Effect of TiO$_2$ Fibers on Properties of Single-Grain Bulk GdBCO Superconductors

P. Hajdová$^{a,*}$, I. Shepa$^b$, E. Mudra$^b$, M. Rajnak$^{a,c}$, J. Dusza$^b$ and P. Diko$^a$

$^a$Institute of Experimental Physics, Slovak Academy of Sciences, Watsonova 47, 040 01 Kosice, Slovakia
$^b$Institute of Materials Research, Slovak Academy of Sciences, Watsonova 47, 040 01 Kosice, Slovakia
$^c$Faculty of Electrical Engineering and Informatics, Technical University of Kosice, Letna 9, 042 00 Kosice, Slovakia

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1. Introduction

The single-grain bulk GdBa$_2$Cu$_3$O$_{7−δ}$ (GdBCO) superconductor is one of the promising high-temperature superconductor materials with significant potential for development in practical applications, such as fly wheel energy storage systems, or magnetic bearings [1–3]. For these applications high critical current density $J_c$ is the most important requirement. It is well known that size and distribution of GdBa$_2$Cu$_3$O$_7$ (Gd211) particles, and addition of non-superconducting artificial pinning centres are essential to enhance flux pinning properties ($J_c$ and trapped magnetic field). An effective way to enhance mentioned properties is introduction of elongated particles with a large surface area. Therefore, various kinds of metal oxides have been introduced already into the superconducting matrix as the second phase particles [4–6].

In this paper, we studied influence addition of ceramic TiO$_2$ fibres on superconducting properties (critical temperature $T_c$ and critical current density $J_c$) of GdBCO bulk superconductor. We were inspired by these works, in which the influence of Ti-based addition on superconducting properties of REBa$_2$Cu$_3$O$_{7−δ}$ (RE denotes a rare earth elements) superconductors has been investigated [7–10].

2. Materials and experimental methods

For the production of pure GdBCO and TiO$_2$ doped GdBCO samples, we used commercially available powders. Nominal composition of mixture powder was:

1 mol. GdBa$_2$Cu$_3$O$_{7−δ}$ (SOLVAY, purity 99.9%, average particle size 30 µm) + $\frac{1}{2}$ mol. Gd$_2$BaCuO$_5$ (TOSHIMA, purity 99.9%, average particle size 1–2 µm) + 20 wt% Ag$_2$O (CHEMPUR, purity 99%) + 0.2 wt% PtO$_2$·H$_2$O (ACROS, purity 79–84%). Additions of Ag$_2$O and Pt were aimed to improve the mechanical properties, and fine dispersion of the Gd211 secondary-phase particles into the Gd123 matrix, respectively. After thorough mixing and milling of powders, the rearranged TiO$_2$ fibres of quantity 0.05 wt% were added to one mixture powder, and mixed with an acoustic mixer to obtain uniform doping effect in a full sample volume. The ceramic fibres [11] with average diameter of 0.49 µm after the calcinations were prepared using the needle-less electrospinning method. Prepared mixture powders together with the thin film NdBa$_2$Cu$_3$O$_{7−δ}$ seeds were uniaxially pressed into the 20 g cylindrical pellets of 20 mm in diameter.

The growth of samples was performed in the air atmosphere using a top-seeded melt growth (TSMG) method. The applied heating profile was as follows: the pellets were heated from room temperature to melting temperature of 1060°C at heating rate of 100°C/h, held for 1 h to ensure decomposition of the mixture powders, under-cooled to 1020°C by rate of 100°C/h, and then slowly cooled down to 980°C at cooling rate of 0.3°C/h. Subsequently, the furnace was cooled to room temperature.

The as-grown samples were cut in halves, and then one half was prepared for measurement of magnetic properties ($T_c$ and $J_c$) at liquid nitrogen temperature. Small specimens with size $\approx 1.5$ mm×1.5 mm×0.5 mm were carefully cut from the equivalent locations of three planes, as shown schematically in Fig. 1. Cut specimens were oxygenated in a flowing oxygen atmosphere at temperature of 410°C for 240 h, to drive the non-superconducting tetragonal Gd123 phase to the desired superconducting orthorhombic phase.
The magnetic measurements of oxygen-annealed specimens were conducted in a vibrating sample magnetometer from Cryogenic Limited. The temperature dependent of magnetization were measured in zero field cooled regime at external constant magnetic field 2 mT which was applied parallel to the c-axis of each specimen. The middle critical transition temperatures $T_{c,m}$ were determined as the $T_c(50\%)$ of the magnetic transition curves. In turn, $J_c$ were calculated from the magnetic hysteresis loops at 77 K using the extended Bean model [12].

