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Characterization of Aluminium Nitride Thin Films

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Abstract: Aluminium nitride (AlN) thin films have been synthesized by evaporation of aluminium and simultaneous irradiation with nitrogen ions, ion-vapour deposition method, at the substrate temperature of room temperature or 473K. The kinetic energy of the incident nitrogen ion beam has been kept at 0·5 keV and the deposition rate has been varied from 0·075 to 0·28 nm/s. The structure of the synthesized films has been examined by X-ray diffraction (XRD) and the surface morphology has been characterized by atomic force microscopy (AFM). In the XRD patterns of both films synthesized at room temperature and 473K, the diffraction lines due to the AlN(10·0), (00·2) and (10·1) planes have been discerned. AFM observations reveal that the surface of the films synthesized at 473K becomes rough as compared with the films synthesized at room temperature. This may be attributed to growth of AlN particles on the substrate kept at 473K. Furthermore, in the films synthesized at the 473K substrate, several aggregated protrusions can be observed on the relatively smooth surface at the deposition rate of 0·28 nm/s, while the surface of the films is uniform on nanometre scale at the deposition rate of less than 0·12 nm/s. The present results suggest that the synthesis of the AlN films with uniform surface is feasible by controlling the substrate temperature and the deposition rate. © 1996 Elsevier Science Limited and Techna S.r.l.

1 INTRODUCTION

Aluminium nitride (AlN) thin films have recently attracted considerable attention because of their unique properties, such as high thermal conductivity, low thermal expansion coefficient, high hardness and electrical resistivity, as well as a high surface acoustic wave velocity and a piezoelectric character. Owing to these unique properties, AlN thin films are considered to be one of the most useful materials applied to electronics fields, and AlN thin films are usually synthesized by chemical vapour deposition (CVD) or physical vapour deposition (PVD) techniques.[1–4]

When one attempts to apply AlN thin films to electronics fields, e.g. insulators and heat sink materials, as well as to the coating materials, it is desired to synthesize AlN films with smooth and uniform surfaces. Both scanning tunnelling microscopy[5] and atomic force microscopy (AFM)[6] have enabled us to improve the resolution of surface characterization to the atomic level, although the surface roughness generally limits the resolution to nanometre scale. In addition, AFM has the advantage of stable measurements for insulating materials like AlN thin films.

To the authors' best knowledge, however, there have been few studies of surface characterization of AlN thin films by AFM in the literature. In a previous article, we reported AlN thin films were synthesized by evaporation of aluminium and simultaneous irradiation with nitrogen ions, ion-vapour deposition (IVD) method, and the surfaces of AlN thin films synthesized both at room temperature and 473K were observed by AFM.[7] In the conditions of the incident nitrogen-ion beam

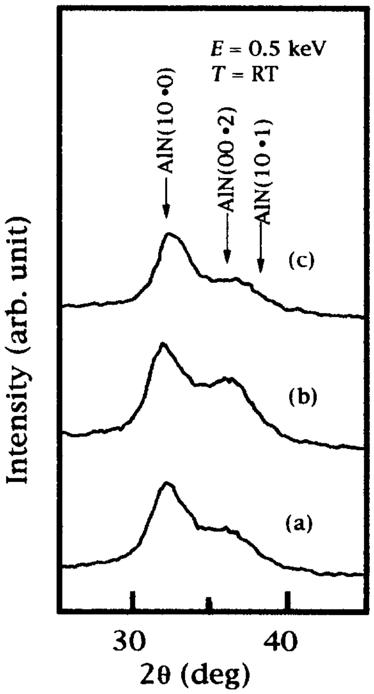

Fig. 1. X-ray diffraction patterns of the films synthesized at room temperature. The deposition rate is (a) 0·075 nm/s, (b) 0·13 nm/s and (c) 0·28 nm/s. The kinetic energy of nitrogen ions is kept at 0·5 keV.

