OPTOELECTRONICS AND ADVANCED MATERIALS – RAPID COMMUNICATIONS Vol. 4, No. 6, June 2010, p. 855 - 858

Characterization of deposited AlN thin films at various nitrogen concentrations by RF reactive sputtering

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AlN thin films deposited onto glass slides using a RF reactive sputtering system at various nitrogen concentrations. Characterizations of the deposited thin films were realized using X-ray diffraction (XRD), atomic force microscopy (AFM) and Uv-Vis spectrophotometer. Morphological and optical properties of deposited films were strongly depend on nitrogen concentration in Ar-N_2 mixed gas plasma. It was shown that deposited AlN thin films were in the amorphous structure except for x=0.4 concentration. The formation of AlN (111) and AlN (220) deposited at x=0.4 concentration were confirmed by XRD studies. Optical properties of deposited AlN thin films were investigated by measuring the transmittance spectra. Transparencies of the deposited AlN films at x=0.4 and 0.3 shown highest transparency concentrations were exhibit approximately ~100% in near infrared regions. Also, refractive indexes of the deposited AlN thin films were change by nitrogen concentrations. As s result, deposited AlN thin films will be use antireflection coatings for optical components.

(Received April 05, 2010; accepted June 16, 2010)

Keywords: AlN thin Films, RF reactive sputtering, Optical properties

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1. Introduction

Aluminum nitride (AlN) thin films with wurzite hexagonal structure have received great interest because of its excellent physical properties like thermal conductivity (3.2W/mK), chemical stability, high hardness, high acoustic velocity, large electromechanical coupling coefficient and a wide band gap (5.8-6.2 eV). AlN thin films are promising candidate for electronic material for thermal dissipation, dielectric and passivation layers, surface acoustic wave (SAW) devices and photoelectric devices [1-14]. For these applications, AlN thin films deposition and characterization are very important. As an optical material, it is important to understand its fundamental optical properties. But, the optical constants of AlN thin film are still unclear [1, 10-13].

Various techniques, such as chemical vapor deposition (CVD), laser chemical vapor deposition (LCVD), pulsed laser ablation (PLD) and molecular beam epitaxial (MBE), have been used to prepare AlN thin films on various substrates under various process parameters. In this paper, RF reactive sputtering carried out for deposition of the AlN thin films on glass slides at various nitrogen concentrations. The properties of an AlN thin film is greatly affected by its microstructure. Microstructures of thin films are depended to deposition conditions and techniques [2, 15]. Also, the dissipation capacity of AlN thin films governed by such microstructure factors as morphology, interface roughness and preferred orientation [2]. These parameters initially will dominate the phonon scattering procedure in AlN films at certain temperature ranges [2, 12].

2. Experimental

AlN thin films were deposited by RF reactive sputtering system where working pressures were ~1.7×10^{-1} Torr. Target materials in our experimental procedures were a commercial Al plate, whose dimensions were 1x1x0.1 cm. RF pulsed power supply was adjust to 200W/600W of 13.56MHz in all deposition process. Using a RF reactive sputtering system, researchers have deposited AlN thin films with different orientation and different properties by changing process pressure and N_2 concentrations because of process pressure and N_2 concentrations would affect the properties of AlN thin films [1]. H.Cheng et al. shown that pressure is very important for AlN preferred orientation of AlN [2]. Mixed gas plasma in RF reactive sputtering system were generated from $(1-x)Ar + xN_2$, where x value was changed to 0.90 from 0.10 step by step (initial step wise was 0.1). x value was calculated from the following equation;

$$x = \frac{P_{N2}}{P_{N2} + P_{Ar}} \quad (1)$$

where, P_{N2} is the partial pressure of the N_2 gas and P_{Ar} is the partial pressure of Ar gas in the Ar-N_2 gas mixing. Distance between the target including Al plate and sample holder located glass slides was 3 cm. All deposition parameters of RF reactive sputtering system were nearly kept constant except for x value and process duration. x values were very important parameters for microstructures properties of AlN thin films [1, 2]. Process duration was only affected the thickness of deposited thin film.

