Superconducting Precursors in Bi/Pb Multiphase Cuprates Fabricated by the Solar Technology and their Comparative Study by Torque Magnetometry Methods

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Received: 8 October 2023 | Revised: 21 October 2023 | Accepted: 28 October 2023

 DOI: https://doi.org/10.48084/etasr.6487

ABSTRACT

A special method has been applied to synthesize HTSC samples of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_y$ with increased local inhomogeneities, using solar energy for the melting and following superfast quenching of the melt (SFAQ-T technology). This study carried out a comparative analysis using low-frequency torsional vibration and resonant vibrating reed spectroscopy methods. The results showed a possibility for the existence of high-temperature superconducting precursors with T_c = 107-138 K and the potential advantages of the application of the resonant vibrating reed method.

Keywords-solar radiation; solar technology; high-temperature superconducting ceramics (HTSC); nominals $Bi_1.7Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_y$; torque magnetometry

I. INTRODUCTION

Among the widely known applications of superconductors is for the production of renewable energy using the environmentally harmless solar and wind energy [1]. HTSC materials containing Bi are among the most promising since they experience the transition to the superconducting state at T_c >100 K (Bi/Pb 2223, T_c = 107 K), and have a high second critical magnetic field record H_{c2} = 150 T [2-3] that is significantly higher than that of standard type II and all other high-temperature superconductors used in technology today. The properties of high-temperature superconducting ceramics are determined by the phase composition and textured structure, depending on their fabrication technology. In [4], a special technology was applied to synthesize HTSC samples with increased local inhomogeneities, called Solar Fast Alloy Quenching Technology (SFAQ-T). Based on glass-crystal and X-ray amorphous precursors, the HTSC samples were synthesized by quenching a melt produced by the heating of precursors with solar radiation at low temperatures. In [5], decomposition-resistant textured superconducting samples of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_{10-y}$ (n = 3-5) were fabricated with critical temperatures of the superconducting transitions T_c = 107-138 K.

Measurement methods are continueously being improved. To determine the critical temperatures of the superconducting (SC) transitions T_c of these samples obtained by this technology, the original torsional oscillation magnetometry method in applied magnetic fields was realized using an automated multipurpose device [6], which has a sensitivity comparable to that of a SQUID magnetometer. In [7-8], the potential possibilities of the Vibrating Reed (VR) magnetometry method for similar purposes were also investigated. The nature of SC precursors in cuprates was the subject of several studies [9-12]. Different superconducting experimental methods have led to different conclusions on the temperature range of superconducting fluctuations. The main challenge was to separate the SC response from a complex into normal-state behavior. For this purpose, in [9], a torque magnetometry method was used, which is a unique thermodynamical probe with extremely high sensitivity to SC diamagnetism. In torque magnetometry, the magnetization M is deduced from the mechanical torque $\tau = M\,H\sin\alpha$, where α is the angle between M and H, experienced by a crystal in an external magnetic field H. The torque is measured as a function of temperature (T), magnetic field strength (H), and the orientation of the sample to the field direction. This approach completely removes normal-state contributions, allowing tracing the diamagnetic signal above T_c with great precision. The results show that the SC diamagnetism vanishes in an unusual universal manner, showing the possibility that this unusual behavior signifies the proliferation of SC clusters as a result of the intrinsic inhomogeneity, which is the inherent property of the cuprates.

