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Investigations of AlN thin films crystalline properties in a wide temperature range by *in situ* X-ray diffraction measurements: correlation with AlN/Sapphire-based SAW structures performance.

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Abstract

Aluminum nitride (AlN) on sapphire is a promising substrate for surface acoustic wave (SAW) sensors operating at high temperatures and high frequencies. To get a measure of the suitability and the temperature stability of such devices, an experimental relationship between the SAW performance and the structural properties of the AlN thin films was investigated in the temperature range between the ambient and 1000°C. The crystalline structure of the AlN films was examined *in situ* versus temperature by X-ray diffraction (XRD). The results reveal that the AlN films remain (002) oriented even at high temperatures. A gradual increase of the tensile stress in the film due to the thermal expansion mismatch with the substrate has been observed. This increase accelerates around 600°C as the AlN film crystalline quality improves. This phenomenon could explain the amelioration in the SAW performance of AlN/Sapphire devices observed previously between 600 and 850°C. At higher temperatures, surface oxidation of the AlN films reduces the SAW performance. The potential of ZnO thin films as protective layers was finally examined.

Keywords: *Aluminum nitride, zinc oxide, surface acoustic wave, high-temperature, protective*

layer.

I. Introduction

Surface acoustic waves (SAW) technology constitutes a promising way to achieve passive wireless sensors able to operate at high temperatures ranging from 200°C to estimated 1000°C. The piezoelectric material constituting such SAW sensors should withstand high temperatures while allowing high operating frequency ranges and having large electromechanical coupling factor (K^2). Materials in the form of thin films such as zinc oxide (ZnO) [1] and aluminum nitride (AlN) [2,3] have been proposed for those applications. In previous works, we have experimentally demonstrated the potential of the c-oriented AlN/Sapphire SAW structure as a strong candidate for high temperature applications up to 1000°C [4], possibly coupled to high operating frequencies [5]. *In situ* measurements of S-parameters revealed that AlN/Sapphire SAW devices exhibit a large and quasi-linear sensitivity to the temperature in the whole range from the ambient to 1000°C. It was also observed that the insertion losses (IL) of the investigated delay lines are fairly constant with small variations below 3 dB in the same temperature range. However, the unexpected decrease of IL observed between 600 and 850°C is still unexplained. Therefore, the first aim of this work is to investigate the evolution with temperature of the AlN thin films crystalline properties to understand this phenomenon. Moreover, as the main drawback of AlN regarding high-temperature SAW applications concerns its sensitivity to oxidation in air atmosphere from 700°C on [6], the second part of the paper explores the potential of ZnO as a passivation layer to protect AlN films at high temperatures.

II. Experimental details

II.1 Thin film deposition

1300 nm-thick (002) AlN films were epitaxially grown on c-plane sapphire substrates by

magnetron sputtering technique in Ar/N₂ gas mixture. The experimental details are fully described in Ref. [7]. 10 nm-thick titanium as an adhesion layer and 100 nm-thick iridium films as electrodes were deposited by e-beam evaporation method onto the AlN surface. SAW delay lines were then fabricated by photolithography and ion beam etching [4]. For the protected structure, a 500 nm-thick ZnO protective layer was deposited on some devices by radio frequency (RF) magnetron sputtering. A 99.9999% pure ZnO target was used. The distance between the target and substrate holder was 80 mm. The system was pumped to a base pressure of $6 \cdot 10^{-7}$ mbar with a turbo-molecular pump. The sputtering system employed an unbalanced magnetron cathode powered by a 100 W RF supply. Before deposition, the target was cleaned using argon and oxygen gases discharge plasma for 50 min using the same conditions as the subsequent film deposition with a shutter shielding the sample. The total gas (Ar + O₂) flux rate during deposition was fixed to 16 sccm and the substrate temperature during deposition was maintained constant at 350°C.

II.2. Thin film characterizations

In situ high-temperature θ/θ XRD measurements were performed with a Bruker D8 diffractometer, equipped with a lynxeye PSD detector and using the CoK α radiation ($\lambda = 0.179026$ nm). The X-ray data were collected at different temperatures ranging from 50 to 1000°C, with a step size of 25°C. Actually, for comparison reasons, the temperature profile was exactly the same as the one used in Ref. [4] in which *in situ* S-parameters measurements were performed. However, note that the chamber was pumped to reach a pressure of $2.6 \cdot 10^{-2}$ mbar (instead of $2 \cdot 10^{-4}$ mbar for IL in-situ measurements). Secondary ion mass spectroscopy (SIMS) measurements were also conducted before and after the annealing process with a Cameca IMS-7F equipment, in order to compare the chemical depth profiles evolution of both unprotected and ZnO-protected AlN/Sapphire samples.

