Delamination strength of YBCO coated conductors under transverse tensile stress*

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Abstract
We present a new experimental technique to measure the delamination strength under transverse tensile stress of $YBa_2Cu_3O_{7-\delta}$ coated conductors for electric power applications. The delamination strength, defined as the tensile stress at which the ceramic layers delaminate from one another, is measured at 76 K for different sample configurations. The delamination strength is reduced by as much as 40% when the conductor is slit to smaller width, a standard fabrication process, and this reduction is due to damage to the ceramic layers near the edges of the conductor. We found that the delamination strength of slit coated conductors can be raised significantly by reinforcing the conductor by laminating it with copper strips and adding solder fillets at the edges. In relatively strong conductors, where the delamination strength is as high as 15 MPa, the critical current does not degrade before actual delamination. This fact greatly simplifies sample characterization of practical high-strength conductors, since only mechanical measurements need to be made. The critical current does, however, degrade significantly as a function of transverse stress before delamination in weak conductors that have relatively low delamination strength below 15 MPa. We discuss how a soft metallic layer in YBCO coated conductors may limit the transverse stress that the superconducting layer experiences in applications.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Remarkable technical advances have been achieved during the past few years in the development of high-temperature superconductors (HTS) for large-scale applications [1]. Research efforts have resulted in superconducting current densities (J_c) in $YBa_2Cu_3O_{7-\delta}$ (YBCO) coated conductors of 2.5–3.0 MA cm^{-2} in self-magnetic field at 77 K, for conductor lengths exceeding 300 m [2]. Major advances in the ability of HTS to carry a supercurrent at high magnetic field have been achieved by enhancing their flux pinning properties by introducing nanoscale defects [3–6]. The ability of YBCO coated conductors to resist mechanical axial strain, an important aspect when used in applications, has improved as well. Although J_c is reduced reversibly as a function of axial strain [7, 8], coated conductors carry a supercurrent up to an irreversible strain limit of 0.5–0.6% [9].

After YBCO coated conductors became available in long lengths, their resistance to transverse tensile stress became an important factor in the success of electric power applications. Transverse stresses that act on the conductor are the result of centrifugal forces in, for instance, generators, and more generally from differential thermal contraction in coil structures. Here, we report the delamination strength, measured with a new experimental technique, of YBCO coated conductors that have a laminar YBCO grain structure. The critical current (I_c) is measured as a function of transverse tensile stress to verify whether the critical current degrades before the conductor delaminates.

2. Experimental details

2.1. Samples under investigation

The coated conductors investigated consist of a YBCO layer 0.8 μm thick and deposited onto an aligned substrate with
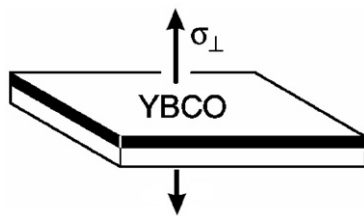

Figure 1. Schematic of the testing mode, where stress is applied to the YBCO coated conductor along its *c* axis, as indicated by the arrows.

Table 1. List of samples.

Sample number	Width (mm)	Slitting	Lamination	Remarks
1	10.0	No	No	—
2	10.0	Yes	No	—
3	4.0	Yes	No	—
4	4.4	Yes	Yes, three-ply	—
5	4.0	Yes	No	Slit after reaction

metal–organic deposition (MOD) [10, 11]. This technique results in a laminar YBCO grain structure with meandering grain boundaries [12]. Grain alignment is introduced within a textured NiW substrate 75 μm thick, by use of the rolling assisted biaxially textured substrate (RABiTS) technique [13, 14]. These samples are designated MOD-RABiTS.

The MOD-RABiTS conductors are slit from 4 cm width to either 1 cm or 4 mm width. This type of slitting is a standard industrial process used to fabricate practical conductors. Some of the 4 mm wide slit MOD-RABiTS samples are laminated to provide electrical and mechanical stability needed in most applications. The conductors are laminated with two copper strips 50 μm thick by soldering the strips to the slit tape ('three-ply') with 62 Sn–36 Pb–2 Ag solder at 179 °C. A list of samples is presented in table 1.

