

Upper critical fields well above 100 T for the superconductor $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ with $T_c=46$ K

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We report specific-heat measurements at magnetic fields up to 20 T on the recently discovered superconductor $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$. The B - T diagram of a polycrystalline $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ sample with $T_c=46$ K was investigated. The temperature dependence of B_{c2} was extracted from the specific-heat curves; the corresponding $B_{c2}(T=0)$ value derived from the Werthamer-Helfand-Hohenberg formula being 150 T. Based on magnetization measurements up to 9 T, a first estimation of the field dependence of the inductive critical current J_c is given. Evidence for granularity is found. The presence of a peak effect is reported, suggesting a crossover in the vortex dynamics, in analogy to the behavior observed in high T_c cuprates.

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I. INTRODUCTION

After the recent discovery of superconductivity in doped quaternary oxyphosphates $\text{REFeAsO}_{1-x}\text{F}_x$, where RE=La, Ce, Pr, Nd, Sm, Gd, this class of compounds has become a subject of considerable experimental and theoretical interests. Soon after the announcement by Kamihara *et al.*¹ of the synthesis of the novel superconductor $\text{LaFeAsO}_{1-x}\text{F}_x$ with the transition temperature T_c of 26 K, T_c values up to 55 K were obtained by substituting La with smaller rare-earth (RE) elements.²⁻⁸ Ren *et al.*⁹ found that under high pressure, superconductivity in the series REFeAsO_{1-x} can be induced by oxygen vacancies instead of F doping. The substitution of oxygen by fluorine, as well as the presence of oxygen vacancies, has the effect of introducing electrons into the REFeAsO system. Kamihara *et al.*¹ found no trace of superconductivity when substituting La^{3+} with Ca^{2+} , i.e., introducing holes into the system. On the other hand, Wen *et al.*¹⁰ found the evidence of superconductivity through hole doping in the system $\text{La}_{1-x}\text{Sr}_x\text{FeAsO}$ with $T_c=25$ K.

Several theoretical works proposed multiband superconductivity arising from Fe-As electronic bands,¹¹⁻¹⁴ suggesting a pairing mechanism of spin fluctuation type. Indeed, the undoped REFeAsO exhibits a spin-density wave (SDW) transition at 130–150 K.¹⁵⁻¹⁸ This transition has been detected by various techniques, e.g., electrical resistivity,¹⁵ magnetic susceptibility,¹⁶ specific heat,¹⁷ Hall coefficient,¹⁵ and Seebeck coefficient.¹⁸ It was proposed that superconductivity is induced by the suppression of the SDW state (through electron or hole doping), T_c being enhanced after the substitution of La by smaller rare-earth atoms, which also leads to a reduced elementary cell.⁹

In the present paper we report a study of the magnetic phase diagram B - T for a polycrystalline $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ sample by means of high-field specific-heat measurements and by magnetization measurements. The sample was prepared in the Hefei National Laboratory² and some details of

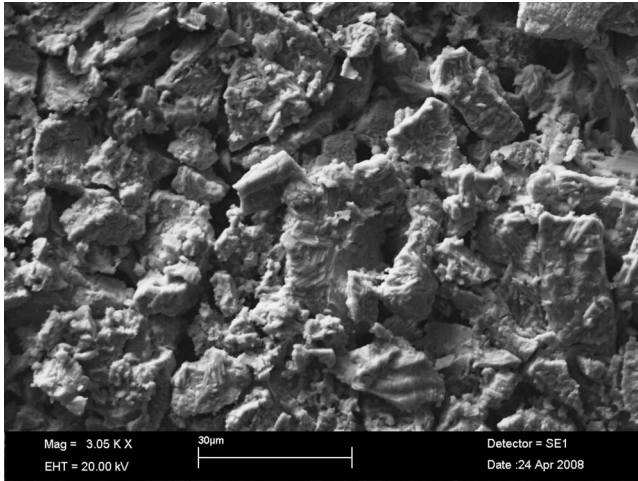
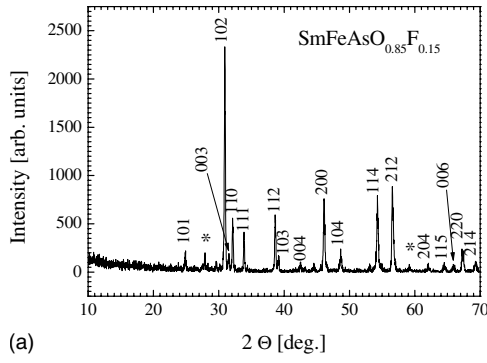
sample preparation are given in Sec. II. Specific-heat measurements at different magnetic fields up to 20 T allowed the determination of the T_c distribution in the sample by deconvolution (Sec. III). These measurements were used for determining the $B_{c2}(T)$ curve up to 20 T. In Sec. IV an estimation of the inductive critical current density J_c is given based on magnetization measurements. The first observation of a peak effect in this family of compounds is reported.

