EFFECT OF NICKEL OXIDE ADDITION ON YBCO SUPERCONDUCTOR PROPERTIES PREPARED BY SOLID STATE REACTION

by

HAIRIL AZWAN BIN HOSNEY

A report submitted in fulfillment of the requirements for the degree of Bachelor of Applied Science (Materials Technology) with Honours

FACULTY OF EARTH SCIENCE
UNIVERSITI MALAYSIA KELANTAN

2017

DECLARATION

I declare that this thesis entitled “Effect of Nickel oxide addition on YBCO superconductor properties prepared 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 : ______________________________

Name : Hairil Azwan Bin Hosney

Date : 5.1.2017

ACKNOWLEDGEMENT

Firstly, thank god because finally I could prepare a thesis in a given time to fulfill the degree requirement. I would like to thank all who have helped me in completing this work. In advance, I would like to have my appreciation to my supervisor Dr. Arlina Bt Ali for providing a very good advice to all the students and willing to spent time to give guidance and take the time to examine the data obtained from the results of the study.

My appreciation goes out to the lab assistant that has provide a very good service in extracting data from the equipment used for testing. The lab assistant also provide a very good assistance while running the experiment.

In addition, thanks also to all graduate students and colleagues who have help in finishing this thesis ever. Without the help and guidance, my research cannot be done successfully and my comrades, namely Ezzaruddin b. Eezahan who equally make the provision of these superconductors.

EFFECT OF NICKEL OXIDE ADDITION ON YBCO SUPERCONDUCTOR PROPERTIES PREPARED BY SOLID STATE REACTION

Abstract

In this project, the YBCO superconductor was added with nickel oxide. The sample was prepared by mixing the chemical powder. Then, the mixed powder grounded and calcine at 900°C. After pelleting with 1 ton of pressure, the sample sintered at 960°C. There are a few test to investigate the performance of the samples. The sample characterizations are analyzed by using three technique such as XRD,XRF and resistivity measurement. The techniques are X-ray diffraction analysis to determine the structure and phase formation of the sample while the multi-meter to measure the resistivity of the sample at room temperature and XRF for elemental analysis. The sample with highest composition that is 0.12 wt% has the highest resistivity. XRD analysis result shows that the sample structure are slightly changing with the addition element NiO with composition NiO 0.08, 0.10, and 0.12 wt%. All the sample have orthorhombic structure and have different lattice parameter. The XRD also shows the addition element has decrease the sample porosity. To measure the sample resistivity, multi-meter was used in room temperature. A type of circuit was built and a battery with a voltage of 1.3 was used. At composition 0.08 wt%, the resistivity measure was 5.7307×10^{-3} Ωm. At composition 0.10 wt%, the resistivity calculated was 9.5513×10^{-3} Ωm. At composition 0.12 wt%, the resistivity calculated was 17.1924×10^{-3} Ωm. XRF analysis shows that there are a very small impurities in the sample. From the tests result, the higher the composition of NiO, the higher the resistivity of the sample. This can be proven by the reading of the resistance by the multimeter test.

KESAN PENAMBAHAN NIKEL OKSIDA KE ATAS SIFAT-SIFAT YBCO SUPERKONDUKTOR DISEDIAKAN MELALUI TINDAK BALAS PEPEJAL

Abstrak

Dalam projek ini, YBCO ditambah dengan bahan nikel oksida. Sampel YBCO ini telah disediakan dengan pencampuran serbuk bahan kimia. Kemudian, bahan tersebut dikisar, dan dipanaskan pada suhu 960°C. Selepas ditekan pada kadar tekanan 1 tan, pelet tersebut dibakar pada suhu 900°C. Beberapa ujian telah dijalankan untuk menguji prestasi sampel-sampel tersebut. Pencirian sampel dilakukan dengan menggunakan dua kaedah. Kaedah-kaedah tersebut ialah ujikaji multimeter untuk mendapatkan rintangan elektrik pada suhu bilik dan pembelauan sinar-X (XRD) untuk menentukan struktur dan fasa sampel dan juga XRF untuk analisa komposisi bahan. Komposisi 0.12 bt% mempunyai kerintangan yang paling tinggi. Keputusan pembelauan sinar-X (XRD) menunjukkan bahawa struktur sampel mengalami sedikit perubahan dengan penambahan NiO daripada komposisi x=0.08 bt% sehingga x=0.12 % bt. Semua sampel mempunyai struktur ortorombik dan mempunyai ukuran kekisi yang tidak sama pada *a*, *b*, *c*.. XRD juga menunjukkan bahawa nikel oksida telah mengurangkan keporosan sampel. Untuk mengukur kerintangan sampel tersebut, multimeter telah digunakan dalam suhu bilik. Sejenis litar telah dibentuk dan satu bateri yang mempunyai voltan sebanyak 1.3v telah digunakan. Pada komposisi 0.08 bt%, kerintangan yang telah dicatatkan setelah membuat pengiraan adalah, 5.7307×10^{-3} Ωm. Pada komposisi 0.10 bt%, kerintangan yang telah dicatat adalah 9.5513×10^{-3} Ωm. Pada komposisi 0.12 bt%, kerintangan yang dicatat ialah 17.1924×10^{-3} Ωm. Ujian meter pelbagai yang dijalankan memberikan gambaran bahawa kerintangan elektrik pada suhu bilik meningkat secara berperingkat dengan komposisi x = 0.08, 0.10 dan 0.12 % bt. Analisa XRF menunjukkan sedikit bahan asing telah dikesan dalam sampel. Daripada keputusan ujian yang telah dilakukan, rintangan meningkat apabila komposisi bahan tambahan meningkat.

TABLE OF CONTENTS

LIST OF FIGURE

LIST OF TABLE

LIST OF ABBREVIATIONS AND SYMBOL

K	Kelvin
F	Force
Q	Electrical charge
B	Magnetic Field
Jc	Current density
Eg	Energy gap
eV	Electron voltage
Bc	Critical Field
Tc	Critical Temperature
Hc	Magnetic Field
Å	spectral lines of the visible spectrum
δ-	change in velocity
wt %	Weightage
°C	Celsius
λ	wavelength
h,k,l	miller indices
V	voltage
Ω	resistance

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The mercury was first experimented for resistance by Heike Onnes. It was discovered that the Helium reach zero resistance when under transition temperature. It means that the material loss all of its resistance under 4.2 K. Superconductivity, is a phenomenon of a zero electrical resistance and repulsion of magnetic field when cooled below critical transition temperature. In normal material, the resistance of metal decreases as the temperature decreases but there are still resistance even at near zero. In superconductor, the resistance drop to zero when it reached the critical transition temperature. Their conducting properties may be altered in useful ways by the deliberate introduction of impurities into the crystal structure (Rana et al., 2016). The difference between the superconductor and the usual conductor can be observed based on their magnetic properties (Rana et al, 2016). There are magnetic fields around the superconductor that can cause it to lift small over the surface when immersed in liquid nitrogen. This phenomenon is called the Meisner Effect. The conductivity of the superconductor is determined by the electrical properties, magnetic properties and structural morphology of the YBCO (Volochova, 2013).

1.2 Problem Statement

The superconductor is mainly used because of the magnetic properties and in the electrical application (Ruppert, 2013). In regular conductor, there are a lot of resistance and this will cause the energy loss to the surrounding. The use of superconductor will solve this but it has to be in their critical temperature. The superconductor was limited by many factors such as low transition temperature, surface defects, and high cost of maintenance due to low transition temperature. These factors can limit the performance of the superconductor in industry. In order to solve this, there are many research has been done to improve the superconductor properties. It was proven by the previous research that by adding a new element to the superconductor, the properties of the superconductor can be enhanced (Volochova et al, 2014) . The superconductors have become a very effective material in electronic application. In this research, the superconductor was added with NiO_2. There are researches that suggesting the addition element will improve the phase formation of the YBCO (Rana et al, 2016).

1.3 Objectives

The objectives of this research are:

i. To study the phase formation of YBCO with NiO addition using X-Ray Diffraction and X-Ray Fluorescence.

ii. To identify the resistivity of YBCO added with NiO at room temperature.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of YBCO

Yttrium-Barium-Copper-Oxide (YBCO) is a type II high temperature superconductor. The superconductivity is depending on the hole and electron concentration present. Previous research finds that YBCO has highest transition temperature (T_c) that is 92 K (Vidya et al., 2011). The unit cell of the YBCO is an orthorhombic structure (Ivan, 2016). The CuO_2 molecule are interconnected with CuO_3 and then provide additional electron density (Rana et al., 2016). The YBCO in bulk form can lift a small magnet when immersed in nitrogen liquid. This is caused by the repulsion of the YBCO magnetic field. This phenomenon is called Meisner Effect. YBCO is a critical temperature and single grain superconductors (Volochova et al., 2014). There are various methods to prepare YBCO such as sol–gel method and solid state reaction. In this research, the method used for synthesize the superconductor is solid state reaction. The YBCO has two main properties that are structural properties and magnetic properties. Addition to the YBCO superconductor will make it has higher transition temperature and higher critical current density and can influence other properties (Volochova et al, 2013). The addition of NiO should be in optimum to lead for better samples.

