

# Determination and Improvement of Deposition Parameters of TiO<sub>2</sub> Thin Films via ALD

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**Abstract:** In recent years, with the development of technology, interest in microelectronics and thin film devices has increased considerably. Future improvements in microelectronics and thin film devices are dependent on the progress of novel materials and new deposition processes. In particular, the continuing drive to some devices such as silicon devices will soon require SiO<sub>2</sub> gate and oxide layers with a thickness on the order of a few nanometers as 1 or 2 nm. Titanium dioxide is a candidate material in microelectronic and thin film devices, a wide band gap semiconductor that exhibits various crystal structures. Similarly, thin film techniques like ALD (atomic layer deposition) attract attention for the preparation of these candidate materials with higher film uniformity and conformity. Present study demonstrates how TiO<sub>2</sub> thin films were growth by ALD technique on silicon substrates at 100 °C, 150 °C and 200 °C temperatures. Different deposition conditions were examined to determine to increase film quality and efficient production of set up. XPS (X-ray photoelectron spectroscopy) technique was used to obtain the surface composition of the TiO<sub>2</sub> films. Film thicknesses and crystal structures of the films were investigated by ellipsometry and XRD (X-ray diffraction) methods. Electrical properties of the films were measured by using four probe techniques, as well. Obtained results were evaluated in terms of repeatability of recipes and application potential.

**Key words:** TiO<sub>2</sub> films, ALD, film grown, optical properties.

## 1. Introduction

Titanium dioxide (TiO<sub>2</sub>) is a wide band gap semiconductor that exhibits various crystal structures such as the tetragonal anatase and rutile. Though TiO<sub>2</sub> is possibly best known for its photocatalytic properties, it is also an interesting material for dielectric and transparent-conductor applications. Owing to its higher dielectric constant, rutile is the structure-of-choice for dielectric applications, while the higher electron mobility of anatase makes it more attractive to transparent-conductor applications [1]. Amorphous TiO<sub>2</sub> thin films are studied increasingly for subwavelength optical structures due to their high

index of refraction and transparency within a broad wavelength region [2]. TiO<sub>2</sub> tends to have n-type conducting character due to intrinsic defects that easily form in its crystal lattice [1]. There are many studies on TiO<sub>2</sub> in the fields of diluted magnetic semiconductors, photocatalyst materials, and electrode materials for electrochromic devices [3].

In the past two decades, it has been considered almost certainly to be the most studied transition metal oxide due to its many unusual physical, chemical, electronic, electrochemical and photoactive properties unexceptionally. TiO<sub>2</sub> has also attracted vast interest in chemical engineering related research such as solar energy conversion via dyesensitized and quantum-dot-sensitized solid-state solar cells, water splitting and general photocatalysis, lithium ion

