THE EFFECT OF CADMIUM DOPING ON BSCCO SUPERCONDUCTORS PREPARED VIA COPRECIPITATION METHOD

By

ALI AGAIL HAMED ABDULGADER

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

April 2004

DEDICATIONS

To my wife, and my daughters, Fatema and Dwaa
for their love, support and understanding….

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

ABSTRACT OF THESIS PRESENTED TO THE SENATE OF UNIVERSITI PUTRA MALAYSIA IN FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF SCIENCE

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

Faculty: **Science And Environmental Studies**

The coprecipitation technique was used in the preparation of cadmium doped $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ and $Bi_{1.7}Pb_{0.3}Sr_2Ca_2Cu_3O_{10}$ polycrystalline ceramic superconductors. In this study, the calcium site was subjected to Cd doping with nominal composition ranging from x=0 to x=0.1 at different sintering times. The superconducting properties of the samples have been investigated. The undoped samples, which exhibits $T_{C(R=0)}$ around 102 –104 K and $T_{C\text{-onset}}$ around 112-116 K, showed large flaky grains around 9-11 μm in size which are randomly distributed. However, at longer sintering time (100 h) the undoped sample showed a better orientation as compared to that of the shorter sintering time (24 hr) sample. The calculated value of Josephson current, I_o, obtained from the AC susceptibility data showed a much higher value (I_o =100.04 μA) as compared to the sample prepared by conventional method (I_o =55.9 μA). 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 decreased in $T_{C(R=0)}$ as the cadmium concentration increased due to the decrease in the 2223 phase and an increase in the formation of 2212 phase.

The temperature dependence of ac susceptibility data, χ', shows the shifting of the onset diamagnetism towards lower temperature as the Cd concentration increased due to the presence of low-T_C phase. The imaginary component, χ'', shows a shift in the intergranular coupling peak, T_p, towards lower temperature as the Cd 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 cadmium concentration increased.

The results of x-ray diffraction (XRD) patterns show that all samples doped with Cd contain 2212 peaks which correspond to the low-superconducting phase. The intensity of these peaks increases towards higher value, as the Cd concentration increases. The volume of 2223 phase decreases gradually as the Cd concentration increases. In

addition, there is a possibility that either Cd^{2+} might have occupied other sites in the sample. When long sintering time was applied, the improvement in superconducting properties was obvious at low doping concentrations x=0.02 where the sample was still dominated by 2223 phase. Above that concentration, the grain size decreased and became shorter and thicker, randomly distributed as compared to the undoped samples. 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.

The study shows that the substitution of cadmium in calcium site does not improve the T_C of the BSCCO system. This is due to the formation of low-T_C phase which weakened the coupling of the grains. However, all samples showed an obvious improvement in T_C as the sintering time increases.

ABSTRAK TESIS YANG DIKEMUKAKAN KEPADA SENAT UNIVERSITI PUTRA MALAYSIA
SEBAGAI MEMENUHI KEPERLUAN UNTUK IJAZAH MASTER SAINS

KESAN PENDOPAN KADMIUM KE ATAS SUPERKONDUKTOR BSCCO MELALUI KAEDAH PEMENDAKAN BERSAMA

Oleh

ALI AGAIL HAMED ABDULGADER

April 2004

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

Fakulti: **Sains Dan Pengajian Alam Sekitar**

Teknik pemendakan bersama telah digunakan bagi menyediakan superkonduktor seramik polihablur $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ dan $Bi_{1.7}Pb_{0.3}Sr_2Ca_2Cu_3O_{10}$ yang didopkan dengan cadmium. Dalam kajian yang dijalankan ini, tapak kalsium telah didopkan dengan Cd secara berasingan yang berbeza dengan komposisi nominal di antara julat x=0 hingga x=0.1 pada masa pensinteran yang berbeza. Sifat kesuperkonduksian bagi sampel-sampel ini telah dikaji. Sampel-sampel tulen yang menunjukkan $T_{C(R=0)}$ di sekitar 102-104 K dan $T_{C\text{-onset}}$ di sekitar 112-116 K, menunjukkan butiran yang berkeping-keping yang bersaiz 9-11 μm dan tertabur secara rawak. Walau bagaimanapun, pada suhu persinteran yang panjang (100 j), didapati superkonduktor tulin menunjukkan orientasi yang lebih baik dibandingkan dengan sampel yang disinter pada jangkamasa yang lebih pendek (24 j). Nilai arus Josephson I_0, yang diperolehi daripada data AC menunjukkan nilai yang lebih tinggi (I_0= 100.04 μA) jika dibandingkan dengan sampel yang dihasilkan melalui kaedah lazim (I_0=55.9 μA). Ini

