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PAPER

Enhanced process stability for the low temperature sputter deposition of aluminium nitride thin films

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Keywords: d_{33}, leakage current, piezoelectric coefficient, AlN, thin film deposition, clamped substrate holder

Abstract

MEMS (micro electro-mechanical systems) operated in resonance and excited piezoelectrically are nowadays used for a broad range of different application scenarios. To enhance the process stability and hence, the reproducibility of key film parameters of sputter-deposited aluminium nitride such as the film stress, the piezoelectric coefficient d_{33} and low leakage current levels, a novel aluminium clamped substrate holder is reported. Compared to the standard molybdenum based solution, where the thermal contact between the wafer and substrate holder varies during deposition, as the wafer can move freely, the substrate temperature variations are substantially reduced due to clamped configuration. Independent of AlN film thickness ranging between 0.5 μm and 2.0 μm the scatter in piezoelectric constant d_{33} and leakage current characteristics represented by the barrier height and the activation energy is reduced up to a factor of 3. These results demonstrate the importance to control carefully the temperature conditions during low-temperature AlN deposition to ensure a high reproducibility in film properties.

Introduction

In recent years, a large amount of silicon based MEMS (micro electro-mechanical systems) sensors and actuators were developed. Due to the detection of e.g. chemical- [1–3] or physical quantities [4–7] a broad range of different application scenarios were made accessible and are commercially available today. Independent of their later use many device architectures are common that they take advantage of either membranes or cantilevers as key elements. Furthermore, MEMS devices are often operated in resonance to increase the sensitivity, by applying either electro-magnetic, capacitive or piezoelectric transducers. In the case of piezo-electrically excited silicon MEMS devices, sputter deposited aluminium nitride (AlN) is often the material of choice [8, 9]. Compared to zinc oxide (ZnO) or lead zirconate titanate (PZT), AlN is preferred as functional material, since AlN is compatible with standard complementary metal oxide semiconductor (CMOS) microfabrication processes [10] and offers a high temperature stability. After post-deposition annealing a stable microstructure up to 1000 °C is demonstrated for sputter-deposited AlN layers in non-oxidising gas atmospheres [11]. When applying reactive magnetron sputter deposition as in the present study for AlN synthetization, the substrate is self-heated by the particle bombardment [12]. The introduced heat leads to thermal induced bending of the substrate/thin film combination due to the different coefficients of thermal expansion (CTE). If the wafer can move freely the thermal connection between substrate and holder is continuously changing, thus leading to an undefined substrate temperature during the deposition process. Sputter deposited AlN thin films show a strong dependence on temperature [13] and hence, a high impact on the electro-mechanical parameters like piezoelectric coefficient d_{33} or the leakage current characteristics are expected. To ensure reliable realization of piezoelectric MEMS devices a high stability in the deposition process has to be ensured.

In this study, the influence of defined temperature conditions on the electro-mechanical properties of low temperature deposited AlN layers is investigated by mechanically clamping the silicon wafer. To increase the thermal conductivity aluminium (Al) is chosen as substrate holder material, thus lowering thermal resistance to

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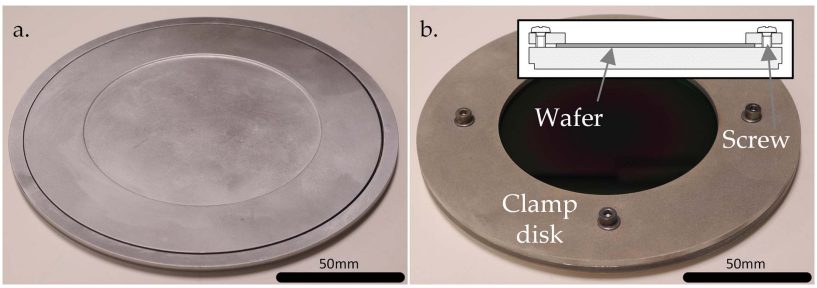

Figure 1. Standard sample holder made of Mo where the wafer moves freely (a) and a tailored substrate holder made of Al for clamping the wafer consisting of a base plate, a clamping disk and fixing screws the latter made of stainless steel (b).

the carousel. Compared to a standard molybdenum (Mo) holder illustrated in figure 1(a) where the wafer can move freely, a tailored wafer holder is realized shown in figure 1(b) where in addition an aluminium disk presses the silicon wafer to an aluminium substrate holder, thus preventing any movement of the silicon wafer during film synthetization [14].