2.2. Measurement technique

Stress is applied along the *c* axis of the YBCO coated conductors (figure 1) by attaching two anvils to the conductor. This straightforward method was chosen over other methods, such as mixed-mode bending [15, 16], because it allows the cooling of samples in a stress-free mode from room temperature to 76 K.

The coated conductor was soldered on a hot plate to two anvils made from Ni–5 at.% W. This material was chosen for the anvils to closely match the thermal contraction of the samples so that no shear stresses develop during cool-down. The thermal contraction of the samples is determined mainly by the substrate material, in this case Ni–5 at.% W. The oxide layer that is formed on the surface of the Ni–5 at.% W of the anvils does not allow a strong solder joint to be made directly to the sample. Therefore, a $\approx$200 μm thick copper layer was brazed to the solder surface of both anvils in vacuum at 1050 °C with 50 Au–50 Cu powder. The brazing procedure breaks down the oxide layer on the surface of the NiW, and a strong solder joint between the copper layers and the sample
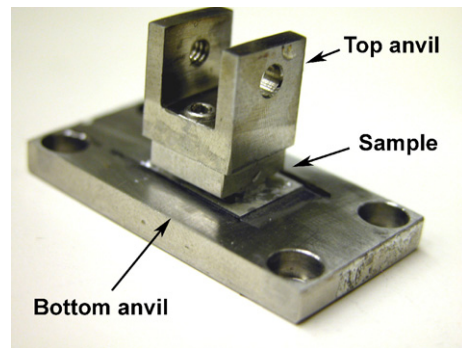

Figure 2. The bottom and top anvils are soldered to the sample. Both anvils are made from Ni–5 at.% W to match the thermal contraction of the sample.
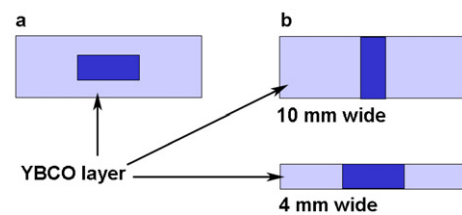

Figure 3. Representation of different sample mounting configurations. (a) The top anvil (dark rectangle) is soldered in the centre of the sample (light rectangle) to measure the internal delamination strength. (b) The top anvil is mounted in such a way that it covers the top of the sample (both 10 mm wide and 4 mm wide) from one sample edge to the other to include the effect of edges on the delamination strength.

can be formed. The thermal contraction of both anvils is still dominated by the much thicker Ni–5 at.% W. This has been verified using strain gauges attached directly to the anvil's thin copper layer.

Samples that are not laminated are soldered with their substrate side to the bottom anvil using 63 Sn–37 Pb solder at 183 °C and corrosive ZnCl$_2$ flux (figure 2). The substrate is first lightly sanded with 500 grit sandpaper to remove most of its oxide layer. Laminated samples are soldered with 97 In–3 Ag solder at 143 °C to ensure that the 62 Sn–36 Pb–2 Ag solder used for lamination does not melt. Care is taken that the 97 In–3 Ag solder does not alloy with the 62 Sn–36 Pb–2 Ag solder. The top anvil, 4 by 10 mm in size, is soldered with 97 In–3 Ag solder at 143 °C in the middle of the sample, on top of the silver cap layer. At this relatively low temperature the solder will not penetrate through the cap layer if the silver is at least 7 μm thick [17].

Two different sample mounting configurations were used in the experiment. The top anvil is mounted in the centre of the sample when the delamination strength is measured without involving the edges of the sample (figure 3(a)). The top anvil is mounted as indicated in figure 3(b), covering the sample from edge to edge, when, for instance, the effect of conductor slitting on the delamination strength is measured.

