Solid State Science and Technology, Vol. 23, No 1 & 2 (2015) 64-72
ISSN 0128-7389 | *http://journal.masshp.net*

GROWTH AND CHARACTERIZATION OF ALN THIN FILM USING THE SPIN COATING METHOD: EFFECT OF ANNEALING TEMPERATURE

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ABSTRACT

This paper reports the growth and characterization of aluminum nitride (AlN) thin film deposited on n-type silicon n-Si(100) using a versatile and cost effective method namely spin coating. The work focuses on the effects of annealing temperature on the structural, surface morphological and optical properties of the deposited AlN thin films. The optical properties of the deposited thin film were determined by using Fourier transform infrared spectrometer. This paper reports the growth and characterization of aluminum nitride (AlN) thin film deposited on n-type silicon n-Si(100) using a versatile and cost effective method namely spin coating. The work focuses on the effects of annealing temperature on the structural, surface morphological and optical properties of the deposited AlN thin films. The optical properties of the deposited thin film were determined by using Fourier transform infrared spectrometer. The optical phonon corresponds to the E_1(TO) of the AlN peak is close to the characteristic value of AlN at 670 cm^{-1}. For X-rays diffraction, diffraction peak at $2\theta = 33°$ corresponds to AlN (100) plane for the sample annealed at 650 °C is clearly observed. While from the field-emission scanning electron microscopy images, the surface morphology of AlN thin films exhibited distinct features of nano-sized crystalline grains. The energy dispersion X-rays spectroscopy results showed the presence of nitrogen, oxygen, aluminum, and silicon elements in the films. Finally, the atomic force microscopy results revealed that the rms surface roughness of the films decreases as the annealing temperature increases.

Keywords: AlN; Thin Film; Spin coating; Silicon

INTRODUCTION

In recent years, aluminum nitride (AlN) thin films are becoming more popular semiconductor in a wide variety of applications such as potential dielectric and passivation layer for gallium arsenide (GaAs) devices, heat sink in electronic packaging applications, component of surface acoustic wave devices [1], and optical coating for spacecraft components [2]. In addition, AlN helps to extend the lifetime of moving mechanical components due to its potential as a wear-resistant hard coating. Besides, AlN thin films are attractive materials for the application as high temperature microelectronic and optoelectronic devices due to its unique properties such as wide band gap (6.2 eV) [3] and high acoustic velocity [4].

Moreover, AlN also possesses some unique properties like moderately high electromechanical coupling coefficient [5], high temperature stability [6], high resistivity, high dielectric constant [7] and low coefficient of thermal expansion. High heat dissipation of AlN can significantly enhance lifetime and efficiency of semiconductor devices. Furthermore, its lattice matching is near to that of silicon and thus less stress is expected to be generated at the AlN/silicon interface. Owing to these properties, AlN films have received a great interest as an electronic material for thermal dissipation, dielectric and passivation layers for integrated circuits, acoustic devices, resonators and optoelectronic devices [8].

From the past, different techniques have been used to grow AlN thin films. These include molecular beam epitaxy, chemical vapor deposition, radio frequency or direct current sputtering [9, 10], pulsed laser deposition [11] and spin coating [12]. In this project, spin coating method has been chosen for the growth of AlN thin films. Spin coating is a simple technique requires considerably less equipment and is potentially less expensive [13] compared to the conventional thin film growth methods as stated above. In general, the annealing temperature is an extremely important growth parameter in influencing the crystal structure of the crystalline material. In this study, AlN thin films were deposited on n-Si (100) substrate by spin coating method followed by the nitridation process prior to the nitridation process, the deposited films were subjected to various annealing temperatures from 250°C to 850°C. The optical properties of the deposited thin films were determined by using Fourier transform infrared (FTIR) spectrometer; and while the structural and surface morphology properties of the thin films were investigated by X-rays diffraction (XRD), field-emission scanning electron microscopy (FESEM), energy dispersive X-rays spectroscopy (EDX) and atomic force microscopy (AFM).

EXPERIMENTAL METHODS

Aluminum nitride thin films were deposited on n-Si (100) substrates by using the spin coating method. Aluminum nitrate hydrate powder was used as aluminum source. Firstly, these powders ware dissolved in the ethanol. Subsequently, a suitable surfactant was slowly added. The precursor was stirred to yield a clear and homogeneous solution. The n-Si (100) substrate was treated with atmospheric plasma prior to the spin coating process. Later, the precursor solution was spin-coated on to the substrate and rotated at 3000 rpm for 30 s. Then, the film was dried through soft baked process on a hot plate at 60 °C for a few minutes. Both the spin-coating and soft baked processes were repeated for a few times to achieve the desired total thickness. The thin film was inserted into a furnace and annealed at various annealing temperatures from 250 °C to 850 °C for two hours. After the annealing process, the films were nitridated in a tube furnace at 1100 °C for one hour under the flow of ammonia (NH_3) gas at 500 sccm. After the completion of the reaction, the furnace was allowed to cool down naturally to room temperature. During the cooling process, nitrogen gas was flown into the tube in order to flush out the excess NH_3.