3. Results and discussion

Transition curves for pure single-grain GdBCO sample (Fig. 2) and for GdBCO sample with the addition of 0.05 wt% TiO$_2$ microfibres (Fig. 3) show that the addition of microfibers caused small decrease of transition temperature $T_c$ to superconducting state. This effect is caused by some change in the charge carrier density, similarly as it was demonstrated with DyBa$_2$Cu$_3$O$_{7-\delta}$ [13]. The charge carrier density can be changed either by differences in substitution of Ba$^{2+}$ ions with Gd$^{3+}$ ions in the Gd123 crystal lattice, or by substitution of Ti$^{4+}$ ions in the Gd123 crystal lattice. Ti can be accommodated at Cu sites due to the similar ionic radius with Cu [13]. The substitution of Gd/Ba ions depends on Ba activity (concentration) during the growth of Gd123 crystal [14].

From thermal and X-ray analyses we know that added TiO$_3$ fibres react with a part of Gd123, according to reaction:

$$2GdBa_2Cu_3O_{6.5} + 3TiO_2 + 2O_2 = 3BaTiO_3 + Gd_2BaCuO_5 + 5CuO.$$  (1)

At the peritectic reaction Gd123 phase decomposes, according to reaction:

$$2GdBa_2Cu_3O_{6.5} = Gd_2BaCuO_5 + L(3BaO \cdot 5CuO).$$  (2)

Melt L will have deficit of barium due to formation of BaTiO$_3$ phase. This will result in higher substitution of Ba$^{2+}$ ions by Gd$^{3+}$ ions in Gd123 crystal lattice, and consequently, in lower charge carrier density and lower $T_c$. Some substitution of Ti$^{4+}$ ions in to Gd123 crystal lattice has not been reported so far, but we cannot exclude it.

The field dependencies of critical current density at 77 K calculated from magnetization measurements are presented in Fig. 4 and 5. The peak effect is observed for all specimens. The most significant effect is decrease...
Fig. 5. Critical current density for the GdBCO+TiO$_2$ specimens at 77 K.

of $J_c$ at zero field for the sample with TiO$_2$ fibre addition. At the zero field, however, it is expected that $J_c$ is mostly influenced by the size and volume fraction of Gd211 non-superconducting particles. Therefore, we can suppose that some coarsening of Gd211 particles was caused by presence of Ti$^{4+}$ ions in the partially melted system. Moreover, this effect was not compensated by TiO$_2$ fibres transformed to BaTiO$_3$ phase.

The addition of TiO$_2$ fibres leads to more pronounced secondary peak in the field dependence of $J_c$ and to lower irreversibility field (Fig. 5). Both these effects are influenced by pinning centres formed by substitutions in Gd123 crystal lattice [14]. Lower peak at the higher distance from the beginning of solidification is caused by lower concentration of chemical pinning centres what suggest lower Gd/Ba substitution in the Gd123 lattice.

4. Conclusions

The single-grain GdBCO bulk sample with TiO$_2$ fibres was successfully prepared by the top-seeded melt growth method in air. The experimentally obtained results showed the following:

1. Formation of BaTiO$_3$ phase leads to deficit of barium in the melt during Gd123 crystal growth and to higher substitution of Ba$^{2+}$ ions by Gd$^{3+}$ ions in Gd123 crystal lattice. As a consequence, the lower charge carrier density and lower transition temperature to superconducting state appear.

2. More pronounced secondary peak effect in the field dependence of critical current density in the sample with the addition of TiO$_2$ fibres can be explained by higher concentration of chemical pinning centres. These latter inform of Gd/Ba substitutions due to lower activity of barium in the melt.

3. The lower secondary peaks closer to the seed may be cause by a decrease of Ba concentration in the melt with the distance from the seed.

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