Development and Application of the Electron Beam Induced Current Technique in a Scanning Transmission Electron Microscope for Electrical Characterization of Nanometer Scale Semiconductor Devices

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Keywords: EBIC, STEM, SEM, GaN, LED, InGaN, quantum well

Abstract

Electron Beam Induced Current (EBIC) is a Scanning Electron Microscope (SEM)-based technique that can provide information on the electrical properties of semiconductor materials and devices. Since the size of electronic devices has reached the nanometer scale, the SEM-based EBIC technique has reached its spatial resolution limits. Therefore, there is a need for a high-resolution EBIC technique. This chapter focuses on the design and implemenation of an EBIC system in a dedicated Scanning Transmission Electron Microscope (STEM). The combination of a high energy electron beam and a thin sample in a STEM improves the spatial resolution to the nanometer scale. The STEM-EBIC technique was used in the characterization of an Indium Gallium Nitride (InGaN) quantum well Light Emitting Diode (LED). A novel sample preparation method using a Focused Ion Beam (FIB) technique and a custom STEM-EBIC sample holder were designed for these experiments. The simultaneous collection of Z-contrast and EBIC images was also demonstrated. These two signals allowed for the nanometer scale electrical and compositional properties of the semiconductor device to be studied concurrently. The relative position of the p-n junction with respect to a thin InGaN quantum well was resolved with the STEM-EIBC technique with nanometer precision. The STEM-EBIC technique is shown to be a powerful tool for the characterization of electrical transport properties of semiconductor materials and devices on the nanometer scale.

Introduction

As the solid-state electronics industry continues to shrink the dimensions of electronic devices and enter into the nanotechnology era, the field of analytical techniques for materials and device characterization is presented with new and additional challenges: structural, chemical, electrical, and optical properties have to be studied with nanometer resolution and higher sensitivity. Adding the vast area of new materials with unknown properties and performances to the increasing complexity of devices creates a need for improved analytical capabilities. Therefore, nano-characterization techniques are needed to investigate the properties of new materials and the performance and failure of devices formed from these new materials. Scanning Electron Microscopy-based and Scanning Transmission Electron Microscopy-based Electron Beam Induced Current (EBIC) are two techniques that can be used for the electrical characterization of materials and devices at the micrometer and nanometer scale. EBIC can provide information on electrically active defects, diffusion of carriers, surface recombination mechanisms, bulk recombination mechanisms, trapping centers, and especially p-n junction width, position, and homogeneity [1,2,3]. This chapter looks at describing how the EBIC technique can be implemented in a commercial dedicated STEM and how to exploit the high spatial resolution obtainable in STEM-EBIC to characterize nanoscale properties of optoelectronic devices. In particular, STEM-EBIC was used to determine the p-n junction location in an Indium Gallium Nitride (InGaN) quantum well Light Emitting Diode (LED).

The STEM-EBIC technique was first demonstrated in the late 1970's by Sparrow and Valde in the correlation of crystal defects and the electrical properties of Si transistors [4]. A few years later, Petroff et al. used the STEM-EBIC method to obtain information on the relationship between dislocation cores and nonradiative recombination properties in GaAlAsP [5]. In the early 1990's, Cabanel and Laval implemented STEM-EBIC to relate the electrical activity of microstructural defects in polycrystalline Si to the presence of impurities [6]. More recently, Cabanel et al. used cross-sectional STEM-EBIC to relate the inhomogeneities in Si p-n junctions to variations in doping concentrations [7]. In this work, the STEM-EBIC method is used to study the cross-section of an InGaN quantum well LED in order to determine the p-n junction location with respect to the quantum well of the device.

In a semiconductor material under observation in a SEM, the incident electron beam generates Electron Hole Pairs (EHPs) or mobile charge carriers within a small volume. These charge carriers will diffuse through the lattice until a recombination or trapping event occurs. However, if an electric field is applied to the sample, either externally or internally, the EHPs generated by the beam will move in response to the field. The electrons and holes will move in opposite directions creating a current that can be detected in an external circuit. In this respect, the specimen itself is used as a charge collector. An internal electric field is most commonly supplied by a p-n junction or a Schottky barrier, while an external electric field is provided by a bias applied to electrical contacts on the device. The microscopy techniques based on the collection of the charge carriers generated in the sample by an incident beam of charged particles are known as "charge collection microscopy" [1,2,3,9].

