Phase Formation Study Of BSCCO-2223 with nano-γAl_2O_3 Addition at Low Sintering Time

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1. Introduction

The ceramic base oxide superconducting properties become popular to discover since 1986 when Berdnorz and Muller found metal oxide ceramics material can posses higher critical transition temperature, T_c. Superconductor has their own properties inter-related each other such as temperature, magnetic field and critical current, I_c. The problem arose when superconductor material fail to conduct large electric especially in high magnetic field. Over past decades, a lot of study raised to overcome this problem. Focusing on I_c, BSCCO-2223 shown the best ceramic can carry large current especially in tape form. Highly density of superconducting core, highly phase pure, large grain size and highly textured microstructures needed to increase the ability to carry large critical current density. In order to get perfect characteristics, previous work shows many trials [5]. Studies had show that the transition temperature for BSCCO system depends on powder production and also sintering hours [3] BSCCO powder highly textured when temperature of calcinations varies from 800^{o}C to 850^{o}C. The calcinations powder is an important features led to the best superconductor production. In this paper, BSCCO powder produced through COP method that promises ultrafine powder. Low calcinations temperature hopes grow the grains in almost perfect growth. Less study report the COP technique, the starting precursor powder preparation. Techniques of preparation are crucial subject to be discussed in this paper which it creates high dependence on starting materials. The COP method has the advantage of better chemical mixing than the solid state reaction method by mixing all cation species on an atomic scale [4]. With this method, the powder reactions gain an advantage as it shortens reaction time and lowers reaction temperature. As a result, small grain size produced and obtained homogenous microstructure. Superconducting phase important in processing material to make sure ceramics compound will initiate as superconductor material. Researchers have been study a lot of material with superconducting properties. By the ages of study, improvement of compound becomes extensive. Therefore, the addition of nanoparticles into bismuth based system was introduced. Several report on addition nanoparticles were proudly present thus enhance the capability superconductor properties, such as addition of nano-MgO, nano-SiC, nano-γFe_2O_3 and others. This paper revealed the formation of BSCCO-2223 with addition γAl_2O_3 as impurities via co-precipitation method.

2. Experimental Procedure

Precursor powder with nominal 2223 composition such $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_x$ was prepared by co-precipitation method mixing of $Bi(CH_3COO)_3$, $Sr(CH_3OO)_2$, $Pb(CH_3OO)_2.3H_2O$, $Ca(CH_3COO)_2$.H2O and $Cu(CH_3COO)_2$ (purity >99%) with acetic acid to form solution (A). Another composition where oxalic acid was dissolved in water:isopropanol (1:1.5) to have concentration of 0.5M named as solution (B). Solution B was poured into stirred solution A in an ice bath and uniform blue suspension was obtained. The slurry was filtered after 10 min of reaction. The precipitated was dried at 80^{o}C for 8h. The dry powder, which is slightly distributed with particle size of 0.1 – 0.6 μm was grounded undergo pre-calcinations process of 12h at 730^{o}C in air to remove the remaining volatile materials. The calcined powder was reground in a marble mortar and heated at 845^{o}C in air for 24h. The powders were reground and add with nano-γAl_2O_3 with 0.2, 0.5 and 1.0 wt % before pressed into pellets of ~12.5mm diameter and ~2mm thickness. The pellets were sintered at 850^{o}C for fixed time 24h and slowly cooled down to room temperature at 36^{o}C per hour. The pure sample and added samples with 0.2, 0.5, 0.7 and 1.0 wt% analyzed composition by X-Ray diffraction (XRD) method using a Bruker D8 Advance Diffractometer with a CuKα source. All samples undergo resistance measurement by using four point probe technique with silver contacts. The cryogenic system used was a Closed Cycle Helium Cryostat.

3. Results & Discussions

Figure 1 shows metallic behavior for all samples with and without nano-γAl_2O_3 addition. The critical transition temperature, $T_{c\ zero}$ drops in the range 97 K to 100 K. Impurity addition reduce the T_c below 100 K. From the graph, the broadening shown by difference of $T_{c\ onset}$ and $T_{c\ zero}$ about Δ4 K to Δ13 K detect low-T_c phase. However, all samples still dominated by 2223-phase.

