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Preparation and superconductivity of MgB_2/Cu tapes

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Abstract

MgB_2/Cu tapes were fabricated by power-in-tube technique using MgB_2 superconducting powder and no post-annealing treatment was performed for the prepared tapes. Both X-ray diffraction and scanning electron microscopy results indicate that there is no obvious texture of grains for the tape samples. However, compared with the bulk sample, evident shifting and broadening of diffraction peaks is observed and T_c is reduced for the tape samples. In addition, it is found that there exists a remarkable difference in flux pinning ability for tapes prepared using wires with different diameters. The difference seems to originate from the different strain and microstructure defects of MgB_2 grains caused by cold working.

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Keywords: MgB_2/Cu tape; Flux pinning; Strain; Defect

1. Introduction

Akimitsu's group reported the superconductivity of MgB_2 on 10th January 2001 at a Conference in Sendai, Japan [1]. Since then it aroused great interest from many research groups. Compared with high temperature cuprate superconductors (HTS), MgB_2 has low anisotropy, large coherence lengths, and transparency of grain boundaries to current flow. Various research groups have already reported the fabrication of tapes, which is necessary for practical cable and magnet applications. At present, there are mainly two methods to fabricate MgB_2 tapes: the Mg diffusion into B tape or B wire method [2] and the power-in-tube (PIT) method [3–8]. Magnesium diffusion is a relatively easy method, which can rapidly convert commercially existent B wires into superconducting MgB_2 tapes [2,9]. However, the PIT method is the most popular for achieving good quality wires and tapes. The PIT approach has been used to fabricate metal-clad MgB_2 wires/tapes using various metals, such as: stainless steel SS [6,7], Cu [10], Ag [10], Ag/SS [10], Ni [11], Cu–Ni [7], Nb, Ta/Cu/SS [12], Fe [8,11]. In most works, researchers focused their interest on achieving high critical current J_c, little attention was paid to the influence of fabrication process on the superconducting properties of tapes. In this paper, MgB_2/Cu tapes were fabricated using wires with $\phi = 1.6$, 2.3 mm by the PIT technique and the flux pinning was investigated by resistance transition broadening measurement under various

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magnetic fields oriented normal and parallel to the tape surface, respectively.

2. Experimental

MgB_2 bulk samples were prepared by solid-state reaction method. Mg flakes $(1 + 5\%)$ and B powder were mixed and pressed into pellets. These pellets were wrapped up in Mo foil and then placed in Mo tubes and heated under a 95%Ar: 5%H_2 gas flow at 900 °C for 2 h. The samples were furnace-cooled to room temperature under mixed atmosphere. Then the samples were pulverized and the powders obtained were packed into Cu tubes with outer diameter of 4.9 mm and wall thickness of 1.3 mm. Then, they were drawn into wires with $\phi = 2.3$ and 1.6 mm, respectively. Short wires with length of ~2.0 cm were cut and uniaxially pressed into tapes with the dimension of $20 \times 4.72 \times 0.78$ mm^3 corresponding to $\phi = 2.3$ mm wire and $20 \times 4.32 \times 0.50$ mm^3 corresponding to $\phi = 1.6$ mm wire and no post-annealing process was performed. This two kinds of tapes are marked as tape A and tape B, respectively.

Powder X-ray diffraction (XRD) for the bulk samples was performed to identify their phase compositions using a Philips PW 1700 X-ray diffractometer with Cu K_a radiation. For the tape samples, XRD were carried out after the surface Cu metal of the tape was cleaved. The microstructure images of the samples were observed by scanning electron microscopy (SEM). The resistance transition broadening was measured by the standard four-probe method under applied magnetic field oriented parallel and perpendicular to the tape surface, respectively.

3. Results and discussion

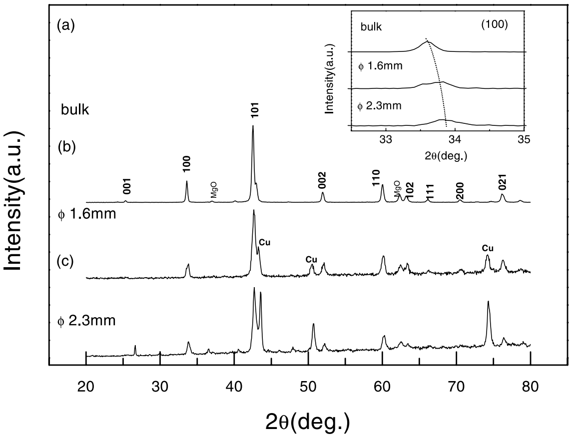

Fig. 1. The XRD patterns of MgB_2 bulk sample and MgB_2/Cu tape samples (tapes A and B). Inset is the enlarged plot of (1 0 0) diffraction peak.

