Modifications of the physical properties of the high-T_c superconductors $YBa_2Cu_3O_{7-\delta}$ ($0.1 \leq \delta < 0.7$) by 3.5-GeV xenon ion bombardment

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The effects of 3.5-GeV Xe ion irradiation on the superconducting (T_c,J_c) and normal (R) properties of the high-T_c superconductors $YBa_2Cu_3O_{7-\delta}$ with $0.1 \leq \delta < 0.7$ have been investigated by means of *in situ* resistance measurements between 4.2 and 105 K. It is shown that the decrease of the T_c offset and the concomitant increase of the resistance in the normal state are strongly enhanced by inelastic collisions which lead to the production of discontinuous tracks of extended defects visualized by high-resolution electron microscopy. These defects act as pinning centers for the flux lines and result in the improvement of the critical current density J_c by a factor of 3.5 ($H=0,T=5$ K) for a Xe fluence of 2.5×10^{11} cm^{-2}. The greater sensitivity to irradiation damage of the phases corresponding to low oxygen contents ($\delta=0.46$ and 0.62) has been attributed to the particular structure of these compounds which exhibit, as has been previously reported, intergrowths of insulating $YBa_2Cu_3O_6$ regions or chains with superconducting $YBa_2Cu_3O_7$ regions or chains.

INTRODUCTION

It is now well established that the mixed valence of copper and bidimensionality are necessary for superconductivity in copper oxides. It is the reason for the crucial role of oxygen nonstoichiometry in these materials. The high-T_c superconductor $YBa_2Cu_3O_{7-\delta}$ is thus a good example of this dramatic influence of the oxygen content upon the value of the T_c offset. Although all results reported in the literature do not always coincide due to synthesis conditions including thermal processes and grain sizes of the polycrystalline samples, it is well accepted that T_c decreases drastically from 92 K for $\delta=0$ to 22 K for $\delta > 0.6$, the value beyond which superconductivity disappears.[1-3] From a crystallographic point of view, a model based on a continuous transition from the orthorhombic $YBa_2Cu_3O_7$ structure to the tetragonal $YBa_2Cu_3O_6$ structure has been proposed. It supposes the occurrence of disordered intergrowths of "O_6" insulating regions or chains with "O_7" superconducting regions or chains which have been more precisely investigated by high-resolution electron microscopy.[4] They could involve the creation of both oxygen and copper vacancies. Such a model supports the partial disproportionation of Cu II into Cu III and Cu I for all the δ range ($0 < \delta \leq 1$) for which noticeable amounts of Cu I have been indeed observed, whatever δ, by x-ray-absorption measurements[5,6] simultaneously with Cu in a $3d^9L$ configuration (L for ligand hole). In that field of research, irradiations provide another way to investigate the influence of point defects and/or extended defects on the physical properties of the new high-T_c superconductors. Numerous results on the effects caused by irradiation and implantation of the new superconducting oxides have already been published.[7-15] In general, one observes a rather high sensitivity of the critical temperature to radiation damage, T_c being thus depressed below 4.2 K for a number of displacements per atom (dpa) of about 0.02, i.e., 2 orders of magnitude less than $A15$ superconductors.[16,17]

We have recently shown that such a tendency could be reversed in the case of the grain surface superconductor La_2CuO_4 (Ref. 18) which exhibits indeed a T_c increase after irradiation by 3.0-GeV krypton ions. This improvement of T_c has been attributed to high sensitivity to the radiation-induced defects of the magnetic structure (spin-density wave) of the grain core with regard to the superconducting behavior of the grain surface.

A point which should be considered to interpret our results, concerns the damage production of high-energy heavy ions in copper superconducting oxides, and more precisely the role of electronic excitation on the defect production. Indeed, recent results obtained at the heavy-ion accelerator GANIL in Caen, demonstrate that in metallic amorphous alloys, for example,[19] the damage rate shows a drastic increase when the energy loss by electronic excitation passes a threshold value.