The first observation of charge collection phenomena in a SEM was reported by Everhart in 1958 when he documented that a "bombardment conductivity" takes place when an ionizing beam "of any sort, energetic particles or photons, serves to create mobile charge carriers" [2]. However, a distinction must be made between charge collection processes due to external versus internal electric fields. If an external biasing source is applied to a specimen via two ohmic contacts, the increase of the conductance of the specimen in the region that is being bombarded can be measured as variations in the current between two ohmic contacts. In a similar way, the variations in the voltage can be measured when a constant current is supplied to the sample. Since these methods rely on measuring the change in conductivity of the sample that is induced by the excess carriers injected by the electron or " beta ray" , they are referred to as β-conductivity techniques [1,2]. If there is no external biasing source, current will only flow if the specimen exhibits electron voltaic effects (i.e. internal potential differences). There are bulk and barrier electron voltaic effects. Bulk electron voltaic effects arise from built in electric fields due to non-uniform impurity distributions within the bulk of a semiconductor material. Barrier electron voltaic effects arise due to built in electric fields at electrical barriers such as p-n junctions and Schottky barrier contacts. The original EBIC acronym stood for Electron Beam Induced Conductivity and was indiscriminately applied to both barrier electron voltaic effect signals and β-conductivity [1]. The technique involving electron bombardment, in a SEM or a STEM, on a sample with an internal electric field due to barrier electron voltaic effects is commonly referred to as Electron Beam Induced Current (EBIC) in the literature and will be discussed in more detail here [1].

EBIC Theory Applied to LEDs

LEDs are semiconductor devices that under appropriate forward biased conditions can emit radiation in the ultraviolet, visible, and infrared regions of the electromagnetic spectrum. The active layer of these devices, or the layer that emits the light, is commonly formed by a p-n junction. A p-n junction is formed when p-type (i.e. semiconductor doped with acceptors) and n-type (i.e. semiconductor doped with donors) material are brought together. Due to the concentration gradient, the holes will diffuse towards the n-type region and recombine with the electrons or majority carriers. Similarly, the electrons will diffuse towards the p-type region and recombine with the holes or majority carriers. Therefore, a space charge region or depletion region is formed around the metallurgical junction. Although the depletion region is depleted of majority carriers, it does contain exposed immobile donor ions. The separation of charge created by the positive and negative immobile ions creates an internal electric field. This built in electric field drives the minority carriers in the opposite direction to the diffusion direction. Equilibrium is eventually reached when the diffusion and drift fluxes are balanced. The built in electric field across the p-n junction reaches a maximum value at the p-n junction, and decreases linearly to zero at the edges of the depletion region [10].

In the EBIC linescan configuration, the electron beam of a SEM or STEM scans the cross-sectional surface of the sample perpendicular to the depletion layer of the device. Figure 1 shows the general experimental setup and beam interaction in the STEM-EBIC experiments. The electron beam is scanned or stepped across the p-n junction of the sample, creating EHPs at each point. These excess charge carriers will diffuse down the carrier concentration gradient of the device. Some of these carriers will recombine or become trapped in defects, and some will reach the depletion region. The charge carriers that reach the depletion region will be driven by the built in electric field and create a short circuit that can be read using a current amplifier. The EBIC signal is formed by scanning the electron beam across the p-n junction of the device and plotting the short circuit current at each point. The electrical transport mechanism of the carriers towards the p-n junction produces an EBIC curve with a maximum at the p-n junction position and exponentially decaying tails on both sides of the depletion layer due to the diffusive nature of the electrical transport in those regions. The carriers created within the depletion region are less affected by the recombination process due to the carrier-depleted nature of this layer. If an EBIC linescan is obtained in low injection conditions (i.e. the generated carrier concentration is less than the equilibrium concentration created by the dopants), the exponentially decaying tails of the EBIC profile can be used to extract the diffusion length and surface recombination velocity of the minority carriers [9,11]. EBIC experiments can also be carried out in high injection, although under these conditions the information about the transport mechanism of minority carriers is lost. Calculations confirmed that high injection conditions were used in the STEM-EBIC experiments. Therefore, the electrical transport theory used to determine the minority carrier diffusion length and surface recombination velocity is no longer valid. However, the p-n junction location, as determined by the maximum in the EBIC signal, can be resolved with nanometer precision with the STEM-EBIC technique since inside the depletion layer the drift component will dominate the transport of the charge carriers.

