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Methods for Producing High-Temperature Superconductors

U. T. Kurbanov
Institute of Nuclear Physics AS of Uzbekistan, Head of the Laboratory of Physics Nanostructured and Superconducting Materials

N. A. Turgunov, K. M. Fayzullaev
Institute of Semiconductor Physics and Microelectronics at the National University of Uzbekistan

R. A. Gulamov
Academic Lyceum "Dustlik" of Namangan Engineering Institute, Uzbekistan

ABSTRACT

This paper presents technologies for obtaining high-temperature superconducting materials.

KEYWORDS: *Superconductivity, gas-phase reaction, evaporation, magnetron sputtering, target, substrate, deposition, plasma, plasma spray.*

The discovery in 1986 by G. Bednorz and A. Müller of high-temperature superconductivity (HTSC) in cuprates, which have a superconducting transition temperature exceeding the boiling point of liquid nitrogen (77 K), made a real revolution in the production of oxide structures, taking into account the entire technological chain "powder - material - product - device. Such a subtle effect as superconductivity, which has an ultra-high sensitivity in brittle oxides with a rapidly degrading structure under the influence of the slightest external and internal influences, required a radical modification of existing technologies and the development of fundamentally new numerous methods for producing HTSCs. This, in particular, is due to the fact that some basic properties of HTSCs (for example, the superconducting coherence length) appear on the nanometer scale, and the corresponding structural features directly determine the quality and characteristics of the finished product [1].

When obtaining HTSC samples with highly efficient parameters, the preparation of high-quality precursor powders is of great importance. Among the methods for obtaining such YBa2Cu3O7-x (hereinafter YBCO) powders, we will mention the following: standard solid-phase reaction and chemical deposition, plasma spray, drying in liquid nitrogen, spray drying and oxidative synthesis, sol-gel method, acetate method and gas-phase reaction. The standard procedure for obtaining superconducting ceramic powders includes several steps. First, the raw materials are mixed in a specific molar ratio using an appropriate "mix-grind" or liquid-phase mixing process. In this case, the homogeneity of the mixture is limited by the size of the particles, and the best results are achieved for particles with sizes smaller than 1 μm. The next step is drying or removing the solvent, which is necessary to maintain the chemical homogeneity achieved during the mixing process.

For multi-component (HTSC) systems, the removal of the solvent by slow evaporation can lead to a very inhomogeneous deposit due to the different solubilities of the components. After drying, the powders are calcined in a controlled atmosphere to achieve the final structural and phase composition. The reaction regime for the YBCO system is determined by process parameters such as calcination temperature and time, heating rate, atmosphere (oxygen partial pressure) and initial phases. Powders can also be directly synthesized from a solution using pyrolysis technology, or obtained by electrodeposition by passing current through a solution. In this case, even small fluctuations of the composition can lead to the formation of normal (non-superconducting) phases, such as: Y2BaCuO5, CuO and BaCuO2. The use of carbon-containing precursors also complicates the formation of the YBa2Cu3O7-x phase and leads to a decrease in the superconducting properties. In turn, the powder for obtaining superconducting films of composition Bi(Pb)-Sr-Ca-Cu-O (hereinafter referred to as BSCCO) can be produced using a solid-state reaction, co-precipitation, aerosol-spray pyrolysis, firing technology, freeze-drying, the method liquid mixing, microemulsion or sol-gel method. The standard approaches for obtaining superconducting precursor powders used in the manufacture of BSCCO tapes and wires are the so-called “one-powder” and “two-powder” synthesis methods. In the first case, the precursor is obtained as a result of the calcination of a mixture of oxides and carbonates. In the second, a mixture of two cuprate compounds is fired [2].

Recently, intensive studies have been introduced to study the properties of multiphase bismuth cuprates synthesized by melt technology under the action of solar energy. The melt technology makes it possible to obtain amorphous and glass-ceramic precursors for the subsequent synthesis of textured impurity phases with characteristic low-angle grain boundaries and controlled distribution. One of the important factors in melting solar technology that affects the formation of textures of crystal nuclei and superconducting phases based on them is a large temperature gradient, which is difficult to achieve with conventional thermal equipment [3]. Smooth technologies and methods for producing composite materials are especially promising for obtaining high-temperature ceramic superconductors suitable for practical use.

