Growth and surface characterization of piezoelectric AlN thin films on silicon (100) and (110) substrates

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ABSTRACT: This work investigates the fundamental growth of c-axis oriented piezoelectric AlN thin films by RF reactive sputtering on p-type (100) and (110) silicon substrates. Substrates are treated with a 1% HF solution before deposition to remove the native oxide followed by back-sputtering using argon ions. X-ray diffraction shows a (0001) oriented columnar texture of AlN grains which is the preferred orientation for piezoelectric applications. TEM shows the presence of a 4 nm thick semi-crystalline interface between silicon and the AlN layer. A basic growth mechanism is proposed from microstructural observations.

1. INTRODUCTION

RF reactive sputtering offers advantages over other processes for depositing AlN thin films on various substrates, and facilitates micromachined device fabrication processes. Okano et al. (1992) suggested that using this technique highly (0001) oriented piezoelectric AlN thin films can be grown at relatively low temperature. Piezoelectric AlN MEMS devices find applications such as microactuators, resonators, acoustic modulators, etc. The surface morphology of AlN thin films with many orientations exhibited a granular, worm-like, and a columnar surface of grains, as detailed by Cheng et al. (1996). In this paper, we have studied the microstructure of AlN thin films with (0001) orientation deposited on Si (100) and Si (110) substrates by RF reactive sputtering using X-ray diffractometry (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Growth observations are presented showing the existence of a semi-crystalline interlayer. From a detailed analysis near the AlN/Si interface region of the sample along its thickness direction from the interlayer the texture, morphology and microstructure of AlN thin films is elucidated.

2. EXPERIMENTAL PROCEDURE

The deposition of AlN thin films was performed at a substrate temperature of 355^{O} C in a Nordiko-2000 RF reactive sputtering system. Prior to that, the p-type silicon substrates were treated using a 1% hydrogen fluoride (HF) solution to remove the native oxide and subsequently kept in vacuum. Back sputtering using pure argon plasma was done to remove any monolayer formed by the residual gases inside the vacuum chamber. The deposition was carried out using optimised sputter parameters as shown in table 1. For a better piezoelectric performance, AlN with a (0001) orientation grown perpendicular to the substrate is required as indicated by Naik et al. (1996). The substrate temperature was kept at 355^{O} C before the deposition started.

Crystalline orientation and texture were determined by XRD (Philips Expert II) with Cu kα radiation generated from a 40 kV, 30 mA X-ray source. A rocking curve scan was also performed at 2θ where the (0001) reflection occurs. The surface morphology and cross-section of the samples were observed by SEM (LEO 1550 FEG). Samples were prepared for cross-sectional TEM (XTEM) studies in a Philips CM30T following the recipe by Keim et al. (2001/2) to study the growth mechanism.

Table 1. AlN deposition parameters

Parameters	Values
Sputter pressure (mbar)	7.2×10^{-3}
RF power (W)	350
Ar:N_2 flow rate (sccm)	7:6
Substrate temperature (deg. C)	355
Back Sputtering	
RF power (W)	150
Ar pressure (mbar)	1.2×10^{-2}
Duration (min)	8

3. RESULTS AND DISCUSSION

3.1 Preferential (0001) Texture

The thickness of the layers was found to be 580 nm as measured by ellipsometry. The deposited films contain mainly the wurtzitic phase of AlN and no other reflections are observed from XRD as shown in Fig. 1 (a). The 2θ - reflection occurs at 35.87^{O} and 35.91^{O} for AlN thin films on Si (100) and Si (110), respectively. The rocking curves measured at 2θ - reflections are shown in Fig. 1 (b) with Gaussian fitted curves, of 3.3^{O} and 2.9^{O} FWHM for films on Si (100) and Si (110), respectively. It shows the predominant (0001) texture of AlN with the c-axis normal to the silicon substrates.

