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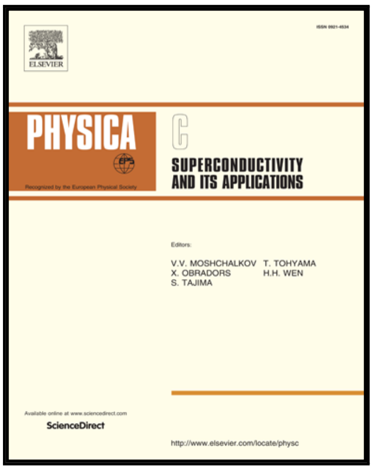

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Highlights

- Biaxial compressive strain effect on FeSe thin crystals
- A notable enhancement of superconductivity in FeSe
- An increase of the onset temperature of enhanced spin fluctuations in FeSe
- A suppression of the structural/nematic transition temperature in FeSe

Enhancement of superconductivity in FeSe thin crystals induced by biaxial compressive strain

X. F. Wang[a,b], Z. T. Zhang[c,d,e,*], W. K. Wang[a], Y. H. Zhou[a], X. C. Kan[e,b], X. L. Chen[a], C. C. Gu[a], L. Zhang[a], L. Pi[b,a], Z. R. Yang[a,e,f,**], Y. H. Zhang[b,a,f]

[a] *High Magnetic Field Laboratory, Chinese Academy of Sciences, Hefei 230031, China*
[b] *University of Science and Technology of China, Hefei 230026, China*
[c] *Institute of Ion Beam Physics and Materials Research, Helmholtz-Zentrum Dresden-Rossendorf, D-01314 Dresden, Germany*
[d] *Hochfeld-Magnetlabor Dresden (HLD-EMFL), Helmholtz-Zentrum Dresden-Rossendorf, D-01314 Dresden, Germany*
[e] *Key Laboratory of Materials Physics, Institute of Solid State Physics, Chinese Academy of Sciences, Hefei 230031, China*
[f] *Collaborative Innovation Center of Advanced Microstructures, Nanjing University, Nanjing 210093, China*

Abstract

We report on the enhancement of superconductivity in FeSe thin crystals induced by in-plane biaxial compressive strain, with an underlying scotch tape as an *in-situ* strain generator. It is found that, due to the compressive strain, the superconducting transition temperature $T_c \approx 9$ K of FeSe is increased by 30%–40% and the upper critical field $H_{c2}(0) \approx 14.8$ T is increased by $\sim 20\%$. In parallel, the T^*, which characterizes an onset of enhanced spin fluctuations, is raised up from 69 K to 87 K. On the other hand, the structural transition temperature $T_s \approx 94$ K, below which an orthorhombic structure and an electronic nematic phase settle in, is suppressed down by ~ 5 K. These findings reveal clear evolutions of the orders/fluctuations under strain effect in FeSe, the structurally simplest iron-based superconductor where the lattice/spin/charge degrees of freedom are closely coupled to one another. Moreover, the presented research provides a simple and clean way to manipulate the superconductivity in the layered iron compounds and may promote applications in related materials.

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[*] Corresponding author
[**] Corresponding author
Email addresses: `ztzhang@issp.ac.cn` (Z. T. Zhang), `zryang@issp.ac.cn` (Z. R. Yang)

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1. Introduction

The high-T_c superconductivity in iron-based superconductors (FeSCs) usually emerges from a magnetically ordered ground state, where chemical doping or physical pressure are used as control parameters [1]. Besides the magnetic order, a nematic order state, in which the four-fold lattice rotational symmetry is reduced to two-fold, becomes growingly important for understanding the superconductivity, because it is shown to be a generic feature among the FeSCs [2], and exhibits a close similarity with the superconducting order parameter in symmetry [3, 4]. The strong couplings between the lattice/spin/orbit degrees of freedom make it experimentally difficult to distinguish which of the orders is the driven force behind [2]. Although there is a general consensus on the electronic nature of the nematic order, its origin concerning whether spin or orbit degree is still under hot debate [2, 5, 6, 7, 8, 9, 10, 11, 12].

