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**Abstract**

The recently discovered Fe–As superconducting materials which show high potential ability to carry current due to their low anisotropy have attracted a great number of attentions to understand their superconductivity mechanism and explore their applications. This paper presents a method to synthesis $SmFeO_{0.75}F_{0.20}As$ polycrystalline by hot press in detail. The magnetization at different temperatures and applied fields obtained by a superconducting quantum interference device are also discussed. In addition, the local magnetization process is presented by magneto-optical imaging technique at the conditions of zero-field-cooling and field-cooling. It is found that the collective magnetization process of the newly discovered Fe–As superconductors is very similar to that of high-$T_c$ cuprates. For instance, the Fe–As superconductors and high-$T_c$ cuprates have the same magnetization features due to strong pining and intergrain weak link. The global supercurrent is significantly lower than local grain supercurrent due to the weak line between the grains.

# Magnetic flux penetration in polycrystalline SmFeO$_{0.75}$F$_{0.2}$As

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The recently discovered Fe–As superconducting materials which show high potential ability to carry current due to their low anisotropy have attracted a great number of attentions to understand their superconductivity mechanism and explore their applications. This paper presents a method to synthesis SmFeO$_{0.75}$F$_{0.20}$As polycrystalline by hot press in detail. The magnetization at different temperatures and applied fields obtained by a superconducting quantum interference device are also discussed. In addition, the local magnetization process is presented by magneto-optical imaging technique at the conditions of zero-field-cooling and field-cooling. It is found that the collective magnetization process of the newly discovered Fe–As superconductors is very similar to that of high-$T_c$ cuprates. For instance, the Fe–As superconductors and high-$T_c$ cuprates have the same magnetization features due to strong pining and intergrain weak link. The global supercurrent is significantly lower than local grain supercurrent due to the weak line between the grains. © *2010 American Institute of Physics*. [doi:10.1063/1.3366606]

## I. INTRODUCTION

The discovery of superconductivity in LaFeO$_{1-x}$F$_x$As (Refs. 1 and 2) with $T_c$=26 K, CeFeO$_{1-x}$F$_x$As (Ref. 3) with $T_c$=41 K, and SmFeO$_{1-x}$F$_x$As with $T_c$=43 (Ref. 4) and 53 K (Ref. 5) has stimulated research activities to understand the fundamental physics in new Fe–As based superconductors, and differ such layered superconducting oxypnictide compounds with high-$T_c$ cuprates. In the new system, the superconductivity is induced by either substitution of doped fluorine on oxygen sites or oxygen deficiency. Doping the parent compounds with fluorine suppresses antiferromagnetic order transition and structural transition from tetragonal to orthorhombic phase at around 150 K. The anomaly observed in temperature dependent resistivity is believed to be associated with both transitions.[6] The anomaly shifts to low temperature and disappears at optimal doping level. Eventually superconductivity emerges.[7] Oxygen vacancies under high pressure can also introduce electrons as fluorine and results in superconductivity.[8] In addition, $T_c$ of Fe–As system depends strongly on the sizes of the rare earth element ions.[8] One of the practical properties of the oxypnictide compounds is exceptionally high upper critical field, $H_{c2}$.[9]

This paper reports synthesis of an oxypnictides compound with both F doping and oxygen deficiency, and studies magnetic properties in the sample at different aspects by a superconducting quantum interference device (SQUID) and magneto-optical imaging (MOI) system. It is found that the collective magnetization process of the newly discovered Fe–As superconductors is very similar to that of high-$T_c$ cuprates. For instance, the Fe–As superconductors and high-$T_c$ cuprates have the same magnetization features due to strong pining and intergrain weak link. The global supercurrent is significantly lower than local grain supercurrent due to the weak line between the grains.

## II. EXPERIMENT DETAIL

Polycrystalline samples with nominal composition SmFeO$_{0.75}$F$_{0.20}$As were synthesized by conventional solid state reaction. The Fe, Fe$_2$O$_3$, and FeF$_2$ powders and presintered powder SmAs were mixed together in argon atmosphere according to the nominal stoichiometric ratio, then ground thoroughly and pressed into cylindrical pellets. The pellets were then coated with thick boron nitride powder, and inserted into a graphite furnace. The pellets were then sintered under a pressure of 4.0 GPa at 1300 °C for 40 h in argon ambient. It should be pointed that the sample composition is different from the most Fe–As superconducting samples, that is, it has nominal 0.2 fluorine doping and 0.05 oxygen depletion. The experimental sample of $1.15 \times 0.875 \times 0.216$ mm$^3$ was cut from the synthesized pellets. Resistivity was measured using PPMS and magnetization was measured using SQUID. The local flux distributions were visualized using homemade MOI system at field cooling (FC) and zero field cooling (ZFC).

