

# Atmospheric Stability of E-Beam Deposited Optical Thin Film Stacks

Ping Hou

Nortel Networks Corp.

Richard Sun and Frank Luo

Angstrom Sun Technologies Inc., Acton, MA

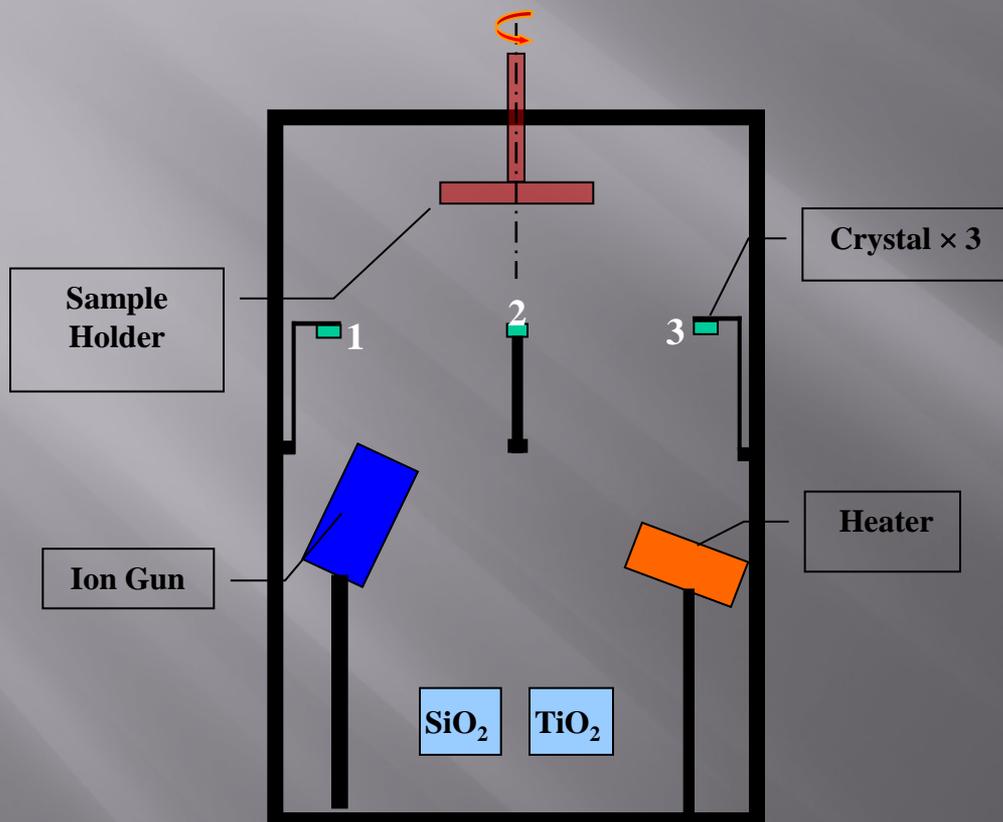
# Introduction

Preventing undesirable changes in mechanical, micro-structural and optical properties for functional thin films remains a challenge. Stress control of as-deposited thin films is one of the critical requirements for achieving optical, electronic, magnetic, and mechanical performances in the state-of-art MOEMS devices. Stress of thin films can be harmful or useful, depending on the applications. Undesired in most of cases, residual stress in thin films may cause structural deformation, cracking, buckling or even delamination. Yet stress may be beneficial to optimize performance in some of optical devices, such as the top mirror with a dome shape formed by precisely controlled residual stress in the tunable vertical cavity surface emission laser (TVCSEL) devices.

Controlling and stabilizing the film stress in a high-performance MOEMS device can present challenges during the fabrication process and in its application environments. In general, the residual stress ( $\sigma$ ) of a thin film can include thermal stress ( $\sigma_T$ ), intrinsic stress ( $\sigma_I$ ), and epitaxial stress ( $\sigma_E$ ), which can be expressed as  $\sigma = \sigma_T + \sigma_I + \sigma_E$ . Thermal stress arises from the temperature changes due to differential thermal expansion between different materials. This kind of stress is almost inevitable in reality when different materials are used. The intrinsic stress is more related to the film growth techniques and can be tuned to certain content in process. The effect of epitaxial stress caused by the lattice mismatch of different materials is more obvious when the film is sufficient thin so that it has a perfect coherent interface with its substrate.