The FeSe superconductor draws intensive attention not only because it is the structurally simplest one among the FeSCs, but also due to its unique and interesting properties [13, 14, 15, 16, 17, 18]. For example, FeSe undergoes a tetragonal-to-orthorhombic structural transition as well as a nematic transition at $T_s \approx 90$ K [11, 14], but no static magnetic order is observed below T_s at ambient pressure [15]. In addition, the superconducting $T_c \approx 8$ K of FeSe sensitively depends on the microstructure and can be significantly enhanced up to 37 K by physical pressure [17], or to 43 K by intercalation of a molecular spacer layer [16], or even to above 100 K in single-layer films [18]. Therefore many efforts are made to investigate the electronic nematic phase in FeSe as well as its relationship with superconductivity [3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13].

Recently, we showed that the $T_c \approx 14$ K of thin crystals of an isostructural $FeTe_{0.5}Se_{0.5}$ superconductor can be enhanced by 14% due to the compressive strain of an underlying-attached commercial scotch tape [19, 20]. However, it is noticed that neither structural transition nor magnetic order is observed in the $\mathrm{FeTe_{0.5}Se_{0.5}}$ [19, 20]. In this paper, we show that the $T_c \approx 9$ K of FeSe thin crystals (of hundreds nanometers) exfoliated from high quality bulk single crystal is increased by 30%–40% and the upper critical field $H_{c2}(0) \approx 14.8$ T is increased by $\sim 20\%$ due to the biaxial compressive strain of the scotch tape. Simultaneously, the T^*, where an onset of enhanced spin fluctuations takes place, is raised up from 69 K to 87 K. On the other hand, the structural and nematic transition temperature $T_s \approx 94$ K is suppressed down by ~ 5 K. Our study reveals clear evolutions of the ordered states and provides informative experimental facts for understanding the relationships between them. Moreover, the presented research provides an effective way to control the superconductivity in layered superconductors and may promote related applications.

2. Materials and methods

High quality FeSe bulk single crystals were grown by $\mathrm{KCl/AlCl_3}$ chemical vapor transport technique as described elsewhere [21]. Chemical composition of the single crystals was determined as to be $\mathrm{Fe_{1.00(5)}Se}$ by energy-dispersive x-ray spectroscopy. Crystallinity of the samples was characterized by X-ray diffraction (XRD) with Cu K_α radiation at room temperature. Resistance were measured using a standard four-probe method in a Quantum Design Physical Property Measurement System (PPMS). Magnetic moment were measured in zero-field cooling sequence using a Quantum Design magnetic properties measurement system (MPMS) at a magnetic field of 20 Oe, which is applied out-of-plane for Bulk-FeSe and in a not well controlled orientation for the strained or non-strained thin crystals due to the small size of the samples.

3. Results and discussion

The XRD pattern of the FeSe bulk single crystal is shown in Fig. 1. Only $(00l)$ diffraction peaks appear, indicating that the c-axis is perpendicular to

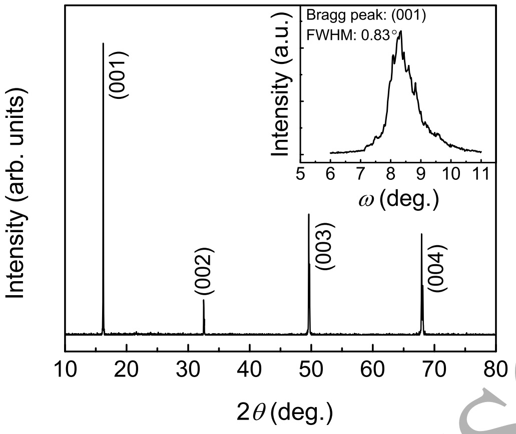

Figure 1: (Color online) (a) XRD pattern at room temperature for the Bulk-FeSe single crystal. Inset shows the rocking curve for the (001) reflection.

cleavage surface of the single crystal. Rocking curve for the (001) reflection was measured to determine the crystalline quality. As shown in the inset of Fig. 1, the full-width at half-maximum (FWHM) of the (001) reflection rocking curve is as narrow as 0.83°, which is much narrower than the FWHM of 5–6° reported for FeSe crystals grown by other techniques [22, 23], proving the very high quality of the single crystals used in this work.