III. Results and discussion

The investigated AlN films were thoroughly studied at room temperature in Ref. [7]. They are fully (002)-oriented, with a rocking-curve width of the (002) AlN XRD peak as low as 0.3° . Fig. 1 shows the evolution with temperature of the XRD pattern around the (002) AlN peak during *in situ* XRD measurements.

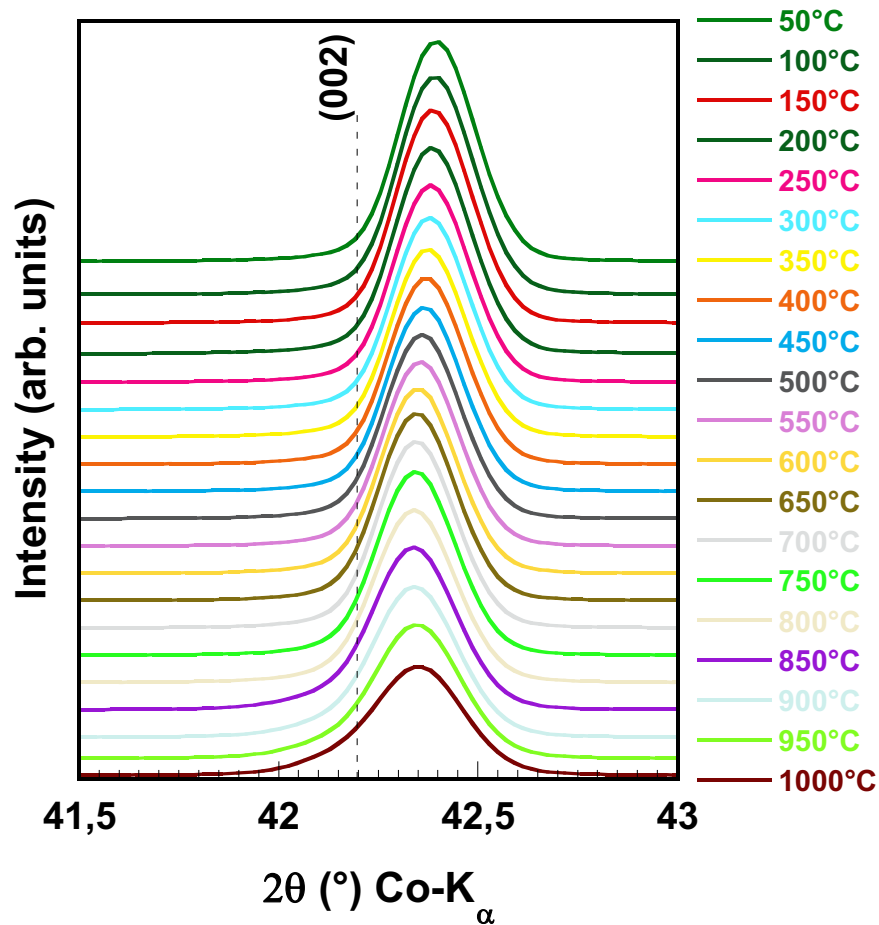


Figure 1: *In situ* θ/θ AlN films XRD patterns (the dotted line at $2\theta = 42.2^\circ$ indicates the peak position for a standard AlN powder)

It appears that the films remain (002)-oriented even at high temperatures. Two more phenomena can be qualitatively observed on this figure. First, the peak decreases in intensity with temperature. This phenomenon, known as Debye-Waller effect, is related to the augmentation

of the lattice vibrations with temperature [8]. We will study this evolution more quantitatively thereafter as it could also be obviously related to any evolution in the AlN thin film properties. The second qualitative effect visible on Fig. 1 is the gradual shift of the peak towards lower diffraction angles. According to the Bragg law, smaller diffraction angles means higher lattice parameter. Thus, this effect is related to the thermal expansion of the AlN films when heated. Again, it is necessary to have a closer look on this phenomenon to extract any significant information on the AlN thin films behavior during heating process.

Fig. 2 shows the AlN c-lattice parameter (deducted from the angular position of the maximum of the (002) AlN XRD peak) as a function of the annealing temperature. The room-temperature c-lattice parameter value was found smaller than that reported for bulk AlN single crystals ($c = 4.979 \text{ \AA}$ [9]), which means that the as-deposited AlN films undergo tensile stress. Fig. 2 points out that this stress expands when the temperature rises because of the thermal expansion mismatch between the film and the substrate [10].

