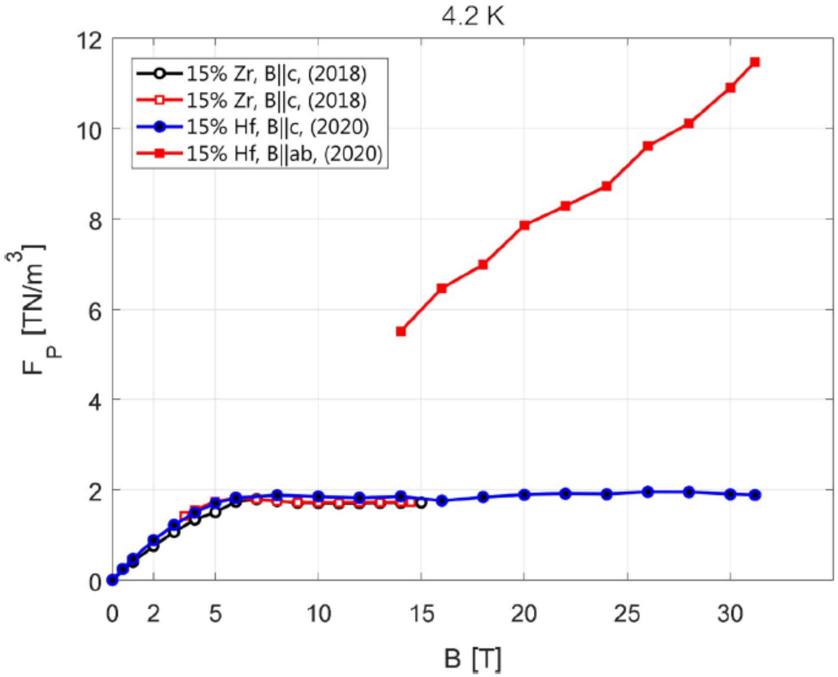
Huikai Cheng, Lee Pullan, Dan Gostovic - **Thermo-Fisher Scientific**

Funded by:
DE-SC0016220 U.S. Department of Energy, Office of Science – High Energy Physics
DE-AR0001374 from Advanced Research Projects Agency – Energy.

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/m^3 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?

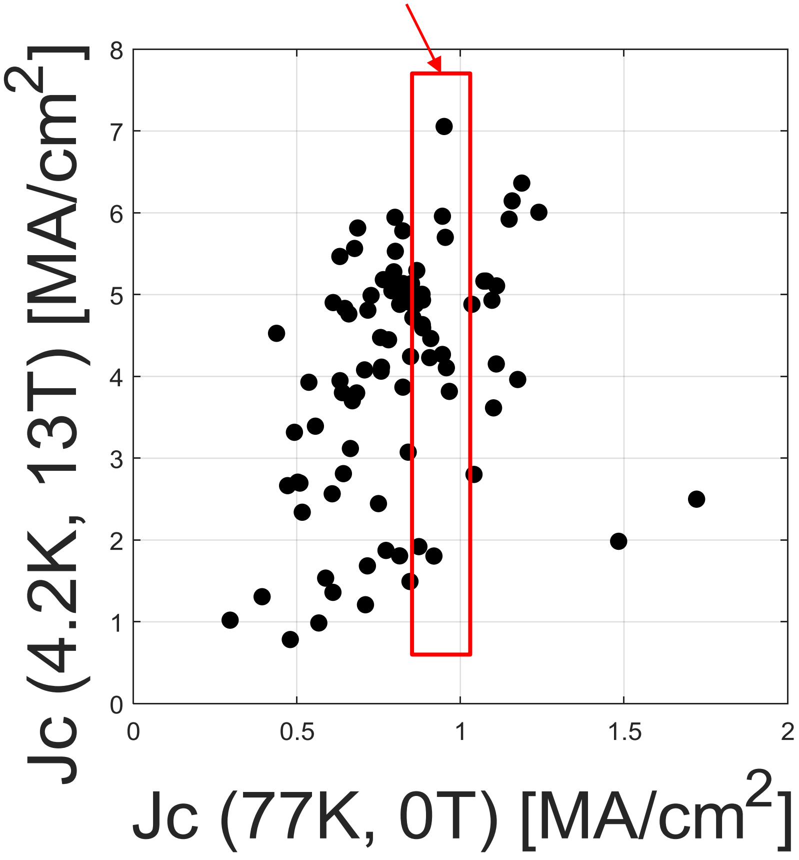
Supercond. Sci. Technol. **33** (2020) 07LT03 (8pp)

Background

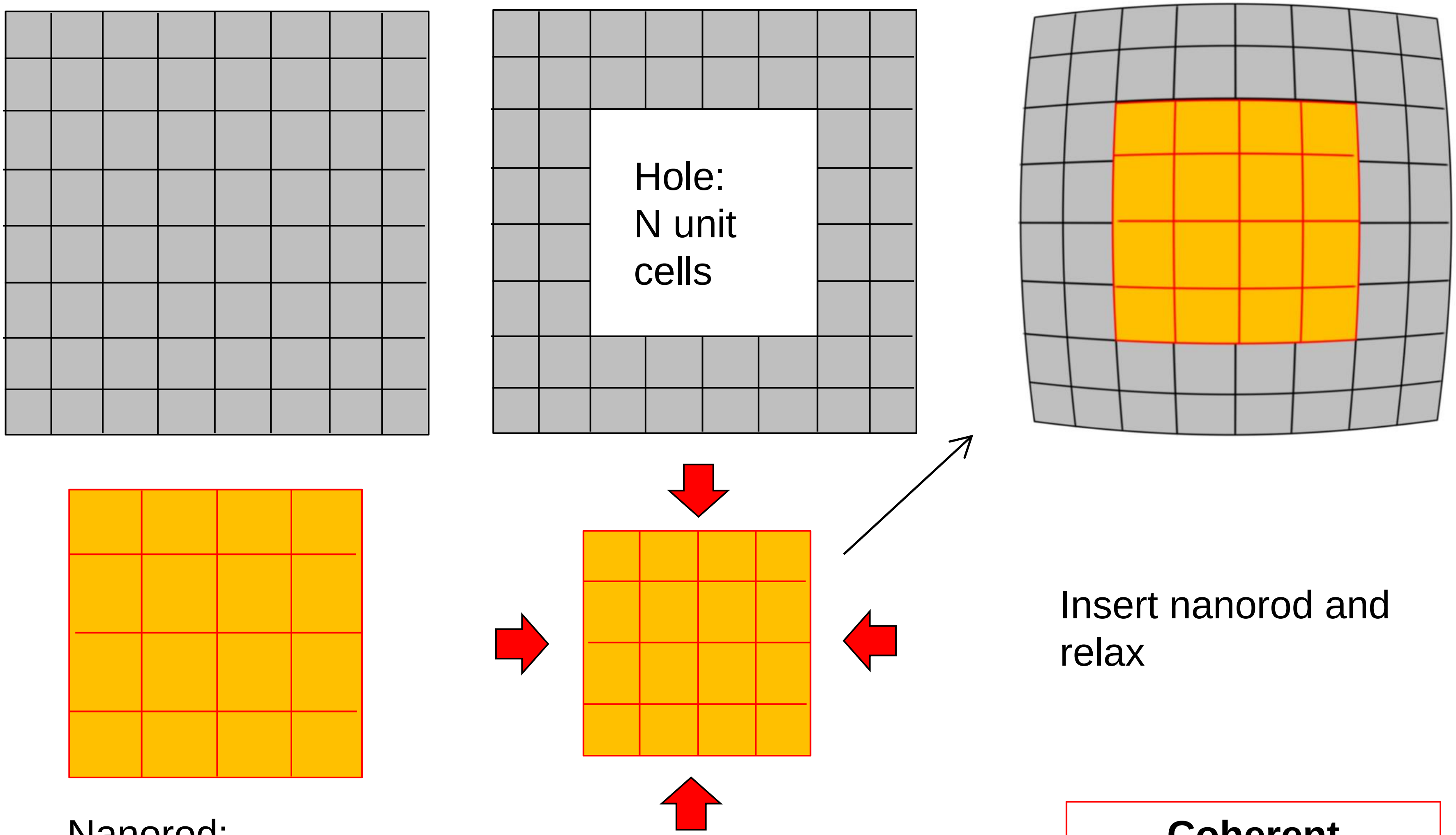
Performance at (77K, 0T) is not indicative of in-field performance at low temperatures and high fields

