Chemical stability and superconductivity in Ag-sheathed CaKFe₄As₄ superconducting tapes

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Abstract
Ag-sheathed CaKFe₄As₄ superconducting tapes have been fabricated via the ex situ powder-in-tube method. X-ray diffraction analyses suggest that the CaKFe₄As₄ phase reacts strongly with the silver sheath at temperature above 500 °C. We therefore anneal the tape at 500 °C and obtain a transport critical current density $J_c$ (4.2 K, 0 T) $\sim 2.7 \times 10^4$ A cm⁻². The pinning potential derived from magnetoresistance measurements is one order of magnitude lower than that of the (Ba/Sr)₁₋ₓKₓFe₂As₂ tapes. Combining with the scanning electron microscopy and magneto-optical imaging results, we suggest that bad connectivity between superconducting grains caused by the low sintering temperature is the main factor responsible for the low $J_c$. However, this system is still a promising candidate for superconducting wires and tapes if we find a compatible sheath material and further optimize the post-annealing process to achieve better grain connectivity.

Keywords: iron-based superconducting tapes, critical current density, powder-in-tube

Introduction
The iron-based superconductors (IBSs) are of great interest from a basic point of view as well as in light of practical application [1]. In spite of the large varieties of IBSs with different crystal structures discovered so far, vortex pinning and anisotropy place fundamental restrictions on the current carrying ability. Furthermore, the high field performance of iron-based superconducting wires and tapes fabricated by the powder-in-tube (PIT) method [2] depends on other extrinsic factors, i.e. the purity of the precursors, the compatibility to the sheath material, the density and texture of the superconducting core, and particularly, the homogeneity of the superconducting phase. After ten years of research and design, the optimally doped (Ba/Sr)₁₋ₓKₓFe₂As₂ has emerged as the dominant material used in iron-based superconducting wires and tapes. On one hand, the quality of the (Ba/Sr)₁₋ₓKₓFe₂As₂ precursor has progressively improved due to mature synthesis processes [3]. On the other hand, plenty of technologies, including flat rolling [4, 5], cold [6, 7] and hot [8] uniaxial pressing, hot isotropic pressing [9–11], as well as double sheath architecture [12] have been applied in the manufacturing process to enhance the texture and density of the superconducting core. Based on these efforts, the critical current density $J_c$ of the (Ba/Sr)₁₋ₓKₓFe₂As₂ superconducting tapes at 4.2 K and 10 T has been gradually improved and now surpasses the level needed for practical applications [13]. Nevertheless, like other doped IBSs, the
superconductivity of the BaK122 system is compromised by the non-uniformly distributed K atoms [14]. The inherent potassium clustering causes an inhomogeneous $J_c$ distribution due to its sensitivity to K content [15]. It deteriorates the overall current carrying capability of the iron-based superconducting wires and tapes, especially at long lengths. Consequently, finding a non-doped, stoichiometric IBS becomes vitally important for practical applications.

The recently discovered CaKFe$_4$As$_4$ superconductor is prominent among stoichiometric IBSs owing to its high superconducting transition temperature of $T_c$ at 16 K [16] which is competitive with the optimally K-doped FeAs122 system. In contrast to the (Ca$_{0.5}$Na$_{0.5}$)Fe$_2$As$_2$ solid solution with $\overline{4}/mmm$ space group, CaKFe$_4$As$_4$ possesses a $P4/mmm$ structure with the Ca and K layers alternately stacked between the Fe$_2$As$_2$ layers. Potassium doping on the alkaline-earth element site unavoidably introduces substantial disorders. Whereas CaKFe$_4$As$_4$ is structurally ordered with Ca and K occupying different layers. In addition, there is no structural or magnetic phase transition [17, 18], so one could consider this system as an optimally-doped ordered system. The upper critical field $B_{c2}$ of the CaKFe$_4$As$_4$ single crystal is 71 T with the field parallel to the $c$ axis and 92 T for the field perpendicular to the $c$ axis [17]. The anisotropy parameter $\gamma$ below 30 K is less than 2. The depairing current density $J_0$ at 0 K, estimated from $J_0 = \frac{\mu_0}{\pi \xi} \phi_0$, is 265 MA cm$^{-2}$, where $\phi_0$ is the flux quantum, $\xi = 2.15$ nm is the coherence length, $\lambda = 133$ nm is the penetration depth [19]. This $J_0$ value is even larger than seen in other FeAs122 superconductors [20]. Further magnetic measurements on CaKFe$_4$As$_4$ single crystals indicate that the critical current density $J_c$ at 10 T is nearly 1 MA cm$^{-2}$ with the field parallel to the $c$ axis [21]. Moreover, it exhibits a more robust temperature dependence than the BaK122 counterpart, making it very promising to be used not only at liquid helium temperature, but also at intermediate temperatures accessible with cryocoolers.

