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Large area mapping of the alloy composition of $Al_xGa_{1-x}N$ using infrared reflectivity

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The energy position of a dip observed in the IR-reflectance spectra recorded from wurtzite *c*-plane $Al_xGa_{1-x}N$ epitaxial films grown on SiC substrate reflects the composition of the alloy. A calibration procedure is presented with the possibility of mapping for large area wafer. The technique is non-destructive, scalable and fast. The limitations are discussed and comparisons with other techniques are made.

In the last few years great progress has been made in the development of optoelectronic devices based on group-III nitrides. Important examples are light-emitting diodes and laser diodes operating throughout the green and UV spectral regions. In most optoelectronic applications the alloy composition is a crucial parameter. The access of the alloy composition is therefore of great interest. Many techniques have been reported for this purpose and among the non-destructive methods the most important one is probably the X-ray diffraction, which also can give information on the crystalline quality of the film [1]. However, X-ray diffractometers are fairly expensive and too long experiment time is required to provide fast feedback to the growth. Optical techniques are thus preferred for characterization. Photoluminescence at low temperature has been useful to determine the composition of $Al_xGa_{1-x}N$. However due to the lack of excitation source this technique is applicable only for $x < 0.5$ [2]. Detailed Raman studies have shown that phonon energies are convenient measures for the quantitative characterization of the Al content in of $Al_xGa_{1-x}N$ alloys [3, 4]. However this method also demands long experimental time, which is not acceptable for feedback to growth. Few reports [3–6] have shown that infrared reflectance spectroscopy can be used for the characterization of nitride alloys, however mainly for the determination of the thickness of the layer. Those previous works were conducted on $Al_xGa_{1-x}N$ alloys grown generally on Si substrate or sapphire.

In this paper we report a technique based on infrared reflectance for determining the alloy composition of wurtzite *c*-plane $Al_xGa_{1-x}N$ epitaxial films grown on SiC.

The substrates used for the structures were Si-face nominally on-axis 4H-SiC wafers with n-type conductivity and resistivity of 0.07–0.2 Ω cm. The layers were grown in a low-pressure hot-wall MOCVD reactor [7] at 1200 °C using ammonia and metal organic compounds (trimethylaluminum TMA and trimethylgallium TMGa) as precursors. A mixture of purified hydrogen and nitrogen was used as a carrier gas. An AlN buffer layer typically 100 nm thick was in most cases grown prior to the $Al_xGa_{1-x}N$ layer, however in some cases the $Al_xGa_{1-x}N$ layer was grown directly on the SiC substrate. The thickness of the $Al_xGa_{1-x}N$ layer varies from sample to sample and was in the range 250 nm to 700 nm. Due to the accumulation of tensile strain in the grown material, cracks were observed typically for thicknesses larger than 450 nm.

High resolution X-ray diffraction measurements were done using a Philips X'pert high resolution X-ray diffractometer (Cu $K_{\alpha 1}$ radiation), with an asymmetric triple-axis analyzer and a symmetric Ge(220) monochromator. The composition was calculated from the 2θ–θ position of the (0002) peak, assuming that Vegard's law is valid [8]. We

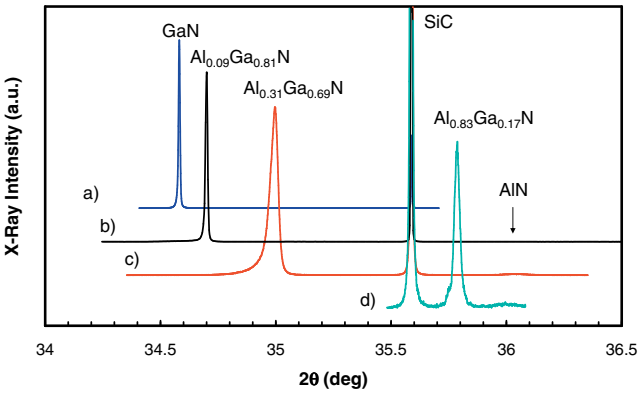

Figure 1 (online colour at: www.pss-rapid.com) XRD data for the (0002) peak obtained for the $Al_xGa_{1-x}N$ layers grown on SiC substrate with a) $x = 0$, b) $x = 0.09$, c) $x = 0.31$ and d) $x = 0.83$, respectively. The thickness of each layer was a) 2.5 µm, b) 1.15 µm, c) 2 µm, and d) 0.37 µm, respectively.

