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Gradual Enhancement of Stripe-Type Antiferromagnetism in Spin Ladder Material BaFe_2S_3 Under Pressure

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The recent discovery of pressure-induced superconductivity in BaFe_2S_3 and BaFe_2Se_3 has generated considerable interest in the 123-type iron chalcogenides. The structure of these materials features iron atoms arranged in quasi-one-dimensional ladders with either stripe-type or block-type antiferromagnetism at ambient pressure, constituting a new parent state for iron-based superconductivity in need of detailed study. Here, we report pressure-dependent neutron diffraction and muon spin relaxation/rotation measurements combined with first-principles calculations to investigate the structural, magnetic, and electronic properties of BaFe_2S_3 under pressure. The experimental results reveal a gradual enhancement of the stripe-type ordering temperature with increasing pressure up to 2.6 GPa and no observable change in the size of the ordered moment. The *ab initio* calculations suggest that the magnetism is highly sensitive to the Fe-S bond lengths and angles, clarifying discrepancies with previously published results. In contrast to our experimental observations, the calculations predict a reduction of the ordered moment with pressure. We suggest that the robustness of the stripe-type antiferromagnetism is due to strong electron correlations not fully considered in the calculations.

Iron-based superconductors (FeSCs) remain an outstanding problem in condensed matter physics, eluding a comprehensive explanation despite more than a decade of intensive research. However, there is widespread agreement that the interrelationships among crystal structure, magnetism, and electronic orders of various types are vital for understanding the origin of superconductivity (SC) in these materials [1–5]. Until recently, the crystal structures of all known FeSCs were characterized by a two-dimensional (2D) square lattice comprised of edge-sharing FeX_4 tetrahedra ($X = \text{Se}, \text{P}, \text{and As}$), with SC typically appearing upon carrier doping, isovalent substitution, or application of pressure. The discoveries of pressure-induced SC in BaFe_2S_3 and BaFe_2Se_3 have introduced a new, quasi-one-dimensional structural template for FeSCs [6–8]. In both systems, the FeX_4 edge-sharing tetrahedra create well separated iron ladders [9], distinct from the planar square lattice of other FeSCs. These compounds therefore represent an important new family of materials that has attracted significant theoretical and experimental attention in the quest to understand iron-based superconductivity [10–27].

BaFe_2S_3 belongs to the orthorhombic space group $Cmcm$ and orders antiferromagnetically below ~ 100 – 120 K in a stripe-type spin configuration [6]. The crystal and magnetic structures are displayed in Fig. 1 (a, b). The antiferromagnetic insulating nature of BaFe_2S_3 at ambient pressure gives way to SC under a pressure of ~ 11 GPa, with a maximum superconducting T_c of ~ 25 K [6, 7]. A recent study of a single-crystal specimen of BaFe_2S_3 reported an additional transition at

0.95 GPa, marked by an abrupt increase of the Néel temperature (T_N) and ordered moment by approximately 50% and 25%, respectively, followed by a rapid suppression of T_N at higher pressures [20]. This was interpreted as a transition to an orbitally selective Mott phase thought to be the true parent state of the SC at higher pressure. To elucidate this reported transition and its relevance to SC, more studies of BaFe_2S_3 under pressure are necessary.

In this work, we investigate the effects of pressure on magnetism in BaFe_2S_3 using neutron powder diffraction (NPD), muon spin relaxation/rotation (μSR), and first-principles calculations. The NPD and μSR results show a gradual increase in T_N with pressure followed by an eventual plateau around the highest pressure measured (2.6 GPa), with no observable change in the magnitude of the ordered moment. This contrasts with the abrupt magnetic enhancement around 1 GPa and subsequent reduction reported earlier [20]. Our density functional theory (DFT) calculations reveal a delicate sensitivity of the magnetism on the Fe-S bond lengths and angles, which may explain the discrepancy with the earlier work. Finally, the DFT calculations predict a monotonic suppression of the magnetism with pressure, in contrast to the initial enhancement observed experimentally. This may indicate that strong electronic correlations not accounted for in the DFT calculations make the stripe-type antiferromagnetism unexpectedly robust against pressure.

