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# $N_2/H_2O$: A New Gas Mixture for Deep Groove Ion Beam Etching of Long Wavelength Quaternary Mushroom Type Laser Structures

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A new technique of structuring waveguides in InGaAsP/InP using $N_2/H_2O$-RIBE has been developed. Deep grooves have been etched into InP and quaternary material with featureless smooth bottom. The dependence of the etchrate on the angle of incidence has been studied. A surface layer with altered optical and electrical properties is created by the etching process and investigated by ellipsometry and C(V)-profile measurements. The preheat process under a $H_2/PH_3$-stream prior to the epitaxial regrowth of InP in a hydride-VPE-system leads to a complete recovery of the surface properties.

## 1. INTRODUCTION

Dry etching is now a well established technique for the fabrication of electronic and optoelectronic devices with dimensions in the micron and submicron range. Especially Ion Beam Etching is a useful tool because the various parameters which control the etching characteristic,e.g. ion energy, ion current density, angle of ion incidence, can be set independently from each other.

While in the AlGaAs-system ion beam etching with Argon as working gas gives reasonably smooth surfaces in the InGaAsP-system problems arise with surface morphology. $N_2$, $N_2/O_2$, $Ar/O_2$ have been reported as working gases for etching InP and quaternary InGaAsP[1]. The published results show smooth surfaces. The reported selectivity, however, between masking material and semiconductor is insufficient for etching deep structures (>2μm) with thin (< 200nm) and nonshrinking masks. Thus, we have developed a new etch process on the basis of N2/H2O, which does not suffer from these deficiencies and is well appropriate for etching of waveguides etc..

## 2. EXPERIMENTAL

### 2.1 ETCHING EQUIPMENT

Ion beam etching has been performed in a Technics MIM II consisting of a Kaufman type ion gun and a stainless steel chamber, which can be pumped to a base pressure $< 10^{-6}$ Torr. The $N_2$-gas is admitted through the ion gun, while the $H_2O$-vapor is fed directly into the etch chamber.

At working conditions the $N_2$-gas pressure amounts to $1x10^{-3}$ Torr in the gun and to $4x10^{-4}$ Torr in the chamber. The $H_2O$-gas-flow is adjusted to reach a partial pressure of $1x10^{-5}$ Torr in the rezipient. Throughout our experiments we etched with an ion beam current density of 1 $mA/cm^2$ and an ion energy of 1000 eV. The samples to be etched were fixed with photoresist on a water cooled stage ($5^oC$ to $70^oC$, usually $14^oC$). The angle of ion beam incidence was varied between $0^o$ and $80^o$ During etching the stage was rotated by 2 rpm.

## 2.2 ETCHING PROPERTIES

When starting an ion gun water partial pressure can rise up to some $10^{-5}$ Torr, because water molecules desorb from the wall due to ion bombardment.

The uncontrolled presence of water vapor strongly influences the etching process. The variation of the water content leads to nonreproducible results. Therefore we fed water directly in a well controlled way into the etch chamber (not through the gun!), which has two main advantages over the pure $N_2$ etching system:

1.) The etched InP surfaces get really featureless smooth.
2.) The titanium etch mask stability for $N_2/H_2O$ is improved even compared to the $N_2/O_2$ gas system.

The reason for that behaviour is the high sticking probability of water molecules on the sample.

At a water partial pressure of $1 \times 10^{-5}$ Torr in the chamber and a $N_2$-pressure of $1 \times 10^{-3}$ Torr in the gun the impingement rate of water molecules is of the same order of magnitude as the ion current density at the substrate. So oxidation and nitridation seem feasible on the InP surface under influence of the energetic $N_2$-ions leading to comparable sputter rates of In and P. The continuous coverage and oxidation of the titanium mask by thermal water molecules (not by energetic $O_2^+$ ions as in the case of the $Ar/O_2$ and $N_2/O_2$ gas system) seems to be responsible for the highest etch selectivity of titanium against InP reported up to now (etchrate ratio InP:Ti > 30:1). Fig. 1 shows the measured etchrate in InP with $N_2/H_2O$ as a function of the angle of incidence of the ions. We observe a rather slow decrease of the etchrate with increasing angle of incidence $\alpha$ compared to the situation of pure $N_2$[1].

