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## Doping effects of Fe ion on magnetic anisotropy of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>v</sub>



T. Ugawa <sup>a</sup>, S. Horii <sup>a,\*</sup>, T. Maeda <sup>a</sup>, M. Haruta <sup>a,1</sup>, J. Shimoyama <sup>b</sup>

<sup>a</sup> School of Environmental Science and Engineering, Kochi University of Technology, Tosa-Yamada, Kami-shi, Kochi 782-8502, Japan

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#### ABSTRACT

We report magnetic alignment of  $YBa_2(Cu_{1-x}Fe_x)_3O_y$  (Fe-doped Y123, x=0-0.1) powders under modulated rotation magnetic fields (MRFs) and roles of Fe ion as a determination factor of magnetic anisotropy in Y123. The Fe-free and Fe-doped Y123 powder samples aligned in the MRF of 10 T showed two different orientation types of the hard axis in Y123 grains. From an X-ray rocking curve measurement for the magnetically aligned powder samples of the Fe-doped Y123, inplane magnetic anisotropy for Y123 grains with the hard axis parallel to the [110] direction was found to be higher than that for Y123 grains with the hard axis parallel to the [010] direction.

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#### 1. Introduction

High critical temperature  $(T_c)$  superconductors (HTSC) with two-dimensional CuO2 layers exhibit a stacked crystal structure alternating a superconducting CuO2 plane and a blocking layer along the c-axis direction. Practical REBa<sub>2</sub>Cu<sub>3</sub>O<sub>v</sub> (RE123; RE: rare earth elements) superconductors with  $T_c \sim 90 \text{ K}$  consist of a bilayer type of superconducting CuO<sub>2</sub> plane and a one-dimensional Cu-O chain as a blocking layer, and exhibit superior critical current properties under high magnetic fields at 77 K intrinsically. Due to this anisotropic crystal structure based on the layered structure, RE123 crystals also show anisotropic critical current densities  $(I_c)$ ;  $I_c(|/ab|) > I_c(|/c|)$  [1]. Furthermore, increase in misorientation angle at a grain boundary of Y123 bi-crystal seriously reduces  $J_c$ even in the critical current properties parallel to the inplane direction [2], which is probably due to the symmetry of the Cooper pairs in the cuprate-based HTSCs. Therefore, the formation of tri- or biaxially aligned microstructure is indispensable for the improvement of critical current properties for RE123.

It is well known that a standard tri- or bi-axial orientation technique is based on epitaxial growth, such as melt-solidification using a seed crystal [3] and thin film growth using a highly oriented substrate [4]. Recently, tri-axial magnetic alignment using a modulated rotation field (MRF) at room temperature has been

reported [5]. Our group has also reported magnetic tri-axial alignment for twin-free RE-based cuprate superconductor powders of  $Y_2Ba_4Cu_7O_y$  (Y247) [6] and REBa $_2Cu_4O_8$  (RE124) [7] as proof-of-principle. In principle, on a substance with uni-axial magnetic anisotropy, the first easy axis of magnetization is aligned parallel to a static field and the hard axis is aligned normal to a magnetically rotating plane [8]. Furthermore, as shown in Fig. 1, both first easy and hard axes can be simultaneously aligned normal to the  $\alpha$  and  $\gamma$  planes, respectively, under a modulated rotating field (MRF) containing both effects of static and rotating fields if tri-axial magnetic anisotropy of a substance is sufficient for the alignment. In the case of a substance with magnetization ( $\chi$ ) relationship of  $\chi_1 > \chi_2 > \chi_3$ , the  $\chi_1$ ,  $\chi_2$ , and  $\chi_3$  axes are aligned normal to the  $\alpha$ ,  $\beta$ , and  $\gamma$  planes, respectively (see Fig. 1).

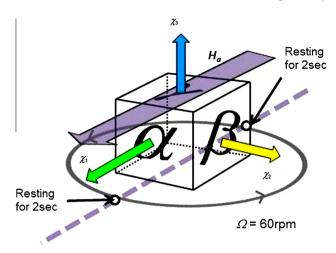
In the case of RE123, tri-axial magnetic alignment, magnetic separation of the a- and b-axes in particular, is predicted to be difficult in RE123 due to the formation of twin microstructure which weakens its inplane magnetic anisotropy in a grain level. The twin microstructure in RE123 is originated from a tetragonal-orthorhombic phase transition corresponding to an increase in the nonstoichiometric oxygen content [9,10]. Very recently, our group has exhibited the high potential of bi-axial magnetic alignment in "twinned" RE123 using fine powders processed by ball-milling [11]. In our previous report, Er123 powders showed two different types of inplane orientation: alignment of the [100] and [110] directions of Er123 grains was accomplished simultaneously. Although the development of tri- or bi-axial orientation techniques based on the magneto-scientific process in RE123 is important from the viewpoint of practical superconducting materials, the origin of the two different types of grain orientation has not been clarified vet.

<sup>&</sup>lt;sup>b</sup> Department of Applied Chemistry, University of Tokyo, Bunkyo-ku, Tokyo 113-8656, Japan

<sup>\*</sup> Corresponding author. Tel.: +81 887 57 2521; fax: +81 887 57 2520.

