# Investigation on the Effect of Current Density on Porous GaN Fabricated by UV-assisted Electrochemical Etching

N. S. M. Razali, A. F. A. Rahim, R. Radzali and A. Mahmood

*Abstract*—**This paper reported the anodization of porous GaN by UV-assisted electrochemical etching with a usage of lower power of UV light in an electrolyte that consist of 4% potassium hydroxide (KOH) with a variations of applied current density (20, 30 and 40 mA/cm². Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-Ray (EDX), Atomic Force Microscopy (AFM) and X-ray Diffraction (XRD) were used to characterize the morphological and structural characteristics while the Raman spectroscopy was used to identify the optical properties of the prepared porous GaN. Top view images from the FESEM demonstrated that the pore uniformity and porosity are affected significantly by the current density. The porous GaN sample etched with 40 mA/cm² produces a higher porosity and larger diameter pore structure compared to other porous GaN sample. This shows that the morphology and structural characteristic of porous GaN are increase with the increase of current density. The EDX result revealed there is no contamination occur, as only significant Ga and N atom presence in all samples. The AFM verified that the surface roughness and the pore depth are increased as current density increased. There were relatively large variations of the peak intensities for 2Theta-scan patterns as exposed by XRD. Raman intensity found to be enhanced with the increase in current density and among the porous GaN sample, the peak for sample fabricated with 30 mA/cm² and 40 mA/cm² was observed to be slightly shifted to lower frequency.**

*Keywords*—**Current Density, Electrochemical Etching, Porous GaN, Surface Morphology, Raman Spectroscopy**

## I. INTRODUCTION

SILICON (Si) based device was extensively used material in electronic and integrated circuit (IC) industry [1] due to low-cost and available in enormous size [2]. However, Si is indirect bandgap materials [1, 3, 4] that cause a poor optical properties [1, 5] at the visible spectrum and this may limits the efficiency as the light emitter for the optoelectronic devices [3].

Besides, porous semiconductors have been demonstrated to be capable of shifting the emission wavelength and enhancing the absorption and luminescence efficiency as compared to the unetched precursors [6]. Among porous semiconductors, porous silicon receives enormous attention and has been investigated most intensively. However, the instability of the physical properties has prevented it from large scale application [7]

Therefore, the Gallium Nitride (GaN) that falls under group of III-nitride materials have gained attention considering its properties to operate in the spectral region from the blue to near UV [6–8]. Besides that, GaN materials provide direct and wide band gap semiconductor [6–10]. Interestingly, the GaN material itself exhibit excellent carrier mobility, super chemical and thermal stability [13] and this enable the materials relevant for device applications in the high temperature and abrasive environment [11].

Despite of, GaN thin films normally grown on poor lattice and thermal mismatch foreign substrates which resulted in high residual stress and lead to high density of structural deficiency [9, 12–14]. This limitations of GaN semiconductor material can be minimized by proposing a porous structure on the surface as this structure were believed to provide larger surface-to-volume (S/V) ratio that enables more optical activities on the GaN surface [6, 7, 11, 15]. On top of everything, porous GaN structure offers shift of bandgap, luminescence intensity enhancement as well as efficient photoresponse as compared to bulk [6, 12, 15].

There were two types of technique available to create porous structure on material surface namely dry and wet etching. Dry etching technique could cause destruction to the semiconductor surface as it involves a very high kinetic energy atom being bombarded to the surface of the material [6–8, 16–18].On the other hand, wet etching used liquid chemicals during process to remove the exposed materials. This type of etching techniques offers high etching rate, minimise etching damage, easy to be implemented and simple to prepare [1–3, 15, 17–20].

Therefore, wet etching such as electrochemical technique is preferable than dry etching to fabricate porous structure on the GaN material. In addition, the electrochemical technique that commonly used rely strongly on the etching parameter such as

anodization time, current density, and illumination condition in order to form uniform and high porosity structure [21].

