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Effects of rolling deformation processes on the properties of Ag-sheathed $Sr_{1-x}K_xFe_2As_2$ superconducting tapes

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ABSTRACT

The powder-in-tube method is widely used in fabricating iron-based superconducting wires and tapes. To make tapes, a multi-pass rolling process is usually adopted. However, the multi-pass rolling process limits the efficiency of tapes. In this work, rolling deformation technique was studied systematically by fabricating $Sr_{1-x}K_xFe_2As_2$ superconducting tapes. The total rolling reduction ratio is about 80% and the difference of superconducting performance of tapes rolled by 2, 3, 5 and 7 passes has been investigated. The critical current density J_c, Vickers micro-hardness and microstructure of the superconducting core indicate that tapes after 2, 3, 5 and 7 rolling passes exhibit a similar trend. The width of the tapes and the area of superconducting cores increase with decreasing the number of rolling passes, but the transport J_c of tapes after different rolling passes seems to be the same, except for the tape rolled by 2 passes, whose transport J_c is lower than the other tapes. Concerning the geometry uniformity for the superconducting cores, the sausaging phenomenon was not observed from the photograph of longitudinal cross-section of all the samples. "Lobes" phenomenon on transverse cross-section can be suppressed through decreasing the rolling passes. Therefore, we can obtain uniform and high-performance Ag-sheathed iron-based superconducting tapes by cutting the number of rolling passes down to 3, which is more advantageous to the large-scale producing in the future.

1. Introduction

Since the discovery of iron-based superconductors in 2008 [1], enormous efforts has been expended to exploit the new superconductors in commercial applications, especially for the fabricating of wires and tapes [2–10]. The 122 type ($AeFe_2As_2$, Ae= alkali or alkali earth elements) exhibit the most attractive performance in iron-based superconductors for the high T_c [11], the ultra-high upper critical fields H_{c2} [12,13] and the low superconductivity anisotropy γ [14]. All of these advantages made the iron-based superconductors potential for high field applications such as the NMR and MRI and so on. A large amount of superconducting wires or tapes are needed in producing these devices. Then how to fabricate high-performance and low-cost long-length superconducting wires or tapes is a challenge.

As in the producing of BiSrCaCuO tapes [15,16] the powder-in-tube (PIT) method is also widely used in fabricating iron-based superconducting tapes [17–19]. This method is simple, scalable, and low-cost. In the PIT method, the precursor powder is loaded into a metal tube and then the tube is mechanically worked to long tapes through a series of deformations such as swaging, wire drawing and rolling [20]. The final performance of superconducting tapes is restricted by many factors such as the quality of precursor, the process of deformation and the technology of heat treatment and so on. With lots of efforts being made to solve the key problems in fabricating iron-based superconducting tapes, the transport J_c has been significantly increased [10,21]. Lin et al. systematically studied the heating condition of Sr-122 tapes and found that the best final heating temperature is 850–900 °C [22]. In order to find a suitable materials for the sheaths of tapes, Zhang et al. [23] and Wang et al. [24] found that the Ag-sheathed tapes almost have no reaction layers between the sheathes and superconducting cores [23,24], and the Ag is the most suitable sheath material for the 122 iron pnictides so far.

The author's group has fabricated the world's first 11 m long Ag-sheathed Sr-122 tape by rolling [21], and the average J_c value can reach about 1.84×10^4 A/cm^2, which is a significant breakthrough for practical applications. The cold pressing [25] or hot

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Table 1
The single and total reduction ratio and the thickness of tapes of every rolling deformation.

Passes	Unit: mm								Single reduction ratio (%)	Total reduction ratio (%)
2-pass	1.90	0.87	0.40						54	
3-pass	1.90	1.11	0.65	0.40					42	80
5-pass	1.90	1.38	1.00	0.72	0.52	0.40			28	
7-pass	1.90	1.51	1.20	0.95	0.75	0.60	0.48	0.40	21	

pressing [5] process can improve the transport J_c by densifying the core of superconducting tapes but the equipment of fabricating the pressed tapes restricts the length of tapes. For large scale applications the long tapes are needed which makes the rolling deformation essential. On the other hands, rolling deformation is one of the most important process in fabricating superconducting tapes which including a series of passes to gradually reduce the tape thickness. Besides the density of powder filling, the initial wire diameter and the diameter of rollers, the reduction ratio and the number of rolling passes are very important parameters in rolling deformation process for iron-based superconducting tapes as well as for the BiSrCaCuO tapes [26].

Effects of rolling deformation processes on the properties of Ag-sheathed iron-based superconducting tapes is systematically studied by fabricating $Sr_{1-x}K_xFe_2As_2$ tapes in this work. The critical current density J_c, the grain size, the distribution of elements, and the Vickers hardness and so on are researched systematically in order to compare the difference of superconducting tapes rolled by different passes to find a proper rolling passes.