This is the case for 3.5-GeV xenon ions which exhibit, as it has been previously shown[20] electronic stopping power 2000 times higher than nuclear stopping power over about 90% of the ions path. This paper deals with the modifications induced by such energetic ions in sintered high-T_c superconductors $YBa_2Cu_3O_{7-\delta}$ with $0.1 \leq \delta < 0.7$.

EXPERIMENT

Sample preparation and characterizations

Sintered samples of $YBa_2Cu_3O_{6.9}$ were prepared from reagent grade powders of CuO, Y_2O_3, and $BaCO_3$ using the conventional ceramic procedure: Heating of the mixture at 1173 K in air for 12 h, cold pressing into a pellet of 10-mm diameter and sintering at 1223 K for 12 h in an alumina boat. The resulting pellet was sliced into rectangular bars of size $5\times1\times0.1$ mm^3 by means of a wire saw and subsequent mechanical thinnings. The bars were then annealed at 673 K for 24 h under an oxygen flow of 3 l/h and finally cooled slowly in the furnace down to the room temperature.

Sixteen bars were thus obtained from the initial pellet and divided into four sets in order to prepare specimens with different oxygen contents $(7-\delta)$ by means of the following procedure. First, the bars of a same set were heated up to 773–973 K in the furnace of a SETARAM microanalyzer at a scanning rate of 5 °C/min under an argon flow. When the desired value of δ was reached $(0.1 \le \delta < 0.7)$, the samples were cooled to room temperature. Second, one of the bars of each set was reduced by heating the TGA apparatus from 298 to 1173 K in a mixed atmosphere of 4 vol.% H_2 in Ar. In this way the oxygen deficiency with respect to the starting material could be determined quite accurately (± 0.02).

The samples were characterized before irradiation by powder x-ray diffraction using Cu $K\alpha$ radiation. Resistivity measurements were made at constant current by a standard four-probe method in a variable temperature cryostat.

Magnetization $M(H)$ measurements $(T=5$ K) were carried out with the help of a vibrating sample magnetometer PAR 155 equipped with a 4.2–300 K He-flow cryostat.

The diamagnetic shielding and Meissner effect were measured using superconducting quantum interference device (SQUID) technique and a magnetic field of 50 Oe for samples irradiated at 300 K.

Irradiation procedure

The irradiations were performed at the heavy-ion accelerator GANIL in Caen with 3.5-GeV xenon-ion flux limited to 5×10^8 ions cm^{-2} s^{-1} in order to avoid any warming effect during irradiation. This was carried out at 105 K in a He-flow cryostat equipped with a composite linear temperature sensor (CLTS) resistance for the temperature regulation. The bars were irradiated along their smallest dimension which was less than the projected range of 3.5-GeV Xe ions, accounting for a mean packing density of 0.8. As a consequence the electronic energy loss effect which varies from 18 MeV/μm at the entrance to 24 MeV/μm at the ion exit could be studied in a pure manner. The *in situ* resistance measurements were made during beam stops. Each time a predetermined value of the fluence was reached between 5×10^{11} and 10^{13} Xe ions/cm^2, a full resistance versus temperature curve was recorded between 4.2 and 105 K, the temperature being measured by means of carbon glass and a copper constantan thermocouple.

Furthermore, irradiations were also carried out at 300 K in order to investigate the radiation effects by magnetic measurements and electron microscopy.

RESULTS

Figures 1 and 2 give, as examples, the results which characterize the variation of the resistance of a $YBa_2Cu_3O_{6.9}$ sample as a function of the temperature before and after irradiations at 105 K. With increasing xenon fluence the following main features can be recognized:

(i) The superconductivity is destroyed for relatively high fluences ($\phi t > 6\times10^{12}$ Xe ions/cm^2).

