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Preparation and characterization of AlN thin films deposited on Si(100) using single source precursor

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Hexagonal aluminium nitride thin films have been prepared on Si(100) by suspension coating pyrolytic method using a new single organo metallic precursor - dichlorophenyl amido aluminium (DCPAA) at temperatures 650 ^{0}C and 950 ^{0}C. XRD studies have shown decrease of peak intensities of hexagonal aluminium nitride at 2θ = 38.3 ^{0} diffracted from (0002) crystal plane, at 2θ = 44.5 ^{0} diffracted from (1011) crystal plane and a comparative decrease at 2θ=68.2 ^{0} diffracted from (1120) crystal plane with increase of temperature. SEM studies have shown a smooth crystalline nature of AlN at 650^0 C but non-uniform crystalline nature at 950^0 C.

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1. Introduction

Aluminum nitride [AlN] thin films are of increasing interest for a number of applications in microelectronic field. Thin films of AlN are grown with strong interest as it has a direct bond gap of 6.2 eV, high thermal conductivity (320W/mk) and high decomposition temperature (2425 ^{o}C). Due to these excellent properties AIN thin films have a great potential for microelectronic devices [1], high power applications and utilization in caustic environments [2]. The initial interest in microcrystalline AlN was for the protection of steel in a salt water environment [3].

AlN is inert and highly transparent in the visible spectrum and hence can be used for protective optical coating. Recently aluminium nitride has attracted attention for use as insulating layers in semiconductor devices [4]. Because of wide direct band gap AlN is a promising material for integrated optics in the UV-region i.e. laser diode [5], detectors [6] and fabrication of frequency surface acoustic wave (SAW) devices [7]. Alloys such as AlGaN have potential applications in microelectronic and optoelectronics [8].

Aluminium nitride thin films have been prepared by various methods like chemical vapour deposition [9-10] ,plasma deposition [11], sputter deposition [12-14] and electron beam evaporation [15].

In this work, we show a new route to the deposition of AlN thin films on Si(100) using suspension coating pyrolytic method with a new single source precursor-dichlorophenyl amido aluminium (DCPAA). The structural properties of the films were investigated by X-ray diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR). The morphologies of the deposited films were studied by using Scanning Electron Microscopy (SEM). The effect of different substrate temperatures on AlN thin films on (100) Si was analyzed.

2. Experimental

The precursor - dichlorophenyl amido aluminium (DCPAA) was synthesised following the conventional solid-state reaction technique. The starting materials anhydrous aluminium chloride and aniline in the ratio 1:1 were taken in a silica crucible and the mixture was heated upto the boiling point of aniline (184 ^{o}C). The reaction occurred with evolution of HCl fumes.

$$C_6H_5NH_2 + AlCl_3 \rightarrow C_6H_5NHAlCl_2 + HCl$$

dichlorophenyl amido aluminium

AlN thin films were deposited on Si(100) substrates by using suspension coating pyrolytic technique. A 0.2 M suspension of precursor - dichlorophenyl amido aluminium was prepared in ethanol. Substrates used for the deposition were Si(100) wafers. The Si (100) wafers were ultrasonically decreased in an ethanol bath, etched in HF acid and subsequently rinsed in deionised water. These were then dried with N_2 gas.

Precursor suspension of 0.2 mol. in ethanol was taken in a precleaned silica crucible and Si(100) wafer was placed in it. The level of suspension was kept 2 mm above the wafer. On evaporation of ethanol a nearly uniform coating of precursor was obtained on Si (100) wafer. A set of precursor coated Si (100) wafers was introduced in the reaction chamber and heated to 650^{O}C in the atmosphere of ammonia under atmospheric pressure for three hours.

Similarly a set of precursor deposited Si (100) wafers was introduced in the reaction chamber and heated to 950 ^{o}C in the atmosphere of ammonia under atmospheric pressure for three hours.

The structures and morphologies of the as -deposited AlN thin films were analyzed by Rigaku Denki X-ray diffractometer with Cu Kα radiation and Hitachi S-2700 Scanning Electron Microscope respectively.

