The Effect of Pb Dopant on The Critical Temperature of BSCCO-2212 Superconducting Crystal

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Abstract A reseach about the study on the critical temperature of BSCCO-2212 superconducting crystal with slow cooling periode of 90 hours objected for knowing the critical temperature of superconducting crystal. An experiment has been carried out on the formation of BSCCO-2212 superconducting crystal by the melt-textured growth at 930^0C. The syntheses were conducted with the molar ratio of the Pb dopant varied between 0 and 0.4 , while the period of the slow cooling process fixed at 90 hours. Characterization of samples with the curve R-T that sample with Pb dopant ratio 0 has the highest critical temperature of 60K and sample with Pb dopant ratio 0.2 and 0.4 has the critical temperature of 57K and 52K respectively.
Keyword : superconductor, Pb dopant, critical temperature

Introduction

Superconductors are materials that conduct electricity have zero resistivity when it is below a certain temperature, called the critical temperature of these materials (Cyrot-Pavuna,1992). Since the discovery of high critical temperature superconductors (HCTS) in 1986, superconducting technology is growing very rapidly. One of the many SKST material studied is a multicomponent system Bi-Sr-Ca-Cu-O, also known as BSCCO superconductors. Interest is related to the high critical temperature Tc of the system and does not contain rare earth elements (Rare Earth) are expensive. Despite having a lower Tc of Tl and Hg-based system, this system does not contain toxic elements. Superconducting BSCCO powder commonly used as a filler tube which in turn silver after a few stages thermomechanical treatment result was obtained in the form of superconducting tape is applied as a current conductor (superconducting wire). BSCCO superconductors consists of three phases and has the chemical formula $Bi_2Sr_2Ca_{n-1}Cu_nO_x$ complete (n = 1,2,3; called BSCCO-2201, BSCCO-2212 and BSCCO-2223). In the family it is known that the structure of superconducting BSCCO-2212 is more stable and has problems weak links lower than BSCCO-2223 (O.Berdan,2012). BSCCO-2212 phase also has a critical current density Jc higher than the liquid nitrogen temperature phase BSCCO-2223 (Verma, 2012). Ease of forming compounds in the solid phase polycrystalline and availability of appropriate methods in growing crystals make BSCCO-2212 is often used as a model for the study of Bi-based superconductors (Darminto, 2002).

In the normal state, superconducting materials showed a decrease in electrical resistivity that is almost comparable to the decrease in temperature. However, the resistivity curve to temperature (RT), which is characteristic of a superconducting material shows that there is a price at which the temperature is the temperature value of the resistivity R suddenly fell very sharply (rapidly). In Figure 1, the temperature at the start of the decline is very sharp resistivity is shown as Tconset. The temperature at which the resistivity starting valuable zero (superconducting) symbolized by Tc zero. The difference between zero and Tc Tconset named as the width of the resistive transition (ΔTc) material in question.

BSCCO-2212 material has a layered crystal structure and composition as a result plural phases multicomponent compounds. The layered structure of the general form alternating arrangement, the cube "perosvkit" containing superconductive CuO_2 layer and the insulator layer Sr-O (Figure 2). CuO_2 layers are separated by a double layer lining with single cations Ca. Each Cu atom bonded to O atom nearest neighbors to form a pyramid configuration coordinate five. Separator layer consists of alternating (Sr-O) - (Bi-O) - (Bi-O) - (Sr-O).