Beside the d_{33}, which is essential for an effective actuation of MEMS devices, the leakage current characteristics of the AlN layers are investigated. Basically, the type of current transport mechanism and the corresponding leakage current levels are strong indicators for the overall film quality dominated by e.g. pinholes, structural defects or impurities [15].

Experimental details

Three different layer thicknesses (i.e. 0.5 μm, 1.0 μm, 2.0 μm) are prepared to study the impact of wafer clamping on the electro-mechanical properties of AlN films during deposition. These experiments are repeated in a time period of 5 weeks to demonstrate the stability and the robustness of the deposition process.

Sample preparation

The AlN thin films were deposited on 4″ phosphorous doped (100) silicon substrates with a bulk resistivity of about 50 $\Omega \cdot$ cm. After pre-conditioning with an inverse sputter etching (ISE), AlN thin films are sputter deposited on silicon substrates with a commercially available DC magnetron sputtering equipment from Ardenne (LS 730S). During deposition, the maximum process temperature T_m of the substrate holder (figure 1(b)) is measured *in situ*. A pyrometer (DIAS Pyrospot DGE 10N) with a working range from 100 °C up to 850 °C, which is installed at the process vacuum chamber, was used. In order to reduce temperature measurement inaccuracies caused by variations of the emissivity ε of the substrate holder, an Agilent T U1241A with a type K thermocouple records the temperature after the sample was transferred out of the vacuum chamber.

By applying a shadow mask circular electrodes with a diameter of 1.0 mm are realized by sputter-deposition of aluminium with a thickness of 200 nm. On the complete backside of all samples again a 200 nm thin aluminium layer is deposited to provide a good electrical contact to ground. For film stress measurements, a capacitive wafer bow gauge (MX 203-6-33 from E + H Metrology) is used.

Electro-mechanical measurements

With a commercial available piezometer PM300 from Piezotest the piezoelectric coefficient d_{33} is determined. Based on the Berlincourt method [16] the sample is clamped at the 1.0 mm diameter contact surface between two electrodes and an oscillating force perpendicular to the piezoelectric thin film surface is applied. The resulting electrical signal is compared to an internal reference what enables a direct determination of d_{33}.

Likewise, these electrodes are used for the leakage current measurements, which were performed using a source measurement unit B2911A from Agilent electrically connected via needle probes at a Süss PM 8 probe station. The measurement procedure itself ramps the voltage in 80 steps for all layer thicknesses two times from 0 V to 20 V, to −20 V and back to 0 V [17]. After having electrically stabilized the piezoelectric layer, 10 current measurements with a cycle time of 250 ms are performed and averaged. The measurements were done automatically with a MatLab script, which controls the integrated chuck with temperature control from ATT A300. The sample temperature was varied between 25 °C and 300 °C in air and due to hysteresis effects at lower temperatures, the evaluation of the barrier height was restricted to temperatures $\geqslant$100 C [18, 19]. When evaluating the basic conduction mechanisms in sputter-deposited AlN thin films the leakage current density J

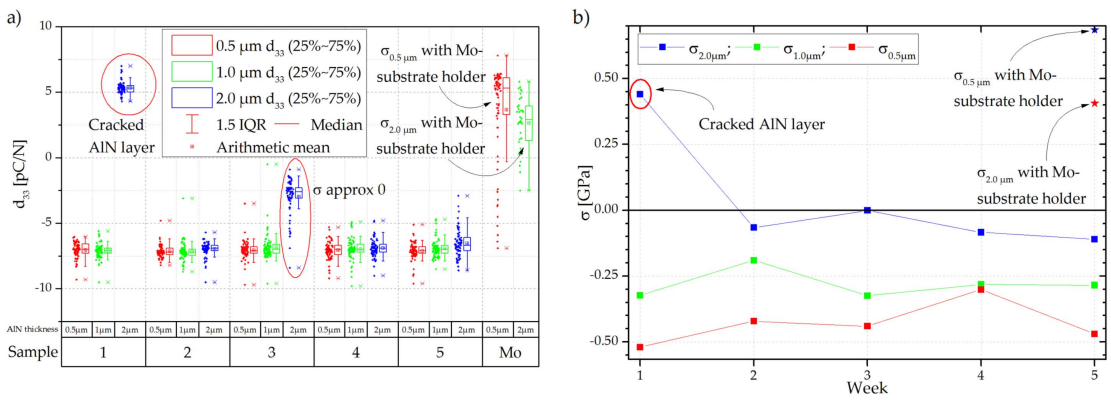

Figure 2. (a) Boxplot of piezoelectric coefficient d_{33} for week 1 to 5 for AlN layer thickness values of 0.5 μm, 1.0 μm and 2.0 μm. Each boxplot represents measurements of 72 individual test structures. (b) The figure shows the layer stress for AlN for week 1 to 5 with the clamped as well as with the standard wafer holder.

