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Photo-assisted local oxidation of GaN using an atomic force microscope

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Abstract
This paper introduces a photo-assisted atomic force microscope (AFM) local oxidation technique which is capable of producing highly smooth oxide patterns with heights reaching several tens of nanometres on both n- and p-types of GaN (and in principle on most semiconductors) without the use of chemicals. The novel methodology relies on UV illumination of the surface of the substrate during conventional AFM local oxidation. A low 1.2 V threshold voltage for n-type GaN was obtained, which can be explained by UV photo-generation of excess electron–hole pairs in the substrate near the junction, thereby reducing the electric field required to drive carrier flow through the tip–sample Schottky barrier. It was demonstrated that the presence or absence of light alone was sufficient to switch the growth of the oxide on or off. The photo-assisted AFM oxidation technique is of immediate interest to the semiconductor industry for the fabrication of GaN-based complementary metal–oxide–semiconductor devices and nanodevices, improves chances for AFM-type data storage, and presents new degrees of freedom for process control technique.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Local oxidation using an atomic force microscope (AFM), sometimes called AFM oxidation, nanooxidation or local oxidation nanolithography (LON), is a promising scanning-probe-based lithographic technique suitable for the fabrication of nanoscale structures and devices [1–8]. The AFM nanooxidation technique uses the very strong electric field that can occur between the AFM conducting tip and the substrate. In normal air or other humid atmosphere, both the AFM tip and the surface of the substrate are covered by a thin film of absorbed water. When the tip approaches sufficiently close to the surface, the two absorbed water layers automatically join and form a 'water bridge' (water meniscus). The oxidant for the oxidation reaction is provided by oxyanions such as OH^- and O^{2-} ions in the water bridge [2, 9]. The combined effect is capable of inducing anodic oxidation on the substrate and forming nanoscale oxide patterns. This technique has been used for local oxidation of various substrates such as silicon, GaAs [10], InP [11], silicon nitride [5, 12], silicon carbide [13], metals [14–18], transition metal nitrides [18] and even oxides [7, 19–21]. Various devices such as field effect transistors (FETs) [1, 7, 22, 23], single electron memories [4], Josephson junctions and superconducting quantum interference devices (SQUIDs) [6, 21] have been demonstrated. Various improved LON techniques have shown enhanced growth rates and oxide heights, for example by employing an UV-induced ozone-rich meniscus [24], ethyl alcohol meniscus [25] or metal deposition [11], making LON techniques of particular interest to researchers interested in mass-produced nanoscale devices.

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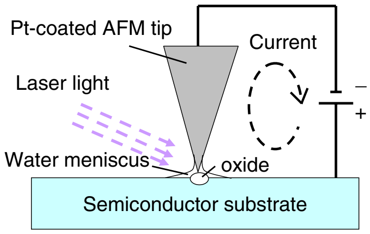

Figure 1. Schematic diagram of photo-assisted AFM local oxidation of GaN.

In earlier work our group achieved AFM oxidation on two important semiconductors, GaN and InN [26], which are of interest for high-speed, high-power and light-emitting electronic applications because of various superior optoelectronic properties. AFM oxidation of GaN is of particular importance to the development of GaN-based metal–oxide–semiconductor FETs (MOSFETs) with low trap density at the oxide/GaN interface. However, GaN and InN are difficult to AFM-oxidize because of high chemical stability. Our studies achieved AFM oxidation of p-type GaN (p-GaN) substrate by using initial deposition of a thin gold layer on the substrate to enhance the initial oxidation current and thus the oxidation rates, producing oxide heights of several tens of nanometres [26], but this technique was debited by intrinsic gold contamination of the oxidized region. Subsequently we found the gold film was necessary only in the immediate vicinity of the region to be oxidized, producing thereby gold-free FM oxidation on p-GaN. Further work found the gold deposition methodology unsuited to enhance AFM oxidation on n-type GaN (n-GaN) substrates due to a Schottky barrier occurring during contact between the Pt-coated AFM tip and the n-GaN substrate (refer to a review on metal/GaN contacts by Liu and Lau [27]). On an n-GaN substrate, it is necessary to use a positive bias voltage to draw oxyions to the oxide/n-GaN interface for anodic oxidation, but applying this bias to the Pt (AFM tip) adjacent to n-GaN creates a revered-biased Schottky junction. This prevents electron transfer from the Pt tip to the substrate, i.e. cuts off most of the oxidation current.

