UNIVERSITI PUTRA MALAYSIA

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By

SALEH AYED AHMAD AL-KHAWALDEH

Thesis Submitted in Fulfilment of the Requirements for the Degree of Master of Science in the Faculty of Science and Environmental Studies
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بِسۡمِ ٱللَّهِ ٱلرَّحۡمَٰنِ ٱلرَّحِيمِ ١٥ يَٰبُنَيَّ إِنَّهَآ إِن تَكُ مِثۡقَالَ
حَبَّةٖ مِّنۡ خَرۡدَلٖ فَتَكُن فِي صَخۡرَةٍ أَوۡ فِي ٱلسَّمَٰوَٰتِ أَوۡ فِي ٱلۡأَرۡضِ يَأۡتِ
بِهَا ٱللَّهُۚ إِنَّ ٱللَّهَ لَطِيفٌ خَبِيرٞ ١٦

سورة لقمان

إلى من تطيب النفس بلقائهم بعد عناء الغربة ،،،

إلى أبي، مثلي الاعلى في العطاء والتضحية ،،،

إلى أُمي، خلجة فؤادي وقرة عيني التي لا تنام ،،،

إلى إخوتي و أخواتي الذين بحبهم تهون الهموم و اليهم أكون ،،،

إلى الاهل والعشيره ... الفخر والانتماء ...

اليكم جميعاً أشد الإعتذار ...

و بإسمكم اقدم هذا العمل المتواضع ...

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Amin

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ABBREVIATIONS AND KEY WORDS

T_c	Critical temperature
B_c , B_{c1}, B_{c2}	Critical magnetic field
BSCCO	Bi-Sr-Ca-Cu-O system
GL theory	Ginzburg-Landau theory
BCS theory	Bardeen, Cooper and Schrieffer theory
LBCO	La-Ba-Cu-O system
YBCO	Y-Ba-Cu-O system
Y123	Family member in $YBa_2Cu_3O_{7-x}$.
Bi(2201)	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n}$, n =0
Bi(2212)	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n}$, n =1
Bi(2223)	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n}$, n =2
TBCCO	Tl-Ba-Ca-Cu-O system
T(2223)	Family member in $Tl_2Ba_2Ca_nCu_{n+1}O_{6+2n}$, n =2
HBCCO	Hg-Ba-Ca-Cu-O system
TGA	Thermogravimetric Analysis
DTA	Differential Thermal Analysis
Calcination	Heating process where the solid state reaction occur
Sintering	Heating process yielding for more compacting of the sample grains and improve its properties

Acetate Precursor	Metal acetate dissolved in suitable solvent
Citrate Precursor	Metal citrate dissolved in suitable solvent
Oxide technique	Solid state reaction method
G_s	Gibbs free energy per unit volume
G_n	Free -energy density of the normal state
H	Applied magnetic field
M	Magnetization
μ_o	Permeability of free space
ξ	Coherence length
λ_L	Penetration depth
ε_F	Fermi energy
θ_D	Debye temperature
v_F	Fermi velocity
k	Boltzman constant
h	Blanck constant
N_s	Superelectron density
$N(\varepsilon)$	Density of state
Δ_0	The zero-temperature energy gap
C_s	Specific heat
λ	Electron- phonon coupling constant
$D_{ph}(\omega)$	Phonon density of the state

$\alpha^2(\omega)$	The electron -phonon coupling strength
μ^*	Coulombic repulsion
a, b, c	Lattice parameters
J_c	Critical current density
MRI	Magnetic Resonance Image
SQUID	Superconducting Quantum Interference Device
$MO(R)_m$	Metals alkoxides
$M(OH)_m$	Metal hydroxides
χ	Susceptibility
AC	Alternating Current
V	Induced voltage
ζ	Filling factor
V	Sample volume
α	Calibration coefficient
f, ω	Frequency and angular frequency
XRD	X-ray diffraction
$\mathbf{d}_{hkl}$	Reciprocal *d* vector
hkl	Miller indices
SEM	Scanning Electron Microscope
IR	Infrared
$\bar{\mathrm{v}}$	Wave number

Abstract of thesis presented to the Senate of University Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

SUPERCONDUCTING PROPERTIES OF SOL-GEL DERIVED Bi (Pb)-Sr-Ca-Cu-O SYSTEM

By

SALEH AYED AHMAD AL-KHAWALDEH

May 1998

Chairman : Assoc. Prof. Abdul Halim Shaari, Ph.D.

Faculty : Science and Environmental Studies

The $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_y$ (2223) superconductor, has been prepared by sol-gel technique, using metal acetate precursors. Room temperature hydrolysis followed by polycondensation and heating yielded a transparent blue gel. The key to successful gel formation was due to a firm control of the pH of the solution. It was required to maintain the pH at 5.5 throughout the sol to gelation process. The decomposition of the polyhydroxyl metal complex to amorphous gel was found to be completed at temperatures in the range of 200 to 250°C producing submicron size particles. The amorphous gel transformed into crystalline powder at 600° C to 700° C.

The effect of heat treatment as a function of sintering time and temperature have been also studied on $Bi_{1.5}Pb_{0.5}Sr_2Ca_2Cu_3O_{10+x}$ system. The 2223 phase was observed in samples sintered at 845° C for 48 h.

The effect of the Pb doping at the bismuth lattice site have been studied in the $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_y$ (x= 0.0, 0.4, 0.5) samples. Two samples doped with 20 and 25 mole % of Pb in the bismuth site yielded single phase sample with T_c (R=0) above 102 K. Undoped sample had mixed phases (2212 and 2223). The study shows that the doped-sample with 25 mole % of Pb is the most suitable ratio that gives rise to the best superconducting properties.

New procedure adopted in this work succeeded in getting good quality gel from carbonates, oxides and nitrates as the starting materials. The study shows that high-purity single-phase superconductor with T_c (R=0) at 104 K was obtained from carbonate and oxide as starting materials. This value is comparable to that obtained from corresponding metal acetates.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia bagi memenuhi keperluan untuk ijazah Master Sains

SIFAT-SIFAT KESUPERKONDUKSIAN DISEDIAKAN MELALUI TEKNIK SOL-GEL SISTEM Bi (Pb)-Sr-Ca-Cu-O

Oleh

SALEH AYED AHMAD AL-KHAWALDEH

Mei 1998

Pengerusi : Prof Madya Abdul Halim Shaari, Ph.D.

Fakulti : Sains dan Pengajian Alam Sekitar

Superkonduktor $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_y$ (2223) telah disediakan melalui teknik sol-gel dengan menggunakan petunjuk logam asetat. Hidrolisis pada suhu bilik diikuti dengan polikondensasi dan pemanasan menghasilkan gel biru lutsinar. Kunci kejayaan pembentukan gel adalah pengawalan terhadap pH larutan. Ia diperlukan untuk mengekalkan pH pada 5.5 sepanjang proses penukaran sol ke gel. Penguraian polihidroksil logam komplek kepada gel amorfus didapati sempurna pada suhu antara julat 200° C hingga 250° C dan menghasilkan partikel bersaiz submikron. Gel amorfus bertukar kepada serbuk kristal pada suhu diantara 600° C hingga 700° C.