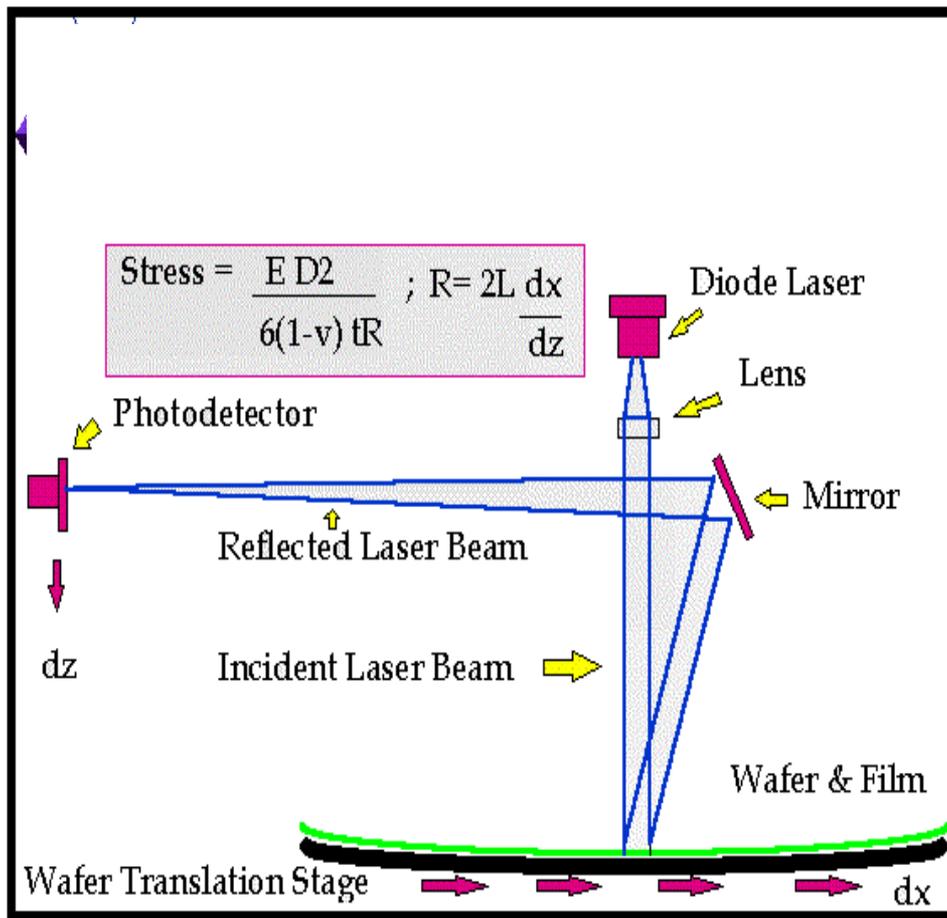
In this paper, the stress evolution of an optical stack ( $\text{SiO}_2\text{-TiO}_2$ ) used in a MOEMS device had been investigated in the atmospheric environment.

# SiO<sub>2</sub>-TiO<sub>2</sub> Stack Was Grown by E-Beam Evaporation with Ion-Beam Assistant Deposition



Thin films of SiO<sub>2</sub>-TiO<sub>2</sub> stack were deposited by the technique of ion-beam-assisted e-beam evaporation at  $2.5 \times 10^{-4}$  Torr. Two MDC e-guns installed at the bottom of the vacuum chamber were used to evaporate SiO<sub>2</sub> and TiO<sub>2</sub> materials, respectively. An MARK II ion gun with a hollow cathode neutralizer was employed to assist the evaporation process. Substrates used for the experiments were 2-inch single-side-polished silicon wafers with. Samples were mounted onto an 8-inch copper holder that rotates vertically at the speed of 32 rpm. To minimize the temperature gradient during the deposition process, the chamber was pre-heated to 150 °C by a quartz-halogen lamp.

# Film Stress Measurement

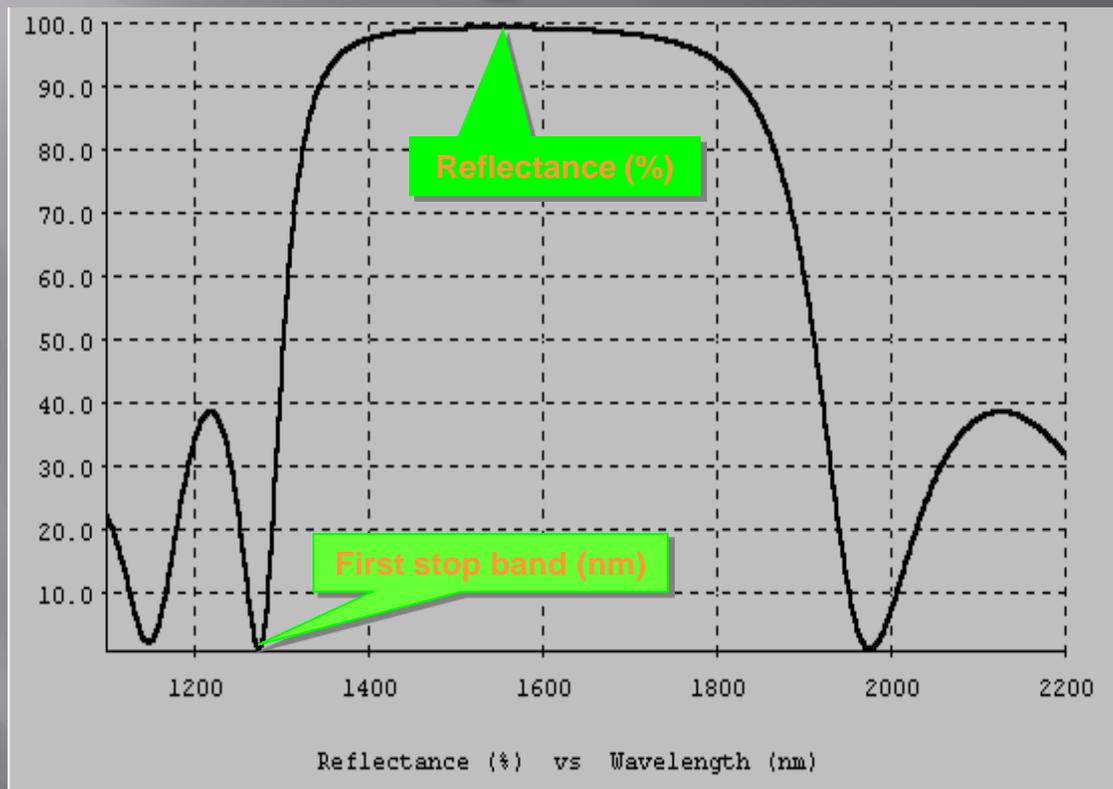


Film stress was measured by a FSM128 stress measurement system. This instrument utilizes a laser optical lever to measure the curvature change of a film stack on a thick substrate. The curvature of a blank silicon wafer was initially measured and recorded as a reference. Then a second measurement was conducted after the e-beam deposition on the same wafer to determine the curvature change induced by the film stress. Stress of a thin film is calculated based on the Stoney's equation:

$$\sigma = \frac{E}{6(1-\nu)} \cdot \frac{t_s^2}{t_f} \left( \frac{1}{R} - \frac{1}{R_0} \right)$$

