Investigation of J_c Suppressing Factors in Flat-rolled $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2/\text{Fe}$ Tapes Via Microstructure Analysis

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Abstract—Pnictide superconductors will be very promising for applications if wires with high critical current density J_c can allow reel-to-reel large-scale fabrication at low costs. To understand the mechanism(s) that limited J_c in flat-rolled $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ (Sr122) tapes, microstructure analysis has been considered to be the most direct and efficient way. Here we report on a high-resolution microstructure imaging and analysis on Fe-sheathed flat-rolled Sr122 tapes, which have a J_c as high as $2.3 \times 10^4 \text{ A/cm}^2$ at 10 T and 4.2 K. The overlapping nature of the Sr122 plates was clearly observed. TEM/STEM analysis showed that, besides the cracks formed during the fabrication process, the SrO_2 phase and cavities caused by the inhomogeneously dispersed Sr and K are the other important factors suppressing the J_c . The wetting phase FeAs at the grain boundaries can be partially substituted by Sn in Sn added samples. Our findings provide insights which pave the way to further enhance the critical current of rolled 122 tapes up to the practical level.

Index Terms—Pnictide superconductors, wires, J_c , microstructure.

I. INTRODUCTION

THE discovery of high-temperature superconductivity at 26 K upon F substitution at the oxygen site in LaFeAsO [1] has aroused worldwide research efforts. Several types of the iron pnictide have been discovered [2]-[6], and tens of superconducting compounds have been developed. Among these compounds, the ‘122’ type ones have attractive properties for potential applications. For example, they exhibit moderately high critical transition temperature T_c , high upper critical field H_{c2} , low H_{c2} anisotropy, low thermal fluctuation, and intrinsic pinning mechanism [7]-[10]. As many of the applications involve the superconducting wires and tapes, polycrystalline pnictide in the form of long wires or tapes have to be developed. Actually, the J_c has been rapidly enhanced particularly for Sr122/Ba122 tapes in the past one year [11]-

[14]. However, it should be noted that high J_c values were only obtained in cold pressed/hot pressed samples [13], [14], which are not easy to be fabricated in large scale. For the flat-rolling method, which can be used to produce long wire/tape for practical application, it is still difficult to fabricate wires/tapes with high J_c values [15], [16]. At the same time, it should be noted that the rapid J_c improvement was only obtained in Ag-sheathed 122 samples [12]-[16], while the J_c of the Fe-sheathed samples, which have a relatively low production cost, is still kept in low values [11], [17].

To understand the suppressing factors of J_c in flat-rolled 122 tapes, a systematic study on the microstructure of sample is necessary to give a clear picture about the relationships between microstructure and superconducting properties. However, there has been no systematic study of the microstructure of the 122 wires and tapes so far. Here we report on a high-resolution microstructure imaging and analysis on Fe-sheathed flat-rolled Sr122 tapes. A detailed microstructural investigation was carried out based on this kind of sample. SEM/EDS analysis was performed for large scale morphology observation and composition analysis, and STEM/EELS was conducted for high resolution microstructure analysis. It showed that c -axis alignment of ‘122’ crystalline can be achieved to some extent. The cracks, SrO_2 phase and cavities are the main factors suppressing the J_c . With Sn addition, the non-superconducting FeAs layers at the some grain boundaries were partially substituted.

II. EXPERIMENTAL DETAILS

A. Samples

The $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ tapes were prepared by a two-step solid-state reaction method. Sr fillings, K pieces, and Fe and As powders were used as starting materials with the nominal composition of $\text{Sr}_{0.6}\text{K}_{0.5}\text{Fe}_2\text{As}_2$. The starting materials were mixed and ground by ball-milling method, and then the milled powders were heat-treated at 900 °C for 35 h. The sintered bulk was ground into powders and mixed with 2.5 wt% Sn by hand. Finally, the powders were filled and sealed into Fe tubes with an outer diameter of 8 mm and inner diameter of 5 mm, which were subsequently rotary-swaged, drawn to wires, and then rolled into tapes with 0.6 mm in thickness. The tapes were finally sintered at 900 °C for 30 minutes.

B. Analysis

The microstructure was studied using scanning electron microscopy (SEM) and transmission electron microscopy

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(TEM). The samples were prepared using a Focused Ion Beam (FIB) Microscope. The chemical composition was analyzed using Energy Dispersive X-ray Spectroscopy (EDS) and Electron Energy-Loss Spectroscopy (EELS) attached to the microscopes. The critical current J_c at 4.2 K and its magnetic field dependence were evaluated at the High Field Laboratory for Superconducting Materials (HFLSM) in Sendai, by the standard four probe method, with a criterion of $1 \mu\text{V}/\text{cm}$.

