Effects of 1-MeV proton irradiation in Hg-based cuprate thin films

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We have studied the effects of 1-Mev proton irradiation on both superconducting properties and normal state resistivity of high-quality $HgBa_2CaCu_2O_{6+\delta}$ (Hg-1212) and $HgBa_2Ca_2Cu_3O_{8+\delta}$ (Hg-1223) thin films. At low proton doses, we observed a linear decrease of the superconducting transition temperature T_c and a linear increase of the extrapolated residual resistivity as proton dose is increased. This is consistent with observations of other high-T_c superconductors while a lower dose threshold for suppressing the superconductivity is found in Hg-1212 and Hg-1223 films. To explain the linear dose dependence of T_c, we propose a model based on the proximity effect. An enhancement of up to 90% in the critical current density at low fields has also been observed in these films at low proton fluences that do not significantly degrade T_c. [S0163-1829(97)03926-X]

I. INTRODUCTION

Ion-beam irradiation has been an effective method for introducing various defects into materials in a fairly predictable and controllable fashion. These defects have two general effects on the material. First, they perturb the crystalline as well as the electronic structures of the original material and therefore can be used to probe many important physical properties of the material. The effect of ion-beam irradiation has been extensively studied in many high-T_c superconductors (HTSc's) during the past ten years. This study has yielded many interesting results,[1–11] including the most striking and puzzling observation by Summers *et al.* of a universal linear dependence of the decrease rate of superconducting transition temperature (T_c) with ion dose ($dT_c/d\phi$) on the nonionizing energy loss of the incident ion beams.[12] Second, ion-beam irradiation has been used to produce magnetic flux pinning centers in HTSc's. This includes creation of weak pinning centers via displacive interactions in low-energy light-ion irradiation[13–17] and strong pinning centers in columnar defects produced by high-energy heavy-ion irradiation.[13,18]

The newly discovered Hg-based cuprates ($HgBa_2Ca_{n-1}Cu_nO_{2n+2+\delta}$ $n=1,2,3,4$) have attracted much attention because of their record high T_c above 130 K that makes-them very promising for many commercial applications. Moreover, $HgBa_2CaCu_2O_{6+\delta}$ (Hg-1212) and $HgBa_2Ca_2Cu_3O_{8+\delta}$ (Hg-1223) phases have a relatively high irreversibility line, staying between that for $YBa_2Cu_3O_{7-\delta}$ (YBCO) and that for Bi- and/or Tl-based compounds.[19] This means that Hg-1212 and Hg-1223 are more anisotropic than YBCO, believed to be a three-dimensional system, and less anisotropic than Bi- and/or Tl-based compounds, regarded as two-dimensional systems. Our recent study on the pinning potential of Hg-1212 films indicated that its original pinning strength is lower than that for YBCO and higher than that for Bi- and/or Tl-based compounds.[20] The Hg-based cuprates, therefore, provide an interesting system for the study of physical properties in order to achieve a complete picture for various HTSc's. We have recently studied the effect of 1-Mev proton beam irradiation on the normal-state resistivity (ρ), T_c, and J_c on high-quality c-axis-oriented Hg-1212 and Hg-1223 thin films. The advantage of using thin-film samples is that for most ion beams with moderate energies, the beam range is much larger than the thickness of the film. One may then assume that the distribution of the defects produced by the beam is uniform across the thickness of the film, which makes the analysis of the result much simpler. In this paper, we report our experimental results and present an explanation of the data.

II. EXPERIMENT

The irradiation experiment was carried out mainly on Hg-1223 films. One Hg-1212 film was also used for J_c observa-

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tions. All samples were grown on $SrTiO_3$ substrates using a three-step fast temperature ramping Hg-vapor annealing process. The details of the film fabrication procedure have been reported elsewhere.[21–23] These films are approximately 700 nm in thickness. Both magnetic and electrical transport properties were measured in the proton-irradiated films. For transport measurement, a 200 μm $\times$ 1.7 mm bridge was patterned on a Hg-1223 film using the wet etching method. After etching, four silver electrical contacts were sputtered onto the four corners of the film. Before irradiation, this sample had a zero-resistance transition temperature (T_{c0}) of 123.6 K and a critical current density of 0.27 MA/cm^2 at 100 K. The original T_c's of the samples used for magnetic measurements are 128.5 K for the Hg-1223 film and 124.0 K for the Hg-1212 film.

