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Synthesis and Characterization of Reactive Sputtered AlN Thin Films

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Abstract: A series of AlN coatings have been elaborated on silicon substrate using dc magnetron sputtering method, by varying the nitrogen flow in the discharge. We have noticed that the coating deposition rate decreases with the nitrogen flow which is conducive to a transition from metallic aluminum coating to (100) and (002) preferentially oriented Aluminum Nitride (AlN). A shorter target-substrate distance gives rise to the (002) orientation, as revealed by the DRX and the SEM characterization.

Key words: Aluminum nitride, thin films, sputtering, preferential orientations

INTRODUCTION

Aluminum nitride, which has the structure of hexagonal wurtzite, is a III-V family compound. AlN thin films have attracted great interest because of their appealing properties such as high values of hardness and thermal conductivity or high resistance to temperature even in hostile environments. Therefore AlN films have been widely used as protective coatings[1-4]. Even though it is used in many other microelectronic applications such as passivation layers, dielectric films, insulating layers[5], solar cell coatings[6] and short wave-length emitters, aluminium nitride is known to be one of the most promising piezoelectric materials for high frequency surface acoustic waves (SAW) devices[7] and thin films acoustic bulk resonators (FBAR)[8] because of its high sound velocity. It is the optimal material for GHz-grade SAW devices. To be applied to SAW devices, the structure of AlN films is required to have a polycrystalline preferential orientation, a homogeneous composition and low surface roughness. Therefore, the needs to study the preferential orientations of AlN films are really significant. The purpose of this study was to investigate the influence of the preparation conditions on the crystallographic orientation by the reactive sputtering method and to explain the change in the orientation theoretically. In this study, piezoelectric AlN thin films have been deposited on Si (100) substrates using a self designed DC magetron sputtering system as shown in Fig. 1.

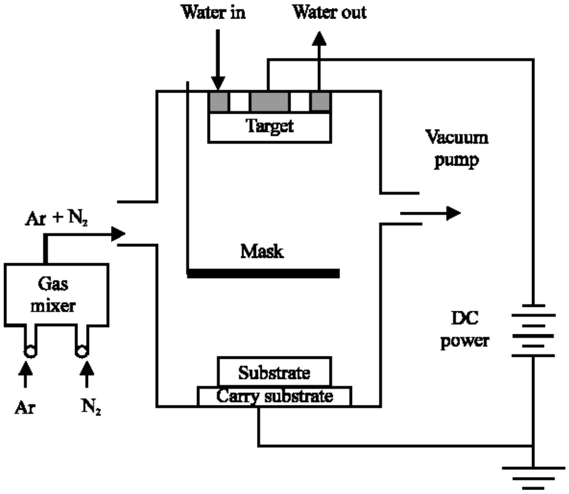

Fig. 1: The self designed DC magnetron sputtering system

MATERIALS AND METHODS

AlN thin films were deposited onto Si (100) substrate using the DC reactive magnetron sputtering in a gas mixture of Ar and N gas. The purity of all the 2 gases was 99.9999%. The aluminum target was 1.3 inch in diameter and its purity was 99.9%. Degreasing of substrates was carried out in the ultrasonic baths of acetone, ethanol and de-ionized water, successively. Native oxide on the Si wafer was removed by etching in diluted HF solution.

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The substrates were dried using N_2 gas and then immediately inserted into the vacuum chamber. The ultimate sputtering pressure of approximately 10^{-6} Torr was obtained using the vacuum pump. The target was pre-sputtered in a pure Argon 99.999% atmosphere for approximately 5 min, to clean and equilibrate the target surface prior to film deposition. Afterwards nitrogen 99.999% was admitted into the chamber, Argon was also continuously added to the nitrogen via a separate valve and we have to keep under control the ratio $N_2/Ar+N_2$ until the total pressure reached is 4.10^{-3} Torr. During the deposition process the total pressure and DC power were kept constant. AlN films were deposited at different nitrogen concentrations and also by varying the interelectrode distance The crystal structures and the preferential orientations obtained of AlN thin films were characterized by X-ray diffraction The crystallite sizes of AlN films and the film thickness were determined by scanning electron microscopy.

RESULTS AND DISCUSSION

All the deposited AlN films were found to have wurtzite structure.The discussion in the following sections focuses on the evolution of preferred orientation of AlN films produced under various deposition condition.

Effect of nitrogen concentration: We have investigated the dependence of crystal orientation of AlN films on nitrogen concentration. Fig. 2 shows the X ray diffraction patterns of the AlN films prepared at different nitrogen concentrations 20-50% with the other parameters fixed (DC power 120W, Pressure 4 mTorr and the interelectrode distance fixed at 3 cm). The films deposited with low N concentrations exhibit improved crystallographic quality. It was also found that high N concentrations are favorable for (0 0 2) preferred orientation while a low N concentration is favorable for (1 0 0) preferred orientation. This result agrees well with other reported work[9-12] and is believed to be resulted from the low deposition rate of AlN films under high N concentration Fig. 3. At low deposition rates, the adatoms have longer time to rearrange in low energy configurations, i.e., close-packed (0 0 2) atomic plane. It was the low sputtering yield of aluminum target by N bombardment (compared to that of Ar) that introduces the low deposition rate.