In this present report, to solve these difficulties, we adopt a methodology derived in part from the techniques found in photochemical wet oxidation on GaN [28] and in photo-assisted scanning tunnelling microscopy [29]. The proposed technique employs laser light during AFM oxidation, allowing nanooxidation on p- and n-GaN without the assistance of gold deposition or other chemicals (excepting the water vapour in air as the 'water bridge'). Figure 1 shows a schematic diagram of the proposed photo-assisted local oxidation (PaLO) methodology, wherein AFM oxidation on n-GaN is made possible through the illumination of light with energy surpassing the band gap of GaN to produce photogenerated carriers for passing through the Schottky barriers, resulting in enhanced or activated AFM oxidation. Our experimental results are surprisingly satisfying, since oxide heights exceeding several tens of nanometres are achievable on both p- and n-types of GaN. Importantly, the same methodology can in principle be applied to most semiconductor substrates, which suggests a new approach to fast, clean and lithography-enabled fabrication of oxides on semiconductors with enhanced growth rates and heights, in a photo-controllable manner. It is believed that the technique's success in local oxidation of both types of GaN also sheds light on future fabrication of complementary metal–oxide–semiconductor (CMOS) devices using AFM technology. Moreover, the photo-control aspects of the proposed technique are of special interest because of its offering a new degree of manufacturing freedom.

2. Experiments

Conventional metalorganic chemical vapour deposition (MOCVD) was used to create both n- and p-GaN films on sapphire substrates. The p-GaN carrier concentration was 1.3×10^{16} cm^{-3} and the n-GaN was 5×10^{16} cm^{-3}. AFM oxidation was performed with an NT-MDT P47H atomic force microscope (NT-MDT Co., Russia) using the contact mode under a continuous negative bias with a conductive Pt-coated silicon tip scanning at 1000 Å s^{-1} at 70% humidity. During oxidation, a 266 nm (deep UV) diode-pumped microchip laser (JDSU, USA) with 2 mW averaged power operated at 6–10 kHz pulse rate was aimed at an incidence angle of approximately 2° onto the GaN substrate. The laser power density on the GaN surface was estimated to be 100 W m^{-2}. Note that usage of the UV laser is key to the success of the photo-assisted oxidation on GaN, since the proposed technique relies on the capability of UV light to generating excess electron–hole pairs and thus photocurrents under the reversed applied bias of the aforementioned Schottky junction. Use of the 266 nm laser is of additional practical benefit in terms of our hardware since the bandwidth of the laser is quite narrow and the 266 nm wavelength has little effect on the light-sensitive diode used as the position sensor when sensing the deflection of the AFM cantilever.

3. Results and discussion

Figure 2 summarizes typical results of photo-assisted AFM oxidation on n-GaN. The AFM micrograph figure 2(a) shows four oxide lines, each formed by moving the AFM tip from top to bottom of the figure and representing from left to right applied sample–tip voltages of 10, 8, 6 and 4 V. Illumination by the 266 nm laser beam during oxidation was the same for all four lines. It can be seen that the 4 V oxide line is quite constant from start to finish, but each of the lines for the higher voltages shows an initial point of greater brightness that will be shown below to correspond to initial growth overshoot in height, after which the growth stabilizes. The stabilized height profiles in figure 2(b) show, as expected, an increase in oxide height with increased applied voltage. Figure 2(c) shows the height profiles of the lines along their length, confirming initial growth overshoot. Such growth rate spiking, to our knowledge, has not been reported in the literature. We speculate that this transient oxide growth behaviour is related to water meniscus instability due to rapid consumption during high growth rates under photo-assisted oxidation. An additional factor or alternative cause might be that the high growth rate results in poor feedback control of the AFM tip height during growth. In figure 2(d) the experimental conditions have been changed. The AFM tip is moved laterally at a constant rate and the AFM tip voltage is held at 10 V, while at the same

