X-RAY PHOTOEMISSION SPECTROMICROSCOPY OF GaN AND AlGaN

G. F. LORUSSO *, H. SOLAK *, F. CERRINA *, J. H. UNDERWOOD **, P. J. BATSON **, Y. KIM #, Y. CHO #, C. KISIELOWSKI #, J. KRUEGER #, AND E. R. WEBER #
*Center of X-ray Lithography, University of Wisconsin, Madison, WI
**Center of X-ray Optics, Lawrence Berkeley Laboratory, Berkeley, CA
#Department of Material Science and Mineral Engineering, University of California, Berkeley, CA.

ABSTRACT

We investigate here for the first time GaN and AlGaN films by using x-ray photoemission spectromicroscopy. As compared to conventional x-ray photoemission spectroscopy (XPS), spectromicroscopy can provide spatially resolved information on the chemical composition of the sample surface. The experimental results where obtained by using MAXIMUM, a scanning photoemission microscope installed on 12.0 undulator beamline at the Advanced Light Source (ALS), Berkelely, allowing for a spatial resolution of 100 nm. We investigate here GaN and AlGaN thin films grown on sapphire substrate by metalorganic chemical vapor deposition (MOCVD). The results clearly indicate the great potential of spectromicroscopy in investigating chemical inhomogeneity, inpurities and localization in GaN and AlGaN thin films.

Figure 1: Secondary electron X-ray photoemission micrograph of AlGaN thin film (sample A). The field is 60 μm by 30 μm. The kinetic energy is E_K=5 eV. This image clearly shows the surface morphology of the sample, indicating a mean grain size of about 2 um, in agreement with other atomic force microscope results.

Mat. Res. Soc. Symp. Proc. Vol. 512 © 1998 Materials Research Society

INTRODUCTION

Gallium nitride and related wide band gap semiconductors are an important class of electronic materials because of potential use in optoelectronic devices operating in the blue range [1]. As a consequence, many efforts have been devoted to investigate the electronic structure of such nitrides in the last few years, also by means of x-ray photoemission spectroscopy (XPS) [2]. Among other results, these investigations indicated a substantial band bending due to Fermi level pinning at the sample surface, related to intrinsic localized surface states [3].

