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Investigation of inversion domains in GaN by electric-force microscopy

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Inversion domains in III-nitride semiconductors degrade the performance of devices fabricated in them. Consequently, it is imperative that we understand their electrostatic manifestation, the growth conditions under which such domains form, and an effective means of their identification. In what is nominally referred to as Ga-polarity samples, N-polarity domains have a polarization that is reversed with respect to the remainder of the surface, and therefore, have a different potential under strain. We have used surface-potential electric-force microscopy (SP-EFM) to image the electrostatic surface potential of GaN grown on sapphire, which is strained due to the thermal mismatch between the substrate and GaN. Employing a control sample with side-by-side Ga- and N-polarity regions, we have established the EFM mode necessary to identify inversion domains on GaN samples grown by molecular-beam epitaxy. This method is not sensitive to topology and has a spatial resolution of under 100 nm. The measured surface potentials for Ga-face and N-face regions are $+25\pm10$ and -30 ± 10 mV, respectively, with respect to the sapphire substrate, where the sign is consistent with Ga- and N-polarity GaN under compressive strain due to thermal mismatch with the sapphire substrate. © *2001 American Institute of Physics.*
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Semiconductor nitrides (AlN, GaN, InN, and their ternaries) have received much attention recently for their potential use in optoelectronic, and high-power/temperature electronic devices.[1–4] The increased use of III-N semiconductors in commercial electronic devices has, therefore, led to a need for a better understanding of material defects that can hinder device performance.[5] One such defect is the presence of inversion domains, or mixed regions of Ga and N polarity within a GaN film. These defects are detrimental to device performance due to additional scattering induced by the electric field normal to the film surface.[6–8] By imaging the voltage at the surface by surface-potential electric-force microscopy (SP-EFM), both a qualitative and quantitative measure of polarization-induced charge distribution across the surface can be obtained. There have recently been several reports concerning EFM on III–V semiconductors,[9–11] but the lack of a well-defined sample has exacerbated efforts to identify the mode of operation necessary to unequivocally determine surface polarity.

In this work, we present SP-EFM results of a standard reference GaN sample intentionally grown to have surface regions of sapphire, AlN, and both Ga- and N-polarity GaN. Using the results from this standard reference, we were then able to appropriately interpret the SP-EFM data of samples grown under typical growth conditions. Moreover, we applied this method to a molecular-beam epitaxy (MBE) sample grown to have nominally Ga polarity in order to determine the presence and density of inversion domains.

SP-EFM is best defined as any configuration of an electric-force microscope that measures the surface contact potential of a given sample. There are various configurations that can be used, but all use an ac voltage signal applied to the tip or the sample. In this work, we apply both an ac and dc voltage to the tip and use a feedback loop to produce a real-time map of the surface potential.[11]

Typically, a lock-in amplifier detects the ω and 2ω signals and a feedback loop adjusts the sample height such that the ω signal goes to zero, thereby forcing the potential difference between the tip and sample to be zero. This technique is known as Kelvin probe force microscopy (KPFM),[12] and a variation of KPFM is used in our atomic-force microscopy (AFM) instrument.[13] In our case, the feedback loop is combined with a scanning mode known as the ''lift mode'' where two scans are taken per data acquisition line. In the first scan, the surface topography is measured at a smaller tip–sample distance, while in the second scan, the SP-EFM data are taken at a larger tip–sample distance that is adjusted so that the previously measured topography is subtracted. Consequently, the contribution from topography is minimized in the SP-EFM data.

We prepared a GaN reference sample by MBE in order to convincingly demonstrate the capabilities of the SP-EFM technique for GaN. The reference sample was grown and patterned such that Ga- and N-polarity regions, as well as bare sapphire, were simultaneously present on the surface. To grow this sample, a 2 in. sapphire wafer was mounted for normal growth and another sapphire wafer was cut in half and mounted so that it masked half of the substrate. An AlN buffer layer was then grown on the substrate, resulting in a sample consisting of an AlN buffer layer on one half and clean sapphire on the other. The substrate was then taken from the chamber, the mask removed, and the sample placed back into the chamber for growth (in air for less than 1 min). GaN was then grown on the substrate at 700 °C in a Ga-rich