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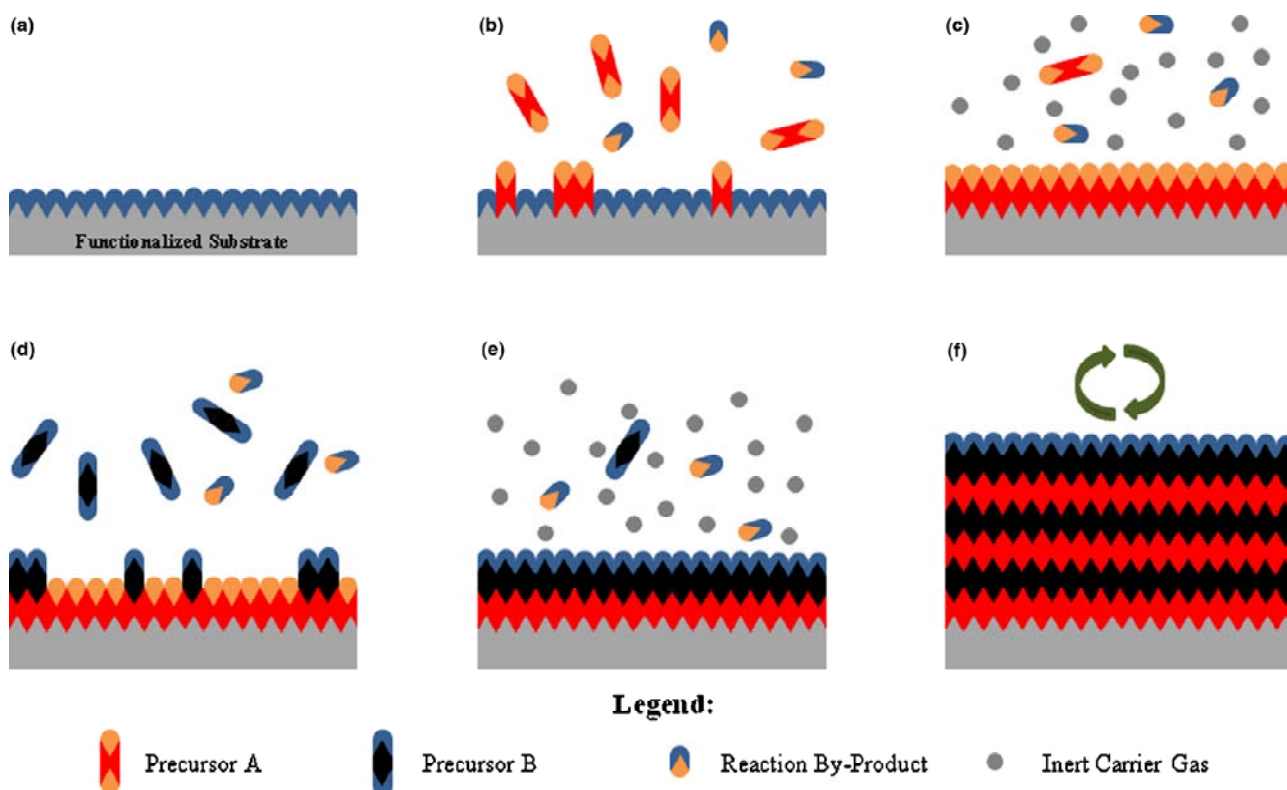
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batteries and supercapacitors, chemical/gas sensing, controlled release, environmental remediation including indoor air purification, wastewater treatment, antifogging, self-cleaning, deodorization and deactivation of bacteria [4-6]. As the firstly reported semiconductor material for photoelectrochemical water splitting, TiO<sub>2</sub> has been intensively studied by a large number of groups in the past decades [7-9]. Although poor ability to light absorption limits its application to a large extent, TiO<sub>2</sub> is still an excellent assistant for other materials arising from its stability over a range of pH [9-12]. TiO<sub>2</sub> protecting layer in the form of ultrathin film usually contains pinholes and cracks, which will activate an oxidative dissolution of the underlying substrate [13].

PVD (physical vapor deposition), CVD (chemical vapor deposition) and spin coating can be given as some of the fabrication ways of TiO<sub>2</sub> films [14]. Aside from these growth techniques, ALD (atomic layer deposition) shows the advantage of conformal growth and precise film thickness control, which are very important to the fabrication of future microelectronic [15] and the other devices. ALD, which is one of the thin film growth techniques, has become advanced fabrication method for a rich variety of applications including complementary metal oxide semiconductor transistors, DRAM memory, energy conversion, photovoltaic, and display devices [16-18]. ALD is a cyclic vapor phase deposition technique, which takes the privilege of temporarily separated and self-limiting reactions of two or more precursors and reactants [19] that thereafter grow an ideally desired film with one specific atomic layer thickness at a time [20]. This technique facilitates the growth of various kinds of coatings including ultra-thin pure and composite layers with precise control of composition by

appropriate design of ALD recipe and process details [21, 22]. In ALD technique, the precursors are pulsed to the reactor chamber one after another with intermediate purging, which results in self-limited surface reactions. A sequence consisting of one pulse of each precursor and intermediate purging is called an ALD cycle shown Fig. 1. Ideally one ALD cycle deposits a monolayer of the target compound, and thus the film thickness is accurately controlled by the total number of cycles. Doping is realized by replacing single ALD cycles of a mother compound by an ALD cycle of a dopant compound [20]. Similarly, ALD is a convenient technique for the production of good quality thin TiO<sub>2</sub> films [23]. Obtained TiO<sub>2</sub> thin films by using ALD techniques have been widely used in energy and environmental science due to its high chemical stability, nontoxicity and favorable energy band structure [24]. As it is known, the properties of nanomaterials depend on their shapes, sizes, crystal structures, defects, impurities, and surface areas, the potential application areas of the films will be intensely affected by these features [25, 26].

