

Influence of Underlayer on Crystallography and Roughness of Aluminum Nitride Thin Film Reactively Sputtered by Ion-Beam Kaufman Source

GABLECH Imrich^{1,2*}, SVATOŠ Vojtěch^{1,2}, PRÁŠEK Jan^{1,2}, HUBÁLEK Jaromír^{1,2}

 ¹ Central European Institute of Technology, Brno University of Technology, Brno, Czech Republic, EU
² Brno University of Technology, Faculty of Electrical Engineering and Communication, Department of Microelectronics, Brno, Czech Republic, EU

*imrich.gablech@ceitec.vutbr.cz

Abstract

This paper deals with deposition of aluminum nitride layer on different underlayers namely silicon (100), amorphous thermal silicon dioxide, and titanium (001). The aluminum nitride layer was deposited using 3-grid radio frequency inductive coupled plasma (RFICP) Kaufman ion-beam source which provides slow deposition rate with low energy of plasma ions. This possibility leads to high homogeneity and very smooth surface. Titanium layer was deposited by argon plasma at low energy of 200 eV to attain the highly oriented c-axis layer. The aluminum nitride was sputtered on substrate which was heated to 350 °C. The nitrogen plasma only at energy of 500 eV was used. It was observed the aluminum nitride deposited on thermal silicon dioxide has the highest root mean square of roughness (RRMS) = 1.49 nm and the lowest intensity of X-ray diffraction in Bragg-Brentano focusing geometry (XRD-BB). The aluminum nitride deposited on silicon (100) shows higher intensity of XRD-BB and the lowest R_{RMS} = 0.48 nm. Although the R_{RMS} = 0.66 nm of aluminum nitride thin film deposited on titanium (001) underlayer was obtained, the highest intensity of XRD-BB was observed. Azimuthally averaged intensity of pole figures obtained using parallel beam setup represents the information about misorientation of individual crystallites. These analyses were performed for aluminum nitride layers deposited on titanium (001) film and silicon (100) wafer. Misorientation determined from full width at half maximum (FWHM) of the pole figures was of about 0.5° lower for aluminum nitride thin film deposited on titanium underlayer than one deposited directly on silicon substrate without silicon dioxide.

Keywords: Aluminum Nitride, Titanium, c-axis orientation, Kaufman ion-beam source, reactive sputtering, AFM, XRD

1. INTRODUCTION

Aluminum nitride (AIN) has attracted considerable huge attention as a piezoelectric layer in numerous MEMS devices such as actuators, sensors, and transducers [1,2]. The significant advantage of AIN film is its compatibility with CMOS technology [3]. Because the AIN is not ferroelectric material it does not require poling unlike the lead zirconate titanate (PZT) as another CMOS technology compatible piezoelectric material [4]. Aluminum nitride shows other extraordinary properties such as dielectric properties connected to wide band-gap, mechanical stiffness, high elasticity, high thermal conductivity, and low thermal expansion coefficient [5].

There have been published a number of papers dealing with the optimizing of its piezoelectric behavior. The characterization of piezoelectric material is important to be performed under the conditions of selected application [6]. For example the piezoelectric coefficient of aluminum nitride thin film is dependent on its surface roughness and grain size. Furthermore, the crucial parameter affecting AIN thin film properties and growth is a suitable underlayer [7]. In most microdevices, there are many materials which are suitable to be used as a bottom electrode for subsequent deposition of AIN film due to their hexagonal orientation for growth of wurtzite, e.g. AI (111), Pt (001), Ti (001), and Mo (110). The titanium electrode is also ideal because the lattice mismatch with aluminum nitride is comparable [5].



The conventional methods of AIN layer deposition are sputtering (e.g. radio frequency magnetron sputtering or ion-beam assisted deposition), metalorganic chemical vapor deposition (MOCVD), plasma enhanced chemical vapor deposition (PECVD), molecular beam epitaxy (MBE), and atomic layer deposition (ALD) [2,8].

In this paper, the aluminum nitride layer is deposited by ion-beam reactive sputtering which concludes to smoother layer than other conventional sputtering techniques. Three various possible underlayers for AIN thin film are investigated in terms of crystallographic orientation, average crystallite size, misorientation of individual crystallites, and roughness of layer surface.

2. EXPERIMENTAL

2.1. Deposition Processes

All depositions of AlN were performed on pieces of silicon wafer (20 x 20 mm) substrates (P-type with the (100) crystallographic plane parallel to surface and resistivity 6-12 Ω .cm) with different types of underlayer. AlN deposition was performed on silicon substrate with native oxide only, silicon substrate with 1 µm thick thermal silicon dioxide , and on titanium layer with (001) crystallographic plane parallel to the surface sputtered on silicon substrate (100) covered by 1 µm thick thermal silicon dioxide as mentioned below.

