Persistent Detwinning of Iron-Pnictide $EuFe_2As_2$ Crystals by Small External Magnetic Fields

S. Zapf,[1,*] C. Stingl,[2] K. W. Post,[3] J. Maiwald,[2,4] N. Bach,[2] I. Pietsch,[2] D. Neubauer,[1] A. Löhle,[1] C. Clauss,[1] S. Jiang,[1] H. S. Jeevan,[2,5] D. N. Basov,[3] P. Gegenwart,[2,4] and M. Dressel[1]

[1]*1. Physikalisches Institut, Universität Stuttgart, Pfaffenwaldring 57, 70550 Stuttgart, Germany*

[2]*I. Physikalisches Institut, Universität Göttingen, Friedrich-Hund-Platz 1, 37077 Göttingen, Germany*

[3]*Department of Physics, University of California, San Diego, La Jolla, California 92093, USA*

[4]*Experimentalphysik VI, Universität Augsburg, Universitätsstraße 1, 86135 Augsburg, Germany*

[5]*Department of Physics, PESITM, Sagar Road, 577204 Shimoga, India*

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Our comprehensive study on $EuFe_2As_2$ reveals a dramatic reduction of magnetic detwinning fields compared to other AFe_2As_2 (A = Ba, Sr, Ca) iron pnictides by indirect magnetoelastic coupling of the Eu^{2+} ions. We find that only ~0.1 T are sufficient for *persistent* detwinning below the local Eu^{2+} ordering; above $T_{Eu} = 19$ K, higher fields are necessary. Even after the field is switched off, a significant imbalance of twin domains remains constant up to the structural and electronic phase transition (190 K). This persistent detwinning provides the unique possibility to study the low temperature electronic in-plane anisotropy of iron pnictides without applying any symmetry-breaking external force.

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The observation of a large in-plane anisotropy in iron pnictides has triggered tremendous research activity, as another potential key ingredient for high-temperature superconductivity was identified [1–9]. Similar to cuprates, the magnitude of the electronic anisotropy is unexpectedly large because it notably surpasses the orthorhombic lattice distortion. In other words, the itinerant electrons do not just follow the lattice anisotropy: instead, there is growing evidence for an underlying electronic "nematic" phase transition which breaks the crystal's rotational symmetry. As the formation of twin domains usually obscures this intrinsic anisotropy, sophisticated methods were already developed to detwin cuprates [10,11]. In the case of iron pnictides, the effect of typical laboratory magnetic fields on the Fe spins is rather weak [12]. Thus, mechanical clamps are commonly used for detwinning single crystals [1]. However, this introduces an explicit symmetry breaking by uniaxial pressure, which must be considered carefully [5]: similar to ferromagnets, where the magnetization depends on the external magnetic field, the intrinsic nematic response can only be measured in the limit of zero symmetry-breaking external force. Indeed, mechanical clamps were found to significantly enhance the transition temperatures in iron pnictides and even induce additional anisotropy above [6].

$EuFe_2As_2$ is a peculiar member of the 122 iron pnictides: similar to related high-temperature superconductors such as Ce 1111 pnictides and ruthenocuprates [13,14], the Eu^{2+} spins order at low temperatures magnetically. Below $T_{Eu} = 19$ K, they are arranged in an A-type antiferromagnetic structure, i.e., within one layer ferromagnetically along the a axis, but antiferromagnetically between neighboring layers (see Fig. 4, Refs. [15,16]). Furthermore, $EuFe_2As_2$ exhibits a structural and spin density wave (SDW) transition at $T_{s,SDW} = 190$ K which can be suppressed by doping or pressure until superconductivity sets in at around 30 K. It is still under debate how superconductivity coexists with local Eu^{2+} magnetism [17–22].