In this paper, we synthesize the CaKFe$_4$As$_4$ precursor with a two-step method. We find that the superconducting core of the CaKFe$_4$As$_4$ PIT tape reacts strongly with the Ag sheath during the post-annealing at temperature above 500 °C. Therefore, we sinter the tape at 500 °C and achieve a $J_c$ of 2.7 $\times$ 10$^4$ A cm$^{-2}$ at 4.2 K and self-field. We demonstrate that bad connectivity between superconducting grains due to low temperature sintering limits the overall critical current and indicates the need for a well-controlled post-annealing method to achieve better connectivity in this system.

Experimental details

In order to improve the homogeneity, we synthesized the precursor according to a two-step method [3]. We firstly prepared the intermediate compounds with nominal compositions CaAs and KAs at 700 °C and 400 °C, respectively. These intermediates were then mixed with Fe powders and As pieces according to the nominal composition Ca$_{1.15}$K$_{1.05}$Fe$_4$As$_4$. Excess CaAs and KAs were added to compensate for the loss of K and Ca during the heat treatment. The mixtures were ball milled for 10 h in Ar atmosphere, sealed in a Nb tube afterwards and directly inserted into a preheated furnace at 900 °C. After 30 h of sintering, the Nb tube was quenched to room temperature [16]. Considering the strong reaction between Ca/Fe and iron pnictides, we chose Ag as the outer sheath material. After grinding the CaKFe$_4$As$_4$ precursor into powders, we loaded them into a silver tube with outer and inner diameters of 8 mm and 5 mm, respectively. The tube was swaged, drawn and rolled into a 0.4 mm thick tape. We cut the tape into short samples and finally annealed them at temperatures between 500 °C and 800 °C.

The x-ray diffraction (XRD) patterns of the precursors and the tapes were performed on a Bruker D8 Advance x-ray diffractometer. We analyzed the diffraction patterns by Rietveld refinement. The thermogravimetric (TG) and differential thermal analyses (DTA) were performed on a synchronous thermal analyzer (Netzsch; STA 499 F3) at a heating rate of 20°C min$^{-1}$. The average composition of the superconducting core is determined by an electron probe micro-analyzer (EPMA). The transport critical current $I_c$ was measured at 4.2 K via a four-probe method with a criterion of 1 $\mu$V cm$^{-1}$ at the High Magnetic Field Laboratory (CHMFL, Hefei). We measured the resistivity of the superconducting core on a Physical Property Measurement System. The microstructure of the tape was analyzed by scanning electron microscopy (SEM, Zeiss SIGMAG). The magneto-optical (MO) image of the superconducting core was obtained using an optical cryostat (Montana Instruments) at Argonne National Laboratory. We polished the superconducting core after tearing off the silver sheath on that side. A Bi-substituted iron-garnet indicator film was placed in direct contact with the superconducting core as a magnetic field sensor [22]. The magnetic field generated from a homemade copper coil is applied with the direction perpendicular to the tape surface.

Results and discussion

Figure 1 shows the XRD pattern of the CaKFe$_4$As$_4$ precursor. We find that most of the diffraction peaks can be well indexed with the $P4/mmm$ space group. There are additional reflections evidencing the presence of a small amount of residual Fe with $Im-3m$ structure. However, we find no trace of KFe$_2$As$_3$ that is usually seen in the ‘1144’ polycrystals. We thus perform Rietveld refinement with two phases. The refinement is successful as indicated by the reliability factors: $R_p = 5\%$, $R_p = 6.59\%$. The fitted lattice parameters are $a = 3.861$ nm and $b = 12.830$ nm. The calculated fractions of the CaKFe$_4$As$_4$ phase and the Fe phase are 97.2% and 2.8%, respectively. The EPMA measurements on 20 points of the superconducting core indicate that the average composition is Ca$_{0.97}$K$_{1.02}$Fe$_4$As$_4$, close to the nominal composition.

