Improvement of critical current density of \( \text{REBa}_2\text{Cu}_3\text{O}_{7-\delta} \) by increase in configurational entropy of mixing

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\( \text{REBa}_2\text{Cu}_3\text{O}_{7-\delta} \) (RE123, RE: rare earth) is one of the high-temperature superconductors with a transition temperature \( T_c \) exceeding 90 K. Because of its high-\( T_c \) and large critical current density \( J_c \) under magnetic fields, RE123 superconductors have been expected to play a key role in superconductivity application. To accelerate application researches on RE123-based devices, further improvements of \( J_c \) characteristics have been desired. In this study, we investigated the effects of high-entropy alloying at the RE site on the superconducting properties, through the measurements of local (intra-grain) \( J_c \) \( (J_{\text{local}}^c) \) by a remanent magnetization method. We found that \( J_{\text{local}}^c \) shows a trend to be improved when four or five RE elements are mixed at the RE site, which results in high configurational entropy of mixing \( (\Delta S_{\text{mix}}) \). All samples exhibited an order of few MA cm\(^{-2} \) which is a criterion for practical application and the highest \( J_{\text{local}}^c \) resulted in a value of around 7.0 MA cm\(^{-2} \) at \( T = 2.0 \) K. Because high-entropy alloying can improve \( J_{\text{local}}^c \) of RE123 superconductors, our entropy-engineering strategy introduced here would be useful for the development of RE123 superconducting materials available under high magnetic fields.

1. Introduction

Since the discovery of high-transition temperature (high-\( T_c \)) Cu-oxide superconductor [1], various kinds of Cu-oxide superconductors have been discovered [2–5]. Among them, \( \text{REBa}_2\text{Cu}_3\text{O}_{7-\delta} \) (RE123, RE: rare earth) in a thin-film form is a promising material for high-field superconductivity application.
because of its high-$T_c$ exceeding 90 K and high critical current density ($J_c$) under magnetic fields [6]. To improve $J_c$ of $RE$123 films, nanoscale disorders such as nanoparticles, nanocomposite structure, defects etc. were introduced [7–10]. However, the current record of $J_c$ of $RE$123 films are far from the limit expected for an ideal $RE$123 material [11]. To achieve higher $J_c$ in $RE$123 materials, further development of the method for $J_c$ engineering is needed. Having considered the structural and physical properties of various Cu-oxide superconductors, we find that the $RE$123 system is relatively difficult to use in superconductivity applications because it contains structural and compositional fluctuations. High-$T_c$ and high-$J_c$ superconductivity of $RE$123 is generally observed in the orthorhombic structure with the space group of $Pmmm$ (#47) [12]. Decrease in oxygen amount in $RE$123 in the blocking layer results in a decrease in hole carriers and suppression of high performance of $RE$123. Recently, the improvement of $J_c$ in $RE$123 film was achieved by over-doping of holes [13]. The improvement of $J_c$ by chemical-composition tuning would be a desired progress because that can be applied together with the nanoscale fabrication techniques mentioned above. Here, we show another strategy to improve local (intra-grain) $J_c$ ($J^{\text{local}}_c$) by introducing high-entropy-alloy-type (HEA-type) $RE$ site in $RE$123 superconductor.