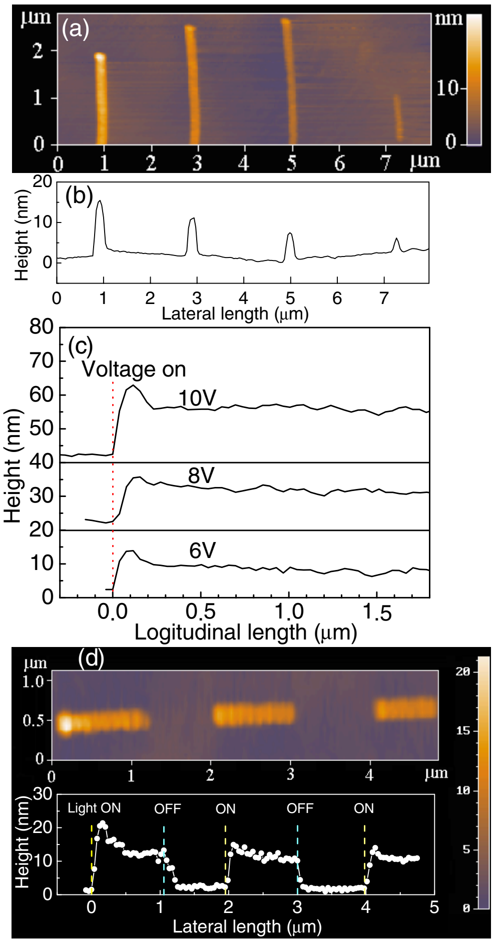

Figure 2. Results of photo-assisted AFM oxidation of n-GaN: (a) AFM image of oxide lines grown at various applied tip voltages; (b) stabilized AFM height profile of (a); (c) longitudinal height profiles for oxide lines in (a) excluding the 4 V line; (d) AFM image (upper) and corresponding height profile (lower) of single uninterrupted AFM pass with tip bias at a constant 10 V but with the laser illumination interrupted twice during the pass.

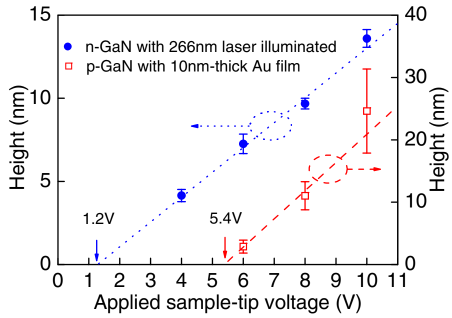

Figure 3. Stabilized oxide heights at different applied voltages on 266 nm laser photo-assisted n-GaN substrate (solid circles) and on 10 nm gold-coating-assisted p-GaN substrate (empty squares), with standard deviation error bars.

time the laser diode is cyclically switched on and off and back on. This process results in the distinctive growth behaviour that can be observed in figure 2(d). Once again, initial spiking of the growth rate results in a regular overshoot of the ridge at each new onset of growth. Interestingly, the amount of spiking decreases with each subsequent growth restart, but the growth rate seems to trend to a constant value. Our experiments are at an early stage and are ongoing. It should be mentioned that photo-assisted local oxidation was applied under identical conditions to our p-GaN substrate. The results (not shown) are essentially the same as those obtained for the n-GaN. Oxide heights reaching tens of nanometres were easily attained. Initial height overshoot was observed. The average height increased with increasing AFM tip voltage. The oxide height on the p-GaN substrate was larger than that on the n-GaN, presumably because the Schottky junction was forward biased for the p-GaN sample, thus allowing larger initial current for PaLO growth.

Significantly, figure 2(d) demonstrates control of an AFM local oxidation process by the use of applied light alone. To our knowledge, this is the first report of such behaviour. It is too early to have a clear understanding of the range of practical control that can be obtained by this methodology. Nevertheless, it seems that light-controlled AFM oxidation offers a new degree of freedom for the remote control of oxide growth and may become an important tool in the development and industrial implementation of future nanosystems, for example AFM-type data storage.

Figure 3 plots the dependence of the n-GaN average oxide height on the applied voltage under UV illumination as measured in the stabilized region seen in figures 2(a)–(c), where the solid circles indicate the height values with standard deviation error bars estimated along the oxide lines. For comparison, figure 3 also shows (empty squares) the oxide height dependence on the applied voltage for gold-film enhanced AFM-oxidation of p-GaN from our previous study [26]. Notably, the threshold minimum AFM voltage required for UV-illuminated n-GaN is a mere 1.2 V. This is remarkably close to zero and is certainly quite small in comparison to the threshold values for our earlier gold-deposited p-GaN (∼5.4 V) and the thresholds measured on various easily AFM-oxidized substrates such as GaAs (∼7 V) [30], Si (⩾2.7 V) [31] and metal compounds (⩾4 V) [32]. In a UV-illuminated Schottky junction, photocurrents in principle are generated automatically even without applied voltage, so the low threshold applied voltage in our test can be explained by the UV illumination creating electron–hole pairs in the substrate near the junction, thus reducing the required applied field for driving carriers flow through the tip–sample Schottky barrier. In our present series of experiments, nearly the whole GaN substrate surface was exposed, more or less, to UV illumination by scattering of the UV light through the substrate despite focusing of the incident laser beam on the area of the tip–substrate contact. In addition to reducing the effective resistance of the tip–substrate junction, the photocurrent produced in the GaN substrate by the incident light at an applied voltage also helped increase the total circuit conductance, hence increasing the oxidation current and resulting in increased oxidation rates and heights. In addition to the low threshold applied voltage, there is another advantage of the photo-assisted AFM oxidation technique. As can be seen from the error bars in figure 3, the smoothness of the oxide line grown using the photo-assisted growth, compared to that grown using gold-assisted growth,

