THERMAL EFFECT ON THE OPTICAL AND MORPHOLOGICAL PROPERTIES OF TIO$_2$ THIN FILMS OBTAINED BY ANNEALING TI METAL LAYER

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Titanium metal layers of different thicknesses were deposited on optical glass, quartz and ceramic substrates at 50 °C and 150 °C substrate temperature with the help of magnetron deposition. Metal layers were converted into rutile phase of TiO$_2$ at different annealing temperatures. The effect of thermal annealing on the morphology and refractive index of the thin film was investigated.

Keywords: Key words: Titanium metal, Titanium dioxide, Annealing and Rutile phase.

Introduction

In recent years, Titanium dioxide (TiO$_2$) has attracted wide attention as a potential photonic material [1-7]. Amorphous and polycrystalline TiO$_2$ thin films can be deposited at low temperatures using conventional, scalable methods [8], allowing for direct integration with other on-chip devices [9]. Due to its high refractive index (n≈2.4), it allows the realization of high-refractive index contrast waveguides with strong light confinement and in dielectric grating elements. TiO$_2$ is transparent over a broad wavelength range that includes the visible and near infrared because of its large bandgap (Eg= 3.1 eV). Additionally, TiO$_2$ is a promising nonlinear photonic material. As compared to silica, its nonlinearity is 25 times higher [10-12] and low two photon absorption for wavelengths above 800 nm [12]. These properties make TiO$_2$ to position in a similar class as silicon nitride: Moreover, TiO$_2$ has higher linear refractive index (2.4 for TiO$_2$ versus 2.0 for silicon nitride at 800 nm) and nonlinear refractive index (9x10-19 m2/W for bulk rutile TiO$_2$ versus 2.4 x 10-19 m2/W for silicon nitrde) [10].

TiO$_2$ has three basic crystalline phases- anatase (tetragonal), rutile (tetragonal), brooktile (orthorhombic) and amorphous phase. Reactive sputtering is considered to be the useful method to deposit both rutile and anatase TiO$_2$. Although, rutile TiO$_2$ films were successfully deposited on a non-heated substrates by RF magnetron sputtering . Thermal annealing can change the structural and optical properties of the deposited films which are reported by several other groups [11]. In this paper, we studied the effect of thermal annealing on Ti metal layer in order to obtain rutile TiO$_2$ films. The structural and morphological properties of TiO$_2$ thin films were determined.

Experimental

A. Ti layer deposition

Ti thin film was deposited on optical glass, ceramic (sital) and quartz with the help of magnetron deposition system Caroline D12A. For all deposition experiments, we used DC power of 300 W, 2x10-5 Torr and the gap between substrate/target was 100 mm. The adhesion of Ti films was improved by heating the substrate but the film morphology strongly depends on the substrate temperature during the deposition process. Ti films of 80-200 nm was deposited for morphological and optical characterization.

B. Annealing process

Usually, Ti thin films deposited below 150 ^{o}C are amorphous and attain crystallinity after annealing process. TiO_2 was obtained by high temperature annealing (oxidation) of Ti films. Annealing process was carried out in muffler furnace by using Conventional Thermal Annealing (CTA). The samples were loaded in the muffler at room temperature and slowly heated till 700 ^{o}C and then maintain the temperature for 1 hr. Once the annealing was complete, the temperature was reduced till room temperature with a slow ramp. In order to obtain rutile phase of TiO_2, the annealing was done between 500-700 ^{o}C for 1-2 h. After annealing, the obtained TiO_2 films were fully transparent which can be observed visually.

Results and discussion

At first we have investigated the effect of annealing on Ti film deposited on ceramic substrate at 500 and 700 ^{o}C respectively. After observing their SEM images, we found that, the TiO_2 film obtained at 700 ^{o}C has bubbles all over the surface which contributes in high surface roughness. We believe that, these bubbles appeared due to heating the ceramic substrate at high temperature during annealing process. On the other hand, TiO_2 film obtained at 500 ^{o}C has lower roughness. It was hard to find any dominant peak on the surface of the layer.

Furthermore, we investigated the effect of substrate temperature during deposition of Ti layer. For this purpose, we used optical glass, ceramic and quartz. Substrate temperature of 50 and 150 ^{o}C were used during Ti metal deposition to test the adhesion and scratch of the layer. After several attempts of tape test, it was confirmed that, Ti layer deposited at both 50 and 150 ^{o}C showed good adhesion on optical glass, ceramic and quartz.

Ti layer of 120 nm was deposited on ceramic at substrate temperature of 50 and 150 ^{o}C. Both of these substrates were annealed at 600 ^{o}C for 1 hour by conventional thermal annealing. It was found that TiO_2 layer obtained after annealing at 600 ^{o}C (substrate temperature of 50 ^{o}C) has less surface roughness as compared to the TiO_2 film obtained after annealing (substrate temperature 150 ^{o}C) as shown in figure 1(a). It can be predicted that, these bubbles has some connection with the pre-heating of ceramic during Ti deposition as seen in figure 1 (b). Ti thin film of 120 nm was deposited on optical glass substrate at 50 and 150 ^{o}C and annealed at 600 ^{o}C for 1 h. From SEM images, it can be observed that TiO_2 layer obtained after annealing at 600 ^{o}C (substrate temperature of 50 ^{o}C) has comparable less surface roughness as compared to the TiO_2 film obtained after annealing (substrate temperature 150 ^{o}C) as shown in figure 1 (c) and (d). Although these roughness are too high, that these layer can’t be used in optical waveguides.

