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In Plane Vortex Dynamic Anisotropy in the Iron Deficient $Fe_{1-y}Se$ Superconductor

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Abstract We present electrical transport measurements in the superconducting dissipative state of crystalline iron deficient $Fe_{1-y}Se$ samples. These iron deficient samples were synthesized using NaCl/KCl flux and are characterized by the presence of correlated defects. The dissipation in electrical transport experiments, when the driving current is perpendicular or parallel to the crystal planes, depends strongly on the direction of the applied magnetic field , ($H = 12\,T$), within the sample plane. There is a dissipation modulation each 60° due to the presence of the correlated defects. We correlate these angular dependent features with the variation of the critical currents (J_c) changing the direction of H confined in the crystals planes. J_c was measured from magnetization loops at fixed temperatures and angles of H always within the basal planes.

Keywords Iron-based superconductors · Electrical transport · Critical currents

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1 Introduction

Among the Fe-based superconductors the 11 family, which has the simplest crystal structure, has been studied intensively. In particular, the $Fe_{1-y}Se$ compound is characterized by the PbO-type structure and a critical temperature of $T_c = 8\,K$ [1], that can be enhanced by Te substitution [2] or by pressure [3]. Nevertheless the synthesis of single crystals of these compounds is rather difficult, since the phase diagram is complex and there is evidence of inhomogeneity in the samples [4].

In a previous work [5], the angular dependence in transport and magnetization measurements while the vortices go in and out of the basal plane of a $Fe_{1-y}Se$ single crystal was studied. The orientation of the interface between the FeSe phases present in the sample coincides with the direction in which the dissipation due to vortex movement is minimum, evidencing the existence of pinning by intrinsic-correlated defects [5]. The purpose of this work is to study the influence of these defects when the magnetic field rotates in the basal plane and the current flows in or out of the plane.

2 Experimental

We synthesized $Fe_{1-y}Se$ samples using NaCl/KCl flux. The obtained crystals are hexagonal prisms, some of them with a platelet shape (typical dimensions: $0.7\,mm^2 \times 0.02\,mm$) and others as tubes ($3.6 \times 10^{-3}\ mm^2 \times 0.2\,mm$). Figure 1a shows a typical hexagonal prism sample in the tube shape. On the surface of the platelet samples, line defects parallel to the sides of the hexagons were observed.

Structural and compositional characterization [5] indicate that the samples present a coexistence of two phases, the superconducting tetragonal β-FeSe, and the ferrimagnetic hexagonal γ-Fe_7Se_8. The c axis of both phases is parallel but for the β-FeSe phase there also exists another preferential c axis orientation at 57° from the platelet normal [5].

The phase coexistence is also evident in their magnetization and transport properties. The samples have a complete superconductor transition with a critical temperature $T_c = 12.0$ K for the platelet and $T_c = 11.0\,K$ for the tube. The transport measurements were performed with a conventional four-wire method in a superconducting magnet at 12 T. The crystals were mounted onto a rotatable sample holder with an angular resolution of 0.05°. The magnetization was measured in a QD-SQUID magnetometer. The magnetization loops show two contributions, one superconducting and other magnetic, which can be assigned to the β-FeSe and γ-Fe_7Se_8 phases, respectively.

3 Results and Discussion

We measured the electrical resistivity of a tube of $Fe_{1-y}Se$ in a fixed magnetic field of 12 T that was applied parallel to the basal plane with the current flowing along the tube. The sample was continuously rotated around the current direction axis. The angle, φ, was measured between the applied field direction and a vector perpendicular to one side of the hexagon (Fig. 1a). In this constant Lorentz force (F_L) configuration, the dissipation in the superconducting state is caused by vortices crossing over the sample always parallel to the basal plane.

