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Optical properties of AlN thin films obtained by reactive magnetron sputtering

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Aluminium nitride films were deposited by reactive magnetron sputtering in an argon-nitrogen atmosphere. The process was controlled by the optical emission spectroscopy using the intensity of an aluminium emission line as an input signal for a nitrogen flow controller. The influence of the substrate temperature and the target sputtering rate on the structure and optical properties of AlN films were investigated.

1. Introduction

Aluminium nitride is a direct, wide band-gap semiconductor ($E_g = 6.2$ eV) with the relatively high refractive index (~2.1) and low absorption coefficient ($k < 10^{-3}$) [1]. Thin films of AlN are transparent for the light wavelength from 0.2 to 12.5 μm, covering the region from infrared to ultraviolet. AlN is thermally and chemically stable and has high electrical resistivity. These properties make its thin film a suitable candidate for protective optical coatings and surface passivating layers of semiconductor surfaces. Parameters of the films depend very much on the stoichiometry, structure and texture, which are related to the method of film production, so the value of the gap given by different authors ranges from 3.6 to 6.0 eV and the refractive index ranges from 1.7 to 2.1 [2], [3].

A stable production of compound films in the reactive sputtering process demands a constant metal flux at the substrate, a constant flow of the reactive gas, and a constant consumption of the reactive gas. For magnetron sputtering, these parameters are often highly interdependent – in particular, the process parameters of reactive dc magnetron sputtering, such as reactive gas pressure and total pressure. So the method of process controlling strongly influences the physical and optical properties of AlN thin films [1]–[3]. Hence the influence of the deposition parameters has been an object of our investigations.

2. Film deposition

Films were prepared in a planar, balanced magnetron sputtering system presented in Fig. 1. An oil diffusion pump evacuated the chamber. The deposition arrangement is vertical and the magnetron discharge is driven by direct current. A target consists of

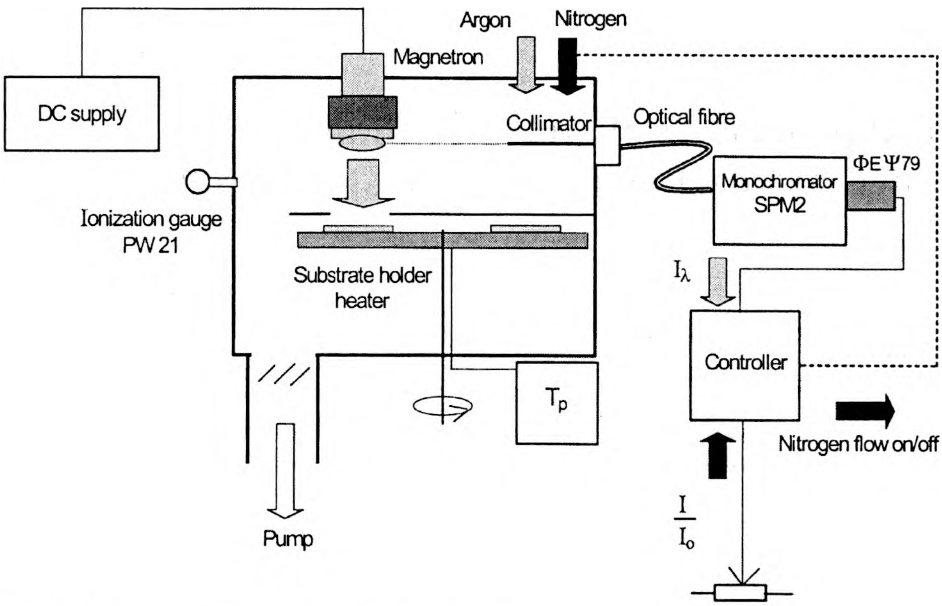

Fig. 1. Schematic drawing of the reactive dc magnetron sputtering system.

pure aluminium disks (100 mm in diameter). The chamber walls and the revolving substrate holder were grounded. The substrate holder was covered by a shutter table with a 20×30 mm window permitting deposition in one of the 8-substrate positions. The target to substrate distance was 35 mm. A controlled resistance heater allowed depositing films at substrate temperature up to 300 °C. Spontaneous heating, by the dc plasma and radiation from the target, gave substrate temperature of approximately 50 °C.

