UNIVERSITI PUTRA MALAYSIA

EFFECT OF STANUM DOPING AND HEAT TREATMENT ON Bi-Sr-Ca-Cu-O SUPERCONDUCTING CERAMICS

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By

AZHAN BIN HASHIM @ ISMAIL

Thesis Submitted in Fulfilment of the Requirements for
the Degree of Doctor of Philosophy in the
Faculty of Science and Environmental Studies
Universiti Putra Malaysia

December 1999

DEDICATIONS

To Assoc Prof Dr Halim,
for his patience, guidance and belief in me

To mak, ayah and family,
for their love and concern

To my wife, Aiza and my children,
for their love, support and understanding

Abstract of thesis presented to the senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Doctor of Philosophy

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

Faculty : Science and Environmental Studies

The influence of Sn doping in calcium (Ca) and copper (Cu) sites either separately or simultaneously on Bi-Sr-Ca-Cu-O system [$Bi_{1.6}Sr_2(Ca_{1-x}Sn_x)_2Cu_3O_\delta$ ($0.00 \leq x \leq 0.20$), $Bi_{1.6}Sr_2Ca_2(Cu_{1-x}Sn_x)_3O_\delta$ ($0.00 \leq x \leq 0.30$) and $Bi_{1.6}Sr_2(Ca_{1-x}Sn_x)_2(Cu_{1-x}Sn_x)_3O_\delta$ ($0.00 \leq x \leq 0.20$)] and the role of heat treatment at various temperature for 30 hours soaking time were studied For Sn-free sample, heat treatment improved the superconducting transition temperature, $T_{C(R=0)}$ from 100 K to 104 K, when the sintered sample was annealed at 820 °C The highest $T_{C(R=0)}$ of 104 K was observed for sintered sample (x=0.02) with simultaneous doping The heat treatment improved the $T_{C(R=0)}$ from 60 K to 94 K for sample doped in Ca site when re-sintered at 855 °C and from 66 K to 100 K for sample doped with Sn in Cu site when annealed at 840 °C For simultaneous doping, the $T_{C(R=0)}$ did not change significantly after heat treatment except for sample with x=0.05 that showed drastic improvement of $T_{C(R=0)}$ from 64 K (sintered) to above 100 K

From XRD analysis, the volume of 2223 phase decreased as the Sn concentration increased All samples with Sn concentration above x=0 10 contained unknown peaks which correspond to the non-superconducting phase Although the lattice parameter of the samples shortened, the crystallographic structure remained in the tetragonal form

All samples indicated the onset of diamagnetism temperature at around 110 K showing the presence of 2223 phase The volume of diamagnetic shielding decreased as the Sn concentration increased It was also observed that the heat treatment process improved the volume of diamagnetic shielding in Sn-free and Sn-doped samples especially for concentrations of x=0 02 and x=0 05 Interestingly the shielded volume for Sn-doped samples in Cu site with concentration of x=0 20 and x=0 25 increased as the heat treatment temperature increased The coupling peak, T_P shifted to lower temperature as the Sn concentration increased due to the enhancement of weak links The nature of the weak links due to the Josephson junction in each doping mode based on the Sn concentrations was different For Sn-free and separately doped (x=0 02) samples the junctions were S-N-S type, whereas for higher concentration of Sn, the weak links was dominated by the S-I-S type For the simultaneously doped S-N-S junction seemed to dominate in samples with $0 \leqslant x \leqslant 0\ 10$ Samples with x=0 05 and x=0 10 showed the transformation from S-I-S type to S-N-S type in this doping mode as compared to the other doping mode

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

KESAN PENDOPAN STANUM DAN RAWATAN HABA KE ATAS SERAMIK SUPERKONDUKTOR Bi-Sr-Ca-Cu-O

Oleh

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Pengerusi : Profesor Madya Abdul Halim Shaari, Ph.D