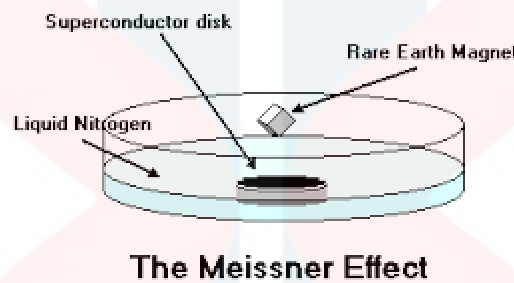

Figure 2.1 Levitation of Superconductor

(imagesco, 2007)

The defining parameter of superconductor is it must be capable to allow large amount of electricity to flow. The YBCO contain CuO_2 that produce anti-ferromagnetism properties. Superconductors are now can be combined with other elements such as Ferro-magnets to enhance the conductivity by modified the magnetic flux lines (Philippe et al., 2014). In other research, addition of element will decreasing the band gap which improves conductivity (Dadras, 2016). Besides that, many of the research shows that impurities in YBCO increase the critical current density of the YBCO which contribute to conductivity of the YBCO (Volochová et al., 2013).

Based on the research done, there are slightly changes in the structure and formation of the superconductor after addition element was added. The crystallite size of the superconductor are increased after addition of Sm (Rana et al., 2016). Increase in crystallite size can be considered as good crystallinity. In other research, it shows that impurity atoms substitute atoms in the crystal lattice of superconductor, suppress conductivity and act as additional pinning center (Li et al, 2015). But there is also research that shows that high composition of Sm can cause to shorter strain .It was cause by the large substitution of Sm ions in Barium lattice. The shortening of the strain might be cause by defects such as vacancies and random distribution of the ions (Rana et al., 2016). In other research, there is also stated that the superconductor with addiition elements have better crystallization (Sun et al., 2015).

2.2 Addition of element in superconductors

The movement of the vortices can cause energy loss in a superconducting material. If CuO_2 is not developed, the flux tends to move because the current flow along the vortex is according to the Lorentz law in equation 2.1.

$$\vec{F} = q\vec{v} \ x \ \vec{B}$$

Equation 2.1

As noted, it is very important to make sure the current through a superconducting material can flow at zero resistivity. Theoretically, the current density of the type two superconductor was higher (Fuger et al, 2010). However, the Jc value can be increased if the vortex motion can be stopped by flux pinning so that the vortex does not move. Flux pinning carried by adding impurities into the superconducting material (Lee et al, 2011). The overall critical current density in a superconducting material is also determined by temperature and the resistance.

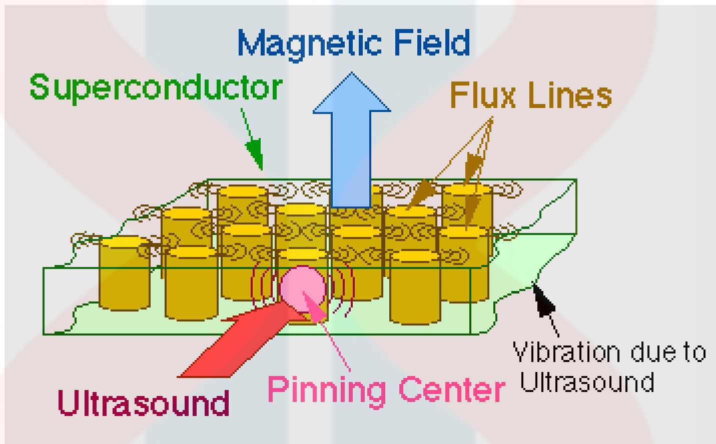

Figure 2.2 Flux pinning center in superconducting material

(Harada, 2003)

Zero resistance is one of the most important parameter that must be focused in order to create a good superconductor (Fallis, 2013). A superconductor must be able to conduct large amount of electricity without releasing heat. Generally, superconductor will reach to zero resistance below transition temperature and it is different by the materials composition. The current that flow in the superconductor is known as current density. The current will create magnetic field around the superconductor. The current density of superconductor are depends on the dimension, microstructure and the flux pinning (Lee et al, 2011).

Based on a few research done before, it shows that Samarium can increase the superconductivity by adding flux pinning center to the superconductor (Lee et al., 2011). Also stated in the research that, the current density of the superconductor are influenced by the concentration of the addition element (Lee et al., 2011). In other research did mention that current density and flux pinning can be improved by adding other elements (Meijuan et al, 2015). The magnetic field around the superconductor makes it suitable for applications that involved high magnetic fields and high temperature such as electronic device.

2.3 Cooper Pairs Theory

The electron pairs move in an orderly manner without causing energy loss. This allows the electron pairs move without any resistance (Pair, 2016). As a result, the two electrons are bound together to form Cooper pairs. Cooper pair bonding force called energy gap Eg is 10-3eV, which will cause low-temperature superconductors.

At transition temperature, the vibration of the virtual phonon is gone. The virtual phonon will produce positive charge between them (Nave, 2010). Electron which has negative charge will attracted, thus will produce two electron moved at the same time. The two electron pairs are called Cooper pairs. Part of Cooper pairs will split and cause excess electrons interact with the other Cooper pairs. This will cause the electron move in straight line without colliding with the lattice atom (Pair, 2016).

While superconductors are cooled to very low temperatures, electrons of a Cooper pair still formed. When superconductors absorb heat, vibrations in the lattice will grow and break Cooper pairs and eliminate the conductivity properties of the material.

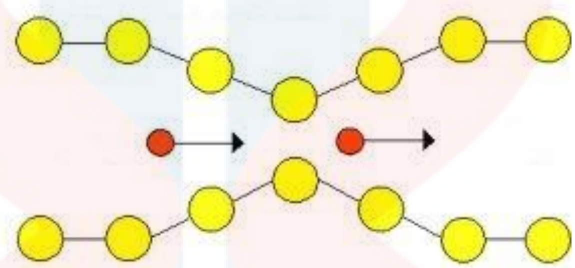

Figure 2.3 The establishment of cooper pairs in the lattice of positively charged ions (Bob Emery, 2002)

Superconductivity can be explained by three factors, namely, the critical temperature (Tc), critical field (Bc) and the critical current density (Jc). Zero resistance phenomena are very important characteristic for superconducting materials(Li et al., 2015). The resulting currents in superconducting coils will causes everything flows without resistance and heat loss. Superconducting materials will have zero resistance below a critical temperature (Tc) (Li et al., 2015). This critical temperature is different for each composition.

The current flowing through the superconductor material is known as the critical current density (Jc). The critical current density is influenced by temperature

(Volochová et al., 2013). The higher resistance, the higher the temperature obtained. The current flows through the material to produce a magnetic field.

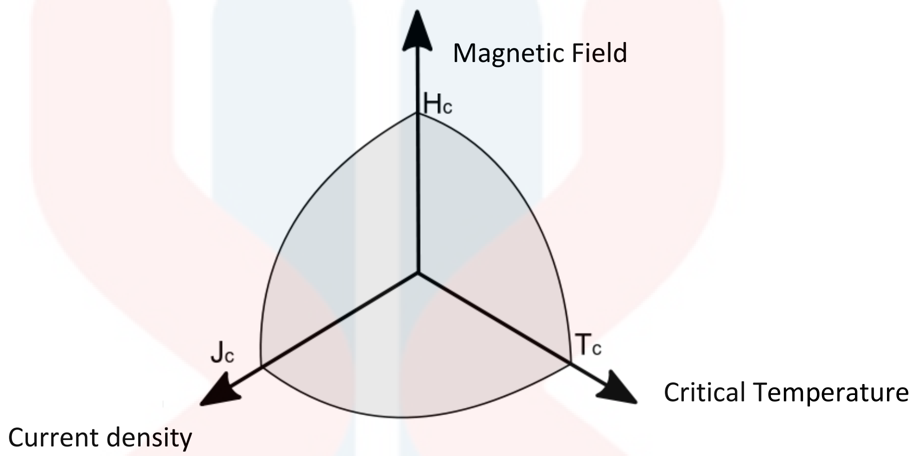

Figure 2.4 The relationship between the parameters Hc, Jc and Ic

2.4 YBCO Unit Cell System

High temperature superconductors LBCO found in 1986 is a CuO based superconducting material (Coombs, 2012). This material is the beginning of the high-temperature superconductor discovery. The CuO is the base of the superconductor (Nave, 2010).

YBCO is a compound of the formula $YBa_2Cu_3O_7$. It Have a Y_{123} phase. YBCO superconductor is a material which has a critical temperature (Tc) above the temperature of liquid nitrogen (77K) and reaches a temperature of 93 K (Bednorz, 2009). Superconductor YBCO has two layers CuO_2 and the CuO_2 chains. In 1987, YBCO material was discovered. Y represents part of rare-earth elements. This material has a rather complicated structure and its oxygen content plays an important role in achieving superconductivity orthorhombic Y_{123} phase structure. Dimensions for YBCO is a = 3.82 Å, b = 3.89 Å and c = 11.68 Å. Reduction of oxygen in the YBCO layer plays an important role in CuO_2. Moreover, the Y atom layer located between layers CuO_2. When the position for the Y and Ba are like Ba-Y-Ba in the structure of YBCO. There is also a chain between the Cu-O-Cu in the crystal (Figure 2.5).

