Development of a Physical and Electronic Model for RuO2 Nanorod Rectenna Devices

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DEVELOPMENT OF A PHYSICAL AND ELECTRONIC MODEL FOR RUO$_2$ NANOROD RECTENNA DEVICES

A Thesis Presented

by

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of

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ABSTRACT

Ruthenium oxide (RuO$_2$) nanorods are an emergent technology in nanostructure devices. As the physical size of electronics approaches a critical lower limit, alternative solutions to further device miniaturization are currently under investigation. Thin-film nanorod growth is an interesting technology, being investigated for use in wireless communications, sensor systems, and alternative energy applications.

In this investigation, self-assembled RuO$_2$ nanorods are grown on a variety of substrates via a high density plasma, reactive sputtering process. Nanorods have been found to grow on substrates that form native oxide layers when exposed to air, namely silicon, aluminum, and titanium. Samples were analyzed with Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) techniques. Conductive Atomic Force Microscopy (C-AFM) measurements were performed on single nanorods to characterize structure and electrical conductivity. The C-AFM probe tip is placed on a single nanorod and I-V characteristics are measured, potentially exhibiting rectifying capabilities. An analysis of these results using fundamental semiconductor physics principles is presented. Experimental data for silicon substrates was most closely approximated by the Simmons model for direct electron tunneling, whereas that of aluminum substrates was well approximated by Fowler-Nordheim tunneling. The native oxide of titanium is regarded as a semiconductor rather than an insulator and its ability to function as a rectifier is not strong. An electronic model for these nanorods is described herein.
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CHAPTER 1: RuO$_2$ NANOROD RECTENNA DEVICES

1.1 INTRODUCTION

The ability to realize the potential advances that nanotechnology materials can give to the field of electronic devices first requires an understanding of the nanomaterial’s physical electronics. The growth of one dimensional metal oxide nanorod structures is an interesting technological development that may lead to further advances in electronic devices for wireless communications, sensor systems, and renewable energy resources. Significant nanorod research has previously been performed in the areas of wireless power transmission[1-3] and solar energy collection[4-7]. Nanorod research has also extended into more diverse applications, ranging from enhanced water electrolysis[8-11] to pharmaceutical sensor systems[12]. As technological demands exceed device size limitations, the need to develop nanostructures becomes ever more prevalent.

1.1.1 Nanorod Growth

Collective understanding of materials processing has vastly improved the ability to control nanorod material growth. Nanorod structures have been found to grow specifically on metal-oxide surfaces[13], a feature explored in a variety of metals[14]. Among the most widely investigated nanorod structures are carbon nanotubes and ZnO nanorods, which have favorable electrical and physical properties[15-21]. In 2002, W. I. Park et. al. demonstrated that individual ZnO nanorods could be well aligned, with uniform diameters, lengths, and densities when grown via metalorganic vapor-phase
epitaxy (MOVPE)[22]. Further studies by X. Wang et. al. in 2004 and O. Lupan et. al. in 2008 have demonstrated growth of self-assembled, hexagonally patterned, and aligned ZnO nanorods for hydrogen nano-sensors and nantenna arrays[23-24].

Though ZnO nanorods have important electrical and physical properties, RuO$_2$ nanorods have also attracted much attention due to their chemical and thermal properties. RuO$_2$ nanorods may be used for a variety of applications, ranging from enhanced electrocatalytic activity[25], to field-emission arrays for vacuum microwave power transmitters and flat-panel displays[26]. In 2012, M. Cross et. al. experimented with RuO$_2$ nanorods applied as a cathode coating to facilitate hydrogen production via electrolysis[8]. Cross et. al. hypothesized that the intrinsic nanorod structure, which establishes a high electric field in its vicinity, combined with the inherent electrocatalytic properties of RuO$_2$ and increased surface area, causes an increased production of hydrogen. It was found that in the electrolysis of water, hydrogen production with RuO$_2$ nanorod-coated cathodes was approximately 7% less efficient than that of a Pt cathode. Hydrogen production was found to be solely dependent on interfacial contact with the liquid, rather than the unique electric field properties of the electrode. The current investigation serves as a follow-up to Cross’s work in order to fully understand the physical and electrical attributes of RuO$_2$ nanorod devices.

In previous publications, RuO$_2$ nanorods have been found to preferentially grow on insulating surfaces[13]. As a result, RuO$_2$ is grown on Si, Al, and Ti substrates, as they respectively form SiO$_2$, Al$_2$O$_3$, and TiO$_2$ insulating native oxides when exposed to oxygen in the air ambient. Nanorod lengths were also found to be dependent on substrate
temperature during the growth process rather than deposition time, provided the reactor ambient contained 5% oxygen fraction[27].

### 1.1.2 Quantum Mechanical Effects

RuO$_2$ nanorods grown in this experiment were metal/insulator/metal (MIM) devices: they consist of two metals separated by an insulating oxide. Electron transport from one metal to the other through the insulator requires an understanding of quantum mechanical tunneling. In quantum tunneling, when a particle approaches a potential barrier that it cannot classically overcome, there is a small probability that it may be found on the other side of the barrier due to the Heisenberg Uncertainty principle. This tunneling effect is a direct result of the wave-particle duality of matter. A diagram of the phenomenon is shown in Figure 1.