These results are very significant for many reasons. First of all, they constitute an unequivocal thermodynamic probe for the emergence of superconducting precursors in the cuprates, as SC emergence is observed via diamagnetism, which is the fundamental and prominent characteristic of the SC, and because such an experimental approach does not resort to any background normal phase effects. As discussed in [9], one could understand the unusual emergence of SC precursors by noting that cuprates are lamellar and perovskite-derived materials that are intrinsically inhomogeneous at nanoscale distances. Evidence for inhomogeneity was observed in Scanning Tunneling Microscopy (STM) and nuclear magnetic resonance [9]. Consequently, some of the spatially inhomogeneous SC gaps "survive" in the form of the SC precursor clusters at temperatures well above T_c. As the temperature decreases, these SC precursor clusters proliferate and grow in size, and eventually percolate near T_c. The emergence of superconductivity could be understood as a percolation process with a temperature scale controlled by the distribution of the SC gaps rather than by T_c. Despite many methods, it has not yet been possible to synthesize long-term stable room-temperature Bi/Pb superconducting phases. The Bi/Pb phase with high T_c = 197 K was obtained at a pressure of 10^3 Torr, but it decomposed at atmospheric pressure [13]. The progressive results achieved in the synthesis of superconductors with increasing critical temperatures of the superconducting transition from 97 to 260 K [14-19] provided an optimistic basis for continuing the research for an unconventional way for the synthesis of Bi/Pb.

This study aimed to compare the high-temperature precursors in $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_y$ using two different methods of torque magnetometry to further substantiate their existence and assess the potential possibilities of the precision VR method for the evaluation of T_c of the high-temperature precursors in this multiphase system.

II. EXPERIMENTAL RESULTS AND DISCUSSION

In [5], SFAQ-T technology was used to obtain HTSC ceramics of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_y$ series (n = 3÷5) and study its properties. The technology to produce SC ceramics using solar energy included the synthesis of precursors and the subsequent production of ceramics from them. The synthesis of precursors was carried out in a melt under the influence of solar radiation with a density of 700-780 W/cm^2, followed by rapid quenching of the melt. Ceramic samples were prepared using standard technology: grinding-pressing-annealing. The annealing of the ceramics was carried out in the range of 500-848°C for 3 to 120 hours. To determine the phase composition of the samples, a diffractometer (DRON-UM1, Cu Kα radiation, Ni filter) and a microscope (NU-2/E, Carl Zeiss Jena, Germany) were used. To determine the critical temperatures of the superconducting transition T_c of these samples, the original vibrating torsional magnetometry method was used to study the torsion oscillations of samples in an applied magnetic field, realized using an automated multipurpose device [6] having a sensitivity comparable to that of a SQUID magnetometer. The appearance of pinned vortices produces a non-zero magnetic moment M in the sample. The interaction between M and H creates a torque. This additional torque affects the oscillating system and makes the dissipation and frequency of the oscillations dependent on the external magnetic field. A unique feature of this method is also the synchronous measurements of two physical quantities, frequency and dissipation. The sensitivity of this method was very high at 10^{-17} W.

Torsion instrumentation is especially sensitive to the existence of superconducting precursors at temperatures $T > T_c$ of the materials under study in external magnetic fields [6, 9]. This approach completely removes normal-state contributions and thus allows one to trace the diamagnetic signal above T_c with great precision. The effect of superconducting precursors is particularly seen in high magnetic fields. Previous measurements of pinning and dissipation processes were carried out with the help of a low-frequency axial-torsion magnetometer and standard methods of magnetic and electric parameter measurements. These measurements showed that the samples obtained using this technology were multiphase, containing SC precursor phases with higher critical temperatures T_c reaching 138 K [6]. However, the concentration of new HTSC phases was so small that, for more precision assessments, a more sensitive method was necessary. Such a method could be the Vibration Reed (VR) method presented in [7]. As the frequency and dissipation of VR could be measured with high precision, the VR method exceeds in sensitivity a conventional ac-susceptometer. Using the VR method, one could study the elastic coupling between the Abrikisov vortex lattice and the crystal lattice, even in the micrometer-sized grains inserted in the normal matrix. Field dependence measurements of VR resonance frequency and dissipation could be used for the precision investigation of the first critical magnetic field H_{c1} temperature dependence without the introduction of free parameters, along with the energy dissipation character caused by the motion of the Abrikosov vortex lattice. In [20], a successful application of the VR method was presented for the investigation of two-phase Bi-

Ca-Sr-Cu-O superconducting ceramics and the precision assessment of their T_c.

One of the purposes of this study was to assess the potential possibilities of the VR precision method to evaluate the T_c of high-temperature precursors in multiphase HTSC samples of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_y$ produced by SFAQ-T.

