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## Processing and characterisation of nano carbon doped MgB2 form of wire and bulk

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# Processing and Characterisation of Nano Carbon

## Doped MgB<sub>2</sub> in Form of Wire and Bulk

A thesis submitted in fulfillment of the requirements for the award of

the degree of

## **DOCTOR OF PHILOSOPHY**

From

## UNIVERSITY OF WOLLONGONG

By

Wai Kong Yeoh

## **Institute for Superconducting and Electronic Materials**

2006

## 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.

Wai Kong Yeoh

2006

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Π

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## ABSTRACT

The objective of this work is to further enhance the critical current density of the  $MgB_2$  superconductor by doping with the two carbon sources: multiwalled carbon nanotube (CNT) and nano carbon. The work in this thesis concentrates on the fabrication and characterization on the CNT and nano C doped  $MgB_2$  with main objective being the enhancement of the critical current density in the high magnetic field. Consequently, introducing effective pinning centres in the form of dopants to enhance the flux pinning will be the main task of this project.

In this project, the effect of carbon doping MgB<sub>2</sub> with carbon nanotubes and nano C on transition temperature, lattice parameters, critical current density and flux pinning for MgB<sub>2-x</sub>C<sub>x</sub> with x = 0, 0.05, 0.1, 0.2 and 0.3 under the various condition was studied. Both types of doping showed excellent  $J_c$  compared to the pure MgB<sub>2</sub>, with significant enhancement observed at higher temperature. Magnetic  $J_c(H)$  was enhanced by a factor of 72 at 5K for a field 8T and a factor of 33 at 20K for a field of 5T for nano C bulk samples, respectively. On the other hand,  $J_c(H)$  of CNT samples was enhanced by a factor of 26 and 13 under the equivalent conditions. In high field, transport  $J_c$  of magnitude 2122 A/cm<sup>2</sup> and 3821 A/cm<sup>2</sup> was observed at 4.2K and 12T for CNT and nano C doped MgB<sub>2</sub>. These results indicate that flux pinning was enhanced by the boron substitution for carbon with increasing processing temperature. However, it was found that the lattice distortion and optimum doping level is different in the CNT and nano C samples which is due to the reactivity of the carbon source, resulting in different carbon substitution rate. Due to better reactivity and homogenous mixing of nano C, nano C doped MgB<sub>2</sub> resulted in better improvement in magnetic and transport  $J_c(H)$ , as compared to CNT doped  $MgB_2$ . This is mainly because CNT fibres with high aspect ratio tend to entangle, which suppressed the reactivity.

The depression of  $T_c$ , which is caused by the boron substitution for carbon, increases with increasing the doping level, processing temperature and duration for both types of carbon doping. By controlling the extent of the substitution and inclusion of carbon, we can achieve the optimal improvement of critical current density and flux pinning in magnetic fields while maintaining the minimum reduction in  $T_c$ . In addition, the values of  $H_{c2}$  and  $H_{irr}$  are higher for CNT doped samples than for the pure MgB<sub>2</sub> at the same value of T/ $T_c$ . The morphology of the CNT doped MgB<sub>2</sub> is similar to that of nano C doped MgB<sub>2</sub>, but different from the pure MgB<sub>2</sub>. The microstructure exhibits noticeable nanoparticles with size around 10-20nm, which are believed to be MgO and MgB<sub>2</sub>.

Magnetization measurements indicate a change in the critical current density with the length of nanotube and not with its outside diameter. This is due to longer nanotubes tending to entangle with each other, preventing their homogenous mixing with MgB<sub>2</sub> and dispersion. Low intensity ultrasonication, as a method of dispersion of CNT's into precursor magnesium and boron powder, was introduced to improve homogeneity of mixing of CNT's with the MgB<sub>2</sub> matrix. Ultrasonication of CNT doped MgB<sub>2</sub> resulted in a significant enhancement in the field dependence of critical current density, while avoiding the side-effects that would occur at higher processing temperatures.

Carbon nanotubes (CNT's) have unusual electrical, mechanical and thermal properties. The elongated CNT's induce anisotropy in  $J_c$  in relation to the direction of applied field in MgB<sub>2</sub>/Fe wires and the value of  $J_c$  for the carbon nanotube-doped wires is insensitive to heating rates. We believe that by taking the extraordinary electrical, mechanical and thermal properties of CNT's, the mechanical properties and thermal stability of CNT doped wire will be substantially improved. Studies on these properties are underway.