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The Effect of Seed on Top-seeded Melt-growth (TSMG) Processing of a RE-123 Superconductor

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Abstract

This study investigated the effects of different kinds of seed crystals with miscut angles and pretreatment on the characteristics of a RE-123 superconductor processed by a top-seeded melt-growth (TSMG) method. When the seed crystal was heat-treated in an oxygen atmosphere, the surface structure was cleaned removing hydroxide. When the seed crystal had a miscut angle, in addition, the surface structure showed a well defined hill-and-valley structure after heat-treatment. A better microstructure, with a well-distributed small RE-123 phase, was obtained using a high miscut angle after heat-treatment in an oxygen atmosphere. As a result of the microstructure improvement, the magnetic characteristics also improved. The experimental result can be explained by reduction of nucleation activation energy.

Keywords : Superconductor, RE-123, seed, miscut, TSMG

I. Introduction

The top-seeded melt-growth (TSMG) method is one of the most promising methods for producing a superconducting magnet applicable to an energy storage system. The superconducting magnet has to have a high critical current density under an applied magnetic field. Though a number of research efforts have been carried out using the TSMG method to increase critical current density, the effect of seed on the manufacturing process appears not yet studied. In this study, we tried to study the effect of seed on the microstructure and the resultant superconducting properties of the RE-123 superconductor processed by the TSMG method.

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II. Experimental

Precursor powders with RE-123=Nd, Gd, and Dy):2Ba:3Cu cation ratios were prepared by the solid-state reaction of Nd_2O, Gd_2O, Dy_2O, $BaCO_3$, and CuO powders. The proportioned powders were ball milled for 24 hours in ethyl alcohol and dried at 120°C for 12 hours in air. The calcination was performed twice at 880°C for 3 hours. The calcined powder was compressed into pellets, 20 mm in diameter and 5 mm in height, at 2 kg/cm^3. We used various kinds of seed crystal such as MgO, NGO, LAO, and STO. The miscut angle of each seeds was 0°, 0.1°, and 4°, respectively. The size of the seeds was 3 x 3 mm. To remove hydroxide on a sample of seed, the seed crystal was heat-treated at 1000°C for 3 hours in an oxygen atmosphere. Phase identification of the heat-treated compacts was accomplished by using an X-ray diffractometer (XRD: Rigaku D-Max III A). Microstructures were observed under an optical microscope (OM) and scanning electron microscopy (SEM: Hitachi S-4700). The magnetic characteristic of the heat-treated compacts was compared by a physical property management system (PPMS) (Quantum Design 6000).

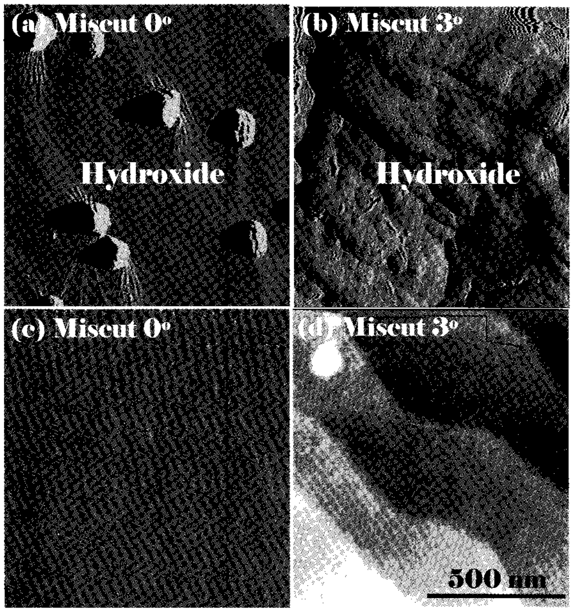

Fig. 1. AFM surface morphology of MgO [001] crystal. (a) exposed to air atmosphere for 24 h, (b) after heat-treatment at 1000°C, oxygen atmosphere for 3 h, and (c) 4° miscutted MgO crystal after the same heat-treatment of (b).