Here we demonstrate the *persistent* detwinning of $EuFe_2As_2$ by small magnetic fields, which yields similar detwinning fractions as commonly used mechanical devices. Whereas previous studies [23] reported only that in-plane magnetic fields of the order 1 T detwin $EuFe_2As_2$ at low temperatures with the crystal's longer a axis parallel to the external field $H \| [110]_T$ [24] and no persistent detwinning was revealed, we have investigated the magnetic detwinning in a broad temperature range, always following a well-defined cooling procedure. First, the sample was cooled from $T > T_{s,SDW}$ to low temperatures in zero magnetic field and the "zero-field cooled" (ZFC) response was measured. Afterwards, when an in-plane magnetic field parallel to the $[110]_T$ direction was first applied and then removed, we call this "field treatment" (FT). Reference measurements along the $[100]_T$ direction as well as details about the measurement techniques can be found in the Supplemental Material [25]. We show that the magnetic detwinning with $a \| H$ can be achieved also above the Eu^{2+} magnetic ordering temperature and that below T_{Eu}, an additional detwinning process with $b \| H$ takes place around 0.1 T. Most strikingly, the detwinned state remains even when the field is switched off (below or above T_{Eu}) and the temperature is raised up to $T_{s,SDW}$. This provides the unique possibility to study the low temperature electronic in-plane anisotropy of iron pnictides without applying any symmetry-breaking external force.

Resistivity and thermal expansion.—Our remarkable observation is a persistent detwinning after the magnetic

field is removed, effective even at temperatures above T_{Eu}. This can be directly seen in Figs. 1(a) and 1(b), which show the temperature-dependent resistivity $\rho(T)$ after FT, normalized to its ZFC value. Although the magnetic field is switched off before measuring, we observe a strong in-plane anisotropy along the orthorhombic axes. The anisotropy is opposite for FT below and above T_{Eu}. Furthermore, its magnitude agrees with values obtained for mechanically detwinned Eu compounds [7,32] and remains virtually constant up to $T_{s,SDW}$.

Concurrent evidence for a persistent structural detwinning is also found in thermal expansion, $\Delta L(T)/L$, which is shown in Fig. 1(c) for ZFC and FT. Apart from transitions at T_{Eu} and $T_{s,SDW}$, the sample is shorter after FT at $T = 4$ K than in the ZFC state. Relative length changes are of the order of 10^{-3}, exceeding the typical magnetostriction of Eu-based materials by at least 2 orders of magnitude. The induced imbalance of twin domains

