

Institute of Sensor and Actuator Systems



Piezoelectric Microsystems: Material Aspects, Devices and Applications





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U. Schmid, M. Schneider

Univ.-Prof. Dr. Ulrich Schmid

- 1993-1998 Study of physics in Munich, Kassel, Nottingham (GB) and Frankfurt/Main
- 1998 Diploma thesis at the microelectronics research lab of the Daimler-Benz AG in Frankfurt/Main

"Preparation and characterization of lateral field effect transistors in 6H-SiC"

- 1999-2001 Ph.D. student at the microsystem research lab of the DaimlerChrysler AG (EADS Deutschland GmbH) in Ottobrunn/Munich
- 2001-2003 Project leader at the EADS Deutschland GmbH in the field of advanced injection technologies
- 2003 Ph.D. degree of the TU Munich with a thesis entitled: "Robust flow sensor for high pressure automotive injection systems"
- 2003-2008 Post doc at the Chair of Micromechanics at Saarland University
- 10/2008 Full professor for Microsystems Technology at the

Vienna University of Technology

- 01/2012 Head of Institute for Sensor and Actuator Systems
- Email Contact: ulrich.e366.schmid@tuwien.ac.at



- 2003-2009 Study of physics at Karlsruhe Institute of Technology (KIT)
- 2008-2009 Diploma thesis at Forschungszentrum Karlsruhe / KIT "Lorentzwinkel-Messungen an hochbestrahlten Silizium-Streifensensoren" "Lorentz angle measurements on highly irradiated silicon strip sensors"
- 2009-2014 Ph.D. student at the Institute of Sensor and Actuator Systems, TU Wien
- 02/2014 Ph.D. degree, TU Wien
 "Einfluss der Schichtdicke und der Substratvorbehandlung auf die elektromechanischen Eigenschaften von gesputterten Aluminiumnitrid-Dünnfilmen"
 "Impact of substrate thickness and pre-conditioning on the electromechanical properties of sputter-deposited aluminum nitride thin films"
- 03/2014 Habilitant at the Institute of Sensor and Actuator Systems, TU Wien
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8 Faculties, ~30.000 students

Electrical Engineering and Information Technology Physics Technical Chemistry Informatics Mathematics and Geoinformation Civil Engineering Mechanical and Industrial Engineering Architecture and Planning Electrical Engineering & Information Technology 2011: 10 institutes

(1st-year students: ca. 350)



3 research groups:

- Micro- and Nanosensors (MNS)
- S. Schmid, Keplinger

Opto-mechanical resonators, microfluidics, technology

- Applied Electronic Materials (AEM)

Nicolics

Packaging, thick film technology, ceramics

- Microsystems Technology (MST)

U. Schmid MEMS, robust materials, technology

Currently circa 30 (state) + 25 (project funded) (of which 20 PhD students) + ca. 10 undergraduate students

MEMS Technology

- Center for Micro- and Nanostructures (ZMNS)
- MEMS Technology Lab/Integrated Ceramic Technology

In total about 250 m² laboratory for sensor realization

Facilities include backside aligner, spray coater, wafer bonder. Key equipment: DRIE, PECVD, LPCVD, electrochemical cell



Research Group: Microsystems Technology

- Expertise in the design, realization and evaluation of MEMS devices and systems
- 2 Post-docs
- 13 Ph.D. students
- 3 research assistants
- 4 technicians
- 1 secretary
- 2 Ph.D. students (external)
- Research topics
- Technology related activities:
- Functional thin films (AIN, SiC)
- Robust thin film systems up to 600°C
- Porosification/Etching techniques
- LTCC/ceramics, flex, silicon, sapphire
- Device related activities:
- Viscosity/density MEMS sensor
- Energy harvesting devices
- High temperature (pressure) sensors
- RF-MEMS switch
- Flow sensors



Research topic:

AlN/ScAlN Thin Film Properties

Motivation: Piezoelectric thin films in MEMS

Typical application scenarios in electronic devices, sensors and actuators:

- SAW: Two port delay line and resonator (b) based sensors ¹
- RF Switches based on PZT actuators (a) ²
- Cantilever based accelerometers (c) ², gyroscopes ³
- Cantilever based detection of adsorbed masses, viscosity, molecules (d) ⁴



¹Tadigadapa, S. and K. Mateti (2009). "Piezoelectric MEMS sensors: state-of-the-art and perspectives." <u>Measurement Science & Technology 20(9);</u> ² Polcawich R (2007) *PhD Thesis,* Pennsylvania State University; ³ S. Günthner, M. Egretzberger, A. Kugi, K. Kapser, B. Hartmann, U. Schmid und H. Seidel; IEEE Sensors Journal, Vol. 6, No. 3, pp. 596 – 604, 2006. ⁴ Tamayo, J., et al. (2013). "Biosensors based on nanomechanical systems." <u>Chemical Society Reviews 42(3): 1287-1311.</u>

