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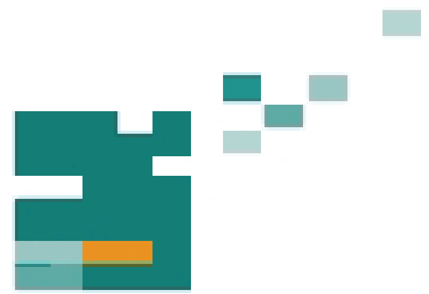

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FACULTY OF ELECTRICAL ENGINEERING AND INFORMATION SCIENCE

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Susanne Jakob
Dipl.-Ing. Helge Drumm

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G. Ecke, T. Baumann, O. Ambacher

Composition Measurements of Group-III Nitride Ternary and Quaternary Compound Nanostructures by AES

Auger Electron Spectroscopy (AES) is a widely applied method for the determination of the composition of surfaces and thin films of compounds and alloys. Nevertheless, the accuracy of the measurements depend heavily on the quality of the standards. Paying attention to this it is necessary to measure own standard samples of the interesting material system under comparable measurement conditions. For these measurements one has to define verry carefully the measurement parameters, for excample primary electron energy, the retard ratio of the analyser and the sample tilt. All parameters must be choosen in order to get the best performance of the Auger spectrometer, that means good spatial resolution, energy resolution and a low detection limit.

AES is a surface sensitive technique, because of this it is essential to eliminate surface contaminations by sputtering of the samples with noble gas ions. However, some additional sputtering effects cause alterations of the surface. The original composition can not be detected directly, because of preferential sputtering[1], sputtering induced atomic mixing, surface roughness, diffusion and segregation. These effects can be excluded as far as possible, if the standard sample of a comparable composition is sputtered and measured under the same conditions. Fig. 1 shows the position of the used standard samples in the system GaN-InN-AlN.

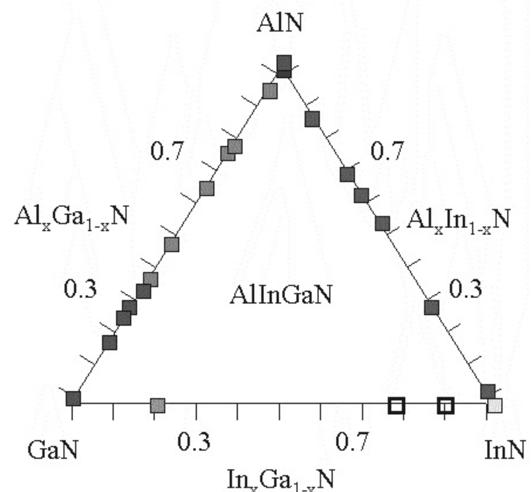

Fig. 1.: Position of the standard samples in the group-III nitride system

There are many publication in which the peak-to-peak-hight (pph) in the diffentiated Auger spectrum is taken as the measure of the Auger intensities in order to estimate the composition of the sample. But it is known, that changes in the Auger peak shape annihilate the proportionality of pph and concentration. We prefer the use of the direct spectrum. Here the measured signal is the Auger peak

area, which can be acquired by subtraction of the background and noise. This critical procedure can be performed by the Shirley approximation [2], but attention should be paid on the position of the borders of the peak area. Also the superposition of two peaks need to be analysed. For example, in InN the In MNN and the N KLL peaks are overlapping even with a good analyser energy resolution, so that both peaks can only be separated by a special peak overlapping approximation.

We carried out the quantification procedures for the binary semiconductors GaN, AlN and InN and than for the AlGaN, the InGaN and the AlInN material systems for two primary electron energies (5 kV and 25 kV) and for two sample tilt angles (0° and 60° with respect to the surface normal). These experimental set-ups lead to the following experimental parameters:

sample tilt (°)	primary electron energy (kV)	primary electron incidence angle (°)	angle between sample and analyser (°)	angle of ion incidence (°)
0	5	0	60	67.5
0	25	0	60	67.5
60	5	60	0	43.4
60	25	60	0	43.4

Table 1: experimental conditions of the quantification measurements

The first step of this work was the estimation of the sensitivity factors for the binary semiconductors GaN, AlN and InN. Table 2 gives the results for the sensitivity factors for the dominant Auger transitions of the three binary semiconductors.

Auger transition	Al (KLL) in AlN	N (KLL) in AlN	Ga (LMM) in GaN	N (KLL) in GaN	In (MNN) in GaN	N (KLL) in InN
5 KV, 0°	0,160	0,220	0,212	0,140	0,497	0,0218
25 KV, 0°	0,181	0,233	0,225	0,155	0,647	0,0462
5 KV, 60°	0,227	0,203	0,258	0,111	0,337	0,0262
25 KV, 60°	0,300	0,281	0,308	0,142	0,405	0,0305

Table 2: Sensitivity factors for the Auger transitions of AlN, GaN and InN

The next steps of the work were the measurements and data evaluation of the ternary nitrides AlGaN, InAlN und InGaN with variable compositions.

In the following paragraph the results are presented for the $Al_xGa_{1-x}N$ system. The procedure has been the same for the other ternary group III-nitrides. Fig. 2 shows the composition of the AlGaN layers, measured by an additional measurement technique such as x-ray diffraction or ellipsometry and the corresponding surface composition measured by AES after sputter cleaning of the sample surface.

