

Thickness Analysis of TiO₂ Thin Films on Quartz by Optical Spectroscopy

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The purpose of this experiment is to develop a method for determining a thin film thickness with a very simple and non-destructive analysis using optical spectroscopy. This is done by creating a calibration chart that correlates optical absorbance with thickness measurements via Scanning Electron Microscopy (SEM). During the experiment sacrificial samples were created and analyzed by SEM, but once the correlation has been made, future samples can be analyzed quickly, cheaply, and non-destructively.

The materials of interest in this study are TiO₂ thin films coated onto quartz substrates. Coatings were prepared by a dip coating method, in which the quartz slides were dipped into a Ti sol-gel, and subsequently oxidized by heat treatment at 500°C [1]. The thickness of the coating was adjusted by controlling the weight percent of Ti atoms in the sol-gel through serial dilution with absolute ethanol, while the dip coating and heat treatment parameters were held constant. Symbolizing, the weight percent of Ti in the original sol-gel as W, then the serial dilutions resulted in weight percents of 0.5W, 0.4W, 0.3W, and 0.2W; these are labeled in the figures as S1-S4 respectively, an uncoated quartz substrate was used as a reference and is labeled as S0. An example of the SEM analysis of one sample (S4) is shown below in Figure 1.

The optical transmittance of each sample was then measured by a Shimadzu UV-3600 spectrophotometer in the range of 250-500 nm. The results are shown in Figure 2a along with the calculated absorbance (Figure 2b). It was hypothesised that the films would have a thickness that is directly proportional to the titanium weight percent, and further, that the peak (257 nm) optical absorbance should be directly proportional to the film thickness. This is justified theoretically by the fact that the optical transmittance of the film exponentially decays with increasing thickness as established by the Beer-Lambert Law. Since absorbance is calculated by taking the negative logarithm of transmittance, the result should be proportional to the film thickness.

In order to test the hypothesis the relative absorbance was plotted against the relative weight percent for each sample, and is shown in Figure 3a along with a dotted line depicting the theoretical prediction for proportionality ($y = x$). The correlation coefficient for the data is 0.97, confirming that the coating thickness is in fact proportional to Ti weight percent. The equation relating the absorbance measurements to the film thickness was then determined by plotting each thickness measurement (t) from the SEM analysis vs. its respective absorbance value (a), and finding a linear fit to the data. The correlation coefficient for this data was also calculated, and has a value of 0.99. The fact that the correlation between absorbance and thickness is higher than that between weight percent and thickness indicates that most of the deviation from theory was introduced in the coating process, rather than in the measurement. The resulting line is expressed mathematically as $t = 56.2a - 4.4$ and is plotted in Figure 3b; this equation permits simple and non-destructive determination of the coating thickness via optical transmission measurement.

References:

- [1] Tada, H. & Tanaka, M. Langmuir **13** (1997), p.360–364.
- [2] This study is supported in part from PSU’s Institute of Sustainable Solutions, Undergraduate Research Mentorship Program, and NSF-REU program.

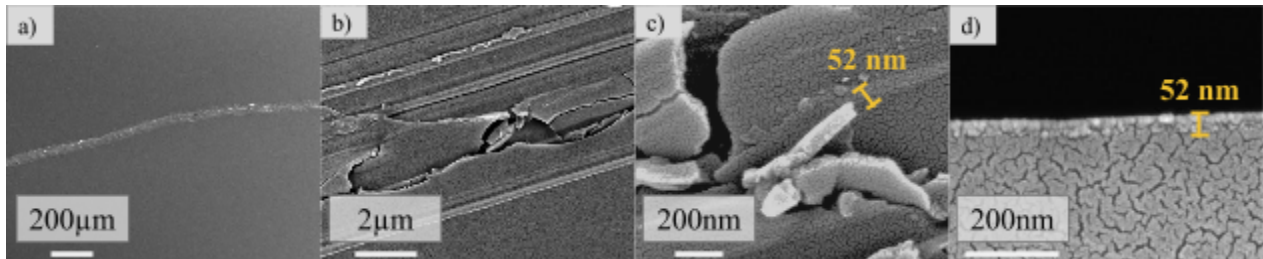


Figure 1: SEM images of the coating. a) and b) A scratch was made along the entire length of the coating using a diamond scribe in order analyze the coating thickness, and to verify the thickness consistency. c) Broken pieces of the coating oriented perpendicular to the detector were used for measurements. d) A cross section of the sample was created to verify coating thickness.

