PS-014

INFLUENCE OF SINTERING TIME ON THE PROPERTIES OF HIGH TEMPERATURE SUPERCONDUCTOR BPSCCO BASED

Eidi Sihombing

Jurusan Fisika
Fakultas Matematika dan Ilmu Pengetahuan Alam
Universitas Negeri Medan

ABSTRACT

The influence of the sintering time of the high temperature superconductors BPSCCO was investigated, Samples were prepared using the solid state reaction. In this study, superconducting BPSCCO ceramics with a fix nominal composition were prepared and sintered at 850^0C for different periods. The structure phases of the sintered ceramics were determined by means of the measurement of the resistivity temperature behaviour and of the XRD analysis. TheX-ray diffraction patterns indicated that: The samples had high quality at the long sintering time. The large interaction between grain boundaries of the two phases happened and it can lead to the variation of lattice parameters. Pb in the BSCCO ceramics were observed to increas the volum fraction of the high Tc phase. The critical temperature for the 100 hour sintered sample was 4 K higher than that of the 60 hour sintered sample.

Keywords: *Sintering time, superconductivity BPSCCO*

INTRODUCTION

Since Maeda discovered superconductivity in the BiSr CaCuO (BSCCO) system, (Maeda et al., 1988), significant research has been focused on the synthesis and the used of various dopant to increase the critical temperature (Tc) of these ceramics in addition to increasing the volume fraction of the high Tc phase in multiphase ceramics. It has been shown that substituting Bi partially with Pb enhances the formation of the high Tc BSCCO phase (Abbas et al., 2012). Current research indicates that ceramics which are a mixture of low and high Tc phases are formed by doping with Pb. The possibility of the existance of a new phase with 85 K low Tc and 110 K high Tc phase was also reported in that work which was the highest Tc reported in the BSCCO system up to that moment.

Superconducting properties can be improved by changing the phase composition, by variation of the quality of grain boundries but also and mainly by changing the macroscopic and microscopic ordering of grains, theis sizes and orientation. Another way of improvement is addition of microscopic particles to the material as possible magnetic flux pinning centre (Misko V.R and Nori F., 2012).

There are three superconducting phases in the Bi Pb Sr Ca CuO superconductor marked according to their stoichiometry as 2201, 2212 and 2223 with critical temperature of about 107 K (Meretliev Sh, 2000). An influence of the addition of nancalcined portion of the mixture to the calcined mixture before sintering on the superconducting proprties.

All heat treatments were carried out with alumina cruicibles in a turbular furnace. The phase contents and the microstructure were analized through X-ray diffraction (XRD) with λ Cu Kα radiation and Scanning Electron Microscopy (SEM). Superconducting materials show electric resistance at temperature close to zero kelvin and they are able to transport electric current without losses. These materials are divided into low and high temperature superconductors.

