Spatially Resolved Characterization of Plastic Deformation Induced by Focused-Ion Beam Processing in Structured InGaN/GaN Layers

R. Barabash[1,2], G. Ice[1], R. Kroger[3], H. Lohmeyer[3], K. Sebald[3], J. Gutowski[3], T. Bottcher[3], D. Hommel[3], W. Liu[4], and J.-S. Chung[1,5]

[1]Materials Science and Technology, Oak Ridge National Laboratory, One Bethel Valley Road, Oak Ridge, TN, 37831-6118

[2]University Of Tennessee, Knoxville, TN, 37996

[3]Institute of Solid State Physics, Bremen, Germany

[4]Advanced Photon Source, Argonne, IL, 60439

[5]Soongsil University, Seoul, Korea, Republic of

ABSTRACT

Polychromatic X-ray microbeam analysis (PXM) results of structural changes caused by FIB machining in nitride heterostructures are discussed in connection with micro-photoluminescence (μ-PL), fluorescent analysis, scanning electron (SEM) and transmission electron microscopy (TEM) data. It is shown that FIB processing distorts the lattice in the InGaN/GaN layer not only in the immediate vicinity of the processed area but also in surrounding volumes. A narrow amorphized top layer is formed in the direct ion beam impact area.

INTRODUCTION

Focused-ion beam (FIB) machining is a promising technique for the realization of novel micro- and nanostructured optoelectronic devices- especially for the important nitride materials system [1], which is difficult to process by conventional etching techniques. For example, there is intense recent research directed at the application of FIB machining to create vertical semiconductor microcavities that possess three-dimensionally confined optical modes for surface-emitting lasers. Because these and other potential optoelectronic devices are sensitive to defect distributions, there is a strong need to assess the damage induced on GaN and related materials by FIB machining.

Although widely used for TEM-sample preparation and for processing of edge-emitters, FIB is increasingly applied to prepare mesa-structures for single-dot spectroscopy and for the preparation of micropillars out of planar vertical-cavity surface-emitting lasers (VCSEL) samples. In such materials induced damage can be important. Of course the damage induced by FIB machining has been previously studied in Si [2] and other materials [3]. For the nitrides however, there are only a few reports with respect to possible ion damage induced by FIB machining and similarly few reports on the induced damage impact on mesoscale structure and optical properties [4]. Below we present the results of polychromatic X-ray microbeam analysis of the damage in InGaN layers structured with FIB. The results are complemented by fluorescence, SEM, TEM and micro-photoluminescence (μ-PL) analyses.

EXPERIMENTAL DETAILS

A FEI Nova 200 NanoLab FIB system was used to prepare reference structure samples consisting of InGaN/GaN multi quantum wells grown by metal-organic vapor phase epitaxy on GaN on sapphire templates (Fig. 1). Some samples were capped with 100nm SiO_2 layer before FIB processing. Both capped and uncapped samples were studied. Each structure consists of several trenches typically 2 µm wide and 20 µm long with varying distances between the trenches. Trenches were etched down to the sapphire substrate using 30 keV Ga-ions with ion-beam currents varying from 300 pA to 7 nA.

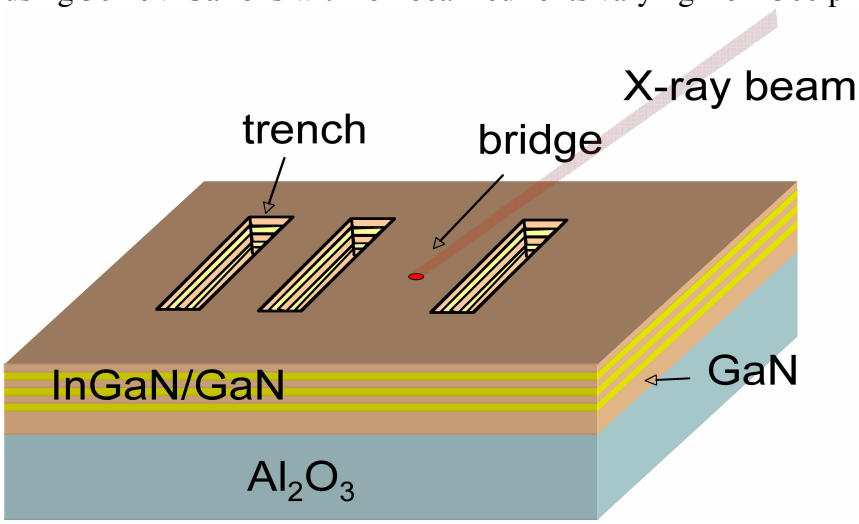

Figure 1. Schematic of the relative orientation of the X-ray microbeam (red spot) and the FIB processed trenches in the InGaN/GaN multilayer with trenches on sapphire.

