

Method for electrical characterization of nanowires

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Abstract

The ability to control conductivity in semiconductor nanostructures is often challenged by surface states trapping the majority of the charge carriers. Addressing this challenge requires a reliable method for assessing electrical properties such as carrier concentration and mobility. Unfortunately, here we are facing another challenge, as the Hall effect is geometrically inapplicable to nanowires while the field effect model is also challenged by the geometry of the common nanowire field effect transistor, and can only yield channel mobility which is very different from Hall mobility. In this paper, we propose a method that combines resistivity and photovoltage measurements with a chemical perturbation to the surface to measure carrier concentration and mobility, as a function of wire diameter, and also to measure the surface state density and the surface band bending before and after the chemical treatment. We apply this method to CVD grown GaN nanowires, before and after a mild HCl etch. Using transmission electron microscope and x-ray photoelectron spectrometry we find that HCl removes the native gallium oxide. The etch is found to reduce the surface state density from 1×10^{12} to $5.3 \times 10^{11} \text{ cm}^{-2}$, which is calculated from a reduction of the critical radius for full depletion from 7.6 to 4 nm, and a reduction of the surface band bending from 0.53 to 0.29 eV, observed using surface photovoltage. On the average, the values of carrier concentration we obtain are about ten times smaller, and the mobility about ten times greater, than values obtained using field effect transistors. Interestingly, the weak size dependence of the mobility disappears after etching, suggesting a causal linkage between the as-grown size dependence of the mobility and the density of surface states. The proposed method provides an experimental handle to the study of surface states and their effects on the electrical properties of nanowires.

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

1. Introduction

Semiconductors are insulators that can be made conductive by means of the controlled incorporation of contaminant. Our ability to control their carrier concentration is what makes semiconductors useful. However, doping technology, as we know it, is facing a major challenge when applied to small structures. As dimensions shrink, the role of charge exchange with surface states becomes dominant in defining the very basic property of a semiconductor—its conductivity [1–4]. The charge trapped by surface states is typically negligible in bulk material. However, as dimensions become smaller the decrease in surface area is slower than the relative decrease in volume, i.e. the ratio of surface to volume increases, until surface phenomena are no longer negligible [5, 6]. To address

this challenge, we need methods to modify and control surface properties, as well as methods to evaluate the effect of these modifications on properties related to conductivity, such as carrier concentration and mobility. A prominent manifestation of the effect of surface states on small structures is full depletion, when surface states trap carriers, emptying the structure completely of its majority carriers [7]. The issue of full depletion in nanowires has been addressed in several studies and several attempts have been made to extract various parameters from resistivity/conductivity data and to develop methods to extract parameters such as carrier concentration and mobility, avoiding the inaccuracies resulting from the known inadequacy of the field effect model for nanowire geometry [8–11]. However, mobility and carrier concentration are not readily separable using conductivity alone. In this

paper, we show how a combination of photovoltage and resistivity measurements may be used to evaluate the effect of a surface treatment on the density of surface states. Using this density of surface states, we are able to calculate the average carrier concentration and mobility, and the surface band bending. We use our model to study the effect of a mild HCl etch on the surface state density, and the surface band bending, in GaN nanowires.

2. Model

Most semiconductor surfaces are naturally depleted due to surface states positioned deep within the forbidden gap. In n-type semiconductors, such surface states typically trap conduction electrons, charging the surface negatively¹. The interplay between the density of these surface traps, N_T , and the carrier concentration within the wire, which is mostly the doping density, N_D ,² along with the surface band bending, ϕ_{BB} , define the depth of the surface space charge region, d . Solving the Poisson equation for a cylindrical nanowire of radius R , where the wire is depleted between $r = r_0$, and R , it can be shown that the internal field is given by

$$E(r) = \frac{qN_D}{2\epsilon} \left(r - \frac{r_0^2}{r} \right) \quad (1)$$

where q is the electron charge and ϵ is the dielectric constant. This field reaches its maximum at the surface ($r = R$). Using the Gauss theorem, we get the relation between this maximum and the density of charge trapped in surface states:

$$E_{\max} = E(R) = \frac{qN_D R}{2\epsilon} \left(1 - \frac{r_0^2}{R^2} \right) = \frac{qN_T}{\epsilon} \quad (2)$$

$$1 - \frac{r_0^2}{R^2} = \frac{2}{R} \frac{N_T}{N_D}. \quad (3)$$

For certain N_T and N_D , all the free carriers within the wire will be trapped at surface traps, i.e. the wire will be fully depleted ($r_0 = 0$). Equation (3) tells us that this happens when the wire radius equals

$$R_0 = 2 \frac{N_T}{N_D}. \quad (4)$$

What equation (4) means is that there are donors that contribute N_D carriers per unit volume and some of them are trapped at surface traps, with areal density (number per unit area) N_T . As the radius of the wire is made smaller both the volume and the surface area become smaller, but not at the same rate. The volume is made smaller faster than the surface area. Therefore, the actual number of donors (N_D times the volume) is made smaller faster than the actual number of surface traps (N_T times the surface area). This goes on, until at a certain radius, R_0 ,

¹ Surface accumulation or inversion are also possible but less common than depletion due to the high density of surface states required to create them. See, for example, [12].

² We assume a full ionization of the dopants, while this may not always be the case, e.g., at low temperatures, a case that is not treated in the basic model presented here. We also assume a uniform distribution of the dopant both radially and longitudinally. Radial non-uniformity will require solving the Poisson equation for the specific conditions of each case.

these numbers equalize, and all the free carriers are actually trapped within an equal number of surface traps leaving the volume of the wire void of mobile carriers. If $\rho(R)$ is the resistivity of the undepleted wire and $\rho(r_0)$ is that of a partially depleted wire, then these resistivities relate like the inverse of the undepleted volumes

$$\rho(r_0) = \rho(R) \frac{R^2}{r_0^2}. \quad (5)$$

Substituting equations (3) and (4) into equation (5), we get

$$\rho(r_0) = \rho(R) \frac{R}{R - R_0}. \quad (6)$$

What this means is that as the wire radius, R , approaches R_0 , or in other words as we approach full wire depletion, the wire resistivity should increase sharply, as would be expected intuitively. This rise in the resistivity is a result of the diminishing carrier concentration. The carrier concentration for a wire of radius R may as well be written in terms of R_0 as

$$n(R) = N_D - \frac{2N_T}{R} = N_D \left(1 - \frac{R_0}{R} \right) \quad \text{for } R_0 \leq R$$

and 0 for $R_0 > R$. (7)

Once we know R_0 and N_D , we can calculate the function $n(R)$.³ We can reliably obtain R_0 by fitting the resistivity data with equation (6). We assume that the function describing the mobility does not vary dramatically approaching R_0 . As we show later, our results justify this assumption. The reason we prefer to work with resistivity rather than conductivity is that while the conductivity goes to zero at full depletion, which is a finite variation, the resistivity goes to infinity, i.e. the magnitude of the effect is greater. The next question is how we obtain the value of N_D . When the depletion is only partial, the relations between N_D , N_T , and the depletion width, d , are given by (substituting $r_0 = R - d$ into equation (3))

$$N_D \frac{\pi(R-d)^2}{\pi R^2} = N_D - \frac{2N_T}{R} \implies N_T = N_D \left(d - \frac{d^2}{2R} \right). \quad (8)$$

Equations (8) and (4) allow us to express d in terms of R_0

$$d = R \left(1 - \sqrt{\frac{1 - R_0}{R}} \right). \quad (9)$$

Now we write the expression for the band bending potential at the surface [1]

$$\phi_{BB} = \frac{qN_D}{2\epsilon} \left[\frac{d(2R-d)}{2} - (R-d)^2 \ln \left(\frac{R}{R-d} \right) \right]. \quad (10)$$

And by substituting equation (9) into (10), we obtain an expression for the surface band bending as function of R_0

$$\phi_{BB} = \frac{qN_D}{4\epsilon} \left[R_0 R - \left(1 - \frac{R_0}{R} \right) R^2 - \ln \left(1 - \frac{R_0}{R} \right) \right]. \quad (11)$$

³ Note that a function $f(R)$ denotes a function of the total wire radius (half its diameter), and not of the radial displacement within a single wire, e.g. $n(R)$ is an average carrier concentration within a wire of radius R .

