

a-plane GaN Shear Wave Thin Film Resonator

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Abstract—This paper focuses on Metal-Organic-Vapor-Phase-Epitaxial (MOVPE) grown on a-plane gallium-nitride (GaN), representing a novel approach for piezoelectric materials with good prospects for Quartz-Crystal-Microbalance (QMB), like sensors with respect to its biocompatibility and frequency filter applications. Material characteristics of gallium-nitride as well as the processing of shear wave resonators and their acoustical characteristics are as well discussed.

I. INTRODUCTION

Metal-Organic-Vapor-Phase-Epitaxy (MOVPE) is a well established process for compound semiconductor layer growth especially of III-V semiconductors as InP, GaAs, and the nitrides GaN or AlN (figure 1). In the case of nitrides, one obtains the a-plane oriented piezoelectric material, well-suited for shear-microwave applications up to 10 GHz, by tilting the c-axis of the piezoelectric unit cell by 90 °C to result in a-plane oriented crystallites. The upper frequency is currently limited to the actual minimum of achievable layer thickness, where the surface is still smooth and pits-free. The lateral size of filters can be minimized due to the lower velocity of the shear wave in contrast to the longitudinal wave. A lower temperature coefficient of the frequency as for longitudinal polarized waves is expected. For sensor applications, shear wave polarized modes will allow a high mass sensitivity and a Quartz Microbalance-like behavior even in liquids.

II. METAL-ORGANIC-VAPOR-PHASE-EPITAXIE OF GAN

A. Principle of Metal-Organic-Vapor-Phase-Epitaxie

Metal organic vapor phase epitaxy, refer to figure 1, uses the high vapor pressure of selected metal-organic compounds to transport them via a carrier gas to a suited and heated substrate surface for epitaxial growth instead of evaporating the pure metal, e.g. in a vacuum as in molecular beam epitaxy. The latter requires in many cases, as e.g., for Ga and Al high temperatures, and is therefore more difficult to

handle. For the group-V-compound in III-V epitaxy, group-V-hydrides are commonly used. The simplified reaction of the GaN deposition is shown in formula 1. Whilst high purity metalorganics, hydrides and carrier gases are applied, which reduces unwanted impurities, a common contamination source is carbon from the metal organic precursors as e.g. trimethyl-gallium (TMGa). Typical growth rates are around 1 nm/s with a thickness control in the sub-monolayer range enabling, e.g., high reproducibility and high quality quantum-wells.

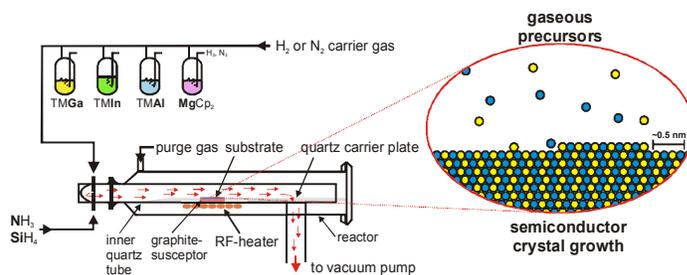


Figure 1. Principle of Metal-Organic-Vapor-Phase-Epitaxie

B. Test Setup for Metal-Organic-Vapor-Phase-Epitaxie of a-plane galliumnitrid

MOVPE growth of a 2.5 μm thick a-plane GaN layer was performed in an AIXTRON AIX 200/RF-S system with a horizontal reactor at 200 mbar and a growth temperature of 1045 °C using trimethyl-gallium (TMGa) and ammonia as source materials in a hydrogen atmosphere. To achieve the a-plane crystal orientation of gallium-nitride, polished two inch r-plane oriented sapphire substrates were used for epitaxial growth.

