

Atom-replaced pins in a Y-based superconductor—single-crystalline perovskite structure including both $\text{PrBa}_2\text{Cu}_3\text{O}_{7-x}$ and $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$

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Abstract

Metal organic deposition using trifluoroacetates (TFA-MOD) provides many uniform superconductors on long metal tapes. The large numbers of long wires have been applied for power grids or superconducting fault current limiters. The related applications worked for a long time without fatal trouble. The quasi-liquid produced during the firing process assisted the perfectly uniform structure in TFA-MOD. On the other hand, when it was desired to introduce artificial pinning centers, the quasi-liquid also enlarged the diameter of the artificial pinning centers to several tens of nanometers. In other words, due to the nature of TFA-MOD, there is very little chance of using TFA-MOD to prepare several nm-sized artificial pinning centers. By proposing atom-replaced pins (ARPs), we aim to overcome the impasse. ARPs are realized by replacing yttrium (Y) with praseodymium (Pr) whose valence number changes from 3+ to approximately 4+. Analytical results suggested that Pr makes pinning centers on a $\text{PrBa}_2\text{Cu}_3\text{O}_{7-x}$ (PrBCO) unit cell, and the weak-linked superconductivity derived from PrBCO extends to the adjacent unit cells in the a/b -plane. J_c decrease by Pr is five times as large as the volume fraction of Pr in the Y-site of the perovskite structure. On the other hand, T_c does not show large degradation in YBCO including 10% PrBCO. These results suggest that PrBCO unit cells are fully dispersed in $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ matrix. With regard to J_c in the magnetic field, ($\text{Y}_{0.98}\text{Pr}_{0.02}$)BCO has been slightly improved compared with pure YBCO only in the region of high temperature and low magnetic field of less than 1 T.

Keywords: superconductor, YBCO, PrBCO, TFA-MOD, atom-replaced pin, J_c - B property, partially stabilized solvent-into-gel

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

Introduction

As second-generation superconducting wires, Y-based coated conductors prepared by metal organic deposition using trifluoroacetates (TFA-MOD) has uniformity in long length [1]. The superconducting wires prepared by TFA-MOD have been applied for power grids [2] and superconducting fault

current limiters [3]. However, $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ (YBCO) wires prepared by TFA-MOD have rarely been applied for coil applications in high magnetic fields, because their superconducting properties in high magnetic fields are insufficient. Such low properties in magnetic fields are attributable to the large size of the pinning centers [1]. Although the ideal pin size at 30 K is considered to be about 5 nm in diameter, the

pin size fabricated by TFA-MOD is reported to grow to over 30 nm because the growth process is a liquid phase [4]. Therefore, many groups have tried to control the pin size with a view to improving the superconducting properties in high magnetic fields [5–8].

On the other hand, in the case of physical vapor deposition such as metal organic chemical vapor deposition [9], pulsed laser deposition [10] could restrict the size of pinning centers. In the process, BaZrO₃, BaHfO₃ and BaSnO₃ (BMO) are generally used for the pinning centers. In the case of physical vapor deposition, some of the pinning centers, such as those composed of Y₂O₃ or Dy₂O₃, grow larger. Since these materials have no perovskite structure, the particles become larger during the growth process through merger with other particles. However, when the pinning material has the same perovskite structure as a matrix superconductor, the growth process of the material is restricted by the surrounding YBCO superconductor unit cell, which has the same perovskite structure. Consequently, a BMO pin of small size could be realized. Although superconductors having BMO pins controlled to about 5 nm in diameter have excellent critical current density (J_c) properties in the magnetic field, they also have degraded critical temperature (T_c) [11]. This T_c degradation is attributable to lost oxygens of YBCO, caused by a direct connection to BMO. Degraded T_c values might be attributable to inhomogeneity of the YBCO structure. Such inhomogeneity might be one of the reasons for unstable operation in Y-based superconducting coil applications in high magnetic fields. Therefore, our objective is fabrication of YBCO films having a mostly oriented perovskite structure, such as single crystal including pinning centers, by TFA-MOD. The structure would not cause great T_c degradation.

