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Development of native, single crystal AlN substrates for device applications

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Ultra-low dislocation density aluminum nitride is a very promising substrate for many device structures based on the III – V nitride system. A better lattice and thermal expansion match than foreign substrates such as SiC or sapphire make AlN the substrate of choice for heteroepitaxial growth of AlGaN alloys, especially those with high aluminum concentration. In this paper, we show crystalline and chemical characterization of native aluminum nitride substrates, as well as characterization of AlGaN epilayers with 40 and 50% concentration of aluminum. The observation of atomic steps in atomic force microscope scans of the bare substrates and epilayers, as well as the narrow Full Width at Half Maximum (FWHM) measured on X-ray diffraction, are an indication of both the good surface preparation of the substrates and quality of the epilayers. An estimation of defect density on epilayers grown by Migration Enhanced Metal Organic Chemical Vapour Deposition (MEMOCVD) resulted in mid $10^6/cm^2$.

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1 Introduction

Native aluminum nitride (AlN) substrates are a relatively new choice for epitaxial growth of III-nitride based devices for optoelectronics and Radio Frequency (RF) applications. For the last three years, Crystal IS, Inc. has been providing native single crystal substrates of AlN with a dislocation density in the order of $1{,}000/cm^2$ [1], which opens the door to the fabrication of devices such as deep ultraviolet (UV) solid state lasers, that were unthinkable before the availability of a native substrate. Another advantage of cutting substrates from bulk crystals is the possibility of preparing different orientations, such as non-polar orientations, which are not readily obtainable by growth on foreign substrates. The lack of both piezoelectric and spontaneous polarization fields, for heterostructures grown on non-polar substrates, has created significant interest in these orientations for high performance light emitting diodes (LEDs) and laser diodes as well as potentially attractive for more reliable RF devices. In addition, the AlN substrates tend to be highly insulating which makes them highly desirable for high power RF devices.

In this paper, we discuss X-ray, atomic force microscopy (AFM), Secondary Ion Mass Spectroscopy (SIMS) and electrical characterization carried out on Crystal IS substrates as well as on epilayers deposited on them.

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2 Substrate characterization

2.1 Crystalline quality

X-ray topography measurements of the substrates have shown high crystalline quality and a low dislocation density, in the order of 1000/cm^2 [1]. The use of crossed polarizers allows the determination of stressed regions on the substrates, as they appear brighter than the relaxed regions. In Fig. 1 we show a picture of a substrate, an image of the same taken through crossed polarizers, X-ray topography images taken at two different rocking angles and, for the sake of comparison, an X-ray topography image of a 2″ in diameter SiC semi-insulating commercial substrate.

2.2 Impurity characterization

The determination of impurity concentration was carried out by SIMS. In order to have a reference, the substrates were implanted with controlled quantities of different impurities that serve as an internal calibration standard. By knowing the natural abundances of the different isotopes it is possible to distinguish background detection from actual impurities on the substrates, assuming that the impurity concentration on our material is low, and carry out the measurements with a control. The main impurity present in AlN is oxygen. In Fig. 2 the results of the measurements for oxygen detection are shown as a function of depth within the substrate. The peaked curve corresponds to the profile of the implanted ^{18}O, the upper flat is the ^{16}O, and the lower flat corresponds to the molecular form ^{27}Al^{15}N. The reason to measure the concentration of this particular molecular form is that this allows the use of a single detector. The flatness of this curve is an indication of almost no charging effects. After the surface peak, the ^{16}O concen-

