ISSN: 2347-3487

Study of the optical properties of SiO_2, and Al_2O_3 films deposited by electron beam technique

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ABSTRACT

Al_2O_3 and SiO_2 thin films have been prepared by electron beam evaporation at different oxygen flows. The influences of oxygen partial pressure on optical properties of Al2O3 and SiO_2 thin films have been studied. We have coated Al2O3 and SiO_2 without some oxygen background in the chamber. The results show that Al_2O_3 thin film does not have absorption even though it is coated without oxygen background in the chamber. On the other hand, without oxygen flow, SiO_2 thin film has some absorption. The packing density of the samples is studied by change in the spectrum of a coating with humidity.

Indexing terms/Keywords

Refractive index, absorption, Packing density

Academic Discipline And Sub-Disciplines

Physics; Thin Film

SUBJECT CLASSIFICATION

Physical Vapor Deposition; optical properties

TYPE (METHOD/APPROACH)

Electron Beam Evaporation

Council for Innovative Research
Peer Review Research Publishing System
Journal: JOURNAL OF ADVANCES IN PHYSICS
Vol. 9, No. 3
www.cirjap.com, japeditor@gmail.com

1. INTRODUCTION

Aluminum oxide (Al_2O_3) has been used as an electrically insulating material over a wide range. Thin films of this material have been applied as insulator layer in electronic devices, such as transistors, metal-oxide-semiconductor (MOS) devices, and electroluminescent panels [1, 2]. Al_2O_3 has an intermediate index in comparison to Magnesium fluoride (MgF_2) and Titanium dioxide (TiO_2). It is one of the most ordinarily used high-refractive index materials in the multilayer dielectric mirror in the UV spectral region and it is used to the manufacture of interference coatings in the UV spectral [3, 4]. Reduction of optical loss for high reflection dielectric mirrors is significant to applications of high-energy UV laser. The design of double layer antireflection "MgF_2/Al_2O_3/glass" is known [5]. Aluminum oxide thin films have been deposited by various techniques, including magnetron sputtering, electron beam evaporation, plasma- enhanced chemical vapor deposition, and filtered cathodic vacuum arc, etc [6, 12].

Silicon dioxide (SiO_2) is one of the low index materials. Although it is somewhat higher index than MgF2 at 1.38 in the visible spectrum, it can often be deposited with less porosity. It is relatively durable and can have a good laser damage threshold [13]. Its use in interference coatings in UV and visible spectral is extensive. Silicon dioxide thin films can be deposited by both magnetron sputtering and electron beam evaporation. The optical properties of Al_2O_3 and SiO_2 film are sensitively dependent on the production process. This is because different deposition techniques to cause different film structures. Generally, the index of refraction is a complex quantity, $N=n+ik$. The optical properties of materials can be determined by n and k. The n is the index of refraction for a purely dielectric material and the k is extinction coefficient. The extinction coefficient k is an indicator of any absorption in the material. The absorption coefficient α defined as $\alpha = 4\pi k/\lambda$, where λ is the wavelength. The ratio of the transmitted intensity I to the initial intensity I_0 through an absorbing medium of thickness t can be found by $I/I_0 = e^{-\alpha t}$. When it is zero the material is completely free from absorption. Dielectric materials have small or zero values of k, while metals have very large values.

Thin film of any material must be having a good density layer and as little as possible absorption. Even if two films were constructed from exactly the same material, their refractive indices would be different if their densities were different. Thin films rarely have the same refractive index as similar bulk materials. The main reason for this is their microstructure that is rarely bulk-like but usually shows a columnar morphology. Optical absorption is an important effect for films used in high power laser technology where high absorption can give local failure of the coating.

The aim of this work is to find the highest packing density and the lowest absorption for Al_2O_3 and $SiO_{2.}$ We have prepared the Al_2O_3 and SiO_2 films by electron beam evaporation on glass substrate. During evaporation Al_2O_3 and SiO_2 by electron beam source, as a rule oxygen gas is injected into the chamber to compensate for the dissociation and oxygen loss from the evaporant. In this paper, we have done with and without some background oxygen in the chamber and each case has been examined to determine higher index and as little as possible absorption. As far as we know, the optical properties of SiO2, and Al2O3 films have been not examined without the O_2 flow. The transmittance spectrum of samples is recorded. This produces a transmittance spectrum with a few peaks. We have collected the wavelength and magnitude of the maxima and minima of transmittance and then the index of refractive and the extinction coefficient of samples have been determined by envelope technique [14, 15].

