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Effect of proton irradiation on the fluctuation-induced

magnetoconductivity of  $FeSe_{1-x}Te_x$  thin films

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#### Abstract

The influence of proton irradiation on the fluctuation-induced magnetoconductivity of high quality  $FeSe_{1-x}Te_x$  (x = 0.4, 0.55) (FST) thin films has been investigated. The measurements were performed with magnetic fields up to 13 T applied in the two main crystal directions. The results were interpreted in terms of the Ginzburg–Landau approach for three-dimensional materials under a total-energy cutoff. The analysis shows that properly-tuned proton irradiation does not appreciably affect fundamental superconducting parameters like the  $T_c$  value, the upper critical fields or the anisotropy. This has important consequences from the point of view of possible applications due to the enhancement of vortex pinning induced by irradiation.

#### Introduction

The discovery of iron-based superconductors has added valuable knowledge to research into the high- $T_c$ superconducting mechanism [1]. To date, several types of iron-based superconductors have been discovered in a variety of crystal structures [2-6]. Among these superconductors, tetragonal FeSe has received special attention due to its simple structure [2]. Since its discovery, this material has been studied as a key compound to find the answers to important questions regarding the superconducting mechanism. It was also shown that the superconductivity in the  $\beta$ -FeSe system is highly dependent on the stoichiometric ratio [7]. For example, in the  $Fe_{1+\delta}$ Se binary phase diagram, the PbO-type tetragonal structure remains stabilized at Fe-rich compounds  $[\delta = 0.01-0.04]$  and bulk superconductivity was found for  $\delta = 0.01$  [7–9]. Furthermore, it was found that in these compounds, the superconductivity is very sensitive to composition and vacancy order [7]. The partial substitution of Fe by Ni, Co, and Cu has also been investigated, with a solubility up to 20% to 30% [10, 11]. However, for each dopant, the superconductivity was depressed within the solubility ratio. Enhanced superconductivity was observed when Se was substituted with Te and  $T_c$  was raised up to 14 K for  $x \sim 0.5$ , [12, 13]. Moreover, Sun *et al* showed that the  $T_c$  increases up to 38 K with high pressure in the FeSe superconductor [14]. Finally, Qiu et al observed the spin fluctuation spectrum and spin gap in the FeSe system through neutron scattering [15]. The NMR results pointed out the enhancement of antiferromagnetic (AFM) fluctuation towards T<sub>c</sub> which indicates the important role of AFM fluctuations for the emergence of superconductivity in FeSe systems [16].

The analysis of fluctuation effects above the superconducting transition is an important way to obtain information about fundamental aspects of superconducting materials such as the critical magnetic fields, the coherence lengths, the anisotropy, or the dimensionality (at present a highly debated issue in iron-based superconductors, see [17–25]. In these materials the Ginzburg number (which is associated with the width of the critical fluctuations region around  $T_c$ ) is half that of conventional low- $T_c$  superconductors and high- $T_c$ 

cuprates, so fluctuation effects are expected to play an important role [17]. Here we apply this technique (sometimes named *fluctuation spectroscopy*) [26] to investigate for the first time the effect of proton irradiation (a useful technique to introduce effective pinning centers for the enhancement of the critical current density) on the fundamental parameters of  $FeSe_{1-x}Te_x$  (x = 0.4, 0.55) (FST) thin films. In particular, we study the fluctuation-induced in-plane conductivity under magnetic fields up to 13 T applied in the two main crystal directions. The results are analyzed in terms of the three-dimensional (3D) anisotropic Ginzburg–Landau (GL) approach [27].