shows a dominant ohmic behaviour at low electric fields $E \leqslant 0.1$ MV cm^{-1} and can be described by equation (1) with the conductivity ρ, the thermal activation energy E_A and the Boltzmann constant k.

$$J(E,\ T) = \rho E \cdot e^{\frac{-E_A}{kT}} \tag{1}$$

At larger electrical field strengths $E \geqslant 0.3$ MV cm^{-1} J is dominated by a Poole-Frenkel (PF) mechanism [17] which is expressed as a function of E and T by the following relations

$$J(E,\ T) \sim E \cdot e^{\frac{-E_A}{kT}} \tag{2}$$

$$E_A(E) = q\left(\phi_B - \sqrt{\frac{qE}{\pi\varepsilon_0\varepsilon_r}}\right) \tag{3}$$

where q denotes the elementary charge, ϕ_B the barrier height, ε_r the relative permittivity and ε_0 the dielectric constant, respectively [20]. A reasonable value for ε_r is 10 for sputter deposited AlN thin films, as reported in a previous investigation [21].

Results and discussion

To estimate the process variation of the AlN reactive sputter deposition process and its impact on the piezoelectric coefficient d_{33} the experiments are performed within a time period of 5 weeks. Every boxplot in figure 2(a) consists of 72 circular electrodes per wafer and indicate the minimum, the maximum, the arithmetic mean and the median value. Basically, the sample holder design has a strong impact on the sign of d_{33} as in clamped configuration a negative sign results, whereas the standard solution where the wafer can move freely has a positive sign. Additionally, the boxplots of the clamped sample holder indicate a substantial decrease in scatter of d_{33} in each week with an overall *average value* of about -7 pN/C, independent of the film thickness. In contrast, the samples with the molybdenum sample holder show a widespread distribution in d_{33} values in each boxplot.

To demonstrate a correlation between d_{33} and the layer stress σ figure 2(b) illustrates the measurement results of the latter parameter as a function of film thickness and week whereas these values were taken from [14]. The samples with 2.0 μm thickness synthesized either in week 1 or in week 3 attract attention due their diverged values. As a consequence of some clamping misalignment between the sample holder and the silicon wafer the sample of week 1 reaches the highest deposition temperature of 160 °C in this investigation. This leads to a change in the sign of the layer stress and to a positive value of 0.45 GPa. In fact, a high stress level is introduced in the AlN layer indicated by cracks, thus influencing the d_{33} measurements. In agreement to all other samples week 3 sample with 2.0 μm thickness proposes that there is a link between a very low stress status of about -8.8 MPa and low average d_{33} values of -2.5 pC/N. All other samples show a constant d_{33} value due to a constant deposition temperature of about 140 °C, which is reflected in a reduced scatter in c-axis orientation of the samples synthesized with the clamped sample holder. In contrast, the temperature distribution on the surface at the standard sample holder may be inhomogeneous, which has a strong impact on σ [22] and hence, on the d_{33} values.

