JOURNAL OF MATERIALS SCIENCE, 32 (1997) 2595–2598

Evolution of texture in melt-grown Y–Ba–Cu–O and Gd–Ba–Cu–O superconductors

R. GOPALAN, A. K. SINGH, T. RAJASEKHARAN
Defence Metallurgical Research Laboratory, Hyderabad-500 058, India

Microstructural studies and X-ray diffraction combined with pole figure characterizations have been carried out on the melt-grown $YBa_2Cu_3O_{7-x}$ and $GdBa_2Cu_3O_{7-x}$ samples. The crystallographic orientation of the domains in melt-grown samples are highlighted based on the above studies.

1. Introduction

Melt growth processes such as MTG (melt textured growth) [1], QMG (quench melt growth) [2] and MPMG (melt powder melt growth) [3] have been successfully used in obtaining high critical current densities (J_cs $> 10^4$ A cm^{-2}) in $YBa_2Cu_3O_7$ (Y-123) superconductors. In the case of $GdBa_2Cu_3O_7$ (Gd-123), it is reported that reduced oxygen atmosphere during melt processing is essential in obtaining high J_c at 77 K [4]. In all these processes, bulk sintered samples are partially melted above their peritectic decomposition temperatures and then slowly cooled at a rate of 1 °C h^{-1} to a lower temperature. Although several variations in melt growth process are reported, the 123 samples thus processed show a typical domain structure in which each domain consists of oriented 123 platelets aligned along their a–b planes. The 123 platelets within each domain will have a common c-axis. Since the critical current density in 123 superconductors is anisotropic, it is of interest to know the c-axis misalignment between the individual domains. This information will enable one to determine the orientation of the individual domains with respect to sample surface. One can obtain these details using X-ray pole figure analysis and such a study is reported in this paper.

2. Experimental procedure

Bulk sintered samples of Y-123 and Gd-123 were melt processed as reported elsewhere [5, 6]. The samples were initially kept at 1130 °C in a preheated furnace for 10–20 min followed by slow cooling at a rate of 1 °C h^{-1} through the peritectic formation temperature of 123 (1030 °C for Y-123 and 1065 °C for Gd-123 in O_2 atmosphere) in flowing oxygen. The samples were heat treated at 930 °C for 24 h and then cooled to room temperature, with long holds between 600 and 400 °C.

X-ray diffraction (XRD) studies of the samples were carried out using a Siemens D500 X-ray diffractometer with CuK_α radiation. Pole figure studies were conducted using a texture goniometer with MoK_α radiation. The Schulz back reflection technique [7] was employed to obtain the pole figure of the samples. The X-ray intensity data was corrected to random times, using powder data for sintered samples. The intensity data was also corrected for defocusing.

Microstructure of the samples were obtained using an optical microscope with stereo projection. The fractured surface of the samples was examined using an ISI 100A scanning electron microscope (SEM).

3. Results and Discussion

3.1. Microstructure

Fig. 1a shows an optical micrograph obtained from a melt-grown Y-123 sample. The microstructure shows that the sample consists of multiple domains, each having a size of $\sim$ 500–1000 μm. A domain is a region which consists of many stacked 123 platelets having a common c-axis. Such a stacking of 123 platelets within a domain is shown in Fig. 1b and c for Y-123 and Gd-123 samples, respectively. The average width of the platelets is found to be $\sim$ 10–15 μm. The stacked platelets microstructure has been described in the literature as a "brick-wall" microstructure [8, 9]. A typical 123 domain identified using a stereo projected optical microscope is shown in Fig. 1d. It is important to mention here that high J_c values ($> 10^3$–10^4 A cm^{-2}) have been reported in such a single domain [10]. These domains form the large three-dimensional network which reduces the effective current flow path in melt-grown samples.