The format of the paper is as follows: In Section II we present the experimental detail. In Section 3 the results of our calculations and measurements are presented. In Section 4 we present results and discussion. Finally, in Section 4 we conclude with a summary of our results.

2. EXPERIMENTAL DETAILS

All Al_2O_3 single layers were deposited on glass (BK7) substrates with the dimension of Ø 25 mm × 3 mm by electron-beam evaporation with exactly the same conditions. Electron beam source is an E-gun in 10 KV, 5 KW, and 270 deg bent-beam. The vacuum system consists of a diffusion pump that is backed with a rotary pump. The base pressure of the system was evacuated to 10^{-6} Torr and deposition pressure was about 6×10^{-5} Torr. To minimize the influence of the substrate, the same block of BK7 material has selected to produce all of the used substrates according to the identical polishing process. Aluminum oxide was placed on water cooled copper crucible. Al_2O_3 and SiO_2 tablets (Merck) are used as the evaporation material. The distance between the substrate and the source is 40 cm. As is common with all materials, increased substrate temperature leads to hard layer. The substrate was heated and the temperature was held constant at 300° C during the thin film growth. Film thickness and the rate of deposition were monitored by a crystal monitor. The samples were obtained by e-gun evaporation at a deposition rate of $6\,A^{\circ}/s$.

3. MEASURMENT OF OPTICAL PROPERTIES

Optical constants can be extracted from transmittance data using a variant of the envelope technique. The films should be basically dielectric with thicknesses such that there are extrema within the wavelength range of interest. Absorption may be included provided that it is not so great that the fringes are destroyed. The technique used is of the class known as envelope methods. Such methods focus on the maxima and minima of transmittance. The first stage of the calculation involves the interpolation of the maxima and minima to generate the envelopes. The ideal envelopes consist of the substrate transmittance and the ideal quarterwave transmittance. When the layer is absorbing, the maxima and minima move away from these ideal envelopes by an amount that increases with thickness but, when absorption is not too high, the envelopes can serve to launch the *n* and *k* calculations. The envelop method fails if the absorption in the film is so high that interference fringes are not noticeable and T_{max} and T_{min} coincide. When absorption is not too high [14-17], $k << (n_f - n_0)^2$

$$T = \frac{16 n_0 n_f n_s}{C_1^2 + C_2^2 \alpha^2 + 2 C_1 C_2 \cos(4\pi t n_f / \lambda)} \qquad (1)$$

Where ,*T* is transmittance, $C_1 = (n_f + n_0)(n_f + n_s)$ and $C_2 = (n_f - n_0)(n_s - n_1)$, n_0, n_s, and n_f are the refractive indices of air, substrate and thin film, respectively.

$$\alpha = \exp(4\pi k t / \lambda) \qquad (2)$$

In the weak and medium absorption regions, the refractive index *n* is given by

$$n = \left(N + (N^2 - n_0^2 n_s^2)^{1/2} \right)^{1/2} \qquad (3)$$

Where

$$N = 2 n_s \frac{T_{\max} - T_{\min}}{T_{\max} T_{\min}} + \frac{n_s^2 + n_0^2}{2} \qquad (4)$$

Where , $T_{\min}$ and $T_{\max}$ are the extreme values of the transmission

$$T_{\max} = \frac{16 n_0 n_f n_s \alpha}{(C_1 + C_2 \alpha)^2}, \quad T_{\min} = \frac{16 n_0 n_f n_s \alpha}{(C_1 - C_2 \alpha)^2}$$

For the absorption coefficient α , we have

$$\alpha = \frac{C_1 [1 - (T_{\max} / T_{\min})^{1/2}]}{C_2 [1 + (T_{\max} / T_{\min})^{1/2}]}$$

4. RESULTS AND DISCUSSION

Fig. 1 shows the dispersion curves of refractive index *n* of Al_2O_3 films, calculated according to above equations using transmission spectral in the spectral range 400–700nm deposited at different oxygen flow. The curves in Fig. 1 show that the refractive indices decrease as the oxygen flow increasing O_2 flow. It is clear that the refractive index has a normal dispersion in the wavelength 400-700 nm. Our calculations show that the calculated values of the refractive index *n* according to the approximate methods [14, 15] are in a good agreement with each other.