The irradiation was performed at the 1.7 MeV Pelletron Tandem accelerator at Texas Center for superconductivity at the University of Houston. A proton beam of 1 MeV was selected to generate point defects in the film. The energy loss of 1 MeV protons in Hg-based cuprates is about 37 keV over the 700-nm thickness, or only 3.7% of the incident energy. This means that the sample is effectively two dimensional, with uniform damage even at high dosage. At 1 MeV energy, the proton collision cross section is of Rutherford form for all nuclei, including O, and therefore does not show any resonances, so there is no preferential bombardment in the sample. The average range of the 1-MeV proton is estimated to be 10 μm, which is much larger than the thickness of the film used in this experiment; thus the defects created are approximately uniform across the thickness of the film. The proton beam line was equipped with an X-Y scanner for uniform irradiation over a 10×10-mm^2 area and a deflector to avoid neutral beam bombardment. The dosage was measured with a current integrator with a 300-V secondary electron suppressor on the sample.

Measurements of resistivity (ρ) and critical current density (J_c) were conducted in a close-cycle cryostat system where temperature control is accurate to within 0.5 K and the minimum working temperature was about 15 K. Platinum wire contacts were made on the sputtered silver pads with silver paste and were retained throughout irradiation and characterization for most of the experiment. J_c was estimated using the criterion of 1 μV/cm. Magnetization (M) was measured in a commercial superconducting quantum interference device magnetometer at temperatures ranging from 5 to 110 K. The magnetic field was applied normal to the film. Magnetic J_c was inferred from hysteresis curves (M-H loops) using the Bean model.

The ρ-T curves obtained for the Hg-1223 film are shown in Fig. 1(a) for proton doses from 0 to 140×10^{15} protons/cm^2 and in Fig. 1(b) for the low-dose range of 0 to 30×10^{15} protons/cm^2. The unirradiated sample had a ratio ρ(250 K)/ρ(150 K) of about 2.05. At low doses, as detailed in Fig. 1(b), the temperature dependence of the normal-state resistivity remains linear, indicating a metallic behavior in the sample while a small number of point defects were introduced by the proton irradiation. The magnitude of the resistivity, however, increases monotonically with the proton dose. Since the ρ-T curves are parallel to each other above T_c, the increase in ρ can be attributed to the residual resistivity due to alloying of metallic Hg-1223 by proton-

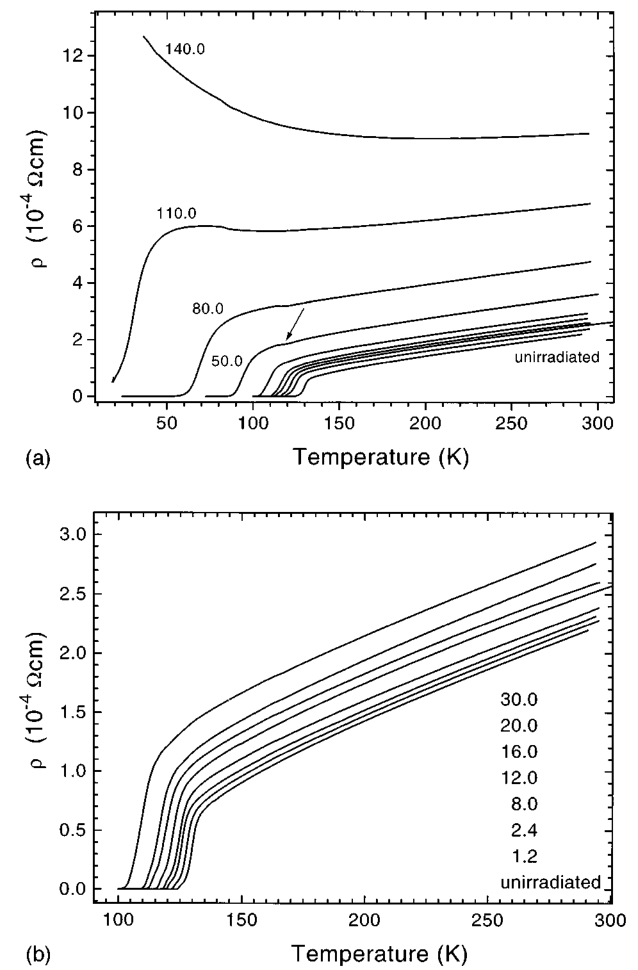

FIG. 1. (a) Hg-1223 film ρ-T curves at different proton fluences where an arrow shows the ''kink'' signifying the onset of metal-insulator transition; and (b) the same ρ-T curves at low doses. Doses are in units of 10^{15} protons/cm^2.

