

High-pressure growth of fluorine-free SmFeAsO_{1-x} superconducting single crystals

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Abstract

Fluorine-free SmFeAsO_{1-x} single crystals of a nominal composition with $x = 0.15$ were grown under the pressure of 3.3 GPa and at 1350–1450 °C by using the self-flux method. Plate-shaped crystals of a few–200 μm in lateral size were obtained. The resistively determined superconducting transition of a single crystal was at about 53.5 K, with a narrow resistive transition width of 0.5 K. The detailed crystal structure was analyzed by using synchrotron-irradiated x-ray diffractometry and high-resolution scanning transmission electron microscopy. A sharp transition, a low residual resistivity, and a large residual resistivity ratio indicate the high quality of our single crystals. The temperature and magnetic field dependences of the magnetization also confirmed the absence of extrinsic impurity phases.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

The recent discovery [1] of a new class of Fe-based superconductors, $\text{REFeAsO}_{1-x}\text{F}_x$ (RE = rare-earth elements), has attracted much attention due to analogies with cuprates: a relatively high superconducting transition temperature (T_c), a two-dimensional layered structure, and the superconducting phase located close to the magnetic instability. Despite the strongly motivated efforts for growing single crystals to understand their basic superconducting nature, up to date only a few groups have reported the successful growth of single crystals in this class of fluorine-doped $\text{REFeAsO}_{1-x}\text{F}_x$ [2–5]. But some important issues are still unsolved even using $\text{REFeAsO}_{1-x}\text{F}_x$ single crystals. For instance, multi-band nature [5–12] was observed as predicted

theoretically [13–18]. In addition, an unusual $\pm s$ -wave pairing symmetry [13, 19–23] was predominantly proposed by theories, but a conventional s-wave symmetry was observed in several experiments [7, 12, 24, 25] in $\text{REFeAsO}_{1-x}\text{F}_x$ (RE = Sm, Nd, Pr) single crystals. Thus, basic consensus has yet to be reached on a number of key issues of the FeAs superconductors and high-quality $\text{REFeAsO}_{1-x}\text{F}_x$ single crystals are still highly demanded.

The large difference in the melting temperatures of the starting compounds (for instance, 1565 °C for Fe_2O_3 , 1530 °C for Fe, 1306 °C for SmF_3 , 993 °C for FeAs, and 900 °C for SmAs) is one of the main obstacles to single-crystal growth. Also the high vapor pressure of As can cause the loss of the element during the growth and raises safety concerns. Thus, we adopted a high pressure synthesis technique that has been routinely used by us for the crystal growth of MgB_2 , $\text{Sr}_{1-x}\text{La}_x\text{CuO}_2$, and MgCNi_3 [26–28]. And,

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we targeted the compound of fluorine-free $\text{REFeAsO}_{1-x}\text{F}_x$ for the following reasons. Firstly, the multi-component nature of $\text{REFeAsO}_{1-x}\text{F}_x$, which contains five elements, complicates the establishment of the optimal growth conditions of single crystals. In contrast, high-quality single crystals of the double-layered $\text{A}_{1-x}\text{K}_x\text{Fe}_2\text{As}_2$ ($\text{A} = \text{Ba}, \text{Sr}, \text{Ca}$) superconductors (consisting of only four elements) have been successfully grown by many groups [29–31]. Secondly, the superconducting transition temperature T_c in $\text{REFeAsO}_{1-x}\text{F}_x$ single crystals grown under high pressure exhibits a rather wide distribution of values. This T_c depends on the doping concentration, which is determined by both the oxygen and fluorine content [2–4]. Thus, reducing the content of the negative ions to keep the doping concentration stable may lead to a narrowed distribution of T_c . Finally, it has been theoretically suggested that producing oxygen vacancies instead of fluorine doping induces more electron carriers in the FeAs layers. This can lead to the lattice shrinkage and the enhancement of the electron density of states, which favor a higher T_c [32, 33]. In this paper, we report on the growth process of SmFeAsO_{1-x} single crystals, eliminating fluorine elements with $T_c \sim 53.5$ K, by using an ultra-high-pressure technique and present an in-depth characterization of the resulting crystals.

