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Pinning phenomena and critical current in proton-irradiated sintered $YBa_2Cu_3O_{7-\delta}$

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In this contribution we deal with proton-irradiation effects on $YBa_2Cu_3O_{7-\delta}$ sintered samples. We irradiated slab-shaped samples with 3.5 and 6.5 MeV proton beams and with fluences ranging from 1.8×10^{16} p/cm^2 to 8.6×10^{16} p/cm^2. We analyzed either intragranular or intergranular properties before and after irradiation by means of magnetic and electric transport measurements. We found that it is possible to enhance both intragranular and intergranular critical current densities with proton irradiation. The optimal ranges of fluences for enhancing intragranular and intergranular J_c are different. By focusing our attention on the possibility of enhancing the transport J_c of bulk materials we show that suitable proton induced defects significantly contribute to the pinning of vortices in intergrain networks, as it is demonstrated by means of different experimental techniques and on different sets of samples. We observed that the enhancement presents nonmonotonic behavior. On the contrary, modulations as a function of field and temperature emerge. Moreover, low-temperature magnetization measurements either show modulations of the intergrain critical current density as a function of field or the appearance of plateaulike features. These results are discussed in the framework of static properties of disordered long Josephson junctions networks. [S0163-1829(96)09729-9]

I. INTRODUCTION

On the basis of all their properties, the polycrystalline high-temperature superconductors (HTSC) oxides should be modeled as an agglomeration of random oriented single crystals or islands with strong superconductivity having their order parameters coupled via Josephson contacts.[1] The large screening currents are to be attributed to the strong intragrain superconductivity, and the transport currents to the weak intergrain superconductivity. The transport critical current, essentially limited by the depinning of the intergrain Josephson vortices, shows a very strong field dependence with a sharp decrease (about one order of magnitude) at very low applied magnetic fields[1–3] with a strong weakening of the Meissner effect at approximately the same fields. These fields roughly correspond to the first average critical Josephson field.

It is generally accepted that the most suitable procedure to increase the critical currents in HTSC is the creation of an optimum density of defects with the right dimension and distribution within the material. Irradiation experiments are one of the most effective methods to produce defects in a rather controlled manner, and hence, of knowing what defect size and distribution produce higher critical currents in these materials. Several groups found an increase of the critical currents in $YBa_2Cu_3O_{7-\delta}$ single crystals upon neutron,[4–6] proton,[7,8] electron,[9,10] and heavy ion irradiation.[11–13] In addition, similar results have been obtained (not only for Y compounds but also for Bi compounds[14–16] and other oxides[17]) also on textured epitaxial thin films,[18,19] textured bulk materials[20] and tapes[13] as well as on *intragrain* currents in sintered samples.[21–23]

On the contrary, concerning transport *intergrain* critical currents in polycrystalline materials, the possibility to obtain, by irradiation, enhancements prevailing on weak link damage is critically dependent on the preirradiation material quality, i.e., on the prevailing of strongly coupled grains on voids and weakly coupled domains.[24–28]

However, sintered materials allow us to study and compare, under exactly the same extrinsic defect distribution, the behavior of the *strong superconductivity* (connected to the single crystal characteristic length scales and to Abrikosov three-dimensional vortices), with the performances of the *weak superconductivity* (connected to different defect networks involved in Josephson-Junction networks). Moreover, good quality polycrystalline HTSC offer a potential field of improvement towards easy applicability.

As a consequence, in the framework of our investigation of flux pinning induced by proton irradiation on sintered materials, we have first focused our attention on comparing the irradiation induced effects on grain properties and on coupled domains within the same irradiation experiment. Such a comparison allows to conclude that a different range of fluence is effective in enhancing intragrain and intergrain critical currents. The simultaneous observation and comparison between both intragrain and intergrain pinning properties gives valuable information on the way the pinning works out.

As a second point, we have specifically looked at the experimental possibility to enhance the transport critical currents of bulk materials by proton induced defects with suitable density. In this paper we show that proton induced defects, or, more likely, their clusters and strain fields created by irradiation, significantly contribute also to the pinning of vortices in intergrain networks, as it is demonstrated by means of different experimental techniques and on different sets of samples.

