How to reduce the Al-texture in AlN films during film preparation*

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The preparation of aluminum nitrogen (AlN) film without Al texture is of great significance for the manufacture of high-performance surface acoustic wave (SAW) device. We research the process factors which bring Al into AlN film due to radio frequency (RF) magnetron sputtering system, and discuss how the process parameters influence the AlN thin film containing Al. In the research, it is found that the high sputtering power, the low deposition pressures and low partial pressure of Ar can lead to growing Al-texture during AlN thin film preparation, and the experiment also shows that filling the chamber with nitrogen gas can recrystallize a small amount of Al composition into AlN film during the annealing process in the high temperature environment.
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Surface acoustic wave (SAW) and bulk acoustic wave (BAW) devices are widely applied in modern communication systems. Aluminum nitrogen (AlN) thin film perpendicular to the *c*-axis preferred orientation is the optimum piezoelectric material for the multilayer SAW devices[1,2].

Considering the thin film preparation method, radio frequency (RF) magnetron sputtering technique is widely adopted[3-6]. However, the AlN film deposited by RF magnetron sputtering often mixes with strong Al texture easily[7-10]. AlN is an important Ⅲ-Ⅴ wide band gap (6.2 eV) insulating compound with the crystal hexagonal wurtzite-type structure[11,12]. AlN thin film mixed with Al texture can reduce the resistivity of the film and conduct electricity worse. The low resistivity of thin film can not only lead to dielectric loss which results in a high insertion loss of the device, but also cut down the coupling coefficient of the piezoelectric material with the improved relaxation frequency[13]. Thus, preparation of AlN thin films without Al texture is vital for SAW devices[14]. However, only the researches on individual process factor for controling the elimination of aluminum but not systematical researches on all the process factors have been reported. In this paper, AlN thin film perpendicular to the *c*-axis is prepared on Si (100) substrate by RF magnetron sputtering system, and how the processing parameters bring Al into the film is studied. After that, through changing the sputtering power, total-pressure and partial pressure of Ar, Al texture disappears and preferential orientation (100) AlN film is deposited successfully.

The AlN film was deposited on Si (100) substrate by RF magnetron sputtering device, and the substrate was tackled with ultrasonic cleaning by hydrofluoric acid (HF) (5%), anhydrous alcohol and acetone before deposition. The sputtering target was pure Al (99.9995%) with the diameter of 60 mm and the thickness of 5 mm. After the sputtering chamber was evacuated below 5×10^{-4} Pa, and the substrate was heated to 300 ºC, high-purity nitrogen and argon (99.999%) gases were introduced into the chamber. AlN films were deposited at appropriate RF power for 120 min after pre-sputtering for 15 min.

In this study, the crystallinity and preferred orientation of the films were studied by Japan's Neo D-MAX2600 (18 kW) X-ray diffraction (XRD). AlN (100) diffraction peaks are at $2\theta = 33.2°$, and AlN (002) diffraction peaks are at $2\theta = 36.0°$.

The parameters in Tab.1 show the different sputtering power for preparation of the AlN/Al thin film. Fig.1 shows

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XRD pattern of the film by changing power with other parameters stable. Sputtering power is necessary for film deposition, and preferred orientation of thin film can be achieved only under appropriate power. Increasing power can improve the energy of bombarded target particle, sputtering productivity and also the adequacy of the reaction of Al and N_2. Over-large power can produce Al texture in the AlN film. With more Al atoms bombarded as the over-large power, Al atoms reacting with insufficient N_2 can deposit Al-rich film. Meanwhile excessive speed of physical reactions, which is also caused by the over-large power, can decrease the ability of atoms migration, which is harmful to film properties.

In our experiment, there is only Al film on the substrate when the sputtering power is 150 W. 100 W is the optimal sputtering power, and if it is less than 100 W, energy of Al atoms is so low that the atoms can not reach the substrate. So precise control of sputtering power is not only important for depositing preferred orientation film, but also an effective method to avoid film to be mixed with Al composition.

Tab.1 Parameters for preparation of AlN thin films with different power values

Condition	Parameter value
Total-pressure (Pa)	1.2
N_2: Ar	20: 12
Power (W)	90 100 120 150
Al target-substrate distance (cm)	3.5
Substrate temperature (°C)	300

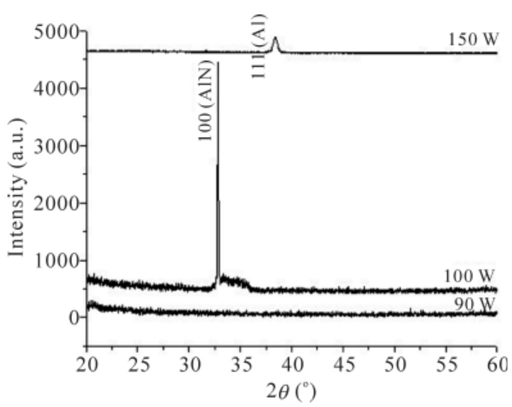

Fig.1 XRD patterns of AlN films at different power values

Tab.2 shows the parameters with different total-pressures for preparation of the AlN/Al film. Fig.2 shows the XRD pattern of the film by changing sputtering total-pressure with other parameters stable. Keeping the distance from the target to the substrate and sputtering power constant, the mean free path (MFP) of sputtering particles is directly related to the sputtering total-pressure. On one hand, the pressure affects the concentration of the working gases and the amount of aluminum atoms bombarded from the target. On the other hand, the low pressure can make bombarding particle without enough energy for migration when it arrives at the substrate, and it is not conductive to crystallization of the film. The XRD measurement shows that the Al atoms can be deposited on the substrate before nitride in low pressure environment, because the Al atoms have so large kinetic energy that there is not enough time for nitride to reach the substrate. When the pressure is 1.4 Pa, it can be seen that there is a (100) diffraction peak at $2\theta=33.2°$. If the pressure exceeds 1.4 Pa, there is no film crystallized on the substrate. So the control of the working pressure is very important for preparing the film crystallized to AlN film without Al texture. For depositing AlN film with right stoichiometric proportion of Al and N, proper working pressure is needed.

