Two types of magnetic shape-memory effects from twinned microstructure and magneto-structural coupling in Fe$_{1+y}$Te

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A detailed experimental investigation of Fe$_{1+y}$Te (y ≈ 0.11, 0.12) using pulsed magnetic fields up to 60 T confirms remarkable magnetic shape-memory (MSM) effects. These effects result from magnetoelastic transformation processes in the low-temperature antiferromagnetic state of these materials. The observation of modulated and finely twinned microstructure at the nanoscale through scanning tunneling microscopy establishes a behavior similar to that of thermoelastic martensite. We identified the observed, elegant hierarchical twinning pattern of monoclinic crystallographic domains as an ideal realization of crossing twin bands. The antiferromagnetism of the monoclinic ground state allows for a magnetic-field–induced reorientation of these twin variants by the motion of one type of twin boundaries. At sufficiently high magnetic fields, we observed a second isothermal transformation process with large hysteresis for different directions of applied field. This gives rise to a second MSM effect caused by a phase transition back to the field-polarized tetragonal lattice state.

magnetic shape-memory effect | antiferromagnets | Fe-chalcogenides

The magnetic shape-memory (MSM) effects are well known in ferromagnetic materials such as Ni-Mn-Ga–based alloys (1, 2) or other Heusler compounds (3). The first type arises due to a field-induced reorientation of one of the crystallographic twin variants. The second type results from a metamagnetic reverse phase transformation. Both effects are rarely found in antiferromagnets because of the absence of a net magnetic moment (4, 5). In the antiferromagnetic parent compound of high-T$_c$ cuprate La$_{2−x}$Sr$_x$CuO$_4$, a strong magnetic anisotropy owing to spin–orbit coupling, a mechanism through which the spin degrees of freedom couple to the lattice, is considered as a driving force for the MSM effect (4). It is generally believed that this first type of macroscopic shape effect by an applied magnetic field requires a twinned microstructure at a ferroelastic transition between a higher-symmetry austenite and a low-symmetry martensite phase (1, 2). The MSM effect of the first type consists of a rearrangement involving the growth of some twin domains at the expense of less-favored orientations. The detailed microscopic process in the case of the metamagnetic shape memory—the second type—requires a transition between 2 different magnetic phases with different lattice geometries; i.e., it is a field analogue of a conventional temperature-driven shape-memory effect. However, the present understanding of the MSM behavior is incomplete and there is no consensus about which materials are expected to display these processes. Observation of both types of MSM effects in the same material is interesting, because it suggests that a high mobility of planar twin walls in the martensitic phase (for the first type) and that of the phase boundaries between different magnetic phases (for the second type) may originate from similar features of the underlying different microstructure.

In the case of Fe-based high-T$_c$ superconductors, magnetic-field–induced persistent detwinning has been reported (6–11). However, a clear physical picture of these detwinning processes including a quantitative theoretical analysis is still lacking. In this article, we provide a detailed identification of the actual microstructures and mechanisms responsible for these field-induced processes by performing magnetization and magnetostriction measurements in pulsed magnetic fields on single crystals of Fe$_{1+y}$Te (y ≈ 0.11 and 0.12), both in the twinned and in the mechanically detwinned state. In particular, our magnetostriction measurements up to a magnetic field of 60 T show that the spin reorientation (10) and magnetostructural transition (11) observed in Fe$_{1.15}$Te are related to 2 different types of MSM effects. Fig. 1 summarizes these processes, as seen in the magnetization measurements up to 60 T (Fig. L4). An initial hierarchical twinning (Fig. L4, Lower Left Inset) of the monoclinic crystal is first transformed at $H_{\text{m}}$ into a single plate consisting only of 2 monoclinic twin variants, which is effectively a single-domain orthorhombic structure with nanotwinning (Fig. L4, Middle Inset). At higher-field $H_{\text{m}}$ and in a second step, the monoclinic lattice structure is replaced by the

Significance

Magnetic shape memory (MSM) refers to a change in shape and/or size of a magnetic material upon applying a magnetic field. There are 2 types of MSM effects; the first one occurs in a twinned magnetically ordered material, in which the crystallographic axes are irreversibly reoriented by the applied magnetic field. In the second type, the applied field drives a magnetoelastic phase transition. In certain iron tellurides Fe$_{1+y}$Te, both types of MSM occur. Notably, the first antiferromagnetic compound found to display an MSM effect is a parent material to the well-studied high-T$_c$ superconductor La$_{2−x}$Sr$_x$CuO$_4$. Observation of MSM effects in 2 known material families related to high-T$_c$ superconductors points to a prominent role of electron–phonon coupling arising through the spin–orbit interactions.