3. Results and discussion

Fig. 1 shows IR spectrum of dichlorophenyl amido aluminium precursor. A broad absorption band at 3450 cm^{-1} is due to N-H stretching vibrations. The absorption band around 2850 cm^{-1} is due to aromatic C-H stretching vibrations. The absorption band at 1500 cm^{-1} is due to C=C ring stretching vibrations. The absorption band at 750 cm^{-1} indicates out of plane bending C-H vibrations of monosubstituted benzene. A broad absorption band in the region 700-550 cm^{-1} with distinct peak at 680 cm^{-1} indicates Al-N bonding in the precursor. A sharp absorption band at 475 cm^{-1} is due to Al-Cl bonding.

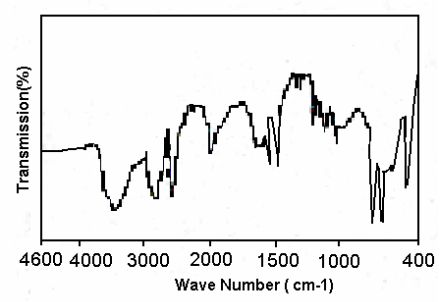


Fig. 1. IR spectrum of dichlorophenyl amido aluminium precursor.

Fig. 2 shows the X-ray patterns of AlN thin film deposited on Si(100) at 650 ^{O}C and Fig. 3 at 950 ^{O}C. All the peaks in the patterns correspond to hexagonal structure of aluminium nitride. The growth direction for AlN thin film is found to be (002). The orientation of the film is greatly controlled by its interaction with the substrate and by kinetics of growth process. This effect is probably due to the grain - boundary separating individual grains as reported by Dovidenko et. al.[16]. These planar defects are mainly generated in the closed - packed plane of AlN during the growth process [17]. No phase corrosponding to other aluminium compounds was detected in the XRD patterns.

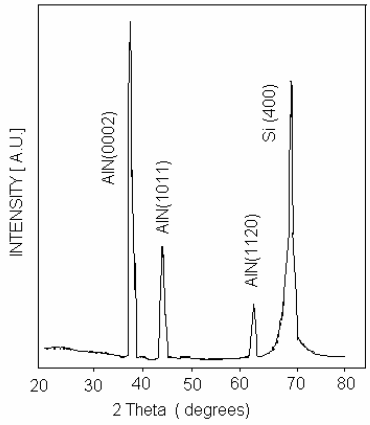


Fig. 2. XRD pattern of AlN thin film deposited on Si(100) at 650 ^{O}C.

Significant changes observed in the XRD patterns are (i) decrease the peak intensity corresponding to (0002) and (1011) planes and (ii)a comparative decrease in the peak intensity corresponding to (1120) plane with increase in temperature.

The XRD patterns at 650 ^{o}C show a characteristic peak of hexagonal AlN at $2\theta = 38.3^{0}$ diffracted from (0002) plane, smaller peak at $2\theta = 44.5^{0}$ diffracted from (1011) plane and a very small peak at $2\theta = 68.2^{0}$ diffracted from (1120) plane. This indicates that the film is polycrystalline with hexagonal structure and is relatively highly oriented in (002) direction[18]. There is decrease in intensities of the peaks at 950^{o}C indicating poor crystallinity of the film as compared to that at 650 ^{o}C. This may probably due to difference in carbon contents in the surface region of the grown film incorporated from the precursor into the substrate during deposition.

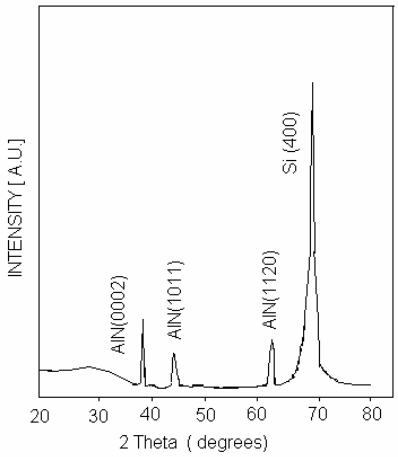


Fig. 3. XRD pattern of AlN thin film deposited on Si(100) at 950 ^{O}C.