induced point defects. According to Mattheissen's rule, $\rho(T)=\rho_i+\rho_L(T)$, where $\rho_L(T)$ is the resistivity of the metal and is independent of the dose. The residual resistivity ρ_i is defined for a superconductor as the zero-temperature intercept of the extrapolated straight line from the linear part of the ρ-T curve—in our study, from $T=250$ to 150 K. As shown in Fig. 2(a), the slope of the normal part of the ρ-T curves, which is defined using all data points from 250 to 150 K, remains fairly constant for proton doses up to 50×10^{15} protons/cm^2. The residual resistivity, however, was found to increase linearly with the proton dose in the same dose range [Fig. 2(b)], which is consistent with Mattheissen's rule. When the dose is further increased ρ_i increases drastically while the slope of the ρ-T curve decreases, indicating a metal-insulator phase transition at high doses. Meanwhile, T_c decreases monotonically with proton fluence. In Fig. 2(c), the onset transition temperature ($T_{c,\mathrm{onset}}$) and zero-resistance temperature (T_{c0}) are plotted as functions of the proton dose. It is clear that both $T_{c,\mathrm{onset}}$ and T_{c0} decrease linearly with the proton dose in the dose range of 0 to 50×10^{15} protons/cm^2 and decrease much more rapidly as the dose is further increased. These effects seem to be universal for most high-T_c superconductors, as reported in other studies with various ion-beam irradiations.[8,9,24–28]

