SPECTROSCOPIC ELLIPSOMETRY STUDY ON E-BEAM DEPOSITED TITANIUM DIOXIDE FILMS

Lianchao Sun
Angstrom Sun Technologies Inc.,
Ping Hou
Nortel Networks

Presented at 3rd International Conference on Spectroscopic Ellipsometry (ICSE03) in Vienna, Austria
July 8, 2003

Introduction

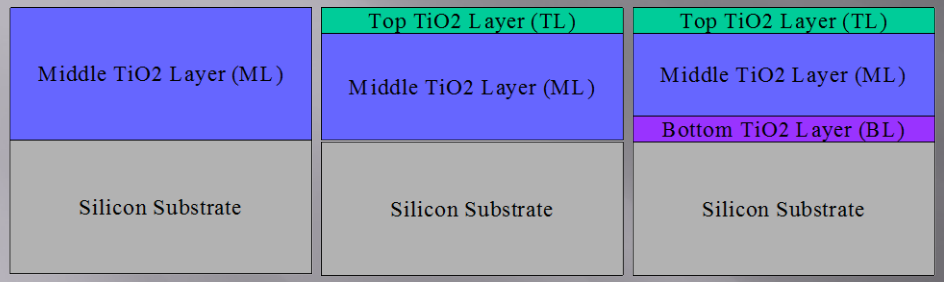
- Titanium oxide (TiO2) thin film has been widely used as optical coatings due to its high index of refraction, generally when high-low index contrast is desired in multiple layer interference coatings. However, structure and properties of TiO2 films are highly dependent on processing technique and conditions. In optical MEMS structure such as tunable vertical surface emission laser (VCSEL), TiO2 is preferred to be prepared with e-beam evaporation because of need to control stress within such a structure. Although e-beam evaporation has long been used to deposit optical thin films, it is still a challenge to deposit homogeneous TiO2 films because there are three different phases such as amorphous, anatase and rutile. In addition, growth structure may also vary from beginning to end of film and thus film density changes. This poses challenges to processing quality control and thus film characterization too.
- Spectroscopic ellipsometry is a nondestructive optical technique. It could be used to extract thickness, roughness and optical constants through proper optical model. At the same time, it is possible to further extract structure information from the relationship between optical properties and film microstructure. In this study, more extensive examination was performed in order to provide fundamental understanding of TiO2 film growth with the use of UV-Vis-Near IR ellipsometry analysis. The established optical model will be verified with destructive technique such as scanning electron microscope (SEM) and transmission electron microscope (TEM).