Equation (6) can be used to fit the resistivity data, yielding R_0 . If we could measure ϕ_{BB} , we could then use equation (11) to calculate N_D . In this way we get the values for all three parameters: N_D , N_T , and ϕ_{BB} . We also get the function $n(R)$. Dividing the resistivity data by $n(R)$ should give the mobility as a function of the wire radius, $\mu(R)$. Therefore, the question of how to find N_D is now reduced to the problem of measuring ϕ_{BB} . We note that our model assumes that N_D and N_T do not vary significantly among wires of the same growth run, and the values we obtain are the mean values. We also use the depletion approximation. While this approximation may not be valid for thin nanowires, the parameter d may still be used as an ‘effective’ depletion width. To measure ϕ_{BB} , we need a technique that is capable of measuring the surface potential. There are several methods that may be applicable, e.g. x-ray photoelectron spectrometry, ultra-violet photoelectron spectroscopy [13]. One of the oldest and most common methods for following the surface potential is the Kelvin probe technique. Due to inherent inaccuracies in measuring the surface potential itself, this method is used mostly for measuring changes in the surface band bending, e.g. surface photovoltage, in which we measure the change in surface potential upon illumination [14]. Measuring the surface potential using surface photovoltage is possible if one can photo-induce a complete flattening of the bands at the surface [15]. Flattening the bands using light, dubbed photosaturation, is not always possible for all semiconductor surfaces, and depends on the surface recombination velocity in each case [16]. However, using the following method, we can test whether we can achieve photosaturation. To this end, we perform a chemical modification of the surface. Any reaction taking place at the surface is supposed to alter the surface state density to some unknown extent [14]. Before and after the surface modification, we measure the surface photovoltage under identical illumination intensity with white light containing photon energies exceeding the bandgap energy. Subtracting the two values, we obtain $\Delta\phi_{BB}$ instead of ϕ_{BB} (provided photosaturation conditions have been achieved). In this way we eliminate possible unknown photovoltage contributions of internal interfaces, which are not sensitive to surface treatments (and therefore cancel out), and keep the desired surface photovoltage alone. We also measure the resistivity in our set of resistor devices and get two values of R_0 , before and after the surface modification. Assuming our surface modification does not affect N_D , we can now calculate N_D . Once we obtain N_D , we can obtain N_T . However, the main surprise is that we can eventually use these values to calculate ϕ_{BB} before and after the surface modification. To verify whether photosaturation was actually achieved, we compare our photovoltage values to the obtained ϕ_{BB} . If they are equal, we can tell that photosaturation has been reached, and the resulting N_D , N_T , and ϕ_{BB} values are correct. If photosaturation is not feasible, we may turn to other methods to evaluate ϕ_{BB} . Once we have N_D , we can extract the carrier concentration and the mobility from our resistivity data, both before and after the surface treatment. Thus, beside this wealth of parameters, the method also affords an experimental handle to the evaluation of the effect of surface treatments on material parameters in nanowires.

