CHPATER 3

ETCH TECHNIQUES ON III-NITRIDES: INDUCTIVELY PLASMA DRY ETCH AND PHOTOENHANCED CHEMICAL WET ETCH

3.1 Overview of Etch Techniques on III-Nitrides Device Fabrication

The potential of the wide bandgap, nitride-based, semiconductors (GaN, AlN and InN) for use in high frequency transistors has been well documented [1-4]. The GaN-based material system is interest not only for high-efficiency blue/green light emitting diodes (LEDs) and laser diodes (LDs), but also for high power applications in defense system, power flow control and microwave transmission.

The ability to remove or etch surface materials is a fundamental process for device fabrication. These applications place stringent requirements on etching process that include reasonable etch rate, high anisotropy, high edge and etched sidewall smoothness, and minimal induced damage on the etched surfaces. In GaN-based photonic devices, the constraints placed on dry etching are minimal. Usually these structures are heavily doped, and etch depths are comparatively large, and thus are fairly resistant to damage. Moreover, the etch proceeds to an n^+-GaN layer, onto which an Ohmic contact is subsequently deposited. Preferential loss of N_2 from the near-surface region during the etch step is actually beneficial in the case because it leads to increasing n-type doping levels and hence lower contact resistances [5-7].

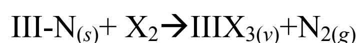
By sharp contrast, in electronic devices such as HBTs or BJTs, the etching requirements are much more demanding. Low damage processes are required to form

mesas for the base and collector contacts without increasing recombination in the base-emitter junction or surface leakage in the base-collector junction. The same situation may occur for gate recessing in HEMTs fabrication. Due to the unusual chemical stability of GaN, ICP (Inductively Coupled Plasma) etch technique is the main stream in GaN-based device fabrication.

To date, there is very limited knowledge about the effects of dry etch damage, and its subsequent removal by chemical treatment or annealing on the electrical properties of GaN materials and devices. Most past work in this area has focused on n-type materials. The sheet resistances of GaN, InGaN, InAlN and InN samples were found to increase in proportion to ion flux and ion energy in an ECR Ar plasma [8-10]. Ren ***et al.*** [11] examined the effect of ECR BCl_3/N_2 and CH_4/H_2 plasmas on the electrical performance of InAlN and GaN channel field effect transistors. They found that hydrogen passivation of the Si doping in the channel may occur if H_2 is a part of plasma chemistry and that preferential loss of N_2 degraded the rectifying properties of the Schottky contacts deposited on plasma-exposed surfaces. Saotume ***et al.*** [12] found pure Cl_2 plasma treatment decreased near band-edge PL intensity of the GaN samples by a factor of approximately five through introduction of non-radiative levels, whereas subsequent photo-assisted wet etching restored this to about half of the original value.

There are two mechanisms involved in the plasma etching, physical and chemical. A physical interaction refers to the surface bombardment by energetic ions accelerated across the sheath. Here the ions' loss of kinetic energy in the surface dominates the interactions. Chemical interactions are standard electronic bonding processes that result in the formation or dissociation of chemical species on the surface. The chemical reaction of ICP etching of III-nitrides can be tentatively written

as:



$$III\text{-}N_{(s)} + X_2 \rightarrow IIIX_{3(v)} + N_{2(g)}$$

Where III could be In, Ga or Al and X_2 represents halogens.

Figure 2-1 is the schematic plot of the chamber in the ICP etcher used in this study. The frequencies are both 13.56 MHz for the top and the bottom RF generators. The reactive gases were introduced from the gas inlet on the top of the chamber. The chamber was made of ceramic and surrounded by the RF coils. The RF resonance makes the reactive chemicals into plasmas and the high-density plasma conditions have been reported to provide faster etch rates. That's very essential in device fabrication, especially for such chemically stable material as GaN.

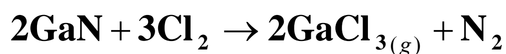
For GaN, the removal of surface material is a fundamental device process step. The most successful etch of GaN has been accomplished using dry etch methods, including RIE, ECR, and ICP. However, exposure to the energetic ions may result in significant damages, which often degrades the material properties and device performance. ICP technique uses lower ion energies than Reactive Ion Etching, and thus in general has lower damage levels as a result. Fortunately, photo-enhanced wet chemical etch can provide low damage etch and low cost.[13] In this work, wet and dry etch of GaN epilayers using photo-enhanced chemical wet etch (PEC) and inductively coupled plasma etch (ICP) are studied.

