Solar technology for the production of Bi/Pb superconducting ceramics and its properties

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Abstract: Based on the experience of isostoichiometric and isostructural oxides production using solar energy, the prospects for synthesizing of Bi/Pb superconducting ceramics are shown. The principle of technology in the Large Solar Furnace (Parkent), the influence of gradient conditions on the formation of the oriented structure of crystallites-nuclei, and the connection of the innate properties of precursors with the lamellar morphology and phase composition of massive ceramics are described. The homophase composition of Bi/Pb ceramics was determined. Graphs of resistance and voltage of BSCCO in the temperature range 80-320K are presented. Anomalous of resistance and magnetic susceptibility in bismuth cuprates synthesized by solar energy were observed. Experimental graphs in resistance and magnetic susceptibility changes represent dependencies characteristic of the potential superconducting transitions. The manifestation of the Meissner effect at room temperature and normal atmospheric pressure and daylight, and under the influence of light is shown. The properties of Bi/Pb ceramics are explained by the connection with the innate properties of precursors obtained by concentrated solar energy.

1. Introduction

Promising examples of "nitrogenous" superconductors practical use in various fields of human life activities force us to continue experimental and theoretical research on the way to searching technologies for creating room-temperature massive superconducting materials, which are necessary for high current energetics and massive products. Results of the using various technologies show the importance of the principle process and conditions influencing the critical characteristics of target superconductors [1-16]. Current parameters direct the dependence on the microstructure perfection and force to search for technologies and conditions that allow the formation of a massive oriented crystalline structure of superconducting ceramics. Application of known texturizing technologies with directed impact of the gradient heat flow led to positive results concerning the thin superconducting films, but it is a very challengingly difficult technology process to create the oriented microstructure in massive ceramics with congenital misoriented microstructure. In comparison with technologies based on "solid state reaction" the melting technologies, based on the principle of growing up monocrystals with oriented crystallization of melt, allows for the creation of a high texture [17]. However, the necessity of maintaining a flat crystallization front for implementation of peritectic equilibrium, a low crystallization process speed, high energy consumption, an inert crucible problem, and the complicated technological equipment confine the wide practical application of these technologies. Developed in

recent years the nontrivial energy-saving, green "Super Fast Alloys Quenching–T" ("SFAQ") technology of superconducting cuprates synthesis is founded on using solar energy for melting and hardening in sharp temperature gradient conditions. [18-23]. The positive results, reached by using this method, raise expectations of progress in obtaining massive high-temperature superconducting ceramics by solar energy. The article presents the features of solar technology and the results of studies of precursors and ceramics of Bi/Pb cuprates to present experimental data to expand the practical applications of solar technologies in the materials science of bismuth superconductors.

2. Synthesis Methods Results and Discussions

Mixtures for bismuth cuprates synthesis by solar energy were prepared from compounds of Bi_2O_3, PbO, SrO, CaO, CuO powders according to formula $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=3-30. Before the synthesis in a large Solar Furnace (LSF) (Parkent), the analysis of optical-energy parameters meeting the requirements for melting and hardening was carried out. These parameters provide the directed microstructure, the eutectic phase amorphization, and the average productivity of $\approx$20kg/sunny day for the above pointed mixture composition above [24]. The directed growth of crystallite-nuclei is accomplished through a sharp temperature gradient in molten bath and remains at firing it. This is one of the most valuable advantages of solar gradient technology, which is very complicated or impossible to implement by the most known processes.

To implement the "solar" technological process of obtaining material by melting or heat treatment it is necessary to arrange the initial powder (mixture) into a melting area (or heat treatment one) so that excludes the way out of the focal the zone and the dropping of drops the mixture from the water-cooled substrate. In the case of the inclined arrangement of a water-cooled substrate, it is necessary to define optimal tilt angle of a substrate and the density of falling concentrated solar flux on the material substrate to direct crystallite growth in a direction of solar flux vector. In this way, the sustainable implement of the molten bath position during the melting of mixtures and the orientation of formed in the melt crystallites in a direction of heat source is reached. The appropriate density of solar flux needed for the melting of mixtures of the superconducting cuprates is approximately defined from the ionization energy of oxides included in the formula of $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=3-30, and data on the energy of formation of bismuth cuprates [25]. The substrate was inclined at an angle of about 30 degrees to the optical axis of LSF (Parkent). Solar flux density in the focal zone of the substrate was calculated [26]:

$S_c = S_0' \cdot cosi$ (S_c– direct radiation, i– angle of incidence of solar flux).

