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The effect of sintering temperature on the crystal growth of the Bi-2223 phase in multifilamentary tape

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Abstract

Bi-2223 tapes with 37 filaments, Ag–Mg–Ni alloy outer sheath and pure Ag inner sheath were heat treated in 7.5% O₂ at different temperatures for 40 h. XRD of etched samples for increasing sintering temperature from 798 to 847 °C showed an increase of eightfold in the absolute intensity of the Bi-2223 (0 0 10) peak, without substantial decrease in the intensity of the Bi-2212 (0 0 8) peak. These changes were correlated with the amount of liquid phase obtained from the Bi-2201 (0 0 6) peak. Optical microscopy also suggested an important role of the liquid phase by revealing significant changes in the morphology of Bi-2223 grains with increasing sintering temperature.

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1. Introduction

The understanding of the material science in Bi-2223 tapes is far from complete. Problems such as porosity, density, [1,2], texturing, [3], and secondary phase formation are as yet unsolved. Currently a lot of evidence supports a nucleation and growth model for the formation of Bi-2223 tapes, [4,5], where the Bi-2223 grows from a transitory liquid using solid Bi-2212 as a template. However little work has been done to encourage the role of the

liquid phase in the removal of pores and the improvement of density and texture.

2. Experimental

Using Merck powder, of nominal stoichiometry Bi_{1.8}Pb_{0.3}Sr_{1.9}Ca_{2.1}Cu_{3.1}O_x, 37 filament, Ag–Mg–Ni alloy outer, Ag inner tape was manufactured at BICC General Superconductors by the PIT process. In tube furnaces that have three-zone control, 20 cm long pieces of tape were heat treated in a temperature gradient of ~1 °C/cm with the tape laid parallel to the gradient. Only one sintering was applied to the tapes. The heating rate was 100 °C/h to 560 °C, 60 °C/h to 700 °C, 20 °C/h till 20 °C below the sintering temperature and then 100 °C/h to the sintering temperature. After a dwell time of 40 h the tapes were cooled at 60 °C/h.

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XRD and optical microscopy was performed on etched tapes.

3. Results and discussion

With etched samples, as the sample area is larger than the X-ray beam, it is possible to compare the absolute peak height between tape samples, all XRD data presented in this paper shows absolute intensities. The XRD data shown in Fig. 1(a), on the left, is for the Bi-2201 (006) peak. At 814 °C and below there is no Bi-2201, then from 822 to 847 °C the amount of Bi-2201 increases. The Bi-2201 phase is known to be stable below 770 °C, [6], and is known to convert to Bi-2223 between 800 and 825 °C in 7.5% partial pressure O₂, [6,7]. Oil quenched tapes, [8], show no Bi-2201 phase but

rather an amorphous phase, this suggests that the Bi-2201 forms on cooling from the liquid phase, and so is indicative of the amount of liquid phase present at the sintering temperature. An increasing amount of liquid phase with temperature is typical of a liquid phase sintering system, [9], the solubility of the Bi-2212 in the liquid phase can be one reason for this, which would explain why Bi-2201 grows from the liquid phase on cooling as the right ions are present. Also the increase in the volume of liquid phase may be due to Bi-2212 becoming more soluble with sintering temperature.

The XRD data for the Bi-2223 phase is shown in Fig. 1(b). The Bi-2223 (0010) peak height increases with temperature to a maximum at 836 °C, at higher temperatures the peak height decreases. The Bi-2212 (008) peak (also shown in Fig. 1) stays low at all temperatures, perhaps starting to

