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Nano-sized Al_2O_3 doping effects on the critical current density of MgB_2 superconductors

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Abstract

MgB_2 polycrystalline bulk samples with addition of 5 and 10 wt.% nano-sized Al_2O_3 powders were prepared by solid state reaction using pre-reacted MgB_2. All the samples were sintered at 850 °C for 40 min in Ar. All the samples were characterized by X-ray diffraction, scanning electron microscopy and magnetic measurements. Results show that the critical current density and irreversibility fields decrease significantly with increasing Al_2O_3 level. J_c values decreased significantly by more than one order of magnitude. The T_c drops slightly from 37.9 to 36.6 K, but acquires a very wide transition width of more than 20 K. Furthermore, the amount of MgO was found to increase with increasing Al_2O_3 level, probably indicating that Mg was replaced by Al, with the excess Mg forming extra MgO. The field dependence of J_c for all the samples is also presented.

Keywords: C. Superconductivity; Magnesium diboride; Chemical doping

1. Introduction

The critical current density (J_c) in MgB_2 has been a central topic for extensive research efforts since the discovery of superconductivity in this compound [1]. Many groups have attempted to improve the critical current density in this superconductor as it has lower H_{c2} and H_{irr} than the commercial low temperature superconductors Nb_3Sn and NbTi. High critical current density values above 10^5 A/cm^2 have been achieved in MgB_2 both in pellets and tapes [2,3]. Experiment results revealed that J_c drops rapidly with increasing magnetic field due to poor flux pinning, although grain boundaries are transparent to the current flow in MgB_2 [4]. Therefore, extensive research has been done on introducing pinning centers into this superconductor. It has been found that inclusions of oxygen or precipitates of nano-MgO can act as effective pinning centers in MgB_2 thin films [5]. Nano-sized chemical inclusions such as Si, SiO_2 and Y_2O_3 were also reported to enhance flux pinning [6–8]. Dou et al. [2] have achieved a J_c enhancement in high fields by more than one order of magnitude, with only slight reductions in T_c by chemical doping of MgB_2 with nano-particle SiC. It also has been found that although C tends to severely react with MgB_2, by controlling the preparation procedure, nano particle C doping can also enhance the J_c at high field [9]. Slusky et al. [10] have shown that Al can aggressively react with MgB_2 and substitute into the Mg position, leading to a loss of superconductivity. However, it was found that Al_2O_3 is more stable than SiO_2 in terms of reaction with MgB_2 [11]. Therefore, Al_2O_3 could be a possible candidate chemical dopant, if doping can produce nano-sized precipitates to act as pinning centers. The magnetic field performance of MgB_2 could then be improved by doping with Al_2O_3. The objective of this paper is to study the effects of Al_2O_3 doping on the superconductivity and flux pinning of MgB_2 superconductor.

2. Experimental

To avoid a high level of Al substitution in the Mg position, pre-reacted and commercially available MgB_2 powder has been used in this work. Samples were prepared using commercially available high purity (99%) MgB_2 (Alfa

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Acer) and nano-sized Al_2O_3 with an average particle size of 30 nm. The starting powders were weighed out according to the nominal composition of $MgB_2 + x$ wt.% Al_2O_3 ($x = 0$, 5, 10). Powders were well mixed through grinding by mortar and pestle. Pellets 10 mm in diameter and 3 mm in thickness were made, then sealed in an Fe tube and subjected to heat treatment at a temperature of 850 °C for 40 min in flowing high purity Ar. The phase formation and microstructures of the samples were determined and investigated by SEM and XRD. The magnetization of samples was measured over a temperature range of 5–30 K using a Physical Properties Measurement System (PPMS). Samples were cut into the form of bars from the as-sintered pellets. All the samples have almost the same size (0.5 mm × 1.7 mm × 2.9 mm). The T_c was determined by measuring the real part of the ac susceptibility at a frequency of 117 Hz and an external magnetic field of 0.1 Oe. T_c was defined as the onset of diamagnetism.

