

MORPHOLOGY AND STRUCTURAL INVESTIGATION OF ALUMINUM NITRIDE LAYERS PREPARED BY MAGNETRON SPUTTERING

Dinara Sultanovna Dallaeva

Doctoral Degree Programme (2), FEEC BUT

E-mail: xdalla02@stud.feec.vutbr.cz

Supervised by: Pavel Tománek

E-mail: tomanek@feec.vutbr.cz

Abstract: Aluminum nitride thin films were obtained by magnetron sputtering of aluminum target. The films surface was studied by atomic force microscopy and scanning electron microscopy. Using of buffer nitridized layer on the substrate allowed the formation of perfect structure films. Lateral force atomic force microscopy was used to study the morphology heterogeneity. The dependence of films structure on the formation conditions has been defined. The objective of the study is to contribute to the improvement of technological process of dry etching and film deposition.

Keywords: Dry etching, epitaxy, substrate, thin film, scanning probe microscopy

1. INTRODUCTION

Study and development of the perspective materials are relevant from technological side and are very important from economical point of view. The research in this field is focused to the development of novel materials with superior properties allowing the fabrication of devices with improved performance [1].

Aluminum nitride (AlN) is a direct wide band gap semiconductor with combined properties of high electrical resistivity and high thermal conductivity [2]. Structure and film texture of AlN have attracted much attention due to wide range of application of this material and its unique properties. Nitrides semiconductors show properties that cannot be found in traditional semiconductor. Materials like silicon and gallium arsenide have not the large enough band-gap for devices design of short-wave spectrum range. Research of AlN formation processes is of a remarkable necessity in the field of optoelectronics. The problem of AlN thin layers manufacturing is the absence of suitable epitaxial substrates of the identical material. Hence the other materials such as sapphire (Al_2O_3) and silicon carbide (SiC) are used for the growth of AlN [3, 4].

There is a number of appropriate methods for thin film growth like sublimation, sputtering, organometallic vapour phase epitaxy, plasma-enhanced, molecular beam epitaxy, etc [5]. Magnetron sputtering is a widely spread method of the thin film deposition. Uniform coatings of this type are necessary in many fields of science and engineering, e.g., in microelectronics, optical industry (thin film sensors, photovoltaic thin films in solar cells, metallic cantilevers and interconnection, etc.) [6]. The technological task of the research is to find a source material for layer deposition from the target. The deposition begins when the discharge occurs. The collision of gas ions and target makes extractions of near-surface atoms, molecules and clusters from the source material, and these particles form the thin film on the substrate.

In its turn the substrate preparation is also a significant procedure which defines the film quality. Dry plasma etching is important when the chemical etching is troublesome. Simple sputtering is

non-selective elimination of surface atoms due to plasma-induced non-reactive gas ions which vertically impinge on the surface of the substrate without any method to control the etch print.

2. EXPERIMENTAL RESULTS

A standard vacuum deposition system was used with two ring-type magnetrons and an ion source. The device schematic is in the figure 1. There is a substrate heater for substrate, a reactor of HF-activated nitrogen plasma, and gas flow regulators there. Crystalline substrate which has to satisfy requirements of deposition of epitaxial growth on it and the source of the desired product are main parts of the deposition process. Fabrication of high-quality thin film is a complicated and multiparametric problem. The main operating parameters are crystal-lattice orientation of the substrate, deposition rate defined by gas supersaturation and gas-dynamic behavior of the reactor. The ionizing efficiency can be improved by using of magnetic field, so ions are generated relatively far from the target and the probability of energy losses is great in ordinary planar diode systems. The magnetic field lines cross the lines of electric field. The mechanism of device is based on the braking of electrons in the crossed magnetic and electric fields. Thus the trajectory of electron in magnetron device changes under simultaneous effect of electric and magnetic fields. Electrons appear out of the cathode as a result of ionization, and consequently they are localized above the surface of sputtering material. Electrons are trapped by magnetic field which makes them move on cycloidal path, and also by electric field repulsion from cathode to anode. As a result, a probability and number of electron collisions with argon molecules and consequently the ionization sharply increase. Ionization rate varies in the deposition area because of inhomogeneity effect of electric and magnetic field in the near-cathode surface. Maximum of ionization occurs in the area where the magnetic lines are perpendicular to electric-field lines, and minimum is obtained in area in which the line directions are parallel. So, the localization of plasma at near-cathode surface allows get significant greater ion current density at lower pressure and hence provides high sputtering rate.

