

# IMPACT OF EVAPORATION RATE ON MULTI-LAYER ANTIREFLECTIVE FILM QUALITY USING SiO, /SiO MATERIAL PAIRS

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## Abstract:

This paper presents the results of research, calculation, manufacturing of three-layer antireflective coating using SiO\_/SiO material pairs on BOC ADWARDS FL500 vacuum evaporator. The results are used to evaluate the effect of film evaporation rate on the quality of multi-layered anti-reflective coating, in the near infrared and short wavelength infrared region.

Keywords: Evaporation rate, SiO<sub>2</sub>/SiO antireflective film, optical thin film quality.

#### 1. Introduction

The quality of optical thin film includes optical properties:  $T(\lambda)$ ,  $R(\lambda)$ ,  $A(\lambda)$ ; Mechanical properties: adhesion, hardness; Spatial characteristics: uniformity and full-surface optical properties [2].

The quality of optical thin film is shown by the adhesion strength of optical thin film and what is the transmission coefficient of film. Durability represents the stability of the thin film and is created by the force that binds the film molecules to the film molecules, between the film molecules and the substrates. Adhesion is the ability of the film to adhere to the surface of the film, due to the force that binds the film molecules to the substrate. Both adhesion and adhere properties depend on many different factors [3]:

- Condition and detailed materials of substrate are coated.

- Coating materials, film thickness, number of layers of the film (the thickness of the film will determine the properties of the thin film).

- Difference between film and substrate in coefficient of thermal expansion, stress, ...

- Coating manufacturing technology conditions: Cleaning conditions, vacuum chamber pressure, deposition velocity, evaporation rate, substrate temperature during deposition film, annealing time, ...

- Environmental conditions and time to use film: Pressure, temperature and humidity.

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In this paper, the author will test the effect of evaporation rate on the quality of antireflective coating.

# 2. Calculation and design of antireflective coatings.

### 2.1. Theoretical basis

Consider the thin film system consisting of L-layers shown in Fig 2.1. The construction parameters comprise not noly the refractive  $n_j$  and thickness  $d_j$  of the layers and plating on substrates have refractive index  $n_s$ , thin films exposed to  $n_m$ refractive index medium.



Figure 2.1. The L-layer thin films: from  $1 \dots j \dots L$ , the index of each layer  $n_j$ , the real thickness for the layer  $d_j$ 

Assuming the thin film has L layers 1... j... L as shown in Fig 2.1 each layer has refractive index n<sub>j</sub>, thickness d<sub>j</sub>, refractive index of substrate n<sub>s</sub>, refractive index medium of transmittance is n<sub>m</sub>, incident angle  $\theta$ , wavelength light  $\lambda$ . The amplitude of the reflection coefficient r and the transmittance t is determined by the following formulas[4]:

$$r = \frac{\eta_m E_m - H_m}{\eta_m E_m + H_m} \tag{2.1}$$

and

$$t = \frac{2\eta_m}{\eta_m E_m + H_m} \tag{2.2}$$

Where: 
$$\begin{pmatrix} E_m \\ H_m \end{pmatrix} = M \begin{pmatrix} 1 \\ \eta_s \end{pmatrix}$$
 (2.3)

 $E_m$ ,  $H_m$  are the electric and magnetic vector, respectively, in the incident medium, and M is a product matrix given by [4]:

$$M = M_{L}.M_{L-1}...M_{j}...M_{2}.M_{1}$$
(2.4)

In the above formula, the  $M_j$  matrix is a 2x2 level matrix of the jth film of system [4]:

$$M_{j} = \begin{pmatrix} m_{11} & im_{12} \\ im_{21} & m_{22} \end{pmatrix} = \begin{pmatrix} \cos \delta_{j} & \frac{i}{n_{j}} \sin \delta_{j} \\ in_{j} \cdot \sin \delta_{j} \cos \delta_{j} \end{pmatrix}$$
(2.5)

Where: 
$$\delta_j = \frac{2\pi}{\lambda} (n_j d_j \cos \theta_j)$$
 (2.6)

With  $n_j d_j \cos \theta_j$  is the effective optical thickness of the jth layer for an refractive angle  $\theta_j$ .

In equation (2.1), (2.3)  $\eta$  represents the admittance of the medium, substrate, or layer and is given by:

$$\eta = \begin{cases} \frac{n}{\cos\theta} & ph\hat{a}ncuc - p \\ n\cos\theta & ph\hat{a}ncuc - s \end{cases}$$
(2.7)

depending on whether the incident radiation is polarized parallel (p) or perpendicular (s) to the plane of incidence. Clearly, for normal incidence of light, the value of the admittance is equal to the refractive index. The angle  $\theta_j$  is related to the angle of incidence  $\theta_0$  by Snell's law:

$$n_m \sin\theta_0 = n_j \sin\theta_j => \theta_j = a \sin\left(n_m \frac{\sin(\theta_0)}{n_j}\right)$$
 (2.8)

The intensity transmittance and reflectance are [4]

$$T = \frac{\eta_s}{\eta_m} \left| t \right|^2 \tag{2.9}$$

$$R = \left| r \right|^2 \tag{2.10}$$

and the phase changes on transmission and reflection,  $\Phi_{T}$  and  $\Phi_{R}$  are given by:

$$\Phi_{\rm T} = \arg\left(t\right) \tag{2.11}$$

$$\Phi_{\rm R} = \arg\left(r\right) \tag{2.12}$$

If the materials in a multilayer are all nonabsorbing, then T + R = 1. If the materials in a multilayer are all nonabsorbing, then T + R = 1. Should one or more materials absorb, then in the above equations the refractive indices of these materials must be replaced by their complex refractive indices  $\tilde{n}$ , defined by  $\tilde{n} = n - ik$ . The absorptance of the multilayer is then calculated from: A=1-T-R. Where k is the extinction coefficient of the material.