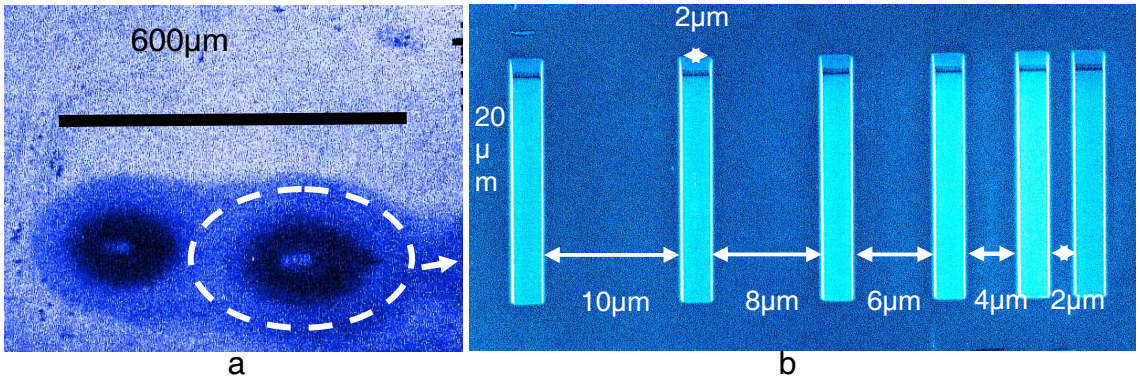

Figure 2. (a) Optical image of the structured region with several structures performed with different ion-beam currents in an uncapped sample; (b) Magnified SEM image of a typical FIB structure with varying distances between the trenches.

Several groups of trenches were processed for each sample. Two such groups in the middle of the shadowed area are shown in Fig. 2a. Typical FIB structures processed with FIB in the InGaN/GaN multi quantum wells on sapphire templates consisted of several bridges with varying distances between trenches (Fig. 2b). Further details of the FIB process can be found in Ref. [1].

Polychromatic X-ray microdiffraction (PXM) was used for spatially resolved structural investigations. PXM was performed with a focused ~0.5 μm diameter polychromatic synchrotron beam which hit the sample surface at 45° incidence (Fig. 1). The Laue diffraction patterns from both the InGaN layer and the sapphire substrate were recorded with a charge-coupled device (CCD) area detector placed at an angle of 90° relative to the incident beam [5 - 7]. To get depth resolved information about the strain distribution in the GaN layers we performed measurements with the so called DAXM technique [5].

With DAXM, a three-dimensional map of the local crystallographic orientation and elastic strain tensor distributions in a sample can be determined. The three dimensional orientation distributions can be used to directly determine the dislocation tensor field. Further details on the experimental setup, the data collection and quantitative analysis of diffraction profiles in PXM and DAXM can be found elsewhere [5 - 7].

The samples were also characterized by scanning electron microscopy (SEM) and by orientation imaging microscopy (OIM) using the field emission microscope of a FEI NOVA 200 focused ion beam system.

RESULTS AND DISCUSSION

Severe damage was observed in the uncapped sample. Micro Photoluminescence (μ-PL) measurements of the uncapped sample show that μ-PL intensity starts to decrease within ~200 μm of the structures area. On the structure itself no μ-PL- signal could be detected independent of beam current. We assume a chemical reaction between the Ga cloud and the uncapped GaN surface (Compare shadows around each structure at Fig.2a) may account for this loss of PL intensity.

In the case of the capped samples there was no change of the μ-PL intensity in the area surrounding the trenches. Therefore it was possible to measure the change of the PL-intensity at the different bridges (Fig. 3).

A scan of μ-PL intensity across the 10 μm- bridge (Fig. 3) starts in the middle of the trench and goes to the middle of the next trench. For each 10 μm - bridge the spectral position of the μ-PL intensity changes. It increases, stays practically constant in the middle of the bridge and decreases again when the laser spot reaches the end of the bridge. The reliable part of the measurement lies between two vertical lines, which correspond to the situation when the whole laser-spot is on the bridge. The change of the spectral position of the GaN signal may be caused by stress relaxation.

TEM analyses in the vicinity of the trenches shows a high density of dislocations and a narrow (~ 2nm) amophized top layer.

To investigate the interaction of stress relaxation and FIB induced structural damage, we used polychromatic x-ray microdiffraction (PXM) method. A typical PXM image for structured InGaN/GaN layers on sapphire substrate is shown in Fig. 4a. Due to the large penetration depth of the x-rays, the sapphire reflections are superimposed on those from the GaN.