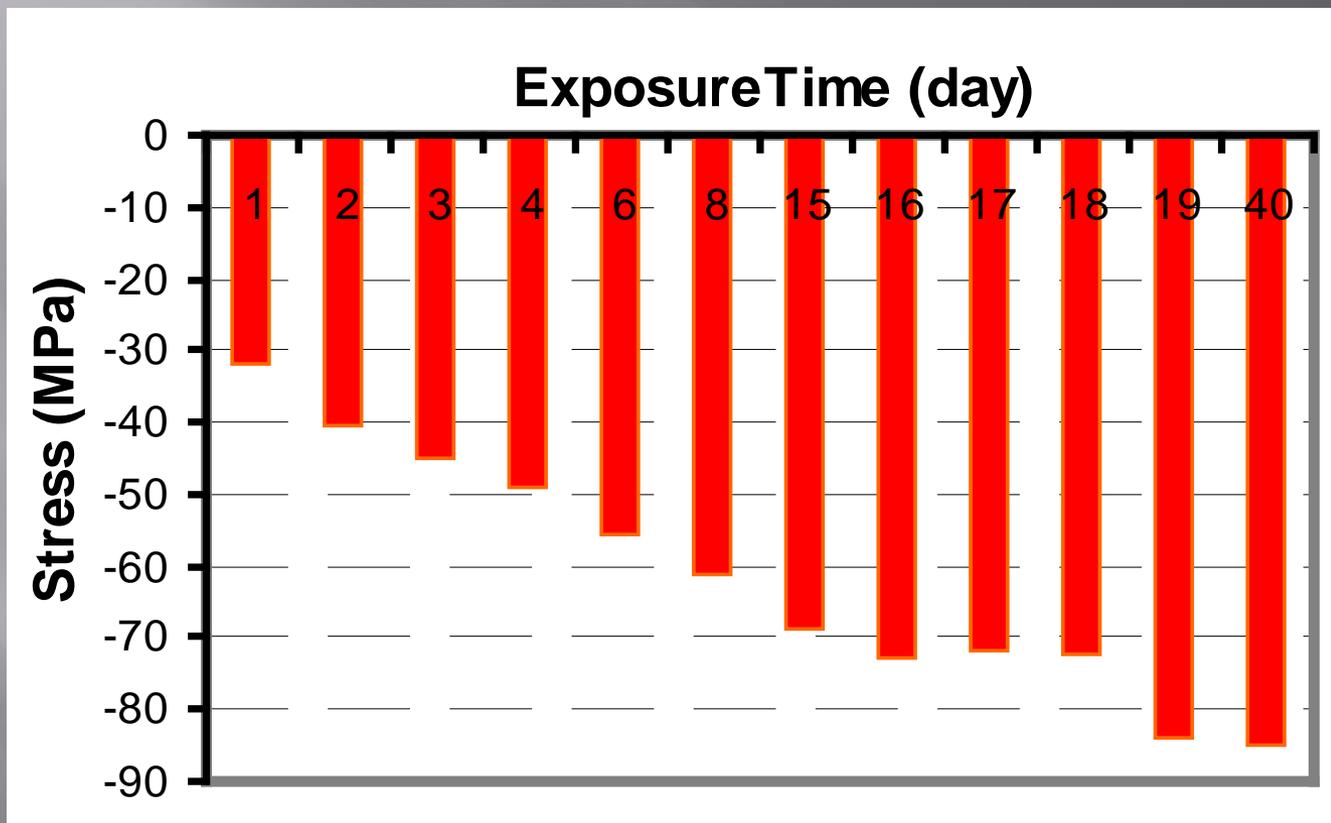
where  $E(1-\nu)$  is the biaxial elastic constants for the substrate, which is 180.5GPa for the (100) Si<sup>5</sup>.  $t_s$  and  $t_f$  are the substrate and thin film thicknesses.  $R$  and  $R_0$  are the substrate curvature measured before and after deposition.