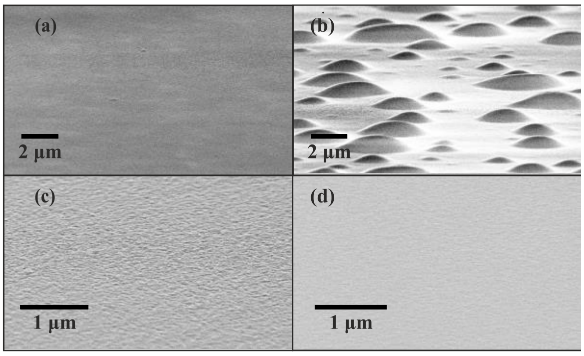

Fig. 1. SEM images of the surface morphology of TiO_2 /ceramic substrate obtained after annealing at 600 °C (a) substrate temperature 50 °C, (b) 150 °C, TiO_2 /optical glass substrate obtained after annealing at 600 °C (c) substrate temperature 50 °C, (d) 150 °C, TiO_2 /quartz substrate obtained after annealing at 600 °C (e) substrate temperature 50 °C, (f) 150 °C.

Ti thin film of 120 nm was deposited on quartz substrate at 50 and 150 oC and annealed at 600 oC for 1 h. From SEM images, it can be observed that TiO2 layer obtained after annealing at 600 oC (substrate temperature of 150 oC) has comparable less surface roughness as compared to the TiO2 film obtained after annealing (substrate temperature 50 oC) as shown in figure 1 (e) and (f). These films are smooth and can be used in the fabrication of various optical components.

The phase of annealed thin TiO_2 films deposited on different substrates was verified by Raman spectroscopy and obtained one weak Raman shift peaks at 143 and two strong peaks at 447 and 612 cm^{-1} which indicates rutile phase.

Refractive index of TiO_2 layer of same thickness deposited on ceramic substrate annealed at different temperatures (500 and 700 °C) was measured. From figure 2, it can be observe that, the TiO_2 films on ceramic substrate annealed at 500 °C has higher refractive index range as compared to sample annealed at 700 °C. The gradient in refractive index can be due to the expansion of layers at different ratio during annealing at different temperature.

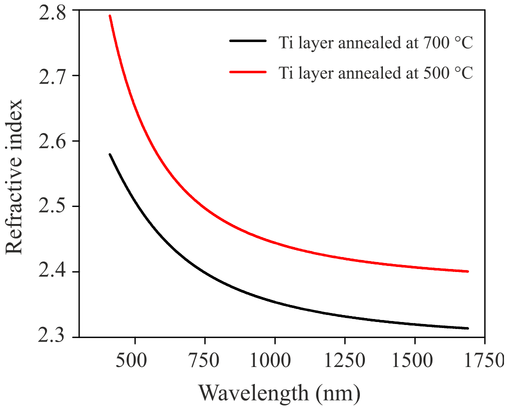

Fig. 2. Refractive index measurement of TiO_2 layer on ceramic substrate

We found different results in case of TiO_2 films of different thickness deposited on quartz but annealed at same temperature. As it can be seen from figure 3, that the film with 38 nm has less refractive index than film with 185 nm in visible range, but it increases in IR region.

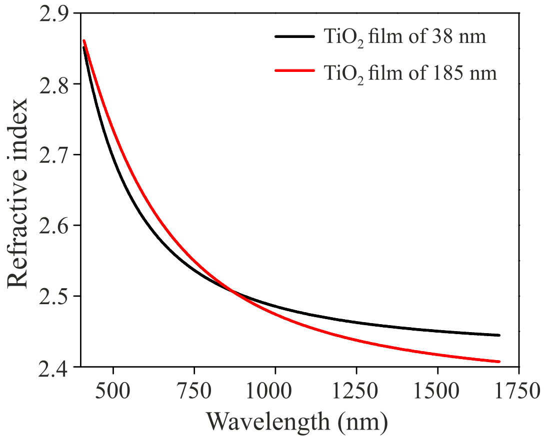

Fig. 3. Refractive index measurement of TiO_2 layer on quartz substrate

The optical properties of TiO_2 film deposited on quartz substrate were measured with the help of Ellipsometer M2000DI. Transmittance spectra were measured in the wavelength range of 350-1600 nm. For the visible wavelength range, the transmittance is around 70-75% but at 413 nm, transmittance peak falls to 51%. At 471 nm and 861 nm, it has high transmittance peaks at 90 and 92% respectively, as shown in figure 4.

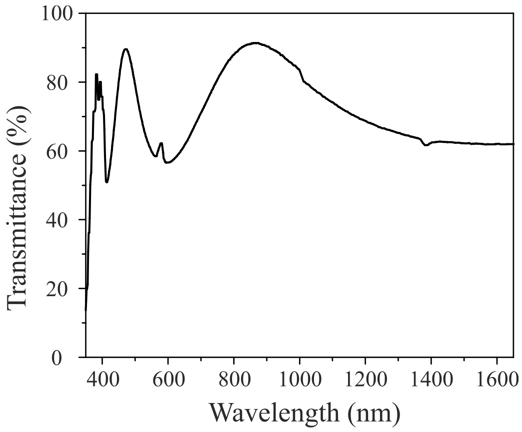

Fig. 4. Transmittance spectrum of TiO_2 deposited on quartz

Conclusion

Titanium metal layers deposited by magnetron sputtering on different substrates were converted into titanium dioxide rutile phase at 500-700 ^{o}C. Polycrystalline phase was determined by Raman spectroscopy. Surface morphology of the dielectric film deposited on different substrates was observed by SEM and found the low surface roughness on quartz substrate when the deposition temperature of Ti metal layer was 150 ^{o}C. These dielectric layers showed high transmittance at 471 and 861 nm.

Acknowledgements

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