#### **Experimental details**

The analyzed FST thin films with a thickness of 110 nm were grown on 001-oriented CaF<sub>2</sub> substrates by pulsed laser deposition (PLD) with a KrF excimer laser (Coherent COMPEX PRO 205F, 248 nm wavelength). During the growth, the pressure was held below  $2 \times 10^{-6}$  Torr, while the base pressure was  $3 \times 10^{-7}$  Torr. The substrate temperature, laser energy density, repetition rate, and the distance between the substrate and target were 400 °C, 3 J cm<sup>-2</sup>, 3 Hz, and 4 cm, respectively. The FST bulk target used for PLD was prepared by the induction melting method for the reaction of Fe, Se, and Te small chips at 700 °C. The nominal compositions of the FST target were Fe<sub>0.94</sub>Se<sub>0.45</sub>Te<sub>0.55</sub>. The samples were characterized by x-ray diffraction (XRD) using Cu K $\alpha$  radiation. Protons with 3.5 MeV energy were irradiated into the films using the MC-50 Cyclotron at KIRAM. The resistivity is measured down to 2 K in a magnetic field up to 13 T with a conventional four-probe method using an Oxford superconducting magnetic system. A 0.1 K temperature interval was used to study in detail the fluctuation effects around  $T_c$ .

#### **Results and discussion**

Figure 1(a) shows the (XRD) pattern of pristine and irradiated FeSe<sub>0.45</sub>Te<sub>0.55</sub> (JP-164) and FeSe<sub>0.6</sub>Te<sub>0.4</sub> (JP-211) thin films (FST). The well-defined (00*l*) peaks perfectly indicate the *c*-axis grown FST films in terms of a PbO tetragonal-type structure. In addition, as shown in figure 1, barring the 00*l* peaks of the CaF<sub>2</sub> substrate, no other extraneous peaks, which are usually associated with an Fe-deficient FST superconductor, are observed in the XRD pattern [7]. Furthermore, a shift of the 00*l* peaks towards high angles in the case of irradiated FST can be seen which indicates the shrinkage of the *c* parameter from 5.8579 Å (JP-164) and 5.8404 Å (JP-211) in pristine samples to 5.8199 Å (JP-164) and 5.8357 Å (JP-211) after irradiation, respectively. The *c* parameter of the pristine samples is smaller than those reported for bulk poly- and single-crystalline FeSe<sub>0.45</sub>Te<sub>0.55</sub> [28], but it is in agreement with that of the thin films [29, 30].

Figure 1(b) shows the Williamson–Hall plot (*FWHM* × cos  $\theta$  as a function of sin  $\theta$ , where  $\theta$  is the Bragg angle, and FWHM denotes the full width at half maximum of the diffraction peaks) [31] for the analysis of any lattice strain in our films. There is a significant change in slope after irradiation in sample JP-164, indicating a reduction of lattice strain by the proton irradiation. However, the slope is unchanged in the JP-211 sample and no radiation-induced changes in the strain are expected. In both samples the proton energy and total dose was the same (3.5 MeV and 5 × 10<sup>15</sup> cm<sup>-2</sup>, respectively). However, while in sample JP-164 the proton current was 100 nA, it was adjusted to a much smaller value (10 nA) in sample JP-211.

Figure 2 depicts the temperature dependence of the in-plane resistivity of pristine JP-164 thin film under different applied magnetic fields. As is clear from figure 2, the normal-state resistivity presents a similar amplitude for both field directions. The  $T_c$  value (20.3 K) is determined from the transition midpoint at the zero applied field. This  $T_c$  value is larger than the one for bulk FeSeTe (14 K) [28], but is in agreement with the one for epitaxial FeSe<sub>0.5</sub>Te<sub>0.5</sub> thin films [29, 30]. Furthermore, one can see that by increasing the applied magnetic field,  $T_c$  is shifted to lower temperatures and the transition width becomes broader. These effects are less evident when the field is parallel to the *ab* layers, which is a consequence of the anisotropic nature of these compounds.

