Microwave absorption study of polycrystalline SmO$_{1-x}$F$_x$FeAs

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Abstract. Measurements of nonresonance microwave absorption have been used to determine the position of the irreversibility line and to estimate the critical current density for the three SmO$_{1-x}$F$_x$FeAs samples with fluorine concentrations $x = 0.06$, 0.08, and 0.1. The irreversibility lines of all samples are characterized with a sharp slope, indicating a strong pinning up to $T_c$. A weak field dependence of the critical current density and an estimate of parameters characterizing the vortex matter allow us to suggest the presence of additional pinning centers in underdoped samples. It is likely that these pinning centers are nanoscale inclusions of a possibly magnetic phase occurring in the fluorine concentration range of 0.06÷0.08.

1. Introduction

Microwave absorption (MWA) study of SmO$_{1-x}$F$_x$FeAs pnictides with various fluorine contents is of high interest on both theoretical and practical grounds. On the one hand, it may help to confirm or disprove the hypothesis of coexistence of superconducting and magnetic phases in underdoped SmO$_{1-x}$F$_x$FeAs. On the other hand, the method allows to estimate the value of the critical current density as well as ranges of magnetic fields and temperatures with non-zero critical current, which can give an insight into possible practical applications of the material.

2. Materials and methods

Polycrystalline SmO$_{1-x}$F$_x$FeAs samples (sintered dense pellets) were prepared and thoroughly characterized as described in Ref. [1]. Optical microscopy investigation has shown that each sample represents a network of well-connected superconducting grains with an average size of about 8 μm. Since samples are air sensitive, they were sealed in glass ampoules with pure helium gas to prevent their degradation. According to wavelength-dispersive X-ray (WDX) analysis the ratio of the nominal and the actual fluorine content in the material is approximately 2.5:1. The doping levels hereinafter thus correspond to the WDX result and are equal to 0.06, 0.08 and 0.1 for the three samples under study. The amount of impurity phases does not exceed the x-ray diffraction resolution limit of ~ 5%.

The superconducting transition temperatures $T_c$ were determined from both four-probe resistivity, DC and AC susceptibility measurements. All methods gave approximately the same half-height $T_c$ values of 35, 45, 53 K for increasing fluorine content. All superconducting transitions demonstrate a two-step behavior which is due to the granular nature of the samples, i.e. the first step corresponds to the establishment of superconductivity within grains, while the second one can be explained by the shielding current flowing across intergranular junctions [2].

The MWA method used here is based on the dissipation of microwave power in superconducting samples by vibrating vortices and can give information about the pinning strength in the system [3-5]. The MWA measurements were carried out on an X-band spectrometer (Bruker BER-418s) using a helium-flow cryostat for obtaining temperatures in the range of 10 - 60 K. A sample was placed in a cavity and cooled down to the measurement temperature below the critical temperature in a zero magnetic field. The MWA hysteresis loop was registered upon sweeping the magnetic field up to 7 kOe and down at a fixed temperature. The shape and temperature changes of the hysteresis loop were analyzed.

Direct information that can be straightforwardly obtained from the MWA measurements is the magnetic field value at which the hysteresis loop collapses. This is a so-called irreversibility field $H_{irr}$. The temperature dependence of irreversibility fields $H_{irr}(T)$ gives an irreversibility line which is a...
boundary between areas with non-zero and zero critical current density on the magnetic field-
temperature phase plane. The hysteresis loop also contains information about the critical current
density, viscosity and the influence of thermal fluctuations. However, to evaluate these data an
appropriate theoretical model should be applied. In the present work we use a model of Shaposhnikova
et al. [3] for a theoretical description of the hysteresis loop and determination of superconducting
parameters by numerical simulation. The shape of the MWA loop is indicative of the pinning type
(bulk or surface) which dominates in the system at certain external conditions.

3. Results
The evolution of MWA hysteresis loops with decreasing temperature for the SmO$_{1-x}$F$_x$FeAs sample
with $x = 0.06$ is shown in Fig. 1. There is a spurious EPR signal at the resonance field of about 1.6 kOe
due to some impurity iron ions present in the glass ampoules used for the sample sealing. The MWA
hysteresis occurs below the critical temperature and its shape changes drastically upon decreasing
the temperature by several Kelvin. Near $T_{c}^{onset}$ the MWA loop is almost symmetrical with respect to the
horizontal axis. Below the temperature of the kink in the superconducting transition ($4÷6$ K below
$T_{c}^{onset}$), which is registered at about 1/3 height of the whole transition, the hysteresis loop becomes
asymmetrical due to the enhancement of the low-field peak ($H \approx 100$ Oe). It is known [4,5] that such
shape of the MWA loop is observed when surface barriers of various origin contribute significantly to
the pinning. In case of ceramic materials this contribution is due to the intergranular pinning which
can be associated with the establishment and progression of intergranular superconductivity in the
sample. It should be noted that the value of this contribution is different for the samples with different
fluorine content. At the same reduced temperature $t = T/T_c = 0.86$ the contribution is maximal for the
underdoped sample with $x = 0.06$ and minimal for the optimally doped sample $x = 0.1$. Besides the
low-field peak, there is an MWA hysteresis in high magnetic fields. The shape of this high-field loop
corresponds to the bulk intragranular pinning and in this region the collapse of the hysteresis loop
occurs at different temperatures. Therefore, the irreversibility line of the sample is defined by pinning
inside grains of the pellet.

