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Critical temperature depression and persistent photoconductivity in ion irradiated $YBa_2Cu_3O_{7-x}$ films and $YBa_2Cu_3O_{7-x}/PrBa_2Cu_3O_7$ superlattices

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We have studied the effect of He^+ irradiation with doses in the range $10^{12}-2\times10^{15}$ cm^{-2} on two high-temperature superconducting structures: $YBa_2Cu_3O_{7-x}$ (YBCO) films and $YBa_2Cu_3O_{7-x}/PrBa_2Cu_3O_7$ (YBCO/PBCO) superlattices. In particular we have focused on superlattices $[YBCO_N/PBCO_M]_{1000\ Å}$ with $N=1$, 8 unit cells of YBCO, and $M=5$ unit cells of PBCO, with a total thickness of 1000 Å. The analysis is presented in terms of depression of the critical temperature (T_c) and modification of the crystalline structure using X ray refinement technique. Single films show a systematic increase in the c-lattice parameter upon irradiation, which is not observed in thin one unit cell YBCO layers in superlattice structures. However, T_c depression resulting from irradiation is deeper in superlattices. These results are explained considering the strained nature of the as-grown $[YBCO_N/PBCO_5]_{1000\ Å}$ superlattices with low values of N. Both structures show persistent photoconductivity, indicating that defects are related to oxygen displacements in the Cu–O chains. © *2000 American Institute of Physics.* [S0003-6951(00)05322-5]

Ion irradiation has been extensively explored as a procedure to modify the properties of high-temperature superconductors by the controlled introduction of defects. Irradiation with heavy ions at high energies is known to cause drastic changes in their normal and superconducting properties. An increase of the normal state resistivity up to the metal–insulator transition is observed when damage increases, together with a reduction of the critical temperature (T_c). Damage mainly originates from electronic stopping and consists basically of amorphous tracks of nanometric width, which act as effective pinning centers, giving rise to enhanced critical currents.[1–3] Irradiation with light ions at low and intermediate energies causes analogous changes in normal and superconducting properties, but a decrease in the critical current is generally observed. This energy range damage results from nuclear elastic collisions, and basically consists of point defects due to atomic displacements.[4] It has been proposed that irradiation induced disorder occurs mainly in the oxygen sublattice, such that the original superconducting properties can be partially recovered with low temperature annealing.

In this letter we report the T_c depression and structural modification in $YBa_2Cu_3O_{7-x}$ (YBCO) thin films and $YBa_2Cu_3O_{7-x}/PrBa_2Cu_3O_7$ (YBCO/PBCO) superlattices induced by 80 keV He^+ ion irradiation at doses up to 2×10^{15} cm^{-2}. Photodoping effect has been measured in both kinds of samples in order to highlight the defect structure responsible for T_c depression. Structure characterization of unirradiated and irradiated films and superlattices was analyzed by x-ray diffraction (XRD). Superlattices allow a more accurate structural characterization than single films since their XRD spectra exhibit a rich peak diffraction profile pattern which is sensitive to structural disorder and defects. Since both YBCO and PBCO are isostructural and show small lattice mismatch, it is possible to grow them epitaxially one on top the other with sharp boundaries.

The c-axis oriented YBCO single films were grown on (100) $SrTiO_3$ (STO) substrates in a high pressure (3.4 mbar) pure oxygen direct-current (dc) sputtering system. Rocking curves around (005) XRD peaks showed full width at half maximum (FWHM) smaller than 0.2°, pointing to a very ordered mosaic structure. Superconducting properties were analyzed by resistivity and diamagnetic shielding measurements. T_c determined using the zero resistance criterium were 90 K, in agreement with the onset of the diamagnetic shielding. Transition widths were smaller than 0.2 K. Critical current was always higher than 10^6 A/cm^2 at 77 K. Superlattices were prepared on (100) oriented STO by sequentially sputtering stoichiometric YBCO and PBCO targets. Superlattices showed a c-axis growth and FWHM of the (005) rocking curves smaller than 0.2° and a T_c dependent on the YBCO layers thickness.[5] In particular we have focused in this letter on superlattices $[YBCO_N/PBCO_M]_{1000\ Å}$ with $M=5$ unit cells of PBCO and $N=1$,8 unit cells of YBCO up to a total thickness of 1000 Å. The structural properties of the superlattices were investigated by high-angle XRD using the SUPREX refinement program.[6] The structural refinement allows a determination of the lattice parameters of the constituent materials,[6] YBCO and PBCO for our multilayers.[5] The analysis showed up a very high degree of structural and