BaFe_2S_3 samples were grown by the Bridgman method, forming small, needle-like single crystals a few millimeters in length. The details of the synthesis

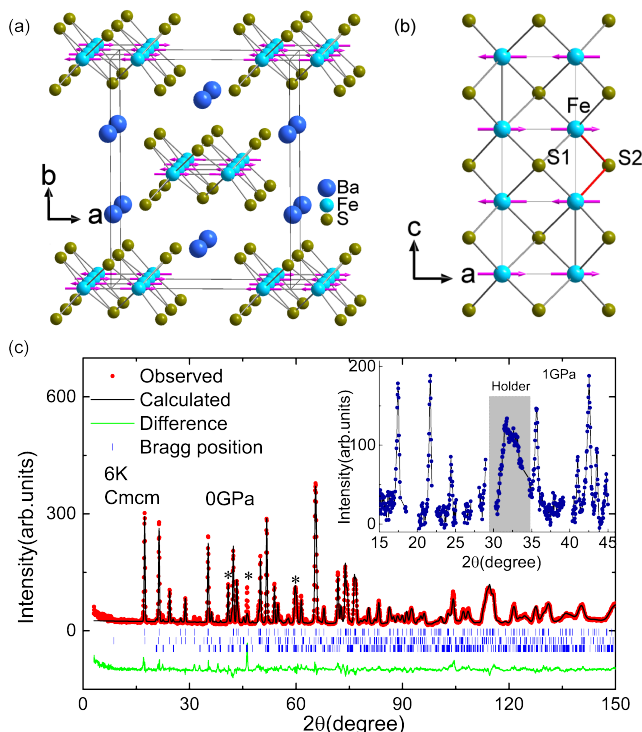


FIG. 1. (Color online) (a) Crystal and magnetic structure of BaFe₂S₃ (Ba = dark blue, Fe = light blue, S = green). The solid lines indicate one unit cell. (b) Isolated view of the one-dimensional, edge-sharing FeS₄ tetrahedra forming the spin ladders. The Fe²⁺ magnetic moments are aligned ferromagnetically along the rungs and antiferromagnetically along the legs. Two distinct S sites exist, labeled S1 (inside the ladder) and S2 (outside). (c) Neutron powder diffraction pattern for BaFe₂S₃ at 6 K and ambient pressure. The pattern was fit with the orthorhombic *Cmcm* model and the stripe-type magnetic phase. Asterisks indicate peaks from the <1% impurity phase Fe₇S₈. Inset: Diffractogram collected at 6 K and 1 GPa. The gray rectangle indicates the signal from the pressure cell.

procedure have been documented previously [24, 28]. Due to the difficulty of co-aligning such crystals, we ground 8 g of single crystals into a powder for subsequent measurement. The NPD experiment was carried out on the High Resolution Powder Diffractometer BT1 at the NIST Center for Neutron Research (NCNR) using a monochromatic beam with $\lambda = 2.0779$ Å. A closed cycle refrigerator was used to control the sample temperature. Measurements were performed both at ambient pressure and in a pressure cell with an applied hydrostatic pressure of 1 GPa. Rietveld refinements of the crystal and magnetic structures were conducted using the FullProf Suite [29]. μ SR measurements at ambient pressure were conducted on the LAMPF instrument at TRIUMF in Vancouver, Canada, and the pressure-dependent experiments were performed on the GPD instrument at the Paul Scherrer Institute in Villigen, Switzerland. A gas-flow cryostat was used for temperature control in both cases. Daphne oil was used

TABLE I. Structural and magnetic parameters of BaFe₂S₃ at ambient pressure and 6 K. The space group is *Cmcm* (No.63) with $a=8.7248$ Å, $b=11.1643(3)$ Å, $c=5.2618(1)$ Å, and the agreement factors are $R_p = 15.6\%$, $wR_p = 17.5\%$, $\chi^2 = 4.5\%$.