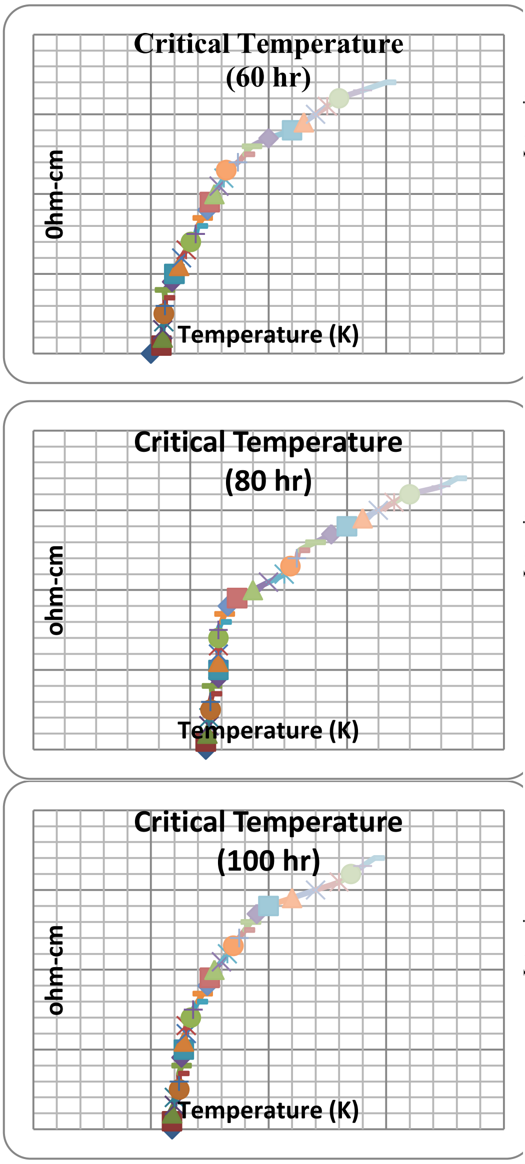
METHODOLOGY

The BPSCCO ceramics were prepared by the conventional ceramic method. Predetermined amounts of high purity 99,99% aldrich starting chemicals (Bi_2O_3; PbO, $SrCO_3$; $CaCO_3$; CuO) were used for the preparation of the ceramics of fixed nominal composition of Bi : Pb : Sr : Ca : Cu are 1.5 : 0.5; 2 : 2 : 3. These powder were well mixed and ground by using a mortar and pastel and it was further calcined at 850^0C for 25 hours inj a furnace. Cacined powders were ground again to form a fine powder. Pellets 15 mm in diameter and 1 mm in thickness were pressed by uniaxial compaction in a die at 500 Mpa. These pellets were sintered at 850^0C for 60, 80, 100 hours in air and then furnace cooled to room temperature. Resistance temperature data were obtained by using four point probe DC method. XRD pattern were obtained by using XRD SHIMADZU 6100 X-ray diffractometers. SEM micrographs were taken by using a ZEISS EVO MA 10.

RESULT AND DISCUSSION

The temperature dependence of the electrical resistivities of the $Bi_{1.5}Pb_{0.4}$ Sr_2 Ca_2Cu_3Oy ceramics sintered at 850^0C for 60, 80 and 100 hours for samples A,B, and C, respectively is given in Figure 1. The onset temperature for all samples is approximately 120 K. This is about 10 K higher than our previously work onset temperature for Pb doped BSCCO ceramics. The resistivity temperature curve is linier up to the onset temperature, in accordance with the metallic character of the samples. Zero electrical resistivity is obtained in a single step at 108, 110 and 112 K respectively for samples A, B and C. The transition width decreases with increasing sintering time which is indicative of better phase homogenity. Pb doped BSCCO ceramics increases the Tc 3-4 K in our work. It is possible to say that the volum fraction of the high Tc phase increases with increasing sintering time and the high Tc phase appears to be the dominant phase in these ceramics.

The XRD patterns of the samples are shown in Figure 2. All the peaks were identified by using the tabulated data.



