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Influence of Sputter Deposition Time on the Growth of *c*-Axis Oriented AlN/Si Thin Films for Microelectronic Application

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ABSTRACT

Aluminum Nitride films were grown on and Si (100) substrate by DC reactive magnetron sputtering at room temperature. Influence of sputter deposition time on properties of the AlN films was studied. Structural optical and electrical properties of the film were investigated. XRD measurements showed the presence of hexagonal wurtzite structure. The optical reflectance spectra of the film were taken and the band gap calculated varied from 4.35 to 5.3 eV. Finally MIS capacitors were fabricated on silicon substrates and variation of dielectric parameter with deposition time was reported.

Keywords: Aluminum Nitride; Thin Films; Sputtering; Preferential Orientations

1. Introduction

Aluminum Nitride (AlN) and its alloys are the most attractive material investigated over the past several years [1-3]. Growth of high-quality epitaxial wurtzite ALN thin film on different substrates have been of great interest due to their peculiar features such as high electrical resistivity (10^{11} - 10^{13} ohm·cm), high thermal conductivity, high hardness (11 - 15 GPa), wideband gap 6.0 - 6.2 eV and high velocity of acoustic waves etc. The combination of these characteristics makes AlN thin films a promising material for many electronic, optoelectronic, acoustic devices such as surface acoustic wave (SAW) [4,5]. The characteristics of AlN films are greatly influenced by their microstructure. Growth of smooth surface and defect less structure were the goal of device development. Various deposition techniques and substrates have been employed in an attempt of achieving high quality growth; the most frequently used substrates are Al_2O_3, SiC, Si (111) and Si (100). The later is of special interest because of the possibility to integrate with the contemporary Si device technology for different functions, e.g. UV detection or emission and Si-ICs on a common substrate.

In the past years several methods have been developed to prepare AlN thin films, such as chemical vapor deposition (CVD), molecular beam epitaxy (MBE), pulsed laser deposition (PLD), ion-beam assisted deposition and reactive sputtering [6-9]. However, these methods are expensive and the required high temperatures to reach satisfying properties are often incompatible with microelectronic processes. Among other techniques, the reactive magnetron sputtering process (RMS) is an attractive deposition technique because it presents advantages of being low temperature and low cost methods, and it allows fine-tuning of the material characteristics [10-12].

2. Experimental

AlN films were deposited with a 99.5% pure Al target DC magnetron sputtering system operated at 60 W DC cathode power and in pure Ar and N_2 gas mixture, which was introduced into the chamber by separate mass flow controllers. The deposition of ALN films can take place in a wide range of temperatures from room temperature up to 400°C. High temperature deposition has the disadvantage of producing degradation of the substrate, incorporation of impurities and thermal damage to the growing film. Hence deposition of AlN films at low temperature has become increasingly important and value [13]. Therefore AlN films were grown on (100) oriented silicon substrate at room temperature. Degreasing of glass was carried out ultrasonically in successive baths of acetone, ethanol and deionized water, and the native oxide on the silicon wafer was removed through an etching step in dilute HF solution. After this initial cleaning process, high purity Ar gas was introduced into

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the chamber and the chamber was evacuated to below 1×10^{-5} mbar. Then prior to each run the target was pre-sputtered with argon gas for 5 minutes with the target shutter closed. During pre sputtering DC power and Ar pressure was kept constant at 60 W and 1×10^{-5} mbar respectively, then nitrogen was introduced into chamber and reactive sputtering was initiated. AlN films were grown on Si substrate for different deposition time keeping the other deposition parameters constant. A summary of deposition parameters and ranges used in AlN thin films is listed in **Table 1**. For electrical measurements, Metal-insulator-semiconductor (MIS) structures were formed by sputter deposition through a metal mask of Al dots on AlN film as top electrode and a continuous Al film on Si wafer backside as bottom electrode.

The thickness of the films measured using stylus profilometer for 2, 4, 6, 8 minutes of deposition time were 49, 81, 102, 114 nm respectively. The type of crystalline structure, orientation and grain size was examined by X-ray diffraction (XRD). Optical studies were carried out by UV-visible spectrometer and band gap was calculated from the obtained reflectance spectra. Finally dielectric parameters were measured using impedance analyzer in frequency range 1 KHz to 1 MHz at room temperature.