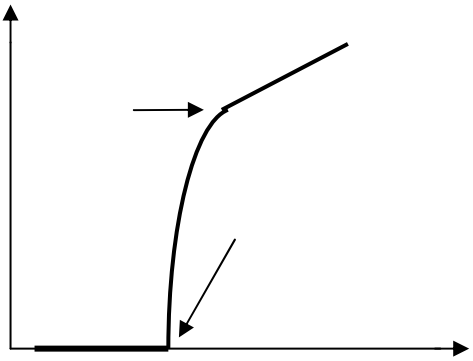


Figure 1. Curve R-T superconducting

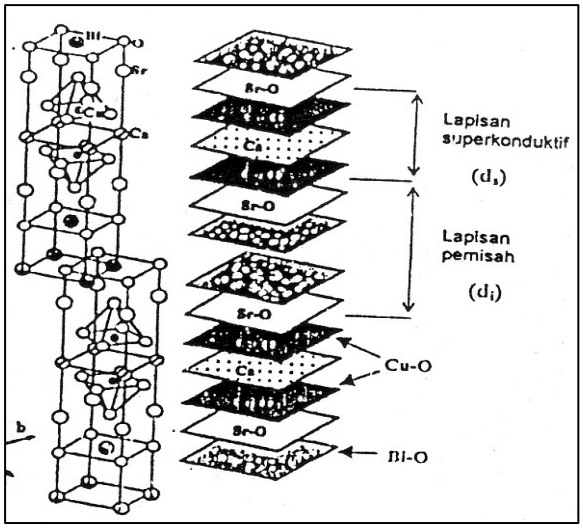


Figure 2. Layered structure of superconducting crystals BSCCO-2212 (P. Majeswki, 1994).

To improve the properties of the material superconductive, has done research on the effects of dopants on the superconducting material. Apparently dopant plays an important role in the formation of BSCCO-2212. Dopants can be a substitution means replacing the original atoms in superconductors with dopant atoms whose size is not much different from the size of the original atoms, or dopants may also be additional means adding dopant atoms into the original atoms superconductors. In addition to oxygen dopants, have also done studies using Pb dopants. The use of Pb dopant atom substitution apparently resulted in Bi by Pb atoms in the Bi-O double layers in the crystal structure. This is due to the similarity measure of ions and valence values of Pb atoms with Bi atoms (Lide-Frederick,1993).

The use of Pb dopant in the synthesis of polycrystalline Bi system in addition to facilitate the formation of the compound concerned, also affects the properties of the resulting compounds [7]. In addition to increased levels of Pb in superconducting BSCCO-2212 single crystals were prepared by the method TSFZ (the Travelling Solvent Floating Zone) are known to have lower critical temperature Tc and normal state resistivity.Synthesis BSCCO crystal samples can be done by different methods such as the method of The Travelling Solvent Floating Zone (TSFZ), alkali halide flux method, alkali carbonate flux method, the method of Self Flux and Melt Textured Growth method. Method Melt-Textured Growth (MTG) is a method of formation of the superconducting crystals discharge (Darminto, 2009). The driving factor in the process of crystal growth methods MTG is cooling (cooling).

In Figure 3, the starting material with a chemical composition that meets the stoichiometry A_2B melted to a temperature above the peritectic temperature Tp. As a result, the A_2B compound breaks down into a mixture of solids and liquid composition range of variation among A2B and X_p (peritectic composition). If the temperature is then lowered slowly so that the cooling process is slow (slow cooling), it will form a mixture of crystals and liquid A_2B range of variation between X_p and X_e composition (eutectic composition) in the range of temperature variation between T_p and T_e (eutectic temperature) . This shows that in order to grow single crystals of the variation in the composition of the melt A_2B must still be maintained between X_p and X_e. For BSCCO-2212, it means that only pure substances can be grown from a melt composition different from the composition variations BSCCO-2212.

Through these experiments will be known critical temperature superconducting BSCCO-2212 crystals that form on every variation of Pb dopant with a molar ratio of 0, 0.2, and 0.4 are used in the synthesis of crystals with MTG method.

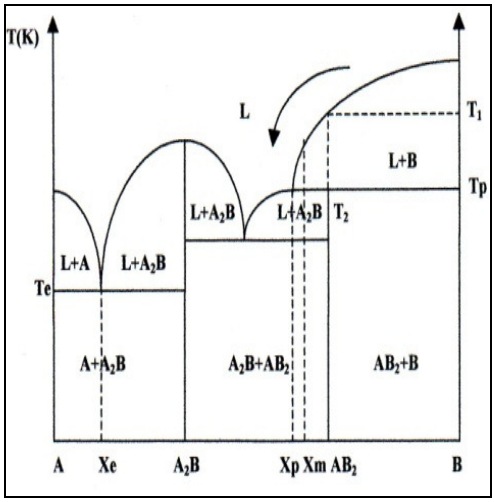


Figure 3. Principles of Crystal Growth Method with MTG

Materials and Methods

Equipment and materials

The equipment used was a tube furnace, furnace rectangular, Sartorius balance, beaker glass, mortal and pastel ceramics, alumina krucibel, and mold samples. Material used is Bi_2O_3 (99.9%), PbO (99.9%), $SrCO_3$ (99 995%), $CaCO_3$ (99.0%), CuO (99.99%), HNO_3 (65.%), Distilled water, and acetone Ag silver paste.