When the index of the film is higher than that of the substrate, the maxima of the transmittance spectrum are equal to the transmittance of the uncoated substrate. Fig. 2 shows the spectral distributions of transmittance *T* the uncoated substrate (glass) in the spectral range 400-700 nm. The maxima of the transmittance spectra of the coated substrate coincide with the transmittance the uncoated substrate. This behavior expresses the optical homogeneity of the films [18]. When film is inhomogonous, the variation of index through the film may be large and the accuracy of the envelope method decreases. Fig. 3(a)-(b) shows the spectral distributions of reflectance *R* and transmittance *T* in the spectral range 400–700 nm. It can be seen that Al_2O_3 films are transparent ($T + R = 1$) and the extinction coefficient is zero ($k = 0$). This also proved by the envelope method. An interesting feature of these results is that Al_2O_3 films do not have absorption without oxygen flow. It seems that to heat substrate up to 300° C enhances the oxidation and repairs the oxygen vacancies [19]. Deposition at lower substrate temperatures results in films with reduced film density. However, higher temperatures ($> 300^\circ$ C) are usually less desirable for technical reasons.

The results on optical properties of SiO_2 films are shown in Fig. 4 and Fig. 5. At a given deposition rate, the refractive index *n* increases with lower oxygen flow as shown in Fig. 4. On the other hand, without oxygen flow, films of increased extinction *k* can be obtained which show absorption as shown in Fig. 5. This can be attributed to the oxygen deficiency. However, increased pressure during film growth will cause a reduction in the film density as shown Fig. 4. Thus, the addition of excess oxygen seems to be opposite for achieving films of high density. On the other hand, it is necessary to provide enough oxygen to remove absorption to increase the laser damage threshold because optical absorption is an important effect for films used in high power laser technology where high absorption can give local failure of the coating.

The value of refractive index is proportional to the packing density of thin film and index is higher for more dense films. On the other hand, the packing density of film depends on energy of deposited atoms or molecules. The average distance traveled between collisions is called the mean free path *MFP*. The mean free path decreases when the oxygen flow increases and the kinetic energy of evaporated particles become lower because of collisions with gas molecules. This results in to reducing surface diffusion and decreasing the packing density. Shamala et al. [20] reported index $n = 1.6$ for Al_2O_3 at wavelength 550 nm. They have been carried out deposition at working pressure 2×10^{-4} Torr, and rate 4 A/s. Such low index can be attributed to low *MFP* high working pressure, i.e., increased pressure during film growth will cause a reduction in the film density.

The increased packing density improves the stability of the films and reduces also the moisture sensitivity [21]. There is a useful expression between refractive index n_f and packing density p [22]

$$n_f = pn_s + (1-p)n_v$$

where n_s and n_v are the refractive index of the solid part of the film and the voids, respectively. The packing density p is defined as the ratio of the density of the thin film to the density of the bulk solid material. To calculate the packing density, we must determine the mass of the films by weighing the substrates before and after coating and taking the difference of the two measurements. This is a difficult procedure because the film mass is much lower than the substrate mass and is probable to many errors. The change in the spectrum of a coating with humidity seems to be a reasonable measure of the density of an optical thin film [23]. Shift in spectrum due to presence of humidity can be used as a measure of the packing density. If there is no detectable shift, the film may be fully dense. The spectrum shift is shown for Al_2O_3 film in Fig. 6 and SiO_2 film in Fig. 7. It is obvious that the spectrum shift of Al_2O_3 film is smaller than SiO_2 film that shows water uptake. Thus Al_2O_3 film is stronger than SiO_2 film.