2. Experiments

For the growth of single crystalline SmFeAsO_{1-x} , pure SmAs powder was prepared by reacting Sm chips and As pieces at 600°C for 5 h and then 900°C for 10 h in an evacuated quartz tube. Starting compounds of SmAs, Fe_2O_3 , and Fe for SmFeAsO_{1-x} were well ground to the nominal composition of $x = 0.15$. The mixture was pressed into a pellet, which was in turn put into a boron-nitride crucible. Then, the pellet sealed with the boron-nitride container was placed in a cubic pyrophyllite cell equipped with a carbon heater. These processes were carried out in an argon-gas atmosphere for safety against toxic arsenic and to protect the compounds from contamination. The whole assembly was pressurized up to 3.3 GPa at room temperature by using a 14 mm cubic multi-anvil-type press. The heat treatment at $1350\text{--}1450^\circ\text{C}$ lasted for 8–10 h and was followed by rapid cooling to room temperature. Finally, the plate-shaped single crystals ranging in size up to $200\ \mu\text{m}$ were selected from the remnant flux after mechanically crushing the final bulk.

Optimum growth conditions were tuned by varying the pressure, the heating temperature, the reaction time, and the cooling rate. In general, slow cooling is favorable to grow sizable single crystals. However, in the case of SmFeAsO_{1-x} , we found that the single phase formed at the reaction temperature tended to decompose into several different phases, such as Fe_xAs_y and $\text{Sm}_x\text{Fe}_y\text{As}_z$ during slow cooling. This was confirmed by using energy dispersive x-ray spectroscopy after synthesis. Thus, in our method, to prevent the decomposition of the single phase, the long heat treatment is followed by rapid cooling. As a consequence, most of single crystals turned out to be smaller than $200\ \mu\text{m}$ in their lateral size. Nonetheless, the size of crystals is sufficient

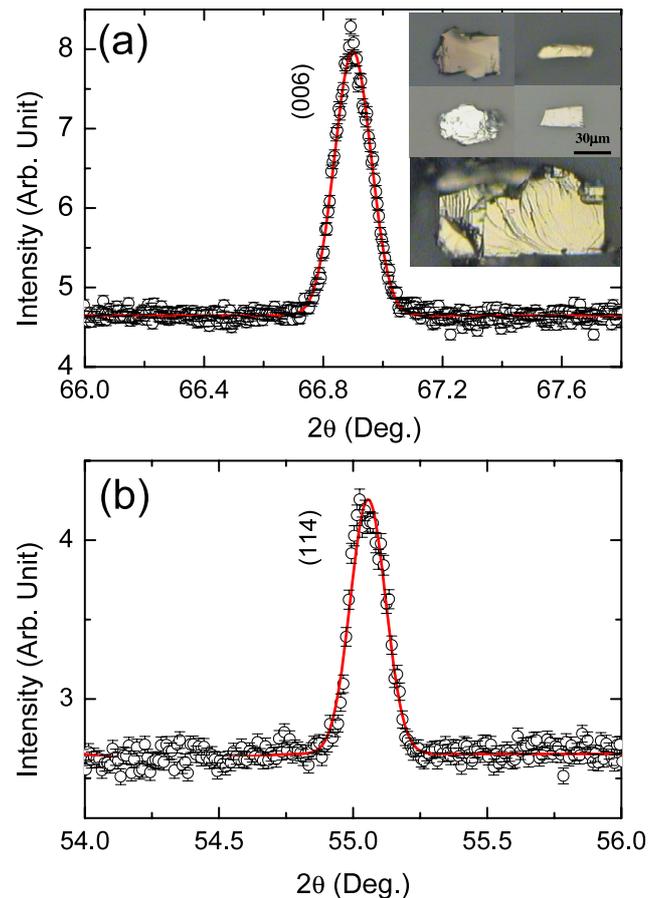


Figure 1. Synchrotron-irradiated x-ray diffractometry (XRD) spectra of single crystalline SmFeAsO_{1-x} with nominal composition of $x = 0.15$. The θ - 2θ scan of (a) (006) and (b) (114) peaks display excellent crystallinity. Solid lines are Gaussian best-fit curves. Inset: optical microscopy image of SmFeAsO_{1-x} single crystals grown from the self-flux method under high pressure.

for transport measurements [34, 35] as well as for key crystal characterizations presented in this study.