II. Sample preparation

The polycrystalline sample with nominal composition $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ was synthesized by conventional solid-state reaction using SmAs, SmF_3 , Fe, and Fe_2O_3 as starting materials.² SmAs was obtained by reacting Sm chips and As pieces at 600 °C for 3 h, followed by 5 h at 900 °C. The raw materials were mixed and pressed into pellets, which were wrapped into a Ta foil, sealed in an evacuated quartz tube, and annealed at 1160 °C for 40 h. The peaks of the x-ray diffraction (XRD) pattern correspond to the tetragonal $P4/nmm$ structure with $a=0.3943$ nm and $c=0.8514$ nm [Fig. 1(a)], SmOF being present as impurity phase. As shown by scanning electron microscopy (SEM) analysis [Fig. 1(b)], the sample is porous and the conglomerate particle size varying between 5 and 30 μm .

III. Specific-heat measurements

The specific-heat measurements were performed on the polycrystalline sample of $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ described above with a homemade calorimeter, using the thermal relaxation technique.¹⁹ The sample, with a mass of 44.950 mg, was mounted on a sapphire plate. A Cernox thermometer with four contacts made of phosphor bronze was on the other side of the plate. The whole setup, including two additional Cernox thermometers and a heater, was sealed inside a vacuum



(b)

FIG. 1. (a) X-ray diffraction pattern for the sample with nominal composition $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$; SmOF impurity phase is indicated with an asterisk. (b) Scanning electron microscope images for the $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ polycrystalline sample showing conglomerate particle sizes varying between 5 and 30 μm .

chamber. Both thermometers were calibrated in magnetic field. Magnetic fields up to 20 T were generated by a superconducting laboratory magnet with 64 mm bore from Bruker BioSpin. The temperature in the variable-temperature insert (VTI) varied between 2 and 50 K.

The variation of C/T versus T in zero magnetic field and at $B=20$ T is shown in Fig. 2. The sample exhibits a broad superconducting transition whose midpoint at $B=0$ is 45.5 K. The onset of the superconducting transition from AC susceptibility is found around 46 K [inset (a) of Fig. 2].

As shown in the inset (b) of Fig. 2, there is an anomaly in the specific-heat curve at temperatures below ~ 15 K. This behavior was already observed by Ding *et al.*¹⁷ and was ascribed to the antiferromagnetic ordering of the Sm^{3+} ions, in analogy to what happens in the electron-doped superconductor $\text{Sm}_{2-x}\text{Ce}_x\text{CuO}_{4-\delta}$.²⁰

The measured specific heat C_{tot} above 20 K can be rewritten as

$$C_{\text{tot}}(B, T) = C_s(B, T) + C_n(T), \quad (1)$$

where $C_s(B, T)$ is the superconducting contribution and $C_n(T)$ is the normal background due to phonons and normal electrons. The main difficulty consists in the separation of

