STUDY ON PROPERTIES OF YBCO SUPERCONDUCTOR WITH ADDITION TITANIUM OXIDE TiO_2 BY SOLID STATE REACTION

by

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DECLARATION

I declare that this thesis entitled "**Study on Properties of YBCO superconductor With Addition Titanium Oxide (TiO_2) By Solid State Reaction** " is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : ____________________________

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Date : 8 JANUARY 2017

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Study on Properties of YBCO superconductor With Addition Titanium Oxide (TiO_2) By Solid State Reaction

ABSTRACT

In this study, the effects of TiO_2 addition on YBCO superconductor were studied. TiO_2 was added into YBCO superconductor samples with 1%, 2% and 3% weight percentage addition respectively were prepared using the conventional solid-state reaction method. The oxide has a triple perovskite-type structure and it also be a first superconductor that have critical temperature. Characterization of sample was performed by X-ray Diffraction (XRD), X-ray Fluorescence (XRF) and measurement the resistivity of superconductor. Using XRD method, the crystal structure and related parameter of compound in YBCO with addition was determined. YBCO sample has the orthorhombic structure and TiO_2 as addition not destroy the structure of YBCO system. The intensity peak was increased with increasing the addition. In XRF the metal element in pure YBCO and with addition was analyzed. The elemental analysis was traced is high percentage concentration of Barium Oxide at all samples. For resistivity measurement, the YBCO superconductor ceramic has properties are zero resistivity. The resistivity was decreased by increasing the addition of TiO_2. In conclusion, the YBCO with addition TiO_2 superconductor improves the superconducting performance of YBCO superconductor.

Mengkaji Sifat YBCO superkonduktor dengan penambahan titanium oksida (TiO_2) dalam Tindakbalas Keadaan Pepejal

ABSTRAK

Dalam kajian ini, kesan penambahan TiO_2 pada YBCO superkonduktor telah dikaji. TiO_2 telah ditambah ke dalam sampel superkonduktor YBCO dengan penambahan peratusan berat 1%, 2% and 3% telah disediakan menggunakan kaedah konvensional dalam tindak balas keadaan pepejal. Oksida mempunyai struktur jenis *triple perovskite* dan ianya juga menjadi superkonduktor pertama yang mempunyai suhu kritikal. Pencirian sampel telah dilakukan oleh sinar x-ray Diffraction, sinaran x-ray Fluorescene and mengukur resistan superkonduktor. Pengunaan kaedah XRD, struktur hablur dan parameter yang berkaitan dengan YBCO dan penambahan TiO_2 telah dikenalpasti. Sampel YBCO mempunyai struktur orthorhombic dan TiO_2 tidak memusnahkan struktur system YBCO. Puncak intensity semakin meningkat dengan peningkatan penambahan TiO_2. Dalam XRF unsur logam tulen YBCO dan YBCO dengan penambahan dapat dianalisis. Analisis unsur adalah Barium oksida mempunyai peratusan tinggi kepekatan pada semua sampel. Bagi pengukuran resistiviti, YBCO superkonduktor seramik mempunyai sifat-sifat yang resistiviti sifar. Resistiviti ini menurun dengan meningkatkan penambahan TiO2. Kesimpulannya, YBCO dengan penambahan TiO2 superkonduktor akan meningkatkan prestasi superkonduktornya.

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LIST OF ABBREVIATIONS

T_C	Critical Temperature
Hc	Critical Field
Jc	Critical Current density
K	Kelvin
YBCO	Yttrium Barium Carbon Oxide
TiO_2	Titanium Oxide
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
SEM	Scanning Electron Microscopy
$CBaO_3$	Barium Carbonate
CuO_3	Copper Oxide
Y_2O_3	Yttrium Oxide
PrO_3	Praseodymium Oxide

CHAPTER 1

INTRODUCTION

1.1 Background of study

Majority application of high temperature superconductors, it is desirable good superconducting and mechanical properties. Besides that, addition is needed to improve mechanical properties of the brittle superconducting oxide YBCO and example of addition is silver or titanium oxide.

The bulk Yttrium barium copper oxide have been widely investigate as a very potential superconductor material because of the potential of large scale engineering use such as high field permanent magnet, levitation platform, trapped fields magnets and many others (Zalga *et al.*, 2006). Beside that the YBCO bulk also has higher threshold temperature of around 92K. This meant for the first time that a material exhibited superconducting behavior at temperatures above that of liquid nitrogen (77K), which is much cheaper and easier to handle than liquid He (Altin *et al.*, 2014)

A great research has been done to determine how the structural and magnetic properties affect the mechanisms of superconductivity in $YBa_2Cu_3O_7$. Despite extensive research on $YBa_2Cu_3O_7$ spanning nearly three decades, there are still inconsistencies between studies. Regarding this issued, there is some investigation on the YBCO addition with titanium oxide (TiO_2) element using solid state method.

This investigation is done to enhance the properties for the superconductor in aspect the thermal conductivity, band gap, and morphology.

The method that used to fabricate the YBCO addition with TiO_2 in this investigation is solid state method. In this solid state method, solids do not react at room temperature over normal time scales and its necessary to heat them to much higher temperatures, often to 1000˚C to 1500˚C in order for the reaction to occur at an appreciable rate (Nakaoka *et al.*, 2014). The factors on which the feasibility and rate of a solid state reaction depend include, reaction conditions, structural properties of the reactants, surface area of the solids, their reactivity and the thermodynamic free energy change associated with the reaction. This solid state method shows that it comfortable to fabricate this superconductor. While in the superconducting state, these materials have the ability to transport large DC currents with no measurable resistive losses.

To do this, a superconductor must be kept below three critical parameters, critical temperature (Tc), critical field (Hc), and critical current density (Jc). Loss-free current flow allows superconducting wires with relatively small cross sections to transport currents at high current densities. It is envisioned that superconductors could be used to develop electrical devices that are smaller, lighter, and more energy efficient. Although some progress has been made, limitations of the materials have prevented wide-scale use of the technology.

1.2 Problem statement

Nowadays in this globalization worlds, the most issued that become a big problem is about the loss of power when its transport from one place to another place. There no heat is generated when the current moves through the conductor. This is a good thing, because we don’t lose any energy in just heating the wire. That might not seem like a problem if you’re just connecting your computer to the wall, but if you’re conducting electrical current from Niagara Falls to New York City, then that becomes a problem. About seven percent of the energy generated by hydroelectric power at Niagara Falls is lost on its way to New York City (Herbert, 2011).

The measurement of superconducting properties of YBCO superconductor with different weight percentages of addition nanomaterials TiO_2 content x= 0.03% prepared by modified combustion route (Rejith *et al.*, 2015). The characteristics of the YBCO superconductor is zero resistance but it not the same as a perfect superconductor. The problem that has been an issued is about the conductivity of electric current in unstable environment which the temperature is not consistent. The temperature which is too low or too high exactly will contribute to the failure of the electric current to be transmitted (Herbert, 2011). Because of this problem the superconductor is invented. However, while the superconductor had save a lot of power loss, there is still much work left to be done, especially in understanding how these compounds actually work and how they can be improved for further used.

1.3 Research scope

Characterization of the sample will be conducted to determine the phase, element and the resistivity of the YBCO superconductor by using XRD, XRF and resistivity testing. Study will be conducted to YBCO sample with different ratio addition of TiO_2 which 0%, 1%, 2% and 3% to identify the change of the properties for each addition.

1.4 Research Objectives

1) To characterize the TiO2 added in $YBa_2Cu_3O_7$ from XRD and resistivity technique.
2) To synthesize $YBa_2Cu_3O_7$ and the optimum amount of TiO_2 as additive.

