ABANT IZZET BAYSAL UNIVERSITY
THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

INVESTIGATION ON THE STRUCTURAL, MAGNETIC AND ELECTRICAL PROPERTIES OF THE AU ADDED YBCO SUPERCONDUCTORS

MASTER OF SCIENCE

GÜLSEVİM AYDIN

BOLU, FEBRUARY 2016

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APPROVAL OF THE THESIS

Investigation on the structural, magnetic and electrical properties of the Au added YBCO superconductors submitted by **Gülsevim Aydın** in partial fulfillment of the requirements for the degree of Master of Sciencein **Department of Physics, Abant Izzet Baysal University** by,

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February 10, 2016

Prof. Dr. Duran KARAKAS ....D. Karakaş....

Director, **Graduate School of Natural and Applied Sciences**

To my daddy…

DECLARATION

I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

Gülsevim AYDIN

ABSTRACT

INVESTIGATION ON THE STRUCTURAL, MAGNETIC AND ELECTRICAL PROPERTIES OF THE AU ADDED YBCO SUPERCONDUCTORS
MSC THESIS
GÜLSEVİM AYDIN
ABANT IZZET BAYSAL UNIVERSITY GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES
DEPARTMENT OF PHYSICS
(SUPERVISOR:PROF. DR. CABİR TERZİOĞLU)

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In this study, a systematic investigation of Au-added $YBa_2Cu_3O_{7-x}$ (YBCO) samples has been undertaken mainly to understand their structural, magnetic as well as electrical behavior.In order to get high quality of superconducting bulk samples, optimum preparation conditions have been provided. The bulk samples were produced by conventional solid state reaction method at 930^0C under the low oxygen atmosphere. The bulk samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), dc electrical resistivity (ρ-T), critical current density and ac magnetic susceptibility measurements. The samples were carried out by means of powder X-ray diffraction for phase analyses, and lattice parameters, SEM for microstructure examinations. From XRD and SEM results, an increase in the intensitiy of main peaks and average grain size but a increase in the lattice parameter c with addition of Au in comparision with the unadded sample.In addition to this, onset-offset critical transition temperature (T_c^{on}, T_c^{off}) was determined from dc electrical resistivity ($\rho - T$) as a function of temperature measurements.Moreover, the transition temperature (T_c) and the critical current density (J_c) are enhanced upon Au added when compared with unadded YBCO superconductor samples. The increament of Au contente decreased the transition temperature and the critical current densities values. From the AC susceptibility measurements, inter-, intra-garin peak temperature and superconducting transition temperature were determined. The possible reasons for the observed improvements in structural, magnetical and electrical properties due to the Au addition in the YBCO samples were discussed by comparison with other studies about addition of transition metals in high temperature superconductors (HTSCs).

KEYWORDS: Superconductivity, X-ray diffraction (XRD), Scanning electron microscopy (SEM), DC electrical resistivity ($\rho - T$), AC magnetic susceptibility

ÖZET

AU EKLENMİŞ YBCO ÜSTÜNİLETKENLERİN YAPISAL, MANYETİK VE ELEKTRİKSEL ÖZELLİKLERİN İNCELENMESİ

YÜKSEK LİSANS TEZİ
GÜLSEVİM AYDIN
ABANT İZZET BAYSAL ÜNİVERSİTESİ FEN BİLİMLERİ ENSTİTÜSÜ
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Bu çalışmada, Au eklenmiş $YBa_2Cu_3O_{7-x}$(YBCO) süperiletken malzemelerin yapısal, manyetik hem de elektriksel davranışlarını açıkça anlayabilmek için düzenli bir inceleme girişiminde bulunuldu. Yüksek kalitede süperiletken malzemeler üretebilmek için gerekli uygun ortamlar oluşturuldu. Malzemeler uygun katıhal tempkime yöntemi kullanılarak 930^{0}C da düşük atmosfer basıncı altında üretildi. Malzemelerin x-ışını kırınım (XRD), taramalı elektron mikroskopu (SEM), dc elektriksel direnç $(\rho - T)$ ve ac manyetik alınganlıkları tanımlandı. Faz analizi ve örgü parametrelerinin belirlenmesi için x-ışını kırınımı incelemesi, mikro yapı incelemeleri için ise SEM ölçümleri yapıldı.Buna ek olarak dc özdirenç ölçümlerinden başlangıç-bitiş kritik geçiş sıcaklıkları ölçüldü. XRD ve SEM ölçümlerinden, altın katkılanmış numuneler saf numuneyle karşılaştırıldığında, ana piklerin şiddetinin ve ortalama taneciklerin büyüklüğünde bir artış olurken, c örgü parametresinde küçülme olduğu gözlendi.Ayrıca, geçiş sıcaklığı (T_c) ve kritik akım yoğunluğunun (J_c), altın katkılanmasıyla, altın katkılanmamış ile karşılaştırıldığında, geliştiği (arttığı) görüldü. AC alınganlık ölçümlerinden de, tane içi, taneler arası ve süperiletkenlik geçiş sıcaklıkları belirlendi. Bu çalışmada, süperiletken YBCO numunelerine altın katkılanmasıyla, numunelerde meydana gelen yapısal, manyetik ve elektriksel özelliklerin sonuçları incelendi. Bu sonuçlar yüksek sıcaklık süperiletkenlere (HTSCs) madde katkılanmasıyla ile ilgili daha önce yapılan çalışmalarla da karşılaştırılarak, YBCO malzemesine altın (Au) katkılanmasının madde üzerindeki etkisinin daha iyi anlaşılması sağlandı.

ANAHTAR KELİMELER: Üstüniletkenlik,Taramalı electron mikroskop (SEM), X-ışını kırınımı (XRD), DC öz direnç (ρ-T), ac manyetik alınganlık

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LIST OF ABBREVIATTIONS AND SYMBOLS

a, b, c: Lattice parameters

AC : Alternatin current

Å : Angstrom

Au : Gold

a.u : Arbitrary unit

C : Celciuss

DC : Direct current

h, k, l : Miller indices

H_c : Critical magnetic field

HTSCs : High temperature superconductors

I_c: Critical current

J_c :Critical current density

K : Kelvin

Oe : Oersted

T_c : Critical temperature

SEM : Scanning electron microscope

XRD : X-ray diffraction

YBCO :$YBa_2Cu_3O_{7-\gamma}$

ZFC : Zero Field Cooled

ξ : Coherence length

ρ: Resistivity

Ω: Ohm

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1. INTRODUCTION

1.1 Overview

Advance in science in common, and in physics in definite, is qualified by many large inventions both in theory and experiment.Time passes and many discoveries occur in physics. Especially with the announcement of superconductivity which is started with the discovered in Hg (T_c=4,2 K) that dc electrical resistance suddenly goes to zero.That point describes by Heike Kamerling Onnes and co-workers in 1911 (OnnesHK, 1911) during the study on pure metals at low temperature.Mercury was the metal which presented the most optimal conditions for the examination of intrinsic properties of metals at low temperature caused by the purity at which it could be acquired.

The necessary technical basis and opportunity for the discovery had been solidly established by Onnes group by the liquefaction of the inert gas Helium in 1908 (Fossheim &SudbØ, 2004). So, liquid helium, the cryogen itself, was conributed to crucial discovery.Gold was experienced to have unmeasurable low resistance in the liquid helium range, in addition to,several elements of the periodic table had been added to the list as tin and lead. Onnes dismissed the prevailing idea that electrons would ' freeze to the atom' and surmised instead that ' the free electrons' would remain free ' while the vibrators' (atom) would become 'practically immovable' (Fossheim &SudbØ, 2004). So, the research in the Leiden laboratory by Onnes groups were inspired to many researcher to explain low–temperature heat capacity and study on thermal and electrical conductivity of solids at low temperatures. Afterward, in same year 1911, the atomic nucleus was discovered.

The Leiden groups had a big problem about magnetic field properties. Because, the upper limit for current which would flow at zero resistance in some superconductors in the self-field of the coil, which we call it, the critical current (I_c) . After 20 years later, the big discovery performed by the scientist W. Meissner an R. Ochsenfeld in 1933, which is the exclusion of magnetic field in the superconducting material. As a result of this, perfect diamagnetism were discovered, which is called " Meissner " (Meissner & Ochsenfeld, 1933). After many study years, Abrikosov expanded 'Meissner effect', and reported in 1957. Abrikosov come up with that there were two class of superconductors, which labelled to be, Type I, where the magnetic flux is completely expelled from the interior of the superconducting sample for values less than a critical magnetic field value H_c; and Type II, where the magnetic flux is completely expelled for small fields (belowH_{c_1}) and partially in higher external fields (up to H_{c_2}) (Abrikosov, 1957). In the same year quantum theory which known as BCS theory, was come out by John Bardeen, Leon Cooper, and Robert Schrieffer. In this theory, electrons form " cooper pair " while moving in the lattice (Cooper, 1956).

After the announcement of superconductivity in the La–Ba–Cu–O(Bednorz &Müller, 1986) system by Bednorz and Müller above 30 K at the IBM laboratory in 1986, a new phenomenon take a big place in the scientists world which is the discovery of Y–Ba–Cu–O (yitrium barium copper oxide) superconductor by Wu at al, which the critical temperature was over 90 K (Wu et al., 1987).This was the milestone of superconductor technology because the critical temperature more over the liquid Nitrogen, easier to coolant and cheaper than the liquid helium. The subsequent discoveries of copper oxides were focused on. In 1988, bismuth lead strontium calcium copper oxide and thallium barium calcium copper oxide ($Bi_2Sr_2Ca_2Cu_3O_{10}$,$Tl_2Ba_2Ca_2Cu_3O_{10}$) (Maeda et al., 1988; Sheng et al., 1988) and; in 1933, mercury barium calcium copper oxide ($Hg_2Ba_2Ca_2Cu_3O_8$) (Schilling et al., 1993) were discovered with the T_c = 110 K, 120 K and 134 K, in turn.Especially, with the discovery of Bi(Pb)-Sr-Ca-Cu-O system,a countless amount of study being done on it (Michel, 1987; Maeda & Taraka, 1988; Tokana, 1988). In 2001 J. Nagamatsu et al (Nagamatsu et al., 2001)found intermetallic superconductor, which the magnesium diboride MgB_2 with T_c = 39 K. In the last years some researchers study on Fe – based superconductors which is found by C. Wang et al in 2008 with T_c = 56 K (Wang et al., 2008).

This thesis consist of four chapters, which are:

Chapter 1 includes the historical overview and basic properties of the superconductivity, the content of the each individual chapter is briefly summarized here, crystal structure and aplication of YBCO systems has been given briefly. A brief history of the manganites has been given.

The chapter 3 includes the experimental techniques which the sample preparation of Au added YBCO and experimental set-up are given. This chapter also consist of a description about the procedures used to perform electric and magnetic transport measurements and a detailed analysis of sample characterization which includes scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD), is given.

In Chapter 4, the study of some researcher about effect of Au on superconducting materials are given briefly. And than the results of all measurements are presented and discussed in detail.

Chapter 5 is a general summary of all chapters and we have shown some important results that obtained during this thesis.

1.2 High Tc Superconductor

Superconductivity has been searched to be presented by many elements: binary and ternary compounds, alloys, organic superconductor and lastly invention of high-T_c superconductors.

A new discovery in higher superconducting transition temperature came in 1986 by two scientists, J. George Bednorz and K. Alex Müller (Bednorz & Müller, 1986), that their remarkable discovery was about high temperature superconductivitiy, T_c=35 K, in defect perovskite as oxide material $La_{2-x}Ba_xCuO_4$. This study were confirmed by other groups. The first attack came from by Houston group, Chu and his friends (Wn & Chu, 1987), that the T_c going above 40 K under 13 kbar pressure (Fossheim & SudbØ, 2004). They replaced the some of ions with smaller ones having the same chemical properties like replacing Ba with the Sr. By the way, Wu from the Huntsville group think about that the similar study have to be experienced at the La-site. Then the replacing of both La and Ba, with Y and Sr, respectively, were achieved in cooperation with the Huntsville group led by Wu and the Houston group. They discovered ' 123 oxides ' with the general formula $LnBa_2Cu_3O_{7-\gamma}$ (Ln=Y, Nd, Sm, Eu, Gd, Dy, Ho, Er, Tm or Yb) (Saxena, 2009), and T_c=90 K region. This new sensational compound $YBa_2Cu_3O_{7-\gamma}$ moved the T_c well above the liquid N_2 (T_c = 77 K) which in the range 91 - 93 K.

After the synthesized of high T_c YBCO superconductor by Wu at al, two more important series of high-T_c were found: one Bi-based, Bi-Sr-Ca-Cu-O, and one Ti-based, TI-Ba-Ca-Cu-O (in 1988) systems that exhibited the superconductivity in the range 60 and 125 K (Maeda et al., 1988; Sheng & Hermann, 1988).