Consider using the example of n = 3-5. The formation of SC phases of samples depends on the temperature and time conditions. After annealing at 500-600°C for 3 hours, a low-temperature superconducting phase 2201 was formed. When annealing at 700°C for 3 hours, the formation of about 50% of the 2212 phase (T_c = 97 K) and the appearance of faint traces of the high-temperature superconducting phase 2223 (T_c = 107 K) were established. More than 90% of the HTSC phase 2223 was obtained after annealing in the 848-850°C range for 90 to 120 hours. The difference in the phase composition of SC ceramics obtained from glassy-crystalline precursors is the formation of homologous SC phases, as evidenced by a series of reflections in diffraction patterns at 2θ, corresponding to certain *hkl*. This effect of the manifestation of a series of reflections was not observed in ceramics synthesized by the method of solid-phase reactions. This feature of ceramics made from glassy-crystalline precursors synthesized using solar technology can be explained by "freezing" the melt by quenching and the crystallization during heat treatment of supersaturated solid solutions. With the increasing duration of heat treatment, as a result of diffusion processes, the number of HTSC phase homologs decreased and the intensity of the reflection, which corresponded to the most stable phase at a given temperature, increased. The unit cell parameters of the homolog phases of type 2223 changed in the following intervals: for a sample of the nominal value $Bi_{1.7}Pb_{0.3}Sr_2Ca_2Cu_3O_y$ a = 5.2832÷5.3539 Å, b = 5.3698÷5.5075 Å, c = 36.506÷37.3855 Å, for a sample of the nominal $Bi_{1.7}Pb_{0.3}Sr_2Ca_3Cu_4O_y$ a = 5.0545÷5.1226 Å, b = 5.6696÷5.8918 Å, c = 36.4798÷37.2383 Å. The degree of texture of the ceramics obtained at 817 °C for 65 hours, shown in Figure 1(a), and at 820 °C for 120 hours, shown in Figure 1(b), was evaluated according to the Lotgering factor [21]. The texture was assessed for reflections [0010], [0012], and [0014] using the Lotgering formula $F = (I_a - I_b)/I_a$ 100 %, where I_a and I_b are the intensity of the selected reflection according to Figure 1. For the HTSC ceramics of the nominal $Bi_{1.7}Pb_{0.3}Sr_2Ca_4Cu_5O_y$, the following values were determined: F = 73%, F = 65%, and F = 64% by reflections [0010], [0012], and [0014], respectively. These results showed an increase in the degree of texture (00L) with increasing temperature and duration of heat treatment.

A study of the superconducting properties of HTSC ceramics of this system by the low-frequency axial-torsion magnetometry method showed that along with the manifested effects that determine the transition to the superconducting state at 107 and 138 K, numerous "bursts" ("chaos" regions) were identified, which were not observed in samples synthesized by the solid-phase reaction method. "Bursts" can refer to homologous phases that appear in diffraction patterns as a series of reflections, or these effects refer to physical phenomena associated with electronic states caused by the action of a concentrated light flux [22]. In the subgroup of compounds 2256-2289, the phase composition was similar to the subgroup 2223-2234 [23], and for the 221920 sample it looks as shown in Figure 2.

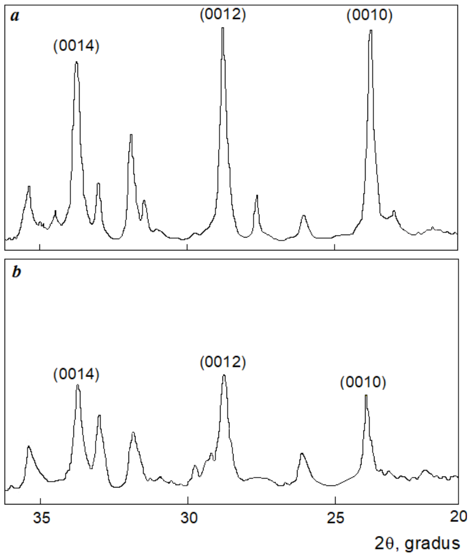

Fig. 1. Diffraction patterns of textured superconducting ceramics with the nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_2Cu_3O_y$, obtained from precursors synthesized in a solar oven: (a) at 817 °C for 65 hours, and (b) at 820 °C for 120 hours.