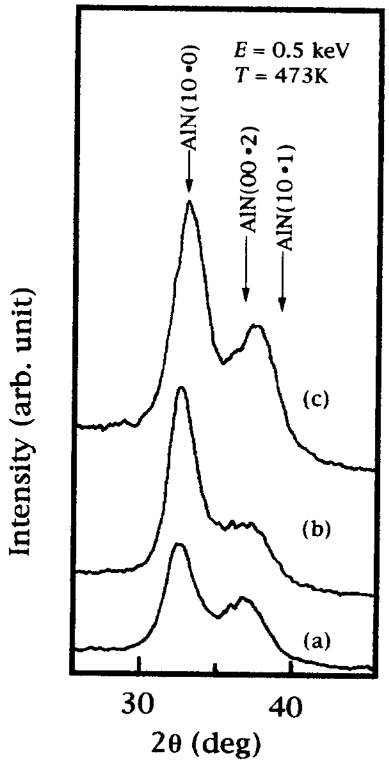

Fig. 2. X-ray diffraction patterns of the films synthesized at 473K. The deposition rate is (a) 0·075 nm/s, (b) 0·12 nm/s and (c) 0·28 nm/s. The kinetic energy of nitrogen ions is kept at 0·5 keV.

energy of 2 keV, the surface of the films synthesized at room temperature is smooth on nanometre scale, but the surface of the films synthesized at 473K is covered with protrusions of 20–70 nm in height. Recently, we have studied the influence of the incident nitrogen-ion beam energy on AlN film structure and found the films synthesized with lower-energy ion beam (0·5 keV) show more highly oriented structure along the *c*-axis.[8]

In the present article, using the IVD method, AlN thin films are synthesized at the substrate temperature of room temperature or 473K by varying the deposition rate under a constant ion-beam energy of 0·5 keV. The surface morphology of synthesized films at the various deposition rates is characterized by observations with AFM and the influence of the substrate temperature and the deposition rate on the surface morphology is examined.

2 EXPERIMENTAL

The schematic drawing of the IVD apparatus used for synthesis of AlN thin films has been shown in our previous article.[7] Nitrogen gas (99·99% pure) and aluminium (99·99% pure) were used as an ion source and a target. Film synthesis was performed on glass and silicon substrates mounted in a high vacuum chamber. After evacuating the vacuum chamber to about $2{\cdot}7\times10^{-4}$ Pa, pure nitrogen gas was introduced to the ionization chamber and nitrogen ions were generated by an arc discharge and then a nitrogen beam was obtained with electric-field lenses for focusing and accelerating. The kinetic energy of the nitrogen ion beam was kept at 0·5 keV and the deposition rate was varied from 0·075 to 0·28 nm/s. Aluminium was evaporated by an electron bombardment and the evaporation rate was monitored by a quartz sensor. Films were synthesized at the substrate temperature of room temperature or 473K.

The thickness of the synthesized film was measured with the Talystep (Rank Taylor Hobson Ltd). The crystallography of the films was determined by X-ray diffraction (XRD) with a Cu target (RINT2500, RIGAKU Co.). The surface morphology was observed by AFM (NanoScope III, Digital Instruments) with a Si cantilever. The microstructure of the films was studied by transmission electron microscopy (TEM) (JEM-2010F, JEOL).

3 RESULTS AND DISCUSSION

Figure 1(a), (b) and (c) show typical XRD patterns of the films synthesized at room temperature

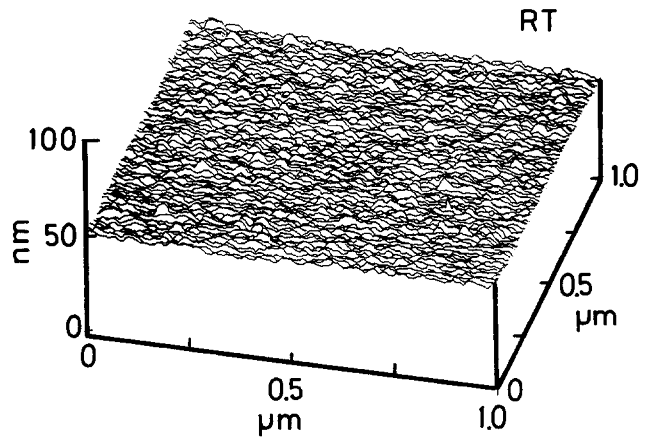

Fig. 3. Atomic force microscope image of the surface of the AlN films synthesized at room temperature.