The transverse stress is applied to the sample with a servo-hydraulic actuator. The top anvil is biaxially gimballed to ensure a proper alignment between the sample and the rest of the measurement device (figure 4). One of the axes where the
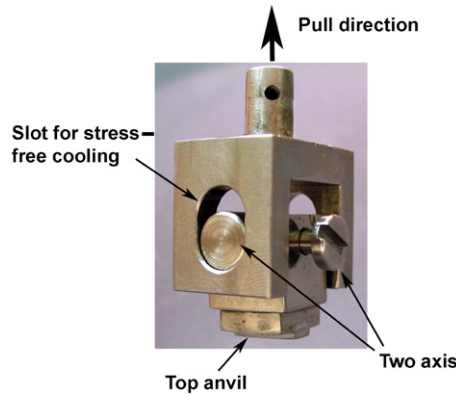
Figure 4. Details on how the top anvil is attached to the servo-hydraulic system, showing the biaxially gimballed top anvil. One of the axes is located in a vertical slot to enable stress-free cooling of the sample. The anvil will move to the bottom of the slot when stress is applied, as indicated in the image.

top anvil is attached is inserted in the centre of a vertical slot. This enables the sample and the anvils to be cooled in a stress-free manner. After cool-down, the servo-hydraulic actuator is activated until the rod of the top anvil reaches the bottom of the slot. At that point, the top anvil starts to pull on the sample. The stress is then increased by 1 MPa s^{-1}, until the sample delaminates. Sample delamination is detected by a steep rise in displacement of the servo-hydraulic actuator and a sharp drop in pulling stress. The delamination stress is measured with a 5% uncertainty.

2.3. Transport current measurements

The critical current of YBCO coated conductors is measured as a function of transverse stress with a four-point transport current measurement. Copper current leads are soldered to both ends of the sample and two voltage contacts are soldered on top of the sample, directly next to the top anvil. The critical current, defined at a criterion of 1 μV cm^{-1}, is measured before stress is applied to the sample with an uncertainty of about 1%.

The critical current as a function of transverse stress is determined in a dynamic mode. A transport current equal to the critical current at zero stress is applied to the sample, before stress is applied. The sample voltage (equal to the voltage criterion before stress is applied) is measured with an uncertainty of about 0.1%, about four times every second at equal time intervals. The transverse stress is then increased at a constant rate of about 0.3 MPa s^{-1} while the current through the sample is kept constant. The sample voltage is thus measured about once every 0.08 MPa. The sample voltage remains constant as a function of stress as long as the critical current of the sample does not change. A rise in sample voltage is measured when the critical current decreases as a function of stress. The (reduced) critical current is estimated with a power law function:

$$I_{\rm c} = I \left(\frac{V_{\rm c}}{V} \right)^{\frac{1}{n}}, \tag{1}$$

where I is the sample current, V is the sample voltage and $V_{\rm c}$ is the voltage criterion. The value of n is determined from the V–I curve at zero stress. This value is most likely not constant
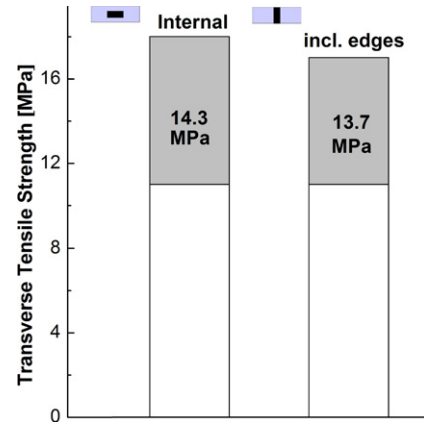

Figure 5. Delamination strength of 1 cm wide MOD-RABiTS samples that were not slit (sample 1 in table 1). The left bar shows the range of the internal delamination strength when the conductor edges are not included. The testing mode is shown in the left top corner of the image. The delamination strength of 14.3 MPa is averaged over four samples. The bar on the right shows the range in delamination strength when the sample edges are included (as indicated above the bar). The average delamination strength when the edges are included is 13.7 MPa and was measured on six samples.

at the electric field of 1 μV cm^{-1}, but will be reduced when $I_{\rm c}$ decreases [18]. Even though inserting the constant n value at zero stress in expression (1) results in an upper limit of the critical current, this method allows us to determine the stress at which the critical current degrades.