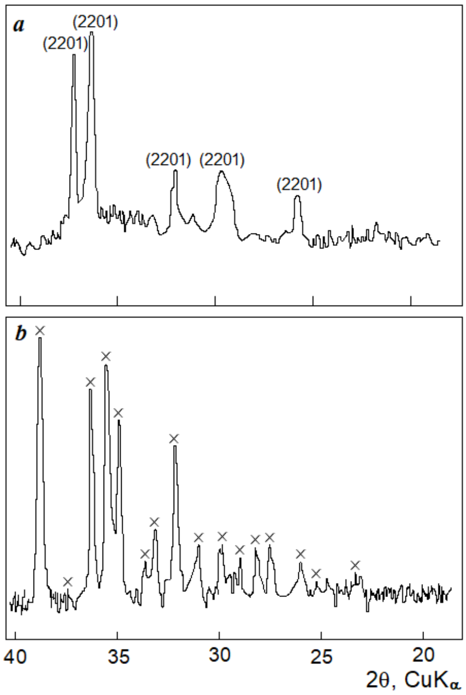

Fig. 2. Diffraction pattern of a precursor with the nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_{19}Cu_{20}O_y$, obtained by improved SFAQ-T: (*a*)-precursor (2201), (*b*) after annealing at 847 °C for 24 hours (× - phase 221920).

Figure 3 shows the form of the microstructure of the annealed ceramics of this type of Bi/Pb (221920) composition.

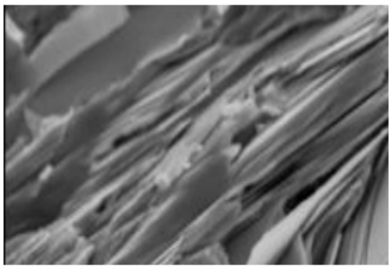

Fig. 3. Microstructure of HTSC ceramics of nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_{19}Cu_{20}O_y$ obtained from precursors synthesized using solar technology.

Ceramic HTSC samples had a compressive strength limit of 12-14 MPa. The phase composition and superconducting effects of 2245 HTSC ceramics [23] were reproduced after aging the samples for 7 years. A direct relationship was identified between the growth in the critical temperature of the transition to the superconducting state and the value of *n*, which allowed the assumption of the existence of HTSC with T_c over 107 K [23].

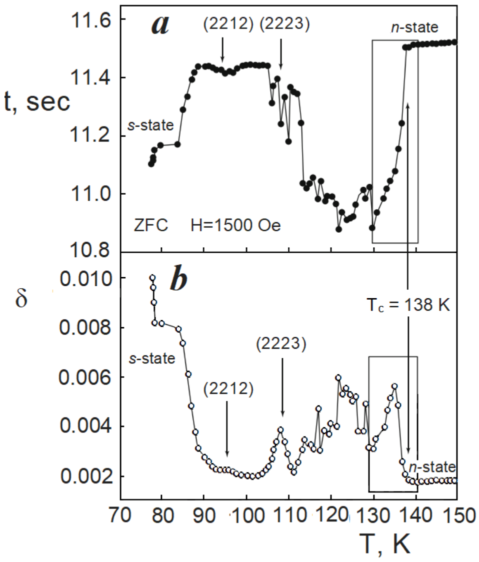

Fig. 4. Temperature dependence of the logarithmic damping decrement *δ* and the period *t* of oscillation in the magnetic field of the ceramic sample of the nominal $Bi_{1.7}Pb_{0.3}Sr_2Ca_3Cu_4O_y$, obtained from precursor synthesized using solar technology (zero field-cooled ZFC-mode): *s*-state and *n*-state are superconducting and normal states, respectively.

