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# Microstructure and $J_C$ Characteristics of Er123 Films With Artificial Pinning Centers

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**Abstract**—Critical current density and surface resistance are evaluated for dilute Zn substituted high quality  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. Dilute Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films are grown on  $\text{SrTiO}_3$  substrates by a pulsed laser deposition technique. Targets used in the experiments are un-substituted, 0.3at. %, 0.5at.%, 1.0at. % and 10at. % Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  ceramics. Crystal structures, field angular dependence of critical current density and surface resistance are evaluated. Zn substituting into  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  has been studied for understanding the origin of oxide superconductivity with substituting level of several %. In this study, dilute Zn below 1.0 at. % is mainly adopted. Further substitution reduces its critical temperature. We intended to introduce zero-dimensional superconductivity killer atoms into  $\text{CuO}_2$  plane as artificial pinning centers. The obtained Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films are  $c$ -axis oriented without peaks from other phases. The sharp drop temperature of surface resistance decreases as the Zn substitution. However, the surface resistance at a low temperature around 20K is almost the same among the  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films with different Zn substitution. We also measured the field angular dependence of critical current density of the Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. There are no strong angular dependences. Dilute zinc substitution increases critical current density for almost of all directions. However, in a high magnetic field of several tesla, pinning force around the field direction of  $a$ -axis is enhanced. Double introduction of one dimensional artificial pinning centers such as  $\text{BaZrO}_3$  nano-rods and zero dimensional artificial pinning centers is thought to be very effective for increasing critical current density for power cable applications.

**Index Terms**— $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  Zn  $J_C$   $R_S$  artificial pinning center.

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## I. INTRODUCTION

SINCE the discovery of high  $T_C$  superconductors (HTSs) with a critical temperature as high as a liquid nitrogen temperature, significant effort has been directed toward fabricating superconducting films with high critical current density ( $J_C$ ). Superconducting cables as developed by Sumitomo Electric Industries [1] by using Bi2223 materials, have a high  $T_C$  and high  $J_C$ . However, HTSs have anisotropic crystal structures as well as transport properties. [2]–[4] Bi family superconducting cables can not feed high superconducting current in a magnetic field around 77.3 K. Critical current density of  $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (RE: Lanthanoid or Y atoms) high  $T_C$  superconducting films at 77.3 K is also relatively lower than that of low  $T_C$  metal superconductors at 4.2 K in magnetic fields. In order to apply high  $T_C$  superconductors for power applications,  $J_C$ s in magnetic fields must be as high as those of metal superconductors at 4.2 K.

Recently, many papers have appeared for the introduction of artificial pinning centers. [5]–[15] These artificial pinning centers are categorized into 4 groups. One is 0-dimensional artificial pinning centers which are atomic substitution of RE [16], Ba or Cu [5]–[8], [17] atoms. Dilute (minute) doping is essential for this type of artificial pinning centers. Next is 1-dimensional artificial pinning centers which are columnar defects induced by nano-particles [9], mosaic surface [18], [19] or heavy ion implantation [20]–[22]. The second is 2-dimensional artificial pinning centers which are grain boundary or thin precipitates. [10], [23] Above 2 artificial pinning centers (1-D and 2-D) have a magnetic field angular dependence. Then, it is effective to improve  $J_C - B$  characteristics by introducing  $c$ -axis correlated artificial pinning centers. The last is the 3-dimensional artificial pinning centers which are insulating nano-particles [24] and nano-low  $T_C$  particles [13], [25], mixed RE atoms [26] and nano-noble metals such as Au.

In this paper, we report relations between the field angular dependencies and their microstructures of  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films with 0-dimensional artificial pinning centers.

## II. EXPERIMENTS

Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films were grown by pulsed ArF excimer laser (Lambda Physik) deposition. The background pressure of the growth chamber (ULVAC) was  $4 \times 10^{-8}$  Pa. The targets used in this experiment was sintered Zn substituted  $\text{ErBa}_2\text{Cu}_3(1-x)\text{Zn}_{3x}\text{O}_{7-\delta}$  ceramics. The incident laser beam was focused on the target surface at  $45^\circ$

through an  $\text{MgF}_2$  window. The focusing lens was also made of  $\text{MgF}_2$  to avoid laser power absorption. The laser irradiated area was about  $2 \text{ mm}^2$ . The laser power was between  $200 \sim 300 \text{ mJ/pulse}$ . The energy density measured on the target was about  $6 \sim 9 \text{ J/cm}^2$  through the window and the lens. The pulse frequency was fixed at  $1 \text{ Hz}$ .  $\text{SrTiO}_3$  (100) substrates were used in this experiment. The substrate was attached by silver paste to a rotating metal substrate holder, which was irradiated by a lamp heater. The substrate temperature was controlled by input power monitored by a thermocouple and calibrated by an optical pyrometer. The substrate temperature was determined using an optical pyrometer. In this experiment, the substrate temperature was fixed near  $750 \text{ }^\circ\text{C}$ .