YBCO is a compound of the formula $YBa_2Cu_3O_7$. Having a Y_{123} phase, YBCO is also trying to show high superconductivity. Existence of Cu-O layer is important in YBCO because the layer will increase superconductivity (BCS Theory and Superconductivity, 1972). However, with the discovery of high-temperature superconductors, it can be shown that Cu-O chains should not be taken serious. The chain is only acting as a storage charge for the structural element. Valence for Cu plays an important role in YBCO . This is because, the electronic structure identified by the valence Cu.

In addition, the structure of the $YBa_2Cu_3O_{7-\delta}$, where $\delta = 0$, indicate where sample YBCO is in orthorhombic phase. When, if $\delta = 1$, YBCO are in the tetragonal phase. Only orthorhombic configurations are superconductors.

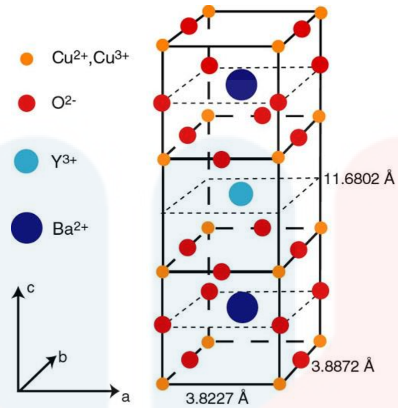

Figure 2.5 $YBa_2Cu_3O_{7-\delta}$ Schematic

(chemwiki,2016)

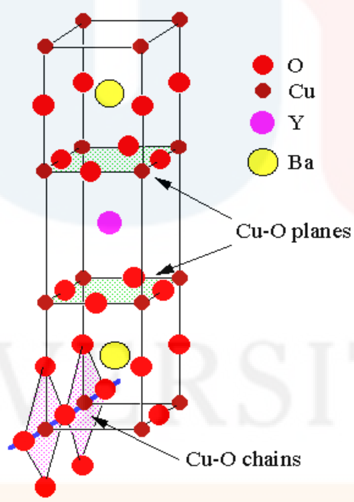

Figure 2.6 Schematic $Yba_2Cu_3O_{7-}$ with CuO and CuO_2 layer

(chemwiki,2016)

2.5 Orthorhombic System

The crystal structure has the systems that it can be categorized. In crystal structure, the system determine by the length of lattice parameters. The orthorhombic structure has different *a*, *b* and *c* number. The orthorhombic has three same perpendicular axis with all of them are different length. If the cell are rotated, it will not changing the appearance of the unit cell. The orthorhombic are usually found in superconductor compound (Ivan, 2016).

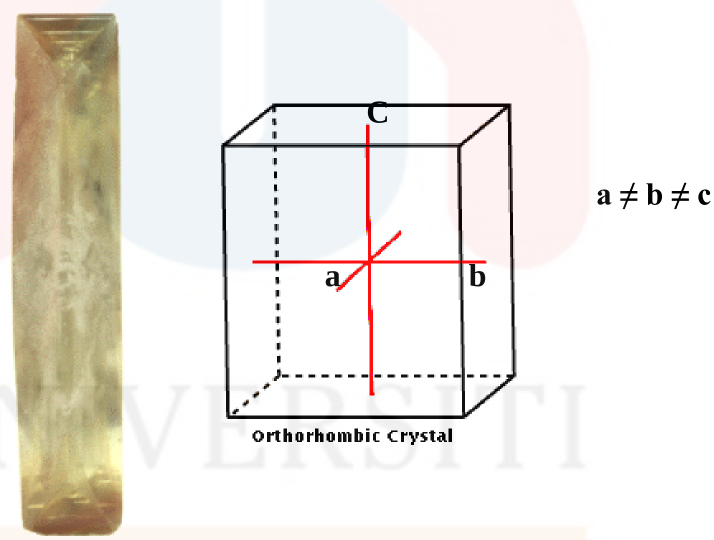

Figure 2.7 Orthorhombic structure

(ledajewelco, 2014)

2.6 Ohm's Law

The Ohm's Law are the basic principle of electricity and current flow. It is important to acknowledge the basic principle of electricity. The ohm's law related the three unit that is current, voltage and the resistance. Three basic units are using the same source of movement that is electron. Voltage is a potential difference between two points that required energy two move the electron. One point is higher than the other point and that provide charge to other point. The voltage is used as a pressure for the current to flow. The voltage are usually sourced from the battery. The higher the voltage, the amount of current can flow. Resistance is the barriers that have to be passed through by the current. The higher the resistance, the lower the current can flow. By using a good conductor, the resistance can be decreased to allow large amount of current. Large amount of current are also provided by high voltage. Current is the electron carrier that ensured the electricity is there. The electron carried by the current are flow through by the pressure from the voltage and electron flow are lower in high resistance material. These three unit that is voltage, current and resistance can be related by using the equation below.

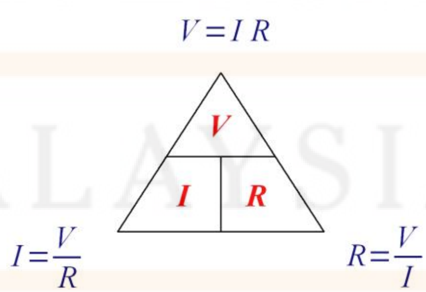

Figure 2.8 Ohm's Law

(R.Nave, 2016)

CHAPTER 3

MATERIALS AND METHOD

3.1 Materials

The materials that have been used are Yttrium oxide, copper oxide, barium carbonate and nickel oxide. There are samples characterization using X-Ray Diffraction (XRD), multi-meter and X-Ray fluorescence. The added element that was nickel oxide are added at different composition with percentage of 0.08%, 0.10% and 0.12 % weightages.

The sample $YBa_2Cu_3O_2(NiO_2)x$ with addition of nickel are prepared by solid state reaction. The yttrium oxide powder has purity until 99%, copper oxide also with purity of 99%, and barium carbonate with purity of 99%. All the powder mixed according to different composition of nickel oxide. The powder weighed to the desired composition and grind in the mortar.

3.2 Sample preparation method

Sample preparation of high temperature superconductor $YBa_2Cu_3O_2(NiO)_x$ with the addition of nickel oxide (NiO_2) with composition x = 0.08, 0.10 and 0.12 wt % prepared by the solid state reaction. All three mix of this powder in accordance with the desired composition. Stoichiometric Equation 3.1 below is a guide to calculate the required composition.

$$\frac{1}{2}(Y_2O_3) + 2(BaCO_3) + 3(CuO) \rightarrow YBa_2Cu_3O_{7-\delta} \qquad \text{Equation 3.1}$$

The solid powder materials are mixed into mortar and grounded homogenously for two hours. The grounded materials are then undergoes calcination at 900°C for 12 hours (Bu et al, 2014). After calcination, NiO_2 added to the mixture with different composition that is 0.08, 0.10, 0.12 wt %. The mixture grounded for one to two hours before pellet preparation. 1 tonne of hydraulic pressure in about 10 minutes is used to press the powder mixture into pellet. It was important that the pellet must show no cracks on the surface. Then, the pellet were sintered and annealed at 950°C for the particles of the powder weld together. Lastly, the sample are ready for characterization.