The inset of figure 2(b) shows an example of the process for the extraction of the background contribution to the resistivity  $\rho_B(T)$  in order to determine the superconducting contribution to the electric conductivity. As can be seen, the normal-state resistivity presents a slight negative curvature that has also been observed in other FST films with similar compositions [32]. To our knowledge there is no theoretical expression for such a  $\rho(T)$  behavior, but we noticed that  $d\rho/dT$  (inner inset) presents a linear temperature dependence down to a temperature (~35 K) that may be associated to the onset of fluctuation effects (hereafter named  $T_{onset}$ ). Then,  $\rho_B(T)$  was estimated by fitting a degree-two polynomial in a temperature range from 35 to 50 K (solid line). The fluctuation-induced conductivity was finally obtained for each applied magnetic field as  $\Delta\sigma(T) = 1/\rho(T) - 1/\rho_B(T)$ . It is worth noting that the



reduced temperature associated to the onset of fluctuation effects  $\varepsilon_{onset} = \ln(T_{onset}/T_c) \approx 0.55$  is in excellent agreement with data in the literature for samples of the FeSe system [25, 33, 34]<sup>6</sup>, and with the theoretical estimate in [35]. It is also close to the  $\varepsilon_{onset}$  value (~0.4) obtained in other Fe-based superconductors (BaFe<sub>2-x</sub>Ni<sub>x</sub>As<sub>2</sub>) with particularly tractable (almost constant) background contributions [27]. Finally, we have checked and found that the background contribution is quite stable to changes in the fitting region. For instance, in sample JP-164, if instead of the interval 35-50 K is chosen 40-50 K or 35-45 K, the variation in  $\Delta\sigma$  near  $T_c$  (21 K) would be only ±6%.

The data are analyzed in terms of a GL approach for 3D anisotropic superconductors, valid for finite applied magnetic fields. This approach includes a cutoff in the energy of the fluctuation modes, which extends its applicability to high reduced temperatures [35]. Such a cutoff scheme has been probed in other iron-based superconductors [20, 27], but also in high- $T_c$  cuprates [36], and low- $T_c$  alloys [37]. It has also been recently taken into account in very recent theoretical works about possible multiband effects on the fluctuation diamagnetism [38]. In the framework of this approach, the fluctuation-induced conductivity in the presence of a magnetic field

<sup>&</sup>lt;sup>6</sup> In this work [34]  $T^* = 20$  K was obtained from the resistivity as the temperature at which  $d\rho/dT$  presents a subtle minimum (of the order of the noise level) in a ~15 K wide plateau. However,  $T_{onset}$  may be reasonably chosen as the temperature at which  $d\rho/dT$  grows above the noise level. This leads to  $T_{onset} \approx 14$  K, i.e.  $\varepsilon_{onset} \approx 0.5$ , in agreement with the values in our work. Measurements in this work of the Nernst, Hall, and Seebeck coefficients present a maximum (or minimum) at  $T^* \approx 20$  K which the authors associate with the onset of precursor superconductivity. However, these observables present an evident curvature around and up to well above  $T^*$ , and it is not clear whether this is directly associated to the actual onset of fluctuation effects. These authors also present data of the fluctuation magnetization, but the resulting  $T_{onset}$  seems to be strongly dependent on the applied magnetic field, and in relation to these measurements the own authors recognize 'some ambiguity due to weakly temperature-dependent normal-state susceptibility'.



**Figure 2.** Temperature dependence of the in-plane resistivity of JP-164 pristine film for magnetic fields applied perpendicular (a) and parallel (b) to the *ab* layers. Inset: normal-state resistivity under zero applied field for the pristine JP-164 film. The solid line is a fit of a degree-two polynomial. The inset is the same background in the  $d\rho/dT$  versus temperature scale.