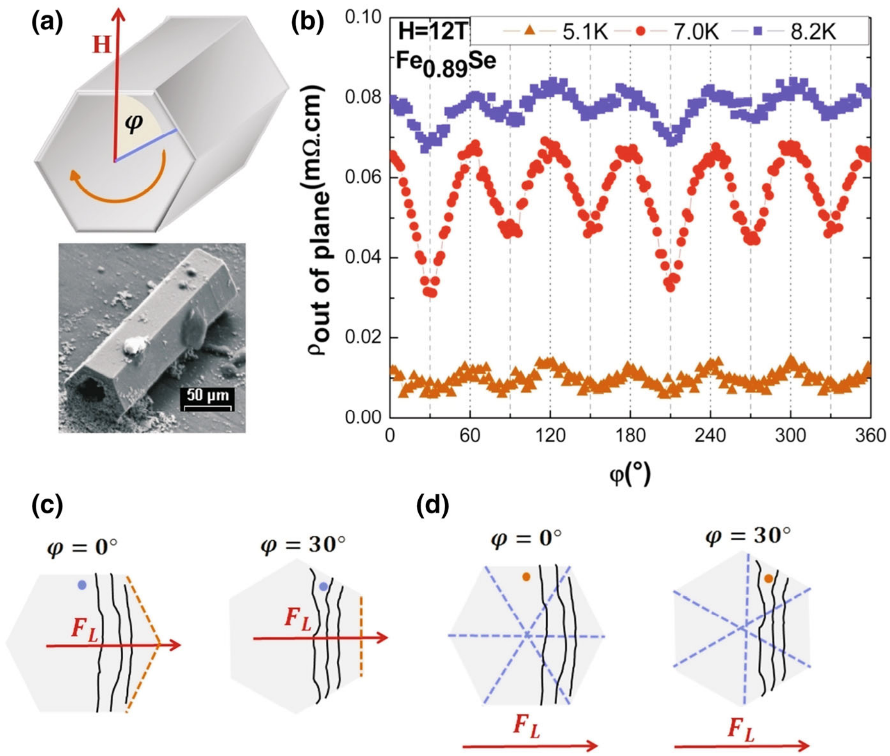

Fig. 1 **a** *Shape* of the sample and definition of the rotation angle. **b** Resistivity perpendicular to the basal plane. **c**, **d** *sketchs* showing the sample profile for the vortex entering the sample and the defects crossing the sample surface (Color figure online)

The results for different temperatures below T_c are shown in Fig. 1b. It can be observed that the dissipation is minimal every 60°. Besides, above T_c we found no measurable angular dependence. This could be understood considering a moving vortex state governed by the effect of the surface barriers and/or the existence of intrinsic-correlated defects in their dynamics. The geometric profiles of the sample in the vortices direction are different if $\varphi = 0°$ or $\varphi = 30°$ (Fig. 1c). In the case of a flat-like profile, as in $\varphi = 30°$, the surface barriers are higher [7], which inhibits the movement of the vortices, making the dissipation lower than in the point-like profile. On the other hand, on the basal plane surfaces, planar defects were observed at $\varphi = 30°$, 90° and 150°. For $\varphi = 30°$, there are defects parallel to the vortices in contrast to the case of $\varphi = 0°$ where no correlated defects are present in this direction. As consequence, the vortices will be more inhibited in their movement if $\varphi = 30°$ on account of either the surface barriers and/or the intrinsic- correlated defects.

In order to clarify this point, we performed a different transport measurement in which the surface barriers were not relevant. In this experiment, a platelet-shaped sample rotates changing the angle φ. The current and the magnetic field were applied in the basal plane (Fig. 2a), and the current direction rotates with the sample. Figure

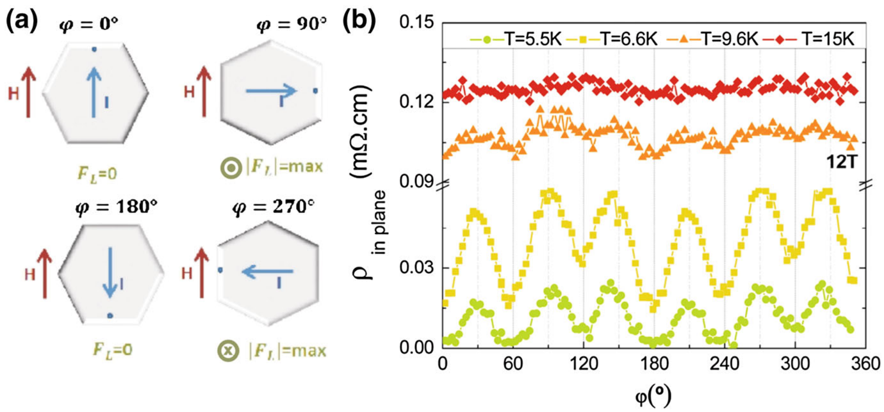

Fig. 2 **a** *sketch* of the sample showing the field, current, and force direction. **b** Resistivity in the basal plane as a function of the angle φ (Color figure online)