The optical properties of the deposited thin films were determined by using a FTIR spectrometer (Spectrum GXFT-IR, Perkin-Elmer) whereas the structural properties of the thin films were investigated by XRD (PANalyticalX'Pert Pro MRD) with a Cu-kα_1 radiation source (λ=1.5406Å). The surface morphology and elementary analysis of the thin films were examined by FE-SEM (NOVA NANO SEM450), AFM (Dimension EDGE, BRUKER) and EDX (NOVA NANO SEM450).

RESULTS AND DISCUSSION

FTIR is an effective technique to investigate the characteristic vibrational modes of the lattice [14]. Figure 1 depicts FTIR reflectance spectra for AlN thin films deposited on Si (100) substrates with different annealing temperatures over the entire range from 400 to 7000 cm^{-1}. It is observed that the optical phonon corresponds to the E_1(TO) of the AlN located at approximately 695 cm^{-1}, 696 cm^{-1}, 680 cm^{-1} and 692 cm^{-1} for annealing temperatures at 250 °C, 450 °C, 650 °C and 850 °C, respectively. Obviously, the peak E_1(TO) shifted towards higher wave number after annealed at all different temperatures, but these peaks are quite close to the characteristic value of AlN at 670 cm^{-1} [15]. The strongest AlN peak was observed for the film annealed at 650 °C. As compared to the reported values, it was found that the detected E_1(TO) and A_1(LO) modes display an upward frequency shift of 10 cm^{-1} and 20 cm^{-1}, respectively. This was most probably associated with the crystalline quality of the deposited thin film. The high frequency shifts of the A_1(LO) mode was attributed to the longitudinal phonon-plasmon coupling effect [16]. This phenomenon was most likely induced by the unintentionally doped carriers in the AlN thin film using present technique [17].

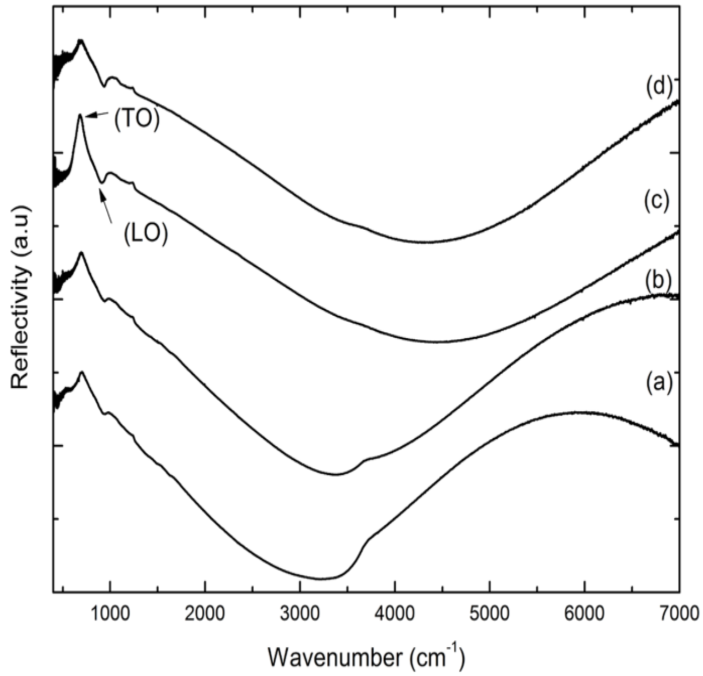

Figure 1: FTIR reflectance spectra of AlN thin films with annealing temperatures: (a) 250 °C, (b) 450 °C, (c) 650 °C and (d) 850 °C

