Effect of Sucrose (C12H22O11) Doping on the Critical Current Density of MgB2

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Effect of Sucrose (C\textsubscript{12}H\textsubscript{22}O\textsubscript{11}) Doping on the Critical Current Density of MgB\textsubscript{2}

Y. Zhang, S. H. Zhou, A. V. Pan, and S. X. Dou

Abstract—MgB\textsubscript{2} bulk samples doped with sucrose were fabricated with the formula MgB\textsubscript{2-x}C\textsubscript{x} (x = 0, 0.08, 0.12, 0.2, 0.3, 0.5). Appropriate amounts of sucrose corresponding to the desired carbon content were added. The doping effect of sucrose was observed. It was found that samples doped with x = 0.2 sucrose have optimized current density at high magnetic field. The optimized sintering temperature for high current density was found to be 850°C. \( H_{c2} \) and \( H_{c1p} \) were both found to be improved due to the sucrose doping.

Index Terms—Critical current, doping, magnesium diboride, sucrose.

I. INTRODUCTION

MgB\textsubscript{2} has been regarded as one of the most promising superconductor materials since its discovery in the year 2001 [1]. This superconductor has already been fabricated in the bulk, wire and film states. The properties of MgB\textsubscript{2} have been studied extensively [2]–[8]. On the one hand, its high \( T_c \), low material cost, and good weak-link tolerance [5], [9] are very advantageous for practical applications. On the other hand, several issues exist that urgently need to be solved, such as its low \( H_{c2} \) and rapid decrease in critical current density (\( J_c \)) under magnetic field compared to Nb-based superconductors. High critical current density and good \( J_c(H) \) performance are crucial for the application of this material in the so called "strong electrical application" field. Intensive studies have been focused on the improvement of critical current density (\( J_c \)) and \( H_{c2} \) since the discovery of MgB\textsubscript{2}. So far, a number of experimental techniques, including chemical doping (addition or substitution), proton irradiation [2], [10], and various types of thermomechanical processing, have been attempted to realize these purposes.

Currently, from more practical and scalable considerations, addition or substitution of nanoparticles into MgB\textsubscript{2}, which causes chemical and nanostructural changes, seem to be an effective way to induce flux pinning centers in MgB\textsubscript{2}, thereby improving \( J_c \) and \( H_{c2} \) [11]. Among the types of chemical doping, nanosize SiC doping has achieved a considerable \( J_c \) improvement. The \( J_c \) of SiC doped MgB\textsubscript{2} has achieved \( 4 \times 10^4 \) A/cm\textsuperscript{2} at 20 K and 4 T [12]. Some other nanosize dopants have also shown \( J_c \) improvement to different levels [13]–[15]. However, doping with nanomaterials gives rise to two concerns: one is agglomeration of the particles when they are mixed with MgB\textsubscript{2} raw materials, which make it hard to achieve an uniform structure [16], and the other factor is the high cost of nanomaterial, incurring economic disadvantages.

Carbon has been proved to be an effective dopant element for improving \( H_{c2} \) and \( J_c \) of MgB\textsubscript{2} superconductor. Tiny carbon particles and carbon nanotubes have shown a strong enhancing effect on the superconducting properties, although agglomeration is still a problem [17]–[20]. Carbohydrate is a very good carbon source for C doping into MgB\textsubscript{2}. Carbohydrate materials provide carbon as an element when they are heated above the decomposition temperature. This freshly obtained carbon is distributed more uniformly than with carbon nanotubes. Commercial sugar has been used recently as a dopant in MgB\textsubscript{2} because it is cheap and easy to find. We have shown that sugar doping has benefits for \( J_c(H) \) improvement [21]. In this paper, the fabrication process was studied more systematically and the parameters were optimized.

II. EXPERIMENTAL DETAIL

MgB\textsubscript{2} bulks were prepared by the in-situ solid state sintering method. Powders of Mg (99%), amorphous B (99%), and 98% sucrose were used as starting materials to achieve the formula of MgB\textsubscript{2-x}C\textsubscript{x} (x = 0, 0.08, 0.12, 0.2, 0.3, 0.5). Appropriate amounts of sucrose based on the corresponding carbon content were added in to the MgB\textsubscript{2}. In order to investigate the effects of sucrose doping on MgB\textsubscript{2} bulks, B powder with 100 nm particle size was mixed with sucrose with the help of de-ionized water. The slurry was dried in a vacuum oven. After drying, the B powder was coated with a sucrose layer. This obtained B and sucrose mixture was mixed with Mg powder. The powder was ground by hand with a mortar and pestle. Appropriate mixtures of these powders were pressed into pellets and sealed in Fe tubes. This packing process was carried out in air. The samples were heated from room temperature to 780°C–1050°C in a tube furnace under Ar atmosphere at ambient pressure with a 5°C/min heating rate, and kept at the set temperature for 1 hour before being furnace cooled to room temperature.