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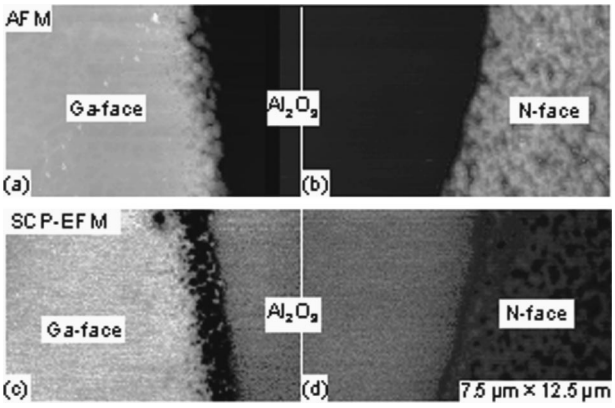

FIG. 1. Simultaneous AFM [(a) and (b)] and SP-EFM [(c) and (d)] images of the reference sample showing regions of Ga- [(a) and (c)] and N-terminated [(b) and (d)] films adjacent to regions where the sapphire substrate remains. In the AFM topography, brighter areas indicate higher regions, whereas in the SP-EFM image, brighter areas indicate regions with a higher surface potential. This image clearly shows that the Ga-face region has a higher surface potential than the N-face one.

environment, resulting in Ga-face growth on the AlN film and N-face growth on the sapphire substrate.[3]

To confirm the polarity of the two different regions, the sample was etched with H_3PO_4 at 140 °C. When Ga-polarity GaN is etched under these conditions, the acid attacks only the defective regions at the surface, leaving the (good) GaN intact. Under the same conditions, N-polarity GaN etches very quickly, resulting in either the complete removal of the GaN or a drastic change in the surface morphology. The effects of hot H_3PO_4 etching are similar to those of KOH studies done previously.[14] By comparing the results of etching on both surfaces, we verified that the surface did in fact consist of Ga- and N-polarity regions. AFM measurements indicated that the Ga-face side was 90–150 nm thick, while the N-face region was 70–160 nm thick. In addition to the reference sample, a typical GaN film was also characterized in this study. It was grown by MBE at ~800 °C to a thickness of 1.2 μm. A series of ten alternating layers of AlGaN and GaN strained layers were inserted below the GaN film to minimize defect formation/propagation. The approximate defect density was found by wet-chemical etching to be about 10^9 cm^{-2}.[15]

Our GaN reference sample contained regions having both Ga- and N-polarity faces, either adjacent to each other or separated by a 100 μm exposed strip of the sapphire substrate. Because the electric fields in both the Ga- and N-polar films are induced by strain between the sapphire substrate and the film, and both Ga- and N-polarity GaN layers are conductive, the potential difference between the film and the substrate will be consistent locally for films of both polarities. Therefore, to ensure that any surface contact potential difference between the Ga- and N-face regions was not an artifact of topographical features, SP-EFM images of both regions were taken in reference to the sapphire. Figure 1 shows AFM and SP-EFM images taken at a point where Ga-face [Figs. 1(a) and 1(c)] and N-face [Figs. 1(b) and 1(d)] GaN regions meet with the sapphire substrate. In Fig. 1, the gray-scale levels of the sapphire have been aligned so that the relative shift in surface contact potential for each surface can be seen. Brighter regions on the SP-EFM image represent higher potentials. In the AFM image, the Ga- and N-polarity regions have thicknesses of 95 and 70 nm with respect to the substrate, whereas in the SP-EFM image they have potentials 25 ± 10 mV higher and 30 ± 10 mV lower than the sapphire, respectively. The N face, therefore, has a surface potential approximately 55 mV below that of the Ga face.