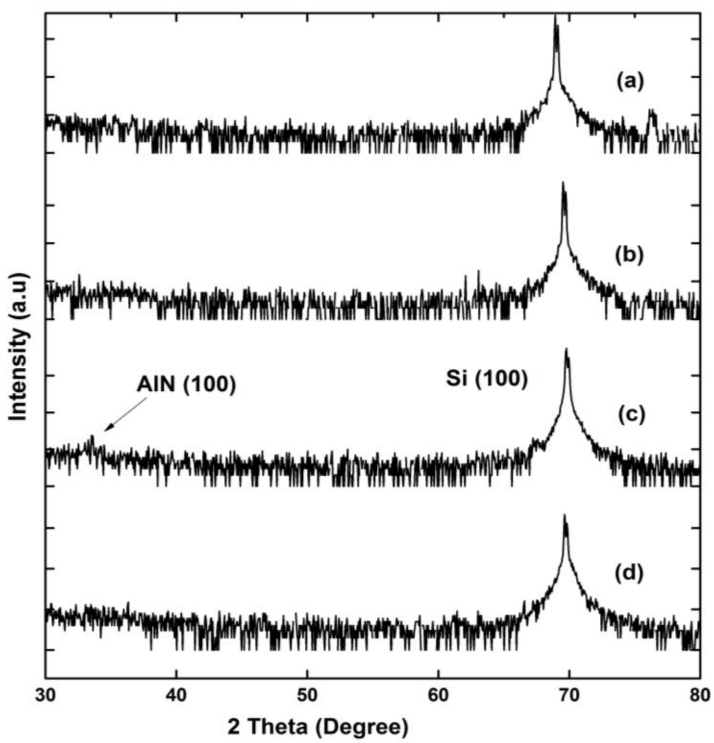

Figure 2: XRD patterns of AlN thin films with various annealing temperatures: (a) 250°C, (b) 450 °C, (c) 650 °C and (d) 850 °C

Figure 2 shows the XRD patterns of the deposited AlN thin films grown at various annealing temperatures. For the thin films annealed at 250 °C, 450 °C and 850 ^{o}C, only peak corresponds to Si (100) plane can be observed. However, it was found that there is a weak diffraction peak at 2θ = 33° corresponds to AlN (100) plane for the sample annealed at 650 °C. However, the AlN (100) diffraction peak cannot be observed for samples annealed at 850^{o}C. This is most probably due to the high annealing temperature has reduced the crystalline quality of the AlN [18]. Note that the formation of AlN at other orientations was not observed in the XRD patterns. This might probably due to the thickness of the deposited film is too thin. Besides that it might also attributed to the formation of the AlN amorphous/polycrystalline structure. Based on the results, it can be concluded that 650 ^{o}C was the most suitable annealing temperature for growth the AlN thin films using spin coating method.

Figure 3 shows the FESEM images of the deposited AlN films on n-Si (100) substrates at various annealing temperatures. It can be seen that the surface morphology of AlN thin films showed distinct features of nano-sized crystalline grains. At annealing temperature of 250 °C, the deposited AlN thin film showed a very smooth and uniform surface. However, as the annealing temperature increases to 450 °C, the obtained film surface was smooth, uniform and with the existence of pores, which indicates the formation of larger grain size. At annealing temperature of 650 °C, the film showed densely packed grains; the surface morphology was clearly observed. The increased in grain size demonstrated that the quality of the surface morphology of the film has enhanced with the increasing of annealing temperature. On the other hand, at annealing temperature of 850 °C, the appearance of bright spotted grains structure was found on the film surface. In addition, the cracking features can be observed in all of the thin films after the annealing process. This is due to the large difference of the thermal expansion coefficient (TEC) between the AlN thin film and Si substrate [13]. In the case where the AlN thin film consists of larger TEC than the substrate, the deposited film experiences compressive stress. The stress introduced the compressive or tensile during the annealing process is known as thermal stress [13]. Hence, the deposited thin films may form cracks or other defects to relieve excessive stress. This usually happens when the temperature increases drastically and vice versa.