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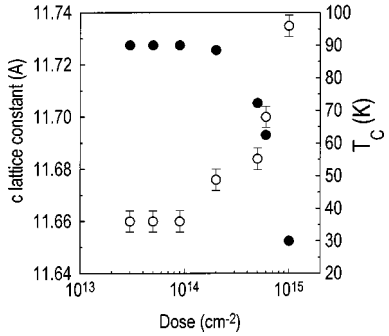

FIG. 1. YBCO thin film critical temperature (black dots) and c-lattice constant (open dots) dependence on He^+ irradiation dose.

chemical order even in the as-grown YBCO one unit cell superlattices: we have found negligible interdiffusion and step disorder pointing that high pressure oxygen growth promotes sharp interfaces.[5] This supports the adequacy of our $[YBCO_N/PBCO_5]_{1000\ Å}$ system for the study of the structural disorder induced by ion irradiation.

The 80 keV He^+ ions were implanted at room temperature and at doses ranging between 10^{12} and 2×10^{15} cm^{-2} at an angle 7° away from the substrate normal to avoid channeling. Single films and superlattices were grown with the same total thickness of 1000 Å to avoid the thickness dependence of the structural disorder introduced by ion irradiation. For the energy selected a projected range of 3500 Å was obtained from the SRIM 96 software, so that the ions would have enough energy to pass through the YBCO or $[YBCO_N/PBCO_5]$ films and to stop in the STO substrates, only causing irradiation effects. Ion current was kept small, in the order of 500 nA, to avoid heating of the samples during irradiation. In this sense, oxygen diffusion and subsequent oxygen removal from the films can be discarded. Photodoping and persistent photoconductivity were analyzed from resistance vs temperature curves measured in a close cycle refrigerator with an optical window. Ion irradiated samples were illuminated with a 40 W Xe lamp for 5–7 h at 95 K. After this period the lamp was turned off and the samples were cooled down to 10 K and kept at this temperature for at least one hour before T_c was measured, to avoid heating effects of the lamp.

The effect of ion irradiation on the superconducting properties of thin films was a monotonic depression of T_c when ion dose was increased up to 10^{15} cm^{-2}, with no changes observed for doses lower than 10^{14} cm^{-2}. XRD analysis did not show significant structural changes of films upon irradiation, although a systematic increase in the c-lattice parameter was observed for increasing doses. These results are shown in Fig. 1. In all cases, FWHM of the rocking curves around the (005) peak were also not significantly altered. Generally speaking, c-axis expansion can be associated with various causes such as oxygen deficiency, film strain, disorder in the oxygen sublattice.

Figure 2 shows the results corresponding to XRD spectra of a superlattice with $N=1$ where irradiation experiments have been consecutively performed on the same sample up to the final dose of 2×10^{15} cm^{-2}. The intensity and width of the $(00l)$ and satellite lines remains the same before and after ion irradiation, and there is no evidence for additional