5. CONCLUSION

The films of SiO_2 and Al_2O_3 have been deposited onto BK7 by electron beam technique. The packing densities of Al2O3 and SiO2 have been examined by shift in spectrum with humidity. The Al_2O_3 films do not have absorption without oxygen. Lower oxygen cause absorption to SiO2 films and it is necessary to provide enough oxygen to eliminate absorption.

FIGURE / CAPTIONS :

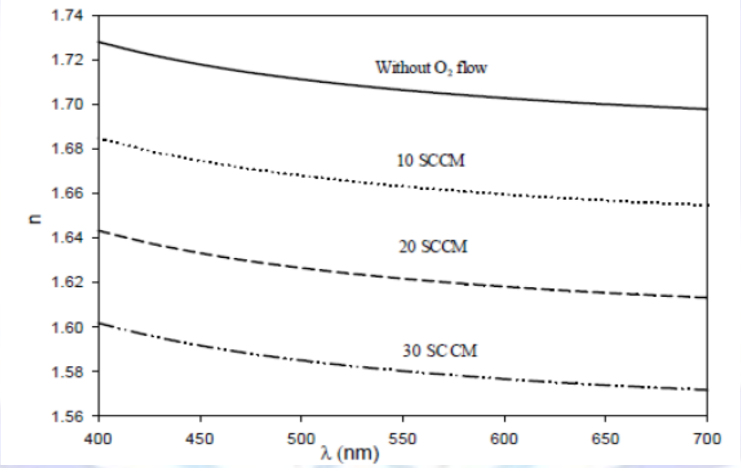

Fig. 1: Dispersion curves of refractive index *n* for Al_2O_3 films with different O_2 flow

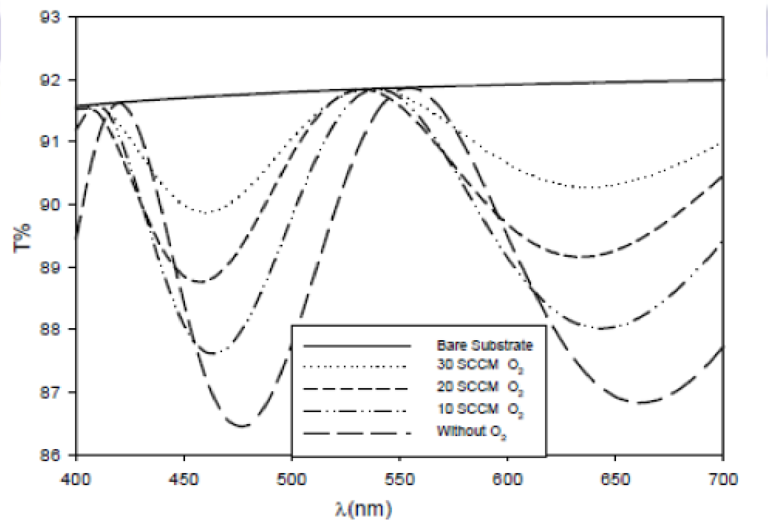

Fig. 2 : Spectral distribution of transmittance *T* for Al_2O_3 films with different O_2 flow and uncoated substrate

