

Substrate Preparation for Manufacturing of Aluminum Nitride Layers

D.Dallaeva¹, P. Tománek¹

¹ Department of Physics, Faculty of Electrical Engineering and Communication, BUT Brno, Technická 8, Brno
E-mail : xdalla02@stud.feec.vutbr.cz, tomanek@feec.vutbr.cz

Abstract:

Aluminum nitrides layers prepared on sapphire substrates are examined. The substrate surface was treated by dry plasma etching. The morphology of aluminum nitride thin films was studied by atomic force microscopy. Lateral force atomic force microscopy was used to study the morphology heterogeneity. The dependence of films morphology on the formation conditions has been defined. The objective of the study contributes to the improvement of technological process of dry etching and film deposition.

INTRODUCTION

Study and development of the perspective materials are relevant from technological point of view and are very important from economical one. 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], [3]. Structure and film texture of AlN has attracted much attention due to its unique properties and wide range of application of this material. Obviously, nitride semiconductors show properties that can not 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 manufacturing process is of a remarkable necessity in the field of optoelectronics. The problem which one meets in AlN thin layers manufacturing is the absence of suitable epitaxial substrates of the identical material. Hence the other materials such as sapphire (Al₂O₃) and silicon carbide (SiC) are used for the growth of AlN [4], [5].

There is a number of appropriate methods for thin film growth like sublimation, sputtering, organometallic vapor phase epitaxy, plasma-enhanced, molecular beam epitaxy, etc [6]. Magnetron sputtering is widely spread method of the thin film deposition. Uniform coating of this type is 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.) [7]. The technological task of the research is to find a source material for layer deposition from the target. The deposition begins when the discharge in material occurs. The collision of gas ions with target makes extractions of near-surface atoms, molecules and clusters from the source material, and these particles form the thin film on the substrate.

By-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.

Study of dry etching process is important to reproducibility improving of the products characteristics which depend on surface cleaning, accuracy of topography, variation of treating parameters, etching and interfaces.

Plasma etching replaces the conventional etching and processing methods in vacuum processing technologies because of suitability [8] and it is one of the key operations of semiconductor structures fabrication [9].

Despite the damage associated with dry etching [10], it can provide highly anisotropic profiles with good reproducibility and uniformity [11].

In case of chemically stable and hard [12] materials, such as Al₂O₃, it is better to use physical etching. Al₂O₃ is good as masking material for optoelectronic structures manufacturing [13]. It was noted that Al₂O₃ can be used as etch stop material for vapor etching [14]. Dry etching is a key process for surface cleaning [15]. Simple sputtering is non-selective elimination of surface atoms due to plasma-induced non-reactive gas ions which vertically impinge on the substrate surface without any method to control the etch print. The etching of Al₂O₃ was described by several authors [16], [17], [18]. This material is also of interest because of its unique properties, such as mechanical hardness and chemical stability [19].

EXPERIMENTAL RESULTS

A standard vacuum deposition system was used with two ring-type magnetrons and an ion source. There is a substrate heater, a reactor of HF-activated nitrogen plasma, and gas flow regulators there. The main parts of the deposition process are crystalline substrate which has to satisfy requirements of deposition of epitaxial growth on it, and a source of the desired product. 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

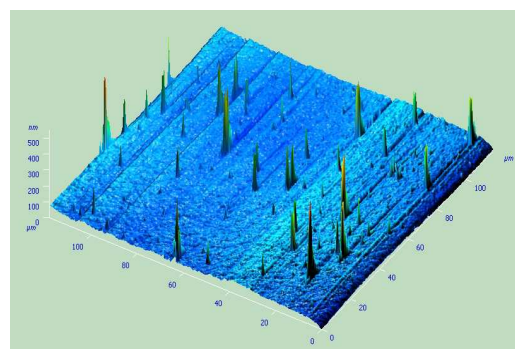
supersaturation and gas-dynamic behavior of the reactor. The ionizing efficiency can be improved by using of magnetic field, so ions are generated relatively far from the target and the probability of energy loss is high in ordinary planar diode systems. The magnetic field lines cross the lines of electric 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 then 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.

In spite of all advantages of this method, there is 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. The 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. So, the high-purity aluminum target was used in this study.

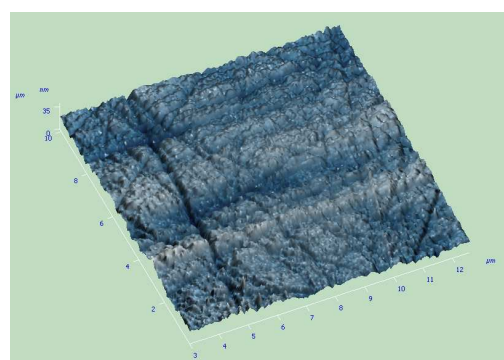
Substrate preparation includes dry etching and nitration by nitrogen implantation into the sapphire substrate (0001 orientation), with subsequent high-temperature annealing at 1400-1600 K in a nitrogen atmosphere. The average Resistance-type heater was used for preheating of the substrate during the 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. 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 259.874 nm and 49.9895 nm respectively (Figs. 1 and 2).