# Optical Spectral Profile of SiO<sub>2</sub>-TiO<sub>2</sub> Stack



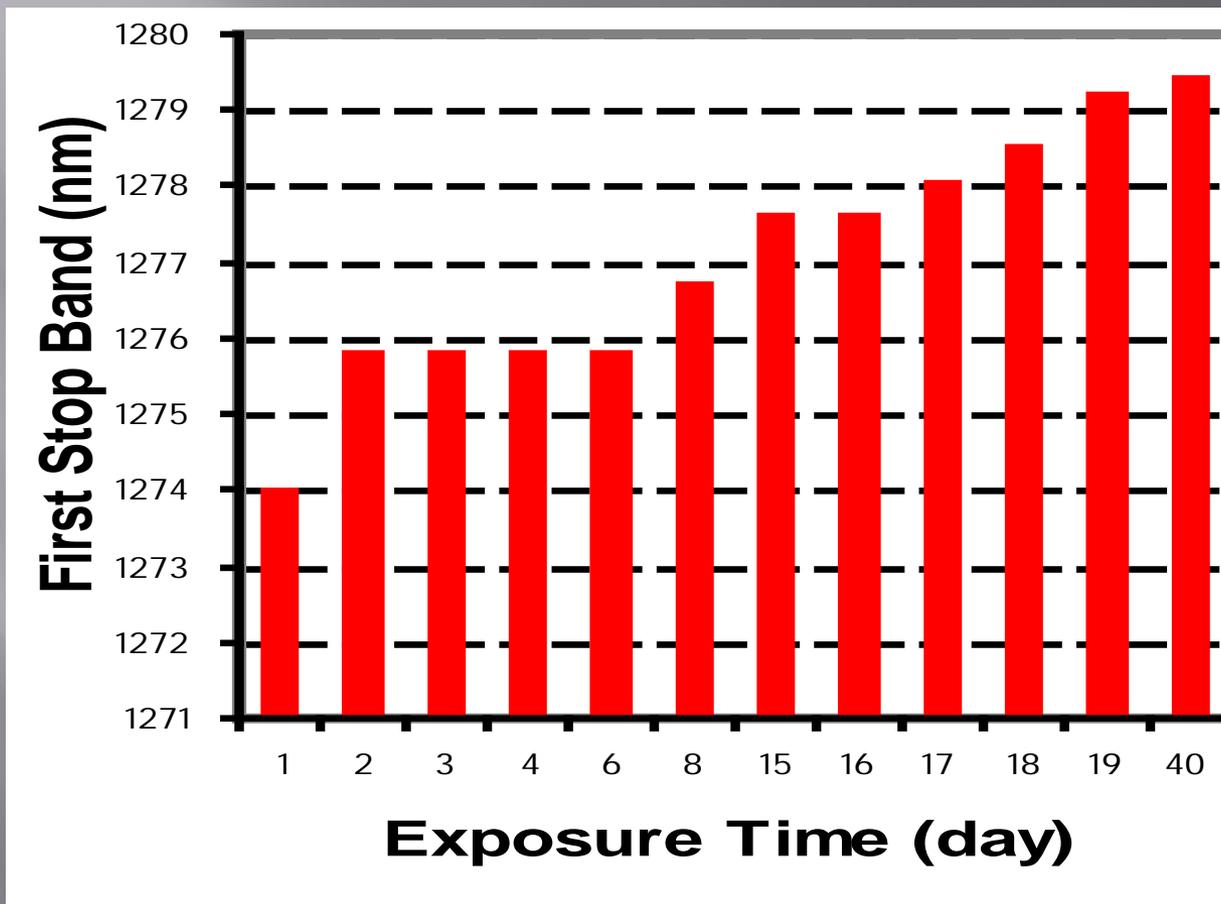
The spectrum of the SiO<sub>2</sub>-TiO<sub>2</sub> stack was scanned by a spectrophotometer (Cary500). A characteristic band position (first stop band) and the maximum reflectance (%) were used to identify the optical spectrum profile changes as the function of exposing time in the air.

# Atmospheric Environment Exposure of SiO<sub>2</sub>-TiO<sub>2</sub> Stack Shows Incremental Compressive Stress.



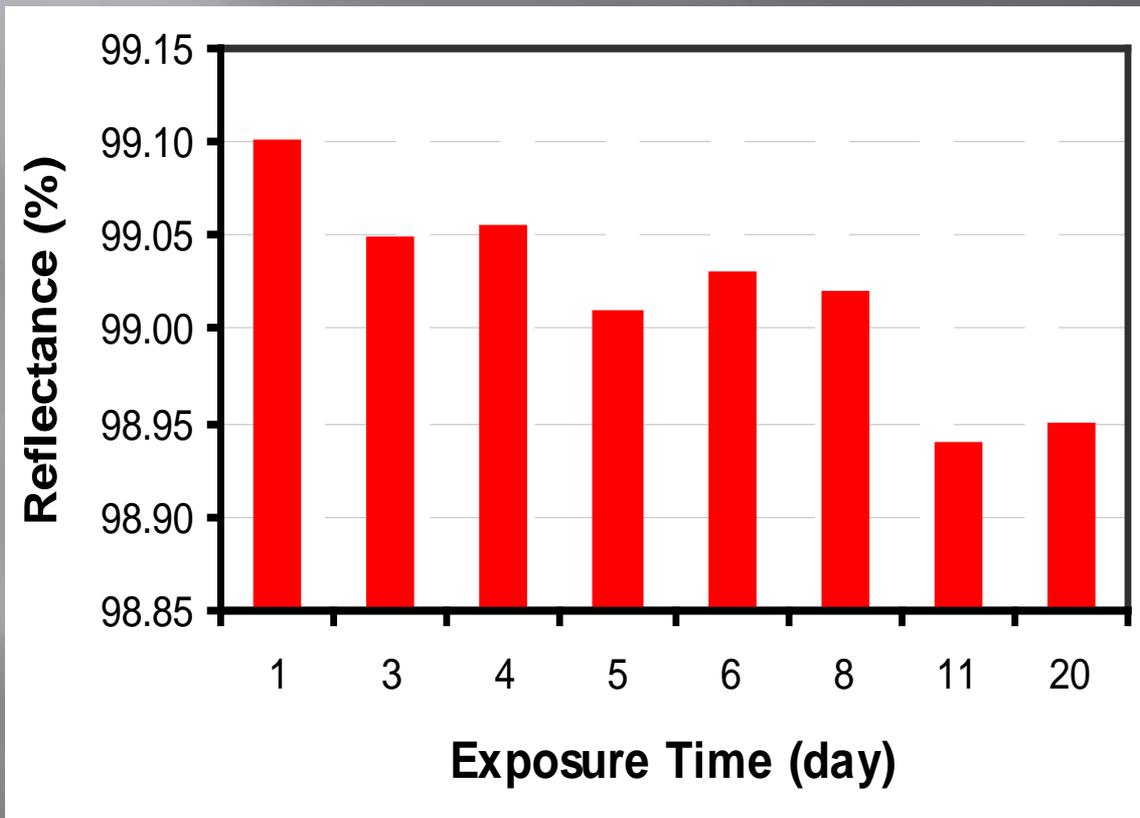
The SiO<sub>2</sub>-TiO<sub>2</sub> stack mirror was kept in the atmospheric clean-room environment and the stress variation was measured in certain amount of time intervals. The film stress shows the compressive stress (negative value), and appears to become incremental compressive along with the exposure time in the air.

