

CHARACTERIZATION OF ALN THIN FILMS DEPOSITED ON THERMALLY PROCESSED SILICON SUBSTRATES USING PE-ALD.

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Abstract: The aim of this work is to study topography and chemical composition of AlN thin films deposited on Si substrates previously exposed to various time of thermal processing using plasma-enhanced atomic layer deposition technique. The samples were heated up to 500 °C for the period of 2 and 4 hours. Chemical composition of wafers and the films obtained are provided by X-ray photoelectron spectroscopy (XPS). Surface topography was investigated using atomic force microscopy (AFM).

Keywords: aluminum nitride, atomic layer deposition, atomic force microscopy, Si single crystal wafers, topography, x-ray photoelectron spectroscopy.

1 INTRODUCTION

Thanks to the large band gap, high breakdown field and high electron saturation velocity, nitrides of third group elements have attracted a lot of attention in the application of various devices such as laser diodes, light emitting diodes, power devices and high electron mobility transistors. Amidst III-nitride compounds, aluminum nitride (AlN) is an attractive material due to its outstanding thermal conductivity, high electric resistance, good piezoelectric properties and ability to maintain those properties under harsh environments. [1] Despite the fact that such techniques as chemical vapor deposition and its variations are broadly used in semiconductor technology, including growth of AlN, they are not compatible with flexible substrates since CVD usually requires high temperatures. Which is why in this scenario ALD methods looks more favorable as it allows deposition at low temperatures (~300 °C). Another huge advantage of ALD method is the possibility of precise control over the film growth (at the Angstrom level). [2]

Silicon wafers play a huge role in nowadays electronics such as solar cells, micro- and nano-electromechanical systems (MEMS/NEMS), due to its great optical and electrical properties. They also are widely used as substrates for various techniques of thin films preparation (ALD, CVD, magnetron sputtering, spin coating, etc). Despite the fact that it is a very common and widespread material there is still a problem of insufficient experimental data concerning its behavior in harsh environments, for example - continuous temperature stress. Furthermore, sometime the preliminary processing of the substrates by heating or annealing is required. Which is why, it is still relevant to conduct studies on silicon wafers exposed to various outer influences. The main goal of this work is to provide analytical data on changes in topography and chemical composition of Si wafers under different periods of stress heating and following etching procedure. This data might prove to be helpful in choosing the right processing procedure of substrate during heterostructures and thin films fabrication processes [3].

The studies of temperatures stresses recently have attracted a lot of attention owing to their large isothermal entropy achieved in commercially nanomaterials. The effects of temperature process influence non-irreversible atomic processes, structural phase transitions, chemical and physical properties of materials [4].

