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Property Variations in Modern REBCO Coated Conductors from Multiple Manufacturers

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Abstract. The complex, multilayer structure of REBCO Coated Conductor (CC) poses significant challenges in the fabrication of high magnetic field devices where large stresses may initiate various forms of damage. Our goal is to peer below the cartoon representations of CC so that, amongst other things, we might better understand whether a CC from one manufacturer is interchangeable with that from another. This involves knowledge of a broad range of electromagnetic, geometric, microstructural, and $J_c(\theta, B, T)$ properties, and their variations that collectively pose challenges for the fault tolerance of REBCO CC devices. Accordingly, comparative measurements of J_c, visualization of flux penetration with Magneto-Optical Imaging (MOI), tape geometry from Scanning Electron Microscopy (SEM) of polished cross-sections, and extensive optical microscopy was performed on recently purchased samples from multiple manufacturers. Our analyses reveal many deviations from or characteristics absent from manufacturers' specifications, while a comparison of different manufacturers' mechanically and laser slit tapes shows a diverse array of slitting characteristics amongst the manufacturers and variation in properties those made to the same specification. Laser slit tapes from several manufacturers reveal ablated edges with damaged regions extending up to 50 μm, comparable to the damaged region found in the mechanically slit CC of this study. Overall, the aim of this study is to flesh out appropriate ways to understand the real conductor below the manufacturers' cartoons to avoid surprises in our REBCO CC coil development program. The goal of this work was to perform a broad array of characterizations of the type needed for validation of purpose for making high field magnets: to our surprise we found a wide range of properties which greatly impact the mechanical strength and electromagnetic performance of solenoids composed of these conductors and reinforced the need for a broad characterization program for each conductor prior to its implementation.

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1. Motivations

Rare-Earth Barium Copper Oxide (REBCO) Coated Conductor (CC) has significant potential to enable new frontiers of superconducting materials research and applications due to its substantial critical temperature (T_c), high critical current density (J_c), and high upper critical field (H_{c2}), particularly at lower temperatures. Despite REBCO's attractive qualities, significant challenges arise from its implementation as a rectangular cross-section tape with a hetero-epitaxial structure quite different from the multifilament stabilization and reinforcement schemes characteristic of (generally round) Low Temperature Superconductors (LTS). The Applied Superconductivity Center at the National High Magnetic Field Laboratory is actively investigating[1] these challenges with REBCO coil technology, one way being through the Little Big Coil (LBC) program which aims to generate record high magnetic fields in a 31 T resistive magnet background using No-Insulation (NI) double-pancake (DP) windings. To enable further progress above the present 45.5 T total field record of LBC3 and to better understand the REBCO CC we wind into magnets, extensive characterization of the tape, both before and after usage, is performed to understand the electromagnetic, mechanical, microstructural, and geometrical constraints and challenges of the tape. An important feature that we consider during the winding of our magnets is the slit-edge of the REBCO CC; REBCO CC is generally made 12 mm wide and then "slit" into narrower pieces of varying size. For most of REBCO's history, mechanical slitting of the CC was the primary method of cutting the tape[2]. However, as REBCO is a brittle ceramic, mechanical slitting results in micro-cracks and delamination at the edge which act as stress-concentrations. We have also observed that slitting can produce quasi-periodic protrusions where Hastelloy is deformed rather than cut on the cutter-opposed face. To avoid these undesirable features, many companies are investigating or switching to laser slitting of the CC. In principle this is a good development, as laser slitting should allow for greater reproducibility and cutting precision and mitigation of the defects mentioned above. Here we show that commercial examples of laser-cut tapes, mostly but not all from production runs, have a wide range of properties: some have trapezoidal cross-sections, quasi-periodic damage, and in some cases, entirely melted edges mixing the Hastelloy substrate with the oxide and Ag layers. In some cases the non-square nature of the edges is very marked. In addition to slitting imperfections, irregularities in delamination strength, critical current density variations, and other property fluctuations are observed in the modern REBCO CC we are evaluating for our LBC program. We believe that such "real" features of the conductor must be taken account of to accurately model and understand our solenoids. To reproducibly use REBCO CC to make compact solenoids, expand our choice of usable conductor, and address the myriad of challenges and imperfections mentioned thus far, REBCO CC from Fujikura, Shanghai, SuperOX, SUNAM, SuperPower, and THEVA were purchased and characterized with the aim to make several small test coils from each manufacturer and compare their performance. Each manufacturer's tapes revealed different $I_c(\Theta,B,T)$ properties, different pinning nanostructures, and different quirks associated with the specific synthesis and slitting combinations for each tape, with differences also arising in tapes made to the same specification from the same manufacturer. Overall, an investigation into modern REBCO CC's electromagnetic behavior, geometrical regularity, slit quality, and its ability to be utilized in a compact solenoid was investigated for several tapes from several manufacturers and a wide variety of behavior and quirks were observed as are further detailed below.

