Superconducting fluctuating diamagnetism in neutron irradiated MgB_2 in relation to precursor diamagnetism in Al-doped MgB_2

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The fluctuation-related diamagnetism above the superconducting transition temperature T_c in neutron irradiated and in Al-doped MgB_2 is studied by means of high-resolution isothermal measurements of the diamagnetic contribution to the magnetization, $M_{dia}=M_{dia}(H,T=\text{const})$. In both the neutron irradiated and the Al-doped compounds, T_c decreases on increasing the fluence and the Al amount, respectively. The magnetic field dependence of M_{dia} is apparently similar in both types of compounds: in the limit $H\rightarrow 0$, $-M_{dia}$ goes as H^n (with n in between $\frac{1}{2}$ and 1), while by increasing the field above a given value H_{up}, an upturn in the field dependence occurs and $|M_{dia}|$ decreases. From the temperature behaviors of H_{up}, it is proved that the origin of the precursor diamagnetism is quite different in neutron irradiated and in Al-doped MgB_2. In the latter, the magnetization curves reflect the precursor diamagnetism typical of heterogeneous systems and unrelated to superconducting fluctuations, due to site dependence of the transition temperature. At variance, neutron irradiated MgB_2 displays different properties. The fluence-dependent transition temperature is practically site independent, the superconducting fluctuations and the related diamagnetism basically retaining the features of the pure (unirradiated) MgB_2. Upon irradiation, the anisotropy parameter involved in the fluctuation spectrum decreases. Correspondingly, also the upturn field H_{up} decreases, consistent with a less anisotropic coherence length in strongly irradiated compounds. The implications of these experimental findings on the disorders induced by heterovalent substitutions and by neutron irradiation in MgB_2 are discussed.

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I. INTRODUCTION

The superconducting properties of MgB_2 are presently of strong interest, both in view of the perspective technological applications and with regards to the mechanism underlying the occurrence of the superconductivity. On the other hand, these properties of MgB_2 are known to be heavily modified when some kind of disorder is introduced. The effects of heterovalent (Al for Mg) substitutions and of neutron irradiation on the transition temperature, the critical current, and the critical fields, on the two superconducting bands and the crossover from clean to dirty regimes, have been subjects of extensive studies (see Refs. 1–6, and references therein). The character of the disorder, related to grain boundaries, to pointlike pinning centers, and/or Al ions, is worthy of particular attention. From specific heat measurements and from the sharpness of the transition in neutron irradiated (nir) compounds, a homogeneous defect structure has been claimed with little, if any, change in the band structure.[7,8] In Raman spectra,[9] disorder-induced violations of the selection rules have been discussed in relation to disorder of similar nature both in nir and in Al-doped (ald) MgB_2 compounds.

In recent years, the fluctuating diamagnetism (FD) in superconductors and, in particular, the isothermal magnetization curves slightly above the transition have been proved to be useful tools in order to get insights on the disorder effects in terms of the site dependence of T_c and/or of the occurrence of phase-fluctuating, nonpercolating superconducting mesoscopic regions. On approaching the transition temperature from above, the superconducting fluctuations (SFs) imply the existence of metastable Cooper pairs, leading to a diamagnetic contribution to the magnetization (see Refs. 10 and 11). It has to be remarked that recent experimental findings in high-T_c cuprate superconductors and in MgB_2 also increased the interest toward SF, since small coherence length, reduced carrier density, high transition temperature, and anisotropy cause strong enhancement of the fluctuations.[12–15] On the other hand, it was also suggested that the SF can reflect the electronic properties of the superconducting state. As additional remarkable feature, in underdoped and overdoped $Y_1Ba_2Cu_30_{7\pm x}$, dramatic deviations from the conventional Ginzburg-Landau (GL) behavior are observed, leading to an anomalous diamagnetism justified on the basis of the existence of superconducting regions at nonzero order parameter strongly fluctuating in the phase.[14,15]

In pure, undoped and unirradiated MgB_2, the following features on FD have been evidenced:[12]

(i) For $T\approx T_c^+$ and in the limit of magnetic field $H\rightarrow 0$, the diamagnetic contribution to the magnetization goes as $-M_{dia}\propto H^{1/2}$.

(ii) In the magnetization curves $M_{dia}=M_{dia}(H,T=\text{const})$, at a temperature-dependent magnetic field H_{up}, the magnetization $|M_{dia}|$ begins to decrease on increasing field, due to field-induced quenching of the fluctuating pairs.

