Pressure-induced lattice collapse in the tetragonal phase of single-crystalline $Fe_{1.05}Te$

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Pressure-induced lattice collapse was discovered in tetragonal (T) phase of single crystal $Fe_{1.05}Te$ at room temperature through x-ray and neutron-diffraction measurements. A remarkable compression along the c axis ($\sim$5%) was observed upon increasing pressure from the ambient condition to 4 GPa. Indexed results demonstrate that the crystallographic structure remains unchanged after the collapse, revealing that the collapse does not break symmetry of crystal structure. The Fe-spin state change was proposed to account for the lattice collapse. The equations of state for the T phase and pressure-induced collapsed T phase were determined from the diffraction measurements.

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I. INTRODUCTION

The discovery of the superconductivity with a critical transition temperature (T_c) of 26 K in $LaFeAsO_{1-x}F_x$ has opened research activity in a family of superconductors.[1] Since then, four types of superconductors, ReFeAsOF (Re =Ce, Pr, Nd, Sm, etc.), AFe_2As_2 (A=Ba, Sr, Ca), A′FeAs (A′=Li, Na), and FeSe(Te), have been found.[2–4] The T_c is enhanced up to 55 K either by chemical substitution Re for La or by introducing oxygen vacancies. This type of superconductors, with such high T_c's but without the copper oxide plane, is likely to have a different mechanism from cuprates. It is believed that the superconductivity in ReFeAsOF and AFe_2As_2 is related to the spin-density-wave (SDW) anomaly in the Fe-As layer.[5] Application of high pressure or chemical doping suppresses the SDW order and drives the superconducting transition.[6,7] Among those superconductors, the binary compound of α-FeSe is a safe system due to the absence of the toxic arsenic. At ambient pressure, FeSe which has a T_c of 8 K has an PbO-type tetragonal structure, with FeSe layers stacked along the c axis.[4] Partial substitution of Te for Se enhances the T_c to 10 K.[8] The T_c is further increased by application of pressure reaching 27 K at 1.48 GPa (Ref. 9) and 37 K at 7–8 GPa.[10,11]

The compound α-FeTe has structure similar to tetragonal α-FeSe at ambient pressure. Neutron-scattering experiments revealed that an antiferromagnetic (AF) transition and structural transition in α-FeTe take place at the same temperature ($\sim$65 K).[12,13] At ambient pressure, α-FeTe has a tetragonal phase at room temperature. This phase transforms to an orthorhombic structure at temperature below 65 K, different from α-FeSe in which the structure transforms from a tetragonal phase to a triclinic phase at temperature below 105 K.[4] Theoretical calculations for the FeTe and FeSe compounds[14] indicated that α-FeTe adopts multiple Femi surface similar to $ReFeAsO_{1-x}F_x$, with hole and electron pockets at the zone center and corners, respectively. An AF instability was found in these two compounds at ground state. However, the strength of instability in α-FeTe is stronger than that in α-FeSe.[14] This implies that α-FeTe should have a higher T_c than α-FeSe after the suppression of the AF transition. Experimental studies showed that the AF transition is tightly correlated with structural properties.[15]

Pressure can reduce the interatomic distances and give rise to distortion of Bragg planes. The AF transition may be suppressed when increasing pressure in the vicinity where superconductivity appears. Superconductivity has been found in nonsuperconducting LaFeAsO and $BaFe_2As_2$ compounds under pressure.[6,16,17] The effect of pressure on structural and magnetic transitions as well as the potential for pressure-induced superconductivity in FeTe is of interest. The structure determinations are step for better understanding the physical properties. Here, we report the experimental investigations of the high-pressure structure of $Fe_{1.05}Te$ by combining synchrotron x-ray and neutron-diffraction techniques. We provide direct evidence for pressure-induced lattice collapse at 4 GPa at which a "transition" from the tetragonal (T) to the collapsed tetragonal (cT) phase also takes place. The Fe-spin state change is proposed to account for the lattice collapse. The equations of state for the T phase and pressure-induced collapsed T phase were determined.