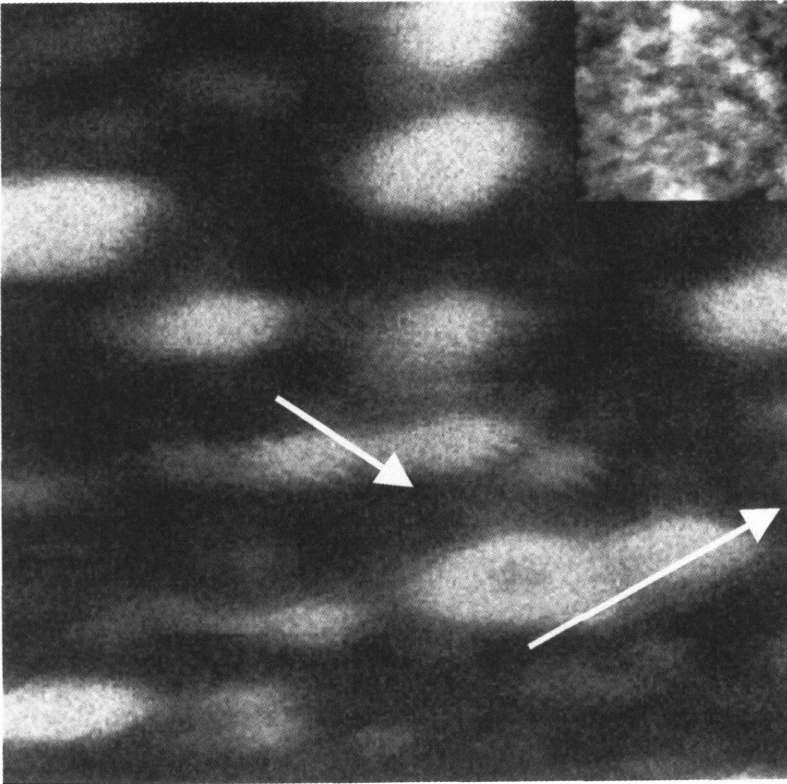

Figure 2: Close-up of the morphological structure of an AlGaN film. The field is 12 μm by 12 μm. The kinetic energy is E_K=5 eV. The grains show a fine structure, related to their crystalline orientation (arrows). The inset shows a AFM image with sam field on the same sample.

Although XPS has been widely used to study GaN and related compounds, no investigation using spectromicroscopy has been reported up to now, according to our knowledge. As compared to conventional XPS, spectromicroscopy [4] can provide spatially resolved information on the chemical composition of the sample surface, as well as standard morphological and chemical analysis. Hence, this technique provides a deeper insight in the puzzling microstructure of these compounds, characterized by lateral inhomogeneity and by dislocation densities several order of magnitude above those in other semiconductors.

We investigate here for the first time GaN and AlGaN films by using x-ray photoemission spectromicroscopy. The experimental results have been obtained by using MAXIMUM [5], a scanning photoemission microscope installed on bl 12.0 undulator beamline at the Advanced Light Source (ALS), Berkeley, allowing for a spatial resolution of 100 nm. We examined GaN and AlGaN samples grown on sapphire substrate by metal-organic chemical vapor deposition (MOCVD). This preliminary analysis clearly indicates the great potential of spectromicroscopy in investigating chemical inhomogeneity, impurities and localization in GaN and AlGaN films, providing detailed information on the chemistry and on the morphology of the investigated systems in the submicron range.

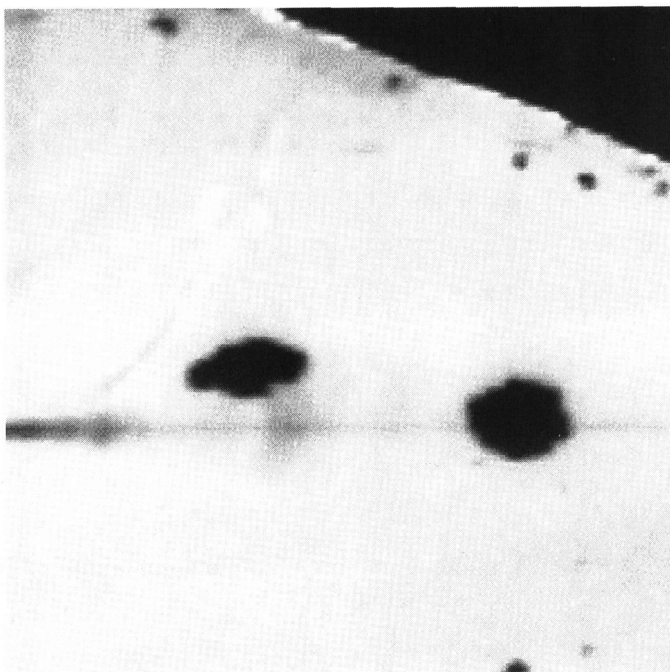

Figure 3: GaN surface with hillocks. The field is 50 μm by 50 μm. The kinetic energy is E_K=5 eV.