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

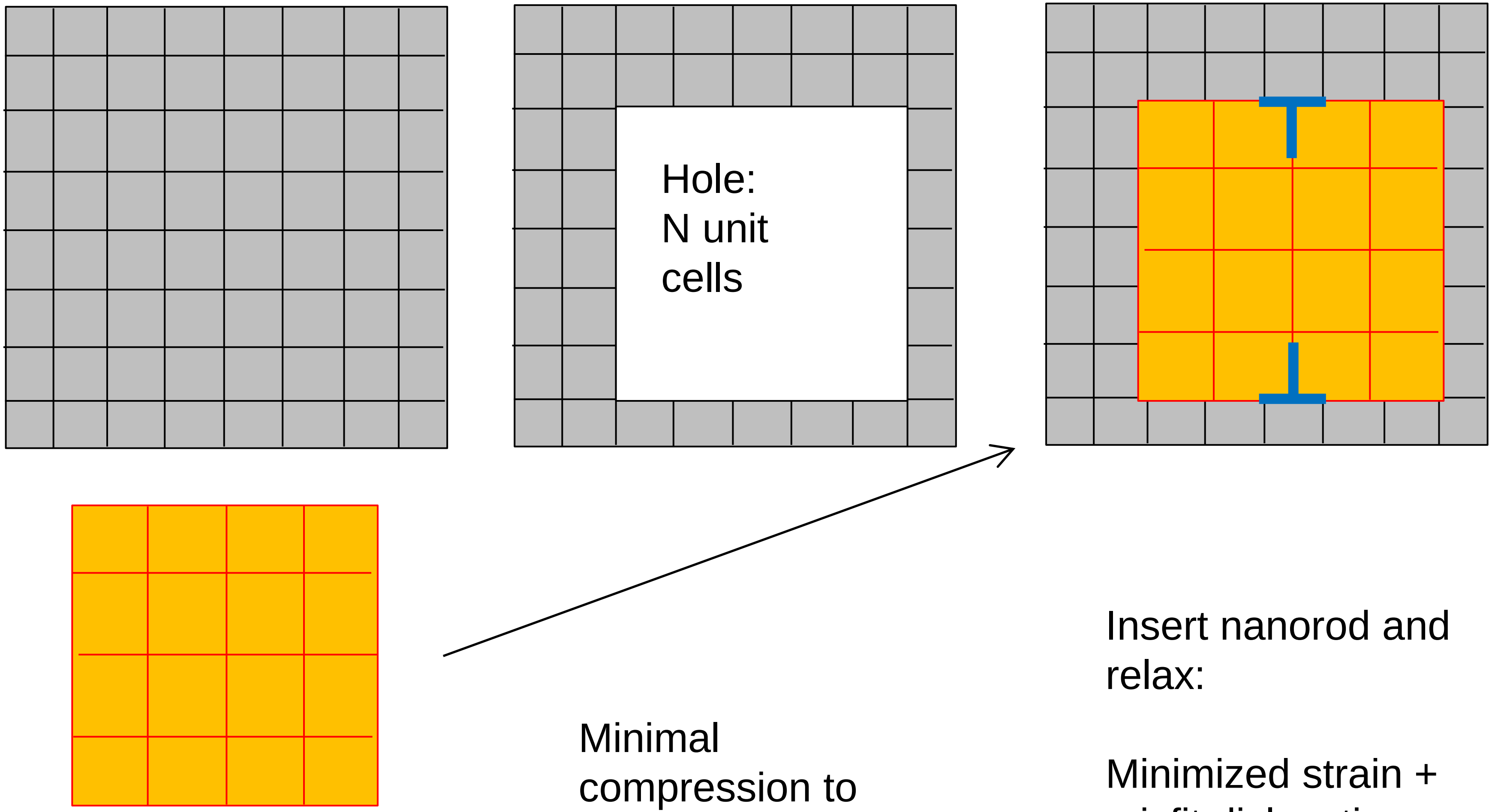
Nanorod:
N unit cells

Compress nanorod

Insert nanorod and relax

Coherent

TEM – Nanorod Self-Assembly: Scenario 2



Hole:
N unit
cells

Nanorod:
N-1 unit cells

Minimal
compression to
match hole

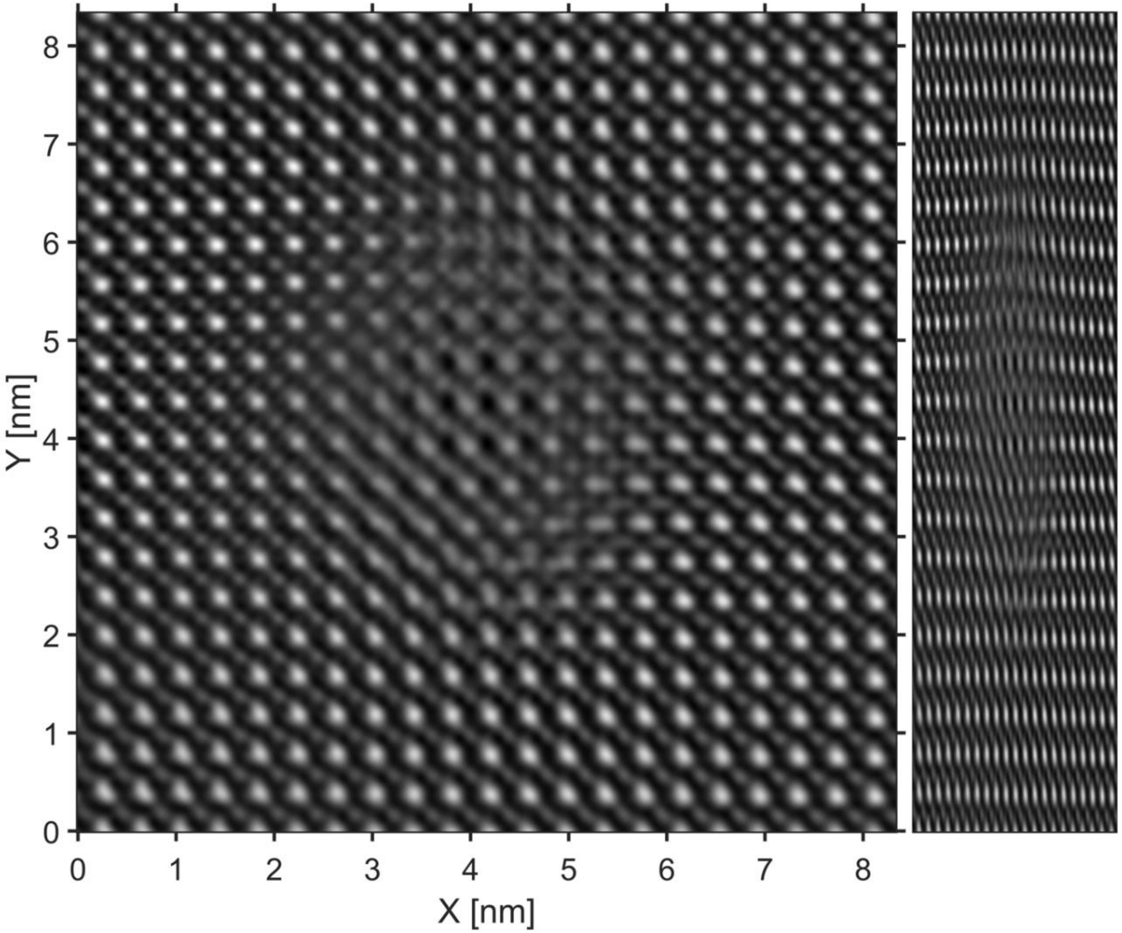
Insert nanorod and
relax:

Minimized strain +
misfit dislocations

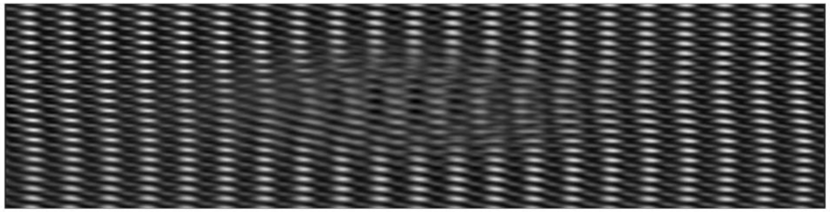
Semi-Coherent

Plane view TEM:

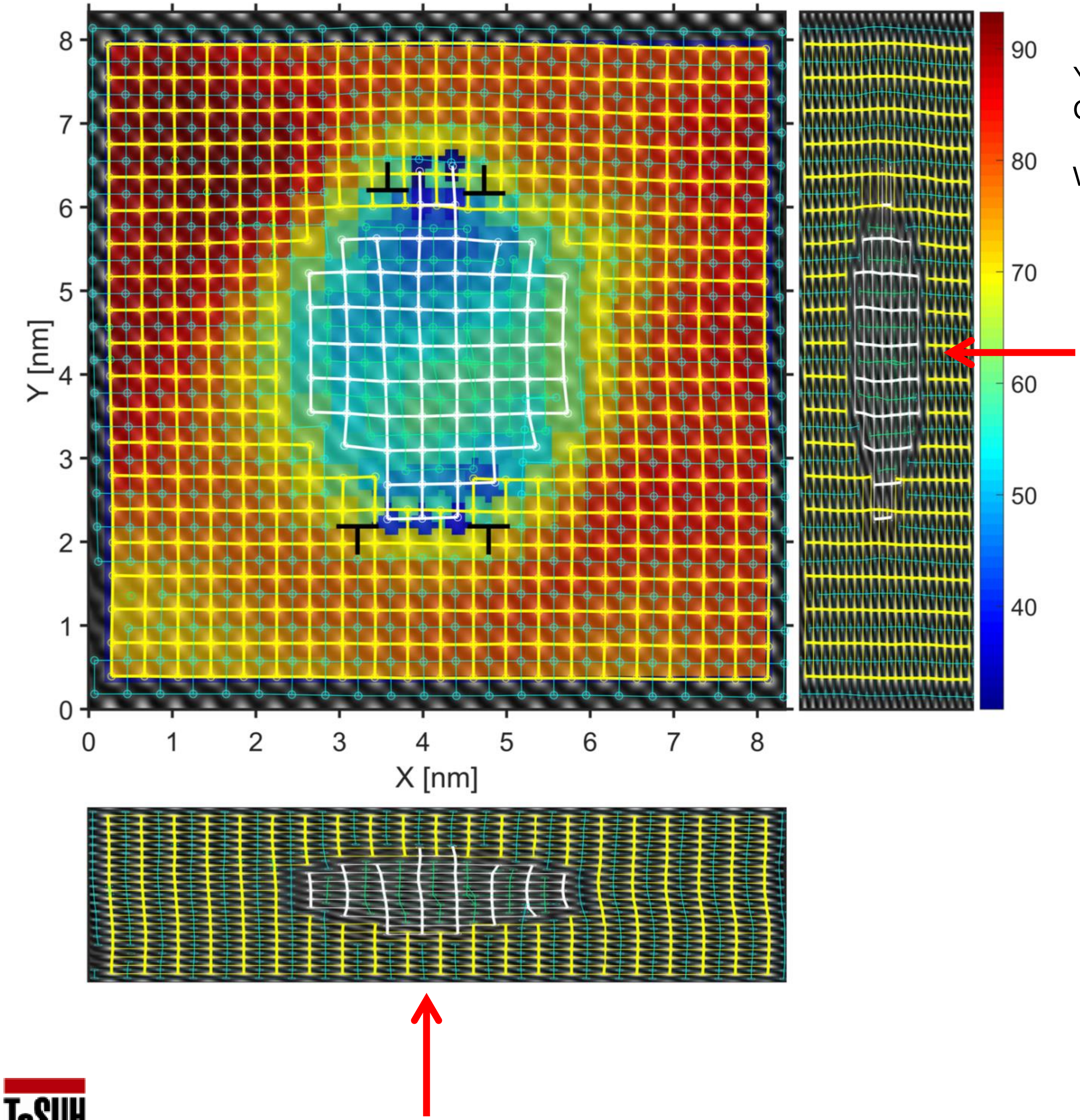
BZO Nanorod in REBCO



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)

