On the Nature of Superconducting Precursors in Bi-Pb-Sr-Ca-Cu-O Compositions Fabricated by Hot Shock Wave Consolidation Technology

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***Abstract*—In this paper, the possibility of critical temperature increasing of superconducting precursor T_c and the current bearing capacity in samples of Bi-Pb-Sr-Ca-Cu-O superconducting system fabricated using hot shock wave consolidation (HSWC) technology and investigated by the vibrating torsional magnetometry method, was studied. The advantage of HSWC technology over the traditional technologies of superconducting composites synthesis is that the high-density materials are made from the Bi-Pb-Sr-Ca-Cu-O superconducting system. After the action of explosive wave the superconductivity is retained. After the explosion a pronounced texture is formed indicating the creation of efficient pinning centers and thus, the increase of current-carrying ability of the obtained material. The critical temperature of potential superconducting precursor T_c of transition to superconducting state increased from T_c=107K for starting sample to T_c=138K, using the HSWC technology for synthesis of samples in range of pressures from P=5GPa up to P=12GPa.**

Keywords-HTSC; shock-wave consolidation; vibrating torsional magnetometry; ctitical temperature of superconducting transition; high-temperature superconducting phases; pinning

I. INTRODUCTION

After the epoch-making discovery of high-temperature superconductors (HTSC) great efforts were devoted worldwide to the further increase of the critical temperature of superconducting transition Tc with aim to reach room temperatures. Such a discovery would cause the creation of a new technological progress using HTSCs with Tc higher than the ones currently used for cooling liquid helium, nitrogen and hydrogen (YBaCuO and MgB_2, as example) thereby opening great possibilities for their applications in electronics and energetics. From this point of view, particular interest attracts the Bi-Pb-Sr-Ca-Cu-O system, because it is characterized by high Tc=107K and the record high second critical magnetic field H_{c2}~150T. In [1], the universal behavior of the superconducting (SC) precursor signifying the proliferation of SC clasters as a result of the inherent intrinsic inhomogeneity of cuprates, was revealed. Understanding of its nature could be very important in fabrication of new HTSC materials with Tc close to the room temperatures. The nature of the superconducting (SC) precursor in the cuprates has been the subject of intense interest. Different experimental researches have led to very different conclusions on the temperature range of superconducting fluctuations. The main challenges have been to separate the SC response from the complex normal state behavior. For this aim, in [1] a torque magnetometry method was used, a unique thermodynamic probe with extremely high sensitivity to SC diamagnetism in four distinct cuprate compounds. In torque magnetometry, the magnetization $\mathbf{M}$ is deduced from the mechanical torque $\tau = MxHsin\alpha$, where α is the angle between M and H, experienced by a crystal in an external magnetic field $\mathbf{H}$. The torque is measured as a function of temperature T, magnetic field strength H, and orientation of the sample with respect to the field direction. This approach completely removes normal-state contributions and thus allows one to trace the diamagnetic signal above T_c with great precision. Results show that SC diamagnetism vanishes in an unusual universal exponential manner showing distinct possibility that this unusual behavior signifies the proliferation of SC clusters as a result of the intrinsic inhomogeneity known to be an inherent property of the cuprates. These results are significant for a number of reasons. First, they constitute an unequivocal thermodynamic determination of SC emergence in the cuprates, as we observe SC emergence directly via diamagnetism, a fundamental and prominent characteristic of superconductivity, and because such experimental approach does not resort to any background normal phase effects. As discussed in [1], one could understand the unusual SC emergence noting that the cuprates are lamellar, perovskite-derived materials that are intrinsically inhomogeneous at the nanoscale scale. Evidence for inhomogeneity is observed on multiple energy scales in scanning tunneling microscopy (STM) and nuclear magnetic resonance measurements [1]. Consequently, some of the spatially inhomogeneous SC gaps "survive" in the form of SC clusters at temperatures well above T_c. As the temperature decreases, these clusters proliferate and grow in size, and eventually percolate near T_c. The emergence of superconductivity may therefore be thought of as a percolation process, with a temperature scale controlled by the distribution of the SC gap rather than by T_c.