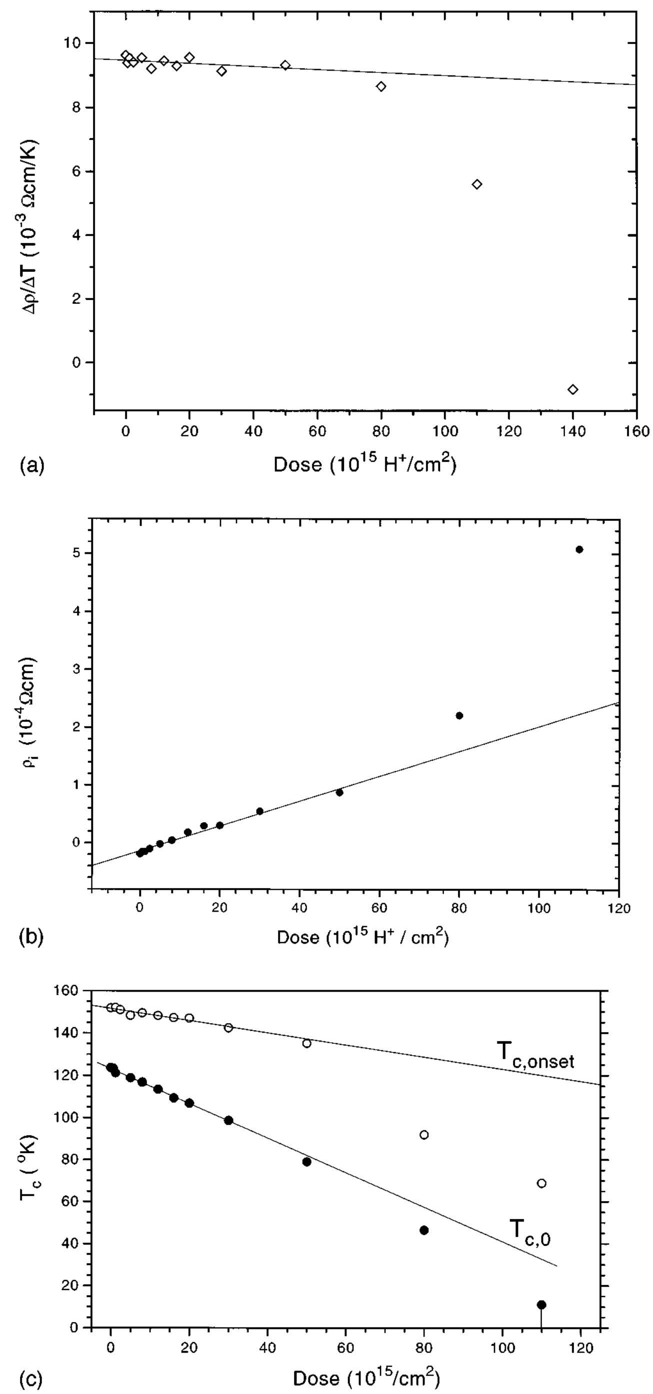

FIG. 2. (a) The slope of the linear portion of the ρ-T curves for the Hg-1223 film from $T=250$ to 150 K, plotted as a function of proton dose; (b) Mattheissen's rule: residual resistivities at different fluences; and (c) onset T_c and zero-resistance T_c of Hg-1223 film at different fluences.

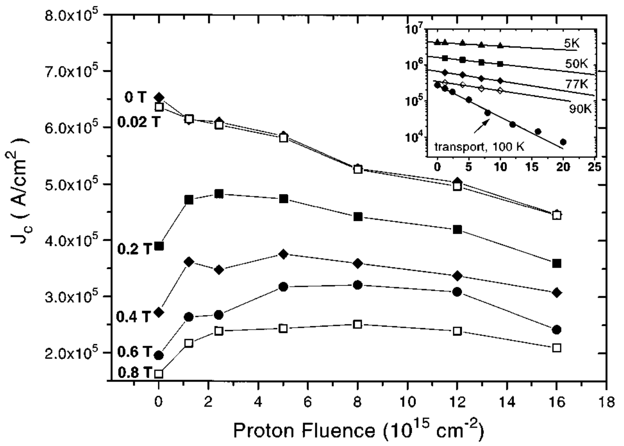

FIG. 3. Critical current density of the Hg-1212 film at different fluences for $T=5$ K. Inset: J_c's for Hg-1223 film at zero external field; from top to bottom, magnetic J_c's at $T=5$, 50, 77, and 90 K; transport J_c at $T=100$ K.

At higher doses, a kink in the ρ-T curve [shown with an arrow in Fig. 1(a)] that is barely evident at the dose $=30\times10^{15}$ protons/cm^2 and becomes visible at dose $=50\times10^{15}$ protons/cm^2, signifies the onset of a metal-insulator transition that reaches critical at a dose of 110×10^{15} protons/cm^2 and is completed by dose $=140\times10^{15}$ protons/cm^2. The flattening of the slope of the ρ-T curves at these doses shows significant damage in the material as overlapping or clustering of point defects becomes prevalent. This is confirmed also by the deviation of T_c from its linear decrease with proton dose, clearly seen in Fig. 2(c), at about a dose of 50×10^{15} protons/cm^2. From TRIM simulations[29] the displacements for 1-MeV proton irradiation in 1-μm-thick Hg-1212 and Hg-1223 films is found to be approximately 3×10^{-5}/(ion Å) and 4×10^{-5}/(ion Å), respectively. For the Hg-1223 films, for example, the proton doses of 50, 110, and 140×10^{15} protons/cm^2 can then produce approximately 47, 100, and 120 displaced atoms per 1000 unit cells, or 0.0029, 0.0064, and 0.0082 displacements per atom (dpa). T_{c0} begins to decrease as the damage level increases beyond 0.000 29 dpa and a linear decrease of T_{c0} with the proton fluence is maintained up to 0.0029 dpa. At higher dose, the fluence dependence of T_{c0} deviates from the linear behavior and superconductivity is nearly destroyed at 0.0064 dpa.

Aside from causing material degradation in the superconductor, it is well known that irradiation-induced defects can also serve as magnetic-flux pinning centers that can enhance the critical current density (J_c) by improving the pinning strength in the material. Since these two effects are competing, we speculate that during the proton irradiation J_c would be improved when the proton dose is low and then be degraded as the dose is high. Meanwhile, the improvement of J_c should also depend on the applied magnetic field, i.e., J_c should decrease at zero or low magnetic field where pinning is insignificant. When the pinning strength needed at a certain applied magnetic field becomes comparable with the pinning strength of irradiation induced pinning centers, an optimized J_c improvement should result. Figure 3 shows the magnetic J_c as a function of proton dose at 5 K and different applied magnetic fields from 0 to 1 T. At zero field and at 0.02 T, a monotonic decrease of J_c with increasing proton dose is observed. The same type behavior was also found in the zero-field measurements of magnetic J_c at other temperatures and transport J_c shown in the inset of Fig. 3. As the field is further increased, for example, close to or above 0.2 T, J_c undergoes an increase at low-proton dose, saturates at around a dose of 2×10^{15} protons/cm^2, and then degrades when the dose is increased further. This same behavior is also seen in YBCO.[14,30]