3. Experimental details

To test our model experimentally, we used GaN nanowires grown by chemical vapor deposition (CVD). All wires were grown under the same conditions on substrates of Si(111) and GaN film on sapphire, using Ni nitrate nanoparticles as a catalyst. Ni cannot make a typical vapor–liquid–solid mode catalyst because it readily forms a Ni nitride and is therefore believed to catalyze nitride nanowire growth by promoting nucleation, while remaining at the substrate–wire interface [28]. The wires were transferred to a substrate of SiN over Si, and were contacted with four contacts each, using e-beam lithography, deposition of Ti/Au and liftoff. Scanning electron microscope imaging was used to measure wire diameter and distance between contacts. Resistivity was calculated from resistance using wire dimensions in scanning electron microscope images. For reference, we also made three back-gated nanowire channel field effect transistors from nanowires of the same growth on p⁺-Si substrate and obtained carrier concentration and mobility using the field effect model. To modify the surface state distribution, we immersed the substrate with the device in 20% HCl for 20 s, followed by a rinse in deionized water. Chlorine and HCl are known to affect the surface state density on GaN surfaces [17, 18]. Surface photovoltage was measured using a Kelvin probe (Besocke Delta Phi) and white light from a Xe lamp. The Kelvin probe (a Au mesh) is brought to a distance of about half a millimeter from the surface of a semiconductor, forming an air gap capacitor with the semiconductor surface. The probe is then vibrated using a piezoelectric crystal to modulate the air gap. The modulation of the air gap results in an AC current through the capacitor. A control circuit applies a DC voltage to the Kelvin probe until the AC current is nullified. The DC voltage required to nullify the AC current is, under ideal conditions, the surface voltage. Under nonideal conditions, there are certain errors to this measurement, for which the actual voltage, dubbed the ‘contact potential difference’ (CPD), deviates from the desired surface voltage. However, when measuring photovoltage, we subtract the CPD under illumination from its value in the dark. Since the errors are insensitive to light, they cancel each other and render the photovoltage measurement, i.e. photo-induced change in the surface voltage, accurate. For an extensive review of the method see [14]. We carried out the measurements on the original Si growth substrate and measured a collective photovoltage value for the entire ensemble of nanowires. We measured the contact potential difference in the dark, and then we irradiated the sample and measured again. These two measurements were repeated after etching in HCl. X-ray photoelectron spectrometry (XPS) was carried out on GaN nanowires grown on GaN film. The measurements were carried out in ultrahigh vacuum using a SSX 100 spectrometer, Surface Science Instruments (USA), with a hemispherical electron energy spectrometer and monochromatized Al K radiation source at a pass energy of 100 eV. The acquisition time was kept constant for all the measurements. The binding energies were corrected by referencing the C 1s binding energy to 284 eV.

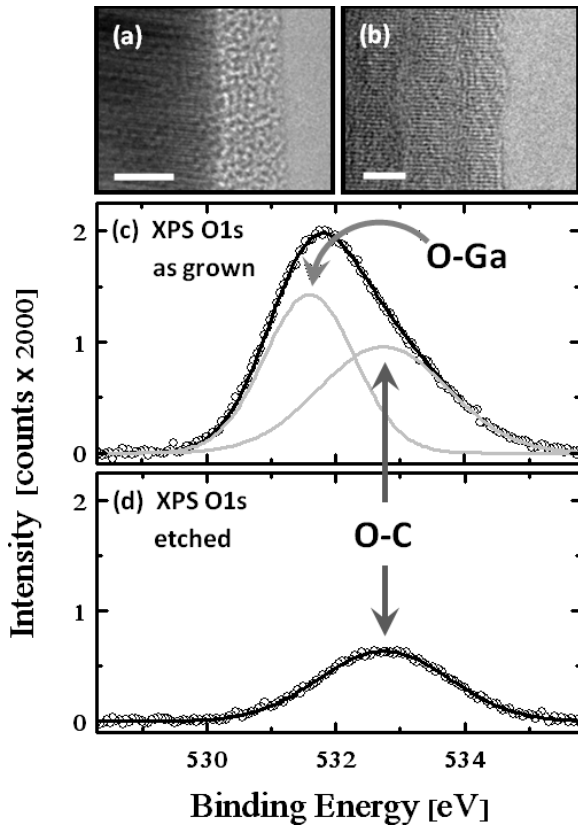


Figure 1. Effect of HCl etch on GaN nanowire surfaces. (a) TEM before etch shows an amorphous layer on the surface (bar is 5 nm) and after etch (b) the amorphous layer is gone (bar is 5 nm). (c) XPS before etch is composed of two peaks, one, at 531.6 eV, is identified as Ga-bound oxygen, the other, at 532.75 eV, is identified as C-bound oxygen. (d) XPS after etch shows no trace of the O–Ga peak and a small decrease in the O–C peak height.

