

Physics

Effective Technology for High Temperature Superconductors Oxygenation

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(Presented by Academy Member G. Tavadze)

ABSTRACT. Good oxygenation of superconductors is a necessary condition in order to achieve a pure high-temperature phase in them. The synthesis under the oxygen pressure can provide homogeneity of multiphase precursor and avoid the carbon wastes in it that is so important for attaining a Hg-based pure high-temperature phase. Oxidation process requires regular delivery of oxygen to the samples of superconductors within a certain temperature range (400-1300K). The temperature is controlled according to the selected algorithm. Present article is dedicated to the effective technology of oxygenation of high-temperature superconductor materials (HTS). Some original solutions for construction of the mobile system of HTS oxygenation OS-1000DMB, the functions of its elements and the technological processes are considered in detail and the results obtained are provided. © 2015 Bull. Georg. Natl. Acad. Sci.

Key words: superconductors, temperature, oxygenation, control system.

In spite of the fact that today there are number of high-temperature superconductor materials, in the nearest future only some of them can be competitive for application in electric-power industry, electronics and some other areas. Therefore, primary goal of our work is to research and develop effective technologies and methods of obtaining superconductors with certain properties, in particular, for production of the high-homogenous powders, thin porous structures, mono-crystals, long ordered composite materials. One of the topical problems in this direction is elaboration of oxygenation technology.

As is known, the sufficiently saturated oxygen concentration is a necessary condition for all the existing high-temperature superconductors in order

to attain a pure high temperature phase in them. For example, the superconductivity in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ is sensitive to the value of oxygen content x . In the materials with $0 < x < 0.65$ the superconductivity T_c is low, while in the materials with $x \sim 0.07$ the superconductivity temperature is the highest $T_c = 93$ K [1,2], for example in $\text{Bi}_2\text{Ca}_2\text{Sr}_2\text{Cu}_3\text{O}_{10+x}$ ($T_c \approx 110$ K) [3], $\text{TlBa}_2\text{Ca}_2\text{Cu}_3\text{O}_{10-x}$ ($T_c \approx 122$ K) [4,5], $\text{HgBa}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+x}$ ($T_c \approx 133$ K) [6,7].

It should be noted that among the above mentioned superconductors, it is especially difficult to prepare the Hg-based superconductors $\text{HgBa}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+2+x}$, where n is equal to 3. Under the regular pressure the highest critical temperature can be achieved ($T_c \approx 135$ K) [8], and in case of high



Fig. 1. General view of the device HTS OS-1000DMB

pressure (30 GPa) it can reach $T_c \approx 160$ K [9]. In low pressure synthesis it is difficult to attain pure state of phase $n=3$, while under high pressure it is possible [10, 11]. However, the pure phase can be attained by means of a regular closed quartz technology if the high-valence dopants are used or a high quality precursor $Ba_2Ca_2Cu_3$ is prepared.

The following two basic requirements must be taken into account to obtain a high quality multiphase precursor: the cation homogeneity and the oxygen content. These requirements can be satisfied if the synthesis proceeds under a certain pressure of oxygen (0.3-1 bar) for a long period of time. The synthesis under the oxygen pressure provides homogeneity of the multiphase precursor and avoids carbon wastes in it that is very important for attaining the Hg-based pure high temperature phase.

Unlike the Y- and Bi-based superconductors, the synthesis of Hg-based superconductor requires "closed" technology. An Au capsule with a sample in it is placed in the quartz tube of evacuated vacuum up to 10^{-3} Tor. In such conditions of synthesis any possibility of the sample oxidation is excluded.

The process of superconducting sample oxygenation presupposes pressure control in the oxygen supply system within the range of stable

temperature of 400-1300K. Temperature is controlled according to the chosen algorithm. In order to achieve an optimal result it is necessary to develop the constructions for special container of superconductor samples, for oxygen supply system, temperature controlling system of the cylindrical open-ended furnace and complex technological devices.

According to the above described requirements, a device for effective oxygenation of high temperature superconductor samples HTS OS-1000DM was developed and tested. Fig.1 shows general view of the device HTS OS-1000DMB.

Main parts of the device are (Fig. 1): high temperature (1273K) cylindrical furnace (1), special container for synthesis of superconductor samples in oxygen environment (2) made from a quartz tube, at the head of which there is a hermetic cover (3) with the closure (4) and the oxygen container and reductor (5) connected to it. On the other side of the heater, there are welded two short tubes for thermocouples (6) and for pressure measuring manometer (7). The manometer tube is a kind of thoriated diaphragm functioning as an aero dynamical cover as well. The container is loaded with the superconductor samples and is placed on an appropriate construction.