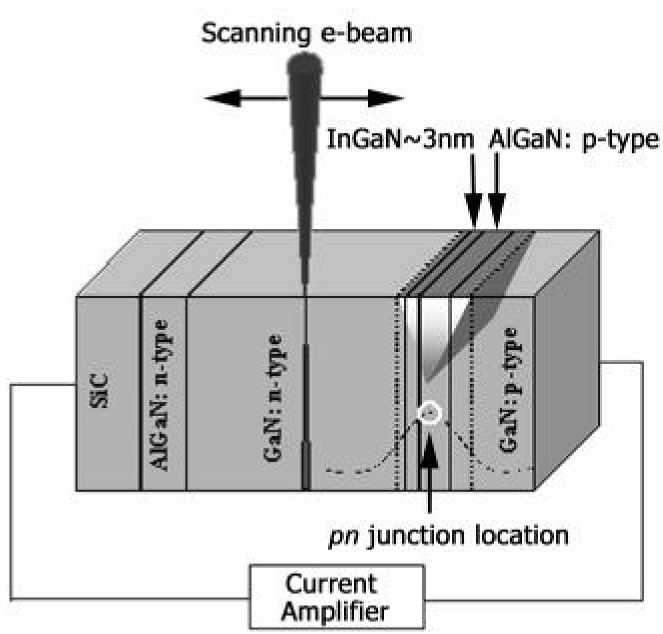

Fig. 1 Schematic of the cross-section of the InGaN-quantum well LED structure and experimental linescan setup used in the STEM-EBIC measurements (Not drawn to scale).

SEM and STEM

In a SEM, a source of electrons is produced by thermionic emission of a tungsten filament in the electron gun. The electrons are accelerated to an energy of approximately 1-30keV. A simple SEM column consists of a condenser lens, an objective aperture, and an objective lens. The electron-optical column provides control of the beam through manipulation of the spot size, beam energy, beam current, and convergence angle, all of which contribute to the resolution and depth of focus in a SEM. The goal of the lens system is to focus the electron beam to a final spot size on the order of 5.0 nm and a beam current in the range of 1pA-1μA on the specimen surface [8].

The electron beam-specimen interactions can be divided into two categories including elastic and inelastic scattering. Elastic scattering primarily gives rise to backscattered electrons (BSE), while inelastic scattering primarily gives rise to secondary electrons (SE), x-rays, and EHP generation. The combined effect of these two scattering processes is to limit the electron beam penetration within the sample. The region in which the electrons interact with the specimen is called the interaction volume. The lateral size of the interaction volume is a function of the mean atomic number, mean atomic weight, and density of the specimen material as well as the beam energy, spot size, and incident angle [8]. For example, a gallium nitride (GaN) sample bombarded with a 1keV electron beam perpendicular to the surface of the sample will have an interaction volume with a diameter of approximately 14nm, while the diameter of the interaction volume created with a 30keV beam will be approximately 4.2μm (Figure 2). The lateral resolution of SEM images is limited by the spot size of the electron beam and the lateral size of the interaction volume created within the specimen. Therefore, the SEM-EBIC technique is limited by the beam excited volume of generated EHPs.

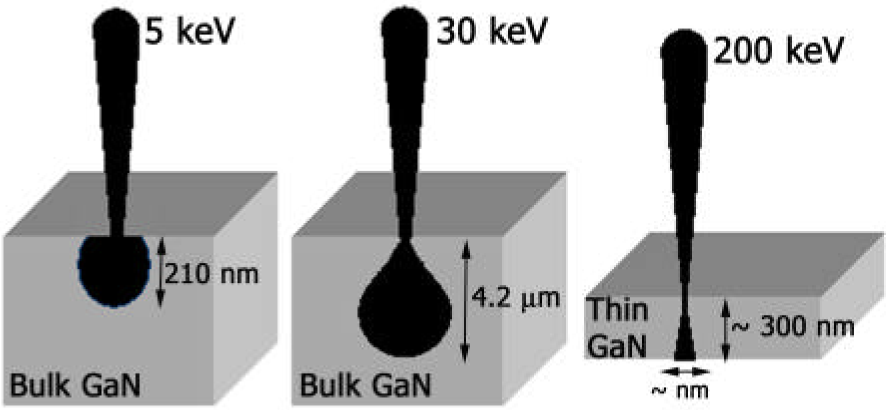

Fig. 2 Schematic illustrating the size of the interaction volume in bulk and thin samples with different accelerating voltages (Not drawn to scale).