| Atom | Site | x | y | z | Occ. | $M(\mu_B)$ |
|------|------|-----------|-----------|---------|-----------|------------|
| Ba | 4c | 0.50000 | 0.1871(5) | 0.25000 | 1.00000 | |
| Fe | 8e | 0.3450(2) | 0.50000 | 0.00000 | 1.053(1) | 1.29(3) |
| S1 | 4c | 0.5000 | 0.609510 | 0.25000 | 1.026(13) | |
| S2 | 8g | 0.2058(6) | 0.3750(6) | 0.25000 | 1.005(16) | |

to transmit hydrostatic pressure, which was calibrated with a superconducting indium plate immersed in the oil with the sample. The uncertainty in the measured pressure was less than 0.1 GPa. The μ SR spectra were analyzed using the least-squares minimization routines in the MusrFit software package [30]. DFT calculations were performed using the Vienna Ab initio Simulation Package (VASP).

We first present the NPD data. Fig. 1(c) displays the diffraction pattern for BaFe₂S₃ at 6 K and ambient pressure. Refinements using the expected orthorhombic structure and stripe-type magnetic order provide a good fit. The refined structural and magnetic parameters at 6 K are summarized in Table 1. Three small Bragg peaks well indexed by Fe₇S₈ were also observed, indicating an impurity level of about 2%. Additional unknown impurities may also be present in small amounts ($\lesssim 1\%$), resulting in somewhat less-than-ideal agreement factors.

The refined ordered moment at 6 K and ambient pressure is $1.29 \pm 0.1 \mu_B$ and lies along the rung direction (*a* axis), as shown in Fig. 1(a). This is slightly larger than the reported values of 1.02 and 1.20 μ_B in Refs. [6, 20]. A portion of the diffraction pattern collected at 6 K and a pressure of 1 GPa is shown in the inset in Fig. 1(c). The data quality is reduced due to the large background from the pressure cell, preventing a satisfactory Rietveld refinement. However, the magnitude of the ordered moment can still be determined through comparison to nuclear Bragg peak intensities, yielding a value of $1.34 \pm 0.1 \mu_B$. Within the experimental uncertainty, then, the magnetic moment does not change from ambient pressure to 1 GPa. We next investigated the ordering temperature by measuring the intensity of the magnetic Bragg peak near $2\theta = 24.4^\circ$ as a function of temperature for both ambient pressure and 1 GPa, shown in Fig. 22(a). The intensity begins to increase below $T_N \approx 100 - 120$ K for both pressures. The dashed line is a guide to the eye, showing an upturn at 120 K. The DC zero-field-cooled magnetic susceptibility measured at ambient pressure with $H = 1000$ Oe, shown along with its temperature derivative in Fig. 2(b), exhibits a broad feature confirming $T_N \approx 100 - 120$ K. This ordering temperature is generally consistent with the earlier reports of $T_N = 105$ K [20] and 119 K [6].

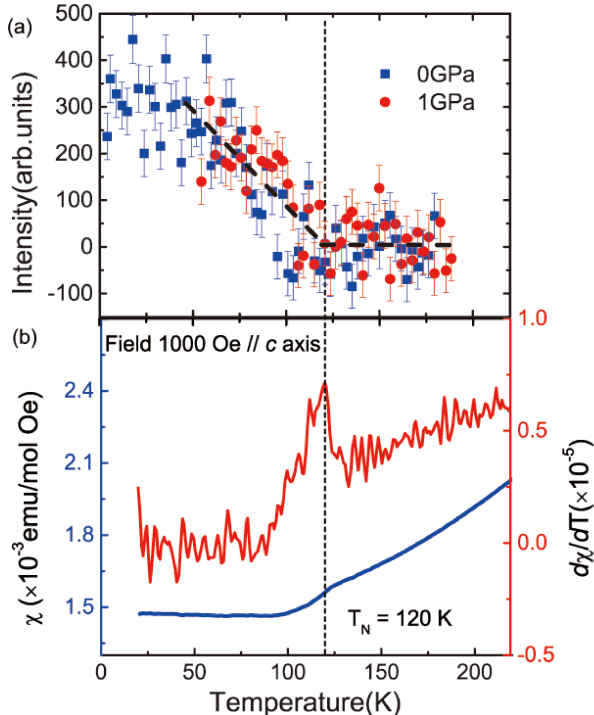


FIG. 2. (Color online) (a) Temperature dependence of the magnetic Bragg peak intensity at $2\theta=24.4^\circ$ in ambient pressure and 1 GPa. No significant change is observed in the ordering temperature. (b) DC magnetic susceptibility of BaFe_2S_3 (blue curve) and its temperature derivative (red curve). The peak in the derivative indicates a magnetic transition at 120 K, in agreement with the neutron data.