Change of electrical polarization due to mechanical deformation of solids

 \rightarrow direct piezoelectric effect

- Deformation due to applied electric field
 → converse piezoelectric effect
- **Non-centrosymmetric** crystal structure (not having a centre of symmetry)
- Common materials:
 - Crystals (quartz, LiNiO3, GaPO4,...)
 - Ceramic thin films (PZT, AIN, ZnO,...)
 - Polymers (PVDF,...)



https://en.wikipedia.org/wiki/Piezoelectricity https://de.wikipedia.org/wiki/Piezoelektrizit%C3%A4t

- Mathematical description of piezoelectric effect:

Mechanical stress

$$T_i = c_{ij}^E S_j - e_{mi} E_m$$

Mechanical strain

$$S_i = S_{ij}^E T_j + \frac{d_{mi}E_m}{\checkmark}$$

pure mechanical

electro mechanical coupling

Motivation: Comparison of Piezoelectric Thin Film Materials

Most typically used piezoelectric thin films in MEMS devices:

- − PZT (Pb (Zr, Ti) O_3) → ferroelectrica, various compositions
- BCZT \rightarrow ferroelectrica, various compositions
- ZnO, AIN \rightarrow piezoelectrica

• Important electromechanical properties:

Material	ε _r	d ₃₁ / pm/V	d ₃₃ / pm/V	C / ms ⁻¹
AIN	10.0	-2.5	5	6000
PZT(25/75, 50/50)	300/165	-15/-12	33/27	2700
BCZT	1000.0	-40.0	80	
ZnO	10.9	-5.8	11	6000

Motivation: AIN related Properties

Material Properties

- ✤ AlN is piezoelectric
- ✤ Direct wide band gap (6.2 eV)
- Good electrical isolation (4-12 MV/cm breakdown field)
- & Low dielectric constant $ε_r$ (~10 $ε_0$)
- ✤ Relative high thermal conductivity (20...300 W/mK)
- ✤ High temperature stability
- ✤ High acoustic wave velocity (~ 6000 m/s)
- ✤ Good temperature stability

Device Related Properties

- Low piezoelectric coefficients
- ✤ CMOS compatible, lead free
- ✤ Requires no high temperature poling step

Crystal structure



Hexagonal wurtzite a: 3.110 Å c: 4.980 Å

(002) basal plane is the most closed packed plane

••

Introduction: Film Synthetization I

Various deposition techniques reported in literature such as

- ADL
- Pulsed laser deposition
- MOCVD
- MBE
- Sputter deposition (DC, RF)





- DC reactive magnetron sputtering system
- Silicon substrates (100), substrates nominally unheated
- Film deposited at different back pressures, plasma powers and gas compositions (N_2 /Ar ratio), electrode distance
- Purity of aluminium target: 99.999%
- Diameter of aluminium target: 150 mm
- Distance between target and substrate: range several cm

Typical AlN layer from our deposition equipment



1000W, 4e-3mbar, 100%N2

Typical example from other groups:





Figure 1. Left: scanning electron microscope (SEM) picture of the texture gradient of an AlN film; right: schematic cross section of a thin nanocrystalline aluminum nitride film.

Mehner et al., JMM, 23 (2013) 095030 (9pp).

Sputter-deposited AIN layers are polycrystalline!

Wet Chemical Etching Experiments I

SEM analysis – Low c-axis orientation

Film deposited at 500 W, $6 \cdot 10^{-3}$ mbar and 75% N₂ (25% Ar)



Surface morphology "as-deposited" Grain size: ~30 nm

Surface morphology after 5 s in H₃PO₄ at 80°C Etch rate: 743,7 Å/s

Surface porosity is very high

Wet Chemical Etching Experiments II

SEM analysis – High c-axis orientation

Film deposited at 1000 W, $4 \cdot 10^{-3}$ mbar and 100% N₂ (0% Ar)







Surface morphology "as-deposited" Grain size: ~ 30 nm

Mean grain size is unaffected

Surface morphology after 20 s in H_3PO_4 at 80°C **Etch rate: 135** Å/s



Surface porosity is low



A. Ababneh, H. Kreher und U. Schmid; Etching Behaviour of Sputter-Deposited Aluminium Nitride Thin Films in H₃PO₄ and KOH Solutions; Microsystem Technologies, Vol. 14, No. 4-5, pp. 567-573, 2008.