During deposition, the oxygen pressure was fixed at  $53.3 \text{ Pa}$  with an oxygen flow rate of  $200 \text{ cc/min}$ . After deposition, the substrate was cooled to a room temperature without any annealing processes. It is reported that additional annealing processes increase the  $J_C$  of  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. [27] However, in order to clarify the  $J_C$  enhancement by the introduction of pinning centers, we do not anneal the films.

The preferred orientations of  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films with artificial pinning centers were determined by measuring the  $\theta/2\theta$  scan of the films by x-ray diffraction (XRD) with  $\text{Cu} - \text{K}\alpha$  radiation. The in-plane orientations of the  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films were evaluated by x-ray  $\phi$  scan (in-plane rotation) using the (102) plane of  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$ . The film crystallinity was estimated by full width at the half maximum (FWHM) of the rocking curve using 005 peak of the  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film.

The film thickness is measured by a scanning electron microscope (JSM-6500F) and a mechanical stylus. The lattice images of the films were observed by a transmission electron microscope (TEM).

Critical currents are measured by a conventional four-probe method in a magnetic field. Samples with a  $100 \text{ }\mu\text{m}$  width are fabricated by a photolithography and a wet etching method. The criterion  $1 \text{ }\mu\text{V/cm}$  is used to define the critical current. The samples were rotated maintaining  $\mathbf{J} \perp \mathbf{H}$  (maximum Lorentz force). The angle  $\theta$  between  $\mathbf{H}$  and the normal to the films which coincides with the crystallographic  $c$ -axis within  $0.5$  degrees.

### III. RESULT AND DISCUSSIONS

Fig. 1 shows a typical x-ray diffraction (XRD) pattern of the as-grown Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films with Zn concentration of 10 at%. The obtained  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films with artificial pinning centers have  $c$ -axis preferred orientation. The figure is almost the same as that of the non-doped  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. Then, it is concluded that Zn in 10 at% Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films is not substituted or Zn is substituted with remaining the 123 crystal structure. The reduced Zn concentration  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films from 1 at% to 0.3 at% also showed the same XRD patterns.

Then we measured surface resistance of the films. Fig. 2 shows surface resistance of  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films with varied Zn substitution. The surface resistance of the 10 % Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films showed low  $T_C$  which is nearly coincide with the bulk  $T_C$  with 10 % Zn substitution. By reducing the Zn substitution,  $T_C$  of the  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films goes up. The optimum substitution level of Zn for Cu in

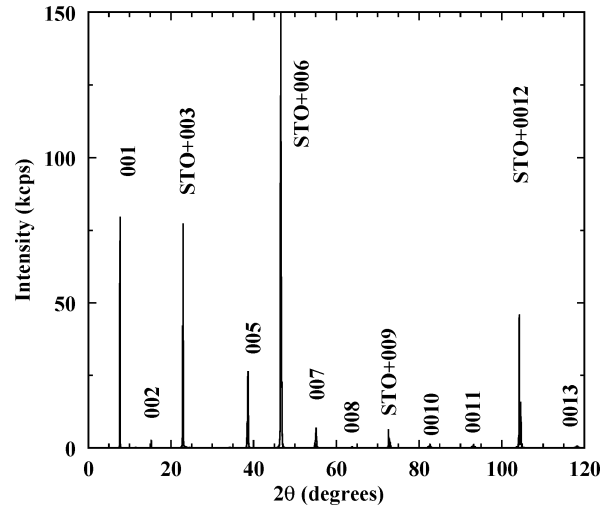


Fig. 1. A typical x-ray diffraction pattern for Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films on  $\text{SrTiO}_3$  substrates.