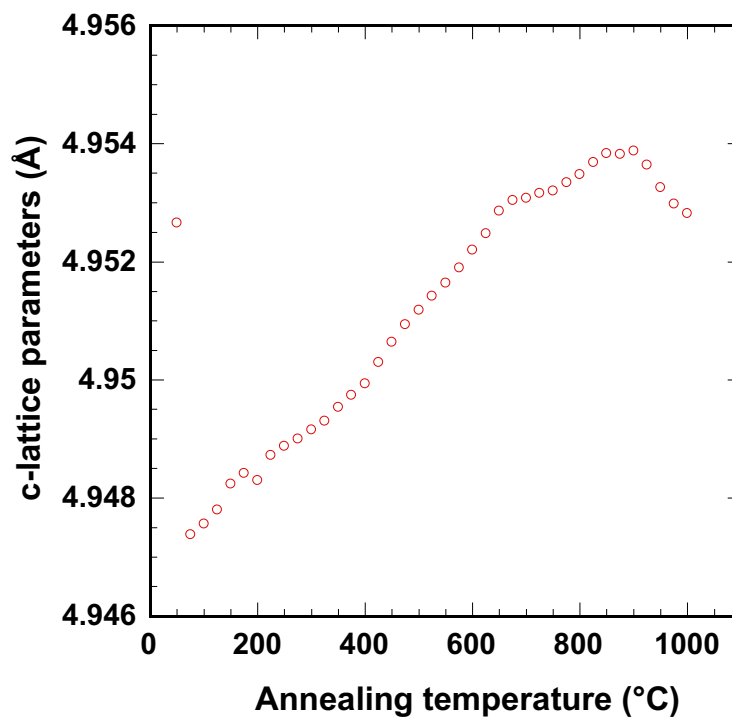


Figure 2: Variation of the c-lattice parameter of the AlN film with temperature

Table I: Room-temperature thermal expansion coefficients (TEC) of AlN and Sapphire bulk crystals [11]

	a-direction TEC (ppm/°C)	c-direction TEC (ppm/°C)
AlN	5.27	4.15
Sapphire	7.28	8.11

Indeed, three different regions can be made out in Fig. 2. Between the ambient and about 650°C, the AlN film thermal expansion is quite linear, with an average c-direction thermal expansion coefficient of 1.8 ppm/°C, that is more than twice smaller than the one of AlN bulk crystal (Tab. I). Hence the film tensile stress steadily increases in this temperature range. This phenomenon can be explained as follows. As the in-plane thermal expansion of the film is mainly driven by the substrate expansion, and the in-plane thermal expansion coefficient of sapphire is larger than AlN one, then the film will be steadily stretched in the plane, and thus compressed out of the plane. This out-of-plane extrinsic compression hinders the intrinsic thermal expansion of the film when temperature rises and thus lowers the c-direction thermal expansion coefficient of the film. A significant reduction in the curve slope occurs at 650°C, indicating a possible modification of the film microstructure, and an even faster increase of the tensile stress with temperature. Finally, the c-lattice parameter starts decreasing from 900°C, representative of a main alteration of the thin film microstructure. We assess that this phenomenon is related to the surface oxidation of AlN films occurring at high temperatures even under low-oxygen environments [4].

In order to establish a correlation between the AlN film structural properties and the SAW device performance, both the variations with temperature of the net peak area of the (002) AlN XRD peak and those of the relative insertion losses (IL curve was reported in a previous study [4]) are shown in Fig. 3. The decrease of the net peak area between the ambient and 500°C is

likely related to the aforementioned Debye-Waller effect. The net peak area then stabilizes, and even starts increasing from 650°C, indicating a significant improvement of the AlN film crystalline quality. This observation matches well with the slope change visible at the same temperature on Fig. 2. However, the reason why the slope curve in Fig. 2 decreases (indicating that the film c-direction expansion is even more influenced by the substrate) when the improvement in crystalline quality occurs is still unclear. The final drop of the net peak area from 900°C supports as well the results presented on Fig. 2, and strengthens the hypothesis that the AlN film oxidation process starts in the neighborhood of 900°C under this low pressure environment.