III. CRYSTAL PROPERTIES AND MATERIAL CHARACTERISTICS

The grown a-plane GaN layers on r-plane sapphire substrates were characterized crystallographically by x-ray diffraction (XRD). The visible different width of the two measured XRD ω scans reveal an anisotropy of the a-plane GaN tilt parallel to the c-axis or the m-axis orientation as shown in figure 2. This difference of width derives from different growth rates within these two directions with a best value of 612 arcsec. From θ 2 θ scans of the (11-20), (10-10), and (0002) reflections the lattice parameters of the crystal were calculated. The strain ϵ for the a-plane (11-20) was found to be $9.4E-4$ and the lattice constant $a_0 = 0.3189$ nm. For the m-plane (10-10) the strain is $-1.65E-3$ and the lattice constant is $m_0 = 0.866 \times a_0 = 0.2762$ nm. Finally, for the c-plane (0002), we have a value for the lattice constant c_0 of 0.5185 nm and a strain of $-1.12E-3$. The piezoelectric polarization along the (0002) direction is $-4.7E-4$ C/m² and the stress in this direction is -0.52 GPa. The stress for the (10-10) direction is -0.625 GPa. Comparisons of the numerical results reveal a compressively strained layer with values well in consensus with literature. Material data were taken from literature [1].

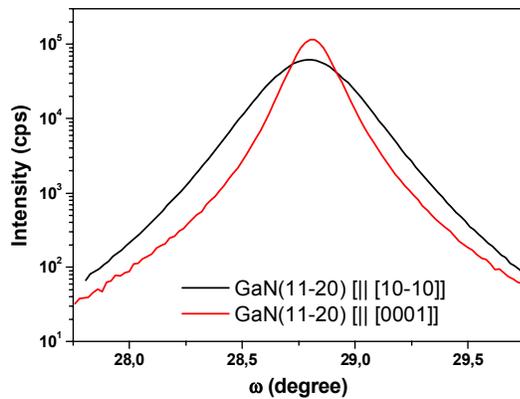


Figure 2. Rocking-curve measured by x-ray diffraction of a-plane GaN on r-plane Sapphire parallel to the m-direction [10-10] and to the c-direction [0001]

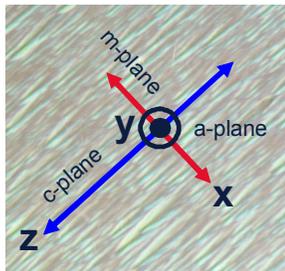


Figure 3. GaN surface with schematic crystal axis. The axis of the a-plane emerges of the paper plane

IV. PROCESSING OF RESONATORS

The resonators' process was complicated by the fact that the actual MOVPE process of a-plane GaN needs r-plane sapphire substrates. Due to the additional fact that r-plane sapphire is quite difficult to process by wet or dry etching, laser assisted processing of sapphire was chosen for membrane structuring.

At first, the a-plane GaN was grown by MOVPE atop polished two inch r-plane sapphire substrates. An excimer laser with a wavelength of 193 nm was then used to drill tapped blind holes with a diameter of 500 μ m into the sapphire substrates [2]. The remaining sapphire membranes had an averaged thickness of 20 μ m. During the laser drilling process, the formation of a conductive layer at the interface of the sapphire and the gallium-nitride layers was observed. It is supposed that the material and optical transition of the sapphire and the gallium-nitride layers (refractive index of gallium-nitride is 2.55 and of sapphire it is 1.75 [3]), absorbed a part of the laser energy and an AlGa_{N_x} highly conductive layer was formed without damaging the topside of the gallium-nitride layer, which served as floating bottom electrode for two serial resonators. A Lift-off process was used for the two half-round top electrodes, each having a radius of 250 μ m and a distance of 30 μ m to each other on the gallium-nitride surface. For the first resonators' run, the orientation of the electrodes were tilted with 45° to the crystallites in c-axis direction in order to facilitate the lithography steps on 6" inch chuck of MA6 mask aligner. Further runs will hence respect the polarization direction of the shear wave.