![Quantum tunneling effect](image)

**Figure 1: Quantum tunneling effect.**

An electron, travelling as a wave, approaches a potential barrier of width $a$. When the electron comes into contact with the barrier, its two options are to be reflected
or to tunnel through the barrier. If the barrier is sufficiently thin, the electron may appear as a transmitted wave on the other side of the barrier. This transmitted wave has the same kinetic energy as the incident wave. This representation of quantum tunneling makes the assumption that there is only transmission and reflection; the barrier does not absorb energy. To determine the probability of electron tunneling through the potential barrier, solutions to the time independent Schrodinger equation are found, written as $\varphi_I$, the incident wave, and $\varphi_T$, the transmitted wave:

$$
\varphi_I = A e^{ik_1x} + B e^{-ik_1x} \\
\varphi_T = C e^{ik_1x} \\
\frac{\hbar^2 k_1^2}{2m} = E
$$

where $A$, $B$, and $C$ are coefficients of the solutions, $x$ is the horizontal position as described in Figure 1, and $k_1$ is the momentum vector and is thus related to electron kinetic energy $E$, particle mass $m$, and the modified Planck’s constant $\hbar$. The transmission coefficient, $T$, and reflection coefficient, $R$, can then be written as

$$
T + R = \left| \frac{C}{A} \right|^2 + \left| \frac{B}{A} \right|^2 = 1
$$

By solving the linear, algebraic, inhomogeneous equations for the coefficients, and rewriting in terms of $E$ and barrier potential $\Phi$, the transmission coefficient can be written as

$$
\frac{1}{T} = 1 + \frac{1}{4} \frac{\phi^2}{E(E - \phi)} \sin^2(2k_1a), \quad E > \phi
$$
There exist a number of models which attempt to describe the phenomenon of electron tunneling through a potential barrier, and the two that are often used in device physics are direct tunneling and Fowler-Nordheim tunneling. Direct electron tunneling, as its name implies, occurs when an electron passes directly from one metal to the other through the potential barrier of the insulator. This phenomenon is observed primarily in the case of transport through very thin insulating oxide layers. Fowler-Nordheim tunneling occurs in a similar manner, however instead of tunneling directly to the other metal, an electron tunnels to the conduction band of the insulator. Under an applied voltage bias, the electron is swept by the electric field into the second metal. Fowler-Nordheim tunneling is common for thicker insulating layers, where the probability of electron tunneling is diminished. A diagram of both tunneling regimes can be seen in Figure 2.

![Diagram of direct electron tunneling and Fowler-Nordheim tunneling.](image)

**Figure 2:** Diagram of direct electron tunneling and Fowler-Nordheim tunneling.
1.2 METHODOLOGY

1.2.1 Synthesis of RuO$_2$ Nanorods

RuO$_2$ nanorod materials were self-assembled using a high density plasma, reactive sputtering process on Si, Ti, and Al substrates. Specific process conditions of substrate temperature, gas composition, and sputter target power were used. These process conditions have been well described in prior publications by Cross et. al., and briefly summarized here[13][27-28]. A 1.5” diameter Ru metal sputter target was operated at a frequency of 13.56 MHz, with a power level of 50W. In conjunction, an electron cyclotron resonant (ECR) plasma was generated above the substrate assembly in order to increase plasma density in the process chamber to $\sim 10^{10}$ cm$^{-3}$. The reactor ambient was maintained by flowing 100 sccm of a 5/95% O$_2$/Ar gas mixture, throttled to a pressure of 15 mTorr. Substrate samples were radiatively heated to a temperature of 460°C. The deposited nanorods were characterized by x-ray diffraction analysis, and were determined to be single crystal, with no alignment with one another. An SEM and TEM image of a nanorod sample is presented in Figure 3. SEM images of RuO$_2$ nanorods grown on different substrates are shown in Figure 4.
Figure 3: SEM and TEM analysis of single crystal RuO₂ nanorods. (a) SEM image of unaligned nanorods. (b) TEM image of nanorod pyramid-shaped tip. (c) TEM diffraction pattern.

Figure 4: SEM images of RuO₂ nanorods grown on (a) SiO₂, (b) Al₂O₃, and (c) TiO₂.
1.2.2 C-AFM Probe Measurement

To electrically characterize the RuO\textsubscript{2} nanorods, a Conductive Atomic Force Microscopy (C-AFM) measurement was performed. C-AFM measurements consist of a metal-coated cantilever tip, touching the RuO\textsubscript{2} nanorod surface. A laser is directed at the back surface of the cantilever tip, and is deflected into a photodiode detector. As the cantilever tip moves across the surface of the material, the laser fluctuates and the photodiode captures the deflection of the laser. Post-processing of the laser fluctuations creates a micrometer-scale scan image of the material surface.

For this experiment, an Asylum Research Dual Gain ORCA-DG C-AFM probe was loaned to the University of Vermont Semiconductor Research Laboratory for use in this investigation. The measurements with this C-AFM probe were performed on RuO\textsubscript{2} nanorod films grown on Si, Al, and Ti substrates. A sample scan of RuO\textsubscript{2} on an Si substrate is shown in Figure 5.
Figure 5: AFM scan profile of RuO$_2$ on a Si substrate. (a) Surface image of device; singular nanorods can be seen at points 1 and 3. (b) Single line scan plot depicting surface height, in μm.
1.3 RESULTS AND DISCUSSION

1.3.1 I-V Characteristics for Si Substrates

In this investigation, the electrical characterization of a single nanorod was attempted. After creating a scan profile of the material surface, the C-AFM probe was carefully placed on a single RuO$_2$ nanorod grown on the Si substrate, and the following I-V characteristics were obtained and shown in Figure 6. Two I-V characteristics were acquired using different gain settings from the measuring instrumentation.