FeSe is a layered compound with weak van der Waals couplings between the layers, therefore thin crystals can be easily exfoliated by using a scotch tape-based mechanical method [19, 20]. The thin crystals, used for resistance and magnetic moment measurements, were carefully chosen in a way that all of them have similar thickness to avoid possible thickness dependence of the measurements. The estimated thickness is of a few hundred nanometers, by using optical microscopy with backlight [19]. In our previous studies on $\mathrm{FeTe_{0.5}Se_{0.5}}$ thin crystals, we provided experimental evidences that the underlying substrate, i.e. the scotch tape, generates compressive strain effect on the thin crystal samples, by means of variable-temperature measurements of lattice contraction and single crystal X-ray diffraction [19, 20]. For instance, the in-plane relative lattice contraction $|\Delta L/L_{300\ \mathrm{K}}| \simeq 3\times10^{-3}$ for $\mathrm{FeTe_{0.5}Se_{0.5}}$ at low temperatures ($< T_c$)

is ~ 2 times smaller than that of the scotch tape $\sim 6 \times 10^{-3}$ [19]. As for FeSe, the $|\Delta L/L_{300\ \mathrm{K}}| \simeq 2 \times 10^{-3}$ is ~ 3 times smaller than the scotch tape [19, 21], which evidences a stronger compressive strain effect of the scotch tape on FeSe than on $FeTe_{0.5}Se_{0.5}$. The compressive strain effect can be released when the thin crystals are separated from the scotch tape by using dichloromethane solvent, which has no effect on the sample quality [19]. In the following, the FeSe bulk single crystal, strained FeSe thin crystal and FeSe thin crystal without strain are respectively labeled as Bulk-FeSe, STC-FeSe and TC-FeSe.

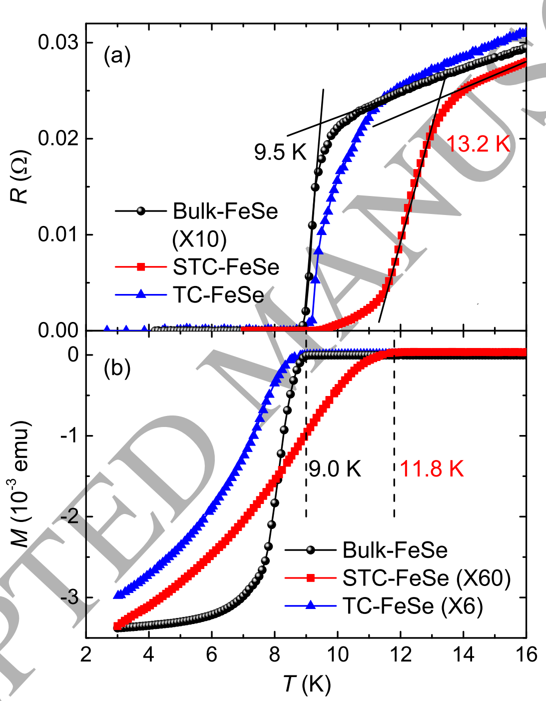

Figure 2: (Color online) (a) Temperature dependence of resistance for bulk single crystal (Bulk-FeSe), strained thin crystal (STC-FeSe) and thin crystal without strain (TC-FeSe). Absolute values of resistance for Bulk-FeSe are enlarged by 10 times for an easier comparison, as is marked in the legends. (b) Temperature dependence of the magnetic moment for Bulk-FeSe, STC-FeSe and TC-FeSe samples. Similarly, absolute values for STC-FeSe and TC-FeSe samples are enlarged by 60 and 6 times respectively. The large difference is due to the amounts of the used thin crystals.