In the present study, TiO<sub>2</sub> thin films were prepared by ALD technique on silicon substrates at 100 °C, 150 °C and 200 °C temperatures in order to determine appropriate recipe. Deposition conditions were tried to determine for increasing film quality and efficient production of set up. XPS (X-ray photoelectron spectroscopy) technique was used to obtain the surface composition of the films. Ellipsometric measurements and XRD (X-ray diffraction) analysis were carried out to determine of thicknesses and crystal structures of the films. Electrical properties of the films were measured, as well. Obtained results were evaluated in terms of repeatability of recipes and application potential.



**Fig. 1** Schematic representation of ALD process. (a) Substrate surface is treated to functionalize the surface. (b) Precursor A is sent and reaction starts with surface. (c) Excessive precursor and reaction by-products are purged with inert carrier gas. (d) Precursor B is sent and reacts with surface. (e) Excessive precursor and reaction by-products are purged with inert carrier gas. (f) Steps b to e are repeated until the desired film thickness is succeeded [18].

## 2. Materials and Method

Depositions of the thin films included in this work were carried out in OKYAY Tech. R&D (Fig. 2) ALD reactor at various temperatures. Prior to the deposition, Si (100) which is used as substrate for production of TiO<sub>2</sub> thin film was cleaned via using standard Radio Corporation of America (RCA) cleaning methods. The pulse and purge period of ALD cycles were optimized for TiO<sub>2</sub> for three different recipes until the self-limiting and uniform growth was observed on Si (100) substrates. For the proper recipe, the precursor dose and purge times were adjusted as 0.15 and 10.0 s, respectively. For some samples grown at 200 °C, optimized recipe was used, where the dose and purge sequence for precursors was (0.015-30-0.015-30) s. The temperature range for film growth was from 100 to 200 °C. The number of growth cycles was varied

from 400 to 1,200 and corresponding film thicknesses of approximately 50 to 150 nm. Various heat treatments have been implemented for characterizing of anatase and rutile phases of TiO<sub>2</sub>.

XPS measurements were performed with a Thermoat a vacuum of  $3 \times 10^{-9}$  Torr (K-Alpha-Monochromated high-performance XPS spectrometer) instrument. Deposited thin films thicknesses were characterized via spectroscopic ellipsometry measurements. Structural properties of the films were acquired by using X-ray diffraction (Bruker D8 Advance & DIFFRAC Measurement Center Software) method. The electrical characteristics, mainly the resistivity, of the materials were obtained with a four-probe resistivity measurement method by using Keithley 2400 source meter. Surface morphology was examined by using SEM (scanning electron microscopy) (an FEI Nova Nano SEM 430 microscope operated at 10 kV).



Fig. 2 Thermal ALD device.

### 3. Results

The TiO<sub>2</sub> films were characterized by XPS technique to obtain the surface composition. The data analysis given in Fig. 3 involved spectra normalization, Shirley background subtraction, and curve-fitting Gaussian–Lorentzian line shapes [27]. Best fits were evaluated using a root-mean-square measure where line shape, peak width (FWHM) and binding energy were adjustable parameters. Shirley backgrounds were used in all fits to narrow scan spectra [28].

