Seed melting during seeded-melt growth process of YBCO superconductors

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Abstract

Melting and re-solidification nature of $SmBa_2Cu_3O_{7-y}$ (Sm123) grains in Ba-Cu-O (Ba:Cu=3:5) liquid containing 0.7 at.% yttrium were investigated at the temperature lower than its melt point. When Sm123 grains/liquid powder compacts were heated to a temperature between two melting points of Ba-Cu-O liquid (1000°C) and a Sm123 phase (1060°C) and held at this temperature for appropriate time, Sm123 grains melted partly in the liquid that was formed by melting of the liquid-forming powder. During subsequent slow cooling, $(Sm,Y)Ba_2Cu_3O_{7-y}$ solidified at the outer parts of the unmelted Sm123 grains, which is distinguished from the core regions by lower Sm_2BaCuO_5 (211) density.

Key Words : Seed melting, YBCO superconductor, resolidification

1. Introduction

It was recently found that SmBa2Cu3O7-y (Sm123) seeds used for top-seeded melt growth (TSMG) processed YBCO samples melted during processing, although the processing temperatures were lower than a melting point (m. p.) of Sm123 [1-3]. The seed melting began at the seed/compact interface and affected the growth nature of the top surface of the TSMG-process YBCO samples. Severe seed melting led to the growth of many undesirable subsidiary Y123 grains at the top surface, deteriorating the superconducting and magnetic properties [4,5]. Due to the complicated heat treatment cycles of the TSMG process, it was difficult to understand the mechanism of the Sm123 seed melting. Therefore, a more simple experiment should be applied to the SmBCO seed-YBCO compact system to understan the SmBCO seed really melts at the temperature lower than its m. p. In this study, single crystalline SmBCO samples were dipped into the peritectic melt at the temperature below its m. p. of the seed and the microstructural variation associated with the seed melting was investigated.

2. Experimental procedure

The SmBCO slab samples used in this study were prepared by a conventional melt process involving slow cooling through the peritectic temperature (Tp). The composition of the SmBCO slabs was $Sm_{1.8}Ba_{2.4}Cu_{3.4}O_x$ (1 mole Sm123 + 0.4 mole Sm211). $Ba_3Cu_5O_8$ (a mixture of $3BaCuO_2$ and 2CuO) powder containing 0.7 at. % yttrium was used as melt-forming powder. 0.7 at. % yttrium is an equilibrium concentration at the processing temperature (1030°C) of this study [6]. At this processing temperature the SmBCO slabs should not melt, because the temperature is lower than

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m. p.(1060°C) of Sm123. If Sm123 slabs melt during heat-treatment due to any reasons, one can observe the microstructural variation associated with the seed melting. The melt-forming powder and the SmBCO slabs were put into the MgO crucible, heated to 1030°C at a rate of 100°C/h, held for 10 h at this temperaturé, cooled to 950°C at a rate of 50°C/h and then cooled again to room temperature.

3. Results and discussion

Figure 1 *shows the microstructure* of the SmBCO slabs heat-treated by the above heating cycles. It can be seen that the SmBCO grains are surrounded by a mixture of $BaCuO_2$ and CuO that were formed by the eutectic separation of the high-temperature peritectic melt. Closely observing the grains, the 211 density in grain interior is higher than in the outer parts. No 211 particle is observed in the regions adjacent to melt.

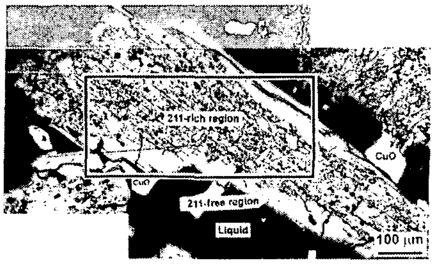

Fig. 1. Optical microstructure of the SmBCO grain dipped in a melt; heated to 1030 °C, cooled to 950°C at a rate of 20 oC/h.

