Photoelectrochemical Etching of Gallium Nitride for Solar Applications

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ABSTRACT

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Solar panels are most efficient when they reflect the least light. This is often
accomplished with an antireflective surface. The goal of this project is to understand how various
3D nanostructures can be imparted on gallium nitride (GaN) surfaces by photoelectrochemical
etching to determine the least reflective surface. In this experiment, GaN was used because of its
durability and stability, both physically and chemically. Although types of silicon and gallium
arsenide semiconductors are often used, GaN is potentially more cost effective and less pollutive
for use in harsh climates including orbiting satellites. The surface structures of GaN wafers were
modified using a wet chemical etching process which included either 0.3 M nitric acid or tribasic
phosphate. The wafers were also simultaneously treated with both UV light and either 2.0 or 3.0
Volts. The reflectivity of the surface structure was dependent on both the etchant and the voltage.
The results show that nitric acid combined with 3.0 V produced the lowest reflectivity which was
was comparable to etched silicon. These results indicate GaN is a potential alternative material
for certain solar applications.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

INTRODUCTION

The goal of this project is to understand how various three dimensional nanostructures can be imparted on gallium nitride (GaN) surfaces by photoelectrochemical etching to determine the least reflective surface for solar panel applications. If GaN can be modified to demonstrate effective antireflective properties, it has the potential to be a useful material for solar applications in both harsh terrestrial climates and satellites.

Solar cells (often referred to as photovoltaic cells) work optimally when they have antireflective coatings and modified surface structures that allow them to convert greater amounts of light energy to usable electricity [1]. In general, unetched silicon has a reflectiveness of about 30%. That is, 30% of the light that hits the wafer is reflected off of it. In previous experiments, etched silicon has displayed a reflectiveness from 10-15% [2]. GaN shows promise as an alternative material to silicon for some applications because of its chemical stability and durability. GaN, however, has a higher band gap than silicon, and higher band gaps often lead to lower efficiency. A band gap is the amount of energy it takes for an electron to be excited into the conduction band, where it will conduct electricity. Therefore, GaN can only become relevant for solar panels if advances in its energy efficiency can be made. One way to increase the energy efficiency of GaN is to improve the quality of antireflective (AR) coatings on its surface.

In terrestrial applications, solar panels are often produced with silicon based compounds in large part because the electronics industry manufactures silicon products very effectively [1]. On satellite solar panels, manufacturers often use Gallium Arsenide (GaAs) panels because GaAs is durable and can withstand ionizing radiation in orbit [3]. However, upon reentry into

2

earth's atmosphere, the arsenic in the GaAs panels contaminates the surrounding area with arsenide particles [3]. The manufacturing process for GaAs panels also can contaminate the environment [4, 5]. NASA has noticed this problem and launched several initiatives to minimize or eliminate pollutants from satellites [6]. If GaN can achieve antireflective properties similar to silicon or GaAs, GaN could become an effective substitute which is cleaner than GaAs and more durable than silicon.

The surface modification process for a solar panel often includes surface texturing, the application of a separate antireflective coating, or a combination of the two processes. To change a surface texture, the semiconductor material is typically submerged into an etchant bath, and a reaction is stimulated in some manner. Each etching process removes some material and leaves behind a unique surface morphology. In the case of GaN, strips of the material come from a manufacturer with a surface layer that contains many partially filled pores. These bottlenecked pores both reflect light and inhibit conductivity. In the etching process, this unfavorable surface layer is removed and dissolved into ionic fluid.

This experiment focuses on the wet etching process to modify the surface of GaN. The wet etching process typically includes either electrochemical, photochemical, or photoelectrochemical means of removing the surface layer. In photoelectrochemical etching, low applied voltage, an intense UV light source and etchants are used to create a distinct surface morphology [7]. Each combination of these variables creates a separate three dimensional surface morphology with antireflective properties [7].

In earlier studies, different surface morphologies have been studied at the molecular level. In LED studies on GaN, many different nanostructures have antireflective properties [8],

3

but hexagonal pyramids are generally optimal compared to triangular or rectangular pyramids or a honeycomb shaped surface [9, 10]. In other studies, it has been shown that hexagonal pyramids also form antireflective surfaces on silicon [9]. Previous studies on GaN have not used voltage as a method of controlling the heights of the pyramids or focused on solar applications.