### 2.2. Calculation

The three-layer antireflection coatings is structured according to[2]: Air|L HH L|Glass

Refractive index:  $n_1 < n_s$ ,  $n_2 > n_1$ ,  $n_3 < n_s$ (outermost layer has a refractive index smaller than the index substrates, the second layer has the largest index of refraction, the first layer near substrates has a refractive index lower than the index substrates).

The three-layer thin films SiO<sub>2</sub>/SiO/SiO<sub>2</sub>

Input parameters: refractive index of substrates  $n_s=1,5239$ ;  $n_1=1,46(SiO_2)$ ;  $n_2=1,95$  (SiO);  $n_3=1,46(SiO_2)$ , environmental index  $n_0=1$ .

The wavelength range:  $\lambda$ =400...1000 nm, the wavelength central  $\lambda_0$ =550 nm.

Thickness of the film layers: 
$$d_i = \frac{550}{4.n_i}$$
;  
 $d_2 = \frac{550}{2.n_2}$ ;

The incident angle of ray  $\theta_0 = 0$ ;

Using the matrix method we have the calculation results on Mathcad:

$$\begin{split} \lambda &:= 360..1000 \\ n_0 &:= 1 \\ ns &:= 1.5239 \quad n_1 := 1.46 \quad n_2 := 1.95 \quad n_3 := 1.46 \\ i &:= 1..3 \\ d_i &= \frac{550}{4.n_i} \quad d_2 = \frac{550}{2.n_2} \\ \theta_0 &= 0 \\ \theta_i &:= a \sin\left(n_0 . \frac{\sin(\theta_0)}{n_i}\right) \\ \delta_{i,\lambda} &:= 2.\pi.d_i.n_i. \frac{\cos(\theta_i)}{\lambda} \end{split}$$

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$$\eta_{i} \coloneqq n_{i}$$
$$M_{i,\lambda} \coloneqq \begin{pmatrix} \cos(\delta_{i,\lambda}) & \frac{i}{\eta_{i}} \sin(\delta_{i,\lambda}) \\ & \frac{i}{\eta_{i}} \sin(\delta_{i,\lambda}) \\ & in_{i} \cdot \sin(\delta_{i,\lambda}) \cos(\delta_{i,\lambda}) \end{pmatrix}$$

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 $\eta s := ns$ 

$$A(\lambda) \coloneqq \left(\prod_{i=3}^{1} M_{i,\lambda}\right) \cdot \binom{1}{\eta s}$$

$$r(\lambda) \coloneqq \frac{n_0 \cdot A(\lambda)_0 - A(\lambda)_1}{n_0 \cdot A(\lambda)_0 + A(\lambda)_1}$$
$$t(\lambda) \coloneqq \frac{2 \cdot n_0}{n_0 \cdot A(\lambda)_0 + A(\lambda)_1}$$

Thickness of the film layers  $d_1 = 94$ nm;  $d_2 = 141$ nm;  $d_3 = 94$ nm

Graph of reflection coefficient of three-layer antireflection coatings by calculation as shown in Fig 2.2 below:



Figure 2.2. Graph of reflection coefficient of three-layer antireflection coatings SiO/SiO/SiO,

Comment: From the graph of the reflectivity of antireflection coating, it is calculated that the maximum reflectivity of thin film in the wavelength range  $\lambda = 400...800$  nm is approximately 4.3%. And there are 2 positions where the coating reflectivity is approximately zero.

#### 3. Results and discussion

The technology of manufacturing 3-layer antireflective coating using  $SiO_2/SiO$  material pairs to evaluate the evaporation rate of the film shown in Table 1. To study the effect evaporation rate on the film quality. The author conducted the experimental process on the BOC ADWARDS FL500 vacuum evaporator: in the film forming process, the technological conditions were constanted: the working mode of the electronic gun (voltage, current), all manufacturing processes are the same and only process films at different film evaporation rate on thin film quality.

## 3.1. Transmittance

The transmission coefficient of the 3-layer film depends on the evaporation rate.