HEA is an alloy containing five or more elements with a concentration range between 5 and 35 at% and hence has a high configurational entropy of mixing ($\Delta S_{\text{mix}}$), which is defined as $\Delta S_{\text{mix}} = -R \sum c_i \ln c_i$, where $c_i$ and $R$ are compositional ratio and the gas constant, respectively [14,15]. Although the field of HEA had mostly focused on structural materials for the use under extreme conditions, various functionalities have been found in HEAs [15,16]. In 2014, superconductivity was observed in a HEA, Ti-Zr-Hf-Nb-Ta [17]. Although the expected pairing mechanisms of superconductivity for the HEA was a conventional type, the unique structural and compositional character were welcomed in the field of new superconducting materials. As reviewed in [18] and [19], many HEA superconductors were discovered after the first discovery by Koželj et al. [17]. Since 2018, we have developed HEA-type superconducting compounds, in which the HEA concept was applied to complicated compounds having two or more crystallographic sites [20]. Comparing the HEA effects for superconductors with various crystal structural dimensionality, we found that the disordering effects introduced by the HEA-type site in layered system (Bi$_2$$_2$$_2$$_2$-based superconductor) [21] and quasi-two-dimensional system (tetragonal $T$Z$_2$$_2$$_2$, $T_r$: transition metals) [22,23] does not suppress its original $T_c$ in pure phases. By contrast, in three-dimensional systems (NaCl-type metal tellurides [24,25] and A15 niobium-based compounds [26]), $T_c$ of HEA-type phases was clearly lower than that for pure phases. Therefore, in a two-dimensional crystal structure, the introduction of HEA-type site does not negatively work on $T_c$ of the superconductor. As a result, we previously reported the synthesis of HEA-type $RE$123 polycrystalline samples and reported that the increase in $\Delta S_{\text{mix}}$ does not suppress superconducting properties including $J_c$ [27]. Let us remind that the technique of mixing $RE$ elements has been used for the improvement of $J_c$ in some $RE$123 materials [28]. To the best of our knowledge, however, superconducting properties including $J_c$ for $RE$123 with remarkably high $\Delta S_{\text{mix}}$ at the $RE$ site was examined in [27] for the first time. In this study, we expanded the study and synthesized various samples of $RE$123 using lighter $RE$ elements including Dy, Ho, Yb and Lu. Here, we show that tuning $\Delta S_{\text{mix}}$ at the $RE$ site could improve intra-grain $J_c$ ($J^{\text{local}}_c$) of $RE$123 superconductors.

2. Material and methods

Polycrystalline samples of $RE$Ba$_2$Cu$_3$O$_{y\delta}$ (RE: Y, La, Nd, Sm, Eu, Dy, Ho, Yb and Lu) were prepared by solid-state reaction and all samples were prepared in air, as described in [26]. Powders of Y$_2$O$_3$ (99.9%), Sm$_2$O$_3$ (99.9%), Eu$_2$O$_3$ (99.9%), Dy$_2$O$_3$ (99.9%), Ho$_2$O$_3$ (99.9%), Yb$_2$O$_3$ (99.9%), Lu$_2$O$_3$ (99.9%), BaCO$_3$ (98%) and CuO (99.9%) were used for the synthesis. To obtain the best superconducting properties of each sample, two-step or three-step sintering was performed in air. Note that annealing in air at a low temperature around 350°C is performed because $T_c$ of $RE$123 decreases with lower oxygen amount. Three-step sintering was performed for all samples except for labelled RE-2 and RE-3 (table 1 for the composition and electronic supplementary material, table S1 for the condition). We also confirmed that sample which showed enough high-$T_c$ exhibited almost same superconducting properties after the three-step sintering. The raw chemicals with a nominal compositional ratio of $RE$: Ba : Cu = 1 : 2 : 3 were well mixed and pelletized with a diameter of 1 cm. The first sintering condition was 930°C for 20 h, followed by furnace cooling. For the second sintering process, the sample was ground, mixed, pelletized in the same manner as the first one and heated at 930°C for 8 h and 350°C for 18 h, followed by furnace cooling. The third sintering was performed in three-step sintering for samples
Table 1. Compositional, structural and superconducting properties of \( \text{REBa}_2\text{Cu}_3\text{O}_{7-x} \) samples examined in this study.

