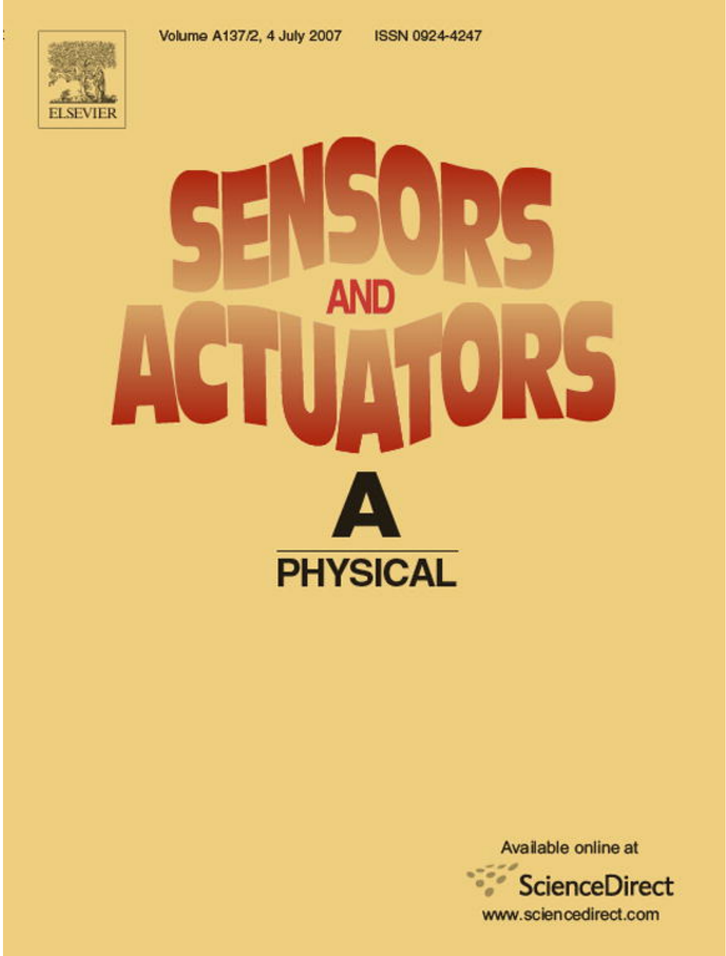
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A study of highly *c*-axis oriented AlN films for diamond-based surface acoustic wave devices: Bulk structure and surface morphology

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Abstract

A systematic study on the optimisation of the sputtering deposition technique for AlN films on diamond substrates is presented. The dependence of the film quality has been analysed versus deposition temperature, for diamond substrates pre-treated with a reactive ion etching (RIE) process for a time duration between 0 and 4 min. These two parameters were found to be critical to obtain highly textured films with the *c*-axis perpendicular to the substrate surface and with a low surface roughness, as required by surface acoustic wave (SAW) applications. In particular, the RIE time was individuated to be crucial in defining the substrate-morphological quality, influencing, in turn, the films structure. The structural characterization of the films was performed by energy dispersive X-ray diffraction (EDXD) analysis and supported by further energy dispersive X-ray reflectometry (EDXR) morphological measurements. Indeed these non-conventional techniques, making use of a non-monochromatized X-ray radiation, are particularly suited when this kind of measurements are required.

Keywords: AlN; SAW; Energy dispersive X-ray diffraction; Sputtering; RIE

1. Introduction

The fast growth of communication media, such as mobile and satellite services, together with new fields of applications in sensing and identification areas (i.e. wirelessly devices), has induced a growing demand of high-performance (i.e. low losses) surface acoustic wave (SAW) devices, operating in the GHz range of frequencies. Nowedays the progress in microfabrication technologies enables mass-production of SAW devices in the 2.5 GHz range [1], but fabrication of SAW devices operating at these (or higher) frequencies, will require an improvement in the line-width resolution limit technology of the interdigital transducers (IDT). However, such improvement requires the use of high-velocity SAW materials such as silicon, sapphire or diamond, as substrates and of coatings made of piezoelectric films, i.e. CdS, ZnO, AlN.

