PREPARATION AND CHARACTERIZATION OF SUPERCONDUCTING THIN FILMS

By

NUR HELMI @ NUR JANNAH BINTI AZMAN

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
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Faculty: Science and Environmental Studies

The future application of high temperature superconductor (HTSs) in cryoelectronic devices, which are capable of operating at temperatures typically between 20 K and 77 K will strongly depend on the development of a reproducible deposition technology of high-quality single and multilayer HTS thin films. In this work, $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ and $YBa_2Cu_3O_x$ thin films have been fabricated by pulsed laser deposition onto MgO single crystal substrate with the (100) orientation. Nine series of Bi(Pb)SrCaCuO thin films with different deposition times (thickness) were annealed at different annealing temperature (850ºC, 860ºC, 870ºC, and 880ºC) under oxygen flow. One series of YBCO thin films were annealed at different duration time under oxygen flow. By varying the deposition parameters, films showing different kinds of particulates and surface morphology were fabricated. The most prominent types of particulates for BSCCO films on MgO substrate are droplets with various shapes and sizes. The droplets are randomly oriented and submicron rod-like features are also observed. Needles, platelets, irregularly-shaped Cu-rich outgrowths, tabular outgrowths and big target fragments are also detected. Droplets that have been observed might be due to the crystallization effect

on the hot substrate. The target morphology, which develops under laser-irradiation, has also been investigated. Morphological changes for the target take the form of periodic structures such as ripples, ridges, and cones. Both x-ray diffraction and resistive measurements indicate that the BSCCO superconducting phase does not form for the as-deposited films since the peak related to the formation of crystalline phases were not observed. Most films showed slight decrease in resistance at 110-120 K after heat-treatment. Zero resistances were registered in the range of 60-68 K. This range is strongly dependent on the annealed temperature. The AFM, SEM, EDX, XRD patterns and T_c measurements indicate that the films were mainly dominated by the 2212 phase, with rather poor evidence for the 2223 phase. The attempt to obtain high T_c superconducting film from a superconducting target of 2223 phase gave poor results probably due to the non optimized heating conditions. Results obtained from characterization of the films, showed that annealing time and temperature, deposition times (thickness) influence the growth of superconducting phases. Since the exposed surface area-to-volume ratio is higher for a film than a bulk sample, this would explain why lead losses are apparently more dramatic for films than for a bulk material. The AFM three dimension image shows the island-growth mode of thin films. This mode may be correlated to the existence of clusters in the plasma. Annealing treatments have shown to be an effective method to change the oxygen in the BSCCO system and vary its superconducting properties. Although the superconducting properties of the films described are not as good as those prepared using an excimer laser, they can be improved further by optimizing various deposition parameters.

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

PENYEDIAAN DAN PENCIRIAN BAGI SUPERKONDUKTOR FILEM NIPIS

Oleh

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Aplikasi masa depan bagi superkonduktor suhu tinggi (SST) dalam peranti krioelektronik yang berkebolehan untuk beroperasi pada suhu di antara 20 K dan 77 K masing-masing sangat bergantung kepada perkembangan teknologi pengendapan untuk filem tunggal dan multi-lapisan superkonduktor suhu tinggi. Dalam kajian ini, filem nipis $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ dan $YBa_2Cu_3O_x$ telah dihasilkan melalui kaedah pengendapan laser ke atas substrat MgO hablur tunggal yang berorientasi (100). Sembilan siri filem nipis Bi(Pb)SrCaCuO dengan masa endapan yang berbeza (ketebalan berbeza) telah disepuhlindap pada suhu yang berlainan (850ºC, 860ºC, 870ºC, 880ºC) dalam aliran oksigen. Satu siri filem nipis YBCO pula disepuhlindap dalam aliran oksigen pada jangkamasa yang berlainan. Dengan mempelbagaikan parameter endapan, filem menunjukkan pelbagai jenis butiran dan morfologi permukaan terhasil. Bagi filem BSCCO di atas substrat MgO, butiran yang paling menonjol adalah titisan dengan pelbagai bentuk dan saiz. Titisan terbentuk secara rawak dan struktur submikron dan bak-rod juga telah dicerap. Jejarum, kepingan, butiran besar tidak malar yang kaya dengan Cu, butiran besar dan serpihan sasaran yang besar juga terhasil. Titisan yang

terhasil kemungkinan disebabkan oleh kesan penghabluran ke atas substrat yang panas. Morfologi sasaran yang terbentuk di bawah sinaran laser juga telah dikaji. Perubahan morfologi sasaran bertukar dengan pembentukan struktur berkala seperti jurang, rabung dan juga kon. Keputusan pembelauan sinar-x dan pengukuran rintangan menunjukkan fasa superkonduktor BSCCO tidak terbentuk untuk filem baru mendap kerana puncak berhubung dengan fasa penghabluran tidak kelihatan. Kebanyakan filem menunjukkan sedikit penurunan rintangan pada 110-120 K selepas rawatan haba pada suhu tinggi dilakukan. Kerintangan sifar direkodkan antara 60-68 K. Had ini sangat bergantung kepada suhu pemanasan dalam aliran udara. Keputusan AFM, SEM, EDX, XRD dan pengukuran rintangan menunjukkan filem secara utamanya didominasi oleh fasa 2212, dengan bukti yang tidak jelas wujudnya fasa 2223. Percubaan untuk mendapatkan filem superkonduktor suhu tinggi daripada bahan superkonduktor fasa 2223 memberi keputusan yang tidak kukuh, kemungkinan disebabkan oleh keaadaan pemanasan yang tidak optimum. Keputusan yang didapati melalui pencirian ke atas filem menunjukkan masa sepuhlindap dengan aliran oksigen dan suhu serta ketebalan filem mempengaruhi pertumbuhan fasa superkonduktor. Disebabkan oleh pendedahan nisbah luas permukaan dengan isipadu lebih tinggi bagi filem dibandingkan dengan sampel pukal, ini dapat menerangkan kenapa kehilangan unsur plumbum lebih ketara bagi filem dibandingkan dengan sampel pukal. Imej tiga dimensi AFM menunjukkan mod pertumbuhan bak-pulau bagi filem. Corak ini mungkin berkait dengan kewujudan gugusan dalam plasma. Rawatan sepuhlindap menunjukkan ianya kaedah yang efektif untuk mengubah kandungan oksigen dalam system BSCCO dan mempelbagaikan sifat superkonduktor. Walaupun sifat superkonduktor yang dihuraikan bagi filem adalah tidak sebaik filem

yang dihasilkan oleh laser ‘excimer’, ianya boleh diperbaiki lagi dengan mempelbagaikan lagi parameter endapan.

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I certify that an Examination Committee met on …… to conduct the final examination of Nur Helmi @ Nur Jannah Binti Azman on her Master of Science thesis entitled "Preparation and Characterization of Superconducting Thin Films" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the 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 has been accepted as fulfilment of the requirement for 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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