1) $4\propto = \angle 70°$, $i = \angle 0°$

$S_4 = S_0 \cdot cosi$

$S'_4 = 7.45 \ wt/cm^2 \cdot cos0 = 7.45 \ Wt/cm^2$

2) $3\propto = \angle 52.5°$, $i = \angle 17.5°$

$S_3 = S_0 \cdot cosi$

$S'_3 = 30.29 \ wt/cm^2 \cdot cos17.5 = 28.88 \ Wt/cm^2$

Allowing for errors in LSF optical system due to inaccuracies of mirror adjustment and dust on their surface, the solar flux density was not less than 400 wt/sm². The substrate position scheme in the Bi/Pb synthesis process and the positions of the mixture and melt on the water-cooled base in LSF are shown in Figure 1a,b,c.

Figure 1.
Water-cooled substrate position scheme of the molten melt (mixture) bath in the technological process of synthesis in Large Solar Furnace (a), prepared mixture charge for melting (b), the synthesis process in a melt on the water-cooled base, inclined at an angle $\approx 30^{\circ}$ to the optical axis of Large Solar Furnace (Parkent).

The technological process of synthesis of Bi/Pb superconductors was preceded by the analysis of previously performed technologies of synthesis of materials of different composition: Al_2O_3, Al_2TiO_5, ZrO_2-Al_2O_3 (eutectic), $SrTiO_3$, $BaTiO_3$, $CaTiO_3$, Bi/Pb (BSCCO), partly presented in Fig. 2 [26].

Figure 2.
Materials, obtained by melting and hardening in the LSF (Parkent) according to the scheme with the slope angle of the water-cooled substrate $\approx 30^{\circ}$.

For the synthesis of materials with different properties, the concentrated solar flux with a power which appropriating to their melting temperatures, color, and bulk density was required. A similar method of the parameter optimization of the LSF optical-energy system was used in the technology of the Bi/Pb synthesis. Before the melting a mixture is heated in the solar flux densities interval $150 - 300 wt/cm^2$. At a solar flux density of $\sim 460 wt/cm^2$, after the melt bath was formed, the melt was formed, the melt was hardened in a dispersant [20-23]. In Fig. 1c it could be imagined the parameters of melt which is discharged by a thin jet onto the dispersant. The melted charge on which the melt is located is a garnish fragment which is represented in Fig. 3a, which is the support for the melt. Plate precursors are shown in Fig. 3b.
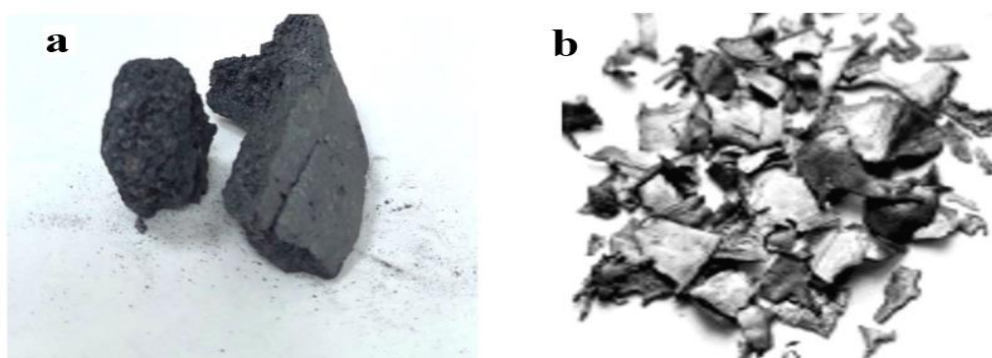

Figure 3.
Chipped pieces of garnish (a); plate-precursors, obtained by hardening melt in dispersant (b).