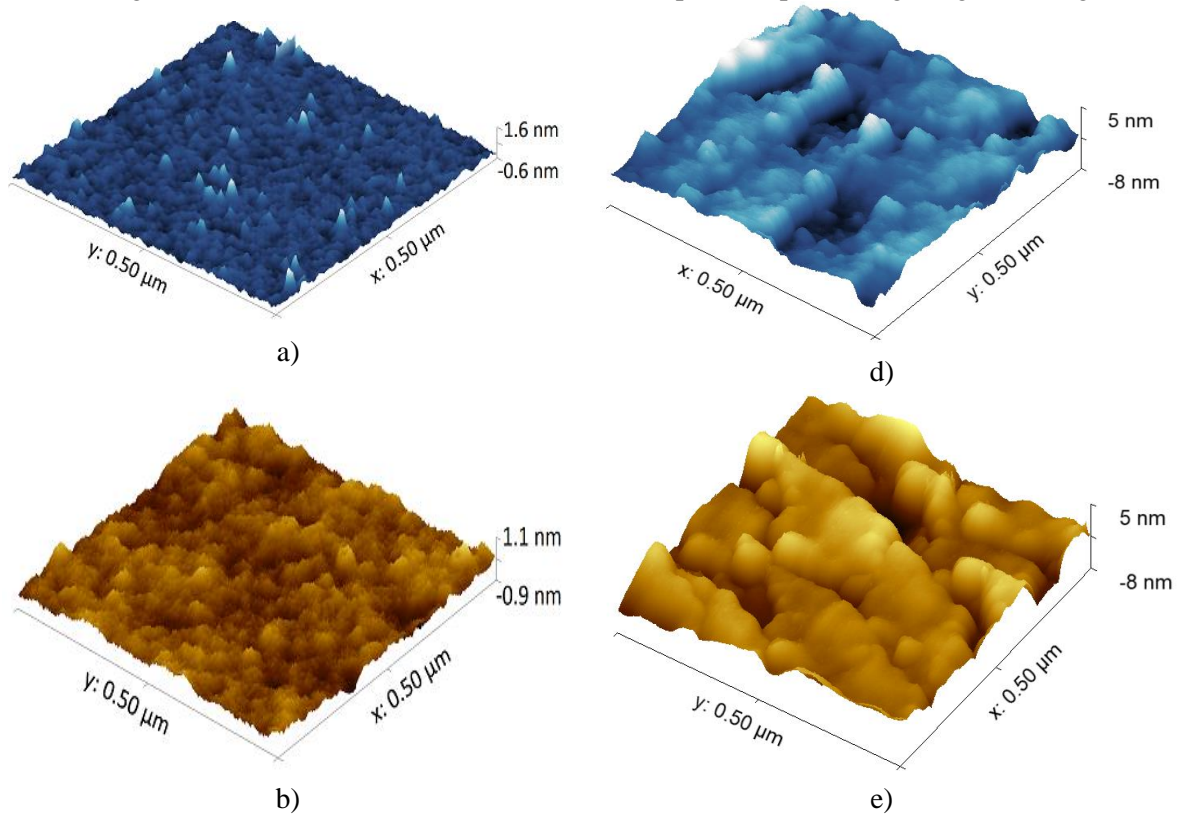
There are several researches in which authors attempted to study behavior of Si wafers under influence of high temperatures. For example, Suzuki T. has reported in his study how high-temperature annealing affects generation of oxidation-induced stacking faults (OSF) in a silicon wafer. D. Gräf et al. have reported that the temperature stress in oxygen atmosphere considerably improves the electric properties of Si wafers [5].

In our paper we focus on structural modifications of Si wafers by thermal annealing and argon ion modification with conditions which have never been tried before. We investigate the changes of chemical composition, crystallographic properties and stereometric analysis of modified surface of Si crystal before and after temperature stressing. For this purposes, X-ray Photoemission spectroscopy (XPS) and stereometric analysis of surface measured by Atomic force microscopy (AFM) were carried out [6].

2 EXPERIMENTAL DATA

Silicon wafers 4 inch, thickness $525 \pm 25 \mu\text{m}$ (100), 1-side polished, p-type (Borron) of MicroChemicals production were used. Different wafers were heated up to $500 \text{ }^\circ\text{C}$ and annealed for two different time periods (i.e. 2 hours and 4 hours). Heating of the samples from room temperature to $500 \text{ }^\circ\text{C}$ was carried out uniformly during 30 minutes in air. The measurements of surface composition by XPS were carried out after cooling of the samples to room temperature. XPS spectra were done in vacuum at following parameters: 15 mA emission current, slot collimation mode, hybrid lens mode and 20eV resolution of pass energy. AlN thin films were deposited using trimethylaluminum (TMA) and N₂/H₂ plasma recipe under, the temperature of the deposition was $300 \text{ }^\circ\text{C}$. \in total 1000cycles were performed which translates into thickness of $\sim 70\text{nm}$ (1 cycle $\approx 0,7\text{\AA}$).

Atomic force microscopy characterizations were carried out for the quantitative analysis of specific microstructural characteristics of samples using tapping AFM mode. All AFM measurements were done at room temperature and normal humidity. Scan rate was 0.580 Hz and probe RTESPA-150 was used. All 512×512 pixel images were obtained by scanning square areas of $0.5 \mu\text{m} \times 0.5 \mu\text{m}$. AFM images for Si substrates with different time of temperature processing are given in fig. 1.