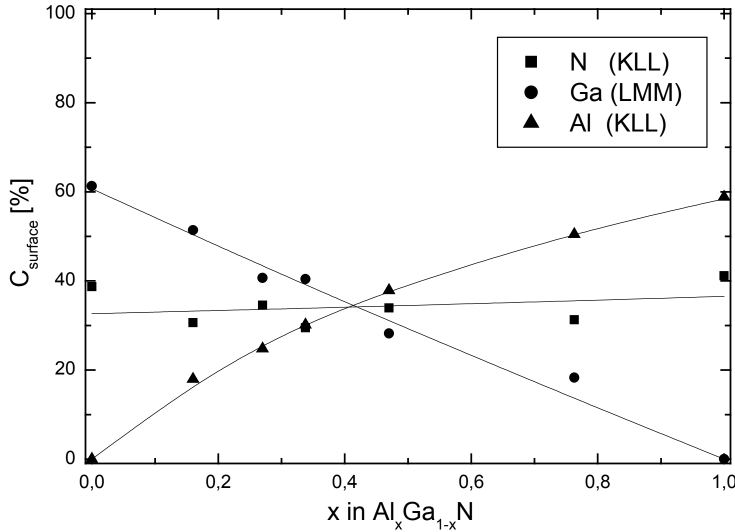

Fig. 2: Surface concentrations measured by AES in dependence on the real film composition of $Al_xGa_{1-x}N$ films for 0°, 5 kV electron bombardment

Fig. 3 demonstrates the measured Al content x at the surface of the sputtering induced altered layer in dependence on the real film composition of $Al_xGa_{1-x}N$ films.

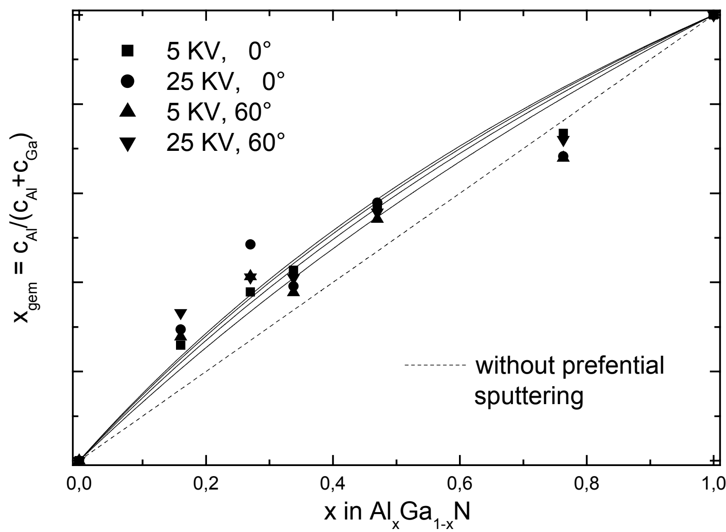

Fig. 3: Surface Al content x measured by AES in dependence on the real film composition of $Al_xGa_{1-x}N$ films

The dashed line demonstrates the theoretical behaviour in the case, that no preferential sputtering would occur. The strength of the bending, of the discrepancy from this dashed line depends on the preferential sputtering Y_{Al}/Y_{Ga} with Y being the sputtering yield. From these dependencies the preferential sputtering behaviour has been calculated for all ternary group III-nitride semiconductor systems for ion bombardment under 67.5° and 43.4°, respectively, with respect to the surface normal for 1 kV Argon.

The preferential sputtering for AlGaN, InGaN and AlInN is summarized in table 3.

	Y_{Ga} / Y_{Al} for AlGaN	Y_{In} / Y_{Ga} for InGaN	Y_{In} / Y_{Al} for InAlN
67.5° , 1 kV, Ar	1.55	2.8	2.4
43.4° , 1 kV, Ar	1.5	3.5	3.2

Table 3: Preferential sputtering behaviour for AlGaN, InGaN and AlInN, measured by AES

With the help of the sensitivity factors and the preferential sputtering behaviour it is possible, to quantify AES measurements of unknown group III-nitride semiconductor layers, even in nanostructures. But the matrix effects have to be corrected during the quantification procedure. These matrix corrections include the attenuation length of the Auger electrons, the backscattering factor and the atomic density correction. Because of the dependence of these three values on the target composition itself, the quantification process becomes an iteration. It could be found, that after 3 to 4 iterations the calculated concentrations converge and the iterations can be aborted. This procedure of the quantification of ternary and quaternary group III-semiconductor layers has been programmed by a simple excel program.

CONCLUSIONS

Finaly, the composition of group III-nitride ternary and quaternary semiconductor materials can be successfully determined by AES with good accuracy. These investigations can be applied even to nanostructures characterising the composition in resonator bars, optical emitters and sensor structures, where the composition can derive from those of unpatterned structures for the same growing conditions. Here the knowledge of the composition is essential because it defines the electrical properties of the device structure.

References:

[1] A.A. Promokhov et al., Vacuum 56 (2000), 247
[2] D.A. Shirley, Phys. Rev. B5 (1972), 4709

Authors:

Dr. Gernot Ecke
Dipl.-Ing. Tim Baumann
Univ. Prof. Dr. rer. nat. habil. Oliver Ambacher
TU Ilmenau, ZMN, P.O.B. 100560
Institute for Micro- and Nanotechnologies
D-98684 Ilmenau
Phone: +49 (0) 3677 69 3407
Fax: +49 (0) 3677 69 3355
E-mail: gernot.ecke@tu-ilmenau.de