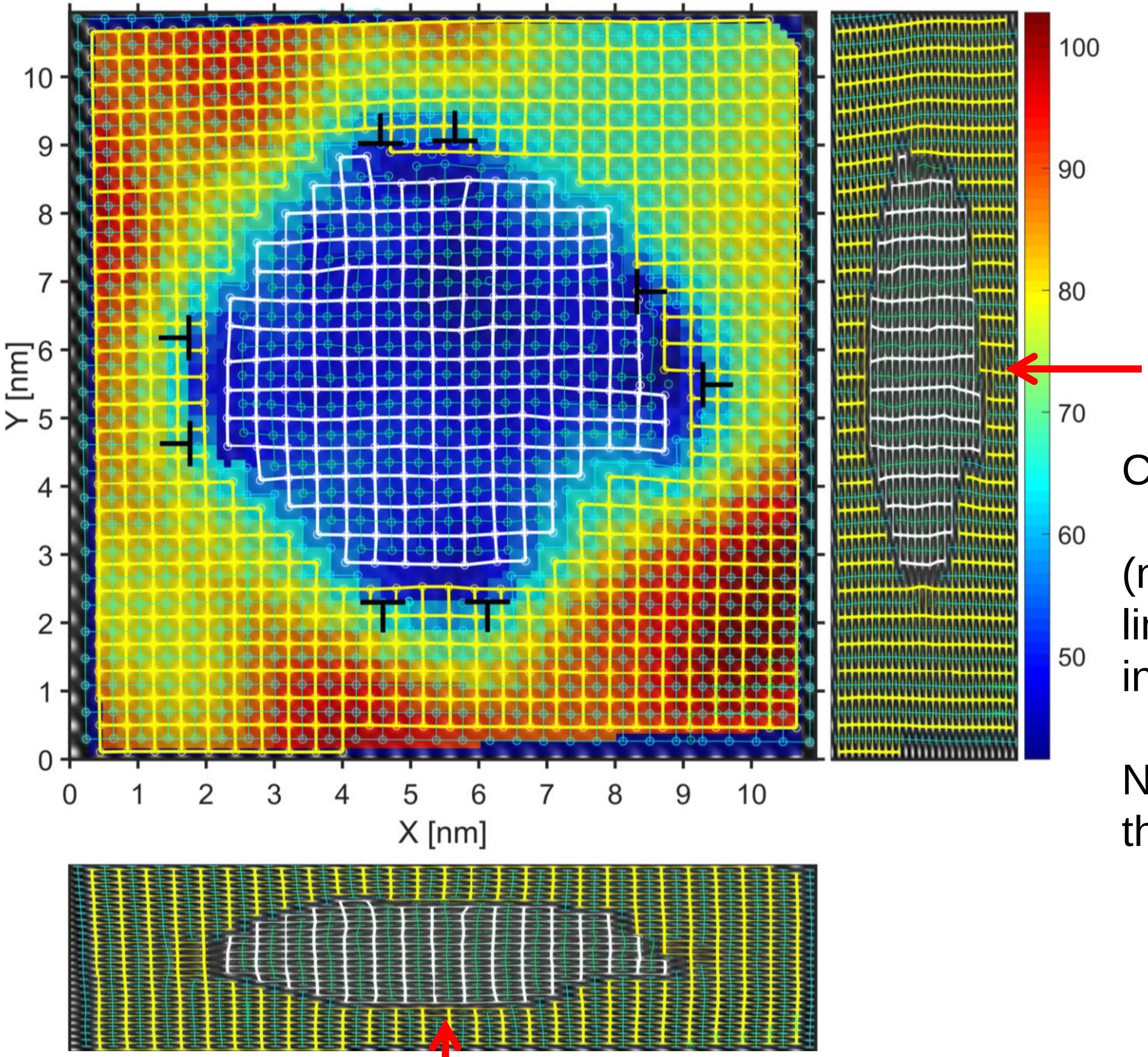


Yellow – peak pair (Ba-RE REBCO columns)
Cyan – peak pair (Cu REBCO columns)

White – peak pair (Ba BZO columns)

Classify as I-0 type:

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

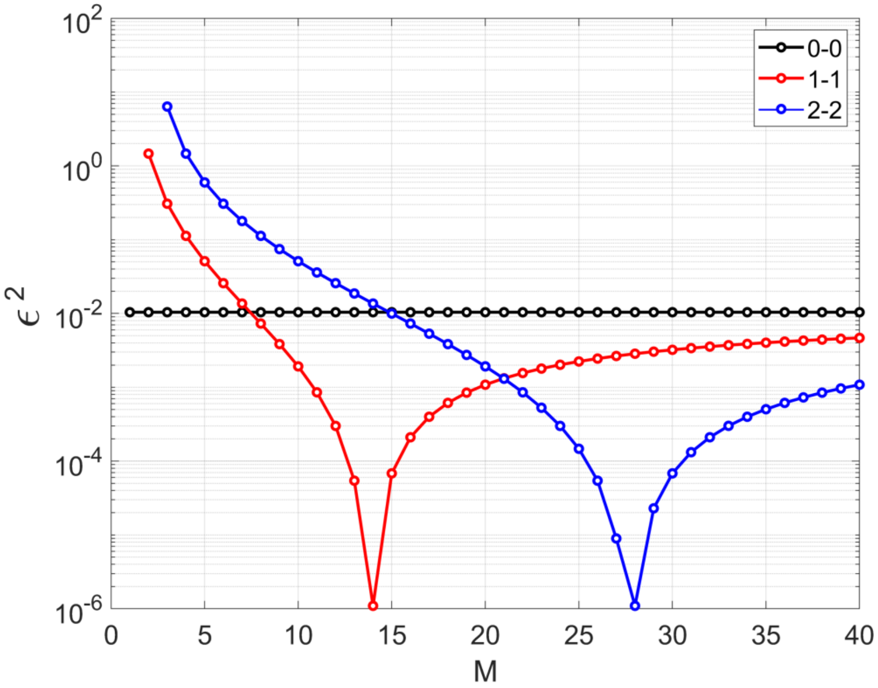


Classify as I-I type:

(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:



a_M and a_N - lattice parameters (matrix and nanorod)

$k = a_M/a_N$

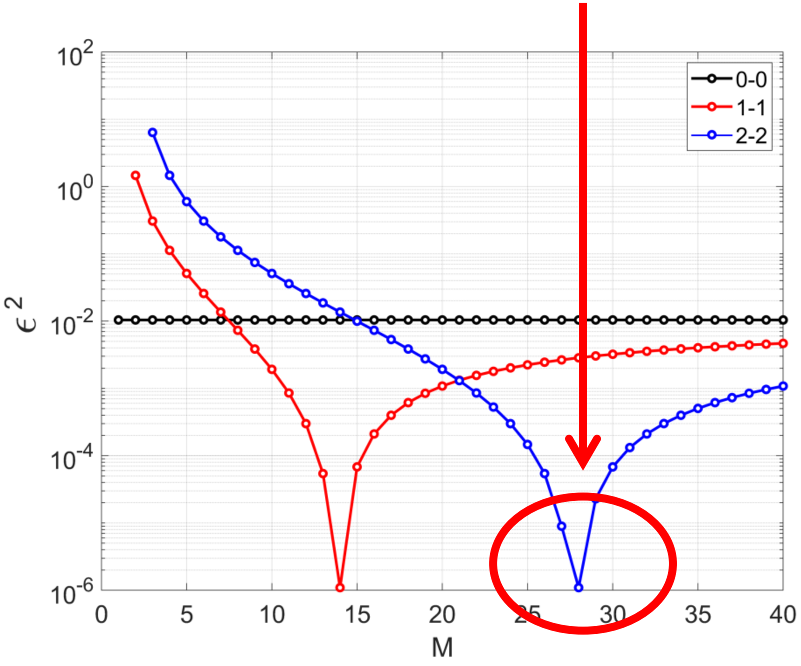
Matrix hole M unit cells
Nanorod N = M – P unit cells

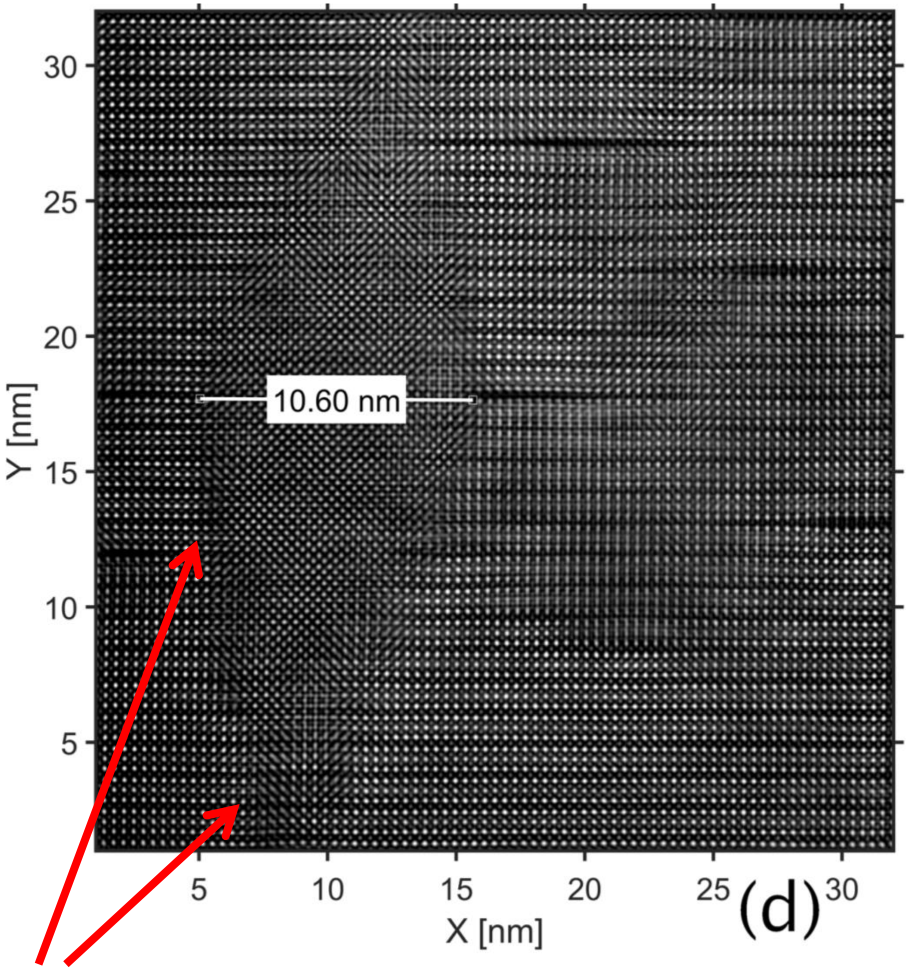
Mismatch strain:

$$\epsilon = \frac{M}{M-P}k - 1$$

Deep minima in strain for ~14 and 28 unit cells.

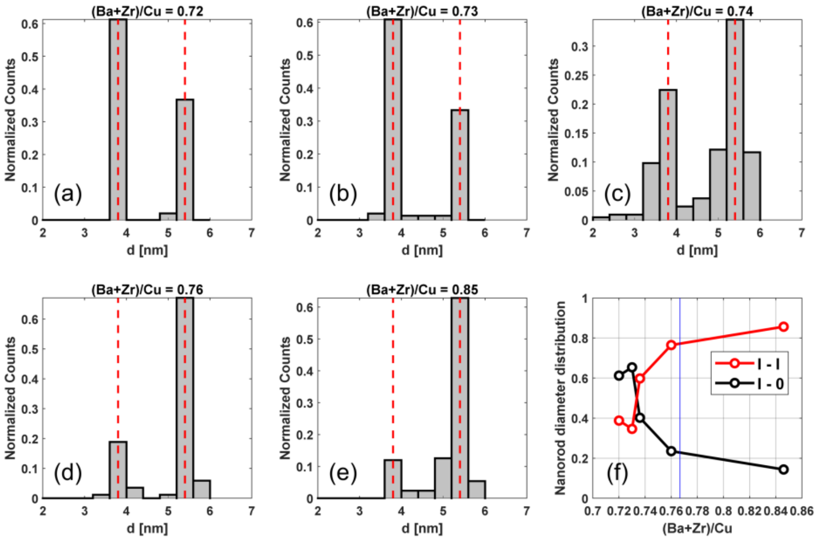
Where is the II-II type?

