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# Composition measurement of epitaxial $Sc_xGa_{1-x}N$ films

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**Abstract**

Four different methods for measuring the compositions of epitaxial $Sc_xGa_{1-x}N$ films were assessed and compared to determine which was the most reliable and accurate. The compositions of epitaxial $Sc_xGa_{1-x}N$ films with $0 \leq x \leq 0.26$ were measured directly using Rutherford backscattering (RBS) and X-ray photoelectron spectroscopy (XPS), and indirectly using *c* lattice parameter measurements from X-ray diffraction and *c/a* ratio measurements from electron diffraction patterns. RBS measurements were taken as a standard reference. XPS was found to underestimate the Sc content, whereas *c* lattice parameter and *c/a* ratio were not reliable for composition determination due to the unknown degree of strain relaxation in the film. However, the Sc flux used during growth was found to relate linearly with *x* and could be used to estimate the Sc content.

**Keywords:** ScGaN, Rutherford backscattering, XPS, composition measurement

**PACS codes:** 81.05.Ea, 81.70.Jb

## 1. Introduction

III-nitride semiconductor alloys offer a wide range of band gaps from 0.7 eV to 6.2 eV [1–3] for applications in short-wavelength optoelectronics. However, low internal quantum efficiencies remain a problem for III-nitride-based ultraviolet light emitters [4]. Challenges include the difficulty of *p*-type doping, alongside the relatively large lattice mismatches between layers within these devices which lead to problems with internal stresses and dislocation generation [4]. Therefore, alternative materials with wide, direct, band gaps but different lattice parameters, such as Sc-based III-nitride alloys, are of interest for improving device performance [5–13].

Both $Sc_xGa_{1-x}N$ and $Sc_xAl_{1-x}N$ alloys are of growing technological interest in this regard and are created by alloying ScN with GaN or AlN respectively [5, 14–24]. ScN is an indirect gap semiconductor with the rock-salt structure and has been of interest recently for thermoelectric device applications [25–30]. ScN-based interlayers are also of use as dislocation-blocking layers in the growth of epitaxial GaN films [31–33]. Recently, experimental studies have shown that $Sc_xGa_{1-x}N$ films can typically be grown with higher quality and lower oxygen incorporation, as well as remaining phase-pure up to higher Sc contents, compared to $Sc_xAl_{1-x}N$ [21–23]. In the past, the band gaps of $Sc_xGa_{1-x}N$ were thought to decrease with increasing Sc content [10, 34], which agreed with early theoretical predictions with a fixed *c/a* ratio [35]. However, recent high-resolution scanning

transmission electron microscopy studies have shown that the band gaps measured from $Sc_xGa_{1-x}N$ are influenced strongly by the presence of lamellar defects: band gaps measured from low defect density $Sc_xGa_{1-x}N$ increase from 3.4 up to 4.36 eV as $x$ increases from 0 to 0.26, consistent with recent high-quality theoretical studies [14, 18]. This change is correlated with a decrease in the *c/a* lattice parameter ratio of the $Sc_xGa_{1-x}N$ alloy as a function of increasing Sc content, leading to a phase transition from the wurtzite to the rock-salt structure (predicted to occur around $x = 0.66$ [10, 14], although in practice spinodal decomposition or other forms of decomposition of the alloy may occur before such high Sc contents are reached). Other important physical properties relevant to devices, for instance the piezoelectric coefficients, are also composition dependent [19, 20, 36]. However, conventional optical absorption measurements of the band gaps are not reliable, as the values obtained can vary by more than 1 eV for the same composition, depending on the types and densities of the defects in the films [18]. Additionally, X-ray diffraction measurements of the *c* lattice parameter (conventionally used for composition measurements of III-nitride alloy films [37–39]) may no longer be a reliable indicator of alloy composition. This is due to the decreasing *c/a* ratio with increasing Sc content and the consequent difficulty of separating the influence of the film strain state and the film composition on the *c* lattice parameter [39]. Therefore, new approaches to composition determination must be assessed and developed in order to understand the composition dependence of the properties of $Sc_xGa_{1-x}N$ alloys and use them effectively in device design and optimisation.