The shape and the surface morphology of the crystals were examined using optical microscopy and field-emission scanning electron microscopy (FE-SEM), respectively. The detailed crystal structure was investigated by using synchrotron-irradiated x-ray diffractometry (XRD) and Cs-corrected high-resolution scanning transmission electron microscopy (HR-STEM). The temperature dependence of resistivity of SmFeAsO_{1-x} single crystals was measured using a standard four-probe technique. The magnetization was measured by using a superconducting quantum interference device (SQUID) magnetometer. For all the measurements in this study, single crystals from a growth batch were used, which revealed almost the same superconducting and structural properties.

3. Results and discussion

Optical microscopic images of SmFeAsO_{1-x} single crystals with nominal composition of $x = 0.15$ are shown in the inset of figure 1(a). The grown crystals ranged $5\text{--}200\ \mu\text{m}$ in

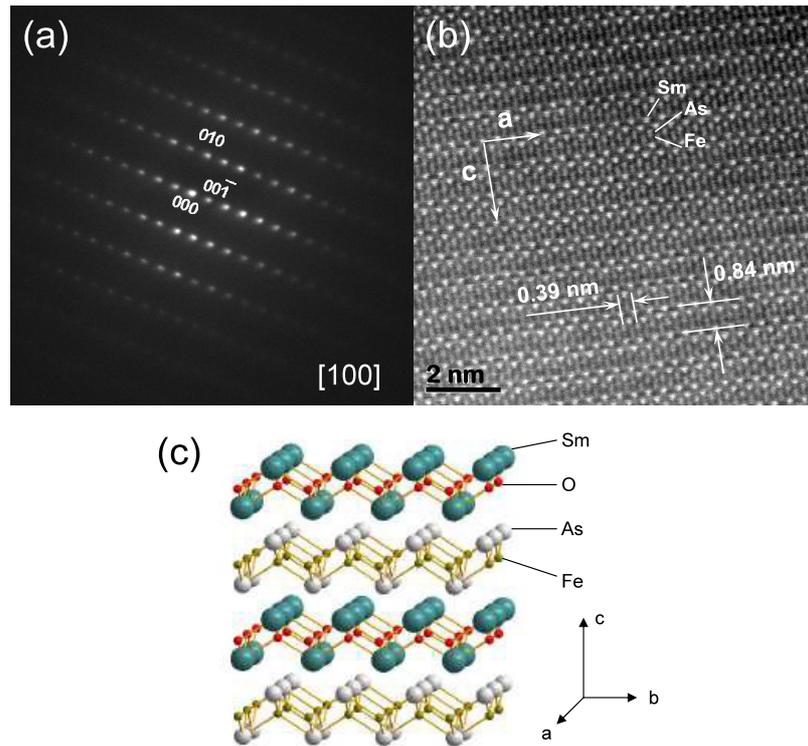


Figure 2. Cs-corrected high-resolution scanning transmission electron microscopy (HR-STEM) images of a SmFeAsO_{1-x} single crystal. (a) A selected-area electron diffraction pattern for the beam direction of $[100]$ and (b) a high-angle-annular-dark-field scanning transmission electron microscopy (HAADF-STEM) image, which show a well-defined structure of alternating SmO and FeAs layers. The images in (a) and (b) are consistent with the tetragonal ZrCuSiAs-type structure of the SmFeAsO compound as schematically illustrated in (c).