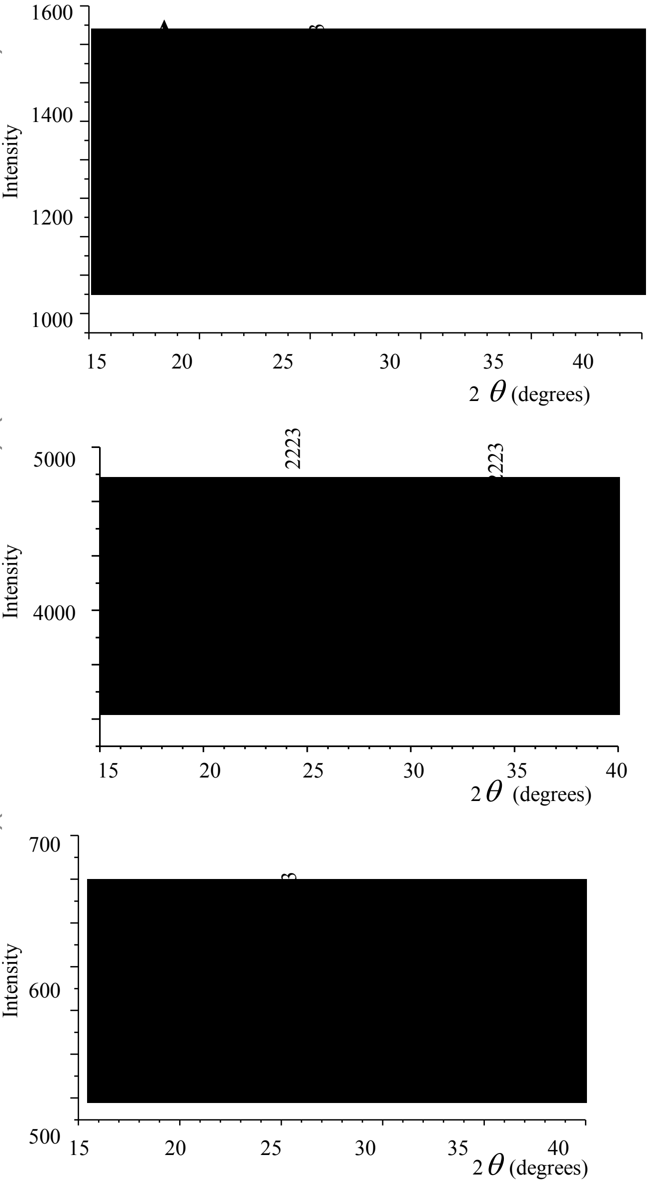


Figure 1. Temperature dependence of the electrical resistivities of the ceramics sintered at 8500C for 60, 80 and 100 hours for samples A,B, and C.

Figure 2. XRD Patterns of Samples A, B, and C

In figure 1, the electrical resistivities of the $Bi_{1.5}Pb_{0.4}$ Sr_2 Ca_2Cu_3Oy ceramics sintered at 850^0C for 60, 80 and 100 hours. Figure 2. The XRD patterns of the samples A, B and C after sintering 60 h, 80 and 100 h respectively.

The XRD analysis showed that the heat treatment causes a very rapid crystallization process. Quantitative analysis of diffraction patterns performed by the Rietveld profile refinement allowed us both to determine lattice parameters of the main crystalline phases (2201, 2212, Cu2 O and CaO) and to estimate their approximate weight fractions (Hermiz et al., 2014. The total amount of the crystalline phases change from about 60h to 100h. The cell parameters of 2201 phase did not change significantly with the sintering time and it was found to be: a = 5.388(2) Å, b = 5.316(2) Å, and c = 24.419(7) Å. In the case of 2212 phase average cell parameters were a = 5.372(1) Å , b = 5.411(1) Å, and c = 30.761(5) Å. The 2212 phase in the samples sintered for 60h, which indicates oxygen deficiency.

All the samples appear to have a multiphase structure which indicates the difficulty in obtaining a single phase material by the conventional ceramic method. Single phase ceramics were obtained by melt-quenching technique and under controlled atmospheres at shorter sintering periods. An analysis of the number of corresponding peaks and their intensities indicates that the high Tc phase is the dominant phase in all three samples in this work. The commonly observed Ca_2PbO_4 impurity phase at $2\theta = 17.8^0$ was also detected in all samples. Few peaks for CuO, Ca_2CuO_2 and Ca_2PbO_4 impurity phase in the $2\theta = 30\text{-}50^0$ range were detected but the number of these peaks and their intensities decreased with longer sintering time.

The most intense peak in XRD pattern of the samples C belong to the high Tc phase with sintering time. The intense low Tc phase peaks in the $2\theta = 30\text{-}35^0$ range in sample A can not be further detected in sample C. Two monoclinic phase peaks (d = 2.953 Å) were detected in all samples which was previously identified as non superconducting phases. This peak were observed to be more intense peaks in our earlier Pb doped BSCCO samples.

Microstructur of SEM images of the material sintered at 850^0C are presented in Fig. 3. The microstructure during the sintering depends on the temperature and time of the thermal treatment. It can be seen that the surface of the sample sintered at 850^0C is composed of crystallites of rather irregular, oval shapes, while that of the sample sintered at 850^0C contains plate-like crystallites.