3. Results and Discussion

3.1. XRD Analysis

AlN films were deposited on Si (100) substrate for different deposition time, keeping the sputtering pressure, target power, substrate target distance and nitrogen concentration constant. XRD analysis were carried out using RIGAKU X-ray diffractometer employing Cu K$_\alpha$ (1.5406 Å) radiation; θ - 2θ scans were performed with step size of 0.05 at a scan speed of 3 sec per step in the range of 10 - 80. The diffraction planes in θ - 2θ method are parallel to the sample surface. Accordingly, this method is very helpful in studying preferred orientation in thin film materials when compared to grazing angle incidence method [15]. The influence of deposition time on the crystalline orientation of AlN films has been investigated. All the deposited films were found to have hexagonal wurtzite structure. **Figure 1** shows the XRD pattern of AlN films on Si substrate for different deposition time. The micro structural properties and the lattice disorders are studied from the analysis of XRD peaks, and are listed in **Table 2**. The mean crystallite size was found to be ~60 nm. By substituting the measured crystallite size, the strain in the films can be determined using the following equation $\varepsilon = (\beta/\tan\theta) - (k\lambda/\sin\theta)$. In addition, it is evident that, both strain and dislocation density increases with increase in deposition time, which reveals the increase of particle collision due to the augmentation of stress in the lattice.

The crystal orientation of AlN films was strongly dependent on deposition conditions. It was found that under low deposition time of 2 min the film presents features at 32.98° and 36° corresponding to (100). As the deposition time increased to 4 min and 6 min, the intensity of AlN (100) plane is decreased, while the intensity of AlN (002)

Table 1. AlN deposition parameters.

| Parameters	Values
Deposition process	DC magnetron sputtering
Magnetron	Disc
Area of cathode (target), inch	2
Material of the target	Al
Discharge voltage, V	510
Discharge current, mA	117
Substrate	Silicon (100)
Substrate temperature in Kelvin	300
Working gases	Ar + N$_2$
Initial pressure in chamber, mbar	1.3×10^{-4}
Partial pressure of active (N$_2$), mbar	5×10^{-5}
Total pressure in the chamber, mbar	1×10^{-3}
Distance between the cathode and anode, cm	4
Deposition time, minutes	2, 4, 6, 8
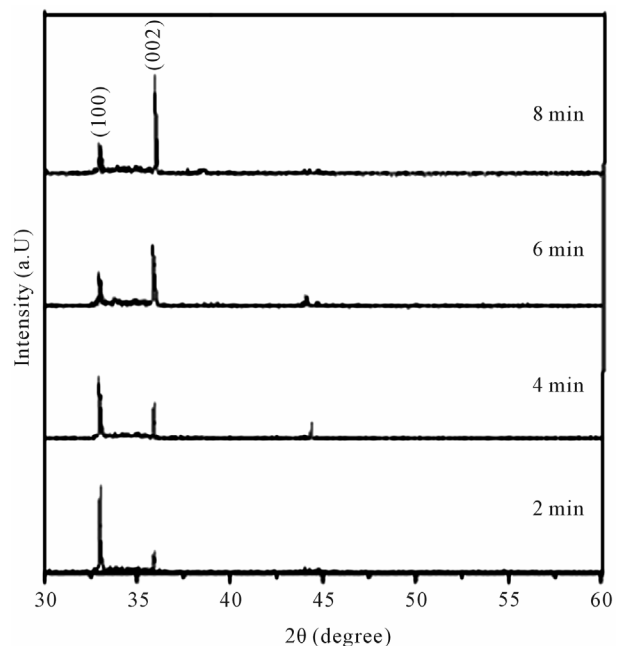
Figure 1. X-ray intensity vs. the diffraction angle 2θ of AlN thin films deposited on Si (100) substrate at different deposition time: (a) 2 m; (b) 4 m; (c) 6 m; (d) 8 m.

Table 2. Structural parameters calculated from XRD data.