lateral size and $0.5\text{--}20\ \mu\text{m}$ in thickness. Most of the crystals were found to have irregular plate-like shapes with flat and shiny surfaces, with smaller crystals displaying cleaner and flatter surfaces. The largest crystal shown in the inset of figure 1(a) reveals a rectangular shape and a terraced surface, which presumably reflects the underlying layered tetragonal structure of the crystal. The crystal orientation was determined by using synchrotron-irradiated x-ray diffractometry (XRD) with $6 + 2$ circle diffractometer on $60 \times 30\ \mu\text{m}^2$ -sized rectangular single crystals. Sharp Bragg peaks of (006) and (114) with a narrow full-width-at-half-maximum of 0.14° and 0.15° , respectively, are seen in figures 1(a) and (b), with the measurement resolution of 0.003° . The x-ray beam spot ($500 \times 500\ \mu\text{m}^2$) was larger than the lateral dimension of the sample. Scattered x-ray beam from the sample holder was the cause of relatively high background level of intensity in figures 1(a) and (b). The solid lines are Gaussian best-fit curves. The results confirm that the direction normal to the rectangular crystal surface is the c -axis while the plate-shaped surface is along the ab -plane. The XRD-peak analysis leads to the estimation of lattice constants as $a = 3.909(1)\ \text{\AA}$ and $c = 8.435(2)\ \text{\AA}$, respectively.

Figure 2(a) shows the electron diffraction pattern in a selected area of a SmFeAsO_{1-x} single crystal for the $[100]$ beam direction, which was obtained from Cs-corrected high-resolution scanning transmission electron microscopy (HR-STEM). The pattern clearly illustrates that the crystal possesses a tetragonal structure, which is consistent with the previous

report [1]. Figure 2(b) shows the high-angle-annular-dark-field scanning transmission electron microscopy (HAADF-STEM) image, which exhibits the real space arrangement of each atomic element constituting the SmFeAsO_{1-x} single crystal. The large and bright dots denote the heaviest element, Sm, and the others indicate As and Fe atoms, as presented in figure 2(b). However, the lightest oxygen atoms were not detected to give information on the oxygen vacancies. The HAADF-STEM image clearly reveals a well-defined structure of alternating SmO and FeAs layers including a wiggle in each layer in the tetragonal unit cell, which directly confirms the structural model of SmFeAsO as schematically illustrated in figure 2(c). The approximate lattice constants, estimated from the HAADF-STEM image, were about $a(=b) \approx 3.9\ \text{\AA}$ and $c \approx 8.4\ \text{\AA}$, respectively, which are consistent with the values obtained from the XRD analysis.

The upper inset of figure 3 shows a field-emission scanning electron microscopy (FE-SEM) image of the specimen prepared for the transport measurement, where a long rectangular single crystal (dimensions: $30 \times 3.5 \times 0.8\ \mu\text{m}^3$) was patterned into a four-probe configuration. Contact leads were made by using photo and e-beam lithographic patterning on the sample surface. Focused-ion-beam etching and Pt deposition were employed to trim the specimens and to reinforce the connection between the crystal and the contact leads, respectively. The main panel presents the in-plane resistivity (ρ) of the SmFeAsO_{1-x} single crystal. As shown in the lower inset of figure 3, the onset of the

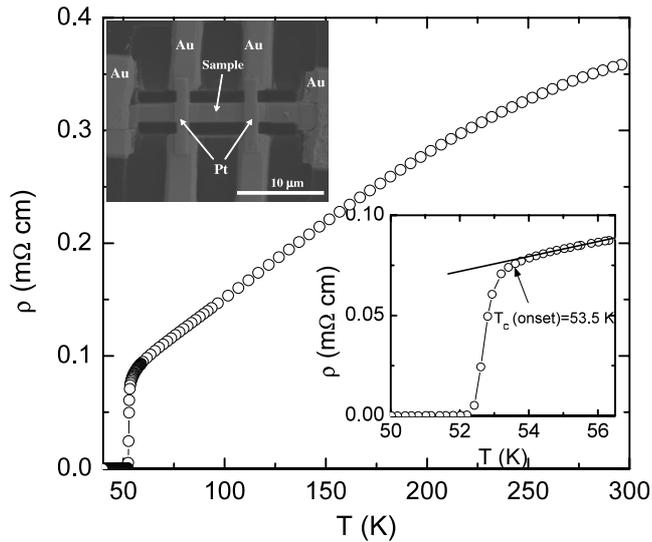


Figure 3. Temperature dependence of the resistivity in zero magnetic field for the SmFeAsO_{1-x} single crystal. The upper inset shows the field-emission scanning electron microscopy (FE-SEM) image of the four-probe patterned crystal for the transport measurements. The inset shows a close-up view of the data near the superconducting transition temperature, denoting the onset of deviation ($T_c(\text{onset})$) from the linearity.