3. Results and discussion

Thickness of the AlN thin films were measured with Filmetrics F20 thin film measurement system. Thickness measurements of the deposited AlN thin films were analyzed 50 times of the different point of the surfaces. The distances of the measurement points were approximately 2mm to each other. Measured thickness of AlN thin films and its statistical data were summarized in Table 2.

Table 1. Measured thicknesses of deposited AlN thin films.

x (N_2 concentration)	0.90	0.80	0.70	0.60	0.50
Duration (min)	60	60	90	90	100
Mean Thickness (nm)	58.57	62.88	90.65	81.12	74.03
Min.Thickness (nm)	50.00	54.29	79.77	81.04	73.93
Max.Thickness (nm)	75.64	72.19	109.38	81.24	74.10
Standard Derivation	4.905	2.865	9.992	0.042	0.04
Deposition rate (nm/min)	0.97	1.05	1	0.9	0.74
x (N_2 concentration)	0.40	0.30	0.20	0.10	
Duration (min)	90	100	80	90	
Mean Thickness (nm)	65.75	64.96	54.82	59.74	
Min.Thickness (nm)	25.00	64.87	54.75	59.64	
Max.Thickness (nm)	65.98	65.04	54.87	60.13	
Standard Derivation	14.294	0.038	0.026	0.149	
Deposition rate (nm/min)	0.73	0.65	0.68	0.66	

Process duration affected the thickness of the deposited thin films in reactive sputtering system. As can be seen table.1, variations of the deposition rates of the AlN thin films via nitrogen concentration shown in Fig. 1. Also as can be seen Fig. 1, while increased x values in Ar-N_2 gas mixing, deposition rates of AlN thin film were increased to nearly constant value (approx 1nm/min), clearly. Because of nitrogen atom is lighter than argon atoms, the particles sputtered from the Al plate will lose less energy through collision with a nitrogen atom than with argon atom [1]. Mean free path is approximately 3 cm in this working pressure that is sputtered Al atoms will collide in 3 cm. Hence, Ar atoms will lose some of their energy by colliding.

Transmittance and absorbance spectra of deposited AlN thin films were realized using Perkin–Elmer Uv-Vis Spectrometer Lambda 2S, which were shown in Fig. 2 at various nitrogen concentrations (x values). As can be seen in Fig. 2, some differences in absorbance and transmittance can be observed clearly.

While as x values increased to 0.4 from 0.1, the transparencies of the growth AlN thin films increased relatively. In the same situation, absorbencies of the deposited films were decreased because of the more nitrogen atoms in the composition. Transmittance of the films deposited 0.4 and 0.3 concentrations were bigger (T~100%) than the transmittance of the glass slides in weak absorption region ($\lambda > 700$ nm) and near infrared region (transmittance of the glass slides were measured approximately 93% in the same regions). Additionally, absorption percentages of the deposited AlN thin films at $x = 0.4$ and 0.3 concentrations were very low according to glass slides (see Fig. 2). Not only, film thickness significantly affected the Uv-Vis spectra, but also electron transitions between the valance and conduction bond in all films decreased the transparency of thin films. In the same time, these samples were more transparent in visible region. Reason of its decreasing surface reflection of the AlN thin films due to the mean roughness of films were affected x values. These are correlated results with those of ref. 2.

