## FABRICATION AND CHARACTERISATION OF SCALN-BASED PIEZOELECTRIC MEMS CANTILEVERS

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## ABSTRACT

Scandium (Sc) doping of aluminium nitride (AlN) increases the piezoelectric actuation potential due to substantially enhanced piezoelectric constants. This work demonstrates the fabrication of MEMS cantilevers actuated by sputter deposited  $Sc_xAl_{1-x}N$  thin films (x = 27%) sandwiched between gold electrodes. Patterning of  $Sc_xAl_1$ . N films is performed by a reactive ion etching process using SiCl<sub>4</sub>. The dynamic actuation potential of the fabricated devices is evaluated with Laser Doppler Vibrometry and with electrical impedance spectroscopy measurements. When applying the Butterworth Van-Dyke equivalent circuit a significant increase of the effective transverse piezoelectric constant  $d_{31}$  is demonstrated.

#### **KEYWORDS**

ScAlN thin films, piezoelectric, MEMS cantilever, impedance spectrum, Laser Doppler Vibrometer.

## **INTRODUCTION**

Piezoelectric thin films are used within MEMS resonators for excitation and sensing purposes. Resonance frequency and damping (Q-factor) of cantilever type MEMS are sensitive e.g. to changes in a fluid environment, to mass or stress loading which can be employed for biosensing applications or measurement of liquid properties such as density  $(\rho)$  and viscosity  $(\eta)$  [1, 2]. For the latter purpose, aluminium nitride (AlN) has been successfully applied to all-electric MEMS resonators for Q-factor measurements as  $Q \alpha (\eta \rho)^{-1/2}$  [2]. The device performance, however, is improved by a higher piezoelectric modulus  $d_{31}$  as the conductance peak  $\Delta G$  is proportional to  $d_{3l}^2$  [2]. Therefore, Sc<sub>x</sub>Al<sub>1-x</sub>N offers great potential especially for such applications since the piezoelectric constants increase with Sc content x. The transverse piezoelectric coefficient  $d_{31}$ increases from  $d_{31} \approx -2$  pm/V for x = 0% up to -13 pm/V for x = 42%[3]. Additionally, the elastic modulus decreases from  $E_1 = 325$  GPa for pure AlN to  $E_1 = 170$  GPa for x = 42%. Once this concentration is exceeded a phase transition from wurtzite to cubic type crystal structure takes place, thus preventing further increase of the piezoelectric modulus. Reported MEMS device based on Sc doped AlN include surface as well as bulk acoustic wave devices [4-7]. But, released micromachined structures, such as cantilevers with Sc<sub>x</sub>Al<sub>1-x</sub>N are not reported in literature. This work focuses on  $Sc_xAl_{1-x}N$  with x = 27% as the piezoelectric active thin film on cantilever devices thereby, avoiding disadvantageous effects near the phase transition, but exploiting the high increase in  $d_{31}$  reported for this concentration [8]. Electrical characterisation is performed

based on a self-actuation self-sensing approach in addition to optical evaluation of the deflection applying Laser Doppler Vibrometry.

# DEVICE FABRICATION

# Work Flow

Piezoelectric cantilevers in different geometrical dimensions are fabricated, each one based on a  $Sc_xAl_{1-x}N$  thin film sandwiched between two symmetric pairs of gold (Au) electrodes. The electrode pairs enable the individual excitation of out-of-plane as well as in-plane eigenmodes. The designs consider different cantilever dimensions to demonstrate the impact of electrode area on mechanical



*Figure 1: Optical micrograph of a typical MEMS cantilever device in top view.* 

deflection and the impedance characteristics. In Figure 1, a typical design of a released cantilever is shown. The top electrode pair is visible and 30  $\mu$ m wide feedlines for sensing and excitation. The fabrication process is based on 100 mm SOI wafers with a device layer thickness of t<sub>b</sub> = 20  $\mu$ m and an additional bi-layer of SiO<sub>2</sub>/Si<sub>x</sub>N<sub>y</sub> (150 nm/80 nm) on top. The bottom Au electrode pair and