![Graph](image)

**Figure 6:** I-V characteristics for RuO$_2$ nanorod grown on Si substrates. Two different gain settings, (a) and (b), are shown.
In Figure 6a, rectifying behavior is clearly seen; the current measured at 2V is 1μA, whose magnitude is significantly larger than the current at -2V. In reverse bias, the rate of current flow is much less than in forward bias: the current measured at -2V was -100nA. The current saturation occurring after 2V is a result of measurement limitations, rather than a device characteristic.

Additionally, in Figure 6b, an alternative nanorod site was measured. The location of the measurement was chosen based on C-AFM surface scan images, which suggested the existence of nanorods in the area. In contrast to Figure 6a, I-V characteristics in Figure 6b appeared to be symmetric in forward and reverse bias, thus not rectifying. It is proposed that the C-AFM probe tip was placed on a cluster of nanorods rather than a single nanorod. Because of the imprecise nature of the measurement, the contact between the probe and nanorod was poor.

Amorphous SiO$_2$ is an insulator with a large band gap of approximately 9eV. In prior investigations, rectifying behavior of the nanorods was attributed to direct electron tunneling through the insulating oxide, which forms a metal/oxide/metal (MOM) structure on the substrate surface. In 1996, M. Hirose reported on an experiment in which electron tunneling was observed through varying thicknesses of SiO$_2$[29]. In this research, tunneling current was measured and plotted as a function of oxide thickness for SiO$_2$ ranging from 3nm to 6nm. Hirose fitted the I-V data with an equation for direct tunneling current modified by the Wentzel-Kramers-Brillouin (WKB) approximation, as well as an equation for Fowler-Nordheim tunneling. It was found that data for samples under 3.3nm were in agreement with I-V data generated by the direct tunneling model,
whereas samples greater than 3.3nm were best fit to the Fowler-Nordheim tunneling model.

In our investigation, the native oxide thickness of SiO$_2$ was assumed to be 1nm, which is well below the threshold for Fowler-Nordheim tunneling found in Hirose’s work. Properties for the oxide materials expected in this investigation are shown in Table 1 and the proposed energy band diagram for RuO$_2$ grown on Si substrates is shown in Figure 7.

**Table 1: Native oxide characteristics for various materials.**

<table>
<thead>
<tr>
<th>Material</th>
<th>Band Gap Energy (eV)</th>
<th>Electron Affinity (eV)</th>
<th>$T_{oz}$ (nm)</th>
<th>$\rho$ (Ω cm)</th>
</tr>
</thead>
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<tr>
<td>SiO$_2$</td>
<td>9</td>
<td>0.9</td>
<td>1</td>
<td>$1\times10^{15}$</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>6.4</td>
<td>2.58</td>
<td>5</td>
<td>$1\times10^{14}$</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>3.2</td>
<td>4.2</td>
<td>8</td>
<td>$1\times10^{10}$</td>
</tr>
</tbody>
</table>

**Figure 7: Electron energy band diagram for RuO$_2$: metal/oxide/semiconductor configuration on Si substrates.** Electron affinity is measured from vacuum level (0eV) down.


A direct electron tunneling model was used by Hirose to explain his experimental data. The model that he used was initially developed by J. Simmons[30], which approximates tunneling current as a function of applied voltage. The Simmons model begins with an expression for the electron tunneling probability, which is obtained with the WKB approximation. The WKB approximation in quantum mechanics is a calculation which approximates solutions to the time-independent Schrödinger equation. If the potential barrier, \( \Phi \), is affected by an applied voltage, \( V_{ox} \), and the kinetic energy of the particle \( E < \Phi \), the particle’s wave function has the generalized solution

\[
\psi(x) = Ae^{\pm \sqrt{2m(E - (\phi + \frac{V_{ox} x}{2}))/\hbar} i x}
\]

where \( \psi(x) \) is the wave function of the tunneling electron, \( A \) is a constant, \( x \) is the horizontal position of the particle (Figure 1), \( m \) is the mass of the particle, and \( \hbar \) is the modified Planck’s constant. If the potential barrier height is non-uniform and is a function of position along the x-axis, the average barrier height can be approximated as

\[
\phi = \frac{1}{T_{ox}} \int \phi(x) dx
\]

For the direct tunneling model, a uniform, rectangular potential barrier is assumed. The number of electrons tunneling in forward bias, \( n_1 \), is given by the density of states function, \( g(E) \), found by solving the Schrödinger equation for an electron trapped in an infinite potential well, and the Fermi-Dirac function, \( f(E) \), found by statistical thermodynamic methods:
where $q$ is the fundamental electron charge, $E_c$ is the conduction band energy, $E_F$ is the Fermi energy, $k$ is Boltzmann’s Constant, and $T$ is temperature in Kelvin. This process is repeated for the number of electrons in reverse bias, $n_2$, and the net sum, $n_1 - n_2$, is found. The current density, $J$, is then an integral over the maximum electrode energy $E_m$:

$$J = \int_0^{E_m} \psi \star (n_1 - n_2) dE$$

The expression for direct tunneling current, $J_{DT}$, can then be expanded as:

$$J_{DT} = \left( \frac{q^2}{2\pi \hbar T_{ox}^2} \right) \left( \phi - \frac{V_{ox}}{2} \right) \exp\left( -\frac{4\pi (2qm)^{1/2} T_{ox} (\phi - \frac{V_{ox}}{2})^2}{\hbar} \right) - \left( \frac{q^2}{2\pi \hbar T_{ox}^2} \right) \phi \star \exp\left( -\frac{4\pi (2qm)^{1/2} T_{ox} \phi^{1/2}}{\hbar} \right)$$

When the Simmons model is applied to the RuO$_2$ on Si physical arrangement, using $q = 1.602 \times 10^{-19}$C, $\hbar = 6.582 \times 10^{-16}$eV s, $m = 9.109 \times 10^{-31}$kg, $T_{ox} = 1 \times 10^9$K, and $\Phi = 3.12$eV, the resulting I-V curve is calculated and shown in Figure 8b. The experimentally measured I-V curve previously shown in Figure 6a is shown in Figure 8a for comparison.
Figure 8: Comparison between (a) experimental data for RuO$_2$ grown on Si substrates and (b) proposed model for direct current tunneling.