CHAPTER 2

LITERATURE REVIEW

2.1 Historical background of YBCO superconductor

In 1911, superconductivity was firstly observed in mercury by Dutch physicist Heike Kamerlingh Onnes of Leiden University. When he cooled it to the temperature of liquid helium, 4 degrees Kelvin (-269˚C), its resistance suddenly disappeared. In year 1957 the superconductor was established by the *BCS theory by Bardeen, Copper and Scheiffer before it replace by the discovery of high temperature superconductive materials by Bedrnoz and Muller which is verified by Tanaka's group (University of Tokyo).After a year, Chu and others has discover an YBCO superconductors which has critical temperature was 90K, which exceed the liquid nitrogen temperature(77K) (Delft *et al.*, 2010). Before the mid-80's, superconductors were metals, and had to be operated at low temperatures, close to the boiling point of He (4.2 K). Due to the costs associated with maintaining such low temperatures, applications were limited to specialty areas such as research laboratories (particle accelerators, high field magnets, SQUIDs) and the medical industry (MRI). These applications succeeded because there were no alternative materials that could compete with the superconductors. This is not the case, however, in power applications, where Cu is the standard. Although energy and space can be saved using superconductors, it does not outweigh the costs of cooling the material or the initial costs and risks of introducing new technology. Superconductivity will improve by the granular coupling of YBCO by making composites of the superconductor with impurities which is silver or titanium and silver doping can enhance the inter granular couplings of YBCO through grain alignment, grain boundary and minimization of grain boundary area (Rani *et al.*, 2012).

2.2 Working principle of superconductor

A superconductor is a material with zero electrical resistance which allows the current flow through it as if it even there. In example, if you start a current flowing in a loop of superconductor, it will keep flowing forever which different with normal conductor that losses to heat and cause the current to dissipated quickly. The concept of superconductor happen when the electron inside them join force to make what are called cooper pairs (BCS pairs) (Mazaheri *et al.*, 2016). Normally, the electrons that carry electricity through a material are scattered about by impurities, defects, and vibrations of the material's crystal lattice (its scaffold-like inner structure). That's what we know as electrical resistance. But at low temperatures, when the electrons join together in pairs, they can move more freely without being scattered in the same way (Nakaoka *et al.*, 2014).

2.3 YBCO Superconductor

Yttrium Barium Copper Oxide, YBCO was a high temperature superconductor ever created. This YBCO is a copper oxide based on material which has critical temperature until 92k. The YBCO has become the most useful material such has been used in magnetic field application because of it is easy to pin flux. Superconductors are now can be combined with other elements such as Ferro-magnets to enhance the conductivity by modified the magnetic flux lines (Kuli, 2006). Besides, YBCO also been used typically in magnetic resonance (NMR) because of the YBCO is so well ordered that all atom of particular species will live in the same electronic environment.

2.4 Properties of titanium oxide

Titanium oxide (TiO_2) is a white solid inorganic powder that has properties such as thermally stable, non-flammable, poorly soluble and not hazardous prove by United Nations' (UN) Globally Harmonized System of Classification and Labeling of Chemicals (GHS). TiO_2 has differences between a TiO_2 pigment and nanomaterial. TiO_2 nanomaterial is a transparent and more effectiveness as ultraviolet absorbers or photo catalyst. Primary particles and higher surface area are smaller it affected the manufacture or various catalyst of enhanced activity.

Titanium oxide (TiO_2) is a compound that have high melting point with low heat capacity, is an excellent for reinforcement of brittle materials as a superconductor ceramic (Hamid *et al.*, 2014). The strength of the ceramics will be increased at the same time superconductor properties will enhance through creation of defects that contribute to the formation of flux pinning centers (Hamid *et al.*, 2014). Titanium oxide also can improve under low magnetic field J_c while for under high magnetic field it can use silicon oxide (SiO_2) (Zhang *et al.*, 2008). TiO_2 also has ability to reside within the YBCO superconducting grains and ability to lower the size or number of porosity. Chemical changes of the titanium oxide will occurred during annealing process including Ti dissolution into the bulk of the crystal phases (Matsumoto *et al.*, 2004).

2.5 Current density of superconductor

In the superconductor there is a current flow that we call a current density. This current density will increase the performance of the superconductor (Rejith *et al.*, 2015). Because of that zero resistance is very important to ensure the superconductor to be able to conduct a large amount of current without loose it. Zero resistance for the superconductor usually achieve when the material is below the transition temperature but influenced by the material composition (Khachan *et al.*). The current density also influenced by the dimension, microstructure and the flux pinning. Through the addition of praseodymium to the semiconductor it can improve the current density. The research state that the current density also influenced by the density of the composition of the superconductor.

2.6 Crystal structure in the superconductor

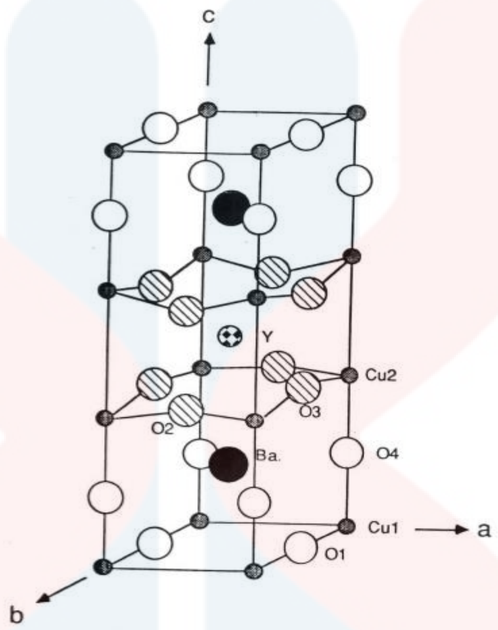

Figure 2.1 (Crystal structure of YBCO)

The crystal structure of $YBa_2Cu_3O_7$ is characterized by the arrangement of copper-oxygen planes and copper-oxygen chains. The stacking sequence of YBCO layers along the c-axis of the crystal goes as follows: CuO_2–BaO-CuO_2-Y- CuO_2-BaO. The perovskite structure layers of YBCO are separated by planes of CuO_2 with yttrium atoms between the copper-oxygen planes. The planes consist of a square lattice of copper atoms bridged by oxygen atoms. Chains of CuO_2 are parallel to the copper-oxygen planes with barium atoms located between the planes and chains. The unit cell of $YBa_2Cu_3O_7$ is shown in figure1. Many studies have shown that the critical temperature and crystal structure of YBCO change with oxygen content (Howe, 2014).

Neutron diffraction and magnetic measurements have shown that Tc is dependent on the charge balance between the copper-oxygen chains and copper-oxygen planes. The chain sites serve as charge reservoirs from which electrons are transferred to the copper-oxygen planes as the oxygen content decreases. It is within the copper-oxygen planes that superconductivity originates. As the oxygen content of $YBa_2Cu_3O_7$ decreases, so does Tc. The material goes through a structural change and the material superconductivity disappears when the oxygen content is below 6.3.

2.7 Effect of Ag addition on the microstructures and superconducting properties of bulk YBCO.

The effect of adding Ag into the liquid phase source ($YBa_2Cu_3O_7$) placed above the Y_2BaCuO_5 preform on the microstructures and current densities is investigated. The addition of Ag led to a significant refinement of Y_2BaCuO_5 particle size and hence, enabled the enhancement of current densities at low fields. Adding Ag to the liquid phase source $YBa_2Cu_3O_7$ resulted in fine-sized Y_2BaCuO_5 and Ag particles in the end product. The addition of Ag enhanced the superconducting properties in the low field regime (<3 T). This improvement in the superconducting properties may be attributed to the enhanced defect density associated with a larger YBCO interface. Because the Directionally Solidified Preform Optimized Infiltration Growth Process (DS-POIGP) is capable of producing YBCO superconducting composites over relatively short time durations with enhanced critical current densities performance, the importance of this process becomes much more interesting from the view point of wire fabrication (Kumar *et al.*, 2013).