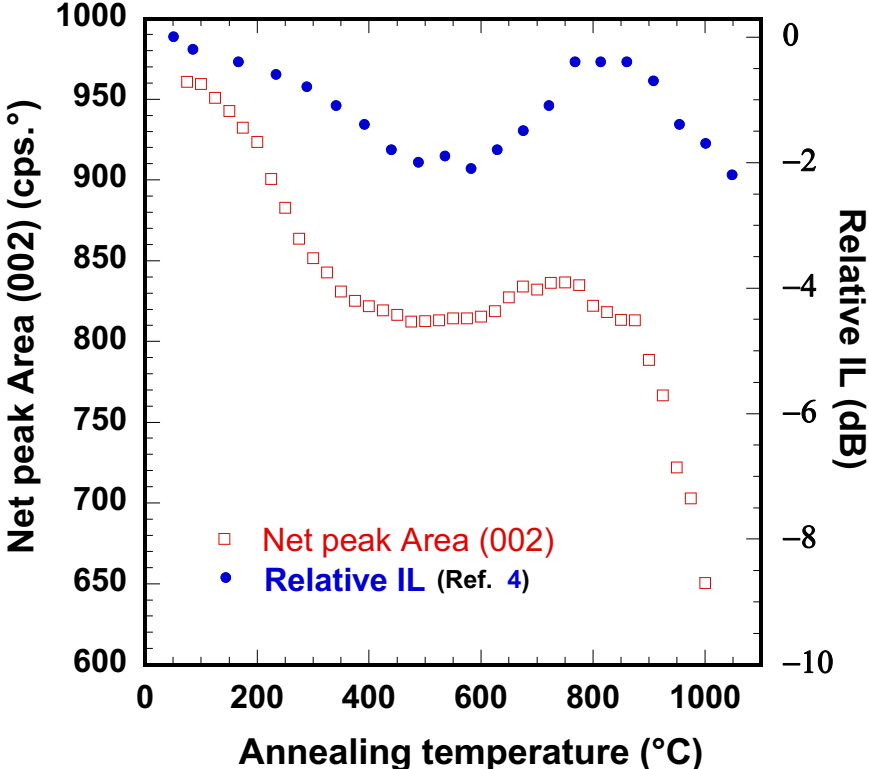


Figure 3: Evolution of the relative IL [4] and the net area of the (002) AlN XRD peak with annealing temperature

Finally, one can observe on Fig. 3 a significant correlation between both plotted curves. At lower temperatures (up to around 500°C), the net peak area decreases as the X-ray photons are more and more scattered by phonons when the temperature rises (Debye-Waller effect). In the

same way, as shown in Ref [6], IL evolution in this temperature range is mainly driven by the propagation losses rise which also comes from the increasing interaction between the surface wave and the thermal lattice vibrations [12]. This phenomenon prevails over the concomitant K^2 increase which was experimentally demonstrated up to 300 °C in Ref. [6]. In contrast, the observed IL decrease between 600 and 800°C remains difficult to interpret on the unique basis of the *in situ* θ/θ XRD measurements presented in this paper. However several assumptions can be made:

- K^2 likely continues growing above 300°C, and this increase could accelerate from 600°C. This hypothesis is drawn from three observations. First, the AlN/sapphire bilayer structure steadily softens with temperature, as evidenced by the quasi-constant negative TCF value of -71 ppm/°C between the ambient and 1050°C [4]. In the same time, the increasing stress evidenced by the present study should lead to an augmentation of the AlN thin film piezoelectric coefficients with temperature, which has been experimentally demonstrated up to 300°C [13]. This phenomenon accelerates in the neighborhood of 600°C as revealed by the slope change on Fig. 2. Moreover, the improvement in the crystalline quality observed in the same temperature range (augmentation of the XRD peak area) also participates to the assumed increase of the piezoelectric coefficients.

- The better AlN crystalline quality observed around 600°C could also lead to a reduction of the SAW propagation losses in the film.

- At last, the IL decrease could also be related to the observed recrystallization of the Ir electrodes around 600°C, which results in a diminution of their electrical resistivity [14].

Further investigations have to be conducted in order to determine experimentally K^2 evolution between 300 and 800°C, as well as that of the electrodes impedance (which is influenced either by K^2 and the Ir resistivity). Subsequent simulation of the device performance should then determine the actual origin of the IL decrease between 600 and 800°C.

In the last part of the experiment, surface oxidation process occurs, leading to a drop in the XRD peak area accompanied by an increase of IL. This oxidation is evidenced by SIMS depth profiles as shown in Fig. 4. Indeed, before annealing, the oxygen presence is only limited to the shallow surface with a native oxide layer of some nanometers [6]. After the annealing process, the oxide overlayer thickness is roughly estimated to be about 400 nm.