Figure 2: Schematic front view on the cantilever (Cant.) and the silicon frame including bonding pads for top (Top-El. pad) and bottom electrodes (Bt–El. pad). Stack sequence of cantilever structure and SOI wafer:  $Si_xN_y$ , Au ( $t_{El}$ =400 nm), ScAlN ( $t_p$ =500 nm), Au, SiO/SiN, Si ( $t_b$ =20 µm), SiO - white. the contact pads comprising a Cr adhesion layer are thermally evaporated on the nitride and patterned with optical lithography and a lift-off process. Subsequently, the piezoelectric Sc<sub>x</sub>Al<sub>1-x</sub>N layer is deposited. A schematic front view of all layers associated with the cantilever is illustrated in Figure 2.

The  $Sc_xAl_{1-x}N$  with x = 27% and thickness  $t_p = 500$  nm is deposited via DC reactive magnetron sputtering from a

100 mm AlSc alloy target. The thin film is prepared in an  $Ar/N_2$  atmosphere at nominally unheated substrate conditions. Au top electrodes are again thermally evaporated and patterned with a lift-off process. Reactively sputtered silicon nitride  $(Si_xN_y)$  is used as mask material for the patterning process of ScAlN and simultaneously acts as electrode passivation. Both films, ScAlN and  $Si_xN_y$  are dry etched by reactive ion etching (RIE), described in the following section. Both the device layer and the handle wafer are etched with a deep reactive ion etching (DRIE) process and finally, the cantilevers are released in hydrofluoric acid by removing the SiO<sub>2</sub>. Finally, the devices are electrically connected to the DIP package with Au wires by thermal bonding.

#### **Reactive ion etching of ScAlN**

Compared to wet chemically etching patterning of thin films with an inductively coupled plasma reactive ion etching process (ICP-RIE) is advantageous due to its high anisotropic etch performance. This prevents underetching effects that may lead to an electrical short circuit of top and bottom electrode. For pure AlN thin films highest etch rates are reported in chloride based gas mixtures [9]. This work



Figure 3: Scanning electron microscopy images of patterned ScAlN after SiCl<sub>4</sub> etching process. (a) Cr-hardmask / ScAlN/ Si- substrate and (b)  $Si_xN_y$  hardmask / ScAlN – after release by  $XeF_2$ .

employs SiCl<sub>4</sub> for the ICP-RIE process to pattern ScAlN and pure AlN thin films with a RIE Oxford 100 system. Prior to the ScAlN etching process the  $Si_xN_y$  hardmask was patterned in the ICP system using a mixture of SF<sub>6</sub> and O<sub>2</sub>. The SiCl<sub>4</sub> process parameters were: pressure p = 15 mTorr, RF power: 225 W, ICP power: 150 W. The etch rate r decreases from r = 24 nm/min for pure AlN to r = 10 nm/min with a Sc concentration of x = 27%. Due to the high etch rate of  $Si_xN_y$  in SiCl<sub>4</sub> (20 nm/min) the thickness compared to ScAlN needs to be larger than 2:1. Figure 3(a) shows the resulting steep sidewalls in an electron microscope (SEM) micrograph that illustrate the anisotropic character of the process. This preliminary test was performed with a chromium etch mask, having a higher selectivity to ScAlN than Si<sub>x</sub>N<sub>y</sub>. The thickness of the etched ScAlN thin film is 500 nm. In Figure 3(b), a SEM micrograph of a released cantilever structure is shown. In contrast to the device fabrication, the release of the structure in Figure 3(b) was performed in a xenon fluoride (XeF2) based process to remove the Si device layer. In addition to ScAlN sidewalls, the Si<sub>x</sub>N<sub>y</sub> mask layer is shown in the picture. For this mask layer an initial thickness of 1600 nm was used to pattern a 750 nm thick ScAlN thin film. The thickness of the remaining Si<sub>x</sub>N<sub>y</sub> after the process is approximately 500 nm.