Later, a Hg-based, Hg-Ba-Ca-Cu-O, series was also synthesized , 135 K. The speciality of these series that the higher T_c's than the 93 K of YBCO. It is significant that whole the high-T_c cuprates (superconductors which containing cupper-oxide) have 'defect perovskite layer' (which sheets of copper and oxygen atoms alternate with sheets of rare earth atom (Saxena, 2009).) and whole except 123 compounds include rock salt type oxide layers. These ceramic superconductor are very different from normal metals, being doped oxides. They are so anisotropic compounds that they could be shown in some cases to possess metalik-like conductivity in direction parallel to the CuO_2 planes- although not free-electron like- while behaving like semiconductors along the c-axis, normal to CuO_2 planes. The superconducting current flows by electron-vacancies or holes.

The most important characteristic properties of high-T_c superconductors are diamagnetism and zero resistivity. At the same time their coherence ξ length is very short because the fact that it allows the achievement of high critical fields, Hc_2. On the other hand, in these materials, due to the thermal fluctuations in the mixed state which rise the creep of the vortex lattice lead to dissipation. In addition to this, due to the some mechanical problem like brittleness of high-T_c ceramic superconductor in bulk form, it restricts the study area of engineering applications.

Doping or substitution of atoms on high-T_c materials changes the superconductivity and the mechanical properties of superconductor that prepare the convenient side in technological applications. The aim of doping or substitution on high-T_c materials to observe the effect of changing some parameters on superconductivity.

In this thesis, the effect of adding gold (Au) to the high-T_c superconductor YBCO (Y123) and observing the changes in the structural, magnetical and electrical properties

1.2.1 Crystal Structure

It has been shown by many researchers that the high-T_c (HTSCs) superconductors are structurally defected which called the perovskite crystal structures. This crystallographic family which have a particular atomic organisation. The general model of compound with perovskite structure is ABO_3. These structure composed of cubes which consist of different chemical elments like; A, B and X in the ratio of 1:1:3 .

In the perovskite structure A and B atoms represent cation and X atoms are anions. The A ion (positive) in the center of each cube, the B ion (positive) at the every corner and the X ion (negative) occupy the midpoints of cube's twelve edge.

The perovskite structure were called as: cubic (see figure 1.1), tetragonal and orthorhombic (see figure 1.2)

First of all at the cubic perovskite a, b and c lattice are equal (a=b=c). Secondly, the structure which known as $KNiF_4$ and has a tetragonal distortion that a, b lattice parameters show equivalent but c is not ($a = b \neq c$). The third one is orthorhombic structure which called as multi-layer perovskite.

1.2.2 YBCO System

This range of the superconductivity is the most studied high-T_c superconductor due to the preparation of suitable and economical side in the technological and engineering applications above liquid nitrogen temperature (77 K).Many comprehensive study were done (Gorur, 2001; Alagoz, 2003; Altınkok, 2011; Yurtcan, 2011; Bektaş,2012; Sarıtekin, 2014; Kutuk, 2014) on this area. For example, current limiters, magnetic bearings, energy storage systems, etc.

YBCO systems are the connection of simple cubic perovskites. In other words, YBCO is a heap of three perovskite unit cells along the c-axis with the general formula as ABO_3 (see figure 1.1).

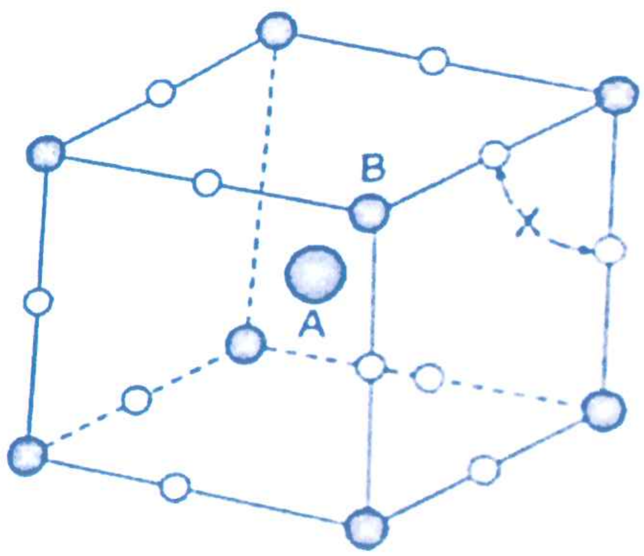

Figure 1.1. The perovskite structure ABX_3

That is have the lattice parameters as a = 3,82 Å, b = 3,88 Å and a = 11,67 Å. YBCO systems consist of two types of conductivities which are non-superconducting and superconducting material, that is to say YBCO exists in either a tetragonal or an orthorhombic crystal structure.

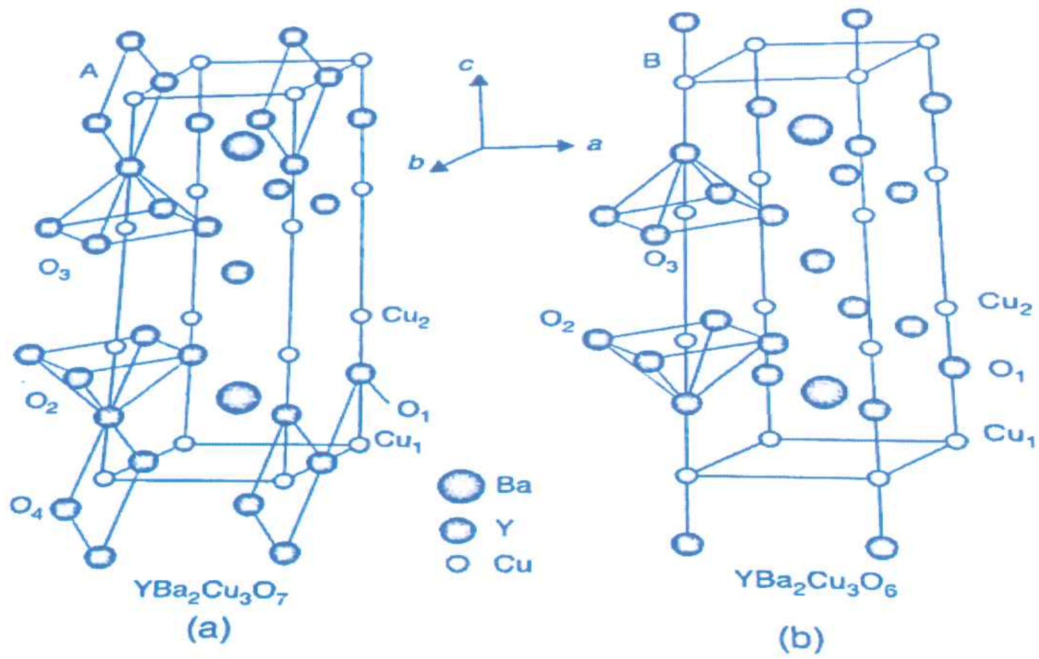

Figure 1.2. The structure of YBCO (a) orthorhombic structure (b) Tetragonal structure (Apetrii, 2009)

First, preparing the non-superconducting as $YBa_2Cu_3O_6$ that obtained under the suitable conditions in an oxygen atmosphere from the total powder of Y_2O_3, BaO and CuO_x, heated to over 900℃. The composed structure is tetragonal (non-superconducting) whichhave a lot of oxygen vacancies. While reducing the temperature to 400℃ more oxygen is absorbed by the structure and many Cu-O chains with variable oxygen content is presented.

These chains break the tetragonal structure and a new phase with the different lattice parameters along the a- and b- directions occur. After the all existing oxygen sites are completed in this structure a new compound which shown as $YBa_2Cu_3O_7$ system occur. Now,it can be realized that the $YBa_2Cu_3O_7$ structure is a modification of $YBa_2Cu_3O_6$ structure. Due to the this modification a new phase substitute for tetragonal structure, which isorthorhombic crystal structure. Only in this orthorhombic phase the superconductivity of YBCO occur (Figure 1.2).

Doping of $SrTiO_3$ had been demonstrated to lead to superconductivity due a reaction process, whereby oxygen was removed (Saxena, 2009). The oxygen content in YBCO determines a more complicated structure (Figure 1.3). The structure of $YBa_2Cu_3O_{6+}x$ is in a tetragonal phase (and insulator) for oxygen content at x=0 to increasing the oxygen content x=6.4, but becomes a superconductor with the values of oxygen content x grater than 6,4. The superconductivity reaches its maximum value at x=6.9, with the increasing on T_c.In Figure 3.1 (b), T_c reached its maximum value, T_c=93K, with the oxygen content came to x=6,94. As a result of this,with the optimal doping of oxygen content to YBCO, the $YBa_2Cu_3O_{7-\gamma}$ orthorhombic phase with γ=0.07 is ocquired.

As all high-T_c compounds, YBCO structure consist of layers, for example one plane at a time from bottom to up, they are; CuO, BaO, CuO_2, Y, CuO_2, BaO and CuO respectively. Superconductivity occurs in the CuO_2 layers in the orthorhombic phase. But the speciality of YBCO is that CuO chains, they transfer holes to the planes. When available oxygen sites are filled superconductivity can be seen. Forth, in this structure the oxygen sites aren't at equal positions in the lattice as in tetragonal structure.

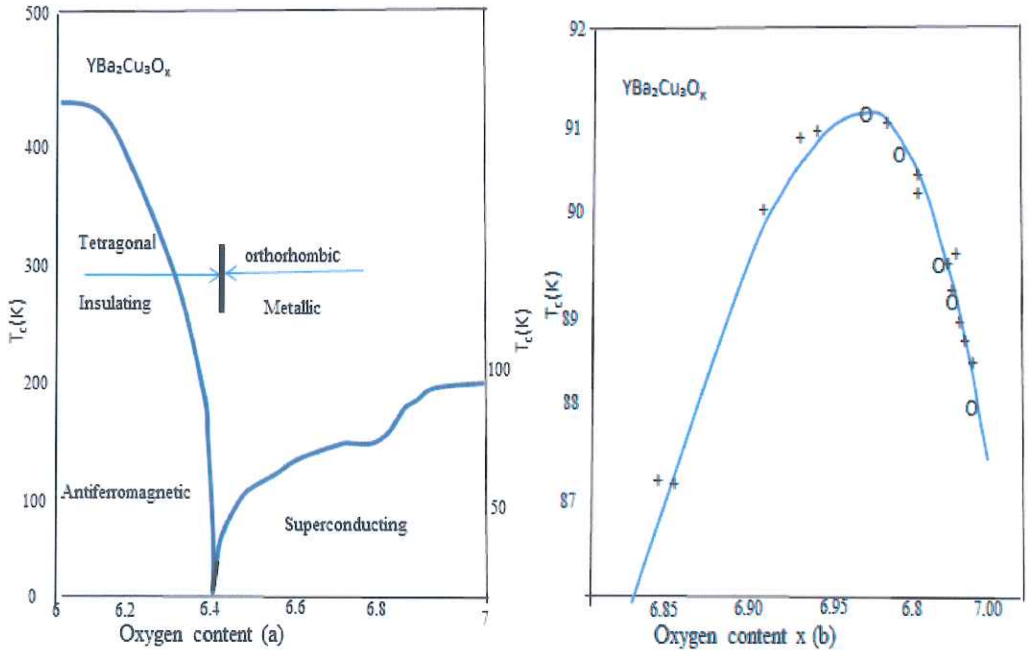

Figure 1.3.(a) Phase diagram of the $YBa_2Cu_3O_x$ system as a function of the oxygen content and (b)Variation of T_c with oxygen content (Apetriie, 2009)

1.2.3 Application of Superconductor

Superconductor have a major potential of technological and engineering applications in the world. The superconductors applications which are produced as thin and thick films, wires and stripes can be listed as below.

The medical applications which are Magnetic Resonance Imaging (MRI), Ultra-Low-Field Magnetic Resonance Imaging (ULF-MRI), Magnetic Source Imaging (MSI), Magneto-Encepthalography (MEG) and Magnetocardiography (MCG), etc.

The other applications utilizing superconductors area are; DC power transport wire and AC power transport wire, Smart Grid system, Superconducting Magnetic Energy Storage (SMES) devices, transformer, motor, generator and bearing, Fugitive Current Limiter (FCL), High-Energy physics accelerators, plasma Fusion reactors, etc. most of above devices used for spacecraft and navy ships. In addition to these, for submarine systems, small frequency receiver, HTS induction heater system etc. are produced.

The other commercial application of superconductivity is on electronic systems that are; Superconducting Quantum Interference Device (SQUID), Superconducting Tunneling Junctions (STJs), Dynamic Josephson Junctions (DJJs), Individual Josephson Junction (IJJ), Transition Edge Sensing (TES), Superconducting Electronic Device Processor (SEDP) etc.

By the way, study on Quantum Computer (QC), Superconducting Digital Redirector (SDR), Superconducting X-ray Detector (SXD), Superconducting Light Detector (SLD) etc. have been continued. Also, study on Magnetic Levitation (MagLev) train, ship repulsion motor and spacecraft motor have been continued too.

2. AIM AND SCOPE OF THE STUDY

The aim of this thesis is to investigate the effect of Au adition on YBCO superconductors. For this purpose we investigate the structural, magnetic and electrical properties of samples.

The Au added $YBa_2Cu_3O_{7-x}$ bulk samples were prepared by the standard solid-state reaction methods with the stoichiometric quantities and high purity of Y_2O_3, $BaCO_3$ and CuO. The weight ratio of Au in YBCO samples were calculated as 1%, 5%, 15% and 20% of YBCO. After the addition of Au powder every samples were regrinding and shaped in the appropriate die. Then,the homogeneous mixture calcining, sintering and annealing were carried out at the convenient temperature.