Process Synthesis

All samples in this experiment on the synthesis method MTG. This method is preceded by a two-stage solid reactions, namely:

-The first stage is to prepare the precursor without Ca according to the nominal composition $Bi_{2-x}Pb_xSr_2Cu_2O_y$ as listed in Table 1.

Table 1. Composition Precursor without Ca

No	Composition of precursor without Ca	Levels of Pb (ratio of molarity)
1	$Bi_2Sr_2Cu_2O_y$	0
2	$Bi_{1.8}Pb_{0.2}Sr_2Cu_2O_y$	0.2
3	$Bi_{1.6}Pb_{0.4}Sr_2Cu_2O_y$	0.4

All material in the form of powder was weighed and mixed according to the composition listed in Table 1. This mixture is then diluted with a solvent assistance HNO_3 and distilled water and stir until a solution is completely homogeneous. Furthermore, the solution is heated to evaporate the mixture of HNO_3 to obtain a blue-black blobs. To ensure that the mixture is called precursor really have free HNO_3 is necessary to do the drying in the furnace. Further grinding manually until the material becomes smooth as talc powder. The precursor form of fine powder is then molded into pellets and calcined at a temperature of 7500C for 40 hours. After calcined precursors were destroyed and crushed recycled back for about 2 hours.

The second step is to mix the powder CaO precursors. CaO powder used here is obtained by deposition of $CaCO_3$ compound. $CaCO_3$ compound deposition process to obtain the compound CaO done with regular solid reaction preceded by solvent mixing distilled water

and HNO_3. The composition of the sample is a mixture of precursors with CaO powder is as indicated in Table 2. Precursor powder mixing with CaO done with the help of acetone until completely homogeneous solution is obtained. Furthermore, acetone was evaporated until mixture is completely free of acetone. Back material is finely ground and molded form of pellets. Then a sample of these pellets are melted in a furnace at a temperature of 9300C for 20 minutes and finally experiencing slow cooling for 90 minutes until it reaches room temperature.

Table 2. The composition of the precursor mixture with CaO

No	composition	Levels of Pb (ratio of molarity)
1	$Bi_2Sr_2CaCu_2O_y$	0
2	$Bi_{1.8}Pb_{0.2}Sr_2CaCu_2O_y$	0.2
3	$Bi_{1.6}Pb_{0.4}Sr_2CaCu_2O_y$	0.4

Data analysis

In all the samples was measured using RT curve CTI-Cryogenics equipment consisting of Refrigeration Cryodine 22/350C the cooling power 10 Watt, 8200 Helium compressor water-cooled, and R measurement system with four electrodes (four point probe). Electrode contacts were made parallel to the sample surface with the distance between the electrodes is uniform. The system is capable of measuring temperatures R to about 10K. Voltage measurement is done by using a Keithley Source Meter 2400 and Keithlry Nanovoltmeter 2182. The temperature is measured with a Si-diode sensor LakeShore DT421, while the heater is made of wire Manganin (54.5 Ohm / m) and monitoring is done by LakeShore 330 Temperature Controller. Temperature stability of the system can reach 200 mK. The entire set of data communication lines and controlled by a PC via an IEEE-488 connection GBIB, based Test Point program. Working principle is schematically as shown in Figure 4.

