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à la Faculté des sciences de base
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programme doctoral en SuperScience
École polytechnique fédérale de Lausanne

pour l'obtention du grade de Docteur ès Sciences
par

Paolino Paperino

acceptée sur proposition du jury :

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Prof Name Surname, directeur de thèse
Prof Name Surname, rapporteur
Prof Name Surname, rapporteur
Prof Name Surname, rapporteur

Lausanne, EPFL, 2020

Wings are a constraint that makes
it possible to fly.
— Robert Bringhurst

To my parents…

Preface

A preface is not mandatory. It would typically be written by some other person (eg your thesis director).

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Lausanne, 12 Mars 2011 T. D.

Abstract

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Contents

1 Introduction

1.1 AlScN as the material of the future

Since the beginning of the decade Aluminum-Scandium Nitride became more and more a common buzzword in scientific publications. The main reasons behind the increased interest on this novel materials are two: IC fabrication compatibility and most importantly increased piezoelectric coupling.

1.2 Material properties

The first occurrency of an alloying of aluminum and scandium was in 1971 (1) and its first application was to improve the strain resistance of Al in aeronautics application. With the advent of miniaturisation in FR front-ends the bulky Quartz crystal started to be replace by MEMS-based resonator such as SAW, BAW, or CMR. The reason being that MEMS devices have a higher throughput and a lower footprint than quartz crystals, allowing for batch fabrication and integrability, still keeping the higher Q that mechanical systems show compared to LC tanks. In parallel to the architecture evolution of RF MEMS a range of new materials to replace quartz have been investigated, PZT, ZnO, AlN. The latter is the most important one to understand how the evolution of AlScN.

1.2.1 AlN

Aluminum Nitride is a binary nitride ceramic whose wirtzite phase exhibits piezoelectric properties. AlN can be deposited on a proper seed layer so that the growth is oriented in the c-axis (the perpendicular to surface axis) in a wurtzite phase. The crystal phase depends on the seed layer over which AlN is deposited. Literature (2) shows that the preferential growth substrate material for AlN is Pt, due to the lattice amtching between the Hexagonal structure of AlN and the cubic phase of Pt.

1.3 Deposition of AlScN

Piezoelectricity in AlN is a consequence of the dipolar nature of the crystalline cell of wurtzite-type crystal. From the first studies using DFT (1) (3) show that the doping of aluminum with scandium increases the piezoelectric coupling coefficient of AlN. The reason lies in the lattice distortion induced by Sc in AlN, causing a structural phase transition. According to (3) a second effect on the piezoelectricity improvement lies in the hybridisation of ionic into covalent bond, due to the lower electronegativity of Sc compared to Al (1.36 vs 1.61). An increase of the concentration of Sc results in an enhancement of response up to 43% Sc followed by a drastic performance drop due to the crystallisation in a rock-salt struture typical of Scandium rather than the wurtzite lattice of AlN. The actual state-of art includes depositions carried with Sputtering (4) (5) (6), co-sputtering (3), Molecular Beam Epitaxy (7) (8) (9), Metal-Organic CVD (10)

1.3.1 Sputtering of AlScN

The first and most simple to describe method for AlScN deposition is sputtering from an alloyed target using reactive sputtering. In this case the Al-Sc percentages are decided during the target fabrication. This technology has been used to fabricate targets with 17.5% Sc concentration (11), 20% Sc concentration (5), a various range of concentration from 6.5% to 28% (4), 40% (12). The upper bound of 40% for a sputtering target comes from the difficulty in alloying aluminum and scandium to form a uniform target, which is critical for a high quality deposition. The absence of higher concentration AlSc targets is nevertheless not critical because according to (13) at a concentration higher than 43% scandium AlScN loses their benefits in term of piezoelectric response. Sputtering is carried out in a chamber with a variable concentration of reactive Nitrogen and inert Argon. Piezoelectric response in thin films depends on the percentage of nitrogen in the sputtering athmosphere (14) as the Al and Sc ions react with N to deposit AlScN films.

1.3.2 Co-Sputtering of AlScN

To overcome the absence of targets, or more in general to achieve a larger flexibility in the Al-Sc ratio, Co-sputtering is a solution. Rather than using a single alloyed target a dual set-up machine with multiple targets allow to change the film composition by changing the sputtering power of each target. The delicate part of co-sputtering lies in surface uniformity as the sputtered particles are impacting the wafer from different angles. In literature a Sc percentage up to 46% (15) has been reached using co-sputtering.