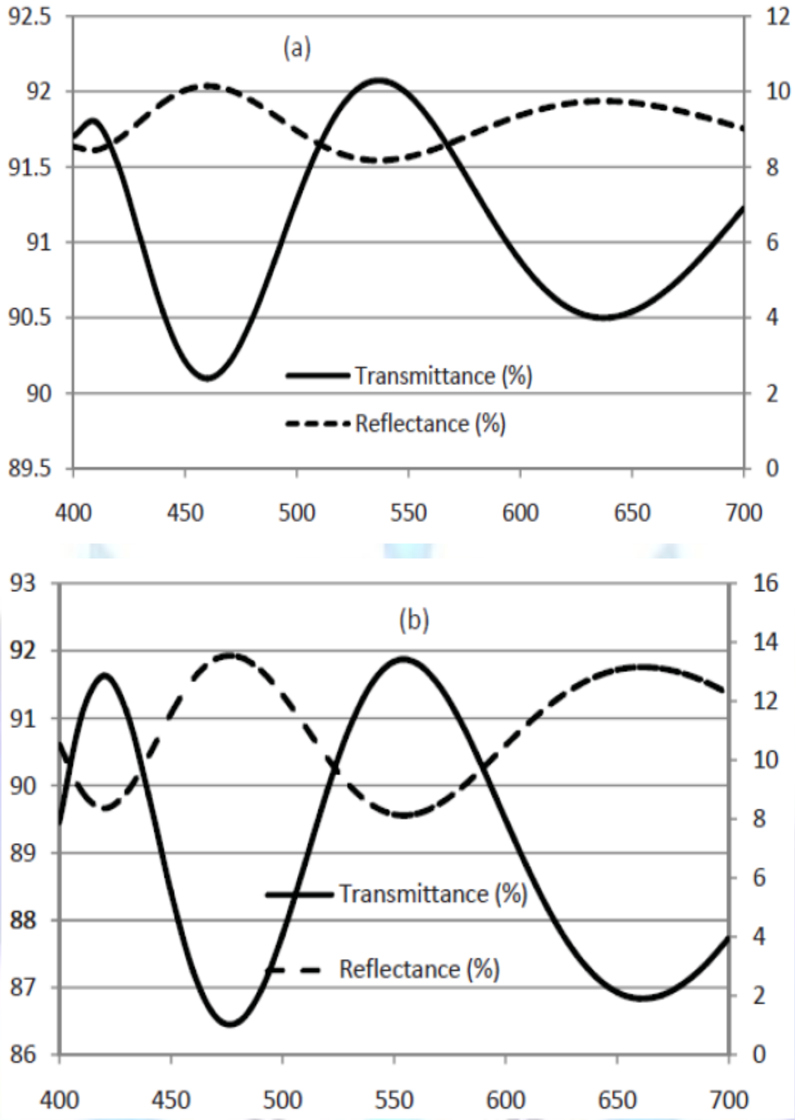

Fig. 3 : Spectral distribution of transmittance *T* and the reflectance *R* for Al_2O_3 films (a) 30 SCCM O_2 (b) without O_2 flow

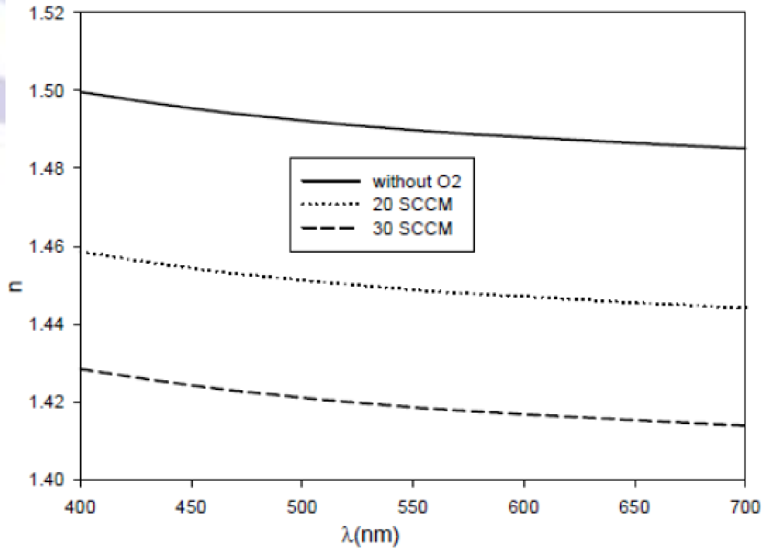

Fig. 4 : Dispersion curves of refractive index *n* for SiO_2 films with different O_2 flow