We obtain the field dependence of these enhancements. A quite common characteristic of the field dependence, as deduced from results obtained by means of all the techniques employed, is that the enhancement presents a nonmonotonic behavior. Moreover, at low temperatures, the after irradiation critical currents present well distinguished modulations at increasing fields above the first average critical Josephson field, H_{c0}. These results are discussed in the framework of static properties of disordered long Josephson junctions (LJJ) networks, with a critical current field dependence modulated by a suitable distribution of vortex density.

Finally, some results obtained on proton-irradiated samples by means of contact measurements, such as voltage-current characteristics, are reported and discussed.

II. EXPERIMENTAL DETAILS

A. Proton irradiation

Irradiation experiments were performed at room temperature in vacuum at the APT scattering chamber of the 7 MV Van de Graaff CN accelerator of LNL-INFN Laboratori Nazionali di Legnaro. Our studies have centered on proton fluences ranging from 1.8 to 8.6×10^{16} p/cm^2 with proton energy of 6.5 MeV. Some irradiation has also been made with 3.5 MeV proton beam. The data of Civale *et al.*[7,11] and of Kirk[29] on single crystal, as well as previous experiment on sintered materials,[23] have been used to select the range of proton fluences at which to perform magnetization and transport measurements.

For 6.5 MeV protons the estimated penetration depth on our samples is about 215 μm and the volume averaged distance between primary knocks in elastic scattering ranges from 20 to 60 Å. For 3.5 MeV the penetration depth is about 85 μm and the volume averaged distance between primary knocks in elastic scattering ranges from 50 to 20 Å. On the other hand, the distance between defects produced by each incident proton ranges from 30 to 1 μm for 6.5 MeV protons and from 20 to 1 μm for 3.5 MeV. All these values have been evaluated by means of a Monte Carlo TRIM simulation.[30] Defect densities increase as the depth increases and the energy decreases down to implantation. As long as protons implant in the bulk and the irradiation affected percent volume is quite the same, no substantial differences are found among the damages produced by 6.5 and 3.5 MeV protons, respectively.[31]

High resolution TEM images of 3.5 MeV proton damage performed on single crystal and reported in literature showed bent or distorted lines of atom columns in the beam direction, with highly anisotropic strain field. The estimated dimension of each pointlike defect is less than 10 Å,[32] although after irradiation with a dose of 2×10^{16} p/cm^2 analysis showed also the presence of small clusters about 30 Å in size.[7] We expect therefore that for both the energies used in the paper, along a single proton track, a lattice consisting of weakly linearly correlated defects with decreasing period sets up, the effectiveness of the correlation only slightly increasing with energy. Due to the produced strain fields, these defects mainly extend in the direction perpendicular to the incident beam.

B. Samples

Three different sets of slab-shaped sintered $YBa_2Cu_3O_{7-\delta}$ slices were used.

(a) Four twin slabs (labeled *S* samples), 0.60 mm thick, irradiated with a proton energy of 6.5 MeV and fluences of (0, 1.8, 3.8, 8.6)$\times10^{16}$ p/cm^2, respectively. These samples were used for magnetic measurements.

(b) Two twin slabs (*A* samples), 0.20 mm thick, cut from a different batch and both irradiated with 3.5 MeV protons. The first sample was irradiated with a fluence of 1.8×10^{16} p/cm^2, characterized and reirradiated with the same fluence (up to a total fluence of 3.8×10^{16} p/cm^2). The second one was irradiated with a fluence of 7.5×10^{16} p/cm^2. These samples were used for magnetic measurements.

(c) Three twin slabs (*W* samples), 0.50 mm thick, cut from a third batch, used for electric transport measurements and irradiated with 6.5 MeV protons and fluences of (0, 1.8, 3.8)$\times10^{16}$ p/cm^2, respectively.

By ''twin'' samples we mean specimens cut from a single pellet with the same accurately measured shape.

Magnetic measurements were performed by means of a Lake Shore 7225 susceptometer/magnetometer in magnetic fields up to 4000 kA/m. The magnetic field was applied perpendicularly to the proton beam direction and parallel to the largest sample surface. Contact measurements were performed by means of a homemade apparatus, equipped with a Keithley 182 Sensitive Voltmeter and suitable scanners for pulsed currents. For these measurements, silver contacts, laid in a four-point arrangement, were deposited under vacuum.