Different choices of etchant will also influence the surface structure of a semiconductor. These etchants can be acids, bases, or neutral. In LED studies, nitric acid has been shown to etch away point and line defects in single crystalline GaN [11]. Nitric and hydrofluoric acids are known to create optimal pores sizes [11, 12]. In general, a smaller pore size leads to greater conductivity. If a conductive polymer is added to the pores and the etched pore size is too small, recombination of the electron hole pairs occurs in the semiconductor, greatly reducing conductivity. This experiment will focus on nitric acid as hydrofluoric acid is highly dangerous.

This study will use trisodium phosphate as a base, which has not been heavily studied. In previous studies, the bases sodium hydroxide and potassium hydroxide have been shown to produce surface morphologies of hexagonal pyramids [13], which are an ideal antireflective surface [10]. In addition, Jung et. al. examined photoelectrochemical etching of GaN with both acids and bases. They found that both bases and phosphoric acid can create hexagonal pyramids independently of each other [14]. This current study builds upon Jung's work by using trisodium phosphate which is a base that has phosphate ions. Jung et. al. did not study antireflective properties; rather, they studied surface morphology for LED applications.

Figure 1 below, summarizes the specific photoelectrochemical process used in this experiment. The independent variables used to modify the GaN wafer include electric current

carrying either 2.0 V or 3.0 and a chemical etchant of either nitric acid or trisodium phosphate.

Each pairing of acid or base and voltage should produce a unique surface structure.

FIGURE 1: Photoelectrochemical Etching Summary

(Figure created by author)
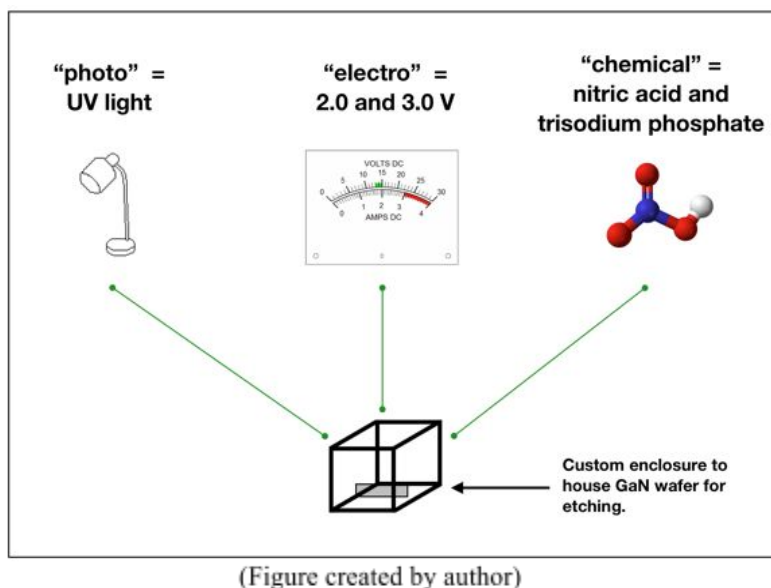
The hypothesis of this experiment is that one or more combinations of UV light, electrical current, and etchant will reduce the reflectivity of GaN to a level comparable to etched silicon.

MATERIALS AND METHODS

All material preparation, experimental procedures, and measurements were performed by the author under the mentor's supervision. The GaN wafers in this study had a silicon-doped GaN thickness of 2 microns with a doping level of $2x10^{19}$ Si atoms/cm3. This doped layer was grown on top of an unintentionally-doped GaN layer, and both GaN layers were grown by metalorganic chemical vapor deposition. The GaN wafers were purchased from a supplier and cut into strips 1.5 cm by 0.5 cm. The strips are then prepared by rinsing them in hot water and

sonicating them in a beaker filled with hexane to remove organic residue. Once the wafers were cleaned and dried, they were stored in concave containers for transportation and storage.