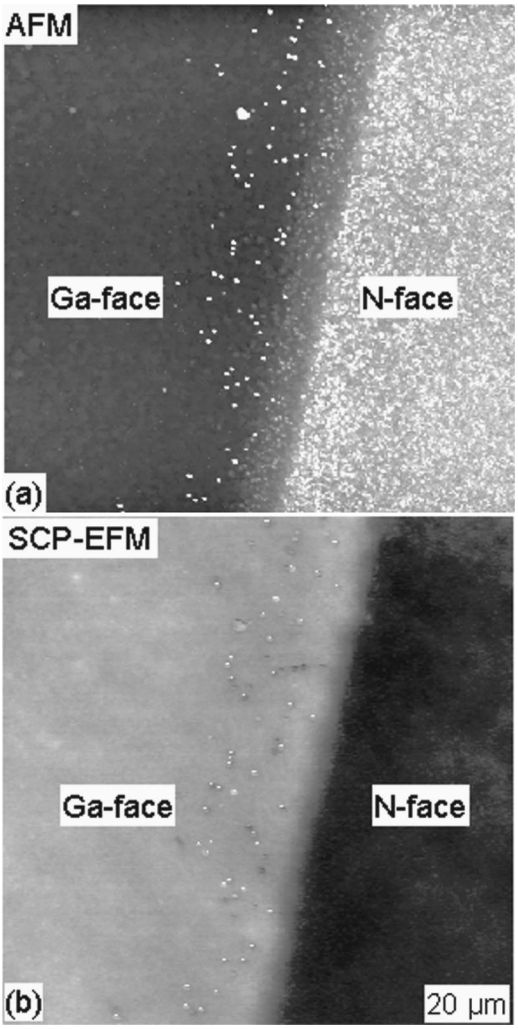

FIG. 2. Simultaneous AFM (a) and SP-EFM (b) images of the reference sample in a region where Ga- and N-terminated films are adjacent to one another. The Ga-face region is again at a higher potential than the N-face one.

This difference in polarity can also be clearly seen in areas where the N- and Ga-polarity regions are juxtaposed. Figure 2 shows an AFM and SP-EFM image taken where two such regions meet. In the AFM image [Fig. 2(a)], the Ga-face region on the left side is 40 nm lower than the N face on the right. The SP-EFM image indicates that although the Ga face is topographically lower, it has the expected higher surface contact potential as compared to the N face (95 ± 15 mV). On this sample, there are also regions where a Ga face is topographically higher than a neighboring N face. In such cases, the SP-EFM image continues to indicate a higher surface potential for the Ga- versus the N-polarity region (90 ± 15 mV), independent of surface topography. The positive sign of the surface potential for Ga-face regions is consistent with Ga-polarity GaN under compressive strain due to thermal mismatch with the sapphire substrate.

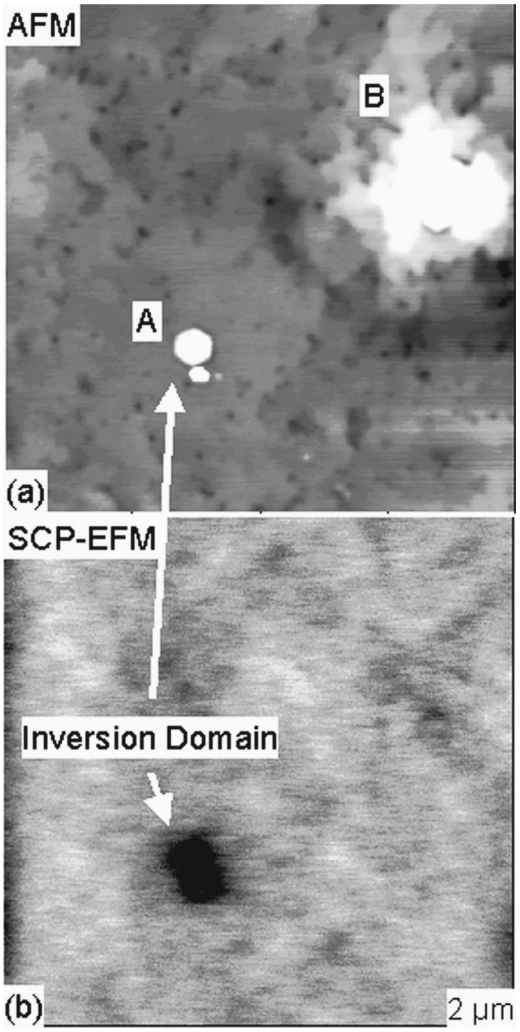

FIG. 3. AFM (a) and SP-EFM (b) images of a MBE sample. The hexagonal column (A) is at a lower potential than the remainder of the surface, indicating that it is an inversion domain.