In [2], special technology was applied to synthesize HTSC samples with increased local inhomogeneities. Using recently developed solar fast alloys quenching technology (SFAQ-T) [3], based on glass-crystal and X-ray amorphous precursors, authors synthesized samples by quenching at liquid nitrogen temperatures of melt which was produced by heating of precursors with solar radiation and obtained decomposition-resistant textured superconducting systems $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_{10-y}$ (n=2-30) with critical temperature of the superconducting transitions up to 200K [2]. To determine the critical temperature of the superconducting transition T_c of these samples it was used an original vibrating torsional magnetometry method to study the torsion oscillations of samples in an applied magnetic field realized using an automated multipurpose device [4], having the sensitivity comparable with that of a SQUID magnetometer, designed at E. Andronikashvili Institute of Physics (Tbilisi, Georgia). The investigation was carried out at low-frequency axially-torsion oscillations (0.1÷1Hz) in a permanent magnetic field with strength H=150mT and showed a significant effect of the background of the experiment, the value of H, initial orientation of the sample and the direction of variation in the temperature of the sample (cooling or warming) on the obtained results. The torsion instrumentation used is especially sensitive to reorientation of magnetic moments of the materials under study in external magnetic fields. As all studied HTSCs possess their own magnetic moments, the experiments of this kind are quite informative when studying structural transitions, especially when such transitions are accompanied by reorientation of magnetic moments including the normal state at $T>T_c$.

Using this method, a wide attenuation peak (ΔT~100K) with a maximum at $T\approx$200K was detected in cuprate superconductors (HTSC) $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_nO_y$ (n=2-30), This peak was particularly pronounced in field cooling (FC) experiments, i.e. after abrupt cooling of the sample in the external magnetic field at the temperature $T<T_c$ with subsequent slow warming up to room temperature with invariance of the applied field. The attenuation peak height depended on the preliminary orientation (before cooling) of the samples θ in the measured permanent magnetic field H. On the one hand, it is well known that, after the FC procedure and subsequent slow warming up, at the temperatures close to the critical temperature T_c, the attenuation peak associated with "melting" of the Abrikosov frozen vortex structure and its disappearance at $T>T_c$ is detected in monophase samples. At the same time, in most multiphase bismuth HTSC samples, synthesized using solar energy and superfast quenching of the melt, the attenuation peak with the maximum at $T\approx$200 K was observed. Depending on the synthesis conditions, the attenuation peak could be two-humped and could be located in the temperature range much wider than T_c of the major superconducting phase. It was assumed that this is due to the existence of frozen magnetic fluxes (after FC) in superconducting "dropping" regions, which gradually (with increasing temperature) transfer into the normal state and release pinned vortex threads. This fact could be a cause of observed dissipative processes, so as also the evidence of the existence of superconductivity at $T\geq$240K.

In [2], the intrinsic inhomogeneity of HTSC was increased using the sun melting of samples followed by their superfast quenching at liquid nitrogen temperatures. In current work for the modification of microstructure, introduction of efficient pinning centers and enhancement of intrinsic inhomogeneity of HTSC samples we use the original HSWC technology [5]. In powder mixtures, shock waves lead to an extremely rapid mass transfer and induce high velocity collisions among suspended solid particles. Such interparticle collisions result in the extreme heating at the point of impact, which can lead to the effective localized melting and significant increase in rates of numerous solid state reactions. As a consequence, the morphology of the initial materials is significantly modified: individual grains are grinded, smoothed and welded together, ultimately resulting in more compact material. Such a morphology change could lead to the better inter-grain coupling and annealing of intra-grain defects. It opens also ways for formation of new HTSC phases at grain boundaries. The application of shock wave pressure proved to be an effective method to generate such superconducting interfaces [6]. The HSWC technology was recently successfully applied for the fast production of superconducting MgB_2 samples avoiding long-time solid state reaction procedures [7]. We present further the experimental results of study of the HTSC samples of Bi-Pb-Sr-Ca-Cu-O system fabricated by the HSWC technology showing the possibility to increase significantly their critical temperature T_c.

II. EXPERIMENTAL RESULTS AND DISCUSSION

The novelty of the proposed HSWC technology is in the consolidation of bulk samples of high density from superconducting powders of mixtures with the dimensions of the order of $\varnothing$~2-5mm, L~50-70mm. The process of consolidation performed into two stages [5]:

1. Explosive pressing of powder precursor mixtures is made at room temperature with 5-20GPa loading for increasing the

initial density and for activation of the surface of mixture particles.