3.2. XRD measurements

In order to obtain orientation of the 123 platelets, general XRD measurements were performed on melt-grown Y-123 and Gd-123 samples and were compared with the XRD of sintered samples. It was observed that the (0 0 l) reflections in melt-grown samples were relatively intense when compared with that of sintered samples, indicating the presence of the preferred orientation. To support this observation, Fig. 2 compares the XRD patterns of sintered and melt-grown Y-123

0022–2461

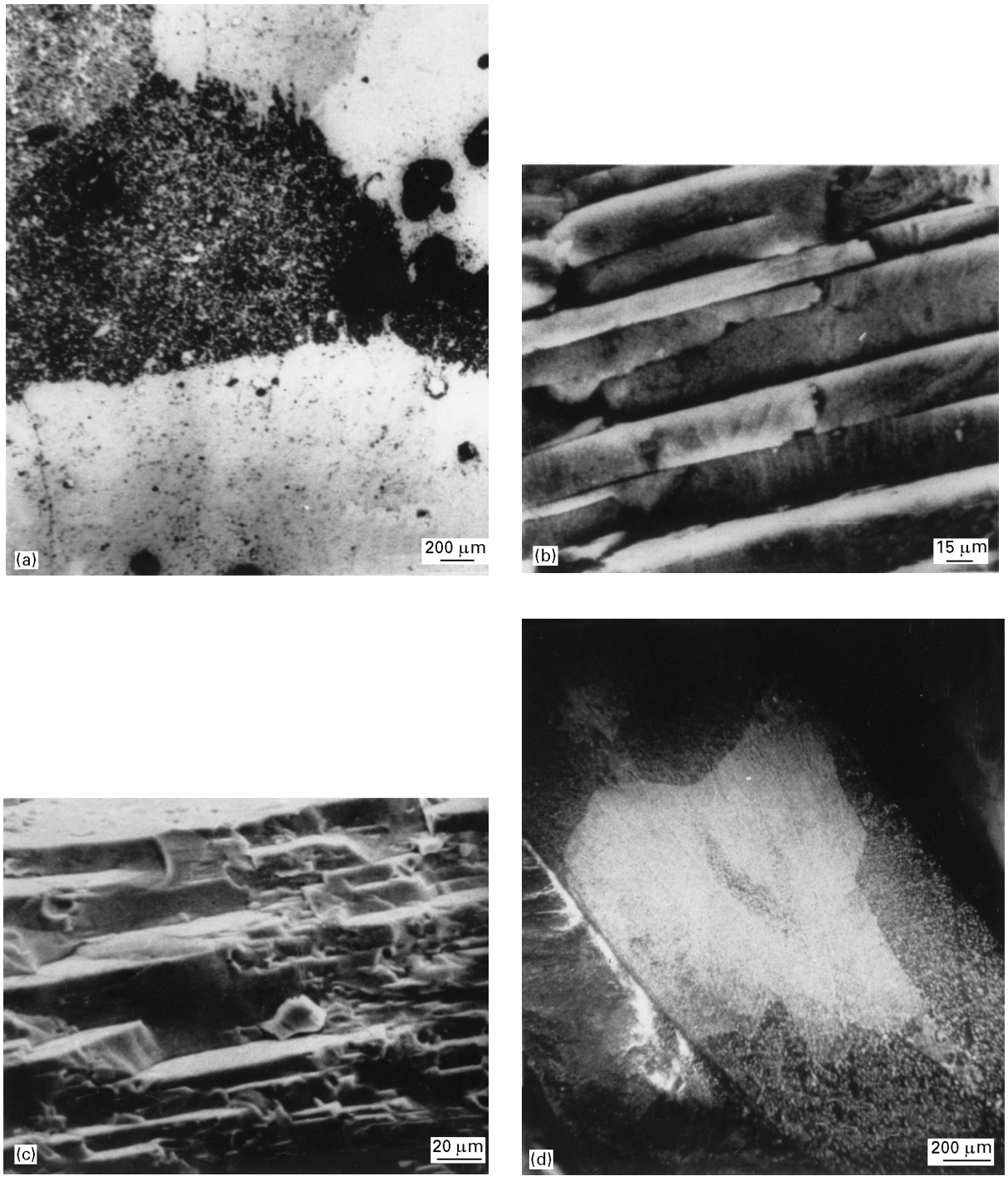

Figure 1 (a) Optical micrograph showing presence of various domains in a melt grown 123 sample, SEM micrographs showing stacked platelets in (b) Y-123, (c) Gd-123 specimens and (d) stereoprojected optical microstructure revealing a single domain in melt-grown Y-123 sample.

samples. The degree of texturing can be expressed as a relative intensity (I_R) of the (0 0 l) to the (1 1 0, 1 0 3/0 1 3) peaks, normalized to the intensities in a randomly oriented sample

$$I_R = \frac{(I_{0\,0\,l}/I_{1\,1\,0,\ 1\,0\,3/0\,1\,3})_{\text{textured}}}{(I_{0\,0\,l}/I_{1\,1\,0,\ 1\,0\,3/0\,1\,3})_{\text{random}}}$$

The I_R values estimated for (0 0 3), (0 0 5) and (0 0 6) reflections in the melt-grown Y-123 samples are given in Table I.