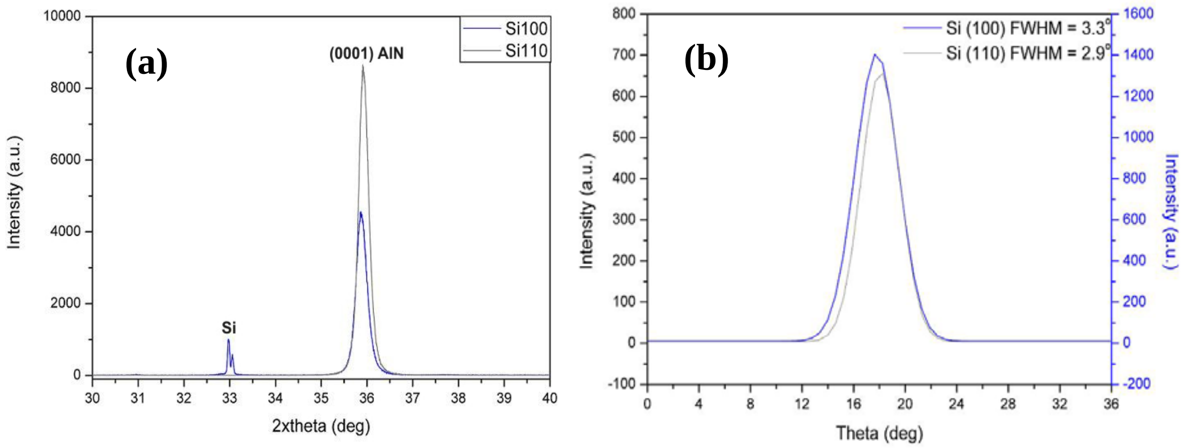

Fig. 1: XRD intensity peaks for (a) 2θ scan and (b) rocking curves (Gaussian fitted) of AlN thin films with (0001) texture deposited on the Si (100) and Si (110) substrate, respectively.

3.2 Surface Morphology

Samples were cleaved into many pieces. Image (a) and (b) of Fig. 2 show the surface morphology of AlN thin films on Si (100) and Si (110) substrates, respectively, with a substrate tilt angle of 5^{O}.

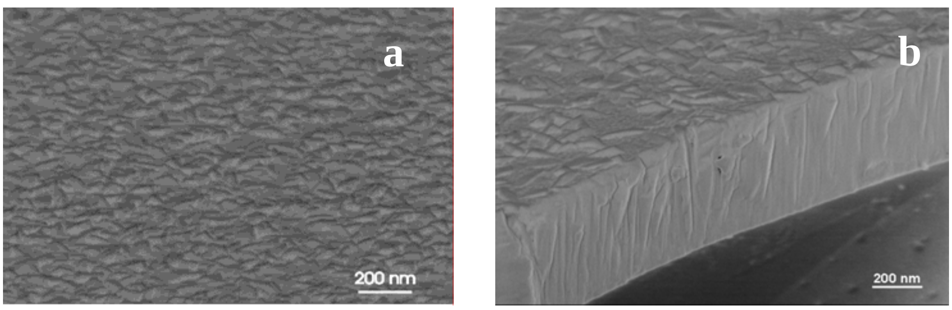

Fig. 2: Surface morphology of AlN thin films on (a) Si (100) and (b) Si (110) substrates using SEM.

The top surface shows an average crystallite size of AlN thin films that varies from 20-50 nm. The crystallites are closely packed together without any trapped pin-holes on the surface.

3.3 Interface properties

TEM images were obtained at the interface between the AlN thin film and silicon substrate to understand the initial stages of growth and subsequent evolution of microstructures. The XTEM lattice images are shown in (a) and (b) of Fig. 3 for the Si (100) and Si (110) substrate, respectively. A clear thin interface with a thickness of approx. 4 nm was observed on both substrates. It can also be observed that (0001) columnar AlN crystallites originate from the interface layer. The interface layers are highly defective and disordered because of a larger mismatch in lattice parameters between silicon substrate and AlN.