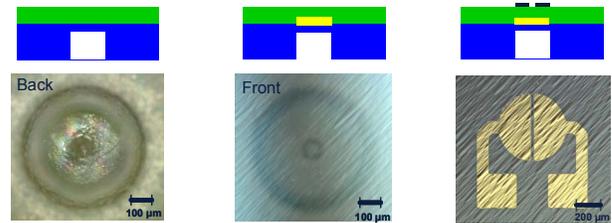


Figure 4. Main steps of MEMS processing of membrane based resonators. Left: Backside view of a $\varnothing 500\mu$ m tapped blind hole in 340 μ m thick 2" r-plane sapphire substrate with 2 μ m a-plane GaN. Middle: Topside view of MOVPE grown a-plane GaN. The tapped blind hole and the damageless surface is clearly visible. Right: Topside view of Lift-off processed gold top-electrodes forming to resonators in serie

V. ACOUSTICAL CHARACTERISTICS

A. Wave Polarization

For the determination of the polarization of the acoustic waves, the harmonic admittance of massless interdigital transducers (IDT) atop c-plane and a-plane gallium-nitride were computed according to the Blotekjaer approach [4] and are shown in figure 5. By changing the orientation of the IDTs, different wave types can be observed in a-plane gallium-nitride.

The curves in figure 5 correspond to the surface waves. The computation is achieved for a grating period equal to 5 μ m,

which corresponds to a wavelength of 10 μm and a metal ratio of a/p equal to 0.5. This yields signatures of waves within the frequency range of 100 MHz to 1 GHz. The signature of a guided wave is a real pole, corresponding to wave propagation under the grating with no loss, where loss can only be due to bulk radiation. The signature of leakage is a non-zero conductance, which corresponds to the real part of the harmonic Y .

Propagation on c-axis direction shows a nicely defined Rayleigh wave. Since the piezoelectric coupling is only capable to excite this type of wave of such a cut, it is the only possible polarization; the velocity here being typically equal to 4000 m/s, pretty high for a Rayleigh wave but slower than the one on AlN. Note also the existence of a well marked longitudinal wave signature near 8000 m/s. Trapping this wave to eliminate its losses could be an issue for high frequency applications or even for immersed sensing applications.

Propagation on the YX cut on the contrary exhibits the typical signature of a shear transversal wave, the pole being very close to the surface skinning bulk wave. This corresponds to the velocity for which the conductance becomes non zero. No longitudinal wave signature occurs, attesting that only pure shear waves can be excited on such an orientation.

The YZ cut is also considered, and found very close from the c-axis curve. Velocities are always slightly higher, and the longitudinal wave seems a little more coupled.

Finally, there is no need for higher frequency computations assuming the above parameters.

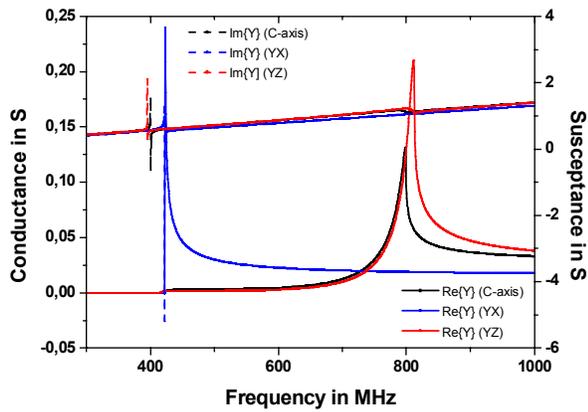


Figure 5. Harmonic admittance of massless interdigital transducers atop Galliumnitrid and a-plane were computed along the Blotekjaer approach

B. Effective Coupling Coefficient

The effective coupling coefficient k_{eff} was calculated on the base of material data supplied by [1] and following the approach of [5]. In comparison to other common piezoelectric materials, gallium-nitride shows even for the transversal polarization a low but sufficient coupling coefficient (figure 6). But its excellent biocompatibility makes gallium-nitride very interesting for bio-sensing applications and has encouraged further investigations.

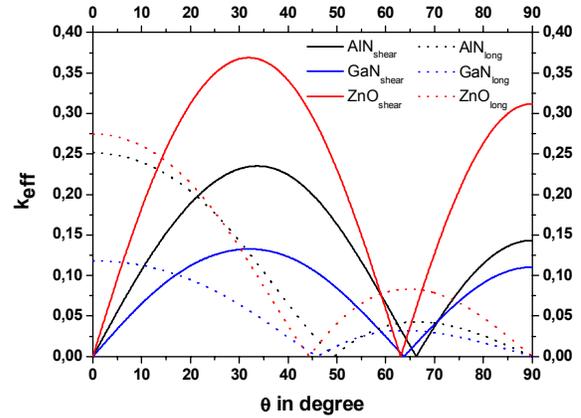


Figure 6. Calculated effective coupling coefficients for longitudinal and transversal polarization of common piezoelectric materials like ZnO and AlN in comparison to GaN

