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A SYSTEMATIC REVIEW ON POROUS GALLIUM NITRIDE UTILIZED PHOTOELECTROCHEMICAL ETCHING TECHNIQUE

Nur Iwani Nor Izaham¹, Ainorkhilah Mahmood^{2*}, Nur Maizatul Azra Mukhtar³, Rosfariza Radzali⁴,
Alhan Farhanah Abd Rahim⁴, A. Razak Abdul Wahab⁵, Faezah Jasman⁶

¹ Faculty of Applied Sciences, Universiti Teknologi MARA, Malaysia
Email: nuriwani1999@gmail.com

² Department of Applied Sciences, Universiti Teknologi MARA Cawangan Pulau Pinang, Malaysia
Email: ainorkhilah_sp@uitm.edu.my

³ Faculty of Health Sciences, Universiti Teknologi MARA Cawangan Pulau Pinang, Malaysia
Email: nurmaizatul038@uitm.edu.my

⁴ Electrical Engineering Studies, College of Engineering, Universiti Teknologi MARA, Cawangan Pulau Pinang, Malaysia
Email: rosfariza074@uitm.edu.my, alhan570@uitm.edu.my

⁵ AiGold Engineering, Malaysia
Email: ajaque7177@gmail.com

⁶ Applied College, Princess Norah Bint Abdulrahman University, Saudi Arabia
Email: fjjasman@pnu.edu.sa

* Corresponding Author

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Abstract:

Wide band gap semiconductor gallium nitride (GaN) has been extensively study in the past few decades due to its great potential for many applications, including optoelectronics, solar cells, and power electronics. Porous GaN is one of the most promising nanostructure improvements to enhance the optical properties as well as the sensing and detection capabilities of a device. One low-cost and harmless method to create nanostructured GaN is photoelectrochemical (PEC) etching. This paper presents a systematic literature review of the PEC etching technique's performance and process. There are 15 selected papers chosen from five electronic databases, including Web of Science, IEEE Xplore, Science Direct, ACS, and Emerald Insight. This paper refers to the Preferred Reporting Items for Systematic reviews and Meta-Analyses (PRISMA) guidelines. The results conclude the specification of the GaN material as well as the growth technique. The pre, during, and post process of PEC has also been discussed in this paper. As a way to find out the performance of PEC etching, the morphological, structural, and optical properties of porous GaN have been analyzed in this study. This paper can be used as a reference for PEC etching of GaN in the future.

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Electrochemical, GaN, Nanostructured, Porous, Review, Systematic

Introduction

Over the past few decades, gallium nitride (GaN) has been thoroughly explored as a wide band gap material for semiconductor applications (J. He et al., 2021; Islam et al., 2022; Liu et al., 2024; Pearton, Abernathy, et al., 2004; Su et al., 2013; Z. Wang et al., 2021; Yan et al., 2024). GaN is a member of the nitride family that has a band gap of 3.44 eV at room temperature and a wurtzite crystalline structure. Due to these characteristics, GaN can be used to detect objects in the visible blind range without the use of any expensive optical filters. It also has the potential to operate in both photovoltaic and photoconductive modes (Aggarwal & Gupta, 2020; Carbone et al., 2019; Fukamizu et al., 2023; Levine et al., 2020; Pearton, 2021). GaN semiconductors have established themselves in a variety of applications, such as ozone detection, flame detection, space communication, and biological sensors (Hirano et al., 2001; Lu et al., 2013; Najda et al., 2022; Novikova et al., 2021; Pearton, Kang, et al., 2004; C. Y. Wang et al., 2007; Yuk et al., 2017). GaN material carries detector properties, such as high breakdown voltages, high carrier mobility, inertness, and resistance to radiation and chemicals at high temperatures.

Additional surface modification is necessary for the integration of GaN materials into devices that are suitable for sensing, optoelectronics, catalysis, and other applications. High quality porous semiconductors are needed to improve the performance of GaN. Dry etching and wet etching methods are often used to create nanostructured GaN (Lim et al., 2018; Meyers et al., 2020; C. Wang et al., 2018; Z. Zhang et al., 2022). Dry etching methods, such as Reactive Ion Etching (RIE) and Inductively Coupled Plasma Reactive Ion Etching (ICP-RIE) might result in surface damages and they lack the desired selectivity for morphology, dopant, and composition (Meyers et al., 2020). Photo assisted electroless, direct current photoelectrochemical (DCPEC), and alternating current photoelectrochemical (ACPEC) are some of the wet etching techniques that can also produce porous formations (Daud et al., 2023; Iwani et al., 2024; Lim et al., 2018; Mahmood et al., 2016; Mahmood, Ahmed, Tiginyanu, et al., 2013; Mahmood, Hassan, et al., 2013; Meyers et al., 2020; Quah et al., 2016; Syuhadah et al., 2020; Z. Zhang et al., 2022). Figures 1 (a–d) illustrate porous GaN formations based on three wet etching techniques compared to the as grown GaN. Electroless etching, or also known as electrochemical oxidation without external bias is a straightforward, affordable, and often used method on both conductive and insulating substrates (Díaz et al., 2002; Li et al., 2002; Mahmood et al., 2016). DCPEC is a conventional method that uses direct current source in a PEC system. However, DCPEC lacks uniformity and etching speed due to the formation of nitrogen bubbles at the semiconductor electrolyte interface (SEI) (Mahmood, Ahmed, Tiginyanu, et al., 2013; Syuhadah et al., 2024). Thus, a sine wave ACPEC etching has been introduced to overcome this deficiency (Mahmood, Ahmed, Yusof, et al., 2013).

The photoelectrochemical (PEC) etching technique stands out among other wet etching methods due to its unique dependence on both illumination and electric fields, which

significantly enhance the etching process and enable controlled nanostructuring of materials (Y. He et al., 2019; Z. Zhang et al., 2021).

