## **UNIVERSITI TEKNOLOGI MARA**

# **COPRECIPITATION AND SOLID-STATE** SYNTHESIS OF TI1212 SUPERCONDUCTORS AND FABRICATION OF DIP-COATED TAPES

### FAIZAH MD. SALLEH

Thesis submitted in fulfilment of the requirements for the degree of Doctor of Philosophy

**Faculty of Applied Sciences** 

September 2007

#### ABSTRACT

This thesis describes the synthesis of T11212 superconductor from various starting compositions via coprecipitation and solid-state methods and fabrication of Tl1212 superconductor tapes. dip-coated Bulk Tl1212 was synthesized from Tl<sub>0.8</sub>Bi<sub>0.2</sub>Sr<sub>2</sub>Ca<sub>0.8</sub>Y<sub>0.2</sub>Cu<sub>2</sub>O<sub>7</sub> nominal starting composition using coprecipitation method with Tl-containing (CP1 sample) and Tl-free (CP3 sample) precursors. The Tl-containing precursor route involves calcination of Tl-containing precursor powder at 600 °C and sintering of the pellets at various temperatures and durations. The best superconducting sample with 1212 phase of 83 vol.% and  $T_{c zero}$  of 94 K was sintered at 870°C for 60 minutes. However, the sample consists of minor TI1201 phase and SrCO<sub>3</sub> impurity. Addition of nano-MgO particles showed enhanced 1212-phase formation and total elimination of the SrCO<sub>3</sub> impurity. The transport  $J_c$  at 40 K in zero magnetic field for the sample with 0.15 wt.% nano-MgO additions was ten times the  $J_c$  of the sample without MgO and  $J_c$  measurements in external magnetic fields (0- 0.9 T) showed improved magnetic flux pinning property. To reduce Tl loss during sintering of the coprecipitated sample, Tl-free precursor route using two-step synthesis method was developed and was used to synthesize Tl1212 using the same composition as above. The TI-free precursor was first calcined at 850 °C followed by additions of appropriate amounts of Tl<sub>2</sub>O<sub>3</sub> and Bi<sub>2</sub>O<sub>3</sub> before sintering at 1000 °C for 6 minutes. This resulted in a higher purity sample with  $T_{c zero}$  of 91 K,  $J_c$  at 40 K of 11.2 A/cm<sup>2</sup> and ultrafine grains with average size of  $0.5 - 1 \mu m$ . For comparison purposes, bulk T11212 was also synthesized using Tl<sub>0.5</sub>Pb<sub>0.5</sub>Sr<sub>1.8</sub>Yb<sub>0.2</sub>CaCu<sub>2</sub>O<sub>7</sub> (SS1 sample) and Tl<sub>0.8</sub>Bi<sub>0.2</sub>Sr<sub>2</sub>Ca<sub>0.8</sub>Y<sub>0.2</sub>Cu<sub>2</sub>O<sub>7</sub> (SS2 sample) nominal starting compositions using the conventional solid-state method. The results show that both SS1 and SS2 samples have  $T_{c \ zero}$  of 94 K and are comparable to the  $T_{c \ zero}$  of the coprecipitated samples. For fabrication of dip-coated Tl1212/Ag tapes, superconducting powders of Tl<sub>0.8</sub>Bi<sub>0.2</sub>Sr<sub>2</sub>Ca<sub>0.8</sub>Y<sub>0.2</sub>Cu<sub>2</sub>O<sub>7</sub> starting composition synthesized from the coprecipitated Tl-containing precursor route and superconducting powders of SS1 and SS2 were utilized. The effects of thermo-mechanical treatments on transport  $J_c$  of the tapes in zero magnetic field were investigated. The results showed that  $J_c$  of the fabricated tapes were very much dependent on the tapes thermo-mechanical history.  $J_{\rm c}$ enhancement was observed for tapes annealed at temperatures around 870 °C in combination with controlled mechanical rolling of 40% - 50% reduction in tapes thickness. The highest transport  $J_c$  (6,538 A/cm<sup>2</sup>) at 40 K in zero field was recorded for the tape using coprecipitated Tl<sub>0.8</sub>Bi<sub>0.2</sub>Sr<sub>2</sub>Ca<sub>0.8</sub>Y<sub>0.2</sub>Cu<sub>2</sub>O<sub>7</sub> powder.

### ACKNOWLEDGEMENTS

I take this opportunity to thank all the people who have helped me and who continue to inspire me.

