Synthesis, Microstructure, and the Transport and Magnetic Properties of Bi-Containing High-Temperature Superconductors with a Porous Structure

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Abstract—Preliminary data on the synthesis and physical properties of polycrystalline $Bi_{1.8}Pb_{0.3}Sr_2Ca_2Cu_3O_x$ high-temperature superconductors of low density with a foam-like microstructure are reported. © 2003 MAIK "Nauka/Interperiodica".

Recently, Reddy and Schmitz [1] reported on the synthesis of a superconducting foam made of $YBa_2Cu_3O_7$ high-temperature superconductor (HTSC). Such a foam may be an almost ideal material for active elements of fault current limiters, since critical currents in the foam immersed in liquid nitrogen can significantly exceed those in bulk HTSCs. This is explained by the fact that the foam contains open pores which are readily filled by a coolant (liquid nitrogen) that provides effective heat removal from the entire volume of a material. In addition, a superconducting foam can exhibit enhanced pinning due to a fractal structure [2], which also increases the critical current and the levitation force.

We have synthesized a low-density HTSC ceramics with a composition $Bi_{1.8}Pb_{0.3}Sr_2Ca_2Cu_3O_x$. The method of synthesis of this bismuth-containing ceramics was similar to that described in [3]. However, we modified the final stage of annealing, so that the growth of HTSC crystallites occurred predominantly in the *ab* plane. Because of a random orientation of grains in a polycrystal, such a growth leads to an increase in the material volume. In addition, calcium carbonate was completely decomposed during the final annealing stage. The excess pressure of carbon dioxide also favors an increase in the material volume. As a result, the density of the material was 0.38 of the theoretical value for Bi_{1.8}Pb_{0.3}Sr₂Ca₂Cu₃O_x. Figure 1 shows an image of the structure of a material under consideration, obtained by the method of scanning electron microscopy (SEM). The SEM measurements were performed on a REM-100U scanning electron microscope operating at an accelerating voltage of 30 kV and a $\times 1000$ magnification. The samples were coated by a layer of aluminum with a thickness of several nanometers, since otherwise the charge of the electron beam was accumulated on the specimen surface in the course of SEM measurements. The Al layer was formed by vacuum deposition. By comparing the images obtained from the Al-coated and uncoated samples, it was shown that the coating had no



Fig. 1. SEM image of a $Bi_{1.8}Pb_{0.3}Sr_2Ca_2Cu_3O_x$ sample with a foam-like microstructure.

effect on the material structure. As can be seen from Fig. 1, the material possesses a porous structure. The size and morphology of pores do not significantly vary within the image field, the pore size changing from a few μ m to several tens μ m. The image exhibits scale-invariant elements which are arranged regularly and rather symmetrically. This conclusion is confirmed by an analysis of the Fourier transform performed for a large number of micrographs. The specific surface area of the samples measured using the thermal desorption of argon and calculated using the Brunauer–Emmet–Teller equation was 6.5 m²/g.

Figure 2 shows the temperature dependence of electric resistivity $\rho(T)$ of a sample measured in a temperature range of 77–300 K. The superconducting transition temperature ("R = 0") is 107 K. Extrapolation of $\rho(T)$ from high temperatures to T = 0 gives a residual resistance of $\rho \approx 0$ that indicates that the effect of grain boundaries is negligible. Such a behavior of $\rho(T)$ is characteristic of single crystals [4]. This result is rather surprising, since the material represents a low-density polycrystal. The absolute value of the electric resistivity $\rho(T)$ has proved to be higher by one order of magnitude as compared to the values available in the literature for bismuth-containing HTSC single crystals [4]. However, we did not perform a correction of ρ with allowance for the actual cross section of our porous material. In our opinion, there is no sense in doing this unless the problem of fractal dimensionality of our samples is solved.

The temperature dependences of magnetization M(T) of HTSC Bi_{1.8}Pb_{0.3}Sr₂Ca₂Cu₃O_x samples with a foam microstructure, which were measured in zero-field-cooling (ZFC) and field-cooling (FC) regimes (at a magnetic field H = 13 Oe) are presented in Fig. 3 (circles) in comparison to the temperature dependences M(T) of a powder-like bismuth-containing compound prepared from the original foam (solid lines). The comparison shows that the diamagnetic response and a difference between the ZFC and FC procedures are greater in the case of the sample with a foam-like structure. This confirms a significant enhancement of pinning in the porous quasi-low-dimensional material.

The levitation force (which depends directly on the critical current and pinning force) of а $Bi_{1,8}Pb_{0,3}Sr_2Ca_2Cu_3O_x$ HTSC sample with a foam-like structure was measured under the following conditions. A permanent Nd–Fe–B magnet (diameter d = 9 mm, height h = 6 mm, and mass m = 2.97 g) was mounted above surface of a pellet made the of $Bi_{1,8}Pb_{0,3}Sr_{2}Ca_{2}Cu_{3}O_{r}$ HTSC (diameter D = 22 mm andheight H = 6 mm). As a reference sample, we used a YBa₂Cu₃O₇ pellet of high density (0.93 of the theoretical value) with the same dimensions, which was sintered by the standard ceramic technology. The measurements were performed at the liquid-nitrogen temperature. Repulsive force between the HTSC sample



Fig. 2. The temperature dependence of electric resistivity $\rho(T)$ of a Bi_{1.8}Pb_{0.3}Sr₂Ca₂Cu₃O_x HTSC sample with a foam-like microstructure.



Fig. 3. The temperature dependences of magnetization M(T) of a Bi_{1.8}Pb_{0.3}Sr₂Ca₂Cu₃O_x HTSC sample with a foam-like microstructure and a powder-like Bi_{1.8}Pb_{0.3}Sr₂Ca₂Cu₃O_x HTSC sample (filled circles and solid lines, respectively).

and the magnet, measured as a function of the superconductor-magnet distance, is shown in Fig. 4. It can be seen that, when interacting with the permanent magnet, the bismuth-containing superconductor with the foam-like microstructure (Fig. 4a) and the yttrium-containing ceramics (Fig. 4b) have practically the same levitation force, although it is known that the maximum levitation force is achieved in single-grain and singlecrystal HTSC samples of 1–2–3 superconductors [5]. Thus, despite a relatively low critical current density in



Fig. 4. Levitation force as a function of the distance between the Nd–Fe–B magnet and (a) a $Bi_{1.8}Pb_{0.3}Sr_2Ca_2Cu_3O_x$ HTSC pellet with a foam-like microstructure and (b) a YBa₂Cu₃O₇ HTSC sample of high density.

our HTSCs with the foam-like structure at the liquid nitrogen temperature (\sim 50 A/cm²) and a low "filling factor," the levitation force turns out to be sufficiently high due to strong pinning.

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