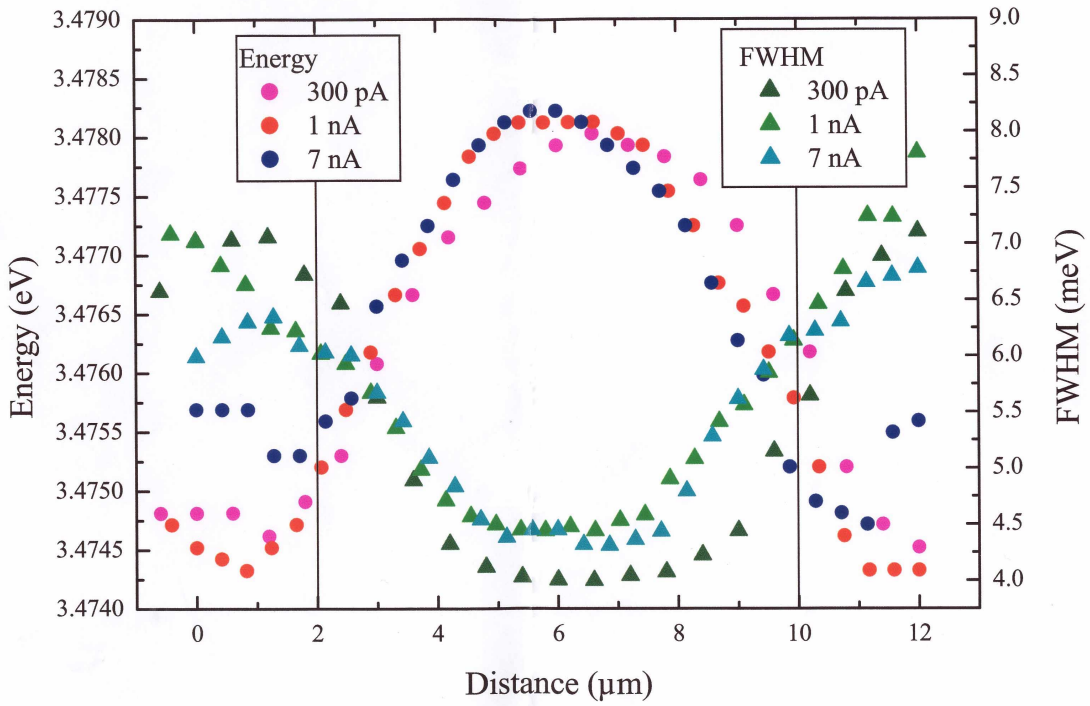

Figure 3. A scan of PL intensity across the 10 µm- bridge for the structures produces with the different beam currents for the capped sample.

PXM provides information about the local shear strain since a lattice distortion causes streaking of the Laue reflections. Lattice curvature can result from both FIB induced elastic as well as plastic distortion of the lattice. In this study, PXM images were obtained in geometry with the trench direction perpendicular to the beam. The total Laue pattern contains zone lines formed by InGaN and sapphire Laue reflections (Fig. 4a). The small dashed rectangle marks the area of interest containing the InGaN and the sapphire reflections.

Fluorescent analysis (Fig. 4b) was performed prior to the PXM analysis to find the exact location of the trenches which are shown as purple horizontal rectangles on the image. This allowed us to choose the correct locations for microbeam probing of the sample along vertical lines passing through the centers of the trenches (dashed line A1-A2) and through the edges of the trenches (dashed lines B1-B2 and C1-C2). The step size between neighboring probe locations along each line was 2µm. The starting probing location is indicated by an "x" on each line.

For PXM measurements the microbeam was scanned both parallel and perpendicular to the trenches to get a 2D map of the structural changes in the trenched region. A portion of the 2D map shown in Fig.5 corresponds to the region containing one upper trench marked by the dashed rectangle in Fig. 4b. Laue patterns were recorded at 50 different sample positions. Due to the small size of the microbeam (0.5 µm) it was possible to obtain spatially resolved data from different locations around the trenches. Because the high-energy (8-25 keV) X-ray beam penetrates through the InGaN/GaN film and probes the sapphire substrate as well, both the substrate and overlayer contribute to the observed Laue patterns. The sapphire reflections do not change position with sample translation and were used as a reference to determine changes in the local orientation of the

InGaN/GaN layer relative to the substrate (Fig. 5). The PXM results show that FIB etching distorts the lattice in the InGaN/GaN layer not only in the immediate trench region but also in the surrounding area. Lattice planes were curved with the curvature radius dependent on the distance from the trench, FIB current and the capping layer.