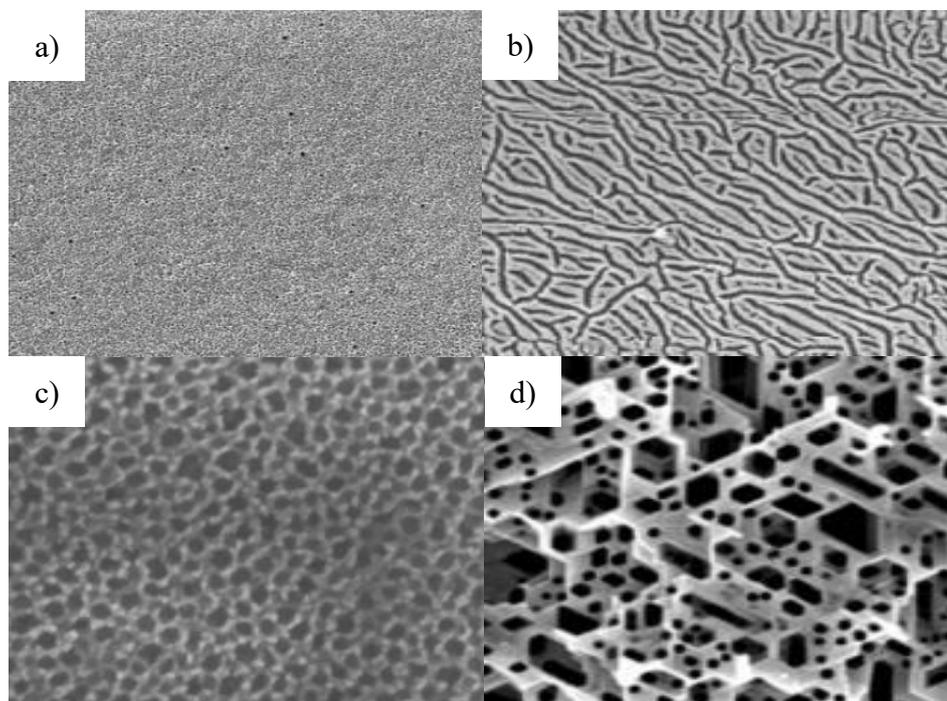


Figure 1: Porous GaN Formation Based On Different PEC Techniques: (a) As Grown GaN (Radzali et al., 2014); (b) Porous GaN Formation Via Electroless Etching (Mahmood et al., 2016); (c) Porous GaN Formation Via DCPEC (Z. Zhang et al., 2021); And (d) Porous GaN Formation Via ACPEC (Mahmood et al., 2013)

However, despite its potential, several issues remain in the field of PEC etching of GaN. These include inconsistencies in porosity uniformity, lack of reproducibility across different studies, variations in etching parameters, and limited understanding of the relationship between etching conditions and the resulting structural, optical, and electronic properties. Moreover, a wide range of experimental conditions and techniques have been reported in the literature, making it challenging to compare and evaluate their relative effectiveness. In light of these challenges, this study aims to conduct a Systematic Literature Review (SLR) focusing on the performance of PEC etching for GaN porosification. The scope of the study covers peer-reviewed publications from 2014 to 2023 that specifically investigate the PEC etching of GaN, including its growth techniques, etching conditions, and characterization methods. The objective of this study is to categorize and analyze existing research in three main areas: (1) the growth techniques and material properties of GaN, (2) the PEC etching processes and influencing parameters, and (3) the morphological, structural, and optical characteristics of porous GaN. By synthesizing these findings, the study seeks to identify trends, highlight research gaps, and provide a clearer understanding of the capabilities and limitations of PEC etching for nanostructured GaN application.

Protocol and Methodology

The aim of this SLR was to evaluate the productivity and performance of PEC etching methods on GaN nanostructures to produce porous GaN. This study has referred to the Preferred Reporting Items for Systematic Reviews and Meta-Analyses (PRISMA) guidelines (Page et al., 2021).

Eligibility Criteria

This study has several inclusion and exclusion criteria for extracting articles and research articles. The excluded documents were proceeding papers, review papers, books, etc. Articles published in languages other than English were excluded, since the language for this analysis was set on only English. The publication dates ranged from 1 January 2014 to 28 August 2023. Publications prior to 1 January 2014 were also excluded.

Information Sources

This systematic literature review (SLR) was conducted by accessing five databases, namely, the IEEE Explore, Science Direct, Emerald Insight, Web of Science (WoS), and ACS Publication. These databases were accessed through the Universiti Teknologi MARA (UiTM) Library E-Resources, which are only available to UiTM staff and students. Each source was last searched and consulted on 28 August 2023 in Universiti Teknologi Mara Pulau Pinang.

Source Strategy

Each database uses a different type of searching algorithm due to each website having different user interfaces. However, this study used the same keywords for all databases, which were (“GaN” OR “Gallium Nitride”) AND (“PEC Etching” OR “Photo-electrochemical Etching” OR “Photo Assisted Electrochemical Etching”). Details of the article search are illustrated in Figure 2, including the filter and limitations based on the setup criteria.

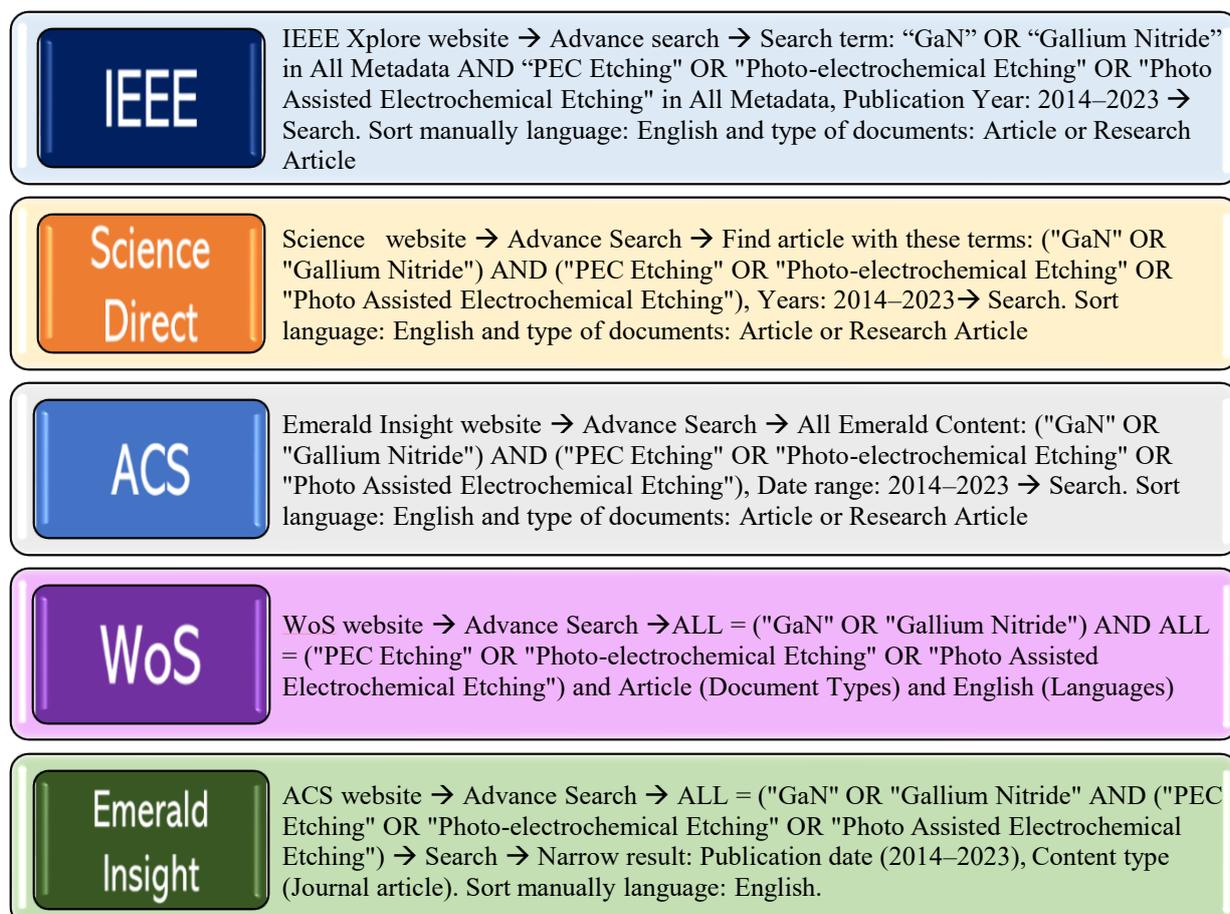


Figure 2: Details Of Article Collection From Database Sources

Selection Process

The selection process is divided into four categories, namely, article collection, duplication screening, title and abstract screening, and full text reading, according to the PRISMA flow diagram, as illustrated in Figure 3. A total of 442 articles (n) were found in the following databases: IEEE Xplore, n = 30; Science Direct, n = 148; Emerald Insight, n = 3; Web of Science, n = 212; and ACS Publication, n = 49. Duplication screening was performed manually using the Microsoft Excel 2016 software, since duplicated articles were observed. A total of 12 articles were found to be duplicated and later removed. The duplication came from two large databases with numerous publications, which were Science Direct and WoS. Finally, 430 articles were eligible for the filtration and criteria screening process.