When preparing the wafers for etching, a piece of metallic tape was cut and placed on the front side of one end of each wafer. Using gloves and tweezers, an electrical wire was attached to the metallic tape. The individual wafers were then wrapped in acid resistant electrical tape. In order to determine that the electrical tape was acid resistant, a sample of electrical tape was weighed at 0.2876 g and was placed in an acid bath of 0.3 M nitric acid. The electrical tape was left to sit in the acid bath for two weeks. Afterwards, the electrical tape was removed from the bath and placed in an oven to dry. The electrical tape was then weighed again to determine if the acid effected it. The electrical tape weighed in at 0.2876 g and it was determined to be acid resistant.

A portion of each wafer was covered with electrical tape so that 0.25 cm^2 of each wafer was exposed to the etchant. The electrical tape was wrapped tightly to prevent the etchants in the petri dish from contacting the wire.

Canned air was sprayed on a petri dish, which would hold the wafers, to remove dust. The wafers were then taped down onto the right side of the petri dish, and a platinum wire counter electrode was taped to the left side of the petri dish. The counter electrode was used to conduct the current through the wafer and solution of water and etchant. The petri dish was then placed in a custom enclosure. The author constructed this enclosure from styrofoam and duct tape. All of the interior surfaces were completely black. The dimensions of the enclosure were approximately 41 cm by 61 cm by 41 cm. The enclosure was designed to prevent the wafers from being exposed to UV radiation until the lamp had been warmed up. The enclosure's design

6

included a removable barrier to prevent the UV light from illuminating the wafer prematurely (Figure 2). The petri dish was placed so that the portion of the GaN that was exposed was directly over the focal point of the UV lamp (model Spectroline SB-100P) to maximize UV exposure. The lamp was always in the same position and a fixed distance to the wafer. The platinum counter electrode and the wire attached to the wafer were hooked up to a voltage controller to apply the voltage. A 0.3 M solution of either trisodium phosphate or nitric acid was poured into the petri dish to a constant level marked in the petri dish. The UV lamp was given 5 minutes to warm up. All times were measured with a stopwatch. After 5 minutes, a positive voltage of either 2.0 V and 3.0 V was applied to the wafer with respect to the counter electrode, and the barrier in the enclosure in front of the lamp was removed so the UV light could illuminate the wafer. The wafers under each scenario were given 10 minutes to etch. During the duration of the etchings, the current was read with the voltage controller every two minutes.

FIGURE 2: Custom Enclosure and UV Lamp Illumination a GaN Wafer Within the Enclosure
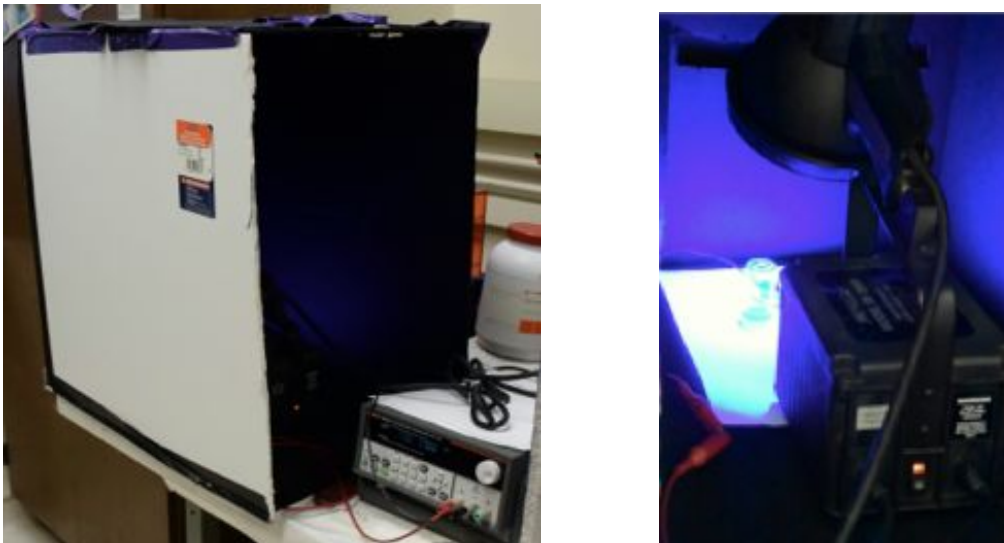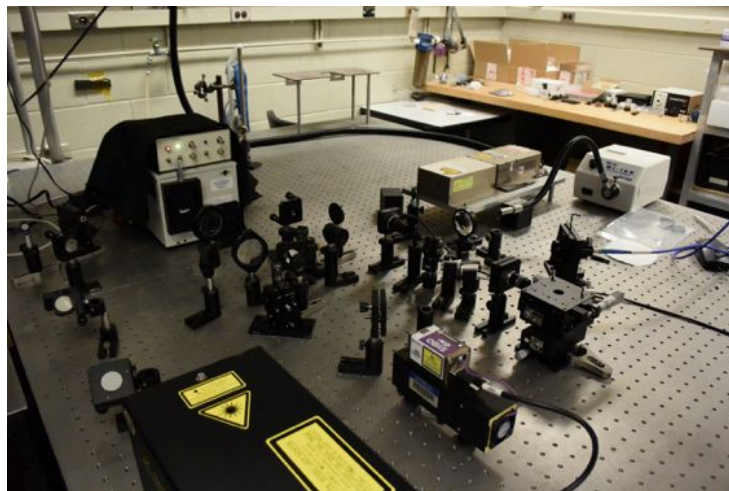

