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High-field non-reversible moment reorientation in the antiferromagnet $Fe_{1.1}Te$

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Magnetization measurements have been performed on single-crystalline $Fe_{1.1}Te$ in pulsed magnetic fields $\mathbf{H} \perp \mathbf{c}$ up to 53 T and temperatures from 4.2 to 65 K. At $T = 4.2$ K, a non-reversible reorientation of the antiferromagnetic moments is observed at $\mu_0 H_R = 48$ T as the pulsed field is on the rise. No anomaly is observed at H_R during the fall of the field and, as long as the temperature is unchanged, during both rises and falls of additional field pulses. The transition at H_R is reactivated if the sample is warmed up above the Néel temperature $T_N \simeq 60$ K and cooled down again. The magnetic field-temperature phase diagram of $Fe_{1.1}Te$ in $\mathbf{H} \perp \mathbf{c}$ is also investigated. We present the temperature dependence of H_R, as well as that of the antiferromagnetic-to-paramagnetic borderline H_c in temperatures above 40 K.

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Since the discovery of the new class of iron-based superconductors in 2008 [1], the superconducting iron chalcogenides $Fe_{1+x}Te_{1-y}Se_y$ [2] were thoroughly studied because of their simple structure and of the absence of arsenic [3–5]. At ambient pressure, the maximal superconducting temperature of 14 K of this system, observed in $FeTe_{0.5}Se_{0.5}$ [6], is much smaller than that of other iron-based superconductors. However, it can be raised under pressure to a significantly higher value, i.e., up to 37 K in $Fe_{1.01}Se$ [7]. Similarly to many heavy-fermion superconductors [8–11], Fe-based superconductors are in the proximity of a quantum paramagnetic-to-antiferromagnetic instability and magnetism is suspected to play a central role for the formation of the superconducting Cooper pairs [12]. Characterizing the magnetic properties of the antiferromagnetic parents of $Fe_{1+x}Te_{1-y}Se_y$ is thus a key to understand the interplay between magnetism and superconductivity in these systems. In the non-superconducting iron-tellurides $Fe_{1+x}Te$, commensurate bicollinear antiferromagnetism with the wavevector $\mathbf{k} = (\frac{1}{2}\ 0\ \frac{1}{2})$ - made of pairs of chains parallel to $\mathbf{b}$ and ferromagnetically-coupled along $\mathbf{a}$ - develops for $x \leq 0.11$ within a first-order transition at $T_N \simeq 60 - 70$ K [5, 13–16]. This magnetic transition is accompanied by a change of the crystalline structure, which is tetragonal ($P4/nmm$, ♯129) above T_N and monoclinic ($P2_1/m$, ♯11) below T_N. While numerous high-magnetic-field studies have been made on Fe-based superconductors to determine their critical field and its temperature dependence (see Ref. [17–21] for $Fe_{1+x}Te_{1-y}Se_y$), only few have been done so far on their antiferromagnetic parents [22–24]. Indeed, in Fe-based superconductors the critical fields $H_{c,2}$ needed to break superconductivity are large, but accessible using non-destructive magnets; in their antiferromagnetic parents, huge magnetic fields are required to break the antiferromagnetic order and drive the sample into a polarized paramagnetic state. In $EuFe_2As_2$, the field-induced antiferromagnetic-to-paramagnetic borderline has been followed only at temperatures just below the Néel temperature $T_N^{Fe} \simeq 190$ K (associated to the Fe-ions moments), due to a huge critical field estimated well above 500 T at low-temperature [22]. In SmFeAsO, despite the smallness of the Néel temperature $T_N \simeq 5$ K a critical field as high as 40 T is associated to the destabilization of antiferromagnetism at sub-kelvin temperatures [23]. Recently, the antiferromagnetic-to-paramagnetic borderline of $Fe_{1+x}Te_{1-y}S_y$ antiferromagnets, for which $45 \leq T_N \leq 70$ K, has been followed down to temperatures of 20 K in fields up to 60 T [24].

Here we investigate by magnetization measurements the high magnetic field-temperature phase diagram of the iron-telluride $Fe_{1.1}Te$ in fields $\mu_0\mathbf{H} \perp \mathbf{c}$ up to 53 T and temperatures from 4.2 to 65 K. A non-reversible reorientation of the antiferromagnetic moments is found to induce a step-like anomaly in the magnetization at $\mu_0 H_R = 48$ T during the rise of the pulsed field, but not during its fall. We also report the observation of the antiferromagnetic-to-paramagnetic borderline field H_c in temperatures just below T_N. From the temperature dependence of H_R and H_c, the magnetic field-temperature phase diagram of $Fe_{1.1}Te$ with $\mathbf{H} \perp \mathbf{c}$ is extracted.