# Atmospheric Environment Exposure of SiO<sub>2</sub>-TiO<sub>2</sub> Stack Increases the First-stop-Band Position.



The first-stop-band position gradually shifts to the long wavelength direction when the SiO<sub>2</sub>-TiO<sub>2</sub> stack exposes to the air for certain amount of time.

## Atmospheric Environment Exposure of $\text{SiO}_2\text{-TiO}_2$ Stack Shows Decreased Reflectance.

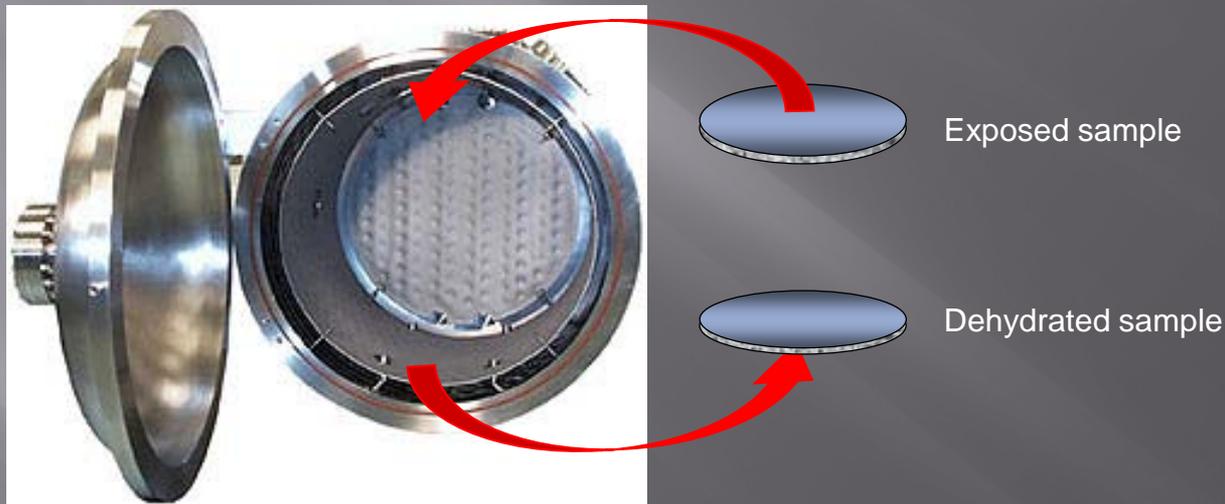


Exposing to the atmospheric environment causes the decrease of reflectance in the six-pair  $\text{SiO}_2\text{-TiO}_2$  reflective mirror.