Determination of Piezoelectric Coefficients



FEM-simulations (d_{33} = 5.5pm/V; d_{15} = 4pm/V)



Folie 18

Mechanical Characterization using Bulge Testing I

- Uniform pressure load
- Displacement is measured
- Describe bending behavior by polynomial

$$w(r, \boldsymbol{\alpha}) = w_0 \sum_{k=0}^{K} \alpha_{2k} \left(1 - \left(\frac{r}{R}\right)^2 \right) \left(\frac{r}{R}\right)^{2k}$$

- → Coefficients determined by bending behaviour
- \rightarrow Applying minimum potential energy approach for C_i determination

$$p(w_0) = C_1(\boldsymbol{\alpha}) \frac{\sigma_f dw_0}{R^2} + C_2(\boldsymbol{\alpha}; v) \frac{Y dw_0^3}{R^4}$$

R. Beigelbeck et al., Journal of Applied Physics, Vol. 116, pp. 114905, 2014.



Mechanical Characterization using Bulge Testing II

Experimental verification of novel mathematical model using a **silicon** membrane

- →despite non-standard bending curve shape
- \rightarrow excellent agreement with predicted value (E=179,5GPa, σ =110MPa)

Analysis of AIN membranes with thicknesses ranging from 1.2 µm down to about 120 nm

- Low variation with film thickness
- Close to results from nano indentation



M. Schneider et al., Applied Physics Letters, Vol. 105, pp. 201912, 2014.





Silicon Surface Pre-Conditioning Using Sputter Etching I





- Forming of a ~6 nm thick layer of amorphous silicon
- Consistent with simulations of Ar-ion penetration depth
- Introduction of surface-near nucleation sites
- Significant increase in film quality at the same deposition parameters

M. Schneider, et al., Applied Physics Letters, Vol. 101, p. 221602, 2012.



Silicon Surface Pre-Conditioning Using Sputter Etching II

- Prior to sputter deposition, the silicon substrate is sputter etched for 5min
- Significantly lower leakage currents are observed for the pre-treated sample in comparison to a reference sample





Tailoring the silicon / AIN interface II

- As expected: linear correlation between d_{33} and d_{31}
 - $\rightarrow d_{31} = -0.389d_{33}$



Piezo constant d_{22} [pm/V]



M. Schneider et al., Journal of Physics D: Applied Physics, Vol. 48, pp. 405301 (7pp), 2015.



Improvement of piezoelectric constant d_{33} :



- 2 port excitation, 180° phase shifted: 10V, 10-100kHz
- LDV Measurement compared to 3 FEM simulations
- Accuracy of extraction of d_{33} and d_{31} improved

 \rightarrow Via plateau shape comparison



High Accuracy in d_{31} and d_{33} determination

P. Mayrhofer et al., Sensors&Actuators A, Vol. 222, pp. 301-308, 2015.



M. Akiyama et al., Adv. Mater. 2009

Piezoelectric constants of $Y_xAI_{1-x}N$

Piezoelectric constant d₃₃:

- slight increase up to x = 6% Yttrium
- below theoretical value



Measured d_{33} at highest c-axis orientation of $Y_xAl_{1-x}N$

Amorphous initial growth layer found in TEM analysis



Mayrhofer, P. M., et al., Acta Materialia, Vol. 100, pp. 81-89, 2015.



Research topic:

Piezoelectric Resonators

MEMS Resonators: Cantilever Manufacturing Process

a) SOI (silicon on insulator) wafer with 20 μ m device silicon thickness and coating of SiO₂ and SI₃N₄

b) Deposition of Cr/Au electrode, piezoelectric AIN layer and Al topelectrode

> - patterning of AIN with a lift-off process using titanium as sacrificial layer and 40% hydrofluoric acid (HF)

c) Patterning of cantilever and backside hole by DRIE etching process

d) Cantilever release by BOX(burried oxide) removing with5% buffered HF acid

dicing, mounting, bonding,.....



M. Kucera et al., Q-factor enhancement of a self-actuated self-sensing piezoelectric MEMS resonator applying a lock-in driven feedback loop, J. Micromech. Microeng. 23 (2013) 085009.



Basic Device & Fluid Properties

Modified Butterworth-Van Dyke equivalent circuit



- C_p ... Parallel capacitance
- R_p ... Leakage resistance
- $R_{m},\,L_{m},\,C_{m}\,\ldots$ Mechanical resonance





Fluid properties

Medium (-)	Density ρ (g/cm ³)	Dynamic viscosity μ (cP)	$1/\sqrt{\rho\mu}\left(\sqrt{cm^3/(g \times cP)}\right)$
Ethanol	0.7855	1.1175	1.0673
DI-H ₂ O	0.9907	1.0471	0.9818
Isopropanol	0.7812	2.1062	0.7796
D5	0.8354	4.9122	0.4936
N10	0.8476	17.235	0.2616
N35	0.8552	65.526	0.1336
N100	0.8627	238.82	0.0697