Scanning electron microstructure (SEM) and wavelength dispersive x-ray (WDX) analysis was carried out for the rectangular region of the Sm123 grain of Fig. 1. Figures 2(a), 2(b) and 3(c) are a SEM micrograph of the rectangular region, the samarium and the yttrium mapping data for Fig. 1(a), respectively. As can be seen in Figs. 2(b) and 2(c), samarium is detected in all parts of the SmBCO grain. The samarium concentration of the core part is higher than that of the outer parts. No yttrium is detected in the core region. On the other hand, both samarium and yttrium are detected in the outer parts of the SmBCO grain containing no 211 particle.

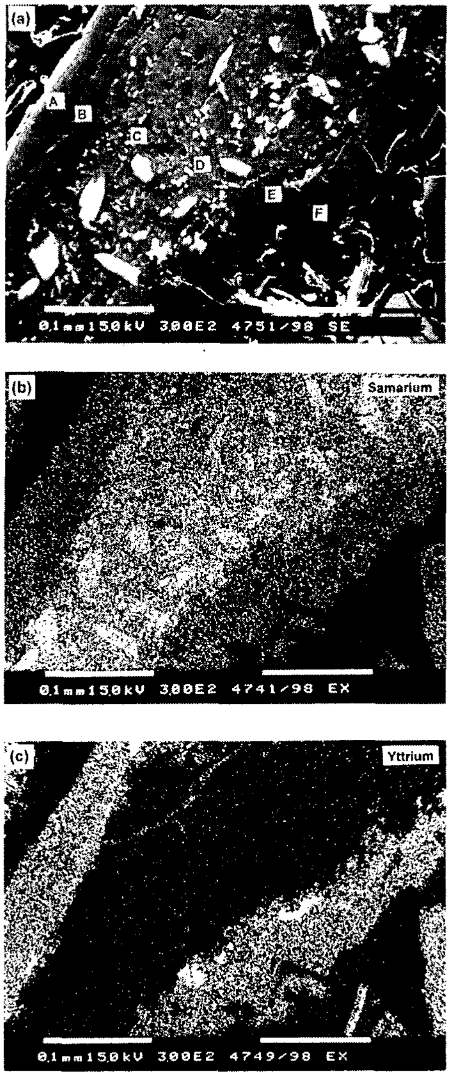

Fig. 2. (a) SEM micrograph of the marked rectangular region Fig. 1, and (b) and (c) are samarium and yttrium mapping, respectively.

Composition of the six points crossing the grain (the marked points as A to F of Fig. 3(a)) was analyzed. The regions marked as A, B, E and F, are solid solution $Sm_{0.5}Y_{0.5}Ba_2Cu_3O_{7-y}$ while the core regions marked by C and D are $SmBa_2Cu_3O_{7-y}$. This result strongly suggests that the grain core did not melt during heat-treatment, while the outer parts melted and resolidified. The yttrium involved in the outer regions seems to be supplied from the peritectic melt containing 0.7 at.% yttrium during the slow cooling to 950°C.

Based on the microstructural investigation of this study, the abnormal melting and re-solidification nature of SmBCO seeds can be explained as follows. When a mixture of melt-forming powder and SmBCO slabs are heated to 1030°C, the melt-forming power melts to form a peritectic melt containing yttrium. When the surface of the SmBCO slabs wets with the melt, it begins to melt. Although the mechanism of the seed melting is not clear yet, samarium solubility in the melt may be the driving force for the seed melting. The melt will take samarium to reach an equilibrium state. As a result of the seed melting, the melt adjacent to the seed contains both samarium and yttrium. As reported in elsewhere [5,7], m. p. of Sm123 and Y123 phases are 1015 °C and 1060°C, respectively. Thus solidification temperature of the melt containing both rare-earth elements ranges between the two melting points. During the subsequent cooling from 1030°C to 950°C, the melt solidifies again, forming $(Sm_{1-x}Y_x)Ba_2Cu_3O_{7-y}$ regions at the outer regions of the SmBCO grain.

In summary, we demonstrated that SmBCO grain melted in the peritectic melt at the temperature lower than a melting point of a Sm123 phase. During cooling after holding at 1030 °C, the dissolved mass solidifies again, forming $(Sm_{1-x}Y_x)Ba_2Cu_3O_{7-y}$ in the outer regions of the SmBCO grain, which is distinguished from the unmelted SmBCO core by the lower 211 density.

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