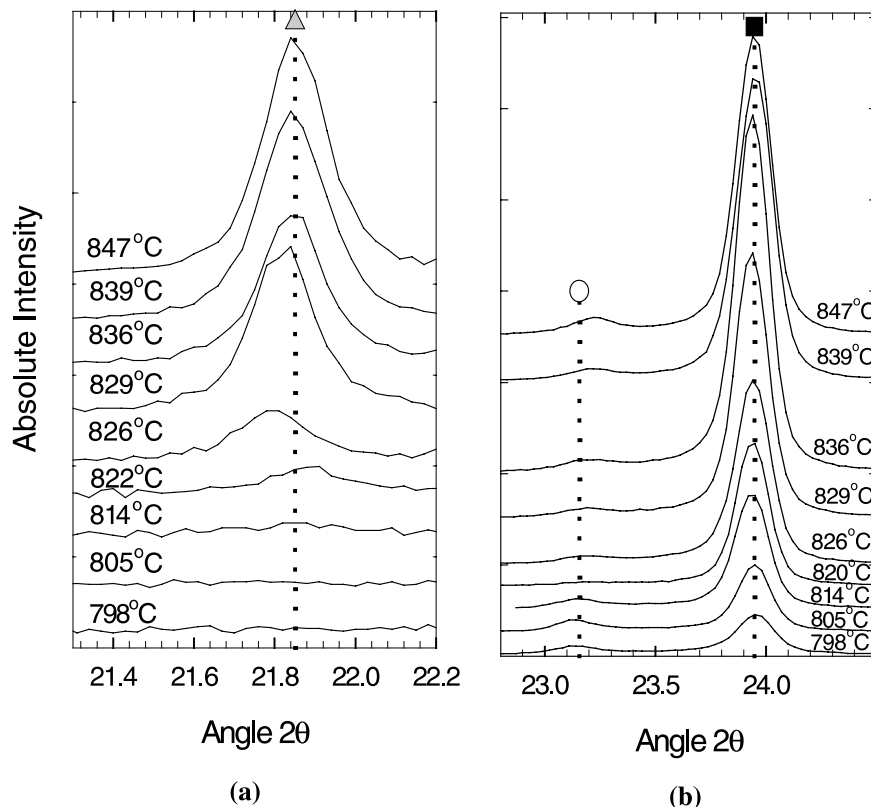


Fig. 1. Absolute intensity/ 2θ XRD data at different temperatures, for the Bi-2201 (006) peak (a) and the Bi-2212 (008) (○) Bi-2223 (0010) (■) peaks (b).

increase at 847 °C. The increase in the Bi-2223 peak height from 798 to 836 °C is by a factor of 8, and so cannot be attributed to the phase percentage as there would have to be an ~80% volume percent of amorphous phase at 798 °C. Large changes in the peak height can however come from changes in texture, long-range crystalline order, and grain size. The FWHM of the peaks was measured but the error in the measurement was larger than the changes, so the grain size may affect the peak height but it is not likely to be the only cause of the changes.

Optical micrographs of etched tape are presented in Fig. 2(a) for tape heat treated at 816 °C and Fig. 2(b) for tape heat treated at 832 °C. At 816 °C there are misaligned Bi-2223 grains that appear as dark patches because they are out of

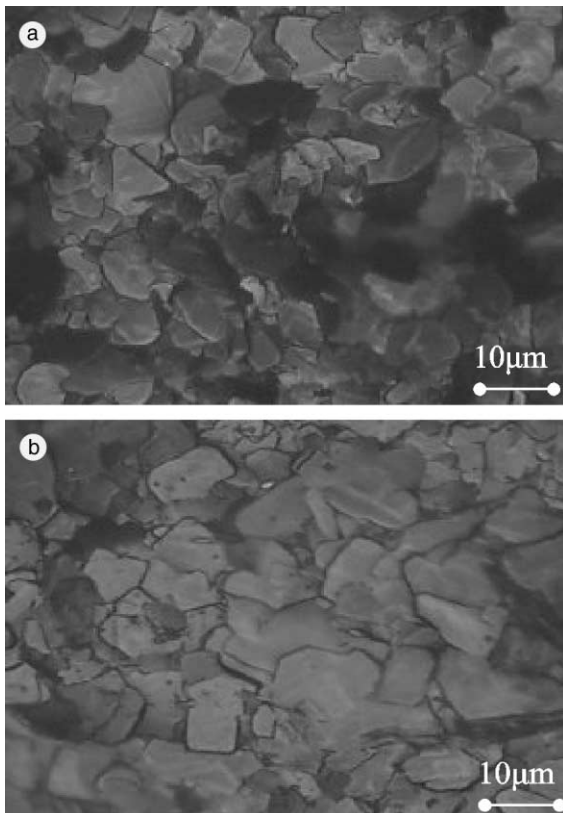


Fig. 2. Optical microscopy images for etched tape sintered at 816 °C (a) and 836 °C (b). The dark grains, left image, are out of focus, misaligned Bi-2212.