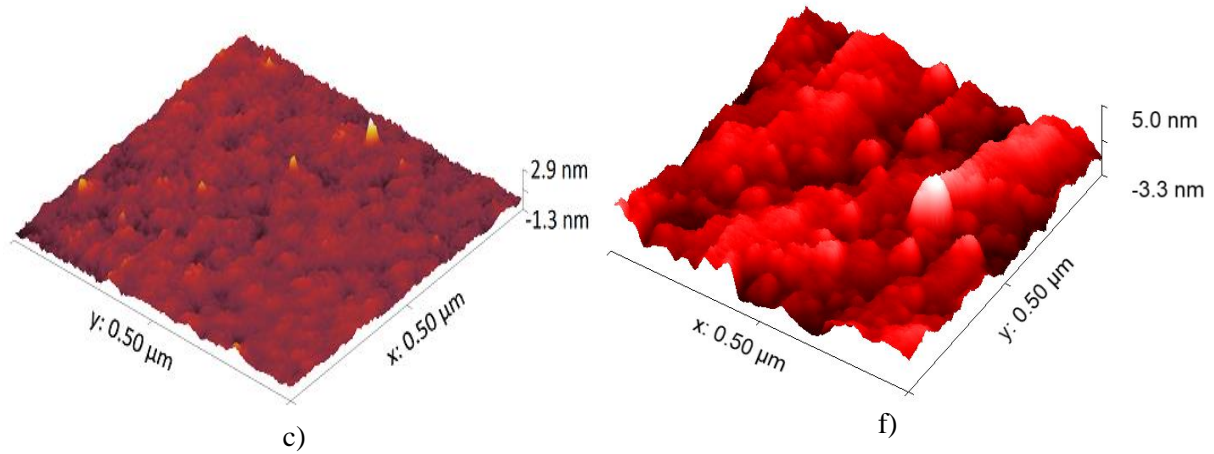
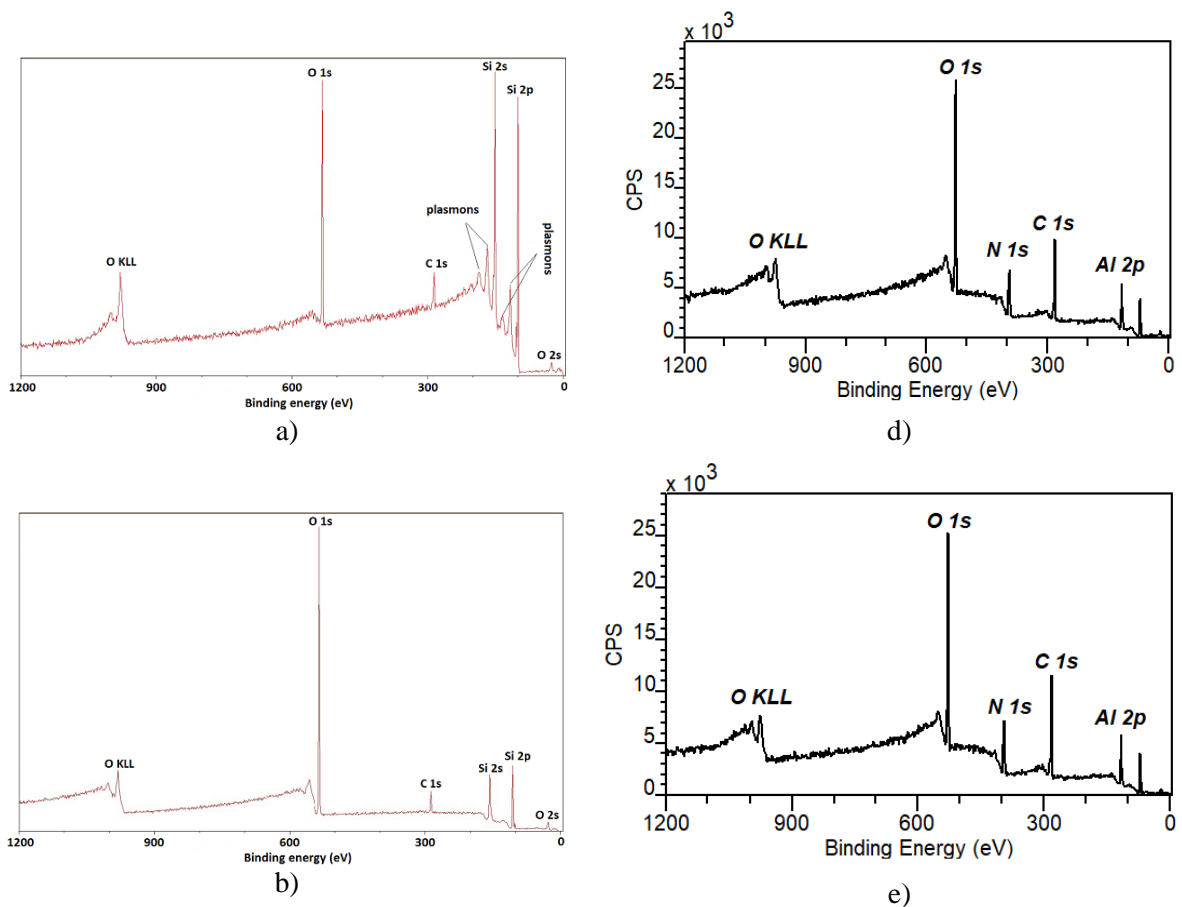