Among rare-earth (RE) atoms forming the REBa₂Cu₃O_{7-x} perovskite structure, praseodymium (Pr) is the only atom reported to be a non-superconducting material [12–15]. It is difficult to obtain a coating solution for PrBa₂Cu₃O_{7-x} (PrBCO) by TFA-MOD, because Pr salt easily reacts with methanol by esterification between carboxylic acid and alcohol. This active chemical nature derives from the large ionic diameter of Pr. In the present work, we obtained YBCO coating solution including Pr by suppressing the above-mentioned chemical reaction. We report YBCO films containing PrBCO unit cells and the J_c - B properties of the film at 30–77 K.

Methods

Pr acetates, yttrium (Y) acetates, barium (Ba) acetates and copper (Cu) acetates in a 0.10:0.90:2.00:3.00 ratio were dissolved into purified and deionized water to realize a transparent blue solution. The blue solution reacted with chemically stoichiometric trifluoroacetic acid (TFA) to yield M-TFA (M = Pr, Y, Ba and Cu), water and acetate solution. The mixed solution was purified by the refluxing method to realize a blue gel including impurities of water and acetate. The impurities were mostly replaced with methanol to realize a blue gel again [16], to yield CS-10%Pr coating solution

(coating solution including 10%Pr). The method of purifying coating solution is reported in detail elsewhere [17]. We also prepared other ratios of coating solution in Pr acetates and Y acetates: 0.00:1.00 (pure Y), 0.03:0.97, 0.06:0.94 and 0.08:0.92. The coating solutions are denoted as CS-YBCO, CS-3%Pr, CS-6%Pr and CS-8%Pr, respectively. The total metal ionic concentration of each coating solution was 1.50 mol l⁻¹.

For each coating solution, gel films were deposited on 10 mm × 10 mm or 10 mm × 28 mm (100) LaAlO₃ single crystals with a spin coater. The maximum spinning rate was 2000 or 4000 rpm. All acceleration rates were 10 000 rpm s⁻¹. All gel films were calcined at 100–400 °C with 4.2% (relative humidity) humidified oxygen gas. Trifluoroacetates were decomposed to realize oxyfluoride at between 200 and 250 °C [18]. Elapsed time at between 200 and 250 °C was 9 h and 43 min. After the decomposition of organic materials, we increased the temperature to 400 °C. The procedure is reported in detail in [18]. The calcined films were fired at 800 °C with 4.2% humidified argon gas mixed with 1000 ppm oxygen gas to yield a tetragonal perovskite structure. The perovskite oxide was annealed below 525 °C in dry pure oxygen gas to form an orthorhombic structure.

X-ray diffraction (XRD) measurements (the two theta vs the omega and rocking curve methods) were performed on the fired film to evaluate the diffraction peaks and the crystallinity. T_c and J_c were measured by a non-destructive inductive method [19–21]. In the method, the third harmonic wave is detected as a voltage. A signal of 50 μV corresponds to 1 μV cm⁻¹, which is the standard criterion to decide J_c of a superconductor. Cross-sectional images were observed by transmission electron microscopy (TEM) to detect other phases on the films or amorphous layers in the films. Depth profiles of metal content from the film surface toward the substrate were measured by secondary ion mass spectroscopy. The sputtering rate was 0.3 nm sec⁻¹. To measure the superconducting properties in a magnetic field, we prepared films on 10 × 28 mm LaAlO₃ single crystals. We formed four silver electrodes on the film by the electron beam method in vacuum. The distance between voltage electrodes was 10 mm. Superconducting properties in high magnetic field were measured in an 18 T superconducting magnet at the National Institute for Materials Science. The magnetic field was 0.1–15 T.