1.3.3 MBE of AlScN

Being sputtering and co-sputtering the most widely used methods for AlScN depostion the majoritz of literature follows these two approaches. Nevertheless, in cases when it is necessary to achieve high cristallinity, a more advanced approach using molecular beam epitaxy has been sused (16). In the quoted example, AlScN thin film was grown on a buffer GaN layer over SiC for a HEMT structure.

1.4 Types of MEMS Resonator

Body 1 Part I

2 State of research

In this chapter we will see some examples of tables and figures.

2.1 Fabrication of AlScN based Contour Mode resonators

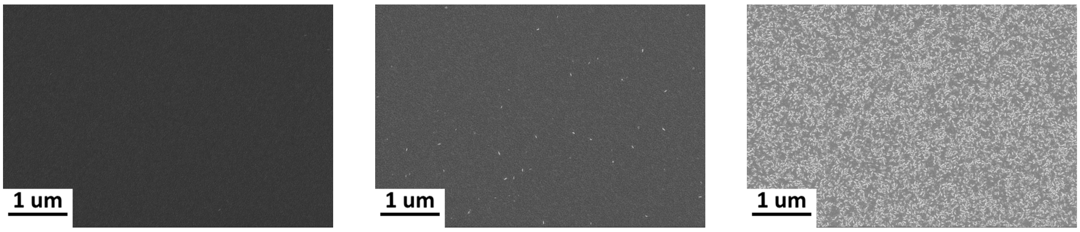
After the results achieved by Lozzi (11) with 17.5% Sc AlScN the objective has shifted to optimie fabrication

2.1.1 Sputtering deposition optimisaton

Carried over from the doctoral studies of Kaitlin M. Howell (17) the first objective of research in AlScN technologies is to optimize the deposition of AlScN in the Spider600 cluster tool at CMi. The Spider600 is a sputtering cluster which allows deposition from different targets in the same fabrication round, without breaking the vacuum between layers. This tools allows or a reactive sputtering of full stacks of the resonator structure. To optimize the deposition three main results were kept in consideration: density of abnormal oriented grains on an SEM image, Rocking curve of X-ray diffraction and measured coupling coefficient k_t^2 to gain as much insight as possible on the process.

2.1.2 Effect of gas flows

it is possible to set two gas flows in the Spider600, Argon as the sputtering gas for the target and Nitrogen as the reactive gas that will form the AlScN film. Three different combination of gas flows were tested: 0/50 10/40 20/30 (Ar gas flux in sccm / N_2 gas flux in sccm). Analysis at the SEM shows that an increase of argon results in a lower density of AOG (see Figure 2.1). SEM does not tell the whole story though, as XRD is used to characterize the crystal orientation of the film. It is shown from XRD that the rocking curve



(a) 20/30 (b) 10/40 (c) 0/50

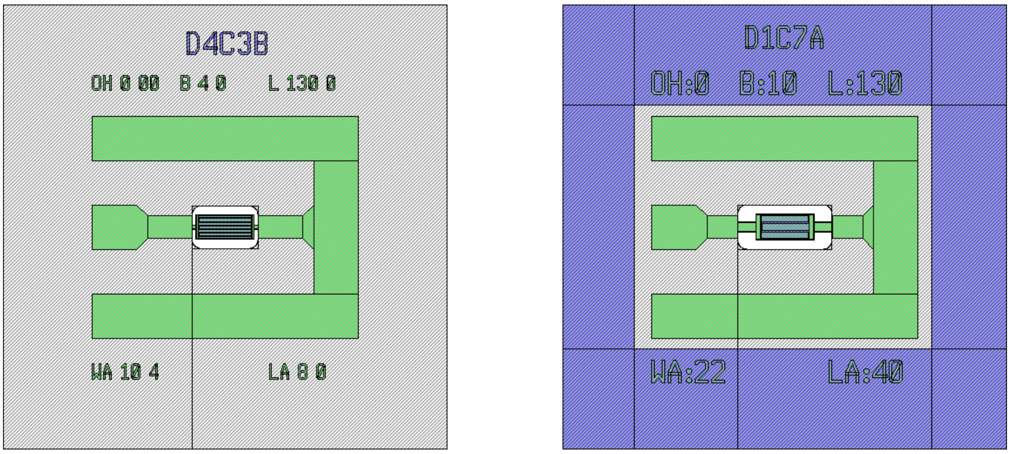
Figure 2.1: Abnormally oriented grains as function of gas flow rates (sccm Ar / sccm N_2)