4. Results

Figures 1(a) and (b) show a transmission electron microscope high resolution image of a typical GaN nanowire surface, before and after HCl etch. An amorphous layer of about 5 nm is observed on the surface of the untreated wire. This amorphous layer is absent in the treated wire. Electron dispersive spectroscopy carried out on the untreated wire showed mostly gallium, nitrogen, and oxygen. To obtain a better idea of the chemistry involved in the etching process, we used x-ray photoelectron spectroscopy (XPS). Since most of the Ga in our sample is bound to N, and vice versa, changes in the Ga and N core levels were not informative. Figures 1(c) and (d) show the O 1s core level peak before and after etching. Before etching, the peak is composed of two prominent peaks.

One of these peaks is absent after etching. The position of this etched peak matches the reported binding energy of O 1s in the O–Ga bond (531.6 eV) [19, 20]. It therefore seems likely that the amorphous layer removed by etching is a native oxide of GaN. Figure 2 shows resistivity data obtained from 19 nanowire resistor devices before and after etching with HCl. Fitting the data with equation (6), we obtain $R_0 = 7.6$ and 4 nm, before and after etching, respectively. Using these results, we calculate $N_T/N_D = 3.8 \times 10^{-7}$ and 2×10^{-7} ,

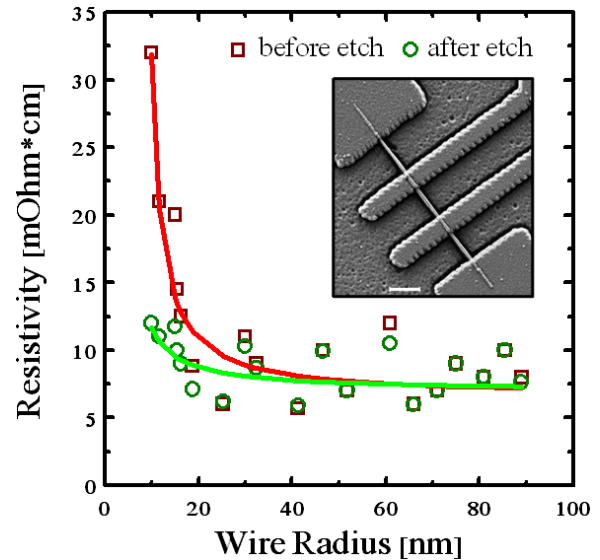


Figure 2. Effect of HCl etch on GaN nanowire resistivity: squares show data before etch, circles show data after etch, and the solid lines show fit. Inset shows SEM image of a device. The bar is 500 nm.

respectively. The photovoltage measured before etching was 0.51 V and after etching was 0.27 V. The difference between these two values is $\Delta\phi_{BB} = 0.24$ eV. For R , we used the average wire diameter in the photovoltage sample, $R_{AV} = 29$ nm, and using relative permittivity of $\epsilon_r = 9.2$ for GaN, we get $N_D = 2.6 \times 10^{18} \text{ cm}^{-3}$. From this value we calculate $N_T = 1 \times 10^{12}$ and $5.3 \times 10^{11} \text{ cm}^{-2}$, before and after etching, respectively. Using these values, we can now calculate the band bending potentials. We get $\phi_{BB} = 0.53$ and 0.29 eV, before and after etching, respectively. These values are very close to the measured photovoltages, suggesting that we were very close to the required photosaturation. Using N_D , we can also calculate the number of donors in a $1 \mu\text{m}$ length of a wire with radius of R_0 (15 nm diameter). At this radius, we have 480 donors per unit length of $1 \mu\text{m}$ donating 480 electrons that are captured by exactly 480 surface traps (per same unit length), leaving the wire intrinsic, in spite of a degenerate level of doping.

