# The Effect of Etching Time on Structural Properties of Porous Quaternary AlInGaN Thin Films

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# Abstract

Using photo electrochemical etching technique, porous silicon (PS) layer was prepared on n-type silicon wafers to generate porous silicon for n-type with an orientation of (111). X-ray diffraction pattern revealed differences between peaks where the high of the peaks and intensity decreased with increasing etching time. The largest crystal size was (30 nm) and the lowest crystal size was (28.6 nm). The atomic force microscopy and field emission scanning electron microscopy were used to study the morphology of porous silicon layer. As the etching time was increased, AFM measurements showed that (RMS), roughness and grain size decreased. FESEM showed a homogeneous pattern and verified the formation of uniform porous silicon.

# Article Info.

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# **1. Introduction**

Porous silicon was discovered in 1956 by Ulhir [1] when performing electropolishing silicon wafer experiments using an electrolyte-containing hydrofluoric acid [2]. Porous Silicon (PS) is a silicon-based nanostructured material formed by electrical processing. Silicon remains uniformly undissolved but it creates very fine holes. Electrochemical dissolution of silicon wafers in aqueous or ethanoic solutions has resulted in PS formation. Interest in PS grew in the 1970s and 1980s because its high surface area was found to be useful in spectroscopic studies as a model of the crystalline silicon surface [3]. Leigh Canham reported his findings on red luminescence in the 1990s [4] showing that certain PS materials can have high PL efficiencies at rooms temperatures in the visible. A shocking finding because PL efficiencies of bulk silicon is very low due to its indirect energy bandgap and limited non-radiative lifespan. The reasons for the partial breakdown of silicon which induces the formation of silicon. Phenomena in quantum confinement result in new effects such as photoluminescence or electroluminescence [5]. PS categories to the diameter of the pore which can be ranged from a few nanometers to a few macrons that depending on the formation parameters [6].

Due to its specific electrical chemical and mechanical features, PS has been shown to achieve effective visible light emissions at room temperature. Different conclusions however are stated on PL from the surface of PS [7, 8]. The first concerns the effect of quantum containment due to the charge carriers in the thin crystalline silicon wall that separates the pore walls. Lately several other alternative models were proposed based on hydrogenated amorphous silicon surface hydrides holes' siloxane and surface states [9, 10]. Many of the PS layer's properties depends on the etched parameters including HF concentration current density temperature and form and resistivity of the Si wafer [11]. Nitride material system has several important properties that make it ideal for near IR to deep UV optoelectronic devices. These properties also allow AlInGaN material to endure challenging applications that involve harsh operating conditions. An III-nitride semiconductor has a wide direct band gap which can be tuned from 0.7 eV for InN, to 3.5 eV for GaN and to 6.23 eV for AlN. Due to its amazing properties III-nitride groups have gained many great attention in the last few years but the methods to prepare high-quality layer without defects and crystal impurities are only at the beginning and need to be more researched and investigated. One of these methods is: Molecular beam epitaxy (MBE) which was used in this analysis to enable the researchers to improve their commitment to find out new methods in terms of preparation [12]. In this work we Study the structural properties of quaternary AlInGaN thin films.

# 2. Experimental work

Quaternary AlInGaN films were deposited on silicon substrate by molecular beam epitaxy the preparation of the n-PS layer is shown in Fig.1 HF main electrolyte acid. And in a horizontal configuration the appliance is built also on n-Si <111> substrates in an electrolyte an HF mixture (40%) is produced from porous silicon generated with a standard technique the all processing during PS formations can be expresses:

 $Si + 2H \rightarrow 2h^+ / SiF_2 + 2H/$ 

## $SiF_2 + 4HF/H_2 \rightarrow + H_2SiF_6$

Anodization is used in the (current-controlled) galvanostatic mode. It is generally favored since irrespective of any evolution during cell electrical impedance anodization it provides the necessary charge for the reaction at a constant pace eventually contributing to homogeneous and reproducible content. It can be modulated by anodization. The modulation is done more effectively by adjusting The PS samples were synthesized by anodic etching using a traditional single-tank electrolyzation cell on n-type Si wafers. Along the (111) crystal plane path, the wafers were polished. The electrolytes were prepared by combining varying volumetric ratios of HF solution and absolute methanol (CH<sub>3</sub>OH).



Figure 1: The schematic diagram of experimental step for preparing n-PS layer vertical arrangement.

The cell that can be used for silicon anodizing is very simple. The anode is silicon wafer. While the cathode is made of platinum or other substance that is HF resistant and conducting. The distance from the center to the platinum Si is round 2 cm. The silicon

wafers used were n-type, <111> double polished with a resistance of 25.cm A selfmade Teflon cell was used to etch a circular area of 1.5 cm<sup>2</sup> on the wafer. By the scribe and cut process the wafers were cut into pieces sufficient to contain this area. Until drying the Si wafer had to be diced first 1.5 to 1.5 cm<sup>2</sup> samples. To extract particulate matter as well organic metallic and ionic contaminants from specimens cleaning is necessary. Aluminum foil is coated with the whole rear half of the aluminum foil as the backside. They jointly sandwiched the sample and aluminum foil into the cell. The wafers were used as usual little was done either on the front side or on the back side. Then the wafer controllable parameters wafer becomes HF concentration time of anodization and illumination. Thus the concentration of HF and the duration of anodization differ. N-type silicon wafers in the experiments. Methanol is used in samples and alcohol is commonly used to disinfect the wafers by immersing it in these chemicals for a few minutes in the ultrasonic baths. Finally, they were rinsed in ultrasonically filtered purified water accompanied by drying in a stream of hot air. Porous silicon (PS) samples were prepared by anodization at a constant time of 5 min, 10 min and 15 min at a current density (50) mA/cm<sup>2</sup>. A photon source, such as halogen lamb, is needed to obtain the nano crystalline porous Si on n-type silicon. An illumination system where the halogen lamb power is 100 watts and the distance between the lamb and Si and intensive light is used to supply the necessary holes. The most effective method of creating holes in the process of electrochemical etching is shown in Fig.2.



