Pressure Induced Stripe-order Antiferromagnetism and First-order Phase Transition in FeSe

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(Dated: November 11, 2016)

To elucidate the magnetic structure and the origin of the nematicity in FeSe, we perform a high-pressure $^{77}$Se NMR study on FeSe single crystals. We find a suppression of the structural transition temperature with pressure up to about 2 GPa from the anisotropy of the Knight shift. Above 2 GPa, a stripe-order antiferromagnetism that breaks the spatial four-fold rotational symmetry is determined by the NMR spectra under different field orientations and with temperatures down to 50 mK. The magnetic phase transition is revealed to be first-order type, implying the existence of a concomitant structural transition via a spin-lattice coupling. Stripe-type spin fluctuations are observed at high temperatures, and remain strong with pressure. These results provide clear evidences for strong coupling between nematicity and magnetism in FeSe, and therefore support a universal scenario of magnetic driven nematicity in iron-based superconductors.

PACS numbers: 74.70.-b, 76.60.-k

In most iron-based superconductors, superconductivity emerges near an antiferromagnetic (AFM) phase, making it important to study the nature of their magnetism [1,2]. For iron pnictides, the magnetic ground state typically has a stripe-type, or ($\pi$, 0), AFM order [3]. The magnetic transition at $T_N$ is preceded by a tetragonal-to-orthorhombic structural transition at $T_s$ $\geq$ $T_N$ where the lattice $C_4$ symmetry is broken, and the orthorhombic phase is denoted as a nematic state. The nematicity can be observed as anisotropy in the in-plane resistivity [4] and spin fluctuations [5], and the splitting of the degenerate $d_{xz}/d_{yz}$ orbitals [6] (so called orbital ordering [7]). Although it is generally believed that the nematicity has an electronic origin, it is still highly debated whether the nematicity is driven by the spin fluctuations or the orbital ordering [8].

Recent discoveries in the bulk FeSe superconductors [9] make this unsolved issue even more elusive: at ambient pressure, the electronic nematicity shows up below $T_s$ $\sim$ 90 K [10, 11], while a magnetic ordered state is absent. Applying pressure above 1 GPa, however, magnetic ordering emerges, and the ordering temperature $T_N$ increases with pressure [12, 13]. Meanwhile, $T_s$ is substantially suppressed at $\sim$ 2 GPa [14, 15]. These seemingly sharp contrasts between FeSe and iron pnictides challenge the existed view of the interplay among nematicity, orbital ordering and magnetism, and inspire various theoretical proposals for the nature of magnetism in FeSe [16-19]. On the experimental side, inelastic neutron scattering measurements suggest the coexistence of stripe and checkerboard spin fluctuations at ambient pressure [20, 21], and transport measurements report high-temperature superconductivity (HTSC) in both the ambient-pressure nematic and the high-pressure magnetic phases [14, 15, 22, 23]. Therefore, resolving the nematicity and the magnetic structure in FeSe not only helps building up the proper theory on the magnetism of FeSe, but also becomes important in understanding the HTSC in FeSe and other iron-based superconductors.

In this work, we present our $^{77}$Se NMR studies on high-quality FeSe single crystals with pressures up to 2.4 GPa and temperatures down to 50 mK. Our main results are summarized in the phase diagram of Fig. 1. From the pressure dependence of the NMR spectral splitting under an in-plane magnetic field, we observe a decrease of the structural transition temperature $T_s$ with pressure. However, the stripe-type spin fluctuations, characterized by the anisotropic $1/T_1(T)$, are enhanced below a weakly pressure dependent temperature $T^* \sim$ 100 K. We find that the magnetic ordering emerges about $\sim$ 0.2 GPa higher than earlier reports [14, 15]. The magnetic transition at $T_N$ above 2 GPa is first-order, and $T_N$ increases with pressure. The S-AFM phase necessarily breaks the $C_4$ symmetry as the magnetic ordering in iron pnictides. The discovery of the strong stripe-type spin fluctuations at high temperatures, the first-order magnetic transition, and the low-temperature S-AFM ground state under pressure clearly reveals a universal magnetic origin of the nematicity in both FeSe and iron pnictides. They also shed light on the important role of magnetism on superconductivity in iron-based superconductors.