III. EXPERIMENTAL RESULTS

A. Intragrain characterization

The experiments were done on *S* and *A* samples irradiated with a fluence range from 1.8×10^{16} p/cm^2 to 8.6×10^{16} p/cm^2, i.e., in a region where the intragranular J_c vs fluence curve shows an increasing monotonic trend.[33] Hysteresis loops up to 4000 kA/m were measured at 5, 15, 25, 50 K for the two sets of samples. Intragranular critical currents were evaluated from the width of the loops by means of the Bean model.[34]

In Fig. 1 the dependence of the intragrain critical current enhancements is shown, as a function of the field, at three different temperatures, for the *S* samples. The average enhancement increases with fluence. At the lowest fluence ($\phi=1.8\times10^{16}$ p/cm^2), and particularly at higher temperatures, the enhancement is rather low or is concealed by a randomization induced by irradiation, which is always present in these experiments.

For the *A* samples the effect is qualitatively the same, with a more pronounced enhancement effect for the two higher fluences (ϕ=3.8 and 7.5×10^{16} p/cm^2). At the lowest fluence ($\phi=1.8\times10^{16}$ p/cm^2) intragrain critical currents are almost unaffected.

In fact, in the employed range of fields the intrinsic pinning structure itself contributes to the magnetic granularity, which manifests itself in the drop of the transport J_c. This drop is due to the appearance of closed loops within macroscopic crystalline grains, where densities of circulating magnetization currents become significantly larger than the vanishing transport J_c. Extrinsic defects provide more pinning

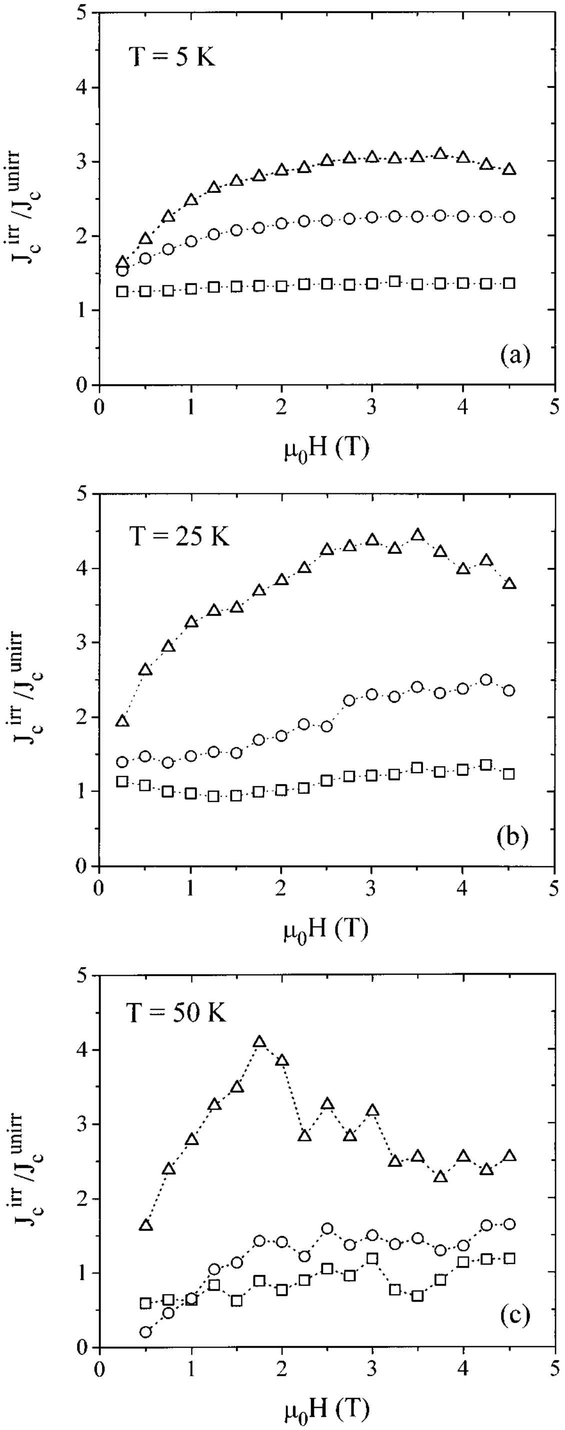

FIG. 1. Intragrain J_c enhancements vs magnetic field at $T=5$ K (a), 25 K (b), 50 K (c) calculated from field hysteresis loops up to 5 T for samples $S1$ (squares), $S2$ (circles), $S3$ (triangles) [irradiated with $(1.8, 3.8, 8.6)\times10^{16}$ p/cm^2, respectively] with respect to the not-irradiated $S0$.

