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High critical current density in Ti-doped $MgB_2/Ta/Cu$ tape by powder-in-tube process

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Ti-doped MgB_2/Cu tapes with Ta as a buffer layer were prepared through the *in situ* powder in tube method by using Mg, Ti, and B powders. The phase compositions, microstructure features, and superconducting properties were investigated by x-ray diffraction, scanning electron microscope, and superconducting quantum interference device magnetometer. It is found that TiB_2 phase was formed in Ti-doped MgB_2 tape. Magnetization measurement results show that the critical transition temperature of $MgB_2/Ta/Cu$ tape with Ti doping is around 38 K. The irreversibility field H_{irr} and critical current density J_c can be greatly enhanced by Ti doping. H_{irr} of the $Mg_{0.9}Ti_{0.1}B_2$ tape is as high as 7.4 T at 10 K. The high critical current density J_c of 1.5×10^6 A/cm^2 (10 K, self field) and 9.3×10^5 A/cm^2 (20 K, self field) are obtained in the $Mg_{0.9}Ti_{0.1}B_2$ tape. In addition, a suitable amount of Ti doping can lead to a high density and fine grain size of MgB_2, which may be the reason for high J_c in Ti-doped MgB_2 tapes. © *2002 American Institute of Physics.*
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INTRODUCTION

The recent discovery of a MgB_2 superconductor with critical temperature T_c around 40 K[1] has generated much interest in both fundamental research and applications. The crystal structure of this material is an hexagonal AlB_2 type structure consisting of alternating layers of Mg atoms and boron honeycomb layers. In addition, unlike high temperature superconductors, MgB_2 has no weak-link problem at grain boundaries,[2,3] providing a high feasibility for fabricating this material to form wires and tapes. Furthermore, the progress in the cryogen free cooling technique at the temperature region of 20–30 K will promote the development and applications of MgB_2. It is reported that high dense MgB_2 wires (160 μm in diameter) were fabricated by the exposure of boron filaments to Mg vapor.[4] The recent results of preparation of MgB_2 wires by powder-in-tube (PIT) using either Ag or Cu sheaths were the first steps to put MgB_2 into application. High critical current density J_c up to 10^4-10^5 A/cm^2 at 10 K were also obtained in the MgB_2 composite wires and tapes with mechanically reinforced sheath.[5] However, the J_c value of MgB_2 tapes is relatively low compared to the conventional low temperature superconductors, due to poor grain connections and the lack of flux pinning centers in this material. Some reports indicated that the microstructure and superconducting properties of superconductors can be easily improved by chemical doping in high temperature superconductors.[6] Recently, it has been found that the partial substitution of Zn or Cu for Mg in MgB_2 will lead to a deteriorative effect on T_c. [7] The largest reduction of T_c was induced by Mn doping, followed by Co, C, Al, Ni, and Fe.[8] These investigations were focused on the transition temperature of MgB_2, not on the flux pinning behavior. Zhao *et al.*[9] studied the effect of Ti doping in MgB_2 bulks. At 5 K, the J_c reached 2×10^6 A/cm^2 in the self field. They did not research the superconducting properties of Ti-doped MgB_2 tape. In this work, we fabricated Ti-doped MgB_2/Cu tapes with Ta as a buffer layer by the *in situ* PIT method at ambient

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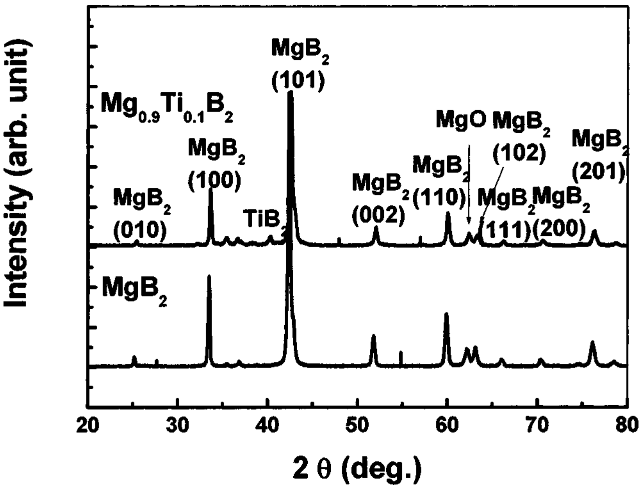

FIG. 1. X-ray diffraction patterns of MgB_2 tape with and without Ti doping. Few impurity phases of MgO are found in both tapes. TiB_2 phases are found in Ti-doped tape.