superconducting transition ($T_c(\text{onset})$) of the crystal occurs at about 53.5 K with a very narrow transition width of $\Delta T_c = 0.5$ K (by adopting the criterion of 10–90% of the normal-state ρ). Our $T_c(\text{onset}) = 53.5$ K is in the range between the zero-resistance transition temperature $T_c(R = 0)$ of 52.8 K and the $T_c(\text{onset})$ of 55 K reported for a polycrystalline sample with the nominal composition of $\text{SmFeAsO}_{0.85}$ [36]. The transition width ΔT_c of our SmFeAsO_{1-x} single crystal is significantly smaller than the 2.5 K and 1–4 K width reported for $\text{SmFeAsO}_{0.7}\text{F}_{0.25}$ [5] and $\text{NdFeAsO}_{0.82}\text{F}_{0.18}$ single crystals, respectively [4, 6], determined by the resistive transition. The residual resistivity (ρ_0) at T_c of our single crystal is about 0.08 mΩ cm, which is also 2–4 times smaller than the $\rho_0(T_c) = 0.35$ mΩ cm for polycrystalline $\text{SmFeAsO}_{0.85}$ [36], $\rho_0(T_c) = 0.17$ mΩ cm for single crystalline $\text{SmFeAsO}_{0.7}\text{F}_{0.25}$ [5], and $\rho_0(T_c) = 0.28$ mΩ cm for single crystalline $\text{NdFeAsO}_{0.82}\text{F}_{0.18}$ [4, 6]. The residual resistivity ratio, $\text{RRR} \equiv \rho(300 \text{ K})/\rho(T_c(\text{onset})) \approx 5$, of our crystal turns out to be twice as large as the value of ~ 2.5 seen previously for $\text{NdFeAsO}_{0.82}\text{F}_{0.18}$ single crystals [4, 6]. Since transport properties such as RRR sensitively depend on the quality of a specimen, our single crystal is considered to be of high quality.

Because of the difficulty with precisely determining the chemical composition of the crystals due to the lightness of oxygen element, the oxygen content in our single crystals was roughly estimated by considering the doping dependence of lattice-constant and T_c . According to [36], the lattice parameter a of polycrystalline NdFeAsO_{1-x} is almost linearly reduced with increasing the oxygen vacancy x while T_c shows a bell-shaped variation with x . The value of T_c rapidly increases up to ~ 50 K with $a \simeq 3.957$ Å for slight doping of oxygen