(ii) The T_c offset decreases more quickly than the T_c onset leading to a transition width δT_c (taken from 10% to 90% of R at the T_c onset) which increases from 1 K before irradiation to 45 K at the fluence $\phi t=9.1\times10^{12}$ Xe ions/cm^2.

(iii) The resistance at 105 K increases by 2 orders of magnitude between the virgin sample and the sample irradiated at a dose of 6.9×10^{12} Xe ions/cm^2 which corresponds to the T_c suppression below 4.2 K. It is noticeable that the slope dR/dT which indicates a metallic state for the virgin samples, gradually decreases toward negative values. The irradiated samples show, above the superconducting transition, semiconducting behaviors (Fig. 2).

The rate of suppression of T_c with fluence $(dT_c/d\phi t)$ and the increase of δT_c clearly depend on the oxygen deficiency of the compounds as it can be seen in Fig. 3. The increase of both parameters is, thus, larger for samples of low oxygen content, i.e., $\delta=0.46$ and 0.62 which show a T_c suppression below 4.2 at smaller fluences ($\phi t > 2\times10^{12}$ Xe ions/cm^2) than that of $YBa_2Cu_3O_{6.9}$. Further, the sensitivity to irradiation of the oxygen-deficient oxides is also detected on the variation of the resistance in the normal state (Fig. 4). In this figure, one can note that the resistances increase exponentially with the fluence in accordance with a metal-semiconductor transformation.

Beside such modifications of the transport properties, enhancements of the critical current density have often been reported after irradiation. This improvement originates from the defects which act as pinning centers for the

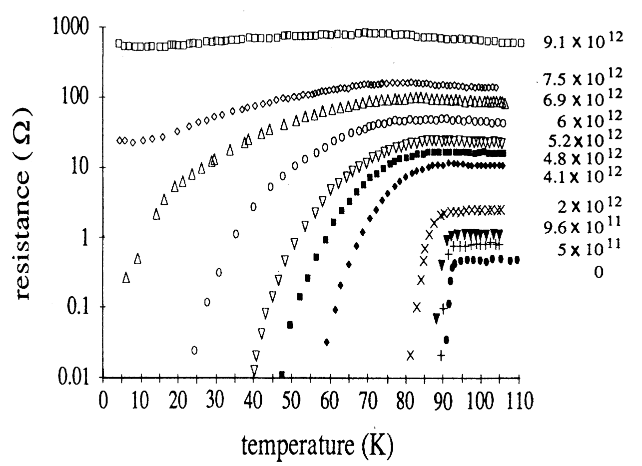

FIG. 1. *In situ* resistance measurements vs temperature for $YBa_2Cu_3O_{6.9}$ bars irradiated at 105 K by 3.5-GeV xenon ions for different fluences.

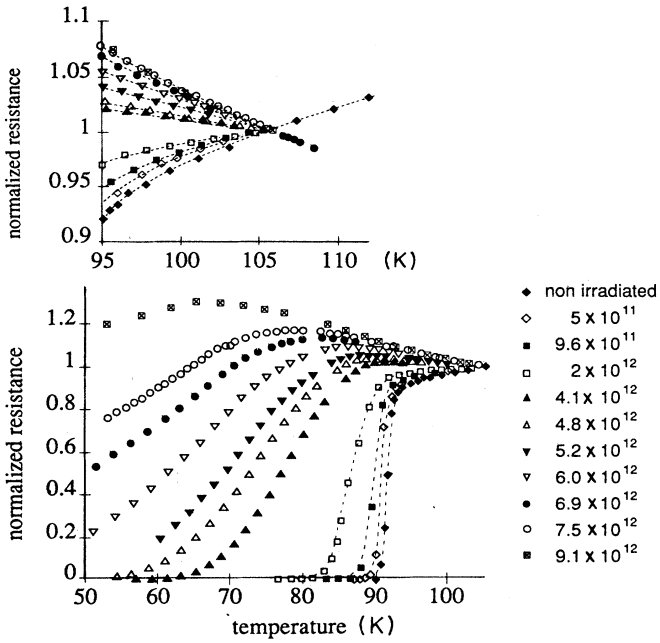

FIG. 2. Normalized transition curves of $YBa_2Cu_3O_{6.9}$ irradiated by 3.5-GeV xenon ions. The upper figure is a magnification of the 100 K region. Note the continuous transition from metallic to semiconducting behavior.

flux lines.