Results and discussion

Preparation of coating solution including Pr-TFA

Pr is the only atom that forms the REBa₂Cu₃O_{7-x} non-superconducting perovskite structure in RE elements [12–15]. In synthesizing the coating solution including Pr-TFA, a problem arises in that Pr-TFA is easily decomposed with alcohol solvent. One of the decomposed contents is metal hydrogen oxide. The hydrogen oxide immediately changes into metal oxide, which disturbs the chemical reaction in TFA-MOD. For this method, methanol is usually used as an

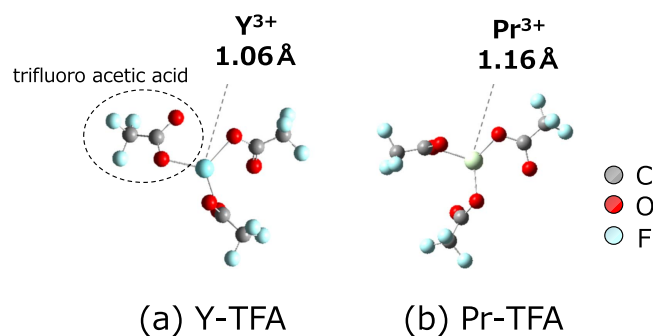


Figure 1. Chemical structure of (a) yttrium trifluoroacetates (Y-TFA) and (b) praseodymium trifluoroacetates (Pr-TFA). The structures were calculated using the Gaussian09 program. A long distance between the center metal and trifluoroacetic basis causes an active chemical nature.

alcohol solvent. If heavier alcohol (such as ethanol or 1-propanol) is used to prevent esterification between alcohol and acetate, the resulting fired film has high carbon content. The carbon content remains as CO_x in Cu-O superconducting planes [22]. If a heavier perfluorocarboxylic acid (such as pentafluoropropionic acid or heptafluorobutyric acid) is used [23], the esterification is usually suppressed in the case of Sm and Nd. In the case of Pr salts, however, it cannot be suppressed. It is reasonable to assume that if Pr-TFA and Y-TFA are mixed in methanol solvent, Pr-TFA simply reacts with alcohol and is decomposed because Pr³⁺ has a larger diameter and is more chemically reactive than Y³⁺, as shown in figure 1 [24]. However, considering the chemical structure of Pr-TFA and Y-TFA, large volumetric TFA ligands surround RE metals of Pr and Y. A mixed solution including Pr-TFA and Y-TFA may modify the esterification condition.

Based on the above idea, we prepared a coating solution in which Y-TFA is partially replaced by Pr-TFA. We firstly succeeded in obtaining Y-TFA mixed with 3% Pr-TFA coating solution. The decomposing condition seems to be determined by the average diameter of Pr³⁺ and Y³⁺. Individual Pr-TFA molecules may react with alcohol to become a decomposed material. However, the above-mentioned mixed material avoids the esterification. We also prepared 10% Pr-TFA mixed with Y-TFA coating solution. In the case of this solution, no precipitation occurred for over two months. The reason for the suppression of esterification has yet to be clarified. We intend to investigate these matters in the near future. Pr has a large ionic diameter and is chemically reactive. If the coating solution includes other materials, such materials may decrease activation energy of the esterification. Therefore, it is necessary to purify the coating solution containing Pr ion. We named this the partially stabilized solvent-into-gel (SIG) method—the PS-SIG method.

Fabrication of (Y, Pr)Ba₂Cu₃O_{7-x} films

We obtained Y-TFA mixed with 10% Pr-TFA coating solution as reported above. With the coating solution, we wanted to establish PrBCO-dispersed YBCO structure in the common perovskite structure. We obtained YBCO film having 10% PrBCO. We measured the film by the XRD 2theta-omega

method. Figures 2(a) and (b) show results of YBCO and YBCO mixed with PrBCO, respectively. Figure 2(a) shows typical YBCO (00n) peaks in 2theta-omega measurements of XRD. The strongest peaks at 2θ = 24° and 47° were LaAlO₃ (100) and (200). Although figure 2(a) shows other phases of Ba₂CuO₃ (200), (400) and (600) at 2θ = 14°, 28° and 42°, and Y₂Cu₂O₅ (400) at 2θ = 34°, the intensities of these peaks were much weaker than that of YBCO (00n). If the film has no amorphous phases, the XRD measurement confirms an almost perfectly oriented structure. Figure 2(b) shows the XRD result of YBCO mixed with 10% PrBCO. Figure 2(b) also shows typical REBCO (00n) peaks and is similar to figure 2(a). According to Araki *et al*, YBCO (006), SmBCO (006) and NdBCO (006) peaks appear at around 46.68°, 46.53° and 46.46°, respectively [23]. The diameter of the RE ion exhibits a tendency of ‘lanthanide contraction’ [13]. If PrBCO alone forms a perovskite structure, the PrBCO (006) peak is estimated to appear at around 2θ = 46.3 from the tendency of ionic diameter. If the film shown in figure 2(b) includes the separated PrBCO layer or structure, then the figure must show the PrBCO (006) peak at approximately 2θ = 46.3°. However, figure 2(b) seems to show that the film has a single-crystalline structure. Therefore, it is assumed that PrBCO is fully dispersed in the YBCO matrix unit cell by unit cell. We wanted to check whether an amorphous layer or structure exists in YBCO mixed with 10% PrBCO film.