2.8 Improvement of critical current density in YBCO superconductor with Nano TiO_2 addition

The optimization of flux line pinning in superconductors is one of the most efficient ways to improve the transport properties of these materials. There is examination on effectiveness of an insulating nanoparticle inclusion in a type II superconductor as a pinning middle with a size close to the coherence length. The microstructure of bulk YBCO added with nanoparticles was characterized by scanning electron microscopy. Sub-micron, randomly oriented particles of this phase were found to form around grain boundaries and within YBCO grains in bulk sintered pellets.

It is observed that the critical current density is higher by a factor of 3 and the (Jc) behaviour is remarkably improved in samples with nano additions. However, the superconductor transition temperature (Tc) slightly decreases from 92K to 90.5K. The flux pinning force is enhanced by 8 times that of pure YBCO sample. Thus these nano particles are effective flux pinning center and enhance the transport critical current density at liquid nitrogen temperature for an applied magnetic field. The experimental results suggest that the added nano sized TiO_2 particle may have high pinning efficiency at a temperature around 77 K.

CHAPTER 3

MATERIALS AND METHOD

3.1 Introduction

This study was conducted in the Material Laboratory of Universiti Malaysia Kelantan, Jeli Campus.

3.2 Materials

For the preparation of the sample it divided into two stages which is sample preparation and sample characterization. For the first stage, there are powder preparation of different sample composition, sample grinding and powder heating. And then, for the second phase which is sample characterization by using X-Ray Diffraction (XRD), resistivity measurement and X-ray Fluorescence Spectrometry (XRF). The high temperature superconductor sample YBCO with addition of Titanium Oxide (TiO_2) are prepared by solid state reaction. There are four powders used to prepare the sample:

1. Yttrium Oxide, Y_2O_3
2. Barium Carbonate, $CBaO_3$
3. Copper Oxide, CuO_3
4. Titanium Oxide, TiO_2

3.3 Methodology

3.3.1 Preparation of sample

The mixing of Yttrium, Barium, Copper and Carbon powder was prepared. Each powder sample was weighing by follow their atomic mass unit (AMU). Then all powder components were mixed together to form YBCO sample.

Atomic Mass Unit (AMU) for each component:

Yttrium = 88.906g/mol

Barium = 137.527g/mol

Copper = 63.546g/mol

Carbon = 12.011g/mol

The mixture's ratio given below:

½ Y_2O_3 = 112.904g/mol

$2BaCO_3$ = 394.676g/mol

3CuO = 238.635g/mol

Total = 746.215g/mol

To prepare 5 g of sample, each of the components should be weighed:

Y_2O_3 =5(112.904)/746.215 =0.757(3)

=2.271g

$BaCO_3$ = 5(394.676)/746.215 =2.645(3)

= 7.935g

CuO = 5(238.635)/746.215 =1.599(3)

= 4.797g

Total = 15.003g

For the preparation of the sample the method used is solid state reaction. Firstly the, barium carbonate, copper oxide, and yttrium oxide are mixed together. And grind the mixture until they exceed the homogeneous state. After grinding process, YBCO sample was place into the crucible and put into the furnace.

3.3.2 Calcination

The YBCO sample has been undergoes calcination process for 24 hours. It involves three stages. The samples were annealed at 900℃ for 12 hours and then furnace-cooled to room temperature (Nakaoka et al., 2014). Partial-melt processing of YBCO is known to significantly improve the microstructure of YBCO phase superconductor and it is employed to obtain the required texturing of the YBCO/TiO_2 superconductor compound. After heat process, the sample should be cooled until it reached the room temperature.

3.3.3 Added of Titanium Oxide

After the calcine process is done the sample added with TiO_2 and reground for until it achieved homogenous state. After that, the sample is press using the hydraulic pressure to compress the powder into pellets shape with 5 tons of hydraulic pressure. After weighing the sample of YBCO and TiO_2, the powder is mixing. Place the mold over the base and insert one pellet caps into the hole. By using the spatula, place 0.11g of the mixture sample of each sample into the hole containing the pellet cap. Then insert the second pellet cap into the hole and this time with flat side down until it completely sinks. Last pump the pellet carver until the rod on the cap meet the carver ceiling then pump it to 5 tons. Leave sample about 2 minutes before take out the sample.

3.3.4 Sintering process

Heat the pellets in furnace at 960°c for 24 hours. Lastly, the pellets sample was cooled until the room temperature. This sintering process is need in order to impart strength and integrity to the sample. There are two precursors thing before this solid state sintering take place which is removal of the pressing lubricant during compaction by evaporation and burning of the vapour beside reduction of the surface oxides from the powder particle in the compact. The samples were ready for the characterization with different analysis.

3.5 Sample characterization

Nine samples of YBCO with addition of TiO_2 were prepared with 3 different ratios for sample characterization. It showed in Table 3.1.

Table 3.1 Superconductor analysis

<table>
<tr><th>Addition's ratio of TiO_2 in YBCO</th><th colspan="3">Testing</th></tr>
<tr><td></td><td rowspan="4">X-Ray Diffraction (XRD)</td><td rowspan="4">X-Ray Fluorescence (XRF)</td><td rowspan="4">Resistivity Measurement</td></tr>
<tr><td>1%</td></tr>
<tr><td>2%</td></tr>
<tr><td>3%</td></tr>
</table>

3.5.1 X-ray Diffraction

Sample for XRD testing were prepared and placed in the sample chamber of XRD. XRD relies on the dual wave/particles nature of X-ray to obtain information about the structure of crystalline materials. A primary uses of the technique is identification and characterization of compound based on their diffraction pattern. As the sample shows the diffraction pattern, there are many of the parameters can be analyzed such as crystallinity, lattice parameter, the orientation of the planes and the components of the sample. Refer to figure 3.1.

XRD analysis has been performed analysis of control and treated sample. XRD system, the data was performed data in the form of table that containing Bragg angles and peak intensity counts, relatively intensity, d-spacing value and full width half maximum for each peak. The Scherrer equation 1 was used to compute the crystalline size.

$$Crsytalline\ size\ (G) = \frac{k\lambda}{b\ cos\theta} \qquad \text{----------} \qquad \text{Equation 1}$$

Where k is constant = 0.94, λ = 1.54056Â and b is full width half maximum.

Figure 3.1 X-Ray Diffraction

3.3.6.2 X-ray Fluorescence

In analysis of YBCO with addition of TiO_2, X-ray Fluorescence Spectrometry (XRF) was used an it to analyze the elemental analysis technique. The figure 3.2 was showed the XRF equipment. XRF is a principle that individual atoms, when excited by an external energy sources, emit X-ray photon or characteristic energy or wavelength (Hardin *et al.*). 5g of sample pure YBCO, YBCO + 1% TiO_2, YBCO + 2% TiO_2 and YBCO + 3% TiO_2 were prepared.

Figure 3.2 X-ray Fluorescence

3.3.6.3 Resistivity Measurement

Resistivity measurement was tested by set up circuit which consists of pellet YBCO sample, battery and multi meter that use to measure the resistivity. The testing was repeated about three times for each sample. Four different compositions of addition TiO2 samples of pellet were tested.