The powder XRD patterns of bulk sample and the surface XRD patterns of tapes A and B are shown in Fig. 1(a)–(c), respectively. For bulk sample, Fig. 1(a) shows that the main phase is MgB_2 with a small amount of MgO and no other extra diffraction peak is observed. For the tapes, Fig. 1(b) and (c) shows diffraction peaks from the Cu sheath in addition to diffraction peaks observed in bulk sample. No obvious texture of grains is observed from XRD patterns for the tape samples, which means it is not easy to form texture by cold working. However, it is found that the peak positions of the MgB_2 phase in the tape samples shift to high angle obviously compared with that of MgB_2 phase in bulk samples, which indicates that cold working gives rise to the variation of the lattice parameter of MgB_2 grains. In addition, evident peak broadening is also observed in XRD patterns of the tape samples, which implies cold working also result in a serious lattice distortion of MgB_2 grains. Shifting and broadening of diffraction eak can be seen clearly in inset of Fig. 1, which shows the (1 0 0) peak of MgB_2 phase for the bulk and tape samples.

SEM is employed to characterize the microstructure of the samples. The surface micrographs of the bulk sample and cross-section micrographs of the tape samples are shown in Fig. 2(a)–(c), respectively. Compared with the bulk, the grain size of tapes is reduced. However, no evident texture of MgB_2 grains is observed from the SEM images of tape samples, which is consistent with the results obtained from XRD of tapes.

Fig. 3 shows that temperature dependence of the resistance at zero magnetic field for bulk sample and tapes. It indicates that the initial transition temperature T_c^{onset} for bulk and tapes A and B is

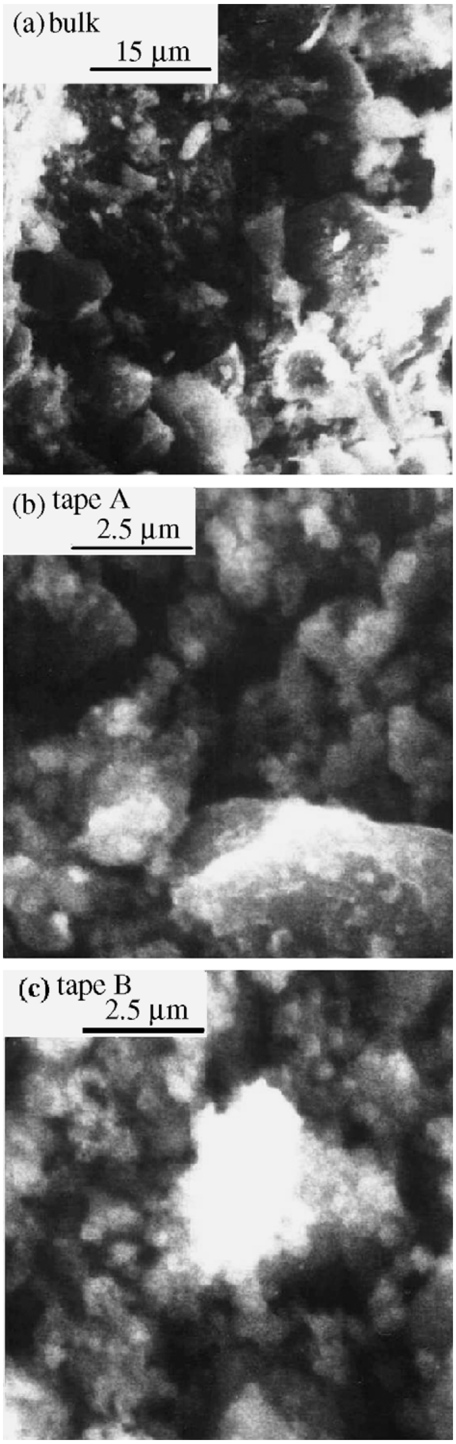

Fig. 2. The SEM images of the samples: (a) bulk sample at a magnification of 1000×; (b) tape A at a magnification of 6000×; (c) tape B at a magnification of 6000×.