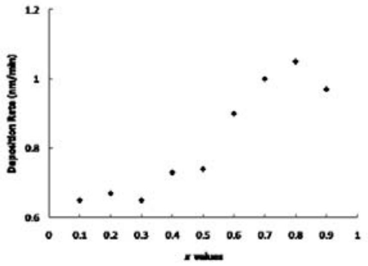
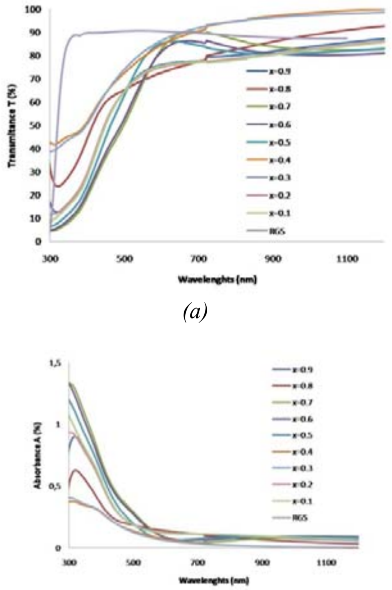


Fig. 1. Deposition rates of AlN thin films via N_2 concentrations in gas mixture.

The refractivity (R) of the films can be calculated from the following equation [15];

$$T = (1 - R^2)\exp(-A) \tag{2}$$

where, T is the transmittance and A is the absorbance values. Refractive index and extinction coefficient of the films are given by the following formula [15, 20];



(a)

(b)

Fig. 2. (a) Transmittance and (b) absorbance spectra of growth AlN thin films.

Variations of the refractive index n of the AlN thin films with x value is shown in Fig. 3. Refractive index of AlN thin films are in the range of 1.4-1.92. These values are lower according to ref.14 [1, 14], But these values are in good harmony with ref. [1]. These lower values are due to the porous structured of AlN thin films [1]. Mean roughness values of AlN thin films are verifying these results [see Table 3].

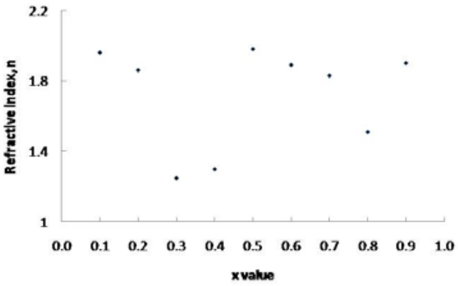


Fig. 3. Variations of the refractive index n of the AlN thin films with x value.

The extinction coefficient *k* can be calculated from the relation

$$k = ((/4(\tag{3}$$

where α is the absorption coefficient and can be calculated following equation;

$$(= (-1)/d\ LN(T) \tag{4}$$

where d is the thickness and T is the transmission of the thin film [16-18].Variations of the extinction coefficient k of the AlN thin films with x value is shown in Fig. 4.

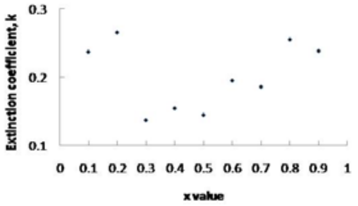


Fig. 4. Variations of the extinction coefficient k of the AlN thin films with x value.

Extinction coefficient is always lower the real part of the refractive index. Like as transparency and absorbance spectra, the values of reel part and imaginary part of refractive index were very low according to other sample's values.

E_g is the band gap energies of the deposited AlN thin films by using various nitrogen concentration and it is determined by Tauc method, which is described elsewhere [19,20]. Variations of the E_g of the deposited films with x value were shown in Fig. 5. Optical band E_g was a variant according to x values. E_g values of AlN thin films are in the range of 2.69-3.14 eV. When determined E_g is small a 6 eV, AlN thin films contain more defects, impurities (oxygen impurities) and some dislocations [1]. When x values increase, concentrations of the defects and impurities will decrease and the band gap will become wider.

The real and imaginary parts of the dielectric constant were determined using the following equation [18],

$$(= (r + (i = [(n + ik)]^2 \tag{5}$$

where $(r$ is the reel part and is normal dielectric constant, $(i$ is the imaginary part and represent the absorption associated of radiation by free carrier. The dissipation factor, tan δ, is expressed as [18],

$$tg(= (i/(r \tag{6}$$

Variation of dissipation factor with x values is shown in Fig. 6. All dissipation factors were calculated for 550 nm for comparison.