2b shows the results for $H = 12\,\mathrm{T}$ and different temperatures. In this case, the results show modulations each 60° but combined with a 180° modulation. In this experimental configuration, the Lorentz force is not constant, since the cross product of the field and the current changes between a maximum and minimum value, crossing zero value (Fig. 2a) for perfect alignment. This force makes the vortices to move, crossing the basal planes of the sample in one direction and, after a half revolution, in the other. If the vortices were rigid rods and we can neglect the intrinsic electronic anisotropy in the plane, the resistivity, ρ, would be proportional to $\sin^2(\varphi)$ [6], explaining the 180° periodicity. Non-zero dissipation for H parallel to the current is attributed to slight misalignment and fluctuations. Concerning the correlated defects, when the angle is $\varphi = 30°$, the vortices move parallel to the defects, in contrast to the case of $\varphi = 0°$. These defects could be channels which facilitate the movement of the vortices.

In summary, both transport experiments show several minima in the dissipation every 60°, which imply a modulation in the critical current with this periodicity. This is due to the correlated defects and not only to surface barriers. This behavior is robust because it was observed in two different samples and with different experimental conditions. In this context, we performed complementary magnetization measurements in a different range ($T = 2$ K and $-0.15\,\mathrm{T} < H < 0.15\,\mathrm{T}$) of the phase diagram. A series of magnetization loops were carried out at different constant values of φ. The magnetic field was applied parallel to the basal plane. The in-plane longitudinal magnetization, M_{L}, and transverse magnetization, M_{T} (parallel and perpendicular to the applied field), were measured with the field always confined to the plane. Moreover, magnetization loops were performed at 20 K with the intention to identify the non-superconducting contribution to the magnetization. We assume that the contributions to the magnetization from the β-FeSe and γ-Fe_7Se_8 are independent and that the magnetization of γ-Fe_7Se_8 is approximately constant in our measurements, provided its high magnetic transition temperature [8].

Figure 3 shows an example of the results obtained in the case of $\varphi = 132°$. In the Bean model, the critical current is proportional to the width of the magnetization

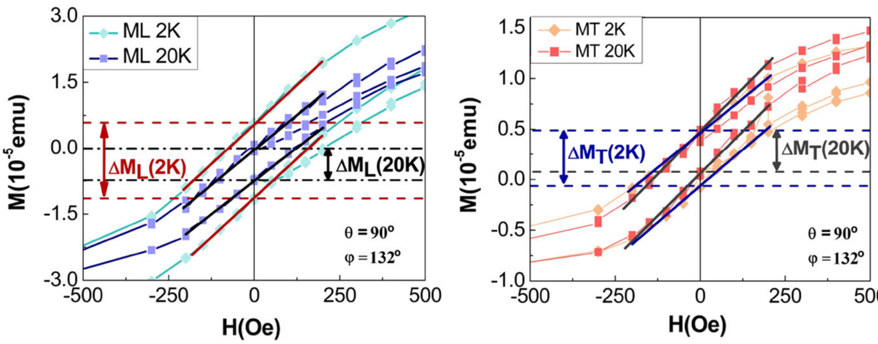

Fig. 3 Longitudinal and transverse magnetization *loops* showing the critera for ΔM definition (Color figure online)

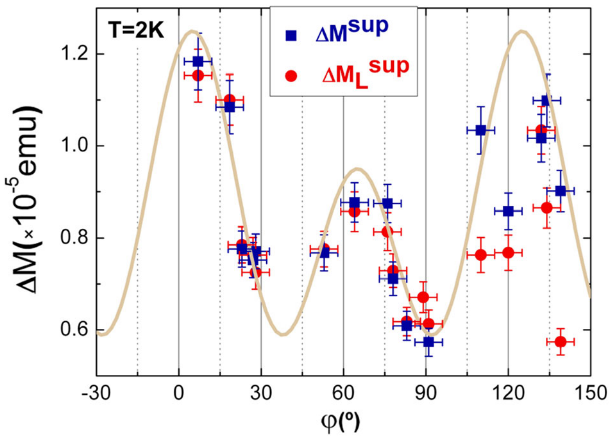

Fig. 4 $|\Delta M^{\text{sup}}|$ (*blue*) and $\Delta M_{\text{L}}^{\text{sup}}$(*red*) versus φ. The *solid line* is a guide to the eye, its modulation is consistent with the data in Fig. 2b (Color figure online)

