



**UNIVERSITI PUTRA MALAYSIA**

**SYNTHESIS AND CHARACTERIZATION OF SAMARIUM DOPED  
Bi<sub>1.6</sub>Pb<sub>0.4</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10</sub> SUPERCONDUCTOR PREPARED VIA  
COPRECIPITATION METHOD**

**IMAD MOH'D KHAIR RASHID HAMADNEH**

**FSAS 2002 51**

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**By**

**IMAD MOH'D KHAIR RASHID HAMADNEH**

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,  
in Fulfillment of the Requirement for the Degree of Doctor of Philosophy**

**September 2002**



## **DEDICATIONS**

To my wife, Lama and my son, Yazan,  
for their love, support and understanding....

To my late father, my mother and the family,  
for their love and concern....



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of  
the Requirements for the degree of Doctor of Philosophy

**SYNTHESIS AND CHARACTERIZATION OF SAMARIUM DOPED  
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COPRECIPITATION METHOD**

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**IMAD "MOH'D KHAIR" RASHID HAMADNEH**

**September 2002**

**Chairman: Professor Abdul Halim bin Shaari, Ph.D**

**Faculty : Faculty of Science and Environmental Studies**

The coprecipitation technique was used in the preparation of samarium doped  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  polycrystalline ceramic superconductor. In this study, four sites (calcium, bismuth, strontium and copper) were separately subjected to Sm doping with nominal composition ranging from  $x=0$  to  $x=0.3$  at different sintering times. The superconducting properties of the samples have been investigated. The pure sample, which exhibits  $T_{C(R=0)}$  around 102 – 103 K and  $T_{C\text{-onset}}$  around 108 K, showed large flaky grains of  $\sim 7 \mu\text{m}$  in size which are randomly distributed. However at longer sintering time the pure superconductor showed a better orientation as compared to that of the short sintering time sample. The calculated value of Josephson current  $I_0$ , obtained from the ac susceptibility data showed a much higher value ( $I_0 = 138.7 \mu\text{A}$ ) as compared to the conventional prepared to that of the sample prepared by conventional method



( $I_0 = 55.9 \mu\text{A}$ ). This indicates better grain connectivity and higher 2223 phase content, which was confirmed by SEM photographs. In addition, the nature of the ultra fine particles of the oxalate powders produced by coprecipitation method have increased the diffusion reaction and shortened the heat treatment procedure for the sample preparation, this leads to better superconducting properties as compared to the samples prepared by conventional solid state technique where its diffusion reaction requires high sintering temperatures for long duration and sometimes several grindings.

The resistivity measurements showed the normal metallic behaviour followed by shifts in  $T_{C(R=0)}$  towards lower temperature as the samarium concentration increased due to the decrease in the 2223 phase and an increase in the formation of 2212 phase. However, the material lost its superconductivity at Sm concentration  $x > 0.20$ . When samarium was doped in  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ , and  $\text{Cu}^{2+}$  sites, it probably brings about changes in the hole carrier concentration which in turn alters  $T_{C(R=0)}$ . Hence it could be deduced that the valency of the dopant has some influence on the electron pairing mechanism.

The temperature dependence of ac susceptibility data  $\chi'$  shows the shifting of the onset diamagnetism towards lower temperature as the Sm concentration increased due to the presence of low  $T_c$  phase. The imaginary component,  $\chi''$ , shows a shift in the intergranular coupling peak,  $T_p$ , towards lower temperature as the Sm concentration increased. Hence it can be deduced that the dynamic magnetic response of the samples are not only phase dependent but also dependant on the intergranular coupling. The calculated  $I_0$  which revealed the quality of the coupling of the grains, showed a decrease

in its value as the samarium concentration increased. For highest doping percentage of samarium the values of  $I_0$  decreased in the following order;  
 $I_0(\text{in Ca}) < I_0(\text{in Cu}) < I_0(\text{in Sr}) < I_0(\text{in Bi})$ .

The results of x-ray diffraction (XRD) pattern show that all samples with Sm concentration above  $x=0.02$  contain unknown peaks which correspond to the non-superconducting phase. The intensity of these peaks increases toward higher value, as the Sm concentration increases. The volume of 2223 phase decreased drastically as the Sm concentration increases, whereas the amount of decrease varied due to the doping at different sites. In addition, there is a possibility that either  $\text{Sm}^{2+}$  or  $\text{Sm}^{3+}$  might have occupied other sites at the same sample.

