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Restoration of superconducting properties of proton-implanted ceramic $YBa_2Cu_3O_{7-x}$ by annealing in oxygen

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500-keV protons were implanted into ceramic $YBa_2Cu_3O_{7-x}$ (Y-Ba-Cu-O) at doses of 1×10^{15} to 2×10^{17} H^+ cm^{-2}. The proton-implanted Y-Ba-Cu-O was annealed in oxygen at temperatures of 400 °C to 700 °C for 90 min to restore superconducting properties destroyed by proton implantation. The resistivities at a temperature of 100 K and the critical temperatures of the annealed proton-implanted Y-Ba-Cu-O depended on the implant dose of protons and the annealing temperature. The critical temperatures decreased as the resistivities at 100 K increased. A critical temperature of 91 K, higher than 87 K of as-sintered Y-Ba-Cu-O, was obtained for ceramic Y-Ba-Cu-O annealed at 500 °C for 90 min after implantation at an implant dose of 1×10^{17} H^+ cm^{-2}.

I. INTRODUCTION

Superconducting properties of $YBa_2Cu_3O_{7-x}$ (Y-Ba-Cu-O) are influenced by chemical and structural disorders introduced by doped impurities. The critical temperature T_c at which resistivity becomes zero is increased by thermally introducing a large amount of hydrogen (H) from a H_2 atmosphere into Y-Ba-Cu-O.[1] A significant increase of T_c has occurred for hydrogenated ceramic $H_{2.7}YBa_2Cu_3O_{7-x}$. Ion implantation also is a superior technique for introducing defects and impurities into Y-Ba-Cu-O. However, this technique leads to a transformation from the superconducting phase to an amorphous phase.[2] Annealing in oxygen (O_2) at high temperatures is performed to restore the superconducting structure destroyed by proton implantation. Also, the annealing serves to produce oxygen (O) lost during proton implantation into Y-Ba-Cu-O.

O atoms in the Cu-O layer between the two Ba atoms in Y-Ba-Cu-O are decomposed easily by heating.[3] H atoms implanted react chemically with the constituents of Y-Ba-Cu-O during annealing, and can result in a number of water molecules by reacting with O atoms.[4] The deficiency of O atoms in Y-Ba-Cu-O results in degradation of the superconducting properties: T_c of annealed proton-implanted Y-Ba-Cu-O decreases, and the resistivity at temperatures above T_c increases since carrier lifetimes are broadened by the defects.[5] Thus the question that we pose is whether or not the superconducting properties of Y-Ba-Cu-O can be improved by proton implantation at high doses.

In this paper we show that the resistivity at a temperature of 100 K and the critical temperature, measured on Y-Ba-Cu-O annealed at high temperatures in O_2 atmosphere after proton implantation at high doses, were improved significantly by conditions of proton implantion and subsequent annealing.

II. EXPERIMENT

Powders of Y_2O_3, $BaCO_3$, and CuO were ball milled for 24 h and then reacted at 930 °C for 3 h in O_2. The synthesized powders were ball milled for 24 h and pressed to a disk with a diameter of 14 mm and a thickness of 1.2 mm at a pressure of 16.5 kbar. The disks were sintered at a temperature of 930 °C in O_2 for 3 h and then were cooled from the sintering temperature to room temperature at a rate of about 10 °C h^{-1}. X-ray-diffraction pattern (XDP) studies revealed that the prepared disks had the orthorhombic phase composition of Y-Ba-Cu-O. The packing factor of the disks was 6.45 g cm^{-3}.

500-keV protons were implanted into ceramic Y-Ba-Cu-O disks with doses ranging from 2×10^{15} to 2×10^{17} cm^{-2}. The calculated range and straggle values of the 500-keV proton for Y-Ba-Cu-O are 2.96 and 0.105 μm, respectively.[6] Proton-implanted Y-Ba-Cu-O was annealed in O_2 at temperatures of 400–700 °C for 90 min. Annealed proton-implanted Y-Ba-Cu-O was cooled from the annealing temperature to room temperature at a rate of about 10 °C h^{-1}.