A key simple summary of this not yet completed work is that no coated conductor is its cartoon and that proper characterization may take much more effort than has so far been expended in the vast majority of cases.

2. Methods

The tapes of this study were subjected to several different characterizations available at ASC to understand their properties and the main conclusions of this paper mainly stem from: microscopy in the forms of Scanning Electron Microscopy (SEM), plane-view Magneto-Optical Imaging (MOI), and electromagnetic characterization utilizing torque magnetometry.

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2.1. Scanning Electron Microscopy

SEM was performed on the Helios SEM at the NHMFL. Every sample was visualized in two different orientations: plan-view SEM begins with removal of the protective copper and silver layers so as to allow direct imaging of the REBCO surface. Removal of these layers is described in several papers[3], and our specific procedure is: Submerge virgin REBCO CC in Ammonium Persulfate (APS-100, a copper etchant) at 60 °C, wait until copper has been completely etched, then expose the silver layer to a mixture of Water (70%), Hydrogen Peroxide (15%), and Ammonia Hydroxide (15%) and wait for complete removal of silver, then, rinse in acetone and dry in nitrogen gas, then begin imaging. Etched plan-view samples were attached to the sample stage/SEM ground via copper tape with double-sided conductive adhesive. Samples were then imaged in bright-field mode to assess the microstructure (grain morphology, impurities, etc.), edge damage from slitting, with some Energy Dispersive Spectroscopy (EDS) to assess elemental composition of anything interesting on the surface. Following plane-view SEM, a polished cross-section of each tape is prepared either through mechanical polishing or ion milling. From this cross-sectional view, the component layer thicknesses are easily obtained and any peculiarities resulting from the slitting process are readily seen on the edges of each tape. EDS is once again performed on anything interesting in the cross-section, such as an entirely melted agglomeration of material resulting from laser slitting.

2.2. Magneto-Optical Imaging

Magneto-Optical Imaging has long been known[4] to be an excellent method of visualizing variation of superconducting parameters on the micro-scale and it has proven especially useful in the analysis of damage done by slitting. A 1 cm long piece of REBCO CC is exposed to cryogenic temperatures in magnetic fields up T while in close proximity to an indicator film (Gadolinium Gallium Garnet) which is then imaged utilizing a conventional camera and polarized light and then digitally analyzed on a computer, revealing where magnetic flux leaks through the "screening" provided by the REBCO CC. Numerous defects which are otherwise imperceptible in SEM or other methods were readily visualized using MOI in this study.

2.3. Electromagnetic Characterizations

The main purpose of the electromagnetic characterizations is to obtain the constitutive $J_c(B,T,\theta)$ relation for each conductor so that we may calculate the I_c limitations present in our solenoids and predict a safe operational range. Transport and magnetization methods are both utilized in this endeavor to address the shortcomings of the other; rotating high-ampacity electrical connections in high magnetic fields are technologically challenging for transport methods whereas magnetization methods have numerous sources of error which are difficult to account for.

2.3.1. 4-Point I-V Curve. The 4-point transport method (measuring voltage and currently independently) is utilized to run current-voltage (I-V) curves of the CC at various environmental conditions. Typically, this is performed in a bath of liquid helium within a 15 T superconducting magnet [5] with currents ranging up to 1400 A. Similar measurements are performed in a bath of LN_2 in fields up to 1 T. A field programmable gate array (FPGA) from National Instruments is utilized to control the Sorenson power supplies and measure the voltages of the superconducting sample and prevent destructive transitioning.

