



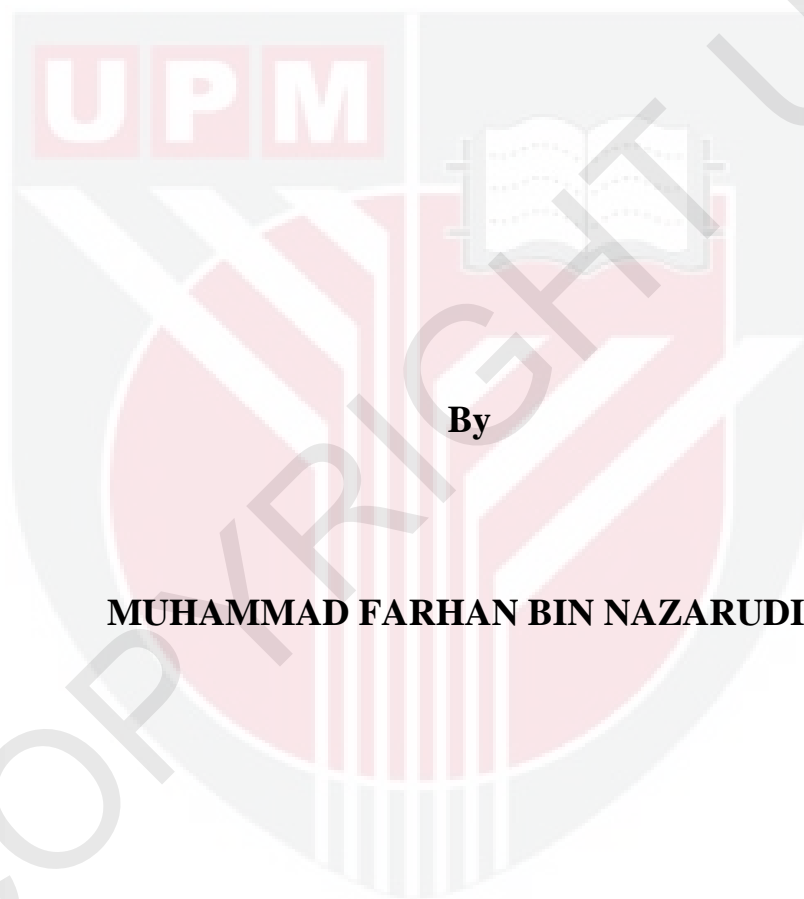
UNIVERSITI PUTRA MALAYSIA

**ELECTROCHEMICAL CHARACTERISTIC OF $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$
SUPERCONDUCTOR SYNTHESIZED VIA CO-PRECIPITATION AND
SOLID-STATE REACTION**

MUHAMMAD FARHAN BIN NAZARUDIN

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By

MUHAMMAD FARHAN BIN NAZARUDIN

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

DECEMBER 2011

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

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December 2011

Chairman : Professor Zulkarnain bin Zainal, PhD

Faculty : Science

High temperature superconductors (HTSC) of microcrystalline ceramic material $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (ErBCO) have been successfully synthesized via co-precipitation (COP) using metal acetate precursors, and conventional solid state method (SS). Comparisons were made on the superconducting properties and electrochemical behavior of ErBCO from both methods. Resistivity versus temperature measurements ($R-T$) showed that all samples exhibited very good metallic behavior. For the COP samples, the sintering temperature was varied from 920-950 °C with the best zero critical current, $T_{C(R=0)} = 91.4$ K obtained for the sample sintered at 920°C. The $T_{C(R=0)} = 90.9$ K was obtained for