Plate precursors` microstructure is represented by oriented nanocrystallites surrounded by an amorphous phase. XRD patterns show crystalline and traces of the X-ray amorphous phases (Fig. 4 a, b, c).
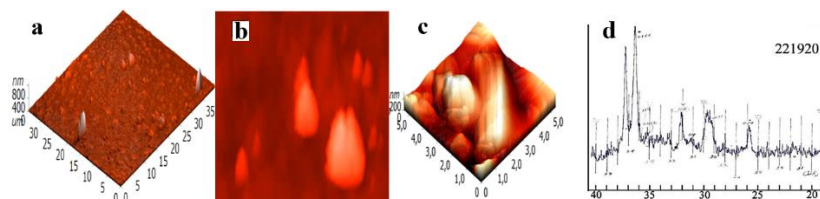

Figure 4.
Oriented nano crystallite nuclei, surrounded by amorphous phase (a, b, c); XRD patterns of precursor with nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=20 (d).

The structure of a ceramics, which is made of glass precursors, consists of mutually disassembled block plates, showed in Fig. 5a. The unit block comprises layers-plates separated by nano-dimensional inter-grain boundaries (Fig. 5b). Such morphology is formed based on the oriented layered structure of crystallite germs shown in Fig. 4 a, b.
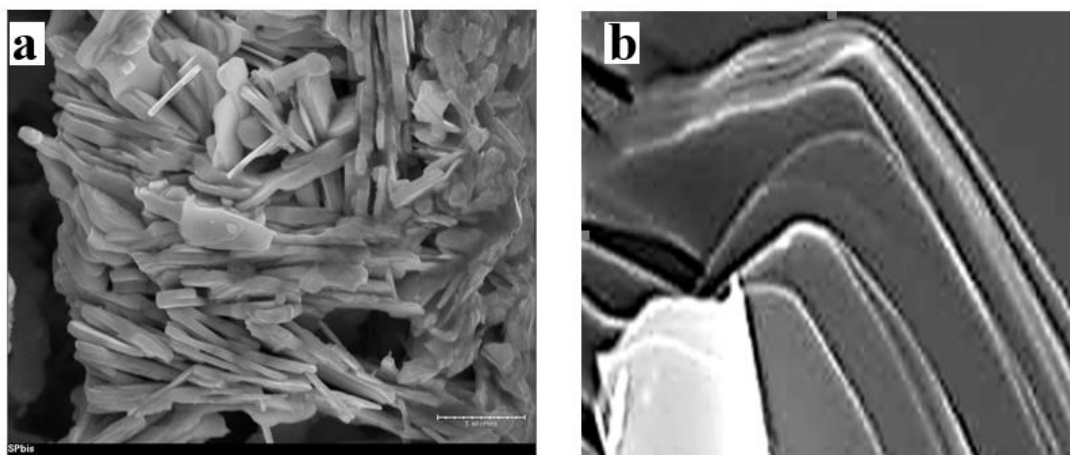

Figure 5.
Disoriented ceramic chip microstructure (a); Laminated unit microstructure with nano-dimensional grain boundaries (b).