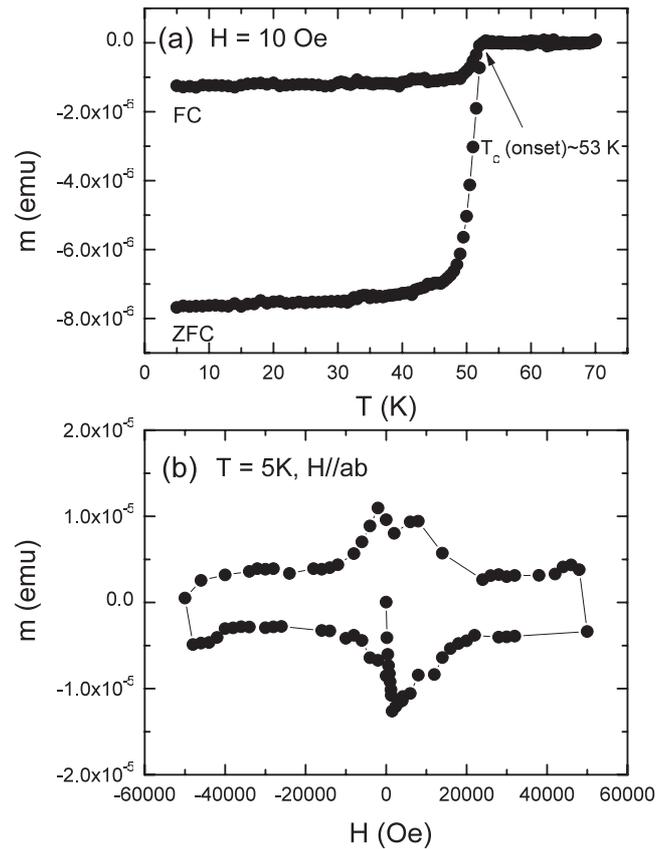


Figure 4. (a) Temperature dependence of magnetic moment $m(T)$ measured on 50 different SmFeAsO_{1-x} single crystals with dimensions in the range of $30\text{--}170 \times 20\text{--}100 \times 10\text{--}30 \mu\text{m}^3$ collected from a single growth batch in 10 Oe for zero-field cooling (ZFC) and field cooling (FC). (b) Magnetic hysteresis loop $m(H)$ taken at 5 K for a unit piece of single crystal with a dimension of $\sim 200 \times 100 \times 20 \mu\text{m}^3$.

vacancies and approaches a maximum value of 53.5 K with $a \simeq 3.944$ Å at $x = 0.15$. With further increasing the doping level x beyond 0.15, T_c decreases down to 20 K with $a \simeq 3.916$ Å while undoped NdFeAsO has $a \simeq 3.965$ Å. Since polycrystalline SmFeAsO_{1-x} and NdFeAsO_{1-x} show close doping dependencies of T_c [36] one may assume that the two materials would have similar relation between T_c and the lattice constants. The lattice constants, $a = 3.909(1)$ Å and $c = 8.435(2)$ Å, for our SmFeAsO_{1-x} single crystals with nominal oxygen deficiency of $x = 0.15$ are slightly longer than those for polycrystals of the same x ($a = 3.897(6)$ Å and $c = 8.407(1)$ Å) [36] and shorter than those for undoped SmFeAsO ($a = 3.933(5)$ Å and $c = 8.495(3)$ Å) [36]. In addition, $T_c(\text{onset}) (=53.5 \text{ K})$ of our single crystals is slightly lower than $T_c(\text{onset}) (=55 \text{ K})$ of the polycrystals [36]. These indicate that our single crystals have the oxygen deficiency less than the starting nominal value of 0.15.

Magnetic moment m of SmFeAsO_{1-x} single crystals was measured as functions of temperature T and applied magnetic field H , as shown in figures 4(a) and (b), respectively. For these measurements, we prepared two sets of single crystals from the same growth batch: 50 irregular plate-shaped single crystals with dimensions in the range of $30\text{--}170 \times 20\text{--}100 \times 10\text{--}$

$30 \mu\text{m}^3$ and a piece of nearly rectangular-shaped single crystal with a dimension of $\sim 200 \times 100 \times 20 \mu\text{m}^3$.

Figure 4(a) presents the temperature dependence of the magnetic moment ($m(T)$) of the collection of SmFeAsO_{1-x} single crystals, taken for zero-field cooling (ZFC) and field cooling (FC) in 10 Oe. The superconducting volume fraction of the crystals could not be estimated because of the resolution limit of the mass measurements for small-size crystals of our collection. However, the saturated ZFC curve indicates the full diamagnetic response of the crystals. The $m(T)$ curve reveals that $T_c(\text{onset})$ of the single crystals is close to 53 K, which is consistent with the value from the resistive transition measurements shown in figure 3. The transition width of this collection of crystals is somewhat broadened ($\Delta T_c \sim 3$ K), which is caused by the variation in the oxygen vacancies and the resulting T_c 's for crystals from the same batch. However, the transition width of the collection of our F-free SmFeAsO_{1-x} crystals is significantly narrower than that of $\text{SmFeAsO}_{1-x}\text{F}_x$ single crystals. The $m(T)$ measurements on $\text{SmFeAsO}_{1-x}\text{F}_x$ single crystals from a growth batch indicated that T_c varied from 45 to 53 K for the nominal value [2] of $x = 0.2$. This seems to indicate that F-free SmFeAsO_{1-x} crystals can be grown more homogeneously with more uniform formation of the superconducting phases than F-doped $\text{SmFeAsO}_{1-x}\text{F}_x$ crystals. The $m(T)$ of the unit piece of single crystal also revealed $T_c(\text{onset})$ of about 53 K (not shown), despite a higher fluctuation of the data due to the resolution limit of measurements.