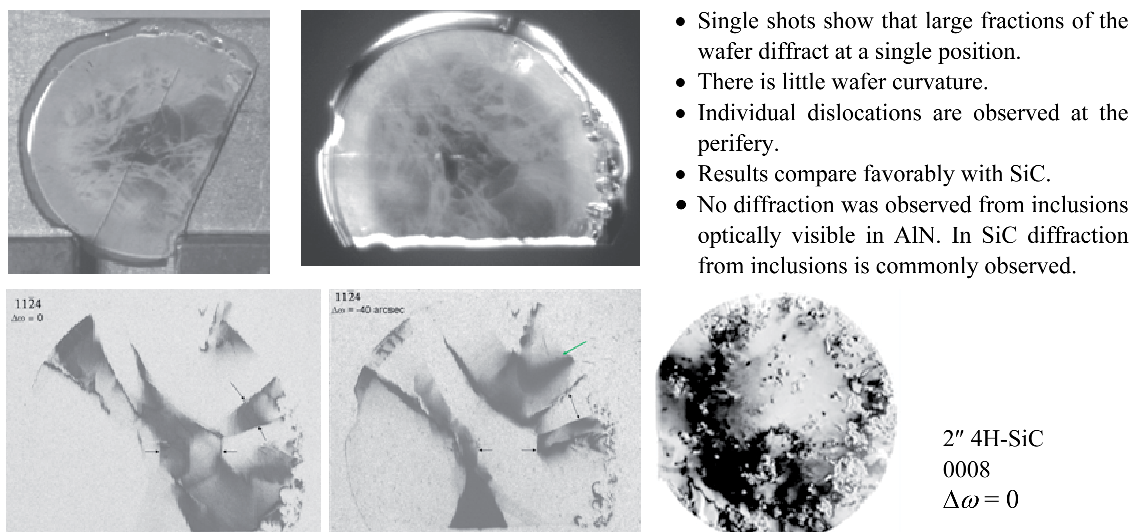

Fig. 1 Upper left: optical image of an AlN substrate. The length of the flat is 22 mm. The image at the right shows a picture of the same substrate taken through crossed polarizers. The main contrast that can be seen that is not due to the optical appearance of the substrate is the round, central darker region. This contrast is due in part to lack of uniformity and roughness on the back of the substrate. Lower left and center: two X-ray topography images of the same substrate taken at an angular distance of 40″, showing the good crystalline quality of the substrates. The black arrows show small angle grain boundaries. $\Delta\omega = 0$ refers to the maximum of the rocking curve. The picture at the center shows the intensity observed at 40″ from the maximum of the rocking curve. Now the areas adjacent to the original black arrows show stronger diffraction intensity. The misalignment between these areas is small, on the order of 40 arcsec. The picture at the right shows an X-ray topography image taken on a commercially available SiC semi-insulating substrate. It shows many more "inclusion" type defects than the AlN substrate.

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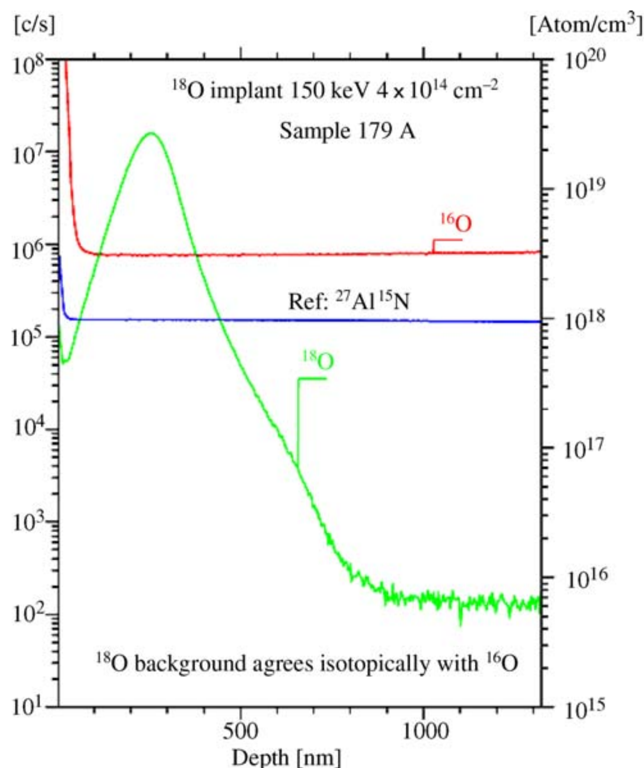

Fig. 2 (online colour at: www.pss-a.com) Oxygen profile as a function of depth in an AlN substrate measured by SIMS. The peaked curve corresponds to the implanted ^{18}O used as a control for background determination. The total oxygen concentration determined in this substrate is $3 \times 10^{18}/cm^3$.

tration is quite constant as a function of depth. The concentration measured is around 3×10^{18} atoms/cm^3. Determination of concentration for other impurities such as chromium, iron, molybdenum, tantalum and, tungsten gave results below the detection threshold.

Electrical resistivity measurements carried out at room temperature resulted in values higher than 10^{12} Ω cm, indicating that the impurities are not electrically active. The resistivity decreases to 10^8 Ω cm for the temperature range between 300 and 560 °C, although the conduction mechanism is not yet understood.

3 Epitaxial growth

In order to develop UV optoelectronic and high power RF electronic devices it is key to obtain good quality layers of AlGaN with high Al concentration. Excellent quality layers grown on native AlN substrates by MEMOCVD [2] and Organo-Metallic Vapor Phase Epitaxy (OMVPE) have been demonstrated. In Fig. 3 we show AFM scans of a bare substrate, where atomic steps can be observed, as well as AFM scans for epitaxial AlGaN layers with 40 and 50% aluminum respectively deposited on nearly-on-axis *c*-face substrates. The two heteroepitaxial layers are of good quality.