# Water Adsorption in the Films?

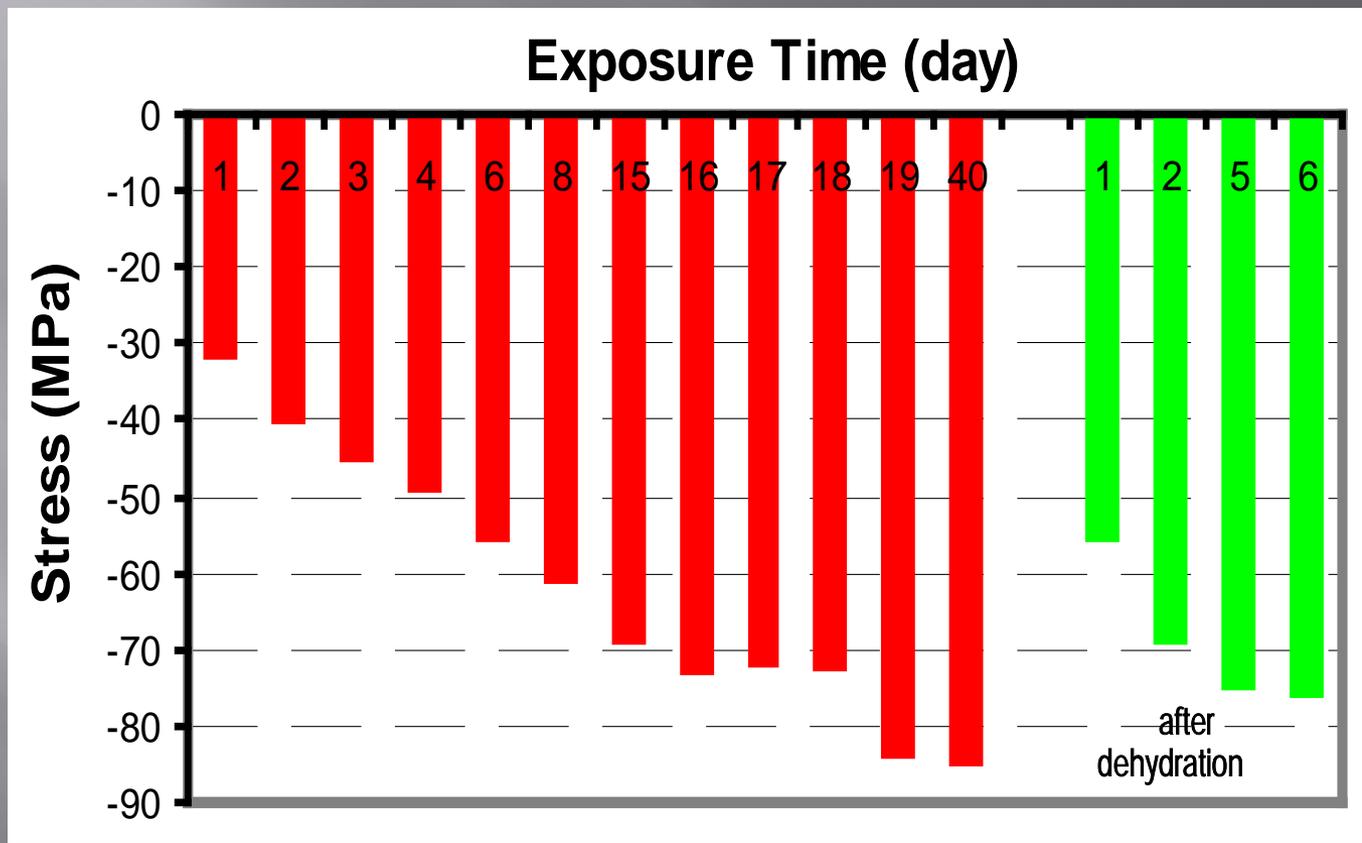


A decreasing trend in stress would be expected if the structural relaxation dominated the film stress evolution. Such an increasing trend of compressive stress leads to the reasonable assumption of water absorption into the films.



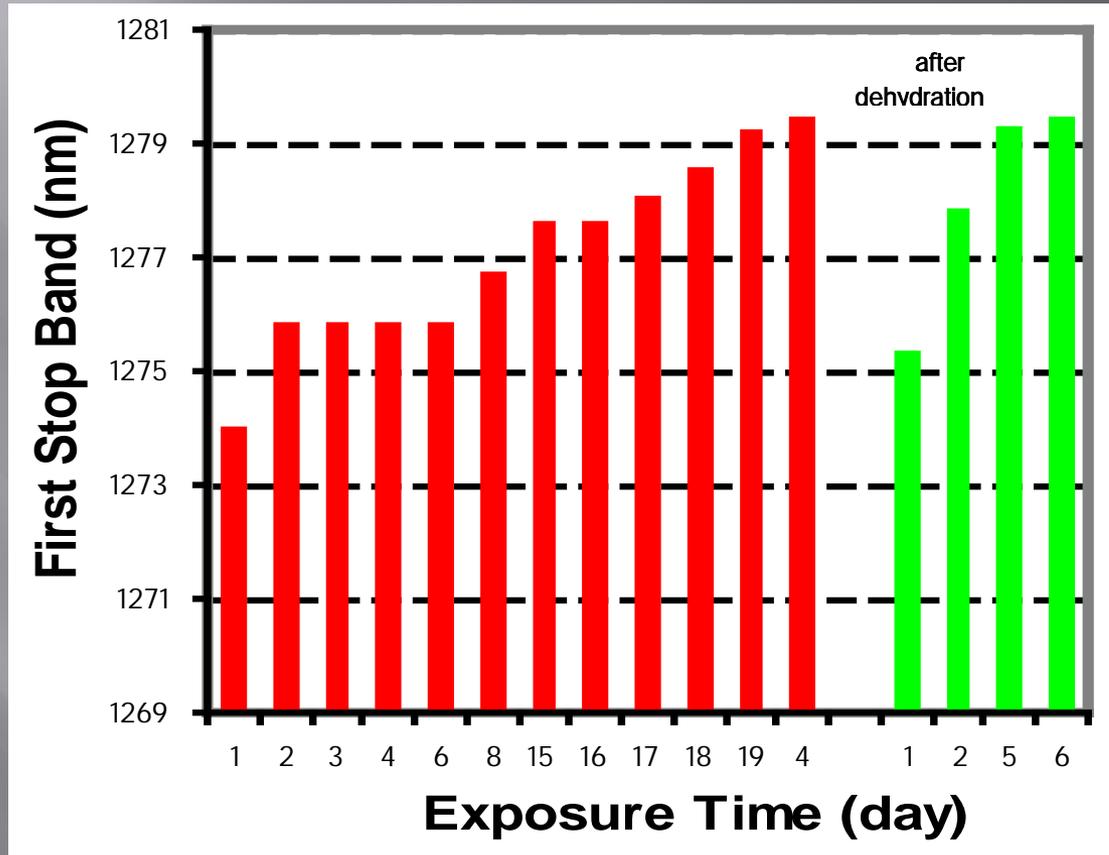
The same atmospherically exposed sample was dehydrated in a vacuum chamber ( $2.0 \times 10^{-6}$  Torr) for 48 hours. In addition, the chamber was also heated up to  $80^\circ\text{C}$  for the last 8 hours to facilitate the water-removing process.