The $Bi_{1.7}Pb_{0.3}Sr_2Ca_{19}Cu_{20}O_y$ system was chosen for the following comparative study. Compared to the similar dependence for the single-phase sample $Bi_{1.7}Pb_{0.3}Sr_2Ca_2Cu_3O_y$, Figure 5 shows the absence of features corresponding to SC precursors, and Figure 6 shows the temperature dependence of critical parameters for SC precursors in the $Bi_{1.7}Pb_{0.3}Sr_2Ca_{19}Cu_{20}O_y$ sample.

The electronic part of the VR acoustic spectrometer contains an acoustic spectrometer operating at a frequency of approximately 1 kHz and instruments for powering a permanent magnet. The sensitivity of the spectrometer provides measurements of the natural frequency of the sample *f* with an accuracy of approximately 0.1%. In the VR acoustic spectrometer, the electrostatic method of exciting bending oscillations of a sample having the shape of a rectangular plate was used. The electronic equipment of the spectrometer allows measurements in the mode of self-excitation of samples at their natural resonant frequencies. The electrode, located in the immediate vicinity of a sample, comprises the capacitance included in the oscillating circuit of the high-frequency generator and serves simultaneously to excite and detect oscillations of a sample. Measurements of the resonant frequencies f_r of the sample oscillations make it possible to determine the elastic modulus *E* according to the relation:

$$f_r = k\frac{d}{L^2}\sqrt{\frac{E}{\rho}}$$

where *d* is the thickness of the sample, *L* is the length of the oscillating plate, *E* is the modulus of elasticity, *ρ* is the density of the sample, and *k* is a constant factor.

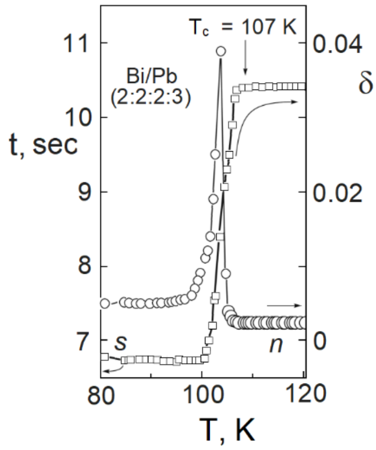

Fig. 5. Temperature dependence of the logarithmic damping decrement *δ* and the period *t* of oscillation in the magnetic field of ceramic samples of the nominal $Bi_{1.7}Pb_{0.3}Sr_2Ca_2Cu_3O_y$, obtained from precursors synthesized using solar technology.

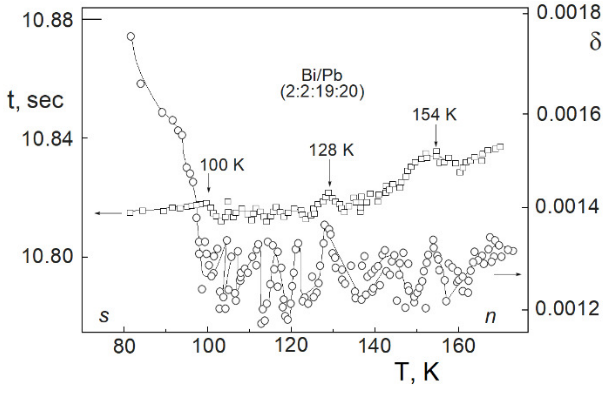

Fig. 6. Temperature dependence of the logarithmic damping decrement *δ* and the period *t* of oscillation in the magnetic field of a ceramic sample of the nominal $Bi_{1.7}Pb_{0.3}Sr_2Ca_{19}Cu_{20}O_y$, obtained from precursors synthesized using solar technology.