The phase ceramic composition of ceramics represented by homologous phases of a series $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=20. For example, in Fig. 6 the diffractogram of ceramics of nominal composition "n=20" is shown with fragments representing a series of reflexes with close values 2θ, indicated in the phase homologs assumption. The parameters of the phase homolog elementary cell given in Table 1 are calculated by extreme and middle reflexes.
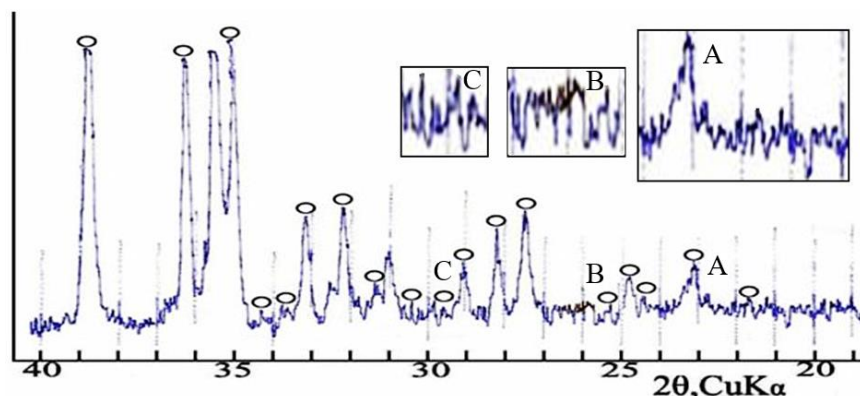
Figure 6.
Diffractogram of ceramics with nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=20 with fragments of phase-homologs.

Table 1.
Parameters of the elementary cell of phase homologs of series $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=20, calculated from a low angle, large angle, and mean reflexes.

Composition	Parameters of a unit cell according to the extreme small-angle reflection		Parameters of a unit cell according to the average reflection		Parameters of a unit cell according to the extreme high angle reflection	
2223	$a_{av.}=3,8609$ $b_{av.}=3,8024$ $c=37,2877$	$c/a=9,6578$ $c/b=9,8064$	$a_{av.}=3,8424$ $b_{av.}=3,7948$ $c=36,9936$	$c/a=9,6277$ $c/b=9,7485$	$a_{av.}=3,8180$ $b_{av.}=3,7782$ $c=36,7764$	$c/a=9,6324$ $c/b=9,7338$
2245	$a_{av.}=3,8955$ $b_{av.}=3,8227$ $c=49,5872$	$c/a=12,7294$ $c/b=12,9718$	$a_{av.}=3,8396$ $b_{av.}=3,8014$ $c=49,3792$	$c/a=12,8605$ $c/b=12,9897$	$a_{av.}=3,8250$ $b_{av.}=3,8075$ $c=49,0224$	$c/a=12,8163$ $c/b=12,8752$
2267	$a_{av.}=3,8523$ $c_{av.}=61,9267$	$c/a=16,0723$ $c/b=16,0150$	$a_{av.}=3,8375$ $c_{av.}=61,4054$	$c/a=16,0014$ $c/b=16,0311$	$a_{av.}=3,8022$ $c_{av.}=61,1119$	$c/a=16,0728$ $c/b=16,0550$
2289	$a_{av.}=3,8549$ $b_{av.}=3,8351$ $c=74,5755$	$c/a=19,3456$ $c/b=19,4455$	$a_{av.}=3,8293$ $b_{av.}=3,8206$ $c=74,0928$	$c/a=19,3489$ $c/b=19,3930$	$a_{av.}=3,8262$ $b_{av.}=3,7805$ $c=73,2240$	$c/a=19,1375$ $c/b=19,3689$
221112	$a_{av.}=3,8512$ $b_{av.}=3,7816$ $c_{av.}=92,6445$	$c/a=24,0560$ $c/b=24,4988$	$a_{av.}=3,8140$ $b_{av.}=3,7905$ $c_{av.}=92,2837$	$c/a=24,1960$ $c/b=24,3460$	$a_{av.}=3,7667$ $b_{av.}=3,8199$ $c_{av}=91,7694$	$c/a=24,3633$ $c/b=24,0240$
221415	$a_{av.}=3,8824$ $b_{av.}=3,8355$ $c_{av.}=111,3436$	$c/a=28,6791$ $c/b=29,0297$	$a_{av.}=3,8583$ $b_{av.}=3,8130$ $c_{av.}=110,7014$	$c/a=28,6918$ $c/b=29,0326$	$a_{av.}=3,8311$ $b_{av.}=3,8023$ $c_{av.}=110,0950$	$c/a=28,7372$ $c/b=28,9548$
221920	$a_{av.}=3,8382$ $b_{av.}=3,8759$ $c_{av.}=142,4241$	$c/a=37,1070$ $c/b=36,7461$	$a_{av.}=3,8264$ $b_{av.}=3,8436$ $c_{av.}=141,9174$	$c/a=37,0890$ $c/b=36,9230$	$a_{av.}=3,7731$ $b_{av.}=3,8428$ $c_{av.}=140,5681$	$c/a=37,2553$ $c/b=36,5796$
222425	$a_{av.}=3,8657$ $b=3,8162$ $c_{av.}=141,6423$	$c/a=36,6408$ $c/b=37,1161$	$a_{av.}=3,8287$ $b=3,8472$ $c_{av.}=140,7450$	$c/a=36,7605$ $c/b=36,5838$	$a_{av.}=3,8032$ $b=3,8220$ $c_{av.}=140,0777$	$c/a=36,8315$ $c/b=36,6504$
222930	$a_{av.}=3,8584$ $b_{av.}=3,8475$ $c_{av.}=141,2492$	$c/a=36,6082$ $c/b=36,7119$	$a_{av.}=3,8439$ $b_{av.}=3,8282$ $c_{av.}=140,7273$	$c/a=36,6106$ $c/b=36,7607$	$a_{av.}=3,8090$ $b_{av.}=3,8251$ $c_{av.}=140,3816$	$c/a=36,8552$ $c/b=36,7001$

