Critical current survival in the YBCO superconducting layer of a delaminated coated conductor

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Abstract
A high-temperature superconducting coated conductor can be practically applied in electric equipment due to its favorable mechanical properties and critical current ($I_c$) performance. However, the coated conductor can easily delaminate because of its poor stress tolerance along the thickness direction. It would be interesting to investigate whether the $I_c$ of the delaminated YBa$_2$Cu$_3$O$_{7-\delta}$ (YBCO) layer can be preserved. In this study, coated conductor samples manufactured through the metal organic deposition route were delaminated by liquid nitrogen immersion. Delaminated samples, including the YBCO layer and silver stabilizer, were obtained. Delamination occurred inside the YBCO layer and near the YBCO–CeO$_2$ interface, as suggested by the results of scanning electron microscopy (SEM) and x-ray diffraction. A scanning Hall probe system was employed to measure the $I_c$ distribution of the original sample and the delaminated sample. It was found that approximately 50% of the $I_c$ can be preserved after delamination, which was verified by $I_c$ measurements using the four-probe method. Dense and crack-free morphologies of the delaminated surfaces were observed by SEM, which accounts for the $I_c$ survival of the delaminated YBCO layer. The potential application of the delaminated sample in superconducting joints was discussed based on the oxygen diffusion estimation.

Keywords: coated conductor, critical current, delamination, YBCO layer, oxygenation

(Some figures may appear in colour only in the online journal)

1. Introduction
High-temperature superconducting (HTS) coated conductors (CCs), also known as the second generation HTS wires, are generally regarded as the main candidate for various HTS electric devices [1–3]. A metallic substrate can provide favorable mechanical strength in the length direction and width direction of the CC. However, in the thickness direction, the adhesion strength of the layer interfaces is weak [4, 5], which can lead to delamination behavior. The stress tolerance in the thickness direction can be exceeded in many ways, such as through thermal cycling [6], electromagnetic force [7], hoop stress [8], etc.

Delamination is generally considered as a failing of the CC, and is a severe threat for practical applications [9, 10]. Therefore, CC delamination has been investigated by many groups in the literature. Takematsu et al studied the structural changes of epoxy-impregnated double pancake coils after five temperature cycles of 77 K to room temperature [6]. In van der Laan et al’s study, two anvils were fixed to each side of the CC...
The sample and tensile strength was applied to measure the delamination strength, which was about 10 MPa for the 0.8 μm thick YBCO layer [4]. In Kesgin et al’s study, a peel test was performed on the CC samples, and it was found that delamination occurred at the interface of the HTS layer and buffer layer or inside the HTS layer [11]. Liu et al studied the delamination behavior under shear stress, and found that there was a correlation between the critical current (I_c) and shear stress [12]. Commonly, the HTS layer of delaminated CC is considered to be degraded or destroyed, and no previous studies have been conducted to investigate the superconducting properties of the delaminated stack with YBCO and silver layers.

Delamination behavior has been studied in many other fields, such as the fabrication of ceramic thin films on plastic substrates. In the study by Kozuka et al [13, 14], a stack of plastic/ceramic-film/release-layer/Si(100)-substrate was fabricated, and then delaminated to obtain part of the plastic/ceramic-film. The ceramic film was not damaged during delamination in spite of its brittle property. YBCO is ceramic in nature [15], thus it is speculated that the superconducting property might also be preserved after delamination. If the delaminated stack with the YBCO layer and silver stabilizer can carry superconducting current, it will be considered as a new type of CC with the silver stabilizer as an underlayer. Silver is quite different from the oxide buffer layers in traditional CCs, thus it might be possible to use the delaminated stack in some new applications.

Therefore, it would be interesting to study whether the I_c of the YBCO layer can be preserved after delamination. In this study, CC samples were delaminated on purpose by liquid nitrogen immersion. The I_c distributions of the original sample and the delaminated stack were measured by using a scanning Hall probe system, and the morphologies of the delaminated surfaces were observed. The potential application of the delaminated stack in superconducting joint technology is discussed through the estimation of the oxygen diffusion process.

