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준 용융법으로 제조한 Y-Ba-Cu-O 초전도체의 방향성 성장 영역에서의 산소확산 과 미세균열 생성

Oxygen Diffusion and Microcrack Formation in the Textured 1-2-3 Region of Partial-Melted Y-Ba-Cu-O Superconductor

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I. Introduction

Superconducting transition temperature (T_c) of $Y_1Ba_2Cu_3O_y$ (1-2-3) oxide is well known to be sensitively influenced by the oxygen content in 1-2-3 phase[1]. By cooling the oxide in oxygen atmosphere from 900-950°C which is usual sintering temperature of this system, the high-temperature tetragonal phase transforms to the low-temperature orthorhombic phase due to the one-dimensional oxygen ordering on Cu(1)-O chain. In the polycrystalline Y-Ba-Cu-O system made by the conventional sintering method, oxygen atoms diffused into 1-2-3 grains through open porosity and grainboundary[2,3]. Meanwhile, the Y-Ba-Cu-O sample prepared by the liquid phase techniques involving the peritectic reaction, shows a different microstructure from the conventional-sintered sample. This microstructure is characterized by large textured 1-2-3 grains and pore-free structure. In spite of absence of open porosity and the grainboundary, the time needed to obtain a T_c of 90 K is shorter than that expected normally, though it is longer than that of the conventional-sintered sample. This paper is aimed to observe the diffusion procedure of oxygen atoms in the textured 1-2-3 region of partial melted Y-Ba-Cu-O oxide.

2. Experimental procedure

The 1-2-3 powder used in this experiment was made by the solid state reaction of Y_2O_3, $BaCO_3$ and CuO powders of 99.9% purity. The powders were mixed and calcined at 900°C for 24 h in air. The calcined powder was crushed in an alumina mortar with a pestle. The crushed powder was uniaxially pressed at 300 MPa and isostatically pressed into cylindrical pellet. The pellet was sintered at 950°C for 24 h in air and then air cooled. The sintered pellet was rapidly heated to 1030°C, cooled to 1000°C with a cooling rate of 30°C/h and then cooled again to 980°C with 1°C/h. The pellet was maintained at 980°C for 10 h and then air cooled. In order to investigate the microstructural change with oxygen diffusion, the pellets were annealed for various time periods at 450°C in air. Microstructure was investigated on a polished surface of a sample by an optical polarized microscope.

3. Results and discussion

Fig. 1 shows an optical polarized micrograph of the partial-melted sample annealed at 450°C for 1 h in air after air cooling from 980°C. The microstructure shows the impinged area of two textured 1-2-3 regions. Small 2-1-1 particles which was trapped in the textured region and remnant liquid phase at the grainboundary are observed. This is a typical microstructure of the Y-Ba-Cu-O sample made by the peritectic reaction, liquid phase + 2-1-1 phase → 1-2-3 phase. The large cracks running along a certain crystal direction were also observed in the textured region. These cracks appears to be formed during the cooling stage from 980 °C. The regions near the thermal cracks have a different brightness from the crack-free region under an polarized beam. The crack-bearing region has an optical twin structure and the crack-free region no twin structure. The observed microstructure indicates that the tetragonal-to-orthorhombic phase transformation in the textured region was made through the thermal cracks which is a fast diffusion path of oxygen atoms. This finding is comparable to the polycrystalline $Y_1Ba_2Cu_3O_y$ of small grain size where diffusion path of oxygen atoms is open porosity and grainboundaries[2].

Figure 2 shows the microstructural change of the marked region of Fig. 1 with annealing time of (a) 6 h and (b) 20 h in air. The orthorhombic region which was started at the thermal cracks extended to the crack-free region with increasing annealing time. The untransformed region of crack free is still observed in the sample (a), which is due to the insufficient annealing time. Whereas almost of the tetragonal region was transformed to orthorhombic phase in the sample (b). It is noticeable that the phase change induced the formation of microcracks running along the (001) cleavage plane [4] of $YBa_2Cu_3O_y$. As well as the thermal cracks, these microcracks would offer an effective diffusion path for oxygen atoms.

The texture-growth techniques such as liquid-phase processing [5], zone-melt texturing [6] and melt-powder-melt growth [7] are known to be effective in increasing high critical current density (J_c) in Y-Ba-Cu-O superconductor. However, a lot of microcracks which is harmful to the J_c, were always involved in their microstructure. The thermal cracks by improper thermal procedure may be reduced by careful control of heating schedule, whereas the microcracks due to the transformation stress appears not to be controllable in the system itself. Attempt to reduce the microcracks such as putting the crack-blunting medium in the textured 1-2-3 region may improve the J_c of the Y-Ba-Cu-O system.

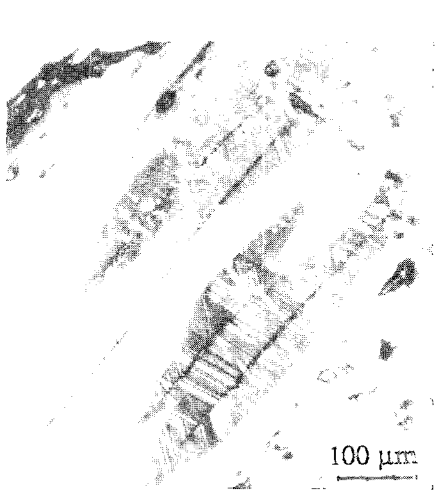

Fig. 1. Microstructure of partial-melted $Y_1Ba_2Cu_3O_y$ showing the oxygen diffusion along the thermal cracks in the textured 1-2-3 region.

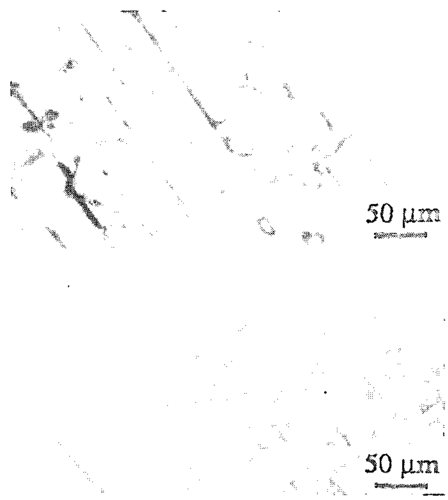

Fig. 2. Microstructural change with annealing time in air. Annealing time of (a) and (b) are 6 h and 20 h, respectively.

4. Conclusion

Oxygen diffusion in 1-2-3 textured region was made through the thermal cracks by improper thermal cooling. The tetragonal-to-orthorhombic phase transformation in the oxygen diffused region accompanied by the formation of microcracks running along the a-b plane. With the thermal cracks, the transformation cracks also offer good diffusion path for oxygen atoms. These results are comparable to the fact that the open porosity and grainboundary is a diffusion path of a polycrystalline Y-Ba-Cu-O oxide [2].

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