the SS sample sintered at the same temperature. X-ray diffraction (XRD) data showed single phase samples of an orthorhombic structure. The formation of non superconducting phases (impurities) was observed when the COP samples were sintered above 920°C. Scanning electron micrographs (SEM) showed grains of size ≤ 40 nm were randomly distributed for all highly compacted samples. However, sample prepared via COP exhibited smaller pores as compared with SS sample. The electrochemical behaviour of the samples had been studied by the cyclic voltammetry (CV), chronoamperometry (CA) and chronocoulometry (CC) techniques. The samples were mechanically attached to glassy carbon (GC) electrode and immersed in NH_4Cl electrolyte solution. NH_4Cl electrolyte was chosen because it has a ligand bearing group with strong complexing ability with copper ion. Multiple cycling tests were carried out to observe the reproducibility and stability of the superconducting surface against the electrolyte. Four characteristics peaks were detected in cyclic voltammogram of ErBCO. There were two sets of peaks observed which could be attributed to the $\text{Cu}^{2+}/\text{Cu}^{1+}$ and $\text{Cu}^{1+}/\text{Cu}^0$ redox couples. This showed that the copper planes in the superconductor played a major role in the surface electrochemical processes. The cyclic voltammetric behavior exhibited from both samples was found to be affected by concentration, temperature and pH of the electrolyte. From CA and CC studies, diffusion coefficient, D was $3.4516 \times 10^{-3} \text{cm}^2/\text{s}$ for COP and $2.3677 \times 10^{-4} \text{cm}^2/\text{s}$ for SS respectively while the amount of charge, Q adsorbed on the electrode surface was $5.4413 \mu\text{C}/\text{cm}^2$ for COP and $4.7383 \mu\text{C}/\text{cm}^2$ for SS, respectively.

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

**PENCIRIAN ELEKTROKIMIA SUPERKONDUKTOR $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$ YANG
DISINTESIS MELALUI TEKNIK KO-PEMENDAKAN DAN KEADAAN
PEPEJAL**

Oleh

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Superkonduktor suhu tinggi, $\text{ErBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (ErBCO) daripada bahan seramik mikrokrystal telah disintesis melalui teknik pemendakan bersama (COP) menggunakan prekursor logam asetat dan tindak balas keadaan pepejal (SS). Perbandingan sifat superkonduktor dan sifat elektrokimia ke atas sampel ErBCO dari kedua dua teknik penyediaan telah dilakukan. Pengukuran kerintangan melawan suhu (R-T) menunjukkan semua sampel mempamerkan sifat logam yang baik. Bagi sampel COP, ia disinter pada suhu pemanasan di antara 920-950 °C dengan takat suhu genting terbaik, $T_{C(R=0)} = 91.4$ K telah diperolehi untuk sampel yang disinter pada suhu 920°C. $T_{C(R=0)} = 90.9$ K telah

diperolehi bagi sampel SS, disinter pada suhu yang sama. Corak pembelauan sinar-X (XRD) menunjukkan pembentukannya adalah fasa tunggal orthorhombik. Pembentukan fasa bukan superkonduktor telah dilihat apabila sampel COP disinter pada suhu 920°C dan ke atas. Mikroskop imbasan elektron (SEM) mempamerkan butiran bersaiz ≤ 40 nm yang padat dan tersebar sekata untuk semua sampel. Bagaimanapun, sampel COP mempunyai saiz liang yang lebih kecil antara butiran berbanding sampel SS. Sifat elektrokimia sampel telah dikaji melalui teknik siklik voltametri (CV), kronoamperometri (CA), dan kronokoulometri (CC). Sampel dilekatkan secara mekanikal pada permukaan elektrod karbon berkaca dan direndamkan di dalam larutan elektrolit 0.1 M NH_4Cl . Elektrolit NH_4Cl dipilih kerana mempunyai kumpulan ligan yang berkemampuan kuat untuk membentuk kompleks dengan ion kuprum. CV dijalankan berulang kali untuk mengkaji keboleholangan dan kestabilan permukaan superkonduktor terhadap elektrolit. Empat puncak ciri telah dikesan di dalam siklik voltammogram bagi ErBCO. Terdapat dua set puncak redoks dikesan dan puncak-puncak tersebut boleh dikaitkan dengan pasangan redoks $\text{Cu}^{2+}/\text{Cu}^{1+}$ dan $\text{Cu}^{1+}/\text{Cu}^0$. Ini menunjukkan bahawa lapisan kuprum di dalam superkonduktor memainkan peranan utama dalam proses elektrokimia permukaan. Sifat siklik voltametri yang ditunjukkan oleh sampel daripada kedua-dua kaedah penyediaan adalah sangat bergantung kepada kepekatan, suhu dan pH elektrolit. Kajian CA dan CC menunjukkan pekali pembauran, D ialah $3.4 \times 10^{-6} \text{ cm}^2/\text{s}$ bagi COP dan $2.3 \times 10^{-6} \text{ cm}^2/\text{s}$ untuk SS manakala jumlah cas elektrik, Q yang terjerap pada permukaan elektrod adalah $5.4 \mu\text{C}/\text{cm}^2$ untuk COP dan $4.7 \mu\text{C}/\text{cm}^2$ bagi SS.