Resistivities of Y-Ba-Cu-O were measured by a four-probe method. Indium was deposited on the surfaces of Y-Ba-Cu-O in vacuum to make good ohmic contacts. Gas chromatography (GCG) studies were carried out to study the decomposition of proton-implanted Y-Ba-Cu-O during annealing. Small masses of about 1 mg cut out from proton-implanted and unimplanted Y-Ba-Cu-O were dropped into a crucible heated to a desirable temperature. Constituents effused from the small masses were introduced into a column of the molecular sieve heated to 80 °C together with helium as carrier gas, and then the constituents were identified by measuring the elapsed time between the injection of the sample and its appearance at a detector. This elapsed time is referred to as the retention time.

III. RESULTS AND DISCUSSION

Figure 1 shows XDP at $2\theta = 32°-34°$ on as-sintered Y-Ba-Cu-O, as-proton-implanted Y-Ba-Cu-O, and annealed proton-implanted Y-Ba-Cu-O. The intensities of the diffraction lines in the range of 32°–34° are very sensitive to the crystallographic structure of Y-Ba-Cu-O. Here proton-implanted Y-Ba-Cu-O refers to Y-Ba-Cu-O implanted at a dose of 1×10^{17} $H^+ cm^{-2}$, and annealed proton-implanted Y-Ba-Cu-O refers to the proton-implanted Y-Ba-Cu-O annealed in O_2 at 500 °C for 90 min. As-sintered Y-Ba-Cu-O was an orthorhombic-phase Y-Ba-Cu-O for which the intensity ratio of the (103,110) and (013) diffraction lines, $R_{XDP,o}$ $[=I(103,110)/I(013)]$, was a value of about 1.7, smaller than 2, for orthorhombic phase Y-Ba-Cu-O. Proton-implanted Y-Ba-Cu-O had a weaker (103,110) diffraction peak and a larger (013) diffraction peak than as-sintered Y-Ba-Cu-O. Furthermore, the diffraction angle of the doublet shifted slightly to lower angles than as-sintered Y-Ba-Cu-O. The proton-implanted Y-Ba-Cu-O then had a low $R_{XDP,p}$ value of about 1.2. Here the $R_{XDP,p}$ is the $R_{XDP,o}$ for Y-Ba-Cu-O implanted with protons.

Layers destroyed by proton implantation seem to extend in Y-Ba-Cu-O from the surface to a depth that is approximately the sum of the projected range and straggle of proton for Y-Ba-Cu-O. X rays diffracted from undamaged Y-Ba-Cu-O lying under the destroyed layer are adsorbed in the destroyed layer. The primary x rays incident upon the undamaged layer are also absorbed in this damaged layer. A rough calculation shows that the intensity of the x rays diffracted from the undamaged layers was only about 2.2% of that from Y-Ba-Cu-O without the destroyed layer. A value of about 233 $cm^2 g^{-1}$ (Ref. 7) for the mass absorption coefficient of Y-Ba-Cu-O for Cu $K\alpha$ radiation and a value of 6.4 $g cm^{-3}$ (Ref. 8) for the density of Y-Ba-Cu-O were used for this calculation. The obtained $R_{XDP,p}$ values can thus be considered to show information for the crystallographic structure of layers destroyed by proton implantation.

The $R_{XDP,p}$ value of proton-implanted Y-Ba-Cu-O became about 1.3 smaller than as-implanted Y-Ba-Cu-O: a number of crystals with the orthorhombic phase in as-sintered Y-Ba-Cu-O changed to crystals with the tetragonal phase because of proton implantation. A fraction of orthorhombic phase Y-Ba-Cu-O destroyed by proton implantation is much less than that of Y-Ba-Cu-O that the proton was implanted at the same dose at low temperatures.[2] The crystallographic structure of Y-Ba-Cu-O destroyed by proton implantation at low temperatures is known to restore the orthorhombic structure only by heating to room temperature.[9] In our experiment a large fraction of orthorhombic phase Y-Ba-Cu-O destroyed by proton implantation seems to restore during proton implantation because proton implantation was performed at room temperature.

