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Low Temperature Growth of Sputtered AlN Films for Layered Structure SAW Devices

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ABSTRACT

AlN films with c-axis oriented perpendicular to the surface were deposited on silicon substrates by reactive RF magnetron sputtering method, at various temperatures (without heating $-400^\circ$C). The structural, morphological and optical properties of AlN films were investigated by X-ray diffraction, scanning electron microscope, atomic force microscopy and Fourier transform infrared absorbance spectroscopy. It was found that the AlN films showed the same highly (002) preferred orientation with low full width of half maximum of rocking curve, which is about $2^\circ$ for all the deposited films. The surface roughness of AlN films determined by AFM is less than 1nm for the film grown at low temperature. This result is very important and means that films with good crystalline quality, low surface roughness can be processed at low temperature. Elastic properties of deposited AlN films were evaluated by realization and characterization of AlN/silicon SAW device.

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I. INTRODUCTION

Aluminum nitride (AlN) has been considered as an attractive thin film piezoelectric material for integrated circuit (IC) compatible surface acoustic wave (SAW) devices. This compatibility requires a deposition process in relatively low temperature. Many techniques, such as sputtering [1–4], chemical vapor deposition [1, 5], laser chemical vapor deposition [1, 6] and molecular beam
epitaxy \cite{1, 7} have been used to fabricate AlN thin films on various substrates. Using silicon as the substrate is highly attractive for device applications due to potential integration with well-developed silicon technologies. In most cases mentioned previously, the deposition temperatures are quite high. High temperature deposition has the drawback of degradation of the substrate and AlN film during growth due to thermal damage. Then, elaboration of AlN films at low temperatures has become very important and valuable \cite{8, 9}. Sputtering technique was adopted here for AlN films deposition.

The choice of AlN as a piezoelectric thin film for SAW applications is due to its high acoustic velocity to allow the achievement of high frequency. In fact, the increase of the operation frequencies of SAW devices can be accomplished using high resolution lithography techniques (e-beam), or high acoustic wave velocity materials. The first solution is highly demanding in terms of fabrication costs, the second one is easier if high quality fast materials can be grown by standard thin films deposition techniques such as sputtering \cite{8}. Furthermore, increasing operating frequency of SAW devices is very important for sensors applications. In fact, the increase of frequency induces the increase of sensor sensitivity \cite{10}. In this work, the high crystalline quality, high acoustic velocity and high piezoelectric coupling AlN thin films deposited at low temperature, is presented, to permit the achievement of high frequency SAW devices based on non-piezoelectric and no high acoustic velocity substrates, such as silicon.

In our previous work \cite{11–13}, we already studied the growth of AlN thin films on silicon substrates using RF and DC reactive magnetron sputtering method, which are interesting of their microstructural, electrical and physico-chemical properties. In this study, we present our latest results concerning AlN thin films deposited at low temperature on Si(100) substrates, after the optimization of growth process. We pointed out the correlation between surface roughness of AlN films and the frequency response of SAW devices based on these films. We have investigated the crystalline structure by X-ray diffraction, the optical properties of AlN films by Fourier transform infrared absorbance spectroscopy (FTIR), and the microstructure by field emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM). SAW devices are realized based on low temperature deposition AlN thin films and we have pointed out the high crystalline quality and piezoelectric properties of these films.

II. EXPERIMENTAL

AlN thin films were deposited by a RF planar magnetron sputtering system on silicon Si(100) substrates. The Aluminum target (purity 99.99\%) diameter was 107 mm and 6.35 mm thick. The distance between the cathode and the substrate holder was 80 mm. The deposition chamber was pumped down to a base pressure of $1 \cdot 10^{-7}$ mbar by a turbomolecular pump prior to the introduction of the
argon-nitrogen gas mixture for AlN thin film production. The gas discharge mixture was Ar/N₂ and the total pressure was kept constant at 5.10⁻³ mbar. The nitrogen percentage in the Ar/N₂ gas mixture was 60%. The RF power delivered by the RF generator was 170 W. The substrate and the chamber wall was grounded. The substrate holder was maintained without heating and after heated to obtain different deposition temperatures until 400°C. In order to perform a better comparison between the different samples, the deposition time of AlN films was adjusted to obtain 1.4 μm thick films for various deposition temperatures. The thickness of deposited films was measured by field emission scanning electron microscopy (FESEM) from the cross section of structure.

