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Effect of sheath materials on the microstructure and superconducting properties of $SmO_{0.7}F_{0.3}FeAs$ wires

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ABSTRACT

Fe/Ti, Nb, and Ta sheathed $SmO_{0.7}F_{0.3}FeAs$ wires were manufactured using the powder-in-tube method. A comparative study was made of the effect of sheath material on the microstructure and superconducting properties of the $SmO_{0.7}F_{0.3}FeAs$ wires. Among these sheath materials, the penetration depth of Ta into the superconducting core is the largest. There was nearly no difference in the critical transition temperature of the $SmO_{0.7}F_{0.3}FeAs$ wires sheathed with different materials, and the upper critical fields were approximately the same. On the other hand, it was found that Nb-sheathed wires had a higher superconducting current density than the others. Impurities existing in the superconducting core were thought to be one of the most important factors that affected the critical current density of the $SmO_{0.7}F_{0.3}FeAs$ wires.

1. Introduction

The recent discovery of superconductivity in iron pnictides has generated much interest due to the unconventional superconducting properties and the underlying mechanism. Shortly after the report of $La(O_{1-x}F_x)FeAs$ with a critical transition temperature (T_c) of ~26 K [1], T_c was rapidly raised to above 50 K by replacing La with other rare earth elements [2–8]. The rather high T_c of the iron pnictide superconductors brings a hope of new superconducting materials, particularly, in the case of the $Sm(O_{1-x}F_x)FeAs$ superconductor, where the T_c has been enhanced to 55 K. At the same time, it was reported that the $Sm(O_{1-x}F_x)FeAs$ superconductor had an H_{c2} over 150 T [9], giving promise of excellent performance in high fields. These properties of $Sm(O_{1-x}F_x)FeAs$ make it a potential material for technology applications. For example, it can be used in high-field applications above 30 K, where conventional superconductors cannot play a role owing to their low T_cs. Many of these applications involve the use of superconductors in wire or filament form. However, $Sm(O_{1-x}F_x)FeAs$ superconducting materials have a layered structure [10], and are mechanically hard and brittle. Therefore, it is difficult to draw this compound into the desired wire geometry, which is similar to the case of cuprates. To solve this problem, the powder-in-tube (PIT) method was tested as a means to fabricate $Sm(O_{1-x}F_x)FeAs$ superconducting wires [11,12].

It has already been shown that the sheath material influences the basic properties of MgB_2 wires and tapes made by the PIT technique [13]. Sheath material selection is more important for $Sm(O_{1-x}F_x)FeAs$ material due to the high sintering temperature (nearly 1200 °C). In general, the sheath materials should not react with the raw materials during the sintering process, and should have sufficient mechanical properties. For the fabrication of $Sm(O_{1-x}F_x)FeAs$ wires, the likely acceptable sheaths reported so far are Nb, Ta and Ti. This paper reports on the effect of sheath material on the microstructure and superconducting properties of $SmO_{0.7}F_{0.3}FeAs$ wires.

2. Experimental details

The $SmO_{0.7}F_{0.3}FeAs$ (nominal composition) wires were prepared by the *in situ* PIT method [11], using Sm, As, SmF_3, Fe and Fe_2O_3 as starting materials. The raw materials were thoroughly grounded by hand in an agate mortar. The mixed powder was loaded into Fe, Nb and Ta tubes. The Nb and Ta tubes both had an 8 mm outside diameter and a 1 mm wall thickness. For the Fe tube, which had an 8 mm outside diameter and a 1.5 mm wall thickness, two layers of Ti sheath (thickness 0.03 mm) were inserted between the Fe and the powder to prevent a reaction between the Fe tube and the mixed powder. The grinding and packing processes were carried out in a glove box containing a high purity argon atmosphere. After packing, the tubes were rotary swaged and then drawn to wires 2.63 mm in diameter. The wires were cut into 4–6 cm lengths, sealed in an Fe tube, then sintered at 1180 °C for 45 h. High purity argon gas was passed through the furnace during the heat-treatment process to minimize oxidation of the samples.

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The crystal structure was studied by X-ray powder diffraction (XRD) using a Philips X'Pert MRD diffractometer with Cu Kα radiation. The microstructure and composition of the sample were analyzed using a scanning electron microscope (SEM, JSM-7600F) equipped with an energy-dispersive spectrometer (EDS, NORAN X-ray Microanalysis System). The superconducting properties of the wires were studied by DC magnetization and four-probe resistivity measurements using a physical property measurement system (Quantum Design PPMS 9T). The inductive critical current density J_c was determined using the extended Bean model [14], with the formula $J_c = 20\Delta M/va(1 - a/3b)$, where ΔM is the width of the magnetization (M) loop, a and b are the length and width of the slab ($a < b$), while v is the volume of the sample, respectively. Slabs cut from the central part of the superconducting core of the wire were used for the above measurements. For the interfacial reaction analysis, we concentrated on mapping the distribution of various phases at the core/sheath interface. Because there were many elements involved in the $SmO_{0.7}F_{0.3}FeAs$ wires, we used a Direct-to-Phase software, which can simultaneously acquire and analyze spectral imaging data. Using this method, the selected region is divided into several parts in different color, while each part possesses the same elements. It should be noted that the absolute composition is not accurately reflected in these EDS patterns. For instance, we have not corrected the raw X-ray yields to take into account the very different efficiencies with which X-rays are produced by F and As.

