UNIVERSITI PUTRA MALAYSIA

SYNTHESIS OF $RBa_2Cu_3O_{7-\delta}$ (R = Gd, Ho, Sm) CERAMIC SUPERCONDUCTOR VIA COPRECIPITATION METHOD AND EFFECTS OF HEAT TREATMENT

AHMAD MUSTAZA BIN AHMAD RUSLI

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By

AHMAD MUSTAZA BIN AHMAD RUSLI

Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for Master of Science

2010

To Ahmad Rusli bin Mohd Sedik, Siti Zaiton binti Sayuti and my lovely sisters and brother.

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

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Chairman: Imad (Khair Moh'D) Rashid Hamadneh, PhD

Institute: Institut Teknologi Maju, ITMA

Preparation of R-123 using conventional solid state method requires high purity oxides and carbonate powders as starting materials, multiple grindings and extended heat treatment to achieve a complete reaction via solid state diffusion. However, high sintering temperature (> 940 °C) and long heating duration (48 - 100 hours) would cause the composition of compound to change, and consequently lowers the quality of the samples. Co-precipitation method (COP) has the capability to overcome these problems because the initial mixtures of cations in the solution are in atomic scale. This enhances

the reaction during the heat treatment and the resulting starting powders are more homogenous with grain size in the nanometer range, higher purity than the powders produced by the solid state method at shorter thermal and processing time.

COP method was used to prepare nanosize metal oxalate (> 50 nm) of R-123 superconductors and the oxalate powders were analyzed via Thermogravimetric Analysis (TGA). Five major drops due to the loss of weight from the bulk sample in the formation of R-123 were observed. Base on the TGA results and previous studies, the calcinations was carried out at 900 – 960 °C. The calcined samples were pelletized and sintered under oxygen atmosphere for 15 hours at 920 ºC, 930 ºC, 940 ºC and 950 ºC. Larger grains (~ 20 μm) which were highly compacted and randomly distributed were observed for the resulting sintered samples by Scanning Electron Microscopy (SEM).

The Ho-123 and Sm-123 samples which were preheated and calcined at 900 ºC, followed by sintering at 920 ºC gave single-phased compounds as confirmed by the X-Ray Diffraction (XRD). However, secondary phase of R-211 can be observed for Gd-123 samples that were treated with the same heat treatment.

In resistivity measurement critical current, I_C and transition temperature, T_C for R-123 samples were observed above 200 mA and 90 K. However, Sm-123 showed decreasing I_C when the sintering temperature was increased and zero transition temperature, $T_{C(R=0)}$ for Sm-123 and Ho-123 sintered at 950 ºC were below 90 K.

Abstrak tesis ysng dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

SINTESIS SERAMIK SUPERKONDUKTOR $RBa_2Cu_3O_{7-\delta}$ (R=Gd, Ho, Sm) MENGGUNAKAN KAEDAH KO-PEMENDAKAN DAN KESAN RAWATAN HABA

Oleh

AHMAD MUSTAZA BIN AHMAD RUSLI

2010

Pengerusi: Imad (Khair Moh'D) Rashid Hamadneh, PhD

Fakulti: Institut Teknologi Maju, ITMA

Sintesis R-123 secara konvensional melalui kaedah keadaan pepejal memerlukan ketulenan oksida yang tinggi dan serbuk karbonat sebagai bahan pemula, pengisaran berulang dan masa yang panjang bagi rawatan haba untuk mencapai satu tindak balas lengkap melalui resapan fasa pepejal. Bagaimanapun, suhu pensinteran yang tinggi (> 940 °C) dan tempoh pemanasan yang lama (48 - 100 jam) boleh mengakibatkan perubahan komposisi sebatian, dan seterusnya merendahkan kualiti superkonduktor.

Kaedah ko-pemendakan (COP) mempunyai keupayaan mengatasi permasalahan ini kerana campuran awal kation dalam larutan berlaku pada skala atom. Ini meningkatkan tindak balas semasa tempoh rawatan haba dan serbuk yang terhasil lebih homogen dengan saiz butiran dalam julat nanometer, ketulenan serbuk yang lebih tinggi berbanding kaedah keadaan pepejal disamping masa proses pemanasan yang lebih pendek.

Dalam kajian ini kaedah COP digunakan untuk menyediakan superkonduktor R-123 bersaiz nano oksalat logam (> 50 nm) dan serbuk oksalat dianalisis melalui Thermogravimetric Analysis (TGA). Lima perubahan utama diperhatikan berpunca daripada kehilangan berat sampel pukal dalam pembentukan R-123. Berasaskan kepada keputusan TGA dan kajian terdahulu, pengkalsinan telah dijalankan pada 900 – 960 °C. Sampel yang dikalsin dipalet dan disinter di dalam oksigen selama 15 jam pada suhu 920 ºC, 930 ºC, 940 ºC dan 950 ºC. Butiran besar (~ 20 μm) yang amat padat dan rawak dihasilkan selepas sampel yang disinter melalui Scanning Electron Microscopy.

Ho-123 dan Sm-123 yang telah melalui pra-pemanasan dan pengkalsinan pada suhu 900 ºC, diikuti dengan pengsinteran pada suhu 920 C memberikan sebatian fasa tunggal seperti yang disahkan oleh X-Ray Diffraction. Walaubagaimanapun, fasa ketaktulenan sekunder, R-211 terbentuk apabila sampel Gd-123 dilakukan proses pemanasan yang sama.

Semasa pengukuran kerintangan arus genting, I_C dan suhu genting, T_C bagi setiap sampel yang telah disinter didapati melebihi 200 mA dan 90 K. Walaubagaiamanapun, Sm-123 menunjukkan penurunan I_C apabila suhu pengsinteran sampel bertambah dan rintangan sifar pada suhu genting, $T_{C(R=0)}$ bagi Ho-123 dan Sm-123 yang disinter pada suhu 950 °C berada dibawah 90 K.

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

Imad (Moh'D) Khair Rashid Hamadneh, PhD
Associate Professor
Faculty of Science
University of Jordan.
(Chairman)

Zulkarnain Zainal, PhD
Professor
Faculty of Science
Universiti Putra Malaysia.
(Member)

HASANAH MOHD GHAZALI, PhD
Professor and Dean,
School of Graduate Studies,
Universiti Putra Malaysia.

Date:

I certify that an Examination Committee has met on 09 December 2010 to conduct the final examination of Ahmad Mustaza bin Ahmad Rusli on his Master of Science thesis entitled Synthesis and Effect of Different Heat Treatment of $RBa_2Cu_3O_{7-\delta}$ (R=Gd, Ho, Sm) Ceramics Superconductor via Coprecipitation Method in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

Members of the Examination Committee are as follows:

Mansor Hashim, Ph.D.
Associate Professor,
Faculty of Science,
Universiti Putra Malaysia.
(Chairman)

Abd Halim Shaari, Ph.D
Professor,
Faculty of Science,
Universiti Putra Malaysia.
(Internal Examiner)

Lim Kean Pah, Ph.D
Faculty of Science,
Universiti Putra Malaysia.
(Internal Examiner)

Ahmad Kamal Yahya, Ph.D
Associate Professor,
School of Physic and Material Study,
Faculty of Applied Science,
Universiti Teknologi Mara,
Malaysia
(External Examiner)

SHAMSUDDIN SULAIMAN, Ph.D
Professor and Deputy Dean,
School of Graduate Studies,
Universiti Putra Malaysia.

Date:

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

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AHMAD MUSTAZA BIN AHMAD RUSLI

Date: 9 December 2010

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