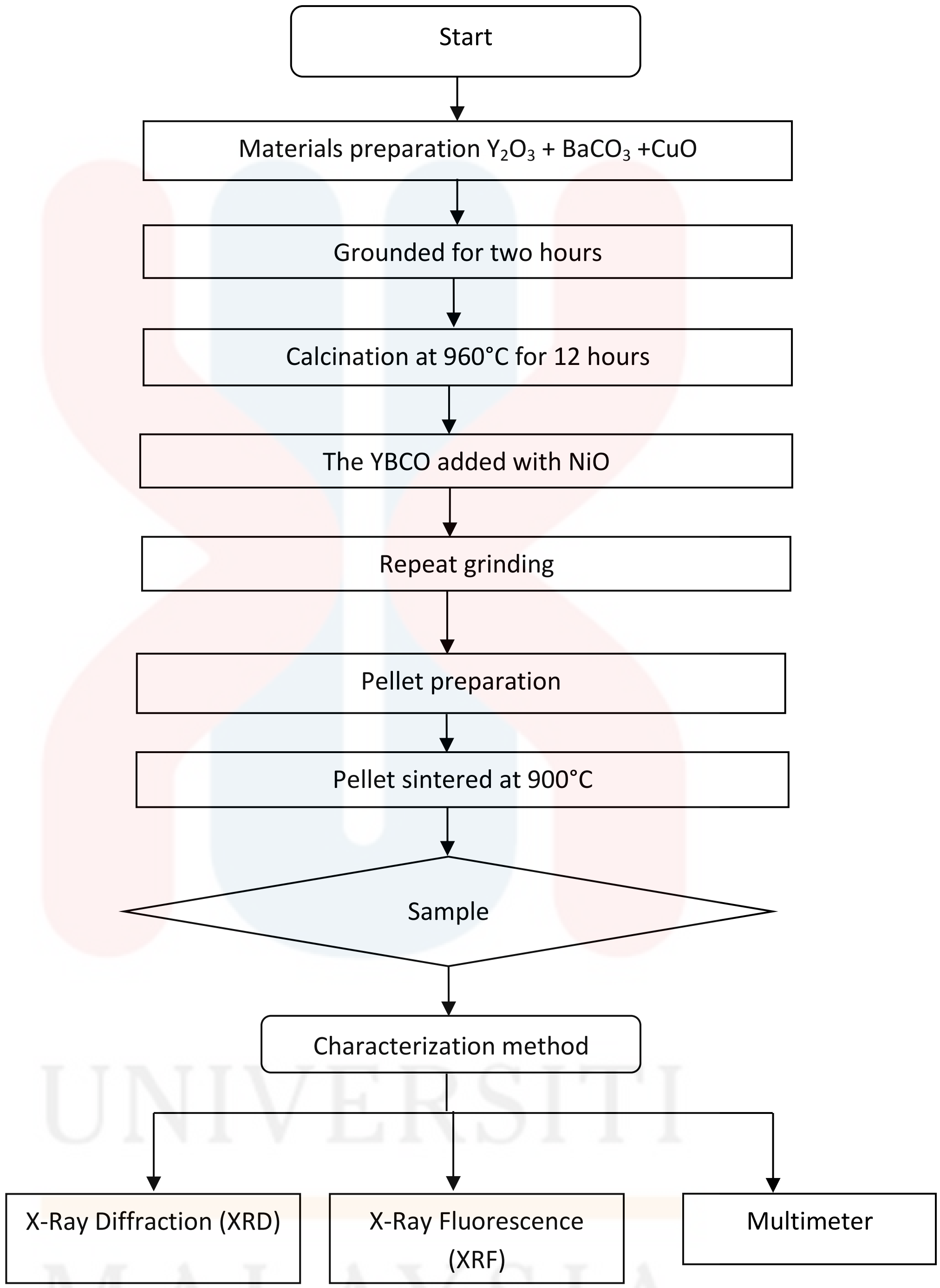

Figure 3.1 Process flow of YBCO synthesize

3.3 Sample characterization

The sample was characterized by using three techniques such that XRD, multimeter and XRF. The XRD characterization was to analyzed the crystal structure and the phase formation of the sample. Multimeter was used to measure the resistance on surface at room temperature. XRF was used for elemental analysis of the sample.

3.4 X-Ray Diffraction

X-Ray Diffraction was used to determine the structure and phase formation of the sample. The sample will be crushed until become powder form for a better results. The XRD shows the diffraction pattern when x-ray source hit the sample and reflected to the x-ray detector. The diffraction pattern can be considered as the fingerprint of the sample.. It was stated in the previous researches that by adding impurities to the YBCO sample, there will be slightly changes in microstructure of the YBCO (Volochova et al., 2014). The a, b and c number of the YBCO samples are should be different and shows the orthorhombic structure. The addition of NiO might be slightly changes the phase formation of the YBCO samples.

For this characterization, the XRD machine was handled by the laboratory assistant who was provided with radiation safety training. The XRD machine used x-ray radiation so only authorized person can operate the machine. The laboratory assistant extracted the data by using D2 Phase XRD machine. The sample was scan at 2θ from 5° to 80°. Then, the raw data analyzed by using EVA software. To analyze the data, the pattern from the graph was compared to the ICDD data.

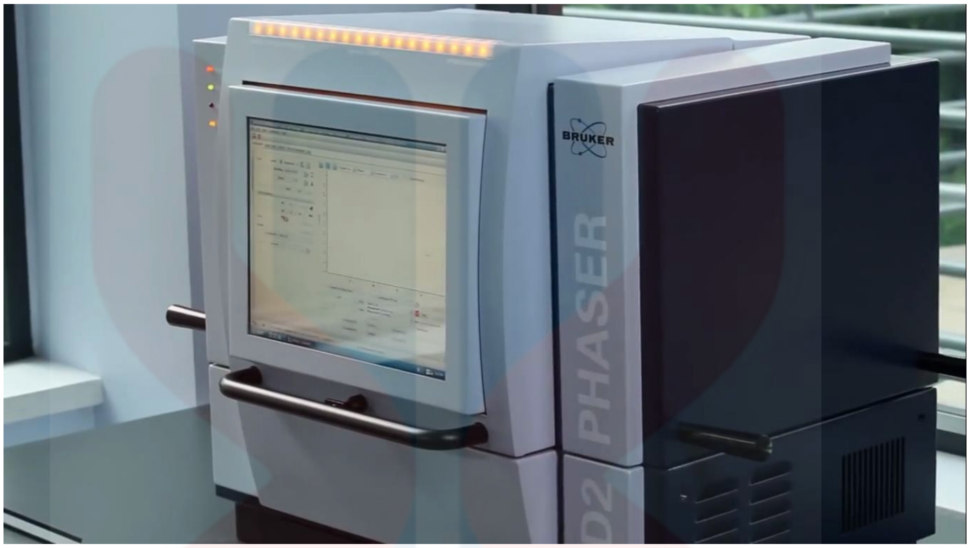

Figure 3.2 D2 Phaser XRD machine

Figure 3.3 The sample was placed in the machine

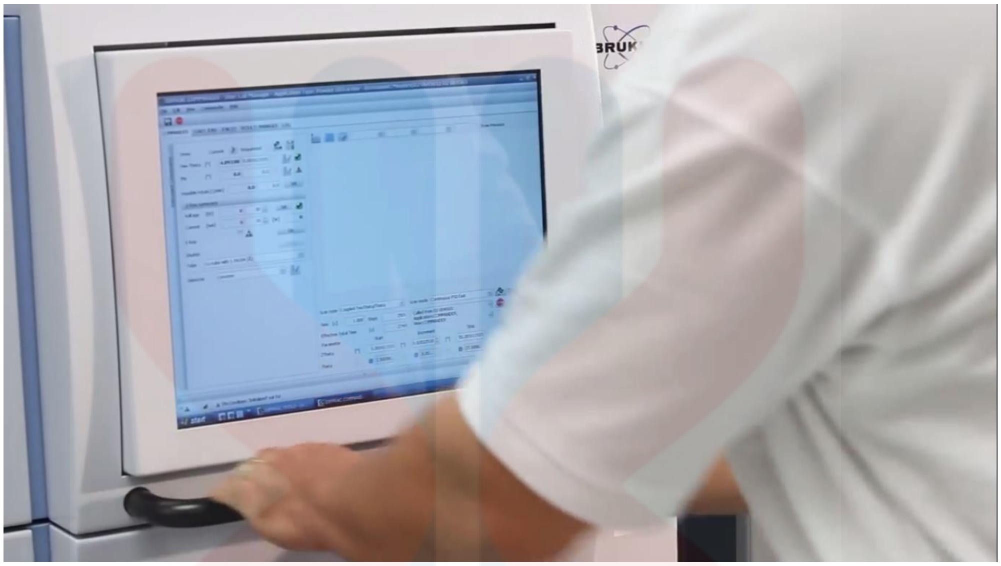

Figure 3.4 The sample ready to be scanned

3.5 Multimeter

Multimeter was used to measure the resistance and the current of the YBCO sample. Although the YBCO are believed to have zero resistance, there will be small amount of resistance in the sample because of the imperfection such as impurities in the sample. The method is used to measure the resistivity at room temperature. A circuit is made and connected to the sample. A dry cell was used as the fixed voltage that is AA battery with 1.3 volt. A circuit was built to run this test. The wire was attached to the sample and the multimeter was attached to the sample to take it resistance reading.