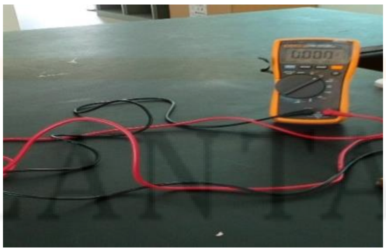

Figure 3.3 Multi meter

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

Synthesize the YBCO superconductor was prepared through conventional solid state reaction. Then the pure YBCO were added with the addition TiO_2 respectively. Finally, the characterizations of the samples were studied by X-Ray Diffraction, X-ray Fluorescence and resistivity of superconductor.

4.2 XRD study

The XRD technique was study the quantitative and non-destructive technique which is used to study the crystal structure and related parameter of compound in the YBCO with addition doping ratio from x=0.00%, 1.00 %, 2.00 % and 3.00% weightage. Figure 4.1 was showed the XRD of the pure YBCO sample while Figure 4.2 was showed the YBCO added with TiO_2 materials at different amount.

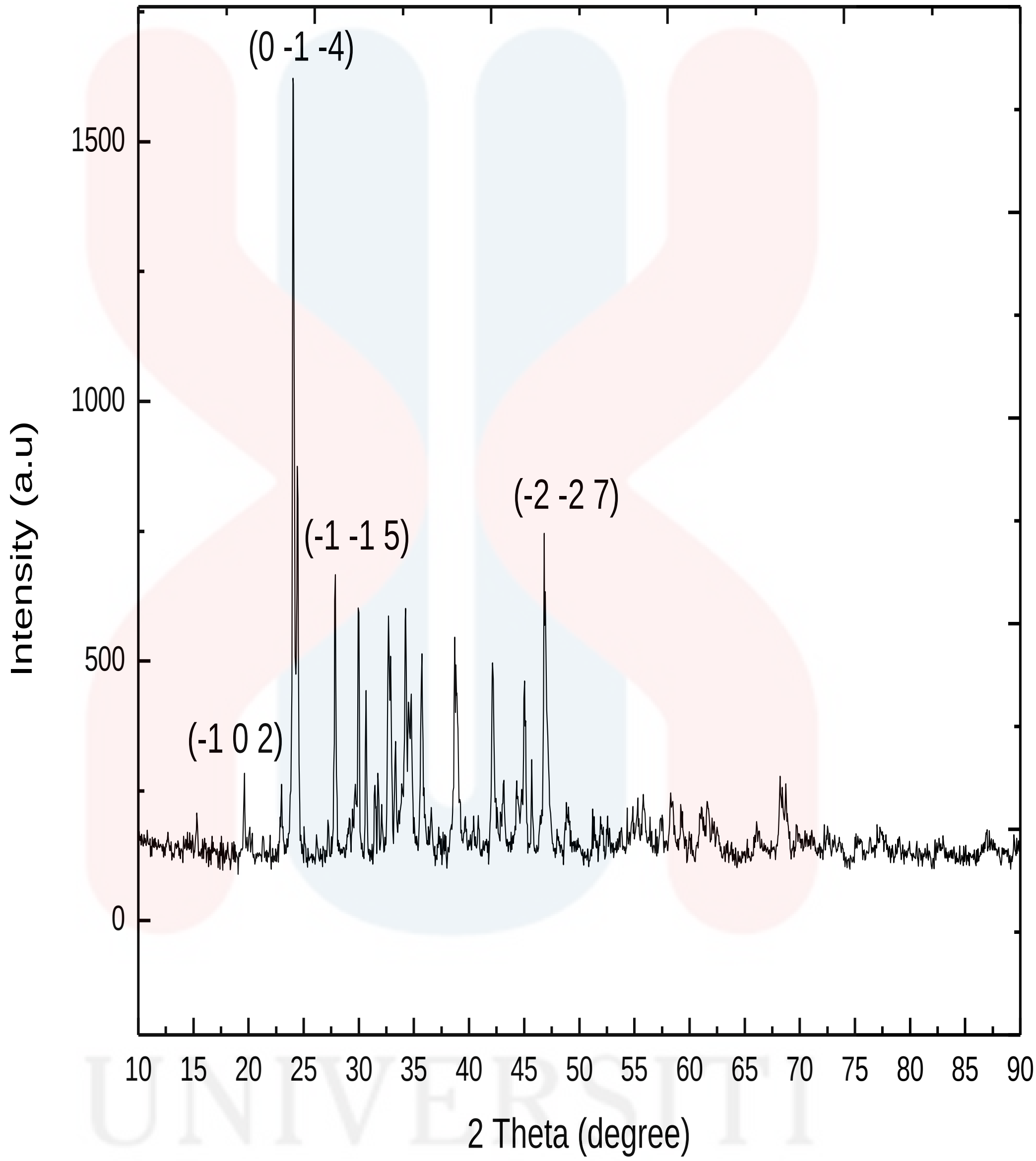

Figure 4.1 X-ray Diffraction of pure YBCO

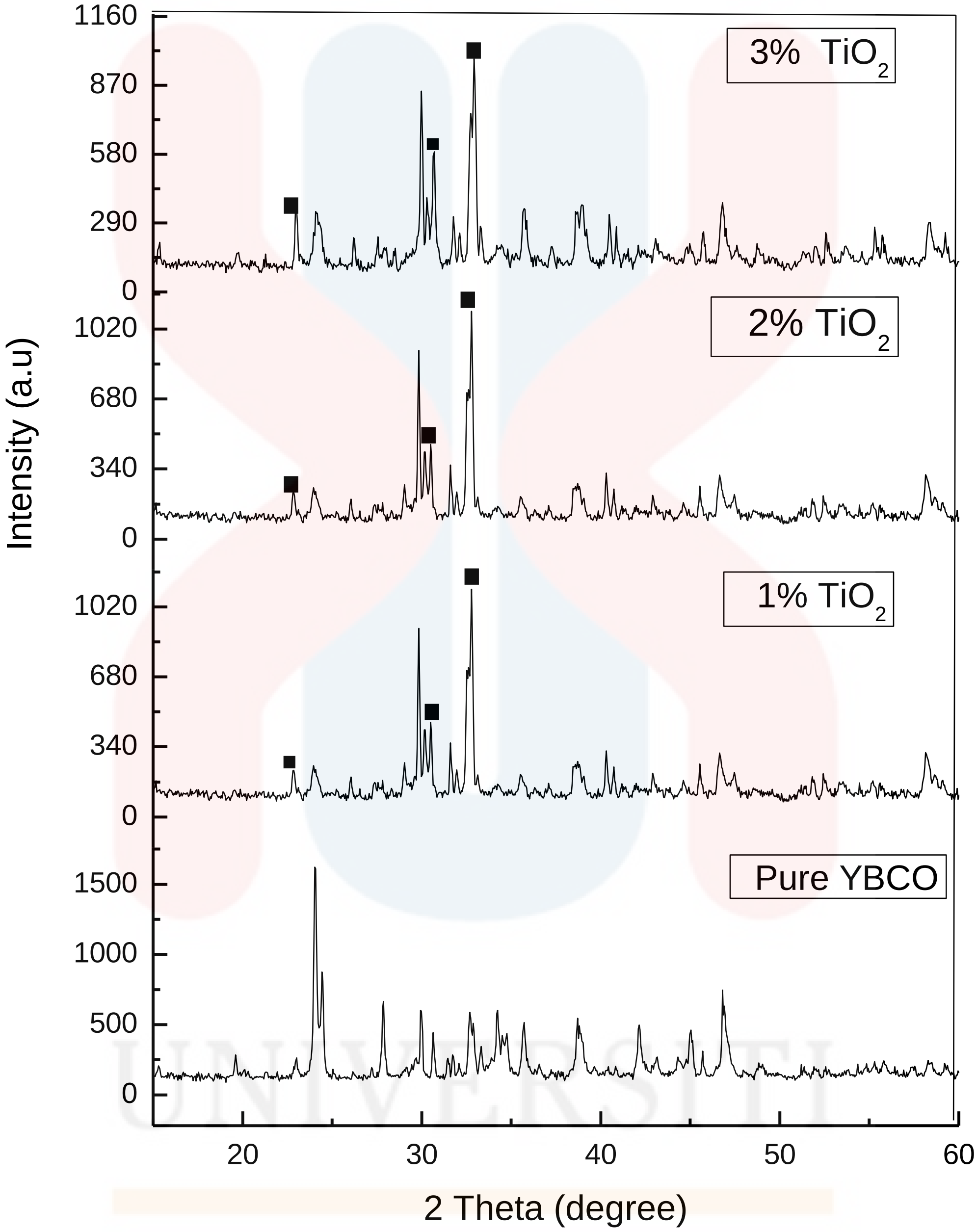

Figure 4.2 X-ray Diffraction of pure YBCO and different amount of TiO_2.