3. Results and discussion

The delamination strength of YBCO coated conductors is an important parameter for the design of electric power applications. The question of what determines the delamination strength of practical coated conductors is addressed below. The measurement procedure to determine the effect of conductor edges on the delamination strength is discussed in section 3.1. The strength of the ceramic layers for MOD-RABiTS is reported and the effect of conductor slitting on the delamination strength is determined in section 3.2. The effect of transverse tensile stress on the critical current is studied in section 3.3. Finally, in section 3.4 a brief discussion is given on how to limit the transverse stress on the YBCO layer of the conductor when used in applications.

3.1. Delamination test configurations

We used two different testing configurations to measure the delamination strength of YBCO coated conductors. The first configuration, where the top anvil is soldered in the centre of a wide coated conductor, was used to measure the 'internal' strength of the ceramic layers without including conductor edge effects (see section 2.2, figure 3(a)). The second configuration was used to measure the delamination strength of coated conductors, including the effect of conductor edges. In that case, the top anvil is soldered to the conductor, as indicated in figure 3(b).

Figure 5 shows the results from both testing modes on a 1 cm wide MOD-RABiTS conductor that is not slit (sample 1
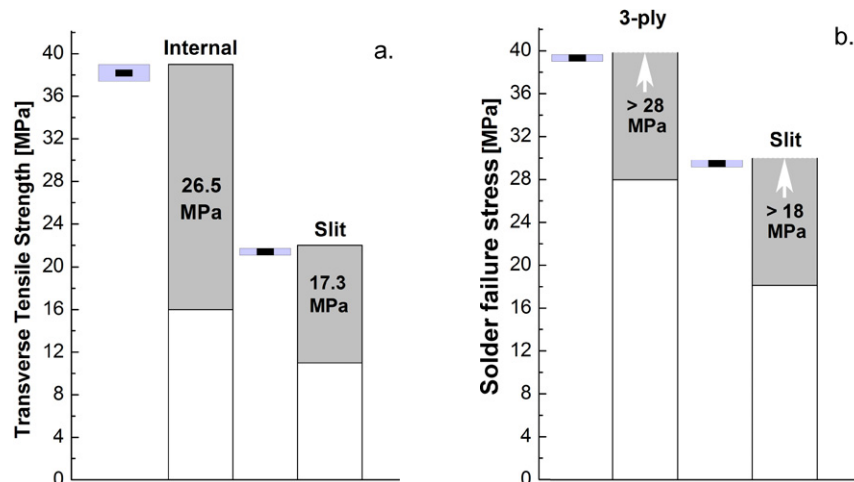
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Figure 6. (a) Delamination strength of MOD-RABiTS conductors at 76 K, showing the degradation from slitting and the significant improvement in delamination strength from lamination. The bar on the left represents the internal strength measured on 1 cm wide conductors (sample 2 in table 1), averaged over eight samples. The bar on the right represents the delamination strength of slit conductors (samples 2 and 3 in table 1), averaged over 13 samples. (b) The bar on the left represents the stress at which the solder joint between the anvil and the laminated samples (sample 4 in table 1). The bar on the right represents the stress at which the solder joint between the anvils and samples that were reacted after slitting broke (sample 5 in table 1). The arrows in the grey bars indicate that the stress at which the solder joint broke is an estimated lower limit of the delamination strength.

in table 1). The left bar shows the range over which the 'internal' delamination strength is measured. Here, the top anvil does not cover the sample up to the edges (as indicated in the top left part of the figure). Only a limited number of samples are measured, since this 1 cm wide unslit conductor is no longer available. The 'internal' strength ranges from 11.0 to 18.0 MPa, with an average of 14.3 MPa. The delamination strength is rather low compared to recent YBCO coated conductors (see section 3.2) and is most likely due to the improvements in conductor quality over the years. The bar on the right in figure 5 shows the delamination strength when the conductor edges are included. The delamination strength ranges from 11.0 to 17.0 MPa, with an average of 13.7 MPa. Scanning electron microscopy and energy-dispersive x-ray spectroscopy studies of the exposed surfaces after delamination of the conductors reveals that they failed within the YBCO layer.