From the XRD results, it is confirmed that by using dichlorophenyl amido aluminium (DCPAA) as single source precursor a thin film of aluminium nitride with hexagonal structure can be prepared at low temperature as much as 650 ^{o}C Fig. 4 shows the FTIR spectrum of a hexagonal AlN thin film grown on Si(100) at 650^{o}C using DCPAA precursor. A characteristic peak for h-AlN associated with its vibrational mode is clearly seen at 680 cm^{-1}.

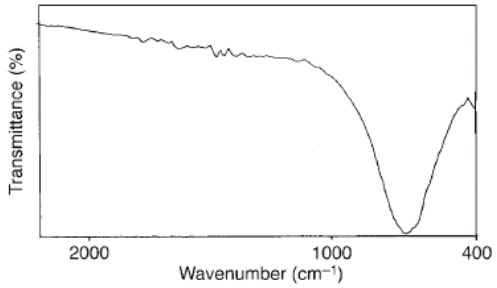
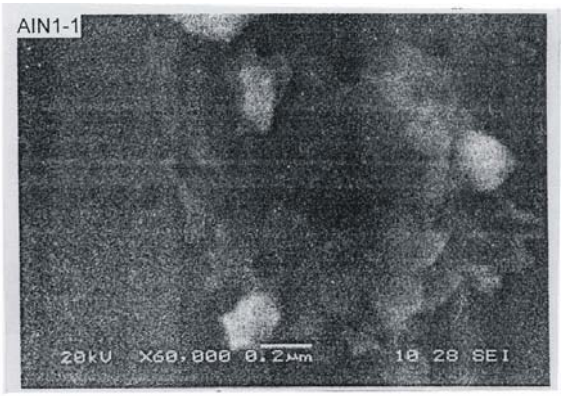
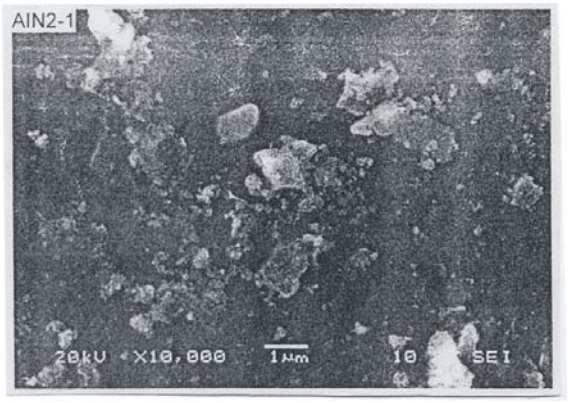


Fig. 4. FTIR spectrum of a hexagonal AlN thin film grown on Si(100) at 650 ^{0}C using DCPAA precursor.

Fig. 5 shows the scanning electron micrographs of AlN thin films deposited on Si(100) using dichlorophenyl amido aluminium presursor. Fig. 5(a) shows morphology of AlN thin film deposited at 650 ^{o}C. It shows fine crystalline and uniform nature of the film formed on the surface.



a



b

Fig. 5. SEM of AlN thin films deposited at (a) 650 ^{0}C, (b) 950 ^{0}C.

Fig. 5(b) shows morphology of AlN thin film deposited at 950 ^{o}C. This micrograph shows poor crystalline and non-uniform nature of the film. Thus on increasing the deposition temperature, there is decrease in crystalline character of the AlN thin film deposited by using dichlorophenyl amido aluminium precursor.

4. Conclusions

Polycrystalline, crack-free h-AlN thin films were successfully deposited on Si(100) by suspension coating pyrolytic method using dichlorophenyl amido aluminium as a single source organometallic precursor. The crystallinity of the films was monitored by using substrate temperature. The AlN thin films deposited on Si (100) at 650 ^{o}C substrate temperature exhibited polycrystalline hexagonal structure but the crystallinity decreased on increasing the temperature to 950 ^{o}C. XRD and SEM studies evidenced better crystallinity of the film deposited at 650 ^{o}C if compared to the crystallinity at 950 ^{o}C.

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