2.3.2. Torque Magnetometry. Utilizing the Lorentz force generated by screening currents resulting from the large filament size of REBCO CC, the torque magnetometer uses a force sensor to measure the tension of fishing line attached to a rotating platform with an ~13 mm long sample of REBCO CC generating a torque as it is rotated ~180° in background magnetic fields up to 31 T. The magnetometer can vary the sample temperature between 4.2 – 50 K and can capture the entire $I_c\big(B(\theta)\big)$ for a given magnetic field in a matter of minutes, a notoriously difficult task near $B \parallel ab$ for transport methods[6].

2.3.3. T_c via SQUID. T 6 x 4 mm lengths of CC were zero-field cooled to ~40 K in a Quantum Design 5.5 T SQUID magnetometer, then warmed in an applied field of 1 mT then warmed to ~120 K. The distribution of the derived critical temperature and the sharpness of the transition were used to compare the various coated conductors and to assess the effects of heating, slitting, and other damage.

3. Results

3.1. Laser Slitting Edge Damage in SEM

SEM of the laser slit tapes yielded an interesting view of the state-of-the-art REBCO cutting methods, mainly revealing melting and geometrical abnormalities in regions comparable in size to the cracks resulting from mechanical slitting.

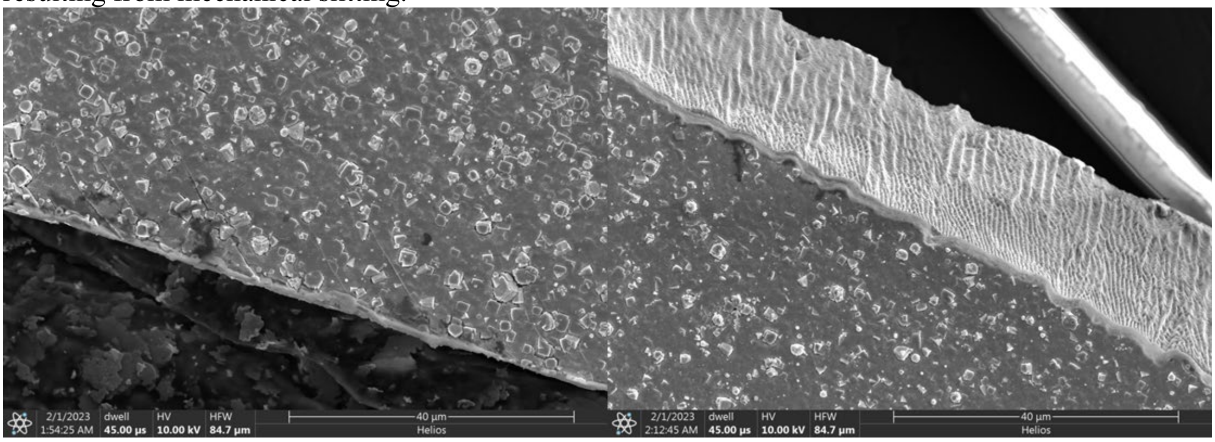

Figure 1. (Left) Mechanically slit tape from SuperOX Japan. The mechanical slitting cracks are uniform in size along the length of the sample, extending ~30 µm into the width of the conductor. (Right) IR Laser slit tape also from SuperOX Japan. The laser ablated area extends ~40 µm into the width of the conductor and has resulted in an obvious slant on the sides of the conductor. Both edges of each sample look like the other.

