

Atomic Layer Deposition (ALD) of Vanadium Oxide Thin Films

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ABSTRACT Microbolometer grade vanadium oxide thin films were grown by Atomic Layer Deposition (ALD) method using VTIP metal organic precursor and water vapor. The ALD process was studied using in-situ QCM system integrated into the reactor. Mass uptake during each half reaction was monitored and analyzed for different half-reaction sequences. An overall mass uptake of 20 ng/cm² was recorded for a complete ALD cycle. Thickness analysis of the vanadium oxide films grown by 2500 cycles yielded a thickness of ~800 Å which corresponds to a 0.32 Å /cycle thin film growth rate. The ALD grown thin films were characterized and their electrical and structural properties were reported. Temperature coefficient of resistance (TCR) value was measured to be around -4.7%/K, and resistivity was found as 77kΩ.cm. In addition, the ALD grown vanadium oxide thin films are found to be oxygen rich in composition that is yielding V₂O₅.

Keywords: Vanadium oxide, ALD, VTIP, Microbolometer, Thermal imaging, QCM

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1. INTRODUCTION

Vanadium oxide is an important material for uncooled infrared (IR) imaging applications [1-3]. Especially for low-cost bolometer systems, different flavors of vanadium oxide thin films have been investigated [4-8]. Although, these films showed potential for higher performance for bolometers, reproducibility of the bolometer grade vanadium oxide films remained a challenge.

Thermal imaging has become a crucial technology in military and civilian applications especially after the achievements in the area that made thermal detectors cost effective and highly efficient [9]. Resistive microbolometers are widely used thermal detectors in which a resistivity difference through the thermally sensitive layer of each micro-pixel occurs due to a temperature increase induced by IR absorption [10]. Vanadium oxide films have been widely used as thermally sensitive layer in microbolometers as these films have appropriate properties which are temperature coefficient of resistance (TCR), resistivity, and 1/f noise [1, 2]. Even though vanadium oxide films have been the choice for room temperature microbolometers, fabrication of these films with reproduceable and stable microstructures which correlates to good TCR and resistivity characteristics, has been a challenge. In this

work, ALD is applied to growth stable and defect-free thin films of vanadium oxide with bolometer grade properties.

There are just a few studies on the microstructure and electrical properties of VO_x thin films in the literature. Gauntt and his co-workers found that microstructure of the reactively sputtered VO_x films depends on the partial pressure of the oxygen, and this dependence shows itself with a change in the resistivity trend where a transition from nanocrystalline to amorphous growth happens [2]. Electrical conduction mechanism of the charge carriers for highly defective nanocrystalline films was proposed to be a combination of band conduction along the defective nanocrystalline VO_x domains and thermally activated hopping mechanism among defect states [11]. As films get more amorphous, hopping mechanism becomes the only way conduction occurs. Few other studies also showed that reactively pulsed DC sputtered VO_x films that provide electrical properties useful for the microbolometer applications have a two-phase structure which is a combination of nanocrystalline and amorphous phases [12-16].

Atomic layer deposition (ALD) is a specific type of chemical vapor deposition method that vapors of different pre-cursors sequentially react with a surface to form a thin film. Each ALD cycle consists of two or more

half reactions. In these cycles, each precursor reacts with the surface, and the remaining gas molecules are purged out before the next precursor is dosed into the reaction chamber. Thus, two precursors do not react with each other in the gaseous state which separates ALD from the conventional chemical vapor deposition technique. ALD method has various advantages compared to the general thin film deposition methods [17]. For example, conformal and pinhole-free thin films can be deposited by using ALD where the process is very precise, highly reproducible and large-area scalable [17]. ALD can be applied to fabricate complex multilayered structures with an extremely well-controlled thickness. Temperature application window for most ALD processes is ranging from 25 °C to 300 °C which enables application to delicate substrates preventing temperature induced damage.

ALD technique has been characterized mostly for Al₂O₃ and ZnO thin films, and the application procedures and chemical processes for such thin films have been better understood compared to more exotic materials [18-20]. For example, trimethylaluminum and water vapor for Al₂O₃ film growth have been applied as a standard reaction to analyze and evaluate ALD mechanisms in a custom reactor [17, 19]. A few in-situ characterization methods have been utilized for ALD reactors including quartz crystal microbalance (QCM) for studying real-time growth rates [18]. In situ characterization methods enable users to understand the nature of each reaction step taking place in the ALD process.