When long sintering time was applied, the improvement in superconducting properties was obvious at low doping concentrations  $x < 0.06$  where the sample was still dominated by 2223 phase. Above that concentration, the grain size decreased and became short and thick, randomly distributed as compared to the pure phase. It is also observed that the superconducting properties and the microstructure improved when the sample was sintered for 48 hours and 100 hours, the high  $T_C$  phase dominates, indicating that the optimum time must be above 48 hours.

Abstrak disertasi yang dikemukakan kepada senat Universiti Putra Malaysia bagi memenuhi keperluan untuk ijazah Doktor Falsafah

**SINTESIS DAN PENCIRIAN SUPERKONDUKTOR  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$   
YANG DIDOPKAN DENGAN SAMARIUM MELALUI KAEDAH  
PEMENDAKAN BERSAMA**

Oleh

**IMAD "MOH'D KHAIR" RASHID HAMADNEH**

**Ogos 2002**

**Pengerusi : Profesor Dr. Abdul Halim Shaari, Ph.D**

**Fakulti : Sains dan Pengajian Alam Sekitar**

Teknik pemendakan bersama telah digunakan bagi menyediakan superkonduktor seramik polihablur  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  yang didopkan dengan samarium. Dalam kajian yang dijalankan ini, empat tapak (kalsium, bismut, strontium dan kuprum) telah didopkan dengan Sm secara berasingan dengan komposisi nominal di antara julat  $x=0$  hingga  $x=0.3$  pada masa pensinteran yang berbeza. Sifat kesuperkonduksian bagi sampel-sampel ini telah dikaji. Di dapati sampel tulen menunjukkan  $T_{C(R=0)}$  di antara 102 –103 K dan  $T_{C_{\text{onset}}}$  adalah sekitar 108 K. Sampel ini menunjukkan butiran besar dan berkeping yang bertaburan secara rawak dengan saiz butiran  $>7 \mu\text{m}$ . Pada masa pensinteran yang tinggi, superkonduktor tulen akan menunjukkan orientasi yang lebih baik berbanding orientasi pada sampel dengan masa persinteran yang lebih pendek.



Nilai perkiraan arus Josephson,  $I_0$ , yang diperolah daripada data kerentanan arus ulang alik, menunjukkan nilai yang lebih tinggi ( $I_0 = 138.7 \mu\text{A}$ ) berbandingkan sampel yang di sediakan secara konvensional ( $I_0 = 55.9 \mu\text{A}$ ). Ini disebabkan oleh penyambungan butir yang lebih baik dan kandungan fasa 2223 yang tinggi dan disahkan oleh gambar foto SEM. Tambahan pula, sifat semula jadi zarah halus serbuk oksalat yang dihasilkan melalui kaedah pemendakan bersama telah meningkatkan kadar tindakbalas resapan dan memendekkan tempoh proses rawatan haba dalam penyediaan sampel. Ini menghasilkan sifat kesuperkonduksian yang lebih baik berbanding dengan sampel yang disediakan menggunakan kaedah lazim iaitu teknik tindakbalas keadaan pepejal, di mana tindakbalas resapannya memerlukan suhu persinteran yang tinggi dan lama serta turut memerlukan beberapa proses pengisaran.

Pengukuran kerintangan menunjukkan sifat logam normal dan diikuti dengan anjakan  $T_{C(R=0)}$  kepada suhu rendah apabila kepekatan samarium meningkat. Ini disebabkan oleh pengurangan pada fasa 2223 dan peningkatan dalam pembentukan fasa 2212. Walau bagaimanapun, bahan ini akan kehilangan sifat kesuperkonduksian pada kepekatan  $x > 0.2$ . Perubahan pada kepekatan pembawa lubang mungkin akan berlaku jika samarium didopkan pada tapak  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$  dan  $\text{Cu}^{2+}$  seterusnya akan mengubah  $T_{C(R=0)}$ . Oleh itu bolehlah disimpulkan bahawa valensi bagi pendopan akan mempengaruhi mekanisme pasangan elektron.