II. EXPERIMENTAL DETAILS

The sample was synthesized by solid state reaction at ambient pressure, as described previously.[18] Phase purity was assessed with powder x-ray diffraction to be a single phase with tetragonal symmetry, as shown in Fig. 1. Temperature dependence of resistance (R) and magnetization (M) obtained at ambient pressure exhibited that R-T and M-T curves of the sample have anomalies at the same temperature near 65 K, as shown in Figs. 2(a) and 2(b). The abrupt de-

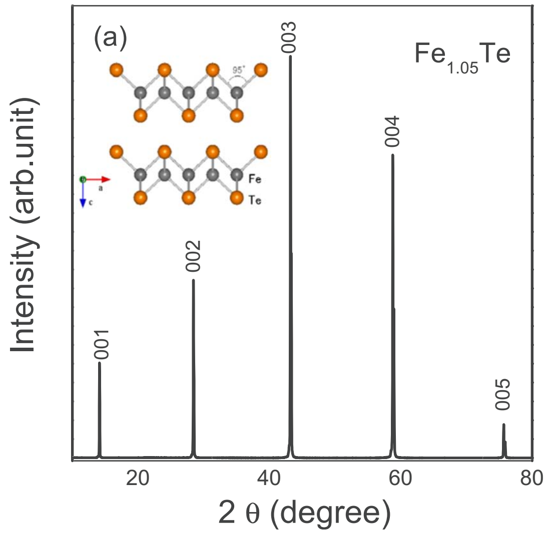

FIG. 1. (Color online) X-ray diffraction pattern of a single crystal $Fe_{1.05}Te$ obtained at ambient pressure and room temperature with Cu K_α radiation.

crease in resistance and magnetization below 65 K is related to the first-ordered tetragonal-monoclinic phase transition, consistent with our previous experimental results.[18]

Angle-dispersive x-ray diffraction (XRD) measurements were carried out at the Beijing Synchrotron Radiation Facility (BSRF). A monochromatic x-ray beam with a wavelength of 0.6199 Å was used. The XRD images were collected using a charge-coupled device (CCD) detector, and the XRD geometry was calibrated with CeO_2. High pressure was created by using a pair of diamond anvils with low birefringence. Diamond anvils used were cut with a 300 μm culet. A spring steel gasket with a 100 μm hole in diameter and 50 μm thickness was adopted. The single crystal sample was loaded into the gasket hole with silicon oil to maintain the sample in a hydrostatic pressure environment. Pressure was determined by ruby fluorescence at room temperature.[19]

Time-of-flight neutron-diffraction experiments were performed at the SNAP (spallation neutrons and pressure)

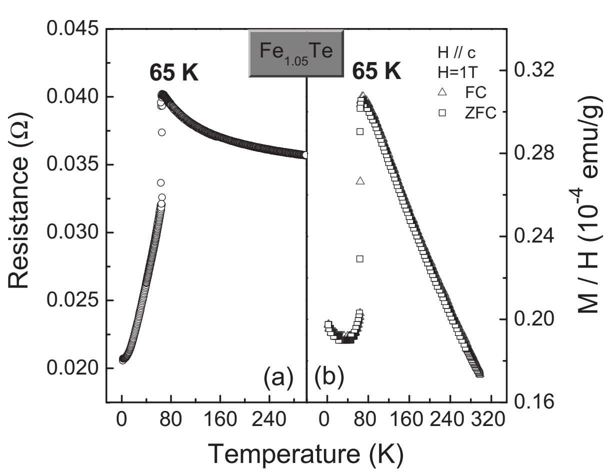

FIG. 2. (a) Resistance and (b) magnetization as a function of temperature of the sample at ambient pressure, showing tetragonal-to-monoclinic phase transition at the same temperature near 65 K.