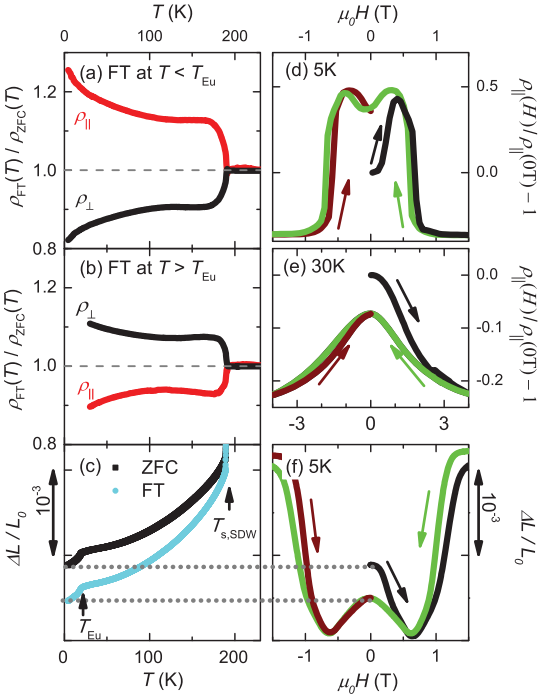

FIG. 1 (color). (Magneto)resistance, thermal expansion and magnetostriction of ZFC $EuFe_2As_2$ ($H\|[110]_T$). (a),(b) Temperature-dependent resistivity $\rho_{FT}(T)$ after field treatment (FT) with 4 T for currents parallel (red lines) and perpendicular (black lines) to H, normalized to ZFC $\rho_{ZFC}(0\ \mathrm{T})$. For FT at (a) $T = 4$ K and (b) 30 K, the opposite behavior is observed. (c) Thermal expansion $\Delta L(T)/L_0$ ($\Delta L\|H$) after ZFC (black line) and FT with 2 T at 4 K (cyan line). (d),(e) Magnetoresistance $\rho_\|(H)/\rho_\|(0\ \mathrm{T}) - 1$ at (d) 5 K and (e) 30 K as well as (f) magnetostriction $\Delta L(H)/L_0$ at 5 K for increasing (black and brown lines) and decreasing (green line) H. All measurements are consistent with a persistent detwinning induced by low magnetic fields that is achievable below or above T_{Eu} and robust against heating up to $T_{s,SDW}$.

stays constant up to $T_{s,SDW}$ (based on a comparison with experimentally determined lattice constants [33]; see Supplemental Material [25]).

Magnetoresistance and magnetostriction.—In order to understand the detwinning mechanism in more detail, we have investigated the field-dependent magnetostriction and magnetoresistance for a ZFC crystal. As the magnetostriction for $T < T_{Eu}$ [see Fig. 1(f)] directly shows, with increasing field the sample first contracts, then expands along H. After decreasing the field to 0 T, the original length is not recovered and the sample remains shorter. Assuming $\rho_b > \rho_a$, which was found for mechanically detwinned iron pnictides [1], the same behavior is visible in the magnetoresistance. Thus, the changes in magnetoresistance can be attributed to the magnetic detwinning of the system and not predominantly to electron-spin scattering, as suggested by Ref. [34]. We conclude from the field-dependent measurements that there are two separate detwinning processes when $T < T_{Eu}$. The first occurs at lower fields where the majority of domains get preferentially oriented with the b axis parallel to H; the hereby induced imbalance of twin domains persists even when the magnetic field is removed. Secondly, the crystal gets detwinned with $a\|H$ at slightly higher fields (< 1 T).

On the other hand, at $T > T_{Eu}$ [see Fig. 1(e)], with increasing field, the sample expands only along H. After decreasing the field to 0 T, the original length and resistance is again not recovered and the sample remains longer. Thus we conclude that above T_{Eu}, only the latter detwinning process with $a\|H$ takes place.

Magneto-optics.—Previous infrared spectroscopy on $EuFe_2As_2$ has revealed that Eu^{2+} spin scattering does not notably influence the reflectivity in the far-infrared (FIR) energy range [35]. Therefore, we have performed low-frequency magneto-optical reflection measurements. Figure 2 exhibits polarization-dependent spectra between 220 and 450 cm^{-1}. We chose $T = 15$ K, 30 K, as well as $H = 0$ T (ZFC), 1.0 T, and 0 T after FT (with 1 T) as representative. Those frequencies are dominated by the SDW gap and an Fe-As phonon mode at $\sim$260 cm^{-1}. (see Ref. [35].) For 1 T and after FT, a notable difference is induced between the two polarizations, consistent with the above described magnetic detwinning. The origin of the anisotropy is a stronger gap opening and an enhanced phonon oscillator strength along the b axis, which was also found in mechanically detwinned Ba 122 compounds [36–39].

Further information on the twin dynamics can be obtained from the false-color plot of the field and frequency dependent relative reflectivity ($E\perp H$) in Fig. 3. For $T = 15$ K $< T_{Eu}$ and increasing magnetic field, $R(H)$ increases rapidly between 0.075 and 0.15 T, afterwards staying almost constant. Thus, $H_1 \sim 0.1$ T can be identified as the critical field where twins preferentially align with $b\|H$. A sharp drop in the reflectivity at $H_2 \sim 0.6$ T marks the second, opposite detwinning process with $a\|H$. For