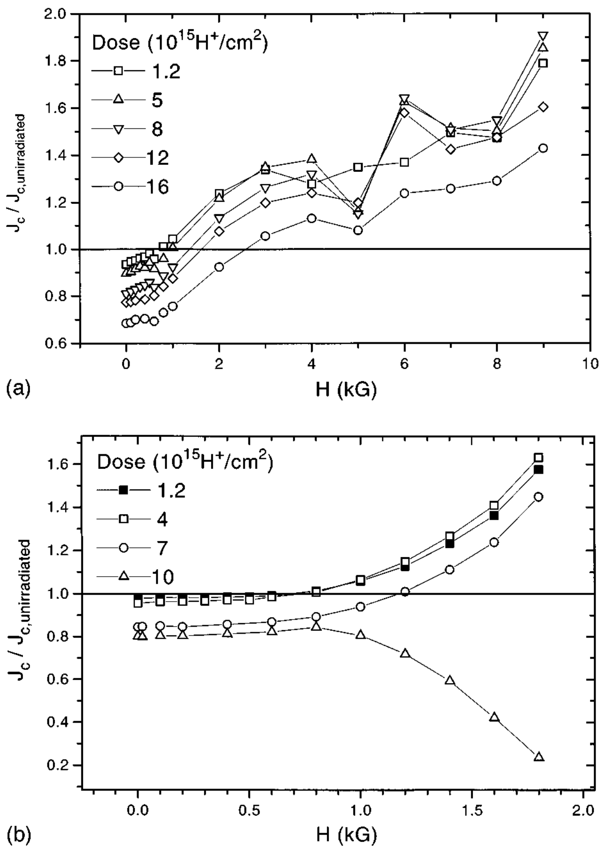

FIG. 4. Normalized magnetic J_c vs field curves at different proton fluences for (a) Hg-1212 film and (b) Hg-1223 film, both at 5 K.

To give a more quantitative description, the normalized J_c, which is defined as the ratio of J_c's after and before the proton irradiation ($J_c/J_{c,\text{unirradiated}}$), is plotted as function of the field in Fig. 4(a) for Hg-1212 and in Fig. 4(b) for Hg-1223 films, respectively. The enhancement of J_c seems to be more significant at higher field and an optimum increase of nearly 90% is observed at 0.9 T for Hg-1212 film for a proton dose of about 87×10^{15} protons/cm^2, where the drop in T_c is still negligible. At higher doses, for example, 16×10^{15} protons/cm^2, this enhancement falls to about 40% at the same field. Similar behavior was also seen on Hg-1223 films as shown in Fig. 4(b), where for the available data enhancement is highest at about 64% increase for a dose of 4×10^{15} protons/cm^2 and a field of 0.18 T. This is about five times higher than the enhancement for Hg-1212 at the same field and comparable dose. By comparison, J_c enhancement in YBCO was reported to be as much as ten times for 1 T and 10×10^{15} 3-MeV protons/cm^2.[13] The minimum field required for J_c enhancement increases monotonically with dose. For the lowest dose with Hg-1212, this occurs at about 0.1 T; with Hg-1223, much lower, about 0.07 T. For the Hg-1223 sample there is no enhancement after 7×10^{15} protons/cm^2. The presence of kinks at about 0.5 T in Fig. 4(a) may likely be caused by an artifact of the measurement system.