2.1.3 Effect of biasing power in high-resistive wafers

In multiple occasion, both with $Al_{0.6}Sc_{0.4}N$ and with $Al_{0.83}Sc_{0.17}N$ it was noticed that according to the substrate of the deposition the k_t^2 was affected. Under the same fabrication process, High resistive (HR) wafers showed a systematic lower coupling compared to standard Test wafers. The Test wafers are SSP 100mm <100> with resistivity of max 100 Ωcm. HR wafers are DSP 100mm <100> with resistivity higher than 10k Ωcm. According to (12) an increase of RF bias power in deposition will lead to a random growth of crystal. it was our belief that in the case of HR wafers the extra resistance of the wafer has to be offset by increasing the bias power, on a range of 3, 4, 6 and 8 W. After XRD analysis it is shown that the theta2theta peak at 36° (Corresponding to (0002) AlScN) has the maximum count intensity at 4W compared to the 2W of the paper, while according to the previous results the crystalline peak disappears with powers higher than 6W. On the side of rocking curve the broadening of the FWHM is proportional to the power. According to this first measurement the growth on an HR wafer benefits from a bias increase but there is a tradeoff between the crystallinity of the film (given from the theta2theta) and the ordered vertical growth of it (given by the Rocking Curve). Investigation is ongoing to find the soft point.

2.1.4 Effects of bottom electrode coverage

According to literature (**?**) (2) the best condition for the growth of an AlN film is to have a seed layer of Pt, that will act also as a bottom electrode to actuate the device. Since adhesion of Pt to the substrate is extremely low, an adhesion layer of Ti is usually employed to solve the problem. The best possible condition for crystal growth happens when the whole wafer is coated with Pt, but this is detrimental for the device performances, because this very large bottom electrode will introduce enormous parasitic capacitances that will reduced the electrical k_t^2. in previous iterations of the project the bottom electrode was patterned with Lift-off so that the Pt electrode would be only below the main resonator body. This meant that while the more "important" regions of the wafers were grown over a Pt substrate, the vas majority of the AlScN was grown on bare Si.



(a) Low coverage (b) High coverage

Figure 2.2: Different coverage approaches to design of resonator. Parasitics rise when the top metal (green) overlaps with the bottom (blue)

2.1.5 AlScN on AlN

In the strive to achieve a better cristallinity for $Al_{0.6}Sc_{0.4}N$ another choice was to follow the approach proposed in (18) for $Al_{0.83}Sc_{0.17}N$. The underlining idea is that the crystalline growth of AlScN can be promoted by a thin film of AlN above the bottom Pt electrode. A batch of 6 wafers was prepared, 2 test wafers and 4 HR wafers, at different Bias power with patterned and non-patterned bottom electrode. Coherently to the result shown in 2.1.3 XRD measurements show that on both Test Wafers and HR wafers, at the same power level and substrate type, the rocking curve peak is narrower for the AlScN wafers where a growth promotion layer of AlN was put. Interesting, the theta2theta peak is higher in AlN-buffered AlScN only in the wafers with the lowest power. In wafers with the higher bias power the AlN-buffered depostions have a lower theta2theta peak compared to the ones directly deposited on the bottom Pt electrode. The main interest in this technique is not only related to the improvement of film quality over Si substrates, but can help in case of non crystalline substrates, as in the case of (18) where AlScN was deposited over a SiO_2 layer.