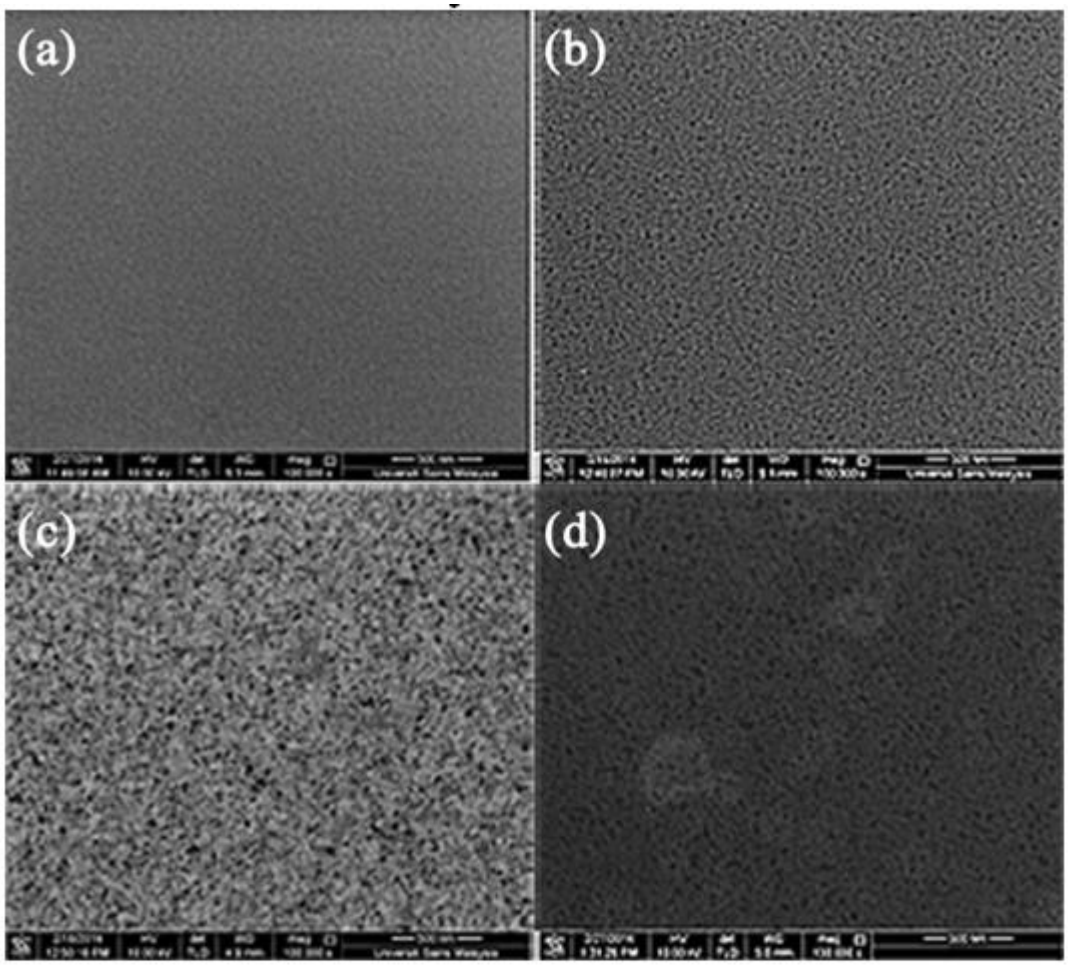

Figure 3: FESEM images of AlN thin with different annealing temperatures: (a) 250 °C, (b) 450 °C, (c) 650 °C and (d) 850 °C

Figure 4 shows the AFM images of AlN thin films with different annealing temperatures measured under scan area of 5 µm × 5 µm. As clearly shown in the figures, all of the samples annealed at different temperatures have a sharp granular surface. The root-mean-square (rms) surface roughness of the deposited AlN thin films annealed at 250°C, 450°C, 650°C and 850 °C were 3.31, 2.98, 2.07and 1.83 nm, respectively. Based on the obtained rms surface roughness values, it was revealed that the rms surface roughness was decreased as the annealing temperature increased.

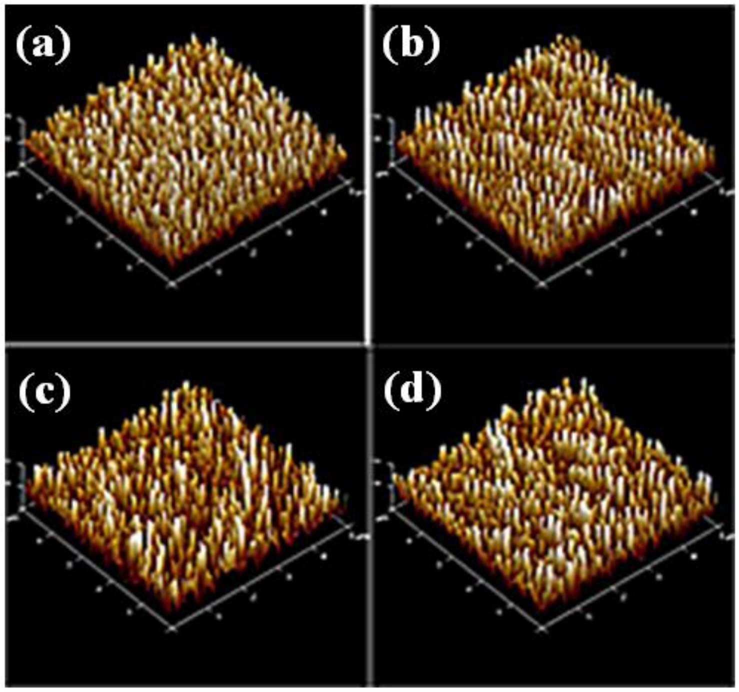

Figure 4: AFM images with of AlN thin films with different annealing temperatures: (a) 250 °C, (b) 450 °C, (c) 650 °C and (d) 850 °C