We performed the electrical-magnetic transport measurements and a detailed analysis of sample characterization which includes scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD).

In the light of these measurements, we aimed to find enhancement of the superconductivity properties.

3. EXPERIMENTAL TECHNIQUES

3.1 Sample Preparation

The effect of Au addition in YBCO(123) system is studied in this work. The superconductor can be prepared by many methods, which are the solid-state reaction methods, chemical routes, powder melting process etc.

The Au added $YBa_2Cu_3O_{7-x}$ bulk samples were prepared by the standard solid-state reaction methods with the stoichiometric quantities and high purity of Y_2O_3 (99,99%) , $BaCO_3$ (99.9+%) and CuO (99+%) (see Table 3.1). The powders were weighted with a precise microbalance.

Table 3.1. The molecular weights, Mass for *2,5* grammas and purity of the compounds.

Compounds	Y_2O_3	$BaCO_3$	CuO
Purity (%)	99,99	99,9	99+
Molecular weights(*g/mol*)	225,80	197,35	79,55
Mass(*g*) (*2,5grammas*)	0,3782	1,3223	0,7995

So, the convenient amounts of chemicals were calculated by using the following balancing equations,

$$(\frac{1}{2})\ Y_2O_3 + (2)\ BaCO_3 + (3)CuO \rightarrow YBa_2Cu_3O_{7-x} + \text{Gases}$$

The starting powders Y_2O_3, $BaCO_3$ and CuO were mixed by a pestle to get a homogeneous mixture. The homogeneous mixture calcining were carried out at 930 ℃ for 24 h with the heating and cooling rates of 5° and 1℃ min^{-1} to get the reacted material, respectively (see figure 3.1). The calcined powder were ground to get the desired particle size. Then the remixed powder shaped in to the desired forms by using a suitable die and pressed under the 230 MPa. Sintering were done in an oxygen atmosphere at temperature 930° C by maintained heating and cooling rate of 3° and 1℃ per minute, respectively. The sample were cooled in an atmosphere of flowing oxygen between 700 and 400 ℃ (Figure 3.2).

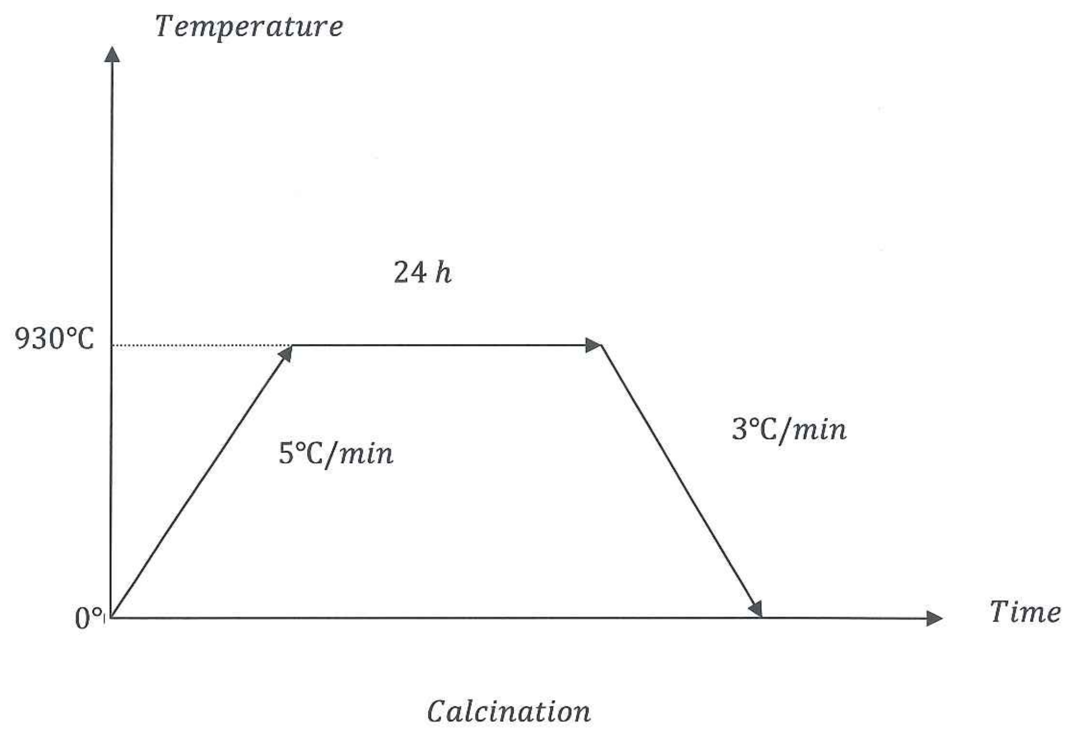

Figure 3.1. Calcinations process of the powder

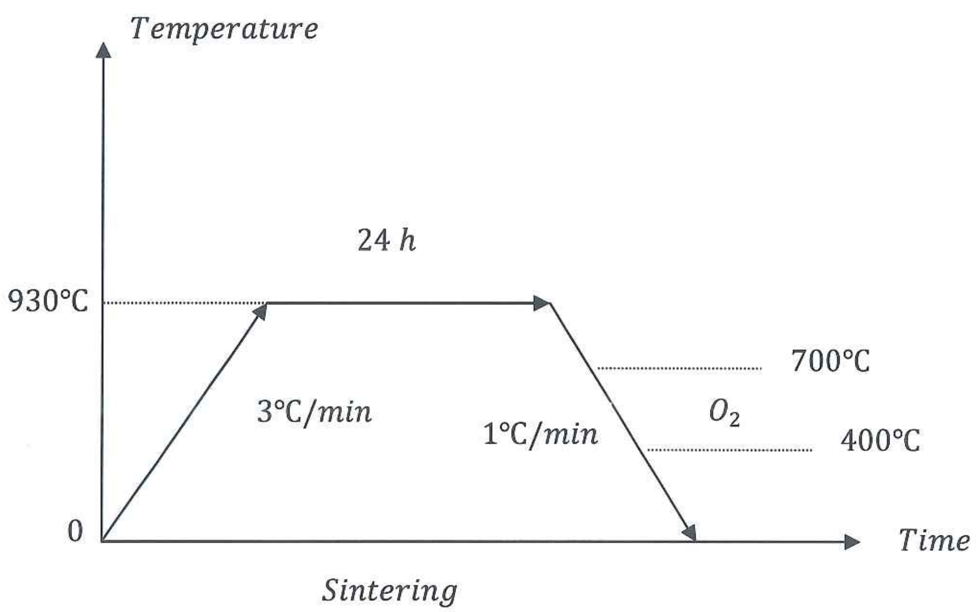

Figure 3.2. Sintering process of the samples

After this stage, the weight ratio of Au in YBCO samples were calculated as 1%, 5%, 15% and 20% of YBCO. After the addition of Au powder every samples were regrinding and shaped in the appropriate die and pressed under the 230 MPa. The calcination and sintering process of Au added in YBCO samples are the same as the pure YBCO sample. In addition to this a pure sample was annealed under the same condition for comparision with the others. All this annealing stage done in a programmable tube furnace from PROTHERM (Model PTF).

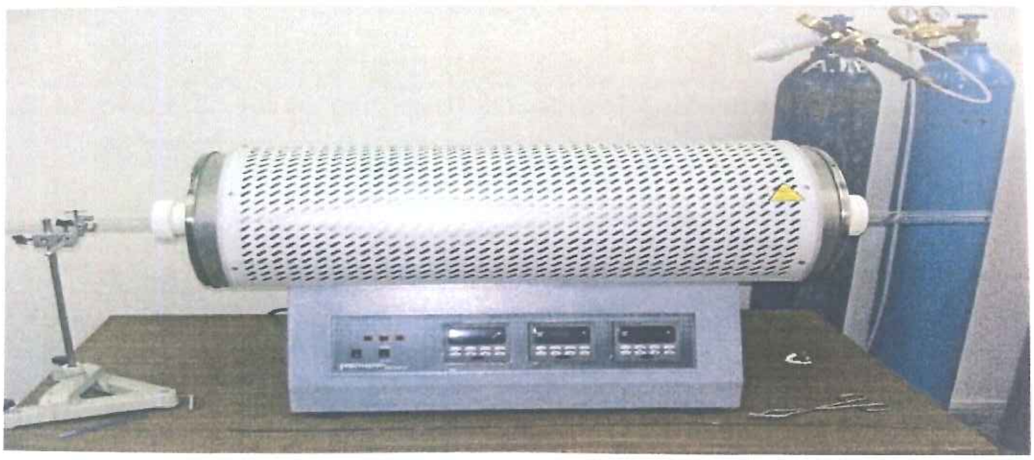

Figure 3.3.PROTHERM (Model PTF 200) programmable tube furnace.

3.2 Sample Characterization

3.2.1 X-Ray Diffraction (XRD) Measurements

In this study, to identified the structural properties and crystalline phases present in the materials, XRD technique was used.The XRD data were collected at room temperature by using a Rikagu D/Max – IIIC (see Figure 3.4) diffractometer with CuKα radiation ($\lambda = 1.5418$ Å) over the range $2\theta = 3^{\circ}$- 70° with the scan speed of $3^{\circ} / min$ and a step increment of 0.02°. The XRD data yield about lattice parameters for pure and Au added Y-123 bulk samples. Phase purity and the lattice parameters were determined from these XRD results.The cell parameters a, b and c are computed with the least square method through d values and (hkl) planes for orthorhombic unit cell structure. The accuracy in determining the lattice parameter c was ± 0.001 Å. From the XRD data the cell parameters for orthorhombic structure can be calculated as:

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$$

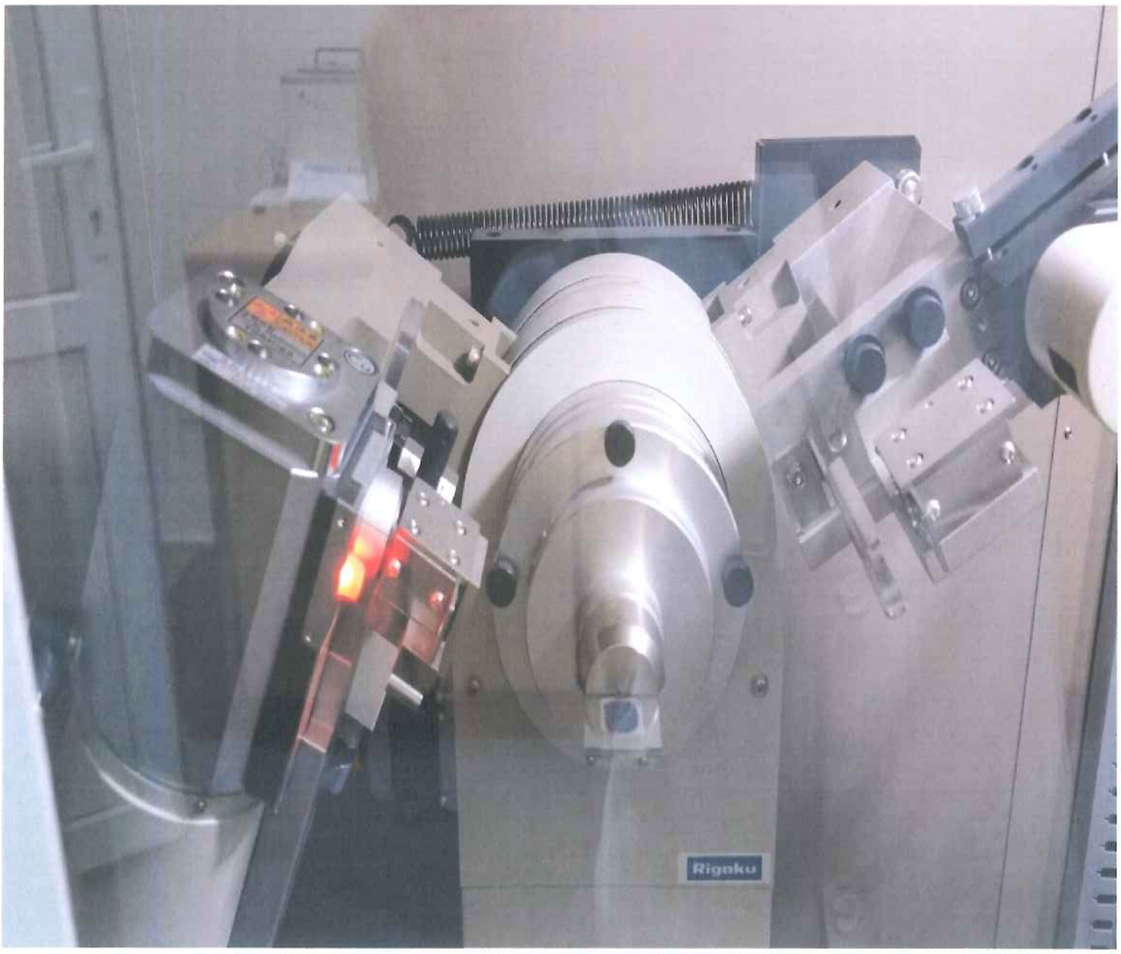

Figure 3.4.A picture from Rigaku X-ray diffractometer system during the powder x-ray diffraction.