The MEMOCVD heteroepitaxial layer shows columnar growth topped by flat hexagonal structures consisting of concentric, closed rings with an elevated central region, as shown on Fig. 3 (center). This feature, which is seen across the whole sample, is distinctly different from structures, attributed [3–5] to screw dislocations, typically observed on epitaxial AlGaN on foreign substrates. Screw dislocations show a spiral step pattern that may have either a pit or a hillock at its center. Studies [4, 5] carried out on this and other material systems suggest that the concentric, closed rings structures correspond to edge

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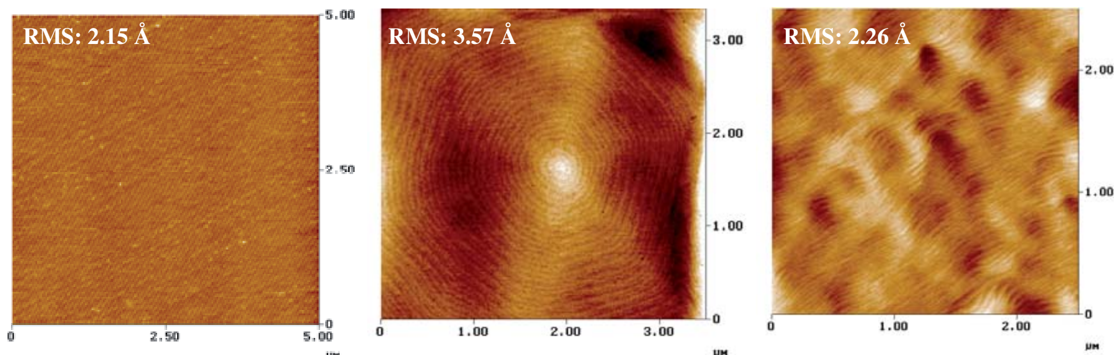

Fig. 3 (online colour at: www.pss-a.com) AFM scans of (left) a bare substrate, (center) 500 nm thick $Al_{0.4}Ga_{0.6}N$ grown by MEMOCVD and (right) 600 nm thick $Al_{0.5}Ga_{0.5}N$ grown by OMVPE. Both layers were grown on 300 nm AlN buffer layers. Atomic steps are visible for the bare substrate as well as in both heteroepitaxial layers. The legends on the upper left corner indicate the RMS roughness measured on the surfaces.

dislocations. Assuming that the center of each structure corresponds to an edge dislocation, the density is on the order of $4 \times 10^6/cm^2$. The spiral pattern associated with screw dislocations is not observed suggesting that the total dislocation density is not significantly higher. Epitaxial layers grown on foreign substrates such as SiC or sapphire have average density of dislocations on the order of $10^7-10^{10}/cm^2$ [6].

Figure 4 shows symmetric and asymmetric rocking curves corresponding to the OMVPE growth of an AlGaN layer with approximately 50% Al. The FWHM measured for the symmetric, (0002) reflection, is 94 arcsec while the one measured for the asymmetric, $(10\bar{1}1)$ reflection, is 97 arcsec, indicative of the good crystalline quality of the epilayer.

4 Conclusions

Crystalline and chemical characterization of native aluminum nitride substrates is presented that shows the good crystalline quality and high purity of our substrates. The main impurity present in the substrate material is oxygen, and the total concentration is in the order of 10^{18} atoms/cm^3. The preparation of an epi-ready surface can be achieved, as shown by the observation of atomic steps in atomic force microscope scans. The epitaxial deposition of AlGaN layers with a defect density as low as $10^6/cm^2$ is very promisory for the development of high power electronics and UV laser and light emitting diodes.

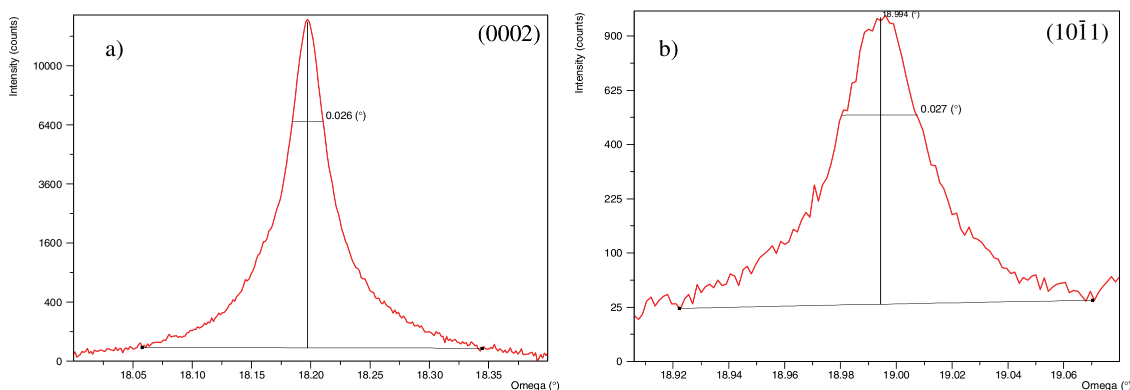

Fig. 4 (online colour at: www.pss-a.com) a) Symmetric and b) asymmetric (right) X-ray rocking curves measured after epitaxial deposition of $Al_{0.5}Ga_{0.5}N$. The epilayer structure consists of a 0.3 μm thick AlN buffer layer followed by a 0.6 μm thick $Al_{0.5}Ga_{0.5}N$ heteroepitaxial layer.

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