In particular, thick diamond films for SAW devices are very attractive as substrates because of their high sound velocity, which allows higher frequency operation at a given line-width resolution of the interdigital transducer (IDT). On the other hand, AlN is the ideal piezoelectric layer for diamond-based SAW applications, due to the good thermal stability, the excellent mechanical and chemical properties [2–4], but most of all for its high acoustic wave velocity. Indeed, compared to other piezoelectric films [5–11], AlN better matches diamond, with the additional advantage of a smaller velocity dispersion.

Different deposition methods, such as chemical vapor deposition (CVD) [12–14], molecular beam epitaxy (MBE), laser ablation and reactive sputtering techniques [15–20], are used to grow AlN thin films on substrates. In particular, *c*-axis oriented AlN films with small surface roughness at relative low temperatures [21] are obtained when sputtering techniques are used. Indeed, the correlation between the sputtering process conditions and good structural properties of the so obtained films, has been widely investigated, even though the results published are sometimes conflicting.

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Table 1
The experimental conditions used during the different sputtering runs are reported

rf power	500 W
Substrate temperature range	200 to 500 °C
Gas pressure	3×10^{-3} Torr
Gas composition	100% 99.999 pure N_2
Gas flow rate	90 sccm
Target	Al 99.999 pure 6 in. diameter
Target substrate distance	∼50 mm
Growth rate	0.3 μm/h
Background pressure	$<1 \times 10^{-7}$ Torr

In order to obtain high quality SAW devices, the AlN *c*-axis should be normally oriented with respect to the substrate. Therefore, understanding the experimental growth conditions to obtain this preferred orientation is of crucial importance.

Generally preferred orientations can be obtained by taking advantage of the by anisotropy of certain properties, such as surface energy or reactivity, as a function of the crystallographic orientation of the deposited materials and of the film formation processes [22].

The main parameters affecting the growth of AlN films are: the film thickness, the bias voltage, the sputtering power, the gas mixture, the gas pressure, the target–substrate distance and the temperature of the substrate. Surface pre-treatments, consisting of a weak dry or wet etching of the substrate surface, are often performed in order to further improve the orientation of polycrystalline films.

In this work, the growth of AlN thin films on diamond substrates by rf reactive diode sputtering technique, both with or without a RIE pre-treatment of different time duration, is systematically studied in order to find the best experimental conditions to obtain highly textured materials.

Energy dispersive X-ray reflectivity and diffraction techniques [23–27] have been used to investigate the AlN films structural and morphological properties. A series of samples grown at different temperatures (from 200 to 500 °C) and pretreated by RIE process in O_2 plasma for different times (from 0 to 4 min), were investigated in this way. The results show that highly textured films with *c*-axis perpendicular to the diamond substrate were obtained. Furthermore, they exhibit very smooth surfaces, which is an extremely important feature for low loss SAW propagation.

2. Experimental

2.1. AlN films deposition

The AlN films were deposited by rf reactive diode sputtering technique, using a commercial MRC 8620 sputtering head pumped down by a cryogenic vacuum system. All the sputtering runs were carried out keeping the conditions reported in Table 1. The background pressure of the system was better then 1×10^{-7} Torr, and a 30 min pre-sputtering was performed before each deposition process, followed by 1–2 h of thermal annealing at 500 °C and slow cooling down: 2–3 h to reach the room temperature. The substrates temperature was varied in the range between 200 and 500 °C (100 °C steps), in order to evaluate the most favourable deposition conditions. Diamond/Si substrates, provided by Sumitomo Chemicals Japan [28], were chosen as substrates. They consisted of 2 in. poly-crystal Si wafers, ∼800 μm thick, coated with a 23 μm thick diamond layer, characterized by $\langle 1\,1\,1\rangle$ and $\langle 2\,2\,0\rangle$ preferred grains orientation and with the free surface optically polished. Before sputtering the AlN films, all the samples were carefully cleaned using a standard cleaning procedure including detergent bath soak, deionised (18 mΩ/cm) water rinse and drying, followed by the RIE treatment in pure O_2 (99.999%) plasma, at a pressure of 100 mTorr and a rf power density of 1.1 W/cm^2 (200 W on a 6 in. target). The duration of the treatment was between 0 and 4 min in steps of 1 min.