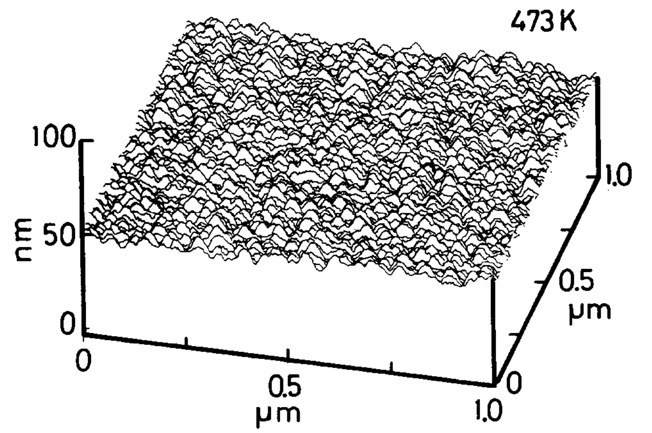

Fig. 4. Atomic force microscope image of the surface of the AlN films synthesized at 473K.

on the silicon substrates at deposition rates of 0·075, 0·13 and 0·28 nm/s, respectively. All films were synthesized at the ion-beam energy of 0·5 keV. The thickness of each film is approximately 300 nm. In these patterns, the stronger peak is assigned to the AlN(10·0) plane and the broad peak is composed of AlN(00·2) and AlN(10·1) planes.

Figure 2(a), (b) and (c) show typical XRD patterns of the films synthesized at 473K on the silicon substrates at deposition rates of 0·075, 0·12 and 0·28 nm/s, respectively. All films were synthesized at the ion-beam energy of 0·5 keV. The thickness of each film is approximately 300 nm.

From Figs 1 and 2, it is found that AlN thin films are synthesized even at room temperature, but the full width at the half maximum of the AlN(10·0) peak becomes much sharper with increasing substrate temperature. This means the film synthesized at 473K is more highly crystallized than the film synthesized at room temperature. This tendency agrees well with our previous results.[8]

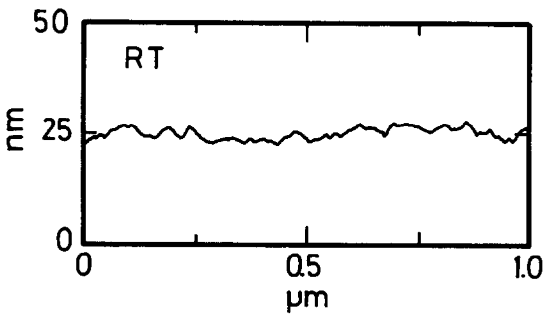

Fig. 5. Cross-sectional profile of the surface of the AlN films synthesized at room temperature.

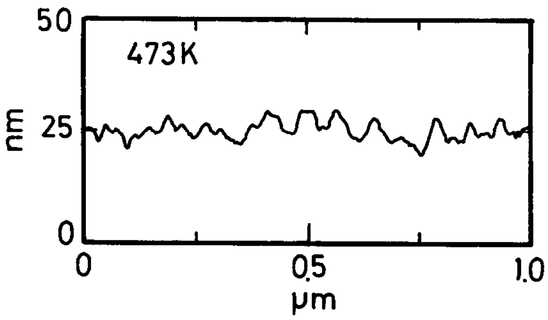

Fig. 6. Cross-sectional profile of the surface of the AlN films synthesized at 473K.