Fakulti : Sains dan Pengajian Alam Sekitar

Kesan pendopan Sn pada tapak kalsium dan kupram samada secara berasingan atau serentak terhadap sistem Bi-Sr-Ca-Cu-O [$Bi_{1.6}Sr_2(Ca_{1-x}Sn_x)_2Cu_3O_\delta$ ($0.00 \leq x \leq 0.20$), $Bi_{1.6}Sr_2Ca_2(Cu_{1-x}Sn_x)_3O_\delta$ ($0.00 \leq x \leq 0.30$) dan $Bi_{1.6}Sr_2(Ca_{1-x}Sn_x)_2(Cu_{1-x}Sn_x)_3O_\delta$ ($0.00 \leq x \leq 0.20$)] dan peranan rawatan haba pada pelbagai suhu dengan tempoh masa 30 jam telah dikaji. Untuk sampel tulin, rawatan haba telah meningkatkan suhu peralihan superkonduktor, $T_{C(R=0)}$ dari 100 K kepada 104 K apabila sampel yang di sinter telah di sepuhlindap pada suhu 820 ^oC. $T_{C(R=0)}$ tertinggi iaitu 104 K telah dicerap untuk sampel x=0.02 yang disinter dan didopkan secara serentak. Rawatan haba telah meningkatkan $T_{C(R=0)}$ dari 60 K kepada 94 K untuk sampel yang didopkan pada tapak Ca apabila di sinter semula pada suhu 855 ^oC dan dari 66 K kepada 100 K untuk sampel yang didopkan pada tapak Cu apabila di sepuhlindap pada suhu 840 ^oC. Untuk pendopan secara serentak, $T_{C(R=0)}$ tidak berubah secara nyata selepas rawatan haba kecuali bagi sampel x=0.05 yang menunjukkan peningkatan drastik $T_{C(R=0)}$ dari 64 K (sinter) kepada lebih 100 K.

Dari analisis XRD, isipadu fasa 2223 berkurang apabila kepekatan Sn meningkat. Semua sampel dengan kepekatan Sn melebihi x=0.10 mengandungi puncak-puncak yang tidak di ketahui yang mana merujuk kepada fasa bukan superkonduktor. Walaupun parameter kekisi-kekisi sampel dipendekkan. struktur hablur kekal dalam bentuk tetragonal.

Semua sampel memperlihatkan suhu peralihan diamagnet di sekitar 110 K yang menunjukkan kehadiran fasa 2223. Isipadu pemerisaian diamagnetic berkurang apabila kepekatan Sn bertambah. Diperhatikan juga rawatan haba telah meningkatkan isipadu pemerisaian diamagnet bagi sampel tulen dan sampel yang didopkan dengan Sn terutama sekali pada kepekatan x=0.02 dan x=0.05. Yang menariknya, isipadu pemerisaian diamagnet untuk sampel yang didopkan dengan Sn pada tapak Cu dengan kepekatan x=0.20 dan x=0.25, didapati meningkat apabila suhu rawatan haba bertambah. Suhu puncak pepasangan, T_P telah dianjakkan ke suhu yang lebih rendah apabila kepekatan Sn bertambah merujuk kepada peningkatan ikatan lemah. Sifat ikatan lemah merujuk kepada simpang Josephson bagi setiap mod pendopan berdasarkan kepekatan Sn adalah berbeza. Untuk sampel tulin dan sampel didopkan secara berasingan (x=0.02), simpangan adalah jenis S-N-S, sebaliknya untuk kepekatan Sn yang lebih tinggi, ikatan lemah didominasi oleh jenis S-I-S. Untuk pendopan secara serentak, simpang S-N-S mendominasi sampel dengan $0 \leqslant x \leqslant 0.10$. Sampel x=0.05 dan x=0.10 menunjukkan transformasi dari jenis S-I-S kepada jenis S-N-S pada mod pendopan ini apabila dibandingkan dengan mod pendopan lain.

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

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I certify that an Examination Committee has met on 29 December, 1999 to conduct the final examination of Azhan bin Hashim @ Ismail on his Doctor of Philosophy thesis entitled "Effect of Stanum Doping and Heat Treatment on Bi-Si-Ca-Cu-O Superconducting Ceramics" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981 The Committee recommends that the candidate be awarded the relevant degree Members of the Examination Committee are as follows

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