The single crystals of $Fe_{1.1}Te$ studied here have been grown by a modified Bridgman method, starting from a nominal cation ration of 1.1:1.0, and all belong to the same batch (cf. Ref. [25] for details on the growth of compounds with slightly different concentrations). Their

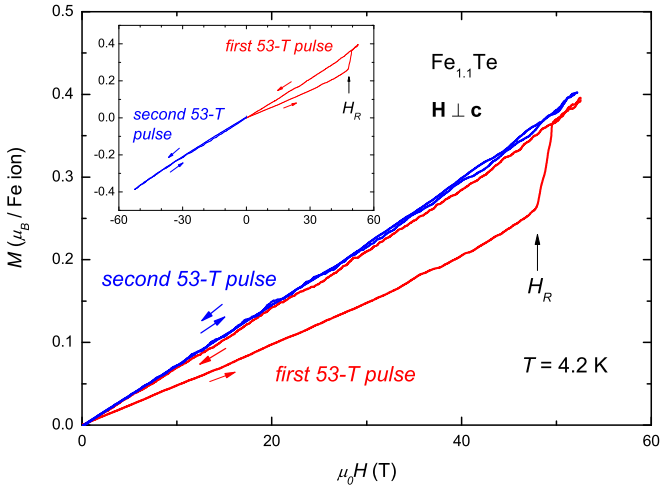

FIG. 1: (color online) Magnetization versus magnetic field of $Fe_{1.1}Te$ at $T = 4.2$ K for $\mathbf{H} \perp \mathbf{c}$, from two successive pulses of magnetic field up to 53 T. An irreversible behavior occurs during the first (zero-field-cooled) pulse, while a reversible behavior is observed during the second pulse (the temperature of 4.2 K was kept constant between the two pulses). The inset shows that a reversible behavior is also observed in a second pulse having an opposite polarity than that of the first pulse.

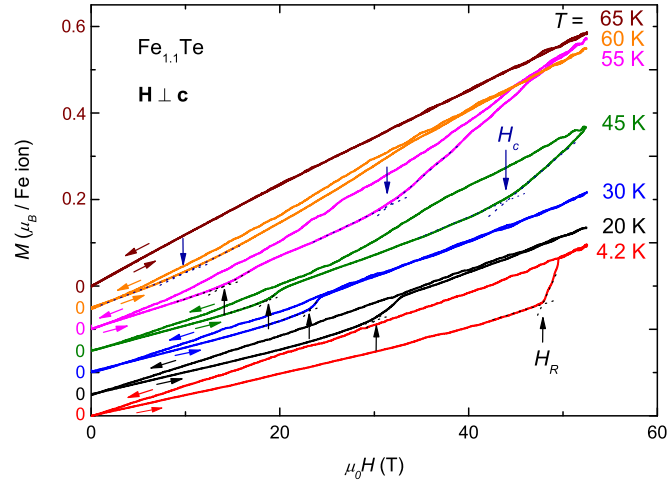

FIG. 2: (color online) Magnetization versus magnetic field of $Fe_{1.1}Te$ for $\mathbf{H} \perp \mathbf{c}$, at temperatures T from 4.2 to 65 K. All curves were measured during zero-field-cooled pulses of magnetic field, i.e., the temperature was raised above T_N (up to 80 K) and then decreased to its target value before each pulse. For clarity, the origin of each curve has been shifted vertically.

