A study of the fabrication and characterisation of high temperature superconductor YBa2Cu3O7 thin films

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A STUDY OF THE FABRICATION AND CHARACTERISATION OF HIGH TEMPERATURE SUPERCONDUCTOR YBa$_2$Cu$_3$O$_7$ THIN FILMS

A thesis submitted in fulfilment of the requirements for the award of the degree

DOCTOR OF PHILOSOPHY

from

UNIVERSITY OF WOLLONGONG

by

AIHUA LI, M. Sc.

The Institute for Superconducting and Electronic Materials, Faculty of Engineering

March 2006
DECLARATION

I, Aihua Li, declare that this thesis submitted in partial fulfilment of the requirements for the award of Doctor of Philosophy, at the Institute for Superconducting and Electronic Materials, the Faculty of Engineering, University of Wollongong, is wholly my own work unless otherwise referenced or acknowledged. The document has not been submitted for qualifications at any other academic institution.

Aihua Li

31 March 2006
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ABSTRACT

Thin films of the high temperature superconductor (HTS) YBa$_2$Cu$_3$O$_7$ (Y123) are of great potential in a wide range of applications, including low-loss microwave cavities and filters, bolometers, various superconducting terminal devices, flux transformers, and dc and rf superconducting quantum interference devices (SQUIDs). They also have the potential to give insight into the fundamental mechanisms governing high temperature superconductivity. Y123 coated conductors, the so-called second generation superconducting tapes, which are based on Y123 thin film technology, also have great applications in carrying large superconducting currents. However, some important challenges or fundamental problems hindering their practical applications need to be solved scientifically. These include: finding the most effective and economic approaches to enhance the superconducting critical current density in high magnetic fields; reducing the fabrication cost with high reproducibility; arriving at a profound understanding of the relationship between microstructure and superconducting performance, etc. The work described in this thesis deals with these challenges, with an emphasis on fundamental studies on supercurrent enhancement through nanotechnology and nanoengineering using advanced thin film fabrication methods, including pulsed laser deposition and spin coating, and on the relationship between microstructure and supercurrent performance.

An overview of the research on HTS thin films in the period from 2000 to the end of 2005 is given in Chapter I. Attention was paid to both Y123 films and coated conductors fabricated using both PLD and chemical methods, on both single crystal and metallic substrates. The related electrical and magnetic properties are also reviewed.
In the first part of the thesis work, Y123 thin films with and without Ag nanoparticle inclusions were grown by pulsed laser deposition (PLD) on YSZ (100), SrTiO$_3$ (100), and LaAlO$_3$ (100) single crystal substrates. A discontinuous layer of Ag nanodots was deposited on the substrates prior to the deposition of Y123 films. The Y123 films grown on such surfaces modified with Ag nanodots were extensively characterised by atomic force microscopy (AFM), X-ray diffraction (XRD), scanning electron microscopy (SEM), AC susceptibility and DC magnetisation. The effects of the density of Ag nanodots, which was controlled by the numbers of PLD shots, on the microstructures and resultant critical current density $J_c$ have been studied systematically. Results showed that under fixed physical deposition conditions $J_c$ increased monotonically with the number of Ag shots, $n$, for films grown on both STO and LAO substrates. At 77 K, the $J_c$ increased from $10^6$ to $3.2 \times 10^6$ A/cm$^2$ for LAO and from $8 \times 10^5$ to $3.5 \times 10^6$ A/cm$^2$ for STO as $n$ increased from 0 to 150. At 5 K, the $J_c$ was enhanced by approximately four times at both low and high fields. These values obtained due to nano-Ag inclusions are comparable to the best results achieved by other prestigious research groups around the world. However, for films grown on YSZ substrate, $J_c$ increased from $2 \times 10^5$ to $2 \times 10^6$ A/cm$^2$ as Ag shots increased from 0 to 30, and decreased to $9 \times 10^5$ for $n \geq 60$. Detailed microstructure investigations indicated that the crystallinity and $ab$ alignment gradually improved as the number of Ag-nano-dots increased.

In the second part of the thesis work, YBa$_2$Cu$_3$O$_y$ films were grown on single crystalline YSZ, SrTiO$_3$, and MgO, and on polycrystalline Ag substrates using non-fluorine sol-gel and spin coating methods. The effects of heat treatment conditions on the phase
evolution and microstructures were investigated using optical microscopy, X-ray
diffraction, and atomic force microscopy. A detailed study was performed on the phase
formation, degree of grain orientation, formation of cracks, and surface morphologies. It
was found that sintering temperature and roughness of substrate surfaces are two key
factors in controlling crack morphologies. With several important advantages, including
precursor solution stability, improved film density, and elimination of HF during
processing, high-quality YBCO films have been achieved on single crystal substrates
with transport critical current densities up to \(10^6\) A/cm\(^2\). An extensive study was carried
out on the alteration of precursor solution stoichiometry and its effects on
superconducting properties. Fluorine-free sol-gel–derived films on the LAO substrate
exhibited epitaxial growth with excellent in- and out-of-plane texture. Experimental
details are given on the sol-gel synthesis chemistry and XRD and TEM characterization
of the YBCO thin films.

The phase evolution of YBCO films prepared by the fluorine-free sol-gel method was
also systematically investigated using in-situ high temperature optical microscope
observations and X-ray diffraction. The conversion sequences and the final resultant
products have been determined for barium, copper and yttrium containing precursors,
respectively. It was found that those conversions are strongly dependent on the
experimental conditions such as water partial pressure. The formation of YBCO starts at
a temperature of around 710 °C and lasts up to 800 °C over about 15 min. Depending on
the barium containing phases and experimental conditions. It is suggested that a- and c-
axis YBCO grains are governed by different reactions.
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