We observed YBCO mixed with 10% PrBCO film by TEM and the results are shown in figure 3(a) (low magnification, ×200 000) and (b) (high magnification, ×4300 000). Figure 3(a) shows a fully oriented structure from the boundary with substrate to the top of the film. We could detect no amorphous layer or structure. Therefore, we assume that PrBCO and YBCO form a single perovskite structure. Figure 3(b) shows a high-resolution TEM image of the film. We could see only an almost perfectly oriented structure in the image. The brightest line in the horizontal direction is Y or Pr, and the other three dot-lines are Cu. The area indicated by open arrow shows slight disorder in the perovskite structure. We believe the slight disorder derives from PrBCO, because the lattice constant of PrBCO is different from that of YBCO. The PrBCO unit cell must have a larger lattice constant than that of YBCO at approximately 800 °C during the firing process. Because of volumetric anisotropy, a large PrBCO unit cell has a low possibility of growing next to another PrBCO unit cell. Therefore, we assume that PrBCO unit cells are fully dispersed. Additionally, we measured the depth profile of metal concentration by secondary ion mass spectroscopy to gain evidence of the existence of Pr. Figure 4 shows the metal depth profile from the surface. The concentration of Al and La included in the substrate increased at around 140 nm from the top of the film, and that of Cu and Y included in the superconductor decreased at the same depth. The film thickness is presumed to be approximately 140 nm. The concentration of Pr from 0 to 140 nm is larger than that at over 140 nm. The superconducting film must include the Pr element. Figures 2–4 confirm PrBCO and YBCO unit cells coexist in a common perovskite structure. The optimal oxygen partial pressure (*p*(O₂)) for YBCO at 800 °C is reported to