All XPS spectral peaks given in Fig. 3 were fitted with Thermo Scientific Avantage software. All spectra showed O 1s and C 1s spectral lines consist of a single peak (a singlet) whereas the Ti 2p spectrum consist of two peaks, a spin-orbit doublet. The C 1s spectral line was seen at 285.0 eV and the O 1s and Ti 2p spectra

were adjusted to this energy. The O 1s spectrum of TiO<sub>2</sub> film has been twinned, one is peaked at 530.2 eV, and the other is peaked at 531.84 eV. The O1s spectrum of the obtained TiO<sub>2</sub> film can be determined into three peaks, one is peaked at 530.2 eV, and the other two are peaked at 531.84 eV and 532.4 eV, after annealing at 550 °C respectively. According to the literature, the main peak at the 530.2 is due to the oxygen lattice and Ti-O bond while the peaks at 531.2 eV correspond to water (O-H) and hydroxide absorbed on the surface and the peak at the 532.9 eV is probably due to the water and hydroxide Ti or basic OH on the surface [29-35].

Related to ellipsometric measurements, thicknesses of titanium dioxide films annealed at various temperatures showed the proper values  $\pm 5$  nm with growth cycles. According to these results, the thin films thickness increases parallel to increasing cycles.

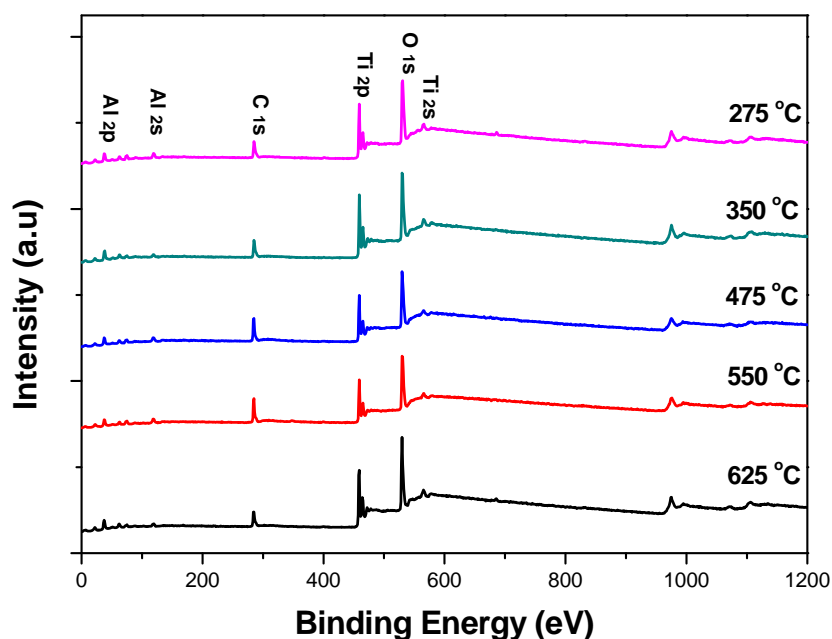


Fig. 3 XPS spectra of ALD-grown titanium dioxide films annealed at various temperatures.