Title and abstract screenings were also performed and each article was roughly scanned, since some researchers did not explain their methodology in detail, which made it difficult to review the results. Some articles were also found to be unrelated to the topic being studied (n = 236). Once the abstracts and articles have been manually scanned, 15 articles were found to be suitable for a full reading and were subsequently used in this study.

Data Collection Process and Data Items

Data were gathered from the methodology and results/discussion sections of each article. Excel was used to analyse and organise the collected data, which were then displayed in each subcategory as a table or graphic. Each category was discussed using the generated table or visuals, followed by a summary, critical remarks, and descriptions. These data extractions were chosen for a specific reason, whilst the data must reflect the goal of this study for future reference. The conclusions and findings that were particularly relevant to the current study question were examined.

Discussion

This section, as divided into three subsections will describe the components and performance of PEC etching of GaN. The subtopic are growth technique and properties of GaN, PEC etching of GaN and PEC characterisation of GaN.

Growth Technique and Properties of GaN

Growth technique and properties of GaN are critical factors because every detail of the material can influence the PEC process and outcome. The GaN type, growth technique, carrier concentration, thickness, and substrate are five crucial details in growth and materials. As shown in Table 1, certain information is unavailable because the authors did not provide them in the selected publications.

The SLR has shown that many researchers used n-GaN and p-GaN, although some of them did not specify the type of doping they used for these types of GaN. Unintentionally doped (UID) GaN and Si-doped GaN are typically used for the n-type GaN, and magnesium-doped GaN is used for the p-type GaN. According to Table 1, n-type GaN is frequently used in PEC etching (Cheah et al., 2015; Heffernan et al., 2020; Hou et al., 2018; Kumazaki et al., 2014; Lee et al., 2017; Matsumoto et al., 2018; Meyers et al., 2020; Radzali et al., 2014; Toguchi et al., 2019; C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2021, 2022). Despite the difficulties of producing pores on p-type GaN thin films, two studies on PEC of the p-type GaN have been published (Lim et al., 2018; Quah et al., 2016). Unlike silicon that can be produced from sand, gallium nitride needs to be grown using specific chemicals and temperatures, which

will appear as a thin film. Numerous techniques are available for GaN growth, including Hydride Vapour-Phase Epitaxy (HVPE), Metal-Organic Chemical Vapor Deposition (MOCVD), Plasma Assisted Molecular Beam Epitaxy (PAMBE), Plasma-Enhanced Chemical Vapor Deposition (PECVD), Laser Molecular Beam Epitaxy (LMBE), Chemical Vapor Deposition (CVD), Atmospheric-Pressure Chemical Vapor Deposition (APCVD), and Pulsed Laser Deposition (PLD) (Al-Zuhairi et al., 2022).