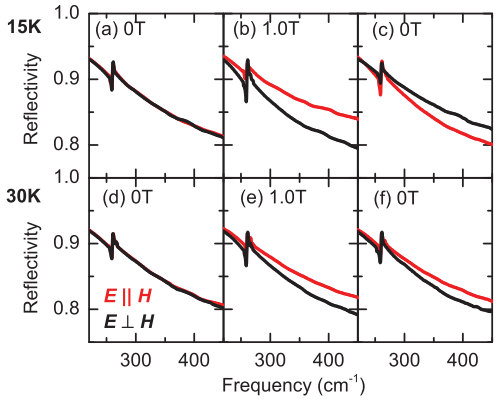

FIG. 2 (color). Frequency dependent reflectivity of $EuFe_2As_2$ at (a–c) $T = 15$ K and (d–f) 30 K, for (a,d) $H = 0$ T (ZFC), (b,e) 1 T, and (c,f) 0 T after FT $(H\|[110]_T)$. The magnetic field induces anisotropy between $R(E\|H)$ (red lines) and $R(E\perp H)$ (black lines) and also changes the Fe-As phonon mode at ~260 cm^{-1}, both corresponding to the magnetic detwinning.

decreasing H, the latter process is reversible with a slightly lower critical field. However, the detwinning at low fields is persistent. At $T = 30\ \text{K} > T_{\text{Eu}}$, $R(H)$ continuously decreases with increasing magnetic field, until it saturates at ~0.9 T. With decreasing H, the reflectivity stays almost constant and the detwinning with $b\|H$ is persistent, even when the field is switched off.

Magnetization.—Since the Eu^{2+} magnetic moments drive the detwinning (comparison measurements on $BaFe_2As_2$ can be found in the Supplemental Material [25]), we also include the field-dependent magnetization, which is dominated by Eu^{2+} moments [16], in Fig. 3. At $T = 15$ K, $M(H)$ exhibits two transitions for increasing—but only the upper one for decreasing—the field. The first transition at ~0.1 T corresponds to the H_1 found in reflectivity. However, the second transition, which can be identified due to an abrupt jump in $M(H)$ as a spin flip, precedes the H_2 transition observed in reflectivity by about 0.1 T. At $T = 30$ K, no transition is visible in $M(H)$. Temperature-dependent magnetization measurements are consistent with our interpretations and are shown in the Supplemental Material [25].

Model.—We suggest a simple model to explain the detwinning process above and below T_{Eu}, based on the competition between magnetocrystalline anisotropy Δ, antiferromagnetic exchange coupling J, and Zeeman energy. Two twin domains have to be considered, one with the easy a axis perpendicular (type $B_\|$) and one with it parallel (type $A_\|$) to H (see Fig. 4). After cooling in zero magnetic field, the crystal is twinned with equally distributed variants. At $T < T_{\text{Eu}}$ and with $H\|[110]_T$, minimizing the energy yields for the two twin variants [see Ref. [40], Supplemental Material [25], and Fig. 4(e)],

