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FABRICATION OF NOVEL III-N AND III-V MODULATOR STRUCTURES
BY ECR PLASMA ETCHING

S. J. Pearton⁽¹⁾, C. R. Abernathy⁽¹⁾, J. D. MacKenzie⁽¹⁾, J. R. Mileham⁽¹⁾,
R. J. Shul⁽²⁾, S. P. Kilcoyne⁽²⁾, M. Hagerott-Crawford⁽²⁾,
F. Ren⁽³⁾, W. S. Hobson⁽³⁾ and J. M. Zavada⁽⁴⁾

⁽¹⁾University of Florida, Gainesville FL 32611

⁽²⁾Sandia National Laboratories, Albuquerque NM 87185

⁽³⁾AT&T Bell Laboratories, Murray Hill NJ 07974

⁽⁴⁾US Army Research Laboratories, RTP, NC 27709

ABSTRACT

Quantum well microdisk laser structures have been fabricated in the GaN/InGa_N, GaAs/AlGaAs and GaAs/InGaP systems using a combination of ECR dry etching (Cl₂/CH₄/H₂/Ar, BCl₃/Ar or CH₄/H₂/Ar plasma chemistries respectively) and subsequent wet chemical etching of a buffer layer underlying the quantum wells. While wet etchants such as HF/H₂O and HCl/HNO₃/H₂O are employed for AlGaAs and InGaP, respectively, a new KOH-based solution has been developed for AlN which is completely selective over both GaN and InGa_N. Typical mask materials include PR or SiN_x, while the high surface recombination velocity of exposed AlGaAs (~10⁵ cm·sec⁻¹) requires encapsulation with ECR-CVD SiN_x to stabilize the optical properties of the modulators.

INTRODUCTION

Whispering gallery-mode microdisk lasers have optical modes strongly confined in a direction perpendicular to the wells comprising the disk⁽¹⁾. Various methods have been reported for directional coupling of light output from these lasers, including patterned asymmetries in the shape of the microdisk resonators⁽²⁾, and use of a double-disk structure.⁽³⁾ A variety of different materials systems have been utilized to realize these low-threshold devices, including InGaAs/InGaAsP,⁽¹⁻³⁾ InGaAs/InGaP, GaAs/AlGaAs⁽⁴⁾ and ZnSe/ZnCdSe.⁽⁵⁾

There is interest in the use of GaN and related alloys for blue/UV emitters and detectors, sparked mainly by the development of highly luminescent light-emitting diode.⁽⁶⁾ To date, the requisite dry and wet etching processes or indeed the growth of the necessary InGa_N quantum wells have not been available to produce a microdisk structure in the nitrides. In this paper we report growth of a GaN-InGa_N multiple quantum well layer structure by Metal Organic Molecular Beam Epitaxy (MOMBE), and a controlled two stage etching process for fabrication of a micro-resonator.

EXPERIMENTAL

The processing of the microdisk lasers is conceptually quite simple and is illustrated in Figure 1 for an optically pumped device. A photoresist, dielectric or metal mask is used during dry etching to form a 1.3μm high cylinder. A selective wet chemical etch (selective for either InP, InGaP or AlN over InGaAsP, GaAs, or GaN respectively, depending on the

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materials system being employed) is then used to undercut the material under the quantum wells, leaving a narrow support post which minimizes interference with generation of the whispering gallery modes around the top disk (see Figure 1 (c)).

For the GaN/InGaN system we need to perform a wet etch undercut to form the disk, but the normal substrate used for growth (Al_2O_3) is extremely difficult to etch. To circumvent this, we grew the InGaN/GaN quantum wells on a AlN buffer layer $\sim 0.5\mu\text{m}$ thick. We then need to develop a selective wet etch for AlN over the GaN/InGaN.

Figure 1. Schematic diagram of the process for fabricating an optically pumped microdisc laser.

RESULTS AND DISCUSSION

After lithographically patterning circular photoresist masks, microcylinders are formed by ECR plasma etching at 170°C in a $\text{Cl}_2/\text{CH}_4/\text{H}_2/\text{Ar}$ discharge (flow rates of 10, 3, 15 and 10 sccm, respectively) at 1 mTorr pressure and a microwave power of 850W. Additional rf power of 150W was applied to the sample position to increase ion energy to $\sim 175\text{eV}$ and thereby improve etch anisotropy. These conditions produce smooth non-selective etching of all of the nitride layers in the structure, at rates between $\sim 1,000\text{\AA}/\text{min}$ for AlN and $2200\text{\AA}/\text{min}$ for GaN.

We noticed that during lithographic patterning of AlN samples, exposure to AZ400K developer produced substantial etching. The active ingredient in the developer in regard to etching AlN is KOH. A systematic study of the wet etching characteristics of AlN in AZ400K was performed to establish the rate-limiting step and dependence of etch rate on material quality.

