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# Molecular Crystals and Liquid Crystals Incorporating Nonlinear Optics

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EFFECTS OF ION IRRADIATION AND THERMAL ANNEALING ON HIGH-TC SUPERCONDUCTOR  ${\rm Ba_2YCu_3O_{7-\delta}}$ 

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Abstract Effects of irradiation of 510 keV 0 ions on thin films of high-Tc oxide superconductor  $\rm Ba_2^{\rm YCu_3^{\rm O}}7-\delta$  (BYCO) were investigated. Increase of critical current density (Jc) by irradiation was observed with a fluence below 8.3 x 10  $^{11}$  0 /cm², while the critical temperature (Tc) did not change in this region. With a fluence above 2.0 x 10  $^{12}$  0 /cm², Tc as well as Jc decreased with fluence, and the superconductivity disappeared perfectly above 5.0 x 10  $^{13}$  0 /cm². The disappeared superconductivities of the specimens recovered partly by annealing at 400  $^{\rm OC}$  for 18 h in O2 flow.

#### INTRODUCTION

Irradiation effects of high-Tc oxide superconductor  $\mathrm{Ba}_2\mathrm{YCu}_3\mathrm{O}_{7-\delta}$  (BYCO) are one of the most important subjects to be studied to consider its application to energy conversion systems as well as fine processing of the compound by high-energy particle beams. Some experimental results have been reported on the decrease of critical temperature (Tc) and the increase of critical current density (Jc) obtained by VSM of BYCO due to irradiation of neutron and charged particles. The authors also have been studying the irradiation effects of BYCO by a neutron reactor and some accelerators in the University of Tokyo. In this paper, we will describe some experimental results on the changes of Tc and Jc of BYCO thin films by the irradiation of oxygen ions( $\mathrm{O}^+$ ) followed by thermal annealing.

# EXPERIMENTAL

BYCO thin films used in this study were grown on  ${\rm SrTiO}_3$  (100) substrates by the activated reactive evaporation method from metals of Y, Ba and Cu at 650  $^{\rm O}{\rm C}$  in  ${\rm O}_2$  flow. The c-axes of these films were

oriented perpendicular to  ${\rm SrTiO_3(100)}$  substrates. Before and after ion irradiation, temperature-dependent I-V characteristics of these films were measured with 30 Hz sine wave current. In the I-V measurements, the conventional four-probe method was used and the films were etched chemically into a pattern of about 300  $\mu m$  in order to reduce the total current. The values of Jc were determined from the top values of the supercurrents, which were defined as voltage values less than 10  $\mu V/cm$ , and those of Tc were determined from the temperature at which the critical current values were less than  $10^{-4} A$ . For the samples irradiated heavily, resistivities were measured by four-probe method.

Ion irradiation experiments were performed with a Van de Graaff accelerator at the University of Tokyo. Ions of 510 keV  $0^+$  impinged on these BYCO films at room temperature in a vacuum of  $2.7 \times 10^{-5} Pa$ . Ion beam currents was about  $1.4 \text{ nA/cm}^2$ , which was chosen to prevent significant heating of BYCO thin films. Ion fluences ranged from  $2.8 \times 10^{11}$  to  $1.1 \times 10^{14}$  ions/cm<sup>2</sup>. For ions of 500 keV  $0^+$ , the penetration depth is estimated to be 540 nm, which is comparable to the thickness of BYCO thin films used in this experiment. Therefore radiation-induced defects distribute almost all over the films.

## RESULTS AND DISCUSSION

The changes of Tc and Jc due to irradiation are shown in Fig.1. In this figure,  $Jc/Jc_0$  and  $Tc/Tc_0$  versus ion fluence are plotted, where  $Jc_0$  and Jc represent the Jc values before and after irradiation at T=0.95  $Tc_0$ , and  $Tc_0$  and Tc represent Tc values before and after

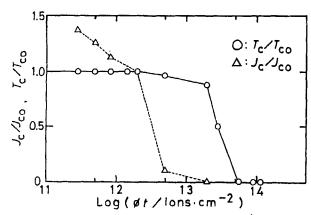


FIGURE 1 Change of Tc and Jc by 510 keV 0 irradiation.

irradiation, respectively. As shown in this figure, both Jc and Tc were degraded at the irradiation above  $2.0 \times 10^{12} \, \mathrm{ions/cm^2}$ . On the other hand, below this fluence the Tc value was not affected and the Jc value was enhanced due to radiation defects, which are considered to act as pinning centers. Considering that atmospheric degradation occurs easily in BYCO thin films and that radiation damage of the grain boundary might degrade transport supercurrents, the observed enhancement of directly measured Jc values in this experiment strongly suggests that defects formed by ion irradiation act as pinning centers.