Previous work reported in [6, 12, 17, 21] succesfully produced a porous strucure on GaN by applying UV-assisted electrochemical etching technique with a very high power of UV light, up to 500W. In this situation, UV light becomes the dominant parameter affecting the etching process, thereby limiting the effect of applied current [19]. The illumination of UV light during the etching process help to excite electrons to generate holes and resulting a more uniform pores structure. This is because, UV illumination etching process consume two sources of energy (UV-light and etching voltage), while without the UV illumination, etching process energy source only depend on the applied etching voltage [11]. There were previous works that study the difference fabricating porous GaN and InGaN with UV and without UV illumination [11,22]. The work reported that porous structure with the illumination of UV produced uniformly larger pores and deeper cavities compared to that obtained without illuminated by UV. The aim of this work is to identify the suitable current density parameter with a usage of lower power of UV light (100W) by applying UV-assisted electrochemical etching technique to fabricate the porous structure on GaN material.

## II. EXPERIMENTAL PROCEDURE

The n-type GaN film grown on sapphire substrate were divided into smaller proportion approximately 1cm x 1cm sample to be fit inside Teflon cell. The GaN wafer sample needs to be cleaned by following the Aqua Regia cleaning procedure before undergoing the etching process.

The UV-assisted electrochemical etching technique were being applied in this work in order to create a porous GaN structure. The 1cm x 1cm GaN sample need to be wrap with the Aluminum (Al) foil before being install through the O-ring so that only the front surface is being exposed to the electrolyte solution. To ensure that there is no electrolyte leakage in the middle of the etching process, the metal plate is being clenched tightly by using two screws. In this project, the potassium hydroxide (KOH) solution were used as the electrolyte due to its great interaction with GaN [11] and it has low etching which allow easy control of the etching process. On top of that, KOH solution has been commonly used in the fabrication of uniform porous GaN [6,23]. KOH was chosen as the electrolyte etchant due to its low etching; this allowed easy control of etching process. The porous structure on the GaN sample was prepared with a different current density of 20, 30 and 40 mA/cm$^2$ for 60 min under the illumination of 100W UV light. The current density was varied from 20 to 40 mA/cm$^2$ as an improvement from work conducted on [9], as from that work, the effect of current density was studied from 5 to 20 mA/cm$^2$ only.

The structural and morphological analysis of the created porous GaN were characterized by the Field Emission Scanning Electron Microscopy (FESEM) (Model: Jeol JSM 7401F) and Atomic Force Microscopy (AFM) (Model: Dimension EDGE, BRUKER). The NanoScope Analysis software was used to evaluate roughness of the surface and the estimated pore depth with scan area of 5 x 5µm$^2$. All of the samples also go through

the Energy-dispersive X-ray spectroscopy (EDX) to determine the composition of each material lie on the porous GaN surface. High resolution XRD (Model:PANalytical X'pert Pro MRD) was used to assess the crystalline quality of the samples. To study the optical chracteristic of the sample, the Raman (Model:Renishaw InVia Qontor) was carried out. A Helium-Neon (He-Ne) laser (λ=633 nm) was used as an excitation source for Raman spectroscopy.
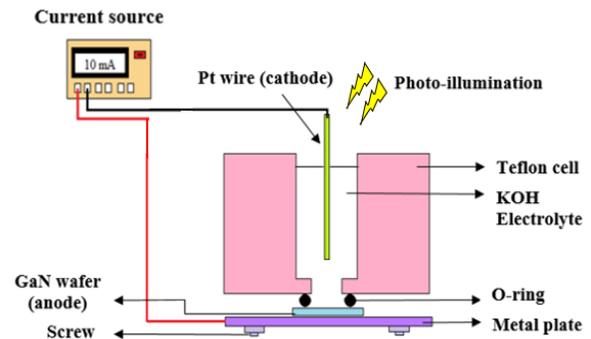


Fig. 1.Schematic diagram of UV-assisted electrochemical etching to create porous GaN structure.

## III. RESULTS AND DISCUSSIONS

### A. Morphological and Structural Properties

The top view image from FESEM and EDX analyses of as-grown GaN and all porous GaN samples subjected to varied current density were shown in Fig. 2. Fig. 2(a) proved that as-grown GaN built on sapphire display the smooth surface structure. For the sample etched with 20 mA/cm$^2$ (Fig. 2(b)), there is effect on the surface which produce a rougher surface. However, there is no exact pore formed on the GaN surface. This phenomenon shows that the applied current density of 20 mA/cm$^2$ is not sufficient to produce porous structure on GaN surface.