The NPD results reported so far indicate that no change to the ordered moment or ordering temperature occurs between ambient pressure and 1 GPa, in contrast to the dramatic enhancement at 0.95 GPa reported previously [20]. To verify our NPD findings, we now turn to the μSR measurements, which we performed on a sample taken from the same powder batch as was used for the NPD experiments. In Fig. 3(a), we display two representative time spectra measured in zero external field (ZF) at 150 K (orange) and 80 K (blue), which are respectively above and below the AF transition. These spectra were collected in the low-background sample environment at TRIUMF. Oscillations of the asymmetry at 80 K originate from the long-range magnetic order, while the featureless spectrum at 150 K reflects the paramagnetic state. The μSR oscillation frequency as a function of dimensionless temperature T/T_N is given by the blue circles in Fig. 3(d), showing a steady increase as the temperature decreases and the ordered moment grows.

In Fig. 3(b), we plot the temperature dependence of the non-oscillating asymmetry component extracted from the ZF spectra at various applied pressures up to 2.6 GPa. The non-oscillating asymmetry is proportional to the paramagnetic volume fraction in the sample, so

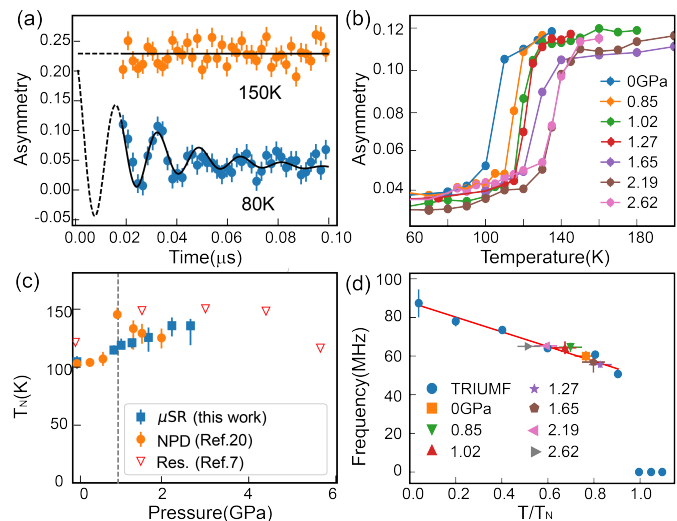


FIG. 3. (Color online). (a) Representative zero-field μSR spectra for BaFe_2S_3 at 150 K (above T_N) and 80 K (below T_N). Filled circles represent data, black curves represent the fits. The dashed black curves represent extrapolations of the fits from the start of the data window at $t = 0.02 \mu\text{s}$ to $t = 0$. (b) Amplitude of the non-oscillating asymmetry component of the ZF μSR spectra as a function of temperature for various applied pressures. (c) T_N as a function of pressure obtained from the present μSR measurements (blue squares) and previously reported neutron diffraction (orange circles; Ref. [20]) and resistivity measurements (red triangles; Ref. [7]). The dashed vertical line represents the critical pressure for the abrupt transition reported in Ref. [20]. (d) Refined ZF oscillation frequencies for the data collected at ambient pressure at TRIUMF (blue circles) and at different applied pressures at PSI (various symbols), plotted versus T/T_N for meaningful comparison.

a bulk magnetic transition is manifest as a significant drop in this asymmetry component as the temperature is lowered. The general trend is a gradual increase in the magnetic transition temperature as the applied pressure is raised from 0 to 2.6 GPa. Interpolating between the data points at each pressure and choosing T_N as the temperature where 50% of the sample is magnetically ordered allows us to quantify T_N as a function of pressure, which we illustrate with the blue squares in Fig. 3(c). At ambient pressure, T_N determined from the μSR data in this way is 105 K, consistent with the susceptibility and NPD results. The upper and lower ends of the error bars in Fig. 3(c) correspond to the temperatures where 20% and 80% of the asymmetry has been lost, respectively, giving a good indication of the temperature range of the bulk transition.