(Photos taken by the author)

After 10 minutes, the UV lamp and applied voltage were turned off. The two wires directly connected to the voltage controller were removed. The petri dish with the wafer and the counter electrode were removed and placed in a fume hood, and the etchant was disposed of into a waste container. The platinum counter electrode was removed first, and rinsed thoroughly with distilled water and left to dry. The electrical tape was then removed from the wafer. The wafer was washed in distilled water and then placed in a bath of deionized water for 3 minutes. The wafer was then dried and placed in a cylindrical vial, and kept for analysis.

An atomic force microscope was used to observe the surface morphology of the wafers. To measure the reflectivity the gallium nitride wafers were exposed to a 1.0 mW continuous laser. A handheld laser beam profiler was used to measure the intensity before the laser hit the sample (incident laser power) and as well as the intensity of the spectrally reflected light (reflected laser power). Three readings were taken for each sample.

In order to measure reflectivity, a laser was relayed through several mirrors and lenses, and light was directed onto the wafer (Figure 3).

FIGURE 3: Laser Setup

(Photo taken by the author)

8

RESULTS

The figure below (Figure 4) summarizes the spectral reflectiveness results for the five conditions in this experiment. For each of the conditions, three reflectiveness readings were taken and averaged. In order to calculate the average spectral reflectiveness, the average reflected laser power was divided by the average incident laser power:

Average spectral reflectiveness = Average reflected laser power * 100%
                                            Average incident laser power