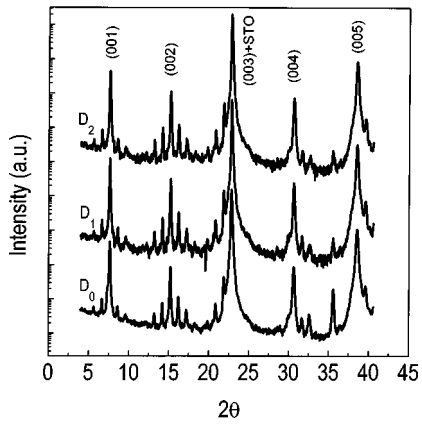

FIG. 2. θ–2θ spectra for a $[YBCO_1/PBCO_5]_{1000\ Å}$ superlattice as-grown ($D_0=0$) and irradiated with two doses ($D_1=5.5\times10^{13}$ cm^{-2}, $D_2=2\times10^{15}$ cm^{-2}). Consecutive spectra have been displaced vertically two decades for clarity.

interface roughness resulting from interdiffusion or amorphyzation during irradiation, which would lead to broadening or disappearance of the satellite lines in the spectra. Then, after irradiation, in all the investigated dose range the superlattice structure persists, with abrupt interfaces. This points that defects caused by irradiation could be mainly related to oxygen displacement, a specie to which x rays are less sensitive, and allows to discard cationic displacement as the source of defects. Especially in the one unit cell thick YBCO superlattices, cationic displacement would give rise to breakdown of the continuity of the layer resulting in significant amounts of roughness. Fitting the spectra with a proper refinement procedure allows the determination of YBCO c-lattice constant, which evolution upon irradiation is shown in Table I. The estimated error is ±0.05 Å and describes the range over which the goodness of the fit did not significantly change.[5] Irradiation causes an increase of the YBCO c-lattice constant in $[YBCO_8/PBCO_5]_{1000\ Å}$ superlattices, while no such changes are observed in $[YBCO_1/PBCO_5]_{1000\ Å}$ samples. We believe that YBCO intralayer epitaxial strain is responsible for this intriguing result. Actually, different levels of YBCO intralayer strain can be recognized in the fresh as-grown superlattice, depending on the number of unit cells in the YBCO layers. This strain arises from the lattice mismatch between YBCO and PBCO. In plane PBCO lattice parameters are about 1% larger than those of YBCO. This results in a tensile in plane strain for the thin YBCO layers, which leads to a compression of the c-lattice parameter. This effect is more pronounced for the

TABLE I. Critical temperatures of as-grown and irradiated $[YBCO_N/PBCO_5]_{1000\ Å}$ superlattices ($N=1,8$). c-lattice constants of the YBCO layers deduced from the refinement of x-ray spectra are included.

Sample	Dose (cm^{-2})	$T_c(R=0)$ (K)	YBCO c-constant (Å)
$[YBCO_1/PBCO_5]_{1000\ Å}$	0	48	11.474
$[YBCO_1/PBCO_5]_{1000\ Å}$	5×10^{12}	46	11.474
$[YBCO_1/PBCO_5]_{1000\ Å}$	5.5×10^{13}	41	11.474
$[YBCO_1/PBCO_5]_{1000\ Å}$	1.5×10^{14}	37	11.474
$[YBCO_1/PBCO_5]_{1000\ Å}$	2×10^{15}	···	11.474
$[YBCO_8/PBCO_5]_{1000\ Å}$	0	82	11.624
$[YBCO_8/PBCO_5]_{1000\ Å}$	10^{15}	29	11.661

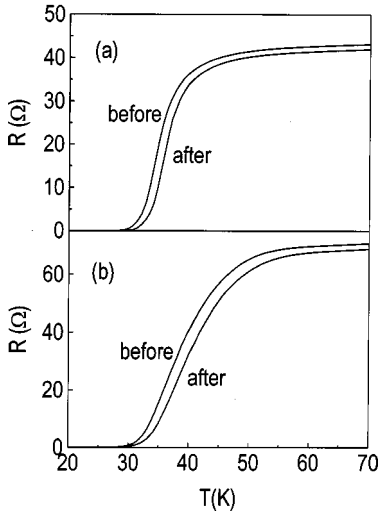

FIG. 3. Resistance as a function of temperature for irradiated (a) YBCO thin film and (b) $[YBCO_8/PBCO_5]_{1000\ Å}$ superlattice before an after illumination. In both cases the total thickness is 1000 Å and the dose is $D=10^{15}\ cm^{-2}$.