C. Measurements of Sample Resonators

A batch of five processed wafers were measured with a manual wafer probe station Cascade Summit 9000 in combination with an Agilent E5071B network analyzer in the interesting frequency range of up to 3 GHz. Resonators of one wafer sample showed that most of them have a nice resonance of the fundamental modes at around 1.5 GHz (figure 7).

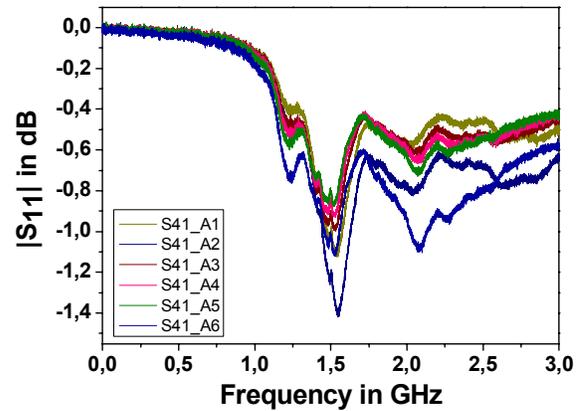


Figure 7. Resonance frequency of several resonators of one wafer. The quality factor is around a value of 8, therefore the signal is quite noisy to resolve a shape signal

In addition, the real and imaginary part of the admittance of one resonator was measured to extract key-parameters of the resonators in general. The quality factor with a value of 8 was really low. This was due to the unpolished surface of gallium-nitride, and the inhomogeneity conducting AlGaIn_x floating bottom-electrode. The remaining parameters were calculated with the Mason model approach. C_0 was found at 74 fF, C_1 was equal to 25 fF, a really high R_1 was found at 590 Ω and the inductivity L_1 at 540 nH. Despite the poor quality of these parameters, the feasibility of pure shear wave

resonators is encouraging for improvement and a better reproducibility for these kinds of resonators.

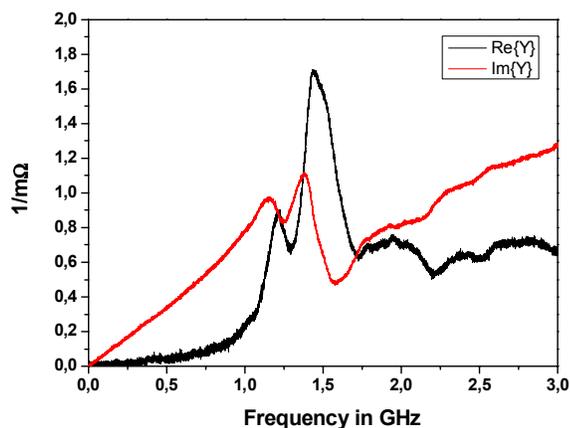


Figure 8. Measured admittance of resonator S41_A6

VI. CHEMICAL IDENTITY AND BIOCOMPATIBILITY

A. X-Ray Photoelectron Spectroscopy

The GaN substrate was characterized with respect to the chemical identity by X-ray Photoelectron Spectroscopy. To investigate the stability of these materials against biological environments, a gallium-nitride sample was put into serum for 24 hours. Figure 9, shows the elemental composition of the GaN substrate after treatment with the serum. The peaks of gallium and nitrogen correspond to the substrate itself indicating the stability of the substrate. Additional peaks (O and C) are also visible, mostly originating from contamination (for example air) of the measurement chambers, which is normal and common for such systems. A part of the intensity of the C-peaks originates from the MOVPE process itself. Carbon represents a major impurity atom in the crystal lattice with a low concentration, limited to value in around $1E18\text{ cm}^{-3}$. However, as it is visible from figure 9, we can say that our substrate is stable against physiological conditions. This is actually interesting and promising for biosensor applications.