Magnetization curves recorded at 5 K in fields up to 15 kG have been reported in Fig. 5 for $YBa_2Cu_3O_{6.9}$ before and after irradiation. We observe an increase of the magnetic hysteresis for the irradiated samples with the increase of the fluence which is consistent with the critical state of hard superconductor. In such a state the penetrated flux lines are pinned on the defects produced here by irradiation.

Using the general formula of Bean[21] for a bar having its largest dimension along the applied magnetic field, the critical magnetization current density J_c can be evaluated

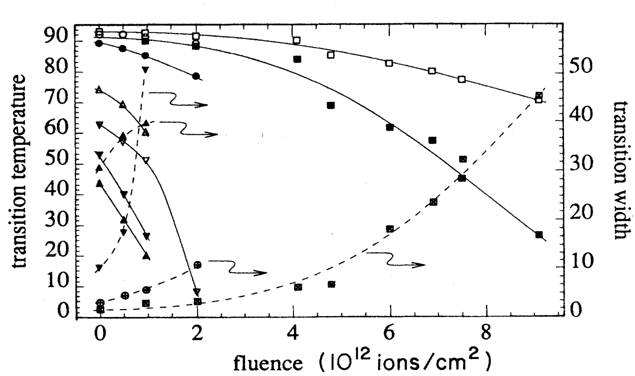

FIG. 3. The reduced transition temperature T_c/T_{c0} (full symbols correspond to 10% while empty symbols to 90% transition) and the transition width δT_c vs fluence for $YBa_2Cu_3L_{7-\delta}$ oxides [δ=0.10 (□,■), 0.28 (○,●), 0.46 (▽,▼), 0.62 (△,▲)] irradiated by 3.5-GeV xenon ions at 105 K.

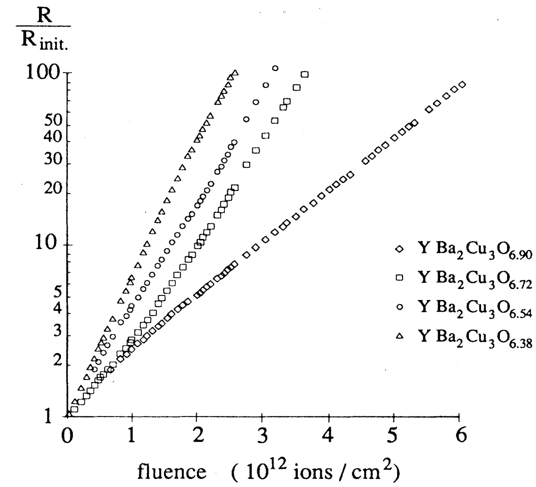

FIG. 4. Exponential variations of the resistance normalized to its initial value at 105 K for $YBa_2Cu_3O_{7-\delta}$ oxides (δ=0.10, 0.28, 0.46, 0.62) irradiated by 3.5-GeV xenon ions.

from the magnetization curves using the relation

$$J_c = \frac{20}{e}\left[\frac{3l}{3l-e}\right](M^+ - M^-)$$

where M^+ and M^- are the sample magnetization in the increasing and decreasing field respectively, l and e being the width and the thickness of the bar.