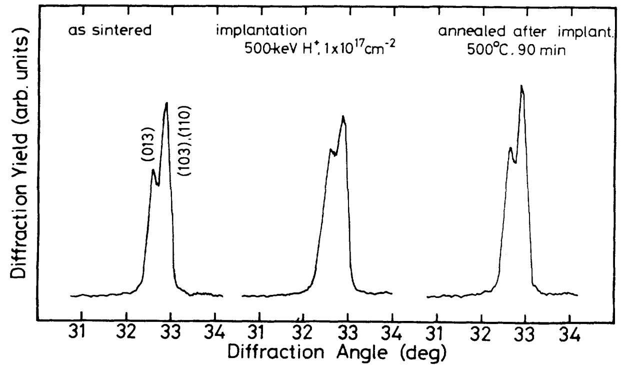

FIG. 1. XDP spectra for unimplanted, proton-implanted, and annealed proton-implanted Y-Ba-Cu-O.

Annealing in O_2 at temperatures above 500 °C was performed to restore the destroyed orthorhombic structure of Y-Ba-Cu-O by proton implantation. Annealed proton-implanted Y-Ba-Cu-O had the approximate same $R_{XDP,p}$ value as as-sinstered Y-Ba-Cu-O. The similar restoration of the destroyed orthorhombic structure of Y-Ba-Cu-O by annealing in O_2 has been observed on Y-Ba-Cu-O implanted at a dose of 1×10^{17} $H^+ cm^{-2}$.[10]

Table I shows $R_{XDP,p}/R_{XDP,o}$ values, representing a fraction of the orthorhombic structure destroyed by proton implantation. The numbers in parentheses in this table are the $R_{XDP,p}/R_{XDP,o}$ values for as-implanted Y-Ba-Cu-O. The $R_{XDP,p}/R_{XDP,o}$ values for as-implanted Y-Ba-Cu-O decreased with increasing implant dose: a fraction of tetragonal phase Y-Ba-Cu-O increased with increasing implant dose. However, the $R_{XDP,p}/R_{XDP,o}$ value did not become smaller than about 0.58, even on Y-Ba-Cu-O implanted at a high dose of 2×10^{17} $H^+ cm^{-2}$. In our experiment the minimum $R_{XDP,p}/R_{XDP,o}$ value is 0.29 for proton-implanted Y-Ba-Cu-O, since as-sintered Y-Ba-Cu-O had a $R_{XDP,o}$ value of 1.7 and tetragonal phrase Y-Ba-Cu-O has a $R_{XDP,o}$ value of 0.5.

Annealing in O_2 gas at temperatures of 400–700 °C for 90 min was performed to restore the destroyed orthorhombic structure of proton-implanted Y-Ba-Cu-O. The $R_{XDP,p}/R_{XDP,o}$ values increased approximately with increasing annealing temperature on Y-Ba-Cu-O implanted at doses below 1×10^{17} $H^+ cm^{-2}$, and decreased slightly at a temperature of 700 °C. Desirable temperatures to restore the destroyed orthorhombic structure of proton-implanted Y-Ba-Cu-O were temperatures ranging from

TABLE I. $R_{XDP,p}/R_{XDP,o}$ obtained on annealed proton-implanted Y-Ba-Cu-O. Here $R_{XDP,o}$ is the intensity ratio of the (103,110) and (013) diffraction lines and $R_{XDP,p}$ is the $R_{XDP,o}$ value of Y-Ba-Cu-O annealed after proton implantation. The numbers in parentheses are the $R_{XDP,p}/R_{XDP,o}$ values for as-implanted Y-Ba-Cu-O (annealing time 90 min).