| label | \( \text{RE} \) site (EDX) | \( \Delta S_{\text{m}} \) (RE) | \( a \) (Å) | \( b \) (Å) | OP | \( T_c \) (K) |
|-------|-----------------------------|-----------------|---|---|---|---|
| RE-1  | Y                          | 0               | 3.81375(10) | 3.8804(2) | 0.0173 | 92.9 |
| RE-2  | \( Y_{0.25}\text{Nd}_{0.63} \) | 0.68R           | 3.85007(13) | 3.9126(2) | 0.0161 | 92.2 |
| RE-3  | \( Y_{0.35}\text{Sm}_{0.3}\text{Eu}_{0.31} \) | 1.09R           | 3.84149(10) | 3.9038(2) | 0.0161 | 93.2 |
| RE-4  | \( Y_{0.25}\text{Sm}_{0.2}\text{Eu}_{0.26}\text{Dy}_{0.2} \) | 1.37R           | 3.8340(2)  | 3.89927(10) | 0.0169 | 92.8 |
| RE-5  | \( Y_{0.25}\text{Sm}_{0.3}\text{Eu}_{0.17}\text{Ho}_{0.28} \) | 1.58R           | 3.8290(2)  | 3.89593(8)  | 0.0173 | 93.0 |
| RE-6  | \( Y_{0.16}\text{Sm}_{0.3}\text{Eu}_{0.15}\text{Ho}_{0.14}\text{Dy}_{0.27} \) | 1.76R           | 3.8163(3)  | 3.8858(12) | 0.0180 | 92.0 |
| RE-7  | \( Y_{0.15}\text{Sm}_{0.3}\text{Eu}_{0.12}\text{Dy}_{0.12}\text{Ho}_{0.31}\text{Yb}_{0.22}\text{Lu}_{0.12} \) | 1.91R           | 3.81933(14) | 3.89045(10) | 0.0185 | 91.4 |

which showed low \( T_c \) and shielding volume fraction (SVF) after the second sintering. The condition of the third sintering was 930°C for 8 h, 350°C for 18 h and 175°C for 12 h, followed by furnace cooling.

Powder X-ray diffraction (XRD) patterns were collected on MiniFlex-600 (RIGAKU), equipped with a D/ tex-Ultra high-resolution detector, with a Cu-Kα radiation by a conventional \( \theta-\phi \) method. Rietveld refinement was performed using RIETAN-FP [29]. Crystal structure images were drawn using VESTA [30]. The actual composition of the synthesized disc-shaped polycrystalline samples was investigated by energy-dispersive X-ray spectroscopy (EDX) on a scanning electron microscope (SEM), TM-3030 (Hitachi), with Swift-ED (Oxford). The compositions were estimated by averaging the EDX analysis result from four points on the surface of the examined samples. The obtained compositions are shown in electronic supplementary material, figure S1 with SEM images.

