Ion damage effects from negative deflector plate voltages during the plasma-assisted molecular-beam epitaxy growth of dilute nitrides

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We studied the effects of ion damage on the optical properties of dilute nitrides grown by plasma-assisted molecular-beam epitaxy. A dual-grid retarding field ion energy analyzer was used to measure the ion flux and ion energy distribution at the substrate position from an Applied-EPI UniBuilbTM rf plasma cell. These changes were measured as the negative deflector plate voltage varied from 0 to -800 V. The largest ion flux resulted with a -100 V setting, while the greatest ion energies occurred with -200 V. Deflector plate voltages more negative than -300 V resulted in a significant reduction in both the ion flux and ion energy. The damage caused by these ions was determined by measuring the pre- and postanneal photoluminescence properties of Ga_{0.8}In_{0.2}N_{0.01}As_{0.99} quantum wells. Comparable optical properties were possible with various combinations of ion fluxes and ion energies, which demonstrate how the ion flux and ion energy each impart an individual effect on the sample's optical properties. An awareness of these effects is crucial because the optical properties of dilute nitrides grown with an improper deflector plate voltage setting can lead to a greater degree of ion damage to the sample. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1940126]

Dilute nitride, narrow-band gap semiconductors have gained a great deal of interest for applications involving the important 1.3 and 1.55 μ m telecom wavelengths.¹⁻⁴ Unfortunately, ion damage incurred during the plasma-assisted molecular beam epitaxy (MBE) growth has led to poor optical properties.⁵ As a result, a number of groups have been involved with research that seeks to remove these damaging ions through the use of magnetic ion traps^{6,7} and dc-biased deflector plates⁸⁻¹⁰ situated at the outlet of the plasma cell. However, none of these earlier attempts ever considered that the removal of ions could deleteriously affect the optical properties of the dilute nitrides. In fact, it was recently shown by Wistey⁹ that the application of either -40 or +18 V still resulted in less ion damage to the sample than when no deflector plates were used-although the samples grown with the negative bias led to slightly poorer optical properties than did its positive counterpart. Work to elucidate the effects of ion damage from a series of negative deflector plate voltages has yet to be completed. Therefore, in this letter, we correlate the effects of ion damage to the optical properties of dilute nitrides by a measurement of the ion flux and its corresponding ion energy, which the sample experiences during MBE growth.

The experimental apparatuses involving our MBE growth chamber, rapid thermal annealing, and photoluminescence (PL) spectroscopy can be found elsewhere.^{10–14} For the purposes of this letter, we briefly discuss the relevant details. Our samples were grown at The University of Texas at Austin in a modified Varian Gen II MBE system equipped with the commercially available Applied-EPI UniBulbTM rf plasma cell. The cell was operated at 425 W with 0.4 sccm of a dilute 1% N_2 in Ar gas mix.¹² The open end of the cell, where the gas exits, directly faced the substrate and resembled a showerhead that comprised 253 holes within a 2-cm-diam circular region. Each hole measured approximately 0.2 mm in diameter.¹⁵ Two tantalum deflector plates, each one square inch, were located at the opening of the plasma cell, and the plates were spaced at 3.8 cm apart. They were isolation mounted with one of the two plates being connected through an electrical feedthrough to a highpotential power supply to provide a negative bias with respect to ground, and the second plate was connected to ground. The power supply had an integrated current meter,

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FIG. 1. Pre- and postanneal photoluminescence peak emission intensities for identical structures grown at various negative deflector plate voltages.

which can measure dc current on the order of μ A's.

The measurements of ion flux and ion energy from the plasma cell were conducted in a separate chamber. It was equipped with a similar pumping configuration as the MBE growth chamber involving cryo- and turbo-molecular pumps. During said measurements, the chamber pressure was 2 $\times 10^{-5}$ Torr. This pressure is identical to that which was measured with the beam equivalent pressure gauge located on the backside of the substrate manipulator during growth of the samples in the MBE chamber. Measurements were taken with a dual-grid retarding field ion energy analyzer, which was placed approximately 12 cm (~ 5 in.) away from the opening of the plasma cell. This position is equivalent to the location of the substrate in the MBE growth chamber. These retarding grid analyzers are not new; they are frequently cited in the literature and regularly used to measure ion energy distributions from plasma sources.^{16,17} They have the added benefit of being simple to construct. Additional details relating to our use of the dual-grid retarding field ion energy analyzer can be found elsewhere.¹⁸ Briefly, it consists of two 15 cm² aluminum meshes and a molybdenum collector plate. The spacing in between the wires was approximately 0.1 mm and the two grids were placed approximately 3 mm apart from each other, as well as from the collector plate. Retarding positive biases were applied on the collector plate and the ion current was measured to ground. Any ions with a sufficient energy (eV) to overcome the retarding voltage will subsequently be measured as ion current. The grid closest to the collector plate was biased at -20 V to return any secondary electrons back to the collector plate,¹⁶ and the outer grid was grounded to decouple any electric fields from the plasma and/or deflector plates with that of the collector plate. 19-21

 $Ga_{0.8}In_{0.2}N_{0.01}As_{0.99}$ triple quantum well (QW) PL samples were grown at 450 °C in the MBE growth chamber. The remainder of the structure, which included the AlAs optical cladding layers, were grown at the GaAs deoxidation temperature of 580 °C. Additional growth-related details can be found elsewhere.²² For this work, the structures of all samples were kept identical, with the only difference between samples being the application of a negative deflector plate bias during the growth of the optically active GaInNAs QWs. After growth, the samples were rapid thermal annealed for 180 s at 850 °C.¹³ The pre- and postanneal PL peak intensities are shown in Fig. 1. Pan *et al.* have shown that a lower degree of ion damage is associated with a larger postanneal increase in the PL peak intensity.⁷ Therefore, the smallest postanneal peak intensity from the -100 Volt de-



FIG. 2. (Color online) Ion flux measured at the substrate position vs retarding positive biases on the molybdenum collector plate. The derivative of the ion flux vs retarding voltage gives the ion energy distribution, which is shown in the inset. High-energy tails of the ion energy distribution are shown for 0, -100, and -200 V deflector plate settings. The derivatives were smoothed using the "FFT Filter Smoothing" function in Microcal Origin.TM

flector plate setting indicates that this sample incurred the most ion damage, when compared to the other voltage settings in this study.