Data-data kerentanan arus ulang-alik,  $\chi'$ , yang bersandarkan suhu menunjukkan berlakunya suatu anjakan pada onset diamagnet akibat suhu yang lebih rendah dan penambahan kepekatan Sm akibat kewujudan fasa  $T_C$  yang rendah. Komponen khayal,  $\chi''$ , menunjukkan anjakan pada puncak gandingan antara butiran  $T_P$  ke suhu yang lebih rendah apabila kepekatan Sm bertambah. Dengan yang demikian, bolehlah disimpulkan bahawa tindakbalas dinamik magnet sampel-sampel bukan hanya bersandarkan fasa tetapi juga bersandar pada gandingan antara butiran. Nilai kiraan  $I_0$ , menunjukkan kualiti gandingan butiran telah menunjukkan pengurangan nilainya apabila kepekatan samarium bertambah. Untuk kepekatan samarium yang paling tinggi, nilai bagi  $I_0$  berkurangan dalam tertib berikut  $I_{0(\text{ dalam Ca})} < I_{0(\text{ dalam Cu})} < I_{0(\text{ dalam Sr})} < I_{0(\text{ dalam Bi})}$ .

Keputusan pembelauan corak sinar-X atau XRD bagi semua sampel dengan kepekatan dopan Sm melebihi  $x=0.02$ , mengandungi puncak-puncak yang tidak diketahui yang berkaitan dengan fasa bukan superkonduktor. Keamatan puncak-puncak berikut didapati meningkat kepada nilai yang lebih tinggi sejajar dengan peningkatan komposisi dopan Sm. Isipadu fasa 2223 berkurang secara mendadak apabila komposisi dopan ditingkatkan, manakala kadar pengurangan berbeza-beza disebabkan oleh pendopanan pada tapak-tapak(site) yang berlainan. Tambahan lagi, terdapat kemungkinan  $\text{Sm}^{2+}$  atau  $\text{Sm}^{3+}$  menduduki oleh tapak-tapak yang lain di dalam sampel yang sama.

Apabila masa pensinteran yang panjang dilakukan, didapati terdapat peningkatan bagi sifat-sifat kesuperkonduksian pada kepekatan dopan yang rendah iaitu pada  $x=0.06$ , di mana sampel masih di dominasi oleh fasa 2223. Pada kepekatan yang lebih tinggi,

saiz butiran akan berkurangan dan bentuknya menjadi pendek dan tebal serta bertaburan secara rawak berbanding fasa tulen. Turut dapat diperhatikan, sifat-sifat kesuperkonduksian dan mikrostrukturnya akan meningkat apabila sampel disinter selama 48 jam dan 100 jam, di mana fasa  $T_C$  yang tinggi akan berlaku dan ini menunjukkan bahawa masa optimum bagi pensinteran perlu dilakukan melebihi 48 jam.

## ACKNOWLEDGEMENTS

### **In the name of Allah, the most Gracious and the most Merciful**

Praise be to Allah the Almighty, for thee (alone) we worship and thee (alone) we ask for help. And praise be upon Mohammad s.a.w who his guidance has led us to the path whom God has favoured.

I am extremely grateful to my supervisor, Professor Dr. Abdul Halim Shaari for most of all, believing in me and for his invaluable advice, patience, guidance, ideas, criticism, encouragement and continuous discussion,

My deepest gratitude goes to my co-supervisors, Professor Dr. Lee Chnoong Kheng and Dr. Zainul Abidin Hassan for the comments, suggestions and wise guidance throughout the research work.

I am very grateful for the financial assistance provided through the Intensified Research Program in Priority Area (IRPA). My special thanks go to Associate Professor Dr. Wan Daud Wan Yusof for his suggestion and support. I am grateful to all the lecturers in the Physics Department for their kind help and discussion.

To Associate Prof. Dr. Fauziah Othman, Mr. Ho, Miss Azilah, Mrs. Faridah and all members of Electron Microscopy Unit, thanks a lot for your kind assistance.



I am extremely grateful to my lab mates; Iftetan Ahmad Taha, Kabashi, Talib, K. P. Lim, Mustafa Dihom, Ali, Zohra, J.Y. Teh, Huda, Mas, Ramadan, Sharmiwati, Jannah, Azman and Abdullah Chik. Thanks a lot for your kind help and understanding regarding this work.

To my friends who never fail to encourage me until the end; Associate Prof. Dr. Ahmad Kamal from UiTM, Dr. Nasri from UNITEN and Isam Qudsiyah, my special thanks go to all of you. I am very thankful to Mr. Razak Harun, Mr. Razi, and other technical staff in the Physics Department for their technical favours. To Mr. Kamal, thanks a lot for your technical assistants.