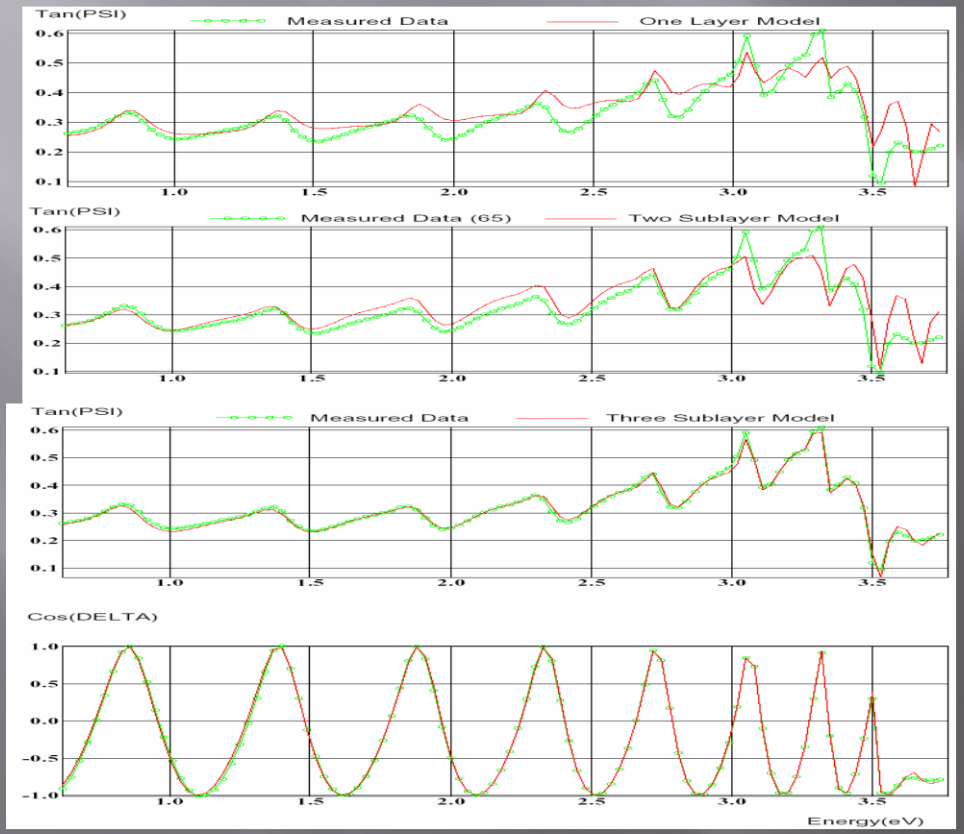
Experimental Details

- 1. TiO2 Film Deposition
  - Technique: Ion-Assisted E-beam evaporation
  - Source Material: Premelted Solid Block from Ti2O3 Tablets
  - Deposition rate: 2Å/sec controlled by quartz crystal
  - Total chamber pressure: 2.5x10-4 torr
  - Oxygen partial pressure: 0.6-1.0x10-4 torr
  - Initial Chamber temperature: 160oC (monitored at near sample holder)
  - Substrate: 2" single side polished Silicon wafer rotated at a speed of 32 rpm during deposition

- 2. Film Characterization
  - Ellipsometry Measurement
    - UV-Vis-Nir Variable Angles Spectroscopic Ellipsometer
    - Measured wavelength range: 210nm to 2000nm
    - Angle of incidence used: 65, 70 and 75 Degree
    - Measurement points: 120pts at each AOI
  - Microstructure
    - Hitachi Scanning Electronic Microscope (SEM)
    - Transmission Electronic Microscope (TEM)
    - Atomic Force Microscope (AFM)