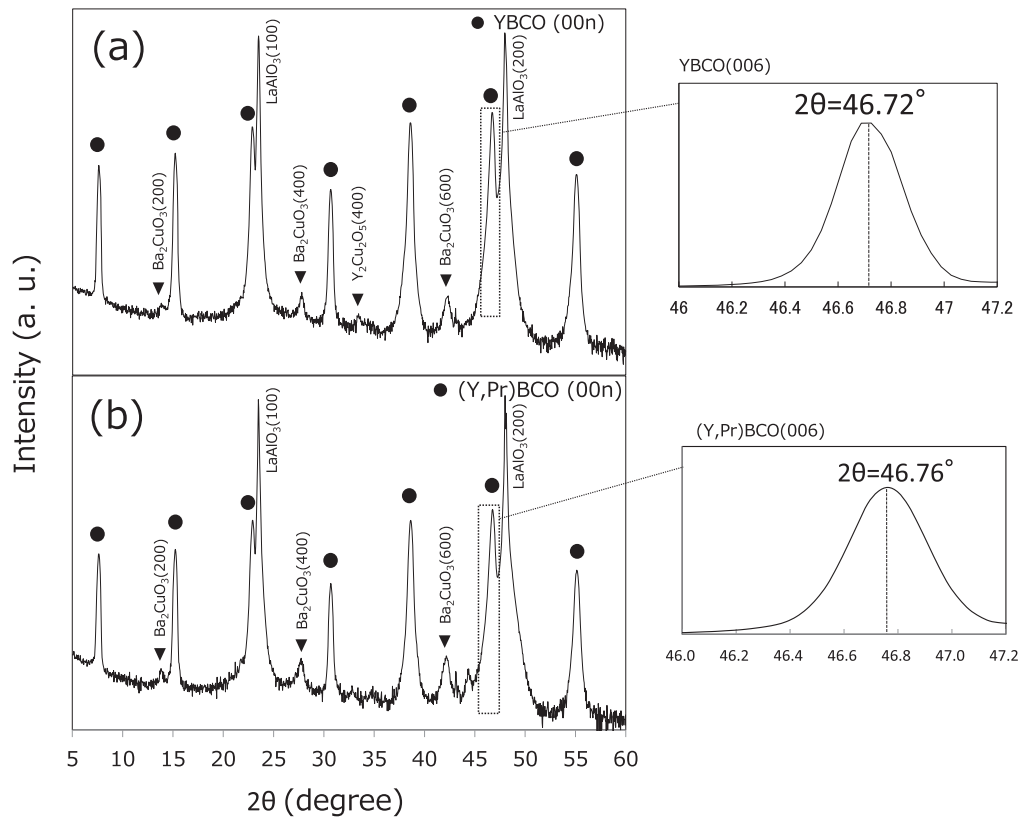


Figure 2. XRD diffraction peaks of (a) $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ and (b) $(\text{Y}_{0.9}, \text{Pr}_{0.1})\text{Ba}_2\text{Cu}_3\text{O}_{7-x}$. Peaks in (b) imply single-crystalline structure.

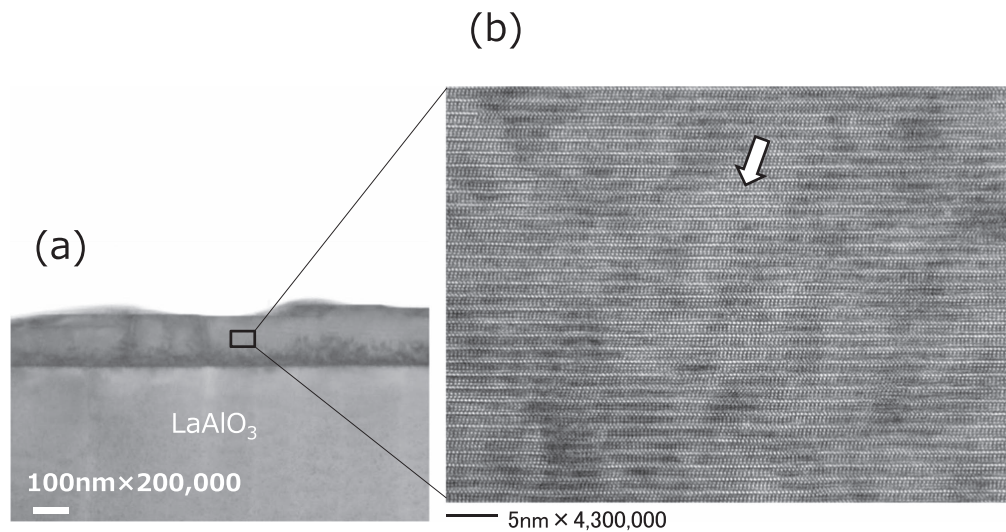


Figure 3. Cross-sectional TEM observation of $(\text{Y}_{0.9}, \text{Pr}_{0.1})\text{BCO}$ film. Magnifications of (a) and (b) are $\times 200\,000$ and $\times 4\,300\,000$, respectively. Although well-aligned structure is confirmed from (b), the local Cu position indicated by open arrow is influenced by atom-replaced pins (ARPs).

be 1000 ppm [25]. This $p(\text{O}_2)$ is the same value for various deposition methods. The optimal $p(\text{O}_2)$ depends on ionic diameter in the perovskite structure. In the case of lanthanum metals, the ionic diameter tends to follow the lanthanide contraction. Optimal $p(\text{O}_2)$ for La, Nd and Sm are considered to be 0.2, 5 and 20 ppm, respectively. Assuming the tendency of the ionic diameter, the optimal $p(\text{O}_2)$ of Pr must be approximately 1 ppm. The optimal $p(\text{O}_2)$ for YBCO is 1000 times larger than that of PrBCO. Generally speaking, the

coexistent structure of PrBCO and YBCO is almost impossible to prepare by physical vapor deposition because of the above-mentioned difference. However, TFA-MOD could provide the coexistent structure. Although the detail of the reason is obscure, the optimal $p(\text{O}_2)$ seems to be the average value in TFA-MOD [23]. In this study, we fixed the $p(\text{O}_2)$ at 1000 ppm. We consider the purified mixed coating solution to be a key to realizing the coexistent structure of PrBCO and YBCO. If an impurity is included in the coating solution, the