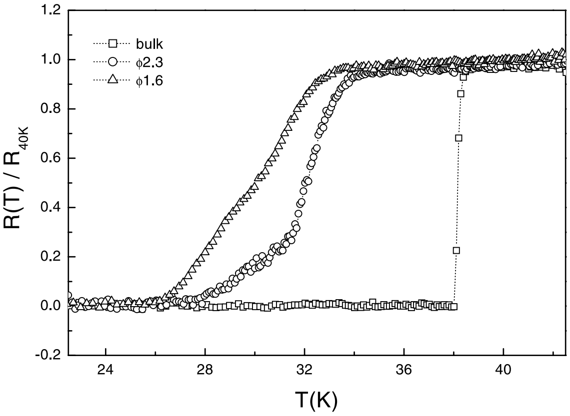

Fig. 3. The temperature dependence of the normalized resistance of bulk and tapes under zero field.

38.3, 33.6 and 32.7 K respectively. Compared with the bulk sample, T_c of the tapes decreases and the transition width increases remarkably. This difference may be attributed to the strain and defects produced during cold working process.

To compare the flux pinning properties of tapes A and B, the resistance transition of the tapes under the action of applied magnetic fields oriented parallel and perpendicular to the tape surface is measured. The measurement results are shown in Fig. 4(a) and (b), respectively. Fig. 4 demonstrates that $R(T)$ curves shift remarkably to low temperatures with the increase of applied magnetic field. For tape B, Fig. 4(a) shows that $R(T)$ curves have not an obvious difference for two kinds of different field orientations, which also implies there does not exist obvious grain texture. For tape A, Fig. 4(b) shows that there exists a difference under two kinds of different field orientations particularly at high magnetic fields, i.e., the shift of $R(T)$ toward low temperatures for $H \parallel$ tape surface is less than that for $H \perp$ tape surface. This implies there exists partial grain orientation due to the cold working though XRD and SEM cannot detect this orientation.

Here we define the zero resistance transition temperature under a fixed magnetic field as the irreversibility temperature T_{irr} and the corresponding magnetic field as the irreversibility field H_{irr}. Fig. 5 shows the irreversibility lines of tapes A and B. It is found that the irreversibility line of

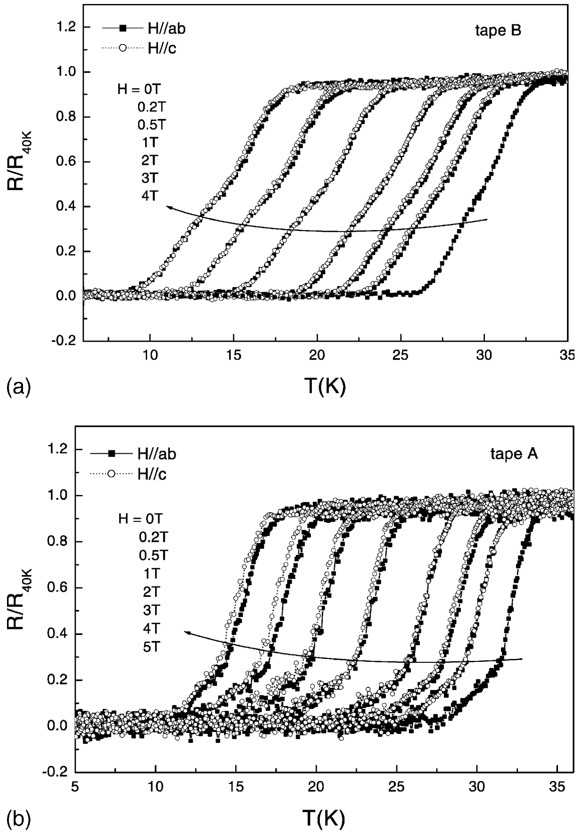

Fig. 4. (a) The temperature dependence of the normalized resistance of tape B at various magnetic fields oriented normal and parallel to the tape surface; (b) the temperature dependence of the normalized resistance of tape A at various magnetic fields oriented normal and parallel to the tape surface.

tape A is shifted to higher temperatures and magnetic fields compared with tape B, particularly in the low-temperature range, which indicates that the flux pinning ability of tape A is superior to tape B. The data shown in Fig. 5 can be fitted according to the equation:

$$1 - T_{\mathrm{irr}}/T_{\mathrm{c0}} = AH_{\mathrm{irr}}^{q} \qquad (1)$$

where T_{c0} is the zero resistance temperature at zero field, A and q are fitting parameters. The fitting results show that $A = 0.23465$; $q = 0.64529$ for tape A and $A = 0.28162$; $q = 0.6038$ for tape B, which is similar to the result obtained in HTS [13]. The solid lines shown in Fig. 5 are fitting lines.