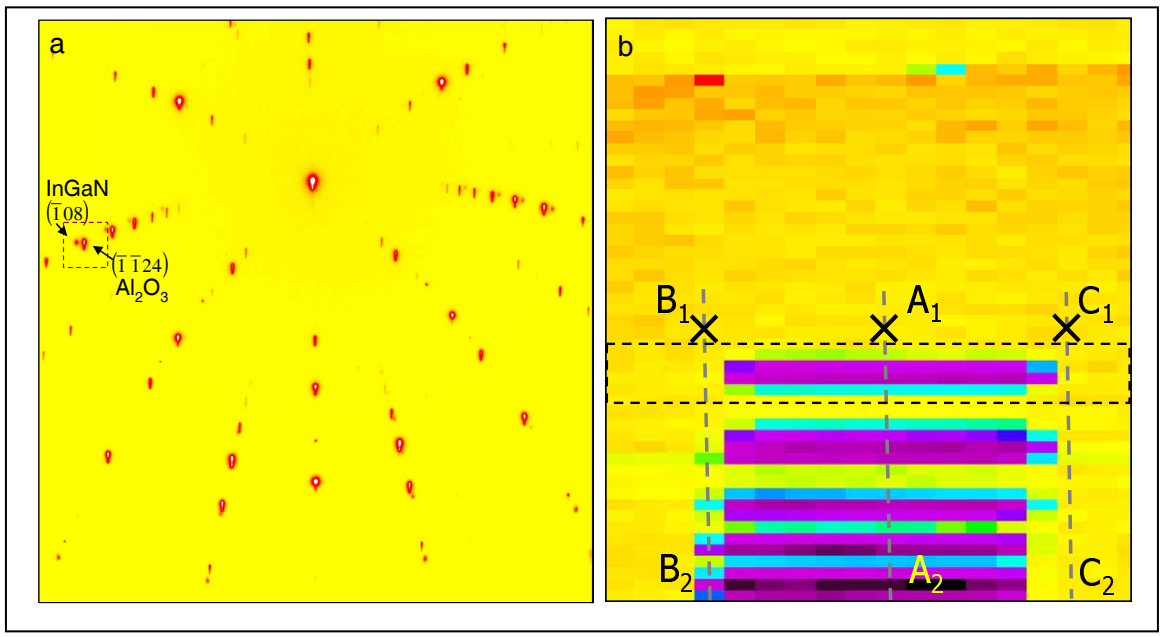

Figure 4. (a) Total Laue pattern with overlapped InGaN (round spots) and sapphire (elongated spots) reflections. The dashed rectangle marks the area of interest containing the InGaN and the sapphire reflections. (b) Fluorescent 2D map showing relative positions of trenches (purple rectangles) and X-ray microdiffraction probing locations (along the lines A1-A2, B1-B2, C1-C2).

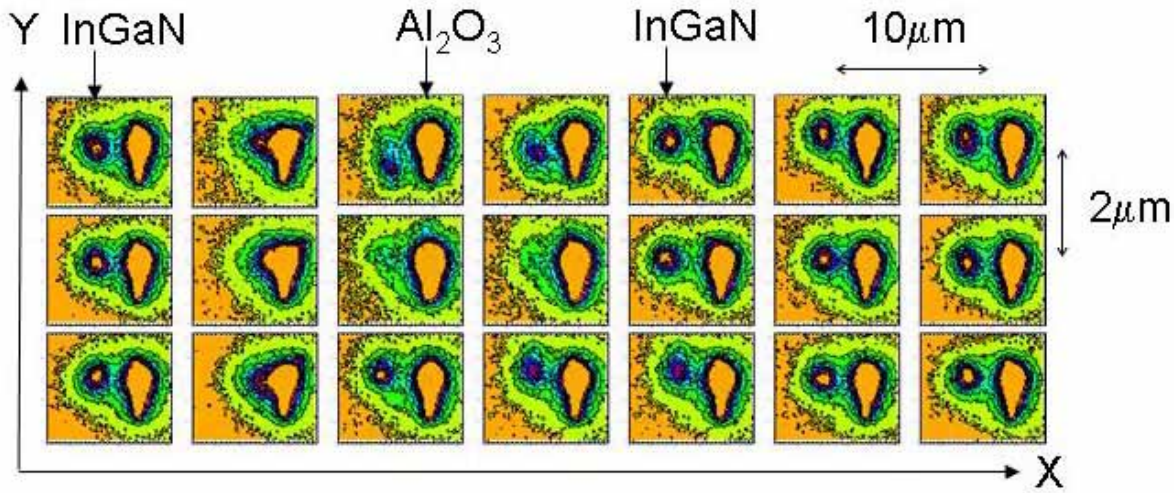

Figure 5. 2D map demonstrates lattice rotations in the InGaN layer relative to substrate.

