INFLUENCE OF Ca DOPING AND ADDITION OFNANO-PARTICLES OF Si AND SiC ON CRITICAL TRANSITION TEMPERATURE OF $YBa_2Cu_3O_7$-8 SUPERCONDUCTOR

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By

WAN NURUL AIN BINTI WAN SHAAIDI

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DEDICATION

To my beloved parents Wan Shaaidi Wan Abdullah and Zaliha Che Idris
for their boundless love and repeated encouragement ..

To my family members
for their wonderful support and concern...

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Currently, intensive research has been carried out to understand the mechanism of superconductivity in $YBa_2Cu_3O_{7-\delta}$ (YBCO) system. However, there is a need to study the influence of Calcium (Ca) on the superconductivity of YBCO. Therefore, it is our aim in this study to investigate the effect of Ca on superconducting transition temperature (T_c) of YBCO by selective doping at Y, Ba and Cu sites, respectively. This study is also focused on the potentiality of the nano-particles of Si and SiC as effective pinning centres in YBCO. Finally the superconductivity of YBCO doped by Ca and nano-particles of Si and SiC was investigated. The polycrystalline samples were prepared via solid state reaction method and they were analyzed by XRD, SEM and four-point electrical measurements.

The XRD results of the Ca doping indicate that all samples can be indexed to Y123 phase with the highest dominant peaks of (103) and (013). The Y211 phase appeared slightly in all site of Ca doping system. SEM micrographs showed larger grain size

giving rise to the reduction of porosity in $(Y_{1-x}Ca_x)Ba_2Cu_3O_{7-\delta}$ and $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$ systems. However, the increasing of Ca concentration doped into Cu site system leads to smaller grain size. It was found that T_c decreased in all the systems. But then it dropped drastically in $YBa_2(Cu_{1-x}Ca_x)_3O_{7-\delta}$ followed by $(Y_{1-x}Ca_x)Ba_2Cu_3O_{7-\delta}$ and finally by $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$.

In order to study the influence of nano-particles on T_c, 0.5, 1.0, 1.5 and 2.0 weight percentages (wt.%) of Si and SiC were added into YBCO samples respectively. XRD showed the dominance of Y123 phase in all the samples. The changes in *a, b* and *c* lattice parameters caused a nonsystematic trend for Si and a reduction in SiC of the orthorhombicity. Meanwhile, the grain size is decreased with the addition of nano-particles. The depression of T_c in nano-Si from 91 K to 77 K is larger than that of nano-SiC which is from 91 K to 80 K attributed to that Si is easier to ionize and become Si^{4+} which may disturbs the overall structure of YBCO.

Finally, the T_c of $(Y_{1-x}Ca_x)Ba_2Cu_3O_{7-\delta}$ reacted with nano-Si was 74 K while that reacted with nano-SiC the T_c was 78 K. Thus, it revealed nano-Si has a stronger effect on T_c suppression than nano-SiC. Besides, lattice defect is increased in the nano-Si case contributing to the larger lattice strain. Therefore, in this research we have discovered that the T_c degradation is determined by several factors which are the ionic radii, charge valency and hole concentration.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains.

PENGARUH PENDOPAN Ca DAN PENAMBAHAN DARI ZARAH NANO Si DAN SiC PADA SUHU PERALIHAN KRITIKAL $YBa_2Cu_3O_{7-\delta}$ SUPERKONDUKTOR

Oleh

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Disember 2012

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Fakulti: Sains

Kini, penyelidikan intensif telah dijalankan untuk memahami mekanisme kesuperkonduksian dalam sistem $YBa_2Cu_3O_{7-\delta}$ (YBCO). Walau bagaimanapun terdapat keperluan untuk mengkaji pengaruh Kalsium (Ca) pada kesuperkonduksian YBCO. Oleh itu, adalah matlamat kami dalam kajian ini untuk mengkaji kesan Ca pada suhu peralihan (T_c) superkonduktor YBCO oleh pendopan terpilih di tapak-tapak Y, Ba dan Cu, masing-masing. Kajian ini juga memberi tumpuan kepada potensi zarah nano Si dan SiC sebagai pusat penyematan berkesan dalam YBCO. Akhirnya kajian kesuperkonduksian YBCO didop Ca dan zarah nano Si dan SiC dilakukan. Sampel polihablur telah disediakan melalui kaedah tindak balas keadaan pepejal dan dianalisis melalui XRD, SEM dan ukuran elektrik empat titik.