Figure 2 shows Arrhenius plots of etch rate for three different samples. The etching is thermally activated with the same value of activation energy, $\sim 15.5 \pm 0.4 \text{ kcal/mol}$, in all cases. Moreover, the rate was not sensitive to agitation and was dependent on the etchant concentration. These are characteristics of reaction-limited etching⁽⁷⁻⁹⁾ of the form

$$R = R_0 \exp(-E_a/kT) \quad (1)$$

where R is the etch rate at temperature T , E_a is the activation energy of the etch reaction, k is Boltzmann's constant, and R_0 is a constant which can be thought of as an attempt frequency for the reaction between $(OH)^-$ ions and the AlN surface. we assume, therefore, that R_0 is strongly dependent on material quality because the AlN with poorer crystallinity will have a greater number of weakened or defective bonds which are more susceptible to attack by the developer solution. Thus in our AlN, the absolute etch rate is governed by the crystalline quality of the epitaxial layer, but the etching has the same activation energy.

FIG. 2. Arrhenius plot of etch rate of three different AlN samples in AZ400K. The polycrystalline AlN is denoted by (●), while the single-crystal data are denoted by (▲) and (■), where the slowest etch rates were obtained for the higher quality AlN.

Figure 3 shows the time dependence of etch depth for the polycrystalline AlN in pure AZ400K solution and in a mixture of 5:1 H_2O :AZ400K solution. There is a linear dependence in both cases and the etch rate was dependent on the etchant concentration which again is characteristic of rate-controlled, rather than diffusion-controlled, etch reactions. If the etch were the latter, one would expect a $t^{1/2}$ dependence for etch depth and an activation energy in the 1-6 kcal/mol range.⁽⁸⁾

FIG. 3. Time dependence of etch depth of a polycrystalline AlN sample in pure AZ400K and in a 5:1 solution of H_2O :AZ400K at 23 °C. The etch rate is dependent on concentration as expected for rate-controlled etchants.

In processing the microdisks, the AlN is then selectively wet etched in AZ400K developer solution for ~30 min. at 85°C to produce an undercut and leave the InGaN/GaN disk supported on an AlN pedestal. The active ingredient in the developer solution is KOH which etches AlN at a rate of ~330Å/min at 85°C. There was no measurable etching of GaN or InGaN under these conditions. We have found that the AlN wet etch rate is strongly dependent on the crystalline quality of the material, with much higher rates (up to ~1 µm/min for polycrystalline AlN Figure 4 shows an SEM micrograph of a typical microdisk after this two step etching procedure. It is important to note that other layer structures are also feasible, including use of AlGaIn/InGaIn quantum wells on a thick GaN buffer. However there have been no reports of a controlled selective etch for GaN with practical rates at this stage.

Figure 4. SEM micrograph of a typical microdisk after two-step etching.

To integrate a quantum wire laser on top of the microdisk is relatively straightforward, and this would allow the quantum wire laser to be pumped by the microdisk since emission from the latter is both within the plane of the quantum wells and vertically. A schematic of the process sequence is shown in Figure 5. After formation of the sidewalls, the microdisk laser is created by a combination of dry and wet etching, and then the quantum wire laser fabrication is completed using the sidewalls as an etch mask.

While the microdisk laser structures generally operate well without the need for surface passivation, AlGaAs for example is known to be prone to oxidation and therefore to degraded surface characteristics. This is a particular problem for laser facets⁽¹⁰⁾ or for surface recombination near the base-emitter junctions of HBTs, especially for small emitter dimensions.⁽¹¹⁾

Figure 5. Process for fabrication of an integrated quantum wire/microdisk laser structure.

The diode diameter dependence of current density in mesa p-n junctions was used to measure surface recombination velocities (S) for both InGaP and AlGaAs. For InGaP, S values of $4\text{--}5 \times 10^4$ cm/s were obtained for both wet- and dry-etched mesas, and the surface was relatively insensitive to changes resulting from annealing or plasma exposure. Surface passivation by $(\text{NH}_4)_2\text{S}_x$ treatment reduced the recombination velocity by a factor of 2. By contrast, AlGaAs displayed a strong sensitivity to the type of processing steps used in photonic and electronic device fabrication, with values of S as high as 9×10^5 cm/s after low temperature annealing, and as low as 3.7×10^4 cm/s after sulfide passivation. (Table I). Therefore, for structures involving AlGaAs, it is necessary to sulfide passivate the surface and then cover the surface with CVD SiN_x to provide long-term stability. This is not necessary when using GaAs/InGaP structures, and we have not found it necessary to employ surface passivation techniques for the nitrides.

SUMMARY

A fabrication scheme for nitride-based microdisk structures has been developed that employs a new selective wet etchant for AlN and ECR plasma etching with a $\text{Cl}_2/\text{CH}_4/\text{H}_2/\text{Ar}$ chemistry. Further work is needed to extend the capabilities of this process to devices involving GaN buffer layers.

TABLE I. Surface recombination velocities (in $\text{cm}^2 \text{s}^{-1}$) for InGaP and AlGaAs $p-n$ junction samples as a function of their processing treatments.

	Initial	250 °C, 60 min	SF ₆	(NH ₄) ₂ S _x
InGaP				
Wet etched	4.4×10^4	4.9×10^4	4.6×10^4	2.2×10^4
Dry etched	5.2×10^4	5.1×10^4	4.9×10^4	2.6×10^4
AlGaAs				
Dry etched	9.0×10^4	9.1×10^5	8.4×10^5	3.7×10^4

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