## III. RESULTS AND DISCUSSION

Figure 1 shows the x-ray diffraction (XRD) pattern for the ground synthesized pellet. The peaks in the XRD pattern can be well indexed to the tetragonal ZrCuSiAs-type structure though there are some tiny peaks from SmOF, SmAs. The impurity is found to be less than 1.9 wt %. The broad peak from 15° to 40° is caused by glass substrate for power diffraction.
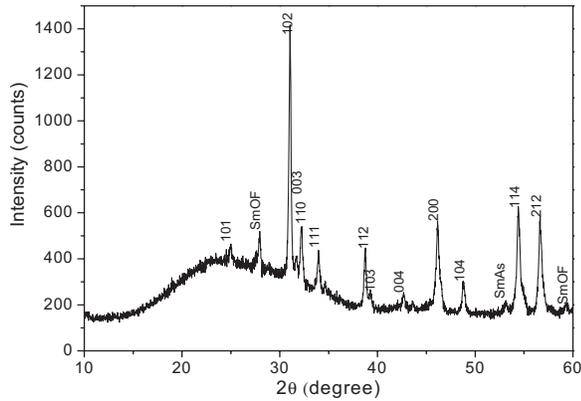
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FIG. 1. XRD pattern for a sample with nominal composition $SmFeO_{0.75}F_{0.20}As$.

Temperature dependence of resistivity obtained by four probe transport measurement at ZFC is shown in Fig. 2. The sample shows metallic behavior, that is, resistance decreases with decreasing temperature until superconducting transition of 45.7 K. The onset $T_c$ of 45.7 K is determined from the intersection of the two extrapolated lines. The residual resistivity ratio $\rho(300\ K)/\rho(52\ K)=3.89$. The resistivity vanished below 41.5 K at zero magnetic fields. The sample shows broad transition since the sample consists of misoriented anisotropic crystalline grains.

Figure 3 shows temperature dependence of magnetization under applied magnetic fields. The paramagnetic background is clearly seen. The magnetic transition under 5 Oe field indicates that superconductivity transition temperature is 45.5 K, which is consistent with $T_c$ obtained from the resistivity measurements. The separation of the magnetization in ZFC and FC indicates that the sample has strong flux pinning. A common feature of type-II cuprates is found in this sample, that is, the onset superconducting transition temperature decreases very gradually with increasing magnetic field whereas the irreversibility field shifts toward lower temperature rapidly, as inset shows. This phenomenon indicates that there is a considerable gap between the upper critical field and the irreversibility field.[10]

Right-hand magnetic hysteresis loops at temperatures between 4.5 and 37.5 K obtained by SQUID are shown in Fig. 4. All loops show slight paramagnetic background. The sample shows significant large hysteresis loops at lower temperatures, though they shrink very quickly with increasing
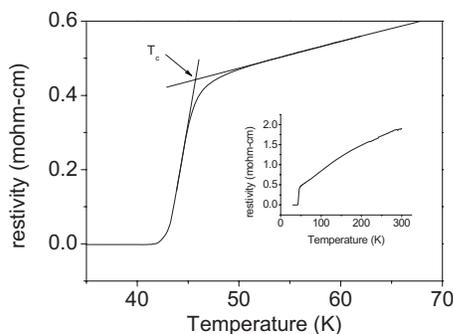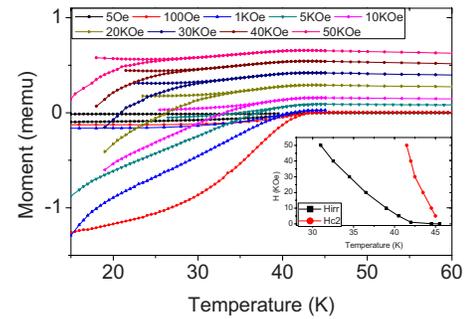


FIG. 3. (Color online) M-T curve under applied magnetic field for ZFC and FC. Inset is $J_c$-T.

temperature. It is suggested that the pinning strength is very strong or intergranular coupling is very good at low temperatures, but it decreases rapidly with increasing temperature. The critical current density, $J_c$, derived from hysteresis loops using extended Bean model is also shown in Fig. 4. It can be seen that at low temperatures, $J_c$ is almost independent in high field region. For example, the $J_c$ reaches maximum 1.3 $MA/cm^2$ at 600 Oe and 0.6 $MA/cm^2$ after 14 kOe at 4.5 K. However, $J_c$ drops quickly with increasing field at high temperatures. The peak effect is also observed at 10, 15, 20, and 25 K. This feature is also reported by other authors.[11,12]