Comparing the experimental data and the I-V curve generated by the proposed direct current tunneling model, it is observed that the change in current for the experimental data in both forward and reverse bias is much more gradual than the abrupt exponential change observed in the proposed model. In forward bias, the current at 2V is 1$\mu$A for both the experimental and model data, however, in reverse bias, the model has a sharp change in current at -3V. This result is unexpected; an electron travelling from the RuO$_2$ nanorod to the Si substrate should see a larger potential barrier when tunneling through the insulating oxide. The difference in electron affinity for RuO$_2$ and Si is 0.85eV, which is not large enough to affect current flow in reverse bias. The experimental data does not exhibit this phenomena, which requires additional explanation in subsequent investigations. Barrier height is also a factor in tunneling probability, as determined in the equation for the transmission coefficient previously described. The relatively large barrier potential of SiO$_2$ (9eV) was evidently not enough to prevent the tunneling current through the device, most likely as a result of the extremely thin native
oxide. The model assumes that measurements are taken at low temperatures, and that there are no thermal contributions towards current, which may account for the differences between the model and the experimental data taken at room temperature. These results suggest that the predominant method of electron transport from the nanorod to the substrate material is direct tunneling through the thin SiO$_2$ layer.

1.3.2 I-V Characteristics for Al Substrates

I-V characteristics from the C-AFM probe measurement of a single RuO$_2$ nanorod grown on an Al substrate is shown in Figure 9.

![I-V Characteristic for RuO$_2$ grown on Al substrates](image)

**Figure 9: I-V characteristic for RuO$_2$ grown on Al substrates.**

The I-V characteristics for RuO$_2$ grown on the Al substrate suggest that the rectification capabilities of this device are very poor. The forward and reverse bias characteristics are almost symmetric; if there is any degree of rectification, it is minimal compared to nanorods grown on Si substrates.
With a band gap energy of approximately 6.4eV, Al$_2$O$_3$ is an insulating material with a band gap that is lower than that of SiO$_2$, which is 9eV. The 5nm thick layer of Al$_2$O$_3$ is considerably larger than the 1nm thick layer of SiO$_2$, making it difficult to determine whether it is the lower electronic barrier which encourages direct electron tunneling, or the thick oxide layer which encourages Fowler-Nordheim tunneling. In 2012, V. Di Lecce et al. investigated metal-oxide-semiconductor (MOS) barrier height extraction in nickel-Al$_2$O$_3$-GaN devices by examining the onset of Fowler-Nordheim tunneling [31]. They fabricated device structures with three different oxide thicknesses of Al$_2$O$_3$: 6nm, 12nm, and 18nm thick samples. It was found that the tunneling current for all three samples was a result of Fowler-Nordheim tunneling. The nickel-Al$_2$O$_3$ interface used in Di Lecce’s experiment is comparable to the RuO$_2$-Al$_2$O$_3$ interface used in this investigation because the electron affinity of RuO$_2$ (4.87eV) is similar to that of nickel (5eV). These results suggest that electron tunneling through the RuO$_2$-Al$_2$O$_3$ interface may also be a result of Fowler-Nordheim tunneling. The proposed electron energy band diagram for RuO$_2$ grown on Al is shown in Figure 10.
Figure 10: Electron energy band diagram for RuO$_2$ metal/oxide/metal configuration on Al substrates.

Similar to Di Lecce’s analysis of the Ni/Al$_2$O$_3$/GaN device, Fowler-Nordheim tunneling is used to model the experimental data. The expression for Fowler-Nordheim tunneling is obtained the same way as the Simmons model for direct electron tunneling, however instead of a uniform, rectangular barrier, the barrier is triangular and the potential varies with horizontal position. The expression for Fowler-Nordheim tunneling is given by

$$J_{FNT} = \left( \frac{q^3 V^2}{8\pi h\phi T_{ox}^2} \right) \exp \left( -\frac{8\pi (2m)^{1/2} \phi^{3/2} T_{ox}}{3hqV} \right)$$

where $J_{FNT}$ is the Fowler-Nordheim tunneling current. The model is applied to the results for RuO$_2$ on Al substrates, using $q = 1.602 \times 10^{-19}$ C, $h = 6.582 \times 10^{-16}$ eV s, $m = 9.109 \times 10^{-31}$ kg, $T_{ox} = 5 \times 10^{-9}$ m, and $\Phi = 1.48$ eV, a side by side comparison is shown in Figure 11.
The proposed Fowler-Nordheim tunneling model well predicts the experimental data in both forward and reverse bias. The measured current level of the experimental data at 0V is -2nA, whereas the current level of the model at 0V is 0nA. This small -2nA offset is a result of an error in instrumentation offset. In reverse bias, the measured current approaches a reverse breakdown voltage much later than the model data, however the rate of current change is greater. Although the proposed model suggests that Fowler-Nordheim tunneling is the predominant mechanism of electron transport through Al$_2$O$_3$, RuO$_2$ nanorods grown on Al substrates are poor rectifiers because they conduct current in both forward and reverse bias. This is a result of the relatively low potential barrier height and thick oxide layer compared to SiO$_2$.