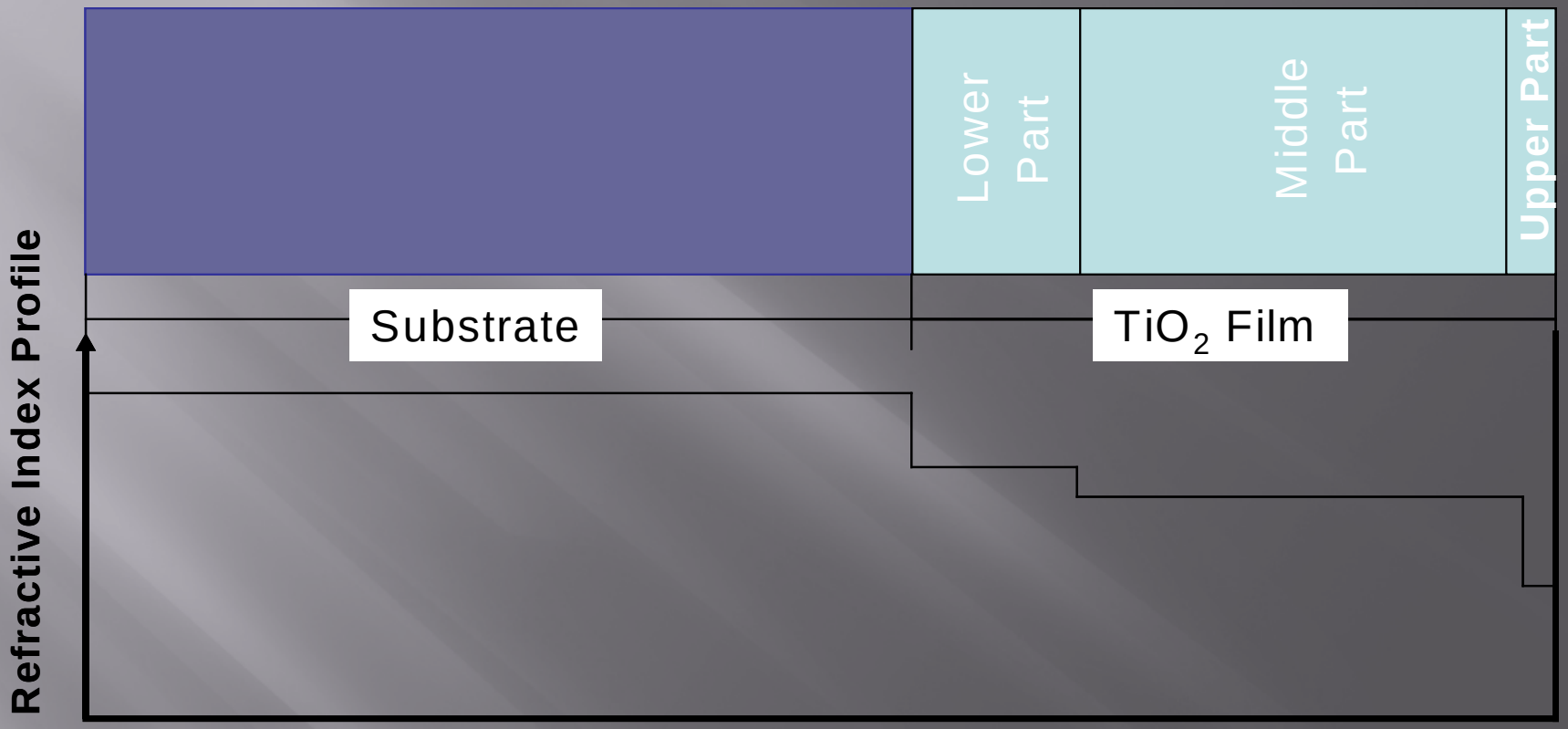
Model and Fitting





SE data were analyzed with three different model: single layer, two sub-layer and three sub-layer. Top layer is described as surface roughness with EMA. Both middle and bottom layers are described with Forouhi Interband model with one absorption band in UV-Vis Range. (Above plots show fitting quality for 65o data only).