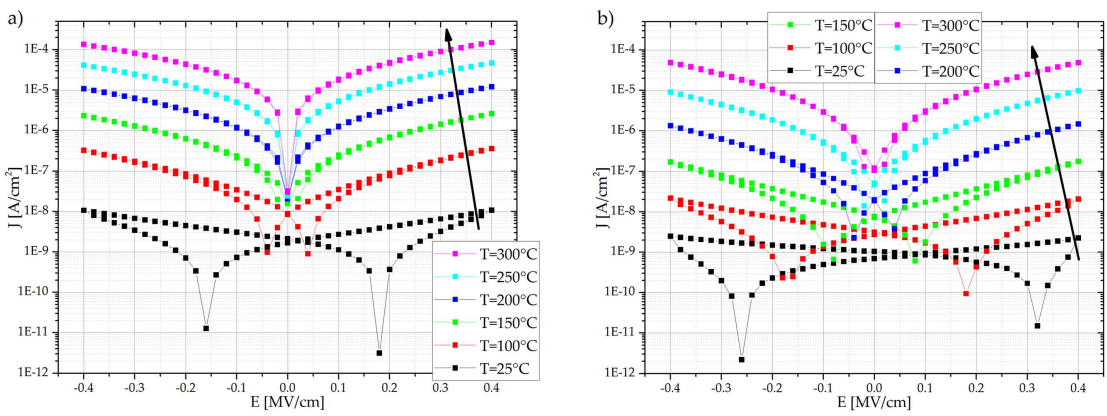

Figure 3. Typical leakage current characteristics at different temperature levels of a 0.5 μm thin AlN film deposited in week 3 with clamped substrate holder at (a) and with free moving Mo substrate holder (b).

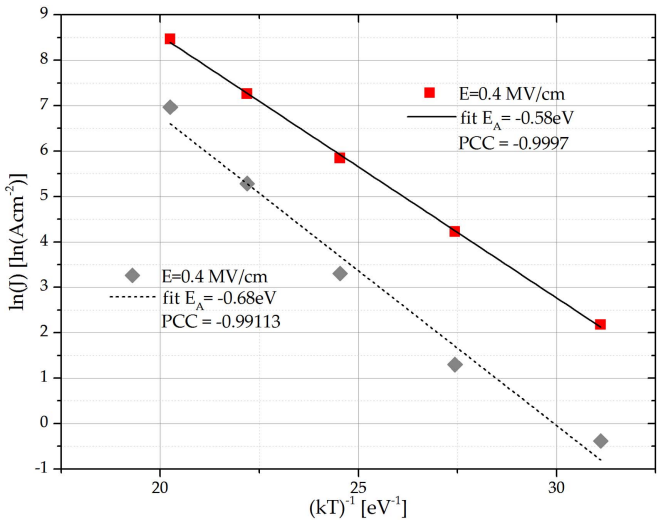

Figure 4. Arrhenius plot of leakage current of sample 3 with 0.5 μm AlN thickness at E $=$ 0.4 MV cm^{-1}. Measurement points are taken at 100 °C, 150 °C, 200 °C, 250 °C and 300 °C in clamped configuration and with the standard Mo substrate holder.

These results indicate a correlation between the biaxial layer stress in the deposited AlN layer and the d_{33} values, which both depend on the crystallographic orientation (c-axis) of the deposited piezoelectric layer. Therefore, a change in the sign of the d_{33} values indicates a corresponding change in the sign of the layer stress.

In figure 3(a) an exemplary J-V characteristics of a 0.5 μm thin AlN sample deposited in week 3 is shown. Besides the expected increase in leakage current density J at enhanced electrical field $E \sim U$, J increases with temperature T. The characteristics show a symmetric behaviour between positive and negative applied voltages and the hysteresis at low electric field levels decrease at higher measurement temperatures. Besides this behaviour figure 3(b) shows a stronger hysteresis effect at the 0.5 μm AlN sample deposited with the standard Mo sample holder.

By arranging ln(J) over $1/k \cdot T$ from equation (1), $E_A(E)$ of the Poole Frankel charge transport is determined for each E. Due to the thermally activated conduction mechanism the measurement results show a high linearity at temperatures above 100 °C. As a representative example, an AlN sample with 0.5 μm of week 3 was selected and the corresponding temperature dependence is displayed as Arrhenius plot in figure 4. The coefficient E_A of the equation (2) is determined by the slope of a linear regression using a MATLAB script. Compared to the measurements of the layers deposited with the Mo holder as illustrated in figure 3, the layers deposited with the clamped sample holder show a higher linearity indicated by a lower Pearson correlation coefficient (PCC).

Applying this approach to all samples from week 1 to 5 for each thickness and also to the reference sample with the Mo holder, the corresponding activation energies are listed in table 1. When comparing these E_A values to previous publications where the same sputter deposition equipment and the same evaluation procedure of the electrical measurements were applied, the standard deviation as an indicator for the process stability shows a

Table 1. Activation energies for AlN thickness of 0.5 μm (E = 0.4 MV cm^{-1}), 1.0 μm (E = 0.2 MV cm^{-1}) and 2.0 μm (E = 0.1 MV cm^{-1}) from week 1 to 5 for clamped and standard Mo sample holder.