The local magnetic structure of the sample was studied by means of MOI technique. Figure 5 shows the magneto-optical images under ZFC condition. Light microscope inspection does not reveal any cracks in the sample. Meissner state was clearly observed at 4.2 K, as shown in Fig. 5(a),
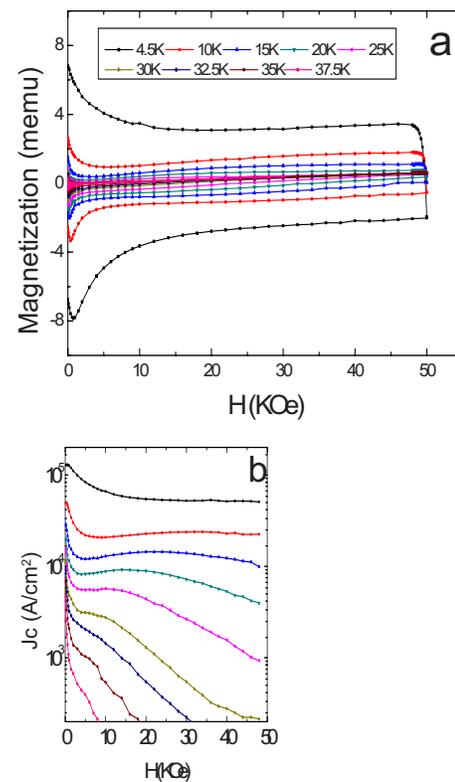




FIG. 2. Temperature dependence of resistivity. The inset is resistivity near $T_c$.

FIG. 4. (Color online) Right-hand temperature dependence of magnetization hysteresis loops (a) and $J_c$-H curve at different temperatures (b).
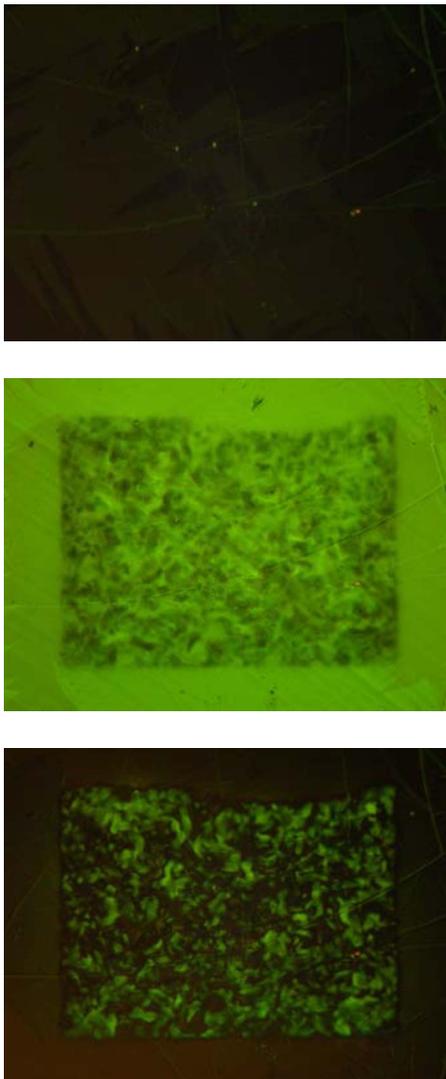
FIG. 5. (Color online) Magneto-optical images on the polycrystalline sample. Top: image taken at external field of 8 Oe showing Meissner state after ZFC. Middle: imaging taken at 160 Oe showing flux penetration easily along intergranular path. Bottom: remanent state after field increased to 800 Oe and decreased to zero.

indicating that the global shielding current flows over the whole sample (the zigzag pattern is magnetic domain in the magneto-optical indicator film). As external field increases, vortex start to penetrate into the sample easily along intergranular path since the pinning strength at intergranular path is more sensitive to the field, but they are still expelled from individual grain, which is shown as black areas in Fig. 5(b). It is the evidence that the pinning strength in the grain is much stronger than that at the intergranular path. As a result, there is no cushion pattern observed in high-$T_c$ superconducting materials.[13] This observation is consistent with magnetization loops at low field region where the magnetization drops rapidly once over the magnetization peak. The easy

vortex motion along the intergranular path was also demonstrated in Fig. 5(c) which was taken at H=0 after external magnetic field increased to 800 Oe. It can be noted that the bright areas in Fig. 5(c) is corresponding to the black areas in Fig. 5(b). The existence of the bright area in Fig. 5(c) indicates that the vortices are pinned in the grains while the vortices at the intergranular path leave the sample along the path. The pinning difference leads to isolated bright area. In summary, the local circulating current around grain is more densely than global shielding current due to the intergrain weak link.[14]

## IV. CONCLUSION

A SmFeO$_{0.75}$F$_{0.20}$As polycrystal with oxygen depletion and fluorine doping is synthesized using hot press apparatus. The sample has superconducting transition at 45.5 K from the magnetization measurement and at 45.7 K from resistivity measurement. Magnetization measurements and magneto-optical images show that the magnetic behavior observed in this work are similar to the high-$T_c$ cuprates in general, and global supercurrent is significantly lower than local grain supercurrent due to relatively weak pining at intergranular path.

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