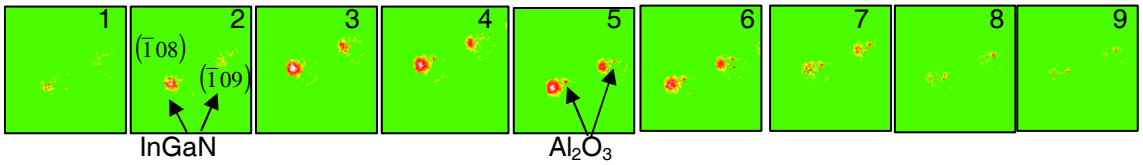

Figure 6. Several 3D depth-resolved (reconstructed) images corresponding to the region with $(\bar{1}08)$ and $(\bar{1}09)$ InGaN Laue spots and two week sapphire Laue spots demonstrate that there is a gradient of the orientation with depth.

With the DAXM technique, the wire shadows part of the diffracted intensity. Images collected with different wire positions were used to reconstruct the Laue images corresponding to different depth inside the sample (Fig. 6). Here each depth increment is 1μm. Depth is shown at each frame in μm. The first frame is closest to sample surface. The series of the reconstructed images in Fig.6 were taken at the first location shown by an "x" on the line A1-A2 in the Fig. 4b within the region of continuous film. Only the $(\bar{1}08)$ and $(\bar{1}09)$ InGaN Laue spots are observed close to the surface (1st and 2nd frames). With increasing depth the $(\bar{1}08)$ and $(\bar{1}09)$ InGaN Laue spots move indicating a change of orientation. At a depth of four - five microns the sapphire Laue spots appear (weak spots to the right of the InGaN spots at the 5th frame). Positions displacement from frame to frame indicates orientation change close to the interface. Finally the InGaN Laue spots disappear. A quantitative analysis of the orientation change with depth and with the distance from the trench is underway.
PXM data suggest the formation of dislocation walls. Although the defect structures clearly self-organize during FIB machining, further measurements will be required to determine whether the defects arise primarily from the inherent misfit dislocations of the original structure or from defects introduced during FIB machining.

CONCLUSIONS

- PXM and DAXM analysis have been used in a 3D spatially-resolved investigation of the local lattice orientation and defect microstructure in FIB structured InGaN layers grown on sapphire substrate.
- The PXM results show that FIB etching distorts the lattice in the InGaN/GaN layer not only in the immediate trench region but also in the surrounding area. Lattice planes become curved with curvature radii dependent on the distance from the trench, FIB current and the capping layer.
- The TEM analysis in the vicinity of the trenches shows a high density of dislocations and a narrow (~2 nm) amophized layer on top, which could be due to direct surface damage by the FIB beam or due to re-deposition.
- FIB induces structural damage of the GaN lattice is discussed in terms of the formation of disconnections causing long-range rotations in the layer.

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REFERENCES

1. H. Lohmeyer, K. Sebald, J. Gutowski, R. Kröger, C. Kruse, D. Hommel, J. Wiersig, F. Jahnke, *Eur. Phys. J.* B **48**, 291-294 (2005).
2. Maaß, R., Grolimund, D. , Van Petegem, S., Willimann, M. Jensen, M., Van Swygenhoven, H., Lehnert, T. Gijs, M.A.M., Volkert, C.A., Lilleodden, E.T., & Schwaiger, R. *Appl. Phys. Lett*. **89**, 151905 (2006).
3. J. R. Greer, W.C. Oliver, W.D. Nix, *Acta Mater.*,**53**,1821-1830 (2005)
4. S.X. Jin, J.Z. Li, J.Y. Lin, H.X. Jiang, *Appl.Phys.Lett.* **76**, 631 (2000)
5. R.I. Barabash, G.E. Ice, (2005). "*Microdiffraction Analysis of Hierarchical Dislocation Organization"* In: Encyclopedia of Materials: Science and Technology Updates, Elsevier, Oxford. 1-18
6. R.I. Barabash, C. Roder, G.E. Ice, S. Einfeldt, J.D. Budai, O.M. Barabash, S. Figge, D. Hommel, *J Appl. Physics*, **100**, 053103-1 -053103-11 (2006)
7. R.I. Barabash, G.E. Ice, W. Liu, S. Einfeldt, D. Hommel, A.M. Roskovski, R.F. Davis, *Physica Status Solidi* (a)*,* **202**, 5, 732-738, (2005)