1.3.3 I-V Characteristics for Ti Substrates

I-V characteristics from the C-AFM probe measurement of a single RuO$_2$ nanorod grown on the native oxide of Ti is shown in Figure 12.
Figure 12: I-V characteristic for RuO$_2$ nanorod grown on Ti substrates.

Rectifying characteristics are evident in the I-V characteristics for RuO$_2$ on Ti substrates, however the degree of rectification is much less than that of Si observed in Figure 6a. A forward bias voltage can be approximated at 1V, however from 1V to 2V, current increases from approximately 1nA to 3.5nA.

It has been proposed by J. Yang et. al. in his study of switching mechanisms for metal-oxide-metal nanodevices that rectification through a TiO$_2$ layer is dependent on oxide properties[32]. In this study, a rectification and switching mechanism was observed at a Pt/TiO$_2$ interface, with 50-nm-thick Pt nanowires grown on a 50-nm-thick TiO$_2$ insulator via nanoimprint lithography. Under negative bias, the device exhibited a rectifying characteristic, turning ON, and under a positive voltage bias, the device turned OFF. This behavior was explained by the drift of positively charged oxygen vacancies in TiO$_2$. An oxygen ion is negatively charged, so an absence of this ion creates a positive charge. When a negative voltage bias is applied to the metal, the influx of electrons
attracts these oxygen vacancies towards the metal-oxide interface, creating an electronic barrier. As a minimum threshold voltage is applied, electron conductive channels puncture this electronic barrier, resulting in exponential electron tunneling and rectifying I-V characteristics. The degree of current rectification was found to be dependent on the location, concentration, and distribution of oxygen vacancies in the oxide layer, as confirmed in experiments which intentionally formed oxide layers with oxygen deficiencies.

In other experiments, TiO$_2$ oxygen vacancies have been shown to act as $p$-type dopants, making the native oxide behave more like a lightly-doped semiconductor than an insulator[33-34]. This is supported by the fact that TiO$_2$ has a relatively small band gap energy of 3.2eV, which is below the 3.3eV bandgap of semiconductor ZnO. In traditional metal/semiconductor junctions, contacts are understood to be ohmic under heavy doping, and rectifying under light doping[35], therefore the rectification properties may be attributed to this phenomena. The energy band diagram for RuO$_2$ grown on Ti is shown in Figure 13.
Figure 13: Electron energy band diagram for RuO$_2$ metal/oxide/metal configuration on Ti substrates.
1.4 CONCLUSION

RuO$_2$ nanorods were grown on native oxides of Si, Ti, and Al to a length of 1\(\mu\)m via a high density plasma, reactive sputtering process. The resulting devices were analyzed by SEM and TEM imaging techniques, along with C-AFM measurements in order to characterize the electronic properties of a single nanorod. I-V characteristic plots are shown, and each device exhibits varying degrees of rectifying characteristics. In comparisons to the literature, it is suggested that direct current tunneling is the predominant mechanism of electron transport through the RuO$_2$-SiO$_2$-Si structure interface due to thin native oxide thickness, while the Fowler-Nordheim tunneling regime is suggested as the transport mechanism for current tunneling through the RuO$_2$-Al$_2$O$_3$-Al structure. Rectification for nanorods grown on Ti is a result of the small band gap of TiO$_2$, creating a quasi metal/semiconductor/metal junction. Energy band diagrams for each device are proposed. This research attempts to provide a theoretical foundation for phenomena observed in experimental findings.
CHAPTER 1 REFERENCES


CHAPTER 2: HYDROGEN DOPING OF GaSb THIN-FILMS ON Si

Effect of In-situ Hydrogen Doping on the Passivation of Acceptor Defects in the Heteroepitaxial Growth of GaSb Films on Si (100) and Si(111) Substrates


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Abstract

GaSb is a III-V semiconductor material that has been used to fabricate lasers, transistors, and thermophotovoltaic devices. This is a direct bandgap material with a minimum gap of 0.726 eV, making it an attractive semiconductor material for many optoelectronic applications. The carrier drift mobilities of bulk materials are relatively high (\( \mu_e \leq 3000 \) and \( \mu_p \leq 1000 \) cm\(^2\) V\(^{-1}\) s\(^{-1}\)), further making GaSb a viable candidate for many high speed electronic applications. The hole drift mobility is especially high relative to similar semiconductor materials, making it an attractive option for III-V CMOS device applications. When combined with the mature information processing capability found in silicon, thin film GaSb heteroepitaxially grown on Si becomes a technology worthy of further investigation.

The characterization of such a heteroepitaxial material system is performed herein; it was found that heteroepitaxial GaSb on Si electronic properties are a strong function of GaSb film thickness. When grown to a thickness of only 2100Å the material exhibited n-type mobilities of \(-749.37\) cm\(^2\) V\(^{-1}\) s\(^{-1}\), where as otherwise identically grown samples, 37800Å thick, exhibited p-type mobilities of up to \(348.2\) cm\(^2\) V\(^{-1}\) s\(^{-1}\). The interpretation of these results is discussed herein.