Time min	d spacing (d_{hkl}) Å	Crystallite size (D) nm	Strain (ε)	Dislocation density (δ) lines/m^2
2	2.7137	61.40	4.48×10^{-4}	2.8492
4	2.7105	61.40	4.82×10^{-4}	2.9491
6	2.7121	57.47	5.47×10^{-4}	3.3807
8	2.7121	57.47	5.78×10^{-4}	3.8147

plane is enhanced. Further increasing the deposition time to 8 min resulted in films with highly oriented (002) (JCPDS file No. 00-0025-1133), with less intense AlN (100) plane.

The mechanism of preferential orientation of AlN film can be explained by the crystalline lattice structure of AlN. The hexagonal wurtzite structure of AlN has two kinds of AlN bonds named B_1 and B_2 [16]. The formation energy of the B_2 bond is larger than that of B_1. {100} plane consists of only B_1 bonds, while {002} and {101} consist of B_1 and B_2 bonds together. Accordingly ad atoms with more energy favor the formation of {002} surface plane. Another factor, which strongly influences the formation of preferred surface planes, is packing habit of such planes. For the development of close-packed surface planes the ad atoms need a longer time to accommodate themselves into the low energy configured lattice sites before the arrival of the next layer of reactive species. A low deposition rate is necessary in such cases [13]. Hence, we predict that at a deposition time of 8 min, the coated film has low deposition rate, which favors the formation of highly oriented (002) preferential plane with enhanced crystal quality, which can provide good piezoelectric response.

3.2. Optical Properties

The optical reflectance of the films has been recorded employing a Perkin-Elmer Lamba-950 spectrometer. The optical energy band gap of AlN films is determined by analyzing the optical data with the expression for the reflectance and photon energy using the Tauc relation

$$2\alpha h\nu = A\left(h\nu - E_g\right)^{n/2} \quad (1)$$

where n is the constant, which is equal to one for direct band gap semi-conducting material and four for and indirect band gap semi-conducting material. $h\nu$ is the photon energy, and α the absorption coefficient, which can be written in terms of reflectance as $2\alpha t = \ln[(R_{max} - R_{min})/(R - R_{min})]$ where t is the thickness of the sample and R the reflectance for any intermediate photon energy. **Figure 2** shows the reflectance spectra of AlN thin film on Si substrate for different deposition time. A fall in reflectance is observed from R_{max} to R_{min} due to absorption of light by the material. **Figure 3** shows, a graph is
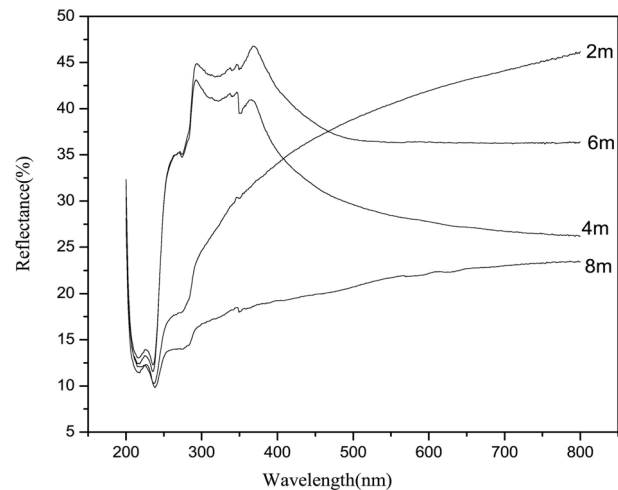
Figure 2. Reflectance spectra of AlN thin film on Si (100) at different deposition time: (a) 2 m; (b) 4 m; (c) 6 m; (d) 8 m the films.
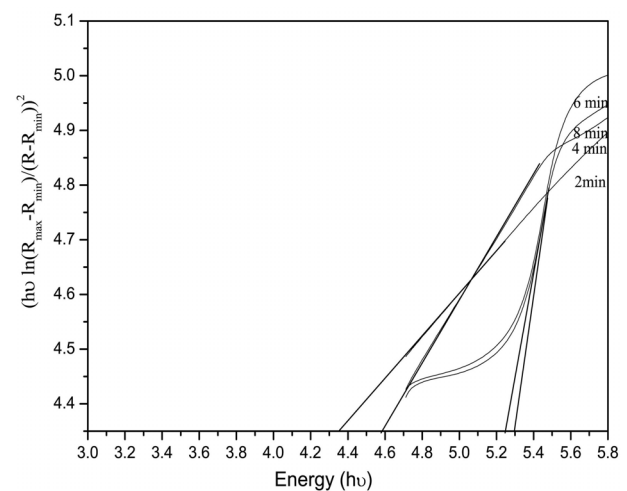
Figure 3. Graph between $(\alpha h\nu)^2$ and $h\nu$.

