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Growth and evolution of residual stress of AlN films on silicon (100) wafer

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ABSTRACT

Aluminium nitride (AlN) thin films are being extensively used for diverse applications. However, its use significantly depends on the minimization residual stress generated during the deposition process. This paper reports the evolution of residual stress in sputter deposited AlN thin films on Si (100) substrates with varying thickness. The deposited films were found to be polycrystalline wurtzite structure with orientations along (100) for low value of thickness (300 nm and 430 nm) and (002) preferred oriented for thicker films (630 nm and 830 nm). Residual stresses in the AlN films were estimated by x-ray diffraction, infra-red absorption and wafer curvature techniques. Determined Residual stresses were found to be matching well with each other and were compressive in nature. Residual stresses in the AlN thin films were found to decrease (from −2.1 GPa to −0.6 GPa) with the increase in film thicknesses.
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1. Introduction

In recent years, aluminium nitride (AlN) thin films are intensively studied as a potential material for wide variety of applications. Its high thermal conductivity (180 $W m^{-1} K^{-1}$) and superior insulating properties ($\rho = 10^9$–10^{11} Ω-m) is employed in the fabrication of III–V based electronic structures [1–3]. Moreover, wide band gap (direct; 6.2 eV) [1,4] of AlN enable it as a promising material for integrated optics in the ultraviolet (UV) region. Because of its wide band gap and ability to form alloy with other group III elements, it is also being used to fabricate high electron mobility transistors (HEMT) and high power transistors [5–7]. In addition, the high acoustic velocity up to 6000–8000 m/s makes AlN an appropriate choice for bulk and surface acoustic wave applications [8,9]. Researchers also exploited its piezoelectric properties (arises due to the lack of a centre of symmetry in the wurtzite crystal structure) in the field of micro-electro-mechanical system (MEMS) based sensors and actuators [10,11].

Thin films of AlN have been deposited on different substrates like SiC [12], sapphire [13] and silicon [14]. Out of them, silicon offers more advantage in terms of large area (upto 200 mm diameter), high crystalline quality, low cost, scope for integration of optical devices with established CMOS technology etc.

Many researchers have been involved in developing different deposition methodologies, like sputtering, pulsed laser deposition (PLD), molecular beam epitaxy (MBE), metalorganic chemical vapor deposition (MOCVD) etc., to get high quality AlN thin films on Si (111) substrates due to its hexagonal arrangement of surface atoms [15–19]. It is well known that AlN films can be deposited in zinc-blend as well as wurtzite structures. However, the wurtzite AlN films are found to have superior piezoelectric properties compared to the AlN zinc-blend structure [10,11]. Moreover, since the piezoelectric coefficient of wurtzite AlN film is found to be maximum in *c* direction. For MEMS applications, Si (100) wafers are predominantly used for the ease of machinability using simple aqueous potassium hydroxide (KOH) or tetra-methyl ammonium hydroxide (TMAH). Therefore, growth of (001) oriented wurtzite AlN films on Si (100) substrates is very important for superior piezoelectric MEMS structures. However, the quality of AlN films as well as its piezo properties can further be enhanced by growing epitaxial (001) wurtzite film. But growth of the epitaxial AlN films on Si (100) surface by sputtering technique is not favourable due to the difference in lattice arrangement between Si (100) substrate surface (having square lattice arrangement) and (001) wurtzite AlN films (having hexagonal lattice arrangement). Thus most of the AlN films deposited on Si (100) substrates are found to be polycrystalline, but their piezo properties can be enhanced by growing the films in (001) direction.

The performances of the MEMS devices like accelerometers, gyroscopes, resonators, RF MEMS switches etc., are greatly

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influenced by presence of residual stresses. Therefore, estimation of residual stress is very important to develop high quality vibrating MEMS structures.

There are very few papers on estimation of residual stress due to the AlN thin film deposition on Si (100) [20–22]. In 1987, Este et al. [23] had tried to control the stress in AlN growth by reactive magnetron sputtering. They have calculated stress by determining the curvature of the films. Sah et al. [21] presented the effect of deposition temperature on the residual stress AlN thin films on Si and GaAs substrates. Variation of residual stress in AlN thin films with the nitrogen gas pressure has studied by Liu et al. [20]. Felmetsger et al. [24] estimated residual stresses in reactively sputtered AlN thin films as a function of Ar gas pressure. Lughi et al. [25] presented the defect and stress characterization of AlN films (on silicon substrate) by Raman spectroscopy. By studying the optical phonon lifetimes using infra-red spectroscopy (FTIR), Pobedinskas et al. [26] estimated the residual stress of sputtered deposited AlN thin films on Si.

Residual stresses can also be discussed in terms of length over which the stresses equilibrate. If the residual stresses vary continuously over large dimensions of the scale of the structure it is known as macroscopic (long range) stresses. On the other hand, if the residual stresses are confined within few atomic dimensions and balance within a grain then we called it microscopic stress. In these cases, the misfitting regions span microscopic or sub-microscopic dimensions [27–29].

The residual stresses can be estimated by different techniques such as x-ray diffraction (XRD), Raman spectroscopy, Fourier transform infra-red spectroscopy (FTIR) and wafer curvature techniques etc [29–33]. Since the sampling volume during the stress measurement using modified $Sin^2\psi$ method (XRD) and FTIR are quite large compared to the polycrystalline grain dimension, these techniques along with the wafer curvature technique are used mainly to determine the macroscopic residual stress in the sample.