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2.1 INTRODUCTION

The demand for further advances in the broad area of high-speed electronics continues, especially with the inclusion of direct band gap semiconductor materials that can also contribute an optoelectronic capability.\textsuperscript{[1-3]} The optical band gap of GaSb produces light beneath the bandgap of Si, and therefore can be easily integrated on a Si substrate as a heteroepitaxially-grown semiconductor. Existing Si technology is at an advanced state of development in terms of data processing capabilities, and therefore presents advantages for the marriage of these two technologies. GaSb as a semiconductor material has attracted attention as a result of it’s high carrier mobilities, especially for that of holes that would be needed in the fabrication of a high speed CMOS technology. Directly combining the advantages of III-V semiconductor materials onto an existing Si substrate, such as in Read-Out Integrated Circuits (ROIC) and/or CMOS requires compatible materials processing, with special attention paid to high processing temperatures. The low temperature growth of heteroepitaxial GaSb thin film materials on a bare Si wafer substrate is reported herein.

2.1.1 Bulk GaSb and the Native Defect

Residual $p$-type conductivity has been observed in undoped bulk GaSb$^{[4,5]}$, and has been determined in a number of investigations to be attributed to the presence of native defects in the GaSb lattice$^{[6-10]}$. In 1966, Y.J. Van der Meulen investigated various characteristics and growth properties of bulk GaSb$^{[11]}$. Mobilities in bulk material were further investigated by Van der Meulen where 6N semiconductor grade gallium and
Antimony was used to synthesize bulk GaSb boules via Czochralski Growth, that were then zone refined by R.F heating at 750°C. Samples were grown in the [100], [110], and [111] directions and all exhibited $p$-type mobilities, ranging from 760 cm$^2$ V$^{-1}$ s$^{-1}$ to 840 cm$^2$ V$^{-1}$ s$^{-1}$.

The work suggested that three fundamental conclusions could be made about the native $p$-type conductivity inherent in GaSb: the unknown acceptor must be related to excess Ga or deficient Sb, the acceptor must be related to a vacancy, and it must be fairly immobile. The first conclusion was confirmed in a related experiment by F.J. Reid et. al., which demonstrated an increase in residual acceptor concentration in Ga rich, non-stoichiometric melts of GaSb, and a decrease in that of Sb rich melts\[12]. The second conclusion was confirmed in a study by R.D. Baxter et. al., where lithium was introduced into GaSb, replacing residual acceptor levels with a shallow acceptor level\[13]. The resulting formation of ion pairs between lithium and GaSb suggest that the native defect in GaSb is a double acceptor, i.e. a trapped state with a charge of $X^{-2}$. A widely accepted conclusion is that the $p$-type conductivity of GaSb is a result of an excess Ga atoms residing in a site-vacancy of Sb.

### 2.1.2 Native Defect Correction

Previous investigations have studied the possible passivation of the native defect for potential device applications\[14,15]. A.Y. Polyakov et. al. in 1992 reported on the passivation of shallow acceptors and donors in bulk GaSb using hydrogen and deuterium\[16]. Undoped GaSb was treated with both hydrogen and deuterium, in hopes of
passivating dangling bonds within the lattice. Samples doped with deuterium were examined via secondary ion mass spectrometry (SIMS) depth profiling. It was found that shallow acceptors, and to some extent shallow donors, were passivated by hydrogen treatment. Following an anneal in hydrogen at 150°C for one hour, the samples exhibited an increase in hole mobility, from 100 cm² V⁻¹ s⁻¹ to 180 cm² V⁻¹ s⁻¹. The increase in mobility results from the decrease in ion impurity scattering caused by the formation of the net neutral (H⁺-Accepto⁻) defect.

### 2.1.3 Thin Film GaSb

In addition to bulk materials, further studies have investigated the growth of thin film GaSb on a variety of substrates, namely Pascal, Dutta, Akahane, Nguyen and Zaixiang. In 1990, Pascal et. al. demonstrated the growth of undoped GaSb, both homoepitaxially on GaSb and heteroepitaxially on GaAs substrates, both using a metalorganic vapor phase epitaxy technique. It was found that the homoepitaxial thin film exhibited $p$-type conductivity, with a measured FWHM value of 0.0418° and a carrier mobility of 860 cm² V⁻¹ s⁻¹ at room temperature. The hole mobility in the heteroepitaxially grown GaSb was significantly reduced by stacking faults resulting from the lattice mismatch between GaAs substrates and the thin-film material.

In a 2004 study performed by T. Nguyen et. al., GaSb was grown on Si (100) substrates using a hybrid plasma-assisted molecular beam epitaxy (PA-MBE) technique. Samples were examined with high-resolution x-ray diffraction (HR-XRD) and Transmission Electron Microscopy (TEM) techniques. The TEM analysis of the (100)
samples showed various threading dislocations and stacking faults at the interface between the thin-film and substrate. In the first 400Å from the interface with the substrate surface, the thin-film material was riddled with defects caused by the lattice mismatch, while the remaining top 1000Å resembled that of bulk GaSb. Electrical properties of the heteroepitaxial material were significantly affected and the electrical properties of the material were dependent on thin film thickness.

In 2009, Zaixiang et. al. reported the growth of polycrystalline GaSb thin-films on amorphous substrates.\textsuperscript{[22]} Co-evaporation of 6N Ga and Sb on soda-lime glass substrates was performed at varying substrate temperatures ranging from 480°C to 560 °C. Samples grown to a thickness of 1μm exhibited a predominant (111) orientation and produced a measured Hall mobility of 130 cm\textsuperscript{2}V\textsuperscript{-1}s\textsuperscript{-1}. It was further proposed that the p-type behavior was caused by Ga anti-sites, where Ga acts as a double acceptor because Ga has two fewer valence electrons than Sb.