Rutherford backscattering spectroscopy (RBS) is an ion scattering technique used for making direct composition measurements without any standard, including compositional depth profiling for thicknesses of up to 2 μm without performing sputtering. The mass resolution of RBS is high, especially for light elements, and an accuracy of $\pm$ 1% is commonly accepted [40–42]. Another direct composition determination method is X-ray photoelectron spectroscopy (XPS), which is very surface sensitive. However, sputtering is required for depth profiling, which could affect the resulting spectrum, and the accuracy, which can be as poor as $\pm$ 10%, is not sufficient. Measurements from X-ray diffraction (XRD) and electron diffraction patterns are also assessed in parallel, may also serve as a guide to the composition, as is standard for conventional III-nitrides. However, the lattice parameters of thin film $Sc_xGa_{1-x}N$ may be affected by strain, which will limit the accuracy of this approach. Therefore, this study aims to evaluate and compare composition measurement methods to determine a suitable technique for use with $Sc_xGa_{1-x}N$ thin films.

## 2. Experimental details

Epitaxial wurtzite-structure (0001)-oriented $Sc_xGa_{1-x}N$ films were grown using molecular beam epitaxy (MBE) with an $N_2$ plasma source, under metal-rich growth conditions, with a substrate temperature of 750 $\pm$ 30 $^{o}$C (measured by optical pyrometry, using a 50 nm layer of IR-absorbing molybdenum deposited on the back of the wafers prior to growth). A 1 μm thick buffer layer of (0001)-oriented AlN grown by metal-organic vapour phase epitaxy (MOVPE) was deposited on the sapphire substrate at a temperature of 1130 $^{o}$C. The $Sc_xGa_{1-x}N$ film compositions were controlled by varying the Sc flux as measured by the current through a beam flux monitor (ranging from 1 nA to 4 nA) while keeping the Ga flux and the $N_2$ flow rate constant at values which produce a GaN growth rate of approximately 260 nm $hr^{-1}$. The resulting film thicknesses ranged from 220 nm to 590 nm. Full details of the growth procedure, film growth rates and microstructural characterisation of these films are given in [17] and full details of the band gap measurements from these films are given in [18] Further details on the growth of the AlN templates are given in [40].

Film compositions were determined using RBS as the reference. RBS measurements were performed using a beam of $^{4}$He at 2 MeV with an incidence angle of 3$^{o}$. The samples were kept under azimuthal rotation during the measurements. A standard detector was placed at 140$^{o}$ and two pin-diode detectors located symmetrically to each other at 165$^{o}$. The RBS data were analysed using the IBA DataFurnace NDF v9.6d [44]. A Thermo Scientific Kα instrument with a monochromated Al Kα X-ray source (1486.6 eV) was used for XPS composition analysis. A 360 s Ar ion etch was done prior to the XPS scan. An inelastic mean free path of 2 nm was assumed to apply for both GaN and ScGaN. Survey scans were performed by focusing X-rays at a spot size of 400 μm with 200 eV while 50 eV was used

for high resolution region scans. Sample charging was corrected by a dual beam charge compensation system using both electrons and low energy Ar cations and the binding energy scale was calibrated using Cu, Ag and Au. XRD for $c$ lattice parameter determination was performed using a PANalytical MRD with a Cu K$\alpha$ source. Transmission electron microscopy (TEM) analysis was carried out using a JEOL 2100 with a high angle annular dark field (HAADF) detector operated in both dark field and scanning transmission electron microscopy (STEM) modes at 200 kV. Cross-sectional TEM samples were prepared by mechanical grinding followed by ion polishing until electron transparency was reached. The *c/a* ratio was measured from the electron diffraction pattern at the $< 01\bar{1}0 >$ and $< \bar{2}110 >$ zone axes.

