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Effect of the starting boron powder on the superconducting properties of MgB

Xun Xu
University of Wollongong

Effect of the Starting Boron Powder on the Superconducting Properties of MgB_2

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XUN XU, B. Eng., M. Eng.

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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.

Xun Xu

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Abstract

The effect of the properties of the starting boron powders on the superconducting properties of MgB_2 has been studied. Low grade boron powders are attractive because of their low cost, but produced lower surface reactivity and larger particle size than high purity (99%) amorphous boron powder, indicating that the low grade powders cannot be used to achieve the same superconducting properties as those of samples made from pure 99% boron powder. However, the low purity boron powders can be improved by using simple physical and chemical processes, leading to enhanced magnetic critical current density, J_c. In order to get high performance MgB_2, it is obviously important to control the phase composition and microstructure of the boron starting powders and the solid state reaction conditions.

Ball milling is an effective method to reduce the boron particle size, so, the effects of ball milling boron powders in different media, such as acetone, ethanol, and toluene, on the superconducting properties of MgB_2 needed to be considered and studied. It was observed that toluene was the most effective medium of them all for enhancing J_c. J_c was estimated to be 5×10^3 A cm^{-2} at 8 T and 5 K for a sample that was ball milled in toluene. This value is much higher than that of the pure MgB_2 reference sample that was not ball milled, by a factor of 20. It was considered that ball milling B using toluene leads to smaller MgB_2 grains, resulting in enhanced J_c at low operating temperatures and high fields.

MgB_2 samples were prepared using as-supplied commercial 96% boron with strong crystalline phase and the same 96% boron (B) after ball milling. The effects of the properties of the starting B powder on the superconductivity were evaluated. It was

observed that samples using ball-milled 96% B, in comparison with the reference sample made from the as-supplied 96% B, were characterized by small grain size and enhanced magnetic critical current density (J_c), which reached 2 $\times 10^3$ A cm^{-2} at 5 K and 8 T. The improved pinning seen in these samples seems to be caused by enhanced grain boundary pinning at high field. MgB_2 samples were also prepared by using 96% boron powder with strong crystalline phase that had been ball milled for various times. Based on Rowell connectivity analysis, when the ball-milling time increased, the connectivity factor, described as the active cross-sectional area fraction (A_F), was decreased. This implies that the inter-grain connectivity became worse. These properties could lead to poor J_c in low field. However, the pinning force strength of samples using ball-milled 96% B is larger than that of the reference sample using as-supplied commercial 96% B powder. These results accompany enhanced irreversibility (H_{irr}) and upper critical fields (H_{c2}).

Furthermore, the magnetic field dependence of the transport critical current density (J_{ct}) and the grain connectivity of MgB_2/Fe wires fabricated from ball-milled boron have been investigated in detail, and strong correlations have been found, as evidenced by differences in grain size, critical transition temperature, and resistivity. It was observed that the samples fabricated by ball milling had relatively small grain sizes, resulting in a weaker field dependence of the J_{ct} in the high field region. On the other hand, the ball-milled boron was associated with poor connectivity between adjacent grains. It is clearly shown that the observed reduction in low field J_{ct} is related to the reduction in the superconducting area fraction that is reflected by the connectivity factor. Even though high temperature sintering could always compensate for the degradation of the J_{ct} in the low field region, the subsequent grain growth in this case was mainly responsible for the degradation of J_{ct} in the high field region. The strong correlation

between the grain size and the connectivity can change the field dependence of the J_{ct}, and both these factors are primarily affected by the sintering temperature and by the presence and extent of ball milling.

In the MgB_2 field, chemical doping is the most popular way to improve the superconductor properties. It has been reported that significantly enhanced critical current density in MgB_2 superconductor could be easily obtained by doping with a hydrocarbon, highly active pyrene ($C_{16}H_{10}$), while using a sintering temperature as low as 600^{o}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.

Using the same concept, low purity boron powders were used to fabricate pure and submicron-sized carbon sphere doped MgB_2 superconductor. The boron powders used showed low reactivity towards MgB_2 formation, as compared to high purity (99%) amorphous boron, which might result from the larger grain size and the existence of crystalline boron or boron oxide in the former. However, the samples prepared from this boron powder showed comparable J_c values at 20 K and in low field (<1 T) to those from a sample prepared from 99% amorphous boron. Doping submicron-sized carbon spheres successfully introduced carbon substitution for boron, and so improved the H_{c2}, H_{irr}, and in-field J_c properties of the MgB_2.