#### **MEASUREMENT RESULTS** Cantilever Deflection



*Figure 4: Z-Deflection of a cantilever excited in the 3<sup>rd</sup> OP Eigenmode measured with Laser Doppler Vibrometry* 

Initial characterization of the bonded cantilever is done optically employing a Laser Doppler Vibrometer (LDV) from Polytec (MSA 400). The study is focused on the analysis of out-of plane (OP) eigenmodes in air at ambient pressure. For that reason, an AC voltage amplitude of 0.5 V is applied to the top electrode pair while the bottom electrodes are grounded. Figure 4 shows the vertical deflection of a 511 x 959  $\mu$ m<sup>2</sup> sized cantilever oscillating in the 3<sup>rd</sup> OP mode (496 kHz), whereas experimental data are gained from the matrix of points covering the cantilever surface. The LDV grid measurements are used to identify the mode type and order. For a further evaluation of the deflection a line scan is performed along the cantilever axis. The vertical deflection amplitude along this line is then used to extract an effective value for the transversal piezoelectric coefficient  $d_{31}$ . For that purpose, a theoretical model describing the cantilever deflection along the center line is fitted to the deflection data.

Table 1: Key parameters of ScAlN or AlN based cantilevers having different width W and length L. Measured resonance frequency f, calculated resonance frequency  $f_{thv}$  Q-factor and conductance peak  $\Delta G$  from BVD equivalent circuit with parameters such as parallel resistance / capacitance Rp/ Cp, motional resonance parameters Rm, Lm, Cm. Out-of plane resonance number num, effective piezoelectric constant  $d_{31}$  obtained from electrical conductance,  $d_{31,V}$  from LDV based optical deflection measurements.

Туре	Num	W /	L/	f/	f <sub>T</sub> /	Q	$\Delta G$ /	$\Delta G/Q$ /	d <sub>31</sub> /	$d_{31,V}$ /	Rp/	Cp/	Rm/	Lm/	Cm/
		μm	μm	kHz	kHz		μS	nS	pm/V	pm/V	MΩ	pF	kΩ	Н	fF
ScAlN1	1	804	1203	18.2	18.0	302	11.0	36.4	5.1	6.0	5.0	320	91	240	320
ScAlN1	2	804	1203	112.6	112.6	165	7.6	46.1	4.2	4.5	0.6	317	132	31	65
ScAlN2	1	511	959	28.4	28.3	748	18.6	24.9	4.7	5.4	4.0	194	54	223	141
ScAlN2	2	511	959	177.4	177.3	320	11.2	35.0	4.0	4.6	31.0	193	89	26	32
ScAlN3	1	327	767	43.7	44.2	875	13.8	15.8	4.2	4.9	0.5	128	95	24	14
ScAlN3	2	327	767	273.3	277.1	436	10.5	24.1	3.7	3.8	0.6	127	144	8	5
ScAlN4	1	1000	1000	25.6	26.0	778	39.0	50.1	4.9	5.7	35.0	337	26	124	312
ScAlN4	2	1000	1000	159.4	163.0	167	9.7	57.9	3.8	4.6	5.0	335	104	17	58
AlN1	1	513	1602	10.6	11.1	660	8.2	12.4	2.6	2.6	41.2	138	122	1070	185
AlN1	2	513	1602	70.7	69.8	532	10.7	20.1	2.3	2.5	4.7	127	94	112	45

In order to describe the cantilever deflection a model is employed as described for instance in Leighton *et al.* that assumes  $T \ll w \ll L$ , where *L* denotes the cantilever length, *w* the width and *T* the thickness [10]. It assumes that the actuation potential of the piezoelectric film upon application of an electric field *E* translates into an out-of plane bending moment  $M_{OP}$ . The total thickness *T* is the sum of the silicon device layer thickness  $t_{b}$ , the piezoelectric film thickness  $t_p$ and the electrode thickness  $t_{El}$ .  $M_{OP}$  is proportional to the induced in-plane strain  $\varepsilon = V/t_p \cdot d_{31}$  which is extracted from the equation to yield  $M_{OP}' = M_{OP}/\varepsilon$ . The deflection  $\Delta z$  at a position *x* along the cantilever length *L* is then given at the resonance frequency via a dynamic calculation as:

$$\Delta z(\mathbf{x}) = d_{31} \frac{M'_{OP}}{m\omega_i^2} \varphi'_{cf,i}(L) \varphi_{cf,i}(\mathbf{x}) QV \qquad (1.1)$$

where  $\omega_i$  is the Eigenfrequency,  $\varphi_{cf,i}$  the mode shape,  $\varphi'_{cf,i}$  its slope at the cantilever tip x = L and the applied voltage *V*. The Q-factor appears due to the employed model that describes the dynamic form by a transfer function which has the absolute value *Q* at the resonance frequency.



Figure 5: Conductance G and susceptance B near the  $l_{st}$  outof plane eigenmode of a ScAlN excited cantilever (W = 511 um, L = 959 um) in air.

#### **Electrical Admittance**

The dynamic response of the cantilever is measured electrically using an Agilent 4294A precision impedance analyser. In analogy to the measurements performed with the LDV, the top and bottom electrode pairs are excited in parallel to further study the OP eigenmodes. The conductance G and susceptance B are measured around each Eigenfrequency and a Butterworth Van Dyke equivalent circuit model is fitted to the frequency response as described in the work of Kucera et al. [2]. In addition to the equivalent circuit parameters, the conductance peak height  $\Delta G$  and the Q-factor are extracted with this approach. A typical measurement of a ScAlN based cantilever design oscillating at resonance in the fundamental mode is shown in Figure 5. The described approach yields the following fitting results:  $\Delta G = 18.6 \,\mu S$  and Q = 748. These parameters are further used to calculate the effective piezoelectric constant  $d_{31}$ . For this purpose, the conductance peak height is calculated as being proportional to the dynamic capacitance change due to the induced piezoelectric charge. The derivation in analogy to Kucera et al., but for OP modes yields for the conductance peak [2]:

$$\Delta G = d_{31}^2 \frac{M'_{OP}}{2\omega_i} \frac{E_P}{\rho L} \varphi_{cf,i}^{\prime 2}(L)Q \qquad (1.2)$$

with the in-plane modulus of ScAlN  $E_P$ , average density  $\rho$  and curve shape functions  $\varphi_{cf,i}$ . For the evaluation Ep = 327 GPa and Ep = 217 GPa was considered for pure AlN and ScAlN, respectively [8].

#### **RESULTS & DISCUSSION**

Cantilevers in different geometric dimensions were fabricated and evaluated electrically and optically. Electrical admittance measurements were used to extract equivalent BVD circuit parameters from a fitting procedure of the data around the corresponding eigenfrequency. The transversal piezoelectric coefficient  $d_{31}$  was calculated from conductance peak height and deflection maxima, respectively. For comparison purposes, a cantilever based on pure AlN is analyzed in addition to the ScAlN based devices. Table 1 gives an overview of the results for four different ScAlN based cantilevers and one excited with a pure AlN film. For each device, the first and second OP modes are evaluated. The piezoelectric constants from deflection and conductance measurements agree reasonable well. On average the ScAlN cantilever exhibit a  $d_{31}$  of -(4.3±0.5) pm/V and  $-(4.9\pm0.7)$  pm/V, respectively. Furthermore, evaluation of the reference AlN based cantilever yields an average  $d_{31}$  = -2.5 pm/V. Q-factors from deflection measurements match the electrically determined values closely. The signal level  $\Delta G$  is clearly largest for the ScAlN cantilever having the largest electrode area as expected theoretically. Theoretically calculated values for the resonance frequencies are close to the measurements, with discrepancies explained by the atmospheric measurement conditions in addition to deviations from variations of sputtered thin film thickness across the wafer. Equivalent motional parameters for a series RLC are shown in table 1 in addition to Rp and Cp which describe the good quality of the fabricated devices in terms of parallel resistance due to leakage current and parasitic capacitance.