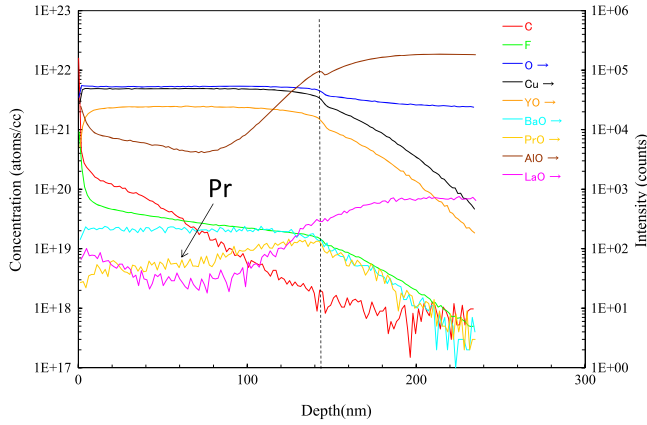


Figure 4. Secondary ion mass spectroscopy depth profile of $(Y_{0.9}, Pr_{0.1})BCO$ film from the film surface. Film thickness is roughly assumed to be 140 nm. The result shows existence of Pr content within the film.

impurity disturbs the well-aligned structure and forms other phases. To yield the coexistent structure, we have to obtain purified coating solution including Pr-TFA. We partially added the Pr acetate instead of Y acetates and the large amount of Y-acetate prevents decomposition of Pr-TFA. This PS-SIG method efficiently realizes the structure.

The superconducting properties of $(Y, Pr)Ba_2Cu_3O_{7-x}$ films

PrBCO is known to form a perovskite structure and to have no superconductivity between 4 K and 77 K. The valence number of Pr at firing temperature must be $3+$. The perovskite structure formed is evidence of the Pr^{3+} . As Tarascon *et al* reported, Pr has a different valence number at low temperature and its perovskite structure has no superconductivity [13]. Alleno *et al* additionally reported that the valence of Pr changes according to the ratio of Pr in $(Y, Pr)BCO$ [26]. In general, Pr is reported to have Pr^{3+} or Pr^{4+} . Thus, we presume that Pr has a valence number of about $4+$ at low temperature, such as at 4 K or 77 K. If the above presumption is right, the larger PrBCO unit cell at the firing temperature of $800^\circ C$ will shrink to a smaller one at low temperature. There are two reasons for the shrinkage: one is that the ionic diameter of Pr^{4+} is only 87% of that of Pr^{3+} [27] and the other is that low-occupation-ratio oxygen sites in the perovskite structure are filled with additional oxygen atoms because of the valence number change of Pr. Such additional oxygen atoms yield an attraction force in the c -axis direction with two Cu^{2+} ions. We measured c -axis length dependence on the ratio of Pr in Y-site and show the result in figure 5. As we expected, c -axis lengths become shorter in film having a relatively large amount of Pr. We also measured the rocking curve ($\Delta\omega$) and figure 6 shows the results. The value of $\Delta\omega$ slightly increases with the ratio of Pr and the increased $\Delta\omega$ indicates low crystallinity. The low crystallinity derives from the shrinkage of PrBCO unit cells.

We measured the J_c dependence at 77 K, 0 T on the ratio of Pr and the results are plotted in figure 7. Although we expected J_c degradation to be proportional to the volume fraction of PrBCO (broken line), J_c degradation is

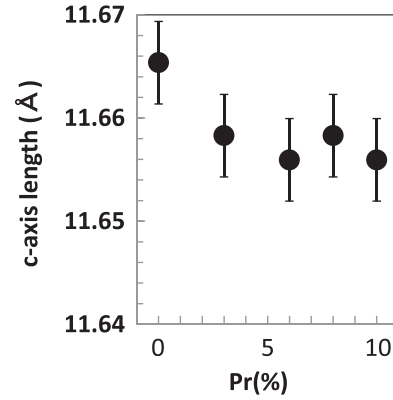


Figure 5. Dependence of c -axis length of $REBa_2Cu_3O_{7-x}$ on the ratio of Pr content in Y-site. The length became shorter than that of pure YBCO film.