### 2. Experimental procedure

#### 2.1. Delamination by liquid nitrogen immersion

Three CC samples, which were produced by AMSC company through the metal organic deposition (MOD) route, were investigated in this study. All the samples had a multilayered architecture of Ag/YBCO/buffer stack/Ni–W substrate, in which the buffer stack was CeO_2/YSZ/Y_2O_3, but with different lamination layers and lengths, as listed in table 1. The labeled I_c of these samples was 90 A (77 K, self-field).

In order to delaminate these samples, two factors found in our preliminary experiments were considered. First, direct peeling can lead to micro-cracks in the YBCO layer, which can be caused by the local curvature exceeding the radius limitation of the CC [16]. Second, the bonding near the edges was quite strong; thus it was difficult for delamination to occur in these areas [5]. The final experimental route was as follows: firstly, the edges of the samples were cut off and the sample’s width was reduced to approximately 3.7 mm; secondly, the samples were put in liquid nitrogen and delamination occurred [17]. As shown in figure 1, the surfaces of the two delaminated parts were black and green, respectively, indicating that delamination had occurred at or near the YBCO-buffer interface. The delaminated stack with YBCO and silver layers is denoted as the ‘delaminated sample’ in this paper.

#### 2.2. Characterization methods

The I_c distributions of samples 1 and 2 were measured using a scanning Hall probe system produced by the Beijing Eastforce company, as shown in figure 2, which was similar to the system used in [18–20]. The sample was first placed in a liquid nitrogen pool and cooled down to 77 K, then it was excited by an external magnetic field two-fold larger than that of the penetration field [21]. A circulating current, which can be considered as the critical current according to the critical state model, formed in the YBCO layer after switching off the external magnetic field. The magnetic field generated by the circulating current was then measured by the Hall probe with two-dimensional scanning. The I_c distribution inside the YBCO layer was then calculated based on the magnetic field distribution. In order to verify the results of the scanning Hall probe measurements, the I_c distributions of the delaminated sample were measured using the same system.
Figure 2. The scanning Hall probe system for the $I_c$ distribution measurement. A close view of the sample zone indicated by the dashed square in figure (a) was shown in (b). The labels are: (1) liquid nitrogen pool, (2) DC source of 12 A for the exciting magnet, (3) Keithley 2700 for the Hall probe signal collection, (4) computer for recording data, (5) Hall probe, (6) measured sample, (7) exciting magnet of the copper coil.

Figure 3. The four-probe device for $I_c$ measurement; (a) original sample 3, (b) delaminated sample 3 (one current lead is loose to show the YBCO surface).
Figure 4. The magnetic field distributions of (a) the original sample and (b) the delaminated sample, which were measured using the scanning Hall probe system. The \( I_c \) distributions of (c) the original sample and (d) the delaminated sample, which were calculated according to the data in (a) and (b), respectively.

Figure 5. The \( I_c \) distributions of (a) original sample 2 and (b) delaminated sample 2, which were obtained by the scanning Hall probe system.
probe system, the $I_c$ values of sample 3 before and after delamination were measured using a four-probe device, as shown in figure 3.

The morphologies of the delaminated surfaces were characterized through scanning electron microscopy (SEM) using a Hitachi SU8010 with an energy dispersive spectrometer (EDS) to analyze the elemental composition. X-ray diffraction (XRD) 2θ-θ scanning was carried out to identify the phases of the delaminated surfaces using a Bruker D8 ADVANCE.

Figure 6. Voltage ($U$–current ($I$) curves obtained with the four-probe method of original sample 3 and delaminated sample 3. According to the $1 \mu V \text{cm}^{-1}$ criteria, the $I_c$ of original sample 3 was about 100 A, while the $I_c$ of delaminated sample 3 was about 53 A.