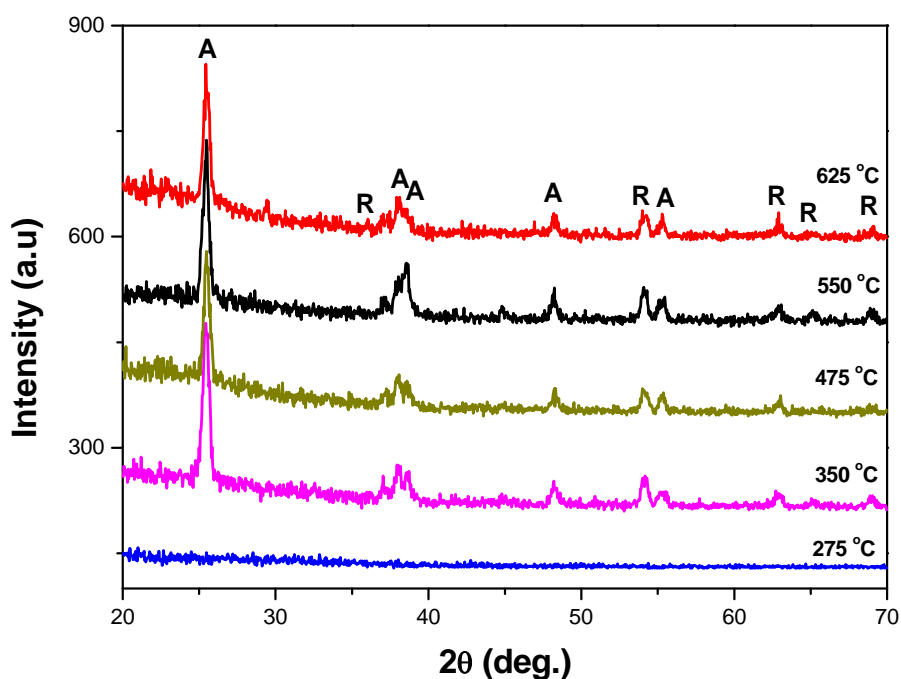


Fig. 4 XRD spectra of ALD-grown titanium dioxide films annealed at various temperatures.

To clarify the crystal structure of ALD grown TiO<sub>2</sub> films, the XRD spectra was investigated which gives anatase and rutile phases after the heat treatment of 275 °C in Fig. 4. According to XRD data, the films grown at 275 °C were amorphous. All the films

annealed at higher temperatures contained crystalline phases of TiO<sub>2</sub>. X-ray diffraction studies revealed formation of anatase phases at  $2\theta = 25.48; 37.92; 38.12; 48.20; 55.28$  and rutile phases at  $2\theta = 36.04; 54.00; 62.92; 64.76; 69.12$ , respectively. Also, the

contents of the anatase and rutile structures and electrical properties of ALD-grown TiO<sub>2</sub> films annealed at various temperatures have been given in Table 1.

Table 1 also shows the calculated carrier mobility, threshold voltage, subthreshold slopes, and I<sub>on</sub>/I<sub>off</sub> ratios of fabricated TiO<sub>2</sub> devices. Extrapolation method in the saturation region (ESR) is used to extract  $\sqrt{I_D} - V_G$  characteristics of the devices [36-38]. Subthreshold slopes are extracted from  $\log(I_D - V_G)$  characteristics with  $dV_G/d\log(I_D)$  relation. Carrier mobility values were extracted from output  $I_D - V_D$  characteristics. Oxide capacitance is calculated using  $C_{ox} = \epsilon_{ox}/t_{ox}$  and  $C_{ox} = \epsilon_0 \cdot \epsilon_r / t_{ox}$  relations, where  $\epsilon_r$  denotes dielectric permittivity of ALD deposited TiO<sub>2</sub>.

It is known that electrical properties of TFT devices follow a similar trend with outcome of XPS measurements. O/Ti ratio in the film decreases with increasing deposition temperature. This results in higher effective doping due to defects [39-41]. At low