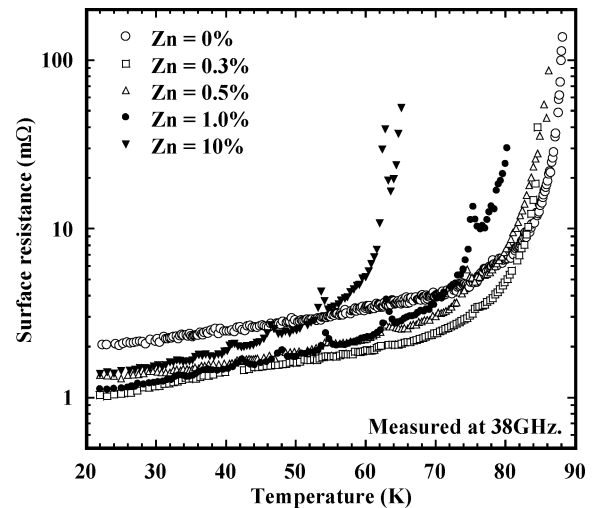


Fig. 2. Surface resistance of  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films with varied Zn substitution for Cu.  $T_C$  depend on the amount of the Zn substitution. However, at a low temperature around  $20 \text{ K}$ , the surface resistance is independent of the Zn substitution.

$\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films may be below 0.3 at%. We used 3N (99.9 %) raw materials to prepare targets. Then, the limitation of doping level is 0.1 at% from the impurity level of raw materials. To get thinner doped films we must use higher purity raw materials. The highest  $T_C$  is obtained in non ( $\text{Zn} = 0\%$ ) substitution  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. However, the surface resistance at a low temperature is the highest for the non ( $\text{Zn} = 0\%$ ) substitution  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. It means that the Zn substitution is effective for the reduction of surface resistance however, substitution in the  $\text{CuO}_2$  plane is too strong. May be another 0-dimensional pinning centers such as RE materials which substitute for Y, or Alkaline earth element Sr for Ba are also available. The most interesting phenomenon is that the surface resistance at a low  $T_C$  around  $20 \text{ K}$  is almost the same for all Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films.

Next we measured the field angular dependence of  $J_C$ . Fig. 3 shows the field angular dependence of  $J_C$  measured at  $77.3 \text{ K}$ .

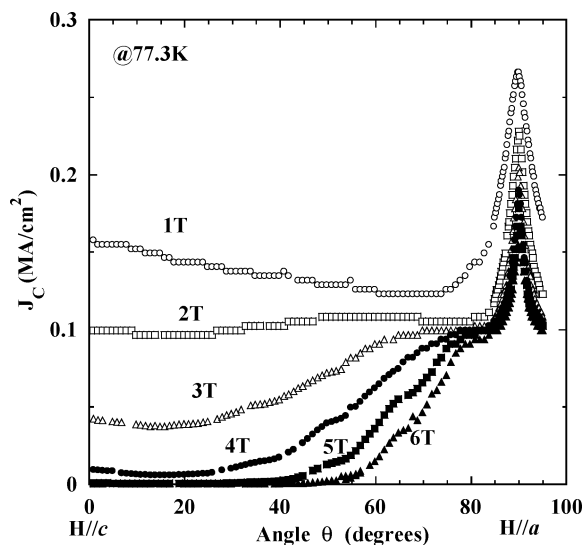


Fig. 3. Field angular dependence of 0.3 at% Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films.

Angle  $\theta$  is the angle between  $c$ -axis of the  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film and the magnetic field. Then the direction of  $90^\circ$  is that of the  $a$ -axis.

In a low magnetic field,  $J_C$  is low at around  $70^\circ$ .  $J_C$  in the magnetic field along the  $a$ -axis is the highest.  $J_C$  in the magnetic field along the  $c$ -axis is not the lowest. Then there are some  $c$ -axis correlated pinning centers. However, no strong anisotropic field angular dependences are observed comparing to the films with 1-dimensional artificial pinning centers [12].

We introduced Zn as 0-dimensional artificial pinning centers which have no angular dependence of the field direction. However,  $J_C$  of the Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film did not increase uniformly for every magnetic direction.

On the contrary, the 0.3 at% Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film showed a  $a$ -axis correlated pinning force in a high magnetic field. Around the  $90^\circ$  ( $75$ - $105^\circ$ ), no reductions of  $J_C$  against the magnetic field up to 6 T were observed. Non doped  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films have greater dependence of the magnetic field along the  $a$ -axis. There may be some defects induced by the Zn addition to the  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. Then we observed cross-sectional TEM images of the 1 at% Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films to clarify the effect of Zn substitution on the microstructure of  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films.