The sample undergoes process calcination by sintered at 900°C about 24 hours. In this group of all samples have the orthorhombic structure. As mentioned before TiO_2 does not destroy the orthorhombic structure of YBCO system. Refer to Figure 4.2 the diffraction pattern of YBCO and YBCO with addition TiO_2, were match a known diffraction. Three peaks were match with TiO_2 pattern which peak is match to TiO_2 at 2θ. In the measurement, the resolution of equipment has seen the known secondary phase of YBCO with addition of TiO_2. It compared with Figure 4.1 that only contain of pure YBCO peak. The intensity of peak was increasing with increasing of the addition amount. Base on the result, it can be seen at 3% addition of $TiO_{2,}$ the peak become more visible compare to the 1%. Few of YBCO peak become shorten and diminished as the percentage of TiO_2 increase. In this study, the orthorhombic TiO_2 peaks was observed at peak 2theta =22.89˚, 30.66˚ and 32.8˚4. Based on previous work by Rejith et al. stated that the interesting to note that even though the nano addition up to 3%wt and peak position of YBCO unaltered without showing any shift.

4.3 Resistivity measurement

The resistivity measurement was done to observe the superconductivity resistivity at room temperature of YBCO and addition with TiO_2. The circuit was setup as shown in the diagram in Figure 4.3. While the schematic diagram of circuit was showed in Figure 4.4.

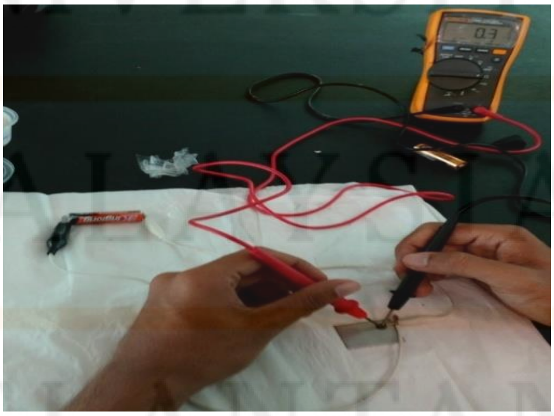

Figure 4.3 Resistivity testing

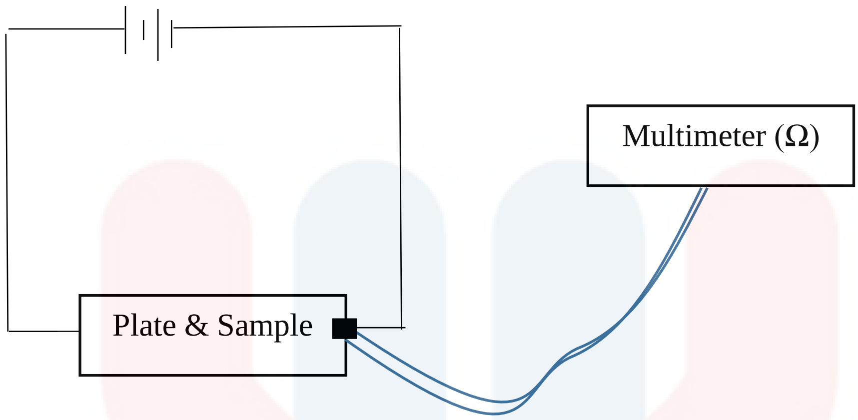

Figure 4.4 Schematic Diagram for Resistivity Measurement

The resistance measurement was done with a Fluke 116 series multi meter and power supply using battery. In this mode a known current is passed through the two wires in the circuit. The current flow and resistance in the circuit was measured using the multi meter.

For this measurement 4 samples were prepared which pure YBCO, YBCO + 1% TiO_2, YBCO + 2% TiO_2 and YBCO + 3% TiO_2. Table 4.1 was showed the relationship of percentages of addition and the resistivity of the superconductor sample shown by multi meter.

Table 4.1 Resistivity data measurement at room temperature

Sample	**Voltage 1.3 V**		**Voltage 1.6 V**	
	Resistance (Ω)		Resistance (Ω)	
0% TiO_2	1.0	1.0	1.5	1.6
	1.1		1.6	
	1.0		1.6	
1% TiO_2	0.9	0.8	1.4	1.4
	0.8		1.5	
	0.7		1.3	
2% TiO_2	0.3	0.3	1.2	1.2
	0.2		1.1	
	0.3		1.4	
3% TiO_2	0.2	0.1	0.3	0.3
	0.1		0.4	
	0.1		0.3	

Refer to the graph in the Figure 4.5 was shown that resistivity of each sample have different value of resistance. The current apply for this circuit is 1.3V. For the sample with 0% addition contain the higher resistivity where is 1.0 Ω which is the largest among the 4 sample. For the sample with 1% addition the resistivity was 0.8 Ω while for 2% addition the resistivity is 0.3 Ω. The lowest resistivity is the sample with 3% addition which is 0.1Ω.

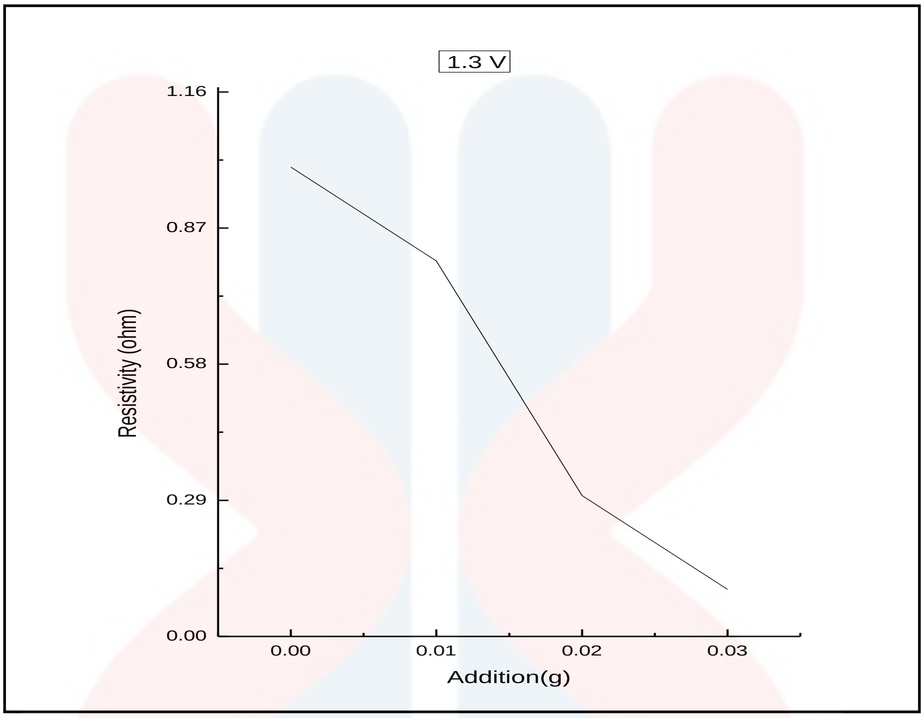

Figure 4.5 Resistivity versus different amount of addition of TiO_2 with apply voltage was 1.3V.