Figure 3: PRISMA Flow Diagram

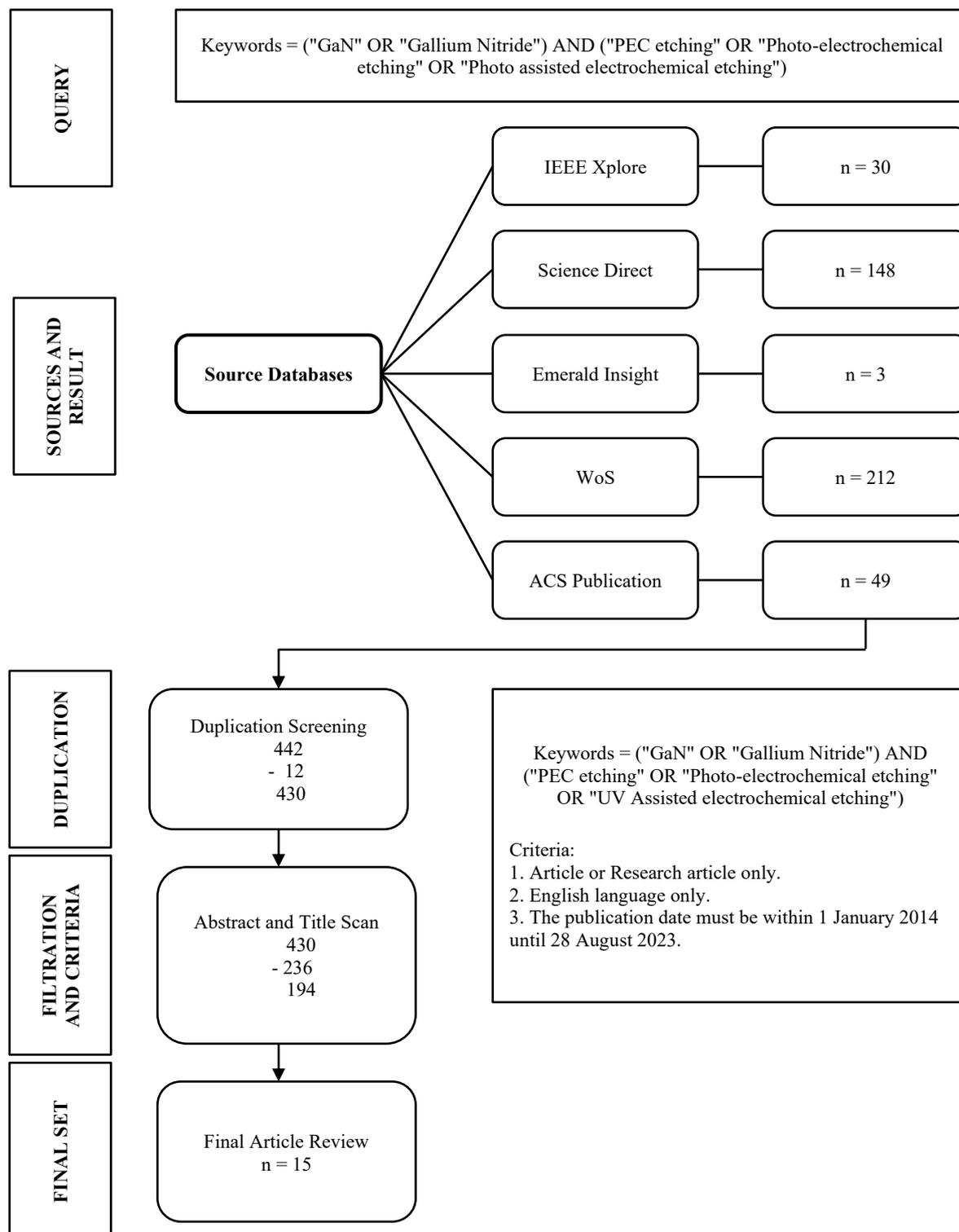


Table 1: Summary Of Growth Techniques And Properties Of GaN

GaN Type		Growth Technique			Carrier Concentration (cm ⁻³)				Thickness (µm)		Substrate		Ref.
n-type	p-type	MOCVD	HVPE	MOVPE	10 ¹⁶	10 ¹⁷	10 ¹⁸	10 ¹⁹	0.1–3.0	3.1–6.0	GaN	Sapphire	
✓			✓							✓		✓	(C. Wang et al., 2018)
✓						✓	✓			✓		✓	(Cheah et al., 2015)
✓	✓		✓	✓					✓				(Meyers et al., 2020)
✓		✓					✓		✓			✓	(Heffernan et al., 2020)
✓			✓				✓			✓		✓	(M. R. Zhang et al., 2017)
✓			✓				✓			✓		✓	(Z. Zhang et al., 2022)
✓			✓				✓			✓		✓	(Z. Zhang et al., 2021)
✓		✓					✓	✓	✓		✓		(Lee et al., 2017)
✓		✓					✓						(Radzali et al., 2014)
✓			✓							✓		✓	(Hou et al., 2018)
	✓					✓				✓		✓	(Lim et al., 2018)
✓					✓				✓		✓		(Kumazaki et al., 2014)
✓				✓	✓						✓		(Toguchi et al., 2019)
	✓					✓				✓		✓	(Quah et al., 2016)
✓				✓	✓				✓		✓		(Matsumoto et al., 2018)

According to the SLR results, the MOCVD, MOVPE, and HVPE were the most popular growth techniques among researchers (Meyers et al., 2020; C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2021, 2022).

Carrier concentration refers to the number of charge carriers (electrons or holes) present in a semiconductor material. The motion of these charge carriers has a significant impact on how electric current behaves in semiconductors. Based on the extracted articles, previous researchers used 10¹⁶, 10¹⁷, 10¹⁸, and 10¹⁹ cm⁻¹ of carrier concentrations.

The SLR results further demonstrated that a carrier concentration of 10^{18} cm^{-1} was preferred (Cheah et al., 2015; Heffernan et al., 2020; Lee et al., 2017; Radzali et al., 2014; C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2022)

In a PEC etching process, the thickness of a GaN thin layer plays an important role in influencing the rates of surface reactions, charge carrier formation, transport and recombination, and light absorption. Thus, it is essential to select the appropriate film thickness for the PEC etching process. Many researchers choose GaN thickness that ranges between 3.1 and 6.0 μm , as indicated in Table 1. This study has determined that GaN thickness range of 3.1 to 6.0 μm was fairly thick. While thicker materials are normally preferred for an effective PEC etching, it is vital to remember that stability must be achieved. If the material gets too thick, it may be difficult to support the surface etching, since thicker materials require greater electrical supply.

The substrate is a base for the components of the thin film. The features of the substrate can have a significant impact on how well the device operates. Several substrates for GaN material have been described, including GaN, Si, SiC, and sapphire/diamond (Al-Zuhairi et al., 2022). This research would only report on two types of substrates, namely, sapphire and GaN. The usage of sapphire substrate outnumbered GaN substrate (Cheah et al., 2015; Heffernan et al., 2020; Hou et al., 2018; Lim et al., 2018; Quah et al., 2016; C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2021, 2022), sapphire substrate is more affordable than GaN substrate, since GaN substrate can only be produced in the same way as GaN thin film.

Another essential aspect of material that was rarely employed by previous studies was the buffer layer. A buffer layer in a semiconductor serves as an interface between two materials that frequently have different crystal structures, lattice constants, or electrical properties. The primary goal of a buffer layer is to eliminate incompatibilities or mismatches between the two materials and to promote the development of superior layers on top. Based on the literature review in this study, Mayer et al. have utilised an AlGaIn buffer layer that has grown beneath the GaN layer (Meyers et al., 2020).

PEC Etching of GaN

Various factors must be considered before, during, and after the PEC etching process. In the last 10 years, researchers from all over the globe have investigated and developed an array of new research studies on PEC. This section focuses on PEC etching processes extracted from the 15 selected articles.

The cleaning and preparation of the GaN wafer is typically included in the PEC etching pre-process. Several studies have utilised a method of cleaning the GaN wafer with acetone, ethanol, and deionized (DI) water (C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2021, 2022). Zhang et al. presented an ultrasonically washed version of the previously described cleaning chemicals. Following these cleanings, these samples were also blow-dried in a N_2 atmosphere (M. R. Zhang et al., 2017; Z. Zhang et al., 2022), and in a H_2 atmosphere (Z. Zhang et al., 2021). Apart from that, Radio Corporation of America (RCA) cleaning has also been utilised to clean samples (Lim et al., 2018; Quah et al., 2016). Another significant cleaning technique to highlight is the use of aqua regia to remove surface contamination, followed by DI water rinsing (C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2022). During storage, GaN epitaxial wafers may form a surface oxide layer. Zhang et al.

addressed this issue by immersing the wafer in 1 M H₂SO₄ solution to thoroughly remove the oxide layer to prevent interference with the etching process (Z. Zhang et al., 2022). Zhang et al. have also conducted a hydrophilic treatment of GaN wafer using a low-temperature UV/O₃ vacuum plasma treatment to enhance the contact between the etchant and GaN epitaxial layer (Z. Zhang et al., 2021, 2022). Another simple yet remarkable approach was by Toguchi et al., who fabricated an etching mask on the surface of the GaN semiconductor using a positive type photoresist film, and the patterned sample was photolithographed, and then, heat baked at 110 °C for 10 min (Toguchi et al., 2019).

Many factors are behind the unique properties of p-type semiconductors. The porous patterns on p-type GaN can be challenging to fabricate. Thus, Meyers et al. studied samples of p-GaN that were treated using an ICP-RIE dry etch first for 15 s, with BCl₃/Cl/Ar prior to PEC etching (Meyers et al., 2020). The ICP-RIE and Electron Cyclotron Resonance-Reactive Ion Beam Etching (ECR-RIBE) were the dry etching techniques used by Matsumoto et al. as the initial etching processes for GaN. They confirmed that PEC etching can reduce and remove the damages caused by dry etching in the near surface region of the GaN sample (Matsumoto et al., 2018). Other than dry etching techniques, Meyers et al. performed a photochemical (PC) etching in 25 mM KOH/DI water solution to confirm the type of selectivity of the etch (Meyers et al., 2020). They claimed that it was preferable to use PC etching before PEC etching, since PEC etch lacks the ability to have a charge-balancing process to remove excessive electrons from the samples (Meyers et al., 2020).