Figure 4(b) exhibits magnetic hysteresis loop of the unit piece of single crystal taken at 5 K in fields ranging from -5 to 5 T aligned to a planar direction⁸. The data do not reveal a ferromagnetic background caused by unreacted iron-based impurity phases. The Bean model, modified for a system with an anisotropy [37], is employed to estimate the planar critical current density within the crystals; $\Delta M = \Delta m/V = (J_{c1}t/20)\{1 - (t/3l)(J_{c1}/J_{c2})\}$, where Δm is the height of the $m(H)$ hysteresis loop in Oe, V is the sample volume in cm^3 , J_{c1} (J_{c2}) is the planar (c -axis) critical current density, t ($\sim 20 \mu\text{m}$) (l ($\sim 200 \mu\text{m}$)) is the thickness (the lateral dimension in the direction perpendicular to the external field) of the single crystal in centimeter. Since $(t/3l)(J_{c1}/J_{c2}) \ll 1$ in our crystal the above equation is effectively reduced to $\Delta M = J_{c1}t/20$. The value of the planar critical current density J_{c1} at 5 K turns out to be⁹ $\sim 5 \times 10^5 \text{ A cm}^{-2}$. Previous reports on the $m(H)$ loop for $\text{REFeAsO}_{1-x}\text{F}_x$ (RE = Sm and Ce) powder samples [38–40] reveal a large ferromagnetic background, with J_{c1} in the range of $\sim 10^5$ – 10^6 A cm^{-2} . Previous data from $\text{SmFeAsO}_{1-x}\text{F}_x$ single crystals show a slight increase of the hysteresis in $m(H)$ in a high field range due to increased pinning [2], which lead to higher $J_{c1}(H)$ values of $\sim 1 \times 10^6 \text{ A cm}^{-2}$. Although one gets only a

rough estimation of the critical current density from the Bean model, the planar critical current density of our single crystal seem to be lower than the previous reports. Along with the weak field-dependence of $m(H)$ of our SmFeAsO_{1-x} single crystals, this fact seems to indicate the presence of a low level of extrinsic impurity phases of our F-free crystals. But $J_{c1} \geq 5 \times 10^5 \text{ A cm}^{-2}$ suggests the presence of intrinsic pinning, presumably from the disorder induced by the oxygen deficiency.

4. Summary

We present the successful growth of SmFeAsO_{1-x} single crystals of the nominal oxygen deficiency of $x = 0.15$ without fluorine doping under high pressure. The rapid cooling after the long heat treatment turns out to be more favorable in maintaining the single crystalline phase. The high-quality XRD peaks and HR-TEM images indicate the good crystallinity of our single crystals, with a well-defined layered tetragonal structure. The T_c from the resistive transition of a single crystal was 53.5 K with a very narrow ΔT_c of 0.5 K. Superconducting properties of SmFeAsO_{1-x} single crystals were investigated by using resistance and magnetization measurements. Relatively low residual resistivity ρ_0 and the large residual resistivity ratio, a weak field-dependence of $m(H)$ in the high field range indicate the high quality of our F-free single crystals. The F-free single crystals of Fe-based superconductors are expected to provide an easier route to find the optimum conditions for the crystal formation and possibly a simpler interpretation of the electronic structure in this family of materials.

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⁸ In spite of complications in the data analysis for anisotropic materials like our SmFeAsO_{1-x} the external field was applied in a planar direction because of a technical difficulty with placing our tiny single crystal to be perpendicular to an external field.

⁹ Since significant anisotropy is supposed to exist between J_{c1} and J_{c2} , the assumption $(t/3l)(J_{c1}/J_{c2}) \ll 1$ may not strictly hold. Then, the value of the planar critical current density at 5 K, $J_{c1} \sim 5 \times 10^5 \text{ A cm}^{-2}$, should correspond to the lower limit of J_{c1} .

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