The MgB\textsubscript{2} pellets were then taken out by cutting the iron tube open. The phase analysis was carried out by X-ray diffraction (XRD) in a Phillips PW1730 Model diffractometer using Cu K\textsubscript{α} radiation. The compositional analyses were performed in an energy dispersive X-ray spectroscopy (EDX) system. The magnetization as a function of temperature T and magnetic field \( H \) applied along the longest sample dimension was measured...
using Quantum Design Magnetic Property and Physical Property Measurement Systems within the field range \( H < 9 \text{ T} \), and within the temperature range of \( 5 \text{ K} < T < 30 \text{ K} \). The magnetic \( J_c \) was derived from the half-width of the magnetization difference between the descending branches (\( M^+ \)) and ascending branches (\( M^- \)) of the magnetization loop, using the following critical state model formula: 

\[
J_c = k\Delta M/d \]

where \( k = 12w/(3w-d) \) is a geometrical factor and \( \Delta M = (|M^+| - |M^-|)/2/V_{\text{eff}}xwxd \), with \( l, d \) and \( w \) being the sample length, thickness and width, respectively. The typical dimensions of the samples used for magnetization measurements are \( 3 \times 2 \times 1 \text{ mm}^3 \). 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.

III. RESULTS AND DISCUSSION

A. Effect of Doping Level

A series of samples doped with different levels of sucrose were synthesized to study the effect of the doping level on the superconducting properties, with the nominal composition \( \text{MgB}_{2-x} \text{C}_x \) (where \( x \) varies from 0 to 0.5). These samples were sintered at 900°C with a 5°C/min heating rate. Fig. 1 shows the transition temperature (\( T_c \)) for the doped and undoped samples, as determined by ac susceptibility measurements. The \( T_c \) noticeably decreased with an increasing sucrose doping level. The \( T_c \) onset for the undoped samples is around 37.7 K. The \( x = 0\text{.5} \) doped sample has a \( T_c \) of 32.0 K. When \( x = 0\text{.2} \), \( T_c \) is 36.8 K. The \( T_c \) decrease might be a result of increased impurity phases introduced by sucrose doping or as a result of C substitution for B in \( \text{MgB}_2 \).

Fig. 2 shows the magnetic field dependence of the \( J_c \) for these samples. It can be seen the doping has strong effects on the \( J_c \) both at 5 K and 20 K. Compared with the undoped sample, samples doped with \( x = 0\text{.08}, 0\text{.12}, \) and \( 0\text{.2} \) experience positive effects. At 5 K and 6 T, the \( x = 0\text{.2} \) sample shows one order of magnitude improvement. Above the \( x = 0\text{.2} \) level, samples such as \( x = 0\text{.3} \) and \( x = 0\text{.5} \) show less improvement.

This can be more clearly shown in Fig. 3, which contains the \( J_c \) performance at 20 K and 4 T. The \( J_c \) reached a peak at \( x = 0\text{.12} \). The \( J_c \) decrease at higher doping levels is a result of \( T_c \) decrease, and also might be related to excessive impurity phase produced by the sucrose. These impurity phases could block the current path, thus resulting in shrinkage of the effective current conducting area. Phase analysis was carried out using X-ray diffraction, and Fig. 4 shows that the impurity phases increased with the doping level. Judging from the \( T_c \) and \( J_c \) performance, we conclude that the doping level of \( x = 0\text{.2} \) is the optimized doping level.

B. Effect of Sintering Temperature

In order to optimize the sintering temperature, samples doped at \( x = 0\text{.2} \) was sintered at temperatures ranging from 780°C to 1050°C. \( T_c \) of these samples is shown in Fig. 5. A pure sample sintered at 900°C was also included for reference. It was found that the \( T_c \) increased with the sintering temperature. This \( T_c \) increase is the sign of improved crystallinity.

Fig. 6 shows the \( J_c(H) \) performance for these samples. The sample sintered at 850°C has the best \( J_c(H) \) performance. At 20 K, the \( x = 0\text{.2} \) sample has higher \( J_c \) both at low field and at high field. At 5 K, the sample sintered at 780°C has higher \( J_c \) than the 850°C one. Higher sintering temperatures...
than 850°C resulted in poorer $J_c$(H) performance. In our experiments, judging from XRD results and $J_c$(H) performance, $J_c$ is related to the amount of impurity phase and grain boundary pinning. At lower sintering temperatures than 780°C, the doped carbon did not react fully with the MgB$_2$, so the doping effect of the carbon was not fully utilized, and $J_c$ at higher field is not high. When the sintering temperature is higher than 850°C, the crystal grain size might grow larger due to the thermal effects which reduce the grain boundary area, thus reducing the grain boundary pinning, and decreasing $J_c$ at high field due to the lack of pinning centers, as indicated in reference [22]. The 850°C sintering temperature is a compromise temperature chosen to produce the best overall $J_c$(H) performance. Fig. 7 is the temperature dependence of $H_{ irr}$ and $H_{c2}$ for the doped

Fig. 6. $J_c$(H) curves for pure MgB$_2$ and sucrose-doped MgB$_{1.8}$C$_{0.2}$ sintered at various temperatures.

Fig. 7. Normalized temperature (T/$T_c$) dependence of the upper critical field, $H_{c2}$, and the irreversibility field, $H_{ irr}$, for pure MgB$_2$ and sucrose-doped MgB$_{1.8}$C$_{0.2}$. The latter was sintered at 850°C.

and undoped samples sintered at 850°C. The temperature was normalized to the samples’ $T_c$. It is obvious that $H_{ irr}$ and $H_{c2}$ both increased after doping with sucrose.

IV. CONCLUSION

Good $J_c$(H) performance was found in sucrose doped MgB$_2$ superconductor. $T_c$ was impeded by the doping, but can still remain reasonably high (above 34 K at doping levels less than $x = 0.2$). $H_{c2}$ and $H_{ irr}$ both shifted towards higher field. This work has opened up way of improving MgB$_2$ superconducting properties by doping with a carbohydrate [23] such as sucrose. Further work is likely to produce even more promising results.

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