Fragile pressure-induced magnetism in FeSe superconductors with a thickness reduction

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The emergence of high transition temperature ($T_c$) superconductivity in bulk FeSe under pressure is associated with the tuning of nematicity and magnetism. However, sorting out the relative contributions from magnetic and nematic fluctuations to the enhancement of $T_c$ remains challenging. Here, we design and conduct a series of high-pressure experiments on FeSe thin flakes. We find that, as the thickness decreases, the nematic phase boundary on temperature-pressure phase diagrams remains robust while the magnetic order is significantly weakened. A local maximum of $T_c$ is observed outside the nematic phase region, not far from the extrapolated nematic endpoint in all samples. However, the maximum $T_c$ value is reduced associated with the weakening of magnetism. No high-$T_c$ phase is observed in the thinnest sample. Our results strongly suggest that nematic fluctuations alone can only have a limited effect while magnetic fluctuations are pivotal on the enhancement of $T_c$ in FeSe.

Unconventional superconductors including high-$T_c$ cuprates, heavy-fermion systems, and iron-based superconductors (IBS) share common features in the intimate relations with magnetic, structural, and electronic orders [1-12]. Understanding the impact of these orders on the stabilization or the optimization of superconductivity is a central topic. Using tuning parameters such as chemical substitution and high pressure, these ordered phases can be controlled, providing a means to disentangle their role on the emergence of superconductivity. In some IBS, such as Ba(Fe$_{1-x}$Co$_x$)$_2$As$_2$, the emergence of the antiferromagnetic (AFM) ordered phase or spin density wave (SDW) phase is accompanied by a nematic phase transition [13-14], in which the rotational symmetry is broken but the translational symmetry is preserved. When the nematic and SDW phase transitions are sufficiently weakened, the superconductivity appears and develops into a dome shape. Recent studies suggest that, near the quantum critical points of nematicity and SDW order, both enhanced nematic fluctuations and magnetic fluctuations can mediate Cooper pairing [11-15]. However, due to the proximity of the nematic phase and the magnetically ordered phase, the interplay among superconductivity, nematicity, and magnetism remains elusive.

FeSe, as another prominent member of IBS, has drawn intensive attention [5-10, 14-18, 57]. Bulk FeSe undergoes a nematic phase transition at $T_N \approx 90$ K, and further becomes superconducting at $T_c = 9$ K, without the presence of magnetism [5-13, 28]. Under pressure, the SDW phase can be induced at around 10 kbar [28, 29, 44], which develops into a dome shape in the temperature-pressure ($T$-$p$) phase diagram and spans more than 40 kbar [30, 31]. Concomitantly, the superconductivity is enhanced in a three-step process and reaches $\sim 40$ K at around 60 kbar. The novel phases in FeSe can also be tuned by combining chemical substitution and pressure [28, 44-52]. In FeSe$_{1-x}$Te$_x$, the nematic phase is suppressed as sulfur content increases, and a nematic quantum critical point is observed without the presence of magnetism [45, 50, 53]. Meanwhile, the SDW phase in FeSe$_{1-x}$S$_x$ only appears when pressure is applied and its onset pressure shifts to a higher value as sulfur content increases [46, 49, 54]. Intriguingly, high-$T_c$ phases appear at both ends of the SDW dome, suggesting that the enhanced magnetic fluctuations may help to increase $T_c$ [46]. A recent result on FeSe$_{1-x}$Te$_x$ shows that, as Te content increases, the nematic phase can also be suppressed, while the pressure-induced SDW phase disappears gradually against $x$ [55]. At $x > 0.10$, no magnetic order is observed under pressure [55]. However, a superconducting dome still forms near the nematic phase, indicating that at least for the FeSe$_{1-x}$Te$_x$ series, the nematic fluctuations alone may also help to promote $T_c$ [55].

In addition to pressure and chemical substitution, the superconductivity in FeSe can also be tuned by sample thickness [26, 53, 54]. Monolayer FeSe grown on SrTiO$_3$ substrate exhibits a remarkably high $T_c$ over 100 K [35], much higher than in the bulk sample under pressure. On the other hand, thin FeSe films cleaved from single crystals exhibit suppression of $T_c$ and $T_N$ as the thickness is reduced [26, 30, 35]. Since the magnetically ordered phase in the bulk FeSe only appears with pressure applied, whether thickness reduction alters the magnetic fluctuations and thus $T_c$ remains unknown yet an important topic. According to Mermin–Wagner theorem, the long-range order with broken symmetry is prohibited in two-dimensional materials [55]. Therefore, with a decreasing thickness, the magnetic order is expected to be weakened, paving a way to critically examine the bordering high-$T_c$ superconductivity. Thus, the ability to simultaneously apply pressure and to alter the sample thickness provides a powerful tool to interrogate the relationship between magnetism and superconductivity in FeSe.