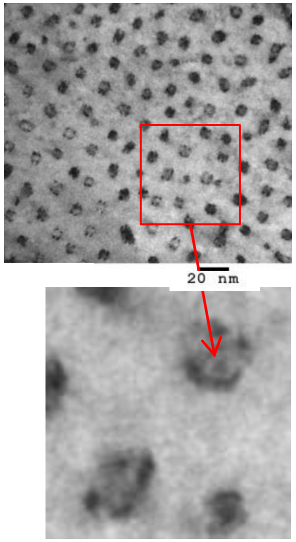


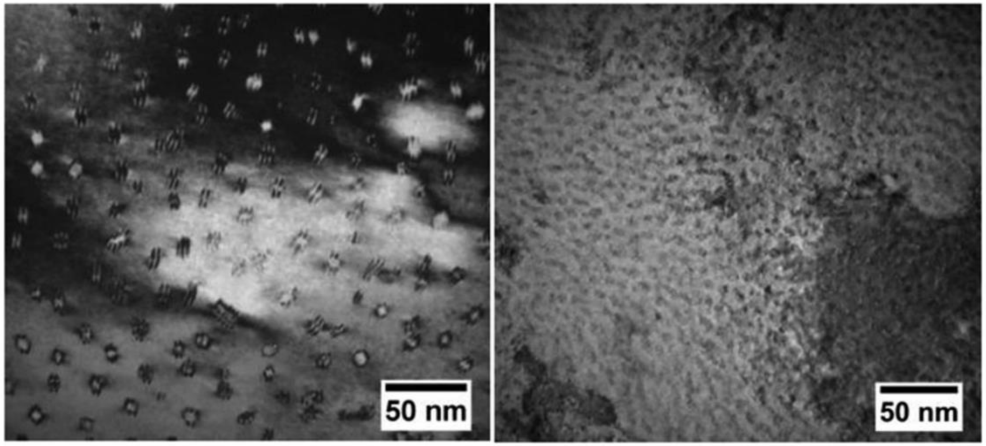


Bottleshape modulation in BZO diameter: from I-I to II-II back to I-I

Nanorods assume discrete sizes:
Bimodal Distribution of Nanorod Diameter







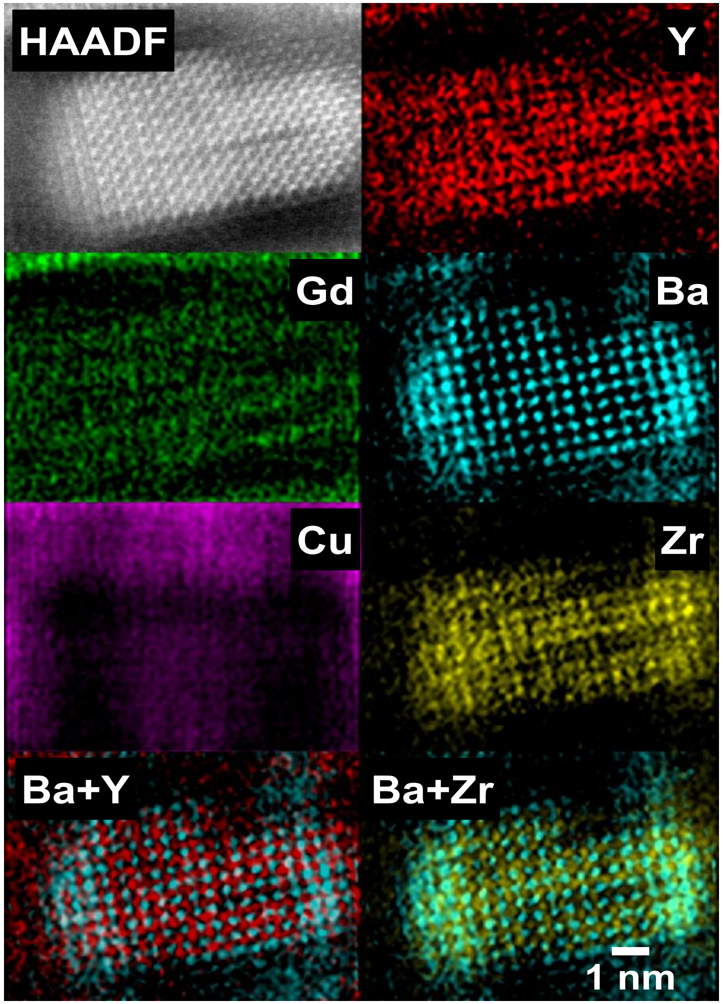
(a) (b)

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 $BaZrO_3$!

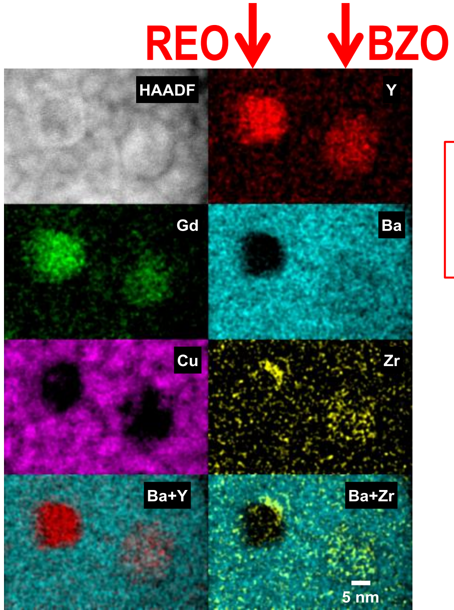
This forms $BaZr_{1-x}RE_xO_3$

For every RE dissolved, we increase amount of $Ba(M,RE)O_3$.

1 $BaZrO_3$ + y Ba + y RE = (1+y) $Ba(Zr,RE)O_3$

REO

BZO



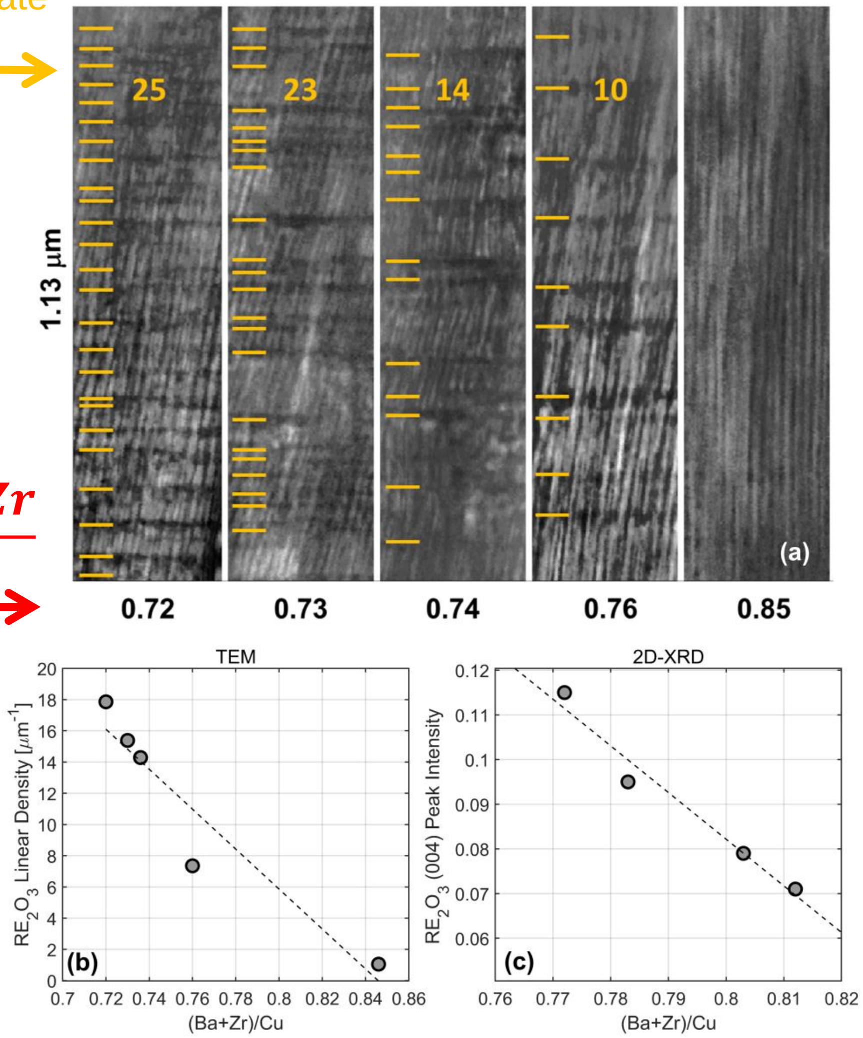
Where do excess RE and Ba come from?

Dopant amount alone does not control amount of nanorods

Where do excess RE and Ba come from?

RE_2O_3 precipitate arrays →

$$\frac{Ba+Zr}{Cu}$$ →



<u>1.) Ba non-stoichiometry.</u>

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

Increase in BZC results in depletion of REO precipitates

Dopant amount alone does not control amount of nanorods

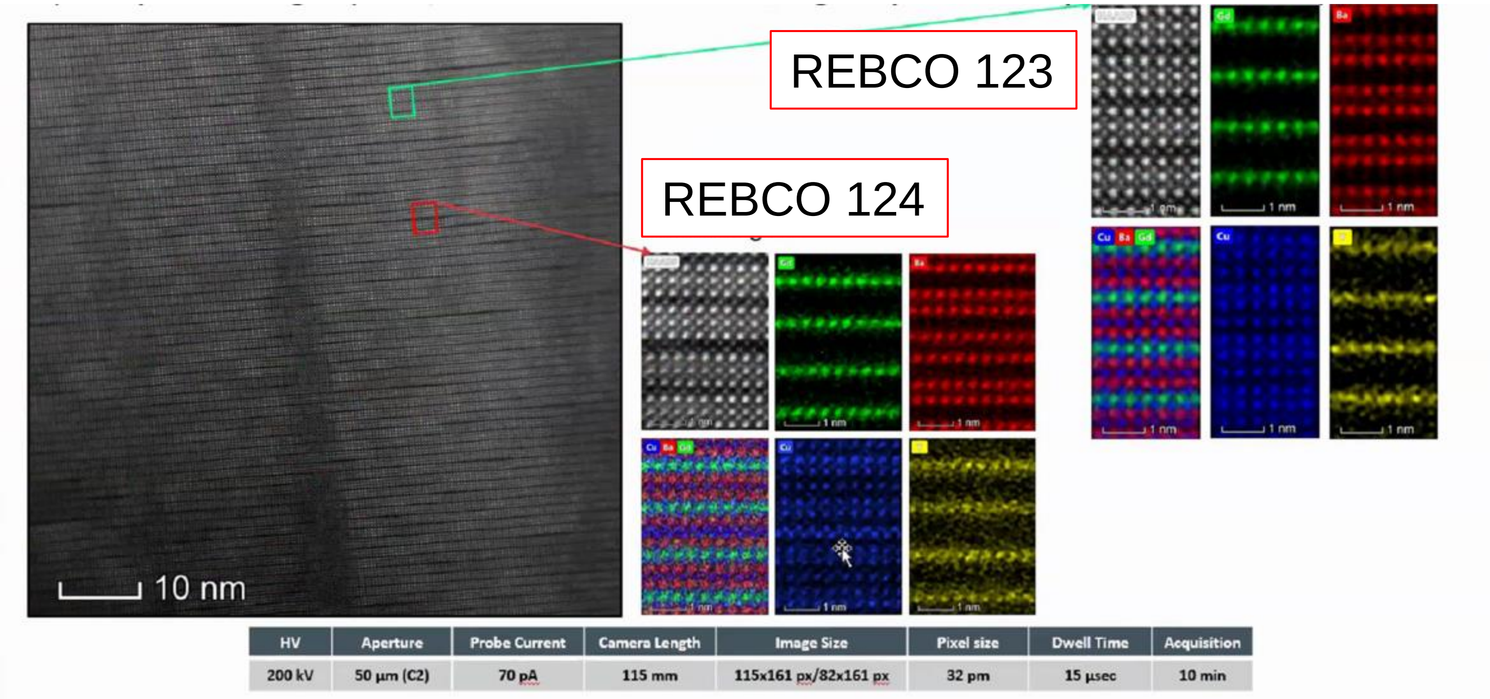
Where do excess RE and Ba come from?