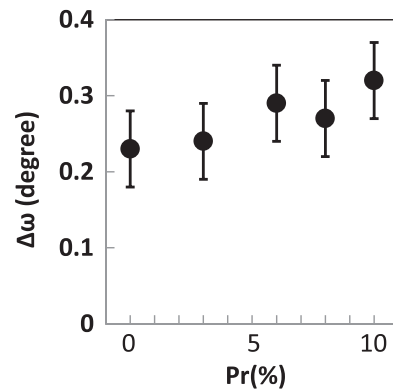


Figure 6. Dependence of delta omega of $REBa_2Cu_3O_{7-x}$ on the ratio of Pr content in Y-site. $PrBa_2Cu_3O_{7-x}$ may cause low crystallinity.

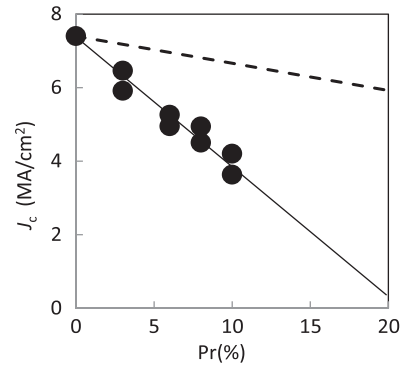


Figure 7. J_c degradation of Pr ratio. J_c decreases with five times as large $PrBa_2Cu_3O_{7-x}$ volume fraction.

approximately five times as large as the ratio of Pr. As reported in the literature on Josephson junctions, PrBCO surrounded by YBCO causes superconductivity to be weak-linked [28, 29]. Therefore, the addition of PrBCO changes the condition of the superconductivity and degrades J_c values. We consider that the PrBCO unit cell influences the adjacent YBCO unit cells. The adjacent unit cells in the a/b -plane seem to be strongly influenced by the shrinkage, because the attraction force of the central one-third perovskite PrBCO unit cell directly influences the adjacent one-third perovskite cells.

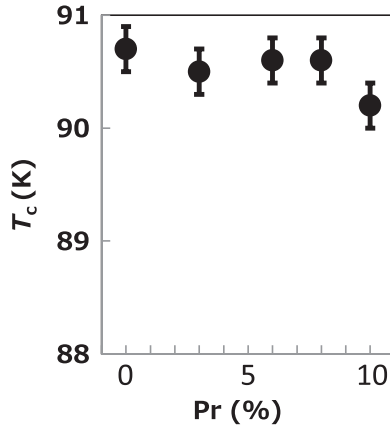


Figure 8. T_c dependence of the film on Pr content. 8% Pr corresponds to about 40% weak-linked superconducting volume.

This is so even if the one-third unit cell adjacent to the PrBCO unit cell has Y as the center metal. In the c -axis direction, the adjacent Y-site is located far from Pr one-third cell. Between the Pr of one-third unit cell and the adjacent Y of one-third unit cell, there are two additional Ba of one-third cells. Therefore, the shrink force does not reach the adjacent Y of one-third cells in the c -axis direction. The shrunk PrBCO unit cell seems to be fully dispersed in the YBCO structure, because five times degradation is maintained till 10% Pr. We call this phenomenon, the ‘five times degradation phenomenon’.

T_c dependence on the Pr ratio is plotted in figure 8. T_c were maintained below 8% Pr. YBCO film with 10% Pr in Y-site has slightly degraded T_c . According to the ‘five times degradation phenomenon’, 10%-Pr-incorporated YBCO films have about 50% weak-linked-superconducting areas. Such a large ratio of weak-linked-superconducting areas slightly degrades T_c at 10% Pr in figure 8.