From Table I, it can be noticed that I_R is >1 ($I_R = 1$ represents random texturing) and essentially it implies that melt-grown samples have appreciable alignment with their c-axis nearly parallel to the sample surface normal (sample pressing direction). The measurement of (0 0 l) pole figures of Y-123 and Gd-123 samples further confirms this observation.

In our earlier studies, we have shown that the c-axis misalignment exit between the domains and is tilted with respect to sample surface normal at angles of ~ 15–40° [11]. To obtain further crystallographic orientation pole figures were analysed in greater detail. In pole figure analysis, intensities of the Bragg diffraction of a particular $\{h\,k\,l\}$ plane were recorded

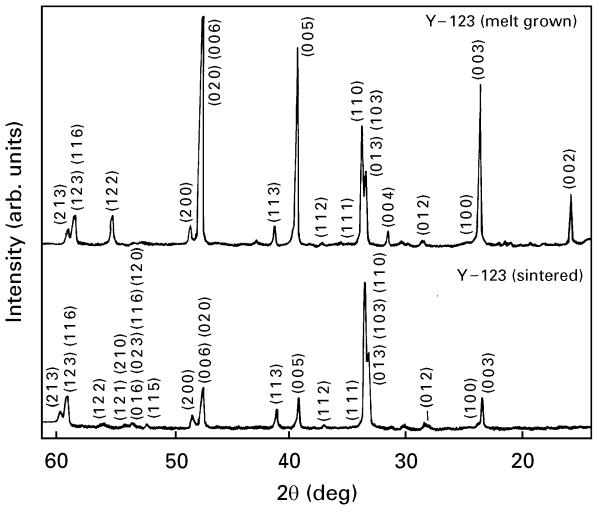

Figure 2 XRD patterns obtained from (a) sintered Y-123 and (b) melt-grown Y-123 samples.

TABLE I Relative intensity (I_R) of the (001) to the (110, 013/103) peaks normalized to the intensities of a random sample for melt grown Y-123 superconductors

Reflections	I_R
(003)	2.27
(005)	2.86
(006)	3.08

by tilting (Φ) the specimen around the axis on its surface and rotating (χ) around the axis normal to the surface with respect to X-ray incident beam. Fig. 3a and b shows pole figures of melt-grown Y-123 sample for reflections corresponding to (005) and (006) planes, respectively. Five intense poles were found at $\Phi = 15^\circ$ and 40° in the case of (005) and (006) reflections. The localization of the poles in the stereographic projection implies preferred orientation. In the case of reflection which occurs at $2\theta = 38.5^\circ$ (CuK_α) for 123 systems, the peak actually corresponds to a mixture of {005} and {104} planes. For the reflection which occurs at $2\theta = 47.4^\circ$ (CuK_α), the peak is a mixture of {006} and {020} planes. Thus, what we have measured using the pole figures represents distribution of two sets of planes having similar values of planar spacing. The pole figures shown in Fig. 3c and d for Gd-123 also represent a similar kind of distribution. If one considers the angular relationship between various crystallographic planes for an orthorhombic geometry, it can be estimated that the angle between [005] and [104] is $\sim 37^\circ$ and the angles among [104]s are 90°. Similarly, the angle between [006] and [020] is 90°. Keeping these relationships in mind we express that the various poles observed on the stereographic projection for (005) and (006) correspond to a mixed pole figure of {001} and {hkl} set of planes. Thus for Y-123, we represent in Fig. 3a, the peak corresponding to A1 as (005) and the peaks at A2–A4 as {104}. In Fig. 3b the intense poles at B1 correspond to (006) and those at B2–B5 correspond to {020} set of planes. Similarly in Fig. 3c and d for