do believe growing $Al_xGa_{1-x}N$ on AlN system implies roughly the same assumption. Typical spectra are shown in Fig. 1 where the (0004) peak of the SiC substrate is observed at 35.59° for all the spectra. A weak broad feature related to the AlN 100 nm thick buffer can be visualized at about 36.04°, whereas the signal of the $Al_xGa_{1-x}N$ layer has a position depending on the composition.

The reflectance spectra were measured using a Fourier Transform Infrared Spectrometer equipped with a KBr beam splitter and a DTGS (dueterated triglycine sulphate) detector which limit the measurement range to 250–4000 cm^{-1}. Measurements were taken at room temperature using a glowbar light source with a beam incident angle of 30°, 1 cm^{-1} resolution and averaged over 10 scans. Typical spectra are shown in Fig. 2. For the SiC substrate the typical reststrahlen band with high reflectance is observed between 800 cm^{-1} and 990 cm^{-1} (Fig. 2a). If a thin (about 100 nm) AlN layer is grown on the SiC substrate (Fig. 2b) a dip at around 892 cm^{-1}, associated to the LO mode of AlN, is observed together with a feature at 679 cm^{-1} related to the TO mode. If we subtract this reflectance spectrum (Fig. 2b) from that of the SiC substrate alone (Fig. 2a) we obtain a similar spectrum as reported from a 0.06 µm thick AlN layer grown on (111)-oriented silicon by molecular beam epitaxy [9]. If the AlN thickness increases a broad band will appear between these two modes (typically 620–910 cm^{-1}, not shown here) which can be considered as the reststrahl band of AlN [10]. Since structures used in this study include a thin AlN nucleation layer, the dip at 892 cm^{-1} and a broad feature at 679 cm^{-1} are expected and are observed for all samples, except from the $Al_{0.56}Ga_{0.44}N$ layer (Fig. 2d) which did not have such an AlN layer. However, as can be seen in Fig. 2 a small shift of the dip at 892 cm^{-1} is sometimes observed which could be associated to stress in the layer [11]. When an $Al_xGa_{1-x}N$ layer is grown on top of the AlN layer an additional dip appears at lower wavenumber than the LO mode of AlN as shown by the arrows of Fig. 2, and the wavenumber decreases further with increasing x.

The position of the dip observed in the IR-reflectance spectra and associated to the LO mode (arrows in Fig. 2) is plotted in Fig. 3 as a function of the Al content as determined by the XRD measurement shown in Fig. 1. This enables the determination of the Al content by measuring the position of the dip in the reflectance spectra. Our data are in fair agreement with those reported in the literature and measured using Raman spectroscopy [4]; those data which are also reported in Fig. 3 for comparison, are obtained from $Al_xGa_{1-x}N$ ($x < 0.5$) layers grown either on thin GaN buffers deposited on sapphire by molecular beam

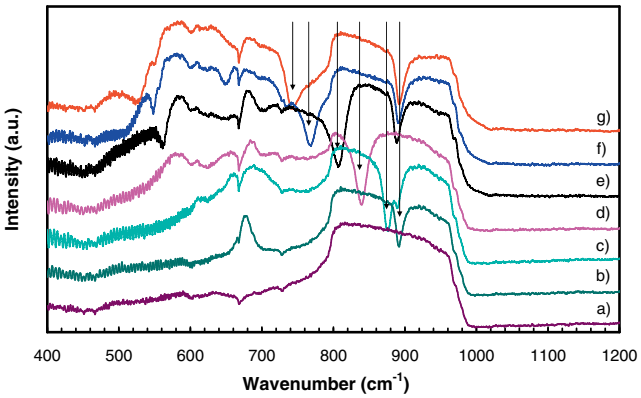

Figure 2 (online colour at: www.pss-rapid.com) Reflectance spectra recorded a) from a SiC substrate and from $Al_xGa_{1-x}N$ structures with b) $x = 1$, c) $x = 0.83$, d) $x = 0.56$, e) $x = 0.31$, f) $x = 0.09$ and g) $x = 0$. Each structure contains a thin (100 nm) AlN buffer layer except for the layer d). The arrows show the positions of the considered absorption peak for the calibration of the composition. The thickness of the AlGaN layer was c) 0.37 µm, d) 0.65 µm, e) 1.15 µm, f) 2 µm and g) 1.45 µm, respectively.