 $<sup>\</sup>label{lem:lemma$ 

<sup>&</sup>lt;sup>1</sup> WPI-Advanced Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan



**Fig. 1.** Experimental configuration of magnetic alignment in a modulated rotation magnetic field (MRF).

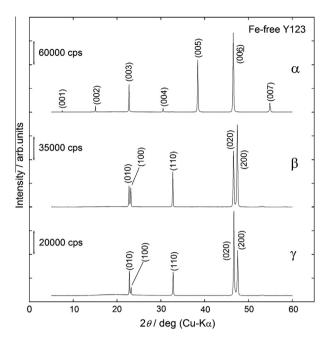
In the present study, in order to obtain a crystallochemical hint for the achievement of tri- or bi-axial alignment of twinned Y123 grains by MRF, we focused on Fe ion as a magnetic dopant into Y123 and clarified their magnetic axes and the degrees of orientation for Fe-doped Y123 powders aligned in various MRFs. Incidentally, Fe ion is preferably substituted for the Cu(1) site in the Cu-O chain [12–14], and the doping of Fe induces the structural change at the chain site [12–14]. Furthermore, this structural change leads to a structural transition to the tetragonal from the orthorhombic phase in RE123 and is expected to induce a substantial change in its twin microstructure.

#### 2. Experimental details

 $YBa_2(Cu_{1-x}Fe_x)_3O_v$  (Fe-doped Y123, x = 0-0.1) powders were synthesized by standard solid-state reaction, and were annealed at 300 °C in flowing oxygen gas to achieve fully oxidized states. Thus obtained powders were mixed with epoxy resin at a weight ratio of powder: resin = 1:10. The Fe-doped Y123 powders in epoxy resin were cured for more than 12 h at room temperature in modulated rotating magnetic fields. Details of the generation of MRF are described as follows. As shown in Fig. 1, the specimens were horizontally rotated at two different steps in a static magnetic field ( $H_a$ ). At the angles of  $0^{\circ}$  and  $180^{\circ}$ , the powder sample was rested for 2 s, whereas the rotation process with  $\Omega$  = 60 rpm was applied at the other angle regions. Here, the original angle, 0°, of the sample was defined with regard to a direction normal to the  $\alpha$  plane of the sample, which was parallel to the transverse  $H_a$  direction. The applied static magnetic fields were  $\mu_0 H_a = 1$ , 5, and 10 T. The first easy, secondary easy and hard axes of magnetization were determined from XRD patterns at the  $\alpha$ ,  $\beta$ , and  $\gamma$  planes of the magnetically aligned powder samples, respectively. The degrees of inplane orientation were evaluated from X-ray rocking curves (XRCs) for the (200), (020) and (110) peaks at the  $\beta$  and  $\gamma$  planes, and the degrees of c-axis orientation were determined from XRCs for the (005) peaks at the  $\alpha$  plane.

#### 3. Results and discussion

Fig. 2 shows XRD patterns at the  $\alpha$ ,  $\beta$ , and  $\gamma$  planes for a Fe-free Y123 powder sample (x = 0) aligned in the MRF of 10 T. Clearly, only (00l) peaks were enhanced at the  $\alpha$  plane, indicating that the first easy axis of Y123 is the c-axis. When one focused on the results of XRD patterns at the  $\beta$  and  $\gamma$  planes, (110), (h00), and (0k0) peaks were enhanced. Here, the (110), (h00), and (0k0)



**Fig. 2.** XRD patterns at the  $\alpha$ ,  $\beta$ , and  $\gamma$  planes for a Y123 powder sample aligned in the MRF of 10 T.

peaks are derived from a [110] direction, the a-axis, and the b-axis, respectively. Therefore, the separation of the a- and b-axes of Y123 in the MRF of 10 T was found to be incomplete, which is due to the orientation of grains with twin microstructures. In detail, magnitude relationship between the (200) and (020) peaks was opposite at the  $\beta$  and  $\gamma$  planes, suggesting that the relationship of magnetic susceptibilities ( $\chi$ ) among the three magnetic axes in Y123 is  $\chi_c > \chi_a > \chi_b$  at room temperature. Furthermore, an interesting feature is that the orientation of the [110] direction ([110] grain) emerged as another orientation type of the hard axis in the Fe-free Y123 in addition to the orientation of the [100] or [010] ([100] grain). That is, two different orientation types of the hard axis in Y123 grains,  $H_a \perp b$  and  $H_a \perp [110]$ , were indicated and this phenomenon has not been confirmed in twin-free orthorhombic Y124 and Y247 particles [6,7].

Fig. 3a and b shows XRD patterns at the  $\alpha$  and  $\gamma$  planes for the Fe-doped Y123 powder samples aligned in the MRF of 10 T, together with the result of the Fe-free Y123 powder sample (x = 0) for reference. As shown in Fig. 3a, only (001) peaks were enhanced also in the cases of the Fe-doped Y123 powder samples. Clearly, the first easy axes for the Fe-doped Y123 powder samples with  $x \le 0.1$ were the *c*-axis and were unchanged by the doping of Fe ion. When one focuses on the result in Fig. 3b, the split (020) and (200) peaks around  $2\theta = 46^{\circ}$  were clearly overlapped at  $x \sim 0.04$ . This result can be understood in terms of the orthorhombic-tetragonal transition induced by the doping of Fe ion. That is, it is suggested that the Fedoped Y123 phase was tetragonal for x > 0.04 and an intrinsic structural change of the one-dimensional Cu-O chain, which originates the orthorhombic symmetry of Y123, was induced by the doping of Fe ion. Another striking feature in Fig. 3b is increase in intensities of the (110) peaks  $(I_{110})$  with the increase in x. In order to understand a relative change of  $I_{110}$  induced by the doping of Fe ion, relationship between an intensity (I) ratio using the (110), (200), and (020) peaks,  $I_{110}/(I_{020} + I_{200})$ , and x was shown in Fig. 3c. Here,  $I_{020}$  and  $I_{200}$  represent intensities of the (020) and (200) peaks. The values of  $I_{110}/(I_{020} + I_{200})$  clearly increased with the increase in x, indicating that number of the [110] grains was relatively increased with x compared to the number of the [100]

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