Finally, substituting N_D and R_0 , into equation (7), we can now calculate the average carrier concentration as a function of the radius and use this function to extract the mobility values from the fitting function of our resistivity data. The results are shown in figure 3, where the average functions $n(R)$ and $\mu(R)$ are compared before and after etching. Before etching, the carrier concentration decreases by 74% over the range of sizes studied. A similar trend is observed after etching with a smaller decrease of 37%. The mobility decreases with the wire radius by 14% before etching and by 0.01% after etching, i.e. after etching the mobility is practically invariant with the radius.

5. Discussion

The purpose of our experiment was to test our model. As input, our model uses a combination of resistivity data as

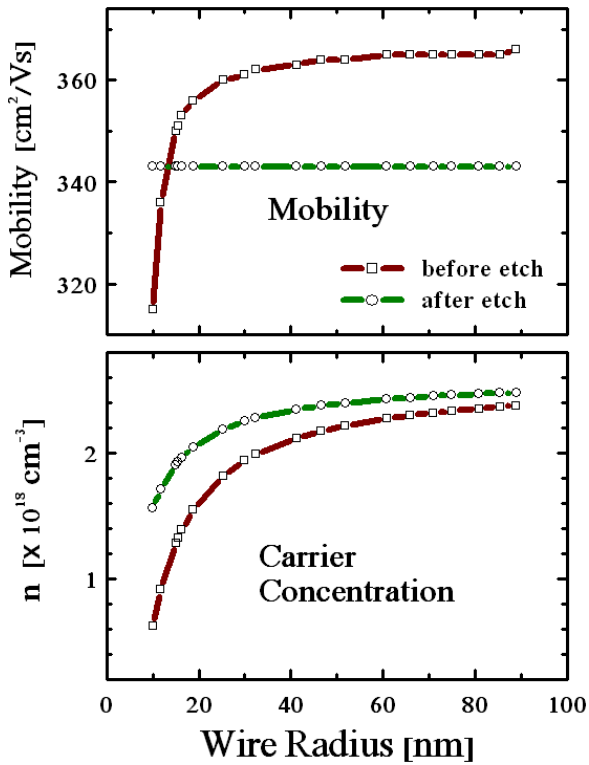


Figure 3. Carrier concentration (bottom panel) calculated using N_D and R_0 , and mobility (top panel), before and after etch, obtained dividing the carrier concentration by the fit of resistivity data.

a function of wire diameter (figure 2) and two photovoltage data items obtained from a multi-wire substrate, before and after a deliberate modification of the surface state density. As an output, it provides four average parameters: doping concentration, N_D , surface state density, N_T , surface band bending, ϕ_{BB} , and full depletion radius, R_0 . The latter three are obtained both before and after the surface modification. These parameters along with the fit to the resistivity data may then be used to obtain mean values of the carrier concentration and mobility as a function of wire radius (figure 3).

We deem our model successful if the results obtained relate reasonably to the range of reported values obtained using other methods. The as-grown surface state density we obtain, $N_T = 1.0 \times 10^{12}$, is within the range of $(1-4.4) \times 10^{12} \text{ cm}^{-2}$ reported for GaN films [17, 21]. Lee *et al* used chlorine treatment and observed an order of magnitude decrease in the GaN surface state density to a range between 2.1 and $3.5 \times 10^{11} \text{ cm}^{-2}$ [17]. They used an electrolytic etch, which is aggressive to GaN, compared with our plain HCl etch, which can only remove the surface oxide. As the effect in our case is milder, one may expect the surface state density to follow the same trend of decreasing upon etching, only to a lesser extent. In our experiment, the surface state density is roughly decreased by 50%, to $5.3 \times 10^{11} \text{ cm}^{-2}$. The drop in surface state density caused by the HCl etch is also reflected in a corresponding decrease of the surface band bending. The available information on band bending in the literature refers to surfaces of GaN films [13]. Since there are no previously reported values of band bending in GaN