By increasing the current density to 30 mA/cm$^2$, as shown in Fig. 2(c), the hexagonal pore are starting to form on the GaN surface. Despite that, it were poor in density , uniformity and porosity, as the estimated porosity is only 6% with an average pore diameter of approximately 35 nm and pore size estimation range from 23 nm to 54 nm. There is an increment in pore formation distribution when the GaN sample was fabricated with the current density of 40 mA/cm$^2$. The GaN surface were moderately covered with hexagonal porous structure, causing increase in porosity to 12% with an average pore diameter of ~53 nm and pore size range from 23 nm to 104 nm. The similar hexagonal pore on GaN surface that fabricate with UV-assisted electrochemical etching were also found in [9]. Summarization of the average pore diameter, estimated porosity and pore size range value for all porous samples are tabulated in Table I.

From the result gained, this affirm that the applied current density plays an important role on pore formation on GaN surface. The most adequate reason for the variation of the pore formation is the reciprocal action between hole and electrolyte ion in the GaN surface during etching process occur [21]. When

the current applied is too low (20 mA/cm$^2$), there is not enough current to allow any hole at the GaN surface and cause minimal pore growth and longer time required to produce the pore. On the other hand, when the current applied were increased, the chances grow bigger for the interaction between hole and GaN surface enable more pore formed. The EDX result revealed significant Ga and N atom presence in all samples. This proved that all samples are not contaminated with other materials during electrochemical etching process.
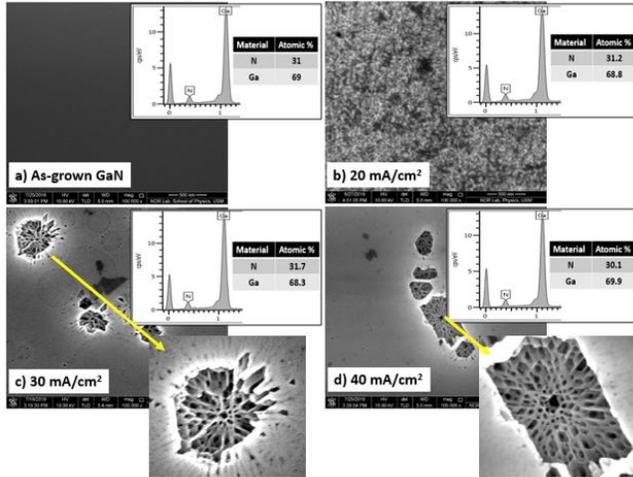
value and estimated pore depth for as-grown and all porous GaN sample. By taking into account for both results from the FESEM (Fig. 2) and AFM (Fig. 3) analyses, it can wrap up that sample anodized with high current density of 40 mA/cm$^2$ produced porous structure that high in porosity and deeper pore compare with other porous GaN sample. This phenomenon prove that current density plays an important part in forming a porous structure on GaN surfaces.
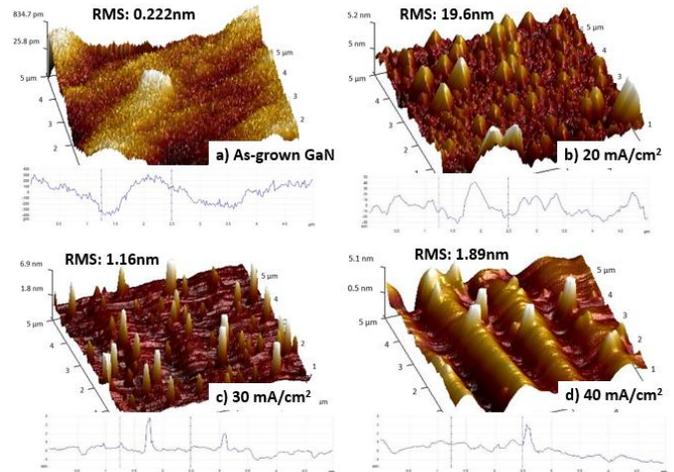


Fig. 2. SEM images and EDX of a) as-grown GaN and all PGaN samples fabricated with current density of b) 20 mA/cm$^2$, c) 30mA/cm$^2$ and d) 40mA/cm$^2$.