The geometry of the tip and sample is usually modeled as a parallel-plate capacitor in the literature. However, it is more realistic to model the tip as a sphere of radius R (<10 nm) and the sample as a conducting plane, resulting in a capacitance[16]

$$C = 4\pi\epsilon_0 R \sum_{n=2}^{\infty} \frac{\sinh\alpha}{\sinh(n\alpha)},$$

where

$$\alpha = \ln\left[1 + \frac{z}{R} + \sqrt{\frac{z^2}{R^2} + 2\frac{z}{R}}\right], \tag{1}$$

and n is an integer. Using Eq. (1) with $R = 10$ nm and $z = 10$ nm, we calculate the capacitance of our system to be 3.6×10^{-18} F. Given this capacitance value, the circular sample surface area that produces the experimentally observed contact potentials has a radius in the range of 5–15 nm. This result supports our assertion that we are, in fact, imaging the GaN film polarity.

After demonstrating the reproducibility of the SP-EFM images on our reference sample, we proceeded to characterize a typical sample. Figure 3(a) shows an AFM image of a sample that has a hexagonal column-like feature (A) and a rough, raised area (B). By imaging these features with SP EFM, we were able to reveal the presence of inversion domains on the surface. The SP-EFM image in Fig. 3(b) shows that the sign of the surface potential is reversed in the area where a hexagonal column exists. The column is 52 ± 10 mV below the potential of the surrounding sample area, indicating that it is a local N-face region on a Ga-face sample. Furthermore, the rough raised area (B) has the same potential as the surrounding area, indicating that it is simply a topographical feature. These observations strengthen the assertion that the SP-EFM method indicates local variation in the surface contact potential and is independent of surface topography.

In conclusion, we have determined unequivocally that the surface potential mode of electron-force microscopy is sensitive to the polarity of the surface charge, thereby allowing the delineation of Ga- or N-terminated regions on GaN surfaces. The surface potential in regions with Ga polarity is about $+25\pm10$ mV with respect to the sapphire substrate, where the sign and magnitude of this value are consistent with predictions. After establishing the utility of SP-EFM by using a reference sample containing side-by-side Ga- and N-polarity regions, we applied this method to a MBE sample and determined the presence of inversion domains. Regions with N polarity in otherwise Ga-polarity films were observed very clearly without any sensitivity to surface topography. SP-EFM is, therefore, a reliable and nondestructive technique for determining the local surface polarity of GaN samples.

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[1] H. Morkoç, *Nitride Semiconductors and Devices* (Springer, Heidelburg, 1999).
[2] S. T. Strite and H. Morkoç, J. Vac. Sci. Technol. B **10**, 1237 (1992).
[3] O. Ambacher, J. Phys. D **31**, 2653 (1998).
[4] S. J. Pearton, J. C. Zolper, R. J. Shul, and F. Ren, J. Appl. Phys. **86**, 1 (1999).
[5] H. Morkoç, R. Cingolani, and B. Gil, Solid-State Electron. **43**, 1909 (1999).
[6] H. Tang, W. Kim, A. Botchkarev, G. Popovici, F. Hamdani, and H. Morkoç, Solid-State Electron. **42**, 839 (1998).
[7] H. Morkoç, R. Cingolani, and B. Gil, Mater. Res. Innovations **3**, 97 (1999).
[8] Sverdlov, G. A. Martin, H. Morkoç, and D. J. Smith, Appl. Phys. Lett. **67**, 2063 (1995).
[9] P. M. Bridger, Z. Z. Bandic, E. C. Piquette, and T. C. McGill, Appl. Phys. Lett. **74**, 3522 (1999).
[10] P. M. Bridger, Z. Z. Bandic, E. C. Piquette, and T. C. McGill, J. Vac. Sci. Technol. B **17**, 1750 (1999).
[11] Q. Xu and J. W. P. Hsu, J. Appl. Phys. **85**, 2465 (1999).
[12] M. Nonenmacher, M. P. O'Boyle, and M. K. Wickreamasinghe, Appl. Phys. Lett. **58**, 2921 (1991).
[13] Digital Instruments Nanoscope III and Dimension 3110 instruments were used.
[14] M. Seelmann-Eggebert, J. L. Weyher, H. Obloh, H. Zimmermann, and A. Rar, Appl. Phys. Lett. **71**, 2635 (1991).
[15] P. Visconti, K. M. Jones, M. A. Reshchikov, R. Cingolani, H. Morkoç, and R. Molnar, Appl. Phys. Lett. **77**, 3532 (2000).
[16] D. Sarid, *Scanning Force Microscopy* (Oxford University Press, New York, 1991).