The resistance and voltage of ceramics of all nominal formulations is affected by the temperature-time firing mode at normal atmospheric pressure. The electrophysical properties were studied using resistive 4-pin [28] and magnetic inductance [29] methods. These methods determined voltage, current, and resistance with a Rigol DM3058E multimeter. The temperature is determined by chromel-copel and chromel-alumel thermocouples, graduated in boiling water and ice. When a sample is placed within of sensing coils of an ac susceptometer [29] the voltage balance is disturbed and measured voltage U is proportional to the susceptibility of the sample. In the graphs of resistance R and voltage U, measured on the same samples, the anomalous effects are repeated in similar temperature intervals of 280-300 K (Figs. 7-10).

The resistance of ceramics obtained in a temperature range of 840-850ºC, by firing during less than 10 hours, represents a resistance drop characteristic of a semiconductor with a temperature increase in the range of 80-320 K (Fig. 7a). After increasing the firing time at 18 hours, the smooth dependence of R(Ω)-TºC (Fig. 7a) is broken, the effects of resistance drop in the temperature range 220-240 K and increased resistance at 310 K (Fig. 7b) appear.

The anomalous effect at 310 K may be due to the influence of the superconducting phase.
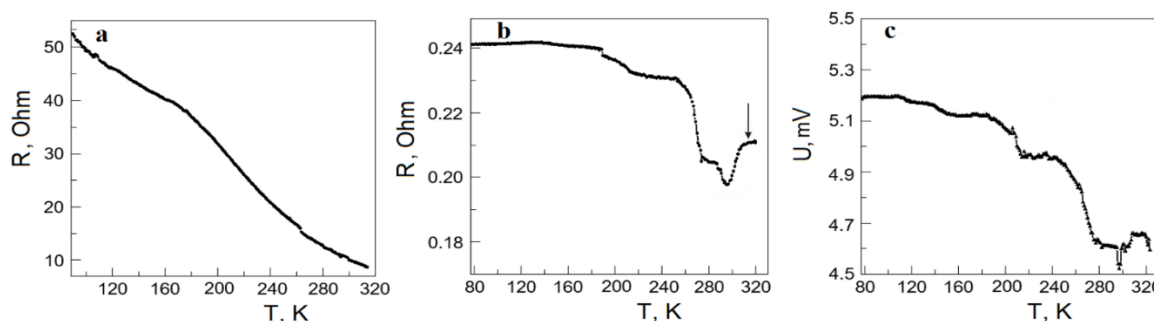
A

Figure. 7.
Dependences of resistances (a, b) and voltage (c) on temperature of cuprate ceramics of nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=20, obtained at a temperature of 840-846ºC, at firing ≈10 h.(a) and ≈18 h (b, c).