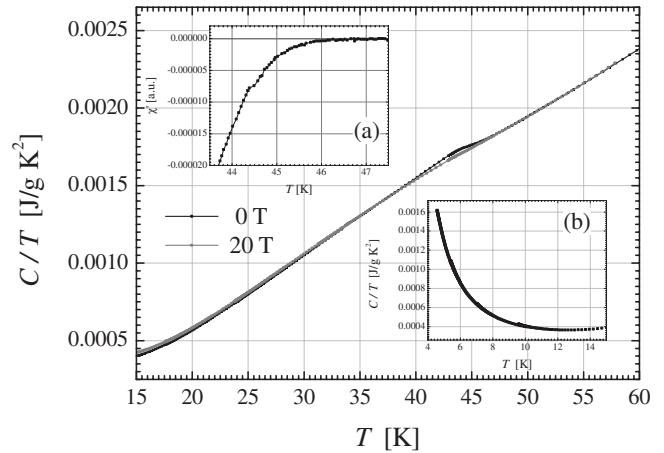


FIG. 2. C/T versus T at zero field (black squares) and 20 T (gray circles). Inset (a): superconducting transition from AC susceptibility. Inset (b): low-temperature specific-heat anomaly, related to the magnetic ordering of the Sm^{3+} ions (Ref. 17).

the singular part $C_s(B, T)$ from the background. For the background we used a quadratic function $C_n(T) = a + bT + cT^2$, and the resulting $C_s(B, T)$ curves are reported in Fig. 3 for $B=0, 6, 10, 14,$ and 20 T. In the following we characterize the superconducting transition by T_{mid} , the midpoint of the superconducting jump; the onset temperature being not well defined. Another point is T_{max} which corresponds to the maximum value of C/T at the superconducting jump (Fig. 3) and is close to the average bulk T_c within the sample. The inhomogeneity of the sample gives rise to a broad specific-heat anomaly, which reflects a wide distribution of T_c . The calorimetric superconducting transition is broadened when a magnetic field is applied.

The distribution of T_c in the present sample was determined using the previously reported method of analysis based on the deconvolution of the specific-heat data.²¹ For a distribution of T_c from 0 K to $T_{c, \text{max}}$ with a weight function

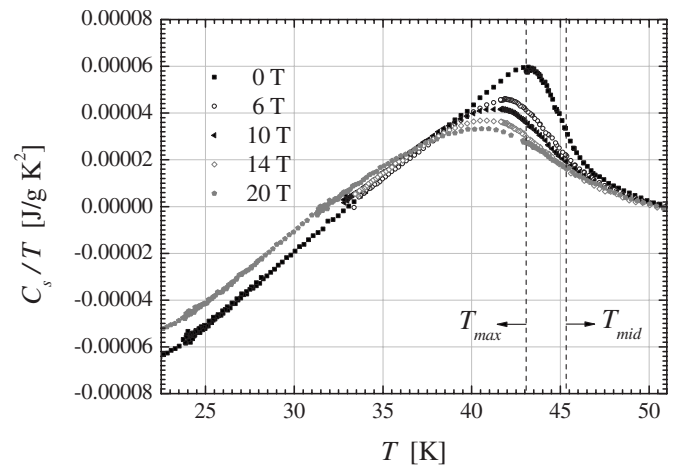


FIG. 3. Superconducting contribution to the specific heat for $B = 0, 6, 10, 14,$ and 20 T. The temperatures corresponding to T_{mid} , the midpoint of the calorimetric transition and to T_{max} , are the maximum of C/T at the superconducting jump are indicated for the curve at zero field.

$f(T_c)$, the electronic specific heat $C_e(T)$ and the entropy $S_e(T)$ at a certain temperature T are given by:

$$C_e(T) = \int_T^{T_{c, \max}} f(T_c) C_{es}(T, T_c) dT_c + \int_0^T f(T_c) C_{en}(T, T_c) dT_c, \quad (2)$$

$$S_e(T) = \int_T^{T_{c, \max}} f(T_c) S_{es}(T, T_c) dT_c + \int_0^T f(T_c) S_{en}(T, T_c) dT_c. \quad (3)$$

The first term of the right side of the equations is the specific heat (Eq. (2)) and the entropy (Eq. (3)) of electrons in the superconducting state, while the second part is the specific heat (Eq. (2)) and the entropy (Eq. (3)) of electrons in the normal state. Here two assumptions are made: in the sample, there is only one normal-state electronic specific-heat coefficient for all the sample parts with different T_c :

$$C_{en}(T, T_c) = C_{en}(T) = \gamma T; \quad (4)$$

the electronic specific heat in the superconducting state can be described using a generalized two-fluid model:

$$C_{es}(T, T_c) = n \gamma T_c (T/T_c)^n. \quad (5)$$

We finally obtain

$$\int_0^T f(T_c) dT_c \equiv F(T) = \frac{n S_e(T) - C_e(T)}{(n-1) \gamma T}. \quad (6)$$