3.1.1 The Inductively coupled plasma etch

There exists no simple wet etch process (i.e., non-electrolytics and non-light-assisted) for pattern transfer in fabrication of devices such as light-emitting diodes (LED's) or laser diodes because of the chemical inertness and high bond strength of the nitrides.[14] The ridge wave guide type structures rely on dry etch for forming the ridge and associated facets in the laser diodes. Several dry etch techniques have been successful in patterning group-III nitrides for device applications. Inductively coupled plasma (ICP) etching offers an attractive dry etching technique. ICP sources are easier to scale up than ECR source, and are more economical in terms of cost and power requirements.[15]

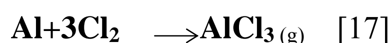
The general trend is to reach maximum etch rate using Cl_2/(Ar, N2, H2) discharge with optimum source power and pressure. The Cl_2/N_2 chemistry is most attractive because it can preserve the near-surface stoichiometry of the nitride materials and minimize the preferential N loss [15].

For ICP, selective etch of GaN relative to AlN and $Al_{.28}Ga_{.72}N$ can be achieved at low DC biases. At –20V, the GaN etch rates were 38 times greater than AlN and a factor of 10 greater than $Al_{.28}Ga_{.72}N$.[16] The reaction in the ICP for GaN is:



$$\mathbf{2GaN + 3Cl_2 \rightarrow 2GaCl_{3(g)} + N_2}$$

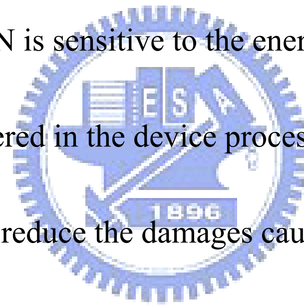
Also for AlGaN, the reaction below happens:



$$\mathbf{Al{+}3Cl_2 \longrightarrow AlCl_{3\,(g)}} \quad [17]$$

ICP plasmas are formed in a dielectric vessel encircled by an inductive coil into which RF power is applied. A strong magnetic field is induced in the center of the chamber. Because the circular region of the electric field exists concentric to the coil, the magnetic field generates high-density plasma. The plasma diffuses from the generation region and drifts to the substrate at low ion energy and low pressure (≤10mTorr). Thus, ICP etch is expected to produce low damage while achieving high etch rates. [15]

Dry etch has been used for patterning of the photonic devices and optoelectronic devices, but the surface of GaN is sensitive to the energetic ion bombardments and the thermal degradations encountered in the device processing. It is of great important to the semiconductor industry to reduce the damages caused by the dry etch process.



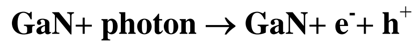
3.1.2 Photo-enhanced chemical etch

Recently, many researchers have demonstrated photo-enhanced wet etch processes for GaN films with etch rate as high as 400nm/min [13,18]. It overcomes the disadvantage of damages caused by the dry etch process.

The photo-enhanced wet etch technique is a very promising method to achieve low-damage process for GaN-based electric and opto-electronic devices. But it also needs to achieve smoother surface for devices. It is shown that a smooth etch occurs for very low solution concentration under conditions that result in a diffusion-limited

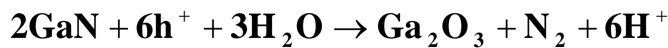
etch process. The surface roughness of 1.5nm and 50nm/min etch rate are obtained using KOH solution and Hg arc lamp. [13]

The reaction in the UV enhanced wet etch gives the photoexcitation of GaN, production of electron-hole pairs occurred when λ≤365nm. [19]



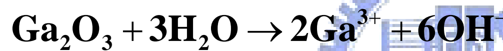
$$\mathbf{GaN + photon \rightarrow GaN + e^{-} + h^{+}}$$

The holes oxidize the GaN to Ga_2O_3



$$\mathbf{2GaN + 6h^{+} + 3H_2O \rightarrow Ga_2O_3 + N_2 + 6H^{+}}$$

The Ga_2O_3 then dissolves into the solution that can be either acidic or basic solution, but not neutral solution.