(iii) H_{up} increases with increasing the measuring temperature (above T_c).

(iv) The scaled magnetization at T_c, for $H\rightarrow 0$, namely, $m_c=|M_{dia}|/H^{1/2}T_c$, that according to general Ginzburg-Landau theories for Gaussian fluctuations[10,11] and scaling arguments for isotropic three-dimensional (3D) superconductors should take the universal value $0.324k_B/\Phi_0^{3/2}$, is enhanced because of the anisotropy in the coherence length.

(v) Two-band evidence in the superconducting fluctuating diamagnetism was detected for temperature slightly above T_c.[13]

On the other hand, an enhancement of the diamagnetism above T_c has to be expected in heterogeneous superconductors. In fact, as a consequence of the disorder, nonpercolating regions become superconducting at a local temperature $T_c^{\mathrm{local}}(\mathbf{r})$ above the bulk T_c^{bulk}. In this case, the magnetization curves for $T>T_c^{\mathrm{bulk}}$ might mimic the ones expected below T_c, for a given volume fraction of the compound. Theoretical descriptions of this anomalous diamagnetism have been given.[16,17] An experimental example, for instance, has been found in YNi_2B_2C.[18] A way to discriminate between precursor diamagnetism associated with diffuse transitions and fluctuation related diamagnetism (possibly enhanced by phase fluctuations of nonzero order parameter as in underdoped superconducting cuprates) is to look at the temperature behavior of H_{up} in the isothermal magnetization curves.[19,20]

In this paper, we report the results of a study of the fluctuating diamagnetism in neutron irradiated and in Al-doped MgB_2. From the comparison of the experimental findings, it will turn out that the disorder in neutron irradiated MgB_2 does not cause diffuse transition. Although the transition temperature decreases on increasing the neutron fluence, a kind of "homogeneous defect structure" or disorder occurs: the transition remains sharp, the FD is fluctuations related, and H_{up} increases with increasing temperature. Furthermore, the anisotropy parameter decreases with respect to the unirradiated MgB_2. At variance, the Al for Mg substitution induces a diffuse transition, with an anomalous enhancement of the precursor diamagnetism of quite a different nature.

II. EXPERIMENTAL RESULTS

The samples used in the present work have been prepared and characterized according to the procedures explained elsewhere.[2,8] For $Mg_{1-x}B_2Al_x$, the doping amount varied from $x=0.1$ to $x=0.3$. The two nir samples we are going to deal with in this paper belong to the P batch:[8] P-3, exposed to thermal neutron fluence of 7.6×10^{17} cm^{-2} and P-3.7 exposed to the fluence of 5.5×10^{18} cm^{-2}. The main properties of the compounds are reported in Refs. 2 and 8.

The diamagnetic magnetization above T_c has been measured by means of the Quantum Design MPMS-XL7 superconducting quantum interference device (SQUID) magnetometer, separating the paramagnetic Pauli-like contribution from the diamagnetic one, according to the experimental procedure detailed in Refs. 12 and 13.

The volume susceptibilities measured in a magnetic field of a few Oersted in pure MgB_2, in the two samples of irradiated MgB_2, and in a typical ($x=0.25$) Al-doped compound are reported in Fig. 1.

In Fig. 2, isothermal magnetization data at typical temperatures around the transition temperature in the P-3 sample are shown. In the plots, the scaled magnetization $m_c = -M_{\mathrm{dia}}/H^{1/2}T_c$ is reported as a function of the magnetic field.

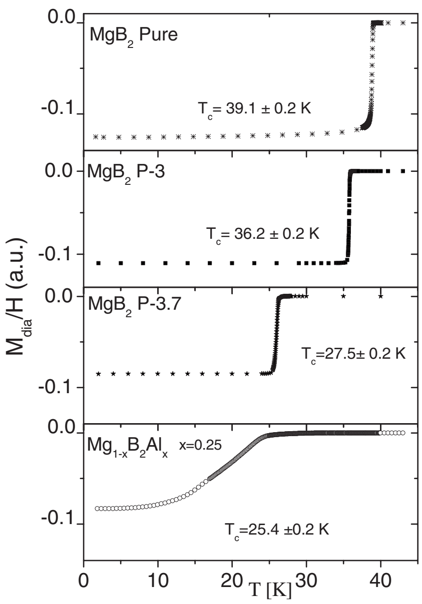

FIG. 1. Temperature dependences of the volume diamagnetic magnetization (measured in the limit of magnetic field $H\to0$) in the two samples of neutron irradiated MgB_2 and in $Mg_{0.75}B_2Al_{0.25}$. For comparison, the data in pure MgB_2 (Ref. 12) are also reported.

