ReBCO processing techniques and cryogenic measurements to enhance 2G HTS quality

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Presenting: Ken Pfeiffer, V.P. Engineering

CEC-ICMC Conference
July 22, 2019

M1Or3B-02
REBCO Coated Conductors
4:25pm Lvl.6-Rm.14

STI
SUPERCONDUCTOR TECHNOLOGIES INC.

Safe Harbor Provisions

Statements in this presentation regarding our business that are not historical facts are "forward-looking statements" that involve risks and uncertainties. Forward-looking statements are not guarantees of future performance and are inherently subject to uncertainties and other factors, which could cause actual results to differ materially from the forward-looking statements. These factors and uncertainties include, but are not limited to: our limited cash and a history of losses; our need to materially grow our revenues from commercial operations and/or to raise additional capital in the very near future, before cash reserves are depleted, to implement our current business plan and maintain our viability; the performance and use of our equipment to produce wire in accordance with our timetable; overcoming technical challenges in attaining milestones to develop and manufacture commercial lengths of our HTS wire; the possibility of delays in customer evaluation and acceptance of our HTS wire; the limited number of potential customers and customer pressures on the selling prices of our products; the limited number of suppliers for some of our components and our HTS wire; there being no significant backlog from quarter to quarter; our market being characterized by rapidly advancing technology; the impact of competitive products, technologies and pricing; manufacturing capacity constraints and difficulties; the impact of any financing activity on the level of our stock price; the dilutive impact of any issuances of securities to raise capital; the steps required to maintain the listing of our common stock with a U.S. national securities exchange and the impact on the liquidity and trading price of our common stock if we fail to maintain such listing; the cost and uncertainty from compliance with environmental regulations; and local, regional, and national and international economic conditions and events and the impact they may have on us and our customers.

Forward-looking statements can be affected by many other factors, including, those described in the "Business" and "Management's Discussion and Analysis of Financial Condition and Results of Operations" sections of STI's Annual Report on Form 10-K for the year ended December 31, 2018 and in STI's other public filings. These documents are available online at STI's website, www.suptech.com, or through the SEC's website, www.sec.gov. Forward-looking statements are based on information presently available to senior management, and STI has not assumed any duty to update any forward-looking statements.

Topics

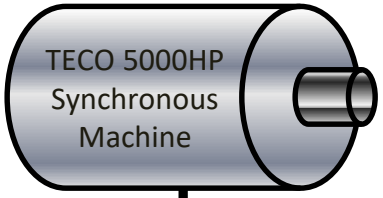
1] Process Control: 2G HTS ReBCO superconductor crystal growth is best accomplished in precisely controlled oxygen partial pressure and at high temperatures (>750°C). STI is working with Fiber Bragg arrays for improved in-situ high temperature process control accuracy & repeatability in large area furnaces to achieve higher yield production and lower costs.

2] Slitting: STI grows ReBCO 2G HTS on foil substrates up to 500+m long x 16mm wide. Some product applications require the superconductors to be as thin as 1mm, like discrete wires. We'll review slitting effects on superconducting wire quality and our findings on different processing techniques used to-date.

3] Metrology: STI is working on cryogenic sensor technology for an inline superconductor measurement machine for characterization of 65K, 0.25T, all-angles & will report on sensor progress.

D.O.E. Program: Ic & Yield Enhancements for 2G HTS

Goal 2: Improve 2G HTS Manufacturing Yield



Goal 1:
Improve 2G HTS In-Field Performance for Electric Machines

1440 A/cm @ 65K for 1.5 Tesla in Rotor

Motor Coil Design

Elect & Mech Specs, Test Criteria

In-Situ Feedback Instrumentation & Control

Reel-to-Reel Ic(B, T, Angle) Test System

2G HTS Improvement Runs

- Increase Thickness (2→7 µm)
- Add (0-15%) Dopants
- Superlattice Insertions mid-plane
- Combination(s)

65Kelvin 0.25 Tesla In-Field @ All Angles Ic/Jc measurements

Thin Film Metrology & Analyses

Fabricate & Test BEST 2G HTS Wire
Multiple 150+ meter runs

Mfg. Yield Analyses

Wind (1)-Pole Motor Coil
Fabricate Cryostat & Test Fixture

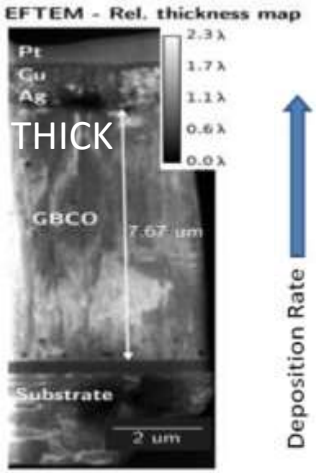
Test & Report FINAL PERFORMANCE

D.O.E. Program: Ic (Amps/cm) Enhancement Technical Approach

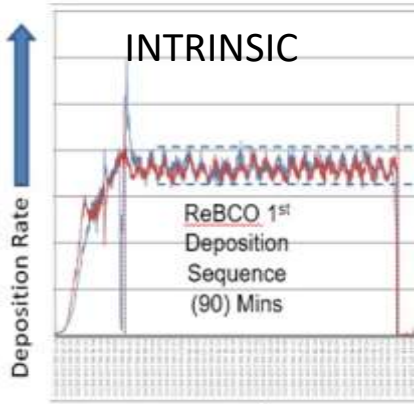
(4) Levers to enhance HTS Cost/Performance Ratio:

1. Ic Lift vs. Temperature (composition control)
2. Increase Pinning forces w/ minimal lattice distortion (optimization for 1.5T/65K)
3. Increase Mfg. Yields (process control - run-to-run yield enhancement)
4. Combined enhancement of 2G HTS: $Re_1Ba_2Cu_3O_{7-d}$
   a. Increased Film Thickness (2→7 μm)
   b. Intrinsic Pinning (gradient concentrations of Re/Ba/Cu atoms)
   c. Extrinsic Pinning (add dopants; Zr, Hf, others)
   d. Insertion of Superlattices

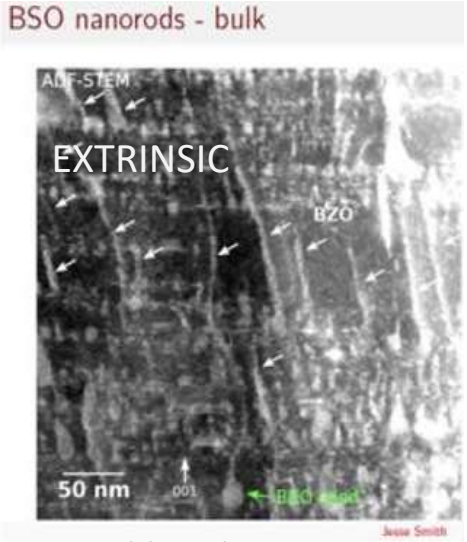
a. X-section Area
b. Diffusion
c. Column Defects
d. Planes



a. Increased Area (μm²)