Cracks resulting from conventional mechanical slitting have long been suspected to act as stress-concentrations[7] under transverse-tensile load and to play a significant role[1] in the degradation of REBCO CC in magnets. The mechanically slit SuperOX tape in the figure above reveals entirely typical cracks compared to what is observed in SuperPower tapes[2]. In comparison, the IR laser slit tape has a 41° slant at the cut Hastelloy edge spanning ~50 µm with a melted edge rather than cracks. EDS confirms that the slanted region is only Hastelloy, no traces of buffer or REBCO was observed past the melted edge. The microstructure of each REBCO layer is nearly identical to one another beyond the slitting region and was uniform across the width. In other laser slit tapes from SuperOX Japan, as well as from Fujikura and Shanghai Superconductor, similarly melted or ablated edges were observed, with some being better than others. The cross-sectional view with the copper and silver sheaths clearly shows the slant associated with the SuperOx laser slitting procedure whereas the Shanghai laser slitting though more perpendicular in its cut severely melts the edge.

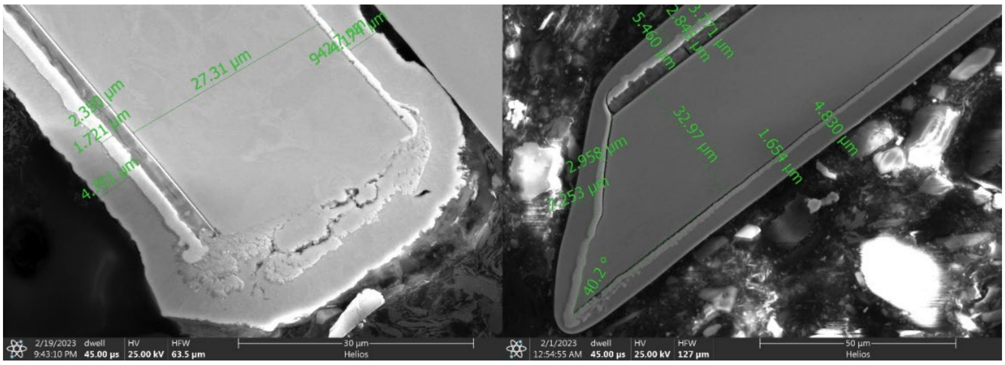

Figure 2. (Left) This laser slit tape from Shanghai Superconductor reveals severe melting of the Hastelloy (to the point of being in direct contact with the REBCO) and a total lack of silver on the edges of the conductor. (Right) This UV-laser slit tape from SuperOX reveals a cleanly laser slit edge, however with a 40° angle associated with the cut.

The negative impact of mechanical slitting cracks are quite clear in the form of crack propagation whereas the melted edges and the severe slant of (some) laser slitting have more complex implications for the stress tolerance of the CC. The severely melted edge of the Shanghai Superconductor samples is particularly concerning regarding the delamination strength of the CC, as it has been shown[9] that defects along the edge can reduce the delamination strength of the CC by an order of magnitude. Despite the potential reduction in delamination strength, it has been reported by Shanghai that the laser slit tapes have greater fatigue behaviour as compared to mechanically slit REBCO tapes[10]. Regarding the SuperOX Japan sample, the 40° slant of the edge is concerning for the typical double-pancake winding method, as not only is the contact edge reduced from ~50 μm to ~10 μm, the conductor will not sit flat and will have a proclivity to bend out of plane, exacerbating the issue of radial expansion and compression of solenoids, especially those composed of REBCO CC