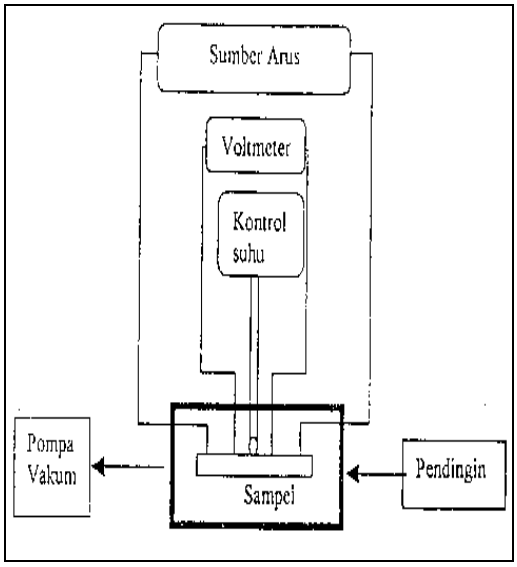
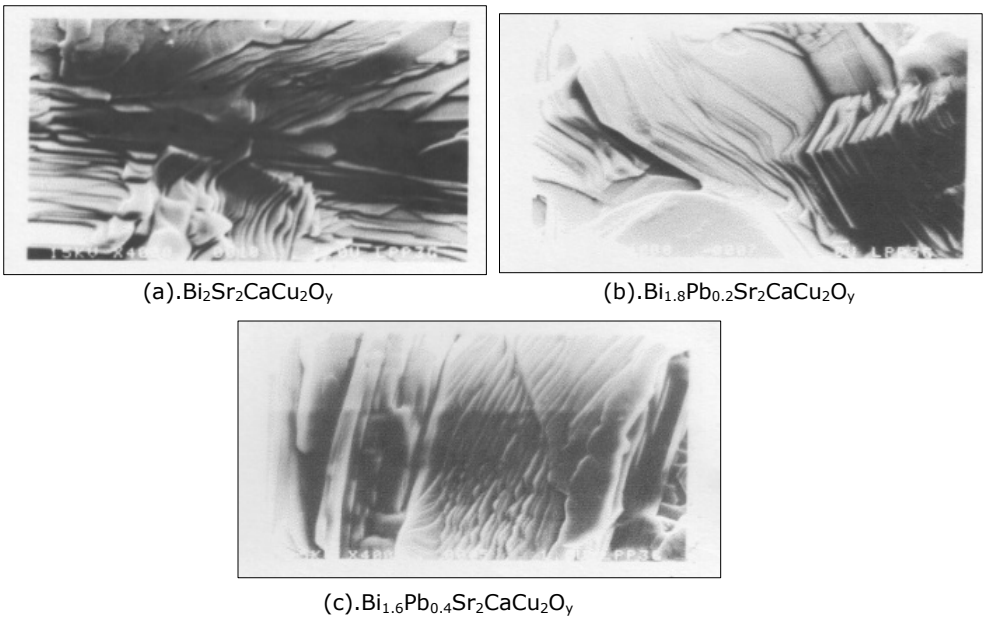


Figure 4. Cryogenic Equipment Scheme for Measuring Curve RT

Results and Discussion

All samples were initially form before disintering pellet diameter of about 1 cm with a thickness of about 3 mm. Once thawed samples obtained in the form of thin plates with a thickness of less than 3 mm, the upper surface is grooved uneven and irregular wide size. The results of the sample surface SEM photo in Figure 5 shows that items such as the formation of superconducting crystal composition layer sheets of paper.



