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Effect of 1.5 MeV Proton Irradiation on Superconductivity in $FeSe_{0.5}Te_{0.5}$ Thin Films

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Abstract: Raising the critical current density J_c in magnetic fields is crucial to applications such as rotation machines, generators for wind turbines and magnet use in medical imaging machines. The increase in J_c has been achieved by introducing structural defects including precipitates and vacancies. Recently, a low-energy ion irradiation has been revisited as a practically feasible approach to create nanoscale defects, resulting in an increase in J_c in magnetic fields. In this paper, we report the effect of proton irradiation with 1.5 MeV on superconducting properties of iron–chalcogenide $FeSe_{0.5}Te_{0.5}$ films through the transport and magnetization measurements. The 1.5 MeV proton irradiation with 1×10^{16} p/cm^2 yields the highest J_c increase, approximately 30% at 5–10 K and below 1 T without any reduction in T_c. These results indicate that 1.5 MeV proton irradiations could be a practical tool to enhance the performance of iron-based superconducting tapes under magnetic fields.

Keywords: superconductor; irradiation; critical current

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1. Introduction

Iron-based superconductors have a reasonably high superconducting transition temperature T_c, very high upper critical magnetic fields H_{c2}, quite a small anisotropy γ and larger critical grain boundary angle than cuprate superconductors, which make them promising for high-field applications such as superconducting magnet and generators [1–5]. The use of superconducting materials for high field applications is limited by the critical current density J_c in magnetic fields, which can be sustained by pinning the vortices (flux pinning) at structural defects with nano-meter sizes such as cracks, voids, grain boundaries and secondary phases [6,7]. The ion irradiation is a useful tool to generate the desired defect structure. Depending on the ion species, ion energy and the properties of the target materials, ion irradiation enables the creation of defects with well-controlled morphology and density, such as point [8], cluster [9–12] and columnar [13–15] defects. Early works on the ion irradiation of cuprate (Cu–O based) high-T_c superconductors (HTS) for improving J_c in the magnetic field have mostly focused on the high-energy, over hundreds of MeV, heavy ion irradiation [13–15]. At this energy range, the irradiation of superconducting materials by the swift heavy ion mainly causes electronic excitation and ionization of the target atoms. As a result, continuous amorphous tracks are formed in a process that can be described as the rapid melting and solidification of nm-sized columns in the path of an ion. Even though the heavy ion tracks proved to be very effective pinning defects, this approach has been limited to fundamental studies of the vortex matter.

Recently, ion irradiation of HTS with a low energy has received a renewed interest as a practical method for increasing J_c in magnetic fields, due to the compact accelerator, lower radioactivity and less costly operation [9–12]. Low-energy ion irradiation utilizes a different mechanism for the creation of vortex pinning defects. The electronic excitation and ionization are low enough so the heat can dissipate without damaging the materials. The low-energy ion irradiation leads to the collision of the ion with the target atom nuclei, resulting in cascade, point and cluster defects. Matsui et al. demonstrated that 3 MeV Au^{2+} ion irradiation to 700 nm thick YBCO films yielded an enhancement in the in-field J_c at 77 K of up to a factor of 4 [9]. Equally impressive results in YBCO commercial tape have been reported by Jia et al. using 4 MeV proton [10]. Recently, we reported a route to raise both T_c and J_c in iron-based superconducting $FeSe_{0.5}Te_{0.5}$ (FST) thin films by low-energy (190 keV) proton irradiation [16,17]. The 190 keV proton irradiation yields the increase in T_c due to the nanoscale compressive strain induced by cascade defects. The irradiation also induced a near doubling of J_c at 4.2 K from the self-field to 35 T through strong vortex pinning by the cascade defects and surrounding nanoscale strain.

In this paper, we report the effect of 1.5 MeV proton irradiation on iron–chalcogenide FST superconducting films. We report the performance of irradiated samples at different temperatures in a magnetic field up to 9 T. We show that 1.5 MeV protons clearly enhance J_c in magnetic fields <1 T with no subsequent reduction in T_c. However, we did not observe a reproducible positive effect in the magnetic fields >1 T. The results are discussed in terms of the spatial distribution of defects produced by fast protons.