Figs. 2 a and b show some typical SEM pictures taken for the case of vertical incidence of the ions. We observe the well-known trench formation. The side wall ripples (see also Fig. 3) reproduce the roughness of the titanium lift off mask (note the featureless smooth bottom). In Fig. 3 a and b we reproduce structures etched under $\alpha = 20^{0}$, a favourable angle for producing waveguide- and laser-structures (no trench, smooth bottom).

The etchrate was proved to be constant in the temperature range from $5^{0}$ to $70^{0}$C. The smoothness of the surface, however, depends on the temperature. Up to $30^{0}$C the etched surface is perfectly smooth, whereas at $70^{0}$C a slight roughness is observed in the SEM-picture.

## 2.3 ETCH DAMAGE AND ANNEALING

The damage created by the $N_2/H_2O$ ion etching manifests itself in the optical and electrical properties of the etched sample surface. To characterize the damage and its possible annealing behaviour we performed ellipsometric (Spectro 2000, Rudolph Research) as well as C(V) Polaron profiler measurements. This was done for n- and p-type substrates before and after ion etching as well as after annealing. Fig. 4 shows the $\Psi$, $\Delta$ -graphs of an undamaged (——), damaged (---) and annealed ( ——) sample. The influence of ion etching on the optical properties is evident. The same behaviour was found for p-type material. From a fit to the $\Psi(\lambda)$, $\Delta(\lambda)$-graphs under variation of thickness and refractive index of the damaged layer we deduce a damage depth of $\sim 30$ nm[2]. This is in reasonable agreement with C(V) profiler measurements shown for n-type material (Fig. 5). The change of the carrier concentration is due to a depletion of phosphorus from the InP-surface during etching[3]. Phosphorus vacancies work as donors, which enhance the surface carrier concentration in n-type InP and compensate carriers in p-type material. A method to remove surface damage was therefor to include a source of phosphorus to reconstruct the InP surface[4]. For annealing we choose the preheat process for epitaxial regrowth in a hydride VPE-system. The etched samples were heated up to 650°C in 4 min with an adjusted $PH_3/H_2$-flow ($PH_3$ = 1,5x10$^{-2}$ atm, $H_2$ = 1 atm). This process resulted in a complete recovery of the etched surfaces. $\Psi$ ($\lambda$) and $\Delta$ ($\lambda$) of the etched and annealed sample are indistinguishable from the unetched one (Fig. 4).

For further characterization of the new etching system the etched samples were overgrown with undoped VPE-InP. The regrown material revealed the same good characteristics as InP deposited on unetched substrates, i.e. low net carrier concentration of $n \approx 3\text{x}10^{15}$ cm$^{-3}$, high electron mobility of $\mu_{77} \approx 40000$ cm$^2$/Vs and good morphology.

## 2.4 APPLICATION TO LASER FABRICATION

The applicability of the new etching system is proved on mushroom type lasers. The mesas (Fig. 6) are dry etched, the undercut etching has been done wet chemically. The regrowth with InP has been done by mass transport. The devices show better yield and performance as compared to completely wet chemically etched structures.

## 3. CONCLUSION

A new RIBE technique has been developed on the basis of $N_2/H_2O$. Smooth etched surfaces are attained with a high selectivity of the etching process due to the titanium mask material. The ion generated damage of ~30 nm depth can be completely cured.