The results of the two different configurations show that including the conductor edges in the experiment has no significant effect on the measured delamination strength of a coated conductor, as long as the conductor is not slit.

3.2. Delamination of MOD-RABiTS conductors

Production of coated conductors in industry has been scaled up during the last several years. The ceramic films are deposited on wide substrates that are then slit to their final width, usually 10 or 4 mm (samples 2 and 3 in table 1). The effect of conductor slitting on the delamination strength of MOD-RABiTS is described in this section. Here, the internal delamination strength of slit conductors is compared to the delamination strength when the conductor edges are included.

The average delamination strength of MOD-RABiTS conductors has increased over the years from 14.3 MPa two years ago, as outlined in section 3.1, to 26.5 MPa recently, as indicated by the left bar in figure 6(a). The increase is most likely due to improvement in the production process. A large

spread in internal delamination strength is measured, ranging from 16.0 to 39.0 MPa, which could be due to inhomogeneity in ceramic layer properties along the length of the tape.

The delamination strength of MOD-RABiTS conductors was measured after the conductor was slit to either 10 or 4 mm width. The total delamination strength of the conductor was measured, including the effect of sample edges, by mounting the top anvil as indicated in figure 3(b). The result shows that the delamination strength of slit MOD-RABiTS conductors is significantly lower than their internal strength, as indicated by the right bar in figure 6(a). The average delamination strength ranges from 10.0 to 22.0 MPa, with an average of 17.3 MPa. The slitting process is well known to induce mechanical damage within the ceramic layers near the edges of the conductors [19]. These areas act as delamination–initiation sites and lower the overall delamination strength of the conductor.

To improve the electrical, thermal and mechanical properties, the slit MOD-RABiTS conductors were laminated with two 50 μm thick copper strips (three-ply architecture, figure 7). The strips were soldered with 62 Sn–36 Pb–2 Ag solder at 179 °C and the slit conductor was sealed at the edges by solder fillets. The lamination with solder fillets extending over the edges of the slit conductor raises the delamination strength to above 28.0 MPa, as is indicated by the left bar in figure 6(b). These samples did not delaminate in our tests, because the solder joint to the top anvil failed at a minimum stress of 28.0 MPa. We had to protect the solder fillets with epoxy to prevent alloying with the solder that was used between tape and anvil. This resulted in a relatively weak solder joint to the top anvil.

Since delamination under transverse tensile stress occurs within the YBCO layer, this indicates that most of the slitting damage occurs within this layer. The YBCO layer in MOD-RABiTS is formed by spin coating the precursor material and

Figure 7. Optical image of the cross section of a copper laminated MOD-RABiTS tape, where both 50 μm thick copper laminates extend over the edges of the 75 μm thick slit coated conductor (sample 5 in table 1). One solder fillet is visible on the left of the image (only part of the cross section with one conductor edge is shown). Figure courtesy of American Superconductor Corporation.

decomposing it at low temperature to primarily CuO, Y_2O_3 and BaF_2 [20]. The next step is a reaction at high temperature to form the ceramic YBCO layer, before the conductor is slit to its final width. Damage from slitting can be reduced by reacting the decomposed layer into the ceramic YBCO layer after the conductor is slit. The delamination strength of these 4 mm slit-reacted conductors could not be measured due to the solder joints with the anvil failing at a minimum stress of 18.0 MPa. The delamination strength of the conductor exceeds this level, which is significantly higher than the delamination strength of conductors that were slit after reaction (see the bar on the right in figure 6(b)).