3. Results and discussion

Fig. 1 shows the XRD patterns for the three different samples prepared for this work. All the samples are revealed to be single phase and can be very well indexed with the space group *P*6/*mmm*. It can be seen that there are no diffraction peaks related to Al_2O_3 in the doped samples, indicating that this powder is reacted with the MgB_2 during the heat treatment. The pure sample consists of a main phase, MgB_2, with minor phases of MgO (<5%) and MgB_4. The amount of MgO impurity increases as the doping level increases. This is possibly due to the decomposition of Al_2O_3, making oxygen available for Mg oxidation.

Fig. 2 shows the T_c for the doped and undoped samples determined by ac susceptibility measurements. The T_c onset for the undoped sample is about 37.9 K which is slightly lower than for samples prepared by the reaction in situ technique [2]. This sample also shows a relatively sharp transition with a transition width of about 2.1 K, larger than the transition width of samples made using the reaction in situ [2]. For the doped samples, the T_c decreases with increasing doping level. T_c values of about 36.9 and 36.6 K were found for the 5 and 10 wt.% doped samples, respectively. Despite the small reduction in the T_c values of the samples, the doped samples show a wide transition width above 20 K. This is in agreement with the increasing amount of MgO impurities in the samples, with the connection possibly due to the oxidation of the surface of grains, resulting in weak links. The grains are disconnected at high temperature, then gradually become connected at lower temperature which results in a large transition width. This phenomenon can be seen also in the weak link superconductors such as YBCO.

An inductive J_c was derived from the height of the magnetization loop ($M - H$) using the Bean Model ($J_c = 20\,\Delta M/[a(1 - a)/3b)$)] with $a < b$ and magnetic field parallel to the longest sample direction). The measured magnetic hysteresis loops at 5, 10, 15, 20, 25, and 30 K for the pure MgB_2 sample are presented in the inset of Fig. 2.

J_c values versus field at 5, 10, 20 and 30 K are plotted in Figs. 3 and 4. Zero field J_c values as high as 4×10^5 and

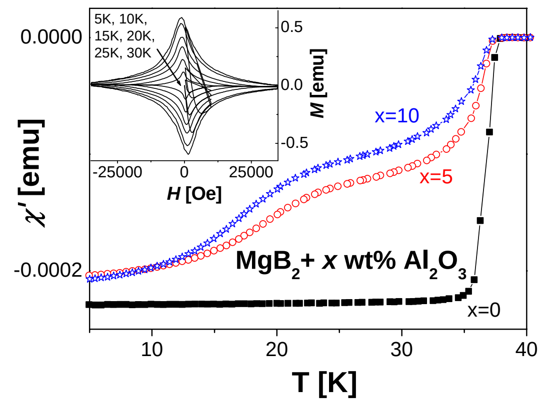

Fig. 2. The Ac susceptibility of MgB_2 samples doped with 0, 5 and 10 wt.% nano-sized Al_2O_3. The inset shows the magnetic hysteresis loops for the pure sample at 5–30 K.

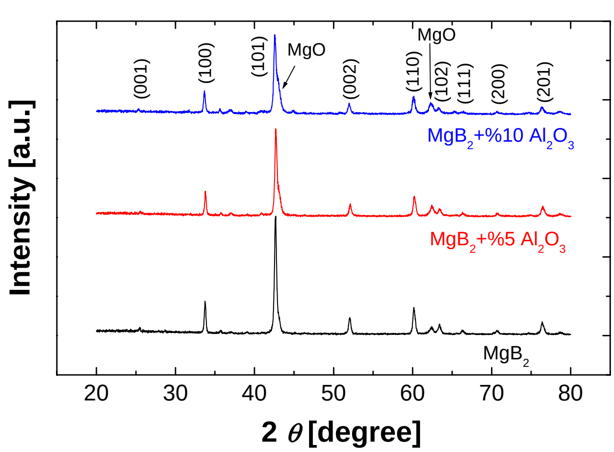

Fig. 1. XRD patterns of $MgB_2 + x$ wt.% Al_2O_3 composition for $x = 0$, 5, 10.