The variation of the square of the resonant oscillation frequency (f^2) of a sample can be considered in the framework of the so-called magneto-mechanical approach [7], according to which when a superconducting sample is displaced relative to the external magnetic field H, a restoring mechanical force acts on each "pinned" magnetic vortex. As a result, the oscillation frequency of the entire sample changes by $\Delta f(H)$, depending on the density of the fixed vortices, the moment of inertia of the superconductor, and its volume. The dependence of the elastic modulus E of the substrate-sample system (in units of f^2) on the magnitude of the magnetic field gives information on the elastic interaction of the Abrikisov vortex lattice with the crystal lattice. This is a convenient method for determining the magnitude of the vortex pinning force. In addition, the $f^2(H)$ dependence provides a simple method for determining the lower critical magnetic field H_{c1} value and the critical temperatures of the superconducting precursors in the multiphase HTSC samples. Figure 7 shows the measured temperature dependences of the square of the natural frequency (f^2) of oscillations of the Bi/Pb system (2-2-8-9) and (2-2-19-20) samples in a magnetic field. Measurements were carried out in Zero Field Cooled mode (ZFC-mode) in the magnetic field 300 mT, turned on after the cooling of a sample to 80 K.

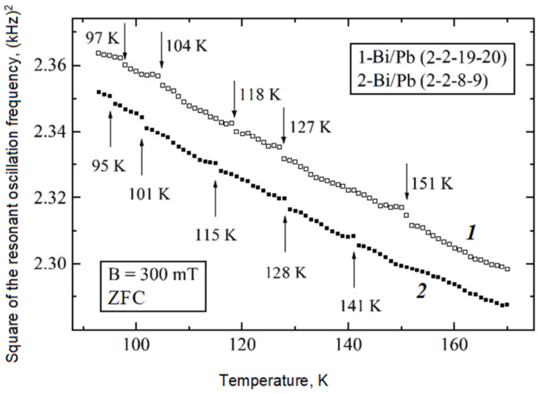

Fig. 7. Temperature dependence of the square of the natural frequencies of the multiphase samples (2-2-19-20) and (2-2-8-9) in a magnetic field of 300 mT.

In the area of ~90-160 K in the samples (2-2-8-9) and (2-2-19-20), there are features near temperatures of 95, 101, 115, 128, and 141 K, which are associated with the existing superconducting high-temperature precursors in the multiphase samples of $Bi_{1,7}Pb_{0,3}Sr_2Ca_{n-1}Cu_nO_y$ system, which are absent in case of a single-phase sample (2-2-2-3) synthesized by solid-phase technology. Thus, it can be concluded that in samples (2-2-8-9) and (2-2-19-20), there are four different SC precursor phases. When comparing the features in Figures 6 and 7, it can be seen that they are in a definite correspondence, but in the case of Figure 7, these features are better separated. This observation can be considered as another confirmation by the VR magnetometry method of the existence of high-temperature precursors in multiphase samples of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_y$ and, in addition to it, shows the potential advantages of applying the VR method to study the superconducting precursors in multiphase HTSC samples.

III. CONCLUSION

In torsional low-frequency and vibrating reed dynamic experiments to investigate the magnetic properties of multiphase cuprate superconductors $Bi_{1,7}Pb_{0,3}Sr_2Ca_{n-1}Cu_nO_y$ (n=3-5, 20) synthesized using SFAQ-T technology, precursor phases were detected near T_c=107-160. Analysis of the nature of the obtained dependences and their comparison with other available results associated with the processes in the vicinity of critical temperature T_c allows inferring the existence of high-temperature superconducting precursor phases. The comparative study of torsional and vibrating reed magnetometries for the evaluation of T_c of the superconducting precursors in the multiphase HTSC Bi-Pb-Sr-Cu-O system fabricated by the SFAQ-T technology was investigated for the first time. The results obtained by both methods were shown to have sensitivity to superconducting diamagnetism, allowing the discovery of new superconducting precursor phases above bulk T_c in these samples. In addition, compared with the low-frequency torsional spectroscopy method, the vibrating reed spectroscopy method has potential advantages for the study of the superconducting precursors in multiphase HTSC ceramics.

ACKNOWLEDGMENT

This research was supported by the Shota Rustaveli National Science Foundation of Georgia (SRNSFG) [STEM-22-1030].

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