loop, i.e. $J_{\text{c}} = K\,\Delta M$, with K a geometric constant [9,10]. Then, for each value of φ $\Delta M_{\text{L}}(2K)$, $\Delta M_{\text{L}}(20K)$, $\Delta M_{\text{T}}(2K)$, and $\Delta M_{\text{L}}(20K)$ were obtained in zero applied field as outlined in Fig. 3. The non-superconducting contribution is subtracted:

$$\Delta M_{\text{L}}^{\text{sup}} = \Delta M_{\text{L}}(2K) - \Delta M_{\text{L}}(20K), \qquad \Delta M_{\text{T}}^{\text{sup}} = \Delta M_{\text{T}}(2K) - \Delta M_{\text{T}}(20K) \quad (1)$$

The results for $|\Delta M^{\text{sup}}| = \sqrt{(\Delta M_{\text{L}}^{\text{sup}})^2 + (\Delta M_{\text{T}}^{\text{sup}})^2}$ and $\Delta M_{\text{L}}^{\text{sup}}$ versus φ are show in Fig. 4. For most cases, the differences are not significant between $|\Delta M^{\text{sup}}|$ and $\Delta M_{\text{L}}^{\text{sup}}$; however, for large angles a little discrepancy was observed probably due to a minimal desalination of the sample. $\Delta M_{\text{L}}^{\text{sup}}$, and therefore J_{c}, shows the 60° modulation observed in the transport measurements.

4 Conclusions

We explored the possibility that the correlated defects observed in the basal plane of Fe deficient $Fe_{1-y}Se$ samples, with coexistence between β-FeSe and γ-FeSe phases, condition the movement of the vortices in the dissipative superconducting state. Two different transport experiments suggested the hindering of the vortex mobility with a 60° modulation in-plane making evident the effectivity of the defects. Also the critical currents, deep in the superconducting mixed state, show a 60° modulation compatible with the presence of intrinsic correlated defects.

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References

1. F.-C. Hsu, J.-Y. Luo, K.-W. Yeh, T.-K. Chen, T.-W. Huang, P.M. Wu, Y.-C. Lee, Y.-L. Huang, Y.-Y. Chu, D.-C. Yan, M.-K. Wu, PNAS **105**, 14262–14264 (2008)
2. M.H. Fang, H.M. Pham, B. Qian, T.J. Liu, E.K. Vehstedt, Y. Liu, L. Spinu, Z.Q. Mao, Phys. Rev. B **78**, 224503 (2008)
3. S. Medvedev, T.M. McQueen, I.A. Troyan, T. Palasyuk, M.I. Eremets, R.J. Cava, S. Naghavi, F. Casper, V. Ksenofontov, G. Wortmann, C. Felser, Nat. Mater. **8**, 630–633 (2009)
4. S. Margadonna, Y. Takabayashi, M.T. McDonald, K. Kasperkiewicz, Y. Mizuguchi, Y. Takano, A.N. Fitch, E. Suard, K. Prassides, Chem. Commun. **43**, 5607–5609 (2003)
5. M.L. Amigó, V. Ale Crivillero, D.G. Franco, A. Badía-Majós, J. Guimpel, G. Nieva, J. Phys. Conf. Ser. **507**, 012001 (2014)
6. K. Kadowaki, Y. Somgliu, K. Kitazawa, Supercond. Sci. Technol. **7**, 519–540 (1994)
7. C. Bean, J.D. Livingston, Phys. Rev. Lett. **12**, 14–16 (1964)
8. P. Terzieff, K. Komarek, Monatshefte für Chemie **109**, 1037–1047 (1978)
9. C.P. Bean, Phys. Rev. Lett. **8**, 250 (1962)
10. C.P. Bean, Rev. Mod. Phys. **36**, 31 (1964)