Various pre-cursors have been used for ALD deposition of vanadium oxide films including vanadyl-tri-isopropoxide (VTIP), Vanadium oxytrichloride (VOCl₃), and bis[2,4 pentanedionato] vanadyl(II) [VO(acac)₂] [21-23]. As the oxidizing agent oxygen or water vapor are used for VO(acac)₂, VTIP, and VOCl₃, thin film growth. Temperature for the process of VOCl₃ and oxygen pre-cursors was given as 490 °C that is higher than conventional ALD reactions. In this particular work, they reported that no film formation occurred at lower temperatures. Musschoot *et al.* applied lower temperatures such as 150 °C to grow vanadium oxide thin films by using VTIP precursor and water vapor [22]. They reported that the ALD process yielded V₂O₅ films with a film growth rate around ~0.2 Å/cycle. It was also noted that oxygen as the oxidizing agent did not yield any film growth. Dagur *et al.* reported that the ALD reaction utilizing VO(acac)₂ and oxygen as chemical precursors resulted in a narrow process temperature window (400 °C - 475 °C), and VO₂ thin films were grown by the process [23]. They found a growth rate of 2.4 Å which was 12 times more than Musschoot's growth rate. Badot *et al.* achieved 0.17 Å /cycle film growth rate by applying the same chemical precursors as Musschoot utilized, and obtained V₂O₅ films [24].

In current work, a custom-built ALD reactor [25] is used to grow thin films of vanadium oxide for microbolometer applications. In-situ QCM analysis of the ALD process is achieved for the thin films. Electrical and structural properties of the vanadium oxide thin films are investigated using Raman spectroscopy and four-point-probe methods.

2. EXPERIMENTAL

A custom-built ALD reactor including an in situ QCM system is used for growing vanadium oxide films [25]. Briefly, the ALD reactor is consisting of two manifolds passing through a static-mixer and the reaction chamber. One of the manifolds is utilized for delivering metal-organic precursors such as TMA and VTIP, and the other manifold provided the oxygen, water vapor and hydrogen gas flow. Nitrogen gas is used to purge the reactor after each ALD cycle. Within the reactor, the substrate mounting plate, two QCM sensor heads, walls of the reactor, static-mixer, and the two manifolds are externally heated. Temperatures of these sections can also be adjusted independently for desired parts of the system. The flow rates for carrier gases are tuned using two mass flow controllers. During operation, a constant volume is first filled and then purged into the reaction chamber. A roughing pump is applied to enable a desired working pressure in the system. The ALD reactor is also equipped with a turbo molecular pump which can be utilized for high vacuum ALD processes running in the molecular flow regime without carrier gases. The ALD system is controlled by several pneumatic valves placed at different regions of the reactor to sequentially let precursor gases inside the reaction chamber. Pressure data is collected in each manifold and in the reaction chamber area utilizing capacitor monometers.

To grow the vanadium oxide films with ALD method, VTIP and water vapor precursors are utilized. VTIP (Vanadium(V) oxytriisopropoxide, Sigma Aldrich Inc., USA) precursor produce a very low vapor pressure at room temperature. To increase the vapor pressure, VTIP container is heated up to 60 °C so that a vapor pressure of 0.63 Torr is generated. The pressure of water vapor at room temperature is obtained around 20 Torr. During a VTIP dosing step, a constant volume vessel located in the metal-organic manifold line is filled until the pressure reached to 0.63 Torr. For the water vapor dosing step, a constant volume located in the water vapor manifold is filled until the pressure value of 18 Torr. The vapor pressure of water is set to this value upon observing the saturation of the reaction which is the condition of a complete reaction after two half reactions are completed. During the film growth, the substrate temperature is increased to 150 °C. To create the same conditions on the QCM surface, the temperature of the QCM assembly is also heated to the same temperature. The temperature of the QCM is controlled using a separate thermocouple located inside a heating cartridge in order to compensate the low thermal conductance through the adhesive tapes that are applied to stick the heating element to the backside of the QCM. Thus, to reach to 150 °C at the QCM head, heater is needed to be set to 220 °C. This difference in the temperature caused a slow rise of temperature in the crystal inside the QCM holder. This temperature drift can potentially alter the frequency shift reading on the QCM that could also change the slope of the mass uptake data. Nevertheless, temperature reading of the QCM was observed to be quite stable during the time windows of each cycle occurring with a sharp elevation in mass uptake. Thus, the temperature drift did not cause a problem for mass uptake reading. Total flow rate in the ALD reactor occurred as 260 sccm (standard cubic centimeters per minute) of which 110 sccm flowed through each mass flow controller unit and 40 sccm was applied to purge the rear side of the QCM to stop film formation on that side. Since QCM works with frequency damping due to mass uptake, film deposition on the back side of the QCM crystal can

affect the mass uptake reading. Considering all the gas flows, base pressure of the ALD reactor is measured as 0.85 Torr. Before filling the vessel of the VTIP precursor in the dosing manifold, an external vacuum pump was applied to reduce the base pressure of the metal-organic dosing manifold.