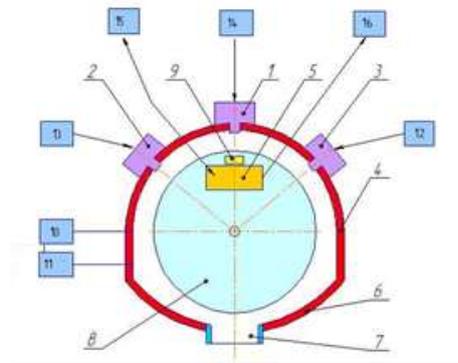


Figure 1: Schematic sketch of vacuum deposition system:

1– ion source; 2, 3 – magnetrons; 4 – frame; 5 – sapphire substrate heater; 6 – chamber shutter; 7 – quartz window; 8 – carousel; 9, 15 – HF-reactor and generator; 10 – fore pump; 11 – turbo-molecular pump; 12, 13, 14, 16 – power supply units

In spite of all advantages of this method there are a lot of features to investigate yet. One of them is a choice of the source with convenient target material, and its formation and preparation. Target is supposed to be without pores and hollows in order to avoid local melting and sprinkling of the material as there is high power at the small area of target. The high-purity aluminum target was used in this study.

Substrate preparation included dry etching and nitration by nitrogen implantation into the sapphire substrate (0001 orientation), with subsequent high-temperature annealing at 1400-1600 K in a nitrogen atmosphere. The average Resistance-type heater was used for preheating of the substrate during the structure formation. The main condition of construction and making of the heater is to

ensure a non-gradient thermal field on the surface of the substrate, and find tools for regulation and maintenance at certain temperature. Water-cooled air-tight feed-through terminals of the heater are in the wall of the vacuum chamber. Temperature of the deposited films is one of the most important parameters of the deposition process. Surface temperature is connected to adhesion strength, surface structure, and level of residual coating stress. By changing the deposition surface temperature it is possible to change the structure of films and thus their mechanical and electro-physical properties. Adhesive strength increases with the temperature growth. The temperature should be chosen according to some necessary prerequisites for material properties, constructive features and requirements to the structure of film, method of deposition, etc. Substrate heater is on the rotator-carrousel and it is possible to use it as heating radiation or for prior degassing of the vacuum chamber.

The values of the absolute height of five highest peaks and the depths of the five deepest pits or valleys within the sampling area of the surface before and after dry etching is equal to 27.3014 nm and 15.2746 nm respectively (Fig. 2).



Figure 2: Al₂O₃ substrate (a) before and (b) after plasma etching

X-ray fluorescence was used for elemental analysis (PHI 5500 ESCA, Physical Electronics). There is gradual decreasing of nitrogen atomic concentration in depth as it is shown in the Figure 3.

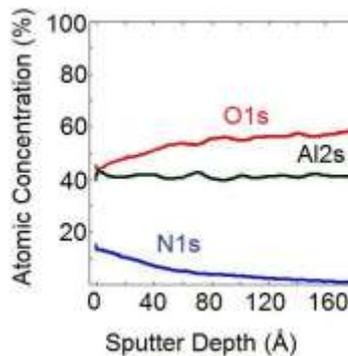


Figure 3: Concentration profile of elements

The morphology of the deposited layers was examined by atomic force microscopy (AFM). Lateral force microscopy (LFM) was used to define the AlN film morphology with nanometers precision. LFM measures frictional forces on a surface. By measuring the “twist” of the cantilever, rather than merely its deflection, one can qualitatively determine areas of higher and lower friction [7]. LFM allows imaging of heterogeneities in materials, thin films or monolayers at high spatial resolution. Furthermore, LFM is increasingly used to study the frictional properties of nanostructures and nanoparticles [8]. AFM-study shows that adhesive strength increases with the temperature growth. The temperature should be chosen according to some necessary prerequisites for material properties, constructive features and requirements to the structure of film, method of deposition etc.