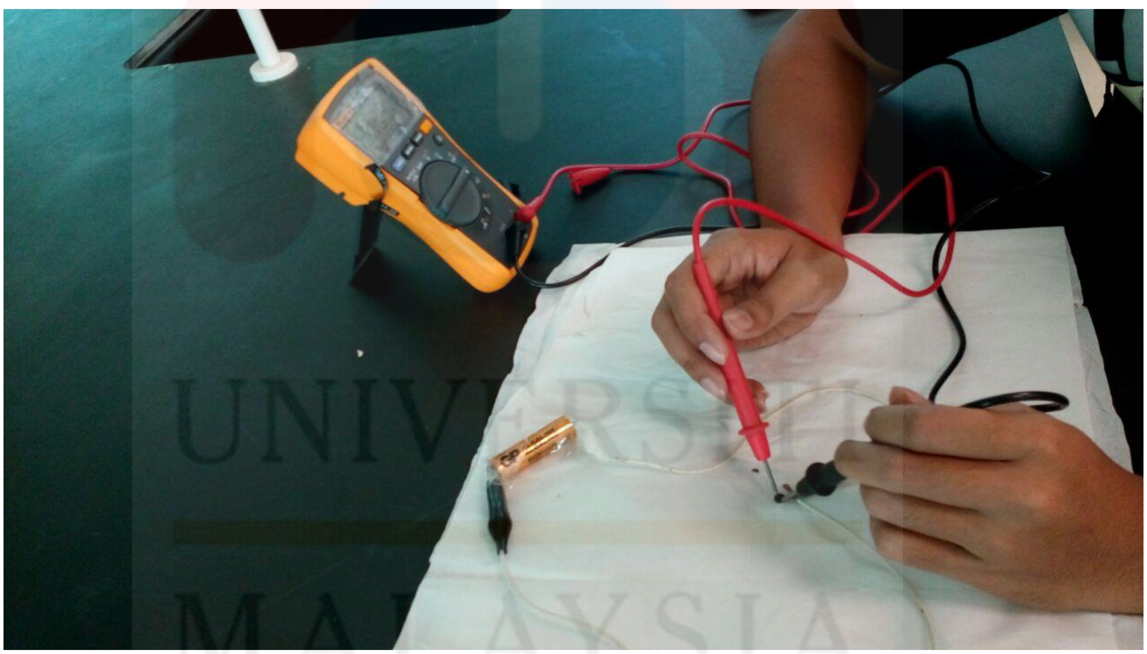

Figure 3.5 Circuit used for resistance at room temperature test

3.6 X-Ray Fluorescence

The XRF characterization was to analyze the element of the sample. Although calculation have been made before synthesize the sample, there might be existence of impurities. It is important to ensure the purity of the sample so that it was not affected the performance of the sample. The sample was characterized in form of powder. The machine used for this characterization was Bruker S2 Phaser. This machine was using x-ray radiation so only authorized person could operate the machine. There are no specific parameters should be considered to run the test. The machine could identified and measure the composition of all the elements in the sample.

Figure 3.6 S2 Phaser XRF machine

Figure 3.7 Computerized setting analysis

Figure 3.8 The sample placed in the chamber

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

In this study, the sample prepared was YBCO superconductor ceramic and addition material used is nickel oxide, (NiO). Different composition of NiO x=0.08, 0.10, 0.12 wt%. are used so we can see the different changes of superconducting materials for samples YBCO(NiO)x. The characterization on the sample that is the X-ray diffraction (XRD) to determine the structure and phase samples, X-Ray fluorescence (XRF) to determine the element composition, and the multi-meter to determine the resistance at room temperature. The sample was prepared by solid state reaction method. All of these characterization results are displayed in the form of graphs, tables, and calculations. In addition, the comparison of the data in the same experimental methods made and changes to the sample was recorded. Problems encountered and the factors that affect the accuracy of the data also discussed.

4.2 XRD Analysis

The XRD analyze the YBCO powder for pure and the addition materials NiO. The graph patterns are constructed by using Origin software. Based on graph plotted in Figure 4.1, the graph shows the phase and the structure of the pure YBCO sample and the YBCO with different ‘x’ composition that is 0.08, 0.10, and 0.12. The highest peak of the pure YBCO sample graph at 2θ is at 24.811. The interplanar spacing (*d)* at the highest peak is 3.49932. The *h, k, l* values of the highest peak are (-2 -1 -1). The structure of the sample are orthorhombic as they have different *a, b,* and *c* value. The lattice parameter of pure YBCO sample was *a*=12.1792, *b*=5.6590 and *c*=7.132.

For the YBCO with 0.08 wt% of NiO, there is almost no change in crystal structure and the phase formation of the sample. But there are small changes in the lattice parameter sizes of the sample. The lattice parameter is *a* = 12.188, *b* = 5.662, and *c* = 7.132. The addition of the nickel oxide does not change the structure and the system of the superconductor that is orthaorhombic. There are peaks altered that showed the interference of NiO in the YBCO sample.

The third sample was the YBCO with composition 0.10 wt%. The lattice parameter for this sample was *a* =12.1792, *b* = 5.659, *c* = 7.1325. Just a small changes from the 0.08 wt% sample, and the peak are almost the same. The structure and the system of the sample are still orthorhombic. Thus, the sample YBCO with addition 0.10 wt% are still have superconducting properties.

Table 4.1 Lattice parameter of YBCO added with NiO at different composition

Sample (Wt %)	Lattice Parameter *A*	Lattice parameter *b*	Lattice parameter *C*
Pure YBCO	12.17920	5.65900	7.1325
YBCO with 0.08 wt% NiO	12.18800	5.66200	7.13200
YBCO with 0.10 wt% NiO	12.17920	5.65900	7.13250
YBCO with 0.12 wt% NiO	14.66730	5.59250	5.18880

The last sample was YBCO with 0.12 wt% addition. The lattice parameter of the sample are *a* = 14.6673, *b*= 5.59250, and *c*= 5.18880. . The table 4.1 above shows the lattice parameter of the sample. The differences are bigger in this sample but the system are still orthorhombic. With 0.12% addition, the YBCO sample still have superconducting properties but starting to change to tetragonal system structure. Tetragonal system structure are not superconducting.

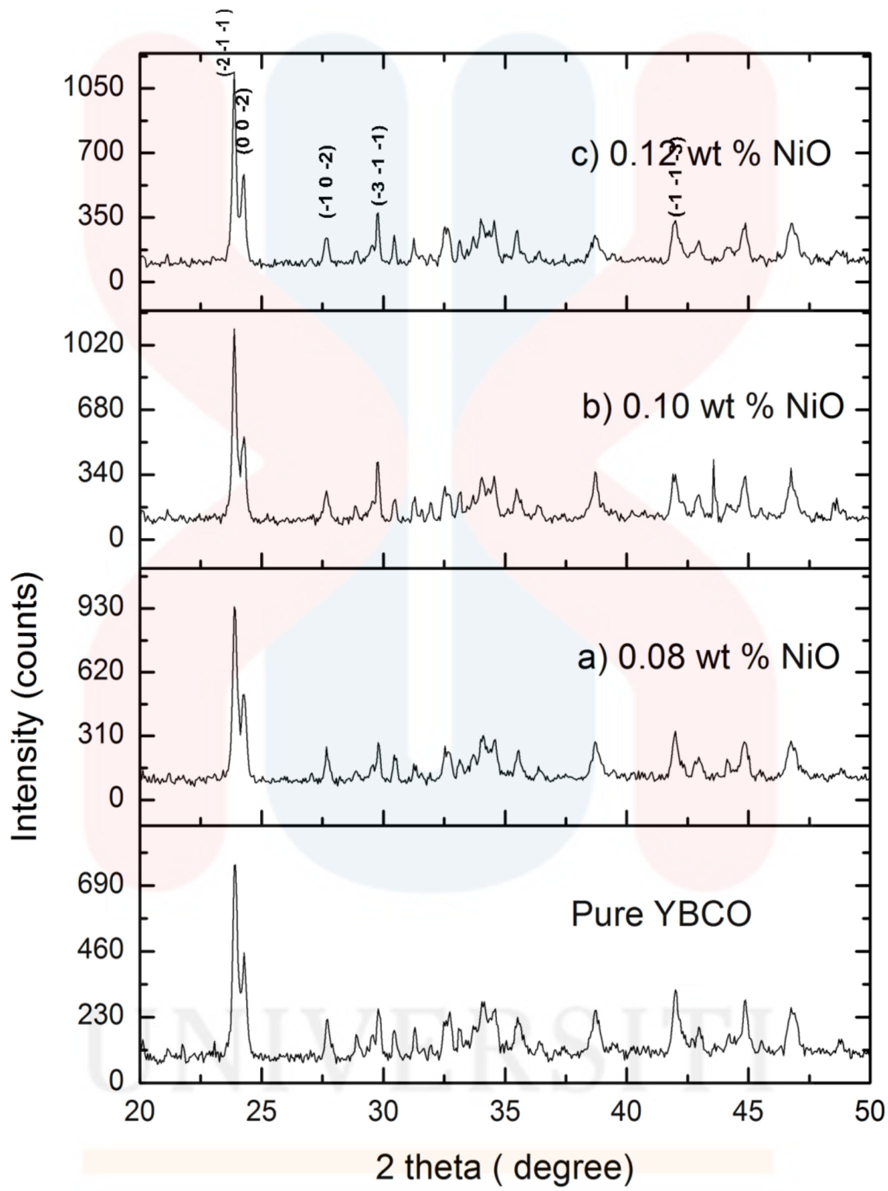

Figure 4.1 XRD pattern of YBCO added with NiO at 0.08, 0.10 and 0.12 wt%

4.3 Electrical resistance measurement at room temperature

Electrical resistivity test performed on four samples of superconducting YBCO to get the electrical resistivity at room temperature, with the different composition categories, namely x = 0.08, 0.10 and 0.12% wt and pure YBCO. Through this test by using multi-meter, the voltage values were fixed by using dry cell. The electrical resistivity at room temperature for three samples of different composition can be calculated based on the calculation shown in appendix. The value of electrical resistance at room temperature obtained during the experiments shown in Table 4.2. Meanwhile, the graph in Figure 4.2 shows the electrical resistivity at room temperature, ρ against the composition x = 0.08, 0.10 and 0.12% wt. The highest resistivity was at composition 0.12 wt%.