To my late father, my mother, brothers and sisters, their love and support keep me going, And last but not least, to my wife, Lama and my son, Yazan, thank you for your love, continuous support, encouragement and understanding.

**May GOD Bless You All.**



I certify that an Examination Committee met on 25<sup>th</sup> September 2002 to conduct the final examination of Imad Moh'D Khair Rashid Hamadne on his Doctor of Philosophy thesis entitled "Synthesis and Characterization of Samarium Doped  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  Superconductor Prepared via Coprecipitation Method" in accordance Universiti Pertanian Malaysia ( Higher Degree) act 1980 and Universiti Pertanian Malaysia ( Higher Degree)Regulations 1981. the committee recommends that candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

**Sidek Abdul Aziz, Ph.D.**

Associate Professor  
Faculty of Science and Environmental Studies  
Universiti Putra Malaysia  
(Chairman)

**Abdul Halim Shaari, Ph.D.**

Professor  
Faculty of Science and Environmental Studies  
Universiti Putra Malaysia  
(Member)

**Lee Chnoong Kheng, Ph.D.**

Professor  
Faculty of Science and Environmental Studies  
Universiti Putra Malaysia  
(Member)

**Zainul Abidin Hassan, Ph.D.**

Doctor  
Faculty of Science and Environmental Studies  
Universiti Putra Malaysia  
(Member)

**Roslan Abd. Shukor, Ph.D.**

Faculty of Science and Technology  
Universiti Kebangsaan Malaysia  
(Independent Examiner)



---

**AINI IDERIS, Ph.D.**

Professor/ Dean,  
School of Graduate Studies,  
Universiti Putra Malaysia

Date: **23 OCT 2002**



This thesis submitted to the Senate of Universiti Putra Malaysia and was accepted as fulfillment of the requirements of the degree of Doctor of Philosophy. The members of the Supervisory Committee are as follows:

**Abdul Halim Shaari, Ph.D.**

Professor  
Faculty of Science and Environmental Studies  
Universiti Putra Malaysia  
(Chairman)

**Lee Chnoong Kheng, Ph.D.**

Professor  
Faculty of Science and Environmental Studies  
Universiti Putra Malaysia  
(Member)

**Zainul Abidin Hassan, Ph.D.**

Doctor  
Faculty of Science and Environmental Studies  
Universiti Putra Malaysia  
(Member)

---

**AINI IDERIS, Ph.D.**

Professor/ Dean,  
School of Graduate Studies,  
Universiti Putra Malaysia

Date:



I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

---

**IMAD "MOH'D KHAIR" RASHID HAMADNEH**

**Date:**





## TABLE OF CONTENTS

	Page
DEDICATION	ii
ABSTRACT	iii
ABSTRAK	vi
ACKNOWLEDGEMENTS	x
APPROVAL SHEETS	xii
DECLARATION	xiv
LIST OF TABLES	xvii
LIST OF FIGURES	xix
LIST OF PLATES	xxix
LIST OF ABBREVIATIONS	xxx
 <b>CHAPTER</b>	
1 INTRODUCTION	1
1.1 Basic Properties Of Superconductors	1
1.1.1 Zero Resistance	2
1.1.2 Meissner Effect	3
1.1.3 Energy Gap	5
1.1.4 Electronic Specific Heat	6
1.1.5 Isotope Effect	7
1.2 A Brief History of Superconductors	8
1.3 Research Objectives	12
2 BACKGROUND OF SUPERCONDUCTIVITY	13
2.1 Microscopic Theory of Superconductivity	13
2.2 Copper Oxide Superconductors	20
2.2.1 Effect of Substitution Doping in BSCCO Superconductor	24
2.3 Non- Copper Oxide Superconductors	38
2.4 Preparation Methods of Superconducting Ceramics	39
2.4.1 Conventional Method	40
2.4.2 Sol-Gel Methods	40
2.4.3 Co-precipitation Methods	43
3 MATERIALS AND INSTRUMENTATION	47
3.1 Sample Preparation and Experimental Details	47
3.1.1 $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ samples	50
3.1.2 $\text{Bi}_{1.6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ samples	50
3.1.3 $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{2-x}\text{Sm}_x\text{Ca}_2\text{Cu}_3\text{O}_8$ samples	51
3.1.4 $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Sm}_x\text{O}_8$ samples	51
3.2 Physical Evaluation	52
3.2.1 Resistance at various temperatures	52