The ultimate pressure in the chamber was 2×10^{-5} hPa, but film depositions were typically performed at pressure of about 1×10^{-2} hPa. The target current was held constant at 1.25 A. Mixed gas atmospheres composed of argon (99.999%) and nitrogen (99.9999%) were used as a sputtering gas. Argon and nitrogen were introduced through independent mass flow controllers. The intensity I of optical emission line of aluminium, 396.15 nm, emerging from the plasma ring was observed via an optical fibre by a monochromator, and measured by a photomultiplier. It was used in the reactive gas flow feedback regulation system described in the previous paper [4]. The intensity of Al emission line obtained for sputtering in pure argon was taken as a reference I_0, and the value of I/I_0 were stabilised by changing the nitrogen flow. The ratio of I/I_0 reflected the flux of aluminium atoms arriving to the substrate. Low I/I_0 value corresponds to low sputtering rate in high nitrogen content atmosphere. Deposition time was in the range of 10–20 min – it increased with decreasing I/I_0 in order to get films of approximately the same thickness. The films were deposited on Corning Glass 0211 substrates. In order to reduce oxygen contamination of deposited films a two step procedure was applied. Firstly, with the shutter covering the substrates, the target was being cleaned by sputtering in a pure argon for approximately 15 min. Next, the argon gas was replaced by the mixture of argon and nitrogen and the sputtering continued for an additional 10 min before removing the shutter. Two series of samples were prepared for different substrate temperatures during deposition, 50 and 250 °C.

3. Structure measurements

Structural measurements were performed on X-Pert Philips diffractometer, using CuKa radiation (Fig. 2). The presented data are detector scans at low angle (1.5°) incidence X-ray beam and with parallel optics. Samples obtained at I/I_0 higher then 0.25 were opaque. Structure measurements detected in them a mixture of aluminium and AlN phases. For lower value of I/I_0 films were transparent and they were composed

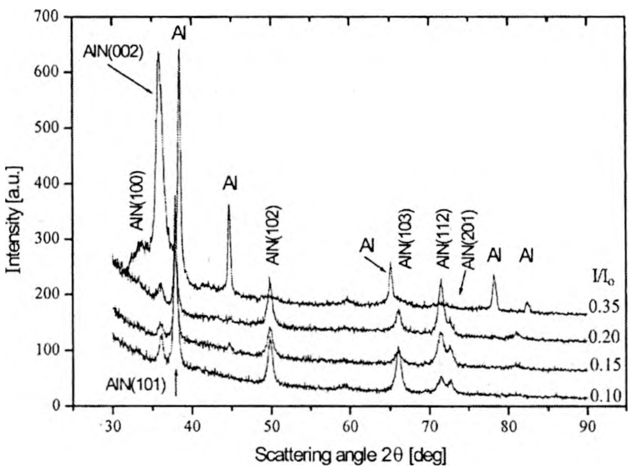

Fig.2. X-ray diffraction pattern for set of AlN films deposited onto heated (T_p = 250 °C) substrates.

mainly of hexagonal structure AlN with a small and decreasing amount of aluminium. The decreasing ratio of I/I_0 is accompanied by the change in film texture. We did not found a significant influence of the substrate temperature on the film structure in the region of I/I_0 where transparent aluminium nitrogen was deposited.

4. Optical measurements

Optical transmission measurements were performed using a Perkin–Elmer Lambda 19 spectrophotometer in the wavelength range of 0.3–2.0 μm. The light beam diameter was limited to 3 mm in order to suppress the possible influence of film thickness inhomogeneity on the modulation depth of interference fringes. Transmission spectra films obtained on unheated substrates, presented in Fig. 3, show the influence of aluminium contamination on absorption in the short wavelength region. Optical

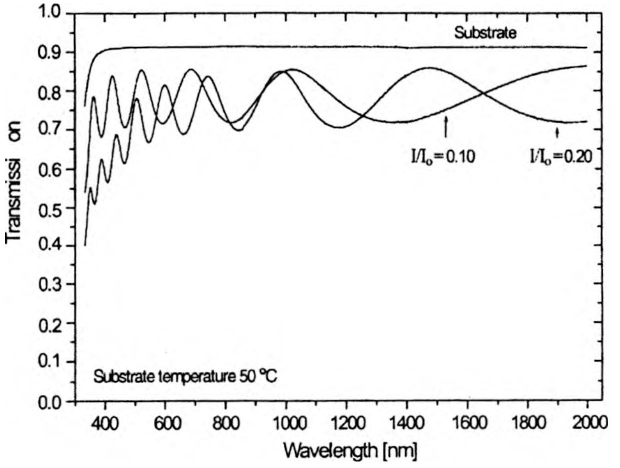

Fig. 3. Transmission spectra for AlN films deposited at substrate temperature of 50 °C.

parameters, refractive index and refraction coefficient were calculated from extrema of transmission interference fringes [5]. In the method the film thickness was also determined. The thickness of films was in the range of 0.50–0.75 μm.