Here, we report our high-pressure investigation of superconducting, nematic, and magnetic phase transitions in FeSe with thicknesses ranging from 140 nm to 5 $\mu$m...
using the concept of a “device-integrated diamond anvil cell technique” we developed (see Methods). Thin flakes of FeSe are first isolated from high-quality single crystals and then they are transferred onto the diamond anvils pre-patterned with electrodes. The diamond anvil integrated with thin FeSe devices (see Fig. 1(a)) then forms a part of the diamond anvil cell for resistance measurements under hydrostatic pressure. We find that, as the thickness is reduced, the nematic phase shows similar pressure evolutions, while the SDW phase shrinks significantly. Importantly, the high-$T_c$ superconductivity can only be observed in thicker samples, in which the SDW phase persists to higher pressure and temperature. In other words, the robustness of the SDW phase is crucial for stabilizing the high-$T_c$ superconductivity in FeSe. Using the constructed $T$-$p$ phase diagrams, we extract the interplay among nematicity, magnetism, and superconductivity.

Figure 1(b) displays the electrical resistance ($R$) as a function of temperature ($T$) normalized to $R(150 \text{ K})$ for samples with the thickness ranging from 140 nm to 5 $\mu$m, collected on warming at ambient pressure. At around 100 K, pronounced slope changes due to the nematic phase transition are observed in all samples. Here, we define the nematic phase transition temperature $T_s$ as the temperature at which the $dR/dT$ is a local minimum (see Fig. 1(c)). To alleviate the effect of the thermal lag due to the usage of a pressure cell, both warm-up or cool-down data are analyzed for extracting $T_s$ accurately (see Supporting Information S2).

Figure 1(d) displays the normalized resistance $R(T)/R(150 \text{ K})$ at $T < 15 \text{ K}$. Like the bulk sample [5,13,20,22,23], thin FeSe samples undergo a superconducting transition at low temperatures. However, a systematic suppression of $T_c$, defined as the temperature at which the resistance reaches zero, is observed as the thickness is reduced. Together with the $T_s$ data, the temperature-thickness phase diagram at ambient pressure is shown in Fig. 1(e). In the thickness range explored, our $T_s$ of around 90 K agrees quantitatively with previous results on the bulk FeSe [5,18–20,22,23]. Considering that the superconducting transitions observed are sharp (Fig. 1(d)) and the suppression of $T_s$ against thickness is systematic, we conclude that the observed phenomena are the intrinsic properties from thickness reduction.

Figure 2 displays the evolution of $R(T)$ under pressure for samples with thicknesses of 5 $\mu$m, 3 $\mu$m, 2.3 $\mu$m, 510 nm, 280 nm, and 140 nm. As pressure increases, the anomaly in $R(T)$ corresponding to the nematic phase transition shifts to a lower temperature, as indicated by the brown arrows. In Supporting Information S3, we show the evolution of $dR/dT$ under pressure for illustration. At $p > 20 \text{ kbar}$, no feature corresponding to the $T_s$ is observed in $R(T)$ or $dR/dT$, suggesting the complete suppression of the nematic phase. Our observations also agree well with previous results in bulk FeSe under high pressure [22,30,31]. Given that the thin FeSe samples in this study possess similar $T_s$ at ambient pressure, the

**FIG. 1** (a) The scanning electron microscope (SEM) image of the device integrated diamond anvil. The inset displays the top view of a 280 nm thick FeSe sample covered with thin h-BN. False colors are used for illustration. (b) The temperature dependence of normalized resistance, $R/R(150 \text{ K})$, for samples with different thicknesses, collected on warming. (c) The temperature dependence of $dR/dT$ for the curves in b. The arrows indicate the temperature of the nematic phase transition. (d) The temperature dependence of $R(R(150 \text{ K})$ at $T < 15 \text{ K}$. The curves in b-d are vertically shifted for clarity. (e) The temperature-thickness phase diagram of measured thin FeSe. $T_s$ (brown) is determined from both warming and cooling data in $dR/dT$ (see Supporting Information S2). $T_c$ is identified as the temperature at which the resistance reaches zero. The regions for nematic, and superconducting phases are coloured for clarity.
FIG. 2 The temperature dependence of resistance, $R(T)$, collected on warming for thin FeSe under high pressure with thickness of (a) 5 µm, (b) 3 µm, (c) 2.3 µm, (d) 510 nm, (e) 280 nm and (f) 140 nm. The anomalies in $R(T)$ corresponding to $T_c$, $T_s$, and $T_m$ are indicated by red, brown and blue arrows, respectively. Except for the ambient pressure data, the curves are vertically shifted for clarity.

