# Characterization of thin films and stack in MOEMS structure with ellipsometry and reflectometry techniques

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# ABSTRACT

Nondestructive characterization on thin films and their stack in MOEMS device is highly desirable. But, it is often a challenging task because the area is usually small. During processing of thin films, the deposition rates, optical properties, and mechanical properties must be fully understood to fabricate a device with desired performance. With the patterned surface, deposition rate of a typical physical vapor deposition (PVD) technique, such as electron-beam evaporation and sputtering, varies at different location due to shadow effect. In this study, spectroscopic ellipsometry and reflectometry were used to characterize the optical properties of electron-evaporation thin films on a flat substrate. On the other hand, microreflectometer was used to monitor the spectrum of deposited multi-stack of optical thin films inside via-holes. Combination of these two techniques provides a practical way to qualify the processing and ensure the device performance.

**Keywords:** Spectroscopic Ellipsometer, Microspectrophotometer, e-beam evaporation, VCSEL, MOEMS, Optical coatings, Titanium oxide, Silicon Dioxide

# 1. INTRODUCTION

Property control of optical thin films in micro electromechanical system (MEMS) devices is very important to achieve the designed performance. In tunable vertical cavity surface emission laser (TVCSEL), two optical mirrors, namely top and bottom mirrors, are used to tune the wavelength through changing the cavity length between these two mirrors. Output of a laser beam quality is controlled by top mirror geometry and transmission behavior, whereas the output power is closely related to the transmission of the bottom mirror. In addition, equally important thing is that the optical alignment of the top and bottom mirror spectrums in process. The first minimum peak on the left side of a mirror spectrum, fist stop band, is most sensitive to the optical thickness changes of a film, and therefore used to characterize the spectrum position. For the convenience of system integration (alignment), the bottom mirror needs to be deposited inside a via-holes structure, in which its size is typically around a few hundreds of microns. Such a design arises potential changes of physical thickness and refractive index of deposited films inside and outside of via holes. It poses challenges of how to detect such a difference non-destructively and subsequently how to control the film properties in deposition process.

In this study, we evaluated the property difference of optical thin films inside and outside via-holes for single  $SiO_2$  and  $TiO_2$  films. Based on real requirements, a method was developed to quality bottom mirror and thus to control the device performance.

# 2. EXPERIMENTAL PROCEDURES

### **2.1 Films Preparation**

 $TiO_2$  and  $SiO_2$  films were deposited by the technique of ion-assisted e-beam evaporation. Substrates used for the experiments were silicon wafers with via holes etched by the photolithography method. The standard via-hole size was shown in Figure 1. To minimize the temperature gradient during the evaporation process, the vacuum chamber was preheated to 150 °C by a quartz-halogen lamp. A thermal couple was placed near the sample holder to monitor the chamber temperature. Two MDC e-guns with 270° beam deflection were installed at the bottom of the vacuum

chamber to evaporate  $TiO_2$  and  $SiO_2$  materials. The source materials were pre-melted  $Ti_2O_3$  tablets and a pure  $SiO_2$  disc. The deposition rates were 2.0Å/s and 3.0Å/s for  $TiO_2$  and  $SiO_2$ , respectively, which were monitored and controlled by three quartz crystals.

An MARK II ion gun was used to assist the deposition process for both SiO<sub>2</sub> and TiO<sub>2</sub> films. The input gas for the ion gun was a mixture of 90 % Argon and 10 % oxygen in volume fraction. The chamber was first pumped down by a mechanical pump and then followed by a 10-inch cryopump to the base pressure  $< 2.0 \times 10^{-6}$  Torr. All process gases were delivered into the chamber by MKS mass flow controllers. The process pressure of the e-beam deposition was maintained at  $2.5 \times 10^{-4}$  Torr by adjusting the oxygen flow versus a close-loop control. The oxygen partial pressure was maintained at  $0.6 - 1.0 \times 10^{-4}$  Torr in this research.