![Figure 14: Side image of thin-film/substrate interface.](image)

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2.2 EXPERIMENTAL METHOD

2.2.1 GaSb Thin Film Growth

The process used to grow the heteroepitaxial films of GaSb on Si(100) and Si(111) were similar to previous investigations performed in our lab and have been reported.\cite{21} The motivation for this new investigation was to study the effect of intentionally doping the GaSb thin film with $\text{H}_2$ and/or $\text{D}_2$, \textit{in-situ}, during film growth. The material was grown by a plasma assisted-sputtering process with an ECR plasma stream source and a thermal effusion cell to supply the Ga reactant. An Ar ambient was used to create the plasma for the Sb sputtering gun operation. Si substrates were cut into 2.6cm x 3.8cm pieces and cleaned via a series of chemical baths: acetone, methanol, and ultra high purity deionized water. An initial Sb layer was first deposited on the Si substrate at a substrate temperature of 420°C, then the Ga effusion shutter was opened and GaSb film was grown at 480°C for the desired time. At optimum conditions, samples were grown at a rate of 35Å/min.

2.2.2 Hall Measurements

Carrier mobility was measured with the four-point van der Pauw Hall technique using the protocol described by the National Institute of Standards and Technology\cite{23}. Measurements were performed in a custom designed probe station, containing four probes in a dark box connected to a switching box, Keithley 220 programmable current source, Keithley 195A digital multimeter, and 100x gain operational amplifier. The switching box facilitated making contact to the current sources and voltage measuring
probes. A 3,500 Gauss neodymium permanent magnet was used to maintain a magnetic field across the sample.

For the purpose of electrical characterization, a combination of Al and AuPd contacts were evaporated onto the four corners of each sample, creating an ohmic contact between external probes and the thin film material surface. Over the course of many measurements, the contacts became scratched. To enhance contact reliability, small indium dots were further melted onto each contact surface, facilitating probe connections.

The samples were placed on a non-conductive platform within the dark box. Four metal probes were manually attached to sample contacts using venire adjustment drives to correct for height and planar location. A variety of voltages are applied to probe contacts in opposing corners, and the resultant current and resistance were measured and calculated. When each of the possible probe combinations was recorded, probes were removed and the sample was then placed on the neodymium magnet. The same process was repeated twice: once for both polarities on the magnet. This data allowed for the calculation of sheet resistivity, hall concentration and hall mobility.
2.3 RESULTS

2.3.1 Thin Film Electrical and Materials Characterization

The samples in total have been characterized with a variety of techniques. HR-XRD results were used to determine crystalline quality via full width half maximum (FWHM) values. Rutherford Backscattering Spectroscopy (RBS) and Secondary Ion Mass Spectrometry (SIMS) were performed to obtain information concerning dopant concentration ($H_2$ and $D_2$) and crystalline quality of the thin film materials.

Characteristics such as thickness, mobility, FWHM values, and carrier concentration were calculated for each sample, and the results were averaged and shown in Table 2 and Table 3.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Run Type</th>
<th>Thickness(Å)</th>
<th>Mobility (cm$^2$/V$^{-1}$s$^{-1}$)</th>
<th>Concentration (cm$^{-3}$)</th>
<th>FWHM (deg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>128B</td>
<td>STD 1hr</td>
<td>2100</td>
<td>-436.00</td>
<td>-4.34E18</td>
<td>0.553</td>
</tr>
<tr>
<td>138B</td>
<td>STD 1hr</td>
<td>2100</td>
<td>-749.37</td>
<td>-1.54E18</td>
<td>0.534</td>
</tr>
<tr>
<td>124A</td>
<td>STD 1hr</td>
<td>2100</td>
<td>-392.33</td>
<td>-1.85E18</td>
<td>broad</td>
</tr>
<tr>
<td>052A</td>
<td>STD 2hr</td>
<td>4200</td>
<td>74.18</td>
<td>3.99E19</td>
<td>0.520</td>
</tr>
<tr>
<td>NTGaS959</td>
<td>STD 18hr</td>
<td>37800</td>
<td>348.2</td>
<td>5.49E18</td>
<td>-</td>
</tr>
<tr>
<td>127B</td>
<td>D2 1hr</td>
<td>2100</td>
<td>-816.10</td>
<td>-1.39E18</td>
<td>0.368</td>
</tr>
<tr>
<td>125A</td>
<td>D2 64min</td>
<td>2240</td>
<td>-1012.60</td>
<td>-8.01E17</td>
<td>0.524</td>
</tr>
</tbody>
</table>

Table 2: Electrical characteristics for GaSb (100). STD denotes a standard run with no deuterium added. D2 denotes a run with deuterium added.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Run Type</th>
<th>Thickness(Å)</th>
<th>Mobility (cm$^2$/V$^{-1}$s$^{-1}$)</th>
<th>Concentration (cm$^{-3}$)</th>
<th>FWHM (deg)</th>
</tr>
</thead>
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<tr>
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<td>2100</td>
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<td>0.520</td>
</tr>
<tr>
<td>124B</td>
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<td>2100</td>
<td>52.22</td>
<td>4.55E18</td>
<td>0.2418</td>
</tr>
<tr>
<td>128A</td>
<td>STD 1hr</td>
<td>2100</td>
<td>46.11</td>
<td>5.69E18</td>
<td>0.0777</td>
</tr>
<tr>
<td>NTGaS957</td>
<td>STD 18hr</td>
<td>37800</td>
<td>-60.00</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>127A</td>
<td>D2 1hr</td>
<td>2100</td>
<td>-148.73</td>
<td>-6.94E17</td>
<td>0.0686</td>
</tr>
<tr>
<td>125B</td>
<td>D2 64min</td>
<td>2240</td>
<td>-41.49</td>
<td>-2.06E18</td>
<td>0.0523</td>
</tr>
</tbody>
</table>