The resistance and voltage tension of ceramics obtained by firing for 30-48 hours represents almost constant dependence with weak abnormal deviations near 310K (Fig. 8 a, b).
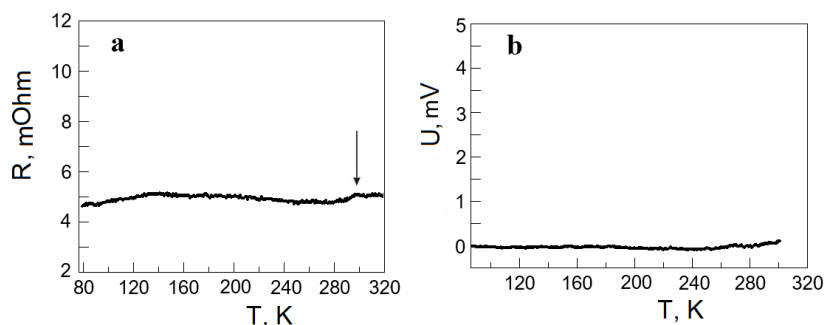

Figure 8.
Dependence of resistance (a) and voltage (b) on the temperature of ceramic cuprates of nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=20, obtained at a temperature of 840-846ºC and firing time ≈30-48 hours [27].

Almost a full-volume content of phases with T_c=120-152 K is obtained in ceramics, fired during 115 h (Fig. 9 a, b). Deviations from the linear relationship occur in a temperature range of 300-320 K
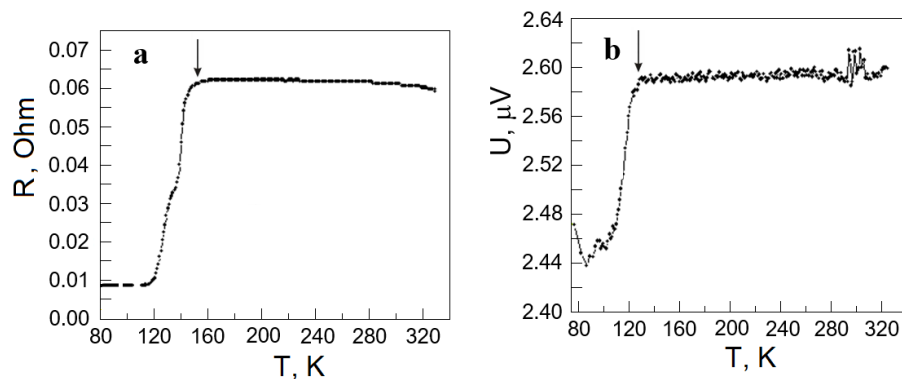
Figure 9.
Dependence of resistance (a) and voltage (b) on the temperature of ceramic cuprates of nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=20, obtained at temperatures 840-846°C, 115 hours.

In firing mode 843-847°C, 20-24 hours ceramic samples with anomalies above 260K in high temperature area were obtained. The resistance and voltage dependences on temperature are shown in Fig. 10 a, b indicating effects near 270 K and 300 K.

These anomalies consist of the torsion oscillation determined of superconducting phase at T_c=295 K [23]. A resistance other than zero could be related to the percolation effects, i.e. the microstructure of the ceramic ship (Fig. 4a) shows a violation of strictly oriented microstructure.
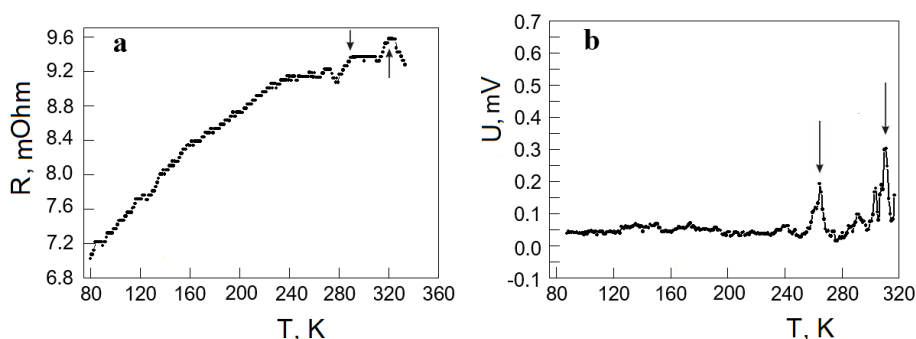

Figure 10.
Resistance (a) and voltage (b) of ceramic made in 843-847°C mode, 20-24 h.