Implantation dose (10^{15} cm^{-2})	Annealing temperature (°C) 400	500	600	700
20	(0.96) 0.96	(0.95) 0.98	(0.89) 0.96	(0.92) 0.96
50	(0.89) 0.90	(0.85) 0.87	(0.83) 0.95	(0.86) 0.96
100	(0.78) 0.84	(0.81) 0.98	(0.80) 0.97	(0.79) 0.95
200	(0.57) 0.63	(0.57) 0.66	(0.58) 0.71	(0.56) 0.74

500 to 600 °C. The $R_{\text{XDP},p}/R_{\text{XDP},o}$ values on proton-implanted Y-Ba-Cu-O annealed at the desired temperatures increased with increasing implant dose below 1×10^{17} cm^{-2}. The restoration of silicon lattices destroyed by ion implantation by annealing is promoted similarly by an increase in the implant dose of impurity ions.[11] However, the orthorhombic structure of Y-Ba-Cu-O destroyed by proton implantation at a dose of 2×10^{17} cm^{-2} was not well restored by annealing since the H atoms produce water by reacting with O decomposed from Y-Ba-Cu-O during annealing as subsequently described.

Figure 2 shows GCG spectra for unimplanted and proton-implanted Y-Ba-Cu-O as a function of the proton-implantation dose. The intensities of the GCG signals were normalized for the weight of the samples. The GCG spectra consisted of three broad peaks. The first broad peak with a centroid at a retention time of 1.1 min is assigned to H_2, N_2, and O_2. However, we could not identify the origin of the peak because of the poor resolution of the GCG used. The second peak is assigned to CO_2. The later appearing peak is assigned to water (H_2O). Here we call attention to the third peak obtained only on proton-implanted Y-Ba-Cu-O as shown in Fig. 2. The O atom of the Cu-O layer between the two Ba atoms in Y-Ba-Cu-O is known to be easily removed by heating, compared with O atoms occupying other sites.[3] The decomposed O atoms produce water by chemically reacting with H atoms implanted into Y-Ba-Cu-O.[4] The intensity of the water peak thus increased gradually with increasing the implant dose of protons and increased steeply at an implantation dose of 2×10^{17} H$^+$ cm^{-2}. This steep increase is in good correspondence with the remarkable decrease in the $R_{\text{XDP},p}/R_{\text{XDP},o}$ value on proton-implanted Y-Ba-Cu-O. A large amount of water produced in this proton-implanted Y-Ba-Cu-O during annealing promotes decomposition of the superconducting structure.[12]

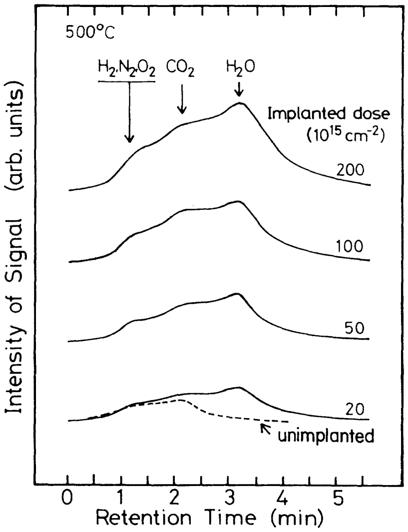

FIG. 2. GCG spectra obtained at a crucible temperature of 500 °C for unimplanted and proton-implanted Y-Ba-Cu-O as a function of the implantation dose of proton.