Figure 2 displays the temperature-dependent resistance $R(T)$ and magnetic moment $M(T)$ at low temperatures for Bulk-FeSe, STC-FeSe and TC-FeSe. For Bulk-FeSe, the superconducting transition temperature is $T_c = 9$ K, characterized by a zero-resistance transition temperature in the $R(T)$ curve as well as a onset temperature of the diamagnetic transition in the $M(T)$ curve. The T_c is quite similar or even a little higher than that of 8 K reported for FeSe [21, 24, 25]. The sharp transition width 0.5–1 K, and the relatively high T_c, indicate the high quality of the sample used, as the rocking curve has revealed.

In comparison, the T_c of STC-FeSe is significantly increased by 31% (9.0 K to 11.8 K) determined by magnetic moment measurements, and by 39% (9.5 K to 13.2 K) determined by the cross-point in resistance measurements [Fig. 2]. In order to preclude the possible influence of the scotch tape on the sample quality, e.g. chemical decomposition, we separated the thin crystals from the scotch tape and got the TC-FeSe thin crystals without compressive strain. Clearly, after the compressive strain is released, the T_c of the TC-FeSe restores back to the bulk value, which confirms the clean strain effect of scotch tape on the FeSe thin crystals. We also rule out the possibility that the change of superconductivity is due to variation of thickness, by considering the two following facts. Firstly, as mentioned above, the used thin crystals were carefully chosen so that they possess similar thickness as possible. In fact, the determination of T_c of either STC-FeSe or TC-FeSe is well reproducible by repeating the experiments, although the absolute values of resistance and magnetic moment show variations due to the geometry and amount of the used thin crystals. Secondly, the enhanced T_c of the strained STC-FeSe restores back to the bulk value when the strain effect is released, which cannot be interpreted as a thickness dependence of superconductivity.

In order to study the compressive strain effect on the upper critical field H_{c2} of FeSe, we measured the temperature-dependent resistance for the Bulk-FeSe and STC-FeSe samples at magnetic fields up to 9 Tesla, as shown in Fig. 3(a) and Fig. 3(b). A progressive suppression of the superconducting transition temperature with increasing magnetic field is observed for both samples. Fig-

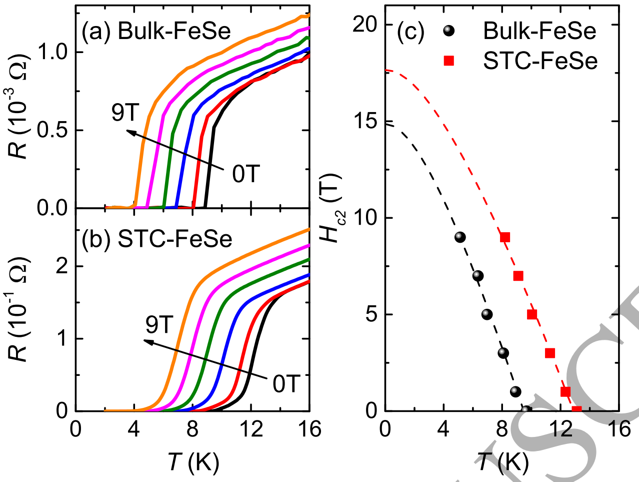

Figure 3: (Color online) (a)-(b): Temperature dependence of resistance below 16 K for (a) Bulk-FeSe and (b) STC-FeSe, measured under different magnetic fields $\mu_0 H^{\parallel c}$ up to 9 Tesla. (c): Temperature dependence of the upper critical field H_{c2} for Bulk-FeSe and STC-FeSe. The dashed lines are the best fits of the WHH model to the data.

