Low-pressure Metalorganic Chemical Vapor Deposition

and Characterization of YBa$_2$Cu$_3$O$_{7-x}$ Thin Films


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ABSTRACT

Low-pressure metalorganic chemical vapor deposition (LP-MOCVD) is a technique which has been used with great success for the growth of compound semiconductors. In this study, we have successfully applied this technique to the deposition of yttrium barium cuprate (YBCO) superconducting thin films grown on a variety of substrate materials. These superconducting films have been deposited in a commercial-scale MOCVD reactor with a capacity of over 100 cm$^2$ per growth run. The EMCORE System 5000 reactor is based on a high-speed rotating disk susceptor (0-2000 rpm) design. Thin films of the binary oxides were initially deposited in order to determine the respective growth rates. This data was then used to calculate trial values of reactant flows for YBCO growth. YBCO layers were grown at 76 Torr on substrates held at 500-550 °C. The β-diketonates of Y, Ba and Cu, heated to 130, 240 and 120 °C, respectively, were used as sources. A 4.5 slpm flow of pure oxygen, representing a partial pressure of 38 Torr, was introduced uniformly at the top of the reaction chamber.

Post-growth annealing was performed in pure oxygen for 30-60 minutes at 900-950 °C. After annealing, initial YBCO layers grown on (100) YSZ substrates exhibited semiconducting behavior above the 90 K onset of the superconducting transition. The zero resistance temperature, $T_C(R=0)$, was 40 K. Under slightly different growth conditions, YBCO deposited on (100) SrTiO$_3$ exhibited a metallic
characteristic, with onset and $T_c(R=0)$ values of 95 and 85 K, respectively. These layers also showed a rather smooth morphology compared to previously reported results using LP-MOCVD. Results of characterization by x-ray diffraction, SEM, EDAX and XPS are reviewed. Evidence of low carbon/carbonate contamination was observed, however a significant substrate/layer interaction was detected in the case of YSZ.

1. INTRODUCTION

Since the discovery of high critical temperature oxide based superconductors (HTSC)\textsuperscript{1,2} many techniques have been investigated to prepare bulk and thin film samples of these materials. Development of high quality epitaxial thin films on suitable substrates is necessary for the application of HTSCs in electronic applications. Potential applications are SQUIDs, Josephson junctions and passive microwave devices such as ring resonators, filters and delay lines. In the future ultrastructured devices incorporating alternating layers of insulators, semi-conductors and superconductors will be outgrowth of present developments.

Presently, several techniques have been used to produce thin film HTSCs. These techniques include sol-gel\textsuperscript{3}, sputtering\textsuperscript{4,5}, evaporation\textsuperscript{6,7}, laser ablation\textsuperscript{8,9}, Molecular Beam Epitaxy (MBE)\textsuperscript{10} and Metal Organic Chemical·Vapor Deposition (MOCVD). Among these, the last three have shown the best results. Laser ablation, however, is limited by the control of large molecule (or droplet) formation and has not been shown to be suitable for large area epitaxy. MBE is limited by low process throughput and the inability to incorporate significant levels of oxygen during growth.

MOCVD is a fast growing, mature, and very promising technology in the compound semiconductor industry for epitaxial growth of III-V and II-VI materials and extremely versatile for the future applications. An important advantage of the MOCVD technique is the ability to perform growth under a high oxygen overpressure. The MOCVD growth mechanism involves the pyrolysis of organometallic compounds which have appropriate vapor pressure. The vaporized organometallic source materials (precursors) are transported by a carrier gas and injected into the reaction chamber. After the film deposition on the heated substrates, the by-products are carried away in the vapor phase and safely exhausted via a scrubbing system.

Several groups have demonstrated MOCVD-grown films of superconducting $YBa_2Cu_3O_{7-x}\textsuperscript{11,12}$ (YBCO) as well as for the Bi\textsuperscript{13} and Tl\textsuperscript{14} based superconducting oxide films. We present here our initial results for the films grown in a commercially available production scale growth system.