Before the sputtering deposition process, the silicon substrate with native oxide only was dipped in standard buffered oxide etchant (49% HF + 40% NH₄F in the 6:1 volume ratio) for 1 minute to remove native oxide. Substrates with thermal silicon dioxide were cleaned in the standard piranha solution (96% H_2SO_4 + 30% H_2O_2 in the 3:1 volume ratio) for 3 minutes, rinsed in deionized water and dried with compressed nitrogen.

The RFICP Kaufman ion-beam source with 3-grid focused ion optics was used for deposition. A titanium target with a purity of 99.995% and argon gas with a purity of 99.9996% were used for titanium deposition. Aluminum nitride was reactively sputtered by pure nitrogen plasma without presence of argon. The purity of nitrogen was 99.9999% and the purity of aluminum target was 99.995%. The sputtering chamber was pumped down to a pressure of $5 \cdot 10^{-9}$ mbar before start of deposition process.

2.2. Diagnostics methods

SmarLab (Rigaku) X-ray diffractometer (XRD) with a linear D/tex detector was employed to perform crystallography analysis in Bragg-Brentano focusing geometry. Pole figure analysis was performed with parallel beam setup. The surface topography and roughness (R_{RMS}) of deposited layers were determined using Atomic Force Microscopy (AFM) Dimension Icon (Bruker) in the ScanAsyst[®] mode.

3. RESULTS AND DISCUSSION

3.1. Titanium underlayer

Deposition of 100 nm thick titanium underlayer was performed on amorphous thermal silicon dioxide with very low energy of ions (200 eV), and low deposition rate of 0.04 Å/s. From X-ray diffractogram (Figure 1a) it is clear that only (001) preferential crystallographic orientation of the crystallites was obtained. Full width at half maximum (FWHM) of diffraction peak was 0.45°. Average crystallite size of 21 nm was calculated according to Scherrer equation. Smooth titanium surface topography of 0.55 R_{RMS} and its corresponding profile are depicted in Figure 1b and Figure 1c respectively.





Fig. 1 XRD and AFM diagnostics of titanium thin film deposited by argon plasma low energy ions (200 eV); a) X-ray diffractogram showing titanium (001) preferential crystallographic orientation; b) surface topography of Ti layer deposited on thermal silicon dioxide; c) surface profile corresponding to the surface topography measurement shown in b).

As the high deposition temperature (up to 400 °C) of subsequent films is required the deposited titanium layer was exposed to 2 hours annealing peak process in vacuum chamber pumped down to $5 \cdot 10^{-7}$ mbar with heating and cooling rate of 5 °C/min up to 400 °C. X-ray diffractogram of annealed thin film (see Figure 2) confirms that the peak of (001) crystallographic plane is without any changes.



Fig. 2 X-ray diffractogram of titanium (001) before and after 2 hours annealing up to 400 °C.

3.2. Reactive sputtering of aluminum nitride

Aluminum nitride was deposited on three different underlayers with different crystallography: Si (100) with face-centered cubic lattice, the amorphous silicon dioxide and titanium (001) with hexagonal close-packed structure with cell parameters very similar to aluminum nitride. To ensure the same deposition conditions, the



sputtering onto all three layers were done simultaneously in one process. Thickness of deposited AIN layer was 200 nm and deposition rate was 0.17 Å/s. During this process the substrate was heated up to 350 °C. Energy of bombarding nitrogen ions was set to be 500 eV. XRD and AFM analyses of this layer are shown in Figures 3a, 3b and 3c.



Fig. 3 XRD and AFM analyses of deposited AIN layers by pure nitrogen plasma with substrate heated to 350 °C; a) X-ray diffractograms of AIN thin film deposited on different underlayers showing only presence of (001) preferential crystallographic orientation; b) surface topography of AIN thin film layer deposited on Ti (001) underlayer and AIN thin film deposited directly on silicon wafer (100); c) surface profiles corresponding to the AFM surface topographies shown in b).