It is reminded that in the absence of any field-induced quenching of the fluctuating pairs for $T\approx T_c$, one should have field-independent m_c (Prange law; see Refs. 10 and 11), while for $T\gg T_c$, the magnetization is linear in the field. In the inset of Fig. 2, typical magnetization curves are shown. It is noted that for $H\geq H_{\mathrm{up}}$, the magnetization $|M_{\mathrm{dia}}|$ decreases on increasing the field.

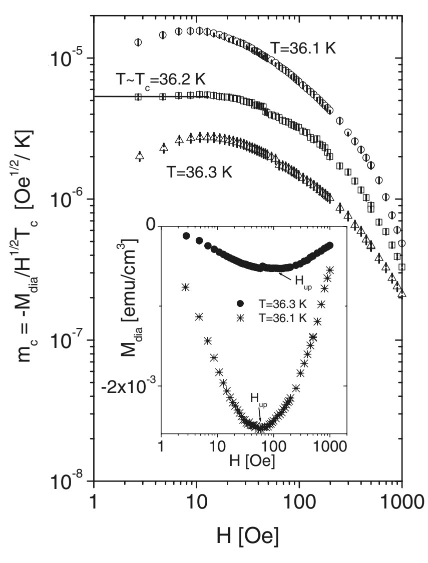

FIG. 2. Field dependence of the scaled magnetization in MgB_2 neutron irradiated P-3 sample, at typical temperatures around T_c. For $H\leqslant50-60$ Oe and $T\approx T_c$, the Prange law (see Refs. 10 and 11) is verified, while for $T>T_c$, the tendency toward $-M_{\mathrm{dia}}$ linear in the field is noticed. The solid line tracks the field dependence that would occur if no field-induced quenching of the fluctuating pairs should occur. In the inset, some representative magnetization curves are shown, with the arrows indicating the upturn fields.

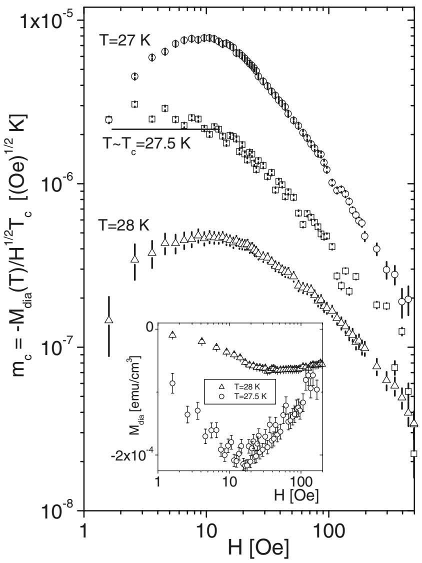

FIG. 3. Scaled magnetization around T_c in the P-3.7 sample. In the inset, two representative magnetization curves are shown (solid line according to the Prange law, see caption to Fig. 2).

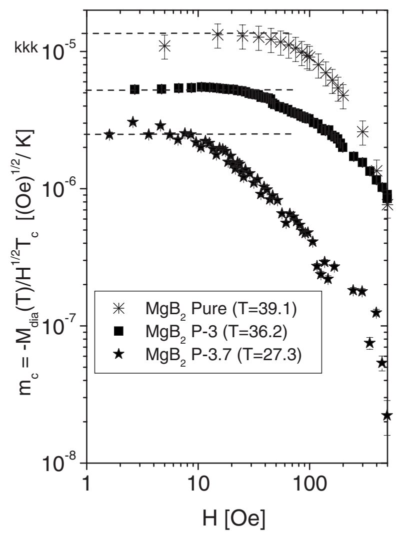

FIG. 4. Comparison of the scaled magnetization at T_c in the two nir samples with the corresponding data in pure MgB_2. The dotted lines are guides for the eye according to a field-independent scaled magnetization.

In Fig. 3, the results for the scaled magnetization m_c obtained in the P-3.7 compound are reported. The noticeable increase in the experimental error with respect to the P-3 sample is due to the decrease of the anisotropy (see the subsequent discussion) and of the transition temperature, both factors implying the decrease of the absolute value of the magnetic moment. Furthermore, it was noticed that the large neutron fluence for the P-3.7 sample induced an increase of the paramagnetic background, thus causing a larger error in the subtraction procedure. The paramagnetic contribution for strong neutron fluence suggests that paramagnetic centers are produced by radiation damage. The lowering of T_c is possibly due to pair-breaking mechanism. Electron paramagnetic resonance measurements are urged at the aim of obtaining insights on the nature of those centers, information that cannot be derived from bulk magnetic measurements as from the SQUID magnetization.