b. Controlled Gradients Re/Ba/Cu elements

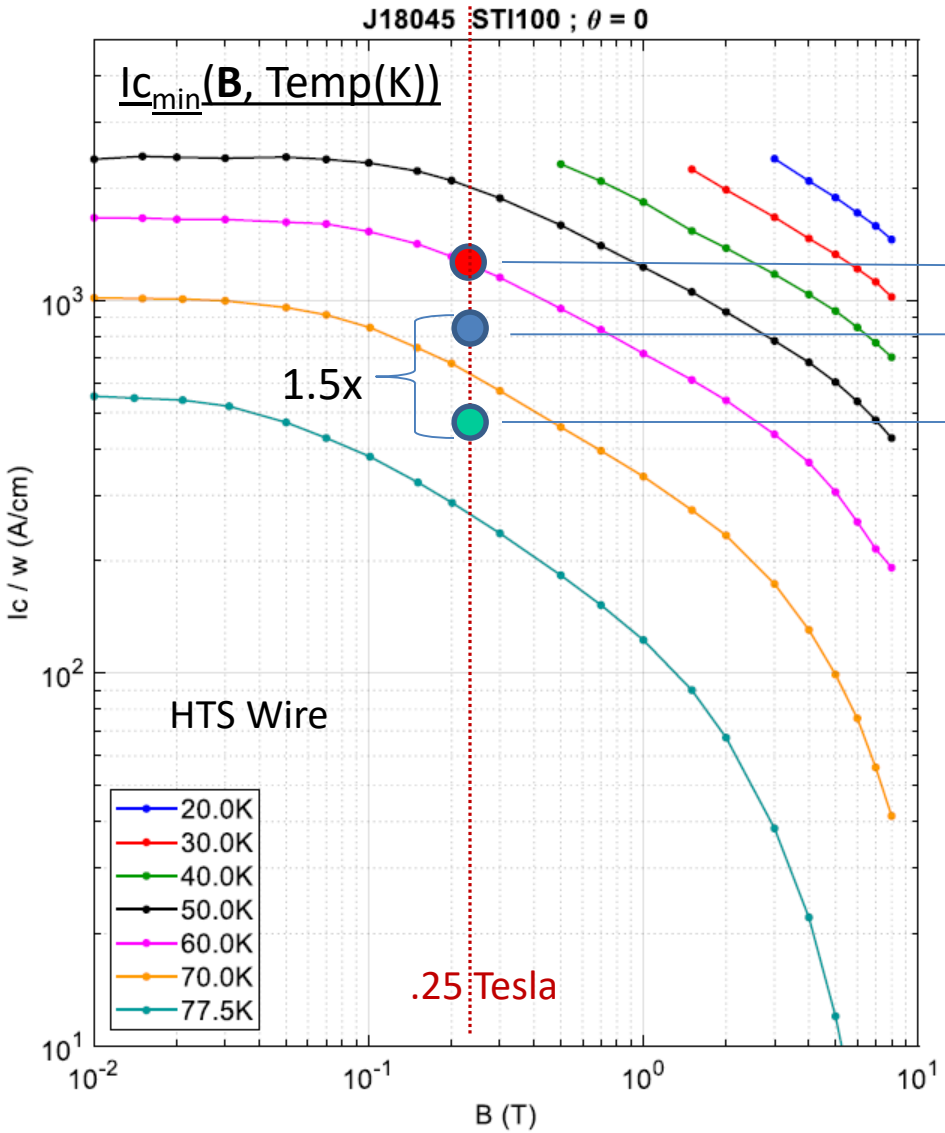


c. Adding dopants

d. Adding 'reset' layers for fixed pinning planes

1st year tried Each 'type' independently to see effect

D.O.E. Program Accomplishments



Data by Dr. Nick Strickland, Robinson Research Inst., NZ

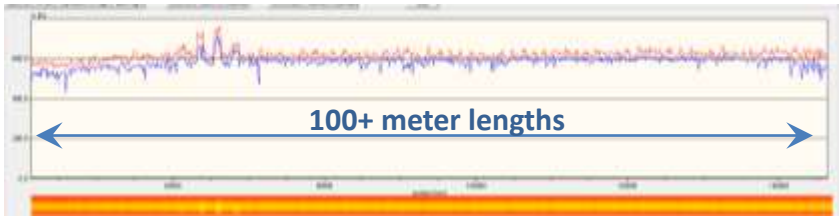
1440 A/cm @ 65K,.25T, All < — GOAL: Year#3 End

850 A/cm @ 65K, .25T, All < — NOW: Year#1 Ended w/ Intrinsic Pinning Only

585 A/cm @ 65K, .25T All < — START: of Program

LENGTH: Year#1 (2x) 15m lengths
Year#2 167+m lengths

10x



Mfg. Yield: Fiber Bragg Thermocouple Arrays

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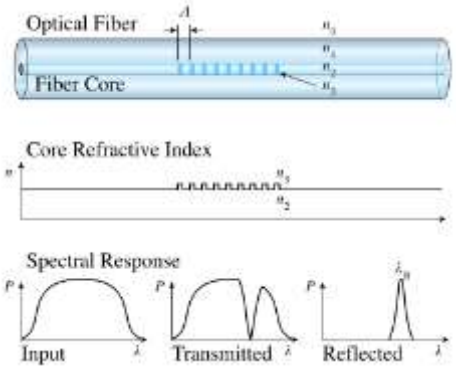
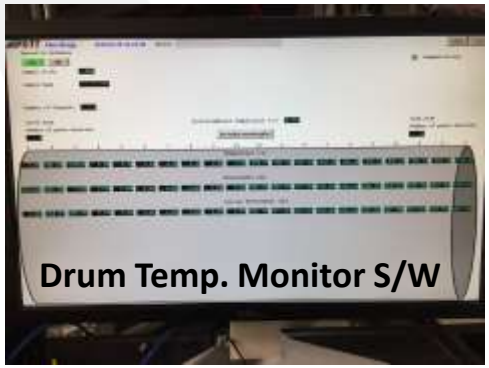
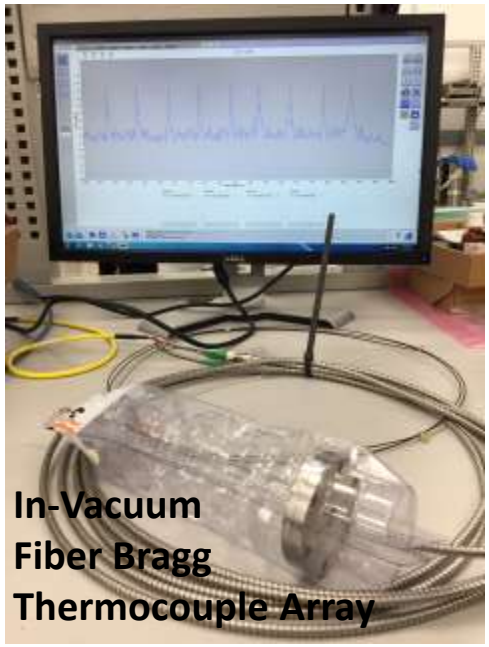


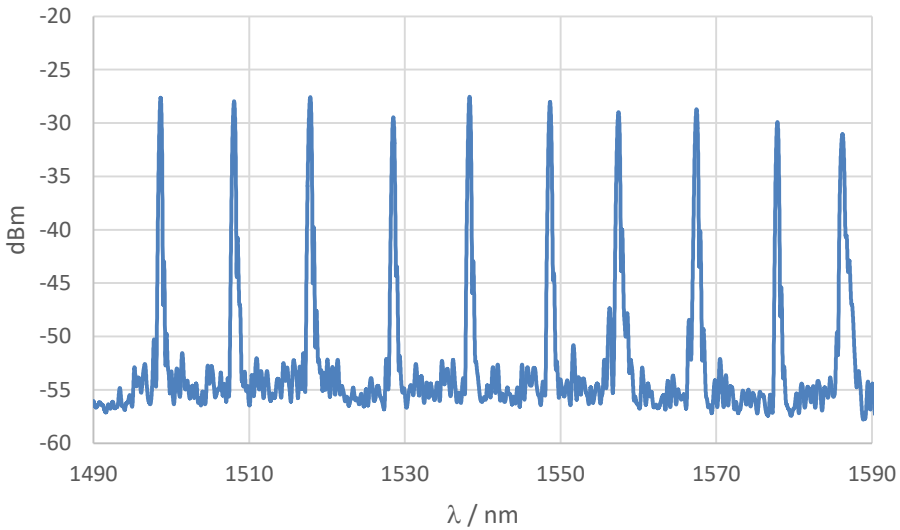


- (5-10x) wavelength varying Bragg dielectric mirrors laser scribed into each Silica fiber core.
- Silica Fiber Bragg array fiber inserted into Inconel vacuum-sealed sheath.
- Std. OEM Fiber Interrogator used to monitor temperature induced wavelength shifts.
  -Calibrated $\Delta\lambda/\Delta T$-