menunjukkan sifat sempadan butiran yang baik dan kandungan fasa 2223 yang tinggi dan ini disahkan dengan fotograf SEM. Tambahan pula, partikel yang bersaiz tersangat halus bagi serbuk oksalat yang dihasilkan melalui kaedah pemendakan bersama membantu proses resapan dan memendekkan proses pemanasan dalam penyediaan sampel, menghasilkan sifat superkonduktor yang lebih baik jika dibandingkan dengan sampel yang dihasilkan melalui kaedah tindakbalas pepejal di mana ia memerlukan masa persinteran yang lebih panjang dengan beberapa kali proses pengisaran.

Pengukuran kerintangan menunjukkan sifat logam yang lazim dan diikuti dengan penurunan dalam $T_{C(R=0)}$ apabila peratus pendopan kadmium meningkat disebabkan penurunan dalam fasa 2223 dan peningkatan fasa 2212.

Kebergantungan suhu oleh data ACS, χ',menunjukkan penganjakan permulaan diamagnet pada suhu rendah yang lebih rendah apabila kepekatan Cd meningkat disebabkan oleh kemunculan fasa rendah T_C. Komponen khayalan, χ'', menunjukkan anjakan dalam puncak gandingan antara butiran T_p, pada suhu rendah yang lebih rendah apabila kepekatan Cd meningkat. Maka, ini boleh disimpulkan bahawa kesan magnet dinamik sampel bukan hanya yang dikira kebergantungan pada fasa tetapi ia juga bergantung kepada gandingan antara butiran. Nilai I_0 menunjukkan kualiti butiran gandingan dan nilai ini menurun apabila kepekatan cadmium bertambah.

Keputusan corak belauan XRD menunjukkan semua sampel yang didopkan dengan Cd, mengandungi puncak-puncak fasa 2212 dan ini menunjukkan kehadiran fasa rendah. Keamatan puncak-puncak bertambah apabila kepekatan Cd bertambah. Isipadu fasa

2223 menurun berperingkat-peringkat mengikut peningkatan kepekatan Cd. Tambahan pula, ada kemungkinan ion Cd^{2+} menghuni tapak kekisi lain pada sampel.

Apabila masa persinteran dilanjutkan, sifat superkonduktor semakin jelas apabila didopkan dengan kepekatan yang rendah, x = 0.02, di mana sampel ini masih didominasi oleh fasa 2223. Apabila kepekatan melebihi daripada 0.02, saiz butiran semakin mengecil, menjadi lebih pendek dan tebal, dengan taburan rawak jika dibandingkan dengan sampel. Ia juga menunjukkan bahawa sifat-sifat superkonduktor dan mikrostruktur semakin baik apabila sampel disinter selama 48 jam dan 100 jam, dengan fasa tinggi paling dominant dan membuktikan bahawa masa optimum persinteran mestilah lebih daripada 48 jam.

Kajian ini menunjukkan bahawa penggantian Cd pada tapak Ca tidak meningkatkan suhu genting, Tc, bagi system BSCCO. Ini adalah disebabkan oleh pembentukkan fasa rendah yang melemahkan gandingan butiran. Walaubagaimana pun, semua sampel jelas menunjukkan penigkatan dalam Tc apabila masa persinteran meningkat.

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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.

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May GOD Bless You All.

I certify that an Examination Committee met on 15/4/2004 to conduct the final examination of Ali Agail Hamed Abdulgader on his Master of Science thesis entitled "Effect of Cadmium Doping on BSCCO 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:

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This thesis submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirements of the degree of Master of Science. The members of the Supervisory Committee are as follows:

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DECLARATION

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.

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ALI AGAIL HAMED ABDULGADER

Date:

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