Figure 4 shows the comparison of the scaled magnetization m_c between pure MgB_2 and the nir samples. As already mentioned, for Gaussian fluctuations and according to scaling arguments,[11,12] the scaled magnetization at T_c has to be written

$$m_c = - M_{\mathrm{dia}}/\sqrt{H}T_c = - 0.324[k_B/{\Phi_0}^{3/2}]\gamma. \quad (1)$$

This equation follows from differentiation with respect to the field of the free energy for an anisotropic superconductor (Ref. 12 and references therein) for randomly oriented powder. γ in Eq. (1) is the anisotropy parameter $\gamma = \xi_{ab}(0)/\xi_c(0)$, ξ_{ab} and ξ_c being the in-plane and the out-of-plane components of the zero-temperature coherence length. In anisotropic superconductors, the factor γ enhances the universal value[10] $m_c = -0.324 k_B/\Phi_0^{3/2}$. In pure MgB_2, the anisotropy parameter γ is $\gamma \approx 7$ and the upturn field H_{up}, for temperature close to T_c, is around 80 Oe.[12] From Fig. 4, it appears that in the irradiated samples, the upturn field decreases (see also the inset in Fig. 3). Furthermore the anisotropy factor has to be decreased by a factor of about 2 for the P-3 sample. Even more marked is the reduction of the anisotropy in the P-3.7 sample. The comparison with the data in pure MgB_2 would lead to the conclusion that the P-3.7 sample is almost an isotropic 3D superconductor (see subsequent discussion).

In Fig. 5, representative results (raw magnetization data) obtained in Al-doped MgB_2 are reported for the sample with $T_c = 25.4$ K. At a first glance, the magnetization curves could be considered similar to the ones measured in nir samples. A strong enhancement of the FD with respect to the pure compound is noticeable, the volume susceptibility reaching a value around 10^{-3} for T close to T_c. For $T \gg T_c$, no anomalous diamagnetic contribution is present, and the paramagnetic magnetization follows the Pauli-like field and it is practically temperature independent. On approaching the bulk T_c, M_{dia} is linear in the field and the upturn field is well above 200 Oe. It should be remarked that the difference between field-cooled and zero-field-cooled data (difference not present in nir samples) in itself is a first indication that the diamagnetic contribution comes from a region of the sample that is below the local irreversibility temperature (see the subsequent discussion).

III. DISCUSSION AND CONCLUSIONS

We first briefly discuss the experimental findings in the Al-doped compound. The Al for Mg substitution acts on the superconducting properties mostly as an electron doping. Other effects are the change of the phonon spectrum (related to the different atomic size) and disorder-induced modification of the coupling between the bands.[3,1,5,2] The transition temperature upon substitution decreases almost linearly down to about $T_c = 25.4$ K for Al amount $x = 0.25$ and then

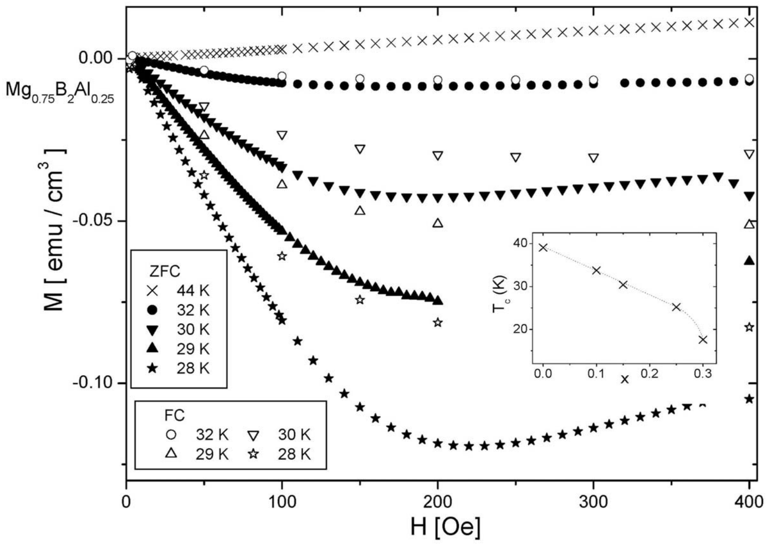

FIG. 5. Representative isothermal magnetization curves in Al-doped MgB_2. The inset shows the doping dependence of the transition temperature.

