

Photo-selective chemical etching of InAs and GaSb to manufacture microscopic mirrors

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1 Introduction

Photochemical processing of materials, particularly in semiconductors, has been a relatively well-known technique used for some time with relatively few applications [1]. However, devices demanding new processing techniques, such as MEMS [2], Leds [3] and lasers with integrated lenses or gratings, etc. have renewed interest in this technique.

The technique can be described as follows [4]: a semiconductor is immersed in an electrolyte, forming a space charge region at the solution–semiconductor interface; at the same time the semiconductor is illuminated with light (normally a laser) of energy greater than the material's band gap, creating electron-hole pairs. The electric field due to band bending at the semiconductor surface attracts one type of carrier to the surface and repels the other type of carrier towards the bulk. Normally, in n-type material, the holes go to the surface and the electrons to the bulk. In this case the excess holes increase the oxidation state of the surface atoms, increasing the etching rate. If the reaction rate is limited by the supply of photo-generated holes at the surface, the etching rate can be adjusted simply by changing the light intensity.

Since the technique uses a beam of monochromatic photons (laser) to locally control reactive interactions near the solid surface, it is particularly useful to perform localized material deposition (writing), solid doping, alloying and/or, as in our case, material removing (etching) without masking assistance [5]. Due to these unique characteristics Laser-Assisted Chemical Etching (LACE) offers advantageous distinctiveness that makes it preferred to other materials processing techniques [6].

Of particular interest in this work is the photochemical etching of semiconductors, also called LACE. This technique has been already used successfully to produce channels with approximately parabolic cross section along GaAs substrates, creating a refraction index negative gradient inside a Graded-Index Separate Confinement Heterostructure (GRIN-SCH) laser structure. This effect was used to deviate the higher order lateral modes out of the laser structure keeping only the central one [7]. In that work the feasibility of a new type of high-power coherent semiconductor laser with lateral emission was demonstrated. As far as we know this was the first time LACE was used successfully to build structures in laser diodes that simultaneously improved their power output and coherence.

In this work we show the results of applying LACE to n-type GaSb substrates to create cavities with nearly parabolic cross sectioned profiles that could be used as micro mirrors or micro lenses to concentrate or deviate light beams emitted from a surface emitting laser diode. A parabolic profile of the mirror is best suited to obtain single mode operation in an unstable resonator [8, 9].

GaSb is an interesting material for the production of lasers and photodetectors in the near and mid infrared region. However, the use of GaSb requires the development of some processing techniques such as etching and polishing. Chemical processing of GaSb is particularly

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difficult because of its reactivity, small band gap and limited solubility of the reaction products [10].

To our knowledge there are no reports on LACE applied to GaSb. There is work on the photoelectrochemical etching of GaSb in aqueous NaOH, NaCl and HCl solutions [10]. However, this method differs from that presented here in that it uses electrical current to achieve the etching process.

2 Experimental procedure

To perform LACE a simple optical system the 514 nm line of an Ar laser beam is focused on the sample surface with the aid of a lens and a mirror to deviate the light downwards to the sample surface which is immersed in the etching solution.

The approximate parabolic profile is produced by the dependence of the etch rate on the intensity of the light. Although the laser used in this work has the Gaussian intensity pattern of the T_{00} principal mode, the central part of the etched cavity closely approximates a parabola. The laser power was fixed at 10 mW. The p-type GaSb substrates were non-intentionally doped and the n-type ones were Te doped crystals with a free carrier concentration of 10^{17} cm^{-3} and 10^{18} cm^{-3} , respectively. The n-type InAs substrates were non-intentionally doped and the P ones were Zn doped with a carrier concentration of $1\text{--}3 \times 10^{16} \text{ cm}^{-3}$ and $1\text{--}3 \times 10^{17} \text{ cm}^{-3}$, respectively. In all cases the surface orientation was (100). The chemical solutions studied were 1 M HCl and 0.2 M H_2SO_4 dissolved in water. The profiles of the etched cavities were measured by means of a profilometer. The mean roughness was determined with an atomic force microscope.

3 Results

Several attempts to etch the samples at room temperature were carried out as described above. However, the p-type GaSb substrates did not show the photochemical effect. This may be due to two reasons; first the excess hole concentration created by the light is negligible compared with the equilibrium carrier concentration or second, the surface electric field in the GaSb surface drives the holes away from the surface and impedes them from participating in the chemical reaction [10].

Neither the p-type nor the n-type InAs samples could be photo-etched; a possible explanation resides in the fact that the Fermi level at the InAs surfaces is always pinned above the conduction band [11] and therefore the surface electric field pulls the holes towards the bulk and away from the chemical reaction site in all the InAs samples, regardless of their conductivity type.

A typical result of the profiles obtained in the n-type GaSb samples is shown in Fig. 1. This corresponds to a sample etched in a 0.2 M H_2SO_4 solution for 15 s.

In Fig. 1, the continuous line shows the cross section of one of the photo etchings on a GaSb-n sample made with a 0.2 M H_2SO_4 solution and a power of 10 mW and the dashed line correspond to the best fitting to a Gaussian profile it can be seen that the agreement is remarkable. The central part of this profile approximates a parabola.

The good fitting of the etched profile with a Gaussian indicates that the etching rates depend linearly on the light intensity since the power distribution of the laser is itself Gaussian.

Figure 2 shows dependence of cavity depth on etching time; the linear dependence is evident. The deviations observed are due to measurement errors and possibly also to variations of the substrate resistivity. A linear fit to these data gives an etching rate of $0.85 \mu\text{m min}^{-1}$.