with an arbitrary orientation is given by [27]

$$\Delta\sigma_{ab} = \frac{e^2}{32\hbar\pi\xi_c(0)}\sqrt{\frac{2}{h}} \int_0^{\sqrt{\frac{c-\varepsilon}{2h}}} dx \bigg[\psi^1\bigg(\frac{\varepsilon+h}{2h}+x^2\bigg) - \psi^1\bigg(\frac{c+h}{2h}+x^2\bigg)\bigg],\tag{1}$$

where  $\psi^1$  is the first derivative of the digamma function,  $\varepsilon = ln(T/T_c)$  the reduced temperature,  $h = H/H_{c2}(0)$  is the reduced magnetic field,  $H_{c2}(0)$  is the upper critical field (in the direction of the applied field) *linearly* extrapolated to T = 0 K, and c is the cutoff constant. It is worth noting that c corresponds to the reduced temperature at which fluctuation effects vanish. This approach is expected to be applicable up to h values of the order of 0.1 [27]. In the absence of the applied magnetic field and also without a cutoff ( $c \rightarrow \infty$ ), equation (1) becomes the classic Aslamazov–Larkin result [39].

Figure 3(a) shows a comparison of the experimental data obtained with H//c on the pristine JP-164 film with the aforementioned theoretical model (solid lines). This last was evaluated by using in equation (1)  $\xi_c(0) = 0.56$  nm,  $\mu_0 H_{c2}^c(0) = 108$  T (a value within the ones in the literature for FST thin films) [29, 40, 41], and c = 0.55 as corresponds to the reduced temperature for the onset of fluctuation effects in this sample (~35 K).  $\xi_c(0)$  and  $\mu_0 H_{c2}^c(0)$  were chosen so that a good agreement is attained in the widest possible temperature interval for each field; note that the low-temperature bound of the Gaussian region in which equation (1) is applicable is limited by the onset of critical fluctuations but also by possible phase fluctuations (see below) and is not *a priori* known. By using the well-known GL expression  $\mu_0 H_{c2}^c(0) = \phi_0/2\pi \xi_{ab}^2(0)$ ,  $\xi_{ab}(0) = 1.74$  nm is obtained, which together with the above  $\xi_c(0)$  value leads to an anisotropy factor of  $\gamma = 3.1$ . As can be seen, the agreement with the experimental data is very good except for data very close to  $T_c$  and under the lowest fields used in the





experiments. The anomalous fluctuation effects in this region of the H-T phase diagram were already observed in other iron-based superconductors and were attributed to the possible presence of phase fluctuations [42], or to a  $T_c$  distribution [43]. The same effect seems to be also present in the fluctuation magnetization of FeSe single crystals recently reported by Kasahara *et al* [34] (see, in particular, figure 2(c)–(e) in that paper).

The data obtained in the same sample but with H||ab are presented in figure 3(b). In this case the theory depends on  $\xi_c(0)$ , c, and the upper critical field in the *ab* direction,  $H_{c2}^{ab}(0)$ . So, the lines in figure 3(b) were obtained *without free parameters*, by using in equation (1) the above  $\xi_c(0)$  and c values, and  $\mu_0 H_{c2}^{ab}(0) = \gamma \mu_0 H_{c2}^c(0) = 335 T$ . As can be seen, the agreement is again excellent; this represents an important consistency check of our analysis. In particular, if the  $\Delta \sigma$  amplitudes were affected by an incorrect background subtraction, or by the presence of an interface between the film and the substrate, the  $\xi_{ab}(0)$  and  $\xi_c(0)$  values resulting from the data for H//c would not explain the data for H//ab. For magnetic fields above the ones presented in figure 3 the agreement with the theory is slightly worse, which could be related to the above-mentioned *h*-limit for the applicability of the theory. It is worth noting that indirect effects like the Maki–Thompson (MT) contribution seem to be negligible in these compounds. The use of the MT term in the fit would lead to an anomalously large pair-breaking parameter ( $\delta_{\text{MT}}$ ) consistent with a negligible MT contribution in the accessible reduced temperature range. This is in agreement with the results obtained in other families of Fe-based superconductors, see [17, 18, 22–25, 27] and in particular [21].