3.3. Effect of transverse tensile stress on I_c

So far, only the transverse tensile stress at which YBCO coated conductors delaminate has been measured. Here, we will investigate whether there is any change in electrical performance under transverse tensile stress before the conductor fails mechanically.

First, the critical current of slit coated conductors is measured in self-field with a transport current before transverse stress is applied. Then the sample voltage is measured as a function of stress (in the configuration where the sample edges are included) while a constant transport current is applied to the tape, as is outlined in section 2.3. The change in critical current due to transverse stress is estimated using expression (1). The resulting normalized I_c at 76 K of a slit MOD-RABiTS conductor (sample 3) as a function of transverse tensile stress is shown in figure 8 (open symbols). The critical current shows no significant degradation up to 9.4 MPa. At higher stress, I_c starts to degrade stepwise to about 96% of its initial value before the sample delaminates at 10.8 MPa. The observed degradation is typical for coated conductors that have relatively low delamination strength (below about 15 MPa). The actual degradation in I_c may be larger than is shown in figure 8. Even though the n value of the conductor is kept constant in expression (1), the n value is reduced as well when I_c decreases. Also shown in figure 8 is I_c as a function of transverse tensile stress for a laminated MOD-RABiTS conductor (sample 4, indicated by the solid symbols). No significant degradation in I_c occurs up to a stress of 25.0 MPa, at which the conductor delaminates. The solder joints of the laminated samples in this particular test were weakened due to alloying with the 97 In–3 Ag solder, causing sample delamination before the solder joints to the anvils failed.

The MOD-RABiTS conductors tested here show similar distinct differences in behaviour, where I_c degrades before the
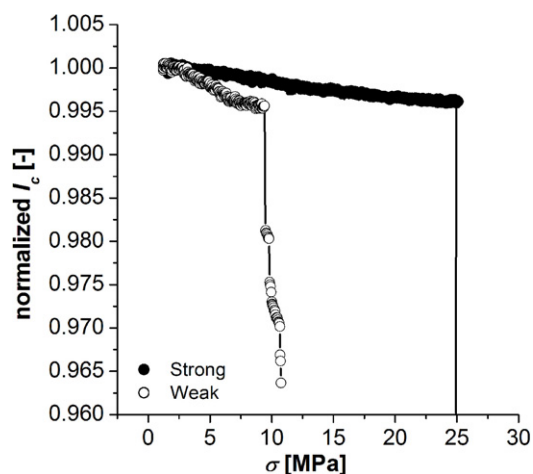

Figure 8. Normalized critical current of two slit MOD-RABiTS coated conductors as a function of transverse tensile stress at 76 K. The solid symbols represent a laminated sample (sample 4) that had a relatively high delamination strength of 25.0 MPa and showed no significant degradation in critical current before delamination. The open symbols represent a slit coated conductor (sample 3) that had a relatively low delamination strength of 10.8 MPa and showed a significant reduction in critical current just before the conductor delaminated.

samples delaminate when they have a relatively low delamination strength, whereas no significant degradation is measured before delamination in relatively strong conductors. This suggests that islands of relative weak material exist in relatively weak conductors, where the microstructure fails locally under transverse tensile stress before complete delamination of the conductor. These areas act as delamination initiation sites that result in complete delamination of the conductor when the stress is further increased. The islands may be the result of local damage due to conductor slitting or areas within the conductor where larger production-related defects exist.

3.4. Limiting the transverse stress

Strengthening YBCO coated conductors to prevent delamination is an important development, although it is not clear at this moment how much transverse stress a conductor will experience in applications. First, cool-down of a typical coil from room temperature to, for instance, 77 K will result in a transverse stress on the YBCO layer. This stress depends in part on the thermal contraction of the winding pack, the epoxy that is used in the winding pack and the backing materials that support the coils. Second, the application itself may contribute additional stress during operation, for instance due to centrifugal forces in generators.