In order to assess the roughness of the etched surfaces Atomic Force Microscope (AFM) images were taken. The results are shown in Figs. 3 and 4. The H_2SO_4 etchant produces the better surfaces with an RMS roughness of 11 nm. This roughness gives a surface figure better than $\lambda/10$ making these cavities suitable for use as micro-mirrors for light of around $2 \mu\text{m}$ wavelength [12, 13].

The roughness of the final surface could be due to the initial cleaning of the sample or to the etching mechanism or etching speed of the chemical solutions employed. The mirror roughness could be further decreased if a detailed study of these factors is carried out.

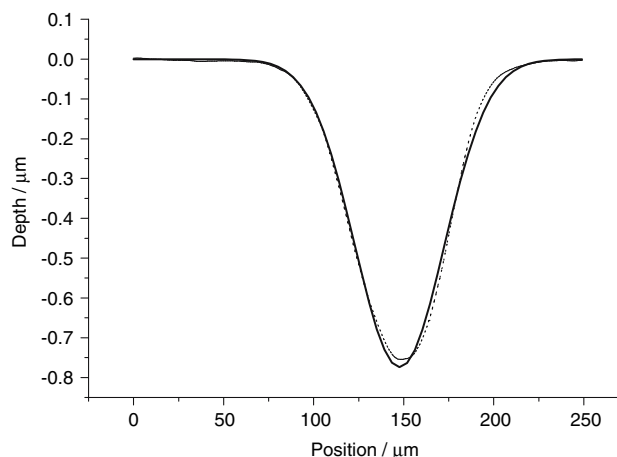


Fig. 1 (a) Etching profile of an n-type GaSb sample etched at room temperature in a 0.2 M H_2SO_4 solution during 15 s. The dashed line is the best fit to a Gaussian profile

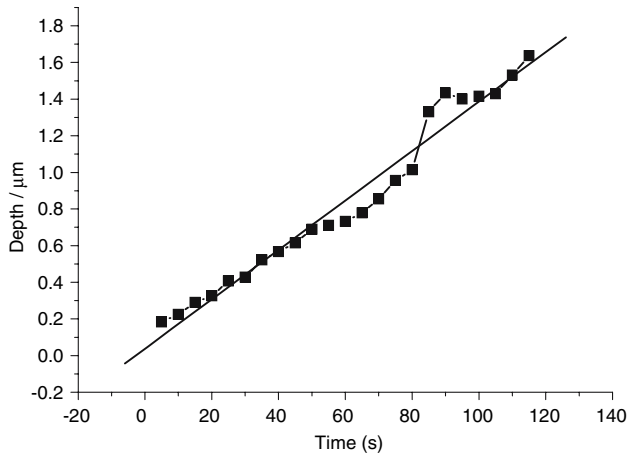


Fig. 2 Cavity depth etched as a function of etching time for a n-type (100) GaSb sample etched with an 1 M H_2SO_4 solution under illumination with a 10 mW argon laser

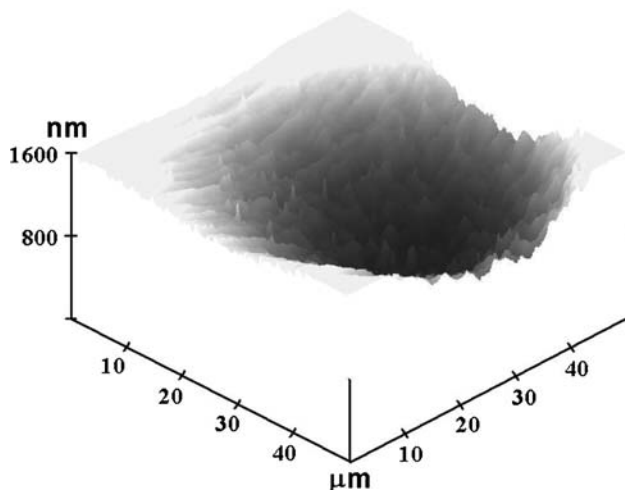


Fig. 3 AFM image of the surface of a cavity etched in a 1 M HCl solution. RMS roughness is 57 nm

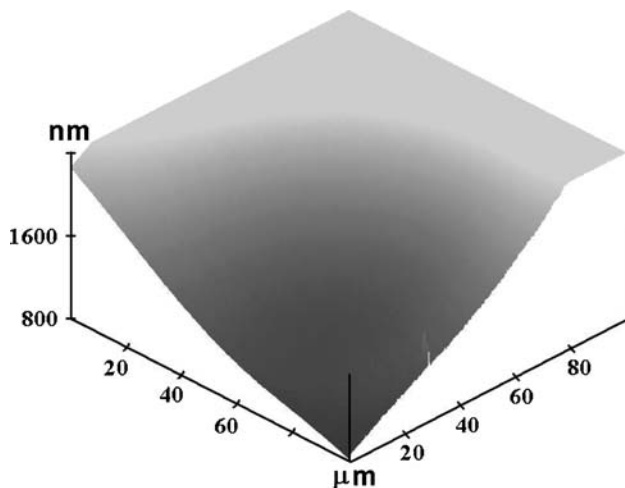


Fig. 4 AFM image of the surface of a cavity etched in a 0.2 M H_2SO_4 solution. RMS roughness is 11 nm

4 Conclusions

In this work LACE was successfully used for the first time to fabricate near-parabolic cavities on GaSb substrates. Varying the exposure time and the incident beam intensity we were able to control the etching depth. The relation between the etching time and the etching depth was linear. These encouraging results open up the possibility of fabricating monomode unstable cavities for vertical cavity surface emitting lasers (VCSEL's), using parabolic micro-mirrors to deviate the higher order lateral beam modes in a similar fashion as suggested in [14] for lateral emission lasers.

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