Spectral dependence of refractive index and absorption coefficient is shown in Fig. 4 and Fig. 5. The refractive indices were in the range of 2.02–2.09 (at 600 nm). In sample $I/I_0 = 0.20$, $T_p = 250$ °C, which has larger crystallites, the value is the highest, $n = 2.08$, and is close to the result reported in literature for bulk single crystal samples [1]. Almost all the films exhibit normal dispersion of refractive indices. One exception is the case of the film deposited at $I/I_0 = 0.2$ and substrate temperature of 50 °C, which has also extinction coefficient significantly higher than the other films. It was the effect of aluminium admission in the film, the Al was detected by X-ray

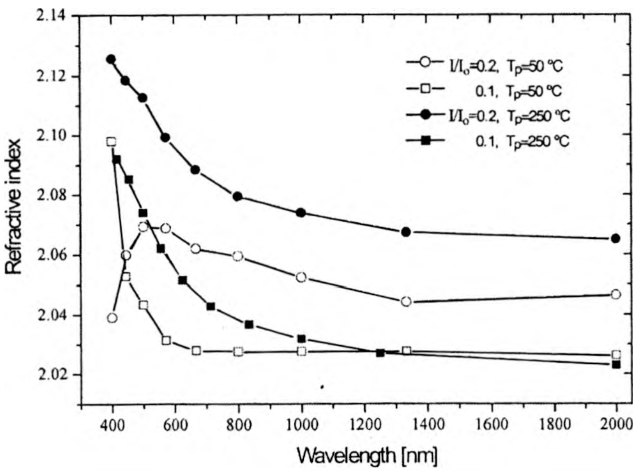

Fig. 4. Wavelength dependence of refractive index of AlN films.

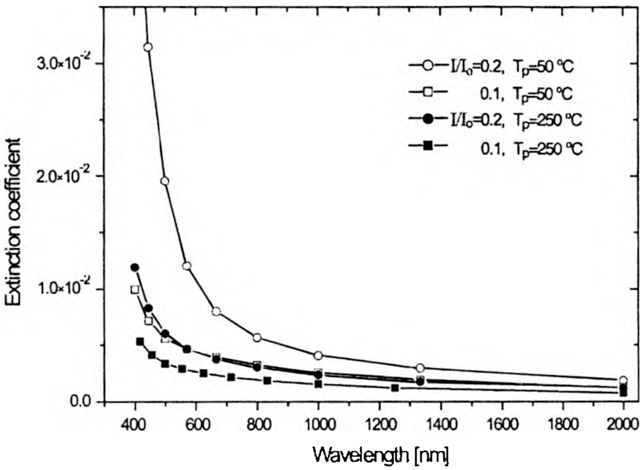

Fig. 5. Wavelength dependence of the extinction coefficient of AlN films.

analysis. The increase in the substrate temperature resulted in decreasing absorption of films and improving the crystalline structure. The contamination of AlN films by a small amount of aluminium is probably the reason for absorption, small but high in comparison to that in a single crystal. The results suggest that there is some optimum of sputtering conditions for obtaining the high value refractive index films as for the lower value I/I_0 a refractive index decreases.

In conclusion, it is observed that the optical parameters are a sensitive function of the preparation conditions. This may be due to some vacancies in nitrogen and some aluminium atoms included in crystallites during sputtering. The magnetron reactive sputtering controlled by the optical emission spectroscopy allows the deposition of the films of aluminium nitride with good optical properties in a reproducible manner.

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References

[1] Strite S., Morkoc H., J. Vac. Sci. Technol. B **10** (1992), 1237.
[2] Gadenne M., Plon J., Gadenne P., Thin Solid Films **333** (1998), 251.
[3] Drusedau T.P., Blasing J., Thin Solid Films **377–378** (2000), 27.
[4] Brudnik A., Czternastek H., Zakrzewska K., Jachimowski M., Thin Solid Films **199** (1991), 45.
[5] Swanepoel R., J. Phys. E: Sci. Instrum. **16** (1983), 1214.

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