2.2. EDXR/EDXD

The energy dispersive X-ray diffractometry/reflectometry techniques (EDXD/EDXR) [29,30] are complementary techniques that can be used jointly for a structural/morphological characterization of the sample under study.

They both make use of a continuous Bremmsstrahlung spectrum X-ray beam, defined as “white”, to irradiate the sample under study. The energy spectrum of the radiation after interaction with the sample (i.e. scattered or reflected, respectively) is acquired by an energy sensitive detector. They can be carried out by using the same energy dispersive X-ray instruments by changing the angular range of the measurement, namely low deflection angles (of the order of 0.1° for reflectometry and 10° for diffractometry).

One major merit of the ED techniques, compared with the conventional angular dispersive (AD) one, is that the experimental set up is static, no motion of the diffractometer arms being needed during the measurements. This characteristic guarantees that the irradiated part of the sample surface is unchanged during the collection time. Moreover, at a parity of statistical accuracy, the time saving is about an order of magnitude, corresponding approximately to the ratio between the number of photons concentrated in a fluorescence line (primary beam in AD) and the number of photons distributed along the Bremmsstrahlung spectrum.

The energy dispersive diffractometer–reflectometer consists of a non-commercial instrument [31] equipped with a water cooled X-ray W anode tube (Philips, model PW2214/20) supplied at 58 kV and 30 mA. The white beam is collimated by two W slits and reaches the sample placed in the center of rotation of the machine arms. The two slits of the detector arm select the portion of the diffracted beam contained in the acceptance angle of the detector, which is an EG&G ultra pure Ge solid state detector (SSD), cooled by an electro-mechanical refrigerator (X-cooler) and connected to an integrated spectroscopy amplifier-multi channel analyzer system (92 × spectrum master) [32].

The experimental set up described above is used to perform energy dispersive X-ray diffractometry measurements [33]. As mentioned above, the only relevant difference between the instrumental configurations used in the two kinds of measurements, both performed in the reflection geometry, is the order of magnitude of the deflection angle, that corresponds to the *q*-ranges, of some 10^{-2} Å for reflectometry and a few Å for diffractometry.

3. Results and discussion

AlN films are polycrystals with a wurtzite hexagonal structure, showing a preferred orientation of the *c*-axis along the normal to the substrate surface. The use of EDXD technique allowed to determine the structural characteristics of the films in order to monitor the influence of the deposition parameters on the films growth. On the other hand, EDXR could be used to provide complementary information on the film roughness, which is another key point in the SAW technology, as discussed in the following.

The goal is to optimize the film growth along such direction, corresponding to the $\langle 0\,0\,2\rangle$ AlN reflection, keeping the surface roughness as low as possible, as required by SAW applications.

3.1. Energy dispersive X-ray measurements

To obtain the statistical distribution of the orientation of the film domains, i.e. the degree of epitaxy along the *c*-axis oriented AlN film, a rocking curve analysis was performed [34]. It was carried out by recording the intensity of the diffracted radiation as a function of an asymmetry paramete $r\alpha = (\vartheta_i - \vartheta_f)/2$, where ϑ_i and ϑ_f are the initial (incidence) and final (deflection) angles and $\vartheta_i + \vartheta_f = 2\vartheta$ is kept unchanged. To perform the rocking curve analysis, the films were placed on a cradle (the sample holder) placed in the optical centre of the diffractometer. Rotating the cradle an asymmetry parameter scan was performed (at the each α value a diffraction pattern is collected), while the diffraction angle 2ϑ remained unchanged.