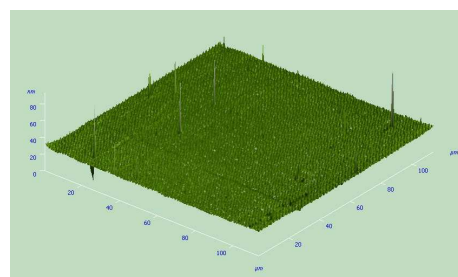


Scan area $120\ \mu\text{m} \times 120\ \mu\text{m}$.
Ten point height 259.874 nm.
(a)

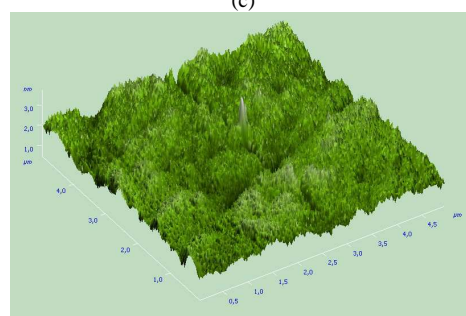


Scan area $10\ \mu\text{m} \times 10\ \mu\text{m}$.
Ten point height 27.3014 nm.
(b)

Fig. 1: Surface of Al_2O_3 before dry etching.



Scan area $120\ \mu\text{m} \times 120\ \mu\text{m}$.
Ten point height 49.9895 nm.
(c)



Scan area $120\ \mu\text{m} \times 120\ \mu\text{m}$.
Ten point height 4.02808 nm.
(d)

Fig. 2: Surface of Al_2O_3 after dry etching.

One of the most important parameters of the deposition process is a temperature of the deposited films is. 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 morphology of the deposited layers was examined by atomic force microscopy (AFM). The statistical distribution of heights is shown in figure 3. Lateral force microscopy (LFM) was used to define the AlN film morphology with nanometers precision (Fig. 4). 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 [20].

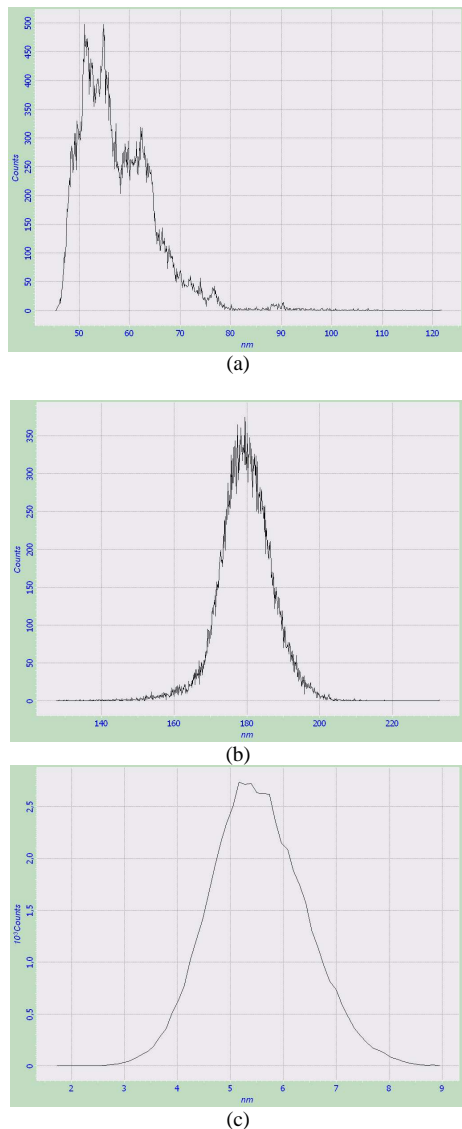


Fig. 3: Height histogram for the samples morphology obtained (a) at 1000K, (b) at 1300 K and (c) at 1500 K.

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.

LFM allows the 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 [21].