A 2500 full cycle ALD reaction was completed with 12 seconds purging duration after VTIP precursor dosing, and 16 second purge duration after water vapor dosing. While the water dosing step occurring, VTIP dosing vessel was set to be filled in the metal-organic manifold to reduce the total cycle time. In this experimental ALD reactor the main goal is to study the ALD reactions rather than growing thin films with high throughput so the time for growing thin films takes longer compared to commercial ALD reactors. Total VTIP vessel filling time adjusted sufficiently for reaching the maximum possible metal-organic precursor vapor pressure for the temperature of 60 °C. The total time for one full ALD cycle was recorded as 38 seconds that consists of 1 second of metal organic VTIP filling, 5 seconds of VTIP vapor dosing, 12 seconds of purging, 1 second of water vapor filling, 3 seconds of water vapor dosing, and finally 16 seconds of the reactor chamber purging. During the reactor purging step, VTIP precursor was also set to fill the metal-organic manifold to reduce the total cycle time. Temperature coefficient of resistance and resistivity measurements are performed using a temperature controlled four-point-probe system. For the Raman spectroscopy a Renishaw inVia Raman Microscope (Renishaw Inc., Gloucestershire, UK) is utilized. A HeNe laser with 632.5 nm wavelength is applied as the excitation source with a spectral power of 5 mW. A 100x objective lens with a numerical aperture of 0.9 is utilized to collect all the Raman data. In-situ QCM mass uptake data is collected to evaluate the ALD process. A multichannel spectroscopic ellipsometer (J.A. Woollam Co. RC2, USA) functioning with a dual rotating compensator is used to measure thickness of the ALD grown thin films.

3. RESULTS AND DISCUSSIONS

Mass per area monitoring for the VTIP + water vapor repeated film growth is given for a 30-cycle process in Figure 1. As it is observed, the ALD process is resulting in a layer by layer uptake of mass that is a typical characteristic of the general ALD method. The data reflects a linear trend which is demonstrated by the line drawn along the stepped-plot. The first 3 cycles are seen to be slightly off the line that is most likely due to the initial conditions of surface of the crystal of the QCM system. Pressure readings of each cycle is overlapped in Figure 2. Vapor pressure value of the VTIP precursor that was heated to 60 °C was measured in the metal-organic manifold when it was isolated from the reaction chamber as 0.63 Torr which is seen as a small step in the red colored line. The pressure reading in the reaction chamber indicated a pressure around 6.5 Torr during each VTIP dosing cycle. This pressure value is due to carrier gas. Vapor pressure of water is indicated with the blue colored line that reads around ~18 Torr in the reaction chamber during the water vapor dosing cycle. It is clearly observed from the figure that the mass uptake occurs during the VTIP dosing cycle, and there is almost no net mass uptake during water vapor cycle. Following the water dosing cycle, a slight mass reduction is recorded in the plot which happens during the purging step. It is most

likely due to the removal of the free radicals from the surface that are created after the completion of the first half reaction. These radicals are being swept away by the carrier gas during the purging step. As a consequence of this mass reduction, a net mass uptake of around ~ 10 ng/cm² per cycle occurs even though the initial amount of the mass uptake was ~ 20 ng/cm².