The superconducting properties were investigated using a superconducting quantum interference device magnetometer on MPMS-3 (Quantum Design). For the precise measurement, the disc-shaped samples were cut into the cube-shaped form with a typical size of \( 0.18 \times 0.17 \times 0.20 \text{ mm} \). The temperature dependence of magnetic susceptibility (4\( \pi \)) was measured after both zero-field cooling and field cooling with an applied field of approximately 10 Oe. The temperature dependences of susceptibility for all the samples are shown in electronic supplementary material, figure S2, and the field cooling with an applied field of approximately 10 Oe. The temperature dependences of susceptibility for all the samples are shown in electronic supplementary material, figure S2, and the estimated \( T_c \) is listed in table 1. All samples exhibited high-\( T_c \) and large SVF, indicating that the samples were oxygenated enough. To estimate \( J_c \) of cube-shaped samples, magnetization-magnetic field (\( M-H \)) loops were measured. From the obtained \( M-H \) loops, \( J_c \) was estimated using the Bean’s model [31]: \( J_c = 20\Delta M/(B(1-B/3A) \text{ (A cm}^{-2}) \text{), where} A \text{ and} B \text{ are lengths determined by sample shape, and} \Delta M \text{ is obtained from the width of the} M-H \text{ curve. Typical results on} J_c \text{ are plotted as a function of field in the electronic supplementary material, figure S3. To estimate} J_c, \text{ remanent magnetization (} m_0 \text{) was measured. Remanent magnetization is defined to be a residual magnetization after the applied magnetic field is turned off [32]. When the field is applied to the sample, two full penetration fields at} H_{p1} \text{ and} H_{p2} \text{ appear at lower field and higher field. When the sample is partially penetrated by the lower field, the contribution to remanent magnetization or the total trapped flux by the pinning centres is only located in the thin penetrated layer. The sample is first fully penetrated with an increasing field at} H_{p1}. \text{ As the applied field further raises, more flux lines get over the pinning barrier and enter the sample. The stronger the pinning strength or the higher the applied field, the more flux lines trapped. The trapped flux lines contribute highly to the remanent magnetization, which has a large value compared with the first full penetration field} H_{p1}. \text{ Therefore, the remanent magnetization sharply increases with increasing applied field until the second full penetration field} H_{p2}. \text{ The derivative of} m_R \text{ with maximum magnetic field} (H_m) \text{ is given by the following equation with respect to the three different} H_m \text{ conditions [33],} d m_R/d \log H_m = \left(3\pi R^3 H_m/4H_{p2}^2\right)\left(1-3H_m/2H_{p2}+7H_m/12H_{p2}^2\right): 0 < H_m \leq H_{p2}, \left(3\pi R^3 H_{p2}/8\right)/(4H_{p2}-6H_m/H_{p2}^2+3H_m^2/ H_{p2}^2-H_m^2/2H_{p2}^2): H_{p2} < H_m < 2H_{p2}, \text{ and} 0: H_m > 2H_{p2}. \text{ In the equation,} d m_R/d \log H_m \text{ exhibits a peak at} H_m = H_{peak} = (6-2\sqrt{2})H_{p2}/7. \text{ Therefore,} J_c \text{ can be calculated from} H_{p2} = J_c/m_R, \text{ where} r \text{ is an average grain size. The} H_m \text{ dependence of} d m_R/d \log H_m, \text{ was measured using a sequence for remanent magnetization measurements. First of all,} H_m \text{ was applied to the sample, and the magnetic field was set to zero, followed by magnetization measurement, which gives} m_R(H_m). \text{ The same measurements were performed at different maximum fields and temperatures. From the} H_m \text{ dependence of} d m_R/d \log H_m, \text{} H_{p2} \text{ was estimated from the peak position} H_{peak}. \text{ The cube-shaped samples were crushed into}

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d\log H_m = \frac{3\pi R^3 H_m}{4H_{p2}^2}\left(1-3H_m/2H_{p2}+7H_m/12H_{p2}^2\right): 0 < H_m \leq H_{p2},
\[
\frac{3\pi R^3 H_{p2}}{8}/\left(4H_{p2}-6H_m/H_{p2}^2+3H_m^2/H_{p2}^2-H_m^2/2H_{p2}^2\right): H_{p2} < H_m < 2H_{p2},
\[
\text{and} 0: H_m > 2H_{p2}.
\]
fine powders by grinding using mortar and pestle. The powders close to 20 µm were selected by micro sieves and used for remanent magnetization measurements. The diameter of powders was estimated using ImageJ [34].