So far, scientists have found various stable 1144 systems with chemical formula $AeAFe$_4$As$_4$, where $Ae$ and $A$ are the alkaline-earth and alkaline elements, respectively. The large
difference between the radii of the $Ae$ and the $A$ ions is necessary for the formation of the 1144 structure [16]. Song et al further utilized density functional theory to study the stability of the 1144 system [23] and found that the $P4/mmm$ structure is sensitive to temperature. It is only stable below a critical temperature of $T' \sim 780$ K ($507\, ^\circ\text{C}$). At higher temperatures, the 122 phase with $I4/mmm$ structure will dominate. In order to obtain a comprehensive understanding of the phase stability during the post-annealing process, we perform TG analysis and DTA on the precursor. Figure 2 shows the TG and DTA curves of the precursor under an argon atmosphere. The loss of the mass due to the evaporation of potassium is not as evident until the temperature is close to the melting temperature $T_m \sim 950\, ^\circ\text{C}$ at which point there is a sharp endothermic peak. At intermediate temperature, there is a sharp exothermic peak at $522\, ^\circ\text{C}$ as shown in the inset, which may be correlated with the critical temperature $T'$ predicted by the theoretical calculation.

In order to further verify this prediction, the CaKFe$_4$As$_4$ precursor is put in an alumina crucible, sealed in a silicon tube and reheated at $800\, ^\circ\text{C}$ for 0.5 h which is much higher than the $T'$. The precursor is then fast cooled to room temperature. However, the diffraction pattern of the precursor changes little after the reheating process, as shown in figure 3(a) and (b), indicating that the CaKFe$_4$As$_4$ phase is stable during the post-annealing process. Therefore, we anneal the CaKFe$_4$As$_4$ tapes at different temperatures for 0.5 h to optimize the sintering temperature. As shown in figure 3(c), all the diffraction peaks of the tape annealed at $500\, ^\circ\text{C}$ for 0.5 h can be well indexed with the $P4/mmm$ space group. There is no evidence for a secondary phase. One can see a great enhancement of the intensities of the $(00l)$ peaks, suggesting a strong $c$-axis texture. In order to quantitatively evaluate the $c$-axis texture, we utilize the Lotgering method to calculate $F = (\rho - \rho_0)/(1 - \rho_0)$ [24], where $\rho = \sum I(00l)/\sum I(hkl)$, $\rho_0 = \sum I_0(00l)/\sum I_0(hkl)$. $I$ and $I_0$ are the intensities of every peak for the textured and randomly oriented samples, respectively. The calculated $F$ value for the Ca1144 tapes annealed at $500\, ^\circ\text{C}$ is 0.565, which is intermediate between the Sr$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ superconducting tape [25] ($F \sim 0.476$) and the Ba$_{0.6}$K$_{0.4}$Fe$_2$As$_2$ superconducting tape [26] ($F \sim 0.590$). The large $F$ value of the CaKFe$_4$As$_4$ tape indicates that the cold working process including drawing and rolling can effectively align the grains with the $c$ axis perpendicular to the tape surface. When the post-annealing temperature increases up to $700\, ^\circ\text{C}$, obvious minor phases appear in the diffraction pattern, namely the KFe$_2$As$_2$ and the CaAgAs phases, as shown in figure 3(d). Moreover, sintering
at 800 °C largely suppresses the intensity of the CaKFe₄As₄ diffraction peaks, making KFe₂As₂ the predominant phase. Based on the above XRD results, we suggest that the CaKFe₄As₄ phase reacts strongly with the silver sheath and produces KFe₂As₂ and CaAgAs at temperature above 500 °C. KFe₂As₂ is a superconductor with diffraction peaks, making KFe₂As₂ the predominant phase. post-annealed at 500 °C, the critical current density exhibits a strong field dependence and decreases to a value at 10 T that is nearly one-tenth of that at self-annealed. The pinning potential at 0.5 T with the Werthamer–Helfand–Hohenberg formula is 242 T and 110 T for the field parallel and perpendicular to the tape surface, respectively. The anisotropy parameter of Bc₂, γ = Bc₂∥/Bc₂⊥, is 2.32 near Tc and decreases to 1.86 at 33.4 K, as shown in the inset of figure 5(b).