3.2.2 Scanning Electron Microscopy (SEM) And Energy Dispersive X-Ray (EDX) Analysis

The microstructure and the surface morphologies analysis of the Au-doped and undoped bulk samples were studied by using a scanning electron microscope (SEM) Philips XL30 SFEG. The eligible and semi-eligible identifications of the sample were searched by SEM equipped with an Oxford X-ray microprobe(EDX), IXRF SYSTEMS Model 550i Analyzer (Figure 3.5). Scanning electron microscope SEM and EDX micrographs were taken from fracture surface of the bulk samples. Surfaces of the bulk samples were enlarged by micrographs about 5000 times. So we examined the surface morphologies of the sample for dimensions, connection and size of the grains.

Figure 3.5.JEOL JSM-6390LV Scanning Electron Microscope

3.2.3 Resistivity Measurements

The electrical characteristics of doped and undoped Y123 superconducting samples measurements were carried out by using CRYOSTAT (see Figure 2.6). The measurements of DC resistivity were performed with the four-probe method on all samples with a Keithley 220 programmable current source and a Keithley 2182A nano-voltmeter system. The contacts of both voltage and current were made by the silver paint (see Figure 3.7). We measure the temperature from 20 K to 120 K dependence of resistivity of the samples with running $5mA$ DC current through the samples in the cryostat. The transition temperature,T_c, was defined as R=0. The calculation of the resistivity,ρ, is made as following relations;

$$R = \rho\frac{L}{A}\ , \quad \text{and} \quad V = I.R \quad , \quad \rho = \frac{R.A}{L} = \left(\frac{V}{I}\right).\left(\frac{A}{L}\right)$$

where L is the distance between the inner electrical contacts , and A is the cross-section of bulks .

In addition to this, the onset critical temperature (T_c^{onset}) where the resistance started to decrease whereas the offset transition temperature (T_c^{offset}) of the samples is obtained as the temperature at which R = 0 Ω

3.2.3.1 Measurement Set-up

The system consist of a closed cycle cooler system which includes the cryostat build on an Edwards model CS2/9 cold head, an intelligent temperature controller (ITC), integral control unit and a water cooled compressor unit, a pair of flexible helium lines. To producing the cooling, Helium is compressed to 16.5 bar in the compressor and then expanded in controlled manner in the cold head.The intelligent temperature controller controls both the heater power to carry out stable control and regulates the speed of the cold head no higher than is necessary. The cold part of the cold head is connected to a copper block and sample, which contains rhodium iron temperature sensor and two wire wond resistor heaters to allow variable temperature operations.

After the sample was placed into the vacuum loading cryostat,the samples were ready to be measured.

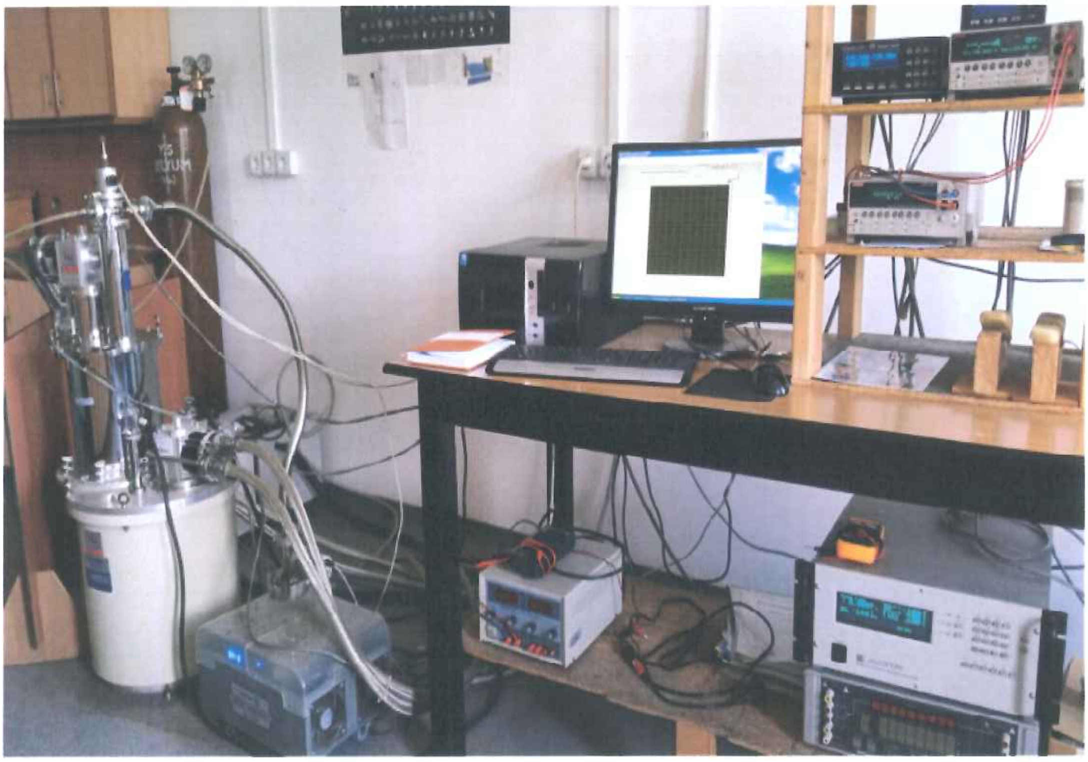

Figure 3.6. He gas contact crycooler and superconducting coil magnet from CRYO Industries.

Figure 3.7. Experimental set-up for the 4-point probe measurement.

3.2.4 Critical current measurements (Current – Voltage (I-V) measurements)

The Current-Voltage measurements of samples were made at liquid nitrogen temperature by using the four-probe method on all samples. Both voltage and current contacts were made with silver paint. The transport critical current experiments were made at the liquid nitrogenous temperature of 77 K in zero magnetic field. The current was applied to the two outer electrical contacts and the voltage drops across the two inner electrical cantacs were measured against current. The Lakeshore Model: 340 temperature controller were used which permit us to accurately monitor the temperature with a stability and accuracy of ±0.02 K and the experimental data were obtained by Labview software. The critical current (I_c) values were determined with the criterion of $1\mu V/cm$. The $J_c$values of the samples produced are evaluated from rato of the I_c to the total cross-sectional area. To calculate the critical - current density (J_c); we divide the critical current by the cross- section area A of the conductor.

$$J_c = I_c/A$$

3.2.5 AC Susceptibility Measurements

Ac susceptibility measurements of the samples were performed using a 7130 AC susceptometer of Lake Shore with a closed cycle refrigerator at 500 Hz frequency. The susceptometer consist of a primary coil and two secondary coils which are a drive coil to apply ac magnetic field, a thermocouple to measure the temperature and a heater to heat the sample. The sample was reassembled with vacuum grease tightly on the sample holder at the centre of one of the secondaries.The coil system was placed in a vacuum sealed pyrex tube. He gas is transferred to pyrex tube in order to heat exchange medium. He gas pressure was 1 bar. The AC was applied along the longest dimension of the sample. The AC susceptibility as a function of temperature (20–120 K) at fixed ac magnetic field amplitudes (H_{ac}) of 2, 4, 6, 8 and 10 Oe was performed on the samples.

Susceptibility data were taken from Lock-in Amplifier were recorded using a computer programme. To investigate magnetic and superconducting properties of materials AC susceptibility measurement were generally used. AC susceptibility measurements are useful in order to compare inter- and intra-grain properties. At high ac fields, two loss peaks can usually be seen in the imaginary part of susceptibility data (Ozturk et al., 2009); a broad peak at low temperature (coupling losses) due to the motion of inter-granular (Josephson) vortices (Muller et al., 1989) and a narrower peak (intrinsic losses) due to the motion of intra-granular Abrikosov vortices near T_c^{on}(Muller et al., 1991). The values of inter-granular critical current densities $J_c(T_p)$ were calculated using Bean critical state model (Bean, 1964) from AC susceptibility measurements. From the χ'andχ'' versus T plots, the values of the T_c^{on}(diamagnetic onset temperature) and T_p (temperature corresponding to χ'' peak) were obtained, respectively.

Figure 3.8. The cryostat assembly ac susceptibility system.

4. RESULT AND DISCUSSION

4.1 Introduction

Some metals, such as copper (Cu), silver (Ag) and gold (Au) etc., have been inspired by many researchers with the discovery of high temperature superconductor (HTSC) (Dzhafarov et al., 1996; Gorur et al., 2005 a; Ozturk,2007; Terzioglu, 2010; Turkoz, 2013). To investigate the effect of the partial substitution on structural, superconducting and mechanical properties of high temperature superconducting materials, especially on $YBa_2Cu_3O_{7-x}$ (YBCO) and Bi(Pb)-Sr-Ca-Cu-O (BSCO) system, a wide range of studies have been performed by several groups (Feng et al., 1994; Mellekh et al., 2006; Bal et al., 2012; Guner et al., 2012; Ozturk et al., 2012; Yildirim et al.,2012; Turk et al., 2014). From the literature review,it is seen the effect of addition or diffusion-doped of metal or metal-based chemical powders on the mechanical, magnetic, electrical and physical properties of superconducting compounds (Ozturk et al.,2013 ; Zalaoglu et al., 2012; Dogruer et al., 2012; Yildirim et al., 2011; Gorur et al., 2005 b). Terzioglu and his co-worker (Terzioglu et al., 2009; Ozturk et al., 2009; Ozturk, 2009) published extensive reports about the effect of Au diffusion-doped on phase formation, superconducting, microstructure and physical properties of $Bi_{1.8}Pb_{0.35}Sr_{1.9}Ca_{2.1}Cu_3O_y$ superconductors. According to the reports given, the reserchers observed both the improvement in the critical current density, critical transition temperature, microstructure of surface morphology, lattice parameter c and high-$T_c$phase, and the decrease in the normal state resistivity for the Au-dopped samples as compared to the undoped samples. On the other hand, study on gold addition in BSCO systems (Grivel & Flukiger, 1994; Liu et al., 1990) showed that Au addition rarely affected the T_c, lattice constant parameters and formation of Bi-2223. In another study, Jin et al. (Jin et al.,1998) found that Au addition reduced the superconductivity of Bi(Pb)-Sr-Ca-Cu-O. However, the addition of Cr, Pr, Ag etc. affects the superconductivity properties of Bi-Sr-Ca-Cu-O systems. Some researchers found that the zero resistivity transition temperature and transport critical current decreased; however, cell parameter a increases while the c parameter decreases as Ce-content increases in Bi-2212 system (Bal, 2012). Furthermore, Ozturk et al. proposed that Pr addition in Bi-2122 superconductor increased the room temperature resistivity and transition temperature (T_c^{onset}) (Ozturk et al., 2012). On the other side, the addition of Ag in the BSCO systems leads to better superconducting properties as indicated by the increase in T_c and J_c (Akdogan et al.,2012) while it reduces the resistance of the normal state and the contact resistance (Comert et al., 1994).

Correspondingly with BSCO systems, many researches have been done to explore the effect of addition in YBCO systems.In the literature review, Dzhafarow et al. scrutinized the effect of Co electro diffusion on the properties of YBCO superconductors and it was found that the Co-doping of YBCO decreased the transition temperature and critical current density (Dzhafarow et al., 2001). Gorur et al.(Gorur et al., 2005 a, 2005 b) have noticeable work on Ag diffusion-doped $YBa_2Cu_3O_{7-x}$ superconductors. They observed that the Ag diffusion into $YBa_2Cu_3O_{7-x}$ increases the critical current density, the critical transition temperature and the lattice parameter *c* with respect to the pure YBCO samples (Gorur et al., 2005 a). Dogan studied the effect of duration of diffusion on Ag diffusion coefficients in YBCO (Dogan, 2005). They obtained that Ag diffusion coefficients in YBCO have decreased with increaseing duration of diffusion. In the other study of Ag-doped $YBa_2Cu_3O_{7-x}$ thin films produced by electron beam deposition techniques performed by Gorur et al.,the lattice parameter *c*, critical temperature and critical current density increased while the normal state resistivity decreased harshly(Gorur et al., 2005 b). Furthermore, Zr added (Guner et al., 2012) and Lu added in YBCO systems (Turkoz et al.,2012) have investigated. Guner et al. observed that T_c^{onset}, T_c^{offset} and J_c increased with increasing the Zr content. Turkoz et al. found that T_c^{onset} and T_c^{offset} increased but $J_c$decreased with increasing the Lu content.Moreover, Dogruer et al. studied the effect of Zr diffusion doped in YBCO systems on activation energy, diffusion coefficient and mechanical properties. They found that physical and mechanical properties of Y123 improved with Zr diffusion(Dogruer et al., 2013). Ozturk et al., researches the influence of Gd diffusion doped on structural and superconducting properties of YBCO superconductors. They showed that the T_c^{onset} and $J_c$values enhance with increasing the Gd diffusion (Ozturk et al., 2007). However, Dogruer et al., investigated the result of Gd addition in YBCO system on microstructural, mechanical and superconducting properties and obtained that the T_c^{onset}, T_c^{offset} and J_c decreased with increment the Gd content.(Dogruer et al., 2012).