centers improving magnetization currents, as it is usually found in single crystal experiments.[33]

B. Intergrain characterization

The topic of the enhancement of intergrain critical current density has been rather controversial.[27,35–37] It is straightforward that in samples characterized by a high percentage of voids, misaligned grains, and spurious phases, the irradiation can only increase the disorder by damaging weak links and breaking useful percolative paths between grains.[25,27] Conversely, if we consider strongly coupled granular superconductors, such as those shown in Fig. 2, some enhancements can be induced (by the lowest defect densities), as anticipated in the Introduction.[38] Although different experimental techniques give information in different ranges of field, temperature, or in different vortex static or dynamic conditions, it is possible to stretch some common trends.

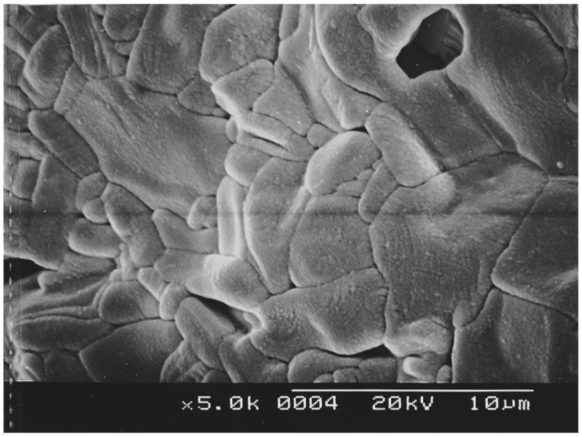

FIG. 2. S sample SEM micrography.

The intergrain characterization, for what concerns ''static'' behaviors, has been performed by means of dc low field hysteresis loops (up to 20 kA/m, i.e., up to a field lower than H_{c1} for grains). Based on the slope of the virgin curve, suitable corrections have been made in order to separate intergrain from intragrain contributions, the former associated with currents threading the weak links and the latter with shielding currents circulating inside the grains.[39] The intergranular critical current densities were thereafter calculated from the width of corrected loops by means of the Bean model.[34] The results show significant enhancements for the two lowest fluences in the S samples ($\phi=1.8$ and 3.8×10^{16} p/cm^2), setting up in given ranges of temperature and field, quite more pronounced at fields which are about 2 or 3 times the estimated value of the first average Josephson critical field (1–4 kA/m) as it will emerge from the below quoted Fig. 3. For what concerns the dependence on temperature of the enhancement peaks, a lowering of the values of such peaks from a maximum value of 3 at 15 K to a maximum value of 2 at 25 K is observed.[35]

Intergranular critical currents were also evaluated by ac susceptibility measurements in corresponding temperature ranges. The ac measurements also support evidence of intergrain enhancements. Also in this case the effect is more pronounced for the two lower fluences. At zero field, where the covered range of temperature is between 82 and 86 K, we obtain an average enhancement factor of 1.3. At a field of 4 kA/m the average enhancement is about 1.5, with a maximum value of 1.7 at 50 K.

Both dc and ac measurements on the A samples confirm qualitatively the S sample behaviors, as shown in Table I, where the main results on A samples, obtained by means of