#### **CONCLUSIONS**

In this study, the fabrication of  $Sc_xAl_{1-x}N$  (x = 27%) based piezoelectric MEMS cantilevers was successfully demonstrated. For characterization, ScAlN thin films with 500 nm thickness are prepared via DC reactive sputter deposition. Patterning of the ScAlN layer is done with an ICP-RIE process based on SiCl<sub>4</sub>. This dry etching process is anisotropic and therefore, prevents due to the high physical character, underetching of the ScAlN thin film. Etch rates of about 10 nm/min were observed and improved etch recipes are currently investigated. The characterization employs measurements of the vertical deflection and analysis of the admittance. The frequency response of conductance and susceptance is fitted to a BVD equivalent circuit model. Further evaluation of the first two OP conductance peaks and the deflection peaks from LDV measurements yield  $d_{31} = 4.3 \text{ pm/V}$  and  $d_{31} = 4.9 \text{ pm/V}$ , respectively. Thus, the reported piezoelectric coefficients are significantly higher compared to values for pure AlN. Potential for further optimization of the ScAlN deposition process remains due to higher, reported  $d_{31}$  values for ScAlN on Si [8].

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#### REFERENCES

- [1] J. Tamayo, P. M. Kosaka, J. J. Ruz, A. San Paulo, and M. Calleja, "Biosensors based on nanomechanical systems", Chemical Society Reviews, 42, 3 (2013), pp. 1287-1311.
- [2] M. Kucera, E. Wistrela, G. Pfusterschmied, V. Ruiz-Diez, T. Manzaneque, J. Hernando-Garcia *et al.*, "Design-dependent performance of self-actuated and self-sensing piezoelectric-AlN cantilevers in liquid media oscillating in the fundamental in-plane bending mode", Sensors and Actuators B-Chemical, 200, (2014), pp. 235-244.
- [3] M. Akiyama, K. Umeda, A. Honda, and T. Nagase, "Influence of scandium concentration on power generation figure of merit of scandium aluminum nitride thin films", Applied Physics Letters, 102, 2 (2013), pp. 021915(1-4).
- [4] W. B. Wang, P. M. Mayrhofer, X. L. He, M. Gillinger, Z. Ye, X. Z. Wang *et al.*, "High performance AlScN thin film based surface acoustic wave devices with large electromechanical coupling coefficient", Applied Physics Letters, 105, 13 (2014), pp. 133502(1-4).
- [5] A. Teshigahara, K.-Y. Hashimoto, and M. Akiyama, "Scandium aluminum nitride: Highly piezoelectric thin film for RF SAW devices in multi GHz range". pp. 1-5.
- [6] M. Moreira, J. Bjurstrom, I. Katardjev, and V. Yantchev, "Aluminum scandium nitride thin-film bulk acoustic resonators for wide band applications", Vacuum, 86, 1 (2011), pp. 23-26.
- [7] K. Umeda, H. Kawai, A. Honda, M. Akiyama, T. Kato, and T. Fukura, "Piezoelectric Properties of ScAlN thin films for piezo-mems devices", 26th Ieee International Conference on Micro Electro Mechanical Systems (Mems 2013) (2013), pp. 733-736.
- [8] P. M. Mayrhofer, H. Euchner, A. Bittner, and U. Schmid, "Circular test structure for the determination of piezoelectric constants of ScxAl1-xN thin films applying Laser Doppler Vibrometry and FEM simulations", Sensors and Actuators A: Physical, 222, (2015), pp. 301-308.
- [9] F. A. Khan, L. Zhou, V. Kumar, I. Adesida, and R. Okojie, "High rate etching of AlN using BCl3/Cl-2/Ar inductively coupled plasma", Materials Science and Engineering B-Solid State Materials for Advanced Technology, 95, 1 (2002), pp. 51-54.
- [10] G. J. T. Leighton, P. B. Kirby, and C. H. J. Fox, "In-plane excitation of thin silicon cantilevers using piezoelectric thin films", Applied Physics Letters, 91, 18 (2007), pp. 183510(1-3).

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