beamline of the spallation neutron source at Oak Ridge National Laboratory. The powder sample used in the neutron diffraction experiment was ground from the same single-crystalline sample was used for the other measurements. Pressure was generated by boron nitride (BN) toroidal anvils in a Paris-Edinburgh high-pressure cell. The wide opening of this kind of cell allows the angular coverage of 38–142° horizontal and ±34° vertical in the configuration with the distance from sample to detector of 50 cm. The neutron-diffraction data were time focused from three detector modules spanning 45°, with its central module at 90° from the incident beam. A collimation with a 2 mm hole in diameter was used to narrow the beam to the sample size and effectively reduce the background. The wavelength range of the neutrons was in the range from 0.5 Å to 3.7 Å. The sample was contained in a chamber of approximately 5 mm in diameter bored in a TiZr gasket filled with a 4:1 mixture of deuterated methanol and ethanol. The methanol and ethanol mixture can remain fluid up to 10 GPa at room temperature, thus offering quasihydrostatic pressure conditions.

III. RESULTS AND DISCUSSIONS

Figure 3(a) shows the x-ray diffraction patterns of $Fe_{1.05}Te$ at ambient and high pressures at room temperature. It is seen that only the (003) reflection of the single-crystalline sample can be detected over the angular range available. No peaks were found in the diffraction measurements with increasing pressure up to 11.5 GPa. This indicates that no phase transition occurs over the pressure range studied. However, distinct shift of peak (003) was observed upon increasing pressure to 4 GPa at which the lattice parameter c decreases about 0.3 Å, signaling pressure-induced lattice distortion in this material.

High-pressure neutron diffraction (ND) was measured on polycrystalline $Fe_{1.05}Te$ sample in order to examine the observed lattice distortion. Figure 3(b) shows the ND patterns obtained at various pressures. All peaks collected at 0.7 GPa can be indexed well as a tetragonal structure. No peak was found under pressure up to 7 GPa, entirely consistent with the high-pressure XRD results. The computed lattice parameters c and a are compared at different pressures, as shown in Fig. 4. The two sets of the c values derived from ND and XRD measurements are in good agreement. A remarkable reduction in c axis was observed under applied pressure, as seen in Fig. 4(a). The c value is reduced about 5% at ~4 GPa. With further increasing pressure, the c value slowly decreases. The striking reduction in c is interpreted as a lattice collapse in the T phase of $Fe_{1.05}Te$. To distinguish pressure-induced collapsed T phase from the "normal" T phase, we call the high-pressure form as cT. Figure 4(b) shows the pressure dependence of the lattice parameter a. Unlike the c axis, the a value exhibits a monotonic decrease with increasing pressure. The different behaviors along the a and c axis indicate that the pressure-induced lattice collapse only occurs along the c direction.

Figure 5 shows the unit cell volumes (V) as a function of pressure (P) in the T and cT phase of $Fe_{1.05}Te$. No visible discontinuity was found in the P-V curve, further supporting