Figure 1: AFM images for: a),b),c) – Si substrates with no proc, 2 hours and 4 hours correspondingly; d),e),f) - AlN film deposited on Si substrates with no proc, 2 hours and 4 hours correspondingly

The average roughness (Ra) for films deposited on Si substrates with 0, 2, 4 hours of thermal processing is 508.6, 314.9, 231.5 pm correspondingly. This suggests that the film takes a more uniform structure if deposited on a substrate exposed to a longer annealing process.

To provide a comprehensive analysis of chemical composition of the wafers before and after annealing and the AlN films obtained on them we employed X-ray photoelectron spectroscopy (XPS) technique. The received spectra are presented in Fig 2.



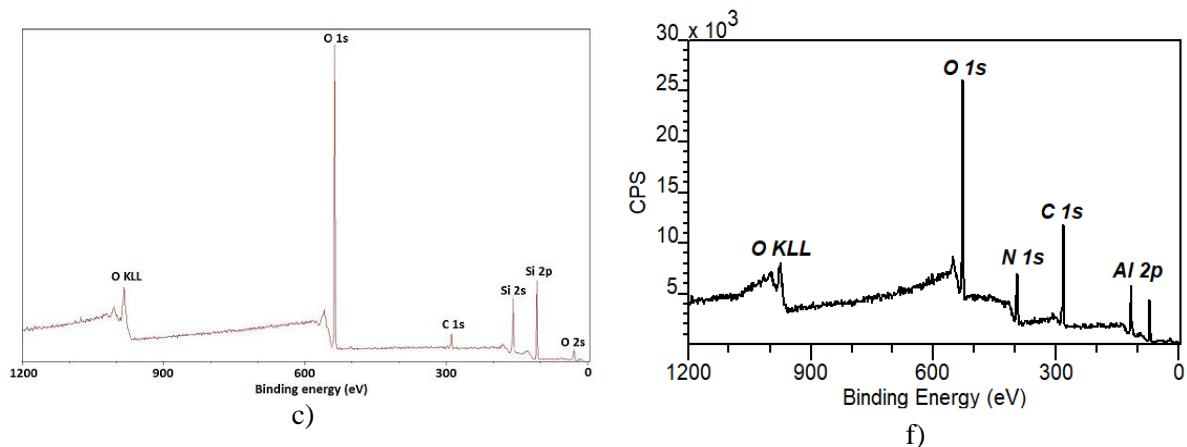


Figure 2: XPS spectra for: a),b),c) – Si substrates with no proc, 2 hours and 4 hours correspondingly; d),e),f) - AlN film deposited on Si substrates with no proc, 2 hours and 4 hours correspondingly

In order to confirm the presence of Al-N structure we also provide here high-resolution elemental peaks of N 1s and Al 2p.