Cieplak et al. (Cieplak et al., 1990) studied the Au subtitution in YBCO system in 1990. In their work, they investigated the consequence of Au addition on structural and superconducting properties in YBCO with x=0.025-0.1. It was found that T_c value of the Au added samples increased and caused a uniaxial lattice expansion along the *c*-axis with increasing Au content (x=0.025-0.1). Nevertheless, the T_c value of the samples with x> 0.1 did not change. Varilci and his co-worker studied the effect of Au diffusion in YBaCuO system and it was found that the Au diffusion in YBaCuO increased the critical current density, the critical temperature and the lattice parameter *c* (Dzhafarov et al., 1995).

In this thesis we studied the effect of Au addition with x=0.1, 0.5, 0.15 and 0.20) in $YBa_2Cu_3Au_xO_{7-x}$ superconductors on the structural, magnetic, electrical and superconducting properties. To the best of our knowledge, no detailed study on the effect of Au addition on electrical, microstructural and magnetic properties of YBCO has been published in the literature.

4.2 X- Ray Diffraction Characterizations

We have prepared the samples with solid state reaction method. Figure 4.1 shows X-ray powder diffraction patterns of pure ($YBa_2Cu_3O_7$) and Au-added superconductors with x=1, 5, 15 and 20%. Hereafter, we will use the abbreviations Y0 (undoped sample), Y1 (Au added sample, x=1 wt.%), Y2 (Au added sample, x=5 wt.%), Y3 (Au added sample, x=15 wt.%), Y4 (Au added sample, x=20 wt.%).

Some of the Miller indices are indicated in the figure. We use the linear least square method to calculate the lattice parameter of the samples. The lattice parameters *a*, *b* and *c* from (*hkl*) peaks of the XRD data are calculated and tabulated in Table 4.1. The XRD patterns revealed that all samples are polycristalline in nature and correspond to orthorhombic structure of YBCO.

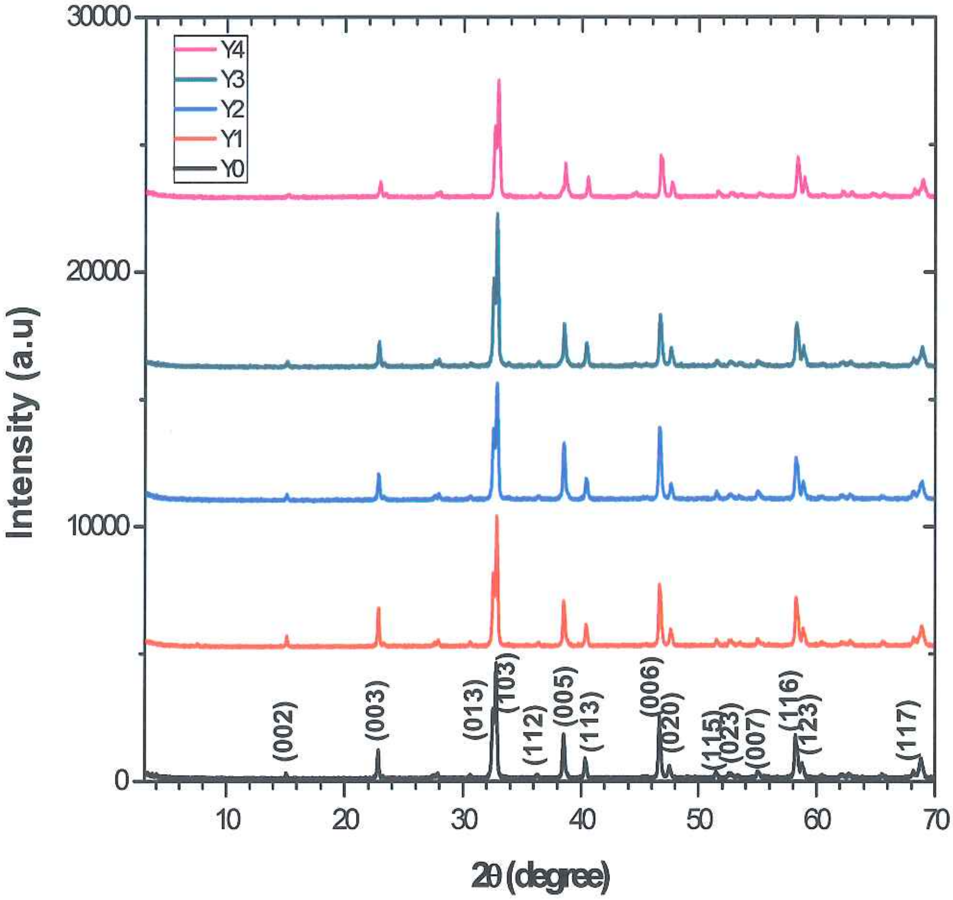

Figure 4.1. The powder XRD patterns for the *Y0, Y1, Y5, Y15*and *Y20* samples recorded at room temperature

The lattice parameter *c* increased with addition of x= 1% (Y1 sample) in comparision with the undoped sample (Y0). However, lattice parameters of *a* and *b* remained essentially constant with Au content up to x=0.1, but remarkably , beyond which the lattice parameter increased (Cieplak, 1990). Appreciable increase in the lattice parameter *c* of Y1 sample has been observed within the experimental limit of 0.001 Å. This result indicates that Au has entered into the structure and caused an expansion of the lattice parameter *c*.

Table 4.1. Experimental data of $YBa_2Cu_3O_{7-\delta}$+Au_x (x= 0, 1, 5, 15 and 20 wt.%) samples.The accuracy in determining the lattice parameter c was ± 0.001 Å.

Samples	Lattice parameter a (Å)	Lattice parameter b (Å)	Lattice parameter c (Å)	Volume(Å^3)	Crystal size(nm)
Y0	3.828	3.889	11.710	174.328	38.2
Y1	3.827	3.886	11.735	174.520	46.6
Y2	3.819	3.885	11.737	174.140	50.1
Y3	3.809	3.883	11.738	173.609	70.1
Y4	3.809	3.881	11.739	173,534	70.2

In addition, the broadening nature of the XRD peaks yields that the particle sizes inferred from the Scherrer-Warren equation are within the nanometer scale(Dogruer M et al., 2012; Yıldırım et al., 2012).From the XRD data findings, we calculated the average crystallite size values for each sample by means of the followin equation:

$$d = \frac{0.941\lambda}{B \cos \theta_B}$$

In this relation,d corresponds to the crystal thickness, λ the wawelenght of the incident X-rays (λ =0.1540 nm). B denotes the full width at half maximum (FWHM) of the Bragg peak when θ_B provides Bragg angle. The grain size of the all sampleswascalculated as shown in Table 4.1.

One can see from the figure that the main peak (*103*) intensity of the Y1 sample increased in comparison with theY0 sample. The enhacement in the peak intensities may point out the enhanced grain growth and orientation in the presence of the Au content, leding to the increase of the critical current density (Vinu et al., 2009; Vinu et al., 2010) which is in good agreement with our critical current density measurements. The increase of intensity and the decrease of full width at half maximum (FWHM) for Y1 sample indicate that crystallite size became bigger when compared with undoped sample Y0. The grain sizes are about 46.6, 50.1, 70.1 and 70.2 nm for Y1, Y2, Y3 and Y4 samples, respectively. Namely, it would be more precise that the crystallite size increased with enhancing the Au content in the crystal lattice. This result is consistent with SEM micrographes which are considered in detailed later.

It has been established (Chien et al., 1988; Xiao et Al., 1988) for the Y-1:2:3 system that all the divalent dopants (Zn^{2+}, Ni^{2+}) substitute for the Cu(2) plane sites, whereas all the trivalent dopants (Au^{3+}, Co^{3+}, Ga^{3+}) occupy the Cu(1) sites. When the Cu(1) site has been doped, an enhance in the oxygen content have been seen. For this reason, Au presumably is in the trivalent state, which indeed is a common valance state for Au (Greenwood & Earnsh, 1984).

Another effective reason of Au addition is that the influential ionic radius of Au^{3+} is 0.85 Å so, it is close to Cu^{2+} (0.73 Å), because the other ionic raius ofAu^{1+} (Au^{1+}, 1.37 Å) is too large for the Cu(1) site.

4.3 SEM Analyses

The surface morphology of the Y0, Y1, Y2, Y3 and Y4 samples was studied by SEM for the determination of the grain sizes and possible precipitation at the grain boundaries. Figures 4.2-6 display the SEM micrographs taken in the secondary electron image mode of the fractured surface of the all samples. It is clear from the figures that the microstructure of the samples are significantly different from ech other. The granular nature and the porosity of the samples is clearly seen in particular in Fig.4.5 for pure sample. The surface of the Y1 sample sample is much smoother and denser than that of the Y0 sample. A significant increase in the average grain size is also evident. This results indicate that the surface morphology of the YBCO sample is improved by content. As can be seen from the figures, the garain size increased with increasing the Au content, which is consistent with grain sizes determined from the Scherrer-Warren equation in the previous section. Based on the results, a close relation among the microstructure, impurities and superconducting properties with the Au content was detected.

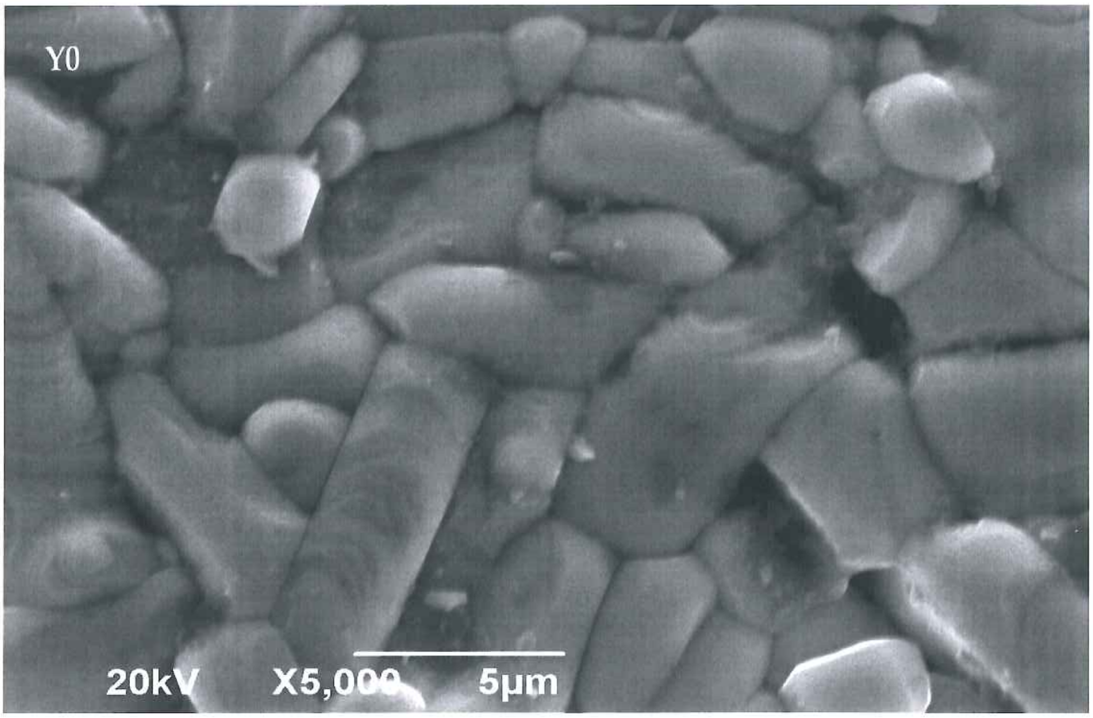

Figure 4.2.SEM micrographs of the of $YBa_2Cu_3O_{7-\delta}$+Au_x (x= 0 and 0 wt.%) sample.