Figure 7. SEM images of (a) the zone with the preserved $I_c$ of delaminated sample 1, (b) the zone with zero $I_c$ of delaminated sample 1, (c) the delaminated part with the buffer layers, and (d) the EDS result at the cross position of (c).
3. Results

3.1. \( I_c \) of the original and delaminated samples

The magnetic field distributions of the original sample 1 and delaminated sample 1 are illustrated in figures 4(a) and (b), respectively. Their calculated \( I_c \) distributions are shown in figures 4(c) and (d). \( I_c \) is low in the central zones of both samples because of the field penetration mechanism, which is consistent with the report by Higashikawa et al [18]. As shown in figure 4(d), a small area of the delaminated sample had zero \( I_c \). The average \( I_c \) percentage of the delaminated sample relative to the original sample was about 50%. A similar result was obtained for sample 2, as shown in figure 5, in which about 45% of the \( I_c \) was preserved in approximately 2/3 of the sample.

In order to investigate the capacity of the delaminated sample to practically transport current, the \( I_c \) of sample 3 was measured by using the four-probe method, as shown in figure 6. According to the 1 \( \mu \)V cm\(^{-1} \) criteria, the \( I_c \) of original sample 3 was about 100 A, while the \( I_c \) of delaminated sample 3 was about 53 A. The preserved \( I_c \) ratio of about 53% is quite similar to the results of the scanning Hall probe method, as shown in figures 4 and 5. Therefore, it is certain that the \( I_c \) can be partially preserved in the YBCO layer after the delamination process used in this study.

3.2. Surface morphology and element analysis

The SEM images of the delaminated sample for sample 1 are shown in figures 7(a) and (b). The porous morphology is common for YBCO thin films fabricated via the MOD method [22]. To explore the reason for the zero \( I_c \) zone, its surface was observed, as shown in figure 7(b). The YBCO film was not continuous in the zero \( I_c \) zone, which might be due to the stress concentration during the delamination process. On the surface of the delaminated part with the buffer layer, there is some porous morphology, indicating the existence of YBCO grains, as shown in figure 7(c). The EDS result shown in figure 7(d) verifies that some YBCO grains are attached to the buffer layer.

In the \( I_c \) distribution result of sample 2 (figure 5), it can be seen that the delaminated sample was badly destroyed, and about 1/3 of it had a zero \( I_c \). The morphologies of delaminated sample 2 can be observed in figure 8. Figure 8(a) shows an SEM image of the zone with preserved \( I_c \), and figure 8(c) shows the zone with zero \( I_c \). The corresponding EDS results are illustrated in figures 8(b) and (d), respectively. However, there is no obvious difference between the two zones. Therefore, more work will be needed to investigate the stress distribution and fracture mechanism in our future studies.
Moreover, it can be inferred according to figure 9 that the YBCO thickness of the delaminated sample was much larger than that of the delaminated part with buffer layers. Therefore, it can be concluded that delamination occurred inside the YBCO layer and near the YBCO–buffer interface.

In MOD-YBCO fabrication, BaCeO$_3$ can form easily because of the interfacial chemical reaction of the CeO$_2$ buffer layer and Ba-containing precursors [23–25], which was also observed in figure 9(b). Moreover, it was reported by Coll et al [26] that the epitaxial relationship between BaCeO$_3$ and YBCO was not favorable, with a mismatch of about 14%. Therefore, the fact that delamination occurred near the YBCO-CeO$_2$ interface might be attributed to the degraded adhering strength caused by BaCeO$_3$.

4.2. Potential application in the superconducting joint

By using the liquid nitrogen immersion method, a delaminated sample with preserved $I_c$ can be obtained. Although more work is required to enhance the percentage of preserved $I_c$, a potential application of the delaminated sample in superconducting joints is proposed in this study. As reported by Park et al [27, 28], there was a very long oxygenation process during the fabrication of the superconducting joint with the CC, thus laser drilling was utilized to decrease the diffusion length ($h$) and the oxygenation duration. The utilization of the delaminated sample might help to shorten the oxygen diffusion process.