2.1.6 Inductively coupled plasma etching

2.2 CMR based oscillator

2.3 Published Papers

- A.Lozzi, M. Liffredo *et als* "Evidence of Smaller 1/F Noise in AlScN-Based Oscillators Compared to AlN-Based Oscillators" (19)

2.4 Attended Courses

- Scanning electron microscopy techniques
- Techniques for handling noise and variability in analog circuits
- Energy efficient autonomous wireless devices
- Design of Experiments
- Enterpreneurial Opportunities Identification and Exploitation
- Piezoelectric Materials Properties and Devices

Body 2 Part II

3 Future research

3.1 N77 N78 and N79 band

The goal of this phd is to exploit the high piezoelectric coupling coefficient of AlScN to satisfy the requirements for the New Radio standards of 5G bands in terms of required fractional bandwidth. From the mBVD model that characterises a

3.1.1 Stepper-based fabrication of electrodes

3.2 AlScN on non-Si substrates

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$$\frac{\mathrm{d}}{\mathrm{d}t}\begin{bmatrix} P_0 \\ P_1 \\ P_T \end{bmatrix} = \begin{bmatrix} \frac{P_1}{\tau_{10}} + \frac{P_T}{\tau_T} - \frac{P_0}{\tau_{ex}} \\ -\frac{P_1}{\tau_{10}} - \frac{P_1}{\tau_{isc}} + \frac{P_0}{\tau_{ex}} \\ \frac{P_1}{\tau_{isc}} - \frac{P_T}{\tau_T} \end{bmatrix} \tag{3.1}$$

$$\bar{I}_f(\vec{r}) = \gamma(\vec{r})\left(1 - \frac{\tau_T P_T^{eq}\left(1 - \exp\left(-\frac{(T_p - t_p)}{\tau_T}\right)\right)}{1 - \exp\left(-\frac{(T_p - t_p)}{\tau_T} + k_2 t_p\right)} \times \frac{\left(\exp\left(k_2 t_p\right) - 1\right)}{t_p}\right) \tag{3.2}$$

4 Another chapter

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A An appendix

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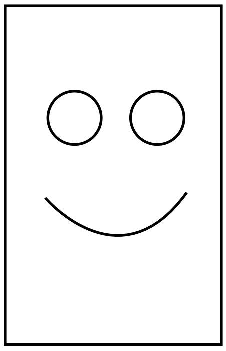

Name :	Mr. Sample CV
Address :	Samplestreet 70 6005 Luzern Switzerland
Date of Birth :	2nd of October 1981
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Legally work :	legally work in EU
Marital status :	with partner
Children :	none
Languages :	Chinese/Mandarin, English, French, German
Education level :	Bachelors degree
Hospitality work experience :	3-5 years
Special experience :	Europe work experience
Date of availability :	September 2009
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Position(s) sought :	Permanent position for graduates
Department(s) sought :	Food & Beverage Bar/Sommelier

Personal profile:

As a Bachelor of Business Administration and after obtaining first relevant international work experience within the hospitality industry, I am now ready to take on new responsibilities to further my professional career.
My key strengths include strong analytical and logical skills, an eye for detail, communication and interpersonal skills.
I enjoy working in a team and help others progress. At the same time I work well independently.
As a highly motivated and driven individual I strive on taking up challenges.

Interests:

Travelling
Foreign Cultures
Photography
Sports

Educational qualifications:

Oct 99 - Feb 02	Higher Diploma (Hotel Management) Swiss Hotelmanagement School, SHL

Employment history:

Mar 04 - Ongoing	Assistant Manager (Rooms Division/Food & Beverage) Hotel Atlantic Kempinski Hamburg www.kempinski.com 5 star business hotel, part of Leading Hotels of the World 412 guest rooms, large function facilities, 3 food & beverage outlets Optimization of bar procedures, reinforcing SOPs Developing & implementing promotions Responsible for day-to-day operations Optimization and streamlining of housekeeping and laundry procedures Implementation of new SOPs Analyzing monthly reports for rooms division performance and sub departments
Mar 03 - Mar 04	Management Trainee Hospitality Graduate Recruitment www.h-g-r.com Leading company for placements within the Hospitality industry. Traineeship covering all aspects of an online recruitment agency.
Mar 02 - Mar 03	Management Trainee (Rooms Division) Hyatt Regency Xian, China www.hyatt.com 5 star business hotel 404 guest rooms, 4 food & beverage outlets Traineeship covering all rooms division departments on operational as well as supervisory level.

Training courses attended:

Mar 02 - Ongoing	OpenOffice - IT Courses
May 01 - Jan 03	Language Course - Chinese

References:

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Hospitality Graduate Recruitment
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