§14. Optimum B/Mg Ratio of Precursor Powder for MgB₂ Wire Fabrication by an *in-situ* Powder-in-tube Process with Mg₂Cu Addition

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From the viewpoint of advanced materials research for future nuclear fusion power plants, an *in-situ* powder-intube (PIT) process using Mg, B and Mg₂Cu as precursor powders is a promising process to fabricate MgB₂ wires at low temperatures below 773 K¹⁾. To achieve highperformance MgB₂ wires applicable for these plants needs optimization of fabrication conditions in the *in-situ* PIT process. Here, we report an optimum B/Mg ratio of the (Mg + B + Mg₂Cu) precursor powder studied by critical current measurements and microstructural observations.

Mg, Mg₂Cu and B powders were mixed in the following molar fraction; Mg : Mg₂Cu : B = 0.94 : 0.03 : x (x = 1.57 - 2.17). The present study regards x = 1.97 (~ 2) as the stoichiometric composition of MgB₂. Hereinafter, B/Mg molar ratio is used to describe the Mg and B compositions of the precursor powder. The precursor powder was packed into metal Ta tubes. The packed Ta tubes were mechanically drawn into wires and heat-treated at 500°C for 200 h to fabricate MgB₂ wires. The critical current density, J_c , was measured by a four-probe-current method. Cross-sectional scanning electron microscopy (SEM) observations were carried out to evaluate area fractions of MgB₂ and other components at a μ m resolution. Transmission electron microscopy (TEM) observations were performed to evaluate nanoscale microstructure in the wires.

Figure 1 shows SEM secondary-electron images of MgB₂ core regions in the circular cross sections of the wires, as denoted in the inserted optical micrograph. At the present magnification with a μ m resolution, the cross-sections consist of the following microstructural components discriminated from image intensities: MgB₂ regions with a medium image intensity and largest area fractions; pores with the lowest image intensity; Mg-Cu-O base compounds with the highest image intensity; residual B regions with a darker image intensity than the MgB₂ regions. One can notice that the area fraction of pores for B/Mg = 1.97 (Fig. 1(a)) is higher than that for B/Mg = 1.87 (Fig. 1(b)).

Bar graphs in Fig. 2 summarize area fractions of the microstructural components described above as a function of B/Mg, accompanied by a line graph showing J_c measured at 4.2 K under self-fields. The J_c graph shows a sharp maximum for B/Mg = 1.87. This indicates that the B/Mg value is an important parameter for the *in-situ* PIT process. The dependence of J_c on B/Mg shows a good correlation with the dependence of MgB₂ area fraction on B/Mg. For example, Both J_c and MgB₂ area fraction exhibit larger degradation for B/Mg > 1.87 than for B/Mg < 1.87. This result is interpreted that B-rich composition of the precursor

powder increases the amount of residual B particles that reduce superconducting areas in the wires, resulting into the serious J_c degradation. TEM observations revealed that there was no remarkable difference in crystal sizes of MgB₂ and other phases such as MgO, Mg₂Cu, etc.

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Fig. 1. Cross-sectional SEM images. (a) B/Mg = 1.97 and (b) B/Mg = 1.87. An optical micrograph of a typical cross section is inserted in (a).



Fig. 2. Area fractions of constituent microstructural parts (bar graphs) and J_c (4.2 K, self-fields) divided by J_c value for B/Mg = 1.97 (line graph).