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Study of superconducting and electromagnetic properties of un-doped and organic compound doped MgB2 conductors

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By

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Institute for Superconducting & Electronic Materials

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DECLARATION

This is to certify that the work presented in this thesis was carried out by the candidate in the laboratories of the Institute for Superconducting and Electronic Materials (ISEM), at the University of Wollongong, NSW, Australia, and has not been submitted for a degree to any other institution for higher education.

Md Shahriar Al Hossain

2008
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ABSTRACT

In this thesis I emphasized on the organic compound doping (specially carbohydrate group, malic acid, C₄H₆O₅) and heat treatment effects on the superconducting properties of MgB₂. I also focused on the basic and fundamental properties of un-doped MgB₂ wires in different temperatures for comparison purpose. And finally I have proposed another new dopant which avoids some problems using carbohydrate in some aspects.

Firstly, I have studied the effects of sintering temperature on the phase transformation, lattice parameters, full width at half-maximum (FWHM), strain, critical temperature (Tₑ), critical current density (Jₑ), and resistivity (ρ) in MgB₂/Fe wires. All samples were fabricated by the in situ powder-in-tube method (PIT) and sintered within a temperature range of 650–900 °C. I have showed that why I have taken such sintering temperature range by analyzing with differential thermal analysis (DTA). The increased FWHM and decreased Tₑ at low sintering temperature region suggested the smaller grain size and poor crystallinity. Strain values also higher at low sintering region. That’s why it was observed that wires sintered at low temperature, 650 °C, resulted in higher Jₑ up to 12 T. The best transport Jₑ value reached 4200 A cm⁻² at 4.2 K and 10 T. This is related to the grain boundary pinning due to small grain size and poor crystallinity due to strain defects. On the other hand, wires sintered at 900 °C had a lower Jₑ in combination with better crystallinity due to higher Tₑ.

The effect of carbohydrate doping on lattice parameters, microstructure, Tₑ, Jₑ, Hₑrr, and Hₑc₂ of MgB₂ has been studied. In this work I used malic acid, C₄H₆O₅ as an example of
carbohydrates as an additive to MgB$_2$. We have described the advantages of carbohydrate doping include homogeneous mixing of precursor powders, avoidance of expansive nanoadditives, production of highly reactive C, and significant enhancement in $J_c$, $H_{irr}$, and $H_{c2}$ of MgB$_2$, compared to un-doped samples. The defects due to the C substitution into boron site lead to the enhancement of $H_{irr}$ and $H_{c2}$. The decrease of $a$-axis lattice parameter and reduction of $T_c$ indicates poor crystallinity due to C substitution. The microstructure was shown both for un-doped and doped samples which were well consistent with FWHM. The $J_c$ for MgB$_2$+30 wt% C$_4$H$_6$O$_5$ sample was increased by a factor of 21 at 5 K and 8 T without degradation of self-field $J_c$ due to C substitution into B sites.

During the evaporation process of the C$_4$H$_6$O$_5$ with B and solvent, freshly and highly reactive C is produced and C substitution for B can take place at the temperature same as the formation temperature of MgB$_2$. By using this chemical route I again evaluated the doping effects of C$_4$H$_6$O$_5$, from 0 to 30 wt% of the total MgB$_2$, on the lattice parameters, lattice strain, amount of carbon (C) substitution, microstructures, weight fraction of MgO, critical temperature ($T_c$), critical current density ($J_c$), and irreversibility field ($H_{irr}$) of a MgB$_2$ superconductor. The calculated lattice parameters show a large decrease in the $a$-axis lattice parameter for MgB$_2$ + C$_4$H$_6$O$_5$ samples from 3.0861(6) to 3.0736(1) Å, with even a 10 wt% addition. This is an indication of C substitution into boron sites, with the C coming from C$_4$H$_6$O$_5$, resulting in enhancement of $J_c$ and $H_{irr}$. Specifically, the $H_{irr}$ of the MgB$_2$ + C$_4$H$_6$O$_5$ samples prepared by the chemical solution route reached around 7 T at 20 K, with a $T_c$ reduction of only 1.5 K. In addition, the self-field $J_c$ of the MgB$_2$ +
C₄H₆O₅ samples was only slightly reduced at an additive level as high as 30 wt%. The interesting thing I found here is maximum C-substitution and the maximum enhancement of all the superconducting parameters up to 10 wt% addition, after that the improvement rate is saturated. From these data I can claim 10 wt% addition is enough for maximum C-substitution and enhancement of superconducting properties. However, residual oxygen after evaporation processing contributed to a large amount of MgO in our MgB₂ + 30 wt% C₄H₆O₅ samples. These problems can be further controlled by the amount of C₄H₆O₅ additive or different evaporation temperatures.

After the successful doping effects of C₄H₆O₅ into MgB₂, then I investigated the behavior of C₄H₆O₅ as a dopant with different sintering temperatures. All the samples were prepared by the chemical solution route. I report the carbon (C) substitution effects of MgB₂ + 10 wt% C₄H₆O₅ on the lattice parameters, critical temperature (Tc), upper critical field (Hc₂), and irreversibility field (Hirr) as a function of sintering temperature in the range from 600 to 900 °C. The additive C₄H₆O₅ as the C source resulted in a small depression in Tc, but significantly increased the C substitution level, and hence improved the Hc₂ and Hirr performance at a low sintering temperature of 600 °C in conjunction with a short sintering period of 4 h. In addition, the low-temperature sintering process resulted in small grain size and higher impurity scattering compared to a pure MgB₂ superconductor which promotes the flux pinning significantly.

Very recently, I have chosen another solid hydrocarbon dopant named pyrene (C₁₆H₁₀) in to MgB₂. There are few reasons behind this decision. Firstly we know all the carbohydrates consist of carbon (C), hydrogen (H), and oxygen (O). During the
evaporation process of C₄H₆O₅, I noticed that the MgO amount is gradually increased with increasing doping level. So our group suggests such special solid hydrocarbon without O content which may reduce the MgO content within the matrix. In this work, we report on significantly enhanced $J_c$ in MgB$_2$ superconductor that was easily obtained by doping with a hydrocarbon, highly active C$_{16}$H$_{10}$, and using a sintering temperature as low as 600 °C. The processing advantages of the C$_{16}$H$_{10}$ additive include production of a highly active carbon C source, an increased level of disorder, and the introduction of small grain size, resulting in enhancement of $J_c$. 