2.) Consume stacking faults:

3 REBCO(124) = 4 REBCO (123) + 1 RE + 2 Ba

124 stacking faults accommodate lattice mismatch along c direction.

Depletion of 124 will affect c-axis mismatch strain

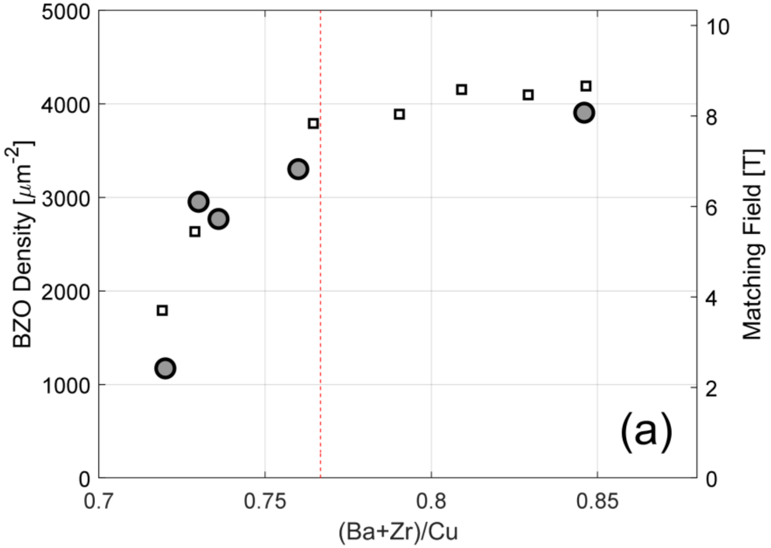


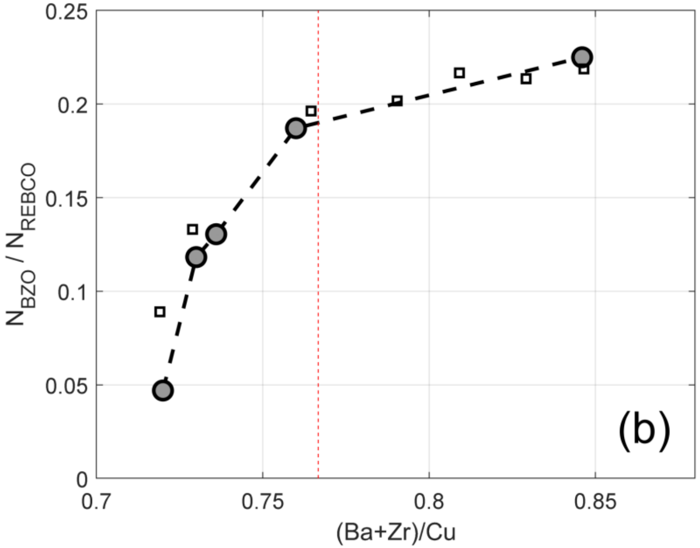
REBCO 123

REBCO 124

HV	Aperture	Probe Current	Camera Length	Image Size	Pixel size	Dwell Time	Acquisition
200 kV	50 µm (C2)	70 pA	115 mm	115x161 px/82x161 px	32 pm	15 µsec	10 min

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)

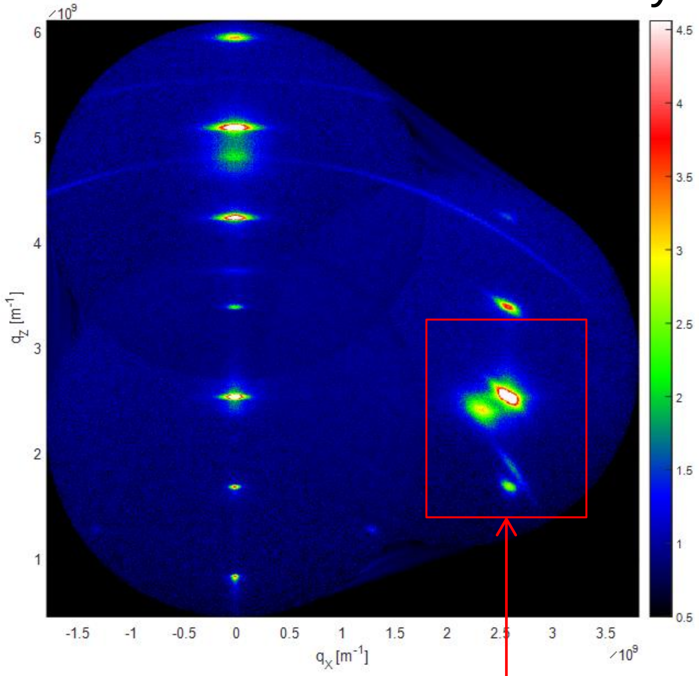
Quick, nondestructive characterization of nanorod density and in-field performance is vital for both:

1.) process control
2.) conductor utilization

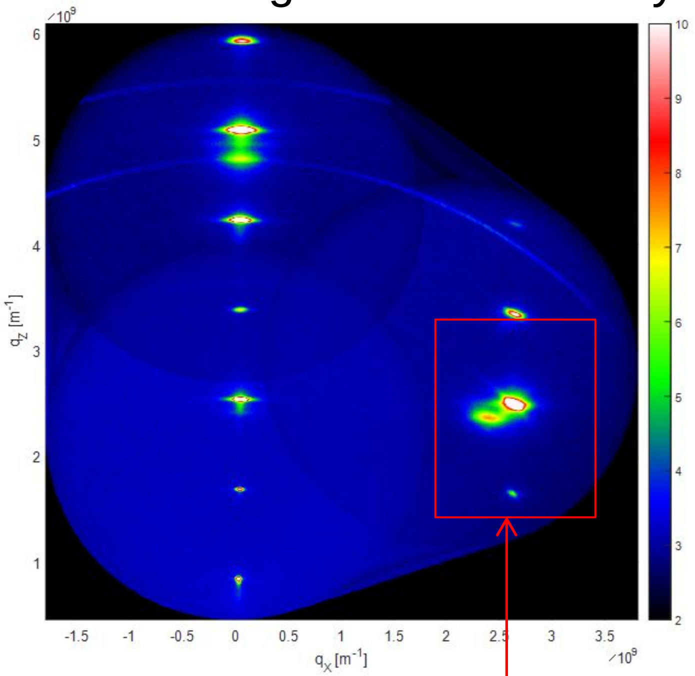
Nondestructive Evaluation:

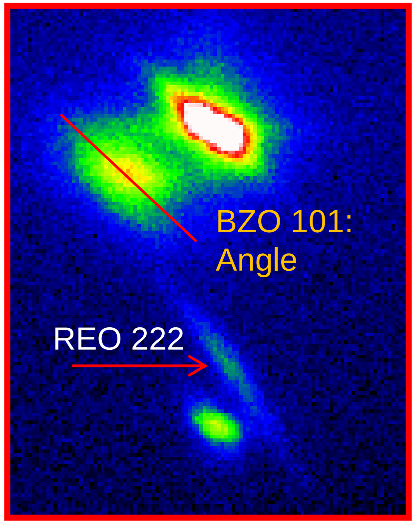
1.) 2D-XRD

15% Zr – low nanorod density



15% Zr – high nanorod density





1.) Streak angle of BZO 101:

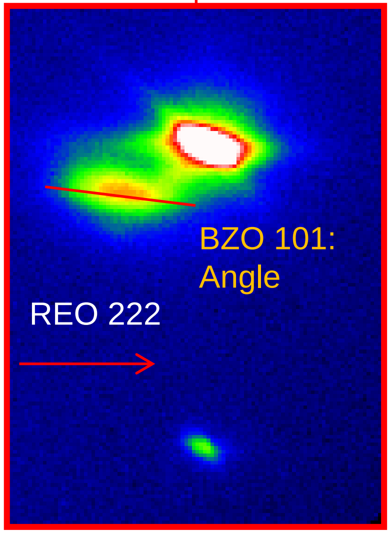
- Along 2θ left
- Perpendicular to c-axis right

2.) REO 222

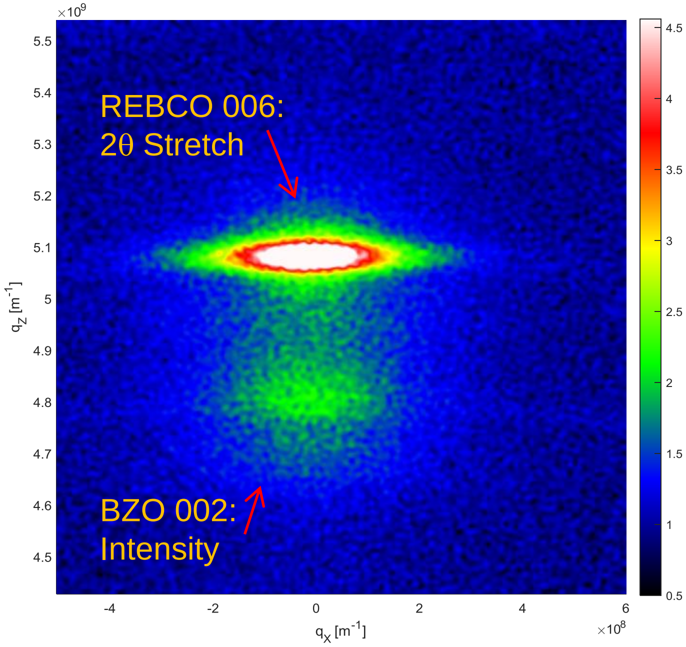
- Strong left
- Absent right

Consistent with TEM:

- Streak angle right = sinc broadening
- REO – consumed at high nanorod density



15% Zr – low nanorod density

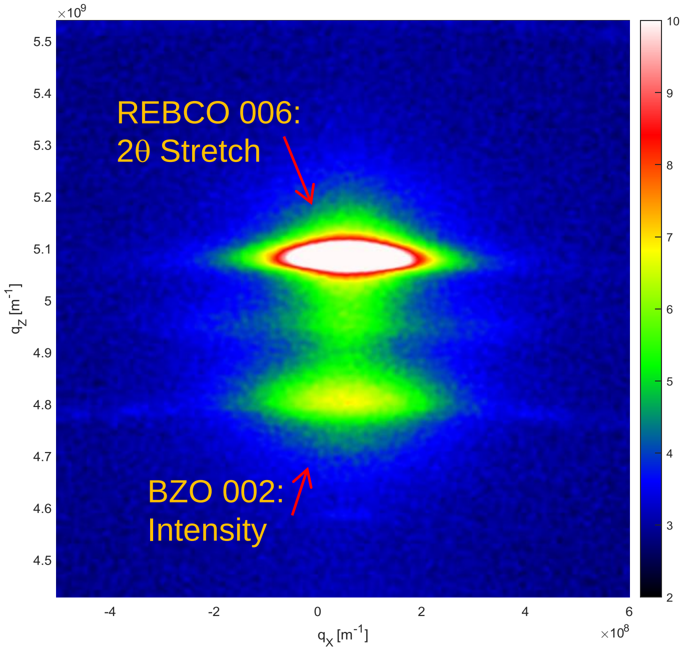


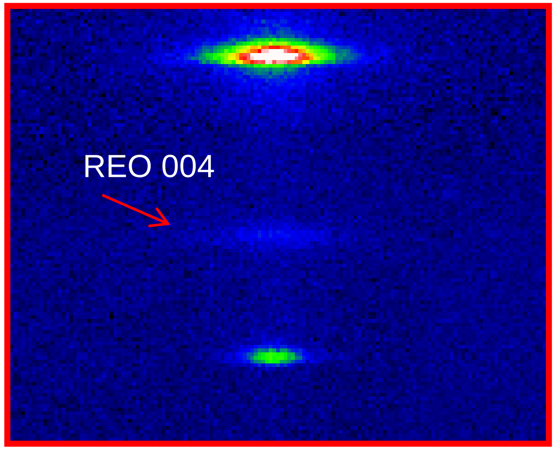
1.) REBCO 006 2θ stretch
- Increased (right)