2. The obtained cylindrical sample is pressed by explosive wave of 5-10GPa, but now at 700-800°C.

The study of superconducting characteristics shows that after the action of the explosive wave, the material retains superconductivity and the explosive pressing of powder precursor mixtures at room temperature with 5GPa, 7GPa and 12GPa loading does not change significantly the superconducting state. After explosion, the pronounced texture is formed, which with the increase of pressure and the temperature up to 700-800°C, could result in the increasing of T_c and the current-carrying abilities of synthesized superconducting Bi-Pb-Sr-Ca-Cu-O material. We present further the results on the critical temperature increase of superconducting transition in Bi-Pb-Sr-Ca-Cu-O HTSC system samples fabricated using HSWC technology. The temperature dependence of magnetic susceptibility χ for starting sample Bi-Pb-Ca-Cu-O (2223) is presented in Figure 1 and the critical temperature of superconducting transition is defined which appeared to be T=107K.

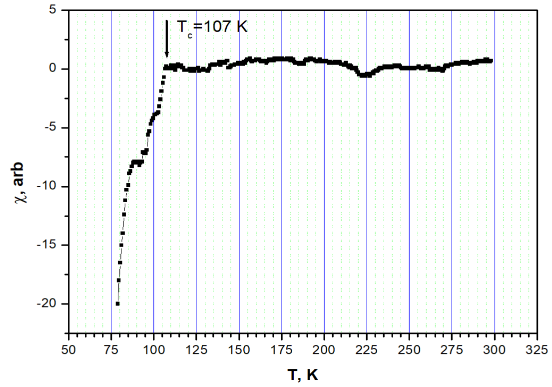

Fig. 1. Dependence of magnetic susceptibility χ on temperature for Bi-Pb-Sr-Ca-Cu-O sample.

We measure the critical temperatures of superconducting transitions with three methods: Two of them are the standard methods of T_c measurement, the measurement of magnetic susceptibility $\chi=f(T)$ and electric resistance and the third one is the original supersensitive torsional mechanical method. At application of the supersensitive mechanical torsional magnetometry method we study the dependence of oscillation period of superconductive cylinder suspended by a thin elastic thread performing torsional oscillations in the transverse magnetic field $t=f(T)$. The mechanical method of measurement of the critical temperature T_c of transition in the superconducting state is a part of methods for study of pinning, dissipation processes and investigations dynamics of Abrikosov vortex lattice in superconductors, is developed by us in the Andronikashvili Institute of Physics, [8-11]. These methods proved to be much more sensitive when compared with the traditional ones for investigation of superconductors such as resistivity and magnetic susceptibility measurements. The measurement of critical temperature using supersensitive mechanical method is based on the change of oscillation period of a superconductive cylindrical sample suspended by a thin elastic thread and performing axial-torsional oscillations in transverse magnetic field $H>H_{c1}$. At transition in the superconducting state superconductor quantized vortices of magnetic flux – Abrikosov vortices are created [12]. Abrikosov vortices are fixed on the crystal lattice defects, they cause the change of superconducting cylinder oscillation frequency. Torn off ones give a change in the dissipation of oscillations. Therefore the investigation of temperature dependence of frequency (period) and dissipation of oscillations give one opportunity to define the critical temperature of transition in the superconducting state, study dissipation processes taking place in vortex matter and reveal new HTSC phases with higher critical temperatures T_c if they are present in the investigated HTSC samples. Due to the high sensitivity of the method (of the order of $\approx 10^{-17}$W) it is possible to record superconducting phases at concentrations much lower than the one of the main phase of the investigated sample.