Figure 3 shows the annealing-temperature dependence of resistivities on unimplanted and proton-implanted Y-Ba-Cu-O, which were annealed at 500 °C for 90 min, as a function of the implant dose. Proton-implanted Y-Ba-Cu-O annealed at other temperatures had a similar temperature dependence of resistivities. The resistivities at a temperature of 100 K, $R_{100\,\text{K},p}$, on annealed proton-implanted Y-Ba-Cu-O increased with increasing implant dose, then decreased, and increased again after reaching a minimum resistivity of Y-Ba-Cu-O implanted at a dose of 1×10^{17} H$^+$ cm^{-2}. The $R_{100\,\text{K},p}$ of Y-Ba-Cu-O implanted with doses of 2×10^{16}–1×10^{17} H$^+$ cm^{-2} was smaller than the resistivity at a temperature of 100 K, $R_{100\,\text{K},o}$, on annealed unimplanted Y-Ba-Cu-O. The critical temperature $T_{c,p}$ of proton-implanted Y-Ba-Cu-O showed an implant dose dependence opposed to its resistivity. The critical temperature $T_{c,o}$ of annealed unimplanted Y-Ba-Cu-O was lower than that of as-sintered Y-Ba-Cu-O, although the $R_{100\,\text{K},p}$ was larger. A similar degradation of $T_{c,o}$ is obtained on Y-Ba-Cu-O annealed in O_2 at high temperatures.[13] This Y-Ba-Cu-O has a large deficiency of O atoms. However, the results shown in Fig. 3 show that the presence of H atoms in Y-Ba-Cu-O was effective in preventing the degradation of its critical temperature during annealing in O_2 at high temperatures. A remarkable increase in the critical temperature, compared with as-sintered Y-Ba-Cu-O, was realized on Y-Ba-Cu-O annealed in O_2 at 500 °C for 90 min after the proton was implanted at doses ranging from 5×10^{16} to 1×10^{17} cm^{-2}.

$R_{100\,\text{K},p}$ and $T_{c,p}$ of annealed proton-implanted Y-Ba-Cu-O are summarized in Fig. 4 as a function of implant dose. $T_{c,p}$ and $R_{100\,\text{K},p}$ depended greatly on the implant dose and annealing temperature. We can find three characteristic regions in the implant-dose dependence of $R_{100\,\text{K},p}$ and $T_{c,p}$. The first region, referred to as region I, is where the implant doses are lower than about 1×10^{16} H$^+$ cm^{-2}. A feature of this region I is that the

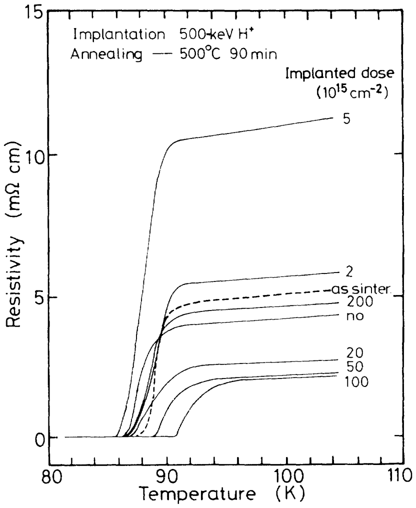

FIG. 3. Temperature dependence of resistivities of proton-implanted Y-Ba-Cu-O annealed at 500° for 90 min as a function of the implantation dose.

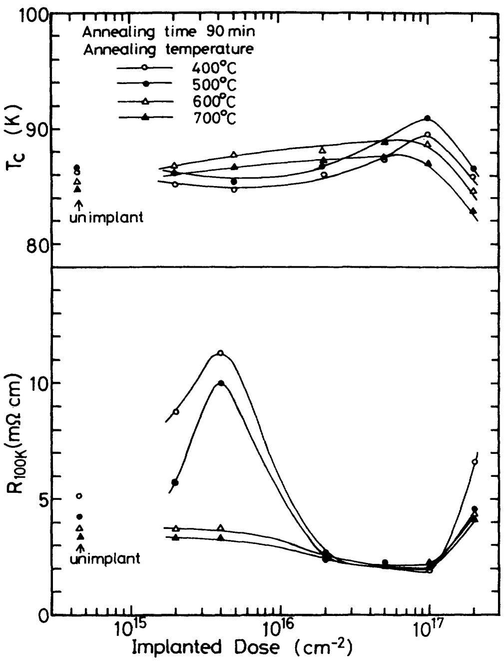

FIG. 4. $R_{100\ K,p}$ and $T_{c,p}$ of annealed proton-implanted Y-Ba-Cu-O.

