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The obtaining of aluminum nitride films on the aluminum sublayer by pulsed DC magnetron sputtering

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Abstract. We report aluminum nitride films deposit with and without an aluminum sublayer on a silicon substrate at magnetron power values ranging from 150 to 300 W. The deposition time is selected so that the thicknesses of the samples in the same order. The phase and elemental composition of the samples are studied by XRD and EDS, respectively. The samples demonstrate *c*-axis preferred orientation that is indirectly confirmed by ellipsometry. In addition, it is shown that the growth rate of aluminum nitride films increases with increasing power. The morphology of aluminum nitride samples is studied by SEM and AFM; it is shown that the roughness of samples obtained on an aluminum sublayer increases. Raman spectra of samples demonstrate A_1 (TO) mode shift associated with the presence or absence of the aluminum sublayer.
Keywords: AlN films, magnetron sputtering, AlN/Al.

1. Introduction

Pulsed DC magnetron sputtering is a method that allows obtaining various films, including aluminum nitride. Hexagonal aluminum nitride is piezoactive and has a piezoelectric coefficient of 5.5 pm/V, due to which it can be used in micro- and nanoelectronics, for example, in radio-frequency filters and resonators [1, 2]. In microelectronics, it is usually necessary to obtain film elements that will be part of a multilayer structure, so there are requirements for the characteristics of aluminum nitride films; for example, they must be highly *c*-axis-oriented and have low roughness.

Some researchers [3, 4] claim that the influence of the substrate on the formation of the aluminum nitride structure is critical, while others [5, 6] argue that deposition parameters are more crucial. The film growth rate and crystallinity increase, and the roughness decreases with the magnetron discharge power increases [7, 8]. However, there is evidence of an increase in internal stresses [8] and degradation in the optical characteristics of AlN films [9] with increasing magnetron power.

The existence of metallic sublayers has an effect on the deposited AlN films [10]. However, the influence of aluminum sublayer on the growth rate, texture and Raman modes of aluminum nitride films is still a debated issue. The purpose of this work was to establish the influence of the Al sublayer on the structure, morphology and physical properties of aluminum nitride films by comparing polycrystalline aluminum nitride films obtained at different magnetron powers with and without an aluminum sublayer.

2. Experiment

To obtain the films, a magnetron sputtering system with the bipolar power source APEL-M-1.5BP-800 DC was used, operating in a pulsed direct current mode with a frequency of 100 kHz, a positive pulse duration of 3 μs, and a duty cycle of 70%. The distance between the substrate and the target surface was 80 mm. The substrate was single-crystalline p-type silicon (Si) doped with boron (B) with a resistivity of 0.85–1.15 Ohm·cm and crystallographic orientation (111). Sputtering and reactive gas (argon Ar and nitrogen N_2, respectively), with a purity of 99.999%, were supplied to the region between the target and the magnetron through a gas distribution ring installed above the magnetron to improve the homogeneity of the gas mixture. Before deposition, the chamber was evacuated to a pressure of $2 \cdot 10^{-4}$ Pa, after which an Al target of 99.95% purity was sputtered with the substrate closed for 10 minutes at a power of 600 W to clean the target surface from oxides and other contaminants.

Two series of samples were obtained: with an aluminum sublayer (AlN/Al/Si) and without it (AlN/Si) on a silicon substrate. In each series, 4 samples were obtained with the magnetron power varying from 150 to 300 W. Linear dependence of growth rate on magnetron power was found experimentally, so the deposition time was chosen to obtain the films with thicknesses around 1000 nm. The remaining deposition parameters were constant: substrate temperature: 200 °C, nitrogen percentage: 20% and gas flow: 100 ml/min. The aluminum sublayer thickness did not exceed 100 nm.

Phase composition of AlN films was investigated by X-ray diffraction (XRD) analysis on ARL X'TRA diffractometer (Thermo Fisher Scientific, Switzerland) with a Cu-Kα radiation source (λ = 1.5418 Å). Shooting range 2θ was from 15 to 85° in steps of 0.05°, with an accumulation of 3 s per point.