My advisor, Associate Professor Dr. Ahmad Kamal Yahya, is the most influential person in my quest for PhD. He has guided me through the academic challenges and supported me in every aspect of my research and professional endeavours. I thank him for his insight, guidance and caring support, and for so much I have learned from him. I shall always look up to him for inspiration.

I am truly grateful to Professor Dr. Abdul Halim Shaari, from Universiti Putra Malaysia who has shown an avid interest in my work and guided me with his constructive comments.

I would like to thank Dr. Muhammad Hafiz Jumali from Universiti Kebangsaan Malaysia for assistance on X-ray diffraction (XRD) analyses that is crucial in this study.

I am also indebted to both, Dr. Imad Hamadneh from Universiti Putra Malaysia and Dr. Zaiki Awang from Microwave Technology Center, Universiti Teknologi Mara for their help on scanning electron microscope micrographs analyses in the research. To both of them, I accord my sincere gratitude.

Sincere appreciation is extended to members of the UiTM Superconductor Group for their invaluable help and encouragements, thank you everybody!

I would also like to thank the Malaysian Ministry of Science, Technology and Innovation (under its IRPA grant No.09-02-01-0007-EA007) for providing the financial support for this research and Universiti Teknologi Mara for the academic sponsorship.

Finally, I would like to give my special thanks to my family for their unconditional love and for always believing in me.

My greatest and ultimate debt and gratitude is due to Allah, the Most Beneficent and the Most Merciful. May He pardon and forgive our weaknesses and endow us with knowledge and wisdom.

ili

### TABLE OF CONTENTS

Page

ABSTRACT	ii
ACKNOWLEDGEMENTS	iil
TABLE OF CONTENTS	iv
LIST OF TABLES	viii
LIST OF FIGURES	x
LIST OF PLATE	xviii
LIST OF SYMBOLS AND ABBREVIATIONS	xxii

CHAPTER 1	INTRODUCTION	1
1,1	Synthesis of Ceramic Superconductor	3
1.2	Fabrication of HTSC Superconductor Tapes	4
1.3	Objectives of Study	8
1.4	Significance of Study	9
CHAPTER 2	BACKGROUND ON SUPERCONDUCTIVITY, SYNTHESIS METHODS AND FABRICATION OF HTSC TAPES	10
2.1	The History of Superconductivity	10
2.2	Properties of Superconductors	11
	2.2.1 Critical Temperature $(T_c)$	11
	2.2.2 The Meissner Effect	12
	2.2.3 Critical Magnetic Field	13
	2.2.4 Critical Current Density $(J_c)$	14
	2.2.5 Relation between $J_c$ , $T_c$ and $H_c$	15

17

#### **CHAPTER 1**

#### INTRODUCTION

The phenomenon of superconductivity has always been very exciting and has been explored both for fundamental and scientific interest and for possible technical applications. Superconductivity refers to a complete loss of electrical resistance to dc current resulting for example in potentially large energy savings for power applications and increased performances for communication electronics. In addition to the property of zero resistance, a superconductor can expel applied magnetic fields so that the field is always zero everywhere inside. The most interesting consequence of this behavior is the ability to levitate permanent magnet over a superconducting surface.

Superconductivity was discovered by Heike Kammerlingh-Onnes in 1911. He was studying the behaviour of metals at the temperature of liquid helium and observed that the resistance of mercury dropped to zero (below 4.2 K). Many other elements, compounds and alloys were later found to be superconductors. Most of the materials have a very low transition temperature to the superconducting state called critical temperature  $(T_c)$  and therefore, cooling to the temperature of liquid helium is usually required. For long, such a low transition temperature restricted the number of potential commercial applications of superconductors. A significant advancement was made in 1986 when G. Bednorz and A. Müller reported that a certain copperoxide-based ceramic material i.e. LaSrCuO with a perovskite structure showed a  $T_c$ of 35 K, which is considered high temperature superconductivity at the time [Bednorz and Müller 1986]. A substitution of yttrium for lanthanum led to the discovery of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>6+ $\delta$ </sub> ( $\delta$ <1) with a T<sub>c</sub> in the range 90-95 K and is often referred to as YBCO or Y123 because of the ratio of Y:Ba:Cu in this material [Wu et al. 1987]. This discovery was very significant because now it became possible to use liquid nitrogen as a coolant. This finding leads to discoveries of other perovskite structured materials that show superconductivity at temperatures above the 77 K