FIGURE 4: Spectral Reflectiveness of Etched And Unetched GaN Wafers
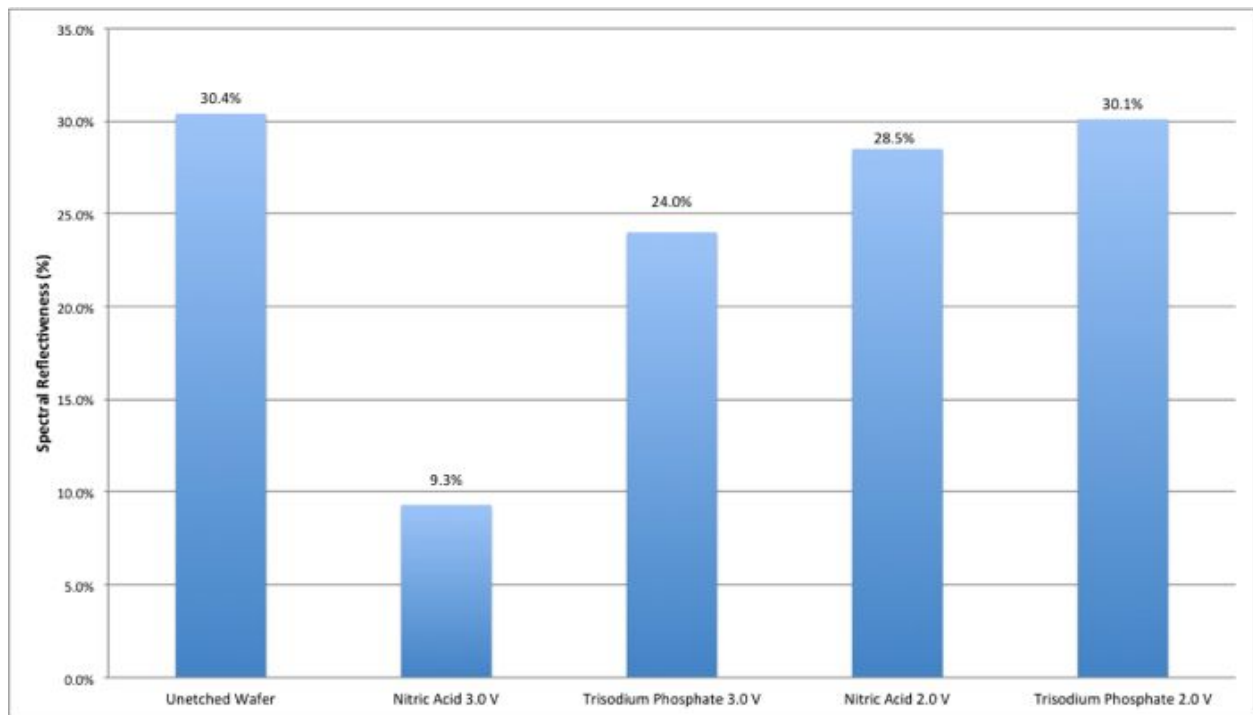
All conditions had less reflectivity than the unetched wafer. The unetched GaN had the highest reflectivity with an **average spectral reflectiveness of 30.4%** (Table 1). The wafer

samples etched with nitric acid at 3.0 V had the lowest average spectral reflectiveness at 9.31% (Table 2). The wafer etched with nitric acid at 3.0 V reflected 21.1% less light than the unetched GaN (Tables 1 and 2). Both the nitric acid and trisodium phosphate wafers etched with 3.0 V had lower reflectivity than the corresponding wafers etched with 2.0 V (Tables 2 and 3). The nitric acid 2.0 V condition had an **average spectral reflectiveness of 28.5% (Table 2)**. The trisodium phosphate 3.0 V wafer was somewhat less reflective than the unetched wafers, 24.0% compared with 30.4% (Tables 1 and 3), however, the trisodium phosphate 2.0 V was similar to the unetched wafer. Overall, nitric acid was more antireflective than trisodium phosphate, and 3.0 V was more antireflective than 2.0 V.

Table 1: Reflectivity of Wafers - Unetched GaN

	Reading 1	Reading 2	Reading 3	Average
Incident laser power	0.996 mW	1.03 mW	1.01 mW	1.01mW
Reflected laser power	0.310 mW	0.303 mW	0.307 mW	.307 mW
Average Spectral Reflectiveness				30.4%

Table 2: Reflectivity of Etched Wafers - Nitric Acid 2.0 and 3.0 Volts

Nitric Acid 2.0 V

	Reading 1	Reading 2	Reading 3	Average
Incident laser power	1.00 mW	1.03 mW	0.999 mW	1.01 mW
Reflected laser power	0.286 mW	0.293 mW	0.286 mW	288 mW
Average Spectral Reflectiveness				28.5%

Nitric Acid 3.0 V

	Reading 1	Reading 2	Reading 3	Average
Incident laser power	0.988 mW	1.01 mW	0.999 mW	.999 mW
Reflected laser power	0.0922 mW	0.0924 mW	0.0944 mW	.0930 mW
Average Spectral Reflectiveness				9.31%

Table 3: Reflectivity of Etched Wafers - Trisodium Phosphate 2.0 V and 3.0 V

Trisodium Phosphate 2.0 V

	Reading 1	Reading 2	Reading 3	Average
Incident laser power	1.01 mW	.998 mW	1.00 mW	1.00 mW
Reflected laser power	.299 mW	.301 mW	.302 mW	.301 mW
Average Spectral Reflectiveness				30.1%

Trisodium Phosphate 3.0 V

	Reading 1	Reading 2	Reading 3	Average
Incident laser power	1.02 mW	1.07 mW	0.989 mW	1.03 mW
Reflected laser power	0.249 mW	0.247 mW	0.245 mW	.247 mW
Average Spectral Reflectiveness				24.0%

The figure below (Figure 5) shows an image of a nitric acid 2.0 V wafer. The area on the left has been etched and although appears lighter in this picture, it reflects less light.