*REF: https://upload.wikimedia.org/wikipedia/en/thumb/d/d1/Fiber_Bragg_Grating-en.svg/400px-Fiber_Bragg_Grating-en.svg.png

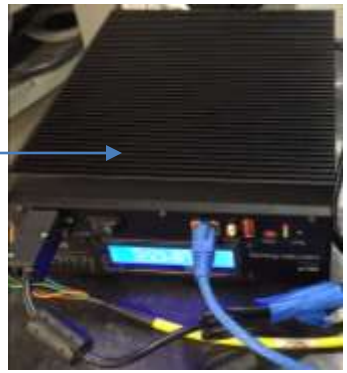
Mfg. Yield: Fiber Bragg Thermocouple Arrays

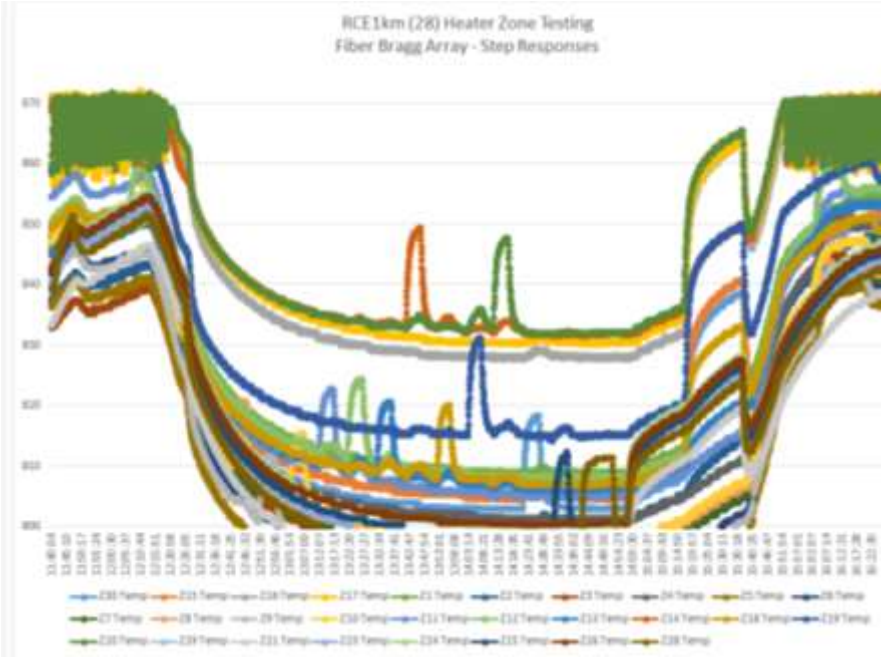


$$T(\lambda) = c_3 \cdot \left[\frac{(\lambda - \lambda_{Ref})}{\lambda_{Ref}}\right]^3 + c_2 \cdot \left[\frac{(\lambda - \lambda_{Ref})}{\lambda_{Ref}}\right]^2 + c_1 \cdot \frac{(\lambda - \lambda_{Ref})}{\lambda_{Ref}} + c_0$$

T – Temperature [°C]; λ – measured Wavelength [nm]

1500-1600nm Optical Fiber Interrogator





Step-Response Testing – All (28) Htr. Zones

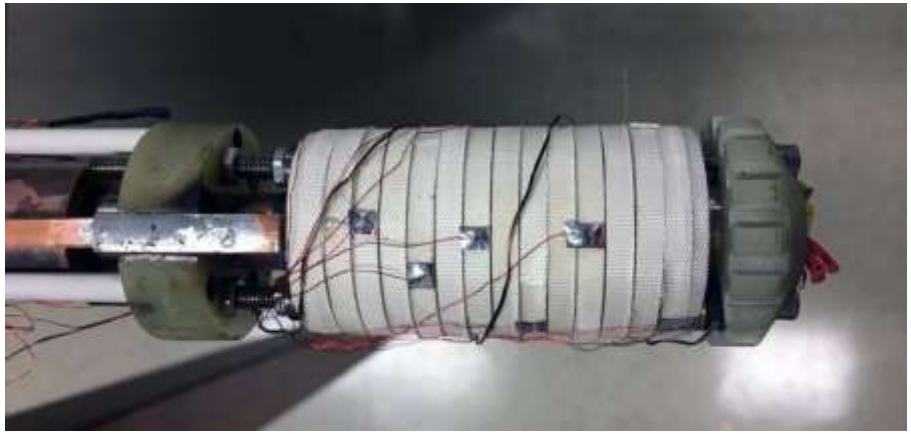
-Determination of 'cross-talk' for heater zones

-Compared fiber Bragg step responses to thermocouple responses during testing. Offsets

-Validated sensitivity to changing drum emissivity during coating processes.