The reduced values of $J_c (J_c/J_{c0})$ have been reported in Fig. 6 versus the applied fluences for $T=5$ K and $H=0$. It is seen that J_c increases with ϕt, reaching a maximum value at a fluence as low as 2.5×10^{11} Xe ions/cm^2 and then begins to decrease slowly but keeping a value which stays higher than that of the virgin sample even at a fluence as large as 2×10^{12} Xe ions/cm^2. The high fluence decrease ($\phi t > 3\times10^{12}$ Xe ions/cm^2) is a result of general degradation of the superconducting material as shown here above. When J_c becomes again equal to $J_{c0}(\phi t = 5\times10^{12}$ Xe ions/cm^2) the T_c offset has been depressed

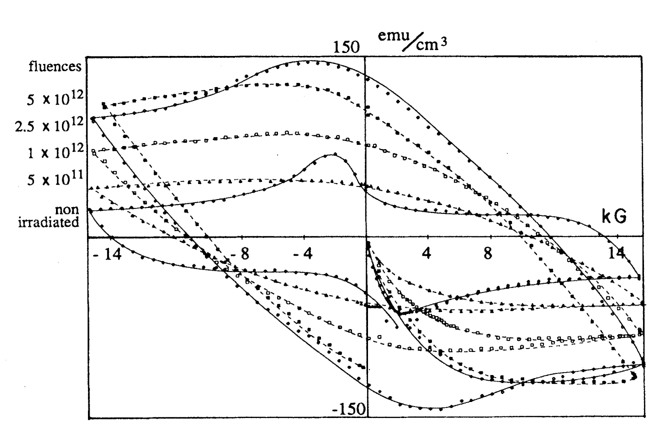

FIG. 5. Magnetic moment vs applied magnetic field at 5 K for $YBa_2Cu_3O_{6.9}$ samples irradiated by 3.5-GeV xenon ions at 300 K for different fluences.

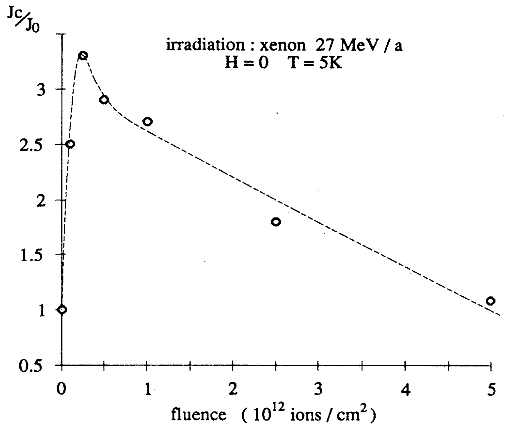

FIG. 6. Critical magnetization current density J_c/J_{c0} vs fluence at 5 K and zero field for $YBa_2Cu_3O_{6.9}$ samples irradiated by 3.5-GeV xenon ions at 300 K. The dashed line is only a guide to the eye.

to about 40 K (82 K for the T_c onset).

Meissner-effect curves which are a measure of the flux expulsion on cooling through T_c in an applied magnetic field of 50 Oe are presented in Fig. 7 for $YBa_2Cu_3O_{6.9}$ after some irradiations at 300 K. The Meissner-effect diamagnetism decreases monotonically with fluence because of the additional pinning defects generated by irradiation. The flux expulsions calculated for a compacity of 1, correspond to 84%, 81%, 72%, and 59% for xenon fluences of 0.5×10^{11}, 10^{12}, and 5×10^{12}, respectively.

DISCUSSION

Figures 2 and 4, which show the variation under irradiation of the resistance normalized to its initial value in the normal state, indicate that these copper oxides cannot be considered as classical metals. In contrast to classical metals, the defects do not only act as diffusion centers but modify also the transport mechanisms which are finally those of semiconductors. They could induce local atomic rearrangements with localized carriers leading to large perturbed potentials in the CuO conducting planes. The bidimensional character of the conductivity which enhances the effect of the defect perturbation should also be considered.