X-ray diffraction using Cu-kα cathode has been employed to determine the crystalline properties of the AlN thin films. The diffracted intensities were collected in θ-2θ scan and ω rocking curve scan modes. The optical properties of AlN films were characterized by Fourier transform infrared spectroscopy. The FTIR spectra were measured over the 400–4000 cm⁻¹ range with a spectral resolution of 4 cm⁻¹. The microstructure were studied by FESEM and TEM in order to determine the dependence of grain size with the film thickness, and the evolution of AlN thin films microstructure in various phases of growth. Eventually, atomic force microscopy measurements, operating in contact mode, were taken to determine the evolution of surface roughness and the surface morphology of AlN films with temperature deposition.

III. RESULTS AND DISCUSSIONS

The preferred orientation of AlN thin films elaborated in different deposition temperature was investigated by XRD. Figure 1 shows the XRD patterns of these films deposited on Si(100) substrates. All the peaks were indexed on the basis of the hexagonal wurtzite type structure. We can observe that the preferred orientation of all the films is the (002) orientation corresponding to the c-axis perpendicular to the surface. The XRD peaks intensities are the same for all the films, which indicate that the film synthesized without heating presents the same degree of (002) preferred orientation. However, from the ω rocking curve scan mode, we have found that the FWHM of rocking curve of all the films is varies from 1.56° to 2.8°, with the minimum value obtained for the film deposited without heating (WH) (1.56°), as shows Fig. 2. This result attests that the AlN (WH) thin film presents the best (002) preferred crystalline orientation with the lower rocking curve FWHM. This evolution of crystalline orientation quality can be explained by the possibility of formation of defects in the films at high temperature by promoting the diffusion of impurities into the films introducing structural disorder [14].

Optical characterization of AlN thin films was carried out to evaluate the phonon modes in the films and a presence of oxygen contamination. As is know, Fourier transform infrared (FTIR) spectroscopy is valuable tool to study
Figure 1. XRD pattern of AlN thin films deposited at various temperatures. (See Color Plate IX)

Figure 2. Rocking curve of AlN film elaborated without heating. (See Color Plate X)
Figure 3. FTIR spectra of AlN films. (See Color Plate XI)

materials structural characteristic and physical properties as the characteristic phonon frequency. Figure 3 shows FTIR reflectance spectra taken from AlN samples. The spectral range shown corresponds to transverse and longitudinal optical phonon energy range. A strong FTIR reflectance peak around 678 cm\(^{-1}\) was observed in these AlN films, and a very little peak was observed around 613 cm\(^{-1}\). The two peaks were attributed to E\(_2\)(TO) and A\(_1\)(TO) phonon modes in the films. The E\(_2\)(TO) mode indicates that the AlN film is oriented c-axis perpendicular to the surface, and the little peak of A\(_1\)(TO) mode means the presence of a very weak (100) crystalline orientation that we doesn’t observed by XRD analysis. This result corroborates the high preferred (002) crystalline orientation of all AlN films. Furthermore, the FTIR analysis allows deducing that the residual stress in our AlN film is constant for all deposition temperature and it is estimated from the FTIR peak position to \(-1.5 \text{ GPa}\).

FESEM and TEM characterizations were made to analyze AlN film microstructure. As show Fig. 4, we can see, from FESEM image, the grain and boundaries of AlN film grown without heating. The average grain size from this image was estimated about 30 nm. TEM analysis was done to investigate the cross section of this film. Figure 5-a shows the image of columnar structure of AlN film (WH), and exhibits dense and crack free film. To analyze the microstructure of the first growth phases of AlN film (WH), we have realized a film with very low thickness about 100 nm. As show figures 5-b, TEM image presents the plane view of this film and we can distinguish the grain and boundaries and the grain size of this film was evaluated about 20 nm. From all these
results, we can conclude that the grain size of AlN film increases with thickness. The formation of columnar structure of our AlN films was established since the first growth phases.