3.2.2	Magnetic susceptibility	53
3.2.3	XRD	56
3.2.4	Morphology	57
4	RESULTS AND DISCUSSION	58
4.1	Resistance at Various Temperatures	58
4.1.1	Effect of Sm doping in Ca site of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$	58
4.1.2	Effect of Sm doping in Bi site of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$	66
4.1.3	Effect of Sm doping in Sr site of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$	73
4.1.4	Effect of Sm doping in Cu site of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$	80
4.2	AC Susceptibility Measurements	90
4.1.1	Effect of Sm in Ca site	91
4.1.2	Effect of Sm in Bi site	114
4.1.3	Effect of Sm in Sr site	133
4.1.4	Effect of Sm in Cu site	153
4.3	X-ray Diffraction Analysis	173
4.1.1	Effect of Sm in Ca site	174
4.1.2	Effect of Sm in Bi site	180
4.1.3	Effect of Sm in Sr site	187
4.1.4	Effect of Sm in Cu site	194
4.4	Morphological Studies	200
4.1.1	Effect of Sm in Ca site	200
4.1.2	Effect of Sm in Bi site	202
4.1.3	Effect of Sm in Sr site	204
4.1.4	Effect of Sm in Cu site	206
5	CONCLUSIONS AND FUTURE DIRECTIONS	208
5.1	Conclusion	208
5.2	Future Directions	212
	REFERENCES	213
	APPENDICES	218
A	Thermo Gravimetric Analysis (TGA) for the pure BSCCO	219
B	SEM micrograph for a metal oxalate mixture of the pure BSCCO	220
C	EDAX Spectrum and Semi Quantitative Analysis of pure $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ sintered for 24 hours	221
D	Schematic drawing of the paths of the intragrain current density $J_{Cg}$ and the macroscopic intergrain current density $J_{Cm}$	222
E	$T_{C(R=0)}$ as a function of Sm concentration at various sites	223
F	% 2223 superconducting phase as a function of Sm fraction at various sites	224
G	Papers Published and Presented Throughout this Thesis	225
	BIODATA OF THE AUTHOR	226



## LIST OF TABLES

<b>Table</b>	<b>Page</b>
2.1: High Temperature superconducting compounds	22
2.2: The lattice parameters in BSCCO superconducting system.	24
2.3: The summarized results of the doping elements in Ca and Cu sites	37
4.1: Summarized $T_{C(R=0)}$ (K) and $T_{C-onset}$ (K) for samples doped with Sm in Ca site with various concentrations of Sm and sintering time (24, 48 and 100 hours).	64
4.2: Summarized $T_{C(R=0)}$ (K) and $T_{C-onset}$ (K) for samples doped with Sm in Bi site with various concentrations of Sm and sintering time (24, 48 and 100 hours).	70
4.3: Summarized $T_{C(R=0)}$ (K) and $T_{C-onset}$ (K) for samples doped with Sm in Sr site with various concentrations of Sm and sintering time (24, 48 and 100 hours).	77
4.4: Summarized $T_{C(R=0)}$ (K) and $T_{C-onset}$ (K) for samples doped with Sm in Cu site with various concentrations of Sm and sintering time (24, 48 and 100 hours).	85
4.5: Summarized data of coupling peak temperature, $T_P$ , first onset temperature of diamagnetism, $T_{C-onset}$ , phase lock-in temperature, $T_C$ and josephson current, $I_o$ for $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}Sm_xCu_3O_8$ samples.	111
4.6: Summarized data of coupling peak temperature, $T_P$ , first onset temperature of diamagnetism, $T_{C-onset}$ , phase lock-in temperature, $T_C$ and josephson current, $I_o$ for $Bi_{1.6-x}Sm_xPb_{0.4}Sr_2Ca_2Cu_3O_8$ samples	130
4.7: Summarized data of coupling peak temperature, $T_P$ , first onset temperature of diamagnetism, $T_{C-onset}$ , phase lock-in temperature, $T_C$ and josephson current, $I_o$ for $Bi_{1.6}Pb_{0.4}Sr_{2-x}Sm_xCa_2Cu_3O_8$ samples sintered at 24, 48 and 100 hours.	150