(a).$Bi_2Sr_2CaCu_2O_y$ (b).$Bi_{1.8}Pb_{0.2}Sr_2CaCu_2O_y$

(c).$Bi_{1.6}Pb_{0.4}Sr_2CaCu_2O_y$

Figure 5. SEM photos

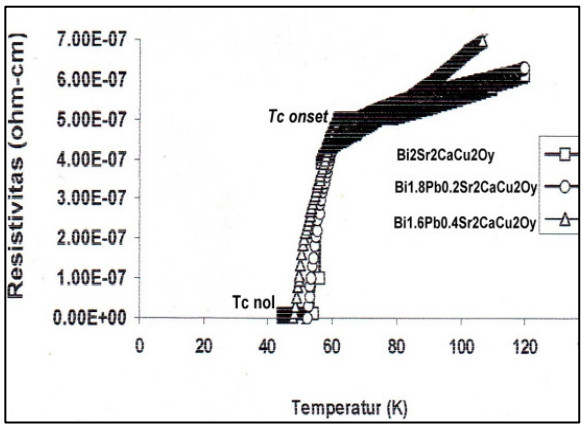


Figure 6. The Resistivity curves for Temperature (R-T) of all samples

The RT curve measurement results showed that all the ingredients have to be superconductive with the sharp transition as shown in Figure 6. In the RT curve (Figure 6) shows that for the sample without Pb dopants obtained Tc onset of 60K and the temperature at which the resistivity values to zero or zero Tc is 56K. This means that when the temperature of the material down the resistivity materials also declined, and when the temperature reaches 60K, the value of resistivity suddenly decreases dramatically / sharply and continued to decline until it finally reaches zero at a temperature of 56K. ΔTc resistive transition width for this sample is 4K. For samples with dopant levels of Pb 0.2 obtained Tc onset of 57K and 53K Tc zero. It also shows that when the temperature decreases the resistivity of the material also decreases and when the temperature reaches 57K resistivity values decreased sharply that eventually reaches zero when the temperature reaches 53K. While the sample with dopant levels of Pb 0.4 shows prices 52K Tc onset and Tc zero at 48K. It is also clear that when the temperature drops then the resistivity of the material is also down, and when the temperature reaches 52K resistivity material decline sharply and continued to decline until it finally reaches zero at a temperature of 48K. These two latter samples also showed the same resistive transition width as the sample without Pb dopant is

4K. The width of the resistive transition is relatively small due to the very sharp decline in the transition area at the RT curve. All prices are measured from samples in this study are summarized in Table 3.

Table 3. Data variable characteristics of the sample

No	Komposisi sampel	Pb (ratio of molarity)	Tc Onset (K)	Tc zero (K)	ΔTc (K)
1	$Bi_2Sr_2CaCu_2O_y$	0	60	56	4
2	$Bi_{1.8}Pb_{0.2}Sr_2CaCu_2O_y$	0.2	57	53	4
3	$Bi_{1.6}Pb_{0.4}Sr_2CaCu_2O_y$	0.4	52	48	4

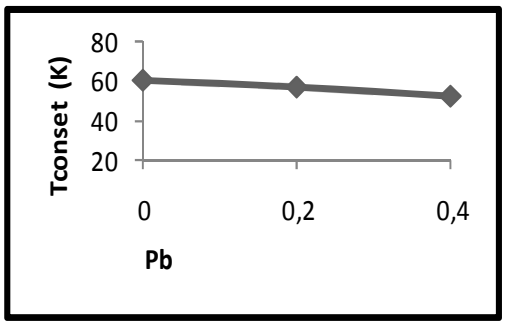


Figure 7. Tc onset of the levels of Pb

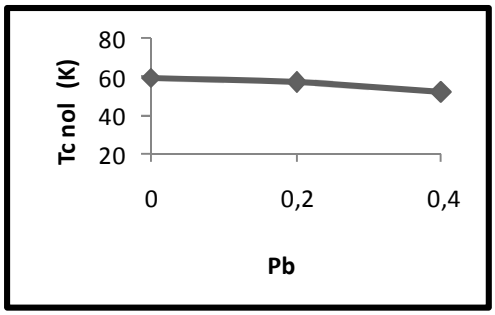


Figure 8. Tc zero of the levels of Pb

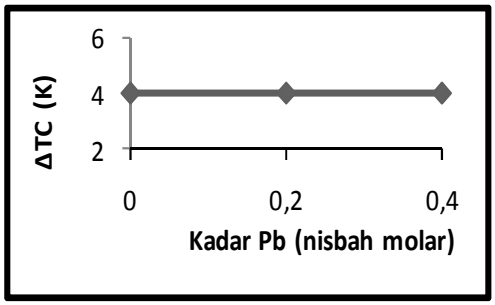


Figure 9. ΔTc onset of the levels

Based on the analysis of the data it can be seen that the samples with slow cooling 90 hours with the addition of Pb levels actually have lower critical temperature of the crystal. This is caused by the melting of the incongruous nature of the material. In addition there is a possibility due to the amount of Pb too much so that the concentration of Cu and Ca concentrations in the sample decreased resulting in increased impurity, both impurities at low Tc phase or impurities were nonsuperkonduktif. This is consistent with experimental results that have been reported despite the use of different methods (O.Berdan , 2012). Additionally, the declining value of critical phenomena can be caused also by the formation of crystal defects caused by the presence of dopant.

Conclusions

Dopant Pb lower critical temperature of the 2212 phase is formed in this study. Highest Tc onset and Tc zero 60K for 56K found in samples without Pb while Tc onset owned 52K sample with the lowest levels of Pb 0.4. Furthermore, it is known that Pb dopants do not affect the sharpness of the curve RT. For all the samples obtained by the resistive transition width 4K relatively narrow so that the RT curve shows a very sharp transition when the material change from the normal state to the superconductive state.

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