## 3. Results and discussion

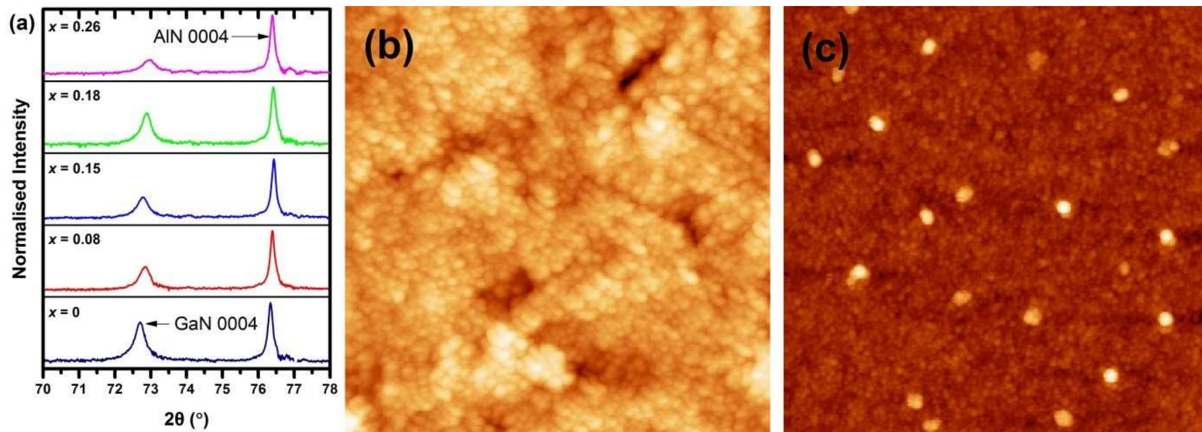


**Figure 1.** (a) XRD ω-2θ diffractograms of the 0004 reflection for $Sc_xGa_{1-x}N$ films grown on MOVPE AlN, and 1 x 1 μm AFM scans of (b) GaN (Z-scale = 28 nm) and (c) $Sc_{0.26}Ga_{0.74}N$ (Z-scale = 12 nm) films. All AFM images were processed using parabolic flattening.

X-ray diffractograms from all films are shown in Figure 1(a), confirming that they are epitaxial with the (0001) orientation. The shift of the GaN 0004 peak position to higher angles with increasing $x$ indicates that the $c$ lattice parameter decreases as the Sc content increases, despite the in-plane compressive strain that would be expected to occur in $Sc_xGa_{1-x}N$ due to its in-plane lattice mismatch relative to AlN. A slight increase in the FWHM of the peaks from 0.23˚ to 0.30˚ as the Sc content increases from $x = 0$ to $x = 0.26$ is also observed due to the relative misorientations of the grains, consistent with the TEM data reported in Ref. [17]. AFM data indicate a relatively smooth film surface with a hillock morphology typical of III-nitride films alloyed with transition metals.