$R_{100\ K,p}$ of annealed proton-implanted Y-Ba-Cu-O decreased abruptly at a temperature between 500 and 600 °C, although $T_{c,p}$ did not show such a drastic dependence as $R_{100\ K,p}$. The $R_{100\ K,p}$ of proton-implanted Y-Ba-Cu-O annealed at temperatures below 500 °C was larger than that of unimplanted Y-Ba-Cu-O annealed at the same temperatures. The $R_{100\ K,p}$ of proton-implanted Y-Ba-Cu-O annealed below 500 °C increased with increasing implant dose and decreased after becoming a maximum at an implant dose of 5×10^{15} $H^+\ cm^{-2}$. On the other hand, the $R_{100\ K,p}$ of proton-implanted Y-Ba-Cu-O annealed at temperatures above 600 °C was smaller than that of unimplanted Y-Ba-Cu-O annealed at the same temperatures, although it decreased gradually with increasing implant dose. However, XDP results show that proton implantation resulted in no significant changes in the crystallographic structure. We therefore conclude that lattice defects induced by proton implantation in region I result in an insignificant change in the crystalline lattice, but they are acting primarily as additional scattering centers. The annealing stage between 500 and 600 °C leads to a large energy required to move the displaced constituents into the original substitutional sites.

The second characteristic region was obtained on Y-Ba-Cu-O implanted at doses ranging from 2×10^{16} to 1×10^{17} $H^+\ cm^{-2}$. The $R_{100\ K,p}$ of annealed proton-implanted Y-Ba-Cu-O was smaller than that of unimplanted Y-Ba-Cu-O annealed at the same temperatures and scarcely depended on the annealing temperature and implant dose. However, the $T_{c,p}$ depended largely on the implant dose and annealing temperature. On Y-Ba-Cu-O implanted at a dose of 1×10^{17} $H^+\ cm^{-2}$, there was an optimum annealing temperature of 500 °C to give Y-Ba-Cu-O with a higher $T_{c,p}$. This restoration of $R_{100\ K,p}$ and $T_{c,p}$ correspond to the restoration of the orthorhombic structure as shown in Table I. The fact that even annealing in O_2 at temperatures as low as 400 °C after proton implantation results in an increase in the fraction of the orthorhombic phase in proton-implanted Y-Ba-Cu-O is similar to the tetragonal-orthorhombic phase transition by proton-implanted Y-Ba-Cu-O with a large deficiency of O atoms when annealed in an O_2 atmosphere.[10] Similarly, H atoms in degraded Y-Ba-Cu-O serve to contribute to the restoration of destroyed superconducting properties during annealing in O_2 atmosphere as in the model described by Really *et al.*:[1] H atoms occupy O vacancies on the *c* or *a* axis, or both, and is coordinated primarily with Cu. This is analogous to a metal and hydrogen system where H occupies a metal-atom-coordinated, interstitial site. The slight increase in $T_{c,p}$ in this system is due to a slightly more favorable configuration of the electronic band structure and/or by a fortuitous elastic distortion of the unit cell caused by insertion of the H atom.

The third characteristic region was obtained on Y-Ba-Cu-O implanted at a dose of 2×10^{17} $H^+\ cm^{-2}$. The $R_{100\ K,p}$ of annealed proton-implanted Y-Ba-Cu-O was larger than that of unimplanted Y-Ba-Cu-O annealed at the same temperatures. The $R_{100\ K,p}$ decreased with increasing the annealing temperature and increased with increasing the implant dose. The $T_{c,p}$ of proton-implanted Y-Ba-Cu-O decreased remarkably at the implant doses of region III. The increase in $R_{100\ K,p}$ and the decrease in $T_{c,p}$ may be due to the large number of lattice defects, as the deficiency of O atoms in the Cu-O layers—generated during annealing as proton implantation in region III—resulted in a large amount of water during annealing. The water also resulted in a poor restoration of the destroyed orthorhombic structure of Y-Ba-Cu-O even at annealing temperatures as high as 700 °C as shown in Table I.

