

Chapter 7

Technology of Cavity Fabrication for Whispering Gallery Modes Laser ($\lambda \sim 3\text{--}4\ \mu\text{m}$)

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Abstract Different wet-etching solutions based on CrO_3 ; HClO_4 and HBr for materials lattice-matched to InAs for whispering gallery modes (WGM) lasers ($3\text{--}4\ \mu\text{m}$) disk cavities fabrication were studied. In the case of WGM-laser cavity – it is crucial to have vertical and smooth sidewalls, free from roughness and other irregularities. Perfect circular disk cavities with diameter from 50 to 300 μm were obtained by using HBr -based wet-etching technology. At a total cavity height reaching 30 μm , the vertical part of the sidewalls was about 10 μm . Based on the obtained disk cavities, WGM lasers are created that produce coherent radiation with a wavelength of $\lambda \approx (3.0\text{--}3.5)\ \mu\text{m}$ in a continuous regime at 77 K and in a pulsed regime up to 125 K.

7.1 Introduction

Semiconductor lasers operating in the middle-IR spectral range are of considerable interest for numerous practical applications. In particular, these lasers constitute the basis for wireless communication systems, for some medical facilities, have promising applications in various fields of biology and ecology [1]. Many technologically and environmentally important gases (e.g. CO , CO_2 , NO_2 , NH_3 and CH_4), show strong absorption lines within the $(2\text{--}4)\ \mu\text{m}$ spectral region, therefore advanced measurement techniques that exploit MIR lasers for gas sensing applications have become well-established tools for characterizing combustible, exhaust and unhealthy gases.

In the frame of semiconductor lasers, generation of the light in active medium owing to an external energy source and providing a feedback for stimulated light emission takes place in the laser resonator. At present time there exist several principle of the forming of the light generation. For example, the Quantum Cascade

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principle (QC) is the most widely used due to possibility to increase the optical gain in the active region. The vertical cavity