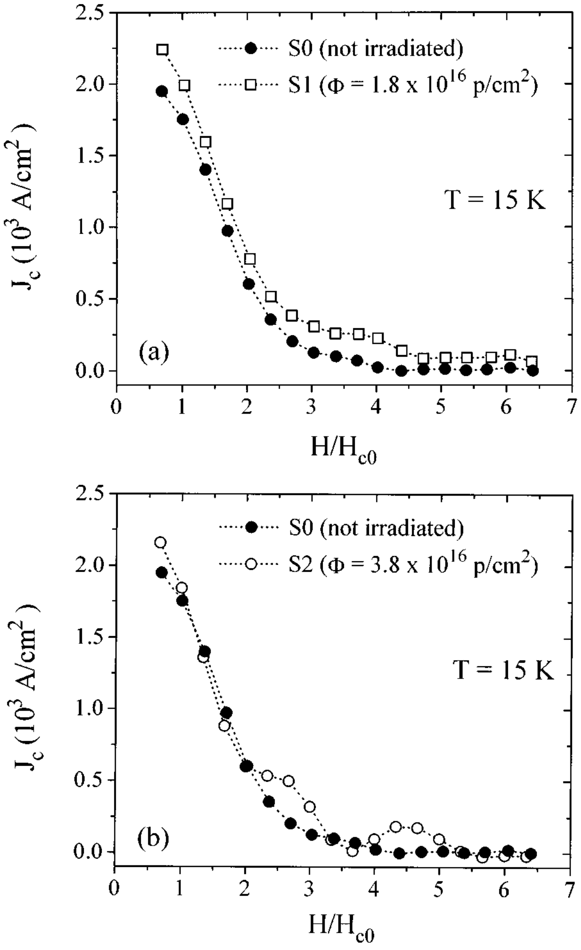

FIG. 3. Intergrain J_c vs magnetic field, as deduced from dc hysteresis loops, for sintered samples $S0$ (not irradiated) and $S1$ (a) and for samples $S0$ and $S2$ (b). H_{c0} is the first average critical Josephson magnetic field; its value is about 3 kA/m.

ac susceptibility measurements, are compared to those already mentioned for S samples.

More significantly, if we observe the behavior of J_c vs H curves before and after irradiation for S samples, as deduced from the above mentioned low field dc cycles at $T=15$ K, we observe some very characteristic features, such as a small plateau for the lowest fluence ($\phi=1.8\times10^{16}$ p/cm^2), and modulations for the subsequent one ($\phi=3.8\times10^{16}$ p/cm^2) (Fig. 3).

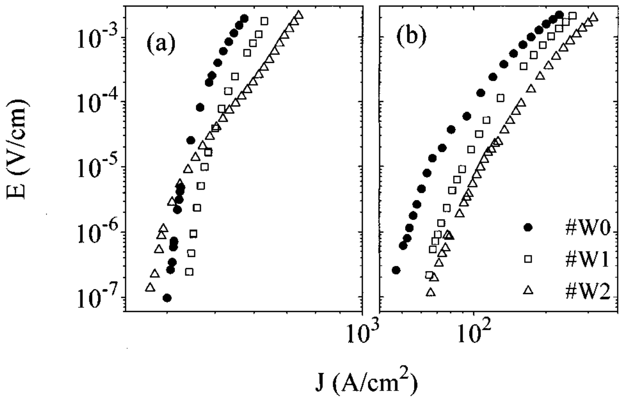

FIG. 4. E-J characteristics at 77.4 K for samples $W0$, $W1$, $W2$ [irradiated with (0, 1.8, 3.8)$\times$ 10^{16} p/cm^2, respectively] under different applied magnetic fields: $H_{\mathrm{dc}}=0$ A/m (a), $H_{\mathrm{dc}}=3760$ A/m (b).

Contact measurements on the W sample set give also an insight on the way this kind of defect operates (Fig. 4). As in the previously quoted experiment, the beam direction is orthogonal to the sample largest surface as well as to the applied magnetic field, while now the transport current flows parallel to the applied field. The sample irradiated with a fluence of 3.8×10^{16} p/cm^2 shows a crossover from damaged behavior at zero field to enhanced behavior at higher fields. In Fig. 5 J_c enhancements as a function of field are plotted; experimental errors due to geometrical uncertainties allow us to conclude only that the enhancement is higher when a magnetic field is applied.

IV. DISCUSSION

If we look at all the results, we deduce that enhancements can be obtained in intergrain critical current density. However, the rather low magnitude order of the J_c enhancement is certainly not as interesting, as the evidence that the ratio of J_c after and before irradiation is a nonmonotonic function of field. In our cleanest results an unmistakable defect-induced modulation of critical current density shows up as a function of the field.

