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Effects of high-pressure annealing on critical current density in 122 type iron pnictide wires

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ABSTRACT

We have fabricated $(Ba,K)Fe_2As_2$ and $(Sr,K)Fe_2As_2$ superconducting wires through an powder-in-tube (PIT) method using hot isostatic pressing (HIP) technique. The transport critical current densities (J_c) have reached 37 kA/cm^2 and 48 kA/cm^2 under self-field, and 3.0 kA/cm^2 and 1.8 kA/cm^2 under magnetic field of 90 kOe, at 4.2 K in the $(Ba,K)Fe_2As_2$ and $(Sr,K)Fe_2As_2$ wire, respectively. Magneto-optical (MO) imaging of the wires confirmed the large intergranular J_c in the wire core due to the improvement of couplings between superconducting grains by high-pressure annealing.

1. Introduction

Iron-based systems (IBSs), such as $LaFeAsO_{1-x}F_x$ [1] and $Ba_{1-x}K_xFe_2As_2$ [2], exhibit high critical temperature T_c, large upper critical field H_c, and relatively low anisotropy compared with cuprate superconductors, so they are very promising for high-field applications [3]. Promising candidates for applications in the iron-based superconductor family are the 122 type compounds, such as $(AE,K)Fe_2As_2$ (*AE* = alkali earths). They have small anisotropies of 2-3, moderate T_cs, large H_{c2}s, and large J_cs [4]. In order to apply 122 type superconducting wires in practical use, weak links between superconducting grains should be reduced because it have been considered to be the main reason for the low and strongly field-dependent transport J_c in the wire. The performance of the wire has been much improved by several methods, the combination of several times of cold press and hot press [5], addition of Ag, Pb, and Sn [6–11], or by texturing tape [9–11], and so on. In this paper, we demonstrate the enhancement of transport J_cs of $(Ba,K)Fe_2As_2$ and $(Sr,K)Fe_2As_2$ superconducting wires only by hot isostatic pressing (HIP) technique without other special treatment such as adding other metals or applying the texturing. This relatively simple process allows us to apply the fabrication of wire for industrial use such as cuprate superconducting wires [12].

2. Experimental detail

$(Ba,K)Fe_2As_2$ superconducting wires were fabricated by *ex-situ* powder-in-tube (PIT) method, and $(Sr,K)Fe_2As_2$ wires were prepared by *in situ* PIT method using $SrFe_2As_2$ and KFe_2As_2. $(Sr,K)Fe_2As_2$ was synthesized from the mixture of stoichiometric amounts of the ternary iron arsenide [13]. Polycrystalline $(Ba,K)Fe_2As_2$, $SrFe_2As_2$ and KFe_2As_2 samples were prepared by the solid-state reaction. We used Ba pieces, Sr pieces, K ingots, and FeAs powder as starting materials. FeAs was prepared by placing stoichiometric amounts of As pieces and Fe powder in an evacuated quartz tube and reacting them at 700 °C for 40 h after heating them at 500 °C for 10 h. A mixtures with a ratio of Ba:K:FeAs = 0.6:0.44:2 was placed in an alumina crucible and sealed in a stainless steel containers in a nitrogen-filled glove box, then heated for 5 h at 1100 °C after heating for 5 h at 600 °C. Mixtures with a ratio of Sr:FeAs = 1:2 and K:FeAs = 1.1:2 were also sealed in the same manner. A mixture of Sr and FeAs were heated at 1000 °C for 10 h after heated them at 1100 °C for 5 h. A mixture of K and FeAs were heated at 950 °C for 24 h. A prepared $(Ba,K)Fe_2As_2$ polycrystalline sample and a mixture of $SrFe_2As_2$ and KFe_2As_2 samples in a ratio of $SrFe_2As_2$:KFe_2As_2 = 6:4 were ground into fine powder in the