Table 1 shows the EDX analyses for AlN thin films annealed at different temperatures. This part focuses on the atomic percentage of the elements that presence in the films. The results show the presence of nitrogen (N), oxygen (O), aluminum (Al) and silicon (Si) elements in the films. The presence of the oxygen in the deposited thin film was associated to the common contamination of AlN in non-equilibrium growth techniques such as spin coating [19]. Figure 5 shows the fluctuation pattern of the atomic percentage for Al, N and O elements with the increasing of annealing temperatures. However, there was a steady decreasing of the atomic percentage of Al element in the films with increasing annealing temperatures from 250°C to 850°C, which indicates the mass-loss has happened during the process.

Table 1: The EDX atomic percentage of AlN thin films on Si substrates with different annealing temperatures

Sample	Element (atomic %)			
	Nitrogen	Oxygen	Aluminum	Silicon
250 ^{o}C	21.62	29.14	27.53	21.71
450 ^{o}C	6.05	46.51	27.38	20.07
650 ^{o}C	21.57	23.26	21.36	33.81
850 ^{o}C	9.52	36.23	20.83	33.41

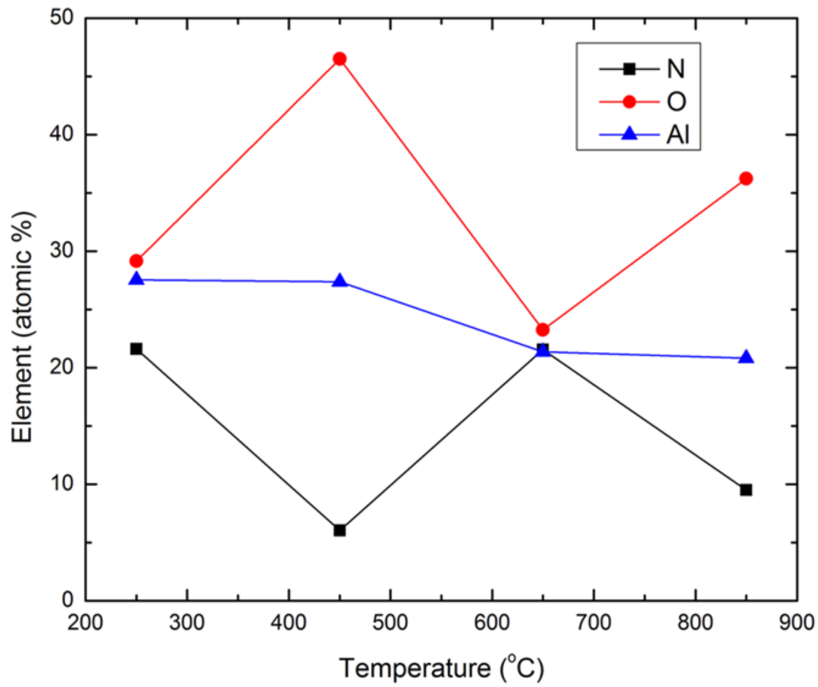

Figure 5: The fluctuation of the atomic percentage of the elements presence in AlN thin films as a function of the annealing temperature

CONCLUSION

In conclusions, the AlN thin film was deposited on n-Si(100) by spin coating method. The effects of annealing temperature on the structural, surface morphologies and optical properties of thin films were systematically investigated. The obtained results also revealed that annealing temperatures play an important role for the growth of AlN thin films and the temperature of 650 ^{o}C was the most suitable annealing temperature for the growth of AlN thin film. At this temperature, characteristics of the AlN thin films were effectively improved. Overall, the results showed that the spin coating is a feasible technique to produce AlN thin film. Although the quality of the thin film produced by this method was not comparable with that produced by the traditional growth methods, the spin coating method has its own advantages, i.e., it is a relatively simple and easy method. However, further optimization need to be done to improve the crystallite quality of the deposited thin films.

ACKNOWLEGMENT

The authors are grateful for the financial support given by the Ministry of Higher Education, Malaysia through the Fundamental Research Grant Scheme (FRGS) (Account no. 203/PFIZIK/6711282).

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ISSN 0128-7389 | *http://journal.masshp.net*

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