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Synthesis of aluminum nitride thin films and their potential applications in solid state thermoluminescence dosimeters

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ABSTRACT

In this work, aluminum nitride thin films were deposited on Si (1 1 1) substrate by magnetron sputtering. The obtained film was studied for thermoluminescence after irradiating it to various doses of γ-rays. Thermoluminescence measurement showed photon emission at an irradiation dose of 100 Gy or higher. Deconvolution of the experimental glow curve indicated that recombination centers in AlN were present below 2 eV trap depth. Irradiated AlN films showed less than 2% fading of TL signals on storage for 1 month in dark conditions and for the same period, light induced fading was also less than 4%. A linear variation of integrated thermoluminescence counts with absorbed dose has been observed up to an irradiation dose of 10 kGy. The deposited film was also characterized by grazing incidence X-ray diffraction, atomic force microscopy and secondary ion mass spectroscopy. Grazing incidence X-ray diffraction measurement of the obtained film has shown formation of polycrystalline wurtzite AlN having preferred orientation along (1 0 0) plane. Secondary ion mass spectroscopy analysis revealed the presence of oxygen in the film.

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1. Introduction

Aluminum nitride is a III–V family semiconductor, which exhibits an excellent combination of physical, chemical and mechanical properties. It can be grown in hexagonal (wurtzite) and cubic (zinc blende and rock salt) crystal structures, also called as α-AlN and ß-AlN, respectively [1]. High quality films of AlN have been used for fabrication of optical, optoelectronic, and acoustic devices [2,3].

For the past several years, extensive work has been done on growth and characterization of AlN films. Films of AlN have been grown by several methods, which include molecular beam epitaxy [4], pulsed laser ablation [5], reactive sputtering [6] and also chemical vapor deposition technique [7]. Most of the properties of AlN like hardness, thermal conductivity, electrical resistivity, and optical and dielectric constants are known today. However, as far as the thermoluminescence (TL) properties are concerned, a few studies based on ultraviolet (UV) and ionizing radiations are available mainly on powder and bulk form of AlN, but no work has been reported on thin films of AlN. Also, the dosimetric applications of AlN have not been well highlighted so far. Among the available work, Benabdesselam et al. [8] and Weinstein et al. [9] reported thermoluminescence of AlN powder and single crystals synthesized from the fine powders of AlN respectively, due to the UV radiation excitation. Trinkler et al. [10,11] showed TL and optically stimulated luminescence (OSL) response of sintered AlN–Y_2O_3 ceramic, after irradiating the sample in ionizing radiations. Similarly, Tanaka et al. [12] also reported γ-ray thermoluminescence in AlN. In these reported works, mostly the mechanism of thermoluminescence has been discussed whereas suitability of AlN as a candidate material for TL dosimetry has not been brought out.

Apart from the bulk, thin films of materials are also being explored for dosimetry applications e.g. carbon doped films of α-Al_2O_3 and diamond films have been found suitable for fabrication of TL/OSL dosimeters [13,14]. Therefore, in the present work, thin films of AlN prepared by magnetron sputtering have been explored for TL dosimetry. Magnetron sputtering has excellent reproducibility; hence films of identical properties can be easily obtained using this technique. Further, the process can be scaled up for production of batches of AlN thin films.

2. Experimental

A pulsed dc reactive balanced magnetron sputtering system coupled with asymmetric bipolar dc generator (reverse bias

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voltage = +36 V) was employed to deposit AlN with a 99.999% pure aluminum disk used as a target. Chemically cleaned single crystal silicon wafers (dimension: 10 mm × 10 mm × 0.5 mm) having (1 1 1) orientation were used as the substrate. Final cleaning of the substrate was done in situ by Ar^+ ion bombardment for 30 min at a negative substrate bias of 800 V and a pressure of 10^{-1} mbar. Here, it may be noted that the kinetic energy of Ar^+ ions in plasma is not sufficient to cause any appreciable mass loss from the substrate and it only removes loosely bonded oxide and dirt particles remaining after chemical cleaning. This was confirmed practically by comparing the weight of the chemically cleaned uncoated substrate and the chemically cleaned uncoated samples underwent plasma cleaning step also. Also, the average roughness of the substrate surface after plasma cleaning was not significantly higher than before. Deposition of AlN was carried out in an atmosphere of argon and nitrogen plasma, at a working pressure of 1.5×10^{-3} mbar, for nitrogen/argon flow rate ratio varied from 4:6 to 7:3. Prior to the deposition of AlN, a thin interlayer of aluminum was sputtered on the substrates to enhance the bonding/adhesion by minimizing the lattice mismatch between the film and the substrate. Numerical values of various deposition parameters are listed in Table 1.

Average thickness of the deposited films was calculated by the weight gain method, assuming the density of deposited AlN films to be the same as that of bulk AlN (3.26 g/cm^3). Further, the thickness value reported in this work is indicative only as porosity in the film was ignored in thickness calculations.