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nitrogen-filled glove box. They were filled in a silver tube with outer diameter (OD) 4.5 mm and inner diameter (ID) 3 mm, then cold drawn into a square wire with diagonal dimension of about 1.2 mm. They were put into 1/8 in. copper tube and redrawn. Both ends of wires were sealed using arc melting. The sealed wires were sintered using hot isostatic pressing (HIP) technique at Japan Atomic Energy Agency (JAEA). They were heated for 4 h at 600–700 °C in argon atmosphere under pressure of 120 MPa. The phase identification of the sample was carried out by means of powder X-ray diffraction (M18XHF, MAC Science) with Cu Kα radiation. Bulk magnetization is measured by a superconducting quantum interference device (SQUID) magnetometer (MPMS-5XL, Quantum Design). Resistivity and current–voltage (*I–V*) measurements up to 90 kOe were performed by the four-probe method with silver paste for contacts. *I–V* measurements were performed in a bath-type cryostat (Spectromag, Oxford Instruments). For MO imaging, the wire was cut and the surface was polished with a lapping film. An iron-garnet indicator film is placed in direct contact with the sample, and the whole assembly was attached to the cold finger of a He-flow cryostat (Microstat-HR, Oxford Instruments). MO images were acquired by using a cooled CCD camera with 12-bit resolution (ORCA-ER, Hamamatsu).

3. Result and discussion

Polycrystalline powders were characterized by powder X-ray diffraction and magnetization measurements. As shown in Fig. 1(a), the X-ray diffraction patterns have strong peaks of 122 phases and do not have peaks of impurity phase of FeAs, which indicate that the 122 phases reacted completely. For $(Ba,K)Fe_2As_2$ polycrystalline sample, the formation of superconducting phase was confirmed by the signal of diamagnetism as shown in Fig. 1(b). The shielding volume fraction for the $(Ba,K)Fe_2As_2$ reached about 110% at 5 K. Onset T_c in $(Ba,K)Fe_2As_2$ is approximately 38 K, which indicates that the composition of obtained $(Ba,K)Fe_2As_2$ is almost optimal potassium content of $x \sim 0.4$. T_cs of PIT wires were also characterized by magnetization measurement with magnetic field of 5 Oe parallel to the cross section of the wire. T_cs of $(Ba,K)Fe_2As_2$ and $(Sr,K)Fe_2As_2$ wires were approximately 30 K as shown in Fig. 1(c). Shielding volume fractions of both wires were more than 100%. Too high fractions may be derived from the shape of the wire. T_c of $(Ba,K)Fe_2As_2$ was reduced after HIP process. T_c of $(Sr,K)Fe_2As_2$ wire was lower than 37 K, which is the T_c of optimal composition of $(Sr,K)Fe_2As_2$ ($x \sim 0.4$) [13]. The distribution of potassium content in 122 phases and impurities may be main reasons for the reductions of T_c.

We investigated the transport J_c of superconducting wires. Fig. 2(a and b) shows the *E–J* characteristics of the wire of $(Ba,K)Fe_2As_2$ and $(Sr,K)Fe_2As_2$ under different magnetic fields at 4.2 K, respectively. Here we adopt $E = 1$ μV/cm as a criterion to define transport J_c. Transport J_cs as a function of magnetic field at 4.2 K for both wires are shown in Fig. 2(c). The transport J_cs at 4.2 K have reached 37 kA/cm^2 and 48 kA/cm^2 under self-field, and 3.0 kA/cm^2 and 1.8 kA/cm^2 under magnetic field of 90 kOe, in the $(Ba,K)Fe_2As_2$ and $(Sr,K)Fe_2As_2$ wire, respectively. Compared with $(Sr,K)Fe_2As_2$ wire, J_c in $(Ba,K)Fe_2As_2$ wire is lower under self-field and higher at high magnetic fields. The self-field J_c in the $(Ba,K)Fe_2As_2$ wire is several times larger than the J_c of $(Ba,K)Fe_2As_2$ wires sintered in vacuum [8]. Especially, they are almost 10 times larger at high magnetic fields. These results suggest that the HIP treatment of the wire effectively contributed for the improvement of J_c.