Fig. 1. Magnetic field dependence of transport J_c at 4.2 K for Fe-sheathed Sr122 tapes. The insets show XRD patterns and M - T curves of the precursors and the tapes.

III. RESULTS AND DISCUSSION

The insets of Fig. 1 show the XRD pattern and M - T curve of the tapes processed by flat rolling. As a reference, the data for precursor powder is also included in these figures. It can be seen that all tape samples exhibit a main phase of Sr-122. The c axis orientation factor calculated by the Lotgering method for these tapes is ~ 0.5 , which is comparable to that of the Ag-sheathed tapes [14]. But compared to the precursor, the susceptibility of the tapes decreases much slow with cooling. This indicates that the crystallization was decreased after the precursor was filled into the Fe tube and re-sintered in tape form [12]. This is different than what happened in Ag-sheathed tapes, where the T_c of the tapes was barely affected [15].

Fig. 2. Typical SEM images of the $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ tapes after peeling off the Fe-sheath, viewed from the tape surface in the normal direction (a) and longitudinal cross-section (b).

However, as exhibited in Fig. 1, the Fe-sheathed flat-rolled Sr122 tapes still possessed a $J_c \sim 2.3 \times 10^4 \text{ A}/\text{cm}^2$ at 10 T and $2.11 \times 10^4 \text{ A}/\text{cm}^2$ at 12 T, respectively. Additionally, these are

the highest values reported in the Fe-sheathed pnictide wires or tapes [11], [17]. From the economic point, the Fe-sheath is more attractive than the Ag-sheath in fabricating Sr122/Ba122 tapes for practical applications.

To understand the mechanism of the lower J_c in Fe-sheathed samples than that in the Ag-sheathed cold/hot pressed samples [13], [14], systematic microstructural studies have been conducted. Typical SEM images of the $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ tape in the normal direction and longitudinal cross-sections are shown in Fig. 2. Clearly, two morphologies of the tape are very different for in-plane and longitudinal cross-section. Based on these images, we found that the tape was composed of different size plates with a typical thickness of $0.8 \mu\text{m}$. Similar to other work [11]-[17], the majority of the plates are aligned with their flat face approximately parallel to the tape surface. These plates are stacked and overlapped, although not perfectly. Some plates are inclined more steeply to the surface in a few places, suggesting the presence of some non- c -axis aligned materials. Although the in-plane image seems to indicate a dense core being formed, a large number of voids were observed in the image of longitudinal cross-sections. In contrast, both of the cold pressed and the hot pressed samples show a dense core [13]-[14]. This indicates that the J_c of the flat-rolled tapes can be increased by enhancing the density of the superconducting core. One possible cause of the formation of the holes/voids could be due to the evaporation of redundant K from $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ during the sintering process, which is confirmed by the EDS/EELS analysis later.

Fig. 3. (a) The typical BF TEM images of $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ specimen prepared by FIB. (b) TEM image of apparently textured grains in polycrystalline $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ tapes.

Shown in Fig. 3(a) is the TEM image of the specimen prepared using FIB. The voids and cracks are clearly seen in the superconducting core. Several long cracks are also observed, which will certainly decrease the conducting area. This is another important reason for the lower J_c in this work than that in the cold pressed and hot pressed samples, where the long cracks were nearly eliminated [11]-[14]. Cracks are often formed during the fabrication process, holes may be caused by the evaporation of K, and SrO_2 may come from the source material. In accordance with the work reported previously [18], large grains about $5 \mu\text{m}$ are formed, as shown in TEM bright field image in Fig. 3(b). These large grains have good crystallinity, as proved by the selected area electron diffraction (SAED) pattern (not shown here). In addition to the

holes and voids observed in the SEM image, some amorphous islands were also detected in the TEM image.

Fig. 4. (a) A STEM image of the hole on the $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ grain. (b) HAADF-line scan across the hole. (c) and (d) EDS spectra and line scan performed on the hole. (e) and (f) EELS spectra and line scan performed on the hole.

To understand the reason why holes were formed inside the grains, EDS- and EELS-line scans have been performed simultaneously across the hole to analyze the elemental composition. As shown in Fig. 4, Sr and O were rich around the hole, while K was hardly detected in the center of the hole. It is proposed that K were evaporated during the heat treatment, leaving behind holes in the grains or between the grains. Actually, the leaking of the K was noticed during the sintering process of the precursor powders.

Fig. 5. (a) A STEM image of the grain boundary. (b) The enlarged image of the amorphous layer with crystallite inside. (c) HAADF-line scan across the grain boundary. (d) EDS-line scan performed across the grain boundary.