-Long-term reliability a concern…(1) failure!

Improved Slitting of 2G HTS ReBCO: Inspired by NHMFL



6/2019

Congratulations to NHMFL team on their 45.5 Telsa ReBCO record field coil insert using 4mm 2G HTS!

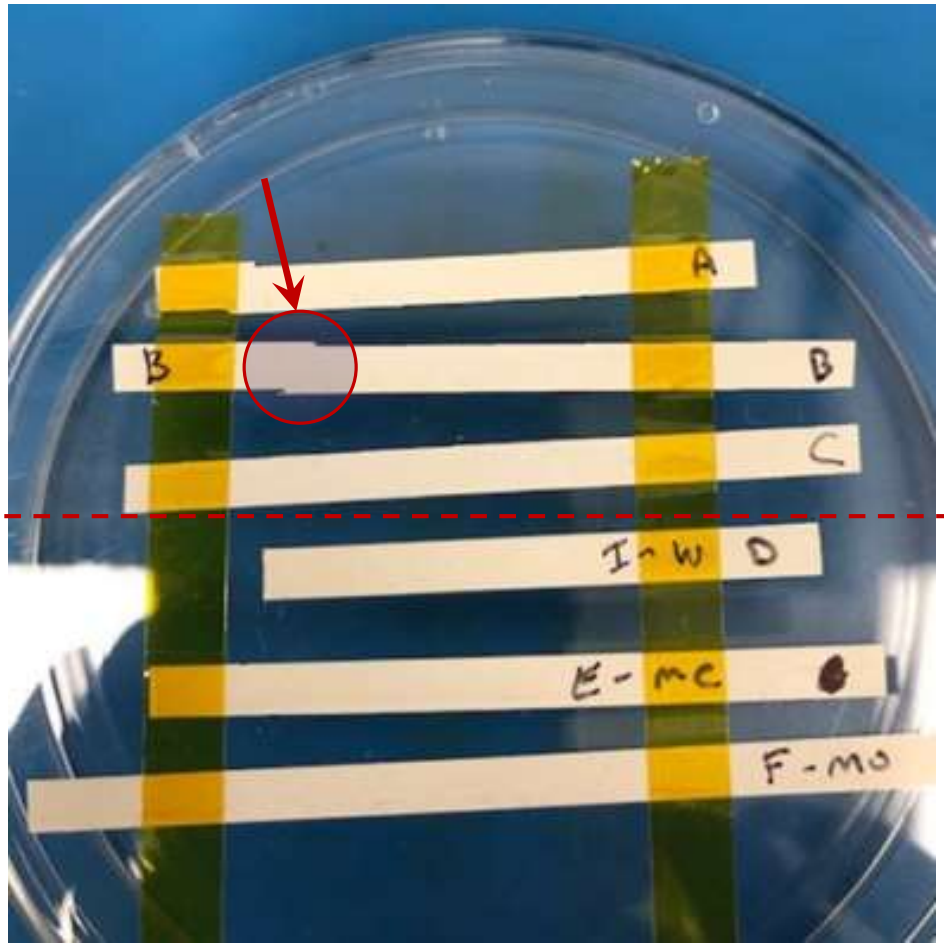
NHMFL Press Release*:

To meet the LBC's requirements, however, those tapes had to be cut lengthwise to 4 mm wide. That's quite difficult to do, even with the greatest care, because REBCO is quite brittle. As a result, the sides of the tape that had been slit were vulnerable to cracking under the mechanical stress of high magnetic fields.

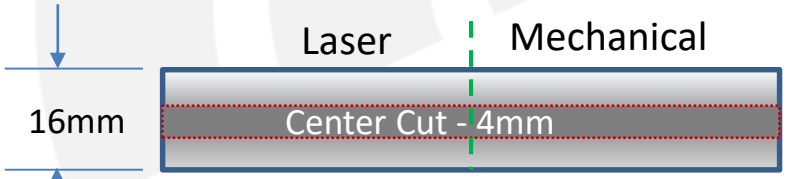
"That was discovered beautifully in these experiments," said Larbalestier. "We found a way to control this damage, which is to insist that we buy material that has one non-slit edge, and we orient the non-slit edge *away* from the center of the magnet. And under these circumstances, so far we are not seeing damage."

* REF: https://www.scienceandtechnologyresearchnews.com/with-mini-magnet-national-maglab-creates-world-record-magnetic-field/

Slitting Sample Comparison: Laser vs. Mechanical



ALL samples were cut from the same continuous piece of 2G HTS ReBCO tape.