Refer to the graph in Figure 4.6, the current apply for this circuit is 1.6V. For the sample with 0% addition contain the higher resistivity where is 1.6 Ω which is the largest among the 4 sample. For the sample with 1% addition the resistivity was 1.4 Ω while for 2% addition the resistivity is 1.2 Ω. The lowest resistivity is the sample with 3% addition which is 0.3Ω. The voltage applied to the circuit effect the value of the resistivity of the sample. From the result we agreed that YBCO superconductor ceramic showed properties are resistivity decreased by increase the addition. Even though the possibility of YBCO to achieve zero resistance because at room temperature.

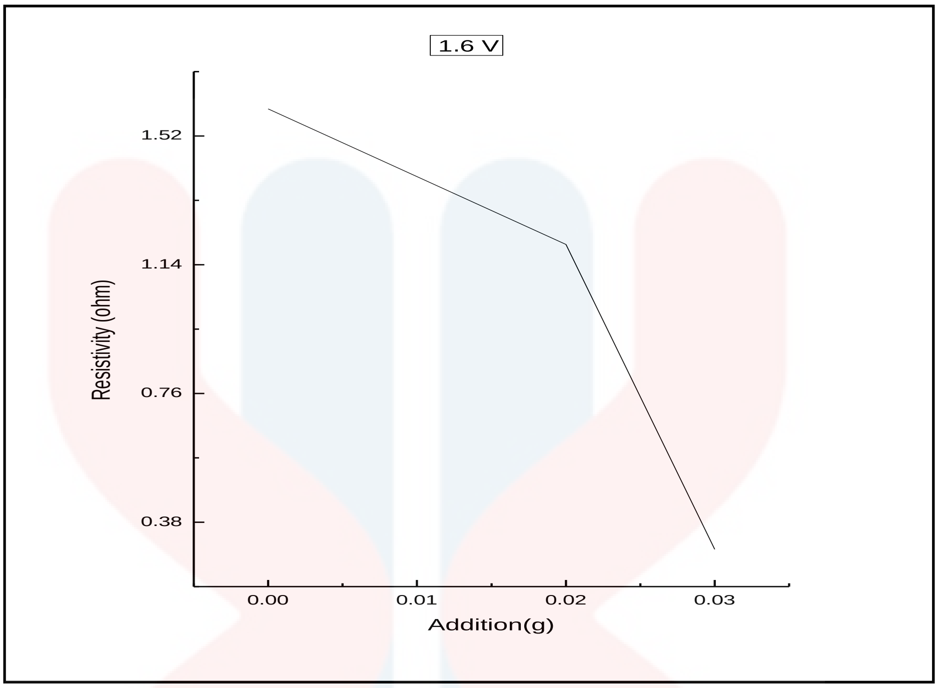

Figure 4.6 Resistivity versus different amount of addition of TiO_2 with apply voltage was 1.6 Volt

4.4 X-RAY FLUORESCENCE ANALYSIS

XRF is one at the most important characterization technique in analyze metal and trace element. Thus X-Ray Fluorescence analysis is based on the generation of characteristics X range from sample irradiated by energetic beam. The elemental analysis was referred in Table 4.2. The capable of measuring the concentration of different elemental in the YBCO sample.

Table 4.2 Elemental Analysis from XRD pattern

Element	**Value Analysis**			
	Pure YBCO	**1% TiO_2 + YBCO**	**2% TiO_2 + YBCO**	**3% TiO_2 + YBCO**
TiO_2	1.50	2.90	3.33	4.71
CuO	33.5	21.7	27.6	29.7

Y_2O_3	17.2	18.6	19.6	25.0
BaO	40.6	36.8	38.4	38.2

All the element under investigation in this experiment were calculated by means of activities (n,Y) reaction. 4 elements were identified. The elements determined with their different concentration are shown in table above. There are few unpredictable elements as foreign elements were presented in this YBCO and YBCO with addition compound. This cause during the sintering process, the apparatus such as crucible use not completely clean. This may cause of the contamination of the sample. From the element concentration result, it shown that the YBCO and TiO_2 element still present such as yttrium, carbon, barium oxide and titanium oxide. It means the compounding of YBCO and TiO_2 was successful.

CHAPTER 5

CONCLUSION AND RECOMMANDATION

5.1 CONCLUSION

YBCO superconductor was synthesized and study on effect of addition TiO_2 at 1% , 2%to 3% using solid state reaction method was prepared. The pure YBCO and YBCO with addition with different weight of TiO_2 sample prepared and have been studied by various characterization techniques. The phase of the YBCO superconductor was confirmed by XRD and the resistivity at room temperature was measured. The elemental of the YBCO were confirmed by the XRF analysis and the YBCO element with the addition of TiO_2 was confirmed. The addition of the TiO_2 was effected the resistivity for each ratio of the sample whereas the resistivity increase as the increasing ration of the addition to the sample. For this resistivity measurement it does not indicate the presence of superconductivity and it focus on semiconducting properties. In this experiment, the resistance change depends on the percentage of the addition in YBCO superconductor. In order fix it to get a better resistivity measurement, the temperature should be controlled and the method for the addition of TiO_2 was changed. With addition of TiO_2 to YBCO superconductor the resistivity and the peak of YBCO in the XRD change which mean certain properties of the YBCO and TiO_2 change.

5.2 RECOMMANDATION

The critical problems for this study during the fabrication process of the YBCO superconductor. The high temperature sintering heat treatment (900-950˚C) is result in loss oxygen, it can easily to overcome through low temperature processing. The temperature of sintering can be varied from 650˚C until 950 ^{o}C.

Measurement resistivity testing depends on temperature. The future research should prepare the YBCO with addition TiO_2 with controlled temperature of sample and environment.

Other recommendation for future study is using a others materials for addition such as zinc oxide, lead oxide or graphene oxide. The selection of addition depends on the properties of materials. The selection of an addition compound that have high melting point with lower capacity for excellent performance of YBCO superconductor. The later coated superconductor development is the high performance YBCO coated superconductor can fabricate.

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Appendix A

XRD analysis of pure YBCO

Pattern: COD 2201629 (Tune Cell) Radiation: 1.54060 Quality: User modified

Formula	C14 H40 Br6 N6 O2
Name	
Name (mineral)	
Name (common)	
Lattice:	Orthorhombic
S.G.:	P b c a (61)
a =	12.34000
b =	10.38000
c =	20.32100
a/b =	1.18882
c/b =	1.95771
alpha =	
beta =	
gamma =	
Z =	4
Mol. weight =	
Volume [CD] =	2602.9
Dx =	
Dm =	
I/Icor =	2.380

Color: colorless: Temperature of data collection: 293.00 K:

Primary Reference
Liang Feng, Li Yi-Zhi, Wu Cheng-Tai, Liu Si-Min, "1,4,7,11,14,17-Hexaazacycloicosane-9,19-diol hexahydrobromide", Acta Crystallographica Section E 58(8) (2002) m950-m952.