Table 3: Electrical characteristics for GaSb (111).
2.3.2 Discussion of Results

The electrical properties of heteroepitaxial GaSb thin film samples grown on Si(100) and Si(111) substrates will be discussed relative to the electrical properties of bulk GaSb crystalline material. In this discussion the samples grown on the Si (100) substrates will be discussed first. As can be seen in the cross-sectional TEM image shown in Figure 1, a large density of stacking faults exists near the interface of the GaSb thin film, and the Si(100) substrate. The conductivity for three samples grown to a thickness of about 2000Å all showed n-type behavior. The stacking faults at the interface are acting as n-type donors. Growing heteroepitaxial GaSb thin films to a greater thickness, e.g. ~4000Å, decreases the relative proportion of the film that has threading dislocations or n-type donors, and the film becomes slightly p-type. This was further confirmed by the observed behavior of a sample that was grown to a greater thickness of 37800Å. The mobility of this sample was p-type, determined by the larger proportion of the film whose conductivity is controlled by the native defects or Sb vacancies, similar to undoped bulk GaSb material.

The effect of doping the ~2000Å thick samples with H$_2$ or D$_2$, during film growth is twofold. The introduction of hydrogen activated by the plasma stream acts to passivate the dangling bonds in GaSb lattice near the Si substrate surface. This result is evident in the increase in the measured electron drift mobility for the samples grown to a thickness of only 2000Å. It is also proposed that the addition of hydrogen will passivate the native bulk-like acceptor defects in the GaSb lattice. In all cases the measured mobility amplitude was observed to increase. The passivation of the bulk-like defects can be
understood as the reduction in ion scattering caused by the formation of the net neutral (H+ donor, Acceptor-) complexes.

Similar to the growth of the 2100Å thick GaSb (100) samples, three GaSb (111) samples were grown to a similar thickness, and in some cases grown side-by-side with the (100) samples during the same run. In all cases the GaSb (111), 2100Å thick samples exhibited \( p \)-type mobilities, with an average magnitude of \( 44.8 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1} \). This is in stark contrast to that of the (100) samples, which were determined to be \( n \)-type as a result of stacking faults located near the substrate interface. The crystal quality of the 2100Å thick GaSb(111) samples were characterized by HR-XRD and RBS. The material quality was determined to be high and the RBS results showed that crystal quality improved with increasing film thickness from the Si substrate surface.

The GaSb(111) film grew off-axis to the Si (111) substrate, by an angle of 4.3° relative to the surface normal. The exact physical cause of this misalignment remains a curiosity, but it was very consistent and observed in all samples prepared. Continued growth proceeded in the 4.3° direction relative to the surface normal as opposed to growing at the plateau jog. This resulted in the formation thick parallel plates growing 4.3° relative to the surface normal. More significantly this resulted in a general decrease in crystal quality beyond a film thickness of 0.4 microns. These crystalline defects eventually acted as donor type defects for the material grown thicker than 4 microns. The measured carrier mobility shifted from \( p \)-type to \( n \)-type behavior for the thicker grown samples.
The purpose of this investigation was to determine the effect of in-situ doping with hydrogen during the growth of the heteroepitaxial GaSb thin film on a Si substrate. For thinner (111) samples grow at thicknesses less than 4000Å, the in-situ doping with hydrogen changed the majority carrier type from $p$ to $n$-type. It is proposed that the hydrogen passivates the acceptor type defects in the GaSb (111) material, likely caused by a Sb deficiency, into a net neutral (H+ donor, Acceptor-) complex.
2.4 CONCLUSION

In thin film heteroepitaxially grown GaSb on a Si substrate, net film majority carrier type is a function of the material’s film thickness. In the case of growth on a (100) oriented Si substrate, lattice mismatch between the thin film and Si substrate creates threading dislocations and stacking faults at the interface of the two materials. These defects act as donor impurities making the material act as $n$-type character. Growing the film thicker, the net majority carrier type transitions to $p$-type, this occurs because the conductivity of the film is governed increasingly by upper portion of the film, which resembles bulk-like GaSb material which is $p$-type. The conductivity of bulk-like material is controlled acceptor type native defects caused by a deficiency of Sb. Ultimately the deficiency of Sb is caused by the relatively higher vapor pressure of Sb relative to that of Ga, which during the vapor phase growth process results in the imbalance. For GaSb (100), the change from $n$-type to $p$-type material occurs between 2100Å and 4200Å, or between one and two hours of growth at the rate of 35Å/min.

The effect of the lattice defects near the interface is healed by in-situ doping the GaSb material film during film growth with hydrogen. When doped with hydrogen, the effect of the interface defects is diminished, increasing electron mobility in the material.

Heteroepitaxial growth of GaSb on (111) oriented Si substrate grows off-axis by 4.3° relative to the surface normal. The material close to the interface is of reasonably good crystal quality. This is due in part to the decoupled nature of the GaSb thin film material from the Si substrate lattice. The off-axis growth direction and a reduction in the typical stress-strain is experienced at an interface. The thin film material acts like a bulk
material and is controlled by its acceptor-like $p$-type impurity. Continued growth of the film results in the formation of stacking faults and dangling bonds which finds the material transitioning to an $n$-type character.

Doping the material during growth with hydrogen causes the thin film material with a thickness less than ~2000Å to grow as $n$-type material. The bulk-like acceptor defects are counter-doped by the hydrogen incorporation.

These results are important in the development of thin film GaSb on Si devices for optoelectronic and thermophotovoltaic applications.
CHAPTER 2 REFERENCES


COMPREHENSIVE REFERENCES