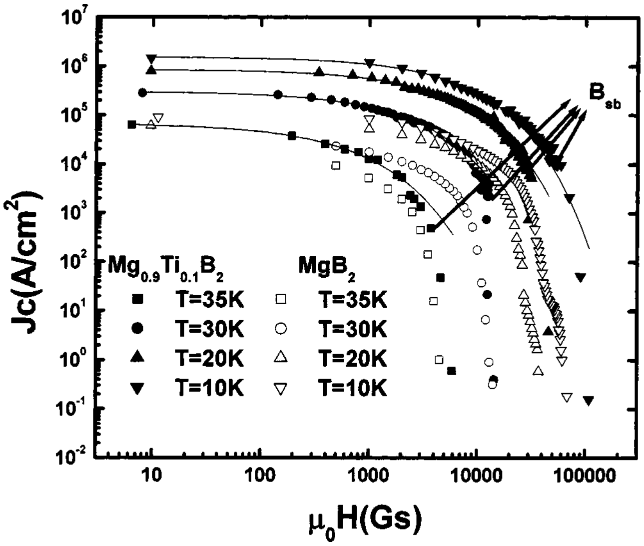

FIG. 2. Magnetic field dependence of J_c for MgB_2/Ta/Cu tape with and without Ti doping at different temperatures. Fitting curves of the equation $J_c(\mathrm{B}) = Jc(0)\exp(-(B/B_0)^{0.65})$ are also shown.

pressure and presented the superconducting properties, structure, and microstructure results. It is found that the Ti doping can significantly enhance the flux pinning. The high critical current density of 1.5×10^6 A/cm^2 (10 K, self field) and 9.3 $\times10^5$ A/cm^2 (20 K, self field) were obtained in $Mg_{0.9}Ti_{0.1}B_2$ tape. Also, the reasons for high J_c in this sample were briefly discussed.

EXPERIMENTS

The MgB_2 composite tapes with and without Ti doping were prepared by the *in situ* PIT method. Mg(99%), Ti(99%), and B(99%) powders were well mixed in air for a short time. 6 wt % extra Mg powder was added in the precursor in order to compensate for the loss of Mg at high temperature. Then, the mixture was filled into a tantalum tube of 7 mm outside diameter and 1.5 mm wall thickness and sealed in a copper tube of 9 mm diameter and 1 mm wall thickness. The tantalum tube was used to prevent the reaction between MgB_2 and Cu effectively. Then the tubes were swaged and drawn to a wire of 2 mm in diameter. The wires were subsequently rolled to tapes of 3.4×0.25 mm. Finally, the tapes were sintered at 900–950 °C for 2–3 h in argon at ambient pressure and followed by furnace cooling to room temperature. The phase analysis was carried out using x-ray diffractometer. The magnetization measurements were performed on a commercial superconducting quantum interference device magnetometer at different temperatures in the magnetic field up to 10 T and the microstructure was examined by a scanning electron microscope (SEM). The transport currents were measured by the standard four-probe technique. The criterion was 1 μV/cm. The available cross section areas of $Mg_{0.9}Ti_{0.1}B_2$ tape were about $0.6-0.7\,\mathrm{mm}^2$. All the samples for this study have the same dimensions of $0.2\times0.2\times0.02\,\mathrm{mm}^3(a\times b\times c)$. The field direction is perpendicular to the ac plane.

RESULTS AND DISCUSSION

The typical x-ray diffraction (XRD) patterns of pure MgB_2 and Ti-doped tapes are illustrated in Fig. 1. It can be observed that the main phase is MgB_2 in the pure MgB_2 sample while only a few impurity phases of MgO are found in the spectrum. However, some TiB_2 phases and fewer MgO phases can be detected besides the MgB_2 phase. Moreover, no MgB_4 is observed in these two samples. The XRD analysis shows that the lattice parameter is not changed by Ti doping, suggesting that Ti atoms do not enter the structure of MgB_2. The critical temperature (38 K) of the Ti doped sample is slightly lower than that of pure MgB_2 (38.4 K).