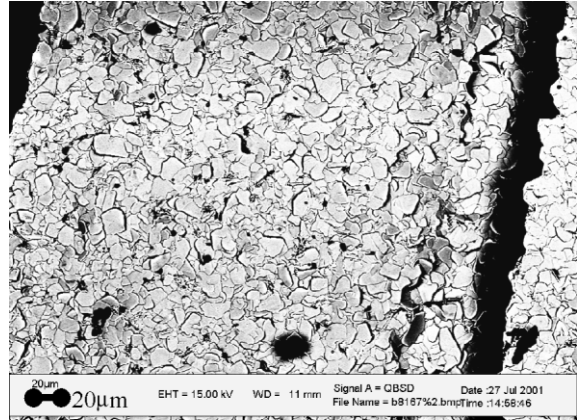


Fig. 3. Backscattered electron microscopy image for etched tape sintered at 816 °C shows no large amount of porosity or secondary phases.

focus. A backscattered electron micrograph of the 816 °C sample is shown in Fig. 3, it demonstrates that the dark patches in Fig. 2(a) are not secondary phases nor porosity. In the optical micrograph of the 836 °C sample the grains are coalescing into large well-textured grains.

The observations show a change in the Bi-2223 grain structure with temperature, this is likely to be a direct result of a re-arrangement in the Bi-2212 grains (the growth templates for the Bi-2223) in a sufficiently large volume of liquid phase. Sufficient liquid between the Bi-2212 platelets will encourage capillary forces to draw grains together, increasing density and texture at the same time.

An alignment of the grains by capillary forces during the early stages of sintering would explain the increase of the XRD Bi-2223 peak height due to texture improvement. A second possible reason for the observed increase in Bi-2223 XRD peak height is that the large textured grains seen in Fig. 2(b) are more efficient at diffracting X-rays than the smaller grains seen in Fig. 2(a). Large grains that are grouped having been drawn parallel by capillary forces would have the long-range crystalline order necessary to diffract X-rays efficiently, however the overall texture of the tape may be unchanged as there is still the same misalignment between groups of grains. This theory would see support from recent TEM work, [10], and MO

work, [11], where grain colonies are well aligned in themselves but poorly with respect to each other.

4. Conclusions

During the first 40 h of sintering the volume fraction of liquid phase increases with sintering temperature, it has been characterised by an increase in the amount of Bi-2201 present after cooling. The Bi-2223 (0 0 10) peak has been observed to increase eightfold with sintering temperature, from 798 to 836 °C. The increase in the Bi-2223 peak height with sintering temperature is thought to be due to a re-arrangement of the Bi-2212 crystals, which occurs in the larger volume of liquid phase.

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References

- [1] A. Kasztler, M. Polak, M. Foitl, H. Kirchmayr, *Inst. Phys. Conf. Ser. No. 167*, 519, Applied Superconductivity, Spain, 1999.
- [2] M.O. Rikel, R.K. Williams, X.Y. Cai, A.A. Polyanskii, J. Jiang, D. Wesolowski, E.E. Helstrom, D.C. Larbelestier, K. DeMoranville, G.N. Riley Jr., *IEEE Trans. Appl. Supercond.* 11 (1) (2001) 3026.
- [3] B.A. Glowacki, *Supercond. Sci. Technol.* 11 (1998) 989–994.
- [4] D.P. Grindatto, J.-C. Grivel, G. Grasso, N.-U. Nissen, R. Flükiger, *Physica C* 298 (1999) 41.
- [5] J.S. Luo, N. Merchant, V.A. Maroni, D.M. Gruen, B.S. Tani, W.L. Carter, G.R. Riley Jr., *Appl. Supercond.* 1 (1/2) (1993) 101.
- [6] Y.L. Chen, R. Stevens, *J. Am. Ceram. Soc.* 75 (5) (1992) 1160.
- [7] H.K. Liu, J. Horvat, R. Bhasale, W.G. Wang, B. Zeimetz, S.X. Dou, I. Kusevic, E. Babic, *Supercond. Sci. Technol.* 11 (1998) 1057.
- [8] T.G. Holesinger, A. Ayala, R.M. Baurceanu, V.A. Maroni, *IEEE Trans. Appl. Supercond.* 11 (1) (2001) 2991.
- [9] R.M. German, *Liquid Phase Sintering*, Plenum Press, New York, 1985.
- [10] L.G. Andersen, S. Bals, G. Van Tendeloo, H.F. Poulsen, Y.L. Liu, *Physica C* 353 (2001) 251–257.
- [11] A. Polyanskii, D.M. Feldmann, S. Patnaik, J. Jiang, X. Cai, D. Larbelestier, K. DeMoranville, D. Yu, R. Parrella, *IEEE Trans. Appl. Supercond.* 11 (1) (2001) 3269.