Details of FeSe single crystal synthesis and characterization are presented in supplemental S1 and S2. The superconducting transition temperature $T_c$ is determined in situ by the RF inductance of the NMR coil (supplemental S3). The NMR measurements were performed under 10.3 T with two field configurations, $H \parallel acb$ (tetragonal $[1 1 0]$ direction) and $H \parallel c$. Daphne oil was used as the...
and $H$ NMR spectra are shown at position from the NMR line splitting below selected temperatures, with evidence of structural transition of Knight shifts, $\Delta K_a, b$ [26, 27]. In the orthorhombic phase, the in-plane Knight shift is anisotropic. This leads to two resonance peaks respectively corresponding to $H \parallel a$ and $H \parallel b$ due to structural twinning. In Fig. 2(a), NMR spectra are shown at $P = 0.56$ GPa for several selected temperatures, with evidence of structural transition from the NMR line splitting below $T_N$. The difference of Knight shifts, $\Delta K_a, b = |K_a - K_b|$, where $K_a$ and $K_b$ are the Knight shifts for $H \parallel a$ and $H \parallel b$ respectively, follows a mean-field like temperature dependence, consistent with a second-order phase transition [26]. Similar behavior of $\Delta K_a, b$ is observed with pressures up to $\sim 2$ GPa (Fig. 2(b)). As presented in the phase diagram (Fig. 1), $T_s$ shows a gradual suppression with pressure below $2$ GPa. At $P = 2.4$ GPa, line splitting is absent above $T_N$ and cannot be resolved below $T_N$ (Fig. 3(a)).

Next we study the magnetic ordering in the pressurized phase. Below $T_s$, the spin-lattice relaxation rates, $1/T_1$, are different for two frequency peaks with $H \parallel a$ and $b$, and data are presented in Fig. 3(a)-(c). For $P > 1.34$ GPa, a magnetic phase transition is clearly seen from the peaked feature in $1/T_1(T)$ (Fig. 3(b)-(c)) and the broadening of the NMR spectra upon cooling. Typical spectral data at $2.4$ GPa are shown in Fig. 3(a)-(c). Below $30$ K, the spectrum with $H \parallel a$ shifts slightly to the high-frequency side, and its FWHM decreases from $\sim 10$ kHz at $T = 30$ K to $\sim 300$ kHz at $T = 26$ K (Fig. 3(a)), signaling the magnetic transition. Followingcooling, the spin-spin relaxation time $T_2$ increases from $\sim 5$ ms at $120$ K to $\sim 20$ ms below $T_N$, which ensures correct analysis on the spectral weight. In Fig. 3(c), the normalized total spectral weight is drawn as a function of temperature at this pressure. The weight drops steeply by $50\%$ from $T = 30$ K to $26$ K across the magnetic transition, develops a plateau-like feature between $26$ K and $18$ K, and then decreases slowly upon further cooling due to RF screening below $T_C$.

The $1/T_1$ shows a sudden drop at $28$ K defined as $T_N$, coinciding with the midpoint of the magnetic transition from the spectral loss. With $H \parallel c$, the spectra keep narrow (Fig. 3(b)), but the total spectral weight decreases when cooled below $30$ K and reaches zero at $26$ K (Fig. 3(c)). Two split broad NMR lines ($\sim 300$ kHz) are seen at $50$ mK (Fig. 3(b)), corresponding to $H_{\text{c}} \approx \pm 0.48$ T.

The split spectra with $H \parallel c$ and the large spectral weight with $H \parallel a$ resemble the $^{75}$As NMR spectra of stripe ordering in the iron pnictides [28]. By applying
The normalized total spectral weight as a function of temperature at high pressures. The data are determined by the RF inductance under the same field. The 1/77T1 for H || a,b (averaged for two frequency peaks below 1/77T1) and H || c, at P = 1.34 and 2.4 GPa. Inset: the anisotropy factor R = (1/77T1/[-m_{Fe}])/(1/77T1/[-m_{Fe}]) as a function of temperature. The dashed and dotted horizontal lines are the theoretical values for the stripe-type (R = 1.5) and for the checkerboard-type (R = 0.5) spin fluctuations.

The disappearance of the paramagnetic peak with H || c indicates that the sample is fully magnetically ordered. The 50% signal loss with H || a,b could be caused by a broad spectrum from distributed magnetic moments, or a very short T2 from phase inhomogeneity. Moreover, we find that the signal loss remains 50% for different pressures and samples, and another plausible explanation is that one of the two domains is out of the NMR window in the stripe phase. In particular, if m_{Fe}c (H^\parallel c) is finite, the signal is lost for H || a but not for H || b. However, further experimental evidences and theoretical understanding are requested to fully settle this scenario.