In order to overcome the resolution limitations found in the SEM, a high-resolution STEM design must be implemented. Originally, STEM capabilities were achieved by using the convergent-beam mode of a Transmission Electron Microscope (TEM). In the traditional TEM mode, two condenser lenses are adjusted to illuminate the specimen with a parallel beam of electrons. The transmitted electrons are then focused by the objective lens to form an image. In the convergent-beam mode, a series of condenser lenses are used to demagnify the original gun crossover to a spot on the specimen [12,13]. Scanning electronics are then used to move the convergent-beam across the specimen. All of the STEM-EBIC work mentioned previously was achieved using a TEM with STEM capabilities.

In 1963, Crewe published the first ideas related to the development of a dedicated STEM instrument [14]. VG Microscopes, Ltd. was the first company to manufacture and sell dedicated STEM systems. They continued to produce STEMs with the electron gun at the bottom of the system until they closed in the mid 1990’s. Currently, several manufacturers including Hitachi, JEOL, and Phillips sell dedicated STEM systems. In a dedicated STEM, the optical design is more closely related to a SEM than a TEM. A source of electrons is produced by a thermionic or field emission source and accelerated to an energy of approximately 200-400keV. In a Field Emission STEM (FESTEM), such as the Hitachi HD-2000 dedicated STEM used in these experiments, the initial crossover is on the order of 5.0 nm. Therefore, the initial crossover produced in a FESTEM is on the same order as the final spot size in a SEM. The condenser lenses, apertures and objective lens focus the beam to a spot on the specimen and provide control over the spot size, beam current and convergence angle. The optics system focuses the electron beam to a final spot size on the order of 0.5 nm and a beam current of approximately 1 nA [12].

While bulk specimens can be analyzed in a SEM, a thin sample on the order of hundreds of nanometers is used for STEM analysis. Several signals, including SE, BSE, x-rays, and EHPs, are produced from the interaction volume that is created within the thin specimen and can be detected and used for imaging. In addition, the transmitted electrons can be detected. These include electrons scattered through large angles and electrons scattered through small angles. The large angle scattered electrons can be detected using a large angle dark field annular detector and used to form a 'Z-contrast' image containing compositional information [12].

The lateral resolution of STEM images is also limited by the spot size of the electron beam and the lateral size of the interaction volume created within the specimen. However, the combination of a thin sample and high energy electron beam improves the spatial resolution of STEM analysis to the nanometer level by decreasing the amount of beam spreading to the order of a few nanometers (Figure 2).

Sample Preparation

The sample used in the STEM-EBIC experiments was an InGaN quantum well LED. The general structure of this device is shown in Figure 1. The device was grown by metal-organic chemical-vapor deposition on a (0001) Silicon Carbide (SiC) substrate. A n-type AlGaN buffer layer was grown on the SiC substrate followed by n-type GaN. A 3 nm InGaN quantum well was grown on top of the n-type GaN material. This layer was followed by p-type AlGaN and finally a p-type GaN contact layer. The as-received sample was a 1 cm^2 section cleaved from the wafer and mounted unpackaged on the edge of a TO header. The can of the TO header made contact to the backside of the device (i.e. n-type material) and a gold bonded wire made contact to the topside of the device (i.e. p-type material).

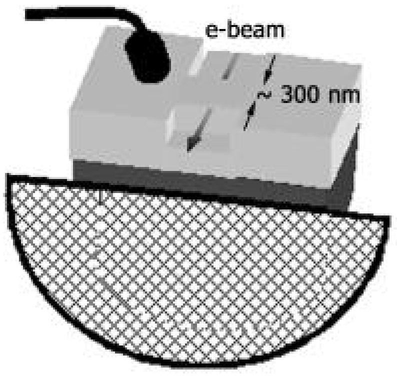

Fig. 3 Schematic of LED after being mechanically polished, placed on a copper mesh grid, and ion milled to create an electron transparent membrane (Not drawn to scale).