2. Materials and Methods

All films in this study were deposited by the pulsed laser deposition (PLD) method using a Nd:YAG laser (λ = 266 nm). We first grew a CeO_2 layer with a thickness of about 80–100 nm on $SrTiO_3$ single-crystal substrate at a substrate temperature of 600–650 °C and oxygen partial pressure of ~115 mTorr. Then, 100–130 nm thick FST films were grown on CeO_2 buffer layers. During the deposition of FST films, the substrate temperature and oxygen partial pressure were kept at 300–360 °C and ~1×10^{-6} Torr, respectively.

Superconducting transport properties were measured using the conventional four-probe method in a physical property measurement system (PPMS, Quantum Design). $T_{c,10}$ and J_c were determined from the ρT and I–V curves using 0.1 ρ_n and 1 μV/cm criteria, respectively. Here, ρ_n means the normal state resistivity above the transition temperature. The current was applied perpendicularly to the magnetic field. The magnetization was measured using a superconducting quantum interference device (SQUID, Quantum Design) magnetometer. Two FST films (sample A and B) were fabricated under the same deposition condition for different irradiation conditions. Each FST film was cut into 3 pieces: one for magnetization measurement before and after irradiation with same film, another for transport measurement before irradiation (pristine) and the other for transport measurement after irradiation (irradiated).

The FST films were irradiated with 1.5 MeV proton doses of 1×10^{15} and 1×10^{16} p/cm^2 in vacuum at room temperature using the 5 MV tandem accelerator of the Wakasa Wan Energy Research Center (WERC). The samples were mounted on a copper plate with a double-faced carbon tape. The incident angle of ions was set as normal to the film surface. The flux was kept around 3.2×10^{12} p/cm^2·s, corresponding to a beam current density of ~500 nA/cm^2. The surface temperature was monitored by a thermocouple. The surface temperature during the irradiation remained below 40 °C.

Prior to the ion irradiation experiment, we ran Stopping and Range of Ions in Matter (SRIM) [18] to estimate ion range and damage profile in our experiment. Based on the simulation results, 1×10^{15} and 1×10^{16} p/cm^2 are estimated to be ~3.2×10^{-5} and ~3.2×10^{-4} dpa (displacement per atm), respectively.

3. Results and Discussion

3.1. Magnetic Measurements

Figure 1a,b compare the temperature dependence of magnetic moment M with $H//c$ for two FST films (film-A and film-B) before and after irradiation with 1×10^{15} and 1×10^{16} p/cm^2 dose, respectively. Both the zero-field-cooled (ZFC) and field-cooled

(FC) magnetizations in 2 Oe magnetic field parallel to the *c*-axis indicate the appearance of superconductivity (obtained by the bifurcation of ZFC and FC) in pristine FST films at 16.8 K for film-A and 16.6 K for film-B. After the irradiation, the superconducting transitions occurred at 16.8 K for film-A and 16.8 K for film-B, indicating that 1.5 MeV proton irradiations with 1×10^{15} and 1×10^{16} p/cm^2 dose have little impact on T_c^{mag}. However, the diamagnetic signal was enhanced with a sharper superconducting transition in the FST film-B irradiated with 1×10^{16} p/cm^2 dose. A degradation of T_c after the ion irradiation is commonly reported in iron-based superconductors [19], although there have been a few reports on an increased T_c in iron-based superconductors irradiated with proton and electron [16,20,21]. In previous work, the Fe(Se,Te) films were covered by Al foil with 80 μm thickness and irradiated with 3.5 MeV protons at doses of 2.68×10^{16} and 5.35×10^{16} p/cm^2, corresponding to 2.30×10^{-3} and 4.59×10^{-3} dpa, respectively [22–24]. The average bombarding energy of the protons on the Fe(Se,Te) film was calculated to be 1.43 ± 0.07 MeV. As a result, the irradiations to doses of 2.68×10^{16} and 5.35×10^{16} p/cm^2 slightly suppressed T_c from 17.7 K for pristine film to 17.3 K and 17.1 K, respectively. Given these results, the primary reason of the almost same T_cs before and after the irradiation in our study would be a lower fluence than that in the previous works.