Keputusan XRD bagi dopan Ca menunjukkan bahawa semua sampel boleh diindeks kepada fasa Y123 dengan puncak tertinggi dominan (103) dan (013). Fasa Y211 muncul sedikit di tapak semua sistem dopan Ca. Mikrograf SEM menunjukkan saiz

butiran yang lebih besar menimbulkan pengurangan keliangan sistem $(Y_{1-x}Ca_x)Ba_2Cu_3O_{7-\delta}$ dan $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$. Walau bagaimanapun, peningkatan Ca yang didopkan ke dalam sistem tapak Cu membawa kepada saiz butiran yang lebih kecil. Didapati, T_c menurun dalam semua sistem. Namun kemudian ia jatuh secara drastik dalam $YBa_2(Cu_{1-x}Ca_x)_3O_{7-\delta}$ diikuti oleh $(Y_{1-x}Ca_x)Ba_2Cu_3O_{7-\delta}$ dan akhirnya oleh $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$.

Dalam usaha untuk mengkaji pengaruh zarah nano pada T_c, 0.5, 1.0, 1.5 dan 2,0 peratusan berat (wt.%) Si dan SiC telah ditambah ke dalam sampel YBCO masing-masing. XRD menunjukkan dominasi Y123 fasa dalam semua sampel. Perubahan dalam parameter kekisi *a*, *b* dan *c* menyebabkan corak yang tidak sistematik untuk Si dan pengurangan di dalam SiC untuk ortorombik. Sementara itu, saiz butiran menurun dengan penambahan zarah nano. Penurunan T_c dalam Si nano iaitu dari 91 K kepada 77 K adalah lebih besar daripada SiC nano iaitu dari 91 K to 80 K disebabkan oleh Si yang lebih mudah untuk mengion dan menjadi Si^{4+} yang mungkin mengganggu struktur keseluruhan YBCO.

Akhirnya, T_c $(Y_{1-x}Ca_x)Ba_2Cu_3O_{7-\delta}$ yang bertindak balas dengan Si nano ialah 74 K manakala untuk tindak balas dengan SiC nano ialah 78 K. Oleh itu, ia menunjukkan bahawa Si nano mempunyai kesan yang lebih kukuh pada penurunan T_c berbanding dengan SiC nano. Selain itu, kecacatan kekisi meningkat dalam Si nano juga menyumbang kepada tarikan kekisi yang lebih besar. Oleh itu, dalam kajian ini kita telah mendapati bahawa penurunan T_c disebabkan oleh beberapa faktor iaitu jejari ionik, cas valensi dan kewujudan lohong kepekatan.

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LIST OF SYMBOLS AND ABBREVIATIONS

Θ_D	Debye temperature
ρ	resistivity
ξ	coherence length
κ	Ginzburg-Landau parameter
λ	penetration depth of magnetic field
a, b, c	lattice parameter
T_c	superconducting transition temperature
B	magnetic induction
B_c	critical field
B_{c1}	lower critical field
B_{c2}	upper critical field
E	electric field
EDX	Energy Dispersive X-ray spectroscopy
F_L	Lorentz force
HTSC	High Temperature Superconductors
I	current
J	electrical current density
J_c	critical current density
k_B	Boltzmann's constant
MgB_2	Magnesium Diboride
p	hole concentration
R	resistance
SEM	Scanning Electron Microscope
V	potential difference

wt.%	weight percentage
XRD	X-ray powder diffraction
Y211	Y_2BaCuO_5
YBCO	$YBa_2Cu_3O_{7-\delta}$
Δ	energy gap
ΔT	Superconducting transition temperature breadth
dc	direct current

CHAPTER 1

INTRODUCTION

1.1 Early discovery of superconductivity

In 1911, Heike Kamerlingh Onnes discovered that the dc resistivity of mercury suddenly dropped to zero when the sample was cooled below 4.2 K (the boiling point of liquid helium). Since then, the phenomenon was termed as superconductivity. In the following years, many metallic elements were found to show superconducting properties. Superconducting transition temperature T_c, is the temperature at which a superconductor loses its resistance totally (Rose-Innes and Rhoderick, 1978). For example, Nb was discovered to have a T_c of 9.2 K in 1930 (Cyrot and Pavuna, 1992). In 1933, Meissner and Ochsenfeld discovered that magnetic field was expelled from a superconductor that was cooled to below its T_c in the presence of a weak external magnetic field (Ginzburg and Andryushin, 1994). In 1957, the theory of superconductivity was formulated by Bardeen, Cooper and Schrieffer (Bardeen et al., 1957). It was named BCS theory which explained the interaction of the conduction electron gas with elastic wave of crystal lattice giving rise to zero resistance. Under this condition, electrons get close to each other to form Cooper pairs (Mourachkine, 2002).