Radiation:		**Filter:**	Not specified
Wavelength:	1.54060	**d-spacing:**	
SS/FOM:			

d	2θ	I	h	k	l	d	2θ	I	h	k	l
7.84377	11.272	13	-1	0	-2	3.33002	26.750	336	-3	-1	-3
7.39830	11.953	3	-1	-1	-1	3.28763	27.101	180	-1	-3	-1
6.25797	14.141	128	-1	-1	-2	3.26605	27.284	29	-1	0	-6
6.17000	14.344	13	-2	0	0	3.22597	27.629	8	-2	-1	-5
5.30376	16.702	48	-2	-1	0	3.19984	27.859	11	0	-2	-5
5.27378	16.798	185	-2	0	-2	3.19684	27.886	4	-3	0	-4
5.19000	17.071	3	0	-2	0	3.18384	28.002	20	-3	-2	-1
5.15416	17.190	119	-1	-1	-3	3.16569	28.166	127	-1	-3	-2
5.13185	17.266	20	-2	-1	-1	3.12899	28.503	47	-2	-2	-4
5.08025	17.442	45	0	0	-4	3.11547	28.630	53	-1	-1	-6
5.02858	17.623	38	0	-2	-1	3.09740	28.800	30	-1	-2	-5
4.70174	18.859	5	-2	-1	-2	3.08500	28.919	225	-4	0	0
4.69772	18.875	34	-1	0	-4	3.07271	29.037	33	-3	-2	-2
4.65678	19.043	40	-1	-2	-1	3.05522	29.207	37	-3	-1	-4
4.32829	20.503	35	-1	-2	-2	3.01787	29.576	53	-2	-3	0
4.27982	20.738	18	-1	-1	-4	2.98950	29.864	34	-1	-3	-3
4.11974	21.553	2	0	-2	-3	2.98513	29.908	37	-2	-3	-1
3.97172	22.366	33	-2	-2	0	2.96895	30.075	463	-2	0	-6
3.92189	22.654	21	-2	0	-4	2.95716	30.198	45	-4	-1	0
3.89797	22.795	23	-2	-2	-1	2.95193	30.253	3	-4	0	-2
3.81274	23.312	44	-3	0	-2	2.92634	30.524	12	-4	-1	-1
3.75806	23.656	3	-3	-1	-1	2.91083	30.690	7	-3	-2	-3
3.69915	24.038	1018	-2	-2	-2	2.89296	30.885	31	-2	-3	-2
3.66875	24.240	113	-2	-1	-4	2.85448	31.311	12	-2	-1	-6
3.63046	24.500	546	0	-2	-4	2.84056	31.469	35	-2	-2	-5
3.61813	24.585	54	-1	-1	-5	2.83935	31.483	155	-4	-1	-2
3.57894	24.858	10	-3	-1	-2	2.83633	31.517	33	0	-2	-6
3.48286	25.555	45	-1	-2	-4	2.78591	32.103	39	-1	-3	-4
3.42619	25.985	43	-2	-2	-3	2.78503	32.113	40	-3	-1	-5
3.38683	26.293	53	0	0	-6	2.76426	32.361	65	-1	-2	-6

Appendix B

XRD analysis of 1% addition in YBCO

Formula: C Ca0.06 Ce F0.2 Fe0.67 La0.56 Mg0.21 Mn0.11 Na0.02 Nd0.24 O9.8 Pr0.08 Si2 Sm0.02 Ti0.01

Name

Name (mineral): Biraite-(Ce)

Name (common)

Lattice: Monoclinic
S.G.: P 1 21/c 1 (14)

Mol. weight =
Volume [CD] = 771.05
Dx =
Dm =
I/Icor = 1.450

a = 6.50500
b = 6.74400
c = 18.56100
a/b = 0.96456
c/b = 2.75222

alpha =
beta = 108.750
gamma =
Z =

d	2θ	I	h	k	l	d	2θ	I	h	k	l
8.78798	10.057	22	0	0	-2	2.92933	30.492	203	0	0	-6
6.29640	14.054	15	0	-1	-1	2.92848	30.501	95	-2	-1	2
6.15978	14.368	2	-1	0	0	2.92250	30.565	33	0	-2	-3
6.03816	14.659	20	-1	0	2	2.90167	30.790	1	-2	-1	1
5.35016	16.556	5	0	-1	-2	2.87582	31.073	27	-2	-1	3
4.68080	18.944	11	-1	-1	1	2.84580	31.409	3	-1	-2	-1
4.54816	19.502	157	-1	-1	0	2.84108	31.463	4	-1	-1	-4
4.49854	19.719	2	-1	-1	2	2.82140	31.688	64	-1	-2	3
4.42028	20.072	102	-1	0	-2	2.80157	31.918	15	-2	-1	0
4.39399	20.193	15	0	0	-4	2.78153	32.155	19	-1	-1	6
4.28757	20.700	79	-1	0	4	2.75556	32.466	99	-2	-1	4
4.16965	21.292	95	-1	-1	-1	2.68681	33.321	18	0	-1	-6
4.09402	21.690	4	-1	-1	3	2.68098	33.395	39	-1	-2	-2
3.69694	24.053	40	-1	-1	-2	2.67508	33.471	77	0	-2	-4
3.68152	24.155	104	0	-1	-4	2.65258	33.763	135	-2	0	-2
3.61824	24.584	43	-1	-1	4	2.65051	33.791	27	-1	-2	4
3.37200	26.411	18	0	-2	0	2.64886	33.812	1	-2	-1	-1
3.31160	26.901	98	0	-2	-1	2.59102	34.591	4	-2	-1	5
3.25097	27.413	7	-2	0	2	2.57597	34.799	16	-2	0	6
3.23947	27.512	7	-1	-1	-3	2.50816	35.771	4	-1	-1	-5
3.16893	28.137	34	-1	-1	5	2.49028	36.037	26	-1	-2	-3
3.14820	28.326	119	0	-2	-2	2.46850	36.366	22	-2	-1	-2
3.13263	28.470	1	-1	0	-4	2.45888	36.513	1	-1	-1	7
3.11716	28.614	8	0	-1	-5	2.45780	36.530	0	-1	-2	5
3.07989	28.968	1	-2	0	0	2.43341	36.909	31	0	-2	-5
3.05332	29.225	2	-1	0	6	2.40640	37.339	5	-2	-1	6
3.01908	29.564	123	-2	0	4	2.36677	37.987	3	-1	0	-6
2.99339	29.824	66	-1	-2	1	2.35306	38.217	0	0	-1	-7
2.95781	30.191	10	-1	-2	0	2.34040	38.432	3	-2	-2	2
2.94404	30.336	2	-1	-2	2	2.32665	38.668	19	-2	-2	1

Primary Reference
Konev A., Pasero M., Pushcharovsky D., Merlino S., Kashaev A., Suvorova L., Ushchapovskaya Z., Nartova N., Lebedeva Y., Chukanov N., "Biraite-(Ce), Ce2Fe2+(CO3)(Si2O7), a new mineral from Siberia with a novel structure type Locality: Biraia deposit, Irkutsk district, Siberia, Russia", European Journal of Mineralogy 17 (2005) 715-721.