![Fig.1. Hysteresis loops registered at different temperatures below $T_c$ for a polycrystalline SmO$_{1-x}$
\textsubscript{F}$_x$FeAs sample with $x = 0.06$. Horizontal arrows show the sweep direction. Vertical arrows indicate
the location of irreversibility fields.](image-url)
The irreversibility lines for the samples with different fluorine content are presented in Fig. 2a. The first feature one may notice is a very steep ascent of all irreversibility lines with decreasing temperature. Comparison of these results with the data for cuprate high-temperature superconductors (HTSC) yields a conclusion that a similar sharp trend of the irreversibility line was reported only for YBCO samples [6], which possess the highest pinning strength among all known HTSC compounds (Fig. 2b). Even LSCO samples are characterized with a weaker dependence $H_{\text{irr}}(T)$, while the irreversibility line of BSCCO ceramics is very flat, especially near $T_c$. It should be noted that all comparisons were drawn between ceramic samples. The revealed tendency is indicative of a strong pinning in SmO$_{1-x}$F$_x$FeAs pnictides. One may also notice in Fig. 2a that the irreversibility line of the optimally doped sample ($x = 0.1$) lies in higher magnetic fields than those of the underdoped samples. It is possible that this fact is connected with the considerably higher $H_{C2}(T)$ values revealed for the SmO$_{1-x}$F$_x$FeAs compound with such fluorine content [7].

![Fig. 2](image-url)

**Fig. 2.** (a) Irreversibility lines of SmO$_{1-x}$F$_x$FeAs samples: circles, $x = 0.06$; diamonds, $x = 0.08$; squares, $x = 0.1$. (b) Irreversibility lines for polycrystalline HTSC compounds: diamonds, SmO$_{1-x}$F$_x$FeAs with $x = 0.06$; white, BSCCO; squares, LSCO; black circles, YBCO.

### 4. Discussion

The critical current density may be estimated from MWA measurements with the help of a theoretical model by Shaposhnikova et al. [3]. In the general case the MWA hysteresis is a complex function of the value of the critical current density $j_c$, the viscosity of the vortex system $\eta$, the amplitude of thermal fluctuations $\langle u^2 \rangle$, and of the distribution of fields $H(x, t)$ and currents $j(x, t)$. These parameters can be estimated by fitting the model loops to the experimental data. The result of a numerical calculation of the MWA hysteresis loop is shown as a dashed line in Fig. 3a. The theoretical curve describes well the experimental loop in the high field range (> 500 Oe). Divergence of theoretical and experimental curves in low magnetic fields can be explained by a contribution of the intergranular pinning which is not considered in the theoretical model, being valid only for the bulk intragranular pinning. Our supplementary experiments of registering the distribution of the local magnetic field on the superconducting pellet surface by a scanning Hall sensor upon varying the external magnetic field have shown the absence of the Bean distribution over the whole of the sample. This implies that it takes place only on the size scale of the grains. Therefore, the average size of individual grains, which was estimated to be 8 µm, was used in the calculations of the critical current density for the three samples under study. For simplicity of the calculations the contribution of the intergranular pinning was omitted.

The critical current density calculated for each sample at $T = 20$ K is shown as a function of magnetic field in Fig. 3b. The following tendency has been revealed: the lower the concentration of fluorine in the sample, the weaker is the field dependence of the critical current density. This observation indicates that the samples with smaller fluorine concentration contain a larger number of pinning centers. It is possible that nanoscale inclusions of a nonsuperconducting phase might act as such centers. The nonmonotonic dependence of the critical current density on the fluorine content
may be explained by the change in the ratio of sizes of nonsuperconducting inclusions and vortex cores. Apparently, the sizes of nonsuperconducting (possibly magnetic) inclusions are maximal at the lowest fluorine concentration and shrink as doping tends to the optimal level. As a result, the effectiveness of pinning centers also decreases with increasing the fluorine content.

The assumption of bigger nonsuperconducting inclusions at the lowest fluorine content under study is supported by estimates of the superconducting parameter \( \frac{\langle u^2 \rangle}{a^2} \). The ratio of the amplitude of thermal fluctuations \( \langle u^2 \rangle \) to the mean distance between pinning centers \( a \) has been obtained at the field of 2 kOe and at \( T = 0.9T_c \) for the samples with fluorine content of 0.06, 0.08 and 0.1, respectively: \( \frac{\langle u^2 \rangle}{a^2} = 0.071, 0.033 \) and 0.037. If the parameter \( \langle u^2 \rangle \) is not changing significantly, then the increase in the ratio \( \frac{\langle u^2 \rangle}{a^2} \) can be explained by a smaller distance between pinning centers, and as a result, either by a greater number of pinning centers or by their larger sizes.

Despite the stronger field dependence of the critical current density for the optimal doping level of 0.1, the absolute value of the critical current density at least at low magnetic fields is higher than that of underdoped samples. This fact is indicative of a large number of pinning centers but evidently their nature differs from that of underdoped samples.

Fig.3. (a) Experimental (solid line) and theoretical (dashed line) MWA hysteresis loops for polycrystalline SmO\(_{1-x}\)F\(_x\)FeAs sample with \( x = 0.08 \) at \( T = 34 \text{ K} \). (b) Magnetic field dependence of the critical current density for SmO\(_{1-x}\)F\(_x\)FeAs samples: circles, \( x = 0.06 \); diamonds, \( x = 0.08 \); squares, \( x = 0.1 \).

5. Conclusion
The MWA study of a series of polycrystalline SmO\(_{1-x}\)F\(_x\)FeAs samples reveals strong pinning of superconducting vortices up to \( T_c \) comparable in strength with the highest values for the cuprate HTSC. Such a remarkable effect may be due to the occurrence of nanoscale possibly intrinsic nonsuperconducting inclusions, which serve as effective pinning centers for magnetic flux lines.

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