1.2 Basic characteristics of superconductors

There are two important properties for a material to be conceived as superconductor. They are:

1. No resistivity ($\rho = 0$)

Below T_c, zero dc resistivity will be observed for a superconductor. According to BCS theory, this occurs at low temperature because the Cooper pairs are able to move coherently without any resistance. Cooper pairs are formed due to the interaction of electron and phonon (Owens and Poole, 2002). An example of superconducting transition for $YBa_2Cu_3O_{7-\delta}$ bulk sample is shown below in Figure 1.1.

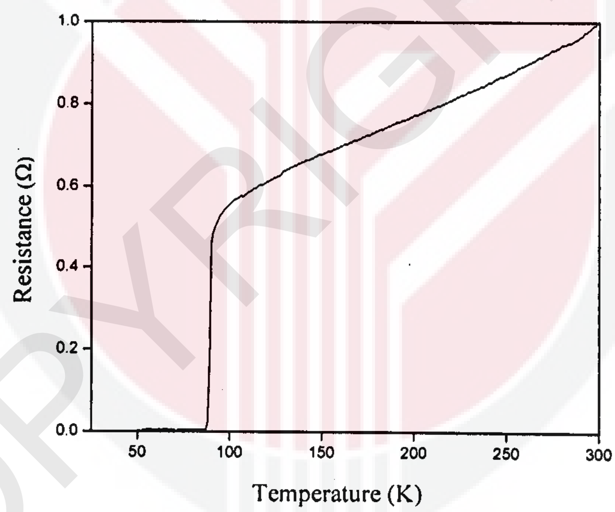

Figure 1.1: Typical transition curve (resistance vs. temperature) for a $YBa_2Cu_3O_{7-\delta}$ bulk sample.

2. No magnetic induction $(B = 0)$

Inside a superconducting material, the magnetic inductance will become zero as it is cooled below T_c or in other words magnetic flux is expelled from the interior of the sample in a presence of weak external magnetic field. This effect is called the Meissner-Ochsenfeld effect. This shows that an applied magnetic field induces a surface current that cancels the applied field within the superconductor so that no magnetic field is presents in its interior (Owens and Poole, 2002).

1.3 High temperature superconductors

The discovery of high temperature superconductors (HTSC for brevity) brings a step closer to the realization of various technological applications with lower cost of cooling. It was first discovered in copper-oxide compound (La-Ba-Cu-O) which superconducts at 35 K by Bednorz and Muller (1986). Impressively, HTSC was found to superconduct at higher temperatures than the non-copper-oxide materials. Basically, HTSC have a perovskite structure with multi-layer of CuO_2 planes as superconductivity occurs between these layers. Earlier studies have shown that the T_c is sensitive to the layers of CuO_2 (Owens and Poole, 2002). In distracted state, oxygen vacancy disorder is correlated to the reduction in T_c, since electrical currents are carried by holes induced in the oxygen sites of the CuO_2 sheets. Broadly speaking, T_c depends on the cation substitutions, chemical elements and oxygen content (Giri et al., 2005). Examples of HTSC are $YBa_2Cu_3O_7$ (YBCO), $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ (BSCCO), $Tl_2Ca_2Ba_2Cu_3O_{10}$ (TLCCO), and $HgBa_2Ca2Cu_3O_{9-\delta}$ (HGCCO) systems (Fossheim and Sudbo, 2005) as in Table 1.

Table 1: Some important HTSC with their superconducting transition temperature, T_c.

HTSC system	Acronym	T_c(K)
$YBa_2Cu_3O_7$	Y123	92
$YBa_2Cu_4O_8$	Y124	80
$Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$	Bi2223	110
$Bi_2Sr_2CaCu_2O_{8-\delta}$	Bi2212	90
$Tl_2Ca_2Ba_2Cu_3O_{10}$	Tl2223	125
$HgBa_2Ca2Cu_3O_{9-\delta}$	Hg1223	153
$HgBa_2Ca_3Cu_4O_{11-\delta}$	Hg1234	134

1.4 Problem statement and research objectives

To date, it has been realized that HTSC have severe current limiting problem at the grain boundary owing to its small coherence length of the order of atomic spacing (Poole et al., 1995). This causes a drastic drop of critical current density, J_c, across the grain boundary even in self field (Ekin et al., 1987). YBCO is one of the HTSCs that shows such a behavior. Although its grain boundary problem is more serious compared to BSCCO, the latter suffers from weak pinning properties which are essential for achieving high J_c. In the past, BSCCO had been well developed as the so called '1st Generation Superconductor'. Now, YBCO becomes a challenge to the scientific community to develop it into '2nd Generation Superconductor'. Due to its isotropic grain nature, it is extremely difficult to align the grains to be in textured form. On the other hand, texturing technique had been successfully developed for BSCCO. Application wise, T_c of YBCO cannot be reduced too much (because of chemical doping or other processing conditions) and must be kept well above 77 K (boiling point of liquid nitrogen). This will eliminates the need for much more expensive refrigeration system involving liquid helium.