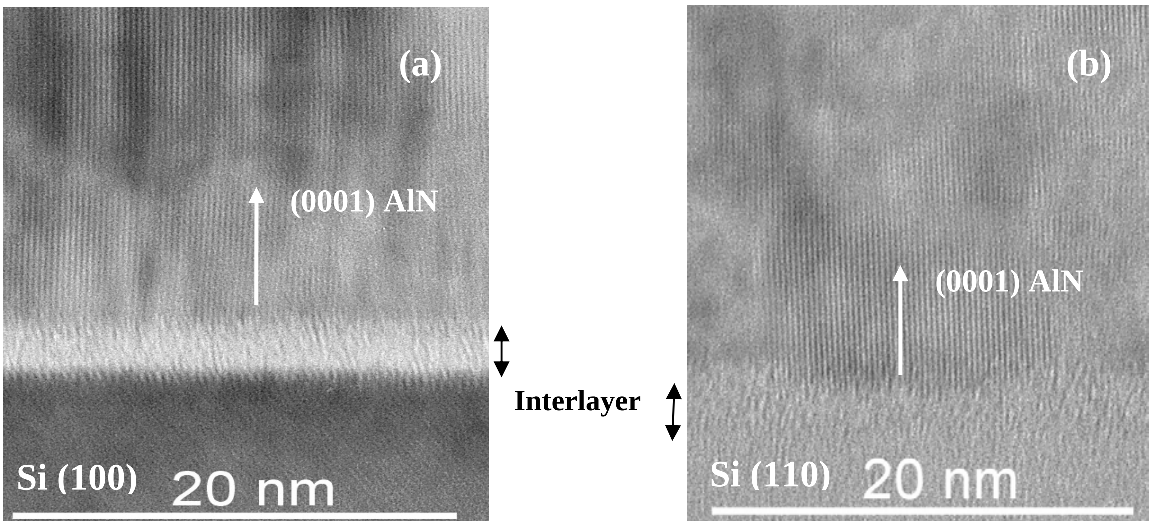

Fig. 3: TEM lattice images at the AlN/Si interface region. The thickness of the interface layer is 4 nm.

To understand the microstructure properties of the interface layers, Dark Field (DF) images were recorded. These are shown in Fig. 4 ((a) and (b)). It shows the presence of various sizes of bright spots within the interface layer indicating that the interface layer is not completely amorphous but semi-crystalline in nature.

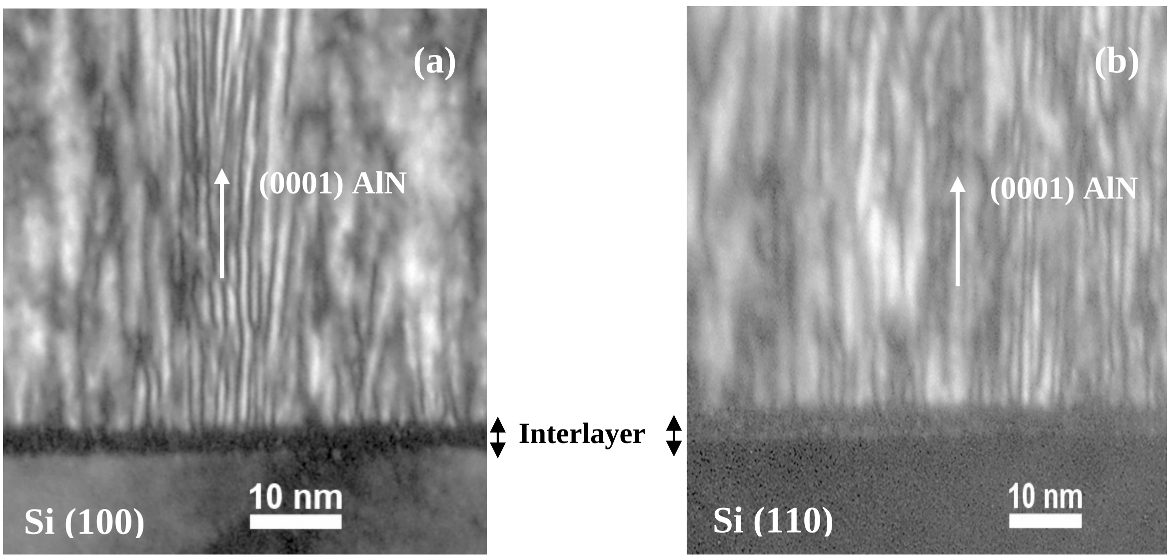

Fig. 4: Dark field TEM images at the AlN/Si interface region.