The above analyses already allow us to rule out other proposed local patterns, such as the checkerboard (\( \pi, \pi \)) spin orders where a zero c-axis internal field is expected on the Se sites (supplemental S4). In fact, it has been suggested theoretically that the lack of magnetic ordering at the ambient pressure is caused by competing interactions, such as strong magnetic frustration from nearest and next nearest neighbor exchange couplings J1 and J2 [16–19]. Our observation under pressure puts strong constrains on these competing theories: naively, our finding of the S-AFM phase suggests a reduced ratio of J1/J2 with pressure. This is also consistent with an ab initio DFT calculation, where the S-AFM state has the lowest energy over other magnetic states under pressure [18].

This high-pressure magnetic order of FeSe naturally resembles that of the iron pnictides [4], and therefore may fit to a unified magnetic phase diagram of the iron-based superconductors generated by the competing exchange interactions. The stripe order with a finite m_{Fe}c clearly indicates a magnetic C4 symmetry breaking because of two choices to select a-axis of the crystals. Furthermore, a strong spin-lattice coupling has to be considered in the magnetic phase transition as shown below. We examined the magnetic transition across T_N from the temperature scan of the 1/77T1T, as shown in Fig. 3(a)-(c) for the high-frequency peak. In fact, the following discussions are valid for the low-frequency peak as well (see Fig. S5). For pressures from 1.34 GPa to 1.86 GPa, the 1/77T1T has a clear divergence at T_N, suggesting a second-order magnetic transition. Surprisingly, the divergence is completely absent at T_N under higher pressures (\( \geq 2 \) GPa), directly evidencing a strong first-order transition with no critical fluctuations. The first-order nature of the transition is further supported by the hysteresis near T_N, with a 1.5 K shift in the temperature dependence of the integrated spectral weight with H || c below 0.1 K/minute (Fig. 3(c)). This strongly implies that the magnetic transition is coupled to a tetragonal-to-orthorhombic structural transition due to the strong spin-lattice coupling [30, 31]. The simultaneous magnetic and structural transition are also supported by the high-pressure XRD data of FeSe [32, 33]. It also suggests that the structural transition is an Ising-nematic transition with a magnetic origin [34, 35].

We are now in a position to discuss the spin fluctuations above T_N from the 1/77T1 data. Fig. 3(d) shows the (1/77T1)_H/[-a,b] and (1/77T1)_H/[-c] below 200 K at P = 1.34 GPa and 2.4 GPa. In the inset, the anisotropy factor R = (1/77T1)_H/[-a,b]/(1/77T1)_H/[-c] at these two pressures are presented, where R \( \approx 1.5 \) holds from 100 K down to 40 K. This value of R is an indication of stripe-type, or (\( \pi, 0 \)), spin fluctuations at temperatures far
FIG. 4: (color online). (a), (b), (c): The spin-lattice relaxation rate divided by temperature \(1/T_1 T\) with \(H \parallel a \& b\) at typical pressures. Only the data measured on the high-frequency peaks are presented below \(T_s\). The \(T_c\) (determined by RF inductance under the same field), the \(T_N\), and the \(T^*\) are marked by the arrows. (d) The plot of \(1/T_1 T\) at 100 K as a function of pressure.

above \(T_N\) (supplemental S4). \(R \approx 1.5\) persists to the highest pressure we measured, and is consistent with the ground state magnetism we presented. The possibility of checkerboard-type spin fluctuations \([21]\) with \(R \approx 0.5\) (see Fig. 3(d) inset) is ruled out at high pressures.

Remarkably, as shown in Fig. 4(a)-(c), the high-temperature \(1/T_1 T\) first decreases upon cooling below 200 K, and then shows an upturn behavior with further decrease of temperature. For each pressure, we define a characteristic temperature \(T^*\) for the onset of the upturn [as shown in Fig. 4(a)-(c)], which indicates enhanced low-energy spin fluctuations. The \(T^*\) barely varies with pressure within our resolution, as shown in the phase diagram (Fig. 3). The error bars of \(T^*\) are taken as distances between two temperatures where \(1/T_1 T\) is enhanced by 10% when cooling/warming away from \(T^*\). Furthermore, the \(1/T_1 T\) at 100 K, as presented in Fig. 4(d), also increases with pressure, consistent with earlier NMR results on polycrystals \([12]\). Therefore, our high-pressure data reconcile FeSe and other iron-based superconductors, where the stripe order and the \((\pi, 0)\) fluctuations tend to be universal, although distinct properties are observed in FeSe at the ambient pressure.