Table 4.2 The resistivity ρ for YBCO with NiO composition 0.08%, 0.10%, 0.12% weightage

Composition of NiO, % wt.	Resistivity at room temperature (ρ) ($\times 10^{-3} \Omega m$)
Pure YBCO	1.9103
0.08	5.7307
0.10	9.5513
0.12	17.1924

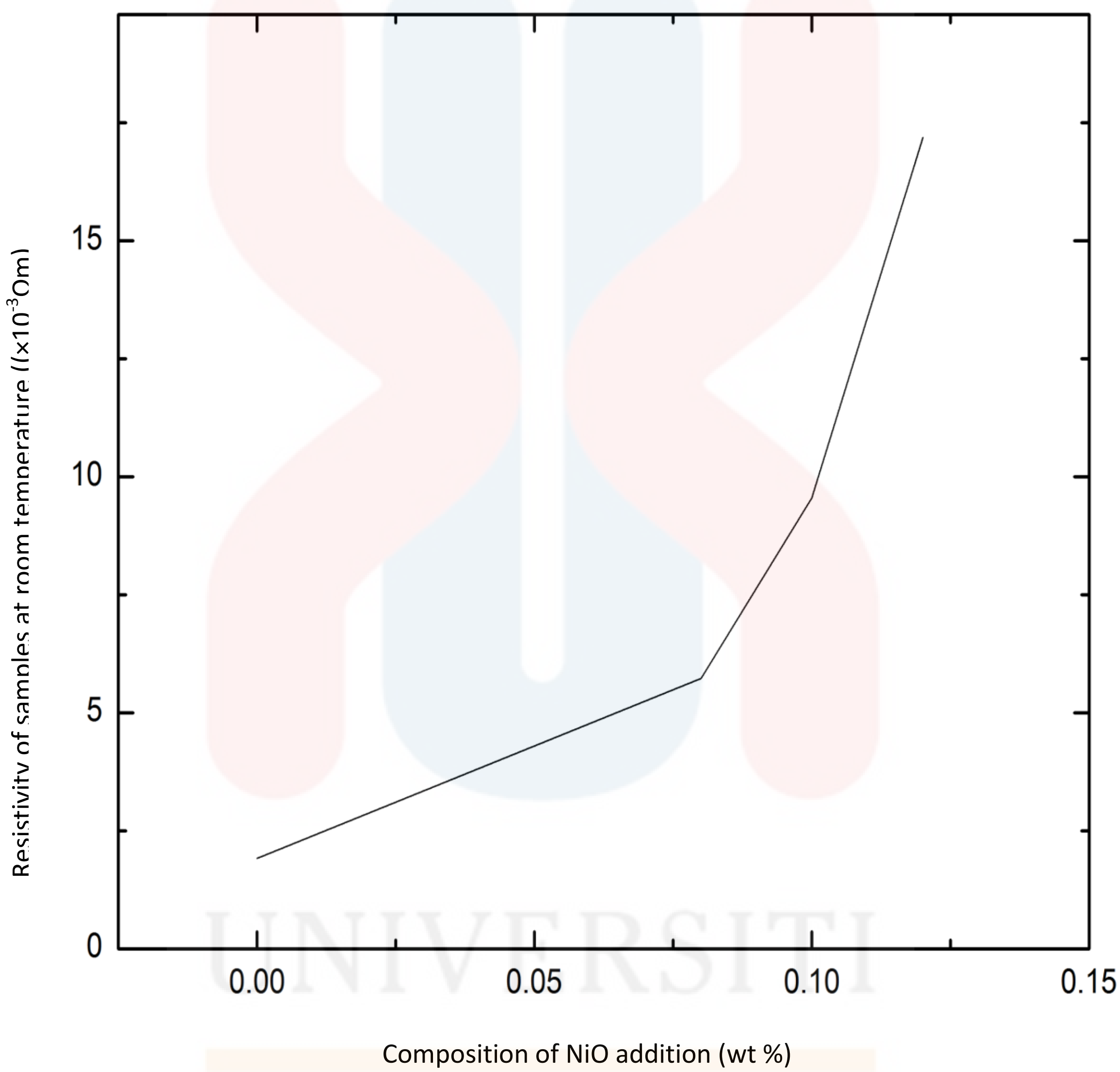

Figure 4.2 Graph of resistivity at room temperature against the NiO addition composition

Based on the calculation of the resistivity at room temperature, it was obvious that the resistance of the sample increase when the composition of the NiO increase. The presence of the NiO may increase the barriers of the atom and increase the band gap. The composition of addition NiO was too high and affected the conductivity of the samples. The barriers for the electron to travel increase the resistance of the sample (Volochova et al, 2014). The resistance of the sample depends on the microstructure and the flux pinning of the sample(Lee et al, 2011).

4.4 XRF analysis

From the XRF result shown, the compositions of the oxide element are not consistent. The composition of Y_20_3, BaO and CuO are having a little bit differences at each composition. But the percentage of the addition element NiO was increasing as the composition increased. It shows the consistent trend of the NiO addition. There was also other elements was detected in the samples such as ZnO, TiO, and CaO but in very small percentage. The other elements might come from surroundings and the mortar used for grinding. These impurities might affected the performance of the YBCO superconductor.

Table 4.3 Composition of Pure YBCO

Elements	Percentage(%)
Y_2O_3	17.2
BaO	40.6
CuO	33.5
NiO	0.0
Other impurities	8.7

Table 4.4 Composition of YBCO with 0.08 wt %

Elements	Percentage(%)
Y_2O_3	24.70
BaO	37.50
CuO	22.70
NiO	8.12
Other impurities	6.98

Table 4.5 Composition of YBCO with 0.10 wt %

Elements	Percentage(%)
Y_2O_3	23.50
BaO	33.12
CuO	23.10
NiO	11.05
Other impurities	9.01

Table 4.6 Composition of YBCO with 0.12 wt %

Elements	Percentage (%)
Y_2O_3	19.30
BaO	40.13
CuO	20.60
NiO	13.13
Other impurities	6.83

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This researched was to identified the performance of the YBCO superconductor with NiO addition. The phase formation of the sample shows small changes of lattice parameter in different composition NiO addition but still showed $a \neq b \neq c$ that was orthorhombic. All the samples were having superconducting properties. The resistance of the sample increased when the composition of the NiO increasing. Based on the graph, it can be seen that the electrical resistivity at room temperature increased with the increasing of NiO composition. There are might be defects in the sample or the synthesizing process that affected the performance of the sample. It was stated earlier that the composition might give effect to the conductivity of sample.

There are not many changes in the phase formation structure of the sample. The sample still maintained its orthorhombic structure which is very important in superconductor. The NiO altered the peak but most of the peak share the same *h, k, l* number. Small changes in lattice parameter of all the sample but it still shows that $a \neq b \neq c$. Thus, the crystal structure of all the samples are orthorhombic. The samples are having a very small impurities other than wanted addition element. The impurities might came from surrounding during sintering, grinding and pelleting. A very tight precaution has to be made to avoid this in next project.

5.2 Recommendation

For further research, start with small amount of addition element. Also, there should be various type of element to be used as addition element such as gold or scandium. The outcome of this research might be different by changing the method. For example, the sol-gel method could be used to synthesize the YBCO sample. The time of calcination for this research was 12 hours. There might be differences if the time of calcination were longer. Besides, the right equipment to characterized the YBCO that was four probe are not used. Furthermore, the defects or impurities might affect the sample in crystal structure.

REFERENCES

BCS Theory and Superconductivity. (1972), (9).

Bednorz, J. (2009). High-temperature superconductivity, (January 1987), 1–9.

Bu, J., Jönsson, P. G., & Zhao, Z. (2014). Author ' s Accepted Manuscript. *Ceramics International*. http://doi.org/10.1016/j.ceramint.2014.10.177

Coombs, T. (2012). *Bulk high temperature superconductor (HTS) materials*. *High temperature superconductors (HTS) for energy applications*. Woodhead Publishing Limited. http://doi.org/10.1533/9780857095299.1.101

Dadras, S., & Ghavamipour, M. (2016). Investigation of the properties of carbon-base nanostructures doped YBa2Cu3O7−δ high temperature superconductor. *Physica B: Condensed Matter*, *484*, 13–17. http://doi.org/10.1016/j.physb.2015.12.025

Fallis, A. . (2013). No Title No Title. *Journal of Chemical Information and Modeling*, *53*(9), 1689–1699. http://doi.org/10.1017/CBO9781107415324.004/

Fuger, R., Eisterer, M., Oh, S. S., & Weber, H. W. (2010). Superior properties of SmBCO coated conductors at high magnetic fields and elevated temperatures. *Physica C: Superconductivity and Its Applications*, *470*(5-6), 323–325. http://doi.org/10.1016/j.physc.2010.01.060

Ghazanfari, N. (2007). Properties of YBCO superconductors prepared by ammonium nitrate melt and solid-state reaction methods u Ozkan, *428*, 1–7. http://doi.org/10.1016/j.jallcom.2006.03.038

Lee, Y., Yamasaki, H., & Furuse, M. (2011). Magnetic-field angle dependent critical current densities and flux pinning in commercial YBCO tapes below liquid nitrogen temperatures. *Physica C: Superconductivity and Its Applications*, *471*(21-22), 1017–1020. http://doi.org/10.1016/j.physc.2011.05.113

Li, G.-Z., Wang, M., & Yang, W.-M. (2015). Enhanced superconducting properties in infiltration processed Y–Ba–Cu–O single-grain superconductor with nano-sized pinning centers. *Journal of Alloys and Compounds*, *649*, 559–563. http://doi.org/10.1016/j.jallcom.2015.07.117

Pair, C. (n.d.). 6.3. Cooper Pair and the BCS theory, 78–83.