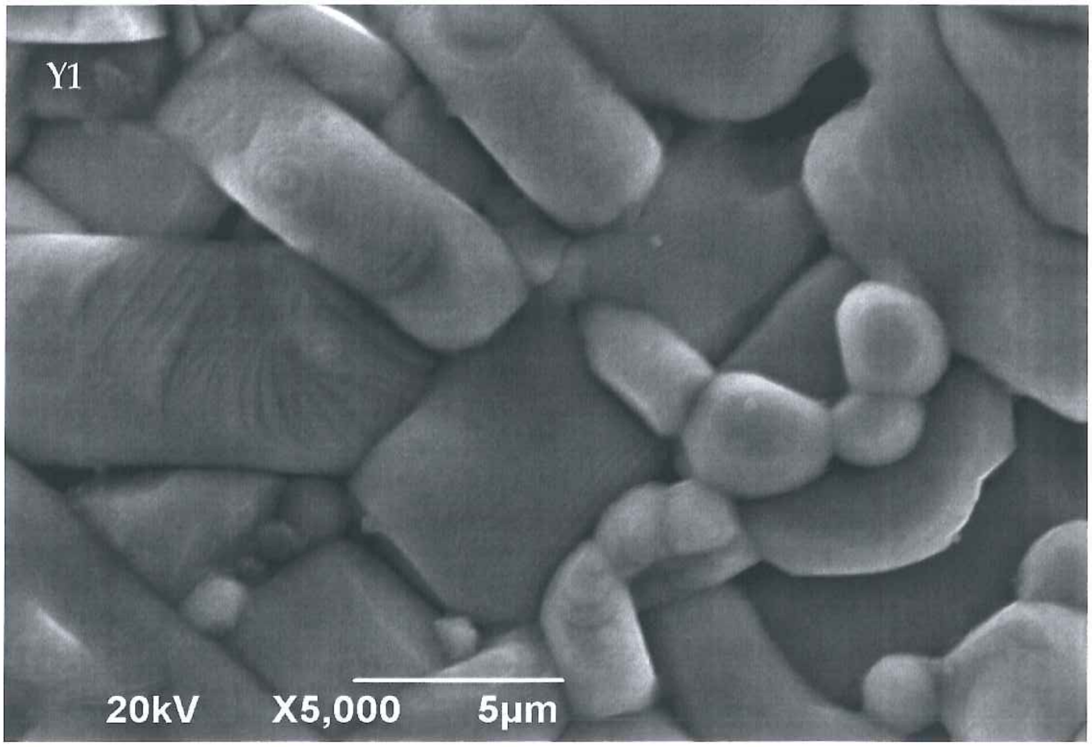

Figure 4.3.SEM micrographs of the of $YBa_2Cu_3O_{7-\delta}$+Au_x (x= 1 and 1 wt.%) sample.

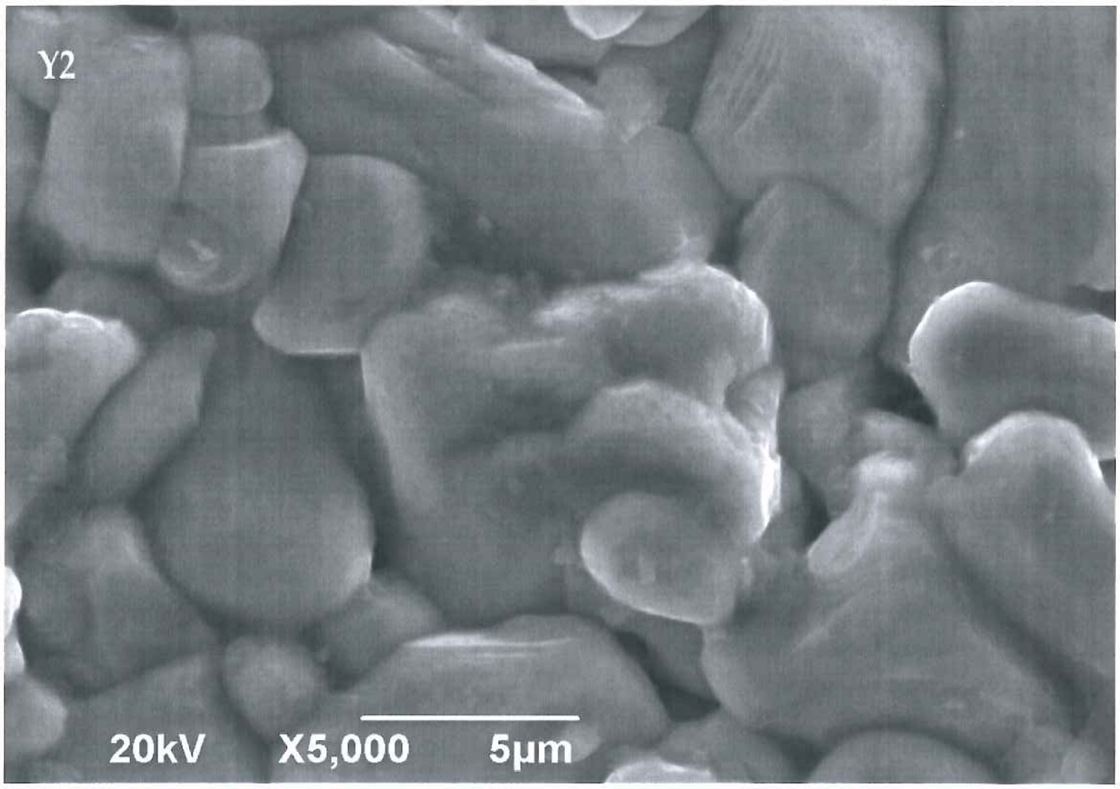

Figure 4.4.SEM micrographs of the of $YBa_2Cu_3O_{7-\delta}+Au_x$ (x= 5 and 5 wt.%) sample.

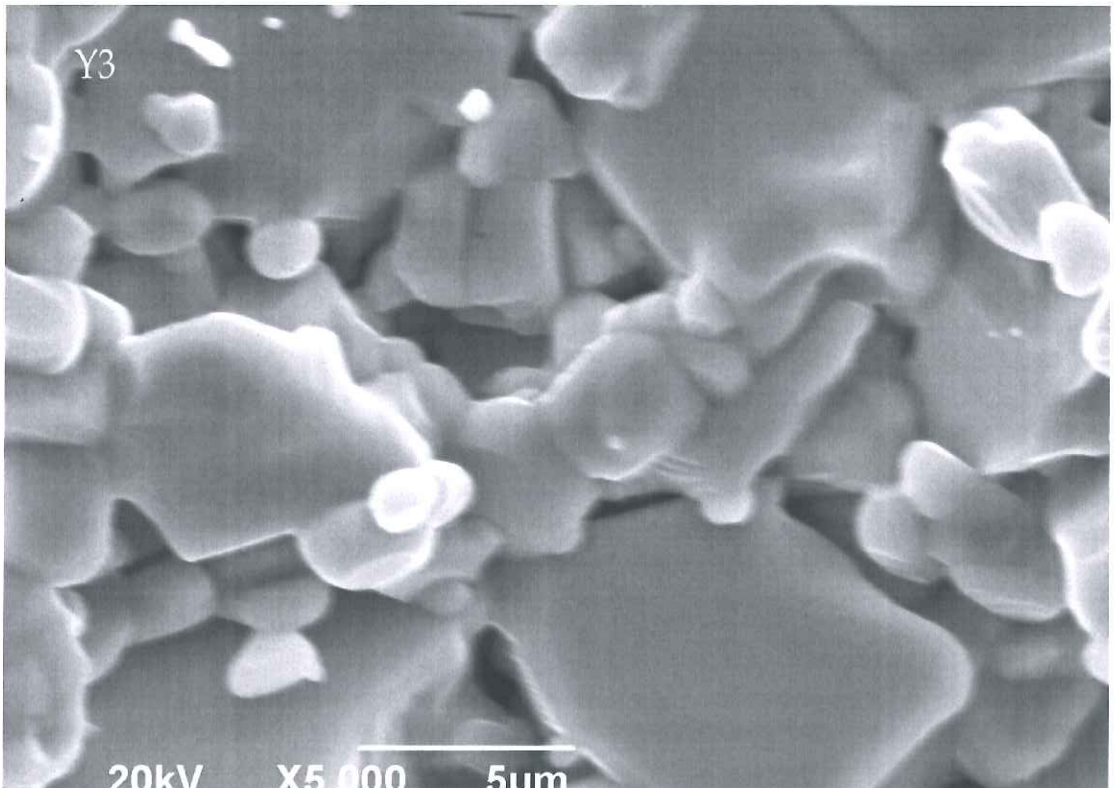

Figure 4.5.SEM micrographs of the of $YBa_2Cu_3O_{7-\delta}+Au_x$ (x= 15, 15 wt.%) sample.

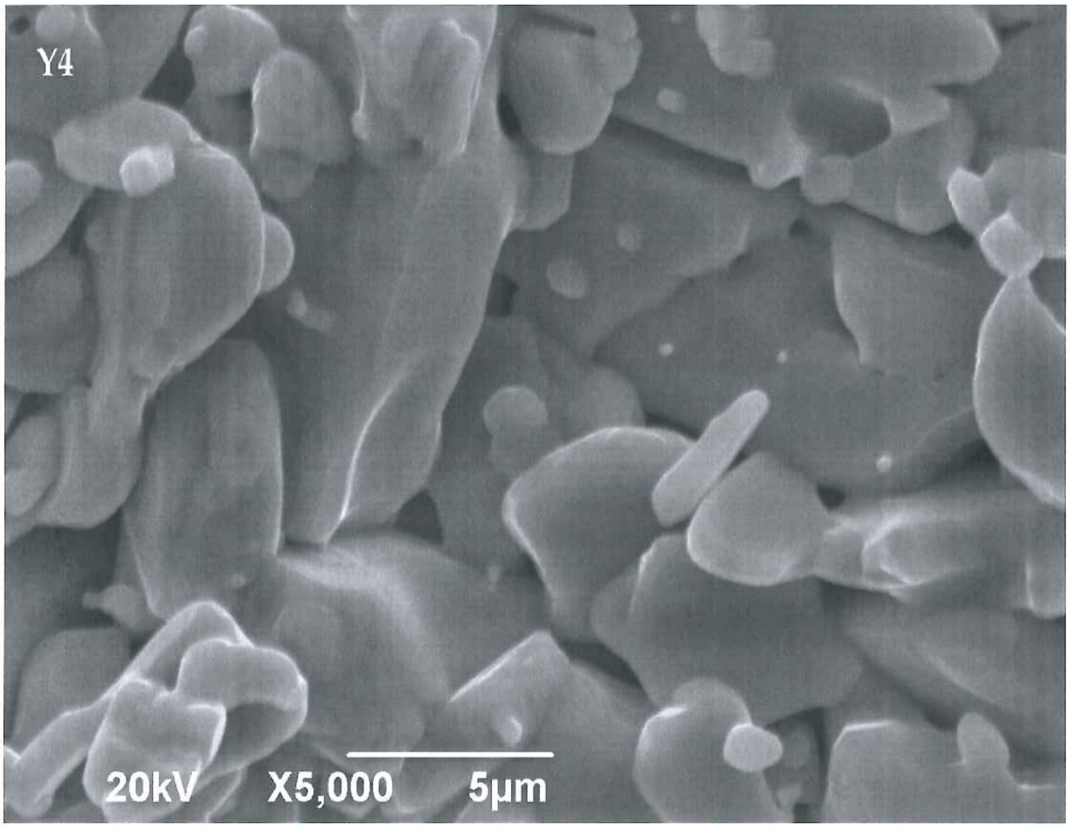

Figure 4.6.SEM micrographs of the of $YBa_2Cu_3O_{7-\delta}$+Au_x (x= 20, 20 wt.%) sample.

4.4 Resistivity Measurements

In this study, the effect of Au addition on superconducting properties of $YBa_2Cu_3O_{7-\delta}$superconductor material is examined in the temperature range of 80-110 K with the aid of the dc electrical resisitivity measurements. The temperature dependence of electrical resistivityfor Y0, Y1, Y2, Y3and Y4 samples is shown in Figure 4.7. Room temperature resisitivity, tarnsition width and the transition temperature values of all the samples are listed in Table 4.2. The Y0 sample showed the lowest room temperature resistivity. As seen from the figure that all the samples show metalic behaviour above the transition temperature. The room temperature resisitivity value increased from $4.1x10^{-4}$ to $2.4x10^{-3}\Omega$.cm with increasing Au content. This increment results in a short-circuiting of the grains due to the increment of porosity of the Au added samples in the normal state (Tepe et al., 2004).

The transition temperature values are found to be 92.40, 94.05, 93.48, 92.69 and 92.16 K under zero applied field for Y0, Y1, Y2, Y3 and Y4 samples, respectively. As can be seen in the table, among them, the Y1 sample showed the highest value of 94.02K, for the starting temperature of resisitivity drop. Thus, the Au content enhanced the transition temperature from 92.40 to 94.02 K. Appreciable increase in T_c may be related to recruitment of the hole density and possible variation in the lattice vibration of YBCO with the substitution of Cu ions by Au ions (Gorur et al., 2005). Furthermore, the transition temperature of the Au added samples decreased from 94.02 to 92.16K with excess Au content.