The key factors in PEC etching processes include electrolyte, source of light, counter electrode, working electrode, reference electrode, and a biasing system, as summarised in Table 2. These factors depend on the kind of components researchers intend to work with and alter. The mechanisms and components used in the following Figures 4(a) to 4(d) are different to one another based on the previously mentioned factors. Figure 4(a) shows a study from Wang et al., which utilises two (2) electrodes and DC current as the current source (C. Wang et al., 2018). Figure 4(b) shows a schematic diagram of two (2) electrodes that use AC current as the current source (Quah et al., 2016). Meanwhile, Figure 4(c) shows a schematic diagram of a system with no electrode (Toguchi et al., 2019), and Figure 4(d) displays a schematic diagram of a system with three (3) electrodes (Kumazaki et al., 2014).

Types of Electrolytes

The electrolyte used in PEC etching is also an important consideration because it affects the efficiency and selectivity of the etching process, as well as the chemical processes that take place at the SEI. Ions are transferred from the surface of the semiconductor via the electrolyte, which increases the etching process. Based on Table 2, various electrolytes have been used by previous researchers, with KOH being the conventional and most common (Heffernan et al., 2020; Lee et al., 2017; Meyers et al., 2020; Radzali et al., 2014; Toguchi et al., 2019). H₂SO₄ and H₃PO₄ electrolytes were also well-known among researchers (Cheah et al., 2015; Heffernan et al., 2020; Kumazaki et al., 2014; Lim et al., 2018; Toguchi et al., 2019). Wang et al. have introduced an effective, yet environmentally friendly etchant for etching GaN, which was an organic electrolyte solution that consisted of ethyl methyl carbonate (EMC) and lithium tetrafluoroborate (LiBF₄) (C. Wang et al., 2018). Another interesting electrolyte study for PEC etching was by Ta., where they demonstrated the use of ethylenediaminetetraacetic acid disodium salt (EDTA-2Na) as an etchant. The ethylenediaminetetraacetic acid ions (EDTA²⁻) have strong coordination capability with almost all metal ions, while Ga-EDTA complex was

used as the gallium precursor to synthesise nanocrystalline gallium nitride (M. R. Zhang et al., 2017). Small molecular weight and mild chemical characteristics are typical for amino acids. The R groups in amino acids, including alanine (Ala), valine (Val), and glycine (Gly) have been utilised as etchants via adjustable control by altering their polarity, and the average pore size and pore density (Z. Zhang et al., 2022). Additionally, three different types of ionic liquids, namely, 1-ethyl-3-methylimidazolium trifluoromethanesulfonate ([EMIM][OTF]), 1-ethyl-3-methylimidazolium bis((trifluoromethyl) sulfonyl) imide ([EMIM][NTF₂]), and 1-ethyl-3-methylimidazolium trifluoroacetate ([EMIM][CF₃OAc]) were also used as etchants. These ionic liquids were more environmentally friendly than inorganic etchants as a type of green reagents (Hou et al., 2018). C₄H₆O₆, C₃H₈O₂, and NH₄OH electrolytes have also been used in a previous research (Matsumoto et al., 2018).

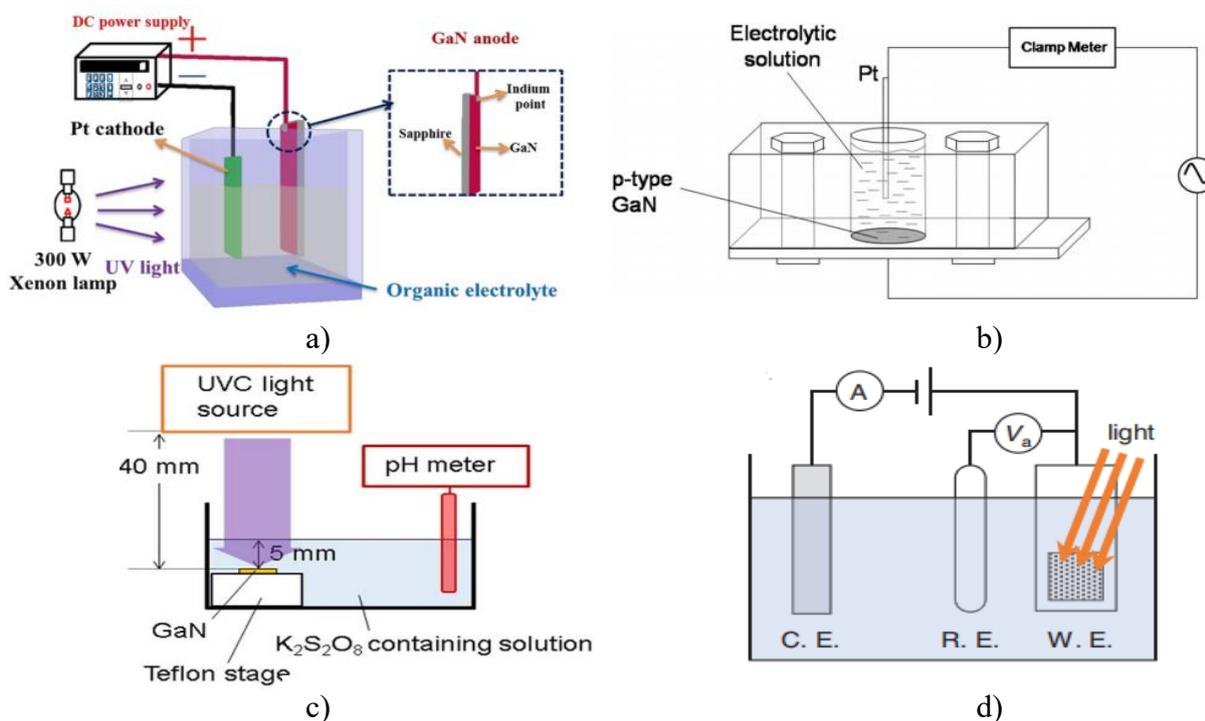


Figure 4: Schematic Diagrams Of Electrode And Electrodeless PEC Etchings: 4(a) DCPEC Etching With Two (2) Electrodes (C. Wang et al., 2018); 4(b) ACPEC Etching With Two (2) Electrodes (Quah et al., 2016); 4(c) Electrodeless PEC Etching (Toguchi et al., 2019); And 4(D) PEC Etching With Three (3) Electrodes (Kumazaki et al., 2014).

Types of Working, Counter, and Reference Electrodes

Working electrodes usually refer to the semiconductor itself, which is GaN. However, to enhance the electrical contact between the semiconductor and the electrolyte, researchers would place a type of contact at the semiconductor surface to act as a working electrode. This contact, which is also an anode terminal, is placed on the semiconductor, while it is submerged in a conductive electrolyte during PEC processing (Kohl, 1998). Indium is the most preferred working electrode, since it can perform an ohmic contact when an electrical bias is applied to the PEC system (Heffernan et al., 2020; Hou et al., 2018; Kumazaki et al., 2014; Lim et al., 2018; Matsumoto et al., 2018; Radzali et al., 2014; Toguchi et al., 2019; C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2022, 2021).