Confirmation of the formation by the superconductive phases in a temperature range above 280 K may be a sample ejection effect at room temperature and normal atmospheric pressure (Meissner effect) shown in Fig. 11 a, b, c.
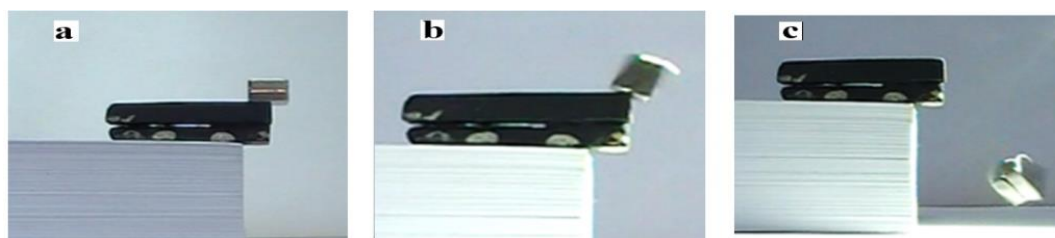

Figure 11.

Ejection of the magnet by a ceramic sample of nominal composition $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=20 at room temperature, normal atmospheric pressure, under the influence of light (a), (b), (c) [23].

The new result is the ejection effect of the magnet from the ceramic of all nominal of the $Bi_{1.7}Pb_{0.3}Sr_2Ca_{(n-1)}Cu_{(n)}O_y$, n=3-20 range at room temperature and normal ambient pressure, daylight (Fig. 11a,b,c) and white light source. Ceramics exhibit no ferromagnetic properties, so the effects of ejection of the magnet from the samples (Meissner effect) in comparison with anomalies on graphs R(Ohm)-T(K) at room temperature and normal pressure could be explained by the assumption of superconducting phases.

3. Conclusion

Depending on the reaction of the formation of the material of a given composition by the variation of the density of the solar flux in the focal zone of the LSF (Parkent), materials with different melting temperatures, formed according to different types of reactions - congruent or incongruent can be synthesized. The properties of isostructural and isostochiometric oxide groups of different compositions showed certain advantages of solar technologies: completeness of synthesis, purity of process, suppression of peritectic reaction, etc. On the example of Bi/Pb cuprate, the possibility and advantages of green "sun" technology for the production of Bi/Pb superconducting cuprate ceramics have been determined. The developed "SFAQ" gradient technology allows us to obtain positive results, namely to synthesize full-volume superconducting ceramics with T_c=110 K and to obtain room-temperature superconducting phases with T_c 280-317 K. The Bi/Pb cuprate synthesis requires of the peritectic interaction between the initial components. Optimum solar technology meets the important requirement of superconducting materials - the formation of oriented crystallites-embryos that are the innate basis of the nano-dimensional plate-oriented ceramic microstructure and the "freezing" of the amorphous eutectic involved in the superconducting phase formation reaction. For the first time, the microstructure of ceramics represented by superconducting homologous phases was obtained by solar technology. Different stoichiometry of phase homology determines the potential difference and, accordingly, the charge concentration at the inter-grain boundaries. In the case of such a process, it is possible an assumption about the negative influence compensation by crystal lattice on conduction electrons. The increase in charge density is directly related to the increase in the Cooper pairs density and critical properties. The mobility and advantages of "green" technology make it possible to offer solar technologies for both the research and synthesis of experimental and industrial batches of materials of a given composition. The use of "solar" technology for the synthesis of materials used in the film technology is advisable, since the average productivity of LSF (Parkent), ranging from 20-30 kg/solar day, can provide raw material for the industrial production of film equipment, chips, etc.

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