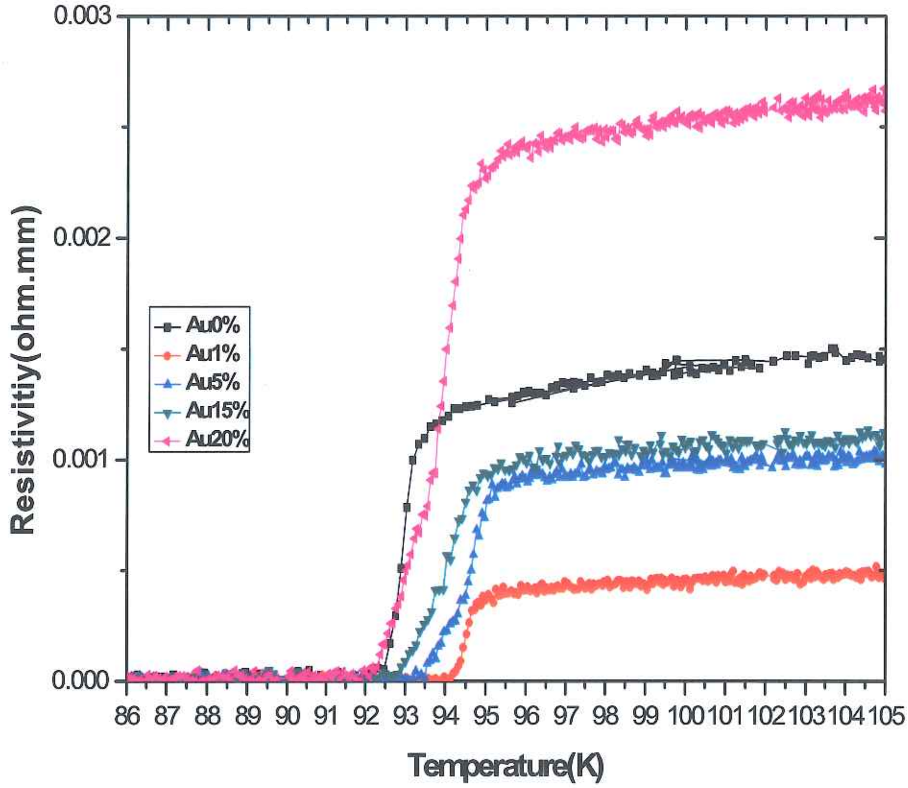

Figure 4.7. Temperature dependence of resistivity for*Y0, Y1, Y5, Y3* and *Y4* samples under zero applied field.

Table 4.2. Experimental data of $YBa_2Cu_3O_{7-\delta}$+Au_x (x= *0, 1, 5, 15* and *20* wt.%) samples.

Samples	T_c^{on}(K)	T_c^{off}(K)	ΔT(K)	Resistivity, ρ,($\Omega.cm$)
Y0	92.40 ± 0.02	93.41 ± 0.02	1.04	1.2×10^{-3}
Y1	94.05 ± 0.02	94.78 ± 0.02	0.73	4.1×10^{-4}
Y2	93.48 ± 0.02	95.05 ± 0.02	1.57	9.2×10^{-4}
Y3	92.69 ± 0.02	94.64 ± 0.02	1.95	9.7×10^{-4}
Y4	92.16 ± 0.02	94.75 ± 0.02	2.59	2.4×10^{-3}

As can be seen from the table, the transiton width (ΔT_c)value of the Y0 sample is observed to be higher than that of Y1 sample. The reduction of broadening in $\rho - T$ curve may be due to the modifying effect of the gold addition on the grain boundaries. Sharper transitions are accompanied by higher critical temperature and higher critical current density values (Ozturk O, 2007), being in good agreement with our critical current density values. However, the ΔT_c values of the Au added samples (Y1, Y2, Y3, Y4) increased with ascending the amount of gold content. This systematical increase in the Au-added sample is due to both the increase of porosity, grain boundary resistivity and weak-links in the crystal structure (Ianculescu et al., 1996), which is consistent with the results of SEM results.

4.5 Critical Current Measurements

The transport critical current density, J_c, measurements were performed at liquid nitrogen temperature for the Y0, Y1, Y2, Y3, Y4 samples. The measured critical current, I_c, and the calculated critical current densitity values for all the samples are listed in Table 4.3. The Y1 sample showed the J_c of 167.52 A/cm^2 at 77K while the Y0 sample showed the J_c of 82.81 A/cm^2. The enhanced critical current density is observed by Au content. The critical current density has an important role to explain the pinning mechanism in a material and quality of the interaction between the grains in the crystal structure. Therefore, the increment of the J_c in YBCO system by Au addition may be caused by the increase of the grain size and their orientations, by improved coupling between Au-added superconducting grains, and by the increased number of flux pinning centres due to the precence of Au in the intergrain region, which is in good agrement with the previous work (Ozturk, 2007). Furthermore, this enhancement of the J_c value by Au content is associated with not only the reduction of porosity and weak links between the superconducting grains but also the increase in the flux pinning centers (Guner, 2012).

However, the critical current density values of the Au added samples decreased with increasing Au content. Eventhough the decrease in T_c is very much less with the enhancement in the Au concentration level, the values of J_c decreased drastically due to the increase in not only the porosity but also the non-superconducting phase scattered along with the samples. As can be seen from Table 4.3, the critical current density is greately influenced by Au content. The higher Au content leads to bad grain connectivity and contributes to decrease of the J_c.

Table 4.3. Some physical properties of *Y0, Y1, Y2, Y3* and *Y4* Samples

Samples	T_c^{on}(K)	$I_c(A)$	$J_c(A/cm^2)$ (at 77 K)
Y0	92.40	6.9	82.81
Y1	94.05	13.1	167.52
Y2	93.48	13.3	131.68
Y3	92.69	10.2	98.08
Y4	92.16	9.1	74.59

4.6 AC Susceptibility Measurements

An impressive change of the electrical transport is strictly related to the magnetic transport of the HTS. AC susceptibility measurements are a strong tool to infer information about the diamagnetic onset temperature, flux dynamic and surface shielding of the materials (Hein RA, 1986). For this reason, we have also investigated the effect of Au addition in Y-123 system by using the low field AC magnetic susceptibility method. AC susceptibility measurements on the samples were performed using a 7130 AC susceptometer of Lake Shore with a closed cycle refrigerator at the constant frequency of 500 Hz. The AC susceptibility as a function of temperature (20–120 K) at fixed AC magnetic field amplitudes (H_{ac}) of 2, 4, 6, 8 and 10 Oe was performed on the samples. For AC susceptibility measurements automatic data acquisition was selected. Figure 4.8. indicates the AC susceptibility ($\chi(T) = \chi'(T) - i\chi''(T)$) as a function of temperature under 4 Oe AC field amplitude for the Y0, Y1, Y2, Y3 and Y4 samples.

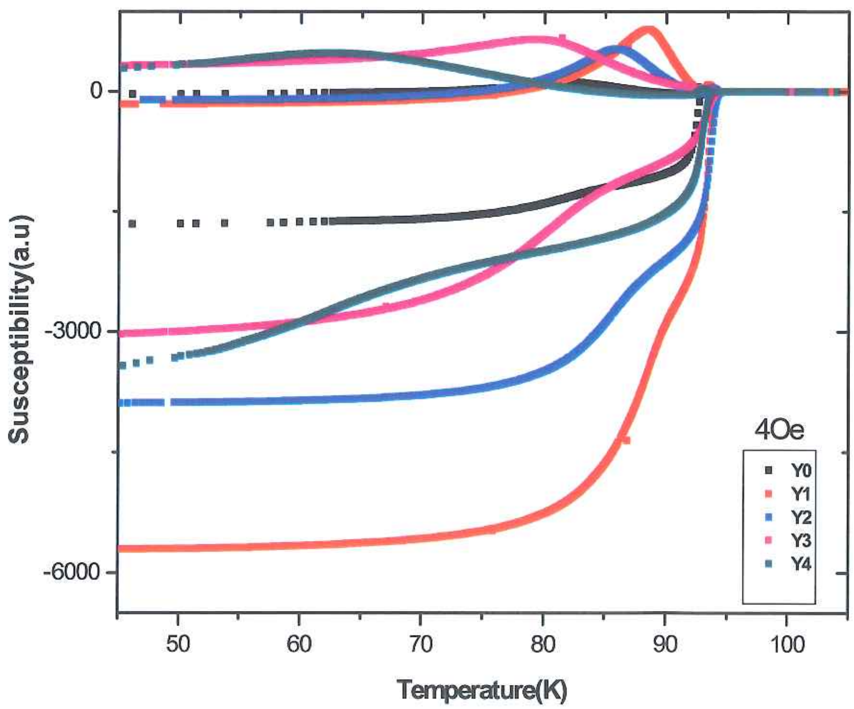

Figure 4.8.AC susceptibility as a function of temperature for 4 *Oe* AC field amplitudes for all samples

As seen from the figure, $\chi' - T$ curve of the sample represents the diamagnetic onset temperature and shielding fraction of the superconducting phases while the $\chi'' - T$ curve displays the dynamics of flux penetration into the superconductor (Yildirim et al., 2011). The maximum of χ'' apears at temperature T_p where the inter-granular field just penetrates into the centre of samples (Müller et al., 1991). When the AC field amplitude level increases systematically, the intensity of the imaginary parts is found to increase for each sample as given in Figure 4.9-13. On the other hand, Figure 4.9-13 confirms that the overall susceptibilty curves of the sample are shifted to slight lower temperature with the enhancement in the AC field amplitudes appiled. Moreover, it is noted that the shift of T_p with the applied field stems from the strength of the pinning force (Terzioglu et al., 2009).

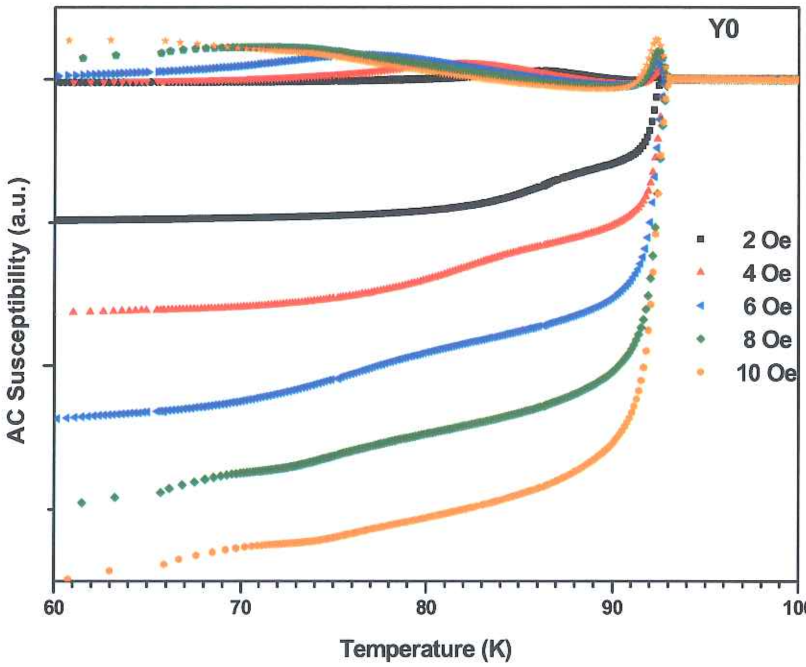

Figure 4.9.AC susceptibility as a function of temperature at different AC field amplitudes for Y0 sample.

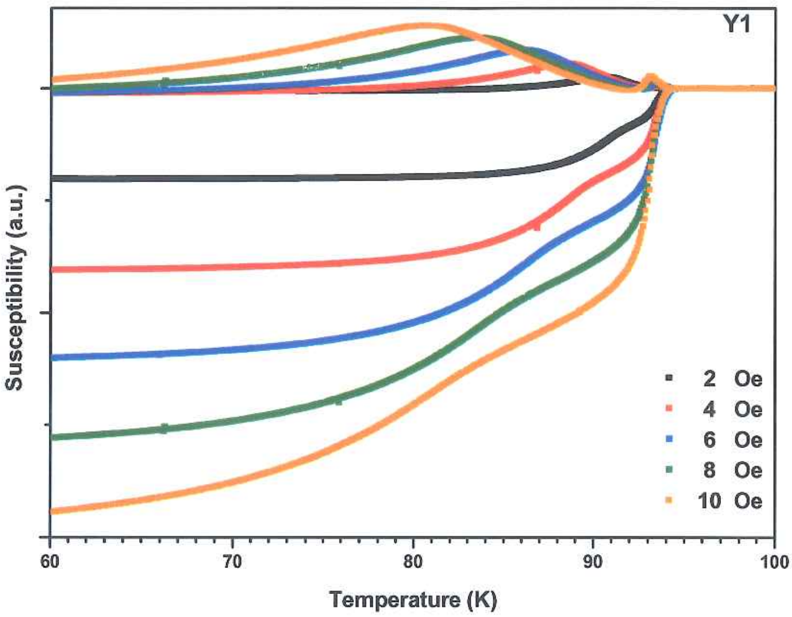

Figure 4.10.AC susceptibility as a function of temperature at different AC field amplitudes for *Y1* sample

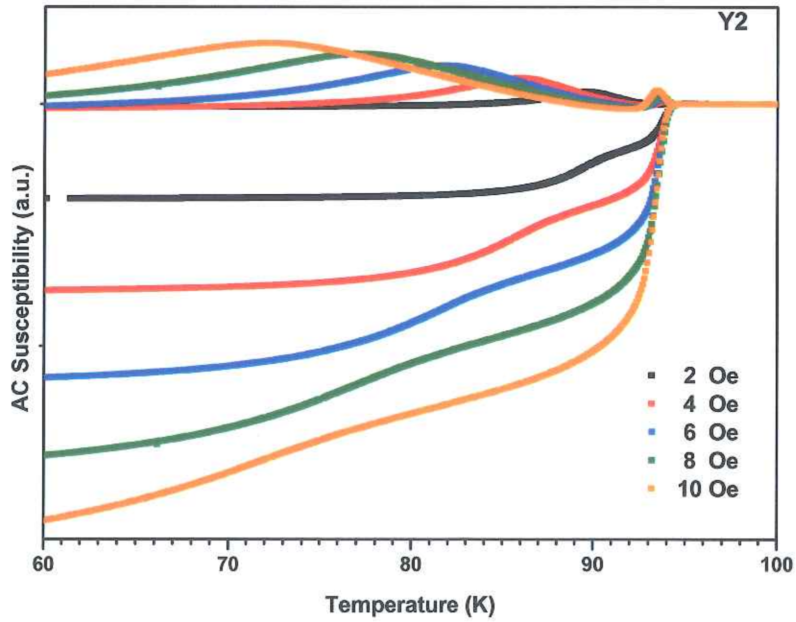

Figure 4.11.AC susceptibility as a function of temperature at different AC field amplitudes for *Y2* sample