The result of optical thin film quality depends on the evaporation rate shown in Fig 3.1a to Fig 3.1d. From the transmittance spectral graph we realize that the transmittance of the film decreases as the deposition rate of the film material increases. Thus, when the consolidation rate of the filmforming material increases, the film thickness increases, resulting in the surface roughness of the film increases, so that the film becomes more porous. Thereby will affect the transmittance of the film. From the spectral transmission graph we see when the film is created with the material's evaporation velocity  $v_{si02} = 0.5A_0/s$ ;  $v_{si0} = 0.4A^0/s$ ;  $v_{si02} = 0.5 A^0/s$ , the largest transmittance of the film is 93.2% in the wavelength range from  $400 \div 700$ nm but when increasing the evaporation velocity of the film-forming material to  $v_{_{SiO2}}$  = 1A<sup>0</sup>/s;  $v_{_{SiO}}$  = 1.1A<sup>0</sup>/s;  $v_{si02} = 1A^0$ /s, the highest transmittance of the film is reduced to 91% also in the wavelength range 400 ÷ 700nm.

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		Current	Voltage	Thicknees film	Evaporation rate	Vacuum chamber pressure
		(mA)	(kV)	(nm)	$(A^0/s)$	(Torr)
	SiO <sub>2</sub>	17	9,96	94	0,7	1,73x10 <sup>-5</sup>
Pattern 1	SiO	8	9,96	141	0,5	5,38x10 <sup>-6</sup>
	SiO <sub>2</sub>	19	9,96	94	0,6	7,25x10 <sup>-5</sup>
	SiO <sub>2</sub>	15	9,96	94	0,5	1,8x10 <sup>-5</sup>
Pattern 2	SiO	8	9,96	141	0,4	5,5x10-6
	SiO <sub>2</sub>	18	9,96	94	0,5	7,15x10 <sup>-5</sup>
	SiO <sub>2</sub>	19	9,96	94	0,8	1,77x10 <sup>-5</sup>
Pattern 3	SiO	8	9,96	141	0,8	5,26x10 <sup>-6</sup>
	SiO <sub>2</sub>	20	9,96	94	0,9	7,2x10 <sup>-5</sup>
	SiO <sub>2</sub>	21	9,96	94	1	1,6x10 <sup>-5</sup>
Pattern 4	SiO	10	9,96	141	1,1	5,4x10 <sup>-6</sup>
	SiO <sub>2</sub>	22	9,96	94	1	7,3x10 <sup>-5</sup>

*Table 1. Three-layer antireflective coating manufacturing technology table, depending on the evaporation rate.* 



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Figure 3.1. Transmitted spectral of the 3-layer film at different evaporation rates

## 3.2. Adherence

To assess the adhesion of the film, the author using incision method. After the film has been incised, we take the film to observe on the microscope we can determine the magnitude of the area to be incised on the coating. This test is under the influence of a defined force based on the thickness or depth of the incision[1]. Measure the size of the incision to assess the durability and adhesion of the coating. With this method, adhesion can be more accurately determined when the film thickness is thin.

Using a diamond incision nose is  $10\mu$ m, the incision force is about 10N (100g), after incision and observation on a biological microscope of 16X magnification, we get an image of the incision on the film and combine with the optical wipe method then observed on microscope. Images of incision

are shown in Fig 3.2.

As we know the film thickness determines the properties of the thin film. Therefore, when the deposition rate increases, the thickness of the film increases, thereby making the adhesion of the film reduced. In the image showing the incision of the film, we realize that as the film deposition rate increases, the peeling of the film becomes clearer and the area of peeling increases.

## 4. Conclusion

Thus, through empirical survey shows that as the film evaporation speed increases, the transmittance of the film in the visible wave range and the near infrared region decreases, the adhesion strength of them also decreases. This is entirely consistent with the theory of thin film has shown.



Figure 3.2. The incision shows the adhesion capacity of a three-layer film at different evaporation rates

## References

[1]. Lê Quang Trà – Luận văn thạc sỹ khoa học, "Nghiên cứu thiết kế chế tạo dụng cụ đánh giá độ bền của lớp phủ trên chi tiết máy", 2009.

[2]. H. Angus Macleod, "Thin-Film Optical Filters", CRC Press 2010.

[3]. Leon I. Maissel and Reinhard Glang, "Handbook of Thin Film Technology", McGraw-Hill Book Company, 1970.

[4]. Michael Bass et al, "HANDBOOK OF OPTICS", Volume IV Optical Properties of Materials, Nonlinear Optics, Quantum Optics, 2010.

# TÁC ĐỘNG CỦA TỐC ĐỘ BỐC BAY TỚI CHẤT LƯỢNG MÀNG KHỬ PHẢN XẠ ĐA LỚP SỬ DỤNG CẶP VẬT LIỆU SiO,/SiO

## Tóm tắt:

Bài báo này trình bày về kết quả nghiên cứu, tính toán, chế tạo màng khử phản xạ 3 lớp sử dụng cặp vật liệu SiO<sub>2</sub>/SiO trên máy bốc bay chân không BOC ADWARDS FL500. Kết quả nghiên cứu sử dụng đánh giá về những ảnh hưởng của tốc độ bốc bay màng tới chất lượng màng khử phản xạ đa lớp, trong vùng cận hồng ngoại, vùng hồng ngoại bước sóng ngắn.

Từ khóa: Tốc độ bốc bay, màng khử phản xạ SiO/SiO, chất lượng màng mỏng quang học.