The greater sensitivity to irradiation of the low oxygen content superconductors is probably due to the particular structure of these compounds which exhibit, as it has been described above intergrowths of insulating $YBa_2Cu_3O_6$ regions or chains with conducting $YBa_2Cu_3O_7$ regions or chains. As a consequence, it should be assumed that the insulating parts of the matrix will be more sensitive to the ionization processes induced by electronic stopping than the conducting ones. As in insulators, the atoms surrounding the Xe-ion trajectories would be preferentially ionized in these regions, i.e., a Coulomb explosion would occur involving outward motions of ions which would then induce atomic displacement and disorder in the neighboring conducting $YBa_2Cu_3O_7$ regions, resulting in a rapid increase of the resistance. However, further experiments would be necessary to explain, in more detail, such resistance variations.

As shown in Fig. 3, the rate of the T_c suppression for $YBa_2Cu_3O_{6.9}$ sample is small at low fluences up to 2×10^{12} Xe ions/cm^2 and increases suddenly in the high fluence region. The rates are thus about -4.5×10^{-12} K/(Xe ions/cm^2) for $\phi t < 3\times10^{12}$ cm^{-2} and -16×10^{-12} K/(Xe ions/cm^2) for $\phi t > 3\times10^{12}$ cm^{-2}. Moreover, it should be pointed out from the Fig. 8 that the T_c offset decreases more rapidly than the T_c onset which stays nearly constant up to 3×10^{12} Xe ions/cm^2. Above this Xe fluence, the T_c onset starts indeed to diminish monotonically with a rate of about -5×10^{-12} K/(Xe ions/cm^2).

The existence of such a threshold fluence value led us to assume the existence of a threshold density of defects above which there are no more continuous superconducting paths in the undamaged zones of the grains. Of course, below this critical defect density, the grain boundaries are much more affected leading to the impossibility to find paths with zero resistance between the grains and thereby to a rapid depression of the T_c offset below 4.2 K.

According to x-ray analysis of $YBa_2Cu_3O_{6.9}$ bars irradiated at 300 K between 5×10^{11} and 5×10^{12} Xe ions/cm^2, it is worthy to note that the undamaged parts of the samples remained orthorhombic. Nevertheless, one observes that the line intensities decrease with increasing Xe fluence which can be attributed to smaller amounts of the superconducting phase due to the extent of disordered zones and displacements of atoms which no longer contribute to the coherent scattering. Moreover, one can see no significant change both in the angular positions of the diffraction lines and in the intensity ratio of the lines (020) and (006) to the line (200) which would indicate a orthorhombic to tetragonal transformation during irradiation as has been reported by Egner *et al.*[22] for $YBa_2Cu_3O_7$ thin films irradiated by H, He, and Ar ions.

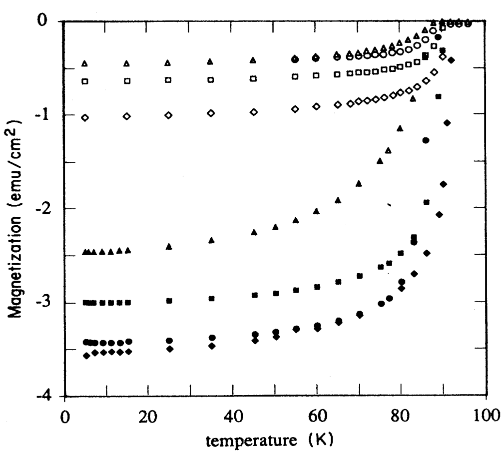

FIG. 7. Magnetization as a function of temperature for $YBa_2Cu_3O_{6.9}$ samples irradiated by 3.5-GeV xenon ions at 300 K (◆, nonirradiated; ●, 5×10^{11} Xe ions/cm^2; ■, 10^{12} Xe ions/cm^2; ▲, 5×10^{12} Xe ions/cm^2). The upper curves are for the samples cooling in a field of 50 Oe (Meissner); the lower curves are for warming in 50 Oe after cooling in zero field (shielding).

