



# Oxygen amount effect on optical properties of aluminium oxide nanostructured films prepared by reactive magnetron sputtering



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## ABSTRACT

A simple method was used to deposit the aluminium oxide thin films. Thin films were prepared by reactive magnetron-sputtering on soda glass substrate and the effect of introduced oxygen flow on optical properties of thin films was studied. Structural and optical properties of thin films were characterized and their optical constants were measured. Results revealed formation of cubic aluminium oxide phase. The average grain size of 25 nm was observed by scanning electron microscopy images. Optical spectra of thin films are measured and the effect of oxygen gas amount in sputtering process on the samples is investigated where the highest transmission value belongs to highest amount of introduced oxygen. A new precise approach has been used to measure optical properties of the films. The refractive index and extinction coefficient of the thin films were also measured where the thin films exhibited refractive indexes more than 1.65 at visible wavelengths.

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## 1. Introduction

Aluminium oxide thin films have been the point of interest for thin film technology since many years because of its optical and mechanical properties for applications as high abrasive and corrosion resistance [1,2], insulating [3], dielectric [4], and hard protective layers [5]. Among different methods to synthesize aluminium oxide thin films, vacuum based methods provide a uniform layer with advantages like high speed and high quality process. Several vacuum methods, such as chemical vapour deposition (CVD) [6], atomic layer deposition (ALD) [7], Pulsed laser deposition (PLD) [8], and sputtering [9–11], have been used to deposit the aluminium oxide films.

Many works have been performed on investigation of optical properties of aluminium oxide thin films to obtain their optical constants [12]. But there are not many reports to obtain the optical constants of thin films from the optical spectra of thin films. These optical constants could be useful information for potential industry applications. As instance, refractive index is an important parameter for optical coatings and the layers with high refractive index have many applications [13,14]. A useful theoretical method

was used to determine these optical parameters only by using the experimental results of transmission and reflection [15].

In this paper, reactive magnetron sputtering method in atmosphere of oxygen and argon gas was used for providing aluminium oxide thin films. The structural and optical properties of aluminium oxide thin films deposited by a radio frequency sputtering process were presented and the optical constants of these films such as transmission, reflection, refractive index, and extinction coefficient are compared theoretically and experimentally. A theoretical method was used to precise determination of the optical contents of films deposited on glass substrate and to study the dependence of these optical constants on the growth condition of oxygen content.

## 2. Theory

Recently, we have reported a precise method to evaluate optical constants of a thin film against the wavelength of the incident light [15]. Consider a thin layer with thickness  $l$  and refractive index  $n_1(\lambda)$  is deposited on a transparent thick substrate with refractive index  $n_2$ . As shown in Fig. 1,  $I_1 = R'I_0$  and  $I_2 = R''I_0$  are the reflected intensities from surfaces 1 (outer surface of the film) and 2 (the junction layer of the film and substrate), respectively.  $R'$  and  $R''$  are reflectances from these two surfaces:

$$R' = (r_{01})^2 = \left( \frac{n_0 - n_1}{n_0 + n_1} \right)^2 \quad (1)$$

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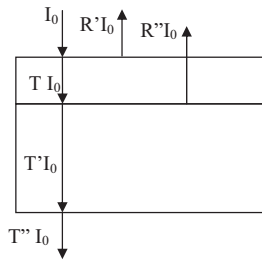


Fig. 1. Schematic description of reflectance and transmittance of film and substrate.

$$R'' = (t_{01}r_{12}t_{10})^2 = \left( \frac{2n_0}{n_0+n_1} \frac{n_1-n_2}{n_1+n_2} \frac{2n_1}{n_1+n_0} \right)^2 \quad (2)$$

where  $r$  and  $t$  are Fresnel factors for reflection and transmittance [15] and  $n_0$ ,  $n_1$  and  $n_2$  are the refractive indexes of the incident medium, film and the substrate, respectively.

Transmittances from surfaces 1 ( $T$ ), 2 ( $T'$ ) and 3 ( $T''$  from outer surface of the substrate) are defined as [15]:

$$T = \frac{n_1}{n_0} t_{01}^2 = \frac{4n_0n_1}{n_0+n_1} \quad (3)$$

$$T' = \frac{n_2}{n_1} t_{12}^2 \exp(-\alpha l) T = \frac{4n_1n_2}{(n_1+n_2)^2} \frac{4n_0n_1}{(n_0+n_1)^2} \exp(-\alpha l) \quad (4)$$

$$T'' = \frac{n_0}{n_2} t_{20}^2 T' = \frac{4n_0n_2}{(n_0+n_2)^2} \frac{4n_1n_2}{(n_1+n_2)^2} \frac{4n_0n_1}{(n_0+n_1)^2} \exp(-\alpha l) \quad (5)$$