3.2. Flux Penetration from Slitting

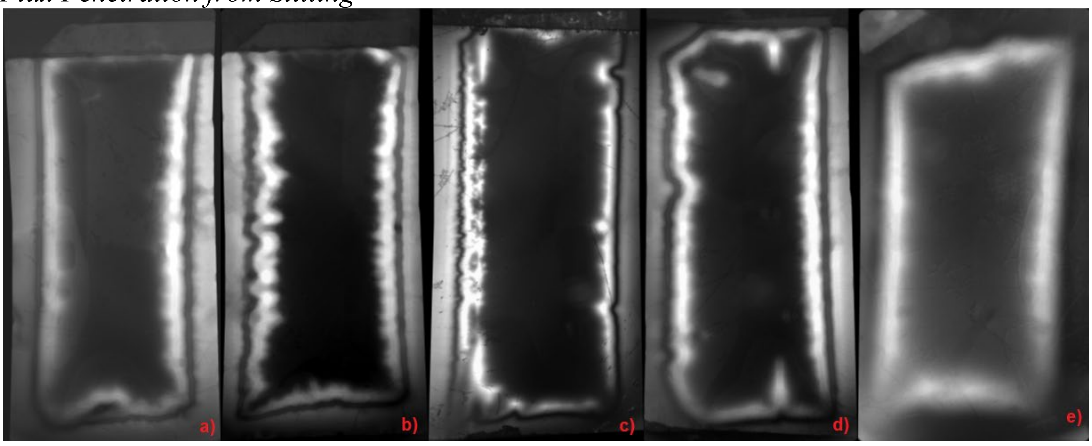

Figure 3. a) This laser slit tape from Fujikura had a rectangular cross-section with few irregularities. Occasional sections of overheated material from slitting observed. b) Mechanically slit tape from Shanghai Superconductor shows irregular flux penetration consistent with edge damage from mechanical slitting. c) SuperOX IR Slit illustrates incomplete slitting of the REBCO. The interesting patterns of small islands of un-slit material which are readily observed in plane-view SEM. d) Mechanically slit tapes from SuperPower have irregularities in the MO images that suggest only very minor cracking. A common defect (scratch on the substrate) extends the length of the sample and beyond. e) THEVA mechanical slit yields a less well-defined image because of the 30° inclined substrate which complicates the induced current flow paths and their effect on the imaging film.

The MOI of the various manufacturers reveals many otherwise unnoticeable defects in both mechanically and laser-slit conductors. The mechanically slit samples in the above image (Shanghai, SuperPower, THEVA) all show the characteristic slitting crack damage, although the SuperPower samples cracks were on the order of 20 μm whereas the others are closer to 50 μm (THEVA is too blurry to notice the cracks in MOI but cracks are readily observed in SEM or optical microscopy).

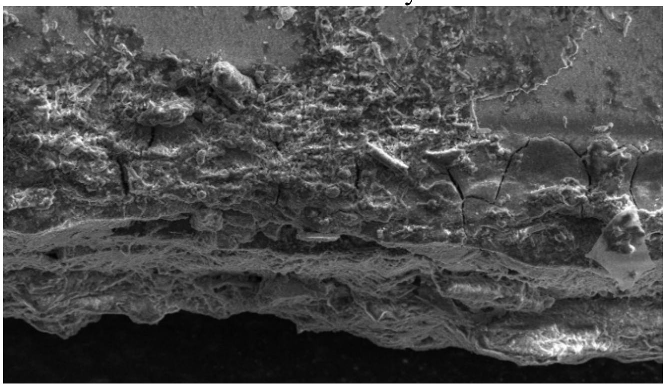

Figure 4. Plane-view image of mechanically slit THEVA tape revealing a rough microstructure and slitting cracks. The rough appearance and irregularity of the slit are likely due to THEVA's inclined substrate deposition (ISD) synthesis method.

The laser slit tapes shown here (Fujikura and SuperOX Japan) represent the extremes of current laser slitting in REBCO CC, with the Fujikura sample being rectangular and only sporadic overheated areas of the film and the SuperOX Japan having incomplete slitting of the REBCO CC and a trapezoidal cross-section. Other inexplicable defects in the middle of the tape (akin to the scratch on SuperPower) were observed in every manufacturer using MOI.

3.3. *Variations in $I_c(B, T, \theta)$ Amongst Manufacturers*

Generation of ultra-high magnetic fields with REBCO CC relies heavily on detailed understanding of the $I_c(B, T, \theta)$ of the conductor so that the local I_c margin throughout the magnet can be mapped. The large temperature and magnetic field ranges being considered for REBCO use requires a large dataset to be acquired for each conductor, as significant variation can exist within[8] a single manufacturer and especially between them depending on the synthesis method and pinning centre scheme. In the figure below, torque magnetometry was used to characterize CCs in a variety of high magnetic fields and temperatures, revealing a wide distribution of critical currents.