Crystal structure and preferred plane of growth of AlN were analyzed by grazing incidence X-ray diffraction (GIXRD) measurement at mono-chromatized CuK_α ($\lambda = 0.154$ nm) wavelength for incidence angle of 0.7°. The experimental GIXRD patterns were matched with JCPDS database (Card no. 25-1133) and peaks of aluminum nitride were identified. Surface morphology and roughness were analyzed by atomic force microscopy (AFM) in semi-contact mode using a silicon cantilever of radius of curvature 10 nm. Chemical composition of the film was measured by secondary ion mass spectroscopy (SIMS) (indigenously developed in Technical Physics Division, BARC, Mumbai, India). The sample was bombarded with a pulsed liquid metal ion source ($^{69}Ga^+$), at energy of 20 keV. For obtaining the composition–depth profile, an argon ion etch gun at 5 keV was interlaced with the primary ion gun. The secondary ion spectra were acquired from an area of 150 μm × 150 μm.

TL measurements were performed using a programmable integrated TL reader system. The TL reader system consists of a sample holder (dimension: 12 mm × 12 mm) made up of 0.4 mm thick Kanthal strip which functions as a heater planchet. A PID controller controls the temperature of the heater strip with an accuracy of ±0.1 °C, using the feedback received from the thermocouple spot welded on the strip [15]. For recording the TL counts, a photon counting module (EMI P25232) interfaced with a computer and equipped with a blue–green sensitive bialkali photocathode having an average dark count of 13 cps was used [15]. During TL measurement, the sample was heated up to 400 °C at a linear heating rate of 4 K/s. Calibrated ^{60}Co was used as a source of γ radiation and the sample was irradiated at a dose rate of 22 Gy/min.

Table 1
Deposition condition for growth of AlN thin film.

Parameters	Numerical value
Base pressure	1.1×10^{-5} mbar
Power density of cathode	3.3 W/cm^2
Pulse frequency	125 kHz
Duty cycle	75%
Target-to-substrate distance	7.5 cm
Total gas (Ar+N_2) flow rate	10 sccm
Substrate temperature	No external heating (~100 °C)
Substrate biasing	50 V
Deposition time for Al interlayer	10 min
Thickness of Al interlayer	0.2 μm
Deposition time for AlN	4 h

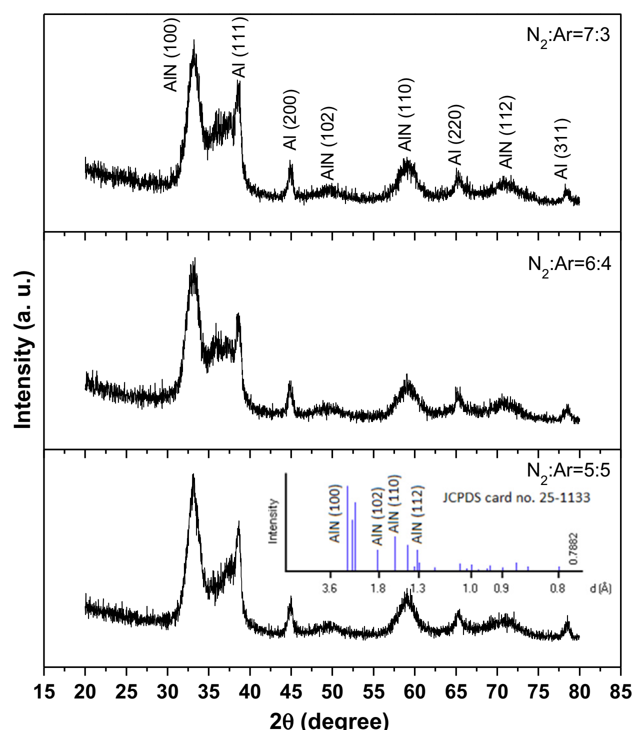

Fig. 1. GIXRD pattern of aluminum nitride films deposited on Si (1 1 1) substrate at various nitrogen/argon flow rate ratios. Film thickness: 1.3 μm at 5:5 flow rate ratio, whereas 1.2 μm at 6:4 and 7:3 flow rate ratios.

3. Results and discussion

3.1. Structural and morphological properties

Fig. 1 shows GIXRD pattern of 1.2–1.3 μm thick AlN films deposited at three different flow rate ratios of nitrogen/argon. For comparison, standard JCPDS of wurtzite AlN is also shown in the inset of Fig. 1. From the GIXRD pattern it is clear that AlN has been crystallized in polycrystalline wurtzite crystal structure (P_{63mc} space group) showing the reflections of (1 0 0), (1 0 2), (1 1 0) and (1 1 2) planes, out of which (1 0 0) was the strongest. Moreover, there is no change in crystal structure and orientation of planes of AlN on varying the nitrogen/argon flow rate ratio. However, besides the reflections of various planes of AlN, reflections of (1 1 1), (2 0 0), (2 2 0) and (3 1 1) planes of Al interlayer were also observed.

Fig. 2(a)–(d) shows AFM micrograph of AlN films prepared at 4:6, 5:5, 6:4 and 7:3 flow rate ratios of nitrogen/argon in order. At lower flow rate ratios, conical shape grains with a wide apex angle were observed that turned to spherical shape on increasing the flow rate ratio. Value of average roughness (R_a) for these films was in the range 7–47 nm and a decrease in surface roughness on increasing the nitrogen/argon flow rate ratio was observed. This observation can be explained as follows. During sputtering, Ar^+ ions are much more effective in ejecting Al atoms from Al target in comparison with lighter N_2^+ ions due to better

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