The heterostructure of (0001)AlN/(0001)Al₂O₃ was produced. Structural and chemical analysis has been effected using an scanning electron microscopy (SEM Quanta 200). This measurement shows the occurrence of AlN film on the Al₂O₃ substrate. Figures 4-6 show dependence of film structure on various temperatures of the substrate.

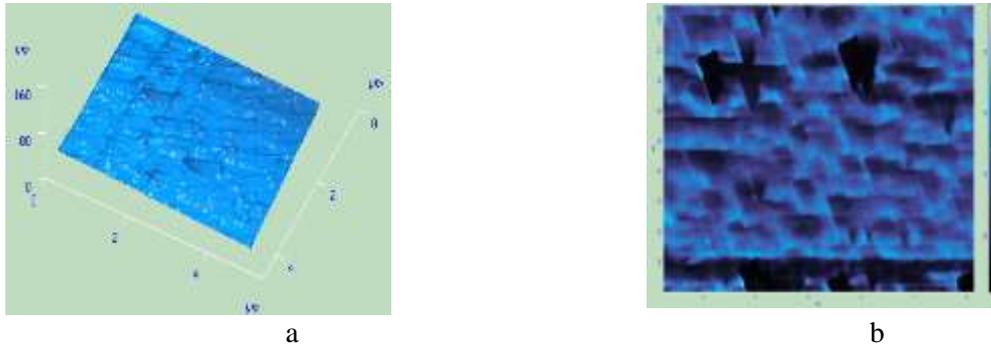


Figure 4: Morphology of AlN nanolayer on the sapphire substrate obtained at 1000 K.
(a – AFM tapping mode image, b – AFM lateral force image)
Scan size is 3×3 μm. Ten point height: 99.6117 nm.

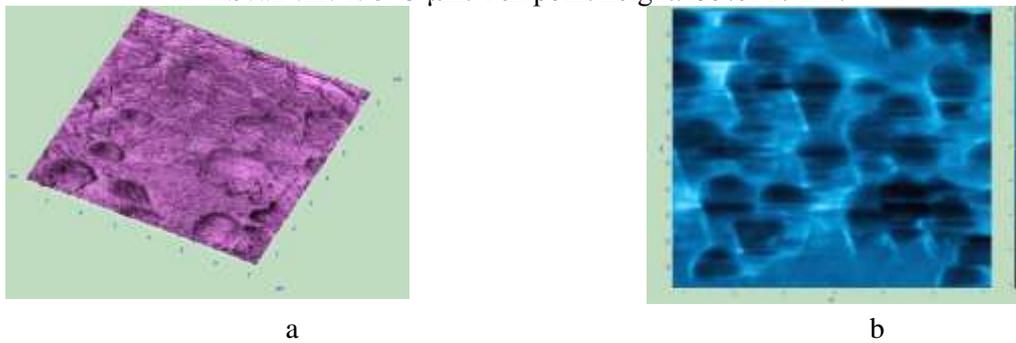


Figure 5: Morphology of AlN layer on the sapphire substrate obtained at 1300 K.
(a – AFM tapping mode image, b – AFM lateral force image)
Scan size is 7.5×7.5 μm. Ten point height: 102.894 nm.