2.) BZO 002 Intensity
- Increased (right)

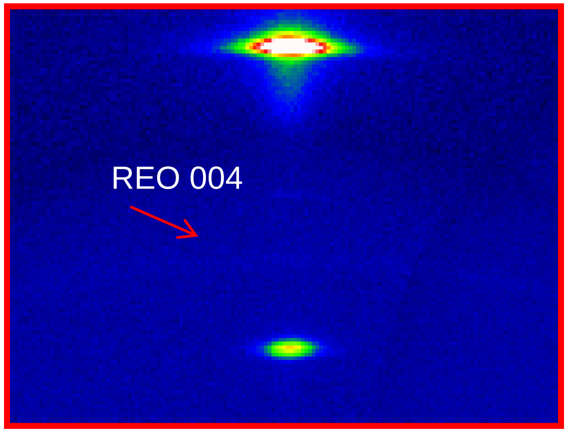
3.) REO 004
- Decreased (right)

15% Zr – high nanorod density

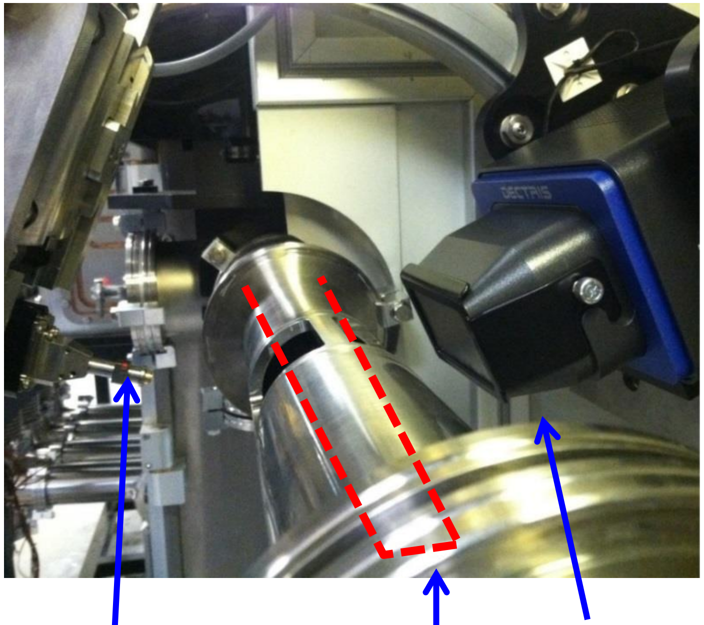




Consistent with TEM:
- Loss of 124 s.f.
- More BZO domains
- REO consumed



Inline Implementation: 2D-XRD

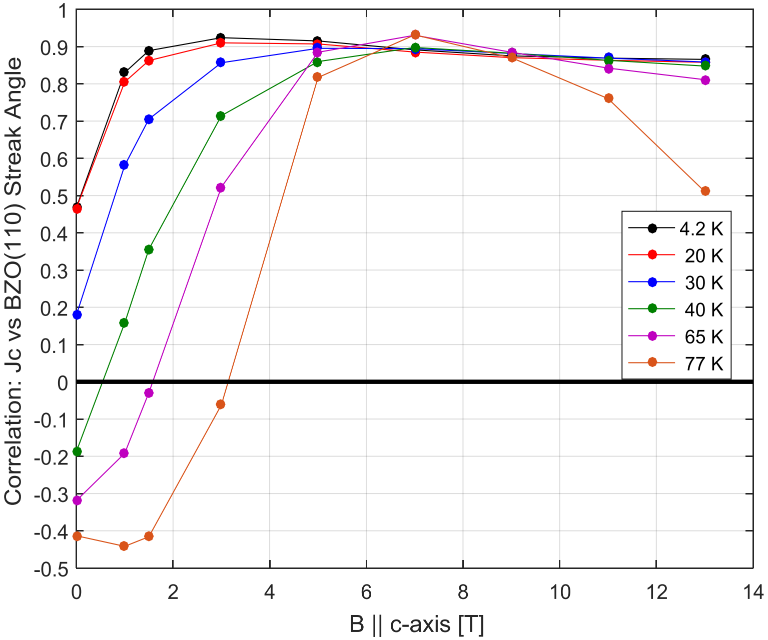


2D Detector

X-ray
Source

Tape path in A-MOCVD

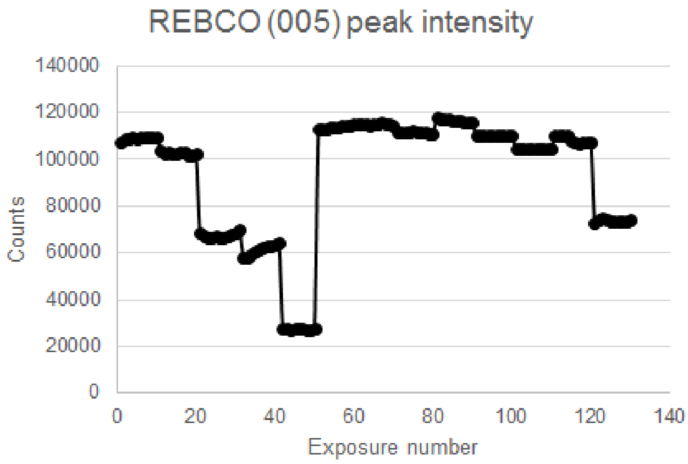
2D-XRD integrated inline with A-MOCVD

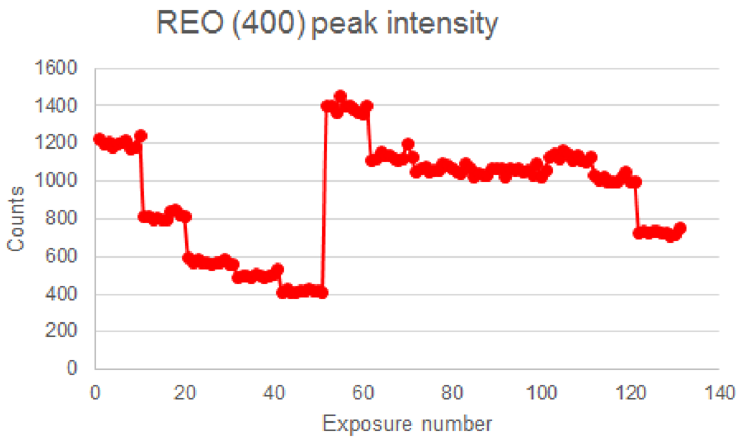


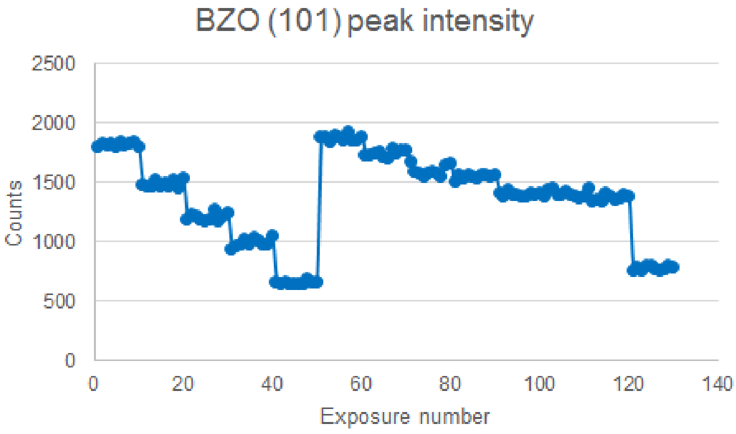
Pearson Correlation – BZO streak angle vs Jc(B,T)

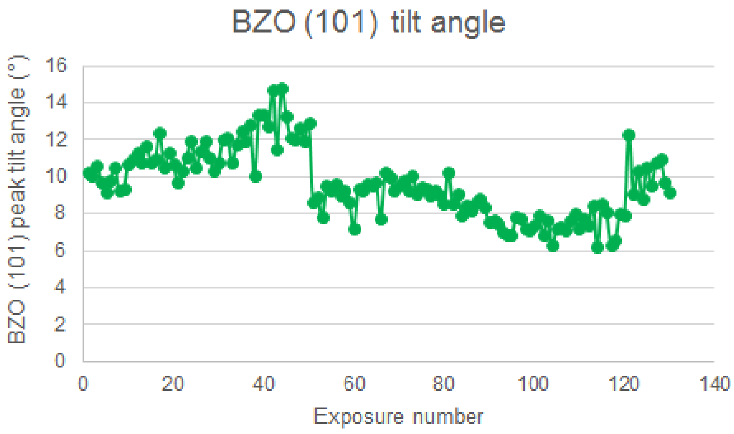
Streak angle correlates well with Jc at lower temperatures:
- At 4.2, 20 K from 2 T
- At 30, 40 K from 5 T