The sample preparation was a variation of the traditional 'H-bar' Transmission Electron Microscopy (TEM) method using Focused Ion Beam Micromachining (FIBM). The TO header was embedded in wax and mechanically polished down using diamond lapping films on an Allied Multiprep™ System. The sample was polished to a final wedge of less than 50 µm, while ensuring that the gold bonded wire was still attached to the p-type material of the device. The backside of the thinned device was attached to a half copper mesh TEM grid with silver paint, while the gold bond wire remained free (Figure 3). The silver paint provided electrical contact between the backside of the device and the TEM grid. In the final step, a FEI 200 TEM with a magnum column and a 30keV Ga$^+$ beam was used to create a 300 nm thick electron transparent membrane. Figure 4(a) shows a SE image taken on the HD-2000 of the region of the sample thinned by FIBM with respect to the gold wire. Figure 4(b) shows the FIBM area and reveals the 'H-bar' shape of the region.

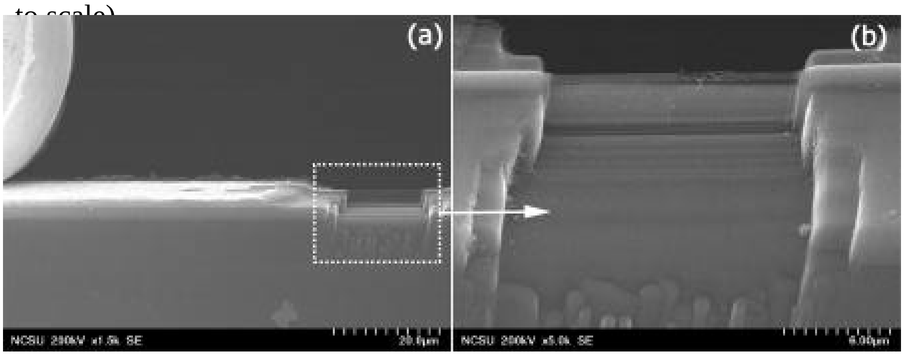

Fig. 4 (a) and (b) SE images of the FIBM region of the LED taken on the HD-2000 at 200keV.

Custom Specimen Holder

The STEM-EBIC specimen holder was designed after a standard single-tilt side-entry HD-2000 FESTEM specimen holder. A ceramic feedthrough was placed in the barrel of a specimen holder and sealed with Torr Seal, a solvent free epoxy resin, to ensure no vacuum leaks through the barrel. Two shielded coaxial single-stranded and silver-plated copper wires insulated with Kapton® were fed through the ceramic feedthrough and sealed with Torr Seal. On the air-side of the barrel, the two wires were soldered to a subminiature A (SMA) connector that was mounted in a plastic handle. The shielding and the Kapton® insulation were removed from the ends of the two wires on the vacuum-side of the barrel in order to allow electrical connections to be made to the sample.

The tip of the specimen holder is a separate component and attaches to the barrel with setscrews. The tip has a 3mm cup, which holds the specimen, and an isolated jewel bearing that makes mechanical contact to the column of the STEM. A piece of mica was glued down on the tip and a hole was cut in the mica around the 3mm cup. The mica was used to create electrical isolation between the specimen and the specimen rod holder and the 3mm hole in the mica allowed for the transmission of the electron beam through the sample.

The fully prepared LED sample was glued onto the mica surrounding the 3 mm cup, while the gold bonded wire was still allowed to move freely. One of the feedthrough wires was glued to the top of the copper grid and silver paint was used to make the electrical connection between the feedthrough wire and the backside of the sample (Figure 5(a)). The end of the second wire was placed in a strip of Indium that was positioned near the 3 mm diameter cup on the tip. The free gold bonded wire was also placed in the Indium strip, thus making contact between the frontside of the sample and the feedthrough wire (Figure 5(a)). Figure 5(b) shows the fully prepared LED sample, including the FIBM area, mounted in the specimen holder.

