

Critical Current Density and Microstructure of NdBa₂Cu₃O₇ Single Crystal

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The top-seeded solution growth method was used to grow NdBCOss single crystals in air. The microstructure of the samples has shown that the oxygenation crack structure developed in the single crystals. The intrinsic critical current density was estimated to be at least twice higher than the value of volume critical current density determined from the magnetization measurements.

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1. Introduction

Single-crystals of REBa₂Cu₃O_{7- δ} (in short RE123, RE = Y or rare-earth element) high temperature superconductors were frequently measured to elucidate different physical and technical parameters of these materials [1]. As-grown crystals have lack of oxygen and they have to be oxygenated. The oxygenation is usually done in flowing oxygen at temperatures 300–500 °C. It has been shown that in the case of RE123 single-grain bulk composite superconductors prepared by top-seeded melt-growth (TSMG) process, with trapped RE₂BaCuO₅ particles, cracks are formed during oxygenation process [2]. In the case of RE123 single-crystals, prepared by flux or pulling techniques, it is always supposed that they are free of cracks.

In this letter we present results on Nd123 single-crystalline sample separated from the crystal, prepared by crystal pulling and oxygenated, showing intensive cracking.

2. Experimental details

The top-seeded solution growth (TSSG) method was used to grow NdBCOss single crystals in air [3]. As-grown NdBCO crystals were cut into sizes of about $1.5 \times 1.5 \text{ mm}^2$ in the a/b -plane and 1 mm in the c -direction and annealed in a tube furnace at 340 °C for 200 h in the oxygen gas flow. Magnetization measurements were done at a temperature of 77 K by a vibrating sample magnetometer with magnetic fields up to 5 T applied parallel to the c -axis of the crystal. The critical current densities,

J_c , were calculated from magnetization hysteresis loops (MHL) using the extended Bean model [4] for rectangular samples, $J_c = \frac{2\Delta M}{a^2(b-a/3)c}$, where ΔM is the difference of the magnetic moments between the increasing and decreasing field branches of the MHL and a , b and c are the sample sizes. The transition temperature, T_c , was determined from the magnetic transition curve taken after zero-field cooling as the onset of this curve in an applied external magnetic field of 2 mT. Microstructure of the crystal was analysed by optical microscopy in normal and polarised light using image processing software after polishing and etching by the 0.5 wt% HCl in ethyl alcohol.

3. Results and discussion

3.1. T_c and J_c

Field dependence of critical current density at 77 K presented in Fig. 1a exhibits a peak effect with the maximum at intermediate external magnetic fields, which is typical for the Nd123 single-crystals with Nd/Ba substituted regions as pinning centres [5]. Transition temperature $T_{\text{onset}} = 94 \text{ K}$ which is also the value typical for Nd123 single-crystals (Fig. 1b).

3.2. Microstructure

Microstructural observation revealed that after oxygenation the sample is full of so-called oxygenation cracks. The polished and etched crystal surface parallel to the $\{100\}$ plane exhibits well pronounced traces of thicker cracks parallel to $\{001\}$ plane with their length nearly identical with the size of the crystal (Fig. 2a). The traces of the $\{110\}$ type twins, which are perpendicular to the $\{001\}$ plane, determine crystal orientation unambiguously. Much thinner cracks mostly perpendicular to the

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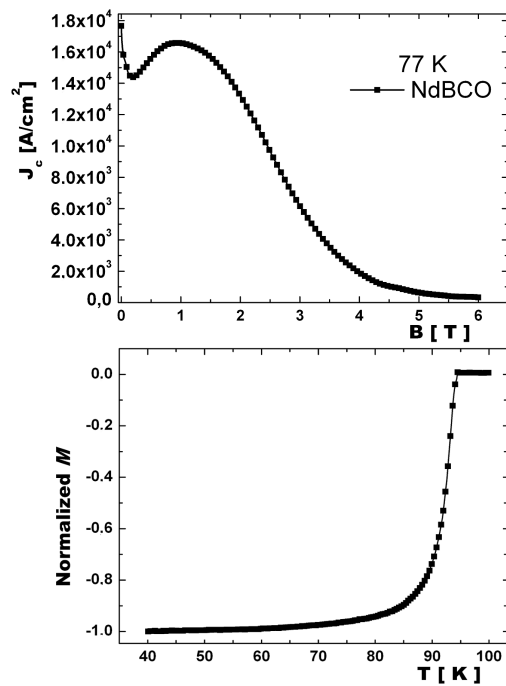


Fig. 1. Field dependence of critical current density at 77 K (a). The magnetic transition curve taken after zero-field cooling in an applied external magnetic field of 2 mT (b).