Figure 2 shows the magnetic field dependence of J_c for the MgB_2/Ta/Cu tapes with and without Ti doping at different temperatures. The J_c values were deduced from the hysteresis loops by using the Bean model of $J_c = 30\Delta M/d$ (where d is the diameter of the sample).[10] In this figure, the symbols presented the experimental data and the lines are the fitting curves of the equation $J_c(\mathrm{B}) = J_c(0)\exp[-(B/B_0)^{0.65}]$. It can be observed that the J_c value is significantly improved by the Ti doping in MgB_2 tape. At 10 K and self field, the J_c of the Ti-doped tape reaches a high value above 1.5×10^6 A/cm^2. As the magnetic field is increased to 1 T, J_c is as high as 2.7×10^5 A/cm^2. However, J_c is only around 1.9×10^4 A/cm^2 at 10 K in 1 T for pure MgB_2 tape. It is worth noting that at 20 K, J_c achieves 9.5×10^5 A/cm^2 in self field. This result means that MgB_2 can be applied in temperatures around 20 K and becomes a possible candidate to possibly substitute the low temperature superconducting materials. Figure 3 illustrates the results of field dependent magnetic J_c and transport J_c for $Mg_{0.9}Ti_{0.1}B_2$/Ta/Cu tape at 20 and 30 K. Because of the limit of measure conditions and heat effects of samples, the maximum measure current is only 80 A. In 20 K, the critical transport current reaches 71 A in 1 T field. They are well fit with the curves of magnetic current density. In addition, a plateau region of J_c can be observed from Fig. 2 at low magnetic fields. At this stage, J_c

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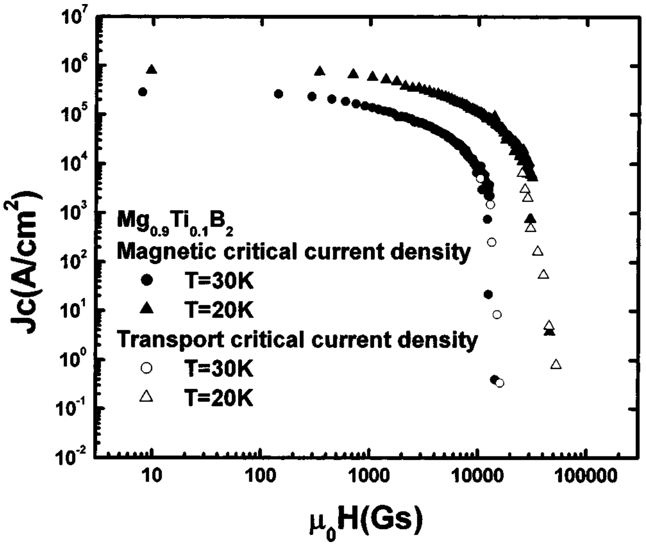


FIG. 3. Magnetic field dependence of magnetization J_c and transport J_c for $Mg_{0.9}Ti_{0.1}B_2$/Ta/Cu tape at 20K and 30K.

has a weak dependence on the field. But when the magnetic filed is increased above a crossover field B_{sb}, J_c begins to decrease quickly. Also, the crossover field decreases with increasing temperature. The crossover field of Ti doping MgB_2 tapes is relatively high compared to pure MgB_2, which indicates the $Mg_{0.9}Ti_{0.1}B_2$ sample has a very strong flux pinning ability. Furthermore, when the field is lower than crossover field B_{sb}, it is found that the experimental data are well fit by $J_c(\mathrm{B}) = J_c(0)\exp[-(B/B_0)^{0.65}]$ at temperatures below 35 K in the $Mg_{0.9}Ti_{0.1}B_2$ tape. This field dependence of J_c suggests that the dominant pinning mechanism in the $Mg_{0.9}Ti_{0.1}B_2$ tape may be explained by the core pinning model[11] when the applied field is lower than B_{sb}. However, when the applied magnetic field is larger than B_{sb}, the law of this curve submits $J_c(B) \sim B^{-3}$. It indicates that three-dimensional pinning model has taken effect.[11] However, a previous result showed that Ti doping decreased the critical current density in MgB_2 tapes. The reason is that the Ti doping level is only 5 mol %.[12] In our work, the tape with 5 mol % Ti doping also deteriorates the critical current density. When the Ti doping level reaches 10 mol %, the J_c value increases significantly. Figure 4 illustrates the temperature dependence of the irreversibility field H_{irr} for $Mg_{0.9}Ti_{0.1}B_2$ and MgB_2 tapes. The H_{irr} value was determined from the closure of the magnetization curves. The H_{irr} achieves 7.4 T at 10 K and 1.4 T at 30 K in $Mg_{0.9}Ti_{0.1}B_2$, which is similar to the result in hot pressed MgB_2 bulks.[13] At 10 K, H_{irr} is increased from 6.1 T in pure MgB_2 to 7.4 T in $Mg_{0.9}Ti_{0.1}B_2$ but the H_{irr} value merges at high temperature, suggesting that the flux pinning ability in the Ti-doped sample at high temperature is not much improved in high fields.