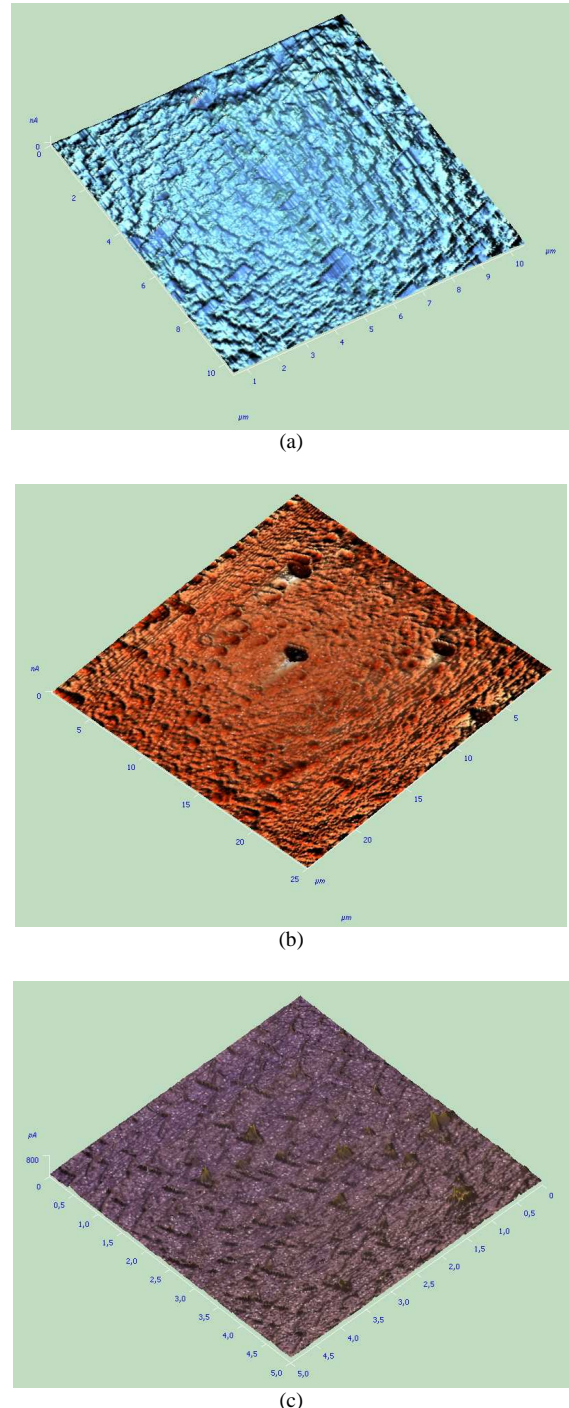


Fig. 4: Morphology of AlN samples obtained (a) at 1000K, (b) at 1300 K and (c) at 1500 K.

The heterostructure of (0001)AlN / (0001)Al₂O₃ was produced and its Scanning electron microscopy (SEM Quanta 200 from FEI) image is in Figure 5.

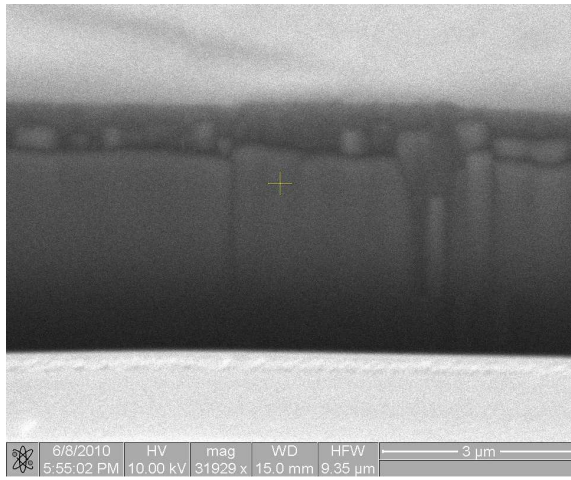


Fig. 5: SEM image of the aluminum nitride layer in cross-section.

This measurement shows the occurrence of AlN film on the Al_2O_3 substrate. There are crystalline columnar grains of AlN in the image of cross-section analysis image. They have flat tops and not sharp shown faceting of the surface.

CONCLUSION

The result of this study is an experimental method for better fabrication of AlN thin films. We have used sapphire substrates for deposition since Al_2O_3 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 allowed 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 Al_2O_3 .

ACKNOWLEDGEMENT

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

REFERENCES

[1] F. Engelmark, AlN and High-Thin Films for IC and Electroacoustic Applications. Uppsala: Acta Universitatis Upsaliensis, 2002.

