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Influence of Magnesium Powder and Heat Treatment on the Superconducting Properties of MgB_2/Fe Wires

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MgB_2/Fe 선재의 초전도성에 대한 열처리 조건과 Mg 분말의 영향

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Abstract

The most common technique to fabricate MgB_2 superconducting wire is by powder-in-tube (PIT) technique. Therefore, the starting powder for the processing of MgB_2 superconductors is an important factor influencing the superconducting properties and performance of the conductors. In this study, the influence of magnesium precursor powders and annealing temperatures on the transition temperatures (T_c) and critical current densities (J_c) of MgB_2/Fe wires was investigated. All the MgB_2/Fe wires were fabricated by *in situ* PIT process. It was found that higher J_c was obtained for MgB_2 wires with smaller particle size of magnesium precursor powders. The J_c also increases with decreasing annealing temperatures.

Keywords : MgB_2, heat treatment, superconducting wires, critical current density

I. Introduction

With the relatively recent discovery of superconductivity in MgB_2, efforts to produce high-performance, economically-viable wires have been revitalized to exploit this low-cost and weak-link-free material with a higher transition temperature, T_c [1, 2]. In this regard, the powder-in-tube process remains as one of the most promising and commonly-used techniques for the development of low-cost MgB_2 conductors [3, 4]. Variation in the superconducting properties of MgB_2 is often reported worldwide, which can be attributed in part to the different thermo-mechanical processing, and the differences between the precursor powders [5-7]. Sintering temperatures and durations also have a strong effect on the critical current density, J_c [8, 9]. Meanwhile, Serquis *et al* [10]. have shown a strong influence of annealing conditions on microstructural development that correlates to J_c. In Cu-clad MgB_2

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wires, the heat treatment is crucial due to the extensive reaction between the superconducting cores and the sheath materials, leading to the formation of Cu_2Mg [11, 12]. These wires require a fast formation method which causes less reaction between the Mg and the sheath materials, and markedly improves the critical current density [11]. Because of the lack of substantial reaction between Fe and MgB_2, Fe is a better choice to be used as sheath material [13].

A systematic study of the influence of magnesium precursor powder and annealing temperature on the superconducting properties was performed on *in situ* MgB_2/Fe wires. Due to the high volatility of Mg during annealing above the melting point of Mg (650°C), 600°C was chosen as one of the three isothermal annealing temperatures to test; the others, 700°C and 850°C, was chosen to be higher, as the kinetics of phase formation plays an important role in affecting J_c.

II. Experimental

Three types of magnesium powders with particle sizes of 44 μm, 12-17 μm and 4-6 μm, and boron powder with 95-97% purity, <1 μm were used as starting precursor powders. Each type of the magnesium powder was mixed with the boron powder in a nominal composition of Mg:2B as shown in Table 1. The powders were packed into Fe tubes and then drawn to about 1.7 mm in diameter using the standard powder-in-tube (PIT) method.

All samples were encapsulated in vacuum quartz tube before heat treatment. Three different heat treatments were conducted: a) 200°C/h up to 600°C for 30 hours, b) 200°C/h up to 700°C for 30 minutes and c) 200°C/h up to 850°C for 30 minutes.

Table 1. The precursor powders of the wires.

Sample name	Magnesium powder (size)	Boron powder (purity, size)
W5	44 μm	95-97%, < 1 μm
W6	12-17 μm	
W7	4-6 μm	

X-ray diffractometry (XRD) was conducted using Cu-K_α radiation in the Bragg-Brentano configuration to determine the phase composition of the samples. Superconducting transition temperature, T_c and critical current density, J_c of the superconducting cores were measured using a d.c. superconducting quantum interference device (SQUID) magnetometer. T_c was defined as the onset of the diamagnetism and magnetic J_c was determined using the Bean model.