drops to about 15 K for $x=0.3$ (see inset in Fig. 5). Correspondingly, the transition loses the sharpness characteristic of the pure MgB_2. The diamagnetic susceptibility at T_c, in the low field limit, increases from $\chi_{\text{dia}} \approx 8 \times 10^{-5}$ emu/cm^3 in pure MgB_2 to about $\chi_{\text{dia}} \approx 10^{-3}$ emu/cm^3 in the sample at Al amount of 0.25, while M_{dia} is practically linear in the field up to almost 100 Oe. The magnetization curves above the transition to the bulk superconducting state show an upturn field that decreases with increasing temperature. As already mentioned, irreversibility effects are detected. All these features are the signature of the anomalous diamagnetism expected in heterogeneous, disordered systems underlying a diffuse transition. In other words, the diamagnetism is not related to superconducting fluctuations and the magnetization curves mimic the ones expected below the transition to the superconducting state in any type-II superconductors, with a certain smoothing of the upturn field, likely due to the distribution of the local critical fields H_{c1}. Evidently, on approaching the bulk T_c from above, nonpercolating mesoscopic superconducting regions are characterized by a nonzero order parameter, thus causing a dramatic increase in the diamagnetism. Besides the early theoretical descriptions,[16,17] the problem of this anomalous diamagnetism has been recently considered by Cabo *et al.*[20] in the light of the detection of nonconventional Ginzburg-Landau fluctuating diamagnetism in underdoped cuprates.[15]

The shape of the magnetization curves for $T \approx T_c^{\text{bulk}}$ results from a Gaussian distribution of transition temperatures, with H_{c1} playing a role similar to that of the upturn field.[21,22] Further work is in progress with regard to this aspect.[22]

A quite different situation is induced upon neutron irradiation. The transition remains sharp (see Fig. 1) and the isothermal magnetization curves above T_c (Figs. 2 and 3) basically show the field and temperature behaviors evidenced in the unirradiated compound, in spite of the marked decrease of the transition temperatures. Furthermore, the values of the scaled magnetization at T_c [see Eq. (1)] evidence the tendency toward isotropic superconductivity, with $\gamma \approx 1$.

In Fig. 6, the temperature behavior of the upturn field H_{up} detected in three Al-doped compounds, for the nir samples and in pure, untreated MgB_2, are collected. In pure MgB_2 and in the slightly irradiated compound, H_{up} increases on

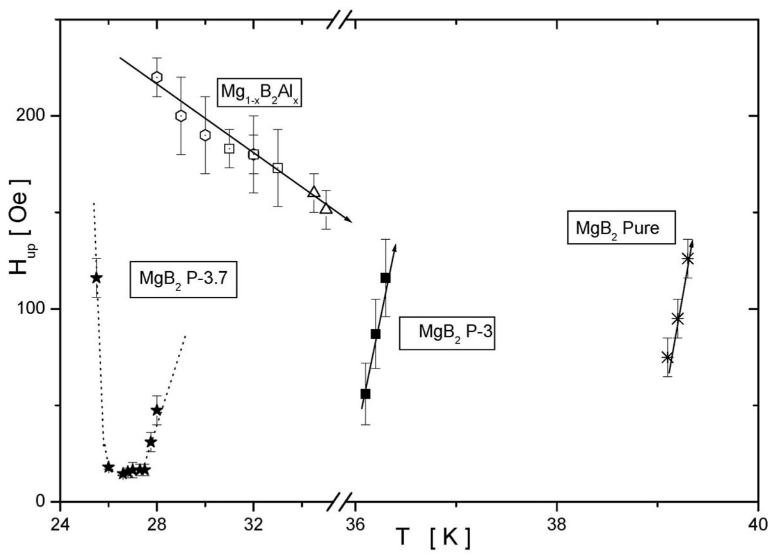

FIG. 6. Upturn field in the isothermal magnetization curves as a function of the measuring temperature in ald (⬡, $x=0.25$; □, $x=0.15$; and, △, $x=0.1$), in nir MgB_2, and in the pure unirradiated compound. The solid arrows are guides for the eyes. The dotted line for the P-3.7 sample tracks the temperature behavior of H_{up} above and below the transition temperature in the strongly irradiated sample.

measuring the magnetization at constant temperatures, all above T_c. On the contrary, in Al-doped compounds, the upturn field decreases on increasing the measuring temperature above bulk T_c. Finally, in the strongly irradiated compound P-3.7, the upturn fields are sketched in Fig. 6 for temperature above and below the transition temperature. The flattening of the data around T_c indicates the change over from the fluctuation induced diamagnetism (for $T \geqslant T_c$) to the incipient magnetization curves occurring below T_c.