- [2] R. Axelbaum, C. Lottes, J. Huertas, and L. Rosen, "Gas-Phase Combustion Synthesis Of Aluminum Nitride Powder". In Twenty-Sixth Symposium (International) On Combustion, (Pittsburgh), pp. 1891–1897, The Combustion Institute, 1996.
- [3] D. Chen, D. Xu, J. Wang, B. Zhao, and Y. Zhang, "Dry etching of AlN films using the plasma generated by fluoride". Vacuum, vol. 83 (2), pp. 282–285, Sep.2008.
- [4] M. K. Guseinov, M. K. Kurbanov, G. K. Safaraliev and B. A. Bilalov, "Magnetron sputter deposition of $(\text{SiC})_{1-x}(\text{AlN})_x$ solid solution films". Techn. Phys. Letts, vol. 31, pp. 138-139, Feb. 2005.
- [5] D. S. Dallaeva, B. A. Bilalov, M. A. Gitikchiev, G. D. Kardashova, G. K. Safaraliev G., P. Tománek, P. Škarvada, and S. Smith, "Structural properties of $\text{Al}_2\text{O}_3/\text{AlN}$ thin film prepared by magnetron sputtering of Al in HF-activated nitrogen plasma". Thin Solid Films, vol. 526, pp.92-96, Dec. 2012.
- [6] S. Pearton, C. Abernathy, F. Rent, J. Lothian, P. Wiskt, A. Katz and C. Constantiner, "Dry etching of thin-film InN, AlN and GaN". Semicond. Sci. Technol., vol. 8, pp. 310-312, Feb. 1993.
- [7] A. V. Soloviev, N. S. Sochugov, K. V. Oskomov and N. F. Kovsharov, "Film Thickness Distribution in Magnetron Sputtering System with the Round Cathode". Russ. Phys. Jour., pp. 491-493, Aug. 2006.
- [8] H. Jansen, H. Gardeniers, M. de Boer, M. Elwenspoek and J. Fluitman, "A survey on the reactive ion etching of silicon in microtechnology". J. Micromech. Microeng., vol. 6, pp.14-28, Mar. 1996.
- [9] K. Suzuki and N. Itabashi, "Future prospects for dry etching". Pure Appl. Chem., vol. 68 (5), pp. 1011-1015, 1996.
- [10] C. D. W. Wilkinson and M. Rahman, "Dry etching and sputtering," Phil. Trans. R. Soc. Lond. A, vol. 362, pp. 125–138, Jan.2004.
- [11] V. Bhagwat, J. P. Langer, I. Bhat, P. S. Dutta, Tamer Refaat and M. Nurul Abedin, "A Comparison of dry plasma and wet chemical etching of GaSb photodiodes". J. Electrochem. Soc., 151 (5) pp. A728-A730, Apr.2004.
- [12] G. Yong Luo, Yan Du and Veena Misra, "Large area nanorings fabricated using an atomic layer deposition Al_2O_3 spacer for magnetic random access memory application. Nanotechnology, vol. 19, pp. 265301, Apr. 2008.
- [13] W. T. Li, D. A. P. Bulla, J. Love, B. Luther-Davies, C. Charles and R. Boswell, "Deep dry-

etch of silica in a helicon plasma etcher for optical waveguide fabrication". J. Vac. Sci. Technol. A, vol. 23(1), pp. 146-150, Jan/Feb 2005.

- [14] Th. Bakke, J. Schmidt, M. Friedrichs, and B. Volker "Stop materials for release by vapor HF etching". In 16th MicroMechanics Europe Workshop, (Göteborg), pp. 103-106, Fraunhofer IPMS, 2005.
- [15] M. A. Lieberman and A. J. Lichtenberg, Principles of Plasma Discharges and Materials Processing, 2nd ed., Wiley Interscience, Hoboken, NJ: Wiley, 2005.
- [16] Dong-Pyo Kim, Gwan-Ha Kim, Jong-Chang Woo, Hwan-Jun Kim and Chang-II Kim, "Dry etching of high-k dielectric thin films in HBr/Ar plasma". J. Korean Phys. Soc, vol. 54 (2), No. 2, pp. 934-938, Feb. 2009.
- [17] Xeng Yang, Jong-Chang Woo, Doo-Seung Um, and Chang-II Kim, "Dry etching of Al₂O₃ thin films in O₂/BCl₃/Ar inductively coupled plasma". Trans. Electr. Electron. Mater., vol. 11 (5), pp. 202-205, Oct. 2010.
- [18] H. Y. Jung, Y. R. Park, H. J. Lee, N.-E. Lee, C. Y. Jeong and Jinho Ahn "Selective dry etching of attenuated phase-shift mask materials for extreme ultraviolet lithography using inductively coupled plasmas". J. Vac. Sci. Technol., vol. 27 (6), Dec 2009.
- [19] B. Braunschweig, S. Eissner and W. Daum, "Molecular Structure of a Mineral/Water Interface: Effects of Surface NanoRoughness of α -Al₂O₃ (0001)". J. Phys. Chem. C, vol. 112 (6), pp. 1751-1754, Jan. 2008.
- [20] Atomic Force Microscopy. Nanoscience Instruments, Inc. [cit. 2013-09-05]. Available from WWW: <<http://www.nanoscience.com/education/afm.html>>.
- [21] M. Munz, "Force calibration in lateral force microscopy – a review of the experimental methods". J. Phys. D: Appl. Phys., vol. 43 (6), pp. 063001, Jan. 2010.