composition has been determined by chemical analysis using energy-dispersive X-ray spectroscopy, performed on two different samples, and by refinement of X-ray single-crystal diffraction data collected at 180 K from a third sample, using an Oxford X-calibur charge-coupled device diffractometer equipped with a cryojet cooler device from Oxford Instruments and a graphite-monochromatized Mo-K_α radiation source ($\lambda = 0.71073$ Å). This last sample was also characterized by X-ray scattering with an incident energy of 15 keV ($\lambda = 0.4$ Å) on the ID06 beamline at the ESRF (Grenoble, France). The evolution of the $(H00)$ and $(H0H)$ Bragg reflections versus temperature showed a crystalline transition from tetragonal ($P4/nmm$) to monoclinic ($P2_1/m$) symmetry space groups at the temperature $T \simeq 60$ K $\simeq T_N$. Additionally, a splitting of both sets of reflections was observed below T_N indicating the coexistence of two crystallographic domains. Our refined lattice parameters are in good agreement with those reported in the literature [15]: $a = b = 3.809(2)$ Å and $c = 6.235(5)$ Å in the tetragonal phase, and $a = 3.833(1)$ Å, $b = 3.783(1)$ Å and $c = 6.262(5)$ Å ($\beta = 89.28$ °) in the monoclinic phase. The magnetic susceptibility was measured for magnetic fields $\mu_0\mathbf{H} \perp \mathbf{c}$ of 1 T and $\mu_0\mathbf{H} \parallel \mathbf{c}$ of 0.5 T using a commercial Magnetic Properties Measurement System from Quantum Design. High-field magnetization measurements have been performed for $\mathbf{H} \perp \mathbf{c}$, at temperatures from 4.2 to 65 K, using the compensated-coil technique in a standard 10-mm bore 60-T magnet (30-ms rise, 120-ms fall) at the pulsed-field facility of the Laboratoire National des Champs Magnétiques Intenses of Toulouse.

Fig. 1 shows the magnetization versus magnetic field $M(H)$ of $Fe_{1.1}Te$ measured at the temperature $T = 4.2$ K, i.e., well below the Néel temperature $T_N \simeq 60$ K of the system, in pulsed magnetic fields up to 53 T. In red is shown the magnetization measured during a first pulse of field applied after a zero-field cooling of the sample. A clear non-reversible behavior is observed. During the rise of the pulse, $M(H)$ is linear below 40 T and increases suddenly by a step-like variation $\Delta M \simeq 0.1$ μ_B/Fe-ion at $\mu_0 H_R \simeq 48$ T (defined at the onset of the step), before becoming linear again, with a bigger slope, above 50 T. During the fall of the field, no anomaly is observed in $M(H)$, which is linear with the same slope as observed in rising field above H_R. The magnetization measured at the same temperature of 4.2 K during a second pulse of field, again up to 53 T, is shown in blue in Fig. 1. The temperature has been kept constant between the two pulses. During both the rise and the fall of this second pulse, a reversible linear behavior of $M(H)$ is observed and follows exactly the magnetization measured during the fall of the first pulse. As shown in the Inset of Fig. 1, the polarity of the field direction does no matter, since a second pulse of opposite polarity than that of the first pulse also leads to a reversible linear magnetization. At 50 T, the magnetization reaches $\simeq 0.4$ μ_B/ Fe-ion, which is still well below the value of the ordered antiferromagnetic moment $m_{AF} = 1$ μ_B/ Fe-ion reported for $Fe_{1.1}Te$ [26] (and much smaller than the antiferromagnetic moment $\simeq 2$ μ_B/ Fe-ion reported for lower Fe-contents [27–29]).

Fig. 2 shows the magnetization of $Fe_{1.1}Te$ at various temperatures $4.2 \leq T \leq 65$ K. For each of these measurements, the temperature has been systematically raised up

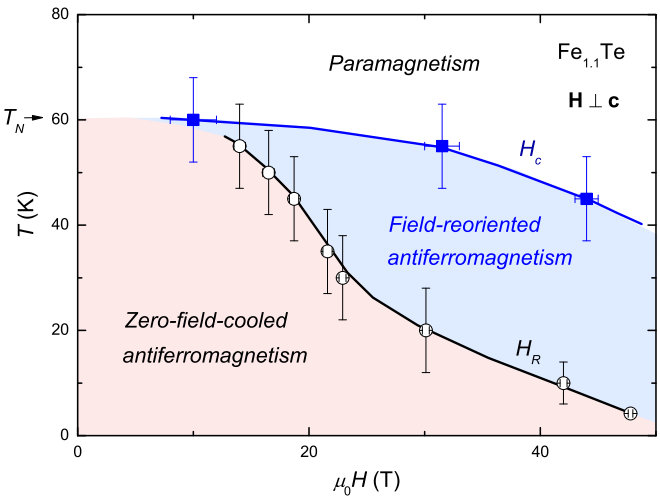

FIG. 3: (color online) Magnetic field - temperature phase diagram of $Fe_{1.1}Te$ extracted in rising fields $\mathbf{H} \perp \mathbf{c}$ (zero-field-cooling condition). The lines are guides to the eyes.

