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Microstructure, texture and superconducting properties of Bi2212 ceramics, deformed by torsion under pressure

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Abstract

A systematic investigation of the effect of high-temperature deformation by torsion under quasi-hydrostatic pressure on the microstructure, texture and superconducting properties of Bi2212 ceramics was carried out. Intercolony sliding was identified as the main mechanism of plastic deformation and basal texture formation. In all investigated deformation regimes the colony thickness did not change, only their length varied. The superconducting properties were analyzed as caused by the action of three main pinning centers: intracolonial lattice defects, low-angle colony boundaries and particles of secondary phases which appear during the decomposition of the Bi2212 phase near the melting point.

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Keywords: Bi2212 ceramics; Plastic deformation; Microstructure; Pinning

1. Introduction

Superconducting $Bi_2Sr_2CaCu_2O_{8+x}$ (Bi2212) ceramics is an advanced material concerning its practical application. It is characterized by a rather high current carrying capacity in strong magnetic fields at 4.2 K ($J_c > 10^5$ A/cm^2, 10 T) [1]. However, in spite of its rather high transition temperature ($T_c \sim 90$ K), the critical current density of Bi2212 rapidly decreases at temperatures above 20 K [2]. There are mainly two facts responsible for this decrease: the large anisotropy of the material [3–6] and the low density of defects [7].

One of the methods for improving the density of defects in Bi2212 is hot plastic deformation. It provides stronger texture and increased density of dislocations which, as known, may act as strong pinning centers [8–11]. Usually deformation methods as hot isostatic pressing (HIP) [11], HIP plus sinter-forging [12], or only sinter-forging [13,14] work with deformation temperatures ranging from 815 to 850 °C. However, since simple procedures of deformation based on uniaxial compression cannot provide high strain values, it is rather difficult to form strong texture together with a high density of dislocations.

The use of complex loading procedures of deformation is more advanced for increasing strain values. One of such procedures is torsion under pressure [15] where quasi-hydrostatic pressure prevents destruction of the material and torsion leads to deformation up to very high strains [16,17]. Recently it has been revealed that applying low quasi-hydrostatic pressure (slightly above 0.6 MPa)

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increases the onset temperature for melting of Bi2212 by almost 60 °C, which essentially expands the useable temperature range for hot deformation [18]. An open question is the influence of the deformation performed near the melting temperature of the Bi2212 phase on structure and on superconducting properties. Thus, the aim of the present paper was to investigate the influence of torsion under pressure on microstructure, basal plane texture, phase composition and superconducting properties of Bi2212 ceramics in a wide range of deformation temperature T_d (795–940 °C).

2. Experimental

Commercial $Bi_{1.98}Sr_{1.88}Ca_{1.03}Cu_{2.00}O_{8+x}$ powder (Hoechst) was pressed at 200 MPa to pellets of 10 mm diameter and about 2 mm height and sintered in air at 855 °C for 24 h. The prepared samples were plastically deformed in the temperature range 795–940 °C by compression between two anvils superimposed by torsion which was performed by rotation of one of the two anvils around the compression direction (Inset Fig. 1). To prevent an interaction with the anvils the ceramics were placed between two plates of monocrystalline (1 0 0)-MgO with dimensions of about 15×15 mm^2. The pressure was initiated via applying a constant load from 50 to 450 kg at 795 °C (Fig. 1). After reaching the desired deformation temperature at a heating rate of 10 °C/min the samples were annealed for 30 min under load to allow equilibration of temperature in the sample, followed by torsion under pressure. The twist rate was 1.5×10^{-3}, 3×10^{-3} or 7.7×10^{-3} rpm (Table 1). The twist angle (α) varied from 45° to 180°. After turning the upper anvil in respect to the lower one by the angle α the torsion procedure was ceased, and the furnace was cooled down to room temperature at a rate of 5 °C/min. At 795 °C the pressure was released (Fig. 1). Afterwards the samples were annealed in air at 850 °C for 96 h and cooled by air quenching. Half of all samples were deformed under the following conditions (150 kg load, twist rate 1.5×10^{-3} rpm and $\alpha = 90°$) which we hereafter designate as "standard conditions".