For the analysis of the mass-uptake on the QCM sensor during the ALD process, the Sauerbrey equation is sufficient considering that the thin films are solid and rigid [26]. The Sauerbrey equation related the change in the resonant frequency of the oscillating quartz crystal of the QCM sensor by

$$\Delta F = \frac{-2NF_0^2}{A\sqrt{\rho_q\mu_q}} \Delta m \quad (1)$$

where m , N , A , F , ρ_q , and μ_q are the mass uptake, the overtone of the crystal oscillator, the surface area of the QCM, oscillation frequency, density of the quartz, and the shear modulus of the quartz used in the QCM, respectively. In order to find the molecular adsorption per unit area (D) related to the mass increase measured by the QCM, Avogadro's number (N_A) and the molar mass (M) are needed

$$\frac{D M}{N_A} = m \quad (2)$$

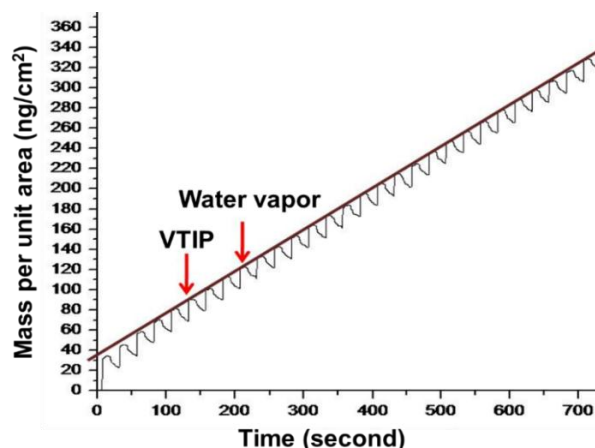


Fig. 1. Mass per unit area for a 30 full cycle VTIP-water vapor ALD growth process.

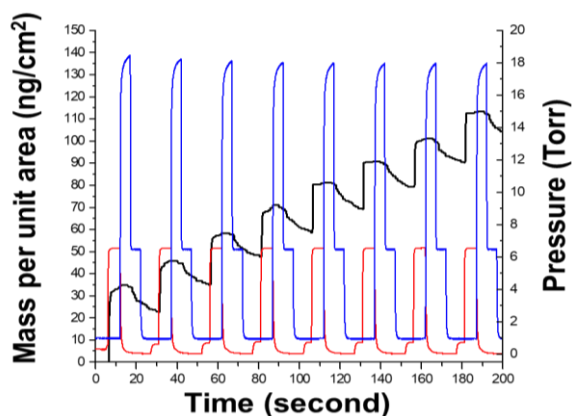


Fig. 2. Mass per cycle data overlapped with pressure data of VTIP and water vapor. (Blue line: pressure of the water vapor, red line: pressure for the VTIP, and black line: the mass uptake data)

A simple calculation is performed to get an estimated number for molecules per unit area. From its molecular structure, VTIP precursor gives a molecular weight of around ~ 244.2 g/mol. A 20 ng/cm² uptake of mass corresponds to 8.19×10^{-11} mol/cm². Using the Avogadro's number, it is calculated that when the VTIP precursor reacts with the surface 4.92×10^{13} molecules adsorbed per unit area. Approximately there are around 10^{15} atoms per cm² on a surface. For comparison, TMA has a molecular weight of 72.09 g/mol, and an average mass-uptake of 39 ng/cm² which corresponds to 3.25×10^{14} molecule/cm². This value is approximately an order of magnitude higher than that of VTIP. In the literature, VTIP and water vapor ALD process was reported to have a growth rate of ~ 0.2 Å/cycle when VTIP precursor was heated up to ~ 45 C [22] whereas growth rate for TMA and water vapor reaction has been reported to be ~ 1.2 Å/cycle [18].

In order to study the saturation trend of the VTIP precursor, an ALD experiment was designed with three repeated VTIP doses followed by a single water vapor dose. Mass and pressure data of this run is shown in Figure 3. After the completion of the first VTIP dose (red colored line) a mass increase of around 20 ng/cm² occurs, and the second and the third VTIP doses result in mass uptakes between 3 and 7 ng/cm². The data demonstrate that after the water vapor dosing, previously witnessed mass decrease starts to take place and an equilibrium is reached. Overall mass increase occurs as 20 ng/cm² for a complete cycle.

Raman spectra of the vanadium oxide films grown by both ALD and sputter deposition methods are shown in Figure 4. The sputtered deposited film is grown by reactive sputtering in an argon/oxygen environment using a pulsed DC and RF magnetron sputtering tool. The spectrum for ALD sample reveals that a low frequency feature appears at 400 cm⁻¹ wave number. From the correlation of transmission electron microscopy analysis in a previous work by authors [3], this feature suggests that the vanadium oxide film is highly amorphous. In addition, a high frequency feature appears at 930 cm⁻¹ wave number with a larger intensity value which is likely due to the high resistivity of the vanadium oxide film. When compared to vanadium oxide thin film grown by sputter deposition which yield a high frequency mode around 890 cm⁻¹, the high frequency mode of the ALD grown sample appeared at a higher wave number which implies the abundant presence of V⁺⁵ valance of vanadium in the resulted films.