Analysis Results



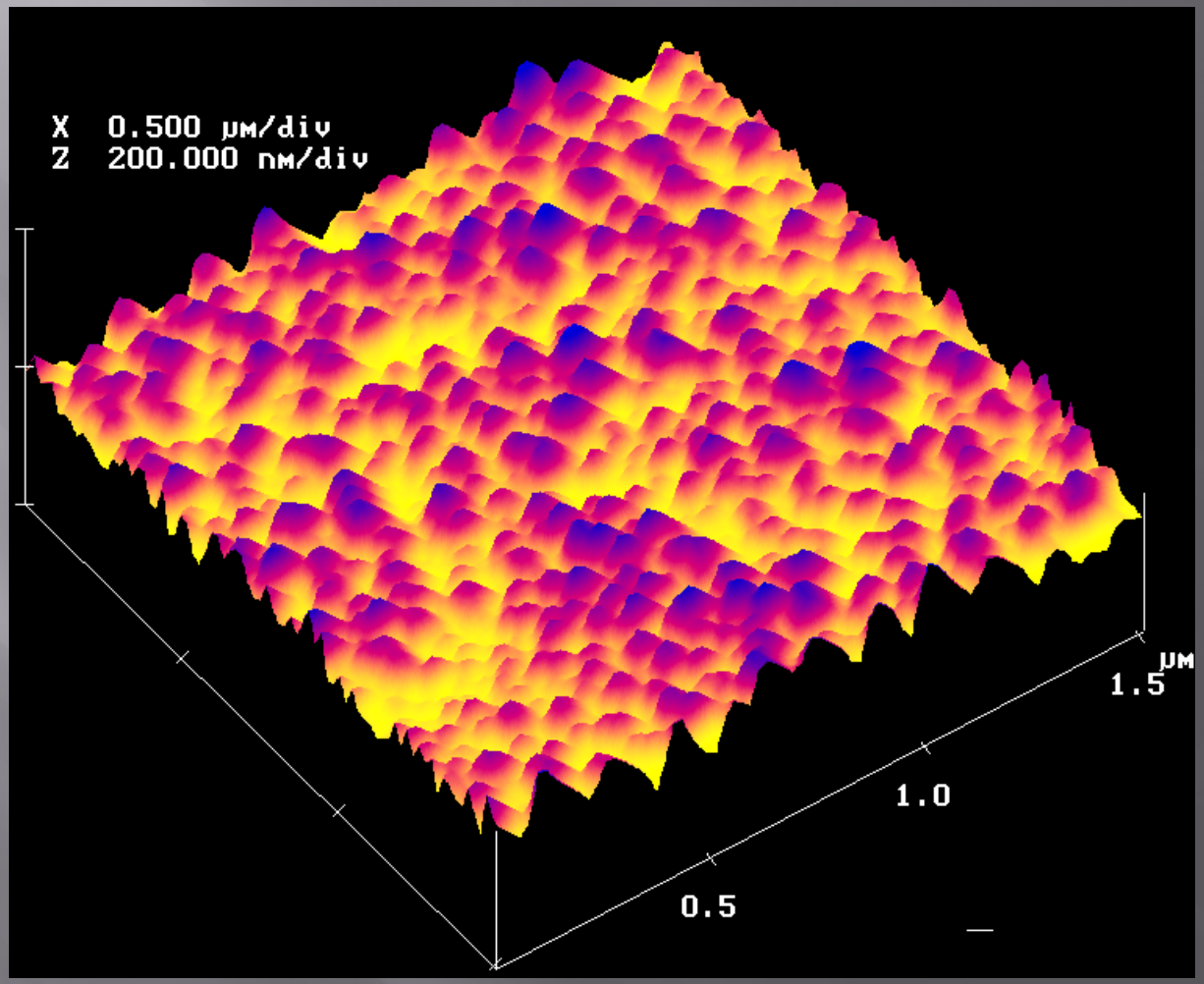
Three sub-layer model gives best fitting.

From three sub-layer model, middle part of the film has lower refractive index than bottom part. Top layer is very rough. Index profile is schematically shown above.