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I certify that a Thesis Examination Committee has met on 6 December 2011 to conduct the final examination of Muhammad Farhan bin Nazarudin on his thesis entitled “Electrochemical Characteristic of $\text{ErBa}_2\text{Cu}_3\text{O}_{7.8}$ Superconductor Synthesized via Co-precipitation and Solid State Reaction” 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.

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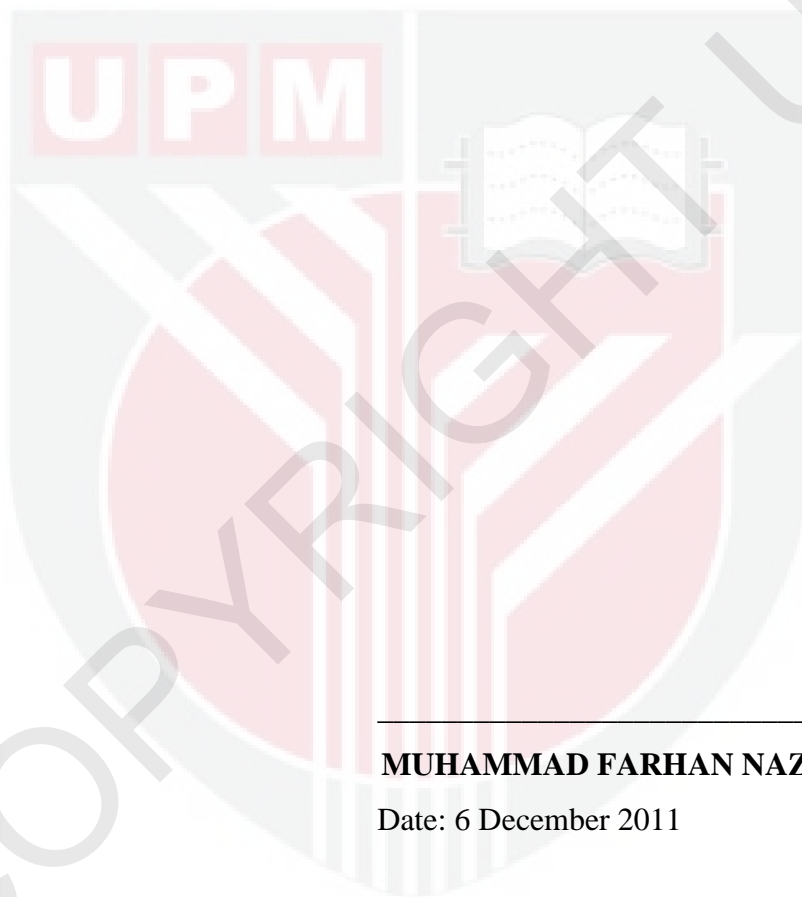
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DECLARATION

I declare that this 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.



MUHAMMAD FARHAN NAZARUDIN

Date: 6 December 2011

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