$F(T)$ is the integral of the distribution between 0 and T and thus represents the fraction of the sample with $T_c \leq T$. The normal-state electronic specific-heat coefficient γ and the parameter n , which in the original Gorter-Casimir model is equal to 3, are chosen in order to obtain

$$F(T_{c, \max}) \equiv \int_0^{T_{c, \max}} f(T_c) dT_c = 1.$$

In terms of the notation introduced in Eq. (1), Eq. (6) can be rewritten as

$$\int_0^T f(T_c) dT_c \equiv F(T) = \frac{n S_e(T) - C_e(T)}{(n-1) \gamma T} = \frac{n S_s(B, T) - C_s(B, T)}{(n-1) \gamma T} + 1, \quad (7)$$

where

$$C_s(B, T) = C_e(T) - \gamma T, \quad S_s(B, T) = \int_0^T \frac{C_s(B, T')}{T'} dT', \quad (8)$$

In Fig. 4 we report the T_c distributions measured at $B=0$ and 10 T. The reduction of the area below the curve at 10 T corresponds to the reduced superconducting volume due to the magnetic-flux penetration. Two reasons can be invoked for the increasing width of the T_c distribution with applied magnetic field: (a) the broadening of the upper critical field (B_{c2}) line in the B - T diagram as an effect of the fluctuations,^{22,23} and (b) the presence of a distribution of B_{c2} , due to the intrinsic anisotropy of B_{c2} in the iron based lay-

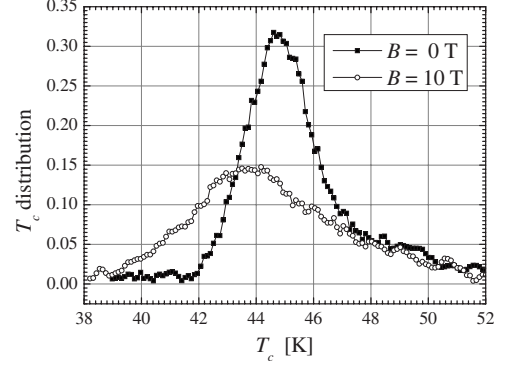


FIG. 4. Distribution of T_c at zero field and at $B=10$ T, obtained by the deconvolution of the calorimetric data.

ered superconductors and the random orientation of the grains within the sample.

The temperature dependence of B_{c2} was determined from the points T_{mid} and T_{max} in the measured specific-heat curves (Fig. 3); these values being better defined than T_{onset} . Compared to other techniques, the calorimetric determination of B_{c2} provides information on the bulk properties of the sample. On the other hand, the results from electrical transport measurements are limited to the percolation paths of the current and the inductive measurements (dc and ac magnetizations) are influenced by magnetic shielding effects.

The slope $dB_{c2}/dT|_{T_c}$ extracted from the $B_{c2}(T)$ data for T_{max} is ~ 5 T/K. The corresponding $B_{c2}(T=0)$ value derived from the Werthamer-Helfand-Hohenberg (WHH) formula is $0.693 T_c |dB_{c2}/dT|_{T_c} \approx 150$ T, thus exceeding the BCS paramagnetic limit $B_p \approx 1.84 T_c \approx 85$ T.

For $\text{LaFeAsO}_{1-x}\text{F}_x$, Hunte *et al.*²⁴ have interpreted the observed upward curvature of $B_{c2}(T)$ as a signature of multi-band superconductivity in this family of compounds, which could lead to a significant raise of $B_{c2}(T=0)$ with respect to the WHH extrapolation. The upper critical field in $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ exhibits a very weak temperature depen-