In EDXD, each point of a rocking curve, i.e. the value at a generic α, is calculated as the ratio between the intensity of the Bragg peak in correspondence of that α-value with respect to the maximum intensity of the same peak along the α-scan. In this way, no normalization to the primary X-ray beam spectrum, usually required in EDXD measurements, is necessary. The peaks on the diffraction pattern are fitted by the sum of a Gaussian and a linear function to model, respectively, the convolution of the Bragg peak with the diffractometer transport function and the almost flat background, containing also the Compton contribution [29]. The integral of the Gaussian components are used to calculate the rocking curves. In case of the energy dispersive mode, since the diffraction pattern is collected simultaneously at any *q* value, all the rocking curves of the peaks visible in the explored *q*-range are collected simultaneously [33–35]. In this way, all the structural information about Si, diamond, and AlN was collected in a single set of measurements.

The study described above was systematically performed as a function of two parameters: the RIE time, determining the morphological properties of the diamond substrate and of the film and the substrate deposition temperature, which influences the film structure.

As will be described in detail in the following, the study shows that both in case of the RIE-treated substrates and of the unprocessed ones, the temperature must exceed a threshold value (corresponding to about 200 °C for the films deposited on the RIE-processed substrates and 300 °C for the others) in order succeed in the films growth.

A first set of diffraction measurements focused on the influence of the deposition temperature parameter on the film structure.

3.1.1. First set of EDXD measurements: unprocessed substrates

A set of measurements was performed on films of AlN grown on the diamond substrate at a substrate temperature ranging from 200 to 500 °C. Various diffraction measurements where performed at different scattering angles in order to detect the total crystalline structure of the deposited film, each scattering angle corresponding to a different *q*-range to be explored. Indeed, the entire diffraction pattern of the AlN film deposited at 300 °C on a 3 min RIE-treated substrate is shown in Fig. 1 as an example. It corresponds to the pattern collected at $2\vartheta = 7.000°$, this scattering angle corresponding to the *q*-range which contains the information regarding the crystallization of the deposited AlN. In fact, the AlN is grown along two preferred orientations only (not a polycrystalline film), the $\langle 0\,0\,2\rangle$ and the $\langle 1\,0\,1\rangle$ one corresponding to a wurtzite hexagonal structure (primitive lattice $P6_3mc$, $a = b = 3.110$ Å, $c = 4.980$ Å). In the inset of Fig. 1, the *q*-range where the AlN reflections appears, is highlighted. Furthermore, the $\langle 1\,1\,1\rangle$ (1) and $\langle 2\,2\,0\rangle$ (3) reflections of the polycrystalline Si and the $\langle 1\,1\,1\rangle$ (2) Bragg peak corresponding to the diamond, are visible too.

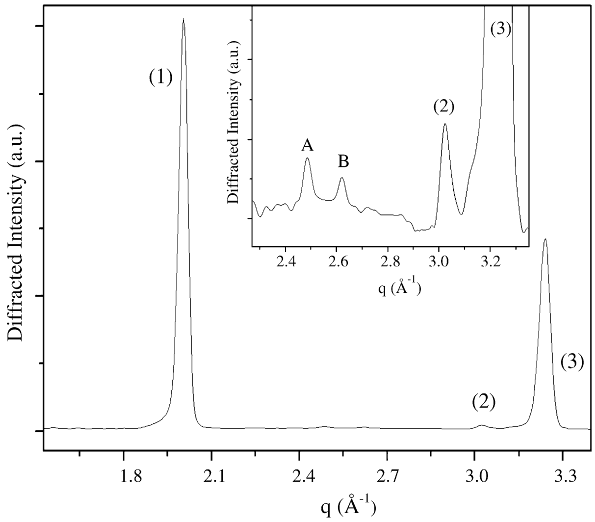

Fig. 1. Diffraction pattern of the 500 °C film: the reflections of the polycristalline Si are visible: (1) Si $\langle 1\,1\,1\rangle$; (3) Si $\langle 2\,2\,0\rangle$; (4) Si $\langle 3\,1\,1\rangle$. In the inset the *q*-range of the ALN $\langle 0\,0\,2\rangle$ reflection (A) is highlighted, the diamond DLC $\langle 1\,1\,1\rangle$ (2) and the ALN $\langle 0\,0\,2\rangle$ (B) reflections are also visible.