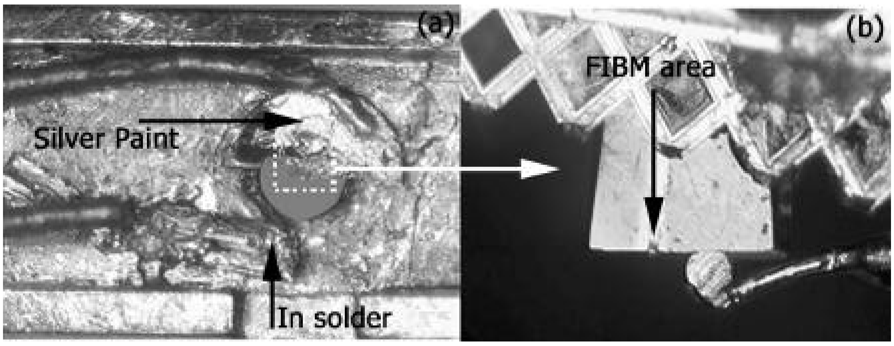

Fig. 5 (a) Customized STEM-EBIC holder showing the device connected to the two electrical feedthroughs with silver paint and In solder. (b) Fully prepared LED mounted in STEM-EBIC.

Analysis and Results

The goal of the STEM-EBIC experiments was to determine the p-n junction location, defined as the maximum of the built in electric field, with respect to the position of the InGaN quantum well. All EBIC experiments were performed at room temperature in a HD-2000 FESTEM with a 200keV electron beam, a 0.5 nm spot size, and a beam current of 350 pA. An EDS acquisition system was used to obtain Z-contrast and EBIC images simultaneously. In the external acquisition system, the output from a Keithley 614 electrometer was fed directly into the auxiliary port of the imaging system. The EBIC and Z-contrast images were collected with a 12.8 ms dwell time and 128 x 100 resolution. The EBIC signal varied between 30 and 55 pA during the EBIC image acquisitions. The highest signal intensity in the Z-contrast image corresponds to the InGaN quantum well because the InGaN layer has the highest mean atomic number in the sample structure (Figure 6). The AlGaN layer corresponds to the lowest signal intensity in the Z-contrast image because it has the lowest mean atomic number in the sample structure (Figure 6). Therefore, the Z-contrast images provided chemical confirmation on the location of the GaN and AlGaN barrier layers with respect to the InGaN quantum well.

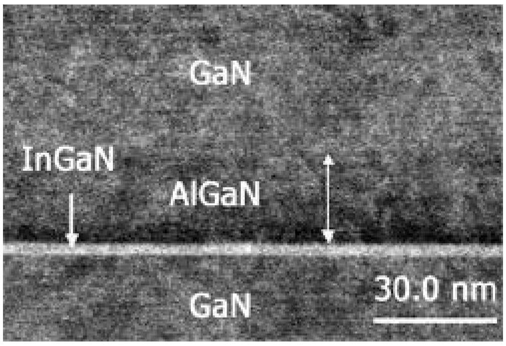

Fig. 6 Z-contrast image of the LED structure, showing the InGaN QW and the GaN and AlGaN barrier layers.