# 2.2 Characterization

Spectroscopic ellipsometry is a nondestructive optical technique<sup>1-6</sup>. With the ellipsometry method, information on film thickness, surface roughness, and optical constants can be calculated by building some proper optical models of film stacks. The assumed film or layer properties (optical, physical, chemical etc.) are adapted in such a model. Then, this model is mathematically treated based on interaction between electromagnetic wave and matter. Calculated results from the models are compared with the ellipsometry angles of Psi and Delta that are experimentally determined. The quality of match between the experimental and modeled data is usually measured by a merit function, such as mean-squared error or overall deviations.

In this study, a variable angle spectroscopic ellipsometer was used to characterize the deposited films. It is a very useful tool to understand the film properties. A typical beam size is 1 to 5 mm. In MOMEMS application, the beam size must be smaller than feature size. With a focused beam, the probing area can be reduced to about 200 $\mu$ m in beam lateral direction. The size in beam propagation direction is dependent on the incident angles. Due to a high aspect ratio of via holes, it is difficult to apply ellipsometry probing beam to via-hole bottom. Therefore, we only apply ellipsometry to either blank wafer or top part of MOMEMS device (outside of Via-hole). It is obvious that another tool is needed to characterize the film or stack inside via holes. Microspectrophotometer (MSP) is an ideal tool for measuring reflection or transmission over a region as small as 8  $\mu$ m in diameter. In addition, for device quality control, this tool is directly used to monitor the bottom mirror reflection spectrum and thus is used to monitor the device performance. For this purpose, a tooling factor was defined by characteristic band position shift inside via-hole to outside of a via hole region.



Figure 1. Via hole geometry with top and side views (left side: top view; right side: side view)

### 2.3 Microstructure Analysis

Surface and cross-section morphologies of as-deposited thin films were analyzed by a HITACHI S4700 emission field scanning electronic microscope (EFSEM) and transmission electronic microscope (TEM).

# 3. RESULT AND DISCUSSION

#### Single layer study

Sets of single layer samples were deposited at different quarter wavelength optical thickness (QWOT). In this research, a parameter of tooling factor (TF) was introduced to describe the thickness difference of a thin film deposited inside and outside via holes. The TF is defined as  $(T_{in}-T_{out})/T_{out}$ , where  $T_{in}$  and  $T_{out}$  are film thickness inside and outside via holes. The witness sample thickness was measured with ellipsometer under parallel beam and thickness at outside of via-holes was measured with focused small beam in ellipsometer. The thickness inside via hole was measured with microspectrophotometer. It is found that a SiO<sub>2</sub> film at outside via hole is around 10% thicker than that of inside via holes (220µm×176µm). For a TiO<sub>2</sub> films, the thickness difference is a function of thickness itself. In general, the thicker the TiO<sub>2</sub> film, the smaller the difference in thickness between inside and outside via holes. The thickness difference for single layer samples is given in Table 1. In addition, with the via-hole size decreases, the difference becomes bigger for both SiO<sub>2</sub> and TiO<sub>2</sub> films. But it seems that TiO<sub>2</sub> changes more than SiO<sub>2</sub> films.

Table 1 Thickness difference for studied single layer samples

Sample ID	Design	Witness	Outside	Inside	<b>TF</b> (%)	Via-Hole Size (µm)
SiO2-1	1QWOT	265.2nm	267.8nm	241.9nm	-9.7	220x176
SiO2-2	2QWOT	510.2nm	535.0nm	482.0nm	-9.9	220x176
SiO2-3	3QWOT	791.4nm	799.2nm	711.2nm	-11	108x93
TiO2-1	1QWOT	164.9nm	171.4nm	153.3nm	-10.6	220x176
TiO2-2	2QWOT	316.2nm	343.0nm	302.0nm	-12.0	220x176
TiO2-3	3QWOT	533.6nm	548.4nm	471.4nm	-14.0	108x93

It is generally believed that such a thickness difference between inside and outside via holes is related to the shadow effect of electron evaporation process. However, a slightly big change in thickness for  $TiO_2$  film is believed to be related to deposition chamber temperature effect. In our previous studies, it has been found that  $SiO_2$  film properties are less affected by chamber temperature changes, while  $TiO_2$  film growth behavior is much relied on chamber and substrate temperatures<sup>7,8</sup>. When growing thicker films, both chamber temperature and substrate temperature will arise and thus cause growth structure changes and also crystallinity difference.