	AlN thk.		
Week	0.5 μm E_A [eV]	1.0 μm E_A [eV]	2.0 μm E_A [eV]
1 (clamped)	−0.53	−0.58	−0.60
2 (clamped)	−0.57	−0.62	−0.63
3 (clamped)	−0.58	−0.59	−0.59
4 (clamped)	−0.56	−0.61	−0.55
5 (clamped)	−0.56	−0.61	−0.56
Mo holder (freely movable)	−0.68	—	−0.63

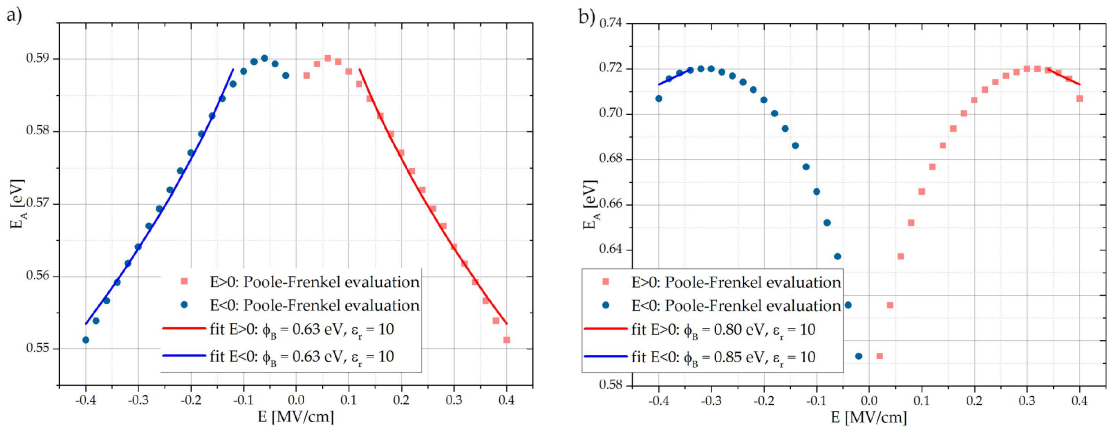

Figure 5. Typical Poole-Frenkel barrier height evaluation of the sample with 0.5 μm thickness from week 3 with clamped (a) and with standard Mo substrate holder (b).

substantial lower value of 0.02 independent of film thickness with respect to values ranging up to about 0.25 for 0.5 μm thin AlN films (see [17, 21]).

By rearranging equation (2) and applying $E_A(E)$ by fitting to the measurement results ϕ_B is determined and is shown exemplary for week 3 for 0.5 μm thin samples in figure 5(a). The fitting curves to determine ϕ_B show the same values for both the negative and positive bias direction as it can be expected from the results presented in figure 3(a). The barrier height for AlN sample synthesized with the Mo sample holder is displayed in the figure 5(b). In comparison to the measurements results in [17], a Poole-Frenkel behaviour starting from E $>$ 0.35 MV cm^{-1} can be assumed. The comparison of the results in figure 5(a) *vs*. Figure 5(b) shows a current characteristic primarily dominated by Poole Frenkel conduction mechanism for the sample deposited using the tailored substrate holder compared to a transitional behaviour between different conduction mechanisms for the sample using the standard Mo holder. This improvement is most likely caused by the reduced amount of defect states in the grain boundaries due to the increased *c*-axis orientation and improved crystallinity of the AlN thin film deposited on the clamped substrate, which was shown by XRD analysis in [14].

A MATLAB script using nonlinear data-fitting based on equation (3) in a least-squares approach is applied to the data to determine ϕ_B. The fit was applied to all samples from week 1 to 5 independent of their thickness and independent of the sample holder type. The ϕ_B values are listed in table 2, which represents the arithmetic average for the two different ranges $E > 0$ and $E < 0$, respectively. When considering the 0.5 μm thin layer, the barrier height varies with a standard deviation up to a maximum value of 0.03 compared to 0.09 [11] and 0.10 [15, 17, 21]. When comparing the results for ϕ_B in this work with those done at AlN layers with thicknesses below 0.5 μm [17], a very good agreement is demonstrated, thus indicating no temperature dependence on the barrier height for thickness values between 0.1 μm and 2 μm.

In figure 6 the results of E_A (table 1) and ϕ_B (table 2) are summarized with boxplots of week 1 to 5 including those gained from the AlN films synthesized with the Mo substrate holder. The remarkable deviation of ϕ_B for 0.5 μm thin AlN samples deposited with the Mo sample holder draw attention to a detailed analysis of the J-V characteristics shown in figure 3. A wider hysteresis shown in figure 3(b) in contrast to figure 3(a) at 100 °C reveal a substantial deposition process improvement when applying the clamped sample holder.

