

Electron Beam Induced Damage in Wurtzite InN

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Interest in InN as a promising candidate for fabrication of high performance high electron mobility transistors (HEMTs) or light-emitting diodes¹ arose with possibilities of the growth of high quality wurtzite InN.² This naturally increases the need for reliable information on the basic structural, electronic and optical parameters and properties of InN. Electron energy loss spectroscopy (EELS) is proven to be a reliable method to measure some of these properties: in the high energy-loss region of EELS the core-level excitation spectra can be used to obtain information on the densities of the unoccupied electronic states,³ while the EELS low-loss region gives information on interband transitions, plasmon excitations and, with care, can provide complex dielectric functions.⁴ However, before conducting any quantitative EELS measurements, we need to know the nature and rate of the electron beam induced damage in InN accruing during observation in the scanning transmission electron microscope (STEM). Here we report the results of the EELS measurements on wurtzite InN using the Cornell UHV VG HB501 STEM with 100 keV electron beam and a focused probe of about 2 Å.

To quantify the effects of the electron-beam induced damage in InN, measurements of the damage in the sample were carried out. Several experiments in spot modes, when the e-beam illuminates single spots of about 2 Å size, were performed. Fig. 1(a,b) shows typical marks (bright spots) of the damage appearing in spot mode. These images were recorded using the Annular Dark Field (ADF) detector of the STEM, which is known to be sensitive to the atomic number, Z , of the material (in the compounds it is average Z). These bright spots indicate that the damaged areas have a higher Z . To understand this we repeat experiments by simultaneously recording electron energy loss spectra of the Nitrogen K -edge and Indium M -edge. Figs. 2(a) and (b) show the time evolution of the damage in the single spot. As is seen the intensity of the N K -edge drops dramatically and the features of fine structure disappear, while spectrum of the In $M_{4,5}$ -edge loses fine structure but not intensity. To be quantitative, we measured the changes of the intensities for both the N K -edge and the In $M_{4,5}$ -edge during the first 70 sec of exposure. The results are presented in Fig. 3, where the slope of the intensity drop for the N K -edge is $7.8 \times 10^{-3} \text{ sec}^{-1}$. It is known that the intensity of the core-level EELS is proportional to the number of the corresponding atoms in the exposed area and, therefore, what is observed here is the ejection of the Nitrogen atoms out of the sample leaving behind metallic Indium. This is also consistent with appearance of the bright spots seen in Fig. 1(a,b), since $Z^{\text{In}} > Z^{\text{InN}}$. A similar mechanism for beam damage was also observed for wurtzite GaN.⁵ For these samples we found that spectra do not show any detectable damage until an exposure of at least 10 sec. A long acquisition time is needed for low noise EELS data and, therefore, in all measurements of N K -edge and In $M_{4,5}$ -edge spectra, 10 sec as the upper limit for acquisition time was used.⁶

References

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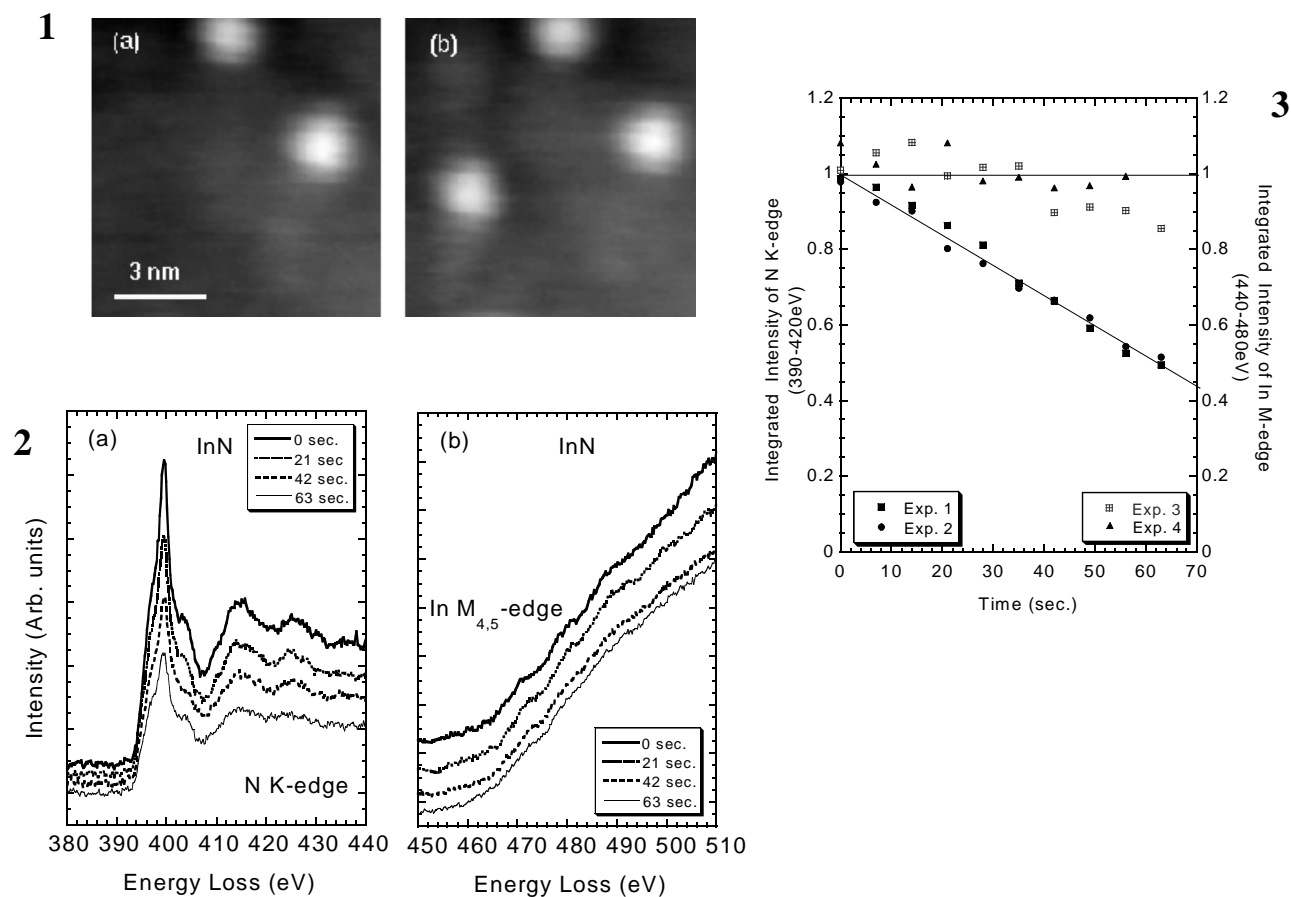


Fig. 1. ADF images of the wurtzite InN with characteristic damaged areas (bright spots) appearing in spot mode after 63 sec exposure.

Fig. 2. The evolution of the damage after the first 70 sec of the exposure: (a) effect on the Nitrogen K-edge and (b) on the Indium $M_{4,5}$ -edge.

Fig. 3. Changes in the integrated intensities of the Nitrogen K-edge (390-420 eV energy range) and Indium $M_{4,5}$ -edge (440-480 eV energy range) with exposure time. Experiments 1 and 2 for NK-edge and 3 and 4 for In M -edge were performed in spot mode. The intensities are normalized to the first spectra.