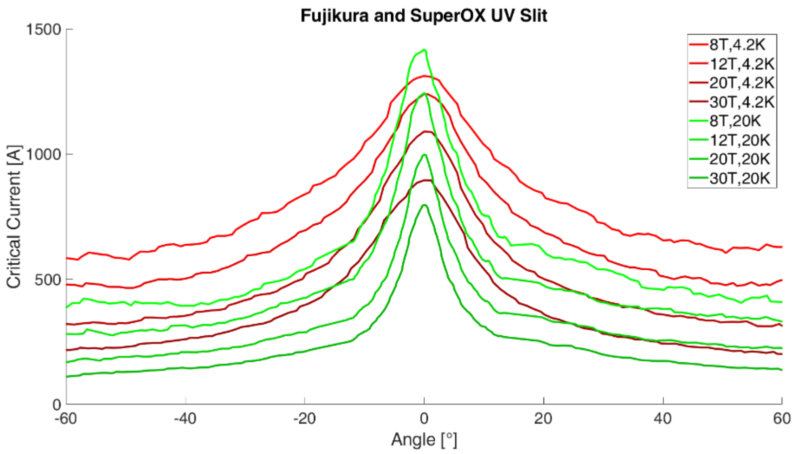

Figure 5. Calculated critical current (Ic) for two samples characterized in the torque magnetometer in the 31 T resistive magnet at NHMFL in roughly 4 hours. The Fujikura (red) and SuperOX Japan (green) show distinct angular distributions (which are not easily parameterized) and possess similar critical currents at higher fields despite the difference in temperature. The critical current in this plot is calculated by converting the torque measured on a rotating platform to critical current flowing at the edge of a conductor according to the bean model.

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4. Implications

This preliminary study represents a glimpse at the characterization routine now being practiced at ASC for the development of our ultra-high-field compact REBCO CC solenoids. The principal point of this incomplete description is that coated conductors being made today are rather far from their cartoon of rectangular, square-edged (even if evenly rounded) objects that can be tightly wound. We do not represent that the results presented here are typical of any manufacturer's product. Although all tapes were purchased in 2021 or 2022, this is many product cycles ago for several manufacturers. We emphasize too that defined specifications for coated conductors are still very sparse, mainly I_c(77,SF) and thickness and width dimensions. Thus the end user of coated conductors must generally develop their own specifications and develop QA procedures to verify fitness for purpose. The purpose of this brief report is to suggest some aspects not normally addressed that may be important for each individual use.

One specific area where we plan more study is on the topic of laser slitting. Our concerns about the cracks introduced by mechanical slitting go back to the time when mechanical slitting was the only way to slit. Delamination of regions near slitting cracks was very evident in some pancakes of LBC3 which reached 45.5 T, admittedly with very high screening current stresses estimated to be ~1 GPa. This message of mechanical slitting being bad was reinforced by recent tension fatigue testing of Shanghai tapes that showed that laser slit tapes had a higher fatigue strength than mechanically slit tapes. We were thus rather surprised at the not completely dense, evidently melted structure of a (we emphasize a) Shanghai tape shown in Figure 2. It does not convey the sense that the REBCO layer is fully protected by such slitting. Clearly more study is needed.

The broader context of studies such as this is that there is now huge demand for coated conductor, especially for fusion use at high fields (typically 20 T at 20 K). Supply chain resilience suggests that at least 2, perhaps more, manufacturers should be qualified for use, even if they manufacture their REBCO CC by different routes with different specifications. Each will have its own quirks, which may or may not be important for each application. The purpose of this short report is to suggest that if any of the observations presented here give you cause for thought, an appropriate characterization scheme may be in order.

Another issue much more heavily addresses is the variability of angular I_c distributions over wide ranges of field and angle. Until recently there was no instrument that could easily, consistently and rapidly measure $I_c(\Theta,B,T)$. Torque magnetometry is now capable of doing this over the range up to at least 31 T from 4-60 K routinely and we believe that this will be vital tool for building future high field magnets with REBCO CC. Future studies of ours will place the purchased conductors whose partial characterization has been presented here into small-scale test coils into harsh stress so as to better understand what properties of the highly non-cartoon real REBCO coated conductors have to be controlled and improved.

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