$$E_{\text{min}}^{B_\|} = E_0 - \frac{M^2(\mu_0 H)^2}{2JM^2 + \Delta}, \tag{1}$$

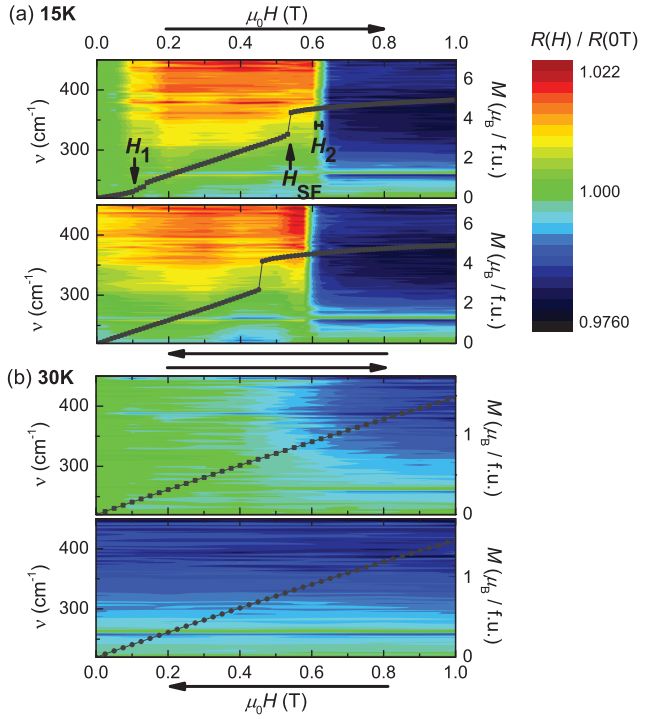

FIG. 3 (color). $EuFe_2As_2$. Field-dependent magnetization $M(H)$ (grey dots) and false color plot of the frequency and field-dependent FIR relative reflectivity $R(H)/R(0\ \text{T})$ $(E\perp H)$ at (a) $T = 15$ K and (b) 30 K $(H\|[110]_T)$. The detwinning fields H_1 and H_2 as well as the spin flip field H_{SF} of Eu^{2+} can be identified $(H_1 < H_{\text{SF}} < H_2)$.

$$E_{\text{min}}^{A_\|} = E_0 + \Delta - \frac{M^2(\mu_0 H)^2}{2JM^2 - \Delta}, \tag{2}$$

with ground state energy E_0. Thus, at low fields, the Eu^{2+} spins of variant $B_\|$ gradually rotate towards H, lowering the system's energy. When $E_{\text{min}}^{B_\|} - E_{\text{min}}^{A_\|}$ exceeds the twin boundary pinning energy, variant $B_\|$ grows irreversibly on the expense of variant $A_\|$, and the crystal gets detwinned with $b\|H$. Increasing the magnetic field further induces a spin flip in variant $A_\|$, but this twin variant is energetically favorable only at slightly higher fields, when the crystal gets detwinned with $a\|H$. At $T > T_{\text{Eu}}$, only a detwinning with $a\|H$ occurs, because the unordered Eu^{2+} spins align gradually along the magnetic field. As characteristic for domain dynamics, these processes are strongly irreversible, leading to a significant persistent detwinning.

Concluding discussion.—The question remains why the magnetic detwinning fields are reduced in $EuFe_2As_2$ by more than 2 orders of magnitude (compared to other iron pnictides). Magnetoelastic coupling usually arises due to spin-orbit interactions. However, the orbital momentum of Eu^{2+} is zero; thus the magnetic anisotropy induced by spin-orbit interactions is negligible. Another possibility to induce magnetic anisotropy is by dipole-dipole interactions. However, the resulting anisotropy is much weaker