These high-pressure data shed important new light on the driving force of the nematicity. First, in the full range of pressure we have measured, enhanced low-energy stripe-type spin fluctuations exist up to \(T^*\). \(T^*\) only accidentally coincides with \(T_s\) (orbital ordering) at \(P = 0\), but surpasses \(T_s\) largely at high pressures. The emergence of high-temperature stripe-type spin fluctuations is hardly understood within an orbital-driven-nematicity scenario \([26]\), but could be explained as enhanced spin fluctuations above the Ising-nematic transition in a magnetic-driven-nematicity scenario \([30]\). Second, the concomitant structural transition above 2 GPa, manifest by the first-order magnetic phase transition, evidences a strong coupling between nematicity and magnetism under pressure. This scenario implies the same underlying physics governing the coupled magnetic and nematic transitions in some iron pnictides \([27]\), and hence suggests a universal picture of a magnetic-driven nematicity in iron-based superconductors \([8]\). Furthermore, it has been proposed that frustrated magnetic interactions at low pressures may favor other magnetic orders, such as the antiferroquadrupolar order \([10]\) and/or the staggered dimmer/trimmer order \([13,14]\). These exotic magnetic states support the same nematic order, but compete with the stripe-order magnetism. With increasing pressure, these competing orders may be suppressed while the stripe correlations grow strongly, which lead to a non-monotonic change of \(T_s\) \([13,18]\) as we observed.

Finally, we address the implications of our data to superconductivity. Besides the RF inductance measurements (supplemental S3), the onset of superconductivity below 1 GPa is also shown by a kinked feature in the \(1/T_1 T\) upon cooling (Fig. 4(a)), which signals bulk superconductivity. Above 1.5 GPa, while both \(T_c\) and \(T_N\) increase with pressures, the \(1/T_1 T\) drops smoothly and fits to \(1/T_1 T = a + bT^\alpha\) with \(\alpha \approx 2.5\) (presented by the solid lines in Fig. 4(b-c)), which is a typical form by taking into account the contributions from both itinerant electrons and spin waves far below \(T_N\). The absence of a kinked feature across \(T_c\) in \(1/T_1 T\) excludes the microscopic coexistence of superconductivity in our observed stripe phase. However, further investigation is needed to address the exact locations and the properties of the superconducting phase. Since superconductivity does not show up in the observed magnetic regions, we speculate that it exists in small inhomogeneous or short \(T_1\) regions as we described earlier. Nevertheless, the proximity of the superconducting phase to the stripe order and existence of strong \((\pi, 0)\) spin fluctuations in the paramagnetic phase, draw a close relation between superconductivity and the stripe-order magnetism, as seen in iron pnictides. Future studies under higher pressures, when
magnetic ordering is suppressed \cite{15}, may shed further light on the pairing mechanism \cite{28,30}.

In summary, we report direct spectroscopic evidence for the strong suppression of the orbital ordering/structure transition under pressure, and for a stripe-order magnetism above 2 GPa in FeSe. The magnetic transition is identified as a first-order type with a $C_4$ symmetry breaking. These pressure effects put constraints on theories of magnetism in FeSe. Although the $T_S$ is not directly detected by the current NMR data above 2 GPa, electronic nematicity and nematic fluctuations are shown to be closely coupled to the stripe-order magnetism, resulting in a first-order magnetic and structural transition under high pressures and persistent strong ($\pi, 0$) spin fluctuations over a wide range of temperature and pressure. These results suggest a strong coupling among lattice, magnetism, and nematicity, and fully support a magnetic-driven-nematicity scenario. Our high-pressure data also reconcile FeSe with other iron-based superconductors by the stripe-order magnetism, and helps to understand the superconductivity on a universal basis.

We acknowledge encouraging discussions with Zhong-Yi Lu, Dong-Hai Lee, Tao Xiang, Kai Liu, Fa Wang, and Qimiao Si. Work at Renmin University of China is supported by the National Science Foundation of China (NSFC) (Grant Nos. 11222433, 11374361, 11374364 and 11574394), the Ministry of Science and Technology of China (Grant Nos. 2016YFA0300504), and the Fundamental Research Funds for the Central Universities and the Research Funds of Renmin University of China (Grant Nos. 14XNLF08, 15XNLQ07 and 15XNFL06).

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