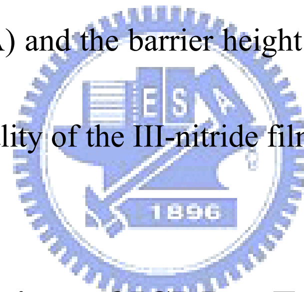
$$\mathbf{Ga_2O_3 + 3H_2O \rightarrow 2Ga^{3+} + 6OH^{-}}$$

In this work, we report the smoother wet etched surface of nitrides. It decreases the damages as compared to the dry etch process. The samples used in this study were n-GaN, UID (un-intentionally doped)- AlGaN and UID-InGaN grown on sapphire substrates by metal-organic chemical vapor deposition (MOCVD, Emcore D75). The electron concentrations were $1x10^{17}$ cm^{-3}, $2x10^{17}$ cm^{-3} and $1x10^{17}$ cm^{-3}, respectively. The inductively coupled plasma (ICP) etcher was used to etch III-nitrides with Cl_2/N_2 as the etching gases. The power level used for the etching was up to about 1200 W. The flow rate of the Cl_2/N_2 gases was kept at 10/10 sccm for all samples. A laboratory-built PEC etcher that was composed of a UV light source and an etch tank

was used to perform the wet etch process. Figure 3-2 is the schematic diagram of the laboratory-built PEC wet etcher used. The etching solution used in PEC etch process was $KOH_{(aq)}$ with molar concentrations ranging from 0.02 M (mol per liter) to 0.10 M. Etch rate (measured by α-step) and surface morphology (measured by atomic force microscopy) were used as the indexes for process parameter optimization for both ICP and PEC etch process. The optimal etch parameters were then used as the hybrid etch conditions. For ICP etch, the optimum condition is as described below: the Cl_2/N_2 flow rate was 10/10 sccm, the ICP/RF powers were 600/100 W, the chamber pressure was 2 mtorr. For the PEC wet etch, the optimum condition is under 100 mW/cm^2 of UV light exposure in 0.04 M of $KOH_{(aq)}$. To perform a hybrid etch process, all samples were etched under the optimal ICP etch process for 300 s, and then etched under the optimal PEC wet etch process for 5 to 90 min. Table I summarizes the materials and treatments used in this study. After hybrid etch process, Ohmic contact metal (Al) and Schottky contact metal (Ni/Au) were deposited by e-beam evaporator through a stencil mask on the samples without any annealing. After metallization, Schottky diode characterizations were performed by HP4145. The ideality factors (n) and the barrier heights (Φ_{Bn}) for these diodes were calculated using:

$$J = A^{*}T^{2}\exp\left(\frac{-e\phi_{Bn}}{kT}\right)\exp\left(\frac{eV_{a}}{nkT}-1\right) \quad (1)$$

The equation is the forward-bias *I-V* characteristics for a Schottky diode, where A^* is referred to as the Richardson constant, V_a is the applied voltage during measurement, k is Boltzmann's constant, T is the temperature in Kelvin. The ideality factors should be in the range of 1 to 2 which corresponds to the thermionic mechanism and tunneling mechanism, respectively. Unity is expected as an ideal value for an ideal Schottky diode. The further away from unity the value is, the less ideal the Schottky contact is. The reverse breakdown voltage (V_b, voltage when the leakage current reaches 10^{-4} A) and the barrier height (Φ_{Bn}) extracted from eq. (1) are used as indexes of surface quality of the III-nitride films.

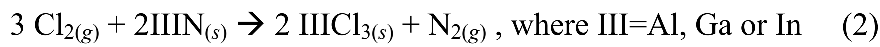


3.2 Parameter Optimizations of ICP Dry Etch and PEC Wet Etch

In this section, the condition optimization of the nitrides etched by the ICP dry etch and PEC wet etch technique will be discussed in detail.