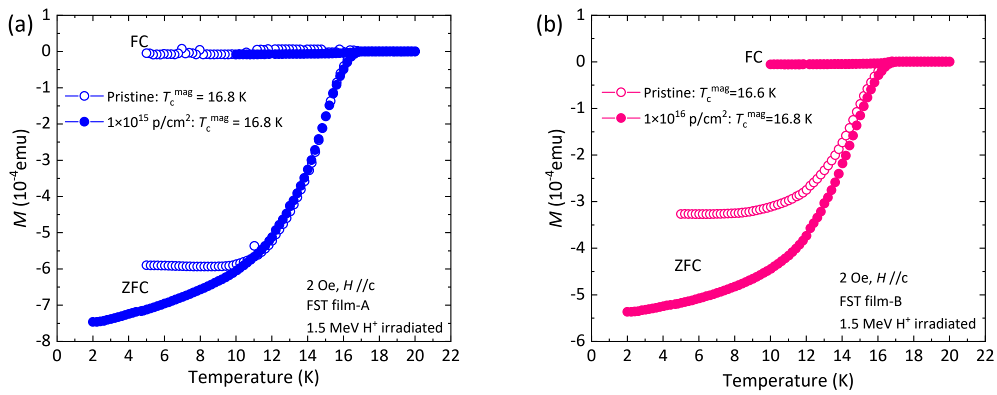

Figure 1. Temperature dependences of magnetic moment *M* for both zero-field-cooled (ZFC) and field-cooled (FC) process at a magnetic field of *H* = 2 Oe applied along the *c*-axis for FST films before and after 1.5 MeV proton irradiation with (**a**) 1×10^{15} and (**b**) 1×10^{16} p/cm^2 dose, respectively.

Figure 2 shows the magnetic field dependence of J_c for the FST film-B at 5, 8, 10 K before and after 1.5 MeV proton irradiation at a dose of 1×10^{16} p/cm^2. The J_c was estimated from the magnetization hysteresis (*M*–*H*) loops using the critical-state Bean model [25,26]. For a rectangular prism-shaped crystal of dimensions $a < b$, we obtained the in-plane critical current density J_c^{ab} in the magnetic field parallel to the *c*-axis as $J_c^{ab} = 20\Delta M/(a(1 - a/3b))$, where ΔM is the difference in magnetization *M*(emu/cm^3) between the top and bottom branches of the *M*-*H* loop. In the inset of Figure 2, the *M*–*H* loop in FST film-B at 5 K before and after the irradiation of a dose of 1×10^{16} p/cm^2 is plotted. A large irreversibility is noticeable up to around 4 T at 5 K. We attained a 30% increase in J_c in the magnetic field below 1 T, which indicates that the irradiation defects contribute to vortex pinning. In contrast, we observed almost no change in the in-field J_c above 1 T. Irradiation with MeV protons could produce mostly random point defects and nanocluster [27] due to ion–nucleus collisions. Sylva et al. reported that 3.5 MeV proton irradiation with 6.40×10^{16} p/cm^2 dose (corresponding to 2.27×10^{-3} dpa) yields J_c improvement of about 40% at 4.2 K and 7 T with respect to the pristine film almost without a decrease in T_c [22]. On the contrary, J_c of 3.5 MeV proton irradiated Fe(Se,Te) films covered with 80 μm thick Al foil decreased by up to 80% after irradiation at 4.2 K. The in-field J_c performance in the irradiated FST films in our study could be attributed to the small number of vortex pinning defects created by the irradiation at low fluence.

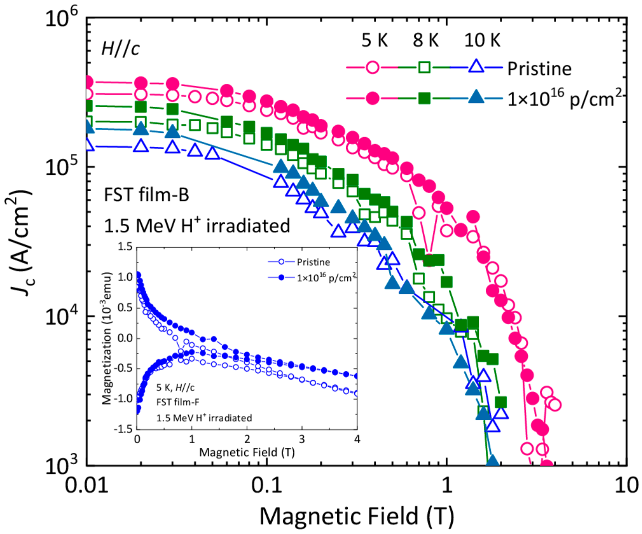

Figure 2. Magnetic field dependence of critical current density $J_c^{ab}(H)$ at 5, 8 and 10 K calculated using the critical-state Bean model for FST film-B pre- and post- 1.5 MeV proton irradiation with 1×10^{16} p/cm^2 dose. The inset shows magnetic hysteresis loop under $H//c$ at 5 K.