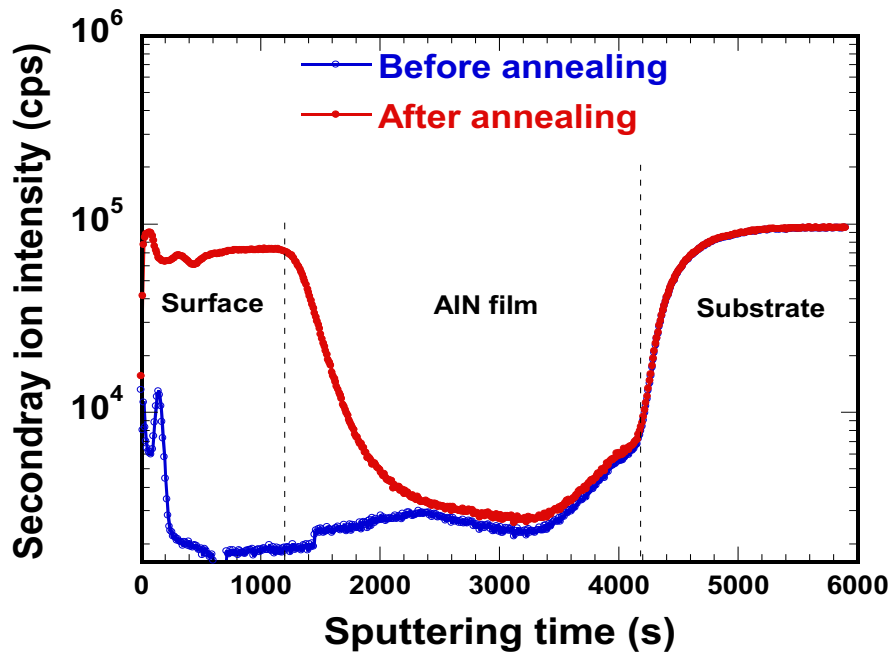


Figure 4: SIMS depth profiles of oxygen element before and after annealing

In order to protect the AlN film of this oxidation process and then preserve its piezoelectric and acoustic properties, a 500 nm-thick ZnO film was deposited on top of the AlN film as shown in the inset of Fig. 5. The new structure was also characterized by *in situ* high-temperature XRD measurements, using the same temperature profile. One can observe that the (002) AlN XRD peak intensity remains almost constant even when the temperature exceeds 900°C, thus demonstrating the benefit of the protective layer (Fig. 5). For more clarity, Fig. 6 shows a comparison between the evolution of this peak intensity in both cases: protected and unprotected AlN/Sapphire structures.

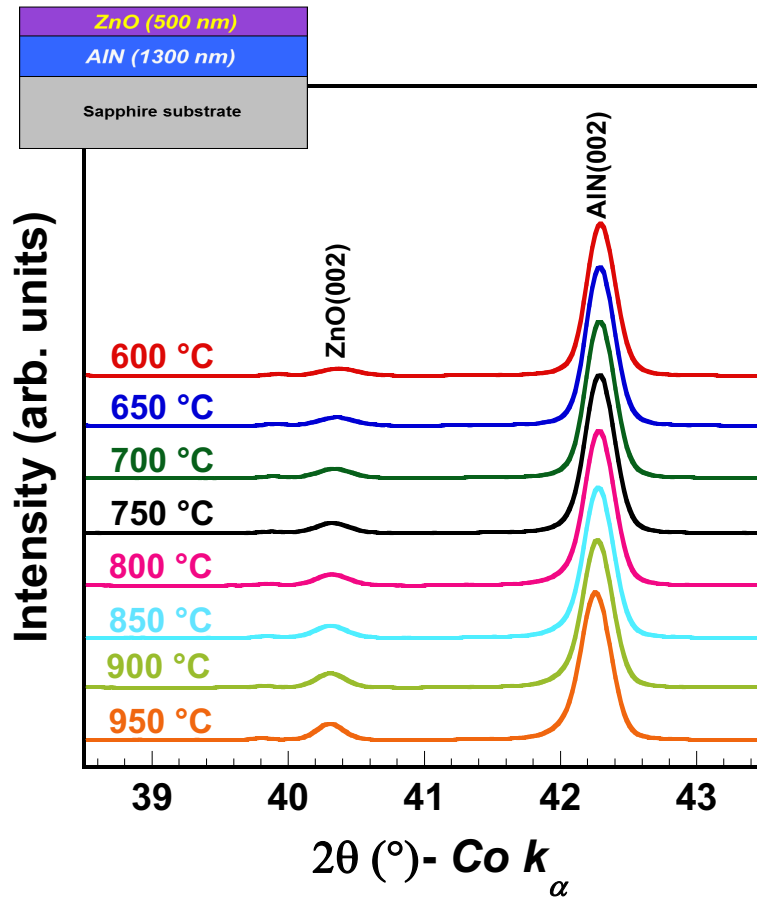


Figure 5: *In situ* θ/θ XRD patterns of the ZnO/AlN/Sapphire samples (schematic representation of the ZnO/AlN/Sapphire structure in the inset).