In iron-based superconductors, vortices at grain boundaries are generally pinned more weakly than vortices in the grains, and the grain boundaries become barriers to current flow. To investigate the quality of the grain boundaries, we performed MO measurement for the wires. Fig. 3(a and b) is optical images of the transverse cross section for the wires. Fig. 3(c and d) depicts MO images of the core region of the wires in the remanent state after applying an 800 Oe field for 0.25 s which was subsequently reduced down to zero at 4.2 K or 5 K. The bright regions correspond to the trapped flux in the wire core. They show uniform and fully trapped magnetic flux compared with our previous report about $(Ba,K)Fe_2As_2$ wires or other IBS wires and tapes [8,14]. It indicates very uniform bulk current flow is present. From the magnetic induction profile, we calculated the intragranular J_c, J_c^{intra}. In this calculation, we roughly estimate it as $J_c^{intra} = dB/dx$ [15]. Fig. 3(e and f) shows the magnetic induction profiles along the dashed arrows in Fig. 3(c and d). The J_c^{intra} decreases gradually as the temperature is increased toward T_c. In $(Ba,K)Fe_2As_2$ wire, trapped magnetic flux shows a smooth single peak and the estimated J_c^{intra} at 4.2 K is approximately 45 kA/cm^2, which is consistent with the value of transport J_c. This result suggests that HIP treatment should contribute to generate strong links between superconducting grains and to enhance intergranular J_c. Recently, it was reported that high core density is responsible for the high J_c performance in cold-pressed Ba 122 tape, which is evaluated by hardness measurement and microstructure investigation [16]. Analogies between HIP wire and cold-pressed tape suggest that the increase in the core density under high pressure is highly effective for the reduction of weak links between superconducting grains and for the enhancement of J_c. In $(Sr,K)Fe_2As_2$ wire, trapped magnetic flux is slightly inhomogeneous, although grains are well connected.

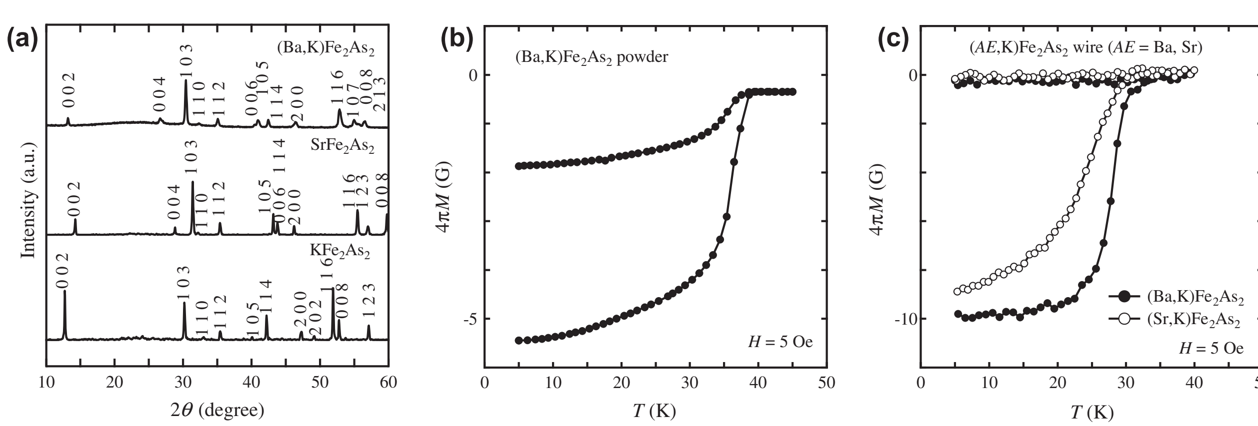

Fig. 1. (a) Powder X-ray diffraction patterns of $(Ba, K)Fe_2As_2$, $SrFe_2As_2$ and KFe_2As_2 polycrystalline powders. Temperature dependence of magnetization of (b) $(Ba, K)Fe_2As_2$ polycrystalline powder and of (c) $(Ba, K)Fe_2As_2$ and $(Sr, K)Fe_2As_2$ PIT wires.