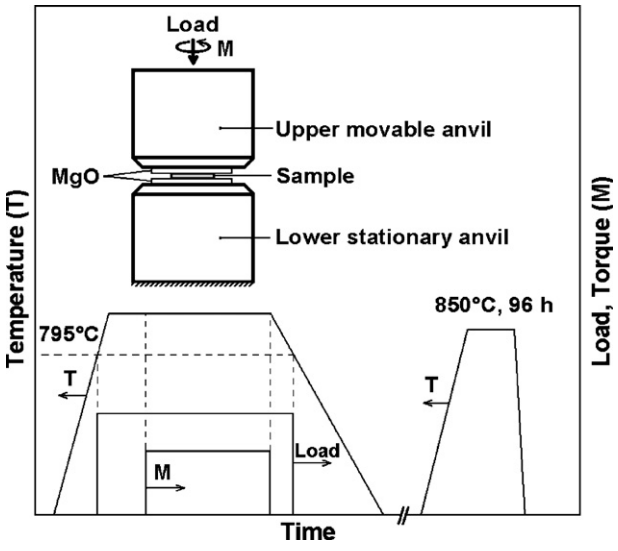

Fig. 1. Procedure of the deformation by torsion under pressure and processing thermal cycle as well as diagram of load and torque application.

X-ray diffraction (XRD) data were collected from the broad faces of the samples using a DRON-3.0 diffractometer with filtered Cu-K_α radiation within the 2θ range 20°–60°. For this purpose one of the MgO plates was removed by abrasive paper and diamond paste. The degree of basal plane texture was determined by analysis of XRD data using the Lotgering method [19]. The Lotgering factor $F = (P - P_o)/(1 - P_o)$ was calculated from the (1 1 5) and (0 0 10) peaks, with $P = I_{(00\,10)}/[I_{(00\,10)} + I_{(1\,1\,5)}]$ determined from measurements of the textured and $P_o = I_{o(00\,10)}/[I_{o(00\,10)} + I_{o(1\,1\,5)}]$ from measurements of the powder. For the Bi2212 powder used in this study P_o is 0.18. The error in determining the F factor didn't exceed 0.01. Moreover the rocking curve of the (0 0 10) peak was measured in a ω-scan mode for a number of deformed samples. Rocking curves were recorded by step scans with step size 0.5° (θ) and counting time of 5 s.

Microstructure and chemical composition of the phases were studied using two JEOL scanning electron microscopes JSM-840 and JXA-6400. The latter one equipped with a wavelength dispersive spectrometer (WDS). The investigation of the microstructure was performed both on samples polished by diamond paste and on samples electrochemically etched by 5% solution of perchloric acid in butylalcohol at a voltage of 0.1 V.

Quantitative analysis of microstructure was carried out on the cross section of the samples after cutting them along a diameter. The mean size of colonies of grains of Bi2212 phase was calculated by measurement of length (L) and thickness (H) of individual colonies (Fig. 2). Further, the mean number of grains per colony was measured. The number of colonies taken into account was approximately 300. Glagolev's point count method [20] was used to determine the total volume fraction of Cu- and Bi-free phases (V_{ns}).

A vibrating sample magnetometer (PAR 150A) was used for measuring superconducting properties at 4.2 K and 30 K in fields up to 7 T parallel to the direction of compression. Bars with dimensions $4 \times 1 \times h$ mm^3, with thickness h varying between 1.0 and 0.2 mm according to the different deformation conditions used, were cut from the central portion of the samples, the largest (4 mm) and smallest (h) sides being along the radius and the compression axis, respectively. From hysteresis loops critical current densities J_c were calculated within Bean's critical state model [21], assuming that the characteristic length of current flow is either the colony or the overall sample size. In addition, the time dependence of the magnetic moment was measured at 1 T and both 4.2 K as well as 30 K for about 30 min. From these relaxation measurements mean effective activation energies were determined within the Anderson–Kim model of flux creep [22].