PrBCO unit cell located in YBCO matrix causes weak-linked superconductivity. The weak-linked superconductivity derived from PrBCO also suppresses the superconductivity of surrounding adjacent unit cells in the a/b -plane. However, considering the T_c result, the suppression area is not large and considered to be about 1.2 nm in diameter, which is about three times as large as the unit cell size of 0.4 nm for REBCO in the a/b -plane.

Measurement of J_c in the magnetic field

We achieved ARPs as discussed above and measured J_c - B properties as shown in figure 9. The film has 2% Pr in Y-site and about 10% weak-linked-superconducting area. J_c - B of the film shows no improvement compared with the data of YBCO in almost all conditions. However, only in the case of 77 K and low magnetic field of less than 1 T, J_c - B properties are slightly improved compared with those for pure YBCO. This might show the potential of PrBCO as a pinning center. In order to improve J_c - B properties, it is necessary to control and optimize the pin size of the weak-linked-superconducting area to the ideal size by another clustering technology. We intend to report the clustering technology in the near future.

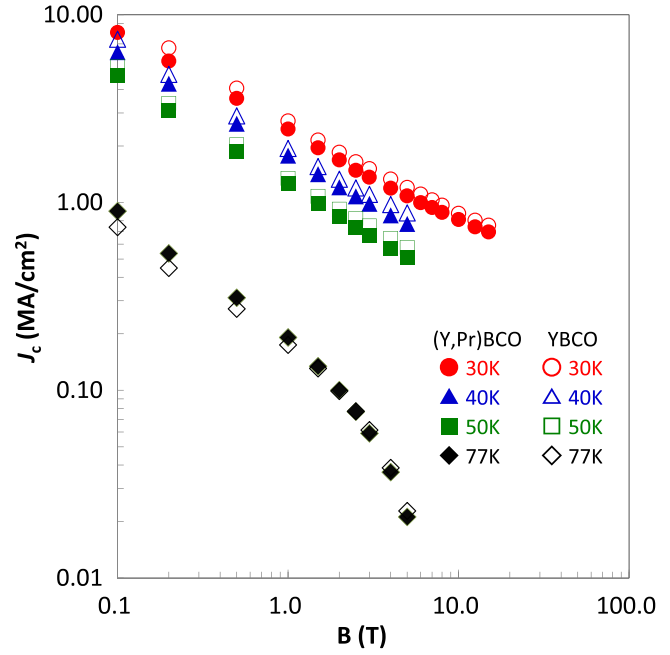


Figure 9. J_c - B properties of $(\text{Pr}_{0.02}, \text{Y}_{0.98})\text{BCO}$ film at 30, 40, 50 and 77 K with closed symbols. Compared with open symbols of YBCO, J_c - B properties slightly degrade in almost all conditions.

Conclusion

Praseodymium trifluoroacetates (Pr-TFA) are easily decomposed in methanol coating solution by esterification. However, a mixed coating solution including yttrium trifluoroacetates and Pr-TFA could suppress the decomposition. The purifying process is the partially stabilized solvent-into-gel (SIG) method. $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$ (YBCO) mixed with $\text{PrBa}_2\text{Cu}_3\text{O}_{7-x}$ (PrBCO) film was detected as a single phase by x-ray diffraction. No other phases and no amorphous layers were detected by transmission electron microscopy; the praseodymium content was detected by secondary ion mass spectroscopy. These results indicate that PrBCO exists in the common perovskite structure of YBCO. Praseodymium has a valence number of $3 +$ at 800 °C and the number changes to almost $4 +$ at lower temperatures. The perovskite unit cell including Pr shrinks and causes weak-linked superconductivity and suppresses the superconductivity of the surrounding unit cells adjacent to the PrBCO unit cell in a/b -plane. Critical current density (J_c) decreases with five times as large PrBCO volume fraction. This is the ‘five times degradation phenomenon’. The critical temperature of YBCO mixed with PrBCO has almost the same value of 90.7 K below 8% Pr. 8% Pr corresponds to about a 40% weak-linked-superconducting area. Therefore, the area of suppressed superconductivity in unit cells adjacent to PrBCO in the a/b -plane is considered not to be large.

Fully dispersed PrBCO has no effect on J_c improvement in high magnetic field. By using another clustering technology, we intend to control the pin size in order to improve the J_c - B properties.

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