ure 3(c) displays the corresponding $H_{c2}(T)$ phase diagram. For the Bulk-FeSe, the obtained zero temperature-limit of $H_{c2}(T)$ is 14.8 Tesla by extrapolation of the Werthamer-Helfand-Hohenberg (WHH) fitting [26], which is quite similar to the reported values by other groups [27, 28, 29]. Actually, by temperature-dependent resistance measurements down to 1.5 K in pulsed high magnetic fields up to 55 T, Her *et al.* showed that the H_{c2} of FeSe in this field orientation is 15 T, which is quite close to our results.[30]. For lower temperatures down to 40 mK, Vedeneev *et al.* observed an upward curvature-like slight deviation from the WHH model, which was interpreted as a signature of multiband superconductivity in these materials [31]. Here, as a result of the compressive strain effect on the thin crystal sample, we show that the $H_{c2}(0)$ of the STC-FeSe sample is increased up to 17.6 Tesla by 19%.

Figure 4 displays the temperature-dependent normalized resistance $R_{\mathrm{N300K}}(T)$ in the whole temperature range. For the Bulk-FeSe sample, a typical metallic conducting behavior is observed above T_c, with a kink located at $T_s \approx 94$ K.

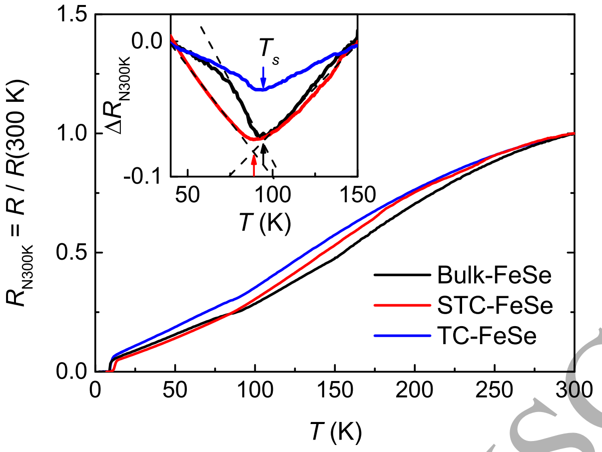

Figure 4: (Color online) Temperature dependence of normalized resistance $R_{\mathrm{N300K}} = R(T)/R(300\ \mathrm{K})$ for Bulk-FeSe, STC-FeSe and TC-FeSe. The inset shows a zoom of the temperature-dependent $\Delta R_{\mathrm{N300K}} = R_{\mathrm{N300K}} - R_{\mathrm{N300K,Linear}}$ around T_s, where $R_{\mathrm{N300K,Linear}}$ denotes a linear background determined from a straight line connecting the two points of $R_{\mathrm{N300K}}(40\ \mathrm{K})$ and $R_{\mathrm{N300K}}(150\ \mathrm{K})$.

Such kink feature is attributed to a tetragonal to orthorhombic structural transition and a concomitant electronic nematic phase transition [11, 14, 24, 25]. For the STC-FeSe sample, however, the kink feature is broader with a lower T_s of $\sim$ 89 K, implying a suppression effect of the compressive strain on the structural/nematic transition.

In FeSe superconductor, spin fluctuation is believed to be a promising paring mechanism [32]. In fact, nuclear magnetic resonance studies showed that the spin fluctuations in FeSe start to settle in at a temperature well above the superconducting T_c [10, 11, 32]. Recently, Rößler *et al.* showed that a change of slope of the temperature-dependent magnetoresistance at a crossover temperature $T^* \approx 75$ K is a characteristic of the onset of enhanced spin fluctuations [33]. In present work, the competition between superconducting ground state and electronic nematic order state is tuned towards the former one via the scotch tape-generated biaxial compressive strain. As a consequence, we expect an increase of the onset temperature T^* of the enhanced spin fluctuations for the