III. Results and discussions

Fig. 1 shows the XRD patterns for sample W7 with different heat treatment conditions. Both W5 and W6 samples also have similar XRD patterns. Some unreacted magnesium was detected for samples heat-treated at temperatures below 850°C. Unreacted magnesium still present in a significant amount for sample annealed at 600°C for 30 hours. This shows the kinetics of the MgB_2 formation at temperatures below the melting point of magnesium is very slow. In all the samples, MgO was present as the main impurity phase.

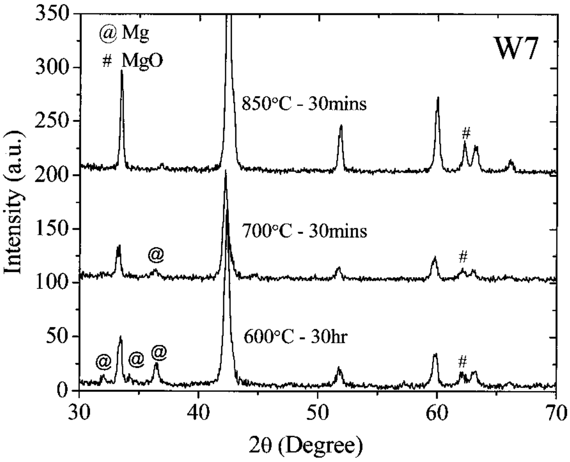

Fig. 1. X-ray diffraction patterns for sample W7 with different heat treatment conditions.

Fig. 2 (a) and (b) show the cross-sectional images of sample W7 heat-treated at 600°C for 30 hours and 850°C for 30 minutes respectively. From the images shown, wire samples heat-treated at lower temperatures contain less porosity and fewer cracks. For the sample annealed at 600°C, which is below the melting point of magnesium, the formation of MgB_2 is by a solid-solid diffusion. For high temperature annealing, which involves a solid-liquid reaction, formation of cracks and large pores size might occur during the heat treatment due to the presence of magnesium liquid as shown in fig. 2(b).

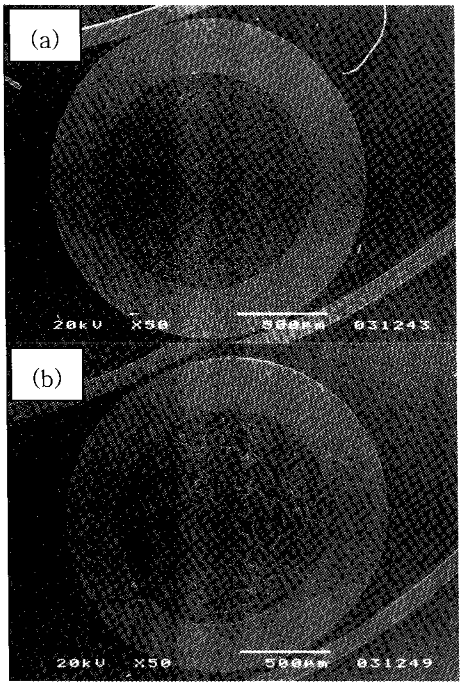

Fig. 2. SEM images of sample W7 heat-treated at (a) 600°C for 30 hours and (b) 850°C for 30 minutes.

Figs. 3 and 4 show the critical temperature, T_c and the magnetic critical current density, J_c, respectively, for sample W7 heat-treated at 600°C for 30 hours, 700°C for 30 minutes and 850°C for 30 minutes. The other two samples, W5 and W6 also have a similar trend in T_c and J_c as W7. The T_c is almost the same for all the samples regardless of annealing temperatures. The sample annealed at 600°C show a slightly narrower ΔT, suggesting that the sample has a higher homogeneity and/or better intergrain connectivity as a result of solid-solid diffusion reaction.