III. Result and discussion

Figure 1 shows the AFM surface morphology of an MgO crystal, with and without the miscut angle, before and after heat-treatment at 1000°C, in an oxygen atmosphere. Figure 1(a) shows the hydroxide formed on the surface of the MgO seed. Generally hydroxide can be easily removed by a heat-treatment for a short time as shown in Figure 1 (b). Figure 1(c) shows a surface morphology of 4° miscut MgO seed after the heat-treatment with the well known hill-and-valley structure formed to reduce total surface energy of MgO.

Because the hill-and-valley structure has many kink sites, in general, there is no activation energy for homogeneous nucleation. Though the nucleation is not homogeneous but a heterogeneous one in this experiment, the existence of kink sites can reduce the activation energy for the nucleation. As a result of easy nucleation, the crystallinity of the RE-123 superconductor seems to be better compared to the high activation energy of plat seed surface.

Figure 2 shows microstructures of a Re-123 (RE=Nd, Gd, Dy, Eu, NED) superconductor, using a MgO seed, cooled down at a rate of 5 K/min from 20°C higher than the peritectic temperature after 20 minutes duration at that temperature. The Nd-123 superconductor (Figure 2(a)), where the ionic radius of a rare-earth element is largest, showed the largest 211 phase and polycrystalline 123 phase. When the ionic radius decreased from Nd to Gd and Dy (Figure 2(c)), the size of the 211 phase decreased and crystallinity of the 123 phase was improved. Especially the Dy-123, showed the best microstructure with well-distributed and small sized 211 phase and a c-axis aligned 123 nearly single crystal.

Figure 3 shows the microstructure of a Gd-123 superconductor using various seeds of MgO, STO, NEO, and LAO with 0°, 0.1°, and 4° miscut angles. When the NGO and LAO seeds were used, there was a severe chemical reaction between seeds and the superconductor. On the contrary, MgO and STO showed good microstructures of large Gd-123 grain with small and well-distributed Gd-211. In addition, heat-treated MgO and 4° miscut STO seeds showed better microstructures compared to no heat-treated MgO and no miscut STO seeds.

Figure 4 shows the magnetic hysteresis curve of

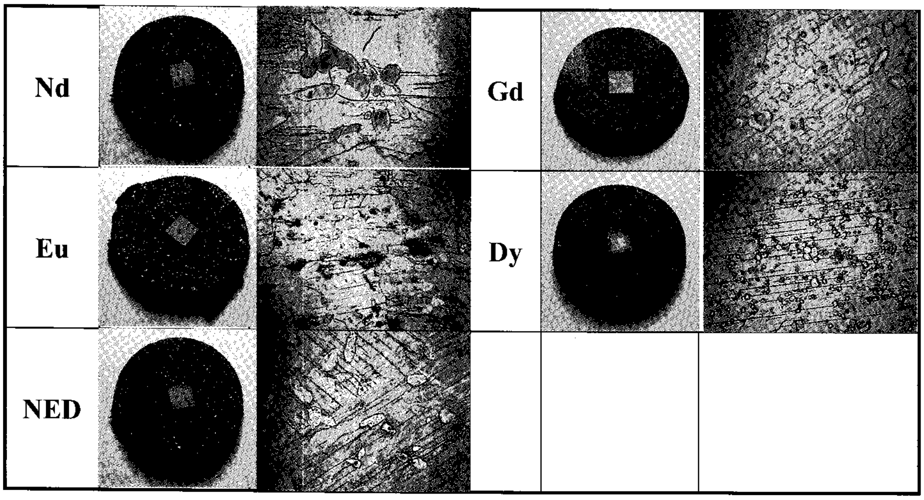

Fig. 2. Optical micrographs of RE-123 (RE=Nd, Gd, Dy, Eu, NED) superconductors processed by TSMG method using no miscut MgO seed.