AFM measurements enable us to characterize the film morphology evolution with growth experimental conditions. We can using this technique measure
Figure 6. Three-dimensional (1 µm * 1 µm) AFM images of various AlN thin films. (See Color Plate XII)

The surface roughness of AlN thin films. In Fig. 6, we show the surface morphology of AlN film elaborated in various deposition temperatures. As show FESEM observations, a granular morphology is observed. All images are exhibited in the quasi-same vertical scale to have a clearly observation. From these images, the rms roughness of AlN film synthesized without heating presents the lower roughness measured at 4.5 Å than the other films. This evolution can be explained by the fact that the high temperature deposition favorite the swelling of grain columns and consequently the increasing of surface roughness.

The SAW transducers were fabricated on the AlN film surface by forming an array of aluminum interdigitated electrodes (IDTs). The thickness of the aluminum layer was 150 nm. It was deposited, patterned and etched using UV lithography technique and wet etching. The individual finger widths were 8 µm and spacings were 4 µm to facilitate harmonic generation [15, 16]. The IDTs were developed on AlN/Si layered structure based on AlN films elaborated without heating and at 400 °C The wavelength of generated SAW is then 24 µm. Figures 7 and 8 show the frequency responses of the both AlN/Si structure SAW devices realized using these AlN films. The represented responses exhibit the fundamental harmonic of AlN/Si SAW devices which have a resonance frequency around 211 MHz. This frequency corresponds, taking into account the wavelength value of 24 µm, to SAW velocity of 5064 m/s. This result involves the good crystalline quality of the both AlN thin films elaborated at
Figure 7. Frequency response of AlN/Si layered structure SAW devices: AlN film deposited at 400°C.

Figure 8. Frequency response of AlN/Si layered structure SAW devices: AlN without heating deposition.
without heating and at 400°C. Concerning the characteristics of the frequency responses in term on insertion loss and rejection, we can observe that the SAW device based on AlN(WH) presents a better response characteristics than the SAW device based on AlN film deposited at 400°C. The rejection and insertion loss of the AlN (WH) are 14 dB and $-33.35$ dB respectively, while those of AlN (400°C) are 10 dB and $-34$ dB. These results are in concordance with the results obtained using AFM measurements presented above. In fact, the structure realized with AlN film (WH) exhibits lower roughness which enable to reduce the propagation losses and consequently the improvement of the rejection and the insertion loss of frequency response. This correlation between morphological properties of AlN thin film and the frequency response characteristics is very interesting to predict the SAW devices performances.

To complete this study, we have measured the electromechanical coupling coefficient ($K^2$) of the two structures AlN/Si based on the films elaborated at 400°C and without heating. The results obtained show a better $K^2$ value for SAW structure based on AlN (WH) measured of 0.54%, while the coefficient of the structure base on film synthesized at 400°C was measured of 0.34%. These results corroborate the structural analysis, which were showed that the AlN film deposited without heating presents a better (002) preferred orientation than the others films. The theoretical value of $K^2$ of this structure, taking into account the film thickness is 0.13%. This difference between the experimental and theoretical values remains difficult to explain. The measurement of TCF was also carried out and shows quasi-similar values for the two structures: $-26.1 \text{ ppm/}^\circ \text{C}$ and $-29 \text{ ppm/}^\circ \text{C}$ for the structures based on films elaborated at 400°C and without heating respectively.

**IV. CONCLUSION**

AlN films were grown by RF reactive magnetron sputtering on Si(100) substrates at different deposition temperatures. We have pointed out the effect of the deposition temperature on structural, morphological and acoustical properties of AlN films. We have showed that we can obtain a high oriented (002) AlN films with a low FWHM rocking curve for the films elaborated without heating. The low surface roughness AlN film can also obtained without heating and allow to have very low acoustic losses which involve the achievement of high performances SAW devices. These results offer the great possibility for integrated circuit (IC) compatible surface acoustic wave (SAW) devices. These physical characterizations were corroborated by forming AlN/Si SAW devices based on AlN film synthesized without heating, which exhibited practical frequency characteristics better than those obtained for the AlN film deposited at high temperature. These results enable to envisage the use of these devices as gas sensors by addition a sensitive layer between the SAW IDTs.
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