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Fig. 1. The field-induced solid–solid transformation processes and underlying microstructure of a Fe$_{1.11}$Te single crystal. (A) Magnetization $M(H)$ obtained at 48 K up to a magnetic field of 58 T (see Fig. 2 for details). Insets show schematics of the microstructural change upon applying a magnetic field. (B) Topography on an area of $5 \times 5$ nm$^2$ measured at 100 K. (C) Three-dimensional topography on an area of $400 \times 400$ nm$^2$ at 40 K indicating a hierarchical twin microstructure of the monoclinic phase. (D) Cartoon of the topography presented in C. (E) Schematic unit cells of 4 different twin variants M1 to M4 in the monoclinic phase derived from the 2 orthorhombic variants O1 and O2 describing the $ab$ and $ba$ deformation of the tetragonal parent lattice. (F) Topography on an area of $20 \times 20$ nm$^2$ depicting a twin-domain wall along the [110] direction across which a periodic modulation of a 1D density wave oriented at an angle of $\approx 90^\circ$ with respect to each other can be seen. (G and H) STM topography on areas of $20 \times 20$ nm$^2$ on either side of a [110] twin-domain wall. Insets display corresponding Fourier transformations. The pink arrows highlight additional spots at (0, 0.5) and (0.5, 0), respectively, confirming the 1D stripe structure. The color-coded arrows (blue, pink, orange, and green) point to the corresponding domains in C and D. (I) Schematic in-plane spin structure of Fe$_{1.11}$Te in the monoclinic phase. The red rectangle represents the $ab$ plane of the unit cell; black and yellow arrows indicate the orientation of the spins at the individual Fe sites.

field-polarized, effectively paramagnetic tetragonal phase (Fig. 1A, Right Inset). A scanning tunneling microscope (STM) was used to determine the twin microstructure, which is shown to fulfill the conditions for self-accommodation of the spontaneous strains at the ferroelastic tetragonal-to-monoclinic transition. The hierarchical arrangement of monoclinic twin domains is identified as a crossing twin pattern described by the continuum theory of martensites (12, 13). Using density functional theory (DFT) calculations, we identified [10$\overline{1}$1] of the monoclinic twin variants as the easy axis, which corresponds to a direction in one of the $ac$ planes of the tetragonal lattice structure. Our experiments provide direct evidence for a giant magnetoelastic coupling in an antiferromagnet, which is closely related to an Fe-based superconductor, namely FeSe.