EXPERIMENTALS

In 1987, the University of Wisconsin, in collaboration with the ALS, started the development of an x-ray microscope system of the scanning type with the goal of reaching a spatial resolution better than 0.1 μm, a spectral resolution better than 300 meV, and a base pressure of 10^{-10} torr. All these goals were achieved in 1992, and the photoemission microscope MAXIMUM was originally installed at the Synchrotron Radiation Center (SRC). During the period from 1992 to 1995, the microscope was successfully used to study semiconductor surfaces, interfaces, biological samples and organic particles. However, during the operation of the microscope it become evident that the microscope performance was severely hampered by the relatively low brightness of SRC, which limited the available flux at the microscope's focus and, consequently, the achievable spatial resolution. In order to overcome these problems, the microscope was moved to the Advanced Light Source (ALS) in April 1995, where it was temporarily installed on the bend magnet beamline 6.3.2. In August 1997, the installation of the microscope in its final location (beamline 12.0) has been completed. The testing of the microscope evidenced a spectral resolution of 250 meV, a lateral resolution of 0.1 μm in photoemission mode, and a flux on the pinhole of 4×10^{14} ph/sec at 130 eV.

The microscope works as in the following. Radiation from the synchrotron source is monocromatized and focused by a Kirkpatrick-Baez system to illuminate a pinhole, which serve as source for the microscope optics. A multilayer coated Schwarzschild objective (SO) produce an image of the pinhole with a 20x demagnification at 130 eV. When a sample is placed at the focus, photoelectrons are collected by a cylindrical mirror analyzed (CMA) electron spectrometer. The sample is mounted on a scanning stage, and by rastering the sample it is possible to produce a 2-d photoemission image.

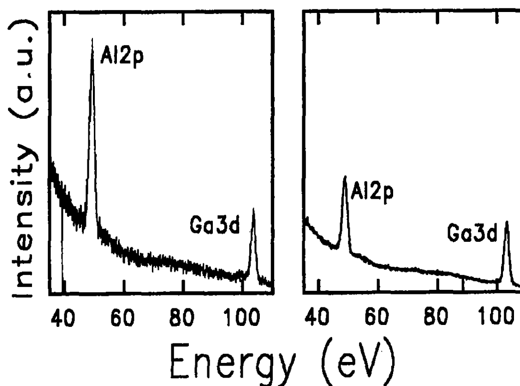

Figure 4: EDCs of the investigated AlGaN films. The results evidence the difference in Al concentration between sample A and B (x=0.23 and x=0.51, respectively).

We investigate here GaN and $Al_xGa_{1-x}N$ thin films deposited by MOCVD on sapphire, having different concentration x of Al and different thickness: sample A (#2922), x=0.23 and 4350 Å thick, and sample B (#2927), x=0.51 and 6540 Å thick. The chemical composition of the films has been analyzed by means of Rutherford backscattering spectrometry (RBS). The results show that both of the films have Ga rich surfaces, especially sample A. Sample A shows a top layer ($5x10^{17}$ atoms/cm^2, 550Å thick) with $Al_{0.135}Ga_{0.44}N_{0.425}$. In the case of sample B, the top layer ($2.5x10^{18}$ atoms/cm^2, 2770Å) shows $Al_{0.255}Ga_{0.244}N_{0.50.}$ Standard XPS spectroscopy has been performed on both the sample.

RESULTS AND DISCUSSION

In Fig. 1 we report a x-ray photoemission micrograph of Sample A. The image evidences the grain structure of the surface morphology. It has been acquired at E_K=5 eV (i.e. imaging secondary electrons) and its size is 60 x 30 μm. The gain size deduced from this image (about 2 μm) is in good agreement with the results of atomic force microscope (AFM) analysis. In Fig. 2 a close-up of the surface grain structure is reported. The image size is 12 x 12 μm, at the same kinetic energy of Fig. 1. The image shows in detail the granular structure of the investigated samples, evidencing also the crystalline orientation of some of the grains (arrows in Fig. 2). These results indicate that X-ray photoemission microscopy can give high quality morphological information on the surface of the samples. In Fig. 3 we report a x-ray photoemission micrograph of a GaN sample grown by MOCVD. The image evidences the presence of hillocks on the surface. It has been acquired at E_K=5 eV (i.e. imaging secondary electrons) and its size is 50 x 50 μm.