$\alpha$  is the absorption coefficient of the film. Using transmittance and reflection, one could obtain the absorption coefficient as [15]:

$$\alpha = \frac{-1}{d} \ln \left( \frac{R'' + T'}{1 - R'} \right). \quad (6)$$

$T'$  could be easily obtained using Eqs. (4) and (5) and experimental value of  $T''$ :

$$T' = T'' \frac{(n_0+n_2)^2}{4n_0n_2} \quad (7)$$

According to Eqs. (1) and (2),  $R'$  and  $R''$  depend on  $n_1$  which is yet unknown. One should presuppose a constant (averaging) value for  $n_1$  to approximate  $R'$  and  $R''$ . It's an assumption, but its effect on  $\alpha$  is so low and negligible. The main effective parameter to the value of  $\alpha$  is transmittance, not  $n_1$  [15].

Solving Eq. (5) against  $n_1$  gives:

$$n_1^2 + ((n_2+n_0) - \frac{8n_2n_0e^{-\alpha d/2}}{(n_2+n_0)\sqrt{T''}})n_1 + n_2n_0 = 0 \quad (8)$$

This equation could give refractive index of the film using the reflectance and transmittance of the film on the substrate.

Using the absorption coefficient, the extinction coefficient of the film would be obtained:

$$\kappa = \frac{\alpha\lambda}{4\pi} \quad (9)$$

Real and imaginary parts of optical susceptibility of the film could be evaluated using the following relations [15]:

$$\begin{aligned} \varepsilon_r &= n^2 - \kappa^2 \\ \varepsilon_i &= 2n\kappa. \end{aligned} \quad (10)$$

This processing method could give a precise spectrum of refractive index and absorption coefficient of thin layers on thick transparent substrates.

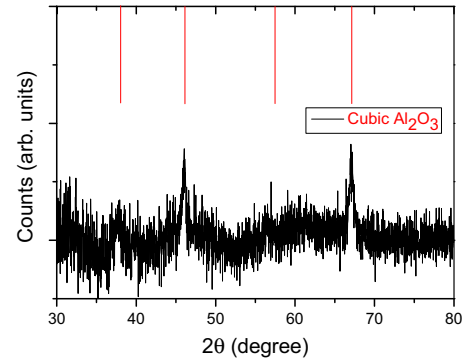


Fig. 2. XRD pattern of aluminium oxide thin film.

### 3. Experimental results and discussion

Soda glass slides were used as substrate for coating. They were cleaned prior to the deposition with ethanol and distilled water in ultrasonic bath and subsequently dried with nitrogen gas. Radio frequency magnetron sputtering was used for the deposition of thin films. Pure aluminium (99.99% purity, Lesker) was used as target for sputtering. The chamber was evacuated with a turbo-molecular pump to a vacuum of  $1.2 \times 10^{-5}$  mbar. High purity argon gas was introduced to the chamber after the evacuation to obtain the vacuum of  $1 \times 10^{-5}$  mbar and immediately the oxygen gas which has been controlled by an independent gas flow meter was introduced to the chamber at different gas flows. The target voltage was 400 V and the sputtering power was adjusted on 100 W. The target-to-substrate distance was 10 cm and the deposition was continued for 2 min. The amount of introduced oxygen was changed from 10 to 40 standard cubic centimeters per minute (sccm) with the steps of 10 sccm.

The structural and morphological properties of the samples were analysed with X-ray diffraction (XRD, Bruker, D8 Advance Model) pattern and field emission scanning electron microscopy (FE-SEM, Hitachi S-4500). The optical spectra (transmission and reflection) of thin films were obtained by Shimadzu (UV-3600 Plus) spectrometer.

The samples are prepared with different oxygen gas during the deposition from 10 sccm to 40 sccm. The XRD pattern of aluminium oxide patterns are obtained and show cubic structure of aluminium oxide without any other peaks related to the impurities or metallic aluminium as shown in Fig. 2. Fig. 3 presents the morphological view of the surface of a typical thin film. The oxygen content did not have remarkable effect on XRD pattern and morphological properties of aluminium oxide thin films.

The thickness of the film of aluminium particles has been obtained 103 nm using ellipsometry method. Optical Transmission spectra of the films measured in the range of 250–2500 nm as

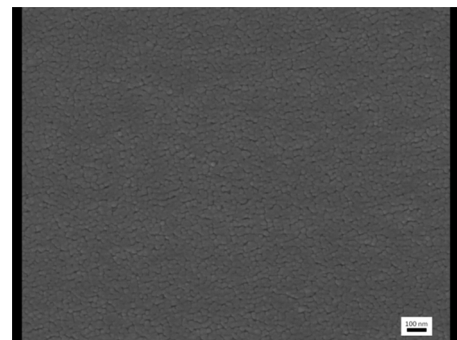


Fig. 3. Plan-view SEM images of aluminium oxide thin film.

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