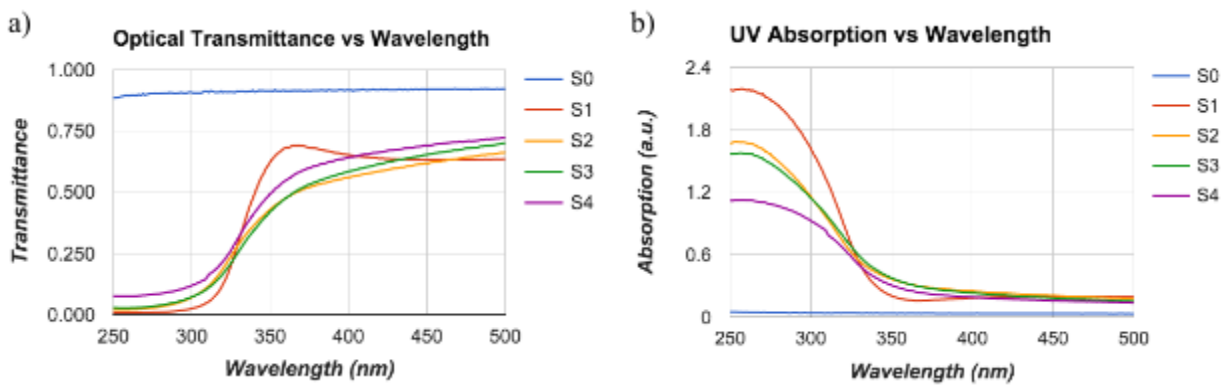


Figure 2: Optical transmittance (a) and absorbance (b) for the four samples from 250-500 nm.

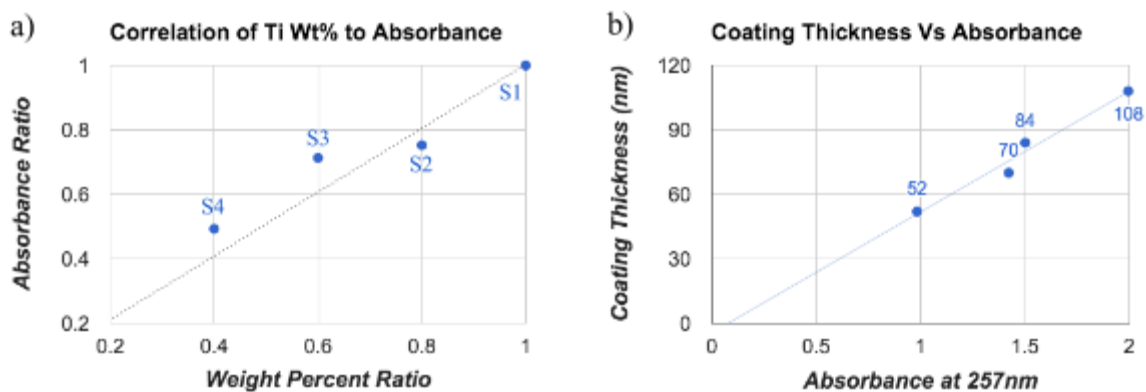


Figure 3: a) Correlation data used to verify the proportionality of Ti weight percent in sol-gel to peak absorbance values of the resulting films. b) Coating thickness data plotted against peak absorbance values to obtain mathematical relationship ($t = 56.2a - 4.4$).