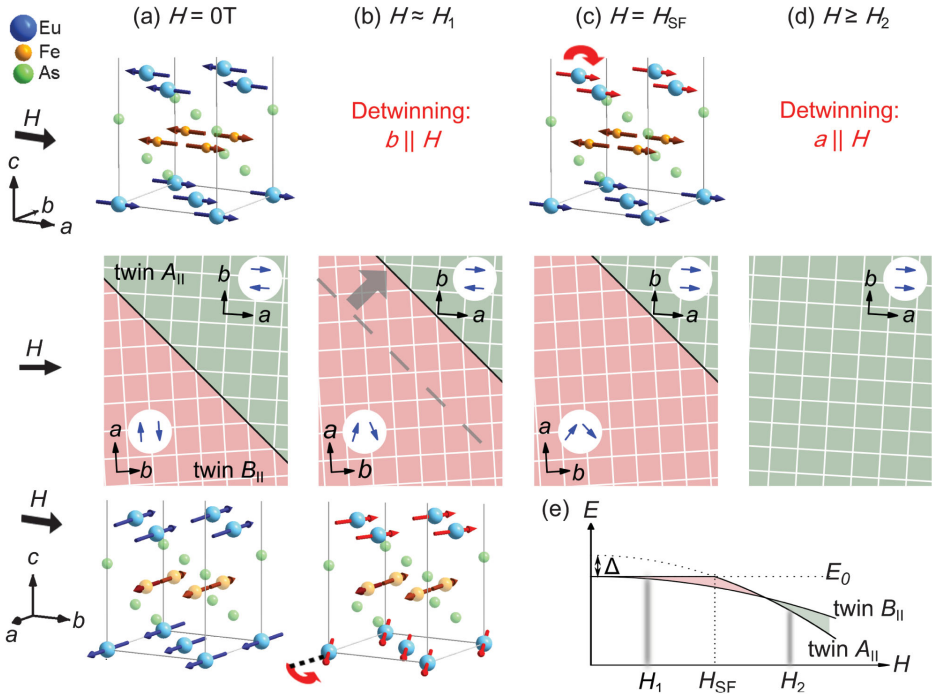

FIG. 4 (color). Sketch of twin distribution and spin configuration of $EuFe_2As_2$ dependent on an (from left to right, increasing) external magnetic field $H\|[110]_T$ at $T < T_{Eu}$. Top and bottom rows show a detail of the $EuFe_2As_2$ crystal and magnetic structure [Eu atoms and spins (blue), Fe atoms (yellow) and spins (brown), and As atoms (green)] that correspond to the twin distribution sketched in the middle row. (a) For $H = 0$ T, the ZFC crystal is twinned and the domains are equally distributed. The Eu^{2+} spins are ordered A-type antiferromagnetically with the spin direction along the a axis. (b) With an external field, twin variant $B_\|$ (red, bottom) with $b\|H$ gets energetically favored and therefore grows at the expense of variant $A_\|$ (green, top) as soon as the energy difference exceeds the twin boundary pinning energy. (c) With a further increasing field, Eu^{2+} spins in the remaining type $A_\|$ twins flip along the field direction. Energetically, twin $B_\|$ is still more favorable. (d) At slightly higher fields, twin $A_\|$ is favored and the crystal is detwinned with $a\|H$. (e) Corresponding energy curves (E_0: ground state). While at low fields twin $B_\|$ is energetically favorable (red area), twin $A_\|$ becomes favorable at higher fields (green area). Detwinning takes place at H_1 and H_2, when the energy gain exceeds the pinning energy of the twin boundary.

[41]. Therefore, other unconventional interactions must cause our observations. From the phase diagram of doped or pressurized Eu compounds, it is well known that the Eu^{2+} and Fe^{2+} magnetic orders are strongly intertwined [42–45]. Furthermore, the magnetic moment of Fe^{2+} is nonzero, leading in $BaFe_2As_2$ to significant magnetoelastic coupling [12]. Hence, we suggest that the Eu^{2+} spins couple indirectly to the lattice via the Fe^{2+} spins.

We have shown by using resistivity, thermal expansion, magnetostriction, magnetoresistance, magneto-optical, and magnetization measurements that $EuFe_2As_2$ can be persistently detwinned by laboratory-scale magnetic fields, yielding similar detwinning fractions as commonly used mechanical devices do. The detwinning is possible below and above the local Eu^{2+} magnetic ordering; however, the mechanism is slightly different: while at $T < T_{Eu}$ the crystal gets detwinned with $b\|H$ at low fields (~0.1 T at 15 K) and with $a\|H$ at high magnetic fields (~0.6 T at 15 K), at $T > T_{Eu}$, only the latter takes place. We propose that the Eu^{2+} moments couple indirectly to the lattice via the Fe^{2+} spins. To our knowledge, this is the first time that such an indirect coupling has been concluded. Most strikingly, a significant imbalance of twin domains remains when the field is switched off and the temperature is raised up to $T_{s,SDW}$. Such indirect magnetoelastic coupling and its persistence up to much higher energy scales could also be interesting for other materials and even technical applications. In summary, the whole effect uncovers a remarkable interdependence between magnetic, electronic, and structural effects and allows examining macroscopically the intrinsic in-plane anisotropy of iron pnictides without the application of any symmetry-breaking external force.

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*sina.zapf@pi1.physik.uni-stuttgart.de

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