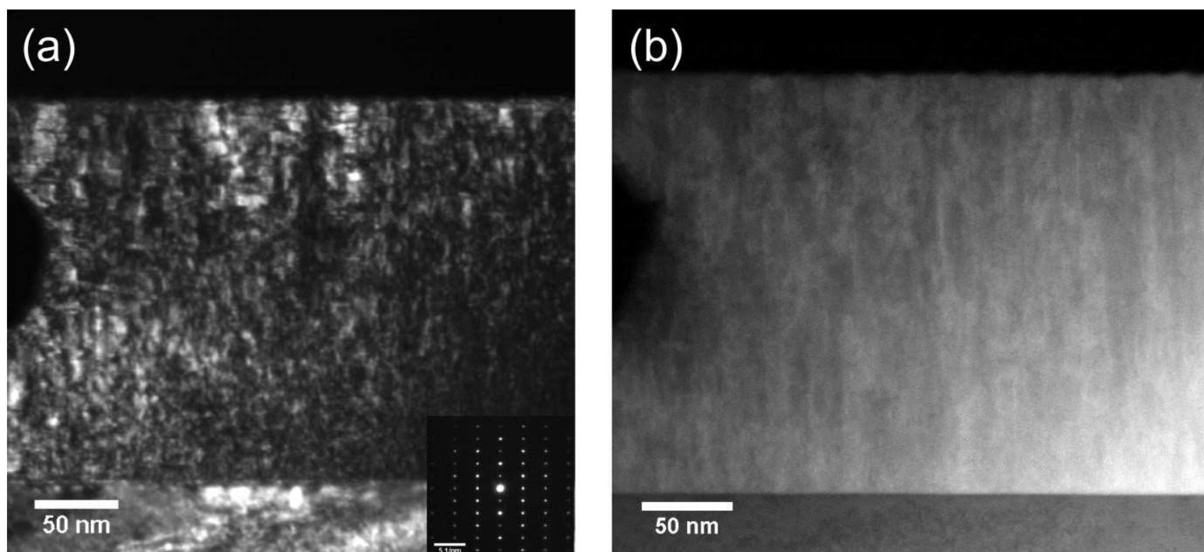

**Figure 2.** (a) Cross-sectional dark field TEM image along the $< 11\bar{2}0 >$ zone axis of $Sc_{0.258}Ga_{0.742}N$ film, insert: diffraction pattern of the $< 11\bar{2}0 >$ zone axis; (b) cross-sectional STEM image of the same film region in (a).

Figure 2 (a) and (b) show typical cross-sectional dark field TEM and STEM images of the $Sc_xGa_{1-x}N$ films with $x = 0.26 \pm 0.01$ (the sample with the highest Sc content that still retains a phase-pure wurtzite structure). The dark-field TEM shows significant non-uniform strain contrast due to a high density of crystalline defects, which are known to include dislocations along with stacking faults and lamellar faults oriented within the (0001) plane [7, 8, 17]. However the single crystal diffraction patterns confirm that the films are epitaxial and single phase. The STEM image indicates that the films are compositionally uniform throughout the film thickness (minor non-uniformities in contrast are consistent with strain effects as revealed in the dark field image).

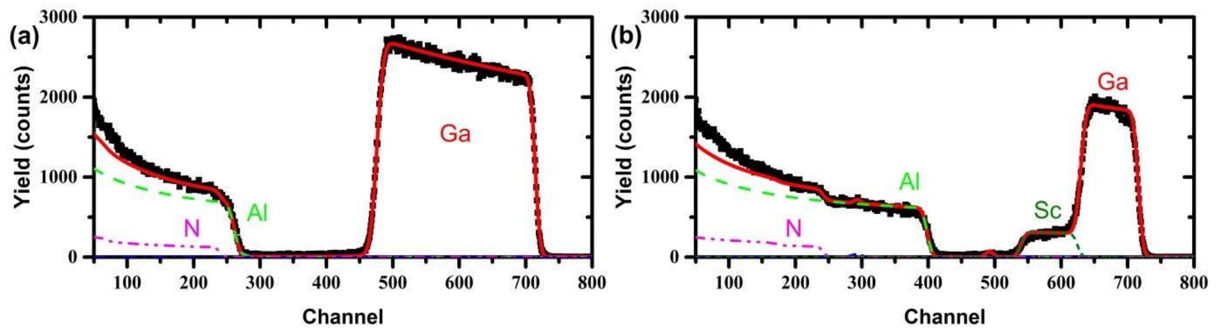


**Figure 3.** RBS spectra (taken at an incident angle of 3˚) for (a) GaN and (b) $Sc_{0.26}Ga_{0.74}N$.

Figure 3 shows typical measured and modelled RBS spectra obtained from both GaN and $Sc_xGa_{1-x}N$, in which a good fit is obtained to the data for all samples. As the signals from Sc and Ga can be distinguished clearly from each other, composition determination through the simulation of the RBS curve is straightforward and a 1% accuracy can be achieved. Therefore, the RBS data are used as a reliable standard to which other composition measurement techniques can be compared. Channeling effects were observed in the measurement with an incident angle of 0˚ (along the growth direction) but minimal effects were observed for other angles of tilt. This indicates that the films are highly oriented in the growth direction but contain some in-plane misorientation in other directions, consistent with the TEM studies of these films reported in Ref. [17]. Figure 4(a) reveals a linear relationship between the Sc flux and $x$ in $Sc_xGa_{1-x}N$ as measured by RBS. For these metal-rich growth conditions, and after suitable calibration using RBS, the Sc flux measured during growth is a very useful guide to the Sc content of the resulting film.