We measured the pinning force which makes it possible to judge regarding the current bearing capacity of HTSC. In Figure 2 the results of T_c measurements are presented which were obtained by the use of the above mentioned three methods for Bi-Pb-Sr-Ca-Cu-O (2223) samples. As it is seen, all three methods give T_c=107K. The method scheme and setup for measurements using the mechanical methods of the T_c, pinning force, dissipation processes and Abrikosov vortex lattice (magnetic flux quanta) dynamics is presented in Figure 3. The temperature dependence of the oscillation period of the suspension system with a superconducting sample suspended by a thin elastic thread and performing axial-torsional oscillations in a magnetic field directed perpendicular to the axis of superconducting cylinder for the HTSC system Bi/Pb (2223) sample synthesized by HSWC technology at $P\approx$5GPa is presented in Figure 4. We see that the critical temperature of superconducting transition after the shock wave effect increases compared with the starting sample, from T_c=107K to T_c=115K. The surface photo of this sample presented in Figure 5 shows the absence of surface contamination.

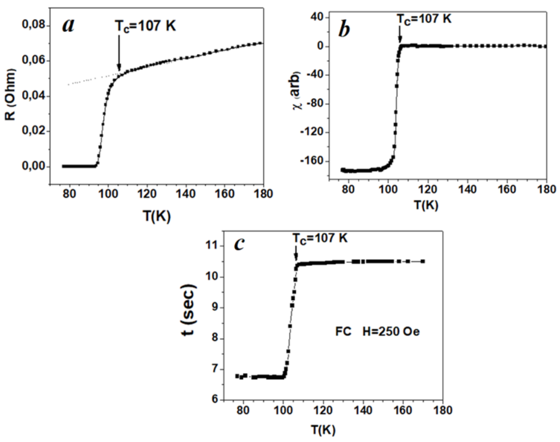

Fig. 2. The temperature dependence of electric resistance R (*a*), magnetic susceptibility χ (*b*) and oscillation period t (*c*) of superconducting sample $Bi_{1.7}Pb_{0.3}Sr_2Ca_2Cu_3O_{10-\delta}$ (2223) suspended on a thin elastic thread and making axial-torsional oscillation in transverse magnetic field.

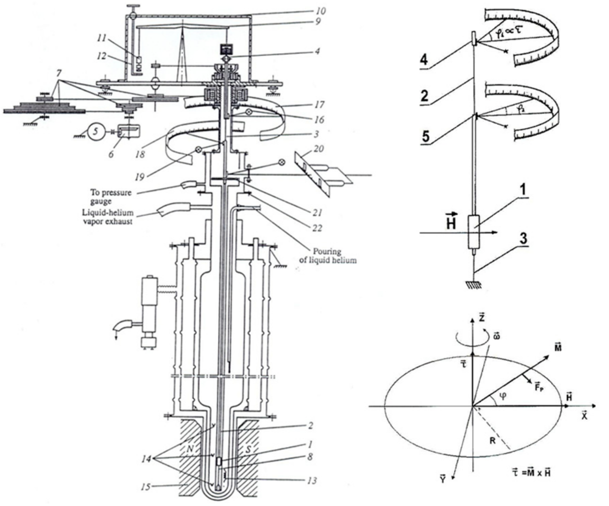

Fig. 3. On the left, the diagram of the device is presented: (1) sample under study, (2) rod, (3) suspension filament, (4) rotating head, (1) motor, (6) reducer, (7) system of pulleys, (8) capron filament, (9) weigh beam, (10) bell jar, (11) weight, (12) manipulator, (13) electric heater, (14) thermocouples Cu-CuFe, (15) electromagnet, (16) illuminator, (17) and (18) circular scales, (19) illuminator, (20) linear scale disk, (21) disk and (22) bearing (demountable) part of the vibrating system. On the right, the schematic diagram and the geometry of the experiment: 1 - sample, 2 - upper elastic filament, 3 - lower filament, 4 - leading head, 5 - glass rod. φ is an angle between M and H.

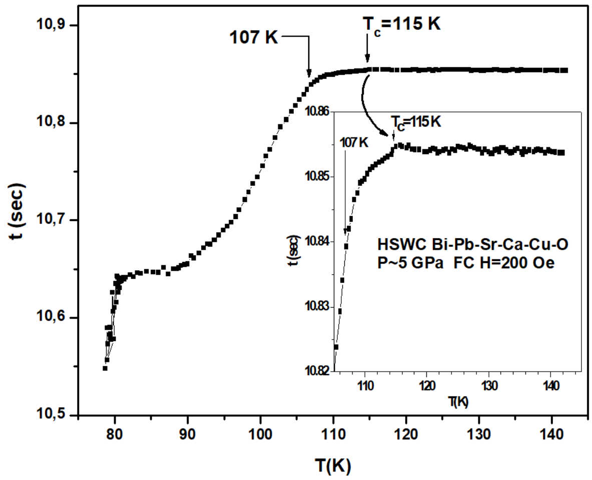

Fig. 4. Dependence of period *t* on temperature T of superconducting sample suspended by a thin elastic thread and making axial-torsional oscillations in transverse magnetic field, at P~5GPa.