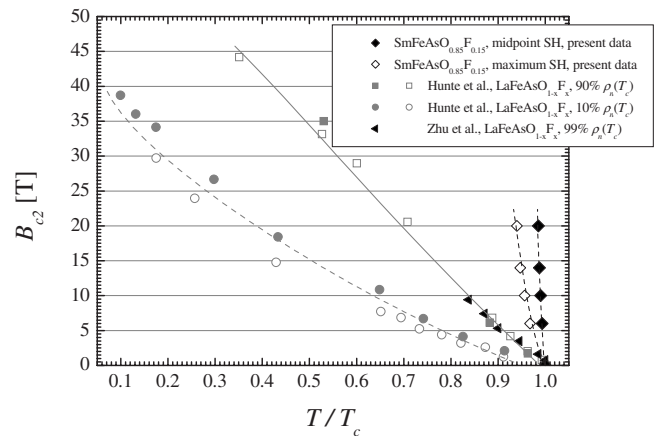


FIG. 5. Upper critical field B_{c2} versus T/T_c for the $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ sample as determined from the midpoint T_{mid} (solid diamonds) and the maximum T_{max} (open diamonds) of the superconducting transition in the specific-heat curves. The B_{c2} values for $\text{LaFeAsO}_{1-x}\text{F}_x$ in Refs. 24 and 25 are also reported for comparison.

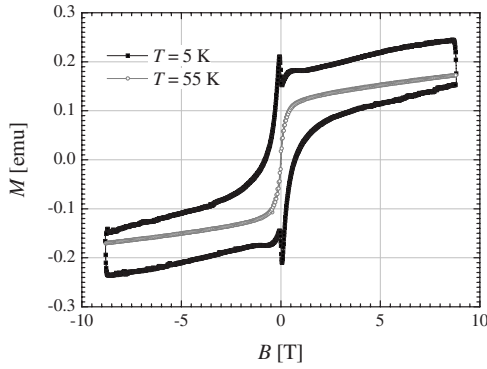


FIG. 6. Magnetic moment as a function of the applied magnetic field B at $T=5$ K (solid squares) and 55 K (open circles). The $M(B)$ curve at 5 K is the superposition of a superconducting contribution and a ferromagnetic background.

dence close to T_c as in Fig. 5. Our data of $B_{c2}(T/T_c)$ and the values measured on $\text{LaFeAsO}_{1-x}\text{F}_x$ by Hunte *et al.*²⁴ and Zhu *et al.*²⁵ are reported in Fig. 5.

IV. Inductive measurements

Magnetization loops $M(B)$ were measured with a vibrating sample magnetometer (VSM). The magnetic field was swept up to 8.8 T and loops were recorded every 2.5 K between 5 and 55 K. The experimental $M(B)$ curves can be understood as the superposition of a superconducting contribution and a ferromagnetic background (Fig. 6). The intensity of the signal for the two contributions in the temperature range 5–40 K is comparable.

Measurements performed above the superconducting T_c show a narrow ferromagnetic hysteresis with negligible temperature dependence, probably due to the unreacted Fe and Fe_2O_3 . The superconducting contribution in the temperature range 5–40 K has been isolated treating the ferromagnetic signal as a temperature independent background. The resulting loops are reported in Fig. 7 for $T=5$ K (a) and 25 K (b).

In presence of flux pinning the sign of the superconducting magnetization depends on the direction of the field sweep, thus resulting in a hysteresis loop between the ascending and the descending branches of the magnetization. The critical current density J_c can be obtained from the irreversible magnetization $\Delta M = (M^+ - M^-)/2$, where $M^+(M^-)$ is the branch of the magnetization for $dB_a/dt < 0$ ($dB_a/dt > 0$). B_a is the applied field, swept at a constant rate: in our case $|dB_a/dt| = 2$ T/min. The value ΔM is proportional to J_c times the length scale of the current flow. The low value of the superconducting magnetic moment for the examined sample suggests the presence of “weak links,” i.e., the current does not circulate through the entire sample. Evidence for electromagnetic granularity was already reported in polycrystalline $\text{LaFeAsO}_{1-x}\text{F}_x$.²⁶ In our case, this was confirmed from the fact that the irreversible magnetization ΔM does not change reducing the sample size, thus indicating that the current carrying length scale is smaller than the sample dimensions. A further confirmation comes from the double transition present in the AC susceptibility curve (Fig. 8).