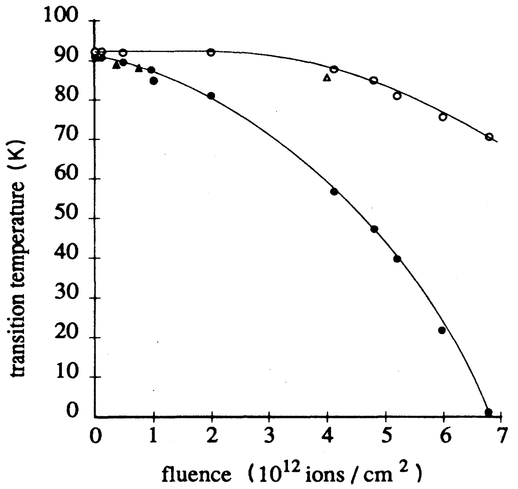

FIG. 8. T_c onset and T_c offset vs fluences for $YBa_2Cu_3O_{6.9}$ samples irradiated at 105 K. The circles correspond to Xe irradiation (⊗ is a room-temperature Xe irradiation), and the triangles to Kr irradiation.

Examinations of microcrystals obtained from the bar irradiated at a fluence $\phi t = 10^{12}$ Xe ions/cm^2 at 300 K by electron diffraction lead to the same conclusion. The electron-diffraction patterns show indeed, beside the orthorhombic arrangement of white dots, the arising of streaks along [100], [010], and [110] directions which involve some local variations of oxygen content and copper coordination. In order to elucidate the nature of the atomic transformations responsible for the T_c decrease and δT_c increase, a high-resolution electron microscopy (HREM) investigation has been performed in addition to the electron-diffraction study. The results will be described in more detail in an upcoming paper. It should be outlined here that they clearly established the occurrence of similar contrast modulations as those previously observed in oxygen-deficient $YBa_2Cu_3O_{7-\delta}$ oxides[4] and corresponding to a doubling of the "a" parameter of the orthorhombic cell. They were interpreted as the intergrowth of $[CuO_2]_\infty$ and $[CuO]_\infty$ chains as sketched in Fig. 9, implying breaking of Cu(1)-O-Cu(1) chains which play, as it is well accepted, a crucial role in superconductivity either directly by carrying the current or indirectly by providing the source for the hole coupling mechanism.

Therefore, it can be concluded that oxygen atoms removed from their lattice site during diffusion by heating in vacuum or oxygen atoms displaced towards interstitial lattice sites during irradiation cause the same detrimental effect on superconductivity. At higher fluences, further displacements of oxygen and copper atoms lead to local disorder and then to the amorphization achieved for $\phi t = 10^{13}$ Xe ions/cm^2.

Similar radiation effects, decrease of the T_c offset and strong broadening of the transition width δT_c, have been reported elsewhere for neutron irradiations.[12,13,23] On the contrary electron irradiations lead to a general shift of the full R-T curve with a transition width remaining approximately constant.[8,24]

From a macroscopic point of view, it has been argued that electron irradiations which formed the simplest point defects, i.e., vacancies and interstitials of oxygen and copper atoms, affect mainly the intragrain (bulk) properties of the superconductors whereas neutron irradiations which produce more extended defects into collision cascades affect more strongly the intergranular links which govern the T_c offset. In both cases it is clearly demonstrated that the damage rate is caused by nuclear energy loss processes with a T_c reduction below 4.2 K observed for a number of displacements per atom (dpa) of about 0.02 dpa. Similar results have been described for ion implantation in thin films of $YBa_2Cu_3O_7$.[13–15,22]

3.5-GeV xenon irradiation strongly enhances the T_c decrease per dpa (ΔT_c/dpa), since a value of two orders of magnitude higher than that calculated for neutron, electron, and low-energy ion irradiations is obtained, which obviously established the crucial role of the electronic stopping power in the defect-creation mechanism. This is confirmed by the observation of HREM photographs[25] of discontinuous tracks of extended defects about 60 Å in size similar to those which appeared in magnetic insulators irradiated by 3-GeV Kr and 3.5-GeV Xe ions[26,27] when the energy deposited by electronic stopping stood below the threshold value necessary for the production of continuous latent tracks.