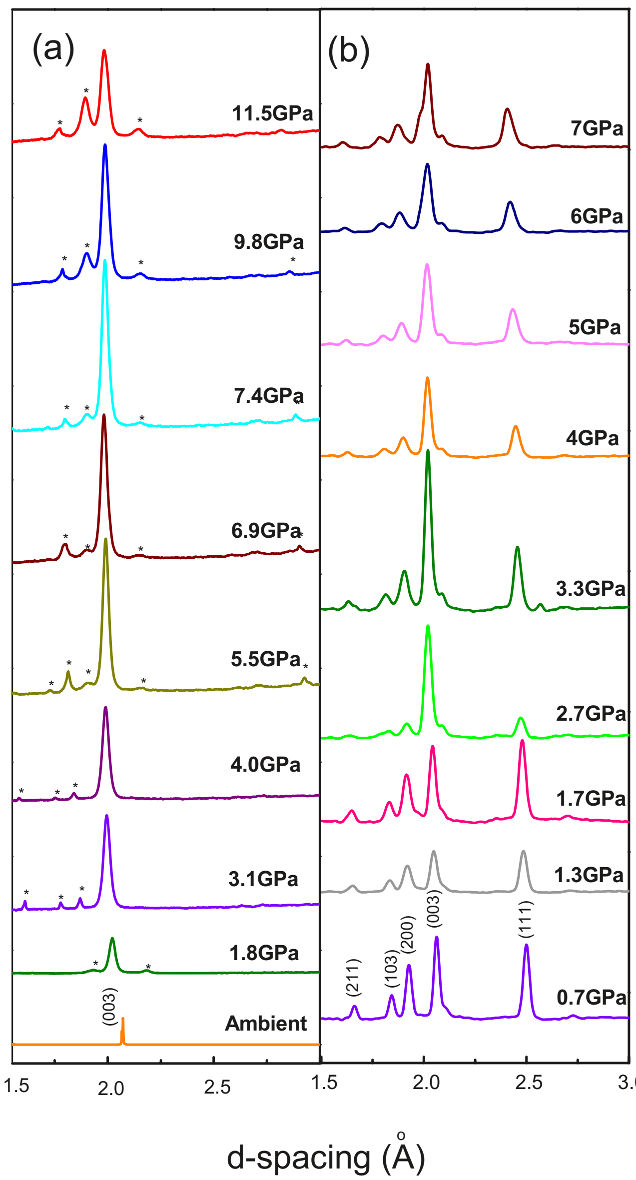

FIG. 3. (Color online) (a) Representative high-pressure XRD patterns of $Fe_{1.05}Te$ obtained with a monochromatic beam ($\lambda = 0.6199$ Å) at 300 K; stars* indicate diffraction peaks from the metal gasket; labeled peak is from the sample. (b) High-pressure ND patterns of the polycrystalline sample at different pressures.

that no phase transition takes place when the lattice collapses. The data were fitted by the third-order Birch-Murnaghan equation of state:[20]

$$P = \frac{3}{2}B_0[(V/V_0)^{-7/3} - (V/V_0)^{-5/3}]\left\{1 + \frac{3}{4}(B_0' - 4)[(V/V_0)^{-2/3} - 1]\right\}$$

where B_0 is the isothermal bulk modulus at zero pressure, B_0' is the pressure derivative of B_0 evaluated at zero pressure, and V/V_0 is the ratio of high-pressure volume and zero-pressure volume of the sample. The resulting parameters are listed in Table I. We obtained $B_0 = 31.3 \pm 1.45$ GPa and $B_0' = 6.6 \pm 1.2$ for the T phase, and $B_0 = 86.7 \pm 6.6$ GPa and $V_0 = 88.7 \pm 0.4$ $(Å)^3$ for the cT phase when B_0' was fixed at 4. These results provide crucial information for further theoretical and experimental investigations of the high-pressure behavior of FeTe parent compound.

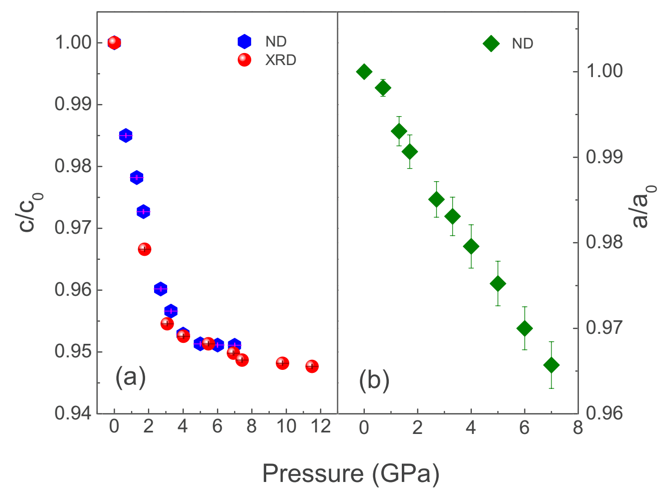

FIG. 4. (Color online) Pressure dependence of the normalized lattice parameters c/c_0 (a) and a/a_0 (b), showing a large lattice distortion along the c axis in the tetragonal phase.