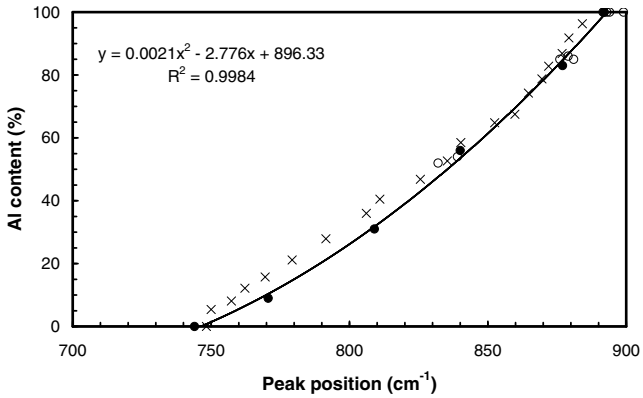

Figure 3 Al content determined from the XRD measurements on the (0002) peak as a function of the dip position (LO phonon) observed in the reflectance spectra. The solid circles are data from the samples for which the reflectance spectra are shown in Fig. 1, the empty circles are from other samples. The crosses are the frequencies of the A1(LO) modes measured with Raman spectroscopy and reported by Davydov et al. [4]. The line shows a fit to all circle data with the equation indicated in the figure.

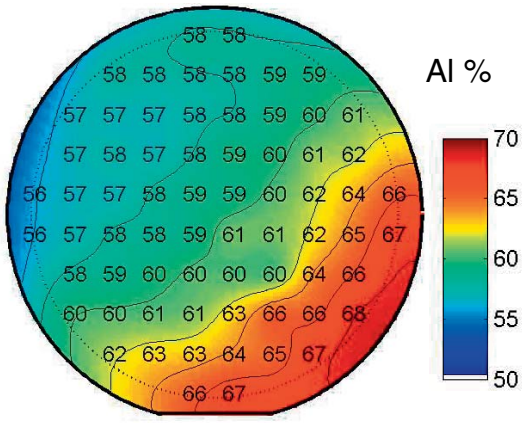

Figure 4 (online colour at: www.pss-rapid.com) Al content map of an $Al_xGa_{1-x}N$ layer grown on a 2 inch diameter substrate. The growth was done without rotation and with non-optimized conditions to favor a non-uniform deposition. The Al content was determined from the calibration using reflectance spectra.

epitaxy, or on sapphire by MOCVD and $Al_xGa_{1-x}N$ ($x > 0.5$) layer grown on Si substrate by hybride vapour deposition.

Using data from Fig. 3 we have mapped the Al content of a 2 inch diameter wafer having an $Al_xGa_{1-x}N$ layer on top. From the growth parameters the average Al content was expected to be about 60%. Since the growth was done without rotation of the substrate and the growth parameters were not optimized for this special case, the thickness of the layer varied from about 0.18 µm to 0.84 µm as measured with reflectance technique in the visible range. The variation of the thickness was thus about 31% (determined sigma/mean, we would like to note that with rotation of the substrate and optimized growth conditions the typical thickness variation is 1% over a 2 inch diameter wafer). This large variation of the growth rate gives rise to a variation (5%) of the Al content in the layer over the substrate; high growth rate gives low Al content (see Fig. 4).

We have demonstrated that the mapping of full wafer to determine the composition of $Al_xGa_{1-x}N$ layer is possible using reflectance technique. The technique is non-destructive, scalable, fast, and therefore suitable for fast feedback to growth.

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