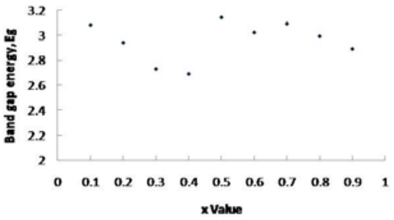


Fig. 5. Variations of the E_g of the AlN thin films with x value.

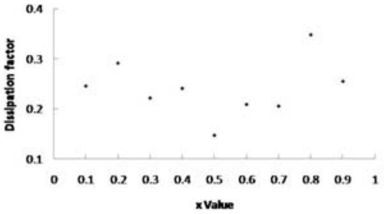


Fig. 6. Variations of the dissipation factors of the AlN thin films with x value

The crystal structures of AlN thin films were detected by x-ray diffraction with Cu-Kα radiation. Fig. 7 shows the XRD analysis results of AlN thin films deposited onto glass slides. As can be seen Fig. 7, AlN thin film at x=0.4 concentration was in (111) and (220) orientations. Peak list of the XRD data was summarized in table 2. Deposited thin films at other nitrogen concentrations were shown the amorphous structures properties.

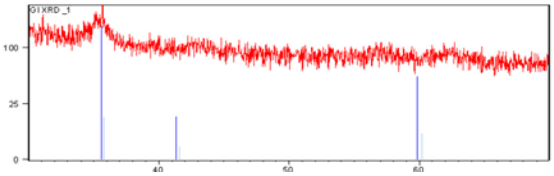


Fig. 7. XRD pattern of the deposited AlN thin film at x *=0.4 concentration.*

Table 2. XRD peak list of AlN thin film.

No.	h	k	1	d(A)	2Theta[deg]
1	1	1	1	2.52010	35.596
2	2	0	0	2.18250	41.335
3	2	2	0	1.54330	59.884

Fig. 8 shows that AFM images of the AlN thin films as an example surface image (x =0.90). Rms roughness of the thin films decreased from 60.20 nm to 12.50 nm. Ref.1 shows that mean roughness of the AlN thin films are correlated with the mobility of atoms, which was governed by the energy of the deposited particles [1, 9]. The grain size of the deposited AlN thin films is approximately 260 nm.

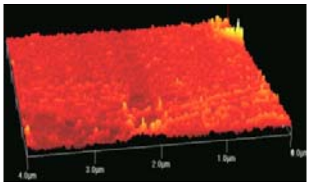


Fig. 8. Example AFM images of AlN thin films at x=0.90.

4. Conclusions

In this paper, AlN thin films were deposited onto glass slides by RF reactive sputtering system at the various nitrogen concentrations. Optical and structural properties of AlN thin films were determined. For all deposited thin films, thickness measurements, deposition rates, transmittance, absorbance, refractive index, extinction coefficient, band gap, dielectric constant, imaginary part of dielectric constant, dissipation factor, XRD, AFM imaging and roughness measurements and calculations were realized using by interferometry, Uv-Vis spectrometry, XRD, AFM tools. All these properties were summarized in this paper. According to optical investigations, deposited thin films at x =0.4 and 0.3 concentrations has a great transparency (~100%) in weak absorbance region and near infrared region. These transmittance values of deposited films are larger than the glass slides' transmittances. These films are good for interference filter, band pass filter, low pass filter, thermal radiation detection systems, lenses for optical devices and some optoelectronics applications. Also, all deposited films can be used in hard coating systems and anti-reflections coatings due to low refractive and absorbance values in visible region according to glass slides. Our experimental analysis results show that optical and structural properties of AlN thin films can be adjusted to desired values by changing nitrogen concentrations in mixed Ar-N_2 gas plasma.

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