Philippe, M. P., Fagnard, J. F., Kirsch, S., Xu, Z., Dennis, A. R., Shi, Y. H., … Vanderbemden, P. (2014). Magnetic characterisation of large grain, bulk Y-Ba-Cu-O superconductor-soft ferromagnetic alloy hybrid structures. *Physica C: Superconductivity and Its Applications*, *502*, 20–30. http://doi.org/10.1016/j.physc.2014.04.025

Rana, K., Thakur, P., Thakur, A., Tomar, M., Gupta, V., Mattei, J. L., & Queffelec, P. (2016). Influence of samarium doping on magnetic and structural properties of M type Ba-Co hexaferrite. *Ceramics International*, 1–6.

Ruppert, V. P. O. C. Y. S. E. (2013). Electrical and Superconducting Properties in Lap Joints for YBCO Tapes, 2089–2092. http://doi.org/10.1007/s10948-012-1905-6

Sun, M., Liu, Z., Bai, C., Guo, Y., Lu, Y., Fan, F., & Cai, C. (2015). Co-doping effects of Gd and Ag on YBCO films derived by metalorganic deposition. *Physica C: Superconductivity and Its Applications*, *519*, 47–52. http://doi.org/10.1016/j.physc.2015.08.011

Vidya, S., Rejith, P. P., John, A., Solomon, S., Deepa, A. S., & Thomas, J. K. (2011). Electrical, optical and vibrational characteristics of nano structured yttrium barium stannous oxide synthesized through a modified combustion method. *Materials Research Bulletin*, *46*(10), 1723–1728. http://doi.org/10.1016/j.materresbull.2011.05.034

Volochova, D., Jurek, K., Radusovska, M., Piovarci, S., Antal, V., Kovac, J., … Diko, P. (2014). Contamination of YBCO bulk superconductors by samarium and ytterbium. *Physica C: Superconductivity and Its Applications*, *496*(C), 14–17. http://doi.org/10.1016/j.physc.2013.04.084

Volochová, D., Piovarči, S., Radušovská, M., Antal, V., Kováč, J., Jurek, K., … Diko, P. (2013). YBCO bulk superconductors doped with gadolinium and samarium. *Physica C: Superconductivity and Its Applications*, *494*, 36–40. http://doi.org/10.1016/j.physc.2013.04.009

Ted Burke, 2012, Tips on writing final year project, from https://batchloaf.wordpress.com/2012/05/02/some-tips-on-writing-a-final-year-project-report/

Ivan A.Parinov, 2016, Microstructure and properties of High- Temperature Superconductors from https://books.google.com.my/books

Taylor C, 2007, Voltage, current and resistance, and Ohm's Law, from https://learn.sparkfun.com/tutorials/voltage-current-resistance-and-ohms-law

William D. Callister, (2009), *The structure of crystalline solids*, Materials Science and Engineering (44) Iowa: Wiley and co

R. Nave, 2010, Cooper pairs and BCS theory from http://hyperphysics.phy-astr.gsu.edu/hbase/Solids/coop.html

Paul J. Schields, 2004, Bragg's Law and diffraction from http://skuld.bmsc.washington.edu/~merritt/bc530/bragg/

James M. Guthrie, 2012, Overview of X-ray Fluorescence from http://archaeometry.missouri.edu/xrf_overview.html

Karl Wirth, 2010, X-Ray Fluorescence (XRF) from http://serc.carleton.edu/research_education/geochemsheets/techniques/XRF.html

Appendices A

Sample preparation calculation $YBa_2Cu_3O_7$

Stoichiometry involved :

$$\frac{1}{2} Y_2O_3 + 2Ba_2CO_3 + 3CuO \longrightarrow YBa_2Cu_3O_7$$

Relative atomic masses (RAM) :

Y = 88.906

Ba = 137.327

C = 12.011

O = 15.999

Cu = 63.546

Sample RAM :

Y_2O_3 = 2(88.906) + 3(15.999) =225.809

$BaCO_3$ = (137.327) + (12.011) + 3(15.999) = 197.335

CuO = (63.546) + (15.999) = 79.545

Sample total Relative molecular masses (Rmm) :

$$\frac{1}{2} Y_2O_3 + 2Ba_2CO_3 + 3CuO = \frac{1}{2}(225.809) + 2(197.335) + 3(79.545) = 746.210$$

Rmm needed for 3 g sample :

Y_2O_3 : $\frac{\frac{1}{2}(225.809)}{746.210} \times 3$ g = 0.4539 g

$BaCO_3$: $\frac{2\ (197.335)}{746.210} \times 3$ g = 1.5867 g

CuO : $\frac{3\ (79.545)}{746.210} \times 3$ g = 0.9594 g

total = 3.0000 g

Mass of Nickel Oxide needed to add to the sample

x = 0.80, 0.10 and 0.12 % wt :

x = 0.80 % wt

$\frac{0.08}{100} \times 1.5$ g = 0.0012g

0.0012 × 3 = 0.0036 g

x = 0.10 % wt

$\frac{0.10}{100} \times 1.5$ g = 0.0015 g

0.0015 × 3 = 0.0045 g

x = 0.12 % wt

$\frac{0.12}{100} \times 1.5$ g = 0.0018 g

0.0018 × 3 = 0.0054 g

Appendices B

Calculation of resistivity at room temperature for YBCO Sample with NiO composition = 0 0.08, 0.10 and 0.12 % wt

The resistivity can be calculated by using equation below

$$\boldsymbol{\rho = \frac{RA}{l}} \qquad \text{Equation b.1}$$

A = area of sample $A = \pi r^2$

R = resistance

L = Length or thickness of sample

By using Ohm's Law, I = $\frac{V}{R}$, the current are calculated. (thickness= 1.028 mm)

Table B.1 Current, resistance,voltage and resistivity of the sample

Sample	Voltage, V (v)	Resistance, R (Ω)	Current, I (A)	Resistivity, ρ (**×10^{-3} Ωm**)
Pure YBCO	1.3	0.1	13	1.9103
0.08 % wt NiO	1.3	0.3	4.3	5.7307
0.10 % wt NiO	1.3	0.5	2.6	9.5513
0.12% wt NiO	1.3	0.9	1.44	17.1924

The resistivity, $\boldsymbol{\rho}$ at room temperature can be calculated by using formula :