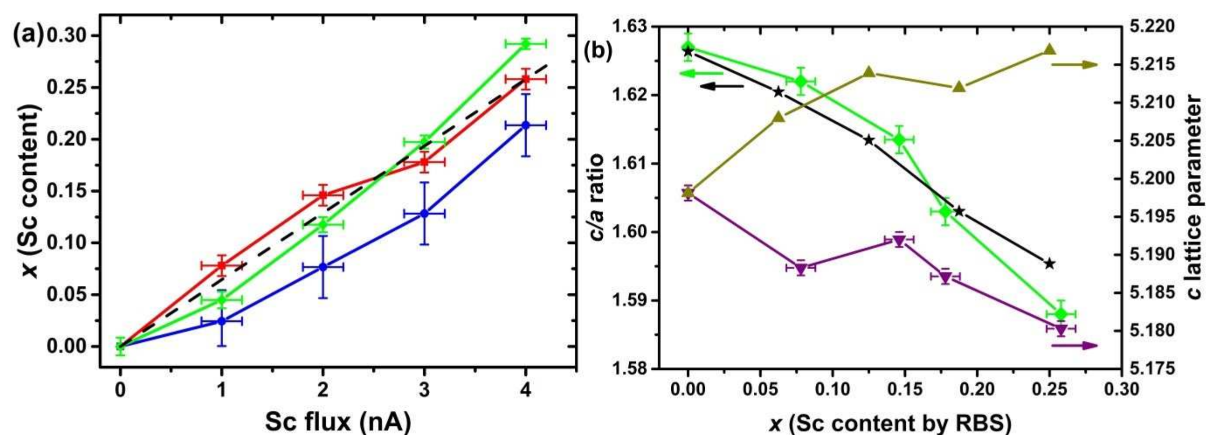


**Figure 4.** (a) The relationship between the Sc flux and $x$ in $Sc_xGa_{1-x}N$ as measured by RBS (red squares and the dotted line indicates a linear relationship), XPS (blue circles), *c/a* ratio measured from electron diffraction patterns and fit to theoretical data from [14] (green diamonds); (b) *c/a* lattice parameter ratio: predicted (black stars, from [14]) and experimental (green diamonds, from electron

diffraction); *c* lattice parameter: predicted (dark yellow upward triangle, from [14]) and experimental (purple inverted triangle, from XRD)

The Sc contents of the $Sc_xGa_{1-x}N$ films were then measured by XPS. By integrating the area of each constituent peak, the ratio of Sc and Ga can be determined and the results are shown in Figure 4(a). When compared to the RBS data, the XPS data consistently underestimate the Sc content by 5–8%. This can be accounted for by two possible factors. XPS carried out using Al Kα radiation is highly surface sensitive, with a sampling depth < 10 nm. The samples were grown using metal-rich conditions and therefore a metal-rich surface layer is expected to be present. A Ga-enriched surface would produce a lower Sc content as measured by XPS [45]. Additionally, ion sputtering is expected to remove the Sc and N faster than Ga from the surface (the bond energy of ScN 6.72 eV/atom [46] is lower than that of GaN 8.92 eV/atom [47, 48], therefore a relatively greater sputtering rate for Sc atoms is expected compared to Ga atoms).

To explore this further, as additional two series of $Sc_xGa_{1-x}N$ samples (full details given in [18]) were investigated using XPS, the Sc content measured by XPS varied slightly at the same Sc flux for the samples with different buffer layers. For instance, at a fixed Sc flux of 1 nA, the measured Sc contents $x$ are 0.06, 0.04 and 0.02 for samples with buffer layers of GaN grown by molecular beam epitaxy, GaN grown by MOVPE and AlN grown by MOVPE respectively. This effect became more pronounced as the Sc flux was increased to 4 nA, where the Sc contents measured by XPS are 0.31, 0.27 and 0.21 respectively. This is likely due to the differences in surface polarity expected to occur between III-nitride films grown by MBE (normally N-polar) and MOVPE (normally Ga-polar) [49, 50], as well as differences in the film strain which are well known to affect the relative incorporation of alloy constituents having different ionic radii [51, 52] and which may also affect the relative surface accumulation.

