§13. Microstructural of MgB₂ Wires Fabricated by Low-temperature In-situ Processes with Mg₂Cu Addition

Hata, S., Kubota, Y., Shimada, Y., Ikeda, K., Nakashima, H. (Kyushu Univ.), Kikuchi, A. (NIMS), Hishinuma, Y., Yamada, S.

An in-situ powder-in-tube (PIT) process using Mg, B and Mg₂Cu as starting powders is a promising process to fabricate MgB₂ wires at low temperatures below 773 K^{1} . We investigated microstructure in the MgB₂ wires using transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM).

Foil specimens for TEM/STEM observation were prepared by focused ion beam (FIB) microsampling and milling techniques. TEM/STEM observation and energy dispersive x-ray spectroscopy (EDX) analysis were carried out at accelerating voltage of 200 kV.

MgB₂ crystals below 300 nm in sizes have plate-like shapes with (001) facets (Fig. 1), suggesting a reaction of solid B and liquid Mg-Cu. The average size of MgB₂ crystals increases with the amount of Mg₂Cu while the amount of residual B decreases. Two kinds of Mg-Cu compounds, Mg₂Cu and Cu₂Mg, are formed during the heat treatment process. Spaces between the plate-like MgB₂ crystals are filled with the Mg-Cu compounds showing orientation relationships with (001) planes of the plate-like MgB₂ crystals (Fig. 2). These Mg-Cu compounds formed between MgB₂ grain boundaries may be effective for enhancing magnetic-flux pinning but not effective for MgB₂ grain growth suppression because these compounds are formed after the crystallization of MgB₂ crystals. From critical current measurements, the optimum amount of Mg₂Cu addition is 3 mol%. This value may be determined from the microstructural advantages and disadvantages of the Mg₂Cu addition in the in-situ PIT process.

This work was partly supported by the Grants-in-Aid for Scientific Research from MEXT and JSPS, Japan. This work was also partly supported by NIFS

1) Y. Hishinuma *et al.*: Supercond. Sci. Technol. **20** (2007), 1178-1183.



Fig. 1. STEM-EDX observation in a MgB_2 wire prepared by heat treatment at 457°C for 200 h. (a) STEM high-angle annular dark-field image, (b) Cu map and (c) Mg map.

(a) HRTEM



Fig. 2. Mg_2Cu phase formed on the (001) plane of MgB_2 phase. (a) HRTEM image, (b) Fourier power spectrum of the square region depicted in (a), and (c) an inverse Fourier filtering image using a doughnut-shaped aperture function depicted in (b).