3.2. Transport Measurement

In transport measurements, the current is forced to flow through the sample in a particular direction, enabling the direct characterization of superconductivity as a function of temperature, applied magnetic field and field angle. However, we observed an obvious degradation of superconducting properties in the transport measurement of the FST film-B. This could be due to sample degradation, sample handling during mounting and unmounting in a measurement system and possible damage by the laser cutting for patterning the bridge on FST films. In this section, we refer to the FST film-A. Figure 3 presents the temperature dependence of the electrical resistivity before and after irradiation for FST film-A with 1×10^{15} p/cm^2 dose of 1.5 MeV proton. The FST films before and after the irradiation showed metallic behavior below 200 K. Additionally, 1.5 MeV proton irradiation with 1×10^{15} p/cm^2 dose has little effect on normal-state resistivity due to the low dpa. On the contrary, the normal-state resistivity shows nearly upwards parallel-shift upon 6 MeV Au-ion irradiation with a dose of 1×10^{12} Au/cm^2, corresponding to 6.42×10^{-3} dpa [11]. We observed no change in $T_{c,10}$ (=17.5 K) before and after the 1.5 MeV protons irradiation with 1×10^{15} p/cm^2 dose. This could be due to the low fluence, i.e., low dpa.

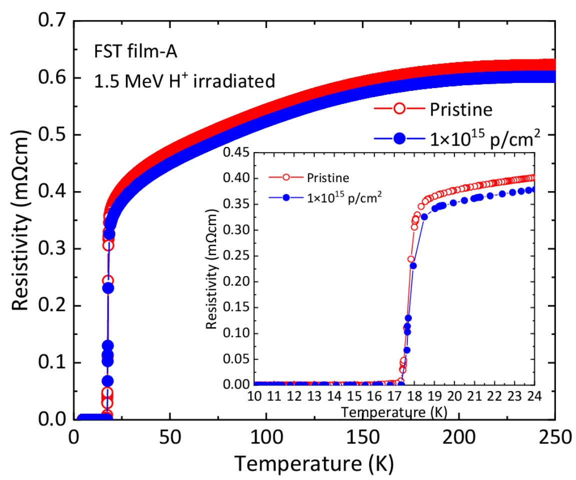

Figure 3. Temperature dependences of electrical resistivity at 0 T for the FST film-A before and after 1.5 MeV proton irradiation with 1×10^{15} p/cm^2 dose. Inset shows a magnified temperature region near T_c.

Figure 4 presents the magnetic field dependence of transport critical current density J_c with $H//c$ for the FST film-A before and after irradiation with 1.5 MeV protons to a dose of 1×10^{15} p/cm^2 at 4.2 K. Comparing J_cs obtained from magnetization and transport measurements, the values of J_c obtained from transport measurement are larger than those of J_c calculated from magnetization measurement. This would come from the difference of criterion to determine the J_c values. The positive effect of the proton irradiation on J_c at 4.2 K is unambiguous in the magnetic field below 1 T. As the magnetic field increased, the difference between pristine and the irradiated FST film became smaller. Similar behavior was observed in $J_c(H)$ (calculated from magnetization measurement in Figure 2) for FST film-B irradiated with 1×10^{16} p/cm^2 dose.

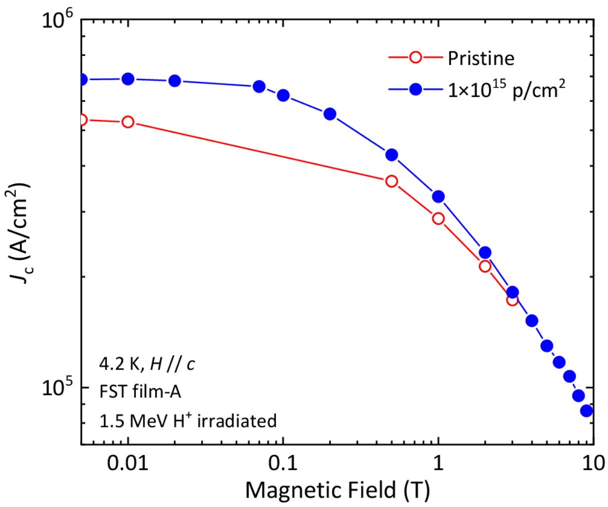

Figure 4. Magnetic field dependence of critical current density J_c obtained from transport measurement at 4.2 K for FST film-A pre- and post-1.5 MeV proton irradiation with 1×10^{15} p/cm^2 dose.