Tab.2 Parameters for preparation of AlN thin films with different sputtering total pressures

Condition	Parameter value
Total-pressure (Pa)	0.1 0.6 1.2 1.4
N_2: Ar	20: 12
Power (W)	100
Al target-substrate distance (cm)	3.5
Substrate temperature (°C)	300

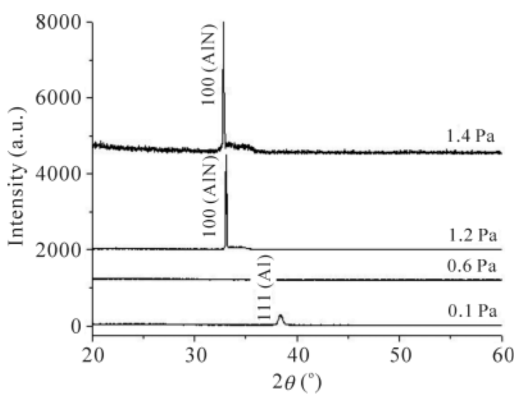

Fig.2 XRD patterns of AlN films at different total-pressures

Tab.3 shows the parameters with different ratios between N_2 and Ar for preparation of AlN/Al film. With increase of argon percentage and decrease of nitrogen percentage, more Al atoms are bombarded down from the substrate by the sputtering atoms. In the environment, the arrival of more Al atoms carrying more energy can lower film's crystallinity, and the AlN thin film rich in Al texture is deposited at last.

The results of experiment in Fig.3 show that adjusting the ratio of N_2 and Ar to 22:10 (sccm), *a*-axis (100) preferred orientation AlN film without Al texture is deposited successfully. On the contrary, with the ratio of N_2 and Ar increasing, there are less Al atoms bombarded, and the Al atoms carry less energy, so there is no crystallized film on the substrate, because Al atoms can not reach the substrate due to the high ratio of N_2 and Ar.

The influence of distance from target to substrate is not obvious. The experiment shows the AlN film is prone to rich in Al when the distance is short. Long distance can increase the collisions among the nitrogen atoms, argon atoms and Al atoms when Al atoms fly to the substrate, and then AlN film is crystallized difficultly.

Tab.3 Parameters for preparation of AlN thin films with different ratios of N_2 to Ar

Condition	Parameter value
Total-pressure (Pa)	1.2
N_2: Ar	20: 12, 22: 10, 24: 8
Power (W)	100
Al target-substrate distance (cm)	3.5
Substrate temperature (°C)	300

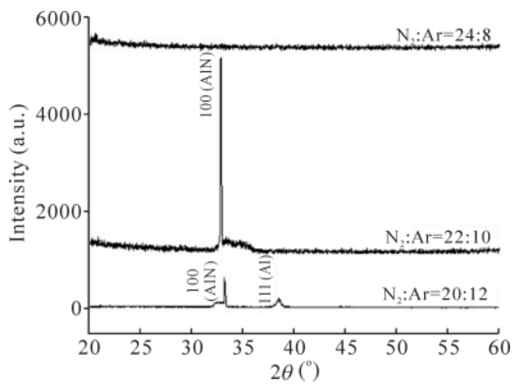

Fig.3 XRD patterns of AlN films at different ratios of N_2 to Ar

The main reason of AlN film rich in Al texture is that aluminum atoms participating in the reaction are increased on the substrate surface. From the results shown in Fig.4, we can see that introducing N_2 to the chamber during annealing treatment in a high temperature is helpful. This approach can reduce the defect and unsaturated bond of AlN film, and re-crystallize the superfluous aluminum with N_2[15,16].

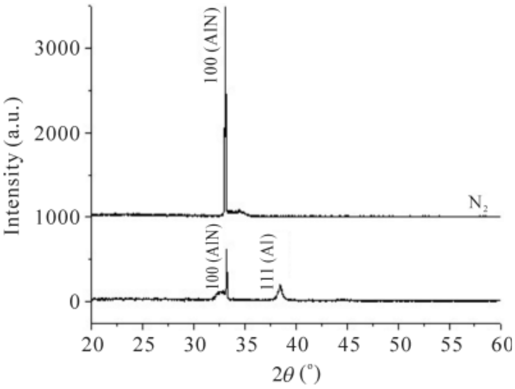

Fig.4 XRD patterns of AlN films at different annealing treatments

In this paper, the parameters which influence the amount of Al texture in the AlN film are researched based on RF magnetron sputtering system on the Si (100) substrate, and how these factors influence the deposition of thin film is analyzed from the point of molecular motion, molecular energy and film growth dynamics. The AlN (100) preferred orientation film without Al texture is deposited under the accurate control. Annealing treatment in the N_2 environment is helpful to reduce the Al composition and improve the resistivity of the film.

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