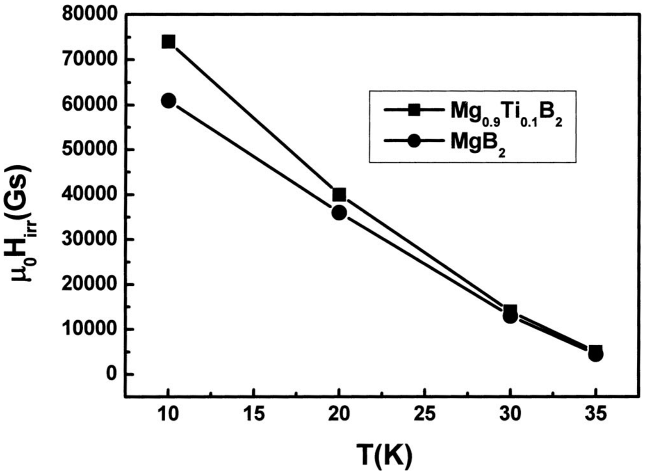
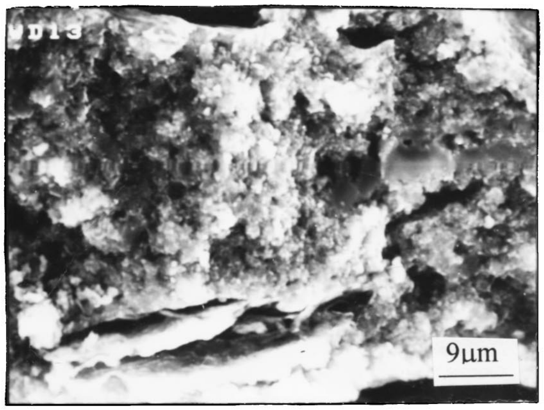


FIG. 4. Temperature dependence of irreversibility field for MgB_2/Ta/Cu tape with and without Ti doping



(a) Pure MgB_2

(b) $Mg_{0.9}Ti_{0.1}B_2$

FIG. 5. Typical SEM photographies for the MgB_2 tape with and without Ti doping. (a) Pure MgB_2 (b) $Mg_{0.9}Ti_{0.1}B_2$.

The typical SEM photograph of the cross section in pure MgB_2 and $Mg_{0.9}Ti_{0.1}B_2$ tapes is given in Fig. 5. More voids formed by the evaporation of Mg can be observed in the pure MgB_2. However, the $Mg_{0.9}Ti_{0.1}B_2$ sample has a much higher density with few voids. In fact, with Ti doping, the temperature of forming the MgB_2 phase is higher than pure MgB_2. Also, the connections between grains are much improved and the fine grains of MgB_2 are found in the Ti-doped tape. A very thin layer of TiB_2 forms around the MgB_2 particles and MgO nanoparticles are observed in Ti-doped MgB_2 samples.[9] Therefore, it can be concluded that the TiB_2 phases in the tapes may prevent the growth of grain size of MgB_2

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and lead to very fine MgB_2 particles. Meanwhile, the fine grain size creates many grain boundaries that may act as the important pinning centers in MgB_2 material and enhances the critical current density of MgB_2.

SUMMARY

In summary, the dense Ti-doped MgB_2/Cu tapes with Ta as a buffer layer are successfully prepared through the *in situ* PIT method. The tapes were sintered at 900–950 °C for 2–3 h in argon. XRD results indicate that the main phase is MgB_2 in the Ti-doped tape, including a few TiB_2 and MgO phases. The irreversibility field is remarkably improved from 6.1 T in pure MgB_2 to 7.4 T at 10 K and 1.4 T at 30 K in the $Mg_{0.9}Ti_{0.1}B_2$ tape. The J_c value is significantly increased to 1.5×10^6 A/cm^2 at 10 K in self field and 2.7×10^5 A/cm^2 at 10 K in 1 T by Ti doping, which is much higher than the best data on other MgB_2 wires and tapes. Microstructure analysis shows that the tapes with Ti doping are very dense and have fine MgB_2 particles, with a thin layer of TiB_2 and MgO nanoparticles, which may be responsible for the significant enhancement of J_c by Ti doping.

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