Summary Table

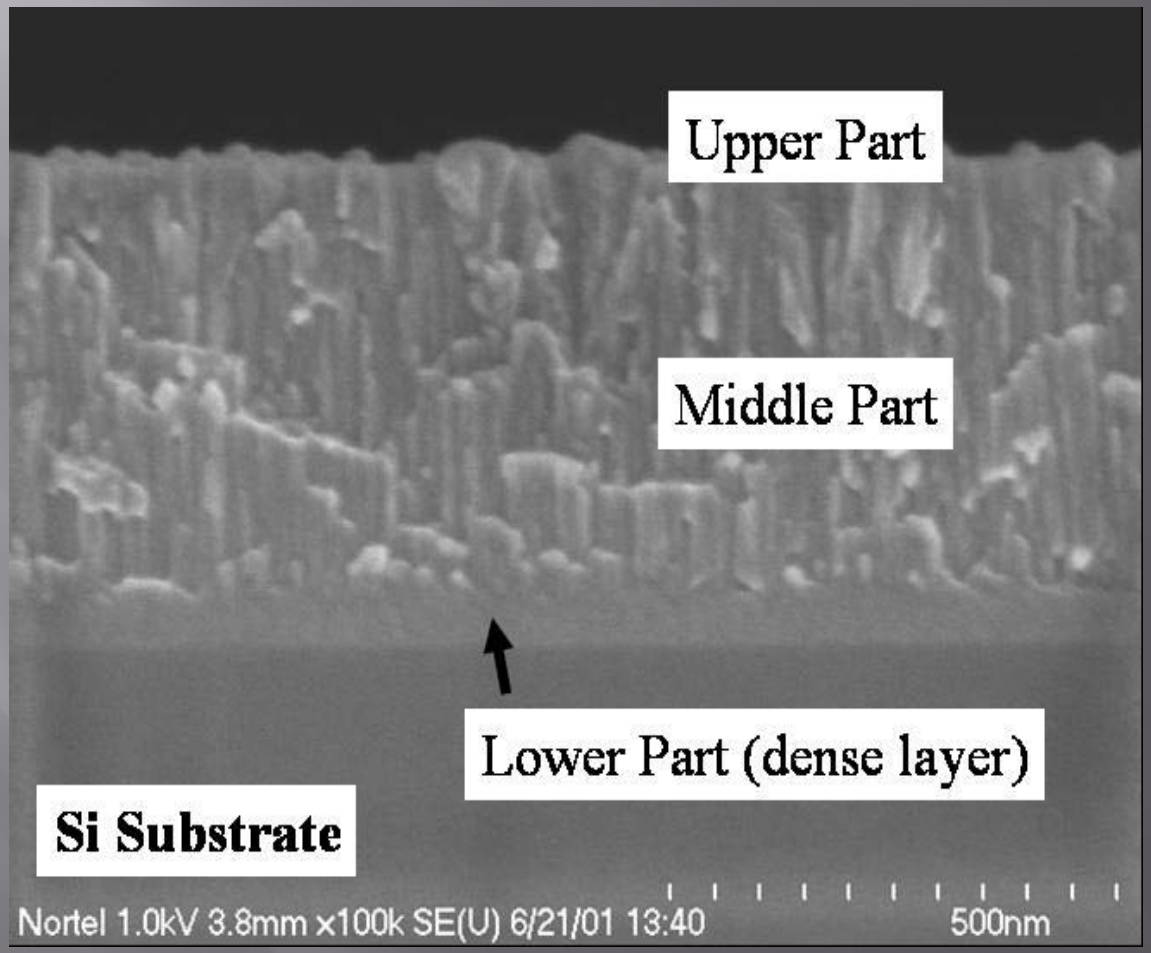
Model	Sub-layers	Thickness (nm)	Refractive Index @1550nm	Standard Deviation for Fitting (χ^2)
One Layer Model	Single	526.9	2.271	0.05
Two Sub-Layer Model	First	21.5	1.939	0.003
	Second	498	2.308	
Three Sub-Layer Model	First (Top)	13.5	1.689	0.0008
	Second (Middle)	463.6	2.252	
	Third (Bottom)	60.1	2.336	

Film Morphology



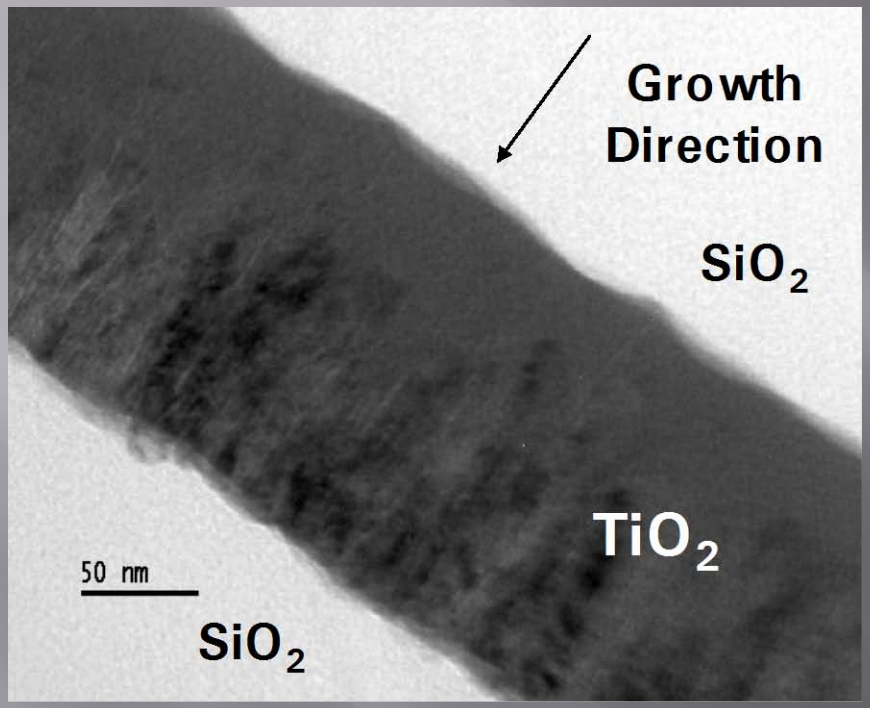
AFM scanning shows very rough surface (Rms: 19nm).