one unit cell based superlattices. As a comparative example, we have quoted in Table I the results obtained for a $[YBCO_8/PBCO_5]_{1000\ Å}$ superlattice: $c(YBCO)=11.62$ Å is closer to the thin film value. Further details about the structural effects of the mismatch strain can be found in Ref. 5. It seems that the high level of epitaxial strain present in the YBCO layers before irradiation in $[YBCO_1/PBCO_5]_{1000\ Å}$ superlattices, hinders the c-lattice parameter expansion after irradiation. T_c depression resulting from irradiation is stronger in superlattices showing a higher degree of epitaxial strain. On the one hand, the structure of YBCO thin film is not affected by irradiation with doses below $10^{14}\ cm^{-2}$, although in $[YBCO_1/PBCO_5]_{1000\ Å}$ superlattices T_c is depressed for doses as low as $5\times10^{12}\ cm^{-2}$. On the other hand $[YBCO_8/PBCO_5]_{1000\ Å}$ superlattice presents comparable variation of T_c than bare thin films after irradiation with the same dose of $10^{15}\ cm^{-2}$. We have checked the reproducibility of the result for this dose in other multilayers where the c-lattice parameters are in agreement within 0.5% with those reported for bulk samples ($N=8$, 12).[5] We conclude that while an evident correlation exists between c-axis expansion and T_c depression in the single YBCO films, the situation is more complicated for the superlattices. If the enlargement of the c-lattice parameter in thin films results from local strains arising from atomic displacements induced by irradiation, the strained nature of $[YBCO_N/PBCO_5]_{1000\ Å}$ superlattices with low values of N may prevent this structural effect to occur.

Concerning the nature of the defects created by irradiation, it is well known that oxygen atoms located in the Cu–O chains are the lightest and the most loosely bound specie in the structure.[7] The 80 keV He^+ ion irradiation will most likely displace oxygen atoms from the chains into vacant oxygen sites along the a axis, leading to disorder in the oxygen sublattice. A direct way to test the presence of the irradiation-induced oxygen disorder is the observation of photoinduced effects upon illumination with visible light. All models for persistent photoconductivity in high temperature superconductors are based on the presence of disorder in the chain structure: charge transfer models,[8,9] electron trapping in oxygen vacancies,[10–12] and photoassisted oxygen ordering.[13] We may expect, therefore, persistent photoconductivity on irradiated YBCO films much in the same way as in oxygen deficient films. In this line Figs. 3(a) and 3(b) show resistance measurements on a single film and a $[YBCO_8/PBCO_5]_{1000\ Å}$ superlattice respectively, before and after illumination. Persistent photoinduced effects show up: after illumination a clear decrease of the resistance is observed, together with an increase of T_c. This points to a damage related with oxygen displacement in the chains. Oxygen displacement from the chains into a-axis positions provides an explanation for the behavior observed in the irradiated superlattices. An expanded YBCO in plane structure due to lattice mismatch with PBCO makes oxygen displacement easier; this is probably the reason why T_c depression is larger in strained superlattices.

Summarizing, XRD analysis of the irradiated superlattices show that the structure is essentially preserved in all dose range, with no signs of amorphization or cationic displacement. Single films show a systematic increase in the c-lattice parameter upon irradiation, which is not observed in thin one unit cell YBCO layers in superlattice structures. This result can be explained in terms of the strained state of the very thin YBCO layer in the superlattice. Both irradiated films and superlattices show persistent photoconductivity, which allows to conclude that defects are related to oxygen displacements in the Cu–O chains.

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