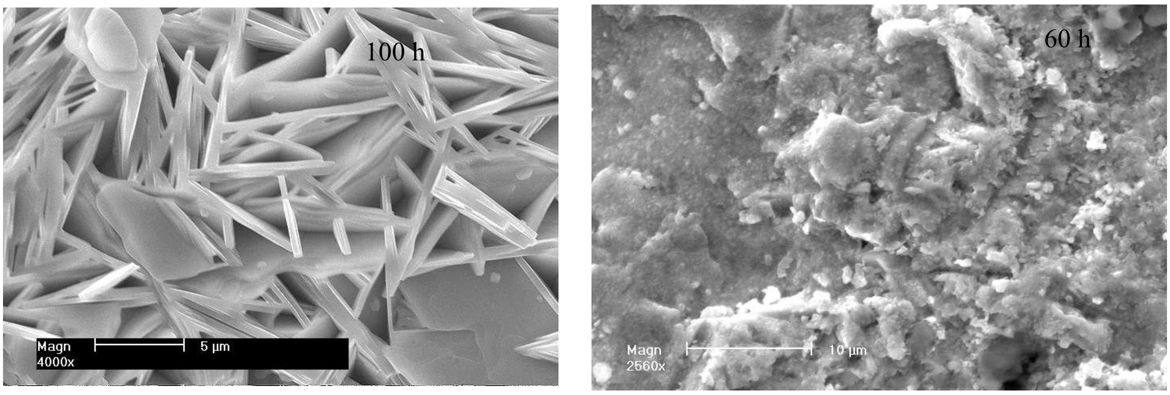


Figure.3. SEM images of the material sintered for 60h and 100 h at 850^0C.

Also different types of evolution of microstructure occur in the case of the samples sintered time at 850^{0}C. During the sintering at 60h the shape of the crystal grains remains the same (oval) and only their radius and number increase. The oval grains disappear while plate-like ones form. Further sintering leads to the growth of their dimensions. It can be also seen that the grains are partly ordered in such a way that their heights are parallel to the sample surface. On the other hand, long-time sintering results where large crystallites are not well connected between each other.

CONCLUSIONS

In the (Bi,Pb)-Sr-Ca-Cu-O system, materials of various electrical and superconducting properties may be produced by conventional method. Materials obtained by this method have very interesting properties and they constitute a new group of superconductors. Depending on the heat treatment conditions, either asuperconductor with the critical temperature between 108-112 K. Best superconducting properties can be achieved at the temperatures 850^{0}C and sintering time 60-100h.

REFERENCES

Abbas M.M., Abass L.K and Salman U., (2012), Influences of Sintering Time on the Tc of $Bi_{2-x}Cu_xPb_{0.3}Sr_2Ca_2Cu_30_{10+\delta}$ High Temperature Superconductors, *Energy Procedia* 18, 215-224.

Hermiz G.Y., Aljurani B.A., Beayaty M.A., (2014), Effect of Mn Substitution on the Superconducting Properties of $Bi_{1.7}Pb_{0,3}Sr_2Ca_{2-x}Mn_xCu_3O_{10+\delta}$, *International Journal Of Engineering and Advanced Technology (IJEAT)*, 3,4, 213-217

Maeda H., Tanaka Y., Fukutomi M., Asano T., 1998, A New High-r Oxide Superconductor Without a Rare Earth Element, *Jpn. J. Appl. Phys. Pt*. 2. 27: L209-10

Meretliev Sh., Sadykov K.B., Berkeliev A., (2000), Doping of High Temperature Superconductors, *Turk J Phy*, 24, 39-48

Misko V.R and Nori F., (2012), Magnetic Flux Pinning in Superconductor with Hyperbolic-Tesselation array of Pinning Sites, *Physical Review B*, 85, 184506

Scholten T, (2013), *Superconducor $Bi_{1.5}Sb_{0.5}Te_{1.7}Se_{1.3}$ Topological Insulator Hybrid Devices,* Master Thesis Faculty of Science and Technology Quantum Transport in Matter Interfaces and Correlated Electron System, University of Twente..