Materials for Energy Technology

MS.4.P113
Distribution of MgO nanocrystals in superconductor MgB_2 wires observed with TEM tomography

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Keywords: MgB_2, superconductor, tomography

MgB_2 has the highest critical temperature (T_c) of 39 K among metallic superconductors [1]. It has other benefit features also such as low weight, abundance and low cost of the start materials, easy to be fabricated into wires, sheets, and etc. For these reasons, the MgB_2 superconductor is considered to be a promising material for large-scale applications in electric power and magnetic field generation at around 20 K with low cost. However, one of the problems of the material is that the critical current density (J_c) is not high enough for practical use. It has been established that doping with carbon or nano-sized inclusions such as oxides can improve J_C and other superconductive properties of MgB_2. MgO is a kind of impurity in MgB_2 which is introduced in the fabrication process of the material and is difficult to be removed. The inclusions of MgO may become positive if they are in nano-size, with appropriate density and distribution because they can act as magnetic pinning centres. Therefore, to understand the size, density and distribution of inclusions of MgO and other impurities in MgB_2 is very important for the improvement of superconductive properties of the material. In the present work, we characterized the MgB_2 wires fabricated with internal magnesium diffusion (IMD) process [2] by means of 3-dimensional (3D) and conventional TEM in order to give a visualization of distribution of MgO nano-inclusions and correlate the size and distribution to superconductive properties of the wires.

Two kinds of MgB_2 wires, without and with addition of SiC, were fabricated by IMD process. The start materials were boron powder (purity 99.9%), magnesium (Mg) rod, and SiC powder. The Mg rod was coaxially inserted into an iron (Fe) tube. The other constitutes were packed into the gap between the Mg rod and Fe tube [2]. The composites were heat treated at 670 °C for 3 hours. The MgB_2 wires with addition of SiC exhibited much higher J_Cs than those without addition of SiC in the magnetic field from 8 to 12 T. The TEM thin film specimens were prepared from the fabricated MgB_2 wires by a focused ion beam instrument, JEM-9310FIB. The tilting series images were obtained in STEM HAADF mode. The observation for obtaining tilting series images were carried out with tilting angles from -69 to 69 degree in a step of 3 degree. The reconstruction of the tilting series images was performed using a software made by Gatan, Inc. JEM-2100F and JEM-3000F were used for the observation and analysis.

Figure 1 shows typical STEM images of the MgB_2 wires. 2 kinds of domains, here called as domain A and domain B, were observed in the MgB_2 wires. Domain A is composed of small crystal grains while domain B is in amorphous status. The crystal grains in the MgB_2 wires without addition of SiC were confirmed to be those of MgB_2 and MgO. The crystal grains in the MgB_2 wires with addition of SiC were identified to be those of MgB_2, MgO, and Mg_2Si. The elemental distribution was analyzed with EDS (energy dispersive X-ray spectroscopy) and EFI (energy filtering imaging). It was confirmed that domain A had elements of boron, Mg, O in the MgB_2 without addition of SiC, and of boron, Mg, O, C, Si in the MgB_2 with addition of SiC. Domain B is composed of boron in the two kinds of MgB_2 wires. It is noticed that the high composition areas of O are distributed surround the B domain. Since the areas are also the high composition areas of Mg, the O should exists in form of MgO. It is considered that the O may mainly come from the surface of the start materials of boron particles. The core of domain B should be the remained unreacted boron. The nano-sized MgO crystals shown bright contrasts in STEM HAADF observation because they have a higher mass-density than MgB_2 or Mg_2Si (density ρ in $g.cm^{-3}$: MgO 3.6; MgB_2 2.57; Mg_2Si 1.99). This made it possible to identify the distribution of MgO in STEM HAADF mode for 3D observation. The 3D observations revealed that the MgO crystals appeared a layer-like distribution surround domain B in MgB_2 wires without addition of SiC, while they distributed more dispersedly in the wires with addition of SiC. It is suggested that in the sintering of MgB_2 in IMD process, Mg reacted firstly with O on the surface of boron particles to form MgO, and then reacted with boron to form MgB_2, finally the Mg was consumed and the unreacted boron was remained. As a result, the nano-crystals of MgO distributed surround domain B in a layer-like shape for the MgB2 without addition of SiC. The results implied that the addition of SiC slowed down the reaction of Mg with O and resulted in the dispersion of MgO. The dispersed nano-sized MgO crystals may act as magnetic pinning centers in the wires. Therefore, the MgB_2 wires with addition of SiC exhibited a higher J_C than those without addition of SiC.

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