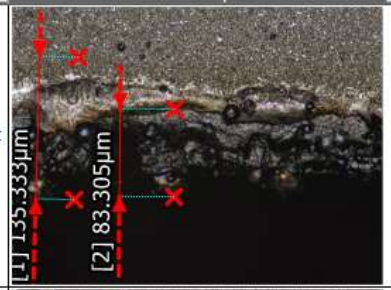
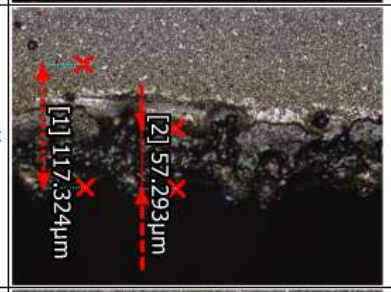
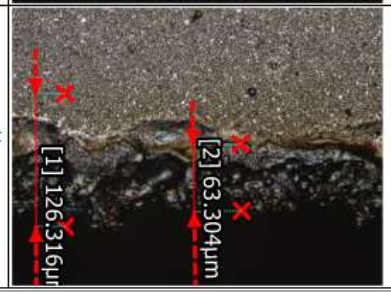


Laser Slit – Thru Backside
Samples A,B,C:
1064nm Fiber-Laser slit from middle of a 16mm wide ReBCO x 65µm thick substrate
Silver-coated

Mechanical Slit – Edges UP
Samples D,E,F:
Mechanical slit from middle of a 16mm wide ReBCO x 65µm thick substrate
Silver-coated

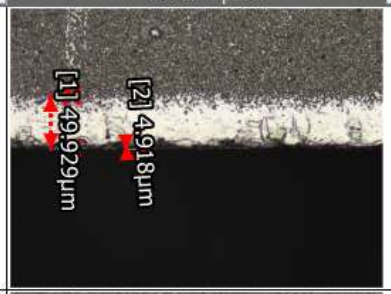
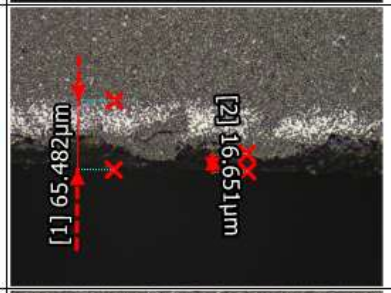
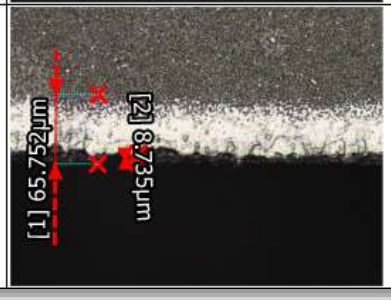
Slitting Sample Edge Data: Laser vs. Mechanical

Laser Slit – slightly more edge area affected

File name	Plane Laser+Optical	Plane Measured values
J19004_Laser_Slit _4mm_C		No. | Measurement name | Measured val 1 | 2 Points1 | 135.3 2 | 2 Points2 | 83.3
J19004_Laser_Slit _4mm_B		No. | Measurement name | Measured val 1 | 2 Points1 | 117.3 2 | 2 Points2 | 57.2
J19004_Laser_Slit _4mm_A		No. | Measurement name | Measured val 1 | 2 Points1 | 126.3 2 | 2 Points2 | 63.3

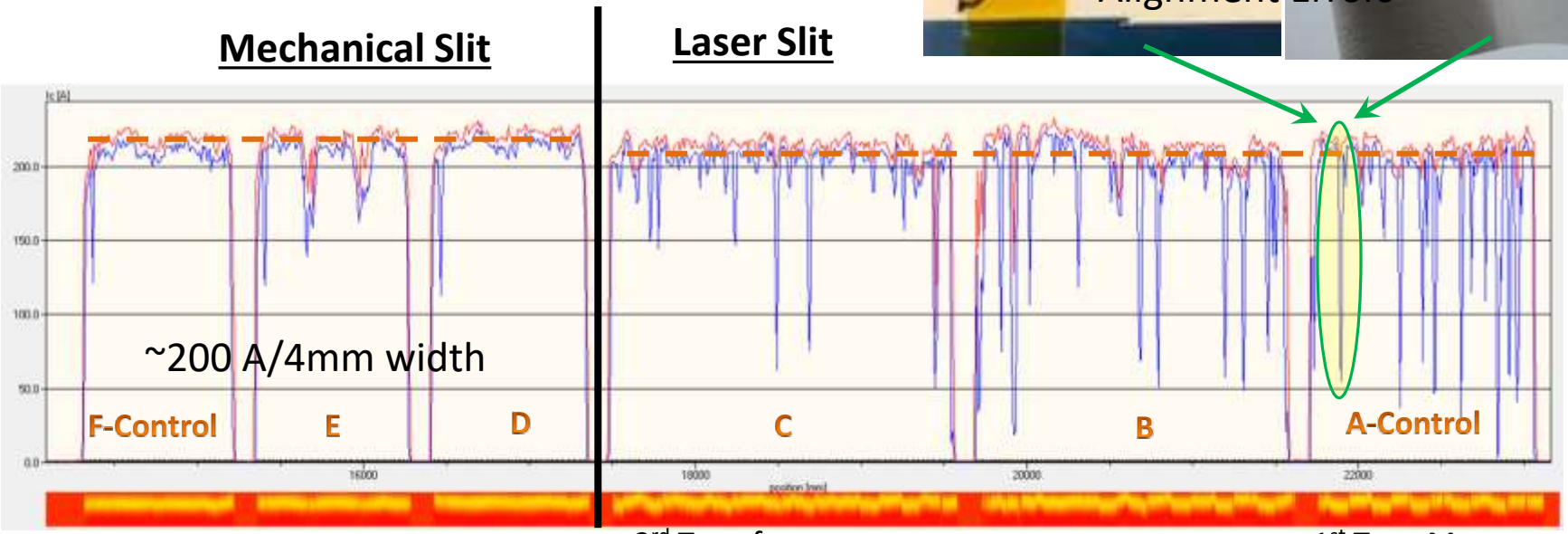
60-80µm Damage/Ag-Melt from Edge
115-135µm Heat Affected Zone (HAZ)