The ASEB-5 spectral ellipsometer (Institute of Semiconductor Physics of the Siberian Branch of the Russian Academy of Science, Novosibirsk, Russia) was used for ellipsometric measurement in the wavelength range from 250 nm to 900 nm in steps of 1 nm with the radiation incidence angles changing from 55° to 70° in steps of 5°. Models of isotropic homogeneous films were used for all measured angles to obtain optical constants and thicknesses.

The morphologies of the samples were obtained using the JSM 6700F scanning electron microscope (JEOL, Tokyo, Japan). The elemental composition of AlN films was obtained on the same device using the Quantax 200 energy dispersive X-ray spectrometry (EDS) analyzer (Bruker Nano GmbH, Berlin, Germany). The accelerating voltage of the electron beam during image acquisition was 15 keV. Elemental composition studies were carried out at an electron beam accelerating voltage of 5 keV.

The surface roughness of the AlN film was achieved using the Dimension Icon scanning probe microscope (Bruker, USA) using the VNCHV-A probe (Bruker, USA), using a stiffness coefficient of the cantilever beam of 42 N/m, a resonant frequency of 300 kHz, and a nominal radius of 8 nm. All data obtained by the probe microscope were processed in the NanoScope Analysis 1.50 software package (Bruker, USA). When processing the data, the tilt plane (1st order) was subtracted, and 0th order line flattening was applied prior to evaluation of the roughness.

Raman spectroscopy was used to analyze the structure of the samples. The T64000 spectrometer (Horiba Jobin Yvon) was used in a single mode, the spectral resolution was no worse than 2 cm^{-1}. Raman spectra were recorded in backscattering geometry. The excitation source was a fiber laser with wavelength 514.5 nm (Inversion-Fiber, Novosibirsk, Russia). The micro-Raman attachment based on a BX41 microscope (Olympus, Japan) was used. To prevent local heating of the films under the conditions of recording spectra, the laser spot was defocused, the spot diameter was about 10 μm, and the light power incident on the sample was approximately 1 mW. The incident light was linearly polarized, while the polarization of the scattered light was not analyzed. The Lorentzian was used as an approximation function for individual peaks in order to analyze.

3. Results and discussion

3.1. Phase and elemental composition

According to XRD data, the obtained AlN samples on both Si (Fig. 1a) and Al/Si (Fig. 1b) were single-phase textured films with a hexagonal AlN phase. In the diffraction patterns, of the entire set of reflections characteristic of polycrystalline AlN, only reflections 002 and 004 were observed. These films had a uniaxial texture along the crystallographic axis *c*, perpendicular to the substrate surface.