It is known that Ca doping increases the critical current greatly (Huhtinen et al., 2007) by enhancing the grain connectivity of YBCO superconductor (Su and Welch, 2005). Recently, Babu et al. (2001) reported significant improvement of superconductivity at the grain boundary of YBCO by doping Ca at Y-site ($Y_{1-x}Ca_xBa_2Cu_3O_y$) to enhance current transport across grain boundary. J_c at 77 K of the samples increases from 0.9×10^4 A/cm^2 to 1.2×10^4 A/cm^2 at 1 T. The T_c was decreased from 89.9 K for pure sample to only 88.1 K for x = 0.05. In order to determine the influence of Ca on the superconductivity of YBCO, it is also important to investigate Ca doping at Ba and Cu site. However, there is no such comparative study so far.

Recently, addition of nano-Si and nano-SiC have been well studied in MgB_2 (Liang et al., 2011). It was observed that the MgB_2 samples reacted with nano-Si and nano-SiC showed an enhancement in J_c compared with the pure sample due to increased flux pinning. However, the T_c is decreased by only 1 K from 37.7 K for the pure sample to 36.7 K for the 10 wt.% nano-Si added sample (Wang et al., 2003). As for nano-SiC reacted sample, the onset T_c is also decreased by 1 K to 36.6 K for the 10 wt.% SiC reacted sample (Dou et al., 2004). This indicates that both of the nano-particle dopants increases J_c and hardly degrades the T_c. So, with the same concept we hope that the pinning effect could be introduced in YBCO.

Hence, the objectives of this work are:

i. To investigate the relative influence of Ca on superconducting transition temperature (T_c) of YBCO by selective doping at Y, Ba and Cu sites, respectively.

ii. To establish the potentiality of the nano-particles of Si and SiC for flux pinning by studying its effect on superconducting transition temperature (T_c) of YBCO.

iii. To study the phase formation and microstructure evolution of YBCO induced by Ca and nano-particles of Si and SiC.

1.5 Scope of present work

In this work, we focus on the characterization of crystal structure, microstructure and superconducting transition temperature (T_c) of YBCO system. This study consists of three parts. First part is on Ca doping at Y, Ba and Cu site of YBCO, respectively. These three series of polycrystalline samples are $(Y_{1-x}Ca_x)Ba_2Cu_3O_{7-\delta}$, $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$ and $YBa_2(Cu_{1-x}Ca_x)_3O_{7-\delta}$ with x = 0.00, 0.01, 0.02, 0.05, and 0.10. Second part is on YBCO reacted with nanoparticles of Silicon (Si) and Silicon Carbide (SiC) for x = 0.5, 1.0, 1.5 and 2.0 weight percentages (wt.%). Third part is $(Y_{0.99}Ca_{0.01})Ba_2Cu_3O_{7-\delta}$ reacted with nano-Si (0.5 wt.%) and $(Y_{0.99}Ca_{0.01})Ba_2Cu_3O_{7-\delta}$ reacted with nano-SiC (0.1 wt.%). The phase composition was determined by X-ray powder diffraction (XRD) technique. The crystal structure parameters were refined by the Rietveld technique using the X'Pert HighScore Plus program. The microstructure was imaged by using Scanning Electron Microscope (SEM) and the elemental analysis was carried out using Energy Dispersive X-ray spectroscopy (EDX). The resistance as a function of temperature was measured using the standard four-probe set-up with a close-cycle helium cryostat from 50 K - 300 K to determine the value of $T_{c\text{-onset}}$ and $T_{c\text{-offset}}$.

1.6 Thesis overview

In Chapter 1, an introduction to superconductivity, motivations and objectives of the research are given. Chapter 2 is on the literature review of the effects of Ca doping and nano-particles additions into YBCO system. Chapter 3 discusses the fundamental properties of superconductivity while Chapter 4 focuses on the description of sample preparation and sample characterization. Chapter 5 presents results and discussion on the data obtained from all the measurements in this study. Finally, conclusions are drawn and suggestions for future work are given in Chapter 6.

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