In Fig. 4 we report the electron distribution curves (EDCs) of the two investigated samples. The relative intensity of the Al2p at E_K=49.9 eV (E_B=75.8 eV) and the Ga3d at E_K=103.4 eV (E_B=22.4 eV) indicates the change in the Al concentration. By accounting for the x-ray photoemission cross section, it is possible to have a quantitative evaluation of the Al/Ga ratio in the two samples. The results agree quite well with the ones obtained by means of RBS and standard XPS, thus allowing an *in situ* evaluation of the surface composition.

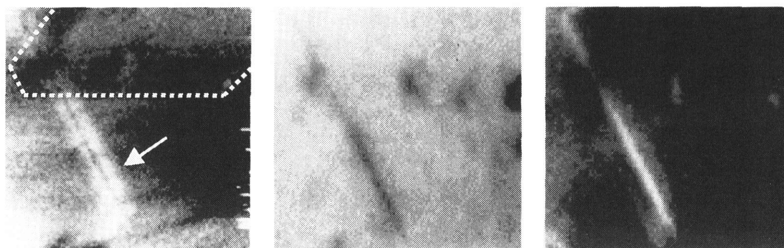

Figure 5: XPS microimages of sample B at (a) 5 eV (secondary electrons), (b) 21 eV (Ga3p), and (c) 50 eV (Al2p). A rhombohedral feature (dashed line) and a crystallographic plane (arrow) are evident in (a).

In Fig. 5, we report three XPS microimages of sample B at different kinetic energy. The energies are 5 eV (secondary electrons), 21 eV (Ga3p), and 50 eV (Al2p), respectively. The size is 100 x 100 µm. In Fig 4a, the secondary electron images shows the morphology of the sample surface. A rhombohedral feature (dashed line) is evident in the upper part of Fig. 1a. The diagonal line (arrow) is a crystallographic plane at 60 degrees with respect to the feature side. The chemical mapping of Ga and Al (Figs. 4b and 4c, respectively) clearly indicate an Al excess and a Ga deficiency in the crystallographic plane.

CONCLUSION

We reported here some preliminary results on GaN and AlGaN by using x-ray photoemisison spectromicroscopy. The analysis clearly indicates the ability of spectromicroscopy to perform simultaneously morphological and chemical analysis *in situ*, as well as microscopic elemental mapping. Our results suggest future promising applications of this technique in investigating chemical and morphological inhomogeneity in AlGaN. This work is mainly intended to stimulate investigators in the field to use MAXIMUM. Beamtime on this spectromicroscope can be obtained by contacting the Principal Investigator: Franco Cerrina, Dept of Electrical and Computer Engineering, University of Wisconsin-Madison. Email: cerrina@xraylith.wisc.edu. Telephone: 608/263-4955

REFERENCES

1. S. Nakamura, M Senoh, N. Iwasa, Jpn. J. Appl. Phys. 34, L797 (1995); S. Nakamura, M Senoh, S. Nagahama, N. Iwasa, T. Yamada, T. Matsushita, K. Kiyoku, and W. Sugimoto, Jpn. J. Appl. Phys. **35**, L74 (1996).
2. G. Martin, S. Strite, A. Botchkarev, A. Agarwal, H. Morkoc, W. R. Lambrecht, and B. Segall, Appl. Phys. Lett. **65**, 610 (1994).
3. V. M. Bermudez, T. M. Jung, K. Doverspike, and A. E. Wickenden, J. Appl. Phys. **79**, 110 (1996).
4. G. Margaritondo and F. Cerrina, Nucl. Instr. and Meth. **A291**, 26 (1990).
5. C. Capasso, A. K. Ray-Chaudhuri, W. Ng, S. Liang, R. K. Cole, J. Wallace, F. Cerrina, G. Margaritondo, J. H. Underwood, J. K. Kortright, and R. C. C. Perera, J. Vac. Sci. Technol. **A9**, 1248 (1991).