Table 2: Key Components And Parameters In PEC Etching

Electrolyte	Counter Electrode	Light Sources		Working Electrode	Etching Duration (min)	Power Supply		Others	References
		Type of Light	Details			Voltage (V)	Current (mA)		
LiBF ₄ , EMC	Pt	Xenon lamp (UV)	N/A	In	N/A	N/A	N/A	Used DC current at room temperature	(C. Wang et al., 2018)
KOH, H ₂ SO ₄	N/A	UV	150 W	N/A	N/A	10–30	N/A	Conducted at room temperature	(Cheah et al., 2015)
KOH, K ₂ S ₂ O ₈ , Na ₃ PO ₄	Electroless	Xe arc lamp	$\lambda = 365$ nm	Ni/Au, Ti/Au	4-32	N/A	N/A	Conducted at 70 °C	(Meyers et al., 2020)
H ₃ PO ₄ , KOH	Pt	HeCd laser beam	5–50 mWcm ⁻²	In	N/A	N/A	N/A	Conducted at room temperature with saturated calomel reference electrode (SCE)	(Heffernan et al., 2020)
EDTA-2Na	Pt	Xenon lamp	300 W	In	3	5	N/A	Used DC current	(M. R. Zhang et al., 2017)
Glycine, Alanine, Valine	Pt	Xenon lamp	N/A	In	5	12	N/A	Used DC current	(Z. Zhang et al., 2022)
Glycine	Pt	Xenon lamp	300 W	In	5	10	N/A	Used DC current	(Z. Zhang et al., 2021)
KOH, K ₂ S ₂ O ₄	N/A	Xe arc lamp	1000 W, $\lambda = 345$ – 364 nm,	Ti/Au	6-20	N/A	N/A	N/A	(Lee et al., 2017)

				100 mWcm ⁻²					
KOH	Pt	Xenon lamp	N/A	N/A	15-45	N/A	N/A	N/A	(Radzali et al., 2014)
[EMIM][OTF], [EMIM][NTF ₂], [EMIM][CF ₃ OAc]	Pt	Xenon lamp	N/A	In	1-7	N/A	N/A	Used DC current	(Hou et al., 2018)
CH ₃ OH-H ₂ SO ₄	Pt	UV lamp	N/A	N/A	90	N/A	60	Used AC current	(Lim et al., 2018)
H ₂ SO ₄ , H ₃ PO ₄	Pt	Xenon lamp	5–40 mWcm ⁻²	Au	5-30	0-4	N/A	Used reference electrode Ag/AgCl	(Kumazaki et al., 2014)
H ₃ PO ₄ , KOH, K ₂ S ₂ O ₈	Electroless	UV	λ = 260 nm, 4 mWcm ⁻²	N/A	0-120	N/A	N/A	N/A	(Toguchi et al., 2019)
CH ₃ OH-H ₂ SO ₄	Pt	UV	N/A	N/A	90	N/A	40-100	Used AC current	(Quah et al., 2016)
C ₄ H ₆ O ₆ , C ₃ H ₈ O ₂ , NH ₄ OH	Pt	Xenon lamp	λ = 360 nm, 5 mWcm ⁻²	Ti/Au	N/A	4	N/A	Room temperature with Ag/AgCl as reference electrode	(Matsumoto et al., 2018)

Additionally, Au, Ni/Au, and Ti/Au have also been used by researchers (Kumazaki et al., 2014; Lee et al., 2017; Matsumoto et al., 2018; Meyers et al., 2020). Other than working electrodes, it is necessary to place a counter electrode in the PEC etching cell setup to enable an effective charge transfer and to regulate etching behaviour. The etching process can be improved with a suitable counter electrode, with better control over the emerging nanostructures or patterns. Based on Table 2, platinum, as a counter electrode, is commonly used by researchers (Heffernan et al., 2020; Hou et al., 2018; Kumazaki et al., 2014; Lim et al., 2018; Matsumoto et al., 2018; Radzali et al., 2014; Toguchi et al., 2019; C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2022, 2021). This is due to platinum having superior conductivity, stability, and catalytic qualities (Papageorgiou, 2004).

Other materials with comparable qualities, for example, Au and Ti can also work well as counter electrodes in a PEC etching setting. Reference electrodes have also been used by previous researchers because the potential of a semiconductor can be measured versus a reference electrode. In this way, the current flowing between the semiconductor and the counter electrode can be recorded as a function of the potential versus the reference electrode (Kohl, 1998). Saturated calomel electrode (SCE) and Ag/AgCl are commonly used as reference electrodes (Heffernan et al., 2020; Kumazaki et al., 2014; Matsumoto et al., 2018). PEC etching setups that do not use electrodes are also available, known as electrodeless/ electroless (Toguchi et al., 2019).

Source of Light

When it comes to PEC etching procedures, source of light is another key factor. It has an immediate impact on the semiconductor material's capacity to produce charge carriers (electron-hole pairs), which in turn has an impact on the speed and effectiveness of the etching operation. Table 2 lists the various types of photon sources that have been employed, with different powers, wavelengths, and power intensities. Details of the sources of light may include the source's power (150–1000 W), wavelength (260–364 nm), and power intensity (4–100 mWcm⁻²). This study has concluded that the most operated light source used by previous researchers in PEC etching was Xenon lamp (Hou et al., 2018; Kumazaki et al., 2014; Lee et al., 2017; Matsumoto et al., 2018; Meyers et al., 2020; Radzali et al., 2014; C. Wang et al., 2018; M. R. Zhang et al., 2017; Z. Zhang et al., 2021, 2022).

Etching Parameters

In PEC etching, the etching duration refers to the amount of time that this process is able to operate. It is a key parameter that has a direct impact on the depth, form, and properties of the etched nanostructures. The etching duration is determined by the desired outcome, the material being etched, and the etching mechanism. Table 2 reveals that previous researchers have employed an etching length that ranges from 4 to 120 min. Meyers et al. discovered that with longer sequential PEC etch durations, the surface morphology of the etched material can become progressively disordered, indicating the formation of a nanoporous layer (Meyers et al., 2020).

The temperature at which PEC etching is performed can also have a significant impact on the resulting nanostructures. The temperatures utilised in the selected studies were not mentioned by the majority of them. Nonetheless, Table 2 shows that room temperature is commonly employed (Cheah et al., 2015; Heffernan et al., 2020; Matsumoto et al., 2018; C. Wang et al., 2018). Low temperatures were not reported, but a high temperature was reported by Meyers et al. (Meyers et al., 2020).

Etching voltage, commonly known as bias voltage, is another critical parameter in the PEC etching process. It represents the electrical potential difference between the semiconductor material being etched and the counter electrode. Different etching voltages have been reported in the 15 selected articles, as listed in Table 2 ranging from 4 to 30 V.

Characterization of Porous GaN

This section will discuss the structural and optical properties of porous GaN etched using PEC. Table 3 summarises the instruments reported in the 15 selected articles. The surface morphologies of the reported samples have been analysed using scanning electron microscopy

(SEM) and atomic force microscopy (AFM), while the structural properties were analysed using X-ray diffraction (XRD). This section also discuss the optical properties of the GaN samples after PEC etching, which were analysed using Raman spectroscopy and photoluminescence (PL).