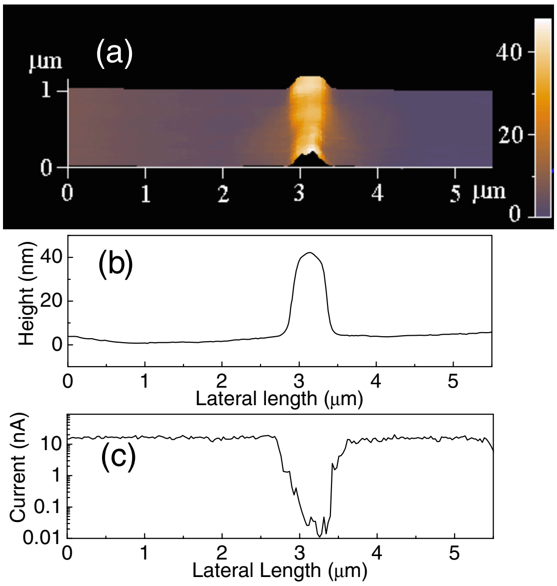

Figure 4. n-GaN substrate after photo-assisted AFM oxidation at an applied voltage of 10 V: (a) AFM image; (b) oxide height; (c) corresponding current profile under illumination.

is greatly improved, which is a feature of particular interest to future device fabrication technology as devices become smaller and manufacturing variance becomes an increasingly critical issue.

To further verify the presence of the oxide, current mapping during illumination by the 266 nm laser was conducted on the n-GaN substrate after photo-assisted AFM oxidation. The illumination was used to improve discrimination between the substrate and the grown oxide because of the limited current sensitivity of the AFM. Improved discrimination can be achieved since the photon energy at 266 nm (~4.66 eV) laser is sufficient to excite electrons from the valence band to the conduction band in n-GaN but not in the gallium oxide, which has a band gap near 4.8–5.0 eV [33]. The AFM height image of an oxide line produced at an applied sample–tip voltage of 10 V is shown in figure 4(a), with the height profile and the illuminated current mapping profile shown respectively in figures 4(b) and (c). The low current seen clearly in figure 4(c) correlates specifically with the peak in height seen in 4(b), which is strong evidence that the peak is insulating oxide. Note that a better focusing of the laser beam was employed to obtain the oxide height of 40 nm seen in figure 4(b), which is considerably higher than that obtained in figure 2. The higher laser power density (estimated to be 300 W m^{-2}) was to help enlarge the photocurrent and thus improve discrimination between the substrate and the grown oxide. On the basis of our experience, we have no doubt that the PaLO oxide height increases with increasing laser power density. Nevertheless, a detailed study of the dependence of oxide height relative to illumination power density is needed and, in fact, is in the process of being performed and will be included in a pending report.

4. Conclusions

This study introduced a photo-assisted local oxidation technique using an atomic force microscope. The proposed technique was shown by experiment to be capable of producing highly smooth oxide patterns with heights reaching several nanometres on both types of GaN. In principle, the PaLO technique should be useful on most semiconductors, without use of chemicals. A remarkably low threshold AFM voltage of 1.2 V was obtained on n-type GaN. The low required voltage was explained by photo-activation of excess electron–hole pairs in the substrate near the junction, allowing easy flow of current through the tip–sample Schottky barrier. It was demonstrated that the presence or absence of the UV illumination could switch the local oxide growth on or off, i.e. growth could be controlled by light alone, an observation we believe to be the first of its kind. It is believed that photo-assisted AFM oxidation will become an important part of future fabrication techniques for GaN-based complementary metal–oxide–semiconductor (CMOS) devices, nanodevices and AFM-type data storage. Moreover, the photo-control aspects of the proposed technique are of special interest because of offering a new degree of manufacturing freedom.

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