The μSR results clearly show a smooth increase of T_N with pressure, possibly plateauing around 2 GPa. The increase in T_N between ambient pressure and 1 GPa is not large ($\lesssim 10$ K) and the transition is somewhat broad, perhaps explaining why it was not observed by our neutron measurements. Overall, the pressure dependence of T_N determined by μSR is in

good qualitative agreement with the kink in resistivity measurements under pressure reported in Ref. [7], shown as open red triangles in Fig. 3(c). However, these results contrast starkly with the earlier report [20] of the dramatic enhancement at 0.95 GPa (vertical dashed line) and subsequent suppression at higher pressure, shown as orange circles in Fig. 3(c). In Fig. 3(d), we plot the refined ZF oscillation frequencies determined at each pressure. Due to time constraints, high-quality data sufficient for resolution of the oscillations under pressure were obtained only at one temperature point per applied pressure. To make a meaningful comparison among different pressures and the more complete set of oscillation frequencies measured at TRIUMF, the temperatures have been normalized by T_N . Displayed this way, the frequencies all lie along the same line. Since the oscillation frequency is proportional to the ordered moment, these results indicate there is no dramatic pressure dependence of the moment size.

The NPD and μ SR results are therefore consistent, demonstrating that there is only a slow and gradual increase of T_N with pressure up to ~ 2 GPa, without any change in the ordered moment. Compared to the results reported in Ref. [20], our sample exhibits a larger ordered moment at ambient pressure ($1.3 \mu_B$ versus $1.0 \mu_B$) and a markedly different response to pressure (gradual enhancement, not dramatic change). We now explore possible explanations for these discrepancies. First, it is known that the magnetic properties can be influenced strongly by small differences in stoichiometry, which may arise from differing synthetic procedures [15]. This may be due in part to slight modifications of the atomic positions based on the details of the synthesis process. Comparing the refined structural parameters of our sample to those of the sample in Ref. [20], we found that the bond lengths and angles between the Fe and S2 atoms show obvious differences between the two samples: the Fe-S2 bond length in the current work is nearly 0.09 \AA shorter than previously reported (2.2700 versus 2.3587 \AA), and the present Fe-S2-Fe angle is nearly 3° larger (70.829° versus 67.985°). The Fe-S2 geometry may therefore influence the magnetism, likely by affecting the magnetic exchange interaction and orbital hybridization between Fe and S atoms. This is supported by our DFT calculations, which show an enhancement of the ordered moment when the Fe-S2 bond length shortens, consistent with the experimental observation.

We now report our DFT calculations investigating the magnetism under pressure. In Fig. 4(a)-(d), we display the orbitally resolved density of states (DOS) of the Fe $3d$ orbitals for zero pressure and 8 GPa. The various colors indicate different Fe $3d$ orbitals and the dashed lines represent the Fermi energy. The calculations correctly predict zero DOS at the Fermi energy at 0 GPa, corresponding to an insulating state, whereas metallic behavior with a finite DOS at the Fermi energy is ex-