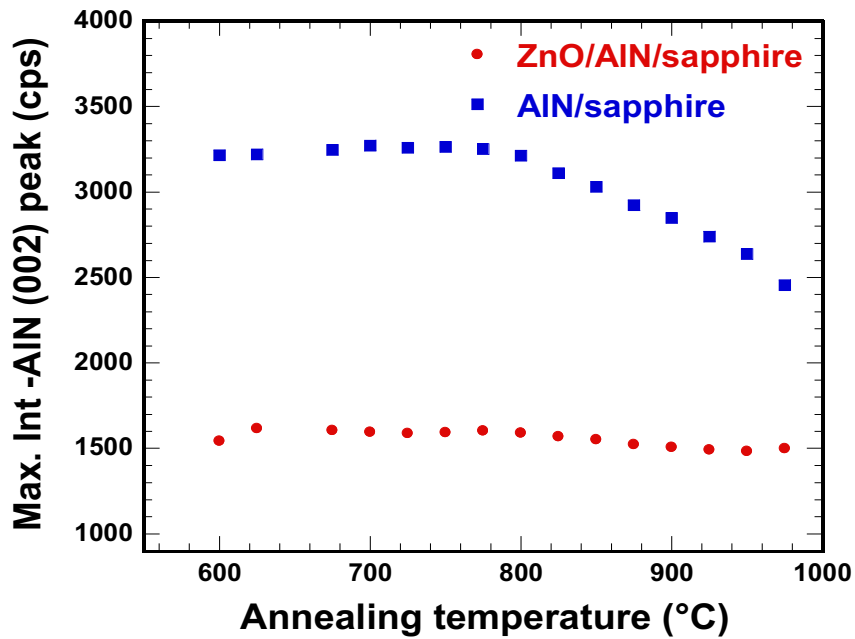


Figure 6: Evolution with temperature of the maximum intensity of the (002) peak of unprotected (square) and ZnO-protected (circle) AlN films.

This new ZnO/AlN/Sapphire structure was also investigated by SIMS analysis. The depth profiles of oxygen (O), zinc (Zn), aluminum (Al) and, nitrogen (N) elements obtained before and after annealing process are respectively shown in Fig. 7-a and 7-b. One can observe that the transition region between the ZnO and AlN films widens significantly during the annealing treatment. The major part of this transition area (etching time between 2000 and 3200 s) is completely N-depleted. As the ZnO film thickness seems mostly unchanged, this means that the transition region corresponds actually to the upper part of the initial AlN layer in which N atoms would have been replaced by O ones. These atoms could come either from the ZnO layer, and/or from the atmosphere after diffusing throughout ZnO. Note that despite the formation of this oxide transition layer, the intensity of the (002) AlN XRD peak has hardly decreased, as shown before. This indicates on one hand that the decrease in the AlN effective thickness has been compensated by an improvement of its crystalline quality, and on the other hand that the ZnO protective layer has hindered, as expected, the oxidation process. Thus, it is now necessary to develop a high-performance non-oxide diffusion barrier that would prevent this phenomenon. In this perspective, chromium nitride (CrN) or silicon nitride (Si₃N₄) films could be relevant candidates