$$\rho = \left(\frac{RA}{L}\right)$$

For sample x=0.08 % wt

$$= \left(\frac{0.3(19.6375}{1.028mm}\right)$$

$= 5.7307 \times 10^{-3}\ \Omega m$

For sample x = 0.10 % wt

$$= \left(\frac{0.5(19.6375}{1.028mm}\right)$$

$= 9.5513 \times 10^{-3}\ \Omega m$

For sample x=0.12 % bt

$$=\left(\frac{0.9(19.6375)}{1.028mm}\right)$$

$=17.1924 \times 10^{-3}\ \Omega m$

APPENDICES C

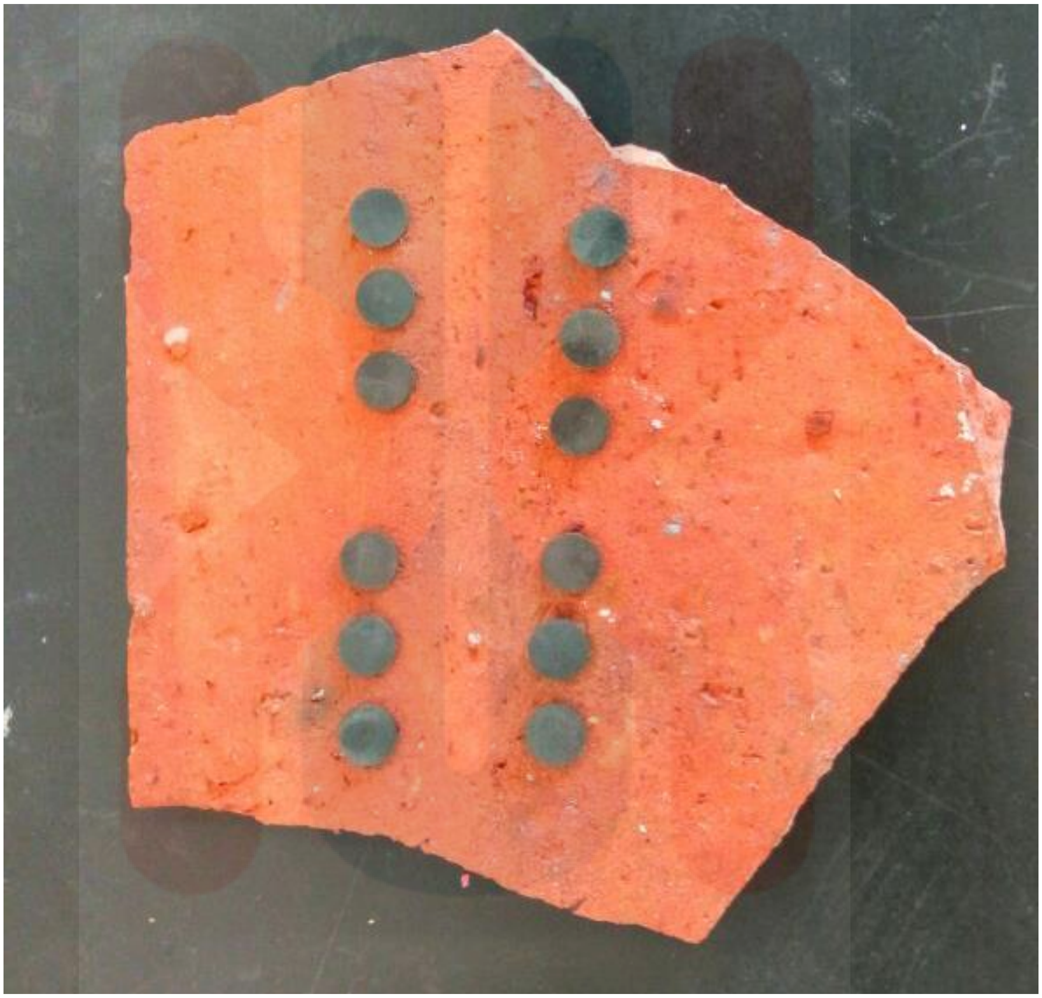

Figure C.1 Samples in pellet form for resistivity test

Pattern: COD 2100876 Radiation: 1.54060 Quality: Quality Unknown

Formula	Ba Cu O5 Y2
Name	
Name (mineral)	
Name (common)	
Lattice:	Orthorhombic
S.G.:	P n m a (62)
Mol. weight =	
Volume [CD] =	492.17
Dx =	
Dm =	
I/Icor =	4.870
a =	12.18800
b =	5.66200
c =	7.13200
a/b =	2.15260
c/b =	1.25963
alpha =	
beta =	
gamma =	
Z =	4

d	2θ	I	h	k	l	d	2θ	I	h	k	l
6.15560	14.377	22	-1	0	-1	2.14400	42.112	4	-4	-1	-2
6.09400	14.524	5	-2	0	0	2.13610	42.275	28	-5	-1	-1
4.16720	21.305	5	-1	-1	-1	2.08360	43.394	31	-2	-2	-2
4.14800	21.404	2	-2	-1	0	2.07400	43.605	75	-4	-2	0
3.58560	24.811	3	-2	-1	-1	2.06260	43.858	41	-2	-1	-3
3.56600	24.950	12	0	0	-2	2.03130	44.570	67	-6	0	0
3.53010	25.208	2	-3	0	-1	1.99150	45.510	321	-4	-2	-1
3.42250	26.014	63	-1	0	-2	1.95360	46.444	6	-6	0	-1
3.07780	28.988	42	-2	0	-2	1.94630	46.629	53	-3	-2	-2
3.04700	29.287	58	-4	0	0	1.92910	47.070	8	-3	-1	-3
2.99560	29.801	999	-3	-1	-1	1.91200	47.516	2	-6	-1	0
2.92900	30.495	668	-1	-1	-2	1.89620	47.937	14	-5	-1	-2
2.83100	31.578	474	0	-2	0	1.87430	48.533	85	-4	0	-3
2.80200	31.913	198	-4	0	-1	1.84680	49.303	27	-6	-1	-1
2.70410	33.101	191	-2	-1	-2	1.80060	50.656	1	-1	-2	-3
2.68310	33.368	7	-4	-1	0	1.79280	50.892	3	-4	-2	-2

Figure C.2 XRD Data of YBCO with 0.08 wt % NiO

Pattern: COD 1006053 Radiation: 1.54060 Quality: Quality Unknown

Formula	Ba Cu O5 Y2
Name	
Name (mineral)	
Name (common)	
Lattice:	Orthorhombic
S.G.:	P n m a (62)
Mol. weight =	
Volume [CD] =	491.59
Dx =	
Dm =	
I/Icor =	4.880
a =	12.17920
b =	5.65900
c =	7.13250
a/b =	2.15218
c/b =	1.26038
alpha =	
beta =	
gamma =	
Z =	4

d	2θ	I	h	k	l	d	2θ	I	h	k	l
6.15470	14.380	22	-1	0	-1	2.14310	42.131	4	-4	-1	-2
6.08960	14.534	4	-2	0	0	2.13480	42.302	29	-5	-1	-1
4.16580	21.312	5	-1	-1	-1	2.08290	43.409	31	-2	-2	-2
4.14540	21.418	2	-2	-1	0	2.07270	43.634	76	-4	-2	0
3.58400	24.823	3	-2	-1	-1	2.06240	43.863	41	-2	-1	-3
3.56630	24.948	13	0	0	-2	2.02990	44.603	67	-6	0	0
3.52820	25.222	2	-3	0	-1	1.99040	45.537	319	-4	-2	-1
3.42250	26.014	63	-1	0	-2	1.95230	46.477	6	-6	0	-1
3.07740	28.992	41	-2	0	-2	1.94550	46.649	53	-3	-2	-2
3.04480	29.309	58	-4	0	0	1.92870	47.080	7	-3	-1	-3
2.99400	29.818	999	-3	-1	-1	1.91070	47.551	2	-6	-1	0
2.92860	30.499	664	-1	-1	-2	1.89530	47.961	14	-5	-1	-2
2.82950	31.595	474	0	-2	0	1.87390	48.544	86	-4	0	-3
2.80030	31.933	195	-4	0	-1	1.84560	49.337	27	-6	-1	-1
2.70350	33.109	196	-2	-1	-2	1.80020	50.668	1	-1	-2	-3
2.68130	33.391	7	-4	-1	0	1.79200	50.917	2	-4	-2	-2

Figure C.3 XRD data of YBCO with 0.10 wt % NiO

Pattern: COD 9011761   Radiation: 1.54060   Quality: Quality Unknown

Formula	Dy0.02 Nb0.46 O3 Ti0.54 Y0.48
Name	
Name (mineral)	Polycrase-(Y)
Name (common)	

Lattice:	Orthorhombic	Mol. weight =	
S.G.:	P b c n (60)	Volume [CD] =	425.62
		Dx =	
		Dm =	
		I/Icor =	6.960

a =	14.66730	alpha =	
b =	5.59250	beta =	
c =	5.18880	gamma =	
a/b =	2.62267	Z =	
c/b =	0.92781		

d	2θ	I	h	k	l	d	2θ	I	h	k	l
7.33370	12.058	12	-2	0	0	1.84930	49.232	2	-1	-3	0
5.22550	16.954	21	-1	-1	0	1.84100	49.469	22	-2	-2	-2
3.68200	24.152	25	-1	-1	-1	1.84040	49.486	106	-6	-2	0
3.68080	24.160	119	-3	-1	0	1.83570	49.621	3	-5	-1	-2
3.66680	24.253	4	-4	0	0	1.83530	49.633	10	-7	-1	-1
3.37660	26.374	60	-2	-1	-1	1.77920	51.310	109	-6	0	-2
3.00220	29.734	999	-3	-1	-1	1.74200	52.488	27	-1	-3	-1
2.79630	31.980	105	0	-2	0	1.74180	52.494	52	-3	-3	0
2.63990	33.930	27	-4	-1	-1	1.73450	52.732	82	-6	-2	-1
2.61280	34.293	6	-2	-2	0	1.68830	54.292	8	-4	-2	-2
2.59780	34.497	4	-5	-1	0	1.65160	55.601	3	-8	-1	-1
2.59440	34.544	87	0	0	-2	1.65130	55.612	52	-3	-3	-1
2.55470	35.098	26	-1	0	-2	1.64200	55.955	23	-1	-1	-3
2.46160	36.471	41	0	-2	-1	1.63010	56.400	17	-7	0	-2
2.44590	36.714	2	-2	0	-2	1.61200	57.091	33	-2	-1	-3
2.44460	36.734	61	-6	0	0	1.59580	57.724	9	-5	-2	-2

Figure C.4 XRD data of YBCO with 0.12 wt % NiO