Table 4.8:	Summarized data of coupling peak temperature, $T_p$ , first onset temperature of diamagnetism, $T_{C\text{-onset}}$ , phase lock-in temperature, $T_{Cj}$ and josephson current, $I_0$ for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Sm}_x\text{O}_8$ samples	170
Table 4.9:	Summarized data of the cell parameters and volume of the unit cell for all samples doped with Sm in Ca site with sintering time (24, 48 and 100 hours).	177
Table 4.10:	Summarized data of the cell parameters and volume of the unit cell for all samples doped with Sm in Bi site with sintering time (24, 48 and 100 hours).	183
Table 4.11:	Summarized data of the cell parameters and volume of the unit cell for all samples doped with Sm in Sr site with sintering time (24, 48 and 100 hours).	190
Table 4.12:	Summarized data of the cell parameters and volume of the unit cell for all samples doped with Sm in Cu site with sintering time (24, 48 and 100 hours).	198



## LIST OF FIGURES

Figure	Page
1.2: The typical curve of resistivity vs. temperature for a superconducting material.	3
1.2: Type I superconductor.	4
1.3: The magnetic field within the material (B) vs. the external magnetic field (H).	5
1.4: The vortices (like little solenoids) shield the penetrating magnetic field through the bulk superconductor	5
1.5: Evolution of critical temperature, $T_C$ from 1911 until 2002.	
1.6: Crystallographic structure of $\text{Bi}_2\text{Sr}_2\text{Ca}_n\text{Cu}_{n+1}\text{O}_{2n+6+\delta}$ system with $n = 0, 1, \text{ and } 2$ .	10
3.1: Flow chart of Bi-Sr-Ca-Cu-O doped with Sm prepared by oxalates precursor.	49
3.2: Dc resistivity measurement (four-point probe methods)	52
3.3: Cross-section view of the primary and secondary coils	56
4.1: Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at $850^\circ\text{C}$ for 24 hours with various concentrations of Sm in $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_\delta$ .	59
4.2: Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples annealed in air at $850^\circ\text{C}$ for 48 hours with various concentrations of Sm in $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_\delta$ .	61
4.3: Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at $850^\circ\text{C}$ for 100 hours with various concentrations of Sm in $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_\delta$ .	62
4.4: $T_{C(R=0)}$ as a function of Sm concentration for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_\delta$ samples sintered at 24, 48 and 100 hours.	63



4.5:	Sm concentration dependence of normalized residual resistance for $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ samples sintered at 850°C for different sintering time.	65
4.6:	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 24 hours with various concentrations of Sm in $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ .	67
4.7:	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 48 hours with various concentrations of Sm in $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ .	68
4.8:	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 100 hours with various concentrations of Sm in $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ .	69
4.9:	$T_{C(R=0)}$ as a function of Sm concentration for $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ samples sintered at 24, 48 and 100 hours.	71
4.10:	Sm concentration dependence of normalized residual resistance for $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ samples sintered at 850°C for different sintering time.	72
4.11:	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 24 hours with various concentrations of Sm in $\text{Bi}_{1-6-x}\text{Pb}_{0.4}\text{Sr}_{2-x}\text{Sm}_x\text{Ca}_2\text{Cu}_3\text{O}_8$ .	74
4.12:	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 48 hours with various concentrations of Sm in $\text{Bi}_{1-6-x}\text{Pb}_{0.4}\text{Sr}_{2-x}\text{Sm}_x\text{Ca}_2\text{Cu}_3\text{O}_8$ .	75
4.13:	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 100 hours with various concentrations of Sm in $\text{Bi}_{1-6-x}\text{Pb}_{0.4}\text{Sr}_{2-x}\text{Sm}_x\text{Ca}_2\text{Cu}_3\text{O}_8$ .	76
4.14:	$T_{C(R=0)}$ as a function of Sm concentration for $\text{Bi}_{1-6-x}\text{Pb}_{0.4}\text{Sr}_{2-x}\text{Sm}_x\text{Ca}_2\text{Cu}_3\text{O}_8$ samples sintered at different sintering time.	78
4.15:	Sm concentration dependence of normalized residual resistance for $\text{Bi}_{1-6-x}\text{Pb}_{0.4}\text{Sr}_{2-x}\text{Sm}_x\text{Ca}_2\text{Cu}_3\text{O}_8$ samples sintered at 850°C for different sintering time.	79
4.16:	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 24 hours with various concentrations of Sm in $\text{Bi}_{1-6-x}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Sm}_x\text{O}_8$ .	81