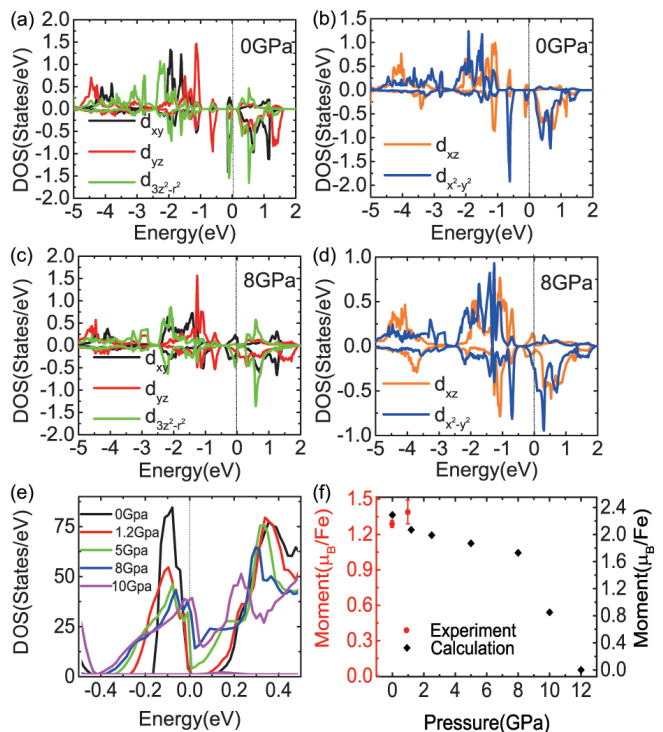


FIG. 4. (Color online) (a)-(d) DFT calculations of the density of states (DOS) of Fe $3d$ orbitals for $P = 0$ GPa and 8 GPa. (e) Pressure dependence of the total DOS. (f) Calculated (right axis) and observed (left axis) ordered magnetic moments as a function of pressure. Note the difference in scale on the vertical axis.

pected for 8 GPa. We found a large difference in the DOS contributed by up and down spins in each orbital (data not shown), implying that all of the Fe $3d$ orbitals contribute to the magnetism. The calculated contributions of each orbital to the combined spin moment of the two Fe atoms per formula unit are 0.40 (d_{xy}), 0.44 (d_{yz}), 0.36 ($d_{3z^2-r^2}$), 0.52 (d_{xz}), and 0.51 ($d_{x^2-y^2}$) at 0 GPa and 0.30 (d_{xy}), 0.32 (d_{yz}), 0.28 ($d_{3z^2-r^2}$), 0.38 (d_{xz}), and 0.42 ($d_{x^2-y^2}$) at 8 GPa. We see that $d_{x^2-y^2}$ and d_{xz} contribute the most to the calculated spin moment, and the calculated contribution from all orbitals decreases as pressure increases. Fig. 4(e) shows the total DOS under different pressures for the stripe AF phase, showing an insulator-metal transition around 5 GPa.

The calculated ordered moment on the iron atoms at different pressures is shown as black diamonds (right axis) in Fig. 4(f), together with the measured ordered moment from the neutron experiments at ambient pressure and 1 GPa shown as red circles (left axis). Note the difference in scale on the two vertical axes. The calculations predict a steady decrease of the ordered moment with pressure, consistent with earlier first-principles studies [19, 25], with a complete suppression of the ordered moment at 12 GPa. This is near the pressure at which SC appears. The calculated magnitude of the ordered moment at 0 GPa is nearly a factor of 2

larger than the experimental result, which likely results largely from strong zero-point fluctuations in this quasi-one-dimensional system. Another possible contributing factor to the reduced moment is the coexistence of localized iron spins and itinerant electrons [17]. Additionally, the predicted decrease of the ordered moment with increasing pressure is inconsistent with the stability of the ordered moment revealed by NPD and μ SR experiments. Given that moderately strong electron correlations are expected to exist in BaFe_2S_3 [14], we suggest correlation effects not properly accounted for in the DFT calculations make the stripe-type magnetism more robust than would otherwise be expected.

In summary, we have studied the magnetic and electronic properties of BaFe_2S_3 through neutron powder diffraction, μ SR, and DFT calculations. Our results confirm the stripe-type antiferromagnetic structure, but with a slightly larger ordered moment than reported in Ref. [20]. This appears to be due to a difference between the bond lengths and angles of Fe and S2. The experimental results show a gradual increase in T_N up to 2.6 GPa with no change in the ordered moment, in contrast to the dramatic increase in T_N and the ordered moment reported previously at 0.95 GPa. Given this lack of consistency, interpretations of the previous results in terms of orbitally selective Mott physics should be taken with caution. Our DFT calculations correctly yield an insulating ground state at ambient pressure, with an insulator-metal transition predicted around 5 GPa. The calculations predict the ordered moment to decrease to zero around 12 GPa, while the observed robustness of the moment and ordering temperature at pressures up to 2.6 GPa suggests that electron correlation effects fortify the stripe-type antiferromagnetism.

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