FIGURE 5: Etched Wafer Image
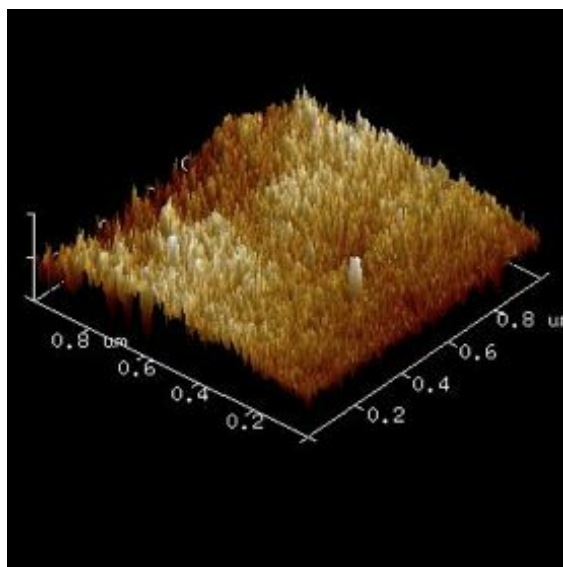

(Photo taken by the author)

The surface morphology of the etched wafers differed from the unetched wafer. The following figures are atomic force microscope photos of the two wafers etched at 3.0 V and a photo of the unetched GaN wafer for comparison. Figure 6 below illustrates how the nitric acid at 3.0 V condition had a rough, needle like surface morphology compared to the unetched wafer (Figure 8). While less needlelike than nitric acid 3.0 V, the tribasic phosphate 3.0 V condition in Figure 7 showed a relatively irregular surface with large differences in height compared to the unetched wafer which was almost completely flat. (Figure 8).

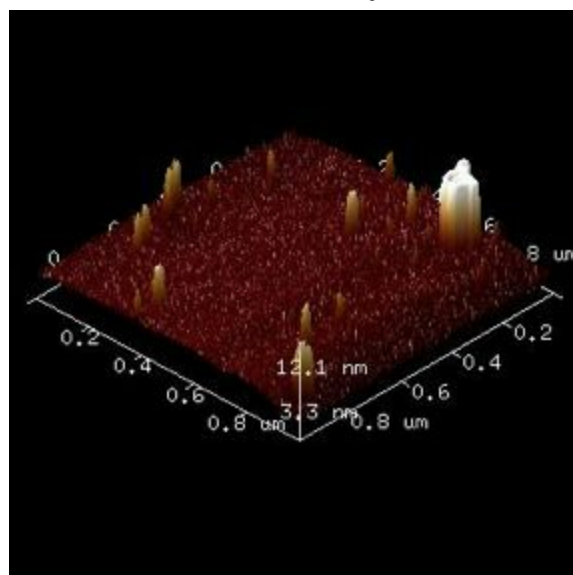FIGURE 6: Nitric Acid 3.0 V Condition as Observed by the Atomic Force Microscope

(Photo taken by the author/mentor)

12

FIGURE 7: Trisodium Phosphate 3.0 V Condition as Observed by the Atomic Force Microscope
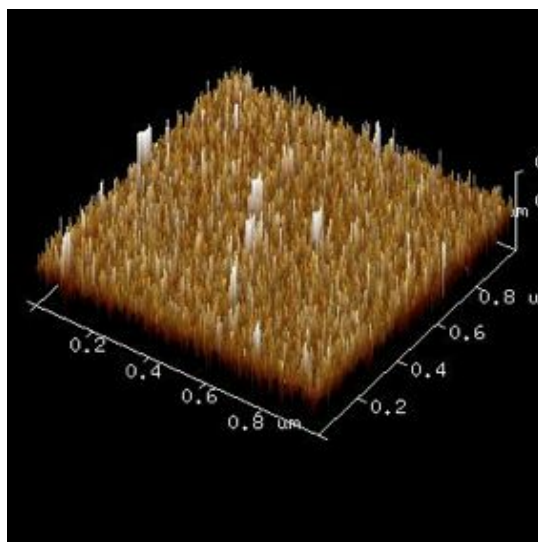
(Photo taken by the author/mentor)