In Figure 6 similar results are presented for *P*≈7GPa. In this case the critical temperature increases further and turns to be of the order of *T*=130 K. Figure 7 shows a rather good sample consolidation. In Figure 8 similar results are presented for pressures of the order of *P*≈12GPa. In this case T_c increases 8 additional degrees at T_c=138K. The use of HSW Consolidation for creation of new superconducting materials allows one to synthesize such high-temperature superconducting systems in which the critical parameters of superconductors can be increased significantly. Pictures presented in Figures 5, 7 and 9 show good consolidation.

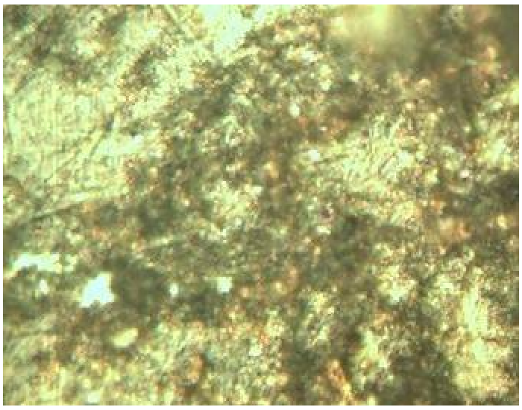

Fig. 5. Polishing of samples (*P*=5GPa) shows that there is no contamination and there is a reasonable consolidation, i.e. good densification results.

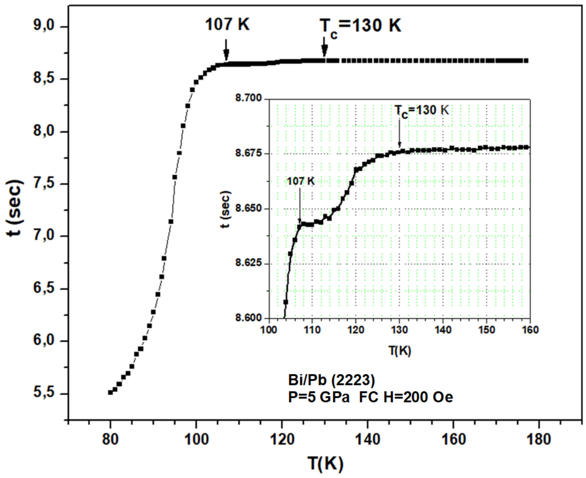

Fig. 6. Dependence of period *t* on temperature T of superconducting sample suspended on a thin elastic thread and making axial-torsional oscillation in transverse magnetic field, at P~7GPa.

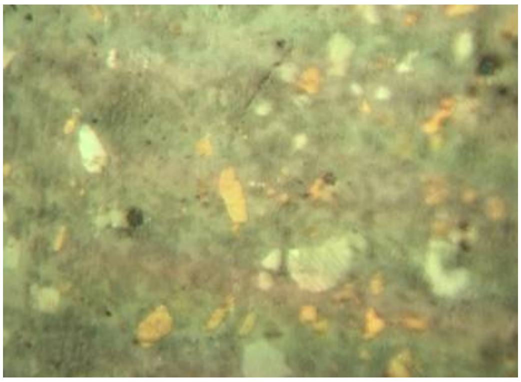

Fig. 7. Sample is compacted at pressure *P*=7GPa. The figure is taken at x350 magnification.