Accounting for magnetization measurements, such extended defects act as pinning centers for flux lines and reduce the sizes of the Meissner and shielding effects as shown in Fig. 7. Further they are also responsible for the hysteretic effect observed on the $M(H)$ curves (Fig. 5) and therefore of the enhancement of the critical magnetization current density as pictured Fig. 6. The results have to be compared with that reported for neutron irradiations. According to Cost *et al.*,[11] measurements of J_c at 7 K and zero field show an increase by a factor of 2.5 at 3×10^{18} neutrons/cm^2 while a decrease of the critical current density measured inductively is described by Küpfer *et al.*[10] for $\phi t = 4\times10^{18}$. It has been argued that magnetization measurements gave the intragrain J_c which is expected to increase by irradiation owing to strong pinning effects inside the grain whereas ac susceptibility mea-

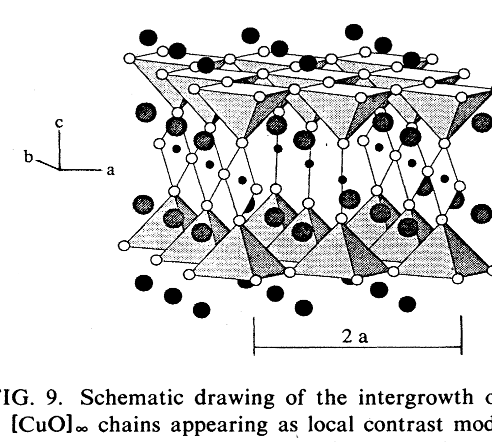

FIG. 9. Schematic drawing of the intergrowth of $[CuO_2]_\infty$ and $[CuO]_\infty$ chains appearing as local contrast modulations in $YBa_2Cu_3O_{6.9}$ microcrystals irradiated by xenon ions at a dose of $\phi t = 10^{12}$ cm^{-2}.

surements led to the intergrain J_c more sensitive to the weak structure of the intergranular material.

In the case of single crystals where the question of granularity can be discarded, it has been found by Umezawa *et al.*[9] that neutron irradiation up to a fluence of 8.1×10^{17} neutrons/cm^2 systematically improved the magnitude of the critical magnetization current and reduced its anisotropy by a factor of 2 in the *a-b* plane with regard to the *c* direction.

CONCLUSION

High-density electronic excitation induced by 3.5-GeV Xe ions causes drastic changes of the superconducting and normal properties of the high-T_c oxides $YBa_2Cu_3O_{7-\delta}$ despite their metallic character in the normal state.

Such a behavior could be explained on the basis of the layer structure of the 1:2:3 compounds which exhibit indeed in the *c* direction a sequence of metallic and insulating slices. In particular it should be outlined that the Cu(1)-O-Cu(1) chains which play a crucial role on superconductivity are surrounded by insulating BaO layers which will be more sensitive to ionization processes induced by electronic stopping as it has been previously shown in magnetic insulators.

As a consequence it can be assumed that secondary displacement collisions could occur in Cu(1)-O chains leading to local disorder and thereby to degradation of T_c and R. Moreover, the T_c onset decrease being considerably less than that of the T_c offset, such effects should affect the intergrain couplings more strongly than the intragrain properties. However, the intragrain pinning is improved even at very low fluences leading to the increase of the critical magnetization current J_c by a factor of 3.5 for an electronic energy loss of about 0.05 eV/Å^3.

Radiation damage investigation by high-resolution electron microscopy of low-dose irradiated $YBa_2Cu_3O_{6.9}$ specimens gives evidence of the formation of discontinuous tracks of extended defects which will be described in a next paper.

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