Results

Scanning Tunneling Microscopy. Since the martensitic microstructure is a salient feature of the materials displaying the MSM effect, we first investigated the microstructure of a Fe$_{1.11}$Te single crystal using a low-temperature STM. Fe$_{1.11}$Te exhibits a direct tetragonal-to-monoclinic phase transition (14, 15) (SI Appendix, sections 1 and 2) and is, hence, prone to strong MSM effects. Cleaving the Fe$_{1.11}$Te crystal in situ exposes Te-terminated surfaces which may contain different amounts of excess Fe (SI Appendix, section 3). In the following, Te-terminated clean surfaces are presented, since they allow us to examine the microstructure. In Fig. 1B, a STM topographic image of Fe$_{1.11}$Te over an area of $5 \times 5$ nm$^2$ taken in the tetragonal phase at 100 K is displayed. The atomically resolved surface with $\delta_{\text{Te-Te}} \approx 3.8$ Å is consistent with the bulk lattice parameter (SI Appendix, section 4). In the monoclinic phase, 4 differently oriented twin variants M1 to M4 are expected (16), which can be discerned from Fig. 1C and D while Fig. 1E depicts how these 4 variants are brought about. The STM topography over a large area of $400 \times 400$ nm$^2$ measured at 40 K, a temperature well below the tetragonal-to-monoclinic structural phase transition, reveals all of the 4 twin variants (Fig. 1C). The surface morphology indicates a hierarchical twinning (17) resembling a chevron pattern with the domain orientation described in Fig. 1D and E. A similar twinning pattern has been recently reported.
for Fe1.08Te (18). In Fig. 1C, an invariant twin plane (110) running along the [110] direction distinguishes domains in which the crystallographic a and b axes are rotated by ≈90°. A typical distance between 2 [110] twin-domain walls is found to be ≈340 nm (SI Appendix, section 3). Across the (110) invariant twin plane, a further microstructural feature has been observed originating from the alternating monoclinic angle β between c and a axes, approximately, at a distance of about 50 nm. This highly regular pattern of crossing twins is the expected self-accommodating martensitic microstructure that combines all 4 monoclinic variants (12, 13); see SI Appendix, sections 5 and 6 for details. In Fig. 1F, the STM topographic image of 20 × 20 nm² with a [110] twin boundary is presented. In addition to atomic modulation, the image displays 1D stripe structures, which are rotated by ≈90° across the [110] twin boundary. In Fig. 1G and H, STM topographic images on either side of a [110] twin boundary are presented. Insets show corresponding Fourier transformations. Besides the regular Bragg spots, the 1D charge modulation can be seen from satellite spots at q = (0, 0.5) and q = (0.5, 0) which correspond to 2 different twin domains. The period of the stripes in real space is found to be 2αTe–Te. This is identical to the antiferromagnetic wave vector qAFM = (0.5, 0) (19) defined in reciprocal space. The AFM wave vector in Fe1.11Te propagates along the a axis, i.e., along the (π, 0) direction (19, 20) as schematically represented in Fig. 1F. Note that the arrows in Fig. 1F indicate individual Fe sites whereas in the STM topography we see Te termination. The 1D stripe structure found here is consistent with results from previous STM experiments conducted with both cold (21–24) and spin-polarized tips (25, 26). In contrast to a charge density wave occurring at 2qAFM expected from the magnetostriction, the 1D stripe order found here at the same wave vector as the qAFM likely stems from the bond order wave induced by the ferro-orbital ordering taking place at the magneto-structural phase transition (24, 27). This scenario evokes an intertwining of spin, charge, lattice, and orbital degrees of freedom. Further, both the SDW and the CDW domains adapt to the underlying martensitic microstructure.

**Magnetization and Magnetostriction.** The presence of modulated martensitic phases suggests that the twin boundaries may be easily movable by the application of external magnetic fields (17). To establish this, we performed magnetization M(H) and magnetostriiction Δl(H) measurements in pulsed magnetic fields up to 60 T. The M(H) measurements with magnetic fields H applied along different crystallographic directions of Fe1.11Te are presented in Fig. 2A and B. The first measurement was conducted at 1.6 K, after cooling the sample from room temperature, annotated as virgin measurement. When the magnetic field was applied along the tetragonal [100] direction (Fig. 2A), the M(H) displays a jump at μ0Hm1 ≈ 48 T upon increasing the magnetic field. This jump is absent in the decreasing part of the field cycle. With a second field pulse, no anomaly, rather a monotonic behavior, was observed during the entire field pulse. The virgin curve could be recovered only after the sample was heated to a temperature above the temperature of the structural transition (57 K) and cooled back again. This behavior indicates that the jump involves an irreversible structural process, related to the monoclinic phase, and is reminiscent of a memory effect: The sample remembers the first pulse, as long as it is kept below the temperature of the structural phase transition. We also measured M(H) with the magnetic field applied along [110] and [001] directions. As depicted in Fig. 2B, no effect is seen in M(H) up to 60 T, indicating that the jump occurs only when the field is parallel to the [100] direction. Fig. 2C shows M(H) measured with H || [100] for Fe1.11Te.