Spectroscopic ellipsometry analysis of the vanadium oxide films grown by 2500 cycles yielded a thickness of ~ 800 Å which corresponds to a 0.32 Å/cycle thin film growth rate. This growth rate is significantly higher than the values that were reported in the literature (~ 0.2 Å/cycle) [22]. This difference is likely due to higher vapor pressure obtained for VTIP precursor in this study.

Electrical properties of the ALD grown vanadium oxide thin films were evaluated using a temperature controlled four-point-probe system. Temperature coefficient and resistance (TCR) value was measured to be -4.7 %/K, and resistivity was found as 77 kΩ cm. As the Raman plot suggested the thin film was highly resistive which is likely in the form of V₂O₅.

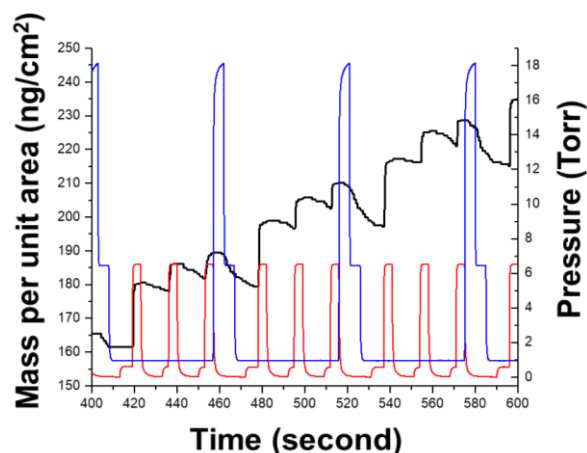


Fig. 3. The ALD process with 3 repeated VTIP doses and 1 Water vapor dose. (Blue line: water pressure, red line: the VTIP pressure, and black line: the mass increase data.

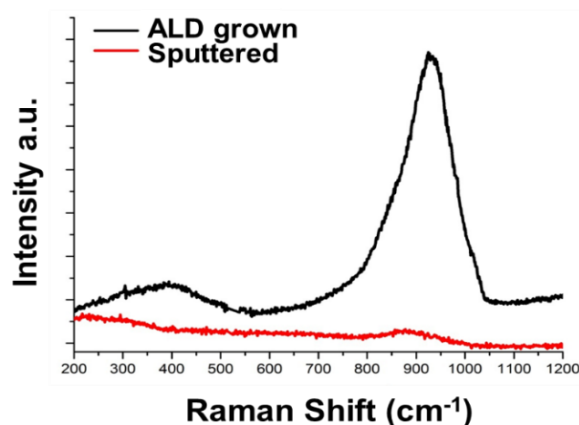


Fig. 4. Raman spectra of the ALD grown and pulsed-DC reactive sputtered deposited vanadium oxide thin films.

4. CONCLUSION

ALD grown vanadium oxide films were studied using an in-situ QCM system in current study. QCM mass increase data showed that with a single VTIP vapor dose, a 20 ng/cm² initial mass per unit area uptake took place which corresponded to 4.92×10^{13} molecules/cm² according to the calculations. As a comparison, performing the same calculations resulted in 3.25×10^{14} molecules/cm² for the TMA metal-organic precursor applied in Al₂O₃ growth in the literature. This simple calculation explains the lower growth rate of VTIP and water vapor ALD process (~ 0.32 Å/cycle) compared to the TMA and water vapor process (~ 1.2 Å/cycle). As an attempt to observe the saturation behavior, multiple VTIP steps followed by a single water vapor step were performed. It was observed that after the initial VTIP step the mass increase still occurred with decreasing amounts. This recommends that the saturation of VTIP vapor half reaction did not occur completely with a single VTIP dose.

The growth rate of the VTIP and water vapor ALD process was observed to be greater than the previously published values. It is likely that when the VTIP metal-organic was heated up it produced higher vapor pressures corresponding to a greater number of VTIP molecules entering the reaction chamber and resulting in a higher growth rate.

Evaluation of the electrical properties of the ALD grown vanadium oxide thin films showed promise for microbolometer applications especially with higher TCR values. To further improve the vanadium oxide thin films grown by ALD process, sub-oxidized high-quality vanadium oxide films can be fabricated by applying reducing steps after each VTIP and water vapor full cycle using a plasma-enhanced ALD process.

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Biography



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