3. Results and discussion

Figure 1a shows a schematic image of the crystal structure of HEA-type RE123 and the concept of HEA-type at the RE site. With increasing number of RE in the site, \( \Delta \text{SVF}_{\text{mix}} \) (ideal value) increases and exceeds 1.5 at% when five RE ions are mixed. Therefore, RE123 samples with more than four RE ions are expected to be HEA-type RE123. In figure 1b, inter-grain global \( J_\text{c}^{\text{global}} \) estimated from the M-H loops is plotted as a function of orthorhombicity parameter \( (\text{OP} = 2|a - b|/(a + b)) \). Here, OP was used to discuss the superconducting properties because, as mentioned earlier, orthorhombicity is a key structural parameter to achieve higher superconducting characteristics in the RE123 system [12,27]. Since OP is a good scale for estimating oxygen deficiency in the RE123 system, the use of OP in plotting superconducting properties is useful to extract the effects of \( \Delta \text{SVF}_{\text{mix}} \) at the RE site of REBa2Cu3O7-δ on the \( J_\text{c} \) characteristics in the system. According to the data in figure 1b, we found that higher \( J_\text{c}^{\text{global}} \)'s are achieved for samples containing four, six and seven RE elements. The results motivated us to study \( J_\text{c}^{\text{local}} \) of RE123 samples with different \( \Delta \text{SVF}_{\text{mix}} \) to clarify the effect of high configurational entropy of mixing to \( J_\text{c} \) characteristics. Then, seven samples having different \( \Delta \text{SVF}_{\text{mix}} \) and almost comparable OP were selected for this study (highlighted with a square in figure 1b). As well, high enough \( T_\text{c} \) and bulk nature of superconductivity of the sample was confirmed through M-T measurements as displayed in the electronic supplementary material, figure S1 after oxygenation. These indicating that the samples were oxygenated enough, and OP are high enough and not a critical parameter to consider for \( J_\text{c}^{\text{global}} \) and \( J_\text{c}^{\text{local}} \). According to the number of RE elements, examined samples are labelled RE-1–RE-7 (table 1).

Through EDX analyses for cube-shaped samples (see electronic supplementary material, figure S1), we confirmed that the actual composition of the examined samples is comparable to the nominal value, as summarized in table 1. Figure 2a shows powder XRD patterns for all the samples (RE-1–RE-7). All the peaks could be indexed with the orthorhombic RE123 model with a space group of \( Pmnm \) (#147). As displayed in figure 2b, no peak broadening was observed among seven samples, indicating that the increase in \( \Delta \text{SVF}_{\text{mix}} \) does not affect crystallinity of the polycrystalline RE123 samples. The major peaks shifted according to the average ionic radius at the RE site. The shift of peaks from RE-2 to RE-7 is originated from the systematic increase of RE element with smaller ionic radius such as Sm, Eu, Dy, Ho, Yb and Lu. As mentioned earlier, however, in the RE123 system, OP is the essential parameter for superconducting properties rather than lattice constants. We, therefore, estimated lattice constants, \( a \) and \( b \), using the Rietveld refinements. The typical refinement result with a reliability factor, \( R_{wp} = 5.8\% \), for RE-5 is shown in figure 2c, which shows that the orthorhombic
model can nicely reproduce the XRD patterns even for a HEA-type sample with five different RE. The estimated lattice constants and the calculated OP are summarized in the electronic supplementary material, table S1. To perform remanent magnetization ($m_R$) measurements, powders with similar diameter are prepared and observed by SEM. As shown in figure 3, the diameter of the powders was almost homogeneous, and the estimation of the average diameter was successful using ImageJ software. The estimated average diameter of powders for RE-1–RE-7 is summarized in table 2.

Figure 4 shows the results of the $m_R$ measurements at $T = 2.0, 4.2, 10.0, 20.0$ K plotted in a form of $d m_R/d\log H_m$ as a function of maximum applied field ($H_m$). Basically, we observed two peaks in the plots; the first peak observed at lower fields is originating from $J_{\text{global}}^c$. Since powder samples were used for the $m_R$ measurements, $J_{\text{global}}^c$ is reasonably quite low. The second peak at higher fields is originating from $J_{\text{local}}^c$. From the peak position ($H_{\text{peak}}$), $J_{\text{local}}^c$ was calculated using the equations $H_{\text{peak}} = (6 - 2\sqrt{2}) H_{p2}/7$ and $H_{p2} = J_{\text{local}}^c r$, where $r$ is average grain size, as described in Material and methods. The estimated $J_{\text{local}}^c$ at $T = 2.0, 4.2, 10.0, 20.0$ K for RE-1–RE-7 is plotted in figure 5 as a function of number of RE elements. At 20.0 K, there is no clear difference in $J_{\text{local}}^c$ among the seven samples except for slightly higher values for RE-1 and RE-4. At lower temperatures, the difference in $J_{\text{local}}^c$ becomes remarkable. At 4.2 K, $J_{\text{local}}^c$ of RE-3, RE-4 and RE-5 is higher than that of RE-1. Furthermore, at 2.0 K, $J_{\text{local}}^c$ of RE-4 and RE-5 is clearly higher than that of RE-1, RE-2 and RE-3. Our results on $J_{\text{local}}^c$ measured for the polycrystalline $RE_{123}$ powders suggest that optimization of $\Delta S_{\text{mix}}$ at the RE site can improve $J_{\text{local}}^c$ of $RE_{123}$ materials at low temperatures.