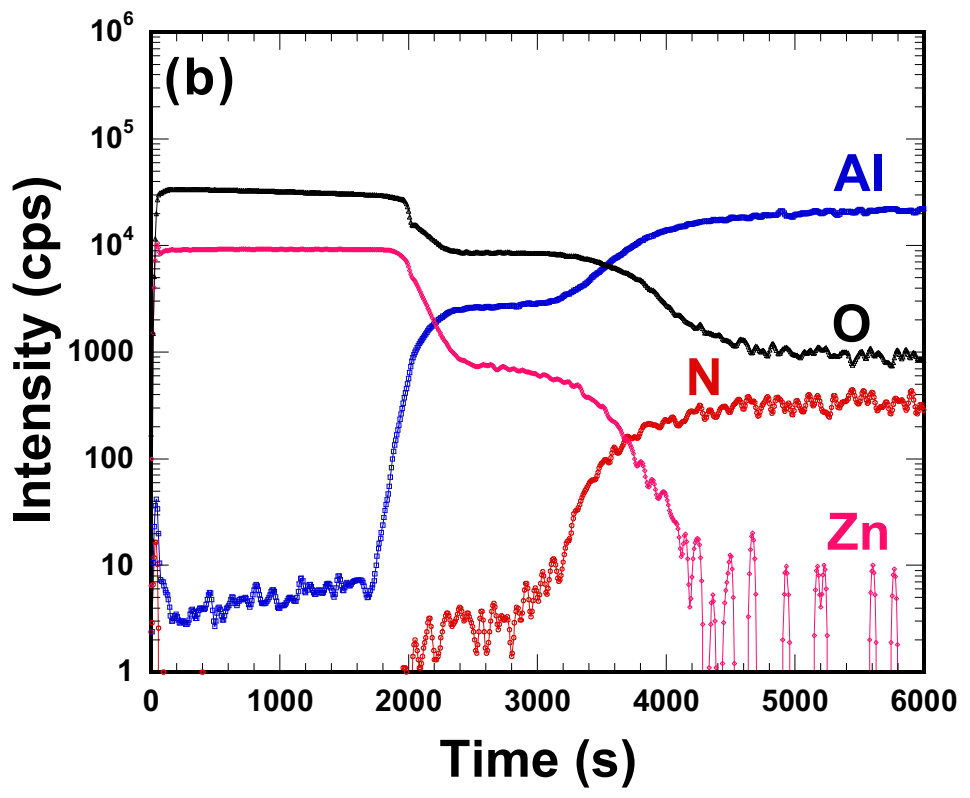
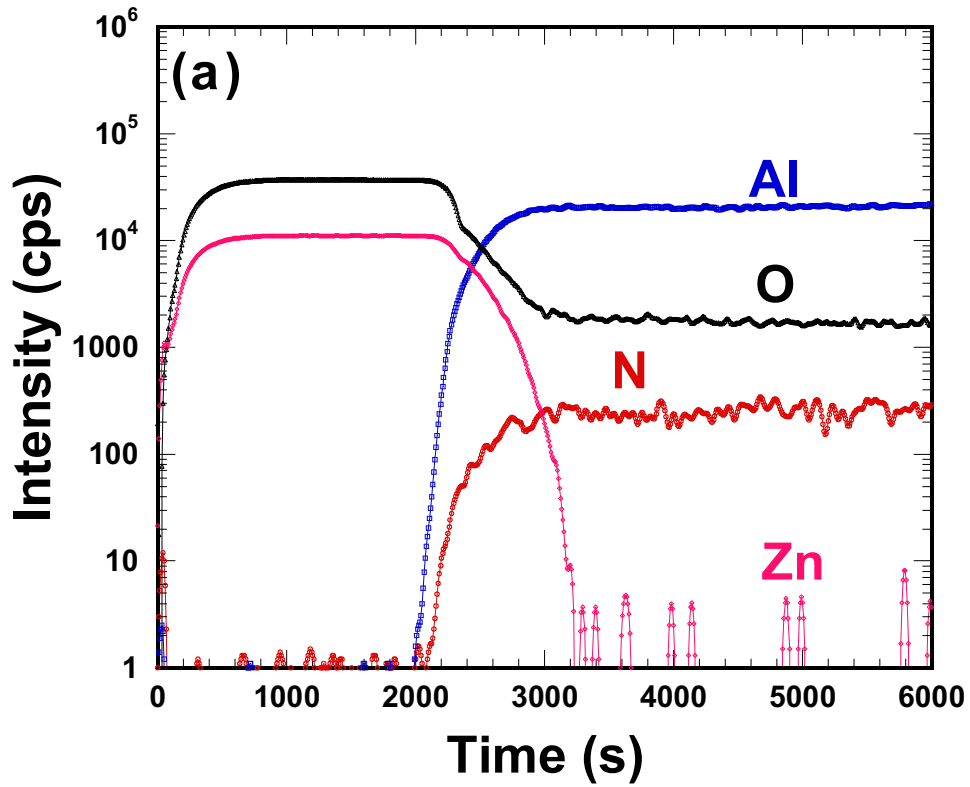


Figure 7: SIMS depth profiles of oxygen, aluminum, nitrogen, and zinc elements in the ZnO/AlN/Sapphire structure measured before (a) and after annealing process (b).

IV. Conclusion

In this study, *in situ* XRD measurements up to 1000°C were performed on AlN thin films deposited on sapphire substrate by magnetron sputtering. Experimental results reveal that the AlN film remains well (002) oriented even at high temperature. A direct correlation between the AlN films crystalline properties (net area of the (002) AlN XRD peak) and the SAW performance in a wide range of temperature was observed. Finally, a 500 nm-thick ZnO protective layer was employed against the surface oxidation process observed on AlN films in the neighborhood of 900°C. XRD measurements show the effectiveness of this layer to hinder oxidation. However, SIMS analysis reveals the diffusion of O atoms (coming from the atmosphere or the ZnO layer itself) throughout the protective layer. These O atoms replace N ones in the initial upper part of the AlN film, which is consequently oxidized. Ongoing experiments are dedicated to the development of a high-performance non-oxide diffusion barrier that would prevent this slow oxidation process.