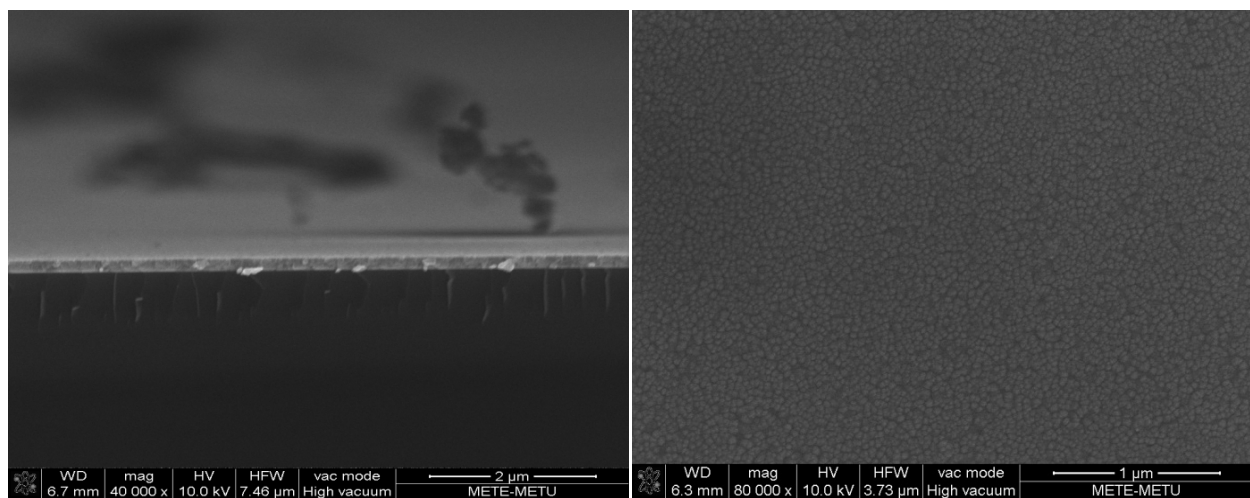
deposition temperatures, O-H bonds passivate the defects and therefore reduce carrier concentration and increase I<sub>on</sub>/I<sub>off</sub> ratio. Typical I<sub>D</sub>-V<sub>DS</sub> characteristics of a device annealed at 475 °C shows maximum value. A maximum I<sub>on</sub>/I<sub>off</sub> ratio of 2.5×10<sup>6</sup> is recorded as 25× improved compared to the highest reported for this kind of devices up to now. After ALD growth, thin films annealed at 475 °C have the optimal Ti:O stoichiometry, therefore the lowest defect density. It can be said that this result is in good agreement with the tendency observed in threshold voltage vs. annealing temperature.

However, when the electrical properties compared with the XRD results, it is understood that the films annealed at 475 °C have the highest anatase content regarding to higher electron mobility.

SEM images of two different samples were given in Fig. 5. SEM images of the ALD grown TiO<sub>2</sub> films were taken at about 6 mm working distance, high vacuum mode, 10 kV accelerating voltage, X40000 for cross-sectional and X 80000 for surface image. SEM

**Table 1** Anatase and rutile content and electrical properties of ALD-grown titanium dioxide films annealed at various temperatures.

T <sub>Annealing</sub>	Anatase (%)	Rutile (%)	V <sub>Threshold</sub> (V)	I <sub>on</sub> /I <sub>off</sub> subthreshold	Slope (V/dec)	Mobility (cm <sup>2</sup> /V·s)
275	73	27	-1.8	10 <sup>2</sup>	6.55	0.330
350	84	16	0.2	10 <sup>2</sup>	5.21	0.400
475	99	1.0	6.5	2.5×10 <sup>6</sup>	0.35	0.670
550	88	12	4.3	4×10 <sup>5</sup>	1.36	0.17
625	96	4.0	7.1	10 <sup>6</sup>	0.35	0.27



**Fig. 5** SEM images of the titanium dioxide films by ALD, annealed at various temperatures: (a) cross-sectional image; (b) surface image.

images of the thin film demonstrated the homogeneous phase distribution of the TiO<sub>2</sub> (Fig. 5b). Some porosity is seen on the surface morphology of these thin films, which is influenced by the growth temperatures, annealing and process parameters. After ALD growth, thin films annealed at 475 °C have the optimal Ti:O stoichiometry, therefore the lowest defect density. It can be said that this result is in good agreement with the tendency observed in threshold voltage vs. annealing temperature.

When obtained results in all experimental studies are evaluated, it can be said that ALD thin films presented at this study are ideal candidates for microelectronic devices and optical applications.

#### 4. Conclusion

In order to improve deposition parameters of TiO<sub>2</sub> thin films via ALD have been investigated and conclusion can be drawn as follows:

As a result, we made a comprehensive study on the deposition parameters for growth TiO<sub>2</sub> films by using ALD technique. For this purpose, various recipe and annealing temperatures have been used and ideal one determined the best one. By the way, it is tried to determine the impact of deposition condition on the structural, electrical properties of the ALD grown TiO<sub>2</sub> film. An ideal recipe showed that the growth temperature is 200 °C. Our results demonstrate that deposition parameters affect the structural, surface and electrical properties of the ALD TiO<sub>2</sub> films, as well.

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