# Dehydrated Film Stress Follows the Similar Changes as Atmospherically Exposed Sample.



The film stress right after the dehydration treatment comes down close to its original level, and then it follows the similar increasing trend when exposed to the air again, indicating the re-adsorption process of water. The incorporation of water into the films appears to enhance the “expanding” tendency and therefore results in a higher compress stress. Removing of water from the films causes the recover of the stress to a certain degree.

# FSB Position after Dehydration also follows the Similar Variation Trend as before.



Again, after the film dehydration in the vacuum chamber, a similar water desorption-adsorption cycle was found for the first-stop-band position change.



# Why Spectrum Changes?

For a reflective mirror with quarter-wavelength multi-layers, the maximum reflectance and the bandwidth are related to the index contrast of the two materials used. A higher index contrast results in a higher reflectance and a wider bandwidth. The shift of the first-stop-band position and the decrease of the reflectance imply the shrinkage of the bandwidth, indicating an unevenly index changes of these two materials ( $\text{SiO}_2$  and  $\text{TiO}_2$ ).

# Absorption of Water into Films Causes the Effective Refractive Index Changes.

Based on effective media approximation (EMA) model, the effective dielectric constants for a multi-component thin film can be expressed as:

$$0 = f_a \frac{\varepsilon_a - \varepsilon}{\varepsilon_a + 2\varepsilon} + f_b \frac{\varepsilon_b - \varepsilon}{\varepsilon_b + 2\varepsilon}$$

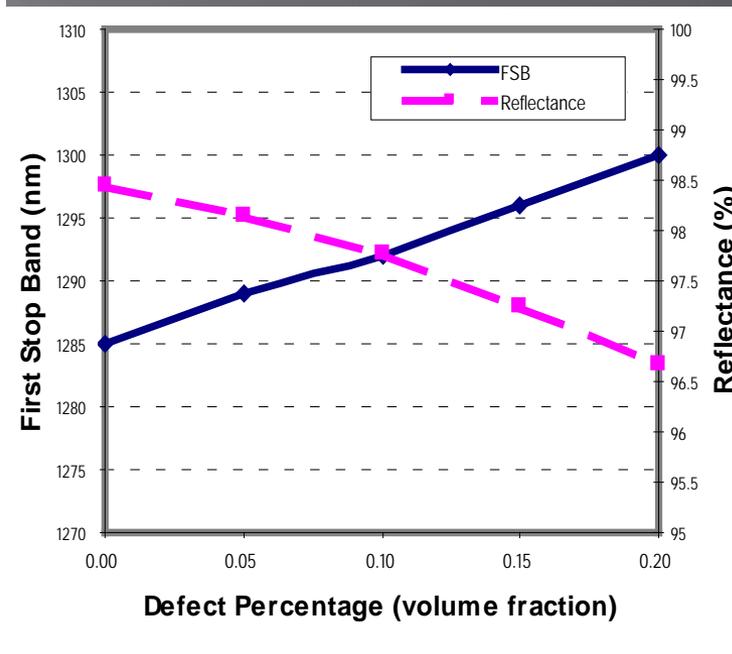
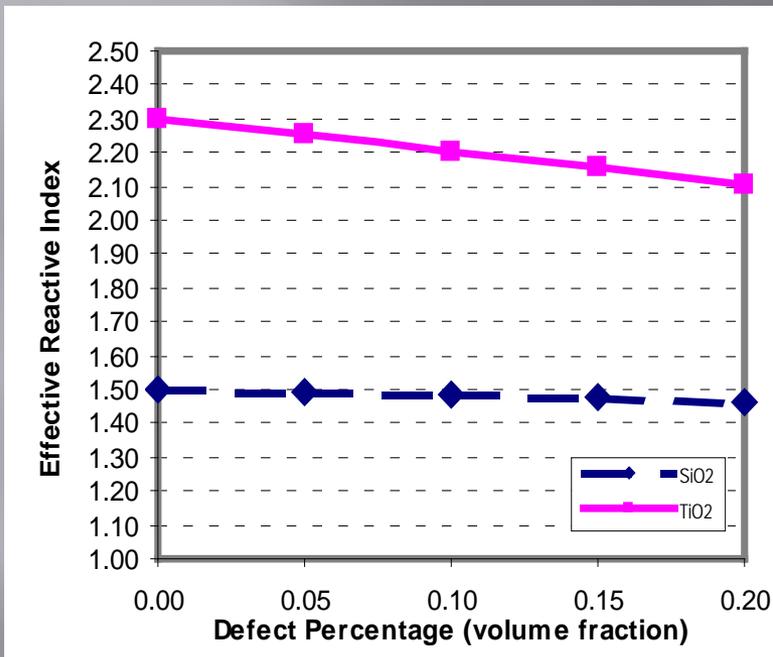
where  $f_a$ ,  $f_b$ ,  $\varepsilon_a$ , and  $\varepsilon_b$  are volume fractions and dielectric constants for components  $a$  and  $b$ , respectively.  $\varepsilon$  is an effective dielectric constant for the composite with component  $a$  and  $b$ , which can be expressed as  $\varepsilon = (n^2 - k^2) - 2nki$ .  $n$  and  $k$  are the refractive index and the extinction coefficient, respectively.