Fig. 4 shows a low magnified cross-sectional TEM image of the 1 at% Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film. From the image, the total thickness of the film is around 500nm. There are no big defects or columnar defects as shown in Fig. 4. However, there are many dark spots in the films. Then, we focus on the dark spots. Fig. 5 shows the high resolution TEM image around the dark spots in the 1 at% Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film. Around the dark spots, we can find many stacking faults in the Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film. Every stacking fault runs along the  $a$ -axis. Very strong pinning force as shown in Fig. 3 is thought to be due to the stacking faults. Then it is concluded that doped Zn in to the  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film was not uniformly distributed in to  $\text{CuO}_2$  plane. Furthermore Zn substitution for Cu in  $\text{CuO}_2$  plane is too strong. Even a small amount of Zn can

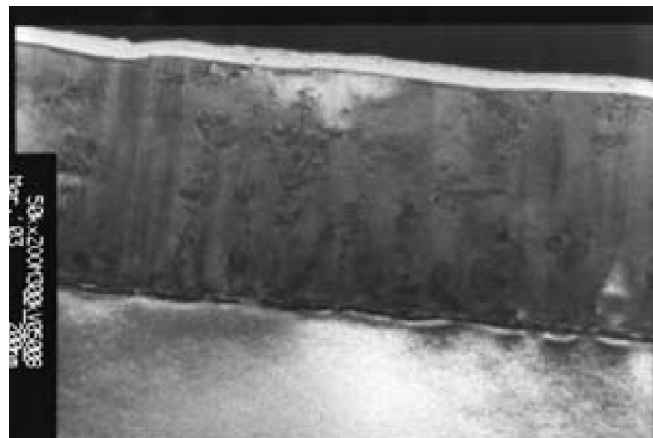


Fig. 4. A low magnified TEM image of the 1 at% Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film.

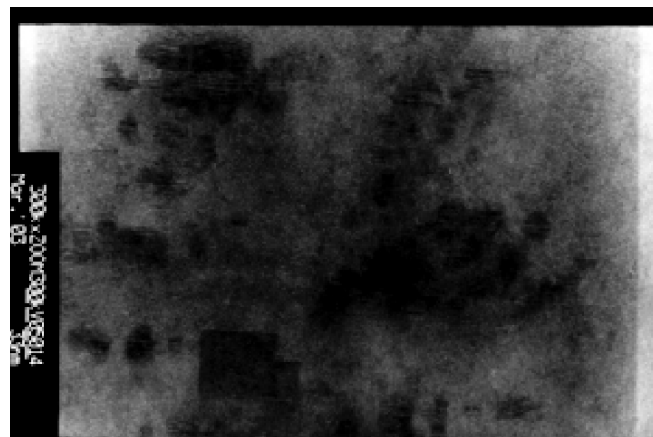


Fig. 5. A high resolution TEM image around the dark spots in the 1 at% Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  film as shown in Fig. 4.

destroy superconductivity. It is difficult to enhance  $J_{CS}$  by substituting Cu atoms in  $\text{CuO}_2$  plane with another atoms. Another 0-dimensional artificial pinning centers which substitute for Ba or RE are strictly required.

#### IV. CONCLUSION

We have evaluated the critical current density and surface resistance of dilute Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. Dilute Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films were grown on  $\text{SrTiO}_3$  substrates by a pulsed laser deposition technique. The obtained Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films were  $c$ -axis oriented with high crystalline quality from the XRD measurements. Zn below 1.0at. % substitution is mainly adopted because, the further substitution reduces its  $T_c$ .

The sharp drop temperature of surface resistance decreases as the Zn substitution. However, the surface resistance at a low temperature around 20 K is almost the same for the  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films with different Zn substitution. We also measured the field angular dependence of critical current density of the Zn substituted  $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films. There are no strong angular dependences comparing to the films with 1-dimensional artificial pinning centers. Dilute zinc substitution increases critical current density for almost of all directions.

However, in a high magnetic field of several tesla, pinning force around the field direction of  $a$ -axis was enhanced. The  $J_C$  enhancement along the  $a$ -axis is due to the stacking faults as observed in the cross-sectional TEM images.

Even a small amount of Zn can destroy superconductivity, then, it is difficult to enhance  $J_C$ s greatly by substituting Cu atoms in  $\text{CuO}_2$  plane with another atoms. Another 0-dimensional artificial pinning centers which substitute for Ba or RE sites are strictly required.

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