Fig. 3. 3D images and cross section line scan of a) as-grown GaN and all porous GaN samples fabricated with current density of b) 20 mA/cm$^2$, c) 30mA/cm$^2$ and d) 40mA/cm$^2$.

TABLE I
POROSITY, AVERAGE PORE DIAMETER AND PORE SIZE RANGE FOR POROUS GAN SAMPLE SUBJECTED TO VARIED CURRENT DENSITY

| Current Density (mA/cm$^2$) | Porosity (%) | Average Pore Diameter (nm) | Pore Size Range (nm) |
|---|---|---|---|
| 20 | - | - | - |
| 30 | 6 | 35 | 23 - 54 |
| 40 | 12 | 53 | 23 - 104 |

TABLE II
SURFACE ROUGHNESS IN RMS VALUE AND ESTIMATED PORE DEPTH FOR POROUS GAN SAMPLE SUBJECTED TO VARIED CURRENT DENSITY.

| Current Density (mA/cm$^2$) | Surface Roughness in RMS (nm) | Estimated Pore Depth (nm) |
|---|---|---|
| 20 | 19.6 | - |
| 30 | 1.16 | 0.83 |
| 40 | 1.89 | 1.53 |

The 3D AFM images of the sample prepared using UV-assisted electrochemical etching method with a varied applied current density were shown in Fig. 3. An increment of surface roughness were expected to occur after the GaN wafer being anodized. The as-grown GaN as shown in Fig. 3(a), display smooth surface with surface roughness Root Mean Square (RMS) value of 0.2nm . By comparing within PGaN sample, sample anodized with 20 mA/cm$^2$ presented highest RMS value of 19.6 nm. This may because of not enough current to allow any hole at the GaN surface, causing an electrolye interact solely with the sample and as a result, no pore structure form on the sample surface. Porous GaN steepness can be determine by the darkness of the surface morphology. Darker color of the surface means the steepness of the porous GaN is higher.

From the estimation analysis, sample fabricated with 40 mA/cm$^2$ shows highest steepness with pore depth of ~1.53 nm. This may because of not enough current to allow any hole at the GaN surface, causing an electrolye interact solely with the sample and as a result, no pore structure form on the sample surface. Table II summarize the surface roughness in RMS

Fig. 4 shows a 2Theta-scan patterns of as-grown GaN and all porous GaN structure prepared with UV-assisted electrochemical etching. From the graph, there is no peak shift occur between porous GaN sample. This indicate that the porosity has minimal effect on peak position shifting. On the other hand, it can clearly seen that there is large differences in term of intensity for all porous sample. The sample etched with current density of 20 mA/cm$^2$ has a highest intensity. This may be due by having rougher surface compare to other sample. As for the Full Width Half Maximum (FWHM), 30 mA/cm$^2$ sample have a lower value of 0.20 compare to 40 mA/cm$^2$ sample. Lower value of FWHM indicates better crystallinity of the sample [26]. The Debye-Scherer equation as in [20] was used to calculate the crystallite size of the sample and it reveals that there is difference between porous sample. The sample prepared with 30 mA/cm$^2$ current density have larger value of crystallite size compare to 40 mA/cm$^2$ as subjected to its smaller FWHM value. The summarization of peak position, FWHM value as well as calculated crystallite size of all porous GaN samples were tabulated in Table III.
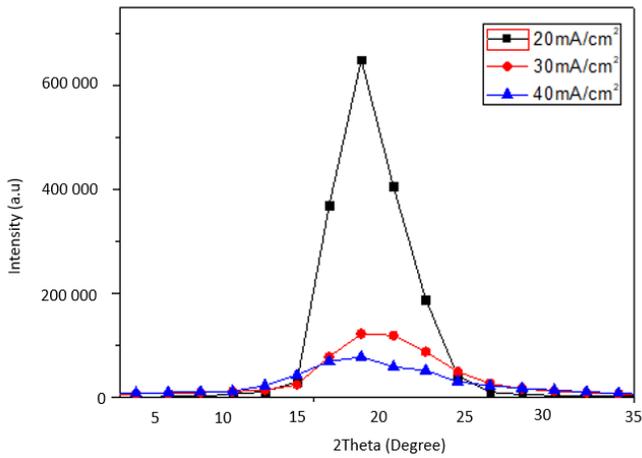
Fig. 4. 2Theta-scan patterns of sample etched by UV-assisted electrochemical etching subjected to varied current density.