Refractive index difference between inside and outside via hole was also evaluated with ellipsometry (for witness and outside via-hole) and microspectrophotometry (for inside via-holes). It was found that the index inside via holes is higher than that of outside via holes, which may be related to a slower deposition rate that leads to a denser film inside the via hole, see Table 2.

Sample ID	Design	Witness		Outside Via		Inside Via		Index Changes (%)	
		n at 633	n at 1550	n at 633	n at 1550	n at 633	n at 1550	n at 633	n at 1550
TiO2-1	1QWOT	2.358	2.290	2.506	2.509	2.556	2.672	2.0	6.5
TiO2-2	2QWOT	2.388	2.329	2.373	2.371	2.456	2.424	3.5	2.2
TiO2-3	3QWOT	2.307	2.233	2.251	2.221	2.320	2.298	3.0	3.5

Table 2. Refractive index difference between inside and outside via holes

#### Via-hole size effect on reflection spectrum

If the shadow effect causes the thickness difference of a film deposited inside and outside via holes, then it is expected that such a difference should be a function of via hole side. To confirm this assumption, some multi-stack  $SiO_2$ -TiO<sub>2</sub> layers were deposited on the via hole substrate, so that it is able to directly measure the reflection spectrum of an as-deposited film inside and outside the via hole with a microspectrophotometer (MSP). Similar spectrum profiles from inside and outside via holes are shown in Figure 2, in which the inside spectrum shifts to the short wavelength direction, indicating a thinner stack thickness. The tooling factor (TF) keeps the same formula as before, but with the characteristic band positions (nm) instead of the film thickness at this time. It was found that the

TF for the spectra shift is a function of the via-hole size (geometry), Figure 3. It had also been found that the laser output power is sensitive to the band position of the bottom mirror inside the via holes, therefore, monitoring such a spectral shift is very critical to ensure the proper alignment of the bottom mirror profile to that of the top mirror in order to maximize the device performance in the interested region.

In such a way, by establishing the empirical relationships between device performance, band position and processing conditions, the design and processing control become possible.



Figure 2 Characteristic band position shift from inside via-hole to outside via-hole



Figure 3 The characteristic band position shift (tooling factor) vs. via-hole size (in area)

#### Microscopic cross-section observation

Morphology of the processed films was inspected with scanning electron microscopy and transmission electron microscopy. It has confirmed the measurement data with nondestructive ellipsometry and microspectrophotometry techniques. For single layer  $SiO_2$  films, a typical cross-section inside and outside the via-holes is shown in Figure 4. Because of amorphous feature and cleavage fracture surface, there are no significant features on the inspected surface. However, for  $TiO_2$  films, beside the thickness difference, the inhomogeneous information is obtained. This specific topic has been reported before. The growth structure changes from beginning to end. As stated before, this is related to the chamber growth temperature changes. On the via-hole wall, the thickness is even thinner than bottom. This might be related to the sticking efficiency on the tiled surface.



Figure 2. SEM cross-section view of SiO<sub>2</sub> film deposited outside (left) and inside (right) a via hole.



Figure 3 SEM cross section of TiO<sub>2</sub> film deposited outside (top left), inside (top right) and via-hole wall (bottom left and right)



Figure 6 TEM bright field image for bottom mirror taken from outside via (left) and inside (right) via-hole



Figure 7. High magnification TEM photos for mirror shown in Figure 6.

# 4. SUMMARY

The combination of non-destructive ellipsometry and microspectrophotometry techniques make it possible in practice to characterize the geometry effect on thickness and optical properties of films deposited inside and outside a via-hole structure or other patterned devices. The difference in film thickness inside and outside of via holes of single  $SiO_2$  and  $TiO_2$  layers were investigated. In all cases, the film thickness inside the via hole is thinner than that of outside, which is believed due to the shadow effect of the electron-evaporation process. A similar trend of thinner stack thickness of  $SiO_2$ -TiO<sub>2</sub> multi-layer inside via holes was also found by the microspectrophotometer

measurement, resulting in the spectral shift toward to the short wavelength direction. Monitoring such a spectral shift is very critical to ensure the proper alignment of the bottom mirror profile to that of the top mirror in order to maximize the device performance in the interested region.

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