![Image](image-url)

**Fig. 2.** Magnetization M(H) and magnetostriction Δl(H)/l measurements on Fe1.11Te. (A) M(H) with magnetic field H || [100] direction of the tetragonal structure at 1.6 K after cooling the sample from room temperature (virgin) in 0 field, by giving a second field pulse at 1.6 K (second pulse), and after heating the sample to 100 K followed by cooling to 1.6 K (re-virgin). (B) M(H) curves with H || [110] and H || [001] directions at 1.6 K. (C) M(H) measured at different temperatures. The black arrows indicate the decrease in detwinning field Hm1, upon increasing temperature. At 42 K, a second jump in M(H) is visible at a field of μ0Hm2 ≈ 50 T, which corresponds to a metamagnetic reverse transformation (main text). The curves measured at 20 K and above are vertically shifted for clarity. (D) Δl(H)/l measured along the [100] direction. The jump is absent in the second measurement, confirming an antiferromagnetic shape-memory effect. In D, Δl(H)/l of a detwinned (by compressive strain) crystal is presented. For all measurements, the magnetic field was kept parallel to the [100] crystallographic direction. (E) Setup used for the application of compressive stress along [010] to mechanically detwin the crystal.
The field $H_{m1}$ decreases and finally it is not observed at and above 60 K. However, at 42 K, while the field is increasing, a second transition appears in a field of $\mu_0 H_{m2} \approx 50$ T. The field $H_{m2}$ also decreases with increasing temperature and disappears at 60 K. Thus, at temperatures near 42 K, $\text{Fe}_{1.11}\text{Te}$ undergoes 2 different types of field-induced reorientations. Similar discontinuities have been previously reported in $\text{Fe}_2\text{Te}$ by Knafo et al. (10). In the following, we show that $H_{m1}$ and $H_{m2}$ are the critical fields required for detwinning and a reverse phase transformation into a spin–polarized tetragonal phase, respectively.

First we address the process occurring at field $H_{m1}$. To this end, we performed magnetostriction experiments (28) on both twinned and mechanically detwinned $\text{Fe}_{1.11}\text{Te}$ single crystals (29–31). The virgin curve measured after cooling the sample from room temperature to 5.8 K displays a jump in $\Delta l(H)/l$ at $\mu_0 H_{m1} \approx 45$ T, as can be seen in Fig. 2D. During the second magnetic pulse at 5.8 K, no change in sample length was observed. This result is consistent with the $M(H)$ behavior and confirms the magnetic shape-memory effect; i.e., the change in the shape of the sample induced by the first field pulse is persistent. The virgin behavior could be recovered if the sample is heated above the structural transition temperature (blue curve in Fig. 2D). In a subsequent measurement, we mounted the same sample on a simple strain device shown in Fig. 2E. This allows for applying a compressive stress along the [010]$\parallel$ direction, which prevents the twin-domain formation once the sample is cooled below the structural transition. In result, the change in length upon applying a field pulse is significantly suppressed at $H_{m1}$ (red curve in Fig. 2D). The $H-T$ diagram of $\text{Fe}_{1.11}\text{Te}$ is presented in SI Appendix, section 7.

Now we focus on the second reorientation transition occurring at field $H_{m2}$. For this purpose, we use an $\text{Fe}_{1.12}\text{Te}$ crystal (Fig. 3A). At 1.6 K and $H \parallel \{100\}_T$ in the virgin measurement, 2 distinct transitions are visible at $\mu_0 H_{m1} \approx 42$ T and $\mu_0 H_{m2} \approx 52$ T. In contrast to $H_{m1}$, however, the jump at $H_{m2}$ is also present when the magnetic field is applied parallel to the [110]$T$ as well as the [001]$T$ directions (Fig. 3B and C), indicating that the second transition is isotropic and involves a different mechanism. According to the $\Delta l(H)/l$ measurements at $H_{m1}$, the sample expands along the a axis due to detwinning, but at $H_{m2}$, the sample shrinks (Fig. 3D). Since the lattice parameter a in the tetragonal phase is smaller than that in the monoclinic phase (15), contraction in the sample length at $H_{m2}$ indicates that the material undergoes a transformation toward the tetragonal phase at $H_{m2}$. This structural transformation has also been observed in $\text{Fe}_{1.1}\text{Te}$ (11). The irreversible behavior at $H_{m2}$ indicates that the second transition also involves a memory effect. However, the behavior can be recovered during the second pulse; i.e., the sample is not required to be heated above the structural transition temperature. The system returns to the distorted martensite state within the waiting time (1 h in the case of magnetization and 2 h in the case of magnetostriction measurements) after the magnetic pulse necessary to cool down the pulsed-field magnet. To obtain the temperature dependence of $H_{m2}$, we performed $\Delta l(H, T)/l$ also on mechanically detwinned crystals. $H_{m2}$