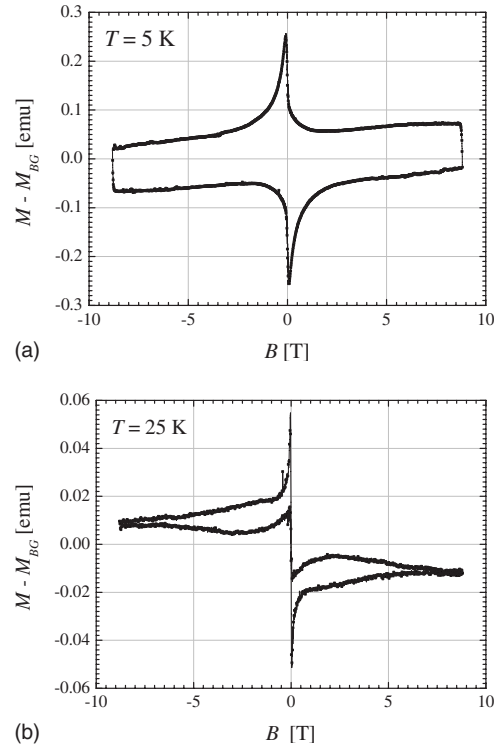


FIG. 7. Superconducting contribution to the magnetic moment as a function of B at (a) $T=5$ K and (b) 25 K. The superconducting signal was isolated from the ferromagnetic background determined above T_c .

In analogy to high T_c compounds,²⁷ the observed granular behavior can be ascribed to nonsuperconducting grain boundaries, to local inhomogeneity, or grain misalignment. A possibility is the loss of fluorine at the grain boundaries as a consequence of the heat treatment performed under vacuum.

In order to calculate the critical current density $J_c(B, T)$ we assumed the current flowing only within the grains. The Bean formula used to calculate J_c from the measured irreversible magnetic moment Δm is

$$J_c = 3 \frac{\Delta M}{\langle R \rangle} = 3 \frac{\Delta m}{V \langle R \rangle}. \quad (9)$$

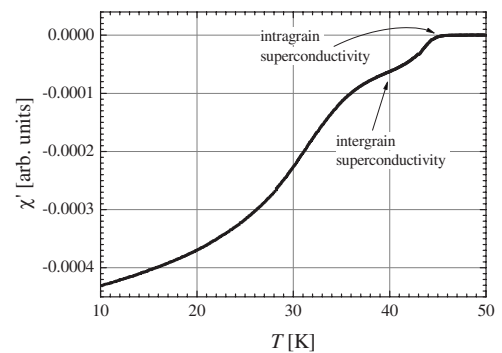
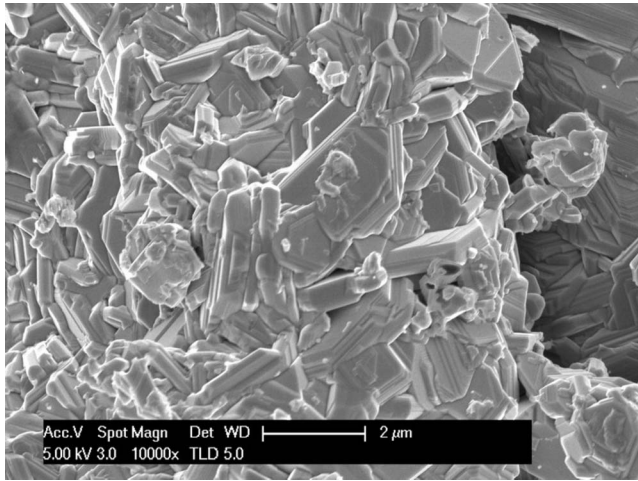
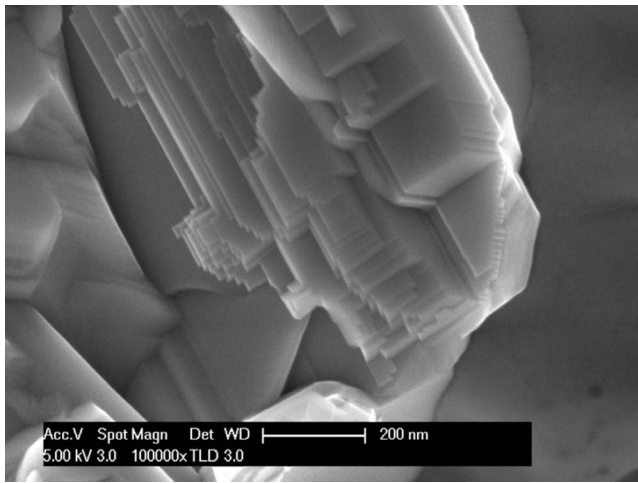


FIG. 8. Real part of the AC susceptibility χ' versus T : the double transition in the curve is determined by the crossover from intragrain to intergrain superconductivity, as a consequence of the granularity of the sample.