The cause of this ion-induced reduction of the optical quality becomes evident when the ion flux to the substrate position was measured, which is shown in Fig. 2. The application of a -100 V deflector plate voltage exhibits the largest ion flux to the substrate, when compared to the other bias settings. Differentiating the values of the collector current with respect to the retarding potential gives the ion energy distribution,²³ which is shown in the inset for the high-energy portion of the ion energy distribution. Note also that the population of ions in the high-energy tail becomes larger for increasing negative deflector plate biases.

The explanation for this lies in a consideration of the sheath voltage within the plasma cell. The polarity of our 13.56 MHz rf sheath is oriented such that its positive edge is adjacent to the bulk of the plasma, while the negative edge forms over the holes of the exit aperture.²⁴

Therefore, larger negative deflector plate voltages can further lower the sheath's potential at the walls of the exit aperture, which would thereby increase the plasma sheath voltage. An ion that traverses across this larger sheath voltage will acquire a greater energy as it leaves the plasma cell through the exit aperture. This would explain the larger number of ions present in the high-energy tail of the ion energy distribution for the more negatively biased deflector plate settings. Increases in the ion flux can also be expected due to the attractive force unto the positive ions from the nearby negatively biased deflector plate.

Although a reduced ion flux associated with the -200 V bias should lead to improved optical properties when compared to no applied voltage, it is shown in Fig. 1 that a comparable optical quality exists between these two samples. This quandary can, however, be addressed by focusing attention to the inset of Fig. 2, which shows the ion energy distribution. A negative deflector plate bias leads to a larger number of ions populating the high-energy tail, which thereby suggests that the deleterious effects resulting from the presence of these higher energy ions are counterbalanced

smallest postanneal peak intensity from the -100 Volt de- the presence of these higher energy ions are counterbalanced Downloaded 07 Dec 2010 to 129.116.230.64. Redistribution subject to AIP license or copyright; see http://apl.aip.org/about/rights_and_permissions



FIG. 3. Simulated ion trajectories for 20 and 200 eV singly ionized positive ions. The location of the deflector plates is represented by the solid black lines, and the potential field by the solid gray lines.

by the benefits gained with a lower overall ion flux. We are currently working to isolate the individual effects on the optical properties from ions with different energies.¹⁸

As larger negative voltages are applied on the deflector plate, a balance exists between two effects. The first effect is an increase in the ion flux and ion energy of the species emanating from the plasma cell. This first effect is, however, counterbalanced by the second effect, which is the ability of the increasing electric field to both: (a) deflect the ions away from the substrate, and (b) subsequently capture these positive ions at the negatively biased deflector plate.

To further illustrate this second effect, the simulated ion trajectories are shown in Fig. 3 for various ion energies and deflector plate settings.²⁵ The simulations show that a 200 eV ion, which resides in the high-energy tail, will be perturbed by a -100 V bias, but would still be expected to impact the sample located approximately 12 cm (~ 5 in.) away from the plasma cell. This would explain why a -100 V bias still results in the most ion flux measured at the substrate position. However, the simulations further show that the -200 V bias exhibits the onset of ion deflection away from the substrate, which is consistent with Fig. 2 where a decrease in the measured ion flux was observed. Voltages more negative than -200 V led to a precipitous drop in the ion flux due to most of the ions being deflected away from the substrate. These deflected ions eventually get collected by the deflector plate, and Fig. 3 shows that a high-energy, 200 eV ion should be collected by about -800 V. Simulations for an ion with a much lower energy (20 eV) show that it would easily be captured by this -800 V bias, thus suggesting that the majority of ions emanating from the plasma cell should be captured by about -800 V. To help corroborate this, the ion current collected at the negative deflector plate is shown in Fig. 4. The onset of current saturation that occurs around -800 V is in accordance with the simulated ion trajectories. Therefore, it is within this -300 to -800 Volt range where the applied deflector plate biases are sufficient to deflect the



damaging ions away from the substrate, but not enough to capture all of these ions with the deflector plate. The sufficient deflection of these ions away from the substrate position did, however, lead to a significant improvement in the sample's optical properties.

In conclusion, we have studied the effects of negative deflector plate voltages on the optical properties of dilute nitrides. Different negative voltages affect the ion flux and ion energy distribution at the substrate position during the plasma-assisted MBE growth, which in turn affects the optical properties of the semiconductor material. Our lower negative deflector plate voltages led to more ion damage because of an increase in the ion flux and an increase in the number of high-energy ions emanating from the plasma cell. Since various combinations of ion fluxes and ion energies led to samples with comparable optical properties, this suggests that the ion flux and ion energy can each contribute its own effect on the optical properties of dilute nitride semiconductors.

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FIG. 4. Ion current collected at the negatively biased deflector plate vs applied deflector plate voltage.

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