Radiation:
Wavelength: 1.54060
SS/FOM:

Filter: Not specified
d-spacing:

Appendix C

XRD Analysis of 2% Addition TiO_2 in YBCO

Formula	C Ca0.06 Ce F0.2 Fe0.67 La0.56 Mg0.21 Mn0.11 Na0.02 Nd0.24 O9.8 Pr0.08 Si2 Sm0.02 Ti0.01
Name	
Name (mineral)	Biraite-(Ce)
Name (common)	

Lattice:	Monoclinic	**Mol. weight =**	
S.G.:	P 1 21/c 1 (14)	**Volume [CD] =**	771.05
		Dx =	
		Dm =	
		I/Icor =	1.450

a =	6.50500	alpha =	
b =	6.74400	beta =	108.750
c =	18.56100	gamma =	
a/b =	0.96456	Z =	
c/b =	2.75222		

d	2θ	I	h	k	l	d	2θ	I	h	k	l
8.78798	10.057	22	0	0	-2	2.92933	30.492	203	0	0	-6
6.29640	14.054	15	0	-1	-1	2.92848	30.501	95	-2	-1	2
6.15978	14.368	2	-1	0	0	2.92250	30.565	33	0	-2	-3
6.03816	14.659	20	-1	0	2	2.90167	30.790	1	-2	-1	1
5.35016	16.556	5	0	-1	-2	2.87582	31.073	27	-2	-1	3
4.68080	18.944	11	-1	-1	1	2.84580	31.409	3	-1	-2	-1
4.54816	19.502	157	-1	-1	0	2.84108	31.463	4	-1	-1	-4
4.49854	19.719	2	-1	-1	2	2.82140	31.688	64	-1	-2	3
4.42028	20.072	102	-1	0	-2	2.80157	31.918	15	-2	-1	0
4.39399	20.193	15	0	0	-4	2.78153	32.155	19	-1	-1	6
4.28757	20.700	79	-1	0	4	2.75556	32.466	99	-2	-1	4
4.16965	21.292	95	-1	-1	-1	2.68681	33.321	18	0	-1	-6
4.09402	21.690	4	-1	-1	3	2.68098	33.395	39	-1	-2	-2
3.69694	24.053	40	-1	-1	-2	2.67508	33.471	77	0	-2	-4
3.68152	24.155	104	0	-1	-4	2.65258	33.763	135	-2	0	-2
3.61824	24.584	43	-1	-1	4	2.65051	33.791	27	-1	-2	4
3.37200	26.411	18	0	-2	0	2.64886	33.812	1	-2	-1	-1
3.31160	26.901	98	0	-2	-1	2.59102	34.591	4	-2	-1	5
3.25097	27.413	7	-2	0	2	2.57597	34.799	16	-2	0	6
3.23947	27.512	7	-1	-1	-3	2.50816	35.771	4	-1	-1	-5
3.16893	28.137	34	-1	-1	5	2.49028	36.037	26	-1	-2	-3
3.14820	28.326	119	0	-2	-2	2.46850	36.366	22	-2	-1	-2
3.13263	28.470	1	-1	0	-4	2.45888	36.513	1	-1	-1	7
3.11716	28.614	8	0	-1	-5	2.45780	36.530	0	-1	-2	5
3.07989	28.968	1	-2	0	0	2.43341	36.909	31	0	-2	-5
3.05332	29.225	2	-1	0	6	2.40640	37.339	5	-2	-1	6
3.01908	29.564	123	-2	0	4	2.36677	37.987	3	-1	0	-6
2.99339	29.824	66	-1	-2	1	2.35306	38.217	0	0	-1	-7
2.95781	30.191	10	-1	-2	0	2.34040	38.432	3	-2	-2	2
2.94404	30.336	2	-1	-2	2	2.32665	38.668	19	-2	-2	1

Primary Reference
Konev A., Pasero M., Pushcharovsky D., Merlino S., Kashaev A., Suvorova L., Ushchapovskaya Z., Nartova N., Lebedeva Y., Chukanov N., "Biraite-(Ce), Ce2Fe2+(CO3)(Si2O7), a new mineral from Siberia with a novel structure type Locality: Biraia deposit, Irkutsk district, Siberia, Russia", European Journal of Mineralogy 17 (2005) 715-721.

Radiation:		Filter:	Not specified
Wavelength:	1.54060	d-spacing:	
SS/FOM:			

Appendix D

XRD Analysis of 3% Addition TiO_2 in YBCO

Formula C Ca0.06 Ce F0.2 Fe0.67 La0.56 Mg0.21 Mn0.11 Na0.02 Nd0.24 O9.8 Pr0.08 Si2 Sm0.02 Ti0.01

Name

Name (mineral) Biraite-(Ce)

Name (common)

Lattice: Monoclinic
S.G.: P 1 21/c 1 (14)

Mol. weight =
Volume [CD] = 771.05
Dx =
Dm =
I/Icor = 1.450

a = 6.50500
b = 6.74400
c = 18.56100
a/b = 0.96456
c/b = 2.75222

alpha =
beta = 108.750
gamma =
Z =

d	2θ	I	h	k	l	d	2θ	I	h	k	l
8.78798	10.057	14	0	0	-2	2.92933	30.492	129	0	0	-6
6.29640	14.054	9	0	-1	-1	2.92848	30.501	60	-2	-1	2
6.15978	14.368	2	-1	0	0	2.92250	30.565	21	0	-2	-3
6.03816	14.659	13	-1	0	2	2.90167	30.790	1	-2	-1	1
5.35016	16.556	3	0	-1	-2	2.87582	31.073	17	-2	-1	3
4.68080	18.944	7	-1	-1	1	2.84580	31.409	2	-1	-2	-1
4.54816	19.502	99	-1	-1	0	2.84108	31.463	3	-1	-1	-4
4.49854	19.719	1	-1	-1	2	2.82140	31.688	41	-1	-2	3
4.42028	20.072	65	-1	0	-2	2.80157	31.918	10	-2	-1	0
4.39399	20.193	10	0	0	-4	2.78153	32.155	12	-1	-1	6
4.28757	20.700	50	-1	0	4	2.75556	32.466	63	-2	-1	4
4.16965	21.292	60	-1	-1	-1	2.68681	33.321	12	0	-1	-6
4.09402	21.690	2	-1	-1	3	2.68098	33.395	24	-1	-2	-2
3.69694	24.053	25	-1	-1	-2	2.67508	33.471	49	0	-2	-4
3.68152	24.155	66	0	-1	-4	2.65258	33.763	86	-2	0	-2
3.61824	24.584	27	-1	-1	4	2.65051	33.791	17	-1	-2	4
3.37200	26.411	11	0	-2	0	2.64886	33.812	1	-2	-1	-1
3.31160	26.901	62	0	-2	-1	2.59102	34.591	2	-2	-1	5
3.25097	27.413	5	-2	0	2	2.57597	34.799	10	-2	0	6
3.23947	27.512	4	-1	-1	-3	2.50816	35.771	3	-1	-1	-5
3.16893	28.137	21	-1	-1	5	2.49028	36.037	17	-1	-2	-3
3.14820	28.326	75	0	-2	-2	2.46850	36.366	14	-2	-1	-2
3.13263	28.470	0	-1	0	-4	2.45888	36.513	0	-1	-1	7
3.11716	28.614	5	0	-1	-5	2.45780	36.530	0	-1	-2	5
3.07989	28.968	0	-2	0	0	2.43341	36.909	20	0	-2	-5
3.05332	29.225	1	-1	0	6	2.40640	37.339	3	-2	-1	6
3.01908	29.564	78	-2	0	4	2.36677	37.987	2	-1	0	-6
2.99339	29.824	42	-1	-2	1	2.35306	38.217	0	0	-1	-7
2.95781	30.191	6	-1	-2	0	2.34040	38.432	2	-2	-2	2
2.94404	30.336	1	-1	-2	2	2.32665	38.668	12	-2	-2	1

Primary Reference
Konev A., Pasero M., Pushcharovsky D., Merlino S., Kashaev A., Suvorova L., Ushchapovskaya Z., Nartova N., Lebedeva Y., Chukanov N., "Biraite-(Ce), Ce2Fe2+(CO3)(Si2O7), a new mineral from Siberia with a novel structure type Locality: Biraia deposit, Irkutsk district, Siberia, Russia", European Journal of Mineralogy 17 (2005) 715-721.

Radiation:
Wavelength: 1.54060
SS/FOM:

Filter: Not specified
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