The etching characteristics of $Al_{0.35}Ga_{0.65}N$, $In_{0.37}Ga0_{.63}N$ and GaN materials were first evaluated. Figure 3-3 shows the etch rate as a function of the ICP power for $Al_{0.35}Ga_{0.65}N$, $In_{0.37}Ga_{0.63}N$ and GaN; the ICP power was up to 1200 W and the other parameters are as indicated in the plot. The etch rates were approximately linear for all three materials at ICP power below 600 W and become saturated at ICP power higher than 600 W. In the linear region, there was no big difference in the etching rate between these materials. However, in the saturation region, the etch rates show a

relationship of $R_{InGaN} > R_{GaN} > R_{AlGaN}$, where R_{InGaN}, R_{GaN} and R_{AlGaN} are the etch rates for InGaN, GaN and AlGaN, respectively. This result is consistent with those of previous reports. [20-21] This is due to the lower formation energy for $InCl_3$ than those for GaN and AlGaN. The total chemical reactions for nitrides etched by chlorine-based ICP can be expressed as eq. (2), and the changes in Gibb's free energy for the end chlorides can be calculated as below in eq. (2) to eq. (5):



$$3\ Cl_{2(g)} + 2IIIN_{(s)} \rightarrow 2\ IIICl_{3(s)} + N_{2(g)}\ ,\ \text{where III=Al, Ga or In} \quad (2)$$

$$InCl_{3(g)}: \quad \Delta G^0_{InCl3} - \Delta G^0_{InN} = -128.4-(-4.2) = -124.2\ kcal/mol \quad (3)$$

$$GaCl_{3(g)}: \quad \Delta G^0_{GaCl3} - \Delta G^0_{GaN} = -108.7-(-26.4) = -82.3\ kcal/mol \quad (4)$$

$$AlCl_{3(g)}: \quad \Delta G^0_{AlCl3} - \Delta G^0_{AlN} = -150.3-(-68.6) = -81.7\ kcal/mol \quad (5)$$

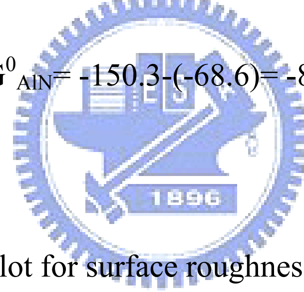
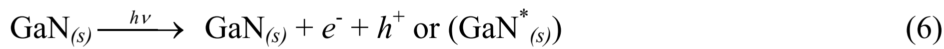


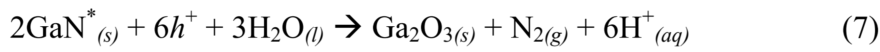
Figure 3-4 is the shows plot for surface roughness as a function of the ICP power. The surface roughness does not show large variation for all samples and is less than 3 nm in all cases. Overall, the surface roughness increases with the ICP power. The increasing roughness as ICP power increases could be due to the combined effect of the energized ion bombardment. The higher ICP power might cause more physical bombardment of the energized ions on the samples, which gives rougher surfaces.

Figure 3-5 is the etch rate *vs.* KOH concentration for $Al_{0.15}Ga_{0.85}N$ and GaN under PEC etch. The etch rates are increasing functions of $KOH_{(aq)}$ concentration. In contrast to the ICP etch, the etch selectivity is much higher for PEC etch. Further, the

relationship of the etch rates is $R_{AlGaN} > R_{GaN}$, which is opposite to that of ICP etch. This implies that different mechanisms apply for the ICP dry etch and the PEC wet etch. The mechanism for the PEC wet etch has been proposed by Bardwell *et al.* as below. [19]

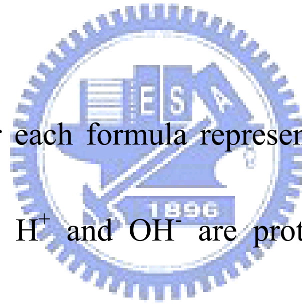


$$GaN_{(s)} \xrightarrow{h\nu} GaN_{(s)} + e^- + h^+ \text{ or } (GaN^*_{(s)}) \quad (6)$$

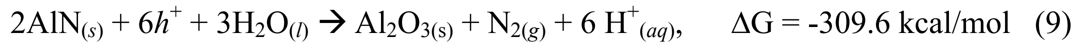


$$2GaN^*_{(s)} + 6h^+ + 3H_2O_{(l)} \rightarrow Ga_2O_{3(s)} + N_{2(g)} + 6H^+_{(aq)} \quad (7)$$

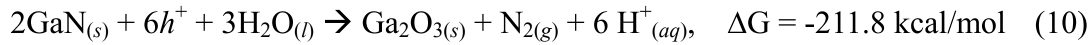
$$Ga_2O_{3(s)} + 3H_2O_{(l)} \rightarrow 2Ga^{3+}_{(aq)} + 6OH^-_{(aq)} \quad (8)$$