to 80 K, i.e., well above $T_N \simeq 60$ K, and then decreased before applying the field. All of these zero-field-cooled curves measured for $T < T_N$ show a strong non-reversible behavior. The moment-reorientation field H_R decreases significantly with increasing temperature, and it vanishes at $T = 60$ K $\simeq T_N$. At $T = 45$ K, additional strongly hysteretic increase in the slope of $M(H)$ is observed at $\mu_0 H_c \simeq 44$ T in rising field and $\mu_0 H_c \simeq 30$ T in falling field (see Fig. 2). For $45 < T \leq 60$ K, H_c decreases rapidly with increasing T and cannot be defined anymore at 65 K. This anomaly marks the destabilization of antiferromagnetism, the moments being polarized paramagnetically above H_c. Fig. 3 presents the magnetic field - temperature phase diagram of $Fe_{1.1}Te$ extracted in rising fields $\mathbf{H} \perp \mathbf{c}$ under a zero-field-cooled condition, in the extended magnetic field and temperature ranges [0-50 T] and [0-80 K], respectively. Whereas the moment reorientation field $\mu_0 H_R$ reaches approximately 50 T at low temperature, the critical antiferromagnetic field $\mu_0 H_c$ is only observed down to 45 K. A rough extrapolation of the H_c versus T line indicates that H_c is expected to reach approximately 80-100 T at low-temperature. Field-induced antiferromagnetic-to-paramagnetic borderlines similar to that found here at H_c have already been reported for $EuFe_2As_2$ [22], SmFeAsO [23], and $Fe_{1+x}Te_{1-y}S_y$ [24] antiferromagnets. Similarly to the magnetoresistivity measurements performed on $Fe_{1+x}Te_{1-y}S_y$ in Ref. [24], and which were related to magneto-elastic effects, our magnetization measurements show a strong hysteresis at the critical field H_c, possibly related to magneto-elastic effects too. The temperature dependence of the magnetic susceptibility extracted from the initial slope, as it fits to experimental data in the range [0-10 T], of our $M(H)$ data for $\mathbf{H} \perp \mathbf{c}$ is shown in Fig. 4 (a). In this graph, the black full squares correspond to data taken in the rise of the pulse, i.e., in a zero-field-cooled condition, and the red open circles correspond to data taken in the fall of the pulse. For comparison, Fig. 4 (b) shows the zero-field-cooled susceptibility extracted in steady fields $\mu_0\mathbf{H} \perp \mathbf{c}$ of 1 T (black line) and $\mu_0\mathbf{H} \parallel \mathbf{c}$ of 0.5 T (red line). Both susceptibilities extracted in the rise of pulsed fields $\mathbf{H} \perp \mathbf{c}$ and measured in a steady field $\mathbf{H} \perp \mathbf{c}$ correspond to a zero-field-cooled condition and have similar temperature dependences. Interestingly, the susceptibility extracted during the fall of pulsed fields $\mathbf{H} \perp \mathbf{c}$ behaves similarly to that measured in a steady field $\mathbf{H} \parallel \mathbf{c}$.

A challenge is now to describe microscopically the magnetic properties of $Fe_{1.1}Te$ under a high field. Different kinds of scenarios can be considered. For example, an

