Design and Characterization of a Microreactor for Thin Film Deposition and in situ Surface Analysis

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Abstract:

Atomic layer deposition (ALD) is a technique capable of precise control of film thickness and conformal film growth due to the self-limiting nature of the precursors involved. The Engstrom research group has built a microreactor that delivers and confines reactants of ALD in a small region for deposition. This microreactor is coupled to an ultra high vacuum (UHV) chamber for surface characterization such that the deposited film is transferred in vacuo, without an air break, to the analysis chamber. The most well-studied precursor of in the ALD community, trimethylaluminum (TMA) is used to characterize the microreactor to verify whether it is able to deposit Al₂O₃ film in ALD fashion with water as the co-reactant.

Summary of Research:

ALD has emerged as a potential approach capable of matching the rapid rate of downscaling of semiconductor devices. The self-limiting nature of ALD precursors brings about two major advantages unique to the technique: precise control of thickness of deposited film and conformal growth [1]. As described in Figure 1, ALD is sequential binary reaction separated by purge steps to prevent any unwanted parasitic reactions between the precursor and co-reactant in gas phase. TMA, the most-studied precursor in the ALD community, is introduced to a surface in alternating sequence with water as the co-reactant. The deposited alumina film is transferred in vacuo to and characterized in a UHV chamber coupled with the microreactor using x-ray photoelectron spectroscopy (XPS).

The thickness of deposited film (~ a few nm) is often in the range that is most effectively probed with surface-sensitive characterization techniques that require UHV, \( p < 10^{-6} \) Torr. Conventional ALD is typically conducted at low to medium vacuum conditions (\( p \sim 10^{-3} - 10^{-2} \) Torr), thus in most cases UHV based analysis of the deposited thin films occurs in a separate chamber, requiring an air break that may significantly alter the surface composition, oxidation state, structure of the deposited film, and/or underlying substrate. Avoiding this air break is critical for fundamental studies of the growth of ultrathin films, particularly in the early stages. Figure 2 describes how a sample is exposed to precursors at the upper stage and transferred down to the lower stage of the chamber without exposure to air for post-deposition characterization.

In this report, we assess the performance of the microreactor probe with various experiments. In a 10-cycle ALD experiment with \( \text{Al(CH}_3)_3 \) and \( \text{H}_2\text{O} \),...
we deposited an ultrathin film of alumina (Al$_2$O$_3$) on a hydroxyl-group terminated SiO$_2$ at substrate temperature of 180°C. From the integrated intensity of a XP spectrum of Al(2p) peak (refer to Figure 3), photoionization cross section, inelastic mean free path, and kinetic energy, thickness of alumina is calculated to be 14.2 Å [2]. Previous studies have reported growth rates ranging from 1.1 to 1.2 Å per cycle, indicating that thickness of the alumina film from ten ALD cycles using the microreactor lies close within the range of reported values [3]. In addition, elemental ratio of oxygen to aluminum is estimated to be 1.52, which corresponds to the stoichiometric ratio of elements in Al$_2$O$_3$ film.

A half-cycle ALD experiment is performed with the same precursor, Al(CH$_3$)$_3$, substrate, and temperature to verify linear correlation between thickness and number of cycles. The thickness of the ultrathin film from this experiment is 1.36 Å, approximately 9.6% of that from the 10-cycle one. This linear relationship between the thickness vs. number of cycles is a unique characteristic of ALD and confirms that the microreactor probe is capable of depositing films via ALD. The absolute atomic density of Al atom is calculated using calibration of semi-infinite Au film and methods described elsewhere [4]. Approximately 1.03 x 10$^{15}$ Al atoms·cm$^{-2}$ are detected from the half-cycle experiment whereas ~ 1.02 x 10$^{15}$ Al atoms·cm$^{-2}$ are present in the two topmost layers of α-Al$_2$O$_3$ <0001> surface [5].

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