The suffixes (*l*, *s* and *aq*) for each formula represent liquid phase, solid phase and aquiform phase, respectively. H^+ and OH^- are proton and hydroxide. As GaN is illuminated by UV photons, the electrons (e^-) and the holes (h^+) are generated in the conduction band and valence band simultaneously. The excited GaN (GaN^*) served as an electron donor and reacts with the h^+ (empty orbital in H_2O molecule) in the solution near the surface. This also explains why p-GaN could not be etched by PEC wet etch without applied bias voltage. The $Ga_2O_{3(s)}$ produced was then dissolved into the solution. The mechanisms of the rate-determining steps for AlN and GaN PEC wet etch are as shown below in eq. (9) and eq. (10).



$$2AlN_{(s)} + 6h^{+} + 3H_2O_{(l)} \rightarrow Al_2O_{3(s)} + N_{2(g)} + 6\ H^{+}_{(aq)}, \quad \Delta G = -309.6\ \text{kcal/mol} \quad (9)$$



$$2GaN_{(s)} + 6h^{+} + 3H_2O_{(l)} \rightarrow Ga_2O_{3(s)} + N_{2(g)} + 6\ H^{+}_{(aq)}, \quad \Delta G = -211.8\ \text{kcal/mol} \quad (10)$$

Again, the calculated free energy for each reaction matches the experimental results, the more negative free energy in the reaction corresponds to the higher etch rate.

The etch rate of AlGaN increases with the $KOH_{(aq)}$ concentration, but the etch rate of GaN does not increase as fast as the etch rate of AlGaN with the $KOH_{(aq)}$ concentration under such intense UV exposure. Figure 3-6 shows the roughness for the samples after PEC wet etch at different KOH concentrations. The roughness remained the same after the samples were PEC wet etched at different KOH concentrations. The fact that AlGaN was not as rough as that of GaN could be explained by eq. (6), eq. (7) and the diffusion limit mechanism (UV-intensity/$[OH^-]$ ration > 1 W/cm^2)[22]. Because the etch rate of AlGaN is higher than that of GaN, the reactant concentration within the boundary layer for the AlGaN should be less than that of GaN. There was less reactant near the surface of AlGaN and hence it was less rough.

3.3 Hybrid etch for AlGaN and GaN

From the study in the previous section, the optimum etching conditions for the ICP dry etch and the PEC wet etch process were obtained. ICP etch of 600 W power and PEC etch with 0.04 M $KOH_{(aq)}$ were selected as the optimal conditions for the hybrid etch technique. A suitable etch rate and acceptable surface roughness were obtained using these conditions. In the hybrid etch, all samples were first etched by ICP under the same conditions with 600 W of ICP power, 100 W of RF power, and 10/10 sccm of Cl_2/N_2 flow rate, and then treated by PEC wet etch for different periods of time. Figures 3-7 and 3-8 are the current-voltage (I-V) plots of the Schottky diodes for GaN and $Al_{0.15}Ga_{0.85}N$, respectively. To distinguish the minute differences in the reverse part of the I-V curves, the logarithms of the absolute values of the currents were determined. The barrier heights (Φ_{Bn}) and ideality factors (n) were then calculated from these curves.

Figure 3-9 and 3-10 show the barrier heights (Φ_{Bn}) and the ideality factors (n) for the Schottky diodes after the hybrid etch for GaN and AlGaN materials, respectively. These two parameters were extracted from the forward part of an I-V curve of a Schottky diode. After the samples were treated by ICP etch, the barrier heights for both $Al_{0.15}Ga_{0.85}N$ and GaN were approximately 0.65 eV. As the ICP-etched samples

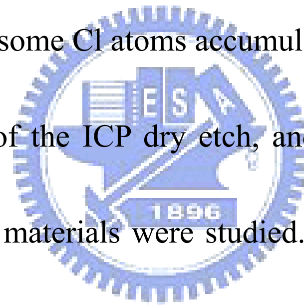
were treated by PEC wet etch, the barrier height increased with the increasing etch time and saturated after about 10 min of PEC wet etch. The same phenomena were observed in both $Al_{0.15}Ga_{0.85}N$ and GaN diodes.