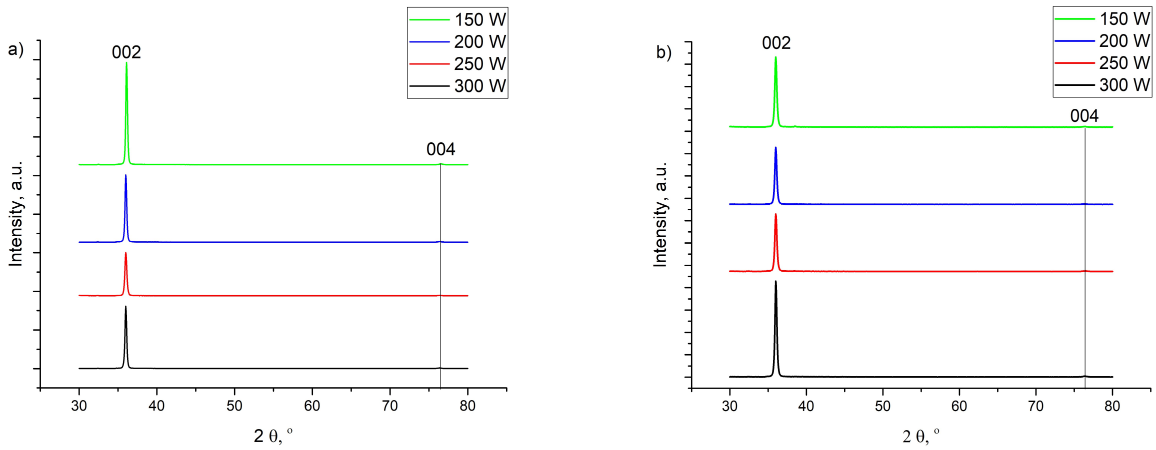

Fig. 1. X-ray diffraction patterns of AlN samples deposited a) on Si; b) on Al/Si.

It was found by EDS that the elemental composition of the samples changed slightly from sample to sample (no more than 2%, which is lower than the error of the method). The values of the atomic fraction of nitrogen were 52–54%, the aluminum fraction was 44–46%, and oxygen and carbon were not more than 2%.

3.2. Ellipsometry

Ellipsometry data indirectly confirmed highly *c*-axis oriented AlN films, which are characterized in literature by a refractive index of 2 and higher [11]. These refractive indexes were typical of all measured films, both deposited on aluminum and silicon, which confirmed the absence of significant influence of the aluminum sublayer on the structure of the films in the utilized magnetron power range.

Growth rate *versus* magnetron power dependence (Fig. 2) was obtained using film thickness values calculated from ellipsometric measurements. This dependence was found to be linear with an error not exceeding 10%. A comparison of both series revealed that there is a slight increase in the growth rate of an aluminum nitride film on aluminum in comparison with silicon.

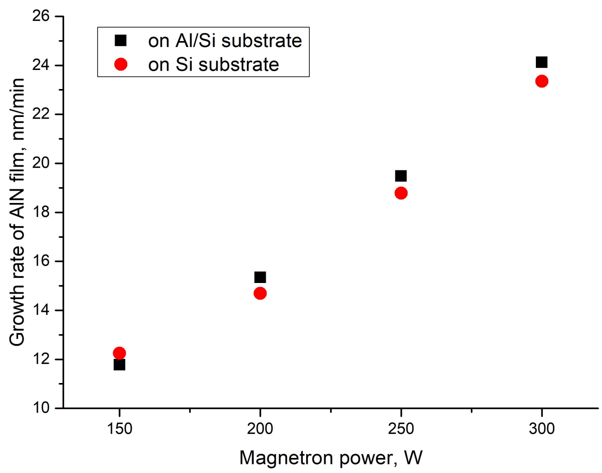

Fig. 2. Dependence of the AlN films growth rate on the magnetron power for two substrates.

3.3. Morphology

The morphology of the samples observed on the SEM images did not have features. Typical images of both series were presented in Fig. 3. These data were in good agreement with the AFM results, which provided information about morphology and roughness.