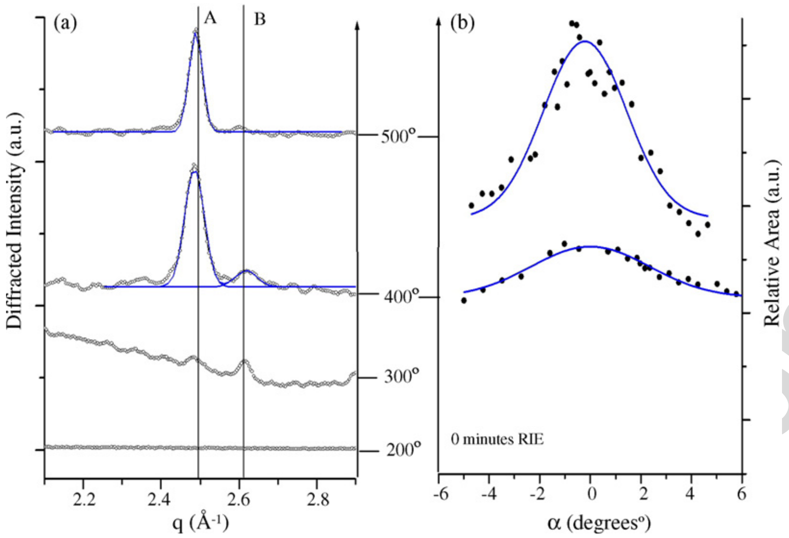

Fig. 2. (a) Diffraction patterns of AlN films grown on the diamond substrate that did not undergo any RIE treatment. The spectra recorded for films grown at different substrate temperatures are reported as a function of the scattering parameter: (A) AlN ⟨0 0 2⟩ and (B) AlN ⟨1 0 1⟩. Increasing the temperature, the desired ⟨0 0 2⟩ reflection gets dominant and the ⟨1 0 1⟩ signal progressively tends to disappears. (b) Rocking curve analysis of the AlN ⟨0 0 2⟩ reflection is reported as a function of the sample growth temperature. The line is the Gaussian fit of the rocking curves: the FWHM is reduced as the temperature increases.

The results of the systematic study are summarized in Fig. 2a, showing the AlN *q*-region of the pattern. The EDXD measurement of the film deposited at 200 °C exhibits the features of the substrate only while, in the patterns corresponding to films grown at higher substrate temperatures, two reflection are visible: (A) AlN ⟨0 0 2⟩ reflection, at $q = 2.50(2)$ Å^{-1}, and (B) the AlN ⟨1 0 1⟩ reflection at $q = 2.63(2)$ Å^{-1} (giving, respectively, lattice parameters of 2.51(2) Å and 2.39(2) Å, in agreement with the literature data). In detail, at 300 °C the relative intensities of the two reflections are comparable, indicating no preferential direction in the deposition. Conversely, when the deposition is performed at 400 °C and 500 °C films with good texture are obtained and the AlN ⟨0 0 2⟩ reflection is the major contribution of the deposited films to the EDXD pattern. In this case RC analysis shows that the AlN ⟨0 0 2⟩ reflection has a FWHM of about 0.37°. For all of the samples, when the asymmetry value α increases (the experimental cradle rotation increases), the peak intensity decreases quite rapidly, demonstrating a good crystalline quality of the films, as shown in Fig. 2b, where the result of the rocking curve analysis of the AlN ⟨0 0 2⟩ reflection is reported. It is notable that the FWHM of the rocking curve decreases as the temperature increases, as shown in Table 2. This show that a good degree of *c*-axis orientation can be obtained, provided the deposition temperature is sufficiently high.