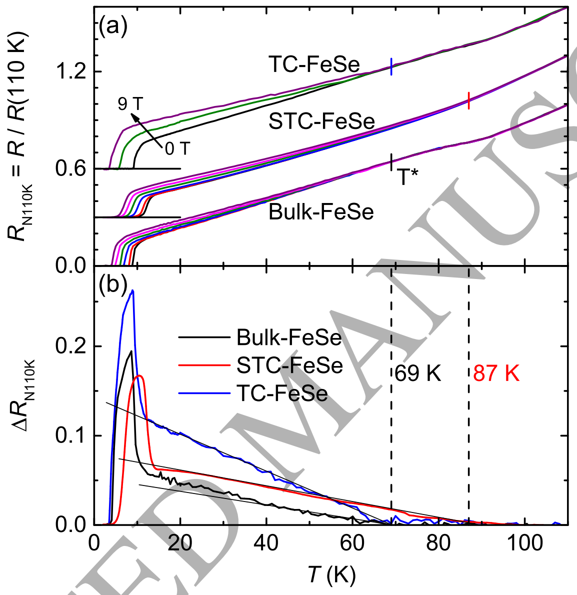

Figure 5: (Color online) (a) Temperature dependence of normalized resistance $R_{\mathrm{N110K}} = R(T)/R(110\ \mathrm{K})$ for Bulk-FeSe, STC-FeSe and TC-FeSe, measured under different magnetic fields $\mu_0 H^{\parallel c}$ up to 9 Tesla. Note: the curves for STC-FeSe and TC-FeSe samples are up-shifted by 0.3 and 0.6 units for clarity. (b) The magnetoresistance $\Delta R_{\mathrm{N110K}} = R_{\mathrm{N110K}}(9\ \mathrm{T}) - R_{\mathrm{N110K}}(0\ \mathrm{T})$ as functions of temperature for Bulk-FeSe, STC-FeSe and TC-FeSe.

STC-FeSe sample. Figure 5 displays the temperature dependence of the normalized resistance at different magnetic fields, and magnetoresistance between 0 Tesla and 9 Tesla. For Bulk-FeSe, the T^* where the slope changes [Fig. 5(b)] is about 69 K, close to the reported 75 K by Rößler *et al.* The difference may be due to the accuracy of determination of the not well-defined T^* in a broad crossover [33]. As expected, the T^* of STC-FeSe sample is increased up to ~ 87 K and that of the TC-FeSe sample restore back to the bulk value. Above T^*, the magnetoresistance is neglectable for all samples, which can be interpreted by the conducting behaviors of a conventional metal [33].

In FeSCs, the electronic nematic order state is found to compete with superconductivity [34, 35]. In FeSe, the very sensitive dependence of superconducting T_c on the microstructure might be a result of the manipulation of the electronic nematic order as an intermediate bridge. However, due to the close couplings of the lattice/spin/orbit degrees of freedom, it is difficult to determine the primary driven force between them [2]. In present work, the biaxial compressive strain on the FeSe thin crystals generated by the underlying scotch tape is considered as an in-plane isotropic effect, which disfavors the orthorhombic lattice distortion in *ab* plane as well as the breaking of four-fold rotational symmetry. Therefore the measured enhancement of superconductivity in the STC-FeSe sample due such strain may arise from a depression of the electronic nematic phase, which is supported by the downward shifted transition temperature T_s.

4. Conclusions

In summary, we studied the enhancement of superconductivity in FeSe thin crystals by the underlying scotch tape generated biaxial compressive strain. The superconducting transition temperature $T_c \approx 9$ K of FeSe is increased by 30%–40% and the upper critical field $H_{c2}(0) \approx 14.8$ T is increased by $\sim$ 20%. In line with these results, the onset temperature T^* of enhanced spin fluctuations increases from 69 K up to 87 K. On the other hand, the structural and nematic transition temperature T_s decreases by ~ 5 K. This study provides informative experimental facts for understanding the relationships between the nematic order and its relationship with superconductivity in FeSe. In addition, the method used is an effective way to tune the superconductivity in layered iron-based superconductors and may promote related applications.

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