When we focused on the grain boundaries, we found strontium and oxygen enrichment (see Fig. 5 (d)), in accordance with the literature [19]. We also studied the grain boundary with high resolution TEM, as shown in Fig. 5 (b). Some SrO_2 particles of about 2-5 nanometers in size could be clearly seen, indicating that some Sr atoms did not crystallize or form into $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$.

This was further confirmed by the elemental mapping (see Fig. 6). Even in a small plate of $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$, the Sr

elements are inhomogeneously distributed, leading to Sr-rich clusters. Compared to the other starting materials, such as the brittle As element and soft K element (Fe powder was used for precursor preparation), Sr is much more difficult to disperse. Therefore, to improve the superconductor property of $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ wires, such as to increase J_c , one must increase the reaction ratio of Sr, i.e. to make all the Sr atoms fully crystallize into the desired compound. This might be achieved by decreasing the initial size of Sr or increasing the milling time of the powder.

Fig. 6. EDS mapping on the $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ grain by STEM.

Fig. 7. A STEM image of the grain boundary and EDS point scan around the grain boundary.

Fig. 8. A high resolution STEM image of the grain boundary with Sn embedded.

It has been proved that the J_c -B property can be significantly improved by adding Sn into $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ [17]. As reported, the grain boundaries are usually rich in amorphous oxide or FeAs layers in the pure samples, and grain boundaries in Sn doped tapes are often filled with Sn rich materials [19]. A STEM image of the grain boundary is shown in Fig. 7 (a). From EDS spectra and point analysis performed around the grain boundary, we clearly observed that Sn existed at the grain boundary. Some areas showed pure Sn (Fig. 7 (b)), which is proved by the high resolution TEM analysis, as shown in Fig. 8. A Sn layer about 3-4 nm thick existed between two $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ grains. There is no other phase presented here, indicating that Sn can penetrate into the grain boundaries where FeAs phase usually inhabited [19]. At the same time, the coexistence of Sn, Fe and As is found in some places (Fig. 7 (c)). It has been reported the Sn added can eliminate the detrimental FeAs wetting phase at the grain boundary, resulting in the distinct enhancement of transport J_c [20]. The observation that the cold pressed samples with no Sn addition also have high J_c [13], it suggests that Sn may not be so critical in the 122 grain connection when the core density is high enough and grain boundaries are clean. One possible function of the Sn is to assist the sintering and homogenization of the composition of $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ phase, similar to the role of Pb in Bi-Ca-Sr-Cu-O superconductor [21].

We now start to answer the crucial questions related to the lower J_c in the Fe-sheathed flat-rolled Sr122 tapes. Firstly, as mentioned above, the grain crystallization was reduced after the precursors were filled into the Fe tube and re-sintered. This is different than the Ag-sheathed samples, where two-step sintering improves the homogeneity and crystallization of the final Sr122 superconducting phases [12], [22]. One possible reason for the reduced crystallization is the reaction between the Fe sheath and the superconducting core, similar to what happened in $\text{SmO}_{0.7}\text{F}_{0.3}\text{FeAs}$ wires [23]. To solve this problem, a new sintering process should be explored to suppress the reaction between the sheath material and the superconducting core, which has succeeded in $\text{SmO}_{0.7}\text{F}_{0.3}\text{FeAs}$ tapes [24].

Secondly, although the in-plane image seems to indicate a dense core being formed, a large number of voids were observed in the image of longitudinal cross-sections. The recrystallization and the shrinkage of the superconducting core during the sintering process may be responsible for the formation of the voids. In this case, the precursors should be further optimized to decrease the shrinkage of the superconducting core during the final sintering process. Actually, the shrinkage of the superconducting core is a common feature in *in situ* processed samples [25], while a large J_c increase has been observed in *ex situ* processed tape when this problem was partially solved [26].

Thirdly, some holes and impurities were detected on the $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ plates. One possible reason for the formation of the holes could be due to the evaporation of redundant K from $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ during the sintering process. At the same time, the Sr atoms are not homogeneously distributed, leading to Sr-rich clusters, even in a small plate of $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$. Therefore, to improve the homogeneity of the superconducting

core, one must investigate the appropriate milling technique to increase the reaction ratio of K and Sr.

IV. SUMMARY

Systematic microstructure investigations were carried out on Fe-sheathed flat-rolled $\text{Sr}_{0.6}\text{K}_{0.4}\text{Fe}_2\text{As}_2$ samples. The analyses show that the transport J_c of the flat-rolled Sr122 tape was restricted by the defects in the superconducting core, mostly by voids/holes and impurity phases. We propose that, reducing the quantity of holes, voids and impurities that currently exist in our samples by such as employing suitable fabrication techniques, the superconductivity properties can be improved in flat-rolled samples.

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