4.17	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 48hours with various concentrations of Sm in $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Sm}_x\text{O}_8$ .	82
4.18	Normalized resistance ( $R/R_{(T=300K)}$ ) as a function of temperature for samples sintered in air at 850 °C for 100 hours with various concentrations of Sm in $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Sm}_x\text{O}_8$ .	84
4.19:	$T_{C(R=0)}$ as a function of Sm concentration for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Sm}_x\text{O}_8$ samples sintered at different sintering time	86
4.20:	Sm concentration dependence of normalized residual resistance for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Sm}_x\text{O}_8$ samples sintered at 850°C for different sintering time.	87
.4.21:	AC susceptibility of pure $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 24 hours.	90
.4.22:	AC susceptibility of pure $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 48 hours.	90
4.23:	AC susceptibility of pure $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 100 hours.	91
4.24:	Applied field as a function of the coupling peak temperature for pure $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ samples sintered at various temperatures.	91
4.25:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sintered for 24 hours sample ( $x=0.02$ )	94
4.26:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sintered for 24 hours sample ( $x=0.06$ )	94
4.27:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sintered for 24 hours sample ( $x=0.10$ )	95
4.28:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sintered for 24 hours sample ( $x=0.20$ )	95
4.29:	AC susceptibility as a function of temperature for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sintered for 24 hours with applied magnetic field 0.1 Oe. (a) Real part, (b) Imaginary part	96
4.30:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sample ( $x=0.02$ ) sintered at 850 C for 48 hour.	99

4.31:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sample ( $x=0.06$ ) sintered at 850 C for 48 hour.	99
4.32:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sample ( $x=0.10$ ) sintered at 850 C for 48 hour.	100
4.33:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sample ( $x=0.20$ ) sintered at 850 C for 48 hour.	100
4.34:	AC susceptibility as a function of temperature for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sintered for 48 hours with applied magnetic field 0.1 Oe. (a) Real part, (b) Imaginary part	101
4.35:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sample ( $x=0.02$ ) sintered at 850 C for 100 hour.	104
4.36:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sample ( $x=0.06$ ) sintered at 850 C for 100 hour.	104
4.37:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sample ( $x=0.10$ ) sintered at 850 C for 100 hour.	105
4.38:	AC susceptibility of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sample ( $x=0.20$ ) sintered at 850 C for 100 hour.	105
4.39:	AC susceptibility as a function of temperature for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ sintered for 100 hours with applied magnetic field 0.1 Oe. (a) Real part, (b) Imaginary part	106
4.40:	Applied field as a function of the coupling peak temperature for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ samples sintered for 24 hours	108
4.41:	Applied field as a function of the coupling peak temperature for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ samples sintered for 48 hours	109
4.42:	Applied field as a function of the coupling peak temperature for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ samples sintered for 100 hours	109
4.43:	Josephson current vs. Sm concentration temperature for $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{2-x}\text{Sm}_x\text{Cu}_3\text{O}_8$ samples.	110
4.44:	AC susceptibility of $\text{Bi}_{1.6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 24 hours sample ( $x=0.02$ )	113





4.45:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 24 hours sample ( $x=0.06$ )	113
4.46:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 24 hours sample ( $x=0.10$ )	114
4.47:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 24 hours sample ( $x=0.20$ )	114
4.48:	AC susceptibility as a function of temperature for $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 24 hours and applied magnetic field 0.1 Oe. (a) Real part, (b) Imaginary part	115
4.49:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 48 hours sample ( $x=0.02$ )	118
4.50:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 48 hours sample ( $x=0.06$ )	118
4.51:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 48 hours sample ( $x=0.10$ )	119
4.52:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 48 hours sample ( $x=0.20$ )	119
4.53:	AC susceptibility as a function of temperature for $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 48 hours and applied magnetic field 0.1 Oe. (a) Real part, (b) Imaginary part	120
4.54:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 100 hours. sample( $x=0.02$ )	123
4.55:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 100 hours sample ( $x=0.06$ )	123
4.56:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 100 hours sample ( $x=0.10$ )	124
4.57:	AC susceptibility of $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 100 hours sample ( $x=0.20$ )	124
4.58:	AC susceptibility as a function of temperature for $\text{Bi}_{1-6-x}\text{Sm}_x\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_8$ sintered for 100 hours and applied magnetic field 0.1 Oe. (a) Real part, (b) Imaginary part	125

