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## 論 文 要 旨

Thesis Abstract

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主論文題名	(Title)						
Innovative 7 Bulk MgB <sub>2</sub>	lechniques for Enha	ncement	of Super	rconducting	; Charact	teristic	s of

内容の要旨 (Abstract)

The objective of this thesis was to study the ways, how to improve critical current density ( $J_c$ ) in polycrystalline magnesium diboride (MgB<sub>2</sub>) bulk. The superconducting devices demand economical operation, a cheap manufacturing route, and a light weight. In view of these requirements, MgB<sub>2</sub> meets most of the criteria making it a promising candidate. Properties such as decent critical transition temperature ( $T_c$ ), cheap and abundant raw materials, and light weight elements are appealing qualities for practical applications. It is also well known that MgB<sub>2</sub> thin films have high  $J_c$  (~ 10<sup>7</sup> to 10<sup>8</sup> A/cm<sup>2</sup>) and upper critical field ( $H_{c2}$ ~ 60T at 0K). However, these values have been never seen in polycrystalline MgB<sub>2</sub> bulks; in fact, the  $J_c$  values are almost two orders of magnitude lower than those observed in thin films. The main reason is poor flux pinning, resulting in low  $H_{irr}$ ,  $H_{c2}$  &  $J_c$ . Poor connectivity between grains and low density are the issue. Sintering has been a standard technique (700 to 850 °C), resulting in good crystallinity and large grain size. Lack of flux pinning at grain boundaries and crystal defects led to considerable reduction of self-field  $J_c$ .

To tackle these issues, we tried to manipulate microstructure with various precursors. In this thesis, we fabricated MgB<sub>2</sub> bulk using solid state sintering at 775 °C for 3 hours. Since, the melting point of boron is very high (~2000 °C) compared to reaction temperature, boron precursor particle size plays a vital role in optimizing  $J_c$ . We used a commercial nano-amorphous boron and the results were astonishing.  $J_c$  of 408 kA/cm<sup>2</sup> was observed at 20 K, self-field. SEM micrographs revealed the nanosized grains in the final microstructure, which approved our hypothesis and was successful in improving self-field  $J_c$ . In order to improve the high field  $J_c$  and upper critical field  $H_{c2}$ , we resorted to carbon doping. One serious issue with the carbon doping was lack of homogeneous distribution of carbon in the matrix. To overcome this issue, we prepared carbon-encapsulated boron (CEB) made from pyrolysis of Diborane, hydrogen and gaseous hydrocarbon. It was found that low wt% of carbon coating leads to the best results.

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内容の要旨(Abstract)

A high-field  $J_c$  and  $H_{c2}$  were observed in 1.5 wt% carbon encapsulated boron based MgB<sub>2</sub> bulk. Tremendous  $J_c$  of 660 and 250 kA/cm<sup>2</sup> were observed at 0 and 2 T; 10 K.  $H_{c2}$  (obtained by extrapolation) was also substantially improved, being almost equivalent to the best records reported in MgB<sub>2</sub> bulk system so far, however at a slight expense of  $T_c$ . To further improve this result, our group studied the effect of co-dopants such Ag and CEB. Microstructural analysis exposed Ag-Mg phases formed in the matrix and optimum performance was observed for 4 wt% Ag. To compensate the loss of Mg reacting with Ag, as well as to increase the Ag-Mg phase fraction, we studied the optimization of Mg precursor concentration. 7.5 wt% excess of Mg resulted in the best result, with highest Ag-Mg phase (2 wt%), high irreversibility field ( $H_{irr}$ ) of 4.76 Tesla and large  $J_c$  such as 440 kA/cm<sup>2</sup> at 20 K, self-field. SEM analysis confirmed existence of secondary nanoscopic Ag-Mg phases (20-40 nm), acting as pinning centers.

The special clean boron precursors first used were expensive, which might make the final product costly. In order to make the processing cheap while maintaining high performance, we explored a novel technique of *high-energy ultra-sonication* using various media such as ethanol, hexane, and distilled water. We successfully produced nano-sized boron via ultra-sonication, and arrived at high  $J_c$  in the final bulk. Beside size refinement, the obtained fine boron powder was free of B<sub>2</sub>O<sub>3</sub>, due to which the MgB<sub>2</sub> bulks were of high quality. SEM analysis clearly revealed that short duration of ultra-sonication results in particle refinement, while prolonged ultra-sonication causes agglomeration of boron particles. MgB<sub>2</sub> bulks fabricated from various systems were studied and the best results were observed in MgB<sub>2</sub> prepared with for 15 min ultra-sonicated boron dispersed in ethanol and for 30 min ultra-sonicated boron dispersed in distilled water. Self-field  $J_c$  of approximately 300 kA/cm<sup>2</sup> at 20 K was achieved (almost 35% improvement compared to a regular bulk MgB<sub>2</sub>).

Author has employed unique precursors such as nano-amorphous boron, carbon encapsulated boron, excess Mg with Ag addition and boron precursor refined via cheap novel ultra-sonication. The ultra-sonication technique is scalable and cost-effective. Its use resulted in 35%  $J_c$  improvement. The results were published in renowned *international Journals*. This improvement in bulk MgB<sub>2</sub> superconducting system makes it commercially viable for industrial sector, for a mass production of rare-earth-free bulk superconducting materials for super-magnet applications.