FIG. 3 The temperature dependence of resistance (upper) and its derivative (lower) in fields up to 14 T with $I\parallel ab$ $\perp B$ for the 2.3 µm thick sample at (a) 18 kbar, (b) 24.4 kbar and (c) 27.6 kbar. Zero field $T_c$ are indicated by the vertical red dotted lines. $T_s$ and $T_m$ are indicated by the vertical dashed brown and dashed blue lines, respectively.

At 17.9 kbar, an additional anomaly manifested as a pronounced upturn is observed in $R(T)$ at 19.1 K (blue arrow). As pressure increases, this anomaly gradually shifts to higher temperatures. At $p \geq 38$ kbar, the anomaly becomes a downward kink. The evolution of this $R(T)$ anomaly under pressure agrees well with the assignments of the SDW phase transition in $R(T)$ reported previously in bulk FeSe, FeSe$_{1-x}$S$_x$ series and FeSe$_{1-x}$Te$_x$ series under pressure [30, 46, 55]. Following the same procedure [30], we obtained the SDW phase transition temperature $T_m$ from either the peak or dip features in $dR/dT$ (see Supporting Information S3).

In 2.3 µm thick sample, a pronounced upturn is also observed in $R(T)$ at 18 kbar. At 24.4 kbar, and 27.6 kbar, sharp superconducting transitions are observed, while no anomaly corresponding to the SDW phase transition is observed. Inspired by the results on bulk FeSe at 60 kbar, in which the anomaly associated with the SDW phase transition emerges when the superconductivity is suppressed by the high magnetic fields [30], we conducted magneto-electrical resistance measurements. Figure 3 displays the obtained results with $I\parallel ab \perp B$. At 18 kbar, the upturn becomes even more pronounced at high fields. The enhanced magnetoresistance in the SDW phase suggests that the Fermi surfaces or/and carrier scattering rates are affected by the pressure-induced antiferromagnetism. At 24.4 kbar and 27.6 kbar, pronounced upturns appears in the $R(T)$ at high fields. The corresponding dip features in $dR/dT$ become visible when the field is larger than 7 T, and remain robust up to 14 T, satisfying the signature of the magnetic phase transition [30]. As pressure continues to increase, the anomaly associated with $T_m$ shows up again in the zero-field $R(T)$ curves, and reaches over 40 K at $p \sim 40$ kbar. At higher pres-
mes, $T_m$ can no longer be unambiguously assigned.

In 510 nm, 280 nm and 140 nm thick samples, the pressure evolution of the SDW phase shows a drastically different behaviour compared with the few-micrometer thick samples. At $\sim 20$ kbar, additional upturn can be observed in $R(T)$ at zero field (see Fig. 2). Utilizing the same strategy of measuring $R(T)$ in high fields, we also confirmed the SDW phase transition in the 280 nm thick sample at 27.8 kbar, and in the 140 nm thick sample at 24.9 kbar (see Supporting Information S3). At $p > 30$ kbar, no SDW phase transition is observed for all three samples with thicknesses of a few hundred nanometers. Hence, the SDW region shrinks substantially in the $T$-$p$ phase diagrams, indicating a rapid weakening of magnetism in these thin FeSe samples.

Finally, we turn to the pressure evolution of $T_c$ in thin FeSe. In all samples measured, $T_c$ first experiences an enhancement at low pressures ($p < \sim 15$ kbar), and then it is suppressed when approaching the critical pressure of the nematic phase transition ($p \approx 20$ kbar). The situation is more complex away from the nematic phase. For the group of thicker samples, namely 5 $\mu$m, 3 $\mu$m, and 2.3 $\mu$m thick samples, $T_c$ is enhanced initially with the accompanying enhancement of $T_m$, and $T_c$ reaches 27.0 K at 35.1 kbar for 5 $\mu$m thick sample, 23.7 K at 33.6 kbar for 3 $\mu$m thick sample, and 28.2 K at 38.5 kbar for 2.3 $\mu$m thick sample. At higher pressure, $T_m$ continues to increase and saturates, while $T_c$ begins to drop. In the thinner group, namely the 510 nm, 280 nm, and 140 nm thick samples, $T_c$ only experiences a slight increase after leaving the nematic phase. In the 510 nm thick sample, $T_c$ reaches 12.6 K at 41.1 kbar, similar to the peak value of $T_c$ when the nematic phase is present. At 48.7 kbar, no zero resistance is observed down to 2 K. In 280 nm and 140 nm thick samples, $T_c$ values measured are less than 7 K, below the maximum $T_c$ values achieved in the nematic phase. Considering the SDW phase is fragile in the thinner group, the suppression of $T_c$ can be naturally associated with the weakening of the magnetism.