Table 2. Barrier height for AlN thickness of 0.5 μm, 1.0 μm and 2.0 μm from week 1 to 5 for sample holder types separated for positive and negative E values.

	AlN [μm] E [MV cm^{-1}]					
Week	0.5 E < 0 ϕ_B [eV]	0.5 E > 0 ϕ_B [eV]	1.0 E < 0 ϕ_B [eV]	1.0 E > 0 ϕ_B [eV]	2.0 E < 0 ϕ_B [eV]	2.0 E > 0 ϕ_B [eV]
1 (clamped)	0.58	0.57	0.61	0.61	0.80	0.80
2 (clamped)	0.63	0.64	0.71	0.65	0.75	0.65
3 (clamped)	0.63	0.63	0.67	0.62	0.64	0.63
4 (clamped)	0.62	0.62	0.72	0.65	0.67	0.59
5 (clamped)	0.62	0.62	0.72	0.65	0.67	0.61
Mo holder (freely movable)	0.85	0.80	—	—	0.60	0.65

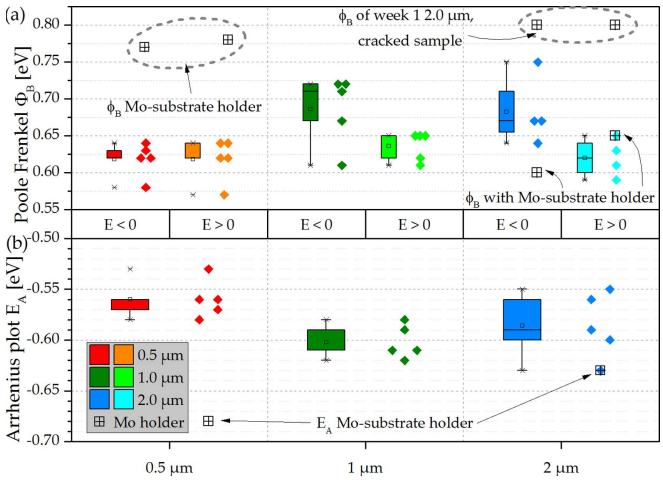

Figure 6. Boxplot of (a) the barrier height ϕ_B and of (b) the activation energy E_A measured at sputter-deposited AlN samples with clamped sample holder of week 1 to 5 and Mo sample holder.

When analysing the E_A values in figure 6(b) the boxplots show similar average values independent of the AlN layer thickness for week 1 to 5 in contrast to the Mo-substrate holder. When applying the clamped configuration during deposition E_A is decreased for the 0.5 μm thin layer, but is within the measurement accuracy of the 2.0 μm layer. Due to the 4 times longer deposition time the quality of the thermal contact of the wafer to the substrate holder may play a minor role compared to the clamped sample holder. The sample with the cracked AlN 2.0 μm layer from week 1 has a 10 times higher leakage current, which indicates the current flow via the cracks, thus motivating the lowered E_A values.

Conclusions and outlook

In this study, a comparison between the electro-mechanical properties of sputter-deposited AlN thin films is performed which are synthesized either with a standard Mo holder, where the wafer can move freely during deposition, or with a tailored Al wafer holder, where the wafer holder clamps the silicon wafer. To indicate the improvement in film quality when using the clamped holder configuration, the piezoelectric coefficient d_{33} and the temperature dependence of the electrical leakage current characteristics were measured. A Poole-Frenkel conduction mechanism is determined for electric field values above 0.1 MV cm^{-1} and the activation energy E_A as well as the barrier height ϕ_B were extracted for AlN layers from 0.5 μm up to 2.0 μm. With the specific Al wafer holder the AlN layer show a substantially reduced scatter in piezoelectric coefficient d_{33}, activation energy values and barrier heights up to a factor of 3. Furthermore, a correlation between d_{33} and layer stress is revealed, as a change in sign of the film stress leads to a change in sign of d_{33}. All in all, it can be summarized that controlling the temperature during AlN deposition in a clamped wafer holder configuration results in a substantial increased reproducibility in key film parameters, even at substantial lower temperatures during synthetization of about 140 °C. These promising results may pave the way for a faster commercialization of AlN-based MEMS sensors and actuators in the near future.

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