Acknowledgments

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Omar ELMAZRIA was born in Casablanca in 1968. He received the M.S. degree in industrial computer science and optoelectronic conjointly from the Universities of Metz, Nancy I, France, and the National Polytechnic Institute of Lorraine, France, in 1993, and the Ph.D. degree in electronics from Metz University, France, in 1996. In 1997, he joined the University of Nancy I as Associate Professor of electronic and communication systems and as Professor in 2003. Was the Head of the Micro and Nanosystems Group within the Institut Jean Lamour, Nancy, France. His research includes thin films for microelectromechanical-systems applications, surface-acoustic-wave devices for sensing applications, and high-frequency filters resonators based on layered structures, including diamond, AlN, and ZnO; microfluidic systems; wireless sensors, etc. He is the author 120 papers in refereed international journals and in proceeding of international conferences.

Prof. Elmazria is a member of the Technical Program Committee of the IEEE International Ultrasonics Symposium, Co-Chairman and member of the scientific committee of the IEEE International Conference on Electronic and Measurement Instrumentation (**ICEMI**), and a member of the Institut Universitaire de France (**IUF**).



Pascal BOULET was born near Rennes (France) in 1967. He did his PhD in Solid State Chemistry, in the frame of the European program “Human Capability and Mobility”. Therefor he performed his thesis work in the network composed of 4 different labs: the institute for physical Chemistry in Vienna (Austria), the chemistry department in Genova (Italy), the National Centre for Scientific Research of Demokritos in Athens (Greece) and the “institut des Sciences Chimiques de Rennes” (France). In 1998, he came back to the University of Vienna(A) for one year as a post Doctorate, then he joined for 6 years, the Joint research center from the European Commission–Institut for transuranium elements in Karlsruhe (Germany). He spent then 6 months at the synchrotron ANKA in Karlsruhe on the Institute for Nuclear Waste Disposal (INE) beamline, and got a permanent position in the CNRS at the institut Jean Lamour in Nancy, France, in 2006. He is in charge of a platform called “centre de compétences XGamma” dealing for all X-ray and Mossbauer spectroscopy facilities for the institute. His research is shared between 3 different fields i) to look for new intermetallic phases showing interesting properties like magnetic ordering or superconductivity ii) to look for new quasicrystalline and approximant phases with rare earth, iii) and characterization of thin film by XRD. He is co-author of more than 100 papers in refereed international journals and in proceeding of international conferences.



Thierry Aubert was born in Epinal, France in 1975. He studied Physics at the University of Strasbourg where he received the M.Sc. degree in 1998. In 1999, he passed the *Agrégation de Physique*, a French national highly competitive exam. Then he worked for 7 years as a high school teacher in Physics and Chemistry. From 2007, he was with the *Institut Jean Lamour* (Nancy, France) where he received the Ph.D. degree in 2010. His research work was focused on the choice of materials for high-temperature SAW applications. He studied in particular the possibility to use AlN/Sapphire bilayer structure for such aim. Between November 2010 and July 2011, he was with the CTR (Villach, Austria) where he obtained new results by achieving *in situ* electrical characterization of langasite and AlN-based SAW devices in high-temperature environment, up to temperatures above 1100°C. Between 2011 and 2014, he was with the *Université de Savoie* (Annecy, France) where he has been recruited as an associate professor. Since September 2014, he is with CentraleSupélec, in the LMOPS laboratory (Metz, France) as an associate professor. He is now the author or co-author of 30 papers in the field of high-temperature SAW devices. His current research activities concern the determination of relevant langasite properties for high-temperature SAW applications, surface degradation of langasite in high-temperature air environment, thermoelastic effects in Pt IDTs, and the use of Ir/Rh alloys as thin films to make high-temperature IDTs.



Quadra LEGRANI was born in France. She studied physics and materials science in University Henri Poincaré of Nancy, France. She received a Ph.D degree in physics from the University of Lorraine, Nancy, France, in november 2012 for his work on the heterostructure for isolated acoustic waves and his potential application in harsh environments. She then performed postdoctoral fellowships at Institut Jean Lamour of Nancy. Since September 2013, she joined the engineering school CentraleSupélec (one of France's leading engineering universities) as associate professor of materials processing. His current research concerns the growth of AlN:Sc material for high temperatures applications.



Denis MANGIN was born in 1960 in Thionville (France). He received B.S. degree in computer science from University of Nancy I, France. From 1990 to 2012 he was working on secondary ion mass spectroscopy (SIMS) in "Centre de Recherches Petrographiques et Géochimiques" in Vandoeuvre, France. In 2012, he joined the Institut Jean Lamour in Nancy, France as Secondary ion mass spectroscopy (SIMS) manager.