The preirradiation structure is characterized by the usual defects with a large random distribution of size and position.[3] Over this distribution we implant an extrinsic defect structure which, due to a well defined unique beam direction and incident energy, introduces an anisotropy in the otherwise isotropic fully random defect distribution.[29] The main characteristics of 3.5 and 6.5 MeV proton-irradiation induced defects appear to be in their structure and resultant strain field, as established by the observation of TEM visible defects in single crystals, as referred in Sect. II A.

We assume that, during the irradiation of our sintered samples, some separate defects coagulate in more extended strain-induced defects in the direction perpendicular to the beam direction. Larger defects growing on the grain bound-

TABLE I. Intergrain J_c enhancements at $H=4$ kA/m at $T=50$ K calculated from ac susceptibility measurements for irradiated S samples with respect to the not-irradiated $S0$ and for irradiated A samples with respect to themselves before irradiation.

	$\Phi=1.8\times10^{16}$ p/cm^2	$\Phi=3.8\times10^{16}$ p/cm^2	$\Phi=7.5\times10^{16}$ p/cm^2	$\Phi=8.6\times10^{16}$ p/cm^2
S samples ($E_p=6.5$ MeV)	1.7	1.55		1.4
A samples ($E_p=3.5$ MeV)	1.12	1.05	0.85	

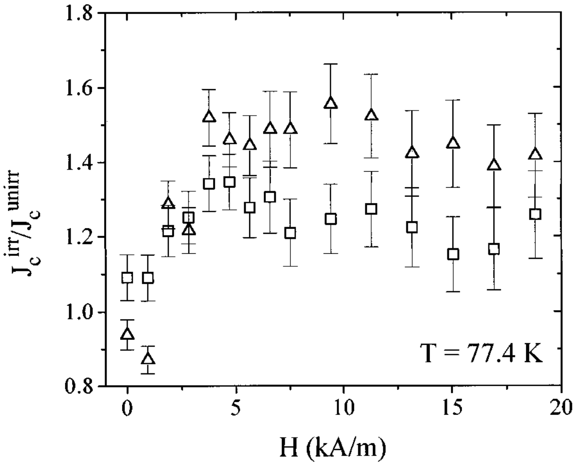

FIG. 5. Ratio between sample $W1$ (irradiated with 1.8×10^{16} p/cm^2) and sample $W0$ (not irradiated) critical current density (circles) and ratio between sample $W2$ (irradiated with 3.8×10^{16} p/cm^2) and sample $W0$ (not irradiated) critical current density (triangles). These ratios, plotted as a function of the applied magnetic field, were deduced from E-J characteristics by the 1 μV/cm criterion.

aries should have suitable dimensions for providing pinning centers for Josephson vortices. In general, both regimes in which the defects act as suitable pinning centers or block (divert) the current flow, coexist, their relative contributions depending on both the tunneling superconducting current and on the geometry of the pinning network.[40]

A realistic model for a strongly coupled granular superconductor was proposed by Rhyner and Blatter, and called limiting interface model.[41] Within this model, the critical current of a sample is limited by the depinning of Josephson vortices across a limiting interface of weakest superconductivity, which extends over the entire sample. Applied to the case of, e.g., an extremely good quality sintered specimen, the interfaces actually become critical paths, which represent an inhomogeneous macroscopic LJJ, whose length scale should be comparable with the sample thickness. We remind that the interdefect distances in intergrain transport paths are to be put in relation both to percolative transport paths and to Josephson junction characteristic penetration length.[42] On the contrary, the interdefect distance in intragrain critical currents must be related to the characteristic length scale of the average grain size, i.e., about 5–10 μm, as well as to the London penetration length λ_L ($\sim$0.5 μm).[37] This means that different length scales are involved in the two cases. If we change the coupling energies of the Josephson junctions by means of defects having distributions ranging from those of a periodic array to random distributions, we obtain different dependencies of the critical current density vs magnetic field. These behaviors depend on the defect ordering and sizes as well as on the typical length scale of the disorder.[1]

The case of periodic columnar defect in a wide LJJ is discussed in a paper of Tinkham,[43] where it is experimentally shown that a LJJ with periodic columnar defects exhibits low field behavior, identical to that shown from defect-free junctions. The appealing fact is that just as screening currents maintain zero field in the junction interior for small applied fields, they also serve to maintain commensurate-field values in the junction interior, when the applied field is close to one of these values. Thus the defected junctions present enhanced critical current densities at fields for which the number of flux quanta in the junction is an integer multiple of the number of defects. As the field is either increased or decreased from one of these commensurate values, well defined J_c peaks set up.