Figure 2 shows temperature-dependent Jc values of the film subjected to a fluence of  $2.8 \times 10^{11} \, \mathrm{ion/cm^2}$ . As shown in this figure, Jc values after irradiation were enhanced in the temperature range investigated. The Jc curve after irradiation rose steeper than that before irradiation. In all cases investigated for Jc, the Jc values were represented as

$$Jc=A(1-(T/Tc))^{2},$$
 (1)

where A is a parameter, and only the value of A changed due to ion irradiation as reported previously. In the fluence range of this ion irradiation experiment, radiation damage had little effect on Tc values, and irradiation-induced defects can act as pinning centers to enhance Jc values below a fluence of  $2.0 \times 10^{12}$  ions/cm<sup>2</sup>, while above

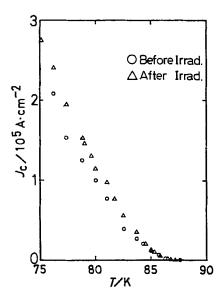


FIGURE 2 Temperature dependence of Jc on BYCO film (fluence: 2.8  $\times$   $10^{11}$   $0^+$  ions/cm<sup>2</sup>).

this fluence, Jc values were degraded due to radiation damage.

On the contrary, in the fluence region of heavy-irradiation above  $5.5 \times 10^{13} \, \mathrm{ions/cm^2}$ , superconductivity perfectly disappeared. Figures 3 and 4 show the temperature dependence of electrical resistance of BYCO specimens before and after irradiation with fluences of  $5.5 \times 10^{13} \, \mathrm{ions/cm^2}$  and  $1.1 \times 10^{14} \, \mathrm{ions/cm^2}$ , respectively. As shown in these figures, the temperature dependence of the resistance changed from metallic one to semiconductor-like one. Onset Tc was retained in the case of such high fluences, and its trace can been seen even with a fluence of  $1.1 \times 10^{14} \, \mathrm{ions/cm^2}$ . As is shown clearly in Fig.3, two-steps drop was observed on the resistance with decreasing temperature. This behavior suggests the coexistence of ortho-I phase (Tc $\!\sim\!\!90$ K) and

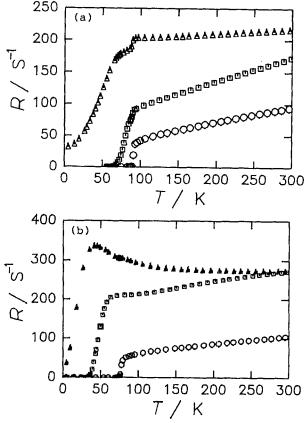


FIGURE 3 Temperature dependence of electrical resistance,  $\bigcirc$ ; before irradiation,  $\triangle$ ; after irradiation with a fluence of (a) 5.5 x  $10^{13}$  ions/cm<sup>2</sup> and (b) 1.1 x  $10^{14}$  ions/cm<sup>2</sup>,  $\square$ ; after annealing (latm  $O_2$ , 400  $^{\circ}$ C, 18 h).

ortho-II phase ( $Tc\sim60K$ ). Oxygen ion irradiation knocked on the structural oxygen atom in the crystal, which may caused the existence of ortho-II phase.

Superconductivities of all these four heavily-irradiated specimens recovered partly by annealing at 400  $^{\rm O}{\rm C}$  for 18 h in  ${\rm O_2}$  flow. As is shown in Fig.3, the temperature dependence of electrical resistances of these specimens changed by annealing to be more metallic in normal condition state, and the values of Tc could be observed. The values of onset, midpoint and end temperatures of Tc are summarized for these four specimens before and after heavy irradiation and after thermal annealing.

TABLE I Critical temperatures of YBCO thin films before and after irradiation and after annealing.

Fluence (ions/cm²)		Before Irradiation	After Irradiation	After Annealing
2.8 × 10 <sup>13</sup>	Tc(onset) Tc(midpoint) Tc(end)	95.0 K 87.0 K 81.0 K	88.0 K 78.3 K 43.4 K	78.0 K 73.7 K 67.4 K
5.5.×10 <sup>13</sup>	Tc(onset) Tc(midpoint) Tc(end)	94.0 K 91.1 K 81.6 K	90.5 K 44.1 K	90.4 K 79.7 K 55.1 K
8.9 ×10 <sup>13</sup>	Tc(onset) Tc(midpoint) Tc(end)	92.0 K 85.3 K 63.9 K	91.0 K 13.7 K	74.8 K 22.5 K
1.1 ×10 <sup>14</sup>	Tc(onset) Tc(midpoint) Tc(end)	78.5 K 74.6 K 69.5 K	77.2 K 15.9 K	59.0 K 47.4 K 29.4 K

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