Proton implantation induced lattice defects and the orthorhombic-tetragonal phase transition in superconducting materials. The lattice defects remaining after annealing or induced during annealing lead to a lifetime broadening of electronic states and, thus, to a smearing of the peak in the electronic density of states. The resistivity is a function of the lifetime of carriers near the Fermi level. On the assumption that the carrier-phonon coupling constant is proportional to the electronic density of states at the Fermi level, $T_{c,o}$ is calculated as a function of the residual resistivity in imperfect materials.[5] The influence of O atoms introduced from the atmosphere into proton-implanted Y-Ba-Cu-O during annealing on the resistivity of annealed proton-implanted Y-Ba-Cu-O seems to be eliminated by subtracting the $R_{100\ K,o}$ of annealed unimplanted Y-Ba-Cu-O from the $R_{100\ K,p}$ of annealed proton-implanted Y-Ba-Cu-O, since the probability of a collision of carriers taking place in unit time is the sum of the probabilities of collisions due to the various scattering mechanisms. Here $(R_{100\ K,p}-R_{100\ K,o})$ is referred to as R. The R values thus reveal approximately the change in resistivity with the presence of H atoms.

Figure 5 shows a relationship between R and $T_{c,p}/T_{c,o}$

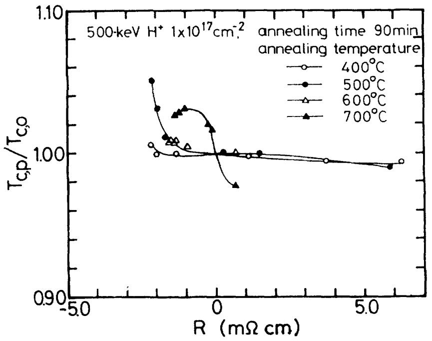

FIG. 5. Relationship between $T_{c,p}/T_{c,o}$ and R for annealed proton-implanted Y-Ba-Cu-O. Here $R=(R_{100\ K,p}-R_{100\ K,o})$.

on annealed proton-implanted Y-Ba-Cu-O. The value of $T_{c,p}/T_{c,o}$ on annealed proton-implanted Y-Ba-Cu-O increased gradually with decreasing resistivity as observed for transition-metal compounds.[5] The $T_{c,o}$ of transition-metal compounds decreases as electron scattering by point defects increases, and the residual resistivity also increases. On our annealed proton-implanted Y-Ba-Cu-O the number of point defects are thus deduced to vary with the number of H atoms. The H atoms in Y-Ba-Cu-O with improved critical temperatures serve to inactivate point defects by terminating their dangling bonds and/or to make a slightly more favorable configuration of the electronic band structure.[1] Also, the H atoms in Y-Ba-Cu-O with degraded critical temperatures serve to generate point defects by reacting with O atoms in the Cu-O layers. The small dependence of the $T_{c,p}/T_{c,o}$ value on the R value seems to be due to an increase of the carrier-phonon coupling constant by the presence of H atoms as in thin Y-Ba-Cu-O films implanted with 300-keV protons at a temperature of 77 K.[2]

IV. SUMMARY

500-keV protons were implanted into ceramic Y-Ba-Cu-O to a dose of 1×10^{17} cm^{-2}. Proton-implanted Y-Ba-Cu-O was annealed in O_2 at temperatures of 300–700 °C to restore the destroyed superconducting structure. H atoms implanted into Y-Ba-Cu-O reacted with O atoms and effused as water into the atmosphere during annealing. This was a mechanism for degradation of the superconducting properties of Y-Ba-Cu-O. However, the H atoms implanted also contribute to an improvement in the superconducting properties. The two occurred in proton-implanted Y-Ba-Cu-O at the same time. That is, whether superconducting properties of Y-Ba-Cu-O are improved or degraded by proton implantation and annealing depended strongly on conditions of proton implantation and annealing. A critical temperature of 91 K, higher than 87 K of as-sintered Y-Ba-Cu-O, was realized on ceramic Y-Ba-Cu-O annealed at 500 °C for 90 min after having been implanted at an implantation dose of 1×10^{17} H^+ cm^{-2}. However, the superconducting properties of Y-Ba-Cu-O implanted at a dose of 2×10^{17} H^+ cm^{-2} were not improved as the decomposition of O atoms was promoted by the large number of H atoms present in Y-Ba-Cu-O.

ACKNOWLEDGMENT

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