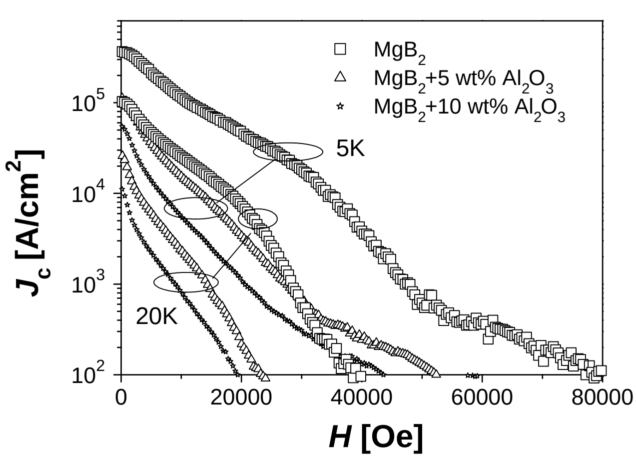

Fig. 3. The J_c field dependence of MgB_2 samples doped with 0, 5 and 10 wt.% Al_2O_3 at 5 and 20 K.

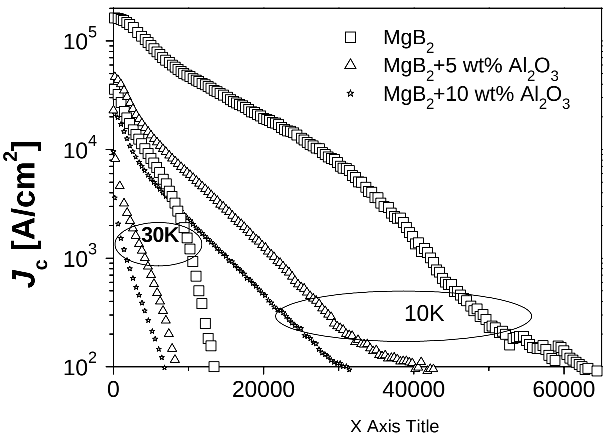

Fig. 4. The J_c field dependence of MgB_2 samples doped with 0, 5 and 10 wt.% Al_2O_3 at 10 and 30 K.

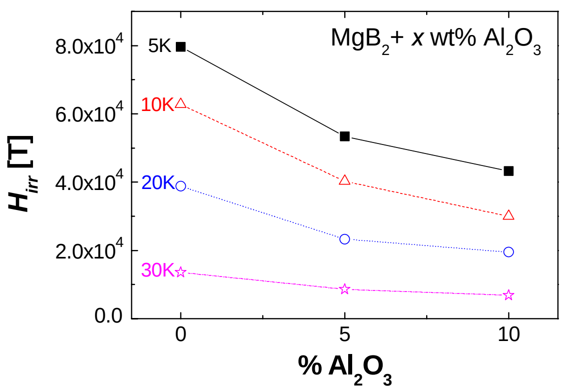

Fig. 5. The irreversibility field for MgB_2 samples doped with 0, 5 and 10 wt.% of nano-sized Al_2O_3 at 5, 10, 20, and 30 K.

2×10^5 A/cm^2 were found for the pure samples at 5 and 10 K, respectively. However, the J_c values decrease rapidly as the magnetic field increases. This again confirms that samples made using the reaction in situ have better performance than samples prepared using pre-reacted powder [2]. For doped samples, the zero field J_c was significantly decrease, together with the performance of the J_c field dependence. This deterioration is greater for sample with higher doping level.

Fig. 5 shows the irreversibility field for pure and doped samples. Here, we defined H_{irr} as the field where J_c drops to 100 A/cm^2. As can be seen, the H_{irr} is significantly decreased by increasing the doping level.

4. Conclusion

MgB_2 polycrystalline bulk samples with addition of 5 and 10 wt.% nano-sized Al_2O_3 powders were prepared by solid state reaction using pre-reacted MgB_2. Results show that the critical current density and irreversibility fields decrease significantly with increasing Al_2O_3 levels. T_c drops slightly from 37.9 to 36.6 K, but with a very wide transition width of above 20 K. The amount of MgO was found to increase with increasing Al_2O_3 levels, probably indicating that Mg was replaced by Al with excess forming extra MgO and possibly producing weak links.

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