In this paper, the growth of AlN thin films of different thickness on Si (100) substrate by dc magnetron reactive sputtering is reported. Phase, degree of crystallinity, orientation parameter and crystallite sizes are estimated using x-ray diffraction (XRD) technique. The macroscopic residual stresses of the deposited AlN films are studied by XRD, FTIR and wafer curvature technique. The residual stress values are compared with the thickness of the AlN films.

2. Experimental

AlN thin films were deposited on 50 mm diameter Si (100) wafers by DC reactive ion sputtering technique with Al (99.5%) target in Ar (10 sccm) and N_2 (10 sccm) gas mixture. Prior to the deposition process, the silicon substrates were cleaned by standard Radio Corporation of America (RCA) cleaning procedure (first developed by RCA Laboratory in 1970). In this process, the wafers were cleaned in two solutions - first in NH_4OH, H_2O_2, and deionised water (DI) mixture and then in HCl, H_2O_2, and DI water mixture. This helps in reducing the contaminations present on the wafer surface. The native oxide generated during the RCA cleaning was then removed in diluted (1%) hydrofluoric acid. Thereafter, the samples were thoroughly cleaned in DI water, dried, and subsequently loaded in sputtering chamber for the deposition. Prior to the depositions, Al target was pre-sputtered in an argon atmosphere in order to remove contaminated surface oxides on the target. For the deposition, substrate temperature is optimized at 550 °C and distance with target to substrate is kept at 8 cm. Rotating stage was used for making uniform film thickness and its rotation speed was round 5 rotations per minute. DC power were fixed around 100 W. Four samples were grown with duration of deposition varying from 25 min to 55 min with 10 min interval.

Thicknesses of the deposited AlN thin films were estimated by using Multi Mode Stylus Surface Profiler (Veeco DekTak 150) at the step sites of the samples. The phase and crystalline structure of the AlN films were analysed using grazing incidence x-ray diffraction (GIXRD) with x-ray incidence angle of 2° (from the sample surface) and detector was scanned (2θ angle) from 25° to 75° using Cu Kα radiation (model: PANalytical XPert Pro MRD HRXRD system). Microscopic investigation was carried out using Field Emission Scanning Electron Microscope (FESEM) (model: Zeiss Supra 55). Residual stress of the thin films was estimated by x-ray diffraction (XRD) and Fourier-transform infra-red spectroscopy (FTIR) (model: Varian 680-IR). The macroscopic/average in-plane residual stress in the thin films is determined by ex-situ wafer-curvature measurements using TOHO FLX-2320 S laser reflectance system.

3. Results and discussions

Thickness of the AlN thin films were measured using the stylus profiler. Four samples with measured thicknesses as 300 nm, 430 nm, 630 nm and 830 nm will henceforth be labelled as S1, S2, S3 and S4 respectively for further discussions. Fig. 1 shows thickness of the AlN films with deposition time. Average growth rate of the AlN thin film was found to be ~17 nm/ minute.

The AlN layers were grown by DC sputtering, in which we found films were polycrystalline wurtzite in nature. The lattice arrangement of Si (100) surface are known to be square and AlN along (002) surface have hexagonal lattice, therefore large mismatch is expected. Thus, hetero-epitaxy seems to be unfavourable in the Si (100) substrates due to the large in-plane lattice mismatch.

The grazing incidence X-ray diffraction (GIXRD) measurements results of the AlN thin films are presented in Fig. 2. All the films are showing polycrystalline wurtzite structure of AlN having grains oriented along (100), (002), (101), (102), (110), (103) and (112) directions. The samples S1 and S2 showed relatively intense (100) peak compared to the (002) peak and intensities of the other peaks are very small. This shows that the growth begins in the (100) orientation and that the growth is competitive between the (100) and (002) orientations. As the thickness increases the growth orientation along (002) is more favoured as compared to (100) direction and is evident from the enhanced intensity of (002) peak as compared to other planes. The intensity of the (103) orientation is also found to be growing in tandem with the (002) peak intensity. The reason for this increase can be understood in

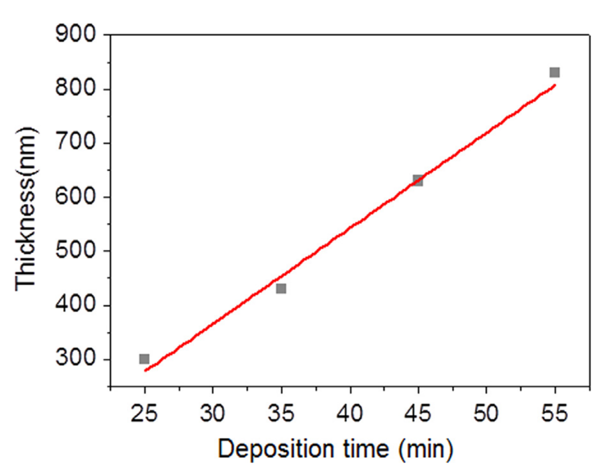

Fig. 1. Thickness of the AlN films with growth time.

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