Table 3: Summary Of The Structural And Optical Properties Of Porous Gan

Reference	Structural			Optical	
	SEM	AFM	XRD	PL	Raman
(C. Wang et al., 2018)	✓				
(Cheah et al., 2015)	✓			✓	
(Meyers et al., 2020)	✓	✓			
(Heffernan et al., 2020)	✓	✓		✓	
(M. R. Zhang et al., 2017)	✓				
(Z. Zhang et al., 2022)	✓		✓		✓
(Z. Zhang et al., 2021)	✓		✓		✓
(Lee et al., 2017)	✓	✓			
(Radzali et al., 2014)	✓		✓		✓
(Hou et al., 2018)	✓			✓	
(Lim et al., 2018)	✓	✓	✓	✓	✓
(Kumazaki et al., 2014)	✓			✓	
(Toguchi et al., 2019)		✓			
(Quah et al., 2016)	✓	✓	✓	✓	✓
(Matsumoto et al., 2018)	✓			✓	

SEM

The SEM image in Figure 5(a) shows that the morphologies of the porous GaN samples are dependent on both the UV density and the applied voltage, while the pore sizes are increased with increasing UV density (C. Wang et al., 2018). By increasing the applied voltage from 10 to 30 V, the pore size of the etched GaN was also increased from 37.5 to 245.5 nm. Consequently, Wang et al. claimed that the organic electrolyte can effectively fabricate pores (C. Wang et al., 2018). This finding was supported by Cheah et al., who reported that 0.1 mm of the pore layer was converted into a porous layer during the PEC etching process when the applied voltage was set to 10 V (Cheah et al., 2015). Similar, but larger pore morphologies were developed when the applied voltages were increased to 20 and 30 V. These findings were obtained because higher voltages have the ability to offer the system an extra energy, which increased the chemical reactions at the semiconductor-electrolyte interface. Larger surface characteristics and more visible etching could also be observed from this interaction. According to [34], [EMIM][OTF], [EMIM][NTF₂], and [EMIM][CF₃OAc] are able to create honeycomb shapes on the surface of the GaN samples, as can be seen in Figure 5(b). Meanwhile, an overlapping hole structure is shown in Figure 5(c), and a deep gully with holes in the surroundings is shown in Figure 5(d). Thus, it can be concluded that the porous structure of GaN has been destroyed after undergoing etching by [EMIM][NTF₂] and [EMIM][CF₃OAc] (Hou et al., 2018). According to Hou et al., the electrostatic attraction of anions in an ionic liquid can be intense due to the differences between them and an aqueous solution, since they can interact with the holes on the interface between a solid and a liquid (Hou et al., 2018). SEM images of porous GaN samples prepared using [EMIM][OTF] showed that increased etching times can result in larger pore sizes (Hou et al., 2018). Radzali et al. have proven that the pore

size and density of GaN samples can increase significantly with longer etching time, once they obtain the hexagonal-like pores, as illustrated in Figure 5(e) (Radzali et al., 2014). Zhang et al. report a complete three-dimensional honeycomb structure, as shown in Figure 5(f), with a smooth and uniform surface using Gly as the etchant. They have also observed a tendency for the pore diameter to increase, with a progressive increase in etching depth (Z. Zhang et al., 2021).

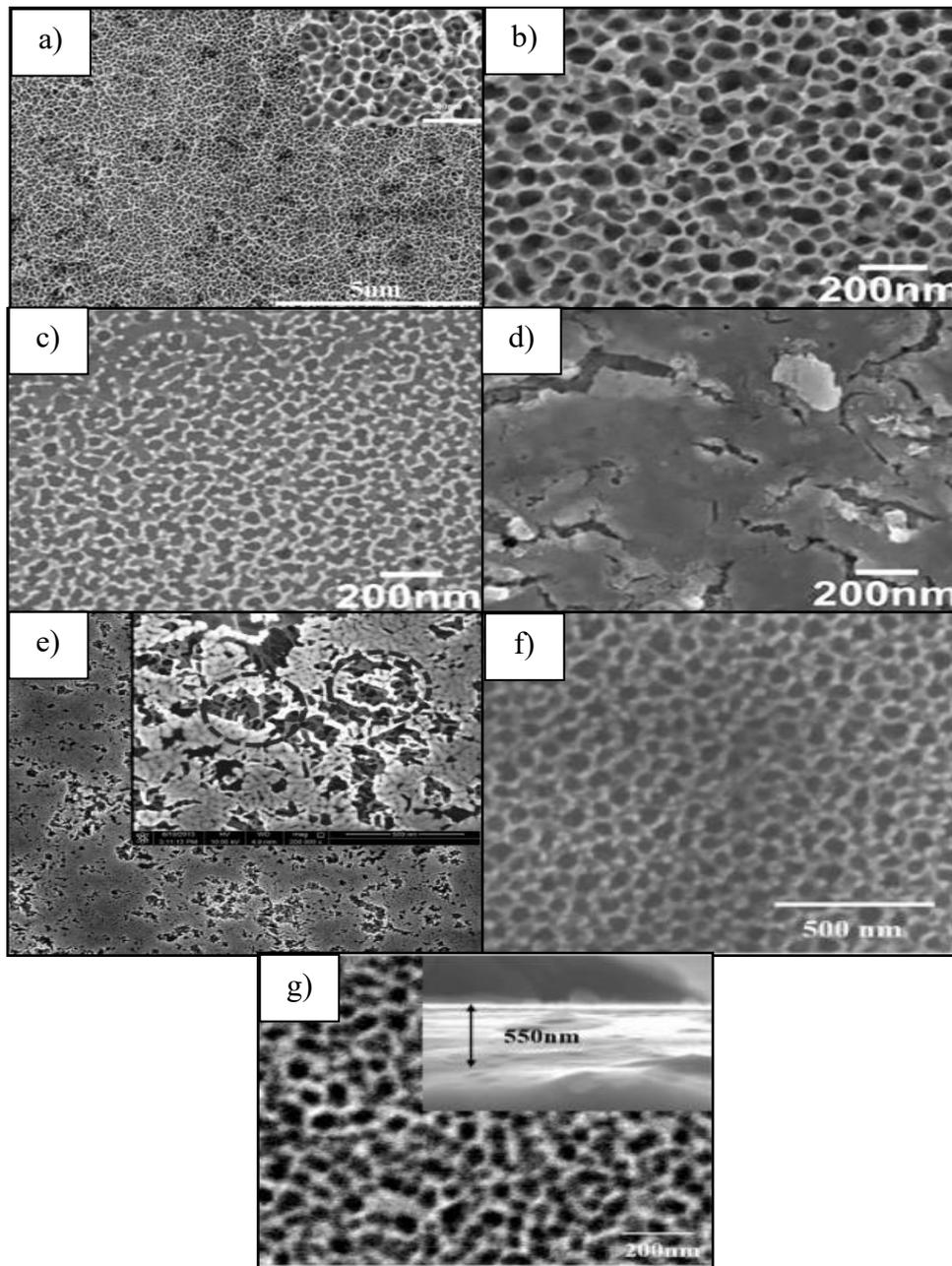


Figure 5: SEM Images Of Porous GaN Samples, As Reported By Previous Studies: (a) (C. Wang et al., 2018), (b) (Hou et al., 2018), (c) (Hou et al., 2018), (d) (Hou et al., 2018), (e) (Radzali et al., 2014), (f) (Z. Zhang et al., 2021), And (g) (Z. Zhang et al., 2022).