Having compared the OP parameter for RE-1, RE-4 and RE-5, we found that OP for those samples is almost the same. Therefore, the oxygen amount would be comparable in those samples. Therefore, the improvement of $J_{\text{local}}^c$ by the increase in $\Delta S_{\text{mix}}$ observed between RE-1, RE-4 and RE-5 would be originating from local structural modification by the disordered RE site and chemical bonds near the RE site. In BiS$_2$-based $RE(O,F)$BiS$_2$ superconductors, local structure modification in the conducting BiS$_2$ layers by the increase in $\Delta S_{\text{mix}}$ was observed [35]. Therefore, we expect that structural modulation was generated.
in the CuO2 planes by the introduction of middle- or high-entropy-alloy-type RE site. The highly disordered RE site and the bonds would modify electronic states and microscopic characteristics of superconductivity in the samples. The lower $J_{\text{local}}$ observed for RE-6 and RE-7 than that for RE-4 or RE-5 may be caused by huge disordered states. Although we have no clear scenario of the enhancement of pinning properties by the increase in $\Delta S_{\text{mix}}$, our current results encourage further studies on the relationship between configurational entropy of mixing and critical current properties in the RE123 materials. Synthesis of single crystals or thin films of HEA-type RE$_2$Ba$_2$Cu$_3$O$_{7-\delta}$ and investigation on local structure modulations and microscopic characteristics of superconductivity are needed to clarify the HEA effects in RE123.

4. Conclusion

Here, we reported the improvement of $J_{\text{local}}$ in REBa$_2$Cu$_3$O$_{7-\delta}$ by the increase in $\Delta S_{\text{mix}}$ at the RE site. Polycrystalline RE123 samples with different $\Delta S_{\text{mix}}$ were synthesized by solid-state reaction. Through
characterization of structural (lattice constants and $OP$), compositional and superconducting properties ($T_c$ and shielding fraction), seven samples labelled RE-1–RE-7 were chosen, and remanent magnetization measurements were performed on those samples. At higher temperatures ($T = 20.0$ K), clear difference in $J_{\text{local}}^c$ was not observed. At lower temperatures ($T = 2.0$ and 4.2 K), higher $J_{\text{local}}^c$ was observed for RE-4 and RE-5 with a higher $\Delta S_{\text{mix}}$ as compared with that for RE-1, RE-2, and RE-3 with zero or low entropy of mixing at the RE site. Although the results of the current work showed the merit of high-entropy alloying at lower temperatures only, there should be optimal conditions on constituent RE element, mixing ratio and $\Delta S_{\text{mix}}$, which will achieve higher $J_{\text{local}}^c$ at higher temperatures as well. If the trial was achieved, the HEA concept can be applied to all RE123 practical materials to additionally improve their critical current properties.

Ethics. Since our work is related to inorganic materials research, it is not relevant to our work.

Data accessibility. The datasets supporting this article have been uploaded as part of the electronic supplementary material [36].

Authors’ contributions. A.Y.: conceptualization, investigation, resources, visualization, writing—original draft and writing—review and editing; Y.S.: data curation and investigation; Y.M.: supervision and writing—review and editing.

All authors gave final approval for publication and agreed to be held accountable for the work performed therein.
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