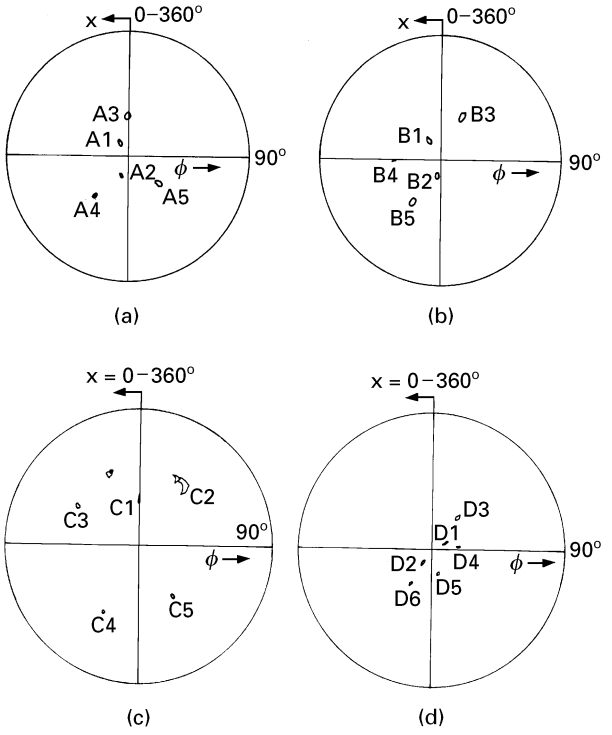

Figure 3 Pole figures showing texture of melt-grown samples (a) (005), (b) (006) reflections of Y-123 and (c) (005), (d) (006) reflections of Gd-123.

Gd-123 the poles at C1–C5 and D1–D5 can be indentified based on the angular relationship between the crystallographic planes. It can be noticed that the {001} pole, which is the measure of c-axis orientation is deviated by a few degrees ($\sim 15^\circ$) around the sample surface normal, in both Y-123 and Gd-123 samples. This misorientation or the lack of perfect texuring in our samples can be attributed to the variations in the temperature gradient that could occur over the length of the specimen during the melt growth process. It should also be mentioned here that the {001} and {hkl} poles are not symmetrically located in the stereographic projection and this may be due to the presence of various domains each having different crystallographic orientation with respect to sample geometry.

Acknowledgement

The authors are thankful to the Director, DMRL for the permission to publish this work.

References

1. S. JIN, T. H. TIEFEL, R. C. SHERWOOD, M. E. DAVIS, R. B. VAN DOVER, G. W. KAMMLOTT, R. A. FASTNACHT and H. D. KEITH, *Appl. Phys. Lett.* **52** (1988) 2074.
2. M. MURAKAMI, M. MORITA, K. DOI and K. MIYAMOTO, *Jpn J. Appl. Phys.* **28** (1989) 1189.
3. M. MURAKAMI, S. GOTOH, N. KOSHIZUKA, S. TANAKA, T. MATSUHITA, S. KAMBE and K. KITAZAWA, *Cryogenics* **30** (1990) 390.
4. M. MURAKAMI, S. I. YOO, T. HIGUCHI and N. SAKAI, *Jpn J. Appl. Phys.* **33** (1994) 1715.

5. T. RAJASEKHARAN, R. GOPALAN and T. ROY, *Pramana – J. Phys.* **37** (1991) L173.
6. R. GOPALAN, T. ROY, T. RAJASEKHARAN and G. RANGARAJAN, *J. Mater. Sci.* **31** 1996) 2557.
7. L. G. SCHULTZ, *J. Appl. Phys.* **20** (1949) 1030.
8. J. MANHART and C. C. TSEUEI, *Z. Phys.* **B77** (1989) 53.
9. A. P. MALOZOEMOFF, in "High temperature superconducting compounds", Vol. II, edited by S. H. WHANG, A. DAS GUPTA and R. LAIBOWITZ (TMS publications, Warrendale, PA, 1990) p. 3.
10. R. GOPALAN, T. ROY, T. RAJASEKHARAN, G. RANGARAJAN and N. HARIBABU, *Physica C* **244** (1995) 106.
11. R. GOPALAN, A. K. SINGH, T. RAJASEKHARAN, G. RANGARAJAN and U. V. VARADARAJU, *J. Mater. Sci. Lett*. **14** (1995) 1043.

Received 14 June
and accepted 13 November 1996