- Viscosity is defined as:
 - μ_f ...dynamic viscosity
 - τ ...shear stress
 - S ... shear rate

 \rightarrow



Cantilever in liquid is approximated by sphere

- Kinetic energy and dissipated energy can be analytically calculated for a sphere in liquids
- Q-factor is defined as $Q = 2\pi E_{kin}/E_{diss}$





- ρ_c ... density of cantilever
- *W*, *T*, *L* ... width, thickness, length of cantilever
- ω ... resonance frequency
- R_{eq}^2 ... equivalent sphere radius (approx. cantilever surface WL)
- $\sqrt{\mu_f \rho_f} \dots$ sqrt of density viscosity product

Study on the In-Plane Vibration Mode I





Piezoelectric area is too small!



Study on the In-Plane Vibration Mode II

Need to evaluate the piezoelectric area @ equal resonance frequency!

- AIN area scaling factor α









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Study on the In-Plane Vibration Mode IV





Study on the Multi Roof Tile-Shaped Vibration Mode





Different roof tile-shaped modes in water



Ord	er	Mode	f _{res} (kHz)	Q		ΔG (μS)	ΔG/Q (μS)
	1	12	53,49		55,2	6,95	0,13
	2	13	152,43		98,1	77,61	0,79
	3	14	317,3		139,8	54,94	0,39
	4	15	555,93		182,7	9,45	0,05

- Electrode shape must reflect the locally ٠ strained areas
- Electrode shape will be mode specific ٠ (to a certain degree)

Optimization of electrode design \rightarrow large increase in measurement signal

arbitrary

arbitrary unit

unit

arbitrary

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Kucera, M., et al., Applied Physics Letters 107 (2015) 053506.



Optimized vs. non-optimized electrode design

100 × higher conductance peak





Frequency f [kHz]

Pfusterschmied, G., et al., Proceedings of the IEEE International Conference on Micro Electro Mechanical Systems (MEMS), 2015.



Roof tile-shaped mode at different temperatures



- Small deviation for density (averaged 0.76% at 20 °C, 0.55% at 40 °C, 1.04% at 100 °C)
- Deviation much larger for viscosity (6.35% at 20 °C, 6.87% at 40 °C and 23.44% at 100 °C)
- Possible solution
 - \rightarrow higher order modes with optimized electrode designs
 - \rightarrow Better calibration method



Good accuracy for density, low accuracy for viscosity measurements

Pfusterschmied, G., et al., Journal of Micromechanics and Microengineering, Vol. 25, pp. 105014 (8pp), 2015.





$$P_{EH} \propto FOM = \frac{e_{31}^2}{\varepsilon_0 \varepsilon_r} \qquad P_{EH} \propto FOM \approx \frac{d_{31}^2}{\varepsilon_0 \varepsilon_r Y_1^2}$$

Evaluation

- Maximum power output
- Variation of load resistance R_L
- Frequency sweep at first Eigenmode





Vibrational Energy Harvesters – Evaluation of AIN and ScAIN II



ScAIN leads to increased power output compared to AIN

26.10.2017 Folie 39

Mayrhofer, et al., ScAIN MEMS Cantilevers for Vibrational Energy Harvesting Purposes, Journal of Microelectromechanical Systems, Vol. 26, No.1, 102-112, 2017.



Research topic:

Energy Harvesting for Wireless Sensor Nodes

Why Energy Harvesting at Aircrafts?





www.tagesschau.de, aufgerufen am 25. 9. 2011
 ICAO Data. Airline Financial Detail Report. Technical report.

AIRBUS GROUP



Dynamic Energy Harvesting:

Ground - Fuselage temperature: 20°C Cruising - Fuselage temperature: -20°C (and even lower) - Temporally restricted energy generation + easy to install

D. Samson et al., Journal of Electronic Materials, Vol. 39, No. 9, pp. 2092-2095, 2010.





COMSOL FEM simulations based on heat conduction equation to evaluate design aspects and to determine the energy output



D. Samson et al., Sensors & Actuators A, Vol. 172, pp. 240-244, 2011.

Energy Harvesting for SHM II





Energy Harvesting for SHM III







Test flight with DLR A320 D-ATRA:



A. Elefsiniotis, et al., Journal of Electronic Materials, Vol. 42, Iss. 7, pp. 2301-2305, 2013.

- Highest Altitude >30,000ft.
- Difference on outside and fuselage temperature due to aerodynamic heating
- Most energy is harvested during takeoff!
- Power peak @~17mW
- Energy Harvested:
 ✓EH Device#1: ~22J
 ✓EH Device#2: ~24J

A. Elefsiniotis, et al., Journal of Electronic Materials, Vol. 42, Iss. 7, pp. 2301-2305, 2013.





Thank you for your attention!

Questions?