A more detailed representation of the pinning efficiency can be obtained from the angular dependence of J_c. We show $J_c(\theta)$ for the FST film-A irradiated with 1×10^{15} p/cm^2 dose of 1.5 MeV proton beam under 1 and 3 T at 4.2K in Figure 5. The pristine film has a less-anisotropic J_c angular dependence at 1 and 3 T without a prominent J_c peak at $H//c$, which is often observed in $YBa_2Cu_3O_y$ films [28]. A small J_c-anisotropy, γ_{Jc} ($J_c^{H//ab}/J_c^{H//c}$), of 1.7 is observed at 1 T. This value is smaller than the value of Fe(Se,Te) films grown on Fe-buffered MgO substrates (γ_{Jc} = 2.6) [29] while it is larger than the value of Fe(Se,Te) films grown on CaF_2 substrates [30,31]. These differences might arise from the difference of the substrate and buffer layer. Upon irradiation with 1.5 MeV proton, the J_c increases for most of the field orientations, retaining a small γ_{Jc} of 1.7 at 1 T, indicating that the vortex pinning defects would be less anisotropic and randomly distributed. At 3 T, there is a significant decrease in J_c in the angular range ±30° from $H//ab$. Iron-based and cuprate high-temperature superconductors commonly possess inherent layered structures, consisting of alternating conducting and insulating atomic planes. In general, the strong J_c peak for $H//ab$ could be ascribed to the vortex pinning by the intrinsic pinning and planar defects such as intergrowths and stacking faults, parallel to the *ab* plane [32–35]. In the iron–chalcogenide Fe(Se,Te) compound, which is composed of only the Fe–Se(Te) layer, $J_c(\theta)$ has a maximum at $H//ab$ due to intrinsic pinning from the Fe–Se(Te) intralayer and Van der Waals interlayer couplings [29,34,35]. Hence, the J_c suppression at around $H//ab$ would occur because of the reduction in the density of intrinsic pinning upon the irradiation.

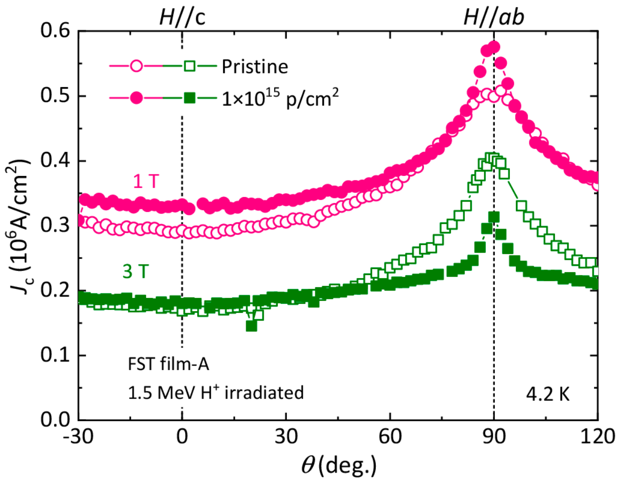

Figure 5. Angular field dependence of the critical current density J_c obtained from transport measurement for FST film-A before and after 1.5 MeV proton irradiation with 1×10^{15} p/cm^2 dose measured at 4.2 K under 1 and 3 T.

4. Conclusions

We conclude a study on the effect of 1.5 MeV proton irradiation on superconducting properties of FST films. Upon the irradiation up to 1×10^{16} p/cm^2 dose, T_c remains virtually unchanged in magnetization as well as in transport measurement. An approximately 30% enhancement of J_c in the magnetic field below 1 T is observed using 1.5 MeV proton irradiation with 1×10^{16} p/cm^2. Transport properties of a pristine film and an irradiated film with a fluence of 1×10^{15} p/cm^2 show a small anisotropy of J_c in the applied magnetic field range at 4.2 K. The enhancement of J_c for almost all the field orientations was accomplished by the irradiation at a dose of 1×10^{15} p/cm^2 at 4.2 K and 1 T. These results indicate that 1.5 MeV proton irradiation is effective in providing less anisotropic pinning defects in the magnetic field below 1 T in iron–chalcogenide superconducting films. Additionally, by fine tuning an irradiation fluence of proton, superconducting properties can be further improved.

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