For dielectric films such as  $\text{SiO}_2$  and  $\text{TiO}_2$ , there is no significant absorption ( $k=0$ ), therefore, above equation can be simplified as:

$$0 = f_a \frac{n_a^2 - n^2}{n_a^2 + 2n^2} + f_b \frac{n_b^2 - n^2}{n_b^2 + 2n^2}$$

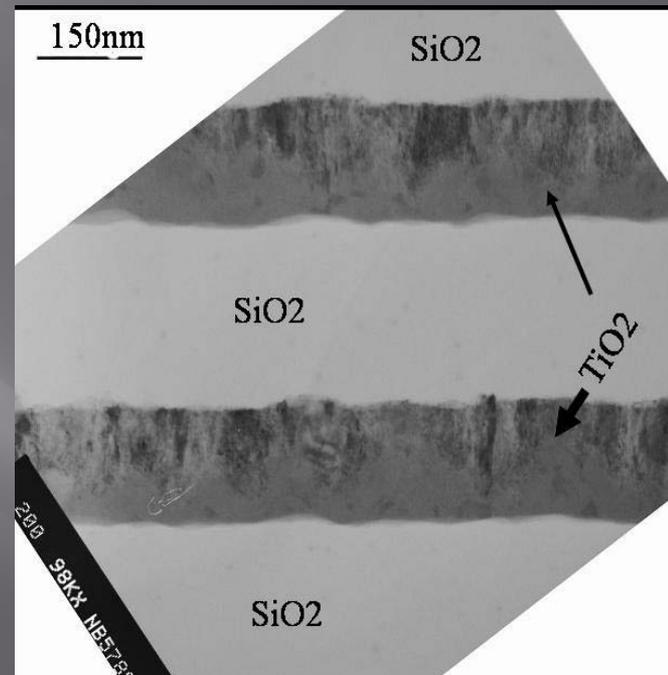
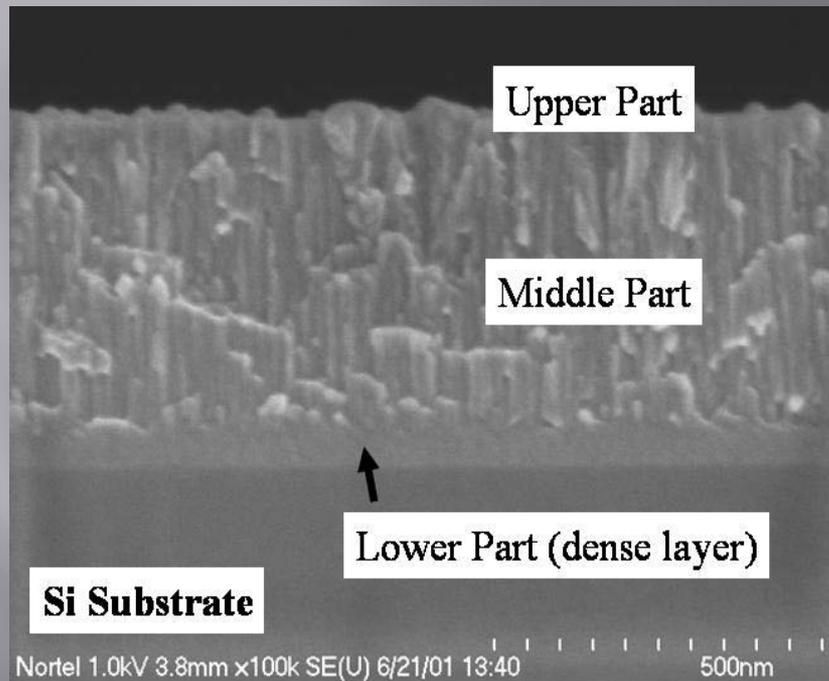
where  $n_a$  and  $n_b$  are refractive index for component  $a$  and  $b$ , respectively.  $n$  is the effective refractive index for the film.

# Theoretic Simulations Show Similar Variation Trends as Experimental Observations.



To better understand the effect of water adsorption on the optical property variations, theoretical simulations had been conducted using the software TFCalc. The refractive indexes of solid SiO<sub>2</sub> and TiO<sub>2</sub> layers are assumed to be 1.50 and 2.30, respectively. Given the same amount of structural defect in both SiO<sub>2</sub> and TiO<sub>2</sub> layers, the changes of the effective refractive index are calculated as the function of air volume fraction in the films. The right figure shows that the decreasing rate of the effective refractive index in the TiO<sub>2</sub> layer is greater than that of the SiO<sub>2</sub> layer, resulting in a reduced index contrast. Accordingly, the variations of the first-stop-band position and the maximum reflectance were also simulated based on the calculated effective refractive indexes at different defect levels (left figure).

# Microscopic Study Show Different Structures of SiO<sub>2</sub> and TiO<sub>2</sub> Layers.



(a) SEM photo showing three sub-layers of an as-deposited TiO<sub>2</sub> film (left); (b) TEM photo for alternating SiO<sub>2</sub>/TiO<sub>2</sub> multiple layers shows the same structure for TiO<sub>2</sub> single layer (right). Therefore, in reality, the effective reflective index of the TiO<sub>2</sub> layer may reduce more than that of the SiO<sub>2</sub> layer, leading to an even smaller index contrast between these two materials.

# Summary

---

The adsorption of water vapor into the micro-defects in the film stack is believed to be the root cause for the evolution of film stress and optical spectrum in the atmospheric environments. It is well known that the output power of a MOEMS tunable laser device is sensitive to the optical alignment of the two mirrors spectra within the interested wavelength regions. Such a spectral profile change may then reduce the device performance. Therefore, a practical way of improving the stability for as-deposited mirror stack is to increase the film packing density, which can be achieved by using the method of ion beam assistant deposition.