Inline Implementation: 2D-XRD


REBCO (005) peak intensity


REO (400) peak intensity


BZO (101) peak intensity


BZO (101) tilt angle

Continuous inline measurement at feature extraction

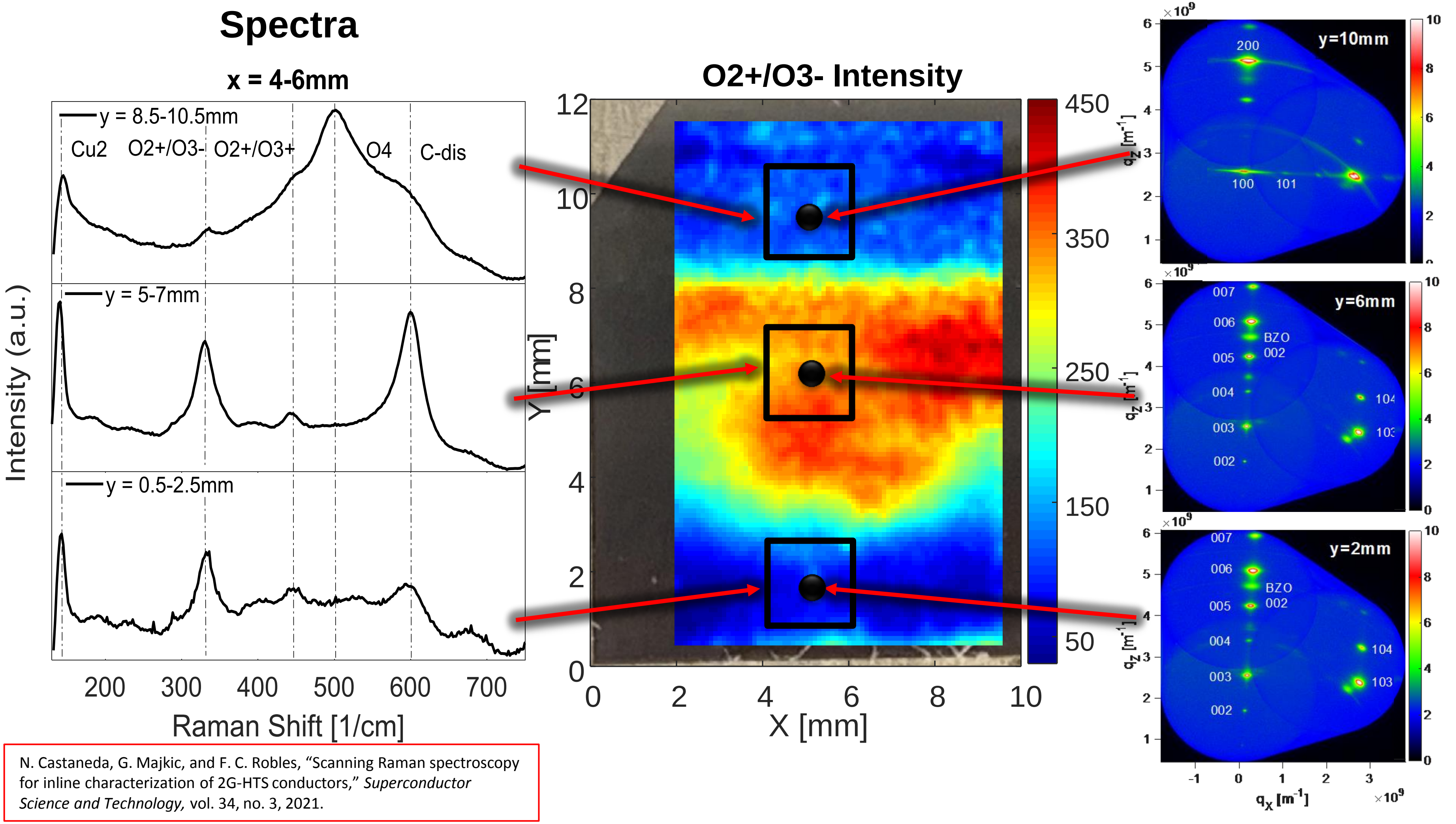
Nondestructive Evaluation:

2.) Scanning Raman

Scanning Raman Capabilities Demonstrated for Defect Detection

Raman Integrated Spectra

2D-XRD

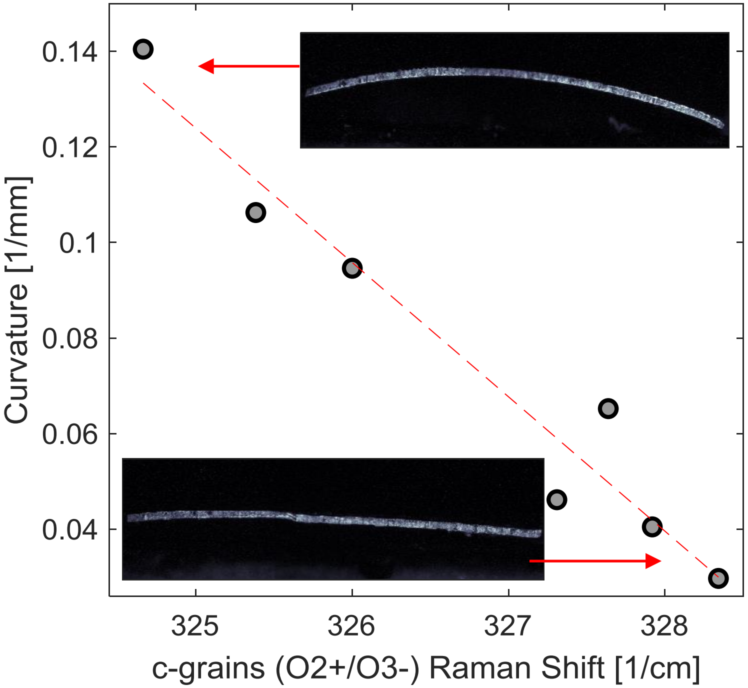


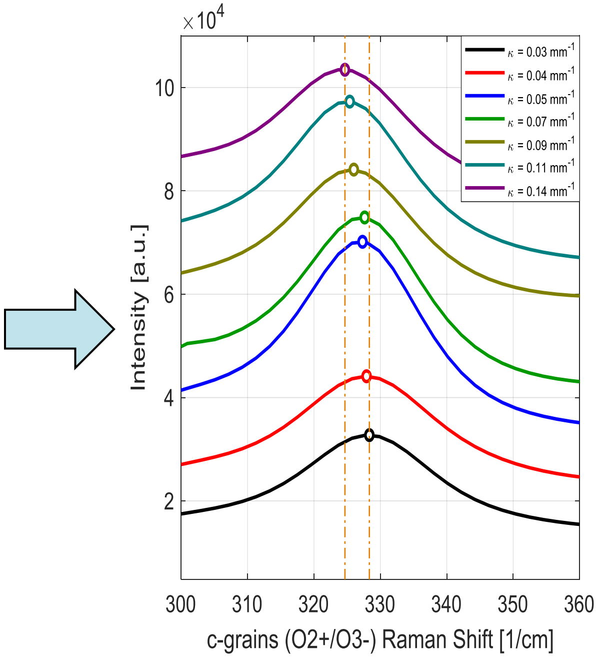
N. Castaneda, G. Majkic, and F. C. Robles, "Scanning Raman spectroscopy for inline characterization of 2G-HTS conductors," *Superconductor Science and Technology,* vol. 34, no. 3, 2021.

NEXT: Can we characterize pinning center density?

Can we characterize pinning center density?

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





Higher nanorod density = higher residual strain = induced curvature

Can be detected via O2/O3- peak shift:

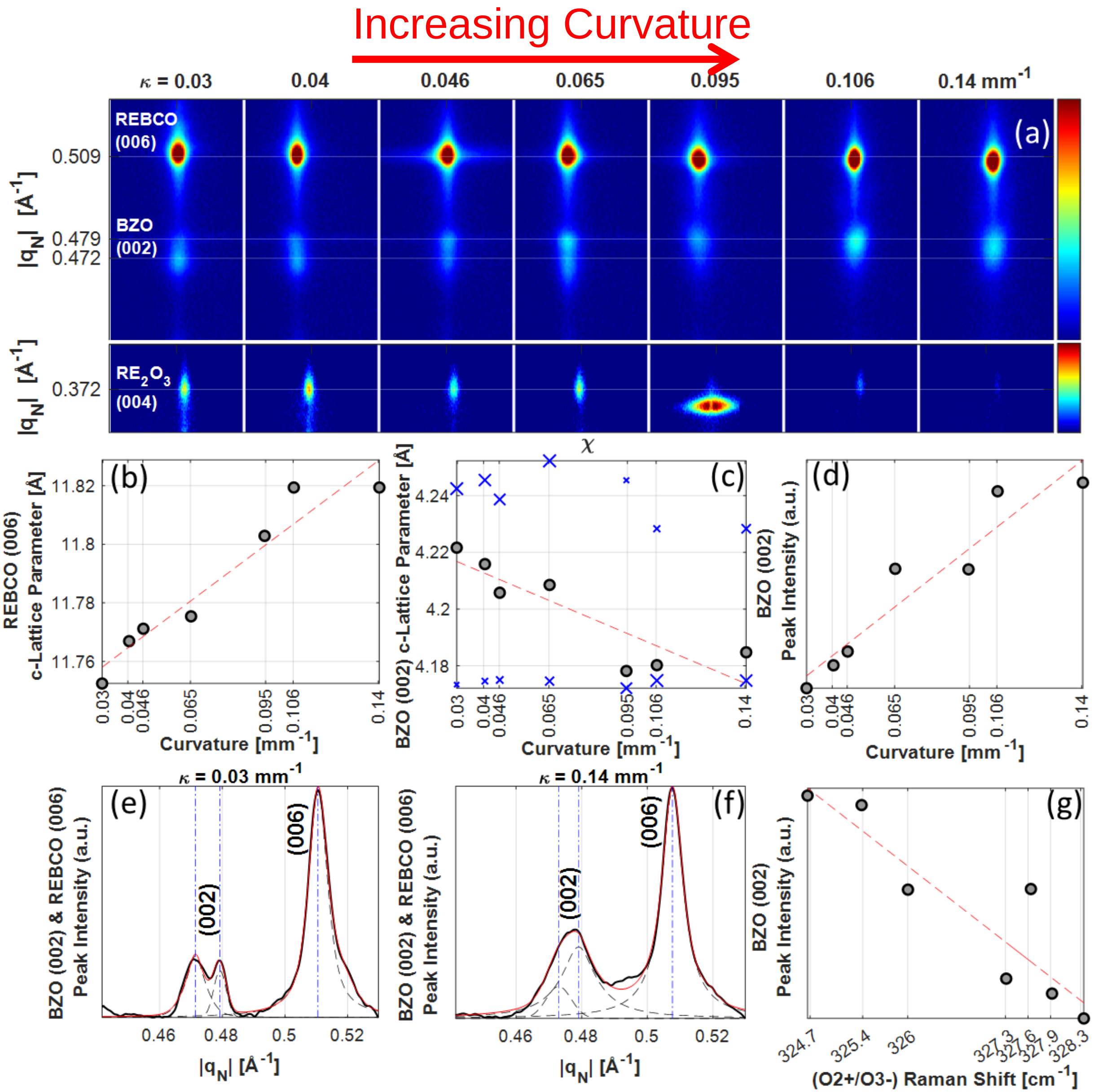
Strong linear correlation between measured curvature and Raman shift