2. Experimental details

The $Sr_{1-x}K_xFe_2As_2$ tapes were fabricated by the *ex situ* PIT method. The starting materials are small Sr pieces (99.5%), K bulks (99.95%) and high-purity Fe (99.99%) and As (99.95%) powders with a ratio of Sr: K: Fe: As = 0.6: 0.4: 2: 2. In order to compensate for the loss of K and As during the milling and sintering procedures, the excess of 10%–20% K and As was added [27]. Then the powder were mixed and thoroughly ground by ball milling in an Ar atmosphere for more than 10 h. The milled powder was sintered in a Nb tube for 35 h at 900 °C. Then the precursor was ground to evenly mixed powder by hand in an agate mortar in the Ar atmosphere. The final powder were filled and sealed into a silver tube (OD: 8 mm, ID: 5 mm), which was subsequently swaged and drawn to a wire of 1.9 mm in diameter with a reducing ratio of about 10%.

The as-drawn long wires were cut into 4 parts evenly then the segmented wires were rolled through 2, 3, 5 and 7 passes which were named as 2-pass, 3-pass, 5-pass and 7-pass, respectively. In order to uniform the parameter, the reduction ratio for each single pass in each rolling deformation was set as the same for a certain type of wires. The reduction ratio was defined as: δ_h/h_0, $\delta_h = h_0 - h_1$, where h_0 and h_1 are the thickness of the tapes before and after the rolling deformation, respectively. The starting diameter of the wires is 1.9 mm and the final thickness of the tapes is 0.4 mm, respectively, then the total reduction ratio is 80%. The single and total reduction ratio and the thickness of tapes after every rolling deformation are shown in Table 1. The diameter of the two rollers is 20 cm and the speed of rolling deformation is 1.2 m/min. Short samples which cut from the rolled tapes were sintered with 880 °C for 30 min in vacuum quartz tubes.

The phase construction of the superconducting core was investigated by x-ray diffraction (XRD) analysis using a Bruker D8 Advance diffractometer with Cu Kα radiation. The grain morphology, microstructure and element distribution of superconducting core after peeling off the Ag sheath were measured by a scanning electron microscope (SEM; Zeiss SIGMA) and an energy dispersive X-ray spectroscopy (EDS). The rolled tapes were sealed in epoxy and polished well by sandpapers and then the photographs of cross section and longitudinal section of the rolled tapes were taken by the microscope (Olympus) with 50 X magnification. The Vickers hardness of superconducting core were also measured on the polished cross section by a Vickers hardness tester (401MVD, Wolpert Wilson Instruments) with 0.05 Kg loaded and 10 s duration in a row at the center of the superconducting core. The transport I_c of the $Sr_{1-x}K_xFe_2As_2$ tapes and its magnetic field dependence were measured at 4.2 K using short samples of 3 cm in length with the standard four-probe method by a criterion of 1 μV/cm at the High Field Laboratory for Superconducting Materials (HFLSM) in Sendai. The transport J_c was calculated with I_c/S, where S is the cross-sectional area of the superconducting core.

3. Results and discussion

The transport properties of the rolled tapes with different number of rolling deformation passes are systemically investigated. Fig. 1 shows the field dependent transport J_c of all the tapes. The critical current density of all the samples exhibit a similar trend with the increase of magnetic field. The transport J_c of 3-pass, 5-pass and 7-pass samples are more than 10^4 A/cm^2 at 10 T, 4.2 K, indicating that the number of rolling passes can be reduced properly without J_c drop. But the J_c value of the 2-pass sample is a little lower than that of the other samples. The degree of grain alignment and the density of superconducting cores are important factors which influence the final transport performance [5,28]. Therefore, the XRD data and Vickers hardness value are shown in the next.

The XRD patterns for the rolled Sr-122 samples with different rolling passes are shown in Fig. 2. For comparison, the data for randomly orientated powder is also included. It can be seen clearly that all the rolled tapes exhibit a well-developed Sr-122 phase, and almost no impurity such as AgSrAs phase can be detected [5], which can be attributed to the chemical stability of the silver sheath. In order to make a clearer comparison of the *c*-axis texture, the XRD data of the rolled tapes was normalized by the intensity of the (002) peak. Compared with the randomly orientated powder, the relative intensity of the (00*l*) peak is strongly enhanced, indicating strongly *c*-axis orientation induced by the rolling deformation. The degree of *c*-axis texture is important to $Sr_{1-x}K_xFe_2As_2$ tapes [29]. It can be quantitatively evaluated with the orientation factor F by the Lotgering method [30]: $F = (\rho - \rho_0)/(1 - \rho_0)$, where $\rho = \Sigma I(00l)/\Sigma I(hkl)$, $\rho_0 = \Sigma I_0(00l)/\Sigma I_0(hkl)$. I and I_0 are the intensities of each reflection peak for the oriented and random samples, respectively. The *c*-axis orientation factor F value of the 2-pass, 3-pass, 5-pass and 7-pass samples are calculated to be 0.54, 0.53, 0.55 and 0.53, respectively. It can be seen clearly that all the rolled tapes exhibit a similar degree of grain alignment and the F value has a very small variation within 0.02. The degree of grain alignment for all the rolled samples is equal to that of the hot-pressing samples [5], but lower than that of the cold pressing tapes [25]. This result indicates that the number of rolling passes has no influence on the degree of grain alignment.

The core density is also an important parameter which correlate with the transport J_c of iron-based superconducting wires and tapes. The Vickers hardness is usually used to indicate the core