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 such as 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 $1 \times 10^{16}$ 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

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 $\gamma$ 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 (\(\lambda = 266\) 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 \(\rho T\) and \(I–V\) curves using 0.1 \(\rho_n\) and 1 \(\mu\)V/cm criteria, respectively. Here, \(\rho_n\) 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 \(1 \times 10^{15}\) and \(1 \times 10^{16}\) 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 \(3.2 \times 10^{12}\) p/cm\(^2\)-s, corresponding to a beam current density of \(\sim 500\) 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, \(1 \times 10^{15}\) and \(1 \times 10^{16}\) p/cm\(^2\) are estimated to be \(\sim 3.2 \times 10^{-5}\) and \(\sim 3.2 \times 10^{-4}\) 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 \(1 \times 10^{15}\) and \(1 \times 10^{16}\) p/cm\(^2\) dose, respectively. Both the zero-field-cooled (ZFC) and field-cooled
3. Results and Discussion

3.1. Magnetic Measurements

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) $1 \times 10^{15}$ and (b) $1 \times 10^{16}$ 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 $1 \times 10^{16}$ 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 $J_{c,ab} = 20 \Delta M/(\alpha(1 - a/3b))$, where $\Delta M$ 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 $1 \times 10^{16}$ 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 $J_c$ 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 $6.40 \times 10^{16}$ p/cm$^2$ dose (corresponding to $2.27 \times 10^{-3}$ 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 $\mu$m 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.
In general, the strong defects would be less anisotropic and randomly distributed. At 3 T, there is a significant reduction in the density of intrinsic pinning upon the irradiation. The in-field hysteresis loop for the pristine film almost without a decrease in critical current density $J_c$ with respect to the pristine film could be attributed to the small number of vortex pinning defects created by up to 80% after irradiation at 4.2 K. The in-field hysteresis loop near $T_c$ with $H_{//c}$ at 5 K is unambiguous in the magnetic field below 1 T. As the magnetic field increased, the angular dependence of $J_c$ became smaller. Similar behavior was observed in the FST film-B irradiated with $1 \times 10^{16}$ p/cm$^2$ dose. Figure 3 presents the temperature dependence of electrical resistivity before and after irradiation with 1.5 MeV protons to a 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 $1 \times 10^{15}$ 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 $1 \times 10^{12}$ Au/cm$^2$, corresponding to $6.42 \times 10^{-3}$ dpa [11]. We observed no change in $T_{c,10}$ (=17.5 K) before and after the 1.5 MeV protons irradiation with $1 \times 10^{15}$ p/cm$^2$ dose. This could be due to the low fluence, i.e., low dpa.

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 4 presents the magnetic field dependence of transport critical current density $J_c$ for the FST film-A before and after irradiation with $1 \times 10^{15}$ p/cm$^2$ dose having $J_c$ peak at $10^6$ A/cm$^2$. The inset shows magnetic hysteresis loop under $H_{//c}$ at 5 K.
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 $1 \times 10^{15} \text{ p/cm}^2$ at 4.2 K. Comparing $J_c$s 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 $1 \times 10^{16} \text{ p/cm}^2$ 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 $1 \times 10^{15} \text{ p/cm}^2$ 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$_2$Cu$_3$O$_y$ films [28]. A small $J_c$-anisotropy, $\gamma_{jc}$ ($(J_c//ab)/J_c//c$), 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 ($\gamma_{jc} = 2.6$) [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 $\gamma_{jc}$ 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 $\pm 30^\circ$ 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.
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