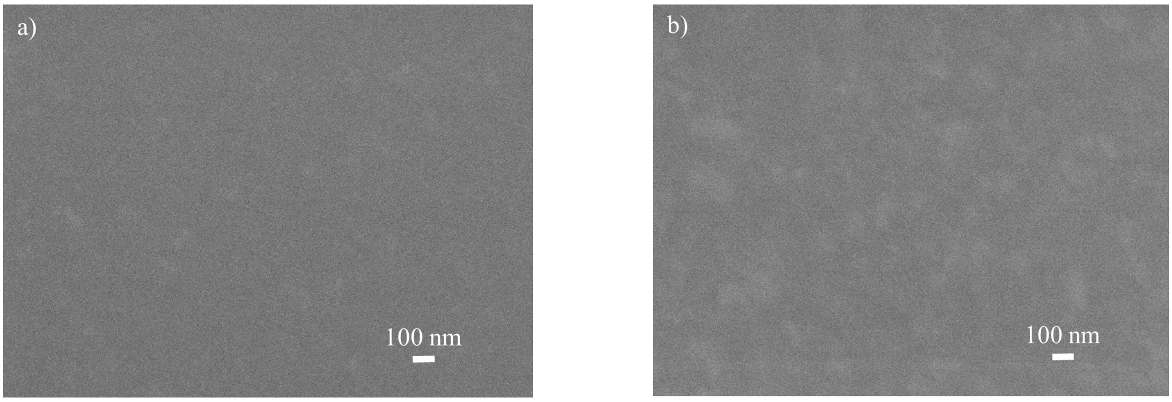

Fig. 3. Typical SEM images of AlN samples deposited a) on Si; b) on Al/Si.

Three-dimensional images of the surface were presented in Fig. 4. The average roughness of AlN films obtained on an aluminum sublayer did not exceed 6 nm and varied insignificantly from sample to sample with a change in magnetron power. The roughness of samples obtained on silicon was lower and did not exceed 2 nm, although the effect of power was insignificant either. Such roughness values are not crucial for practical applications of aluminum nitride films.

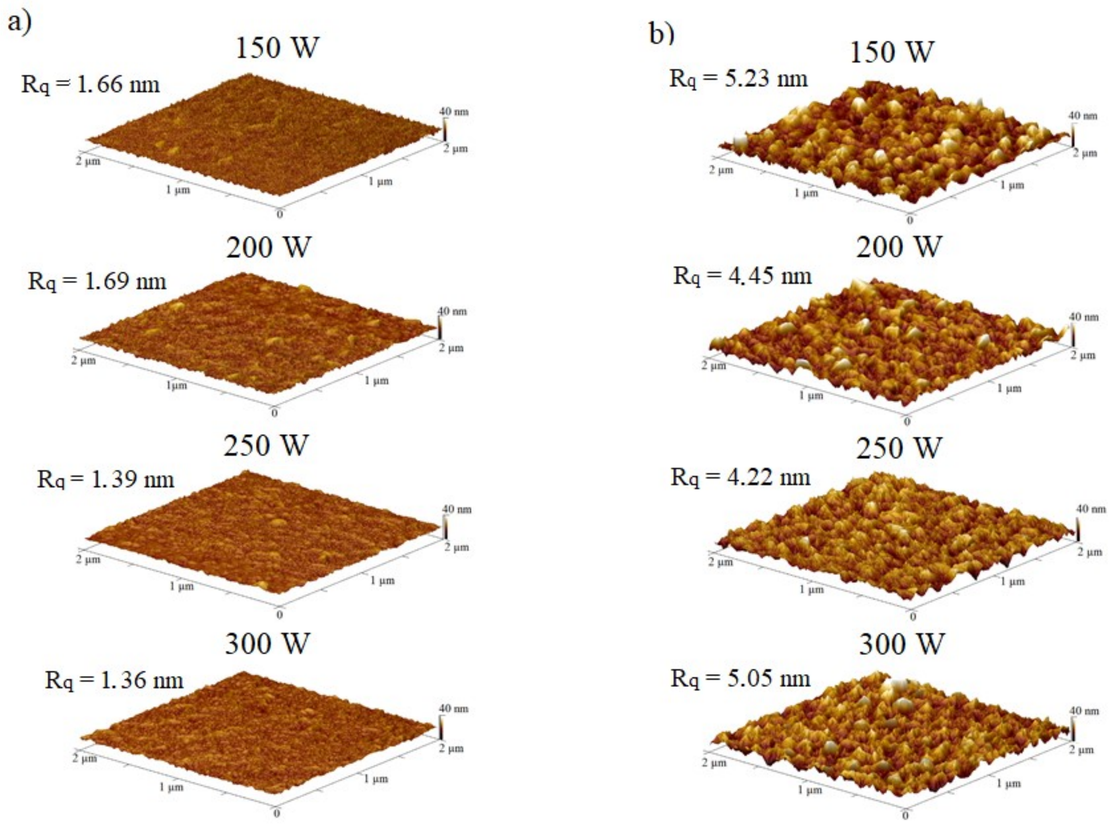

Fig. 4. 3D images of the morphology of AlN samples deposited a) on Si; b) on Al/Si.