Figures 3 and 4 display AFM images of the surfaces of AlN films synthesized at room temperature and 473K, respectively. Both films were synthesized at the deposition rate of 0·075 nm/s and the thickness of each film is approximately 100 nm. The topographic data were acquired at 256 points per scan with 256 scans per image. These images were corrected by subtracting the background slope.

From these images, it can be seen that the surface of the films synthesized at room temperature is much smoother than the surface of the films synthesized at 473K. In order to characterize the surface profile in detail, the typical cross-sectional profiles are calculated from the AFM images and shown in Figs 5 and 6 for the films synthesized at room temperature and 473K.

From these figures, it can be said that the surface of the films synthesized at room temperature is smooth on nanometre scale, while the surface of the films synthesized at 473K is covered with particle-like features. The height and the base diameter of the typical particle-like feature are 10 nm and 60 nm, respectively. The values of the average surface roughness of these films are evaluated to 0·98 and 2·0 nm for the films synthesized at room temperature and 473K. Both values are approximately one third of those of the films synthesized with nitrogen ion beam of 2·0 keV.[7] This result suggests that the surface of the films can be smooth with decreasing the incident ion-beam energy.

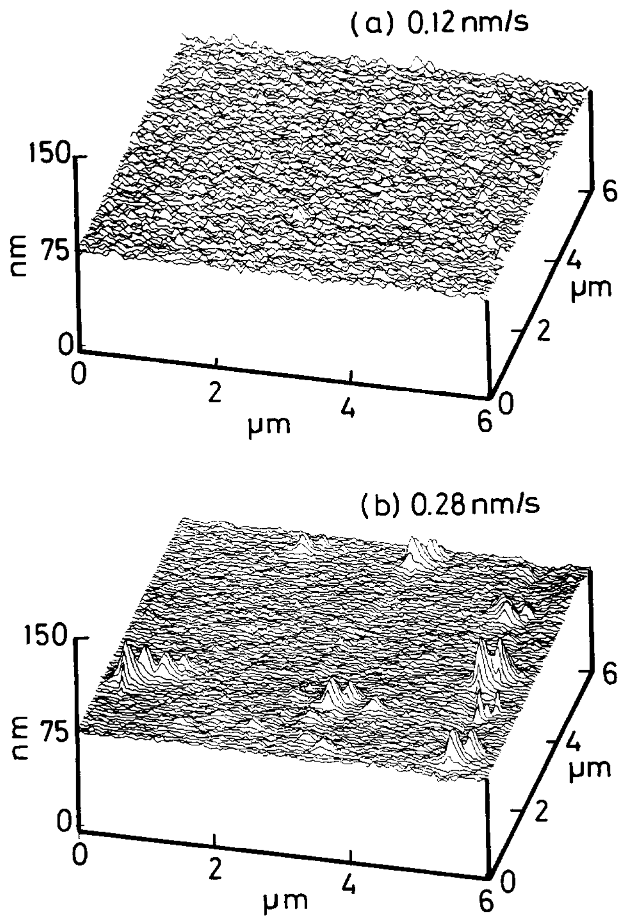

Fig. 7. Atomic force microscope images of the surface of the AlN films synthesized at 473K. The deposition rate is (a) 0·12 nm/s and (b) 0·28 nm/s. The kinetic energy of nitrogen-ion beam is kept at 0·5 keV.

As shown in Figs 1 and 2, dominant X-ray diffraction lines of AlN can be seen in the films synthesized at 473K rather than in the films at room temperature. Therefore, the influence of the deposition rate on the surface roughness was studied at the substrate temperature of 473K by varying the deposition rate under the constant ion-beam energy of 0·5 keV.

Figure 7 (a) and (b) display AFM images of the surfaces of the films synthesized by varying the deposition rate of 0·12 and 0·28 nm/s, respectively. The thickness of these films is approximately 100 nm. The scan size is extended to 6 μm $\times$ 6 μm in order to observe the surface widely.