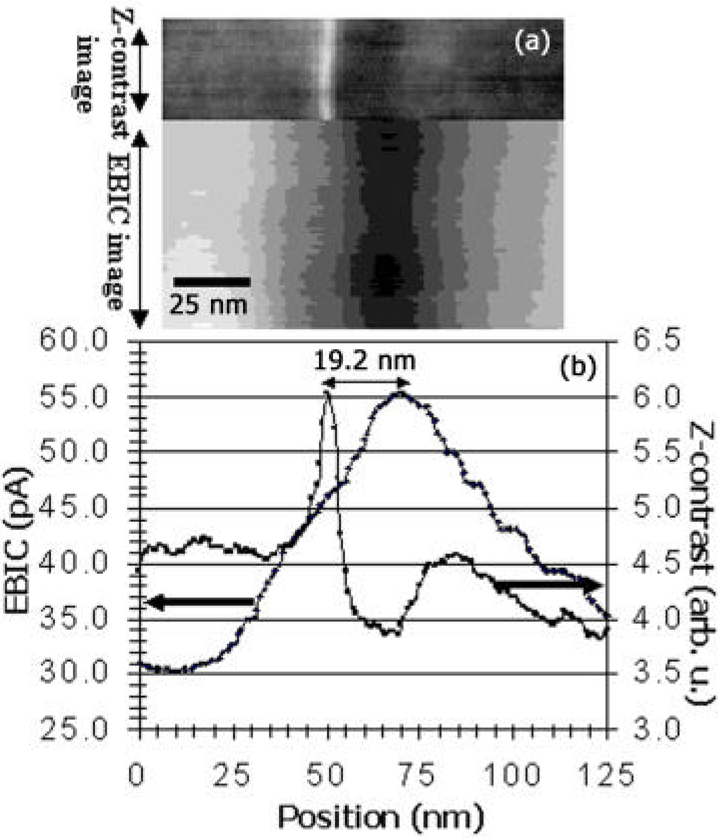

Fig. 7 (a) EBIC and Z-contrast images acquired with the Oxford ISIS EDS system. (b) Mean Z-contrast and EBIC linescans showing p-n junction is located (19± 3) nm from center of InGaN QW.

In order to determine the p-n junction location, a mean Z-contrast linescan was obtained by averaging 28 lines in the Z-contrast image and a mean EBIC linescan was obtained by averaging 70 lines in the EBIC image (Fig. 7(a)). A comparison of the maximum in the mean Z-contrast linescan to the maximum in the mean EBIC linescan revealed the position of the p-n junction with respect to the center of the InGaN quantum well. The relative position of the p-n junction with respect to the InGaN quantum well was found to be (19 ± 3) nm from the center of the InGaN quantum well, as shown in Figure 7(b).

A Noran Vantage EDS system was used to simultaneously obtain Z-Contrast and EDS linescans (Fig. 8). The elemental In EDS linescan confirmed the position of the InGaN quantum well that was previously determined with the Z-contrast image.

Conclusions

The EBIC technique can be implemented in a SEM or a STEM in order to obtain electrical information about electrical transport properties of semiconductor materials and devices. Due to the high energy electron beam (200keV) and the thin specimen (300nm), the STEM-EBIC technique has been shown to be superior in terms of spatial resolution when compared to the SEM-EBIC technique. The increased resolution is an important feature due to the decreasing size of electronic and optoelectronic devices and the overwhelming need for micro- and nano-characterization techniques in the upcoming nanotechnology industry. The sample preparation and electrical connections for STEM-EBIC are challenging. However, the introduction of a novel sample preparation method as well as the design and construction of a custom specimen holder was described here and proved to be a viable solution. The capability of the Hitachi HD-2000 dedicated FESTEM in conjunction with a customized STEM-EBIC holder to simultaneously collect chemical (i.e. Z-contrast) and electrical (i.e. EBIC) images of semiconductor samples was also demonstrated. Finally, the STEM-EBIC technique resolved the position of the p-n junction with respect to the InGaN quantum well with nanometer precision. The STEM system is a useful and powerful tool for the nano-characterization of structural, compositional, and electrical properties of semiconductor materials and devices.

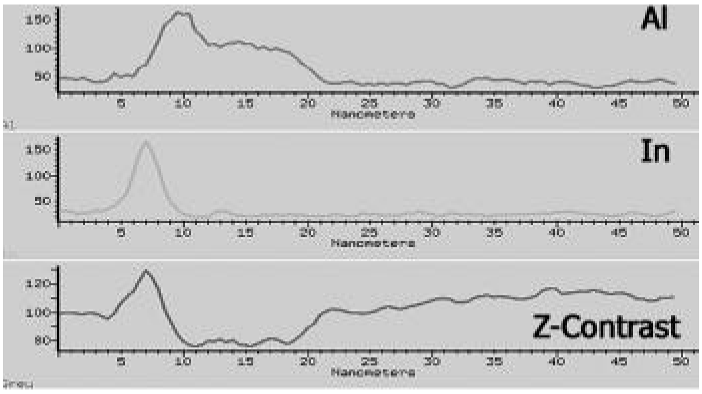

Fig. 8. EDS linescans of Al, In, and EBIC and Z-Contrast linescans acquired with the Thermo Noran Vantage EDS system.

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