Mechanical Slit

File name	Plane Laser+Optical	Plane Measured values
J19004_Mech_Slit _4mm_F		No. | Measurement name | Measured val 1 | 2 Points1 | 49.9 2 | 2 Points2 | 4.9
J19004_Mech_Slit _4mm_E		No. | Measurement name | Measured val 1 | 2 Points1 | 65.4 2 | 2 Points2 | 16.6
J19004_Mech_Slit _4mm_D		No. | Measurement name | Measured val 1 | 2 Points1 | 65.7 2 | 2 Points2 | 8.7

50-65µm Edge Damage from slit

Ic Data: Laser vs. Mechanical



3rd Try – fewer alignment Errors

Better Alignment
Fewer Dropouts

1st Try – Max. alignment Errors

- Achieved Laser Slit Ic values 95% of Best-Known Mechanical Slitting technique
  - MECHANICAL: ~220 A/4mm
  - LASER: ~210 A/4mm
  - Our best performance to-date on 65µm Hastelloy substrate w/ 3+µm ReBCO
- Laser cutting speeds (~50mm/sec/line)
  - i.e. (5) cut-lines → 10mm/sec
- Need to reduce the Heat Affected Zone
- Need to minimize spot size & foil 'flatness' in cut zone to achieve EQUAL performance.

Slitting Improvements for 2G HTS ReBCO

- STI characterized extent of laser slit edge damage with Optical & Laser confocal microscopes after silver removed via chemical etchant.
  - Could not easily measure edge defect features on laser slit samples past melt-edge
  - TEM/SEM/Other techniques may be useful here?
- Many possible Laser configurations…just started w/ off-the-shelf technology for testing.

<u>Next Steps:</u>

- Complete multiple 1.0m long wrap-tests with statistical Ic degradation analyses to determine effects of Bi-Axial stress and compare to best Mechanical Slits.
  - 1st need to remove alignment induced cracks with improved fixturing.
  - Multiple Batches, multiple film thickness, etc..

<u>STI's Goal</u>: Better processing technology for higher yields & stronger/more-robust 2G HTS.

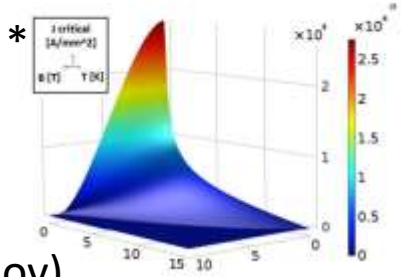
2G HTS Metrology: Speeding-up performance characterization

Investigating ways to characterize the critical surface of 2G HTS by length

- Very High Speed sensing
- Multi-Pass per condition (B, Temp, Field < +/-90°)

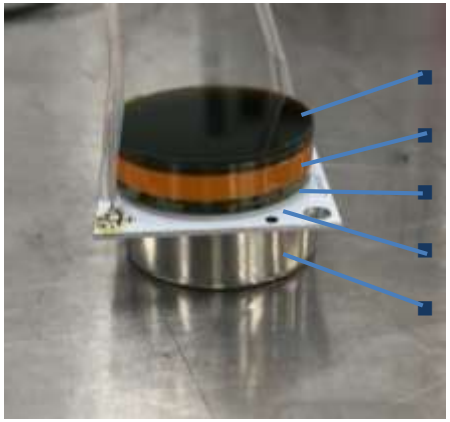
  -or-
- Multi-Condition per fixed position (B, Temp, Field < +/-90°)
- Measures flux expulsion (Meissner Effect) & flux pinning (Abrikosov)
- Works at 77K LN_2 (1 atm.)→65K LN_2(0.2 atm.)
- Self-Field to 1.5+ Tesla , possibly higher

*



Concept: A low-power COB LED array w/ Magneto-Optics 'stack' on top

- <50milliWatts power dissipation in LN_2. [36V/.001 Amps]
- >=25mmΦ illumination area for 16mm wide 2G HTS measurements



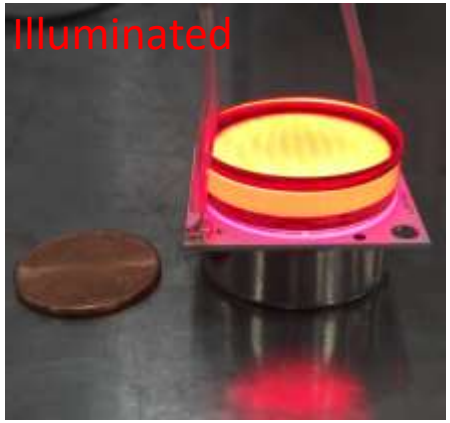
- Top Polarizer
- 2.5mm Magneto-Optic crystal
- Bottom Polarizer
- LED Array
- NdFeB Permanent Magnet