From Fig. 7 (a), it is found that the films synthesized at the deposition rate of 0·12 nm/s show a rather uniform surface. The surface of the films synthesized at the deposition rate of 0·075 nm/s is also found to have a uniform surface. However, from Fig. 7 (b), the characteristic features can be observed on the surface of the films synthesized at the deposition rate of 0·28 nm/s. On the surface of the films synthesized at the deposition rate of 0·28 nm/s, several aggregations of sharp protrusions are formed on the smooth surface like islands in the sea. The height of the protrusions varies from a few nm to approximately 30 nm. These aggregated protrusions seem to be remnants of the preferential nucleation and growth of AlN. This difference in the surface morphology caused by the deposition rate may be explained by the assumptions that (1) the films have grown uniformly until the deposition rate of 0·12 nm/s and (2) at the deposition rate of 0·28 nm/s preferential nucleation and growth have occurred.

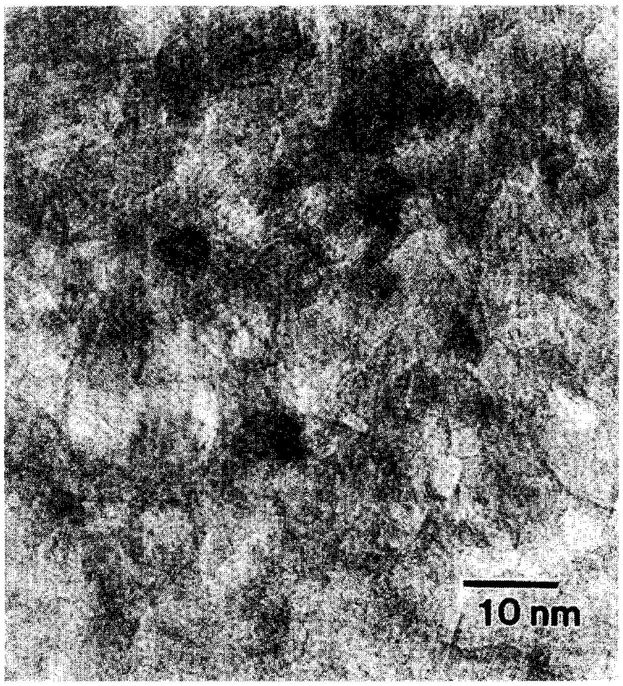

Fig. 8. Transmission electron microscope photograph of the AlN films synthesized at 473K. The deposition rate is 0·28 nm/s and the kinetic energy of nitrogen-ion beam is kept at 0·5 keV.

In order to examine the microstructure of the films, TEM observations were applied for the films synthesized at 473K and the deposition rate of 0·28 nm/s. Figure 8 shows a typical TEM photograph and it is found that the film shows a polycrystalline structure and the typical size of grains is 10 nm or less, and in addition, the preferential orientation has not occurred. TEM observations, however, do not provide significant information about the aggregated protrusions. Therefore, it is necessary to observe the surface morphology in the early stage of AlN deposition with different deposition rates in order to clarify the mechanism of formation of aggregated protrusions.

4 CONCLUSIONS

Aluminium nitride thin films have been synthesized from nitrogen gas and aluminium by the IVD method at the substrate temperature of room temperature or 473K. The kinetic energy of nitrogen ions has been kept at 0·5 keV and the deposition rate has been varied from 0·075 to 0·28 nm/s. The surface of the films is characterized by observations with AFM. The surface of the films synthesized at room temperature is smooth on nanometre scale and the surface of the films synthesized at 473K is covered with particle-like features. In the case of the film synthesis at the 473K substrate, the films synthesized at the deposition rate until 0·12 nm/s show uniform surface, but on the surface of the films synthesized at the deposition rate of 0·28 nm/s several aggregations of sharp protrusions are formed on the smooth surface. These observations suggest that the preferential nucleation and growth of AlN have occurred at the high deposition rate.

From the present study, it can be concluded that it is feasible to synthesize AlN thin films with smooth and uniform surfaces by controlling the substrate temperature and the deposition rate. Furthermore, AFM observations of the nucleation and growth process of AlN films are now in progress.

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