Similar results were observed in the reverse breakdown voltages. The breakdown voltages of the as-grown GaN and $Al_{0.15}Ga_{0.85}N$ were 7.56 V and 10.20 V, respectively. After IPC etch treatment, the breakdown voltages were 0.75 V and 4 V for GaN diode and $Al_{0.15}Ga_{0.85}N$ diode, respectively. Further treatment by PEC wet etch after ICP etch recovered the breakdown voltage of the diodes to higher values. As shown in Fig. 3-11 and Fig. 3-12, when the PEC wet etch time increased, the breakdown voltages of the diodes recovered to higher values. Based on the Schottky characteristics observed for the GaN films, it takes approximately 30 min for the PEC wet etch (0.04 M) to recover the barrier heights and breakdown voltages. The PEC wet etch rate is about 0.5 nm/min when $[OH^-]$ is 0.04 M, the depth of damages caused by ICP etch is then estimated to be at least 150 Å (0.5 nm/min x 30 min). After removal of 150 Å of GaN from the surface, the electrical properties of the diodes did not show any improvement with further wet etch treatment.

As already known, the ICP etch is composed of two mechanisms; one is chemical reaction, the other is physical bombardment. The energetic ion bombardment can cause surface damages as deep as 1000 Å and degrades the electrical and optical

properties. [23-24] In this study, the interface states on the damaged surface induced by ion bombardment caused degradation of the Schottky properties including higher ideality factors (n), lower barrier height (Φ_b) and lower breakdown voltages (V_B). As the interface states were removed, the barrier height of the diodes was recovered. Under the conditions of high intensity of UV exposure and low $KOH_{(aq)}$ concentration, the PEC etch can etch the damaged region away without creating further damage. Due to the diffusion limit mechanism of the PEC wet etch, a smoother fresh surface with fewer interface states can be obtained. Figure 3-13 shows the AFM images of the surface morphologies of GaN after various amount of PEC wet etch treatment. The as-grown GaN film had a very smooth surface. After ICP etch, the etch pits appeared at the surface. After further treating by PEC etch, the surface became smoother, even as smooth as the as-grown film. The damages caused by ICP etch at different etching times were also investigated. Figure 3-14 shows the properties of the GaN diodes treated by ICP etch. The barrier heights of the diodes decrease rapidly for the first 50 s. After that, the barrier heights remain almost constant with the increase of the etching time. This implies that the surface damages were primarily created at the early stage of the ICP treatment. Another possible reason for the Schottky property recovery is the passivation effects of Ga_2O_3 formed on the surface during PEC wet etch. [25] To verify whether the passivation effect occurred, the as-grown epitaxy films were

treated by PEC etch under the same condition as that of the hybrid etch. The result is as shown in Fig 3-15, the barrier heights and ideality factors almost remain constant with increasing PEC wet etch time. This implies that the passivation effect does not play a key role in the improvement of the damaged surface, and the improvement is caused by the removal of the damaged layer by PEC wet etch. We think there must be other factors that affect the electric properties, like residual Cl and its compounds. According to the SIMS data, as shown in Fig. 3-16, there exist residual Cl atoms near the surfaces of both GaN and AlGaN after etch. The ESCA spectra, Fig. 3-17 and Fig. 3-18, also show that there are some Cl atoms accumulating near the surface.\



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In summary, the effects of the ICP dry etch, and PEC wet etch and the hybrid ICP/PEC etch on GaN-based materials were studied. When the ICP power is higher than 600 W, the etch rates for nitrides show a relationship of AlGaN > GaN > InGaN for Cl_2/N_2-based ICP etch and the etch rates increase with the ICP power up to 1200 W. As for the PEC wet etch, the etch rate of AlGaN is higher than that of GaN, and due to the diffusion limit mechanism, the etch rate increases with the $KOH_{(aq)}$ concentration under 100 mW/cm^2 of UV exposure. The calculated Gibb's free energy for the ICP etch and the PEC wet etch matches the experimental results. Based on the DC characteristics of the Schottky diodes after ICP etch, the depth of the damages caused by the ICP etch was at least 150 Å under our experiment conditions. This

study shows that a nearly damage-free surface can be obtained under hybrid dry ICP etch and wet PEC etch; this could be a very practical technique for the fabrication of electronic devices such as gate recess in the HEMTs.

3.4 References in Chapter 3

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