## REFERENCES


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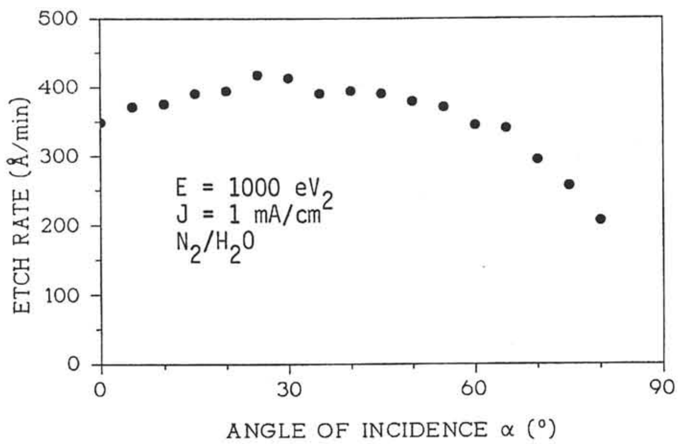


Fig. 1 Etchrate of InP versus angle of ion incidence

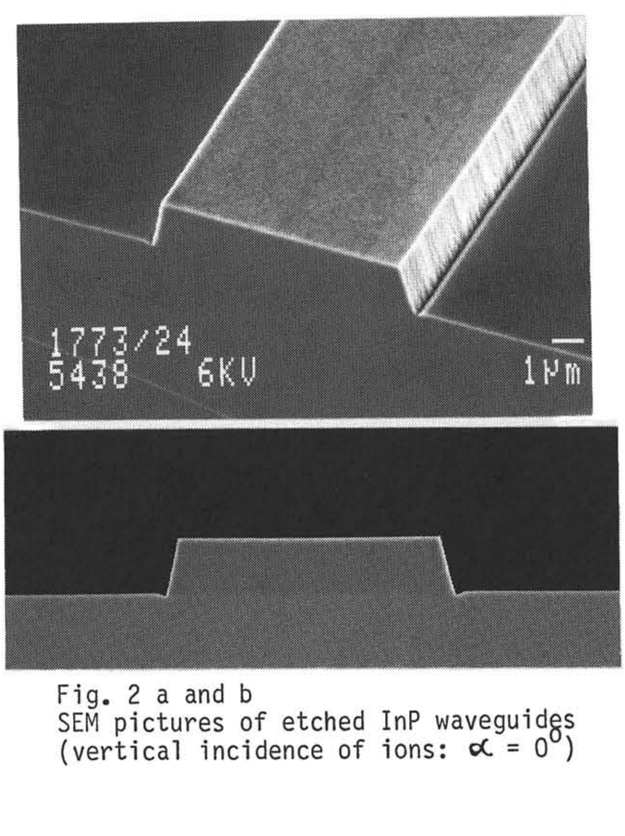


Fig. 2 a and b
SEM pictures of etched InP waveguides (vertical incidence of ions: $\alpha = 0^0$)