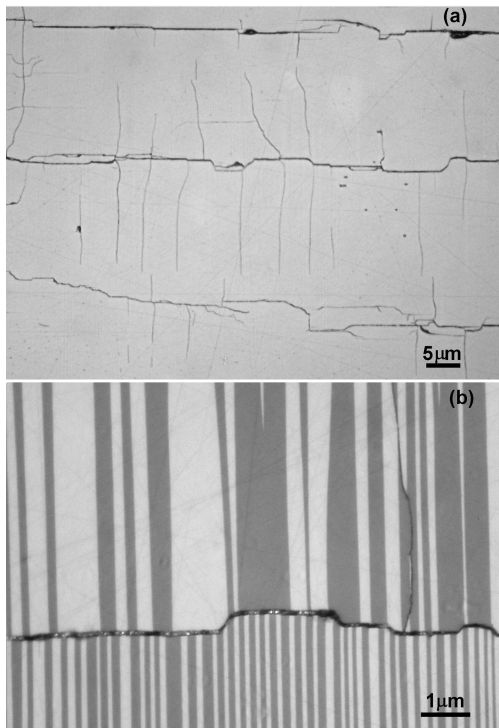


Fig. 2. Thicker crack traces along $\{001\}$ plane and thinner crack traces along the (001) direction seen after polishing and etching on the surface of the Nd123 crystal parallel to the $\{100\}$ plane (a). Discontinuity in twin pattern along the crack traces (b).

(001) direction starts from the surface of thicker cracks. Detailed view in polarised light, showing the discontinuity in twin pattern across the crack (Fig. 2b), confirms that the crack does not develop during sample grinding and polishing.

Cracks were observed also on the polished surface of the crystal parallel to the (001) crystal plane (Fig. 3a). Here, the discontinuities in the twin structure, located inside the twin complexes, are caused by cracks, which were seen as the traces perpendicular to the (001) plane on the (100) sample surface. The orientation of observed cracks is more or less random with some preferences of the $\{100\}$ or $\{110\}$ planes (Fig. 4). Location of the cracks along the twin complex boundaries was also detected (Fig. 3b).

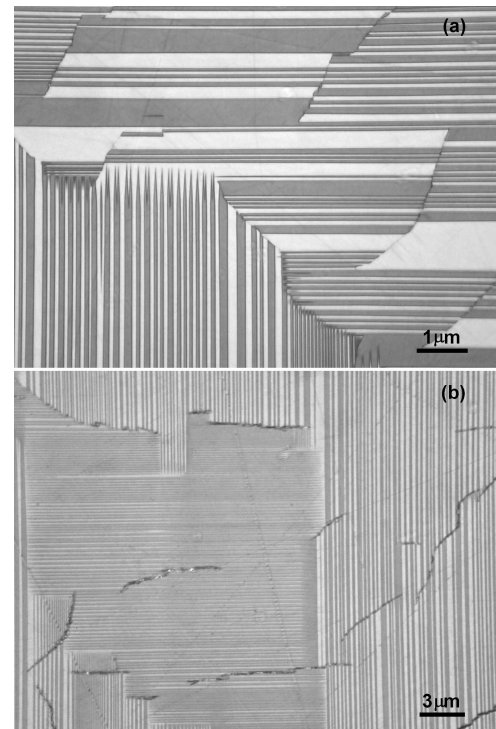


Fig. 3. Discontinuities in twin pattern along the crack traces seen after polishing on the surface of the Nd123 crystal parallel to the $\{001\}$ plane (a). Randomly oriented crack traces sometimes located at the twin complex boundaries (b).

The reason for cracking during oxygenation of tetragonal N123 crystal is that the lattice parameters are shorter with higher oxygen content. Consequently the surface oxygenated layer is under tension stress causing cracking [2]. These cracks in fact allow oxygenation of the sample in a reasonable short time as oxygen can flow to the sample along created cracks [6]. As the cracks, which are perpendicular to the $\{001\}$ plane reduce the effective sample cross-section, the critical current density estimated from magnetization measurements using the sample dimensions is only apparent. According to [7], the reduction of intrinsic critical current density, J_{c0} , for super-current flowing along the $\{001\}$ plane is propor-

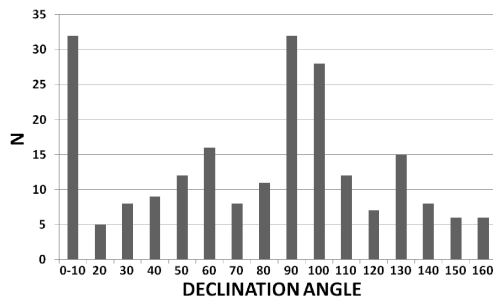


Fig. 4. Frequency N of crack traces declination from $\langle 100 \rangle$ direction seen on the crystal surface parallel to the $\{001\}$ plane.

tional to the l/d parameter,

$$J_c = J_{c0} [1 - 0.93(l/d)^{1/2}], \quad (1)$$

where l is the mean crack length and d is the crack spacing of the cracks perpendicular to the $\{001\}$ plane. In our case the projection of the crack length to the $\{100\}$ direction is the effective length of the cracks. Measurement of these parameters on the micrographs from the etched $\{001\}$ surface estimated mean values: $l = 5.2 \mu\text{m}$ and $d = 9.8 \mu\text{m}$. Then, $l/d = 0.53$ which is the value close to what was measured in TSMG YBCO bulk superconductor [8]. This value points out that intrinsic value of critical current density should be three times higher than the volume critical current density determined estimated from magnetisation measurements.

4. Conclusions

The microstructure analyses of the NdBCOss single crystals have shown that the oxygenation crack struc-

ture developed in the single crystals. The intrinsic critical current density was estimated to be at least twice higher than the value of volume critical current density determined from the magnetization measurements.

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