Figure 2: Schematic of the experimental setup of the illumination assisted method.

# 3. Results and discussions

The X-ray diffraction spectra of porous on the n-Si substrate at different etching times are shown in Fig.3. These revealed a separate distinction samples at different anodization between the times based on the phases of the sample the X-ray radiation diffracts at various angular angles with respect to the incident beam. An expansion of diffraction peaks was found as the crystal size was decreased to the nanometer scale and the diameter of the peak closely associated with the size of the peak. Fig.3 also shows the X ray diffraction pattern of AlInGaN after etching time of 5, 10 and 15 min, which revealed that at all cases the diffraction peaks were at 2 $\theta$ , equal to 34.46, 34.45 and 34.44 corresponding to Si (111), quaternary AlInGaN (0002), buffer layer AlN (002) and AlInGaN (004) respectively. In addition, the intensity of preferred orientation AlInGaN (002) became lower with increasing the etching time while at orientation are reverse which attributed to less the preferring of orientation (002) and formed Nano

particles depending of the pores sites added and sequent the quantum confinement effect become very clear. Table (1) summarized the XRD parameters.



Figure 3: XRD spectrum of (0002) porous quaternary Al<sub>0.08</sub>In<sub>0.08</sub>Ga<sub>0.84</sub>N thin film grown on Si (111) substrate at three etching time 5, 10, and 15 min.

AFM measurements demonstrate that the surface of the etched PS layer consists of a matrix of uniformly spaced nanocrystalline Si pillars and voids at etching time of 5 min, 10 min and 15 min. The average diameter diminished as the etching time increased. A variation in the microstructure was found for various times of the porous silicon surface where pores varied greatly as seen in the images of the AFM. The PS layers surface comprises of a nanocrystal line Si uniformly dispersed matrix that has the same orientation and AFM images often show void also reveal voids that composition this attributed to longer etching time caused an increasing in root mean square and roughness show in Fig.4.

Fig.5 shows FESEM images of the etched surface prepared by a porous sheet consisting of a large thick pore aligned uniformly at 5, 10, and 15 minutes of etching time. In structure pores in (FE-SEM) images and a homogeneous pattern a group of PS is presented and confirms the growth of uniform porous structures on the silicon. The influence of growing time of 15 min on surface morphology.

Table 1: FWHM, miller indices, phase and grain size of the porous quaternary AlInGaN thinfilms grown on Si substrate.

Time (min)	2θ (Deg.)	FWHM (Deg.)	d <sub>hkl</sub> (Å)	C.S (nm)	Phase	hkl
5	34.460	0.2730	2.6005	30.5	Hex AlInGaN.Si	(0002)
10	34.450	0.2822	2.6013	29.5	Hex AlInGaN.Si	(0002)
15	34.440	0.2930	2.6020	28.4	Hex AlInGaN.Si	(0002)



Figure 4: 2- and 3-D AFM images of porous quaternary Al<sub>0.08</sub>In<sub>0.08</sub>Ga<sub>0.84</sub>N thin films grown on Si (111) substrates with different etching times of: a- 5 min, b- 10 min, and c- 15 min.





(c)

Figure 5: SEM images for porous quaternary n-Al<sub>0.08</sub>In<sub>0.08</sub>Ga<sub>0.84</sub>N thin films grown on Si (111) substrates at different etching times: a) 5min, b) 10 min, and c) 15 min.

#### 4. Conclusions

Quaternary AlInGaN thin films were deposited on silicon substrate by molecular beam epitaxy using N-type porous silicon synthesized by electrochemical etching at different etching times (5, 10 and 15) min. X- ray diffraction patterns showed the formation of porous silicon and that the structure thin film size increase of the Si nanosized of the Si peaks the particle size of the porous layers is nanostructured were decrease. The investigation of atomic force microscopy has demonstrated an improvement in surface roughness with the increase of etching time. Field Emission scanning electron microscope (FESEM) of PS at various etching periods revealed, a homogeneous pattern and confirmed the development of uniform porous structures on the silicon wafers.

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# **Conflict of interest**

Authors declare that they have no conflict of interest.

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# تأثير وقت الحفر على الخصائص التركيبية للأغشية الرقيقة الرباعية المسامية للالمنيوم- انديوم-غاليوم-نترات

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الخلاصة

استخدام طريقة النقش الكهر وكيمائي لانتاج طبقات السيليكون المسامي من سليكون نوع n-type والتي تكون باتجاه (111). اظهر فحص حيود الاشعة السينية فرق بالشدة وطول القمم حيث انها تقل شدتها وطول القمة بازدياد وقت الحفر للسيليكون المسامي. ان الحجم الحبيبي وجد اكبر قيمة له هي 30 نانومتر واقل قيمة هي 28.6 نانومتر. تحليل نتائج مجهر القوة الذرية ومجهر القوة الالكترونية بين ان الخشونة تقل ايضا بازدياد وقت الحفر. ولوحظ ان النمط متجانس للسيليكون المسامي.