The results for the same sample after proton irradiation (5 × 10<sup>15</sup> cm<sup>-2</sup> with 3.5 MeV energy) are presented in figure 4. In the insets the temperature dependence of the resistivity  $\rho(T)$  is shown under different field amplitudes and for both  $H \perp ab$  and  $H \mid ab$ . The  $T_c$  value (estimated as 13.2 K from the transition midpoint of the measurement with H = 0) is suppressed up to ~7 K with respect to the pristine sample. Moreover, a resistive tail persists down to ~10 K, from which a transition width of ±3 K may be estimated. The background contribution to the resistivity was again obtained by fitting a degree-two polynomial. The fitting region was 25–35 K, consistent with the one in the non-irradiated case (relative to the  $T_c$  value). The resulting



**Figure 4.** The temperature dependence of the fluctuation conductivity  $\Delta \sigma$  in irradiated JP-164 film under various applied magnetic fields perpendicular (a) and parallel (b) to the *ab* layers. The solid lines correspond to equation (1). Inset: temperature dependence of the in-plane resistivity in irradiated JP-164 film for magnetic fields applied perpendicular (a) and parallel (b) to the *ab* layers, respectively.

fluctuation contribution to the conductivity is presented in the main panels of figure 4. In the theoretical approach, this time we have introduced a pre-factor *f* to account for a possible reduction in the superconducting volume fraction caused by the irradiation. Also, due to the broadened resistive transition, the  $T_c$  value is a free parameter, together with the upper critical fields (or equivalently, the coherence lengths). These parameters were chosen so that a good agreement is attained in the widest possible temperature interval for each field amplitude and orientation. A relatively good agreement was also obtained (solid lines) but with some important differences relative to the pristine sample: the used  $T_c$  value (15.1 K) is above the transition midpoint but still within the transition width. The effective superconducting volume fraction has strongly reduced to 0.18. The upper critical fields extrapolated to T = 0 K resulted in  $\mu_0 H_{c2}^c(0) = 50 T$  (a factor ~2 smaller than in the non-irradiated sample) and  $\mu_0 H_{c2}^{ab}(0) = 110 T$ , leading to an anisotropy factor of ~2.2 (slightly smaller than in the non-irradiated sample). The corresponding coherence lengths are  $\xi_{ab}(0) = 2.56$  nm and  $\xi_c(0) = 1.17$  nm. Finally, the cutoff constant (c = 0.35) is found to be slightly smaller than in the pristine FeSe<sub>0.45</sub> Te<sub>0.55</sub> thin film.

A second thin film (JP-211) with composition FeSe<sub>0.6</sub>Te<sub>0.4</sub> but slightly different  $T_c$  (~18 K) was also studied with H//c. For the irradiation process we used the same proton energy (3.5 MeV) and total dose (5 × 10<sup>15</sup> cm<sup>-2</sup>), but with a proton current of 10 nA (10 times smaller than the one used in sample JP-164). As can be seen in the  $\rho(T)$  curves presented in the insets of figure 5, this time there is no appreciable change in  $T_c$ as a post-irradiation effect, although a transition broadening of ~2 K is observed. The fluctuation contribution to the conductivity was obtained by following the same procedure as in sample JP-164 for the background contribution (a degree-two polynomial between 35 and 50 K). The result is presented in the main panels of figure 5. In the case of the pristine sample, a good agreement with the theory (solid lines) was achieved by using  $T_c = 18.0$  K (corresponding to the resistive transition midpoint for H = 0), c = 0.65 (as corresponds to the reduced temperature for the onset of fluctuation effects),  $\xi_{ab}(0) = 1.98$  nm, and  $\xi_c(0)$ = 0.82 nm. These last values are in rough agreement with the ones obtained for JP-164, taking into account the slight difference in the  $T_c$  value. After irradiation a relatively good agreement with the theory was also observed, although with some differences similar to the ones found in JP-164: the used  $T_c$  value (18.6 K) is



**Figure 5.** The temperature dependence of the  $\Delta\sigma$  for (a) the pristine and (b) the irradiated JP-211 film under different magnetic fields applied perpendicular to the *ab* layers. The solid line corresponds to equation (1). The insets show the corresponding resistivity around  $T_{c}$ .

