Integrating RHEED-TRAXS and Molecular Beam Epitaxy for Real-time Compositional Control of Functional Oxide Deposition Process

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Oct 11, 2011, 3pm, 224 Hurtig Hall

Real-time chemical analysis during film growth by Molecular Beam Epitaxy (MBE) has been unattainable because traditional Ultra High Vacuum (UHV) tools such as X-ray Photoelectron Spectroscopy (XPS) or Auger electron spectroscopy (AES) cannot be used at pressures above 10^{-8} torr, and MBE growth pressure are typically 10^{-6} to 10^{-5} torr. Real-time chemical analysis and stoichiometry control is important, however, because stoichiometry changes of less than one percent in materials such as functional oxides can cause measurable changes in their physical properties[1]. Indirect measurements of stoichiometry, such as surface Reflection High Energy Electron Diffraction (RHEED) pattern, are often misleading. RHEED - Total Reflection Angle X-ray Spectroscopy (RHEED-TRAXS) have been shown in this work to be a viable real-time relative stoichiometry analysis tool for MBE deposition processes. Despite the limitations in detecting low atomic number elements (Z<10), this work showed that RHEED-TRAXS could be used to provide useful chemical information during the deposition of multi-element metal oxides such as barium hexaferrite (BaM, $BaFe_{12}O_{19}$). While progress has been made through this work to qualify relative atomic ratio, quantitative stoichiometry measurement still faces challenges.

This RHEED-TRAXS study first involved designing a detector positioning system compatible with the UHV environment and the existing MBE chamber. Systematic factors that can impact the measured x-ray intensity were identified and characterized to provide consistent spectrum evaluation and data processing. Critical angle of the substrate signal, such as the Si Kα line, was established as a reference for calibrating the measurement geometry and determining elemental sensitivities. Using this reference, increases in film elements intensities such as the Mg x-ray Kα line, were measured during film growth such as magnesium oxide (MgO) deposition and related to the growing film thickness. Substrate Si Kα x-ray intensity attenuation through the MgO layer also provided a way for film thickness approximation. In the deposition process of

BaM, RHEED-TRAXS was used for monitoring Fe Kα line, Ba Lα line, Mg Kα line and Si Kα line signal intensity variations during the deposition. The intensity changes were related to the film stoichiometry changes during MBE processing. The correlation between thickness and composition with absolute and relative x-ray intensities based on calibrated geometry in this study is an excellent example of the potential of RHEED-TRAXS for real-time compositional analysis and further quantitative development.

Reference:

[1] I. Kanno, H. Kotera, K. Wasa, T. Matsunaga, T. Kamada and R. Takayama, *Journal of Applied Physics* 2003, *93*, 4091-4096.