Table 2
The FWHM values, obtained by a Gaussian fit of the rocking curves of the AlN ⟨0 0 2⟩ reflections, of samples deposited on diamond substrates treated for various times and at various temperatures are reported

Re-treatment duration (min)	FWHM (°)		
	AlN ⟨0 0 2⟩, 300 °C	AlN ⟨0 0 2⟩, 400 °C	AlN ⟨0 0 2⟩, 500 °C
0	Not visible	5.50 ± 0.05	3.86 ± 0.05
3	0.25 ± 0.05	0.23 ± 0.05	0.10 ± 0.05
4	Not visible	Not visible	0.61 ± 0.05

3.1.2. Second set of EDXD measurements: RIE processed substrates

In order to obtain high quality AlN film growth at lower deposition temperatures, sets of samples were prepared by depositing the AlN films on diamond substrates previously submitted to a short RIE treatment. The purpose of this treatment is to introduce small, controlled defects at the substrate surface, which favorites the growth of AlN films with a preferred *c*-axis orientation.

The results of the deposition on untreated substrates shown in the previous paragraph were compared with those of a second set of diffraction measurements carried out on AlN films grown on diamond substrates previously submitted to a 3 min of RIE treatment. The collected patterns are shown in Fig. 3a, as a function of the scattering parameter and deposition temperature, while the rocking curve results for each AlN, are plotted in Fig. 3b (dots) as a function of the asymmetry parameter. The Gaussian fit of each rocking curve is shown with the continuous line.

- For the film deposited at 200 °C, a deposition along the ⟨1 0 1⟩ direction is obtained (while no crystalline AlN sample was obtained on substrates not submitted to RIE). This indicates that the critical temperature to obtain crystals is lower when treated substrates are used.
- At 300 °C, the AlN ⟨0 0 2⟩ reflection appears and it gets dominant when the temperature is further increased, while the ⟨1 0 1⟩ signal progressively tends to disappear.

Comparing the result of the RC analysis for these two samples (Fig. 4), it is evident that films characterized by a much higher degree of epitaxy, are obtained when the deposition is performed

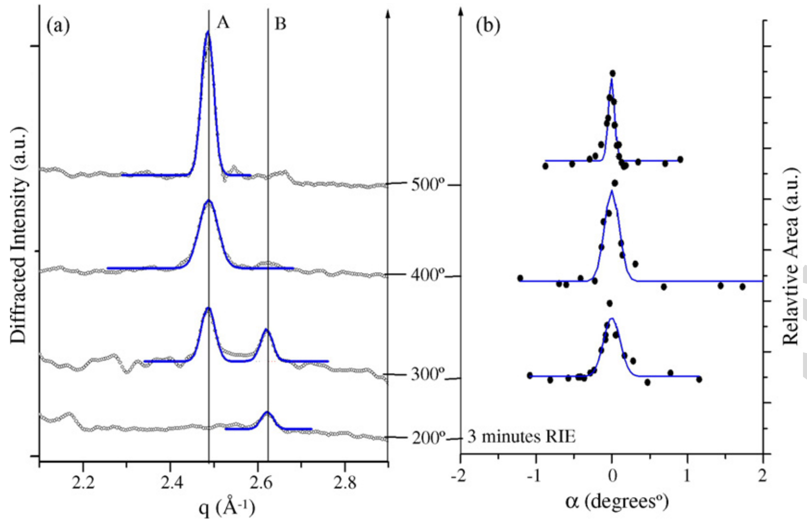

Fig. 3. (a) Diffraction patterns of AlN films grown on the diamond substrate that was previously submitted to 3 min of RIE treatment. (b) Rocking curve analysis of the AlN ⟨0 0 2⟩ reflection.