Methods of vacuum co-evaporation imply simultaneous or sequential (layer by layer) co-deposition of HTS components evaporated from various sources using, for example, electron beam guns or resistive evaporators. Films obtained by this technology are inferior in their superconducting properties to samples produced by laser or magnetron sputtering. Vacuum co-evaporation methods are used in two-stage synthesis, when the structure of the films deposited at the first stage and the oxygen content in them are of no fundamental importance.

Laser evaporation is highly efficient in the deposition of HTSC films. This method is easy to implement, has a high deposition rate, and allows you to work with small targets. Its main advantage is equally good evaporation of all chemical elements contained in the target. By evaporating targets under certain conditions, one can obtain films of the same composition as the targets themselves. Important technological parameters are: the distance from the target to the substrate, as well as the oxygen pressure. Their correct choice makes it possible, on the one hand, to prevent overheating of the growing film by the laser-evaporated plasma energy and the corresponding formation of too large grains, and, on the other hand, to establish the energy regime necessary for film growth at the lowest possible substrate temperatures. The high energy of the deposited components and the presence of atomic and ionized oxygen in the laser plume make it possible to fabricate HTSC films in one stage [4]. In this case, single-crystal or highly textured films with the c-axis orientation are obtained (the c-axis is perpendicular to the plane of the substrate). The main disadvantages of laser evaporation are: (a) the small size of the area in which it is possible to deposit stoichiometric films; (b) inhomogeneity of their thickness; and (c) surface roughness. Due to the strong anisotropy of HTSCs, only films with a c-axis orientation have good transport and screening properties. At the same time, films with a-axis orientation (the a axis is located in the ab plane of the substrate), which have a long coherence length in the direction perpendicular to the surface and are highly smooth, are convenient for fabricating high-quality HTSC Josephson junctions consisting of successively deposited layers. HTSC - normal metal" (or "dielectric - HTSC"). Films with mixed orientation are undesirable in all respects.

Magnetron scattering makes it possible to obtain YBCO films in one stage, which are not inferior in their superconducting properties to samples grown by laser evaporation. At the same time, they have a more uniform thickness and a higher surface smoothness. As in the case of laser evaporation, the formation of plasma during magnetron sputtering generates high-energy atoms and ions, which allow one-stage production of HTSC films at low temperatures. The target-substrate distance is also important here. When the target is close to the substrate and the medium pressure is insufficient, the substrate is subjected to intense bombardment by negative oxygen ions, which destroy the structure of the growing film and its stoichiometry. To solve this problem, a number of approaches are used, including protection of the substrate from bombardment by high-energy ions and its location at an optimal distance from the gas-discharge plasma to ensure a high deposition rate and successful film growth at the lowest possible temperatures. The in situ YBCO thin films, which were fabricated by off-axis magnetron sputtering and had optimal electrical properties, have already demonstrated the superconducting transition temperature and critical current density, respectively: Tc = 92 K and Jc = 7×106 A/cm2. Varieties of pulsed laser deposition used to produce YBCO films and wires with a high texture, fabricated on various single- and polycrystalline substrates with and without sublayers, make it possible to achieve a critical current density of Jc = 2.4×106 A/cm2 at a temperature of 77 K and zero magnetic field.

The essence of the method of chemical precipitation from the vaporous phase of metal-organic combinations is the transport of metal components in the form of vapors of volatile metal-organic compounds into the reactor, mixing with a gaseous oxidizer, vapor decomposition, and condensation of the oxide film on the substrate. This method makes it possible to obtain thin HTSC films comparable in their characteristics to samples prepared by physical deposition methods. The comparative advantages of this method over the latter include: (a) the possibility of depositing homogeneous films on non-planar parts and large areas; (b) higher deposition rates while maintaining high quality; (c) the flexibility of the process at the stage of debugging the technological mode, due to the smooth change in the composition of the vapor phase [5].

The main goal in terms of material strength is the selection of adjacent layers with similar temperature and crystallographic properties, as well as the prevention of a chemical reaction between them. It should be noted that the critical current density of films on a polycrystalline substrate decreases significantly in the presence of high-angle intercrystallite boundaries, which lead to the problem of weak bonds. Therefore, for tape applications, it is very desirable to grow films with a highly ordered crystal structure in the plane, that is, to create an ordered structure not only along the c axis, but also in the direction of the a axis. A common problem of films obtained by liquid-phase epitaxy and electrophoretic deposition, which have high deposition rates and rather low cost, is the formation of cracks due to stress relaxation during sample cooling and differences in the thermal properties of the substrate and film.

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