FIGURE 8: Unetched Wafer as Observed by the Atomic Force Microscope

(Photo taken by the author/mentor)

DISCUSSION

This study used a wet etching process and modified GaN samples to determine potential techniques for improving the antireflective properties of a GaN wafer. Specifically, the conditions included nitric acid at 2.0 V and 3.0 V and trisodium phosphate at 2.0 V and 3.0 V. In all conditions, the study also used a consistent amount of UV light to modify the GaN surfaces. After the surface layer was modified, the wafers were studied for antireflective properties.

The condition which produced the most promising result for antireflectivity and proved the experiment's hypothesis was the nitric acid at 3.0 V condition. In this case, the resulting spectral reflectiveness was 9.31%. This is likely in part due to the ability of nitric acid to dissolve point and line disjunctions. The higher voltage level in this optimal condition contributed to increased roughness of the surface as seen in the atomic force microscope photos (Figures 6 and 8). This result is a considerable improvement compared to unmodified GaN (measured at 30.4%) and unmodified silicon (approximately 30%). In addition, this result is broadly consistent with etched silicon which has displayed reflectiveness between 10-15% in previous studies [6]. This experiment suggests that GaN has further potential for even lower levels of reflectivity as additional modifications are attempted.

When analyzing voltage as an independent variable, this experiment illustrated that reflectiveness was minimized for both materials in the 3.0 V condition. The decline in reflectivity for the nitric acid conditions when increasing voltage from 2.0 V to 3.0 V was 19.2% (a reduction from 28.5% to 9.31%). The decline in reflectivity in the trisodium phosphate condition by increasing voltage from 2.0 V to 3.0 V was 6.1% (a reduction from 30.1% to 24.0%). These declines in reflectivity corresponded to observations of increased roughness and

14

larger visual surface differences in the atomic force microscope photographs of the samples etched with 3.0 V versus those etched with 2.0 V (Figures 6, 7, and 8). For future experiments, it would be interesting to measure a broader range of voltages. A future experiment might consider increasing the upper limit from 3.0 V to 5.0 V. It would also be helpful to refine the changes in voltage to increments of 0.1 V or 0.2 V to identify more precisely the minimal reflectivity level. These two modifications could help make GaN even more potentially relevant for commercial applications.

In this experiment, nitric acid proved to be a more effective etchant for lowering reflectivity than trisodium phosphate. When trisodium phosphate was used at 2.0 V, the resulting reflectivity was 30.1%, and this constitutes only a minimal improvement over the unetched GaN wafer (30.4%). In the condition using trisodium phosphate and 3.0 V, the resulting reflectivity was 24.0%. This condition was somewhat better that the unetched GaN wafer or nitric acid etched with 2.0 V condition (28.5%) condition but not nearly as effective as nitric acid condition etched with 3.0 V (9.3%). Overall, nitric acid 3.0 V was much less reflective than trisodium phosphate at either voltage. It is possible that smaller voltage increments in future studies of trisodium phosphate would produce improved reflectivity results, but it appears that nitric acid is a superior etchant for reflectance.

GaN etched with trisodium phosphate was not as reflective as might have been expected. There are two potential explanations for this result. The first is that most studies on etching of GaN used strong bases like NaOH or KOH while a weak base Na_3PO_4 was used in this study. Thus, the trisodium phosphate did not completely dissociate in water leading to a lower concentration of hydroxide ions. There was a lower concentration of hydroxide ions in the

etching solution than what had been used in previous studies using strong bases. The second, is that in the chemical equation $Na_3PO_4 + 3\ H_2O = 3\ Na^+ + 3\ OH^- + H_3PO_4$, when trisodium phosphate dissociates, it is possible that the phosphate ions are interacting with and contributing to the etching of GaN. If so, then it is likely that the PO_4^{3-} is interfering or altering the etching process resulting in a different structure than would be apparent with hydroxide as the only present anion.