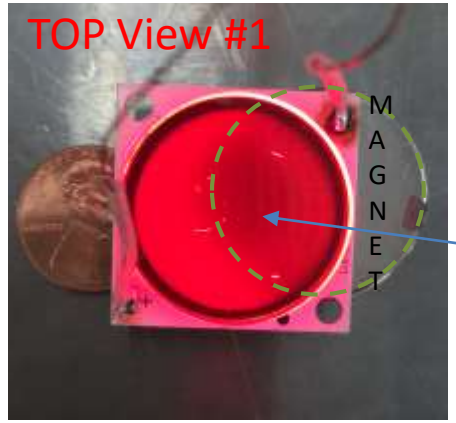
Optical stack cost <$1k

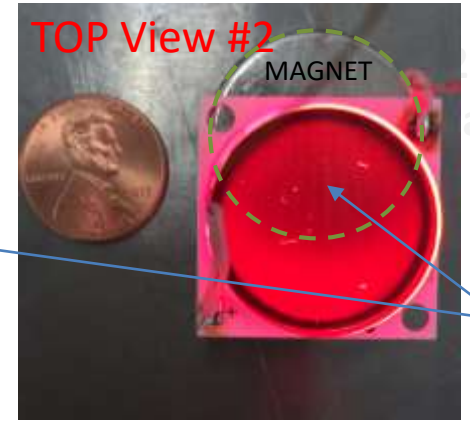
(1)-meter Length

*Ref: https://cdn.comsol.com/wordpress/2017/03/critical-surface-plot.png

2G HTS Metrology



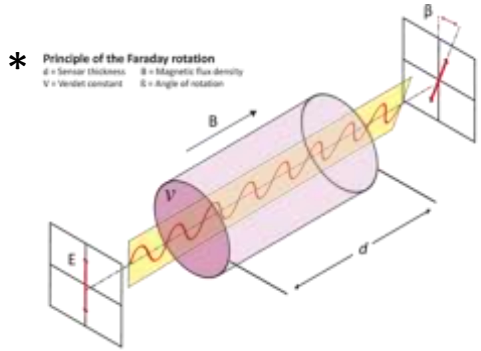




Room Temp
In-Air Testing

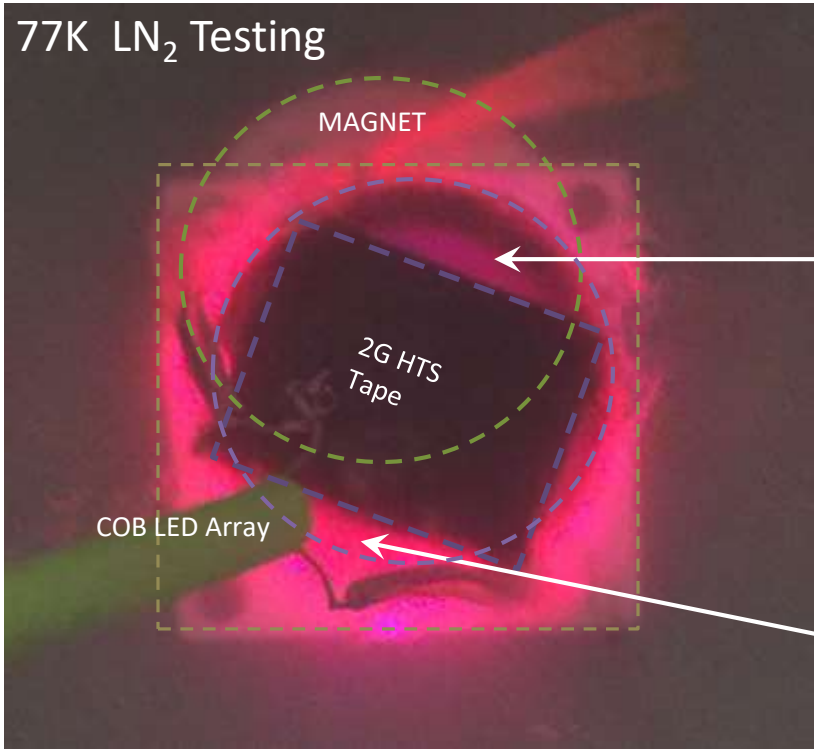
B-Field
'shadow'
visible

- Verdet constant increases with lower wavelengths & lower operating temp's.
- Polarization rotation increases with higher Verdet constant & higher **B**-fields.
- - Should work better @(77K) cryogenic temps & 0.5 to 2 Tesla.
- COB LED array has performed well with >50x LN_2 immersions → Room Temp.
- NdFeB Permanent magnet field (~0.3-0.5) Tesla, in region-of-interest
- Height Magnet to M.O. crystal = <3.0mm, small air gap/retain high flux density



*REF: https://matesy.de/en/products/materials/mo-sensors-magnetooptics

2G HTS Metrology



22mm Diameter

<u>Several possible modes of operation:</u>
Backlit Illumination Mode

Magnet Side:
Flux expulsion region @ edge of tape is clearly illuminated

Non-Magnet Side:
No 'shadow' or 'ring' visible

<u>Next Steps:</u>
-CCD camera & Image processing software for data collection
-Determine Statistical correlation to D.C. Ic values
-Area & Light intensity-

For information on Superconductor Technologies' Conductus[tm]
2G HTS wire please contact;
Adam Shelton , [E]: ashelton@suptech.com

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