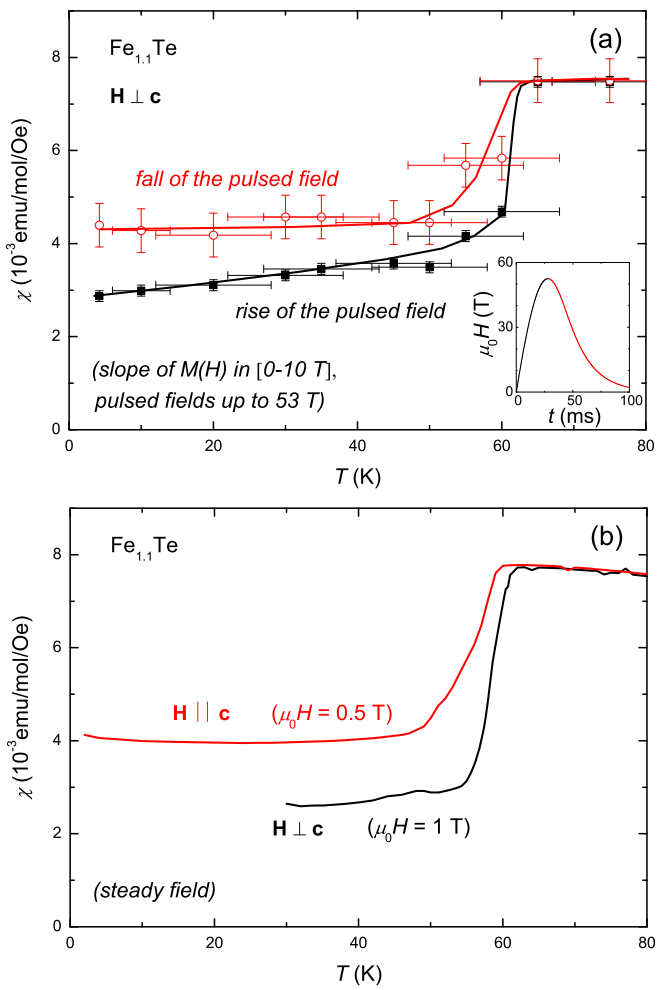

FIG. 4: (color online) (a) Magnetic susceptibility versus temperature of $Fe_{1.1}Te$ extracted from the slope in the range [0-10 T] of the magnetization versus field $M(H)$ for $\mathbf{H} \perp \mathbf{c}$. The irreversible behavior of the high-field magnetization is emphasized here by the different susceptibilities for the rising and decreasing parts of the magnetic field pulse. The lines are guides to the eyes. (b) Magnetic susceptibility versus temperature of $Fe_{1.1}Te$ in steady magnetic fields $\mathbf{H} \perp \mathbf{c}$ of 1 T and $\mathbf{H} \parallel \mathbf{c}$ of 0.5 T.

irreversible spin-flop-like alignment of the antiferromagnetic moments m_{AF} at H_R during the rise of the pulse could lead to an alignment of the moments perpendicular to the field direction. Within this hypothesis, the condition $\mathbf{H} \perp \mathbf{m_{AF}}$ would be fulfilled for both susceptibilities measured in a steady field $\mathbf{H} \parallel \mathbf{c}$ and during the fall of pulsed fields $\mathbf{H} \perp \mathbf{c}$, which would explain their similar temperature dependences. Alternatively, the step-like anomaly in the magnetization at H_R could be driven by a more subtle change of the magnetic structure, as a modification of the magnetic wavevector (cf. Refs. [30, 31]) and/or the alignment of the moments from one of the two non-equivalent magnetic Fe-sites (cf. Ref. [32]). The microscopic model should also describe how a remanent moment reorientation is induced once a magnetic field higher than H_R has been applied and then removed. Knowing that a change of the lattice structure almost coincides with the development of antiferromagnetism at T_N [14, 28, 29], magneto-elastic couplings could possibly play a role in $Fe_{1.1}Te$ in a high field. Several mechanisms of coupling between the elastic and magnetic degrees of freedom, for example via an orbital ordering [33], a spin-nematic mechanism [34], or based on symmetry considerations [35], have been proposed in the literature. The question is whether such theories could help understanding the non-reversible moment reorientation observed here. To go further experimentally, magnetostriction under pulsed magnetic field (by strain gage [36], dilatometry [37], or diffraction techniques [38]) could lead to valuable information about the field-induced distortion of the lattice. Also, the high-field magnetic structure of $Fe_{1.1}Te$ could be determined by neutron diffraction in pulsed field [39].

In conclusion, we have studied the magnetic properties of the antiferromagnet $Fe_{1.1}Te$ in pulsed magnetic fields $\mathbf{H} \perp \mathbf{c}$ up to 53 T. In addition to the observation, at temperatures close to T_N, of the critical field H_c associated to the antiferromagnetic-to-paramagnetic borderline, we have evidenced a non-reversible moment reorientation at $\mu_0 H_R \simeq 50$ T at low temperature. The irreversible step-like anomaly in the magnetization at H_R could result from a spin-flop-like reorientation of the antiferromagnetic moments, and/or a change of the magnetic structure (wavevectors, ordered moments etc.). Further efforts from both experimental and theoretical sides are now needed to understand the remanence of the high-field reoriented antiferromagnetic phase. Better knowledge of the magnetic properties of the parent compounds $Fe_{1+x}Te$ will surely help understanding the role played by magnetism and magneto-elastic coupling for the appearance of superconductivity in $Fe_{1+x}Te_{1-y}Se_y$, which coincides with an antiferromagnetic-to-paramagnetic quantum instability coupled with a lattice structure modification.

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