nanowires, we have no adequate reference. On the other hand, we know that, in nanowires, the band bending ϕ_{BB} decreases with the wire radius R , as evident in equation (11), i.e. thin wires have a smaller band bending than films. In the sample used for photovoltage measurements, the average radius was 29 nm. As can be seen in figures 2 and 3, the effect of surface trapping at this radius is substantial. This means that the average surface band bending should be considerably smaller in nanowires than in films. We obtain an average band bending of 0.53 eV on our as-grown wires. This value is indeed smaller than values reported for GaN films (1 eV) [22]. As expected, this value decreases further upon etching (0.29 eV) with the decrease in surface state density. Hence, our method provides a new means for the evaluation of the two fundamental surface parameters affected by surface treatments in nanowires, surface state density and surface band bending, and their response to the surface treatment.

The carrier concentration in undoped GaN nanowires is typically at or near a level of degeneracy [23]. One of the explanations lays part of the blame on an artifact of the field effect model [24]. As the Hall effect is inapplicable geometrically to one-dimensional structures, the common method for measuring carrier concentration and mobility is using a nanowire channel field effect transistor. The main source of error is attributed to the modeling of the metal-oxide–nanowire capacitor, which assumes the oxide and the gate surround the nanowire channel, whereas in most cases the geometry is of a wire laid on a planar oxide and gate [24]. Another source of error is the field-induced charging and discharging of the nanowire surface state [25]. These systematic errors have been shown to yield an overestimate of the carrier concentrations and an underestimate of the mobility by up to an order of magnitude. Indeed, the value we obtain for the doping concentration $2.6 \times 10^{18} \text{ cm}^{-3}$ is almost an order of magnitude smaller than values we measured using back-gated nanowire channel field effect transistors ($1 \times 10^{19} \text{ cm}^{-3}$), while the average mobility (about $400 \text{ cm}^2/\text{Vs}$) is over an order of magnitude higher than the field effect mobility (about $20 \text{ cm}^2/\text{Vs}$).

The radius dependence of the calculated mean mobility (figure 3) shows a mild decrease with the wire radius (14% over the range studied). A decrease in field effect mobility has also been observed in GaN nanowires by Motayed *et al* [26]. Over the same size range, they observed a drop of 72% in mobility with size. The reason for this difference is likely surface roughness scattering, an inherent shortcoming of the field effect method. Surface roughness scattering affects field effect mobility as the increasing field forces charge carriers to flow very close to the surface. This mechanism is not relevant to any other type of transport except for that in the channel of a field effect transistor. Thus, it is also irrelevant to our experiment, especially because the electric field in the surface space charge region applies a centripetal force on the electrons, confining them to the wire center, keeping them away from the surface. Interestingly, our results show that after etching, along with the reduction in the surface state density, the size dependence of the mobility is practically gone. This observation points to a possible causal linkage between the

density of surface states and the size effect on the mobility in our nanowires, possibly remote charge scattering by charges trapped in the oxide [27].

We note that our method is limited to depleted surfaces. Although these constitute the majority of semiconductor surfaces, the method will not be adequate for semiconductors known to have surface accumulation, e.g. InAs, InN. Our model considers nanowires at room temperature with a uniform distribution of dopants. For low temperatures, incomplete dopant ionization will need to be considered. Cases of non-uniform dopant distribution will require a specific solution for the Poisson equation for each specific case. The use of surface photovoltage is also limited to surfaces that can be photosaturated. If photosaturation is not achievable, other methods should be used, e.g. x-ray or ultra-violet photoelectron spectrometry.

6. Conclusion

We proposed and tested a set of simple experiments and a model for electrical and surface characterization of semiconductor nanowires. Electrical properties of nanowire-structured semiconductors, as also shown here for GaN, are sensitive to the condition of their surfaces. Knowledge of this sensitivity is a prerequisite for any electronic or photonic utilization of nanowires. Our proposed method affords a tool for such evaluation of the effect of surface treatments on the surface state density and surface band bending as well as on the size dependence of carrier concentration and mobility.

Acknowledgments

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