In an ideal case, every proton would produce exactly the same defect distribution no matter its impact point in the irradiation plane or time of incidence. Then planar correlation takes place among defects on surfaces perpendicular to the proton beam and parallel to the applied field. With reference to the damage evaluation, made in Sec. II A, the average distances between defects produced by each incident proton can be estimated about 10 μm, nicely matching the average grain dimension. Consequently the characteristic features of two spatially periodic systems, e.g., the periodic soft vortex lattice (VL) and the nearly periodic rigid defect lattice, should be found. The overlapping is accompanied by commensurability effects, such as the adjusting of the soft VL structure to the periodicity of the defects in order to maximize the pinning forces. As a consequence, at fields for which the VL becomes commensurate with the defect lattice, peaks in the J_c vs H curve should be observed. In the reality, this nice rigid correlated defect lattice does not set up with low energy protons on sintered materials. Then we expect J_c vs H curve features ranging from those obtained for periodic defect lattice to trends due to more random disorder, as provided from preirradiation defect structures.

Within this framework we analyze our experimental results. In the curve J_c vs H/H_{c0} plotted in Fig. 3(b), two peaks may be observed. The value of the first average Josephson magnetic field H_{c0} can be determined from the slope of the virgin magnetization curve.[44] For sample $S2$ at $T=15$ K, H_{c0} is about 3 kA/m and the first peak appears for a $H_{\rm peak}/H_{c0}$ value of about 2.5 and the second one for a $H_{\rm peak}/H_{c0}$ value of about 4.5.

Following Ref. 1, we write

$$r_0 \approx \pi\lambda_j \frac{H_{c0}}{\Delta H_{\rm peak}},$$

where $r_0=(s_p+d_p)$, s_p is the size of the pinning centers, d_p the distance among pinning centers, and $\Delta H_{\rm peak}/H_{c0}$ the peak distance. If we put $r_0\approx 10$ μm (i.e., equal to the average distance between defects produced by each incident proton) the magnitude order of the Josephson junction characteristic penetration length reaches a magnitude order of a few microns, in agreement with the literature values.[37] In Fig. 3(a) it is possible to see that, for sample $S1$ irradiated at the lowest fluence ($\phi=1.8\times10^{16}$ p/cm^2), a plateaulike feature emerges, i.e., a feature more similar to those produced by random disorder in a network of LJJ.

Correspondingly, in voltage-current characteristics, the crossover between damaged and enhanced behaviors shows the near recovery at suitable fields of the zero field dynamics of junctions in the transport network, due to fluxon propagation which has been seen previously only near zero field. These results can be interpreted in the framework of an equivalence to defect-free junction in zero field near commensurate fields, but with a reduced critical current density.[43]

V. CONCLUSIONS

There is much to be learned about flux dynamics and pinning by studying intragrain and intergrain critical current behavior under the same proton induced defect distribution.

Different ranges of fluences drive larger intragrain and intergrain critical current enhancements, respectively. In particular, lower fluences (ϕ=1.8 and 3.8×10^{16} p/cm^2), i.e., larger defect distances, can have a better matching with intergrain critical currents. These same fluences are rarely affecting intragrain critical currents. This demonstrates that intergrain critical currents, mainly dependent on weak pinning, i.e., Josephson junction pinning, operate better in particular LJJ length scales. The enhancements of intergrain critical current densities are depending on the field and temperature in a rather characteristic nonmonotonic way. Our cleanest low-temperature magnetization measurements show either modulations of critical current densities as a function of field or the setting up of plateaulike features. This effect nicely matches the general trend predicted by the Fehrenbacher-Blatter LJJ model.[1]

Electric transport measurements show, in the non-Ohmic region, a crossover from damaged to enhanced behavior, when a magnetic field is applied. This trend should represent a signature of those junctions with defects, which recover their defect-free behavior in the vicinity of commensurate-field values.[43]

Future work should be concentrated on the investigation of the same properties on defect-free preirradiation structures, as those obtained with high pressure sintered, rather well oriented materials. Defect structures matching commensurate fields seem now to be the most stimulating task in the field of high temperature superconductivity.

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