The Sc contents of the $Sc_xGa_{1-x}N$ films were subsequently assessed using lattice parameter measurements derived from both XRD and electron diffraction patterns. Because the *c* and *a* lattice parameters of $Sc_xGa_{1-x}N$ are expected to vary differently as a function of $x$, both the *c/a* ratio from the electron diffraction patterns and the *c* lattice parameter from XRD need to be compared to theoretical predictions in order to link the lattice parameter data to the composition. As shown in Figure 4(b), the *c/a* lattice parameter ratio of $Sc_xGa_{1-x}N$ derived from electron diffraction data decreases as the Sc content increases, in line with theoretical predictions [14]. This is reasonable, since the $Sc_xGa_{1-x}N$ film thicknesses are significantly larger than the critical thicknesses for strain relaxation predicted from theory [15], and therefore the films are expected to be strain-relaxed. However the absolute *c* lattice parameter values as measured by XRD deviate slightly from the predicted values. This may be related to the presence of hydrostatic strain, which may be caused, for example, by substitutional impurities and/or variations in carrier concentrations. Importantly though, the change in *a* lattice parameter as a function of Sc content is much greater than that of the *c* lattice parameter: the *a* lattice parameter increases by 3.4% as Sc content increases from $x = 0$ to $x = 0.3$ while the *c* lattice parameter increases by only 0.3% [14]. Therefore the trends in *c/a* ratio are dominated by the (relatively large) change in the *a* lattice parameter, whereas small absolute variations or small measurement errors in the *c* parameter alone could be enough to cause significant deviations from the expected trend. Therefore measurements of trends in the *c* lattice parameter are not reliable for composition determination. Instead, as both experiment and theory are consistent for the variation of *c/a* ratio as a function of Sc content, the film composition can be estimated by fitting the measured *c/a* ratio to the theoretical trend. However, comparing the RBS results and $x$ values estimated from the *c/a* ratio, it is clear that $x$ is underestimated at low Sc contents but overestimated at high Sc contents (Figure 4(a)), likely due to the influence of the aforementioned issues regarding the *c* lattice parameter. Therefore, Sc concentration determination using *c/a* ratio measurements also has limited reliability.

**4. Conclusion**

In conclusion, the compositions of a series of $Sc_xGa_{1-x}N$ films were analysed using RBS as an accurate reference method and compared to three other composition measurement approaches. Surface-sensitive XPS analysis results in Sc content values $x$ which are 5–8% lower than values from RBS and which vary depending on the III-nitride buffer layer on which the films were grown. This is attributed to the presence of a surface metal accumulation layer in which the Ga content may be enriched compared to the rest of the $Sc_xGa_{1-x}N$ film. In future it may be possible to quantify $Sc_xGa_{1-x}N$ film compositions grown on a specified buffer layer using XPS with the aid of a calibration curve based on high-quality reference RBS data. On the other hand, although overall the correct trend is observed from *c/a* ratio as a function of Sc content, the film composition cannot be determined accurately due to the uncertainty in the strain state of the $Sc_xGa_{1-x}N$ films, the uncertainty in the effect that the defect microstructure may have on the *c/a* ratio of the $Sc_xGa_{1-x}N$ films, and the lack of accurate experimentally determined reference lattice parameters for $Sc_xGa_{1-x}N$, combined with uncertainty in the accuracy of the reference values obtained from DFT calculations. In contrast, a linear relationship was found between the Sc flux and the Sc content determined by RBS (the reference technique), such that after calibration the Sc flux used during growth can be used to provide a reasonable estimation of the amount of Sc incorporated during the growth of $Sc_xGa_{1-x}N$ films.

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