In contrast to the thickness dependence of physical properties in many 2D materials [59–61], a reduction of thickness to hundreds of nanometers can already alter the physical properties of FeSe under pressure. Based on our $T$-$p$ phase diagrams and previous results [28, 30, 44], we propose a schematic pressure-thickness phase diagram at $T = 0$ K showing the nematic and SDW phase boundaries, as shown in Fig. 4-g. From the schematic phase diagram, it can be seen that the nematic phase boundary is nearly unaffected by the thickness reduction over a wide range of thicknesses, as shown by the nearly horizontal phase boundary above $\sim 100$ nm. On the other hand, when the thickness is reduced, the SDW phase becomes more fragile, and the pressure range over which the SDW phase exists shrinks significantly. As presented above, the weakening of the SDW order has an observable effect on the reduction of $T_c$.

Why is a robust SDW phase crucial for realizing high-$T_c$ superconductivity in FeSe? In superconductors driven
by magnetic quantum criticality, the superconductivity is optimized near the magnetic quantum critical point, at which magnetic fluctuations are critically enhanced \[1,4,11,62\]. Such a scenario highlights the importance of magnetic fluctuations as an effective pairing glue. Thus, it is natural to speculate that magnetic fluctuations are critically enhanced near the magnetic quantum critical point, the superconductivity is optimized near the magnetic quantum critical point, and the superconductivity is optimized near the magnetic quantum critical point.

In summary, using thickness reduction and high pressure as tuning knobs, we systematically investigate the evolution of superconductivity, nematicity, and magnetism in thin FeSe with thicknesses ranging from 5 \(\mu m\) to 140 \(\mu m\). We find that, as the thickness is reduced, the nematic phase boundary remains robust on \(T-p\) phase diagrams, while the magnetically ordered phase shrinks significantly. Meanwhile, in all samples measured, a local maximum of \(T_c\) is observed outside the nematic phase region, not far from the extrapolated nematic endpoint, suggesting that the nematic quantum fluctuations can enhance superconductivity. However, associated with the weakening of magnetism, the superconductivity is also weakened. The pressure-induced high-\(T_c\) superconductivity no longer exists in our thinner samples, where magnetism is fragile. Our observations strongly suggest that nematic quantum fluctuations alone can only have a limited promotion of \(T_c\), while magnetic fluctuations are pivotal for the stabilization of high-\(T_c\) superconductivity.

**METHODS**

**Crystal growths.** High-quality single crystals of FeSe were grown by chemical vapor transport with a eutectic KCl-AlCl\(_3\) mixture as the transport agent \[21,41\]. High-quality h-BN single crystal with the length of about 7 \(mm\) was grown under high pressure using a flux method. For detailed sample characterizations, see Supporting Information.

**Device integrated diamond anvil cell.** The diamond anvil cells used were equipped with two low side angle diamond anvils (Type IIa) with a culet diameter of 800 \(\mu m\) and a bevel of 1000 \(\mu m\). Stainless steel gaskets with a 10 \(\mu m\) thick insulation layer were prepared following the method in Ref. \[64\]. The anvils inlaid in the piston were then patterned with micro electrodes by photo-lithography and PVD coating. Thin flakes of FeSe were exfoliated from single crystals using the “blue tape” (from Nitto Denko Co.) and then attached to the silicone elastomer poly-dimethylglyoxime (PDMS, Gelfilm from Gelpak) stamp. Well-chosen flakes were then transferred onto the patterned electrodes. For 140 \(nm\), 280 \(nm\), 510 \(nm\) and 2.3 \(\mu m\) thick samples, a thin layer of h-BN

**FIG. 5 The temperature exponent \(\alpha\) for the resistance as a function of pressure for \(p > 16\) kbar and \(T\) between 100 K and \(\max\{T_m, T_s\}\). For detailed data analysis procedure, see Supporting Information S4.**
was transferred onto the sample for encapsulation. The dimensions of the resulting devices were determined in a dual beam focused ion beam system (Scios 2 DualBeam from Thermo Scientific). High-purity glycerine was used as the pressure transmitting medium. The pressure was determined by the spectral measurement of ruby fluorescence at room temperature [65].

**Measurements.** Magneto-electrical resistance measurements were carried out in a Physical Property Measurement System by Quantum Design with a base temperature of 1.8 K and fields up to 14 T.

**ASSOCIATED CONTENT**

The Supporting Information is available.

Figures S1 – S17 on the characterizations of FeSe and h-BN, ruby fluorescence spectra and additional transport data.

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