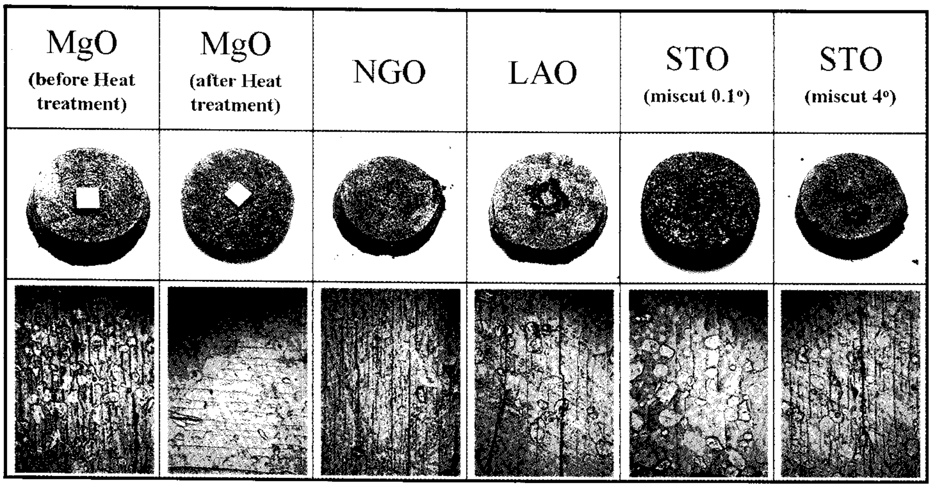

Fig. 3. Optical micrographs of Gd-123 superconductors according to various seed crystal. (a) no miscut NGO, (b) no miscut LAO, (c) no miscut MgO, (d) no miscut MgO annealed at 1000°C, (e) 0.1° miscut STO, (f) 4° miscut STO.

the RE-123 superconductors as shown in Figures 2 and 3. The Gd-123 superconductors of Figures 3(b) and (f), using heat-treated MgO and 4° miscut STO seed, showed 5 times higher than the others using no heat-treatment and no miscut.

IV. Conclusion

The effects of different kinds of seed crystals with miscut angles and pretreatment were investigated with respect to the resulting microstructure change and change in superconducting properties of RE-123 superconductors processed by a TSMG method. When the ionic radius of rare-earth elements decreased from Nd to Dy, a better microstructure was obtained. The pre-heat-treated and high miscut seeds also showed a better microstructure. The experimental results were explained by the reduced activation energy for nucleation of superconducting 123 phases. The reduced activation energy could speed up the peritectic reaction for 123 phase formation and thus resulted in the better microstructure. As a result of microstructure improvement, the magnetic hysteresis characteristics of superconductor also improved.

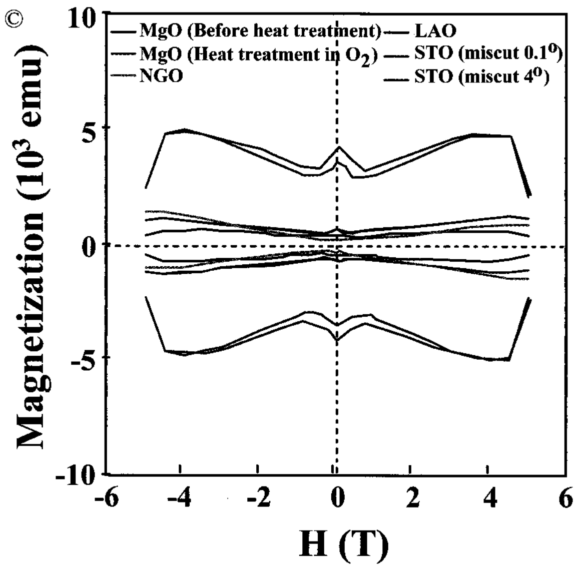

Fig. 4. Magnetic hysteresis curves of RE-123 (RE=Nd, Gd, Dy, Eu, NED) superconductors as shown in Figs. 2 and 3.

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