3. Results and discussion

The SEM image in Fig. 1 shows an area of the interface region from the Fe/Ti sheathed samples. The three parts in this SEM image display the superconducting core on the left ("C" region), the pure Fe sheath on the right ("S" region), and the Ti layer in the middle ("R" region), respectively. From the elemental map (c) and corresponding EDX spectra (c), we can see that the phase present in S region is almost pure Fe (95 at.%) with a small amount of As (3 at.%) and Ti (1.6 at.%). For the R region, Fe (27.1 at.%), As (41.5 at.%) and Ti (30.7 at.%) elements were detected. This means that some As leaked into the Fe sheath across the Ti layer during sintering, which is further proven by the brittle sheath of Fe/Ti sheathed samples. Because the Ti material has poor ductibility, the Ti layer inserted in the Fe tube would tend to be torn during the fabrication process.

The elemental map (a) and corresponding EDX spectra (a) in Fig. 2 show that there is nearly no As (2 at.%) present in the S region, indicating a low penetration of As into the Nb sheath. On the other hand, it can be seen that the interfacial reaction layer is about 60 μm thick. In contrast, as seen from the elemental map (b) in Fig. 3, high percent Ta element (13.8 at.%) can be found to a depth of 200 μm into the superconducting core. From this aspect, Ta is not a good sheath material for $SmO_{1-x}F_xFeAs$ fabrication using the *in situ* PIT method, even though the high hardness of Ta is favorable for obtaining a dense superconducting core.

Although the thicknesses of the reaction layers in the interface region are different from sample to sample, the SEM images of the superconducting core show very little difference. Typical SEM images of the superconducting core for all the samples are shown in Fig. 4. Multiple layers forming large grains can be easily observed in all of the samples. This is also a common feature of $SmO_{1-x}F_xFeAs$ superconductors [15]. At the same time, some particle-like impurity phases and voids are observed. The plate-like grains of the superconducting phase are greater than 5 μm in size. As reported in [16], the impurities and voids, as well as the large grain boundary angles, make it difficult for superconducting current to flow through the wire.

In order to obtain more information about the phases in the superconducting core, XRD measurement was performed on superconducting slabs cut from the various sheathed wires, as shown in Fig. 5. It can be seen that the main phase in all three samples is SmOFeAs. Some peaks marked by asterisks and crosses can be indexed to impurity phases, such as SmAs and SmOF. Judged from the relative height of SmOF diffraction peak to those of SmOFeAs, the impurity phase content in the Fe/Ti sheathed wires are much higher than the other samples. This is also in accordance with the results of interfacial analyses. However, it should be noted that the XRD measurements were performed on the superconducting core, while the interfacial analysis were carried out on the interfacial region. The phases obtained by EDX and XRD measurement may have some differences.

The temperature dependence of resistivity for the $SmO_{0.7}F_{0.3}FeAs$ wires is shown in Fig. 6. The samples reveal a metallic behavior in that resistance decreases with decreasing temperature until it drops to ~55 K. We can see that the T_c of the $SmO_{0.7}F_{0.3}FeAs$ superconducting cores is much the same for the Fe and Ta sheathed wires. On the other hand, the resistivity at 55 K ($\rho_{55\,K}$) of the Nb sheathed samples shows a lower value than the others. Although the nominal F doping value is high, the T_c is lower than that of bulk samples doped to the same F level [5]. This is a common phenomenon in SmOFeAs wire preparation [11]. In wire samples, the composition is difficult to control. The residual resistivity ratio ($\rho_{300\,K}/\rho_{55\,K}$) of these samples is about 3, a little lower than that for the bulk samples [10]. The existence of a superconducting phase was

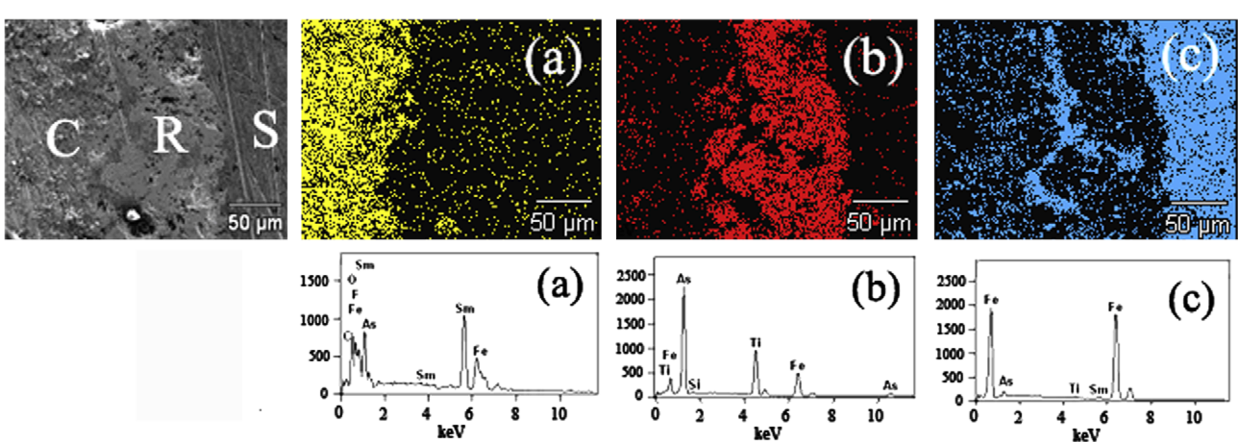

Fig. 1. SEM image of the interface region from Fe/Ti sheathed samples (S: sheath, R: reaction layer, C: superconducting core), and the corresponding elemental maps and EDX spectra.