We define the onset transition temperature under the fixed magnetic field as the upper critical temperature T_{c2} and the corresponding magnetic field as the upper critical field H_{c2}. The upper critical field versus temperature for tape samples is shown in Fig. 6. It is found that the $H_{\mathrm{c2}}(T)$ line of tape A is also higher than that of tape B in H–T plane. In addition, for tapes A and B, the variation of $H_{\mathrm{c2}}(T)$ is more slowly than predicated by the Werthamer–Helfand–Hohenberg model [14]:

$$\mu_0 H_{\mathrm{c2}}(0) = 0.7T_{\mathrm{c}}(\mathrm{d}H_{\mathrm{c2}}/\mathrm{d}T)|_{T_{\mathrm{c}}}.$$

Compared to the tape samples with bulk sample, on the one hand, the cold working plays a role of deteriorating the superconducting properties of MgB$_2$, on the other hand, the cold working also

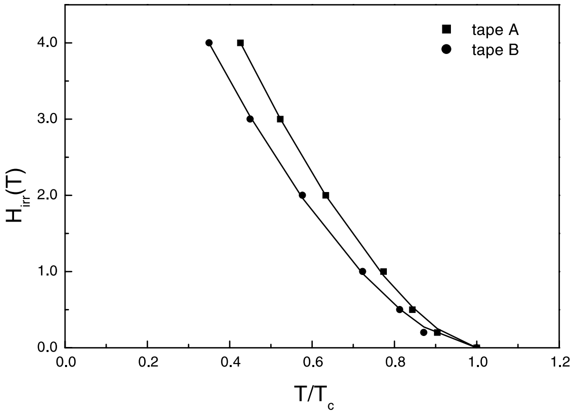

Fig. 5. The irreversibility field versus normalized temperature for tapes A and B. The solid lines are fitting lines according to Eq. (1) (see text).

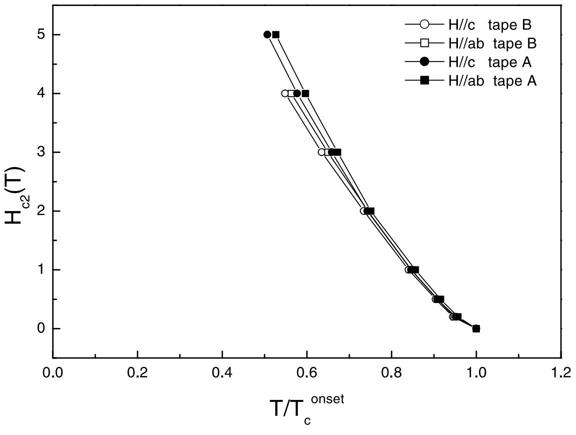

Fig. 6. Upper critical field versus normalized temperature for tapes A and B.

introduces lattice distortion and microstructure defects, which seems to be beneficial to flux pinning.

4. Conclusions

MgB_2/Cu tapes were fabricated by PIT technique using MgB_2 superconducting powder and no post-annealing treatment was performed for the prepared tapes. Both XRD and SEM results indicate that cold working cannot result in obvious texture of grains of MgB_2. However, compared with the bulk sample, cold working can gives rise to evident lattice distortion and microstructure defects. Our results indicate that a modest cold working during the preparation of tapes and post-annealing treatment of tape may be necessary to obtain high quality MgB_2 tape materials.

Acknowledgements

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References

[1] J. Akimitsu, Symposium on Transition Metal Oxides, 10 January 2001, Sendai, Japan, 2001.
[2] G.C. Che et al., cond-mat/0105215, 2001.
[3] G. Grasso et al., Appl. Phys. Lett. 79 (2001) 230.
[4] M.D. Sumption et al., cond-mat/0102441, 2001.
[5] C.F. Liu et al., cond-mat/0106061, 2001.
[6] K.J. Song et al., cond-mat/0106124, 2001.
[7] H. Kumakura et al., cond-mat/0106002, 2001.
[8] S. Soltanian et al., Phys. C 361 (2001) 84.
[9] P.C. Canfield et al., Phys. Rev. Lett. 86 (2001) 2423.
[10] B.A. Glowacki et al., Supercond. Sci. Technol. 14 (2001) 193.
[11] H.L. Suo et al., cond-mat/0106341, 2001.
[12] W. Goldacker et al., Supercond. Sci. Technol. 14 (2001) 787.
[13] D.S. Fisher et al., Phys. Rev. B 43 (1991) 130.
[14] N.R. Werthamer et al., Phys. Rev. 147 (1996) 288.