![Fig. 3. Magnetization ($M$($H$) and magnetostriction ($\Delta l$($H$))/($l$) measurements on $\text{Fe}_{1.12}\text{Te}$. (A) $M$($H$) curves measured with applied magnetic field $H \parallel \{100\}_T$ direction of the tetragonal structure at 1.6 K after cooling the sample from room temperature (virgin) in 0 field and by applying a second field pulse at 1.6 K (second pulse). (B) $M$($H$) curve with applied field $H \parallel \{110\}_T$ at 1.6 K. (C) $M$($H$) curve measured at 1.6 K. (D) $\Delta l$($H$)/$l$ measured along the [100]$T$ direction, both during the first (virgin) pulse and during the second pulse. The magnetic field $H$ was kept parallel to the [100]$T$ crystallographic direction. (E) $\Delta l$($H$)/$l$ measured along [100]$T$ at different temperatures on mechanically detwinned crystals. (F) Magnetic field vs. temperature phase diagram of $\text{Fe}_{1.12}\text{Te}$ with $H \parallel \{100\}_T$ displaying a field-induced transformation of a martensite (twinned phase) to austenite via a detwinned martensite phase, i.e., an effectively single-variant orthorhombic state, which however still can be nanotwinned into 2 monoclinic variants (shown in inset).]
We thank Ulrike Nitzsche for computational assistance. In Fe$_{1+y}$Te, which is structurally superconducting, reflects the magnetic moment of Eu combined with magnetoelastic coupling of the Fe sublattice (38), but the evolution of microstructure during this process is unknown at present. Recently, the possibility of thermal shape-memory effects and anomalously large, "superelastic" strains has been demonstrated for micropillars of CaFe$_2$As$_2$ (39). The extremely large spontaneous strains of order 10% in CaFe$_2$As$_2$ stem from an entirely different mechanism, namely, an isostructural tetragonal-to-collapsed tetragonal transition under pressure. A shape-memory behavior then requires one to exploit also the symmetry-breaking martensitic transition at ambient pressure, which however seems to be associated with plastic deformations. Notably, Fe$_{1+y}$Te displays a similar isostructural, so-called type-0, transition from tetragonal-to-tetragonal phase under pressure. The high-pressure phase, however, is ferrimagnetic (40). Cycling applied pressure, temperature, and an additional magnetic field for the transition from the monoclinic ground state to this tetragonal ferrimagnetic phase in Fe$_{1+y}$Te, similar to the cycle proposed in the work by Sypek et al. (39), may offer a way to a magnetic-field control of this type of superelasticity and shape-memory effects.

Conclusions

The field-induced detwinning in Fe$_{1+y}$Te, which is structurally related to the Fe-based superconductors, underlines the relevance of spin–orbit coupling in inducing a giant magnetoelastic coupling. As a corollary of our study an intriguing question arises: Whether the presence of magnetic shape-memory effects in 2 materials which are closely related to high-T$_c$ superconductors, namely, the cuprates and the Fe-based superconductors, just a coincidence or whether spin–orbit interaction is one of the necessary ingredients for high-T$_c$ superconductivity. This implies the coupling of electrons and phonons via the spin channel, which is considered an important constituent for superconductivity in Fe–pnictide superconductors (41). Such an underlying commonality of shape-memory effects in members of 2 classes of high-T$_c$ materials is expected to trigger future inquiry regarding the role of elastic degrees of freedom and anisotropic spin–lattice couplings for the mechanism of high-T$_c$ superconductivity.

Materials and Methods

Single crystals of Fe$_{1+y}$Te ($y = 0.11$, 0.12, and 0.14) were grown using chemical vapor transport (CVT) with iodine as a transport reagent (42, 43) (SI Appendix, section 2). For STM, conducted at a base temperature of 4.5 K and in ultrahigh vacuum, single crystals were cleaved in situ at $\approx 25$ K. The $\Delta M(\mu T)$ and the $\Delta(H)/I$ measurements were performed in the pulsed magnetic fields up to 60 T at the high magnetic-field laboratory in Dresden-Rossendorf (HLD-EMFL). The DFT calculations were done using spin14.00 (22), using the generalized gradient approximation (GGA) in the PBE parameterization as an exchange-correlation functional.

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