The tail structure in the magnetoresistance curves may imply a possible thermal activated flux flow (TAFF) behavior. According to the TAFF model, the resistivity can be expressed as ρ(T, H) = ρ(T = 0)exp(−U(T)/T))/U(T)/T, where U is the thermally activated energy or pinning potential. Assuming that ρ(T = 0, U/T) is a temperature independent constant, the thermal activation energy is U = U₀(1 − T/Tc), and we can obtain the Arrhenius relation:

\[ \ln \rho(T, H) = \ln \rho(T = 0) - U₀(T)/T + \ln(U₀(T)/T), \]

where lnρ(T = 0) = lnρ(T = 0) + U₀(T)/T. The pinning potential can be evaluated from the slope, U₀(T)/T. We depict the field dependences of U₀ for the two field directions in figure 5(c). The pinning potential at 0.5 T with the field perpendicular to the tape is 650 K. It is much smaller than the K-doped 122 single crystal and tape [13, 33], implying weak thermal barrier against vortex motion. The pinning potential shows a weak power-law field dependence, namely U₀ ∝ BΓ, with n = 0.12 and 0.14 for field perpendicular and parallel to the tape surface, respectively, manifesting a small anisotropy.

Figure 6(a) presents the optical image of the transverse section of the CaKFe₄As₄ tape showing a typical superconducting core with a saddle shape which arises due to the stronger deformation in the middle as compared to the two edges. Figure 6(b) shows a SEM image of the longitudinal section of the superconducting core. As a result of the low temperature sintering, the average size of the grains is as small as 2 μm. There are clearly observable voids and pores, limiting the actual current path along the tape. Figure 6(c) shows a MO image of the CaKFe₄As₄ superconducting tape. MO images reveal the normal component of the magnetic induction, that is, the vortex density, at the imaged sample section of the superconducting core. As a result of the low temperature sintering, the average size of the grains is as small as 2 μm. There are clearly observable voids and pores, limiting the actual current path along the tape. Figure 6(c) shows a MO image of the CaKFe₄As₄ superconducting tape. MO images reveal the normal component of the magnetic induction, that is, the vortex density, at the imaged sample section of the superconducting core.
density. The transverse feathering feature may be due to residual damage and cracking arising in the rolling process. The bright contrast along the center-line of the sample represents negative vortices which are generated by the return field from the vortices trapped in the thicker sample sections on either side. Based on the SEM and MO images, we suggest that bad connectivity between superconducting grains is responsible for the low $J_c$ at high magnetic field.
Conclusions

In summary, we have fabricated Ag-sheathed CaKFe$_4$As$_4$ tapes by ex situ PIT method. We find that CaKFe$_4$As$_4$ strongly reacts with the silver sheath during the post-annealing process at temperature higher than 500 °C. Therefore, we sintered the tape at 500 °C and obtained a self-field $J_c$ of 2.7 × 10$^4$ A cm$^{-2}$ at 4.2 K. The $J_c$ exhibits a strong field dependence and decreases to 2.2 × 10$^5$ A cm$^{-2}$ at 10 T. Magneto-transport and MO imaging reveal weak-link effects in the tape. The pinning potential derived from the magnetoresistance measurement is one order of magnitude smaller than that of the Ba$_{1-x}$K$_x$Fe$_2$As$_2$ tapes. Coupled with the SEM image, we conclude that the bad connectivity between the grains due to low temperature sintering is the main factor limiting the critical current density at high magnetic field. Considering the inherent high critical current density in the CaKFe$_4$As$_4$ single crystal, the polycrystalline wires and tapes are still promising for practical application if we use a sheath material that is chemically inert to CaKFe$_4$As$_4$ at high temperature and apply a proper post-annealing process to improve the grain connectivity.

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