TABLE III
PEAK POSITION, FWHM AND CRYSTALLITE SIZE FOR POROUS GaN SAMPLE SUBJECTED TO VARIED CURRENT DENSITY

| Current Density (mA/cm$^2$) | Peak Position (cm$^{-1}$) | FWHM (cm$^{-1}$) | Crystallite Size (nm) |
|---|---|---|---|
| 20 | 34.58 | 0.14 | 620.49 |
| 30 | 34.58 | 0.20 | 434.34 |
| 40 | 34.58 | 0.25 | 347.47 |

### B. Optical Properties

From Raman spectra of the porous GaN sample prepared with different applied current density as shown in Fig. 5, the Raman intensity was found to be different with the difference usage of current density. The 20 mA/cm$^2$ sample display an abnormal Raman spectra may be due by having no exact pore on the GaN surface sample. Therefore, by comparing 30 and 40 mA/cm$^2$ porous GaN sample, the 40 mA/cm$^2$ sample exhibited higher Raman intensity and lower FWHM. The smallest value of the FWHM represents good crystalline quality. This emphasize an improvement in the crystalline structure of porous GaN. The peak for sample prepared with 30 mA/cm$^2$ and 40 mA/cm$^2$ was observed to be slightly shifted to lower frequency. This showed that stress relaxation has occur in both samples, in which pore structure was found. From the Raman spectra analysis, it can be concise that the material optical properties could change due to presence of porous structure, as supported in [23] stating that higher density of pore contribute to stronger scattering effect. The peak position, FWHM and relative intensity are summarize in Table IV.

### IV. CONCLUSIONS

The porous structure was succesfully fabricated on GaN surface by implementing UV-assisted electrochemical etching technique with an application of lower power UV lamp. From the result gained, this etching technique produced a hexagonal pore shape but different in porosity and pore sizes corresponding to the variations of current density applied. This phenomenon indicate that the current density affected the porous formation on the GaN surface, as both porosity and pore diameter increased with the increase of current density. By correlating within porous GaN sample anodized with 30 and 40 mA/cm$^2$, sample prepared with current density of 40 mA/cm$^2$ exhibited higher RMS value, produced deeper pore, higher in porosity and present larger pore diameter. As for the optical characteristics, higher porosity of pore resulting in upgraded the optical behaviour of the material.
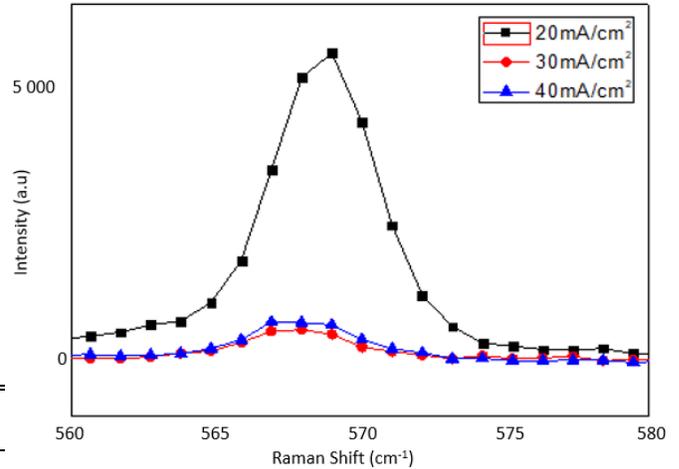


Fig. 5. Raman spectra of porous GaN sample etched by UV-assisted electrochemical etching subjected to variations of current density.

TABLE IV
PEAK POSITION, FWHM AND RELATIVE INTENSITY FOR POROUS GaN SAMPLE SUBJECTED TO VARIED CURRENT DENSITY

| Current Density (mA/cm$^2$) | Peak Position (cm$^{-1}$) | FWHM (cm$^{-1}$) | Relative Intensity |
|---|---|---|---|
| 20 | 569.01 | 6.27 | 0.11 |
| 30 | 518.66 | 8.42 | 0.88 |
| 40 | 566.92 | 7.32 | 0.89 |

### V. ACKNOWLEDGEMENT

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