(a)



(b)

FIG. 9. (a) FE-SEM image showing grain sizes varying between less than 0.1 and 2–3 μm . (b) Magnification of a submicrometric grain.

No data about the real current length scale $\langle R \rangle$ is known at present, and the determination of J_c is thus uncertain. In the present case, an average conglomerate particle size of $\langle R \rangle \approx 10 \mu\text{m}$ can be estimated from SEM measurements, the value of J_c obtained by setting this value in Eq. (9) can thus only be considered as a lower limit. Substantially higher J_c values can be obtained when considering decoupled grains inside the conglomerate particles. We have found from field emission SEM (FE-SEM) analysis that the grain size varies from 50–100 nm to 2–3 μm (Fig. 9). Therefore, an upper limit for the intragrain J_c value can be estimated if we assume a grain size of $\sim 0.1 \mu\text{m}$, which is roughly of the same order of magnitude as Nb_3Sn , V_3Ga , NbTi , and MgB_2 .

Figure 10 shows the inductive J_c values from 5 to 40 K plotted versus the magnetic field. The $J_c(B)$ values have been represented for both $\langle R \rangle = 0.1$ and $10 \mu\text{m}$, a more precise answer will be given once the correct length scale of these compounds will be determined.

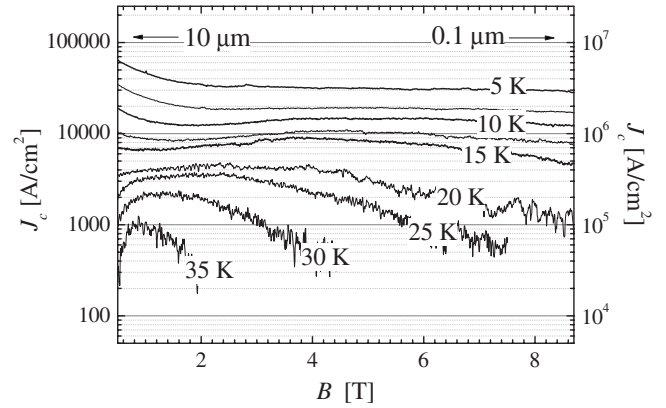


FIG. 10. Inductive critical current density J_c versus B from 5 to 40 K. J_c has been determined for $\langle R \rangle = 10 \mu\text{m}$ (left axis) and $\langle R \rangle = 0.1 \mu\text{m}$ (right axis), the current carrying length scale within the sample being not measured yet.

A. Peak effect and B - T diagram

As shown in Figs. 7(b) and 8, the irreversible magnetization and thus the $J_c(B)$ curves exhibit the peak effect for $T > 10$ K. In many cuprates^{28–31} and in some low T_c superconductors such as Nb_3Sn ,³² CeRu_2 , NbSe_2 ,³³ V_3Si ,³⁴ and MgB_2 ,^{35,36} the magnetization peak effect has been observed and several models have been proposed to explain the increased pinning as the magnetic field increases to the peak field B_{peak} .

In the case of low T_c superconductors, the peak effect appears approaching the B_{c2} line. Close to B_{c2} and thus to the vortex melting, thermal fluctuations are responsible for the softening of the shear modulus in the quasiordered Bragg glass state of the vortex matter.³⁷ This leads to a better accommodation of the vortices on the pinning centers, with the consequent formation of a highly disordered vortex glass corresponding to a sharp increase in the critical current density.