In Figure 3a, the highest diffraction peak of aluminum nitride layer (FWHM = 0.266° ; crystallite size = 32.8 nm) deposited over titanium underlayer is evident. This layer has smooth surface with $R_{RMS} = 0.66 \text{ nm}$ (see Figure 3b and 3c). The lowest peak (see Figure 3a) and the highest $R_{RMS} = 1.49 \text{ nm}$ was achieved for aluminum nitride layer (FWHM = 0.262° ; crystallite size = 33.3 nm) deposited on thermal amorphous silicon dioxide (see Figure 3b and 3c.). Aluminum nitride layer (FWHM = 0.252; crystallite size = 34.9 nm) deposited directly on polished silicon wafer (100) without native oxide has higher peak (see Figure 3a) than previously mentioned layer and the smoothest surface with $R_{RMS} = 0.48 \text{ nm}$ (see Figure 3b and 3c).

The pole figure orientation analysis of (001) crystallographic plane was performed for aluminum nitride on silicon and titanium underlayers to determine misorientation of individual crystallites. From the azimuthally averaged intensity of pole figure (see Figure 4), it is clear the misorientation of AIN crystallites on titanium underlayer is lower (FWHM = 4°) than on silicon (100) substrate without silicon dioxide (FWHM = 4.5°).





Fig 4. Azimuthally averaged intensity profiles extracted from pole figures for AIN (001) crystallographic plane deposited on Ti (001) underlayer and for AIN (001) crystallographic plane deposited directly on silicon wafer (100) without silicon dioxide.

CONCLUSION

In this paper, the influence of underlayer material on deposited aluminum nitride crystallography and roughness is described. In case of silicon dioxide underlayer, the lowest diffraction peak and the highest roughness of 1.49 nm was reached. Despite of deposition on the pure silicon wafer (100) the aluminum nitride thin film grows in c-axis direction. This layer has the lowest roughness of 0.48 nm and slightly higher intensity of diffraction peak than the layer deposited on silicon dioxide. Aluminum nitride layer deposited over titanium underlayer has the highest diffraction peak and negligibly higher roughness of 0.66 nm. On the other side, aluminum nitride layer deposited on titanium layer has according to the pole figure 0.5° smaller misorientation (FWHM = 4°) compared to the aluminum nitride layer deposited directly on pure silicon wafer (FWHM = 4.5°). Titanium underlayer proves to be excellent as bottom electrode due to its lattice parameters similar to aluminum nitride lattice which is obvious from azimuthally averaged intensity obtained from pole figure of (001) plane diffraction.

ACKNOWLEDGEMENTS

Research described in this paper was financed by the National Sustainability Program under grant LO1401. The work was also carried out with the support of the CEITEC Nano Core Facility under the CEITEC – open access project, ID number LM2011020, funded by the Ministry of Education, Youth and Sports of the Czech Republic under the activity "Projects of major infrastructures for research, development and innovations". For the research, infrastructure of the SIX Center was used.

REFERENCES

- POLLA, D. L. a FRANCIS, L. F. Processing and characterization of piezoelectric materials and integration into microelectromechanical systems. *Annual Review of Materials Science*, 1998, vol. 28, pp. 563-597. ISSN 0084-6600.
- [2] SARAVANAN, S. et al. Surface Micromachining Process for the Integration of AIN Piezoelectric Microstructures. SAFE 2004, 7th Annual Workshop on Semiconductor Advances for Future Electronics. 2004. 9789073461437. Veldhoven, the Netherlands public.
- [3] ABABNEH, A. et al. c-axis orientation and piezoelectric coefficients of AIN thin films sputter-deposited on titanium bottom electrodes. *Applied Surface Science*, 2012, vol. 259, pp. 59-65. ISSN 0169-4332.



- [4] JACKSON, N. et al. Flexible-CMOS and biocompatible piezoelectric AIN material for MEMS applications. *Smart Materials and Structures*, 2013, vol. 22, no. 11. ISSN 0964-1726.
- [5] DOLL, J. C. et al. Aluminum nitride on titanium for CMOS compatible piezoelectric transducers. *Journal of Micromechanics and Microengineering*, 2010, vol. 20, no. 2. ISSN 0960-1317.
- [6] AL AHMAD, M. a PLANA, R. Piezoelectric Coefficients of Thin Film Aluminum Nitride Characterizations Using Capacitance Measurements. *Ieee Microwave and Wireless Components Letters*, 2009, vol. 19, no. 3, pp. 140-142. ISSN 1531-1309.
- [7] IRIARTE, G. F. et al. Influence of substrate crystallography on the room temperature synthesis of AIN thin films by reactive sputtering. *Applied Surface Science*, 2011, vol. 257, no. 22, pp. 9306-9313. ISSN 0169-4332.
- [8] FIGUEROA, U. et al. Production of AIN films: ion nitriding versus PVD coating. *Thin Solid Films*, 2004, vol. 469, pp. 295-303. ISSN 0040-6090.