3.4 Discussion

Investigations from bright and dark field images reveal that a few atomic layers of AlN grains at the interface layer are semi-crystalline on both silicon substrates. This semi-crystalline nature can be explained from the following facts: First, the mismatch in lattice parameters causes AlN grains to nucleate in an amorphous phase initially, and they are highly defective. Second, the mobility of ad-atoms at the inter-layer is not enough to find the minimum surface energy plane due to a relatively low substrate temperature, therefore no epitaxial growth of AlN thin films occurs. Based on a microstructure analysis of the AlN thin films, a growth model is proposed as shown in Fig. 5. It consists of a transition layer which is a semi-crystalline AlN thin film of thickness less than 4 nm, followed by a direct columnar AlN layer which is running through the entire thickness of the film. They are strained and have dislocation defects. The semi-crystalline inter-layer thus forms nucleation sites for preferential columnar orientation of AlN which has a minimum surface energy related to other faces as mentioned by Stevens et al (1994). These inter-layers are not hydrogenated AlN (AlN:H) because the substrates were back sputtered with argon before deposition. Also, we do not expect silicon nitride formation at the interface since the deposition was done at relatively low temperatures, as mentioned earlier by Bing-Hwai et al. (2002).

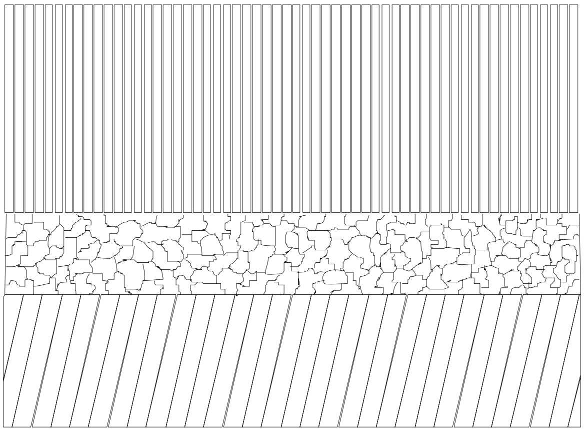

Fig. 5: Schematic representation of the growth mechanism of AlN thin film on Si substrates

4. CONCLUSIONS

Preferential (0001) oriented piezoelectric AlN thin films deposited on silicon (100) and (110) substrates have been studied for their orientation, surface morphology and interface properties. SEM and XTEM observations show dense, columnar crystallites of AlN. Microstructural investigations show two distinct layers: (1) a transition and (2) a columnar AlN layer. The inter-layer (1) between the AlN film and silicon substrate is semi-crystalline in nature and it is believed to be the origin of nucleation sites for columnar growth of AlN grains. The lowest surface energy is along the (0001) direction and it is consistent with all deposited films regardless of the nature of (100) or (110) silicon substrate, which is an essential prerequisite for piezoelectric thin film applications.

REFERENCES

Cheng C C, Chen Y C, Wang H J and Chen W R, 1996 J. Vac. Sci. Technol. A **14** p 2238.
Hwang B H, Chen C S, Lu H Y and Hsu T C, 2002 Mat. Sci. Eng. A **325** p 380.
Keim E G, Bijker M D and Lodder J C, 2001 J. Vac. Sci. Technol. A **19** (4) p 1191.
Keim E G, Nguyen L T, Lodder J C, 2002 Proc. Microscopy & Microanalysis Meeting, (Quebec, Canada) p 1346CD.
Naik R S, Reif R, Lutsky J J and Sodini C G, 1996 J. Electrochem. Soc. **143** (20) p 691.
Okano H, Takahashi T, Tanaka T, Shibata K and Nakano S, 1992 Jap. J. Appl. Phys. **31** p 3446.
Stevens K S, Ohtani A, Kinniburgh M and Beresford R, 1994 Appl. Phys. Lett. **65** p 321.