The lattice collapse in tetragonal phase was also found in $CaFe_2As_2$ compound at 2.5 GPa where the c parameter is reduced about ~ 1 Å.[21,22] First-principle study[23] and high-pressure inelastic neutron-scattering results[24] indicated that this strange behavior is caused by the strong suppression of Fe-spin state. As a result, the Fe-As bonding reduces and the corresponding As-As bonding enhances. These results indicated that the spin state of iron is a key factor that controls the lattice distortion in $CaFe_2As_2$ system. Pressure-induced lattice collapse in α-FeTe should have the similar origin to

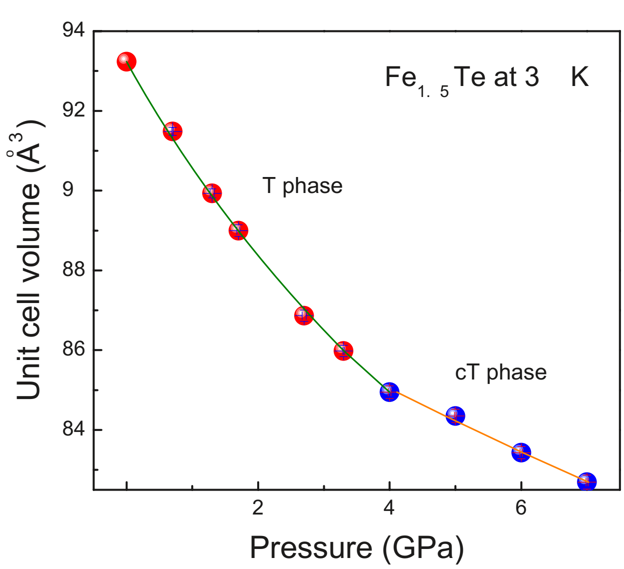

FIG. 5. (Color online) Unit cell volume of the tetragonal phase and collapsed tetragonal phase as a function of pressure; solid circles, experimental data; curves, third-order Birch-Murnaghan equation of state fit to the data obtained from ND measurements.

TABLE I. Bulk modulus B_0, volume at ambient pressure V_0 and pressure derivative B_0' of tetragonal phase T and collapsed tetragonal phase cT in $Fe_{1.05}Te$ compound.

P (GPa)	Phase	B_0	V_0 (Å^3)	B_0'
0–4	T	31.3 ± 1.45	93.2 ± 0.1	6.6 ± 1.2
4–7	cT	86.7 ± 6.6	88.7 ± 0.4	4 (fixed)

$CaFe_2As_2$. Pressure may change Fe-spin state which leads to the reduction in the Fe-Te bonding. Compared to $CaFe_2As_2$, the contraction along the *c* direction of FeTe compound should be small because the iron radii of Te is larger than that of As. If that would be a case, the lattice collapse in the FeTe compound should be slightly weaker than that in $CaFe_2As_2$. The Fe-spin state change must play an important role in the lattice collapse of the FeTe compound.

IV. CONCLUSION

We have investigated high-pressure behaviors of single crystal and polycrystalline $Fe_{1.05}Te$ compound through the measurements of x-ray and neutron diffractions at 300 K. Pressure-induced lattice collapse was found around 4 GPa at which the T phase "transforms" to the cT phase. The equations of state for the T and cT phase were determined from x-ray and neutron-diffraction measurements. These results are expected to simulate further theoretical calculations on the electronic structure of this material at high pressure.

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