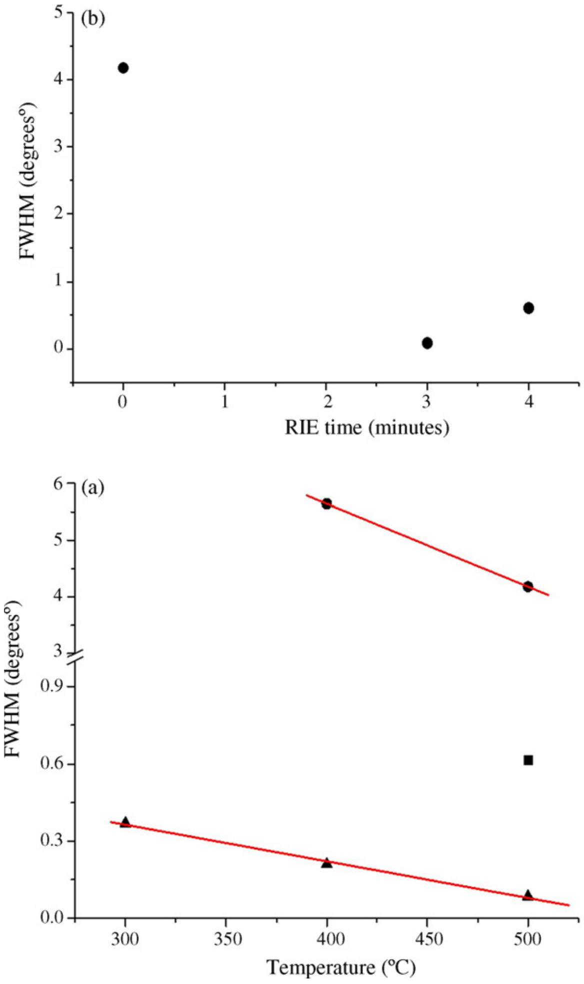

Fig. 4. (a) The trend of the FWHM of the rocking curves of the AlN ⟨0 0 2⟩ reflection, deduced from the Gaussian fit, is plotted as a function of the deposition temperature (dots: no RIE, triangles: 3 min RIE, square: 4 min RIE) and fitted by a linear function (straight line). (b) FWHM of the rocking curves of the AlN ⟨0 0 2⟩ reflection (500 °C) as a function of the diamond RIE time.

on 3 min RIE-treated substrates. Moreover, the dependence of the films degree of epitaxy (i.e. the FWHM the of the AlN ⟨0 0 2⟩ reflection, Table 2) on the temperature appears to be less dramatic for RIE processed films (see Fig. 4).

3.1.3. Third set of EDXD measurements: the influence of the duration of the RIE treatment of the substrate on the structural order of the films

The diffraction patterns of AlN films grown on the diamond substrate submitted to 4 min of RIE treatment are reported in Fig. 5a and the relative Rocking Curve analysis of the AlN ⟨0 0 2⟩ reflection in Fig. 5b.

The effect of a longer RIE time on the film deposition was very important: the substrate had to be heated up to 500 °C to obtain an ordered crystalline film and, in any case, the AlN ⟨0 0 2⟩ reflection is no longer the only contribution (although it remains the major one) of the films to the EDXD spectra. In Table 2 the FWHM of the RC analysis on the ⟨0 0 2⟩ AlN reflection is shown.

One hypothesis to explain this behaviour, is that a longer RIE time has a negative effect on diamond substrates morphology, reducing the efficiency of the film growth along the preferential direction. Indeed the RIE treatment induces small defects that, acting as nucleation sites, promote the growth of high quality film. However, when a critical number and size of the defects is exceeded, the nucleation rate increases and a large number of non-regular crystallites are formed, inducing a disordered growth of the material. From this point of view, 3 min appears to be the optimum RIE-time in order to obtain the best film texture.

The X-ray reflectometry technique was then applied to get further information on the film surfaces. It is sensitive to surfaces and interfaces morphology at the Angstrom resolution [36,37]. The method is based on the optical properties of X-rays [38] (Snell rule). In the small angle approximation, the reflected intensity is a function of the momentum transfer q (as in the case of diffraction) that, in turn, depends on both the reflection angle and the energy of the X-rays [39]. When an X-ray beam impinges