III. DISCUSSION

The effect of 1-MeV proton irradiation on Hg-1223 is found to be very similar to that on other HTSc's in both normal state and superconducting state. The damage level that affects the superconductivity in Hg-1223, however, is much lower than that for other HTSc's.[3,18,27] For example, loss of superconductivity occurs at ~0.035 dpa for YBCO (Refs. 3 and 18) and at 0.02 dpa for Tl-based cuprates,[27] compared to 0.0064 for Hg-1223. A possible explanation is that some sites associated with Hg^{2+} and O^{-2} in the Hg-1223 have much lower activation energies. This is inferred from the low annealing temperature for oxygen diffusion in Hg-1223. We have found that the T_{c0} of a Hg-1223 film can be increased by 15–20 K when it is annealed at a temperature as low as 200 °C in flowing oxygen.[31] For YBCO, an annealing temperature above 400 °C is needed to diffuse oxygen into the sample. Furthermore, our recent hot-stage x-ray diffraction study of the Hg-1223 film in flowing O_2 suggests that Hg is activated at temperatures above 425 °C,[32] while in Tl-based cuprates Tl is stable at a much higher temperature near 800 °C.[33]

Most of the proton-irradiation effect on HTSc's is well understood. However, the linear T_c decrease with ion fluence in the low-dose region, an effect that is universal to most HTSc's including YBCO, Bi- and/or Tl-based compounds, and the Hg-based cuprates, remains a mystery.

This has triggered many theoretical models, yet no consensus has been reached to date.[34–37] For example, a depairing mechanism caused by Cu and O vacancies was proposed by Jackson *et al.*[34] However, inconsistencies still exist,[35,36] moreover, the linear relation between the T_c depression and the defect density derived from their model applies only to the case where $\Delta T_c \ll T_{c0}$,[34] yet in Hg-1223 we found this linearity to extent to $\Delta T_c > 45$ K [Fig. 2(c)] which is more than $\frac{1}{3}$of the unirradiated T_c.

The question is as follows: why is the superconductivity greatly affected at such a low damage level as observed in Hg-1223 film? Assuming superconductivity in a unit cell is completely destroyed if one atom is knocked out, then the volume-ratio threshold of the damaged material is less than 5% at 0.0029 dpa for Hg-1223. This value is much lower than the threshold for the destruction of superconducting percolation in a two-dimensional system which is around 30%. This discrepancy indicates that several unit cells must be affected to a certain extent when one atom is knocked out. A hint of such an effect was already seen in our earlier study of microstructures in proton irradiated YBCO using high-resolution transmission electron microscopy, where a large stressed volume around a point defect was observed.[38] Identical features were also observed in proton-irradiated bulk Hg-based cuprates.[39] Assuming a poorer superconductivity exists in those distorted unit cells, the observed T_c would be dragged down by the proximity effect between the original and the distorted volumes.[40,41] We therefore propose the following model based on the proximity effect.

Let us divide the proton dose range into three different regions: low dose, high dose, and very high dose. In the low-dose region, which is below 5×10^{15} protons/cm^2 for Hg-1223 films, few point defects are produced so that the volume fraction of distorted unit cells is too small to affect the superconductivity of the sample; thus T_c is nearly unchanged during the irradiation. In the high-dose region, which is between 5×10^{15} protons/cm^2 to 50×10^{15} protons/cm^2 for Hg-1223 films, we may picture the sample as multilayers of original superconductor (layer A) sandwiched by materials in distorted unit cells (layer B). The thickness of layer A is much bigger than the thickness of layer B since the dose is still low. As the dose is increased, the relative thickness change of layer A is then much smaller than that of layer B. One may assume that the thickness of layer A is approximately a constant while the thickness of layer B is increased linearly with the proton dose. A linear decrease of T_c is then expected based on the proximity between layer A and layer B as shown from both theory and experiment.[40,41] The very-high-dose region begins at above 50×10^{15} protons/cm^2 as the volume fraction of the distorted unit cell becomes comparable to the threshold at which superconducting percolation is destroyed. T_c decreases much more rapidly than the linear behavior because now the relative change of thickness in layer A is comparable to that in layer B; this has also been predicted by the proximity effect.[41] Finally, when the threshold is passed and superconducting percolation no longer exists, the current has to flow through the damaged material in layer B, and a drastically reduced T_c should be observed.

IV. SUMMARY

In summary, the effect of 1-MeV proton irradiation on Hg-based thin-film superconductors is qualitatively similar to that on other HTSc's but quantitatively more sensitive to the proton doses. We proposed that the proximity effect plays a role in the degradation of the superconductivity, particularly in the linear decrease of T_c with proton dose at low doses. J_c is enhanced by as much as 90% at optimum dose of around 5×10^{15} protons/cm^2, where T_c decrease is fairly insignificant.

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