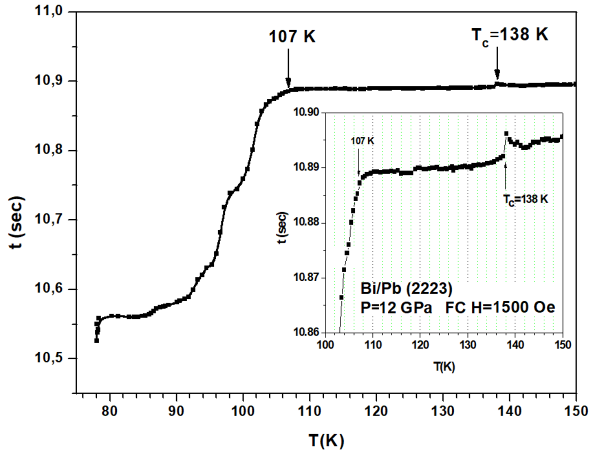

Fig. 8. Dependence of *t*-period on temperature *T* of superconducting sample suspended on a thin elastic thread and making axial-torsional oscillation in transverse magnetic field, at *P*~12GPa.

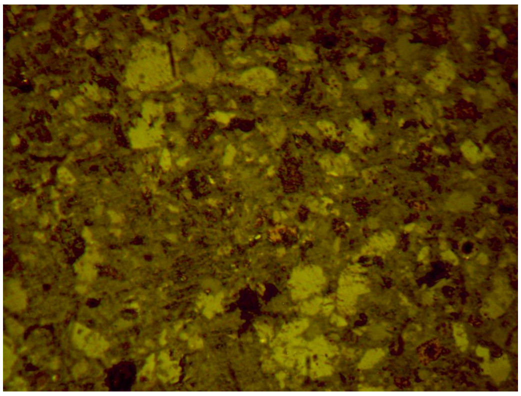

Fig. 9. Sample is compacted at pressure *P*=12GPa. The figure is taken at x200 magnification.

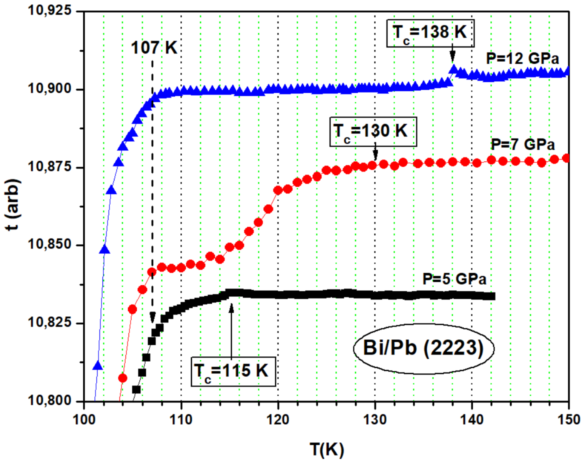

Fig. 10. Dependence of period *t* on temperature *T* of superconducting sample suspended on a thin elastic thread and making axial-torsional oscillation in transverse magnetic field, at *P*~5GPa, *P*~7GPa and *P*~12GPa.

The effect of HSWC on critical transition temperature in superconductive state T_c of the high - temperature superconductive system Bi-Pb-Sr-Ca-Cu-O at room temperature and at $P\approx5$GPa, $P\approx7$GPa and 12GPa is presented in Figures 4, 6 and 8. They show that pressure of $P\approx5$GPa increases the critical temperature of transition into superconductive state *T* from T_c=107K up to T_c=115K, HSWC with $P\approx7$GPa makes T_c=130K and HSWC with *P*=12GPa makes T_c=138K(differentiation of 31 degrees).

III. CONCLUSION

The possibility of increasing of T_c and the current bearing capacity in HTSC samples of Bi-Pb-Sr-Ca-Cu-O systems fabricated using HSWC technology and vibrating torsional magnetometry method was studied. The advantages of hot HSWC technology over traditional technologies of the synthesis of superconducting composites are:

- The materials of high-density are made from Bi-Pb-Sr-Ca-Cu-O superconducting system.
- After the action of explosive wave the superconductivity is retained.
- After the explosion, a pronounced texture is formed indicating the creation of efficient pinning centers and thus, the increase of current-carrying ability of the obtained material.
- The critical temperature of potential superconducting precursor T_c of transition to superconducting state increased from T_c=107K (starting sample) to T_c=138K, using the HSWC technology for synthesis in a range of pressures from *P*=5GPa up to *P*=12GPa with 31 K increase of T_c on the 12GPa case.

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