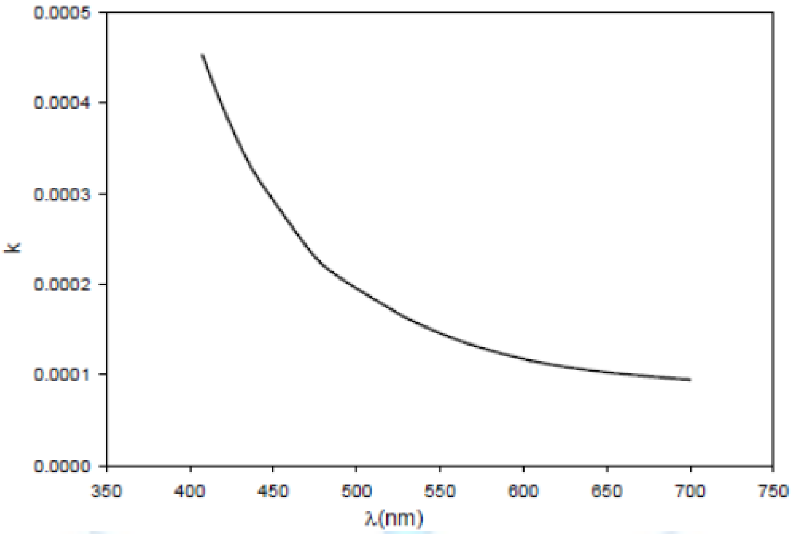

Fig.5 : Dispersion curves of extinction coefficient *k* for SiO_2 without O_2 flow

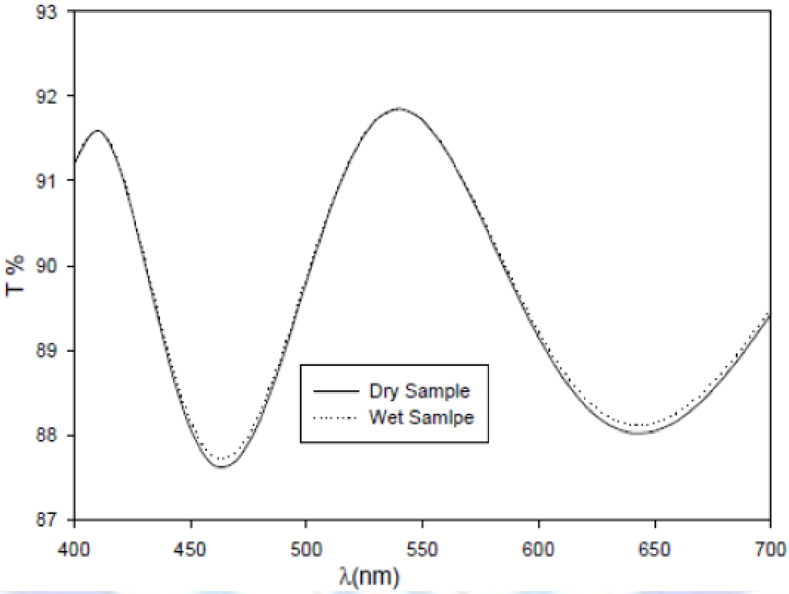

Fig. 6 : The change in the spectrum of transmittance with humidity for Al_2O_3

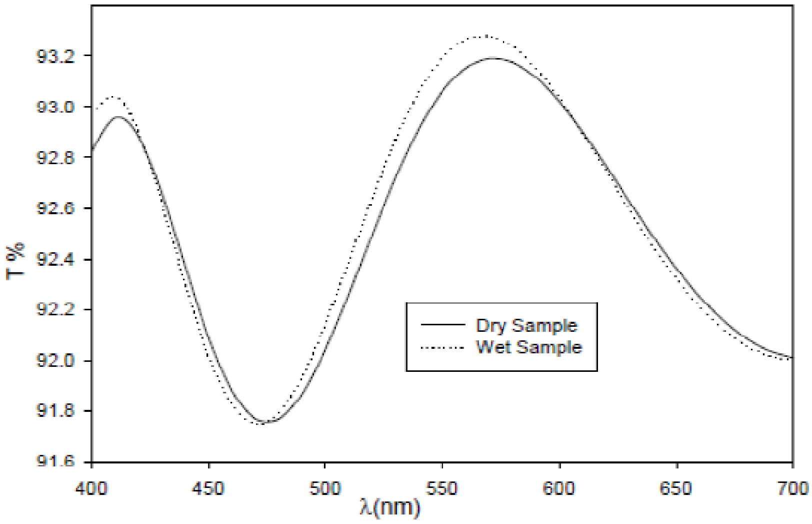

Fig. 7 : The change in the spectrum of transmittance with humidity for SiO_2

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