The honeycomb structure on the wafer that was not immersed in H_2SO_4 solution was uneven, with apparent etchant residue and a rough honeycomb wall (Z. Zhang et al., 2021). Comparatively, amino acids can produce more useful foam-like structures when used as an etchant. Amino acids utilised by Zhang et al. included Gly, Ala, and Val. Gly is able to create the biggest pore density and the smallest average pore size in the field of GaN PEC, as shown in Figure 5(g) (Z. Zhang et al., 2022). These findings showed that the foam-like structure has a consistent shape and distinct texture. Meanwhile, Ala and Val took up more room and electron mobility, which led to further pore size expansion during the etching process (Z. Zhang et al., 2022).

AFM

AFM analysis showed that PEC etching of highly doped n-GaN, which was not possible with the Xe arc lamp, was made possible by the Q-switched 355 nm laser (Lee et al., 2017). Surface roughness of 1.6 nm and 3.6 nm RMS were attained after timed etching down to an $n-Al^{0.20}Ga^{0.80}N$ layer, which was nearly 300 nm deep. Surface roughness could be improved using a refined PEC etching technique (Lee et al., 2017). According to Lim et al., a non-porous film has the lowest RMS surface roughness, which could be due to the film's relatively flat surface topography and the distribution of hillocks without any cavity (Lim et al., 2018). The film that was etched in $H_2SO_4: CH_3OH$ (2:1) solution has the highest RMS surface roughness among the porous films (Lim et al., 2018).

The same result was obtained by Quah et al., where they stated that a non-porous p-GaN film, which originated from a uniform distribution of hillocks with comparable diameter, has the lowest RMS surface roughness compared to the porous samples (Quah et al., 2016). Another interesting PEC etching technique was proposed by Meyers et al., where they started with dry etching the p-GaN sample before conducting the PEC etch (Meyers et al., 2020). Following the dry etch of the p-GaN surface and each succeeding PEC etch, the surface morphology of the sample changed. ICP-RIE etching somehow roughened the surface of the sample, with the surface morphology of the sample progressively developing a nanoporous morphology after 8 min (Meyers et al., 2020).

XRD

By using Gly as the etchant, it was discovered that the XRD diffraction peaks after the etching perfectly matched the peaks of GaN crystals, thus proving that Gly was a mild and effective etchant, and not damaging to the crystalline structure (Z. Zhang et al., 2021). Zhang et al. obtained similar results, where the diffraction patterns of foam-like GaN, which corresponded to GaN etched with Val, Ala, and Gly, showed no obvious heterogeneous peaks or clear shift in peak positions before and after etching. However, the F-GaN crystal surface, which was obtained using three different amino acids as etchants, showed no changes in its diffraction peaks and crystal structure (Z. Zhang et al., 2022). The GaN (0002) and (0004) peaks for the as grown sample can be observed at $\sim 34^\circ$ and $\sim 72^\circ$, respectively. It is important to notice that GaN peaks can still be observed in all porous samples, demonstrating that the GaN layer was not completely etched away during the etching process and has retained its epitaxial characteristic (Radzali et al., 2014).

Photoluminescence

Cheah et al. reported that when the free carrier concentration of GaN thin films was increased from 10^{17} to 10^{18} cm^{-3} , a noticeable redshift occurred to the near-band edge (NBE) emission,

which was a narrowing of the band gap in PL analysis (Cheah et al., 2015). They have also concluded that the high free carrier concentration in n-GaN was mostly responsible for the significant luminescence signal of the n-GaN sample compared to the UID GaN sample. Following the PEC etching procedure, according to Radzali et al., no change was observed in the band-edge luminescence. This result was in line with the fact that anodic etching of porous GaN structures is not linked to any blueshift (Radzali et al., 2014). Radzali et al. also claimed that compared to an untreated n-GaN surface, there was a noticeable increase in the defect-related yellow luminescence peak at approximately 535 nm. The production of the ridge structures, which could be assigned to surface-terminating dislocations, and the development of this peak may be connected (Radzali et al., 2014). On the other hand, a significant peak observed in the PL spectrum at approximately 365 nm, while a much smaller peak can be seen at approximately 373 nm due to the presence of silicon and other impurities in the GaN layer (M. R. Zhang et al., 2017). Compared to planar GaN, the major peaks of porous GaN exhibited a small redshift because of the relaxation strain at the surface (Hou et al., 2018).

Raman Spectroscopy

Radzali et al. obtained the optical phonon region of GaN at 567 and 734 cm^{-1} of the spectrum, which corresponded to the E_2 (high) and $A_1(\text{LO})$, respectively. However, their study found that the as-grown sample was absent in the $E_1(\text{TO})$ phonon mode, although it was present in the porous samples. The occurrence of this phonon mode may be due to the instability of the porous samples' crystal lattice, which would increase the scattering of light from the sidewalls and could alter the polarisation of the light (Radzali et al., 2014). Zhang et al. observed that four peaks, namely, E_2 (low) and E_2 (high), a LO phonon peak $E_1(\text{LO})$, and a sapphire substrate were visible in the Raman spectrum of the honeycomb-like GaN obtained using PEC, which proved that this material shared the same lattice properties as planar GaN. These results have also proven that the crystal properties of GaN were not harmed when Gly was used as the PEC etchant (Z. Zhang et al., 2021). Zhang et al. have also revealed that the porosity of the foam-like structure was gradually expanded when the concentration of amino acid as etchant was increased, eventually leading to the release of compressive stress. A decrease in peak width has also occurred, along with a decrease in the half-peak width (Z. Zhang et al., 2022).

Conclusion

This study has presented a comprehensive systematic review of research conducted over the past decade (2014–2023) on the procedures and effectiveness of PEC etching in producing porous GaN. The reviewed studies were systematically categorized into three main themes: (1) the growth techniques and fundamental properties of GaN, (2) the methods and parameters involved in the PEC etching process, and (3) the morphological, structural, and optical characteristics of the resulting porous GaN. Through this structured analysis, the study has successfully achieved its objectives by identifying and synthesizing key findings that demonstrate the effectiveness of PEC etching as a promising method for fabricating nanostructured GaN, particularly for use in optoelectronic and photonic applications. The findings of this review contribute meaningfully to both academic understanding and practical advancement by offering an organized overview of the progress made in PEC etching and highlighting the potential of low temperature techniques in nanofabrication. While PEC etching shows strong performance and versatility, several challenges remain, including achieving uniform porosity and improving control over etching parameters. Future studies are recommended to address these issues through the integration of advanced control systems, optimization of electrolyte conditions, and real-time monitoring techniques. Further

investigation into device-level applications of porous GaN, such as its incorporation into photodetectors or LEDs, would also enhance the practical relevance of ongoing research in this area.

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