Comparison with 2D-XRD on same samples

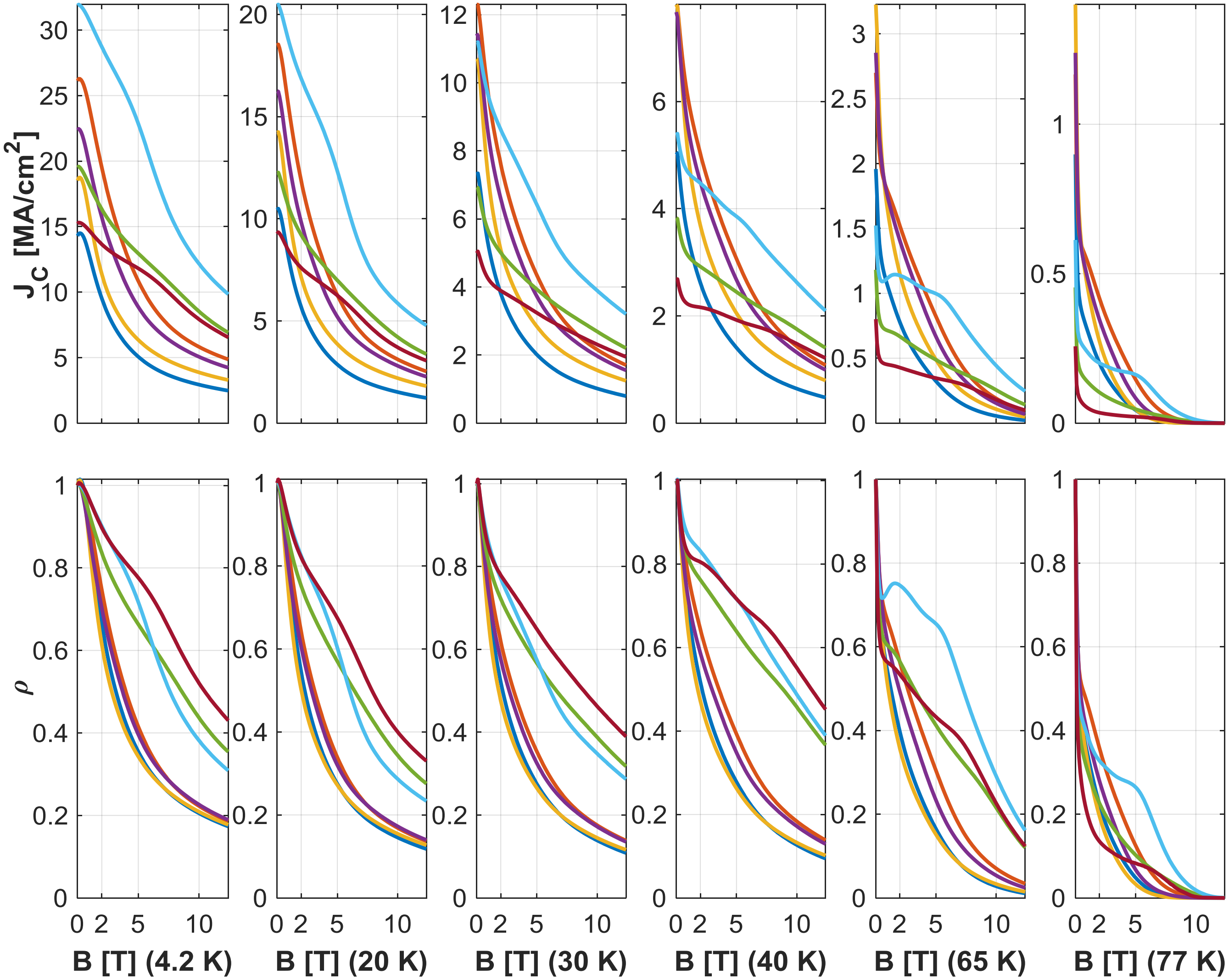
- REBCO c-axis lattice parameter increase with curvature and BZO(002) intensity.
- BZO *c*-axis bimodal, (4.23 and 4.17 Å) – more 4.17 at higher curvature.
- REO intensity decrease with curvature
- Strong linear trend between BZO(002) intensity and Raman 02/03- shift.

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

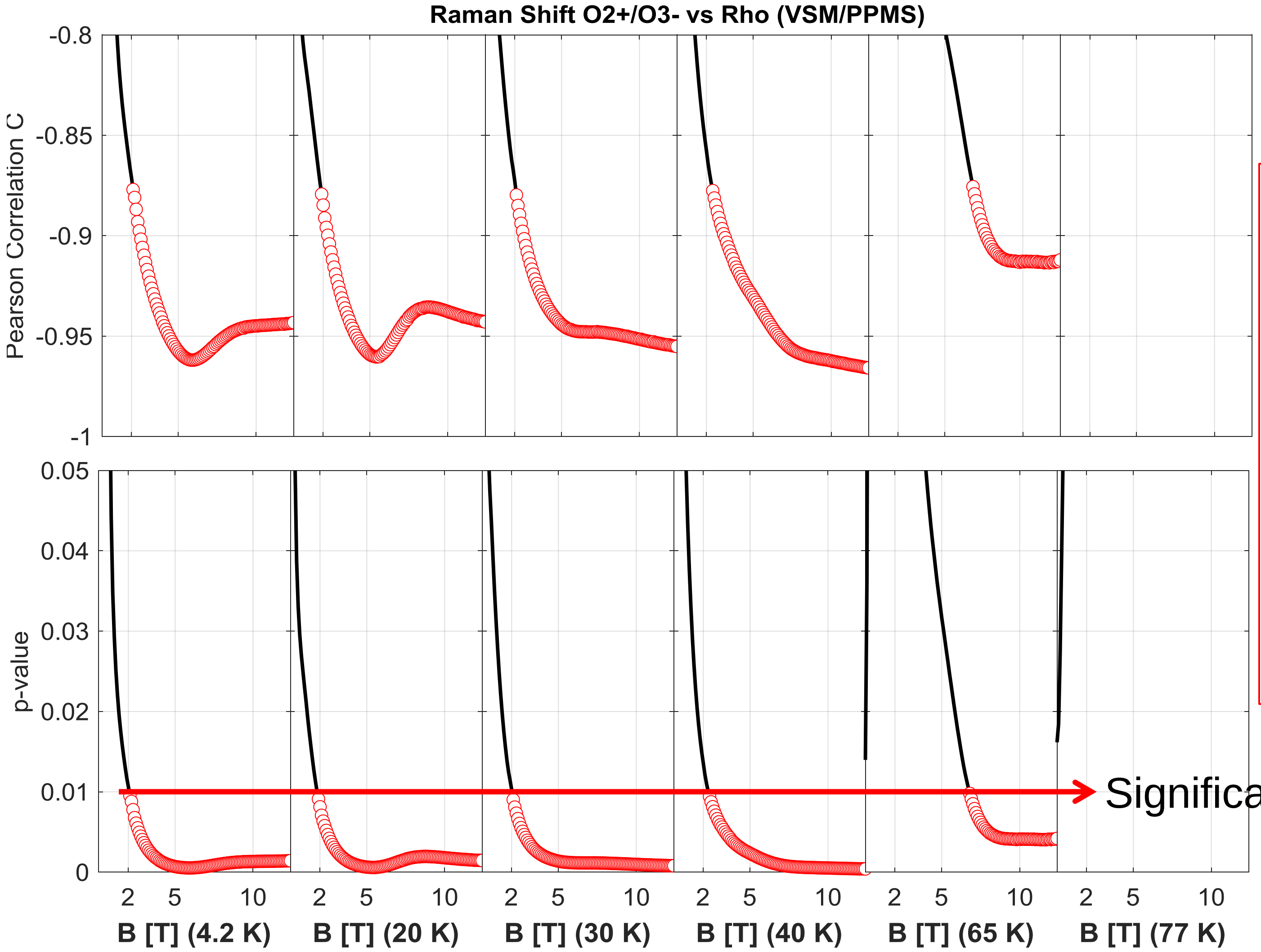
$J_C(B,T)$

Retention Factor

$$\rho(B,T) = \frac{J_C(0,T)}{J_C(B,T)}$$

Both Jc(B,T) and ρ(B,T) vary widely over the selected samples

Can Raman O2/O3- Predict Jc(B,T) and Retention $\rho(B,T)$?



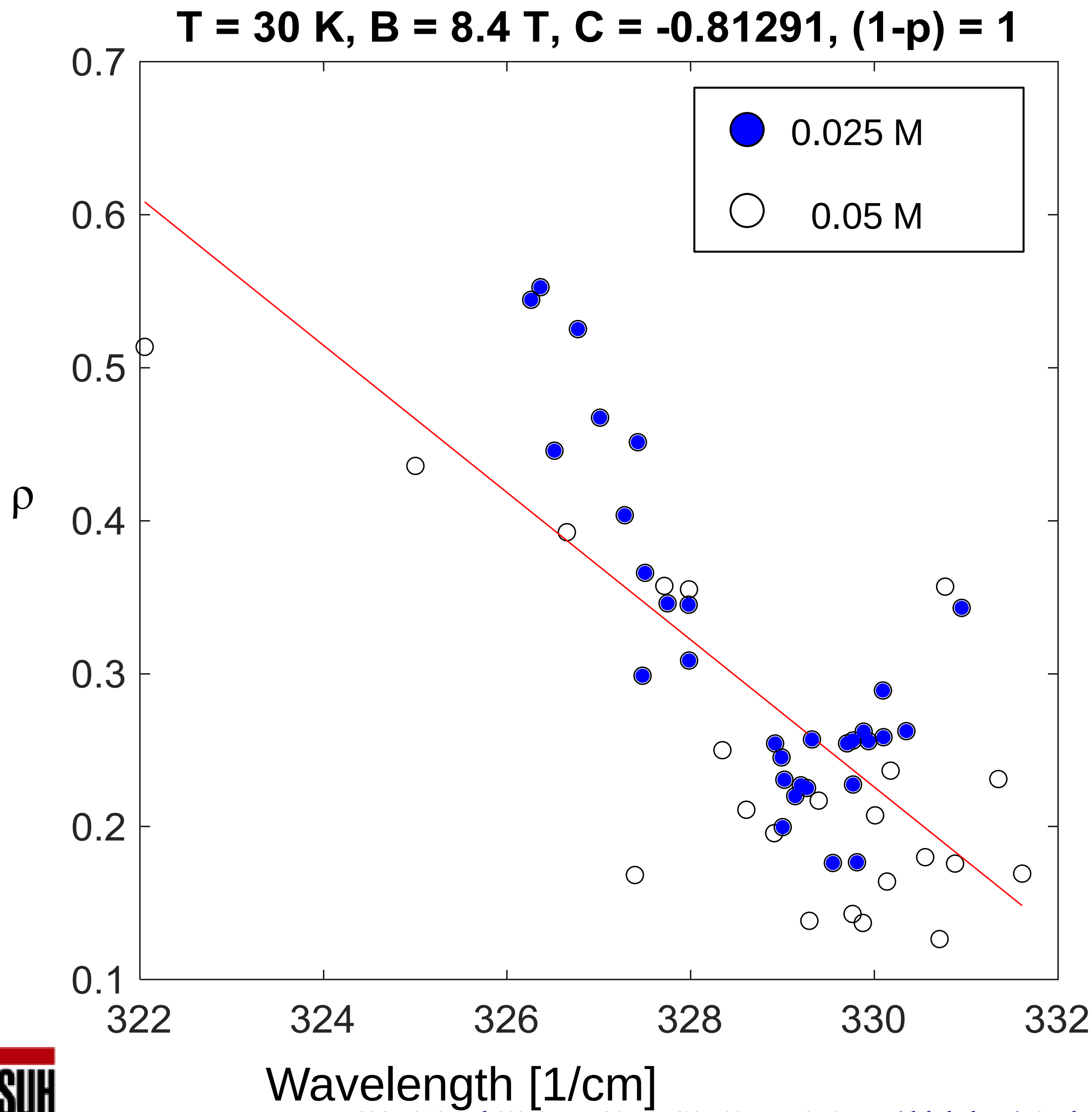
Significance Level: $p<0.01$

For Jc(B,T), no strong correlation

However, for Retention ρ(B,T):

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

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