In the highly anisotropic high T_c cuprates, the mixed state can be described in terms of two-dimensional (2D) vortices confined in the CuO_2 planes and coupled through the separating layers by Josephson and electromagnetic coupling.^{38,39} The characteristic length of electromagnetic interaction is the in-plane penetration length λ_{ab} . In the case of Josephson coupling, the characteristic length is given by $\lambda_J = \Gamma d$, where Γ is the anisotropy value and d is the distance between consecutive CuO_2 planes. In that respect, the magnetization peak effect appears in the same region of the B - T diagram corresponding to the transition from a lattice of three-dimensional (3D) flux lines to a lattice of 2D pancake vortices and it results

$$B_{\text{peak}} = \frac{\Phi_0}{\ell^2}, \quad (10)$$

ℓ being the shorter length between λ_{ab} and λ_J .^{30,39,40} In optimally doped YBCO with $\Gamma < 10$, the peak effect is associated to a crossover from elastic to plastic creep in the vortex motion.³¹

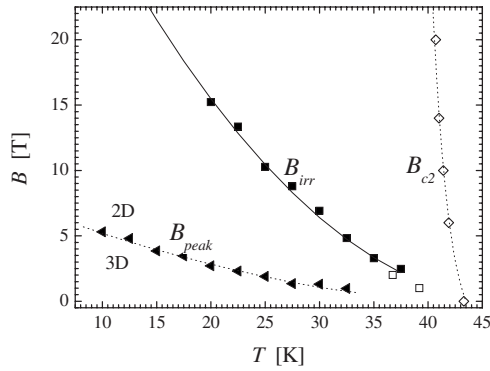


FIG. 11. B - T phase diagram for $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$. Data points in the $B_{c2}(T)$ curve were determined from specific-heat measurements. The irreversibility line and the $B_{\text{peak}}(T)$ line were extracted from magnetization measurements. The solid line is a fit $B_{\text{irr}} \propto [1 - (T/T_c)]^2$.

In $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ the peak in magnetization appears for values of the applied magnetic field much lower than B_{c2} . Our scenario is in principle similar to what is proposed for high T_c superconductors. Due to the layered structure of the quaternary oxyphnictides, an anisotropic behavior of the electronic properties is expected. For $\text{LaFeAsO}_{1-x}\text{F}_x$ Singh and Du⁴¹ predicted a resistivity anisotropy $\rho_c/\rho_{ab} \sim 15$. Therefore, the observed magnetization peak effect can be ascribed to a 2D-3D crossover with consequent enhanced vortex pinning. Due to the random orientation of the grains within the sample, this crossover holds only in the grains whose ab plane is not parallel to the applied magnetic field. Recent muon spin rotation experiments yield a penetration length $\lambda_{ab}(T=0) \approx 200$ nm.⁴² Using the expression for the peak field in Eq. (10), we find a crossover length spanning between 20 and 50 nm in the temperature range 10–35 K, suggesting that Josephson interactions have to be taken into account.

In Fig. 11 we show the B - T phase diagram for $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$. The maximum of the magnetization peak B_{peak} reveals the crossover in the dimensionality of the vor-

tex lattice. The irreversibility field B_{irr} was determined from the extrapolation of the Kramer plot, i.e., $\Delta M^{0.5} B^{0.25}$ versus B at different temperatures. The open squares in Fig. 11 correspond to the separation of the zero field cooled (ZFC) and the field cooled (FC) $M(T)$ curves, measured at $B=1$ and 2 T. The irreversibility line follows a $[1 - (T/T_c)]^2$ law, as predicted for the melting line in the collective pinning theory.⁴³ The upper critical field B_{c2} corresponds to the maximum of the superconducting jump in the specific-heat measurements.

V. CONCLUSIONS

The temperature dependence of the upper critical field B_{c2} for the recently discovered $\text{SmFeAsO}_{0.85}\text{F}_{0.15}$ superconductor was determined from specific-heat measurements up to 20 T. The high value of the slope $|dB_{c2}/dT|_{T_c} \approx 5$ T/K suggests an extrapolated $B_{c2}(T=0)$ around 150 T using the WHH formula.

We propose a range of values for the critical current density J_c , extracted from magnetization measurements, taking into account different length scales ranging between 10 and 0.1 μm . The granular behavior of our sample renders the correct estimation of J_c quite difficult, due to the uncertainties in determining the current carrying length within the sample.

The first observation of a peak effect in this family of compounds is also reported. In analogy to the behavior observed in high T_c cuprates, this could indicate a 2D-3D crossover of the vortex lattice.

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