plotted between $(\alpha h\nu)^2$ and $h\nu$ using Equation (1). The extrapolation of linear portion of the plot of the energy axis $(\alpha h\nu)^2 = 0$ gives the value of the energy band gap of the thin film. It is found that the energy band gap of the AlN film varies from 4.35 to 5.3 eV. It is clear that the energy band gap of AlN films increases with increase in the deposition time. This variation in band gap is attributed to the internal strain in the films which increases

with the deposition time as shown in the **Table 2**. The refractive index (η) of the sputter-deposited AlN is dependent on the sputtering conditions since it is related to the composition and density of the films.

For polycrystalline AlN films obtained by DC reactive sputtering, the refractive index varies from 1.9 to 2.1. The refractive index of our AlN samples, determined from the dielectric measurement of ε and δ, revealed that they are in the range of 1.1 - 1.2, for the AlN samples prepared at a deposition time of 8 minutes. The decrease in the refractive index values is observed in the presence of impurities (such as oxygen) or nitrogen vacancies present in the film [17].

3.3. Electrical Properties

The variation of the dielectric parameters, such as C, ε and tanδ of MIS capacitors with deposition time was studied in the frequency range from 10 KHz - 1 MHz using impedance analyzer at room temperature. Plots of the dielectric constant ε, loss factor tan δ and capacitance C as a functions of the frequency for different deposition time of AlN films are shown in **Figures 4(a)-(c)**. The capacitance of the thin insulating film almost remains constant in the frequency range from 10 KHz - 1 MHz, with its value varying from 0.8×10^{-8} to 1.8×10^{-7} μF. It is observed that, as the deposition time increases the capacitance decreases with least value at 8 minutes of deposition time.