The samples annealed at lower temperatures show a higher J_c values as shown in fig. 4. From the SEM images shown in fig. 2, sample annealed at lower temperature has a more uniform core and less porosity. High temperature annealing causes the vaporization of the unreacted magnesium thus resulting in a decrease in the wire core density. This increases the superconducting area of the wire and causes the J_c to be higher. In addition, sample annealed at high temperatures will also promote grain growth hence reduces the grain boundary pinning which lead to a lower J_c.

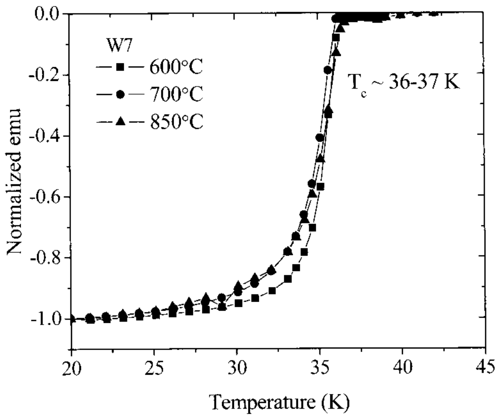

Fig. 3. Magnetic moment measurements as a function of temperature for sample W7 annealed at 600°C for 30 hours, 700°C for 30 mins and 850°C for 30mins.

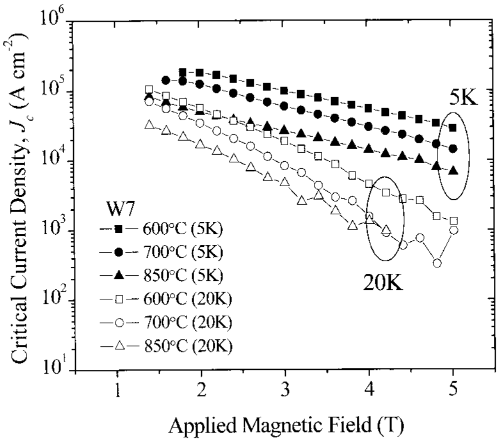

Fig. 4. Critical current density, J_c as a function of magnetic field at 5K and 20K for sample W7 annealed at 600°C for 30 hours, 700°C for 30 minutes and 850°C for 30 minutes.

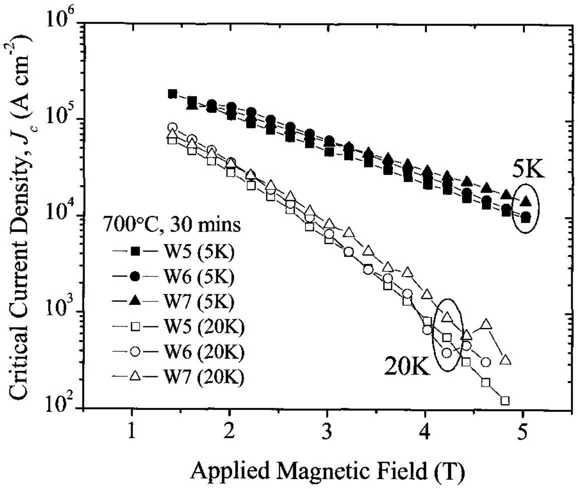

Fig. 5. Comparison of critical current density, J_c as a function of magnetic field at 5K and 20K for samples W5, W6 and W7 annealed at 700°C for 30 minutes.

Fig. 5 compares the critical current density, J_c for samples W5, W6 and W7. The sample with the smallest particle size of magnesium powder, W7 shows the highest J_c value among the three samples. When using small particle size of magnesium powder as a precursor, the magnesium is expected to react more uniformly and quickly, leading to the formation of MgB_2 with small grain size [5]. Therefore, it increases the grain boundary pinning, which in turn improves the J_c of the wire.

IV. Conclusions

In this study, we found that the MgB_2/Fe wires fabricated with smaller particle size of magnesium precursor powder and annealed at lower temperatures have smaller pores size, grain size and less porosity. Therefore, the results show the critical current density, J_c increases with decreasing annealing temperatures and with decreasing magnesium particle size.

Acknowledgments

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