0.5 K above the transition midpoint but still within the transition width. The in-plane coherence length was found to be  $\xi_{ab}(0) = 2.17$  nm, close to the one before irradiation. The amplitude of fluctuation effects is a factor 5 smaller than in the pristine sample. By assuming that  $\xi_c(0)$  is also roughly the same after irradiation, such a reduction may be attributed to a reduction in the effective superconducting volume fraction ( $f \approx 0.2$ ) caused by the proton irradiation.

In figure 6 we compare the  $H_{c2}(T)$  curves obtained from the shift of the resistive transition (as determined by using a 50% criterion on the normal-state resistivity), with the ones resulting from the analysis of fluctuations (lines). These last were obtained from the coherence lengths through

$$\mu_0 H_{c2}^c(T) = \frac{\phi_0}{2\pi\xi_{ab}^2(0)} \left(1 - \frac{T}{T_c}\right),\tag{2}$$

$$\mu_0 H_{c2}^{ab}(T) = \frac{\phi_0}{2\pi\xi_{ab}(0)\xi_c(0)} \left(1 - \frac{T}{T_c}\right).$$
(3)

The excellent agreement observed in the case of the pristine samples is an important consistency check of our analysis. In the case of the irradiated samples this comparison is complicated by the broadening of the resistive transition. However, as can be seen in the insets in figure 6, the  $H_{c2}(T)$  curves obtained from the analysis of fluctuations are still within the ones resulting from the 50% and the 90% criteria.

Let us finally mention that the reduction of the  $T_c$  and  $H_{c2}$  values in the JP-164 film after irradiation (not observed in sample JP-211) could be due to local heating effects because of the temperature increase of the film during the irradiation process. These local heating effects may accelerate the displacement of interstial atoms and create vacancies and as result there is change in the lattice strain in the irradiated JP-164 film, as indicated in figure 1(b). In spite of the differences in the proton current, the reduction in the amplitude of the fluctuation



**Figure 6.** (a) The main panel shows  $H_{c2}(T)$  for pristine JP-164 estimated from 50% of the normal-state resistivity for both field orientations. The upper and lower insets show  $H_{c2}(T)$  estimated from 50% and 90% of the normal-state resistivity in the irradiated JP-164 for magnetic fields applied perpendicular and parallel to the *ab* layers, respectively. (b) The main panel shows  $H_{c2}(T)$  for pristine JP-211 estimated from 50% normal-state resistivity for the applied magnetic fields perpendicular to *ab* layers. The inset shows the  $H_{c2}(T)$  estimated from 50% and 90% normal-state resistivity in the irradiated JP-211 for magnetic fields applied perpendicular to ab layers. The inset shows the  $H_{c2}(T)$  estimated from 50% and 90% normal-state resistivity in the irradiated JP-211 for magnetic fields applied perpendicular to ab layers. In all the cases the solid lines were evaluated from the analysis of fluctuation effects.

effects after irradiation is similar in both films due to a reduced superconducting volume fraction which seems to be related to the proton energy and dose level which cause similar scattering centers in both films.

## Conclusions

Superconducting FST thin films were grown by PLD. The effect of proton irradiation on the superconducting fluctuation effects above  $T_c$  was analyzed on two different samples in terms of a GL approach for 3D anisotropic superconductors. The analysis provided information on fundamental superconducting parameters like the coherence lengths, the anisotropy factor, and the effective volume fraction. Irradiation reduces the amplitude of fluctuation effects by a factor as large as ~5, which could be attributed to a proportional reduction in the effective superconducting volume fraction. It was found that a comparatively large value of the proton current leads to an annealing effect and thus has a detrimental effect on the superconducting properties. However, once properly tuned, the irradiation has a moderate effect on the upper critical fields, the anisotropy, and even on the  $T_c$  value. This is important from the point of view of potential applications of these materials, because it would allow the use of irradiation to enhance pinning properties without a detrimental effect on other fundamental superconducting parameters.

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