The dielectric constant of AlN films was between 6.0 and 6.8, which is similar with reported values for AlN [15]. The real part of dielectric constant increases with increasing deposition time and decreases with increasing frequency. The observed dependence of the dielectric constant on the dielectric film thickness (deposition time) for thinner films (less than ~1000) is attributed to defects such as voids, stresses, in homogeneity, grain boundaries, discontinuities etc, which are normally present in vacuum-deposited films, rather than to the non-stoichiometry of the deposits resulting from an excess of oxygen or metal atoms. Some of these defects are removed by self-annealing or aging processes, but others require more thermal energy such as is provided by an annealing process. As the films become thicker, the density of voids decreases, resulting in a higher value of dielectric constant, which evidently becomes thickness independent. Adam *et al.* [17] reported the dependence of dielectric constant on film thickness. The dielectric constant was between 4 and 11 for thicker layers ($\geq$100 Å) and decreased to values between 2 and 6 for thicknesses below 100 Å. And Dimitrova *et al.* [18] reported low dielectric constants (6.8 - 7.1) due to the presence of nitrogen vacancies in the AlN lattice.
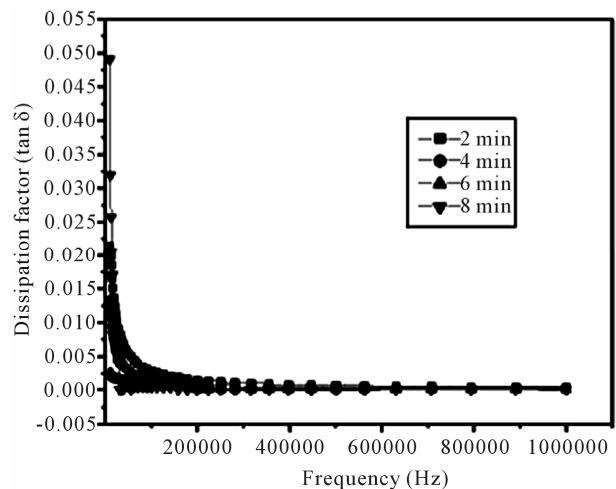
The dielectric dissipation factor tanδ of an insulating
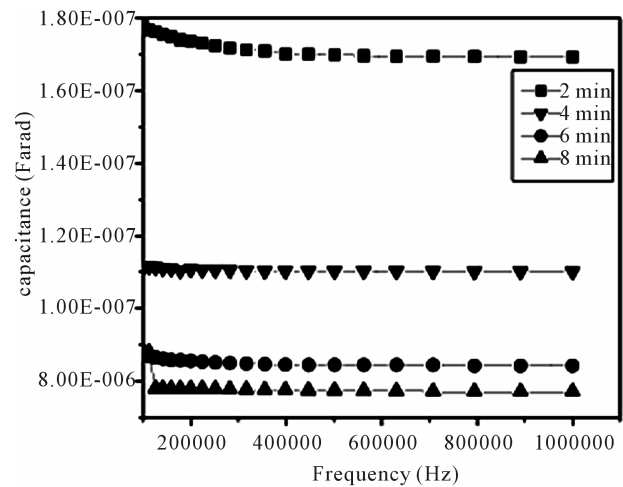
(a)
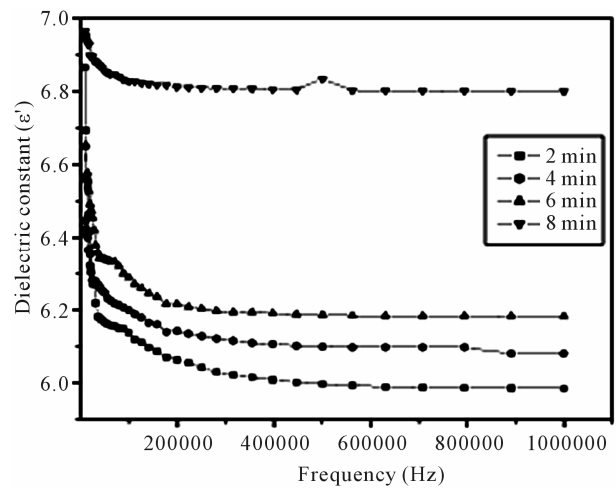
(b)

(c)

Figure 4. (a) Graph between capacitance and frequency for different deposition time; (b) Graph between dielectric constant and frequency for different deposition time; (c) Graph between dissipation factor and frequency for different deposition time.

material is the tangent of the loss angle δ. In a perfect dielectric, the voltage wave and the current are exactly 90° out of phase. As the dielectric becomes less than 100% efficient, the current wave begins to lag the voltage in direct proportion. The amount the current wave deviates from being 90° out of phase with the voltage is defined as the dielectric loss angle. The tangent of this angle is known as the loss tangent or dissipation factor [19]. Hence a good dielectric film should have minimum dissipation factor. In the present study, the dissipation factor varies from 0.0011 to 0.004 and is independent of the deposition time. It almost remains constant for all the films with different deposition time.

4. Conclusion

AlN films have been prepared by DC reactive magnetron sputtering on Si (100) substrates for different deposition time. The evolution of preferred orientation and morphology of AlN films deposited at 50% nitrogen concentration on Si (100) substrate was studied. The XRD analysis of the films revealed that, at a deposition time of 8 min, the coated film favored the formation of highly oriented (002) preferential plane with enhanced crystal quality, which can provide good piezoelectric response. The optical reflectance spectra show reflectance at 240 nm. The band gap increased with the increase in the deposition time and the values of refractive index were in the range of 1.1 - 1.2, for the samples prepared at 8 min of deposition time. The MIS structures fabricated using AlN at different deposition time showed a significant improvement of electrical characteristics with deposition time as we go from 2 min to 8 min.

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