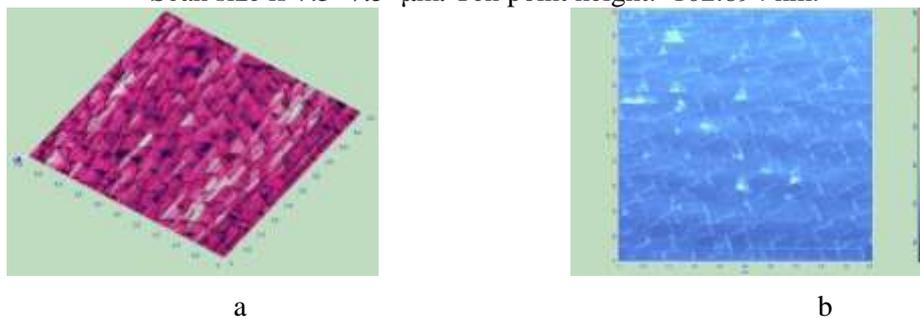


Figure 6: Morphology of AlN layer on the sapphire substrate obtained at 1500 K.
(a – AFM tapping mode image, b – AFM lateral force image)
Scan size is 5×5 μm. Ten point height: 12.4285 nm.

3. CONCLUSION

The result of this study is an experimental method for better formation of AlN thin films. We have used sapphire substrates for deposition since Al₂O₃ has electro-physical, mechanical, and thermal properties which are suitable for extreme conditions devices. Dry etching and nitridization of sapphire substrate were carried out before the sputtering of Al target. These technological features al-

lowed to reconstruct the surface and to create a thin intermediate layer from sapphire to aluminum nitride. The presence of the nitridization sapphire layer provides a good condition for consequent growth of AlN epilayers in the (0001) plane. The morphology and structural investigation were executed on each step of the film formation. Effectiveness of dry etching is proved by changing of morphology roughness of the substrates Al₂O₃. X-ray fluorescence analysis reveals the successful nitridization of the substrate near surface area. AFM measurements shows that the higher the temperature of Al₂O₃ substrate the better the morphology perfection of the AlN thin film.

ACKNOWLEDGEMENT

Research described in the paper was financially supported by the European Centre of Excellence CEITEC CZ.1.05/1.1.00/02.0068, by project Sensor, Information and Communication Systems SIX CZ.1.05/2.1.00/03.0072, as well as by grant GAČR 102/11/0995 "Electron transport, Noise and Diagnostics of Schottky and Autoemission Cathodes".

REFERENCES

- [1] Engelmark, F.: AlN and High- Thin Films for IC and Electroacoustic Applications, Uppsala, 2002, ISBN 91-554-5421-6
- [2] Axelbaum, R., Lottes, C., Huertas, J., Rosen, L.: Gas-Phase Combustion Synthesis Of Aluminum Nitride Powder. In: Twenty-Sixth Symposium (International) On Combustion/The Combustion Institute, 1996, p. 1891–1897
- [3] Guseinov, M., Kurbanov, M., Safaraliev, G., Bilalov, B.: Magnetron sputter deposition of (SiC)_{1-x}(AlN)_x solid solution films: Techn. Phys. Letts, 31, 2005, p. 138-139, ISSN 1063-7850
- [4] Dallaeva D., Bilalov, B., Gitikchiev, M., Kardashova G., Safaraliev G., Tománek P., Škarvada, P., Smith, S.: Structural properties of Al₂O₃/AlN thin film prepared by magnetron sputtering of Al in HF-activated nitrogen plasma: Thin Solid Films, 526, 2012, p.92-96, ISSN 0040-6090
- [5] Pearton, S., Abernathy, C., Rent, F., Lothian, J., Wiskt P., Katz, A., Constantiner, C.: Dry etching of thin-film InN, AlN and GaN: Semicond. Sci. Technol. 8, 1993, p. 310-312, ISSN 1361-6641
- [6] Soloviev, A., Sochugov, N., Oskomov, K., Kovsharov, N.: Film Thickness Distribution in Magnetron Sputtering System with the Round Cathode. p. 491-493, ISSN 0021-3411
- [7] <http://www.nanoscience.com/education/afm.html>
- [8] Munz, M.: Force calibration in lateral force microscopy – a review of the experimental methods: J. Phys. D: Appl. Phys., 43 (6), 2010, p. 063001, ISSN 1361-6463