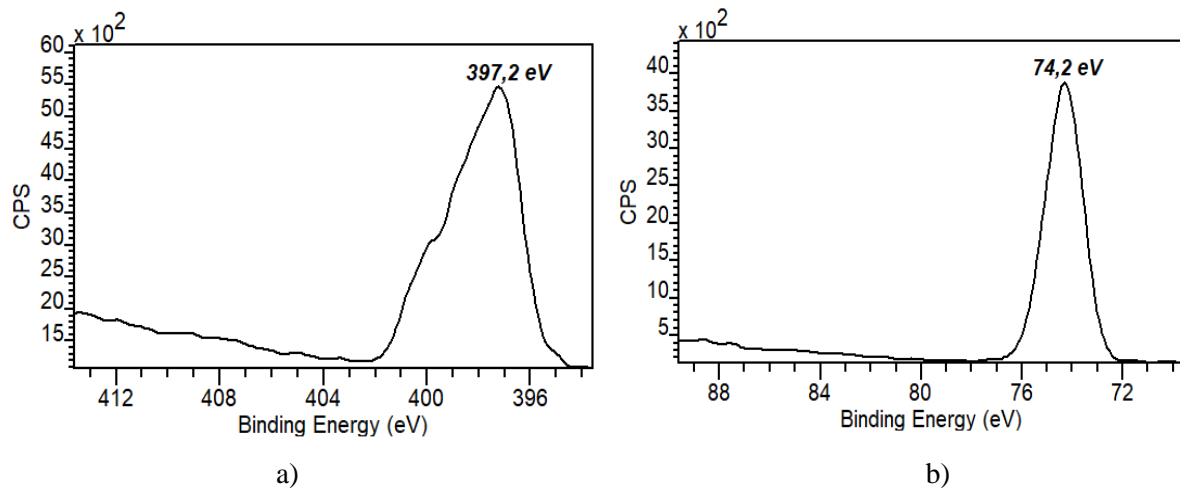


Figure 3: High-resolution elemental spectra for a) N 1s; b) Al 2p

As can be seen from from fig. 3. binding energies of N 1s and Al 2p amount to 397,2 and 74,2 eV correspondingly. These are exact energies at which formation of Al-N bond occurs [7].

3 CONCLUSION

In this study we used AFM and XPS methods to investigate morphological and compositional changes that take place in Si wafer during thermal processing of various durations. Furthermore, aforementioned wafers have been used as substrates for deposition of AlN thin films using ALD technique. The results obtained suggest that continuous annealing process of a wafers leads to a more uniform topography of the film. After analyzing the XPS data, it became clear that on the surface of the silicon wafers there were small impregnations of carbon and an oxide film that thickens during the annealing process. The same analysis of the AlN films indicates the presence of well-defined N1s and Al 2p peaks of binding energy at which formation of Al-N occurs.

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REFERENCES

- [1] H. Kim, N. Do Kim, S.C. An, H.J. Yoon, B.J. Choi, Improved interfacial properties of thermal atomic layer deposited AlN on GaN, *Vacuum*, 159 (2019) 379–381. doi:10.1016/j.vacuum.2018.10.067.
- [2] Abdulagatov, A. I., Ramazanov, S. M., Dallaev, R. S., Murliev, E. K., Palchaev, D. K., Rabadanov, M. K., & Abdulagatov, I. M. Atomic Layer Deposition of Aluminum Nitride Using Tris(diethylamido)aluminum and Hydrazine or Ammonia. *Russian Microelectronics*, 47(2), (2018) 118–130. <http://doi.org/10.1134/S1063739718020026>
- [3] Dallaev R., G.T. Córdova, D. Sobola, S. Stach, A. Méndez-Albores, L. Grmela, Ș. Țălu, Stereometric Analysis of Effects of Heat Stressing on Micromorphology of Si Single Crystals, *Silicon*. (2019). doi:10.1007/s12633-019-0085-4.
- [4] Sun, X., Gao, K., Pang, X., Sun, Q., & Li, J. Thermodynamic energy variation diagram to speculate preferred growth orientation of magnetron sputtered PbSe thin films on monocrystalline silicon substrates. *Applied Surface Science*, 452, (2018) 1–10. <https://doi.org/10.1016/j.apsusc.2018.04.200>
- [5] D. Gräf, U. Lambert, M. Brohl, A. Ehlert, R. Wahlich and P. Wagner, Improvement of Czochralski Silicon Wafers By High- Temperature Annealing, *J. Electrochem. Soc.*, 142(9) (1995) 3189-3192. DOI: 10.1149/1.2048711
- [6] Gotoh, K., Cui, M., Takahashi, I., Kurokawa, Y., & Usami, N. Development of spin-coated copper iodide on silicon for use in hole-selective contacts. *Energy Procedia*, 124, (2017) 598–603.
- [7] L. Ravi, B. Krishnan, Epitaxial growth of AlN microwalls on wet etched GaN template by MOCVD, *Superlattices Microstruct.* 123 (2018) 144–153. doi:10.1016/j.spmi.2018.07.011.