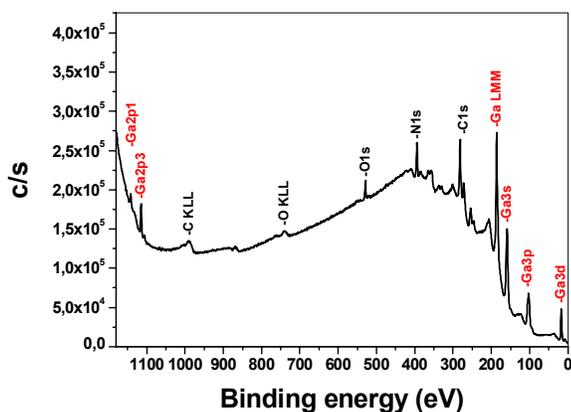


Figure 9. X-ray Photoelectron Spectroscopy of a gallium nitride sample.

B. Cell-adhesion on GaN surfaces

In a next step, we investigated the biocompatibility of our system. For this, cell-adhesion assays were performed in serum-free culture media. Human skin fibroblast cells and endothelial cells were used as model-cells. For the cell-adhesion assays, substrates were placed in a six-well plate, and covered with media. A known amount of cells were suspended, and incubated with substrates for one hour. After this time, non-adherent cells were washed-off from the surface. Micrographs were taken, and cells were counted. The medium was replaced by a medium having growth factors (serum), and the substrates were further incubated at $37\text{ }^{\circ}\text{C}$ for several days.

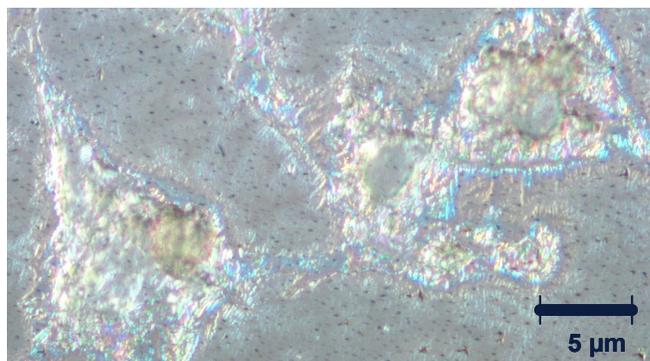


Figure 10. Human endothelial cells after 24h on gallium nitride

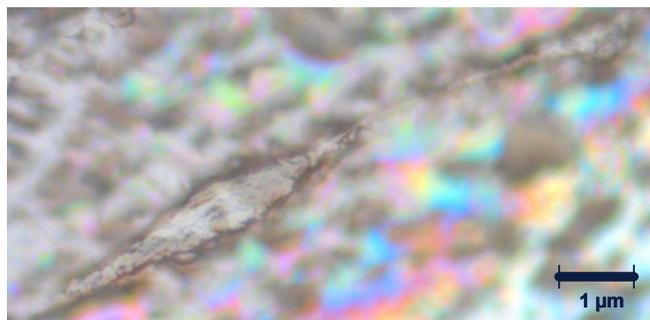


Figure 11. Human fibroblast cells after 24h on gallium nitride

From the micrographs, figures 10 and 11, it is visible that cells adhered to the GaN surface. Attached cells were similar to the cells which adhere to the culture-plate, indicating that above this concentration, cells appear to be “feeling well” on such surfaces.

VII. SUMMARY

MOVPE growth a-plane gallium-nitride was investigated under the aspects of use as piezoelectric layer for thin film resonators. The crystallographic characterization indicates high-textured and well-oriented crystallites which will in future allow high Q resonators. The lower wave velocity is suitable for further miniaturized frequency filters. The shown biocompatibility of gallium-nitride is, although the low efficient coupling factor quit, interesting for bio sensor applications.

Our ongoing work will focus on a facilitated processability of resonators, a deeper understanding of wave propagations and investigating MOVPE growth of a-plane oriented highly textured piezoelectric materials.

ACKNOWLEDGMENT

R. B. G. thanks to Katja Samm, Anke Wörz, David Eisele, Bernd Neubig, the staff of IMTEK-RSC and especially to Sylvain Ballandras for scientific discussions concerning process technology, cell assays and simulations.

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