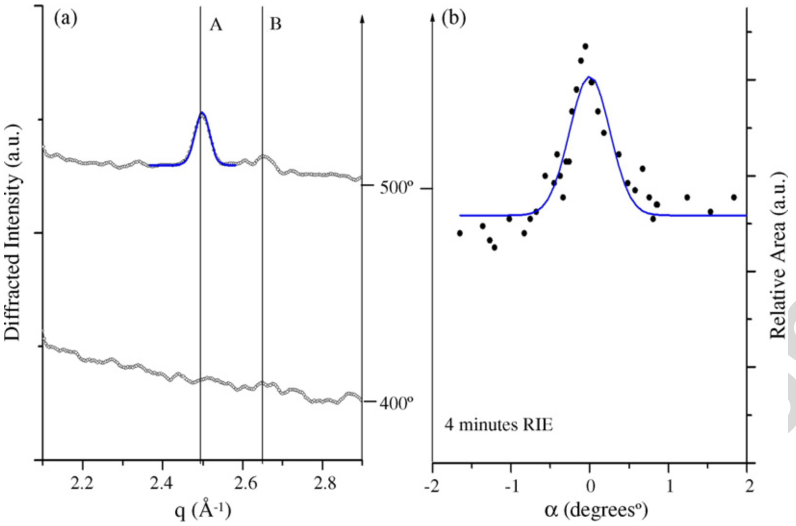

Fig. 5. (a) Diffraction patterns of AlN films grown on the diamond substrate that was previously submitted to 4 min of RIE treatment. (b) Rocking curve analysis of the AlN ⟨0 0 2⟩ reflection.

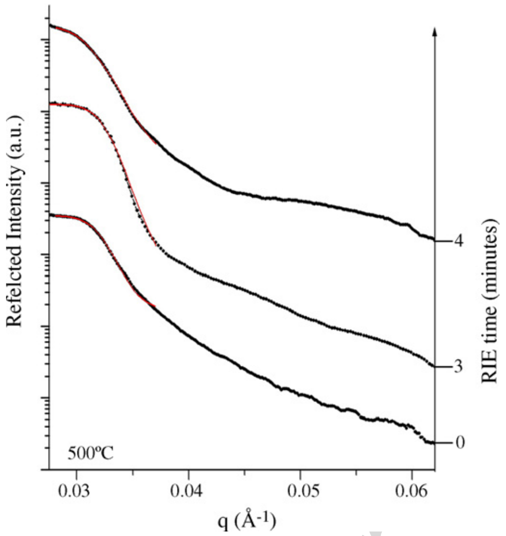

Fig. 6. EDXR data for the 500 °C samples: experimental data and treshold fits. A film roughness always below 20 Å is deduced.

a film deposited on a substrate, it is partially reflected at the two interfaces (air-film and film-substrate) and the two reflected components interfere producing an almost squared sinusoidal modulation of the reflectivity pattern. The period of the modulation is connected with the film thickness, while the inclination of the threshold and the damping of the oscillations are related to both the surface and the interface roughness.

EDXR measurements were performed to evaluate the surface roughness of the films deposited at 500 °C, which show the best structural characteristics at the different RIE treatment times (see Fig. 6). In our case, since the films are rather thick (microns), the oscillations frequency is too high to be observed, since they are washed out by the convolution effects due to the limited resolution of the detector. Therefore, the reflectometry patterns could provide the roughness of the surfaces, only. Importantly, films with quite smooth surface can be obtained, the roughness value being always below 20 Å.

4. Conclusions

We report on the systematic EDXD/EDXR study of AlN films deposited on diamond substrates for SAW devices applications. Both the submission of the substrate to preventive RIE treatments and the deposition temperatures were found find to play a relevant role to obtain highly-textured films with the *c*-axis perpendicular to the substrate surface and with smooth surfaces, as required by SAW applications. In particular, when the deposition is performed at higher temperatures, AlN films with a preferential ⟨0 0 2⟩ orientation and characterized by a very narrow rocking curve (i.e. highly epitaxial) are obtained. The films mosaicity and their degree of epitaxy, also at lower temperatures, are enhanced by using substrates that have preventively been submitted to a RIE treatment below 3 min. Conversely, when the RIE treatment of the substrate is prolonged for longer times, it has a negative effect on the structure of the AlN film. This behaviour can be attributed to the excessive concentration and size of the defects that, above a certain threshold, tend to disturb the correct deposition process rather than to favourite the ordered nucleation of the sputtered material.

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