3.4. Raman spectroscopy

The spectra of all samples were deconvoluted into the characteristic region from 580 to 750 cm^{-1} (Fig. 5) into two Lorentzian functions. While the position of the second peak changed slightly and fluctuated around 655 cm^{-1} from one sample to another, the position of the first peak varied for both series. Both of observed peaks were characteristic of hexagonal aluminum nitride [12, 13].

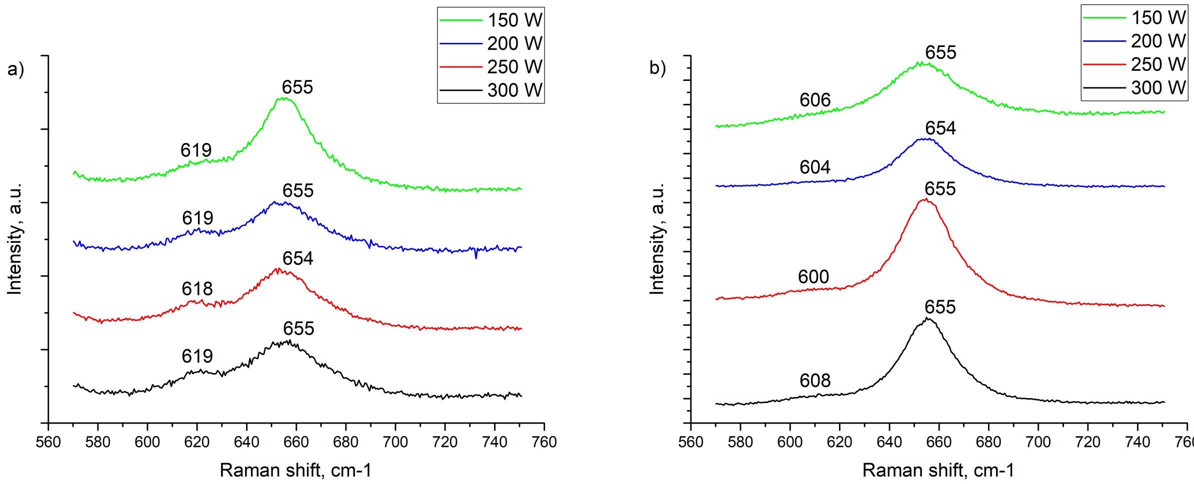

Fig. 5. Raman spectra of AlN samples deposited a) on Si; b) on Al/Si.

So, the second peak had a position of about 655 cm^{-1}, which is consistent with the literature data for the E_2 AlN mode [12–15]. However, all spectra revealed the presence of a less intense first peak, which corresponds to the A_1 (TO) mode [12]. The maximum position of this peak was from 600 cm^{-1} to 608 cm^{-1} for samples obtained on aluminum, while the samples obtained on silicon had a maximum position of the first peak of about 618–619 cm^{-1}.

It is worth noting that according to the literature, the position of this peak (at room temperature) varied from 607 cm^{-1} [14] for massive AlN crystals to 611 cm^{-1} [15] for AlN films grown on α- Al_2O_3. It can be assumed that the shift in the position of the peak was associated with the occurrence of internal stresses or structural defects. The blue shift of the A_1(TO) mode in films grown on silicon was caused by compressive strain [13].

4. Conclusion

We can conclude that films deposited on an aluminum sublayer compared in their characteristics with films deposited on pure silicon. All films obtained demonstrate the presence of an orientation perpendicular to the substrate. The growth rate of films with and without a metal sublayer barely differs. Films grown on silicon have higher smoothness, while films deposited on an aluminum sublayer show a less internal stresses, according to Raman spectroscopy.

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5. References

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