Fig. 2. General view of the container loaded with the superconductor samples

Fig. 2 shows general view of the container loaded with the superconductor samples.

The process of oxygenation is controlled by a mobile system of operation and control (MEISSA-1), which is specially elaborated for technological problems. The whole cycle of oxygenation is controlled and operated by the controller CN7200 produced by Omega and by the thermocouple (6) of K-type programmed in accordance with the chosen algorithm.

The oxygenation process is going on as follows: MEISSA-1 provides regulated voltage for the furnace, where the container with superconductor samples is placed and the heating process begins. At the temperature of 800K the oxygen is delivered along with the uniform growth of pressure up to the temperature of 1200K.

Fig.3 (a,b) shows the samples of XRD $Ba_2Ca_2Cu_3O_y$ synthesized: (a) on the air and (b) synthesized under the oxygen pressure. In Fig.3 (a) quite a strong peak of $BaCO_3$ is observed while in Fig.3 (b) only $BaCuO_2$ and Ca_2CuO_3 phases are observed indicating that a good precursor is obtained with the following ratio of the cations Ba:Ca:Cu=2:2:3

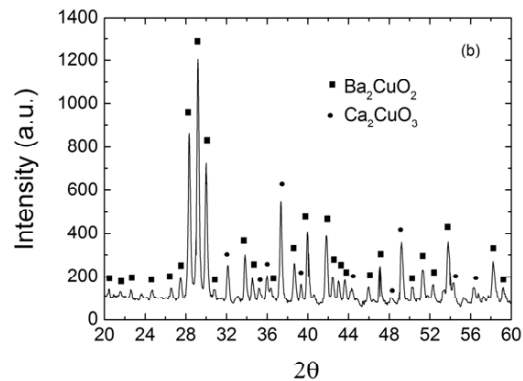
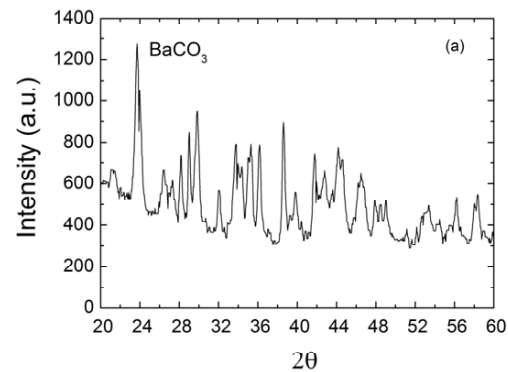


Fig. 3. XRD $Ba_2Ca_2Cu_3O_y$ samples, (a) synthesized on the air, (b) synthesized under oxygen pressure

[12].

The developed mobile device HTS OS-1000DMB and the tested technology allows us efficiently to oxygenate high homogenous powders obtained by the methods of PVD and CVD as well as the thin porous structures, mono crystals, long-range ordered composites and high temperature superconductive materials [13].

Acknowledgements

This work has been fulfilled by financial support of the Shota Rustaveli National Science Foundation, Grant No. FR/423/6-260/12.

ფიზიკა

მაღალტემპერატურული ზეგამტარების დაჟანგბადების ეფექტური ტექნოლოგია

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(წარმოდგენილია აკადემიის წევრის გ. თავაძის მიერ)

მაღალტემპერატურულ ზეგამტარში სუფთა მაღალტემპერატურული ფაზის მისაღწევად, აუცილებელი პირობაა რომ მათში ჟანგბადის კონცენტრაცია იყოს საკმარისად გაჯერებული. ჟანგბადის წნევის ქვეშ სინთეზი გვაძლევს არამხოლოდ მრავალფაზიანი პრეკურსორის ერთგვარონებას, არამედ ასევე აღმოფხვრავს მასში კარბონატის ნარჩენებს, რაც მნიშვნელოვანია Hg-ფუძიანი სუფთა მაღალტემპერატურული ფაზის მისაღებად. დაჟანგბადების პროცესი ითვალისწინებს ზეგამტარი ნიმუშებისათვის გარკვეულ ტემპერატურულ დიაპაზონში (400-1300K) ჟანგბადის რეგულირებად მიწოდებას. ამასთან ტემპერატურის მართვა ხდება არჩეული ალგორითმის შესაბამისად. წარმოდგენილი სტატია ეძღვნება მაღალტემპერატურული ზეგამტარი (მტზ) მასალების დაჟანგბადების ეფექტურ ტექნოლოგიას. დეტალურად განხილულია დამუშავებული მობილური დაჟანგბადების სისტემის HTS OS-1000DMB კონსტრუქციის ორიგინალური გადაწყვეტილებები, ელემენტების დანიშნულება, ტექნოლოგიური პროცესი და მიღებული შედეგები.

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Received August, 2015