In the study of alternative solar panel materials, GaN has not been widely studied for solar or satellite applications. Much of the prior research on GaN has focused on its interactivity with conductive polymers and its optimization for doping levels in the context of LED applications. It is possible that GaN has been overlooked for solar applications because it is more of a specialty solar panel material, and many researchers focus on silicon products that can be manufactured relatively easily. GaN may have difficulty displacing silicon in high volume uses, but the applications in harsh weather environments and satellites remain relevant. Specifically, solar panels on satellites release arsenide when they burn upon reentry to earth's orbit. GaN may be highly useful in niche applications that require a particularly durable or environmentally friendly material such as solar satellite applications. For these types of special situations, GaN merits study as a solar material.

To date, since GaN has not been the focus of extensive solar research, there has been quite limited investigation into the reflectivity of GaN's surface and specifically into the use of reflectivity as the dependent variable. In addition, this experiment incorporates a few other unique approaches in the study of GaN as an antireflective material. First, trisodium phosphate has not been commonly used as an etchant for GaN in solar applications. This experiment

analyzed trisodium phosphate as an etchant because it could serve as a starting point for analyzing how pH affects the etching of GaN through the use of potential phosphate buffers. Further, when GaN was previously etched with trisodium phosphate it created hexagonal shapes on the surface which are often associated with antirelectiveness [10]. Second, this experiment uses the relatively uncommon photoelectrochemical etching process. Other etching experiments have focused on two factor electrochemical etching where each experimental condition is faster without adding UV light or two factor photochemical etching where the conditions are simpler to set up without electrical current. Although photoelectrochemical etching is somewhat slower, this method permits a broader choice of independent variables and offers many opportunities for future research.

CONCLUSION

The primary purpose of this experiment was to assess whether the surface structure of GaN could be modified in a photolelectochemical etching process to make GaN competitive with silicon in terms of its antireflective properties. If GaN is able to display comparable antireflectiveness, certain applications such as harsh weather and satellite solar panels may be able to take advantage of the superior durability and stability of GaN. In this experiment, the optimal condition for surface modification of a GaN wafer was nitric acid and 3.0 V. In this condition, the modified GaN wafer demonstrated a spectral reflectiveness of 9.31%. This result is significantly better than the unmodified GaN wafer which displayed reflectiveness of 30.1%. More importantly, the optimized GaN wafer compared favorably to modified silicon surfaces

which have demonstrated reflectiveness in the range of 10-15%. This suggests GaN has the

potential to be a new material for use in the solar industry for certain applications.

The reflectiveness measurements were averaged from three trials in each condition.

While a larger sample size would be preferable, the sample size was sufficient to support the

validity of the conclusions.

Based on the findings in this experiment, there are at least five possible opportunities to

expand the conclusions. Many of these extensions involve modifying the two independent

variables of voltage and etchant choice. An additional research opportunity focuses on taking the

results for reflectivity and expanding them into the important area of real world energy

conversion. First, it would be interesting to expand the range of voltages from 2.0 V to perhaps

5.0 V to help find the optimal voltage level for each etchant. Second, with additional time, the

voltage levels might be measured in increments of approximately 0.25 V to refine the results.

Third, additional trials might examine higher molarities of each etchant. It is possible that

trisodium phosphate and nitric acid could display greater antireflectivity at higher concentrations.

While this study used nitric acid at a molar concentration of 0.3 M, stronger concentrations

might produce even greater changes in surface morphology and better antireflective surfaces.

Fourth, additional etchants which also contain nitrate ions but are safer to manage might produce

similar results to nitric acid. The sample etchants with these properties are potassium nitrate and

sodium nitrate. Fifth, it would be useful to extend the antireflectivity results for the wafers in this

study to an experiment focused on actual energy conversion efficiency. This future extension

would continue the comparison of GaN to other materials in current use.

In summary, this experiment illustrated that photoelectrochemical etching can produce significant reductions in the reflectivity of the surface of GaN. As a result, GaN merits further study for solar and satellite applications where its durability and stability could prove useful.

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