Based on the above experimental results, it can be concluded that the delaminated sample is a YBCO/silver/copper stack, as sketched in figure 10(a). Silver has a large oxygen diffusion coefficient ($D$) in the order of $10^{-6}$ cm$^2$ s$^{-1}$ at 450°C–600°C (a typical temperature range for the oxygenation process of YBCO) [29], and this is much larger than that of YBCO (typically $10^{-14}$ cm$^2$ s$^{-1}$ along the c-axis direction [30]).

If the delaminated sample is used to connect two CCs, as shown in figure 10(b), and partial melting treatment is used to enable inter-diffusion of YBCO surfaces similar to Park’s study [27, 28], the oxygenation process can be estimated as follows: firstly, the oxygen diffusion will be in the silver layer, and its duration can be neglected since silver has a much larger $D$ value than YBCO. Secondly, the oxygen diffusion in YBCO is anisotropic, $D$ in the $a$-$b$ plane of YBCO being $10^4$–$10^6$ times that in the $c$-axis direction [31], while $h$ in the $a$-$b$ plane is around $10^3$ times that in the $c$-axis direction. The characteristic time ($\tau$) can be estimated through the one-dimensional diffusion model ($\tau = h^2/π^2D$) [31]. Therefore, oxygen diffusion in YBCO is mainly along the c-axial direction, as shown by the arrows of figure 10(b). The oxygenation of the two YBCO layers would be similar to that in the study of Kim et al [32], in which the YBCO layer exposed in oxygen required 15 h to be fully oxygenated. Thus, the oxygenation duration for the proposed joint structure in figure 10(b) is estimated to be about 60 h, since $h$ is approximately doubled compared to that in [32]. Moreover, the porous MOD-YBCO film has a much larger equivalent $D$ value along the c-axis direction [31], resulting in a more rapid oxygenation than the YBCO layer fabricated via MOCVD in [32].

**Figure 9.** XRD patterns of (a) delaminated sample 1, and (b) the delaminated part with the buffer layers.

### 3.3. Phase detection by XRD

The XRD patterns of delaminated sample 1 and the delaminated part with the buffer layers for sample 1 are shown in figures 9(a) and (b), respectively. According to the XRD database, YBCO (PDF#86-0053), Y$_2$O$_3$ (PDF#88-1040) and Ag (PDF#87-0720) can be identified in figure 9(a), and BaCeO$_3$ (PDF#85-2155), YBCO (PDF#86-0053), NiWO$_4$ (PDF#72-1189), CeO$_2$ (PDF#81-0792), NiO (PDF#78-0643) and Ni (PDF#87-0712) can be identified in figure 9(b). It can be seen that the peak of YBCO is much stronger (about ten-fold) in the delaminated sample than in the delaminated part with the buffer layers.

### 4. Discussion

#### 4.1. Location of delamination occurrence

As shown in figure 7(c), there are YBCO grains on the surface of the delaminated part with the buffer layer, but the morphology of the buffer layer cannot be observed, indicating that delamination has occurred inside the YBCO layer.
Therefore, the delamination behavior of the CC might be a beneficial factor and result in new application potential. There are more tasks to investigate in our future studies: first, the reason for the decreasing $I_c$ is still not clear, which could guide us to a higher percentage of $I_c$ preservation; second, post treatment, such as annealing, could be used to improve the properties of the delaminated sample; third, the delamination of other types of HTS CCs might be different from the samples in this study.

5. Conclusion

In this study, coated conductors manufactured via the MOD route were delaminated by liquid nitrogen immersion. The $I_c$ distributions of the original sample and the delaminated sample were measured by a scanning Hall probe system. It was found that $I_c$ survived in the delaminated sample, with a preserved percentage of about 50% relative to the original. The four-probe method was utilized to verify the $I_c$ result and the validity of current transportation. SEM observations, EDS detections and XRD analysis were conducted to obtain more information about the delaminated surfaces, and the location of lamination occurrence was inside the YBCO layer and near the YBCO-buffer interface. The potential application of the delaminated sample in superconducting joint technology was discussed based on an estimation of oxygen diffusion, and a short oxygenation duration is expected. Further study of the $I_c$ survival in the YBCO layer of the delaminated coated conductor will be carried out in our future research.

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