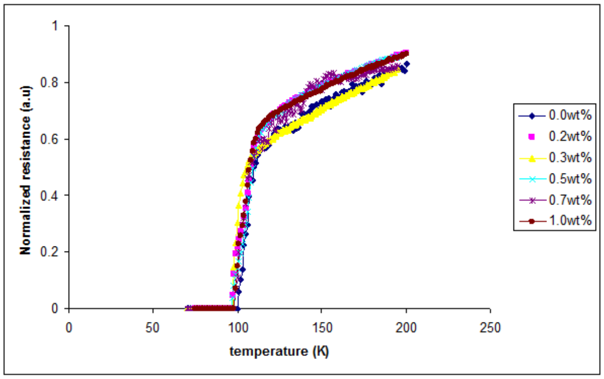

Figure 1. Normalized resistance as a function of temperature

Measurement of resistivity versus temperature for different amount of nano-γAl_2O_3 addition less than 1 wt% show single phase which also shows powder mainly composed of Bi-2223 phase. Increasing the addition led to decrease slightly the zero resistivity temperature. Low level of Al addition or oxygen content may cause the decreasing of T_c.

Figure 2 shows XRD pattern for samples $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_x$ pure and nano-γAl_2O_3 added. Peaks (002) at the position 2θ = 4.775^o shows consistent peaks for every samples. However, (013) peak disappear when 0.3wt% nano-γAl_2O_3 was added into (Bi, Pb)$_2Sr_2Ca_2Cu_3O_x$. The intensity of peak (1011) reduce at 2θ = 31.45^o when it comes to 0.3wt% nano-γAl_2O_3 pattern. Major crystalline phases present in the pure samples are 2223 phases with nominal composition (Bi, Pb)$_2Sr_2Ca_2Cu_3O_x$, Ca_2PbO_4 and CuO. The samples show majoring 2223 phase. XRD pattern for that sample will reduce secondary phase, noted by Ca_2PbO_4. The secondary phase was detected at 2θ= 17.85^o. The overlapping secondary phase reduces at that point. The major secondary phases involved directly in sequential reaction are Ca_2PbO_4 and CuO. Previous work stated, deal with BSCCO-2223 without lead, Pb added show 2212 phase appear most. The addition of lead as catalyst to enhanced the 2223 phase. However, observed that Ca_2PbO_4 is formed when Pb added to the composition, in addition to the common impurity phases. The appropriate Pb added only can enhance formation 2223 phase. Almost all diffraction peaks describe as tetragonal lattice cell of Bi-2223 phase. The measured lattice parameters are a = 5.4 Å, c = 37.1Å. The volume fractions can be estimated from the intensities [3],[7]. Where *I* is the peak intensity of the phase obtain in x-ray diffraction pattern. The fractions of Bi-2223/Bi-2212 (%) were estimated as 86.8/13.20, 87.89/12.11, 89.15/10.85, 87.67/12.33, 84.98/15.02 and 85.59/14.41 for samples 0.0, 0.2, 0.3, 0.5, 0.7 and 1.0wt% nano-γAl_2O_3 added respectively.

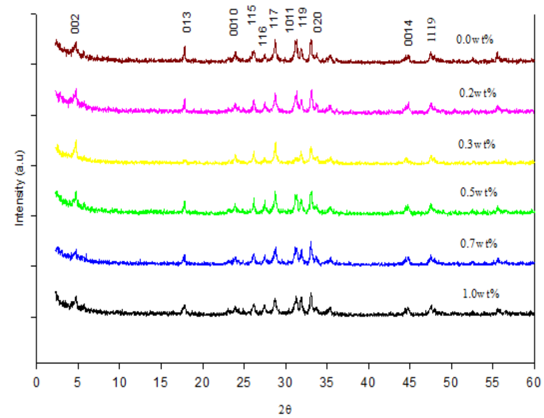

Figure 2. XRD pattern for pure and nano-γAl_2O_3 added into BSCCO-2223 samples sintered at 24 hours

4. Conclusion

The precursor powder of BSCCO-2223 superconducting ceramic has been successfully prepared via co-precipitation method using metal acetate and the addition of nano-γAl_2O_3 through conventional method. XRD data showed a high production of the Bi-2223 for all samples with and without addition ($V2223_{phase}$ ~ 93 to 96%). The addition of nano-γAl_2O_3 not reacted with the precursor hence the data still showing high fraction of Bi-2223 phase. The calculated lattice parameters are, a = 5.40 Å, c = 37.10Å.

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