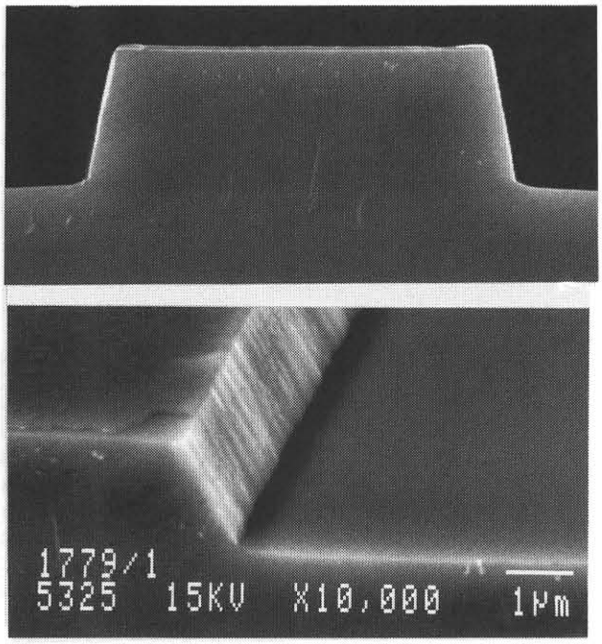


Fig. 3 a and b
as in Fig. 2 however $\alpha$ = $20^{o}$

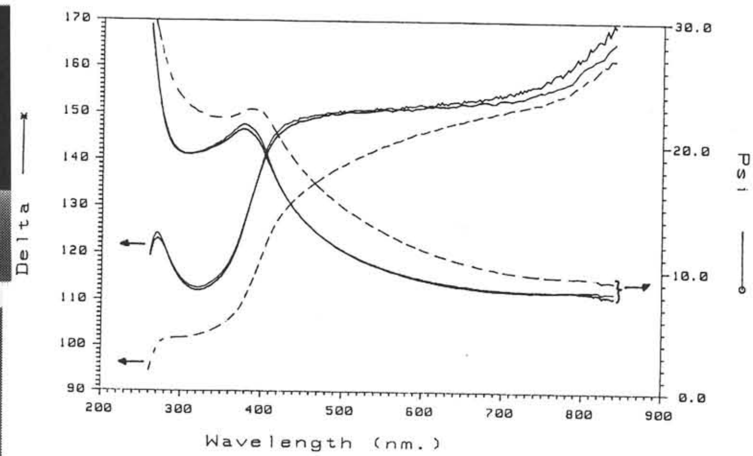


Fig. 4 $\Psi(\lambda), \Delta(\lambda)$ graphs for n-InP
(——) lower curve: undamaged surface
(----): etched surface
(——) upper curve: etched and annealed surface

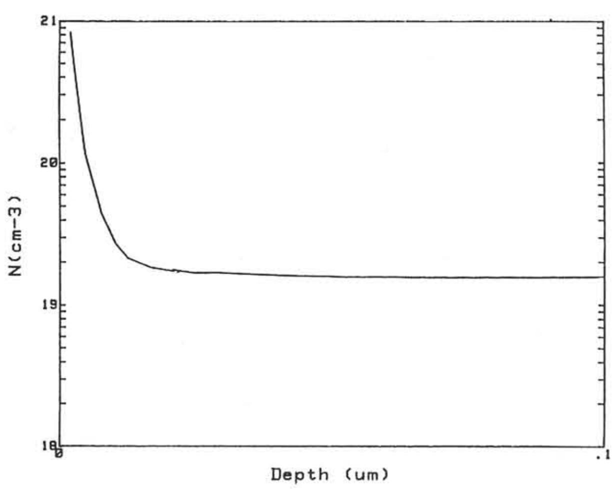


Fig. 5 Electrical carrier concentration as function of depth for an etched n-InP substrate

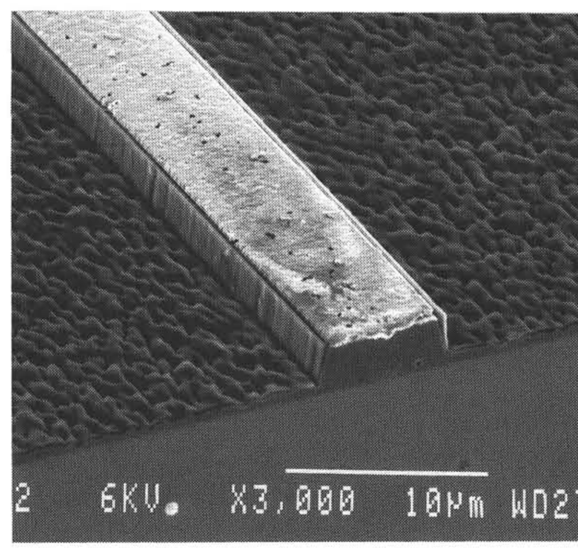


Fig. 6 Mushroom type laser structure. The waveguide was underetched to 1,5 um width and regrown by mass transport. The surface roughness is due to mass-transport and $SiO_2$ passivation.