Delamination of conductors under transverse tensile stress played no significant role when coils where made from $Bi_2Sr_2CaCu_2O_x$ (BSCCO 2212) or $Bi_2Sr_2Ca_2Cu_3O_x$ (BSCCO 2223) tapes. These conductors are certainly not stronger than YBCO coated conductors. The delamination strength at 77 K of BSCCO 2223 tapes with a pure silver matrix ranges from as low as 3.0 MPa to about 14.0 MPa [21]. Only tapes that are reinforced by lamination with steel strips, with solder fillets covering the edges, have significantly larger delamination strengths.

The main differences between YBCO coated conductors and BSCCO tapes are their architecture and the mechanical properties of the different materials within the conductors. First of all, YBCO coated conductors have a layered structure, whereas BSCCO tapes consist of a large number of ceramic filaments embedded within a soft silver matrix. Second, YBCO coated conductors consist of a fairly strong substrate, while the annealed (during the various heat treatments) silver matrix in BSCCO tapes has not only a relatively low Young's modulus of 76 GPa [22], it also has a *very low yield strength*. The silver matrix is already yielding during cool-down, due to the stress resulting from the mismatch in thermal contraction between the silver matrix and the ceramic filaments [23]. At 76 K, Ni–5 at.% W (the substrate material of the MOD-RABiTS conductors) has a Young's modulus of about 128 GPa and a tensile yield strength of 255 MPa [24]. The yield strength of work-hardened copper (the lamination material of MOD-RABiTS conductors) at room temperature is as high as 333 MPa [22].

The transverse tensile stress that a conductor experiences during cool-down is a direct result of the difference in thermal expansion coefficient (α) of the conductor and other components such as the support structure of the coil. The mismatch in α results in a transverse *strain* in the conductor. The transverse stress experienced by the conductor is the result of this strain and the mechanical properties of the components in the wire (Young's modulus and yield strength). The soft silver matrix in BSCCO tapes absorbs most of the strain because of its relatively low yield stress. This limits the transverse tensile stress on the filaments in the tape. The YBCO coated conductors that are available at present do not consist of soft metallic components. As a result, the transverse stress on the YBCO layer will be significantly higher than the stress on the BSCCO filaments in a BSCCO tape, even when they are used in the same application.

Here we suggest a method of reducing the transverse tensile stress on the YBCO layer in coated conductors by incorporating a soft metallic layer within the conductor. One of the hardened copper laminates could, for instance, be replaced with soft annealed copper, in the case of laminated MOD-RABiTS conductors. This copper layer will yield at low stress, limiting the transverse stress on the YBCO layer. This will result in a relaxation of the conductor requirements for delamination strength.

4. Conclusions

A new experimental technique has been developed to measure the delamination strength of YBCO coated conductors under transverse tensile stress. Measurements at 76 K have been performed on various types of coated conductors that were produced with metal–organic deposition on biaxially textured substrates (RABiTS). The results show that the delamination strength of RABiTS coated conductors degrades significantly when the conductors are slit, due to localized damage to the microstructure of the conductor. The delamination strength of slit conductors can be significantly improved by external reinforcement with lamination (as is currently done with RABiTS conductors), or by reacting the YBCO layer after the conductor is slit. We were not able to delaminate these stronger

samples, but were able to provide an estimate of the lower limit of the delamination strength; the stress at which the solder joint between anvil and sample failed.

The critical current of YBCO coated conductors degrades significantly only under transverse tensile stress before the conductor delaminates in the case where localized damage due to, for instance, slitting exists and causes a relatively low delamination strength of less than 15 MPa. Relatively strong conductors show no significant degradation in critical current before delamination.

We proposed an alternative approach to prevent delamination of coated conductors in applications, where a soft metallic layer is incorporated within the conductor. This layer will lower the transverse stress on the ceramic layers during cool-down by absorbing most of the applied strain. Lowering the transverse stress due to the mismatch in thermal expansion between different materials within the application may ease the conductor requirements.

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