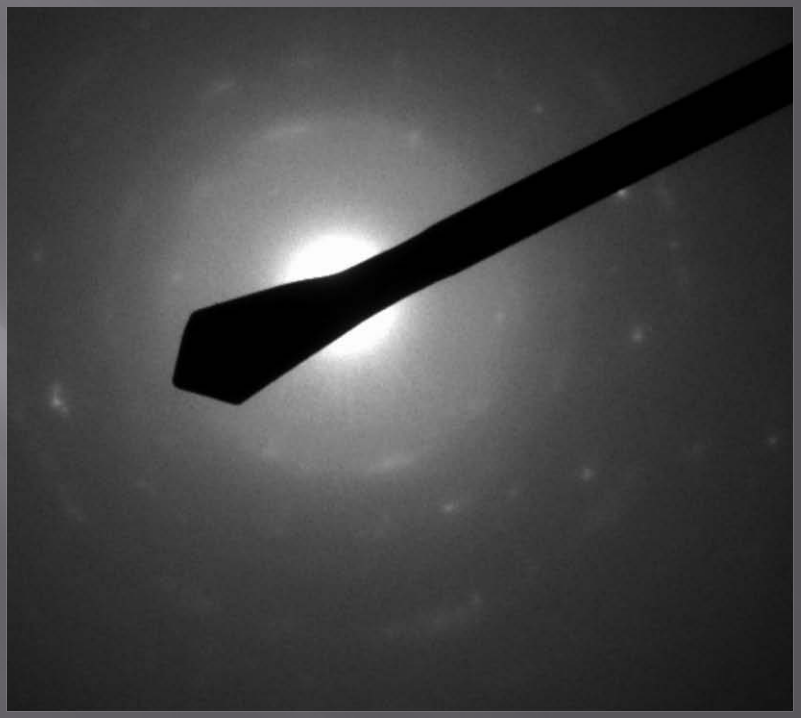
SEM Cross Section View



SEM photo from cross section shows film growth changing from dense part to columnar structure.