From the experimental data, it seems possible to conclude that the irradiation induces a kind of homogeneously defected structure, which is reflected in the spectrum of the superconducting fluctuations and the related diamagnetism above the transition. The reduction in the anisotropy factor γ implies that the coherence length along the c axis and in the plane are forced by the irradiation toward a common value, at least for the sample exposed to the fluence of 5.5 $\times 10^{18}$ neutrons/cm^2.

The decrease in the upturn field upon irradiation is more difficult to handle, the role of the magnetic field in suppressing the superconducting fluctuations in a two-band superconductor being hard to treat in the framework of the microscopic Gor'kov theory accounting for nonlocality and short-wavelength fluctuations.[11] In the crude assumption of the so-called zero dimensional model for the superconducting droplets responsible of the FD (see Refs. 12 and 13), one approximately writes for the single particle magnetization

$$M^0_{\text{dia}}(\varepsilon,T) = -k_B T H[4\pi^2\xi^2 d^2/5\Phi_0^2]/[\varepsilon + (2\pi^2\xi^2 H^2 d^2/5\Phi_0^2)], \tag{2}$$

where $\xi = (\xi_{ab}^2\xi_c)^{1/3}$ is an effective coherence length, ε the reduced temperature, and d an average size of the fluctuating SC droplets responsible of the FD, which can be assumed to be of the order of ξ. Under these simplifying assumptions, the upturn field in the isothermal magnetization curves is given by

$$H_{\text{up}} \approx \sqrt{2.5}\sqrt{\varepsilon}\Phi_0/\pi\xi_0 d. \tag{3}$$

According to this equation, the reduction of H_{up} upon neutron irradiation would mean that the effective almost-isotropic coherence length is increased. This is somewhat surprising in view of the remark that the π band at larger value of the coherence length should be brought by the irradiation to the dirty regime more easily than the σ band. On the other hand one should remark that also the upper critical field H_{c2}, which in the clean limit is inversely proportional to ξ^2 was found to decrease for the sample P-3.7.[8] An effective, common coherence length shorter than ξ_π can be assumed to occur in the dirty regime for both the two bands.[1]

As regards the possible persistence of two-band effects[13] in the spectrum of the superconducting fluctuations, first we remark the following. The two-band spectrum is observable in the magnetization curves only for magnetic field perpendicular to the c axis and for temperature rather far from T_c. On approaching the transition, the two bands merge and the magnetization curve is no longer structured.[13] A careful experimental attempt has been performed on the P-3.7 sample to look for evidence of a double structure in the magnetization curves. However, the signal to noise ratio for temperatures rather far from T_c is too poor to allow a firm conclusion. Furthermore, the tendency upon neutron irradiation toward the isotropy condition can be expected to wipe out any double-structured magnetization curve.

Summarizing, from the isothermal magnetization curves above the superconducting transition temperature, the different nature of the diamagnetism occurring in Al-doped MgB_2 with respect to the one in neutron irradiated MgB_2 has been evidenced, thus emphasizing the quite different character of the related disorders. Upon Al doping, a diffuse transition occurs, with site dependent transition temperatures which imply a strong anomalous diamagnetism above the bulk T_c^{bulk}, originated from the region of the sample having $T_c^{\text{local}} > T_c^{\text{bulk}}$. At variance, the neutron irradiation, although decreasing the transition temperature, introduces a kind of "homogeneous disorder": the transitions remain sharp and the spectrum of the superconducting fluctuations revealed by the fluctuating diamagnetism retains most of the features typical of unirradiated MgB_2. The effect of the magnetic field in suppressing the superconducting fluctuations above a certain H_{up} is confirmed, with two significant modifications with respect to untreated MgB_2. The upturn field H_{up} as well as the anisotropy factor decrease on increasing the neutron fluence. The implications of these findings have been discussed, suggesting a crossover to a dirty regime for both the two bands, with a common almost-isotropic spectrum for the superconducting fluctuations, corresponding to an increased effective coherence length.

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