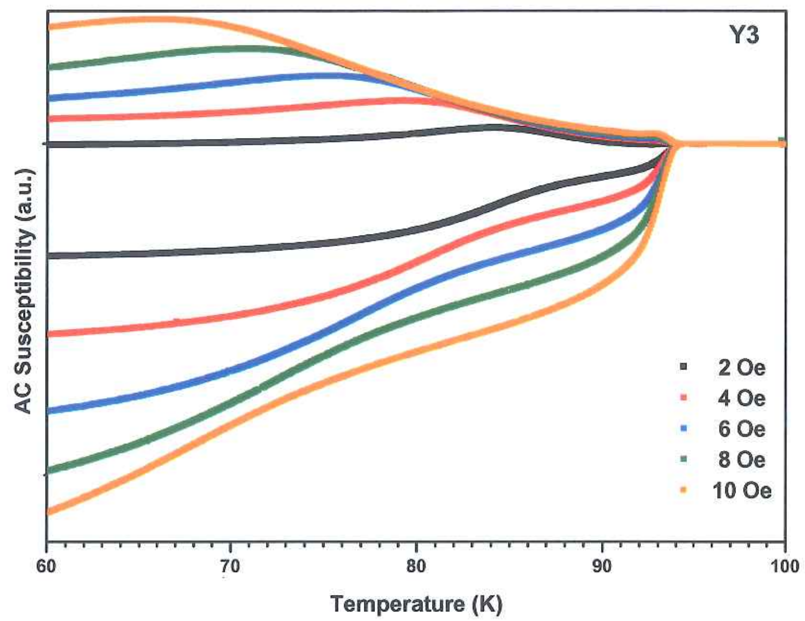

Figure 4.12.AC susceptibility as a function of temperature at different AC field amplitudes for *Y3* sample

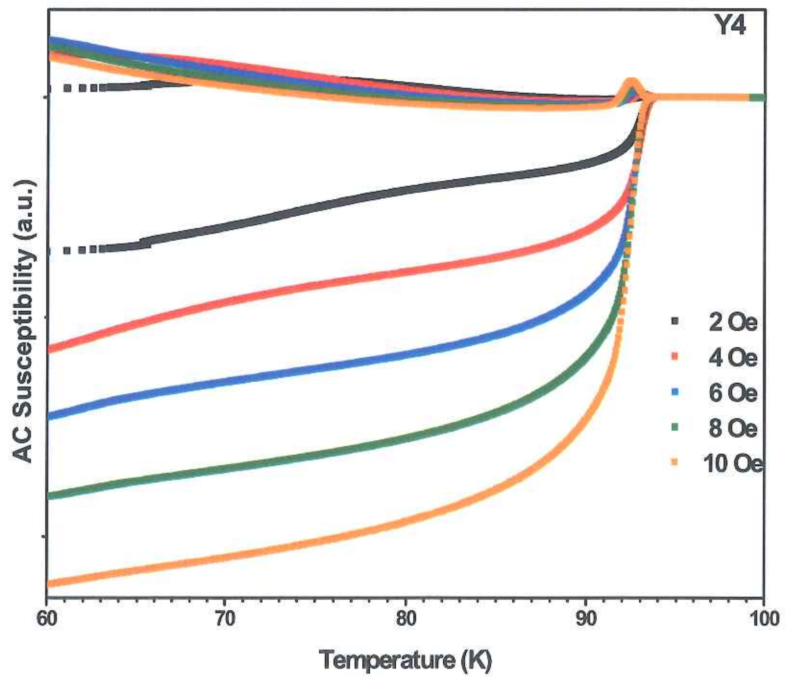

Figure 4.13.AC susceptibility as a function of temperature at different AC field amplitudes for *Y4* sample

The temperature values of the complex ac susceptibility belonging to the samples studied in this thesis are shown in Figure 4.8. It is clear from the figure that the susceptibility-temperature curves are observed to shiftto much higher temperaturewith increasing the Au content. This result is consistent with the resisitivity as a function of temeperature measurements.

The values of inter-granular critical current densities $J_c(T_p)$ were calculated using Bean critical state model (Bean, 1964) from AC susceptibility measurements as listed in Table 4.5. This result is in good agreement with our transport critical curent result.

Table 4.4. Onset and Peak temperature (K) results of the samples under various applied magnetic field.

Samples	T_c^{on}(K)	T_p(K)				
		2Oe	4Oe	6Oe	8Oe	10Oe
Y0	92.74±0.02	86.23	81.73	76.83	72.44	61.85
Y1	94.24±0.02	90.64	88.75	86.5	83.82	80.83
Y2	94.19±0.02	89.41	86.2	81.7	77.34	72.69
Y3	94.07±0.02	84.7	79.45	75.23	71.46	65.06
Y4	93.61±0.02	75	62.63	54.86	52.41	52.12

Table 4.5. Peak temperature (K) results of the samples for 4Oe applied magnetic field

Samples	$J_c^{inter}\ (A/cm^2)$	$T_p(K) for\ 4Oe$
Y0	8.6	84.09±0. 02
Y1	20.7	86.67±0. 02
Y2	12.3	80.33±0. 02
Y3	8.1	78.03±0. 02
Y4	2.0	67.01±0. 02

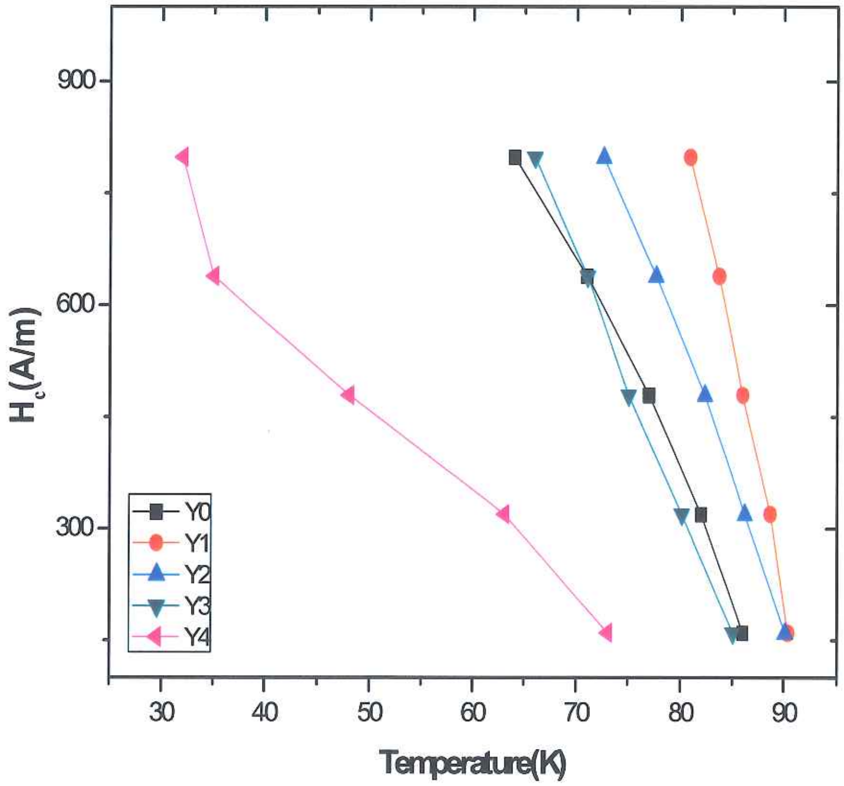

Figure 4.14. The peak temperature dependence as a function of AC magnetic field, for *Y0, Y1, Y2, Y3* and *Y4* samples

5. CONCLUSION

Along the master thesis, we shed light on the crucial variations in the crystal structure, electrical, magnetic, flux pinning and superconducting properties of the bulk Y-123 cuprate ceramics with the Au impurities embedded in the superconducting core at different concentration level with the aid of the available experimental techniques such as dc electrical resistivity, critical current density, scanning electron microscopy and powder X-ray diffraction measurements. At the same time, we evaluate the crucial parameters (being charge of many fascinating applications) as regards the average crystallite sizes, critical transition temperatures, room temperature resistivities,transition widths, surface morphologies, granular natures, porosities, pinning mechanism behaviors, lattice constant parameters *a*, *b* and *c*, weak-interaction between the superconducting grains. Similarly, the ac susceptibility measurement results enable us to determine the differentiation between the diamagnetic onset temperatures, flux dynamics and surface shielding. It is experimentally found that the characteristics given above depend strongly on both the Au foreign impurity levels. Taking into consideration all the findings, the major results to be deduced from this work are as follows:

- The dc electrical resistivity and powder XRD measurement results declare that the Au impurities introduce successfully into the Y-123 polycrystalline structures produced in this work.
- Based on the dc resistivity evidences, the room state resistivity increases considerably with the enhancement in the Au inclusions in the crystal lattice. In this respect, the Y0 sample showed the lowest room temperature resistivity of 4.1×10^{-4} Ohm.cm whereas the maximum value of 2.4x10-3 Ohm.cm is noted for the Y4 material. This is attributed to the short-circuiting of the superconducting grains due to the increment in porosity of the Au added samples in the normal state.
- As for the critical transition temperature values, the Au content makes the transition temperature enhance harshly from 92.40 K until the value of 94.02 K as a result of the improvement in the mobile hole concentration, grain misorientations, grain boundary weak connection, homogeneities in the oxidation states, local structural distortions, defects, disorders, grain boundary coupling and average

crystallite size. Moreover, it is important to stress here that the transition temperature of Au added samples decreased from 94.02 to 92.16 K with the excess Au doping in the Cu(1) sites throughout the stacked layers. Under the circumstances, the optimum concentration level of the Au concentration in the Y-123 crystal matrix is found to be about x=1 wt.% for the applications in the potential engineering, sensoring, industrial, technological and large scale fields.

- The critical current density results verify the formation of effective nucleation centers in the intragrain and inter-grain boundaries by highly dispersed Au nanoparticles for the flux pinning of the vortices in the Y-123 crystal structure. Namely, the J_c value dramatically increases from to 82.81 A/cm^2 (for the Y0 sample) to 167.52 A/cm^2 (for the Y1 material) with the increase of Au content level in the Y-123 crystal structure until the content of x=1 wt.% after which the $J_c$value tends to degrade with the excess Au concentration, in fact the value falls into the global minimum point of 74.59 A/cm^2. This is related to the fact that the presence of the excess Au inclusions in the Y-123 crystal system damages significantly the pinning mechanism and grain boundary coupling due to the enhancement in the local structural distortions, lattice strains and misorientations of the grains. In other words, the increment of Jc in the YBCO system stems from not only the increase of average grain size and number of flux pinning centers, but also the improvement in the coupling between Au-added superconducting grains.

- As for the powder XRD experimental results, each sample is polycristalline in nature and corresponds to orthorhombic structure of YBCO. The characteristics peak intensities belonging to the Y-123 phase improves considerably with the enhancement of the Au content level in the crystal lattice up to the concentration of x=1 wt.% above which the phase begins to regress immediately due to the increase of the local structural distortions, lattice strains and grain misorientations. In this respect, the Y1 sample with the crystallite size of 68.7 nm exhibits the best crystallinity and interaction coupling between the grains. Similarly, the lattice parameter *c* increases with the addition of x= 1% (Y1 sample) in comparison with the undoped sample (Y0). However, lattice parameters of *a* and *b* remain essentially constant with Au content up to x=0.1% beyond which the parameter enlarges regularly.

- According to the SEM micrographs, the surface morphology is obtained to improve with the Au concentration level up to the x= 1%. Namely, the Y1 sample exhibits the smoothest and densest surface morphology with the best connectivity between the superconducting grains and largest crystalline distribution.

- Furthermore, the deduced magnetic onset temperature, shielding fraction and flux dynamics behaviors of the superconducting ceramics prepared in our research for this thesis show that the overall susceptibility curves belonging to the samples exhibiting the superconducting characteristics shift to slight lower temperature with the enhancement in the AC field amplitudes applied due the their intrinsic nature. Similarly, the imaginary part intensities tend to increase with the enhancement of AC field amplitude level. However, it is fair to conclude that among the samples, the Y0 one presents the maximum strength of the pinning force with its higher susceptibility-temperature curves. To sum up, the concentration level of x= 1% favors the Y-123 superconducting ceramics for usage in the large scale applications.
- The long and short òf it is that this thesis establishing the links between processing, crystallinity and crucial properties of ceramics with respect to the Au concentration level along with the Y-123 superconducting matrix may open up a newly/novel and feasible area for the engineering applications of the Y-123 superconducting materials.

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