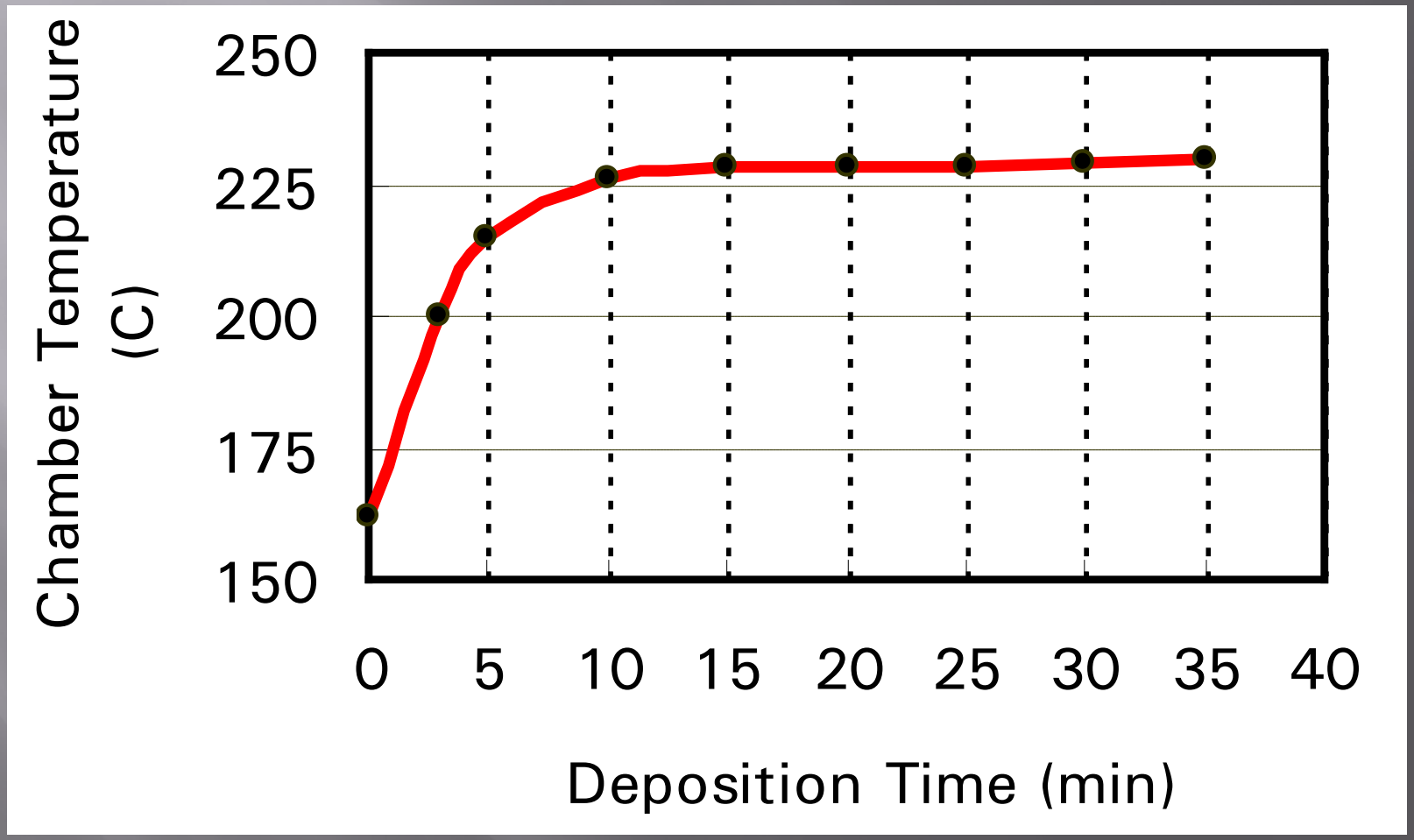
TEM Study





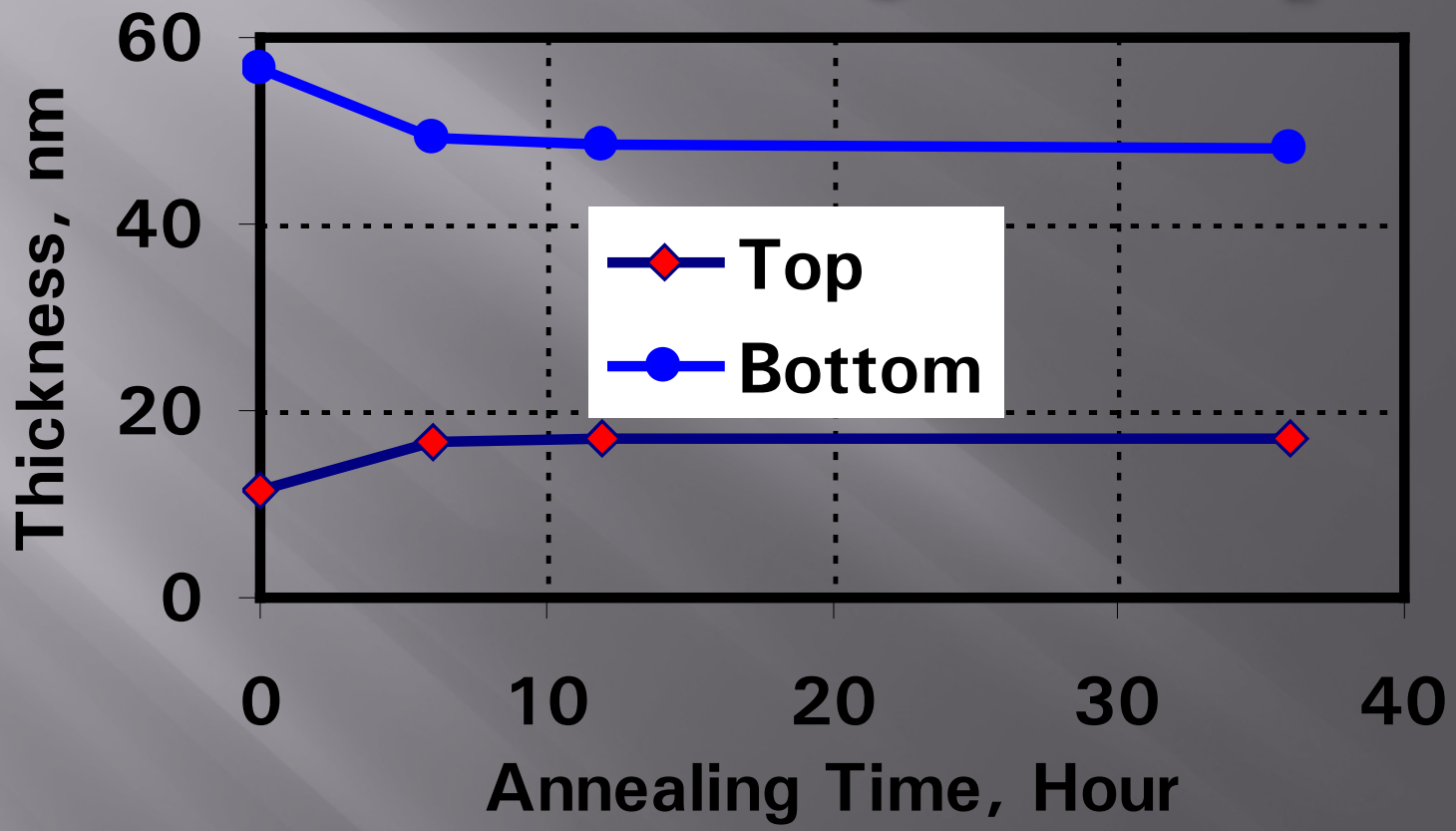
BF TEM Image shows TiO2 sandwiched between SiO2 in real mirror structure reveals the dense part is amorphous and upper part partially consists of crystalline feature.

Recorded Chamber Temperature



Growth structure is related to deposition temperature changes inside chamber

Effect of Annealing on TiO_2 Film



TiO_2 film was annealed at 250°C for different time. Top surface layer thickness increases at beginning.

Summary

- Spectroscopic ellipsometry has been successfully used to characterize the structural inhomogeneity of TiO_2 films.
- Three sub-layer model need to be used in SE analysis to describe the inhomogeneity of the as-deposited TiO_2 films. In such model, top layer is defined as surface roughness with EMA. Both middle and bottom layers are defined with Forouhi Interband transition plus one absorption band in UV-Vis range.
- Three sublayer model was confirmed by SEM and AFM observation, which corresponds to rough surface, columnar growth structure and dense part at film bottom.
- It is found that such structural inhomogeneity is related to the chamber temperature changes during the deposition.
- With TEM analysis, film grown at early stage mainly consists of amorphous structure which is dense and has higher index. While it grown in columnar structure at higher deposition temperature which contains partial crystalline phase with relatively lower index.
- Further annealing at 250^{o}C will increase surface roughness due to crystallization of the film.

Further Information Available

- www.angstec.com
- info@angstec.com
- Phone: (978) 263-6678
- Fax: (978) 263-6675