The SEM cross section image, at 30% N_2 concentration Fig. 4 shows a well developed columnar structure and a dense film with small grains.

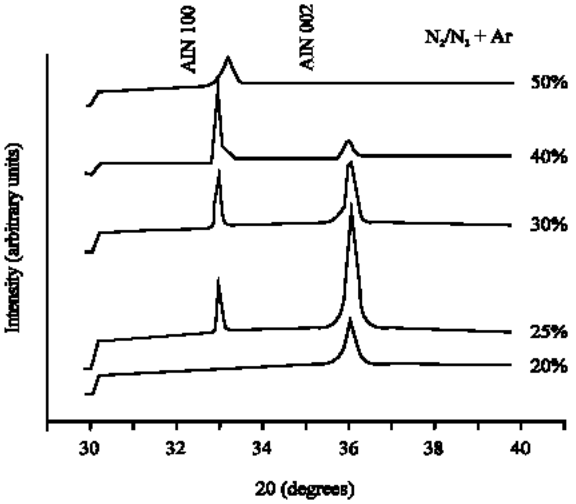

Fig.2: XRD patterns of AlN films deposited at various N_2 concentrations (Total pressure 4mT, DC power 120W and target subtrate distance 3cm)

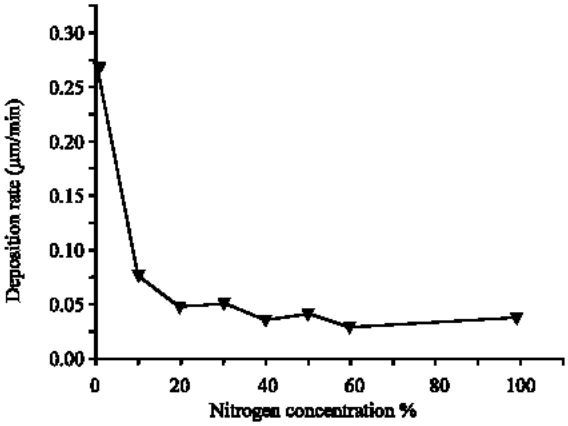

Fig. 3: Effect of N_2 concentration on the deposition rate

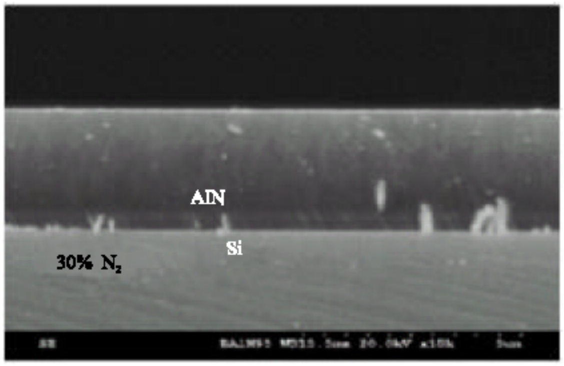

Fig. 4: SEM cross section image at 30% N_2 concentration

Effect of the Substrate(Si)-Target(Al) distance D: The influence of the distance D on the crystalline orientation of AlN films have been investigated. Fig. 5 shows the XRD patterns of AlN films deposited at different distances D, with the target power, sputtering pressure and N_2 concentration fixed.

when the distance D was approximately 2 cm, 100, 002 planes co-existed in the film. With increasing distances D, the film only had the 100 plane. If the distances D were too great, the films would not grow on the substrate. The deposition rate decreased rapidly from 0.1 to 0.03 μm min^{-1} with an increase in distance D from 2 to 4 cm.

This demonstrates that the impact of the distance D is also great, an increasing D is conducive to the 100 preferential orientation of the film[13-14]. In summary, a shorter distance D is conducive to the growth of AlN 002 films, whose c-axis is perpendicular to the substrate. On the contrary, a longer distance D is advantageous for the growth of AlN 100 films, whose c-axis is parallel with the substrate.

The relationship between the mean free path of the sputtering particles and the interelectrode distance D affects the orientation of the films. When λ >D, more sputtering particles reach the substrate directly without collision and the energy of the sputtering particles is greater, which is beneficial for the formation of bond B_2, and consequently, the growth rate of the 002 plane is faster. When λ < D, most of sputtering particles will make collisions once or many times before reaching the substrate, thus decreasing the energy of the particles, or forming small AlN clusters. In this case, the growth rate of the 100 plane is higher Fig. 6.

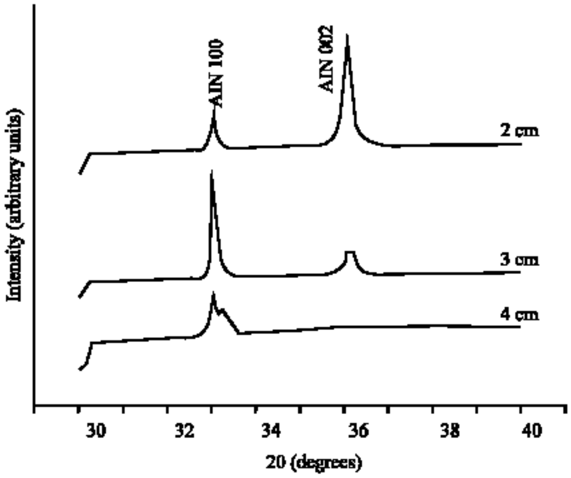

Fig. 5: XRD patterns of AlN films at different Substrate Target distances (Total pressure 4mT, DC power 120W and N_2 concentration 40%)

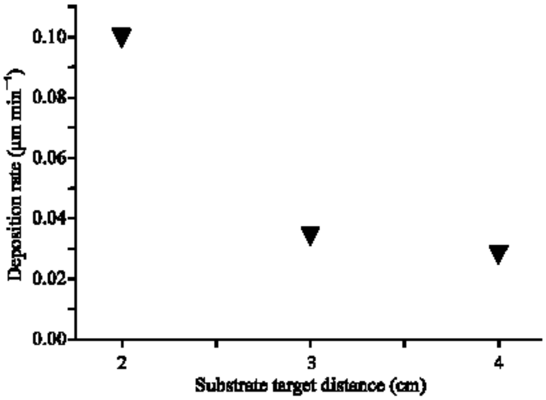

Fig. 6: Effect of Substrate Target distance on the deposition rate

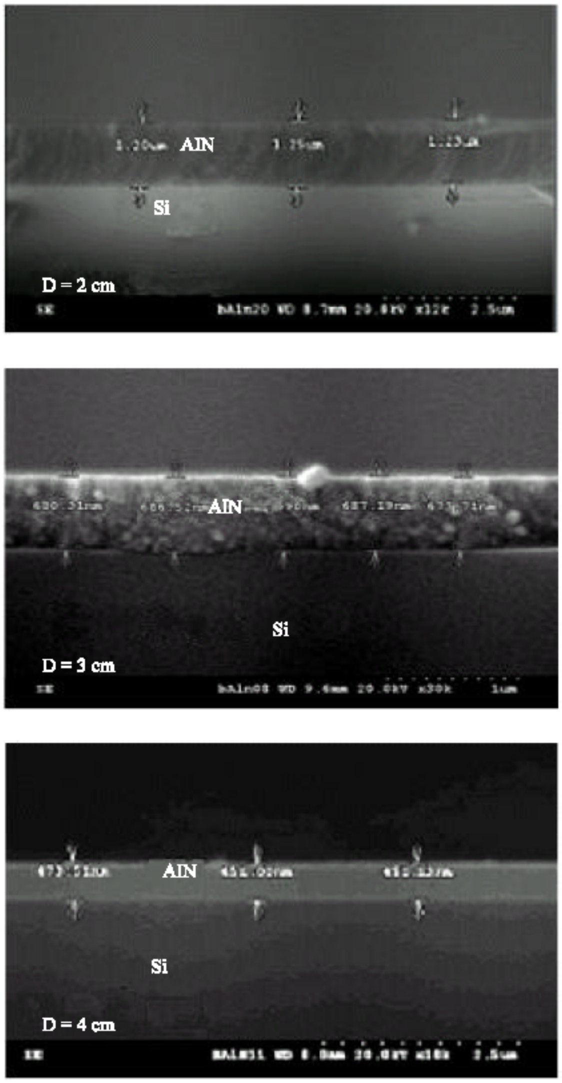

Fig. 7: SEM cross section images at different Substrate Target distances

The results obtained can be summarized in the following. As the interelectrode distance goes down, the preferred orientation of the film changes from (100) to (002) orientation. Through the microstructure study of the film using SEM cross section images, as shown in Fig. 7, it is proven that the columnar structure is well developed and a dense film can be deposited with the decrease of the distance, also the film thickness decreases with the interelectrode distance.

CONCLUSION

This paper reports on the preparation of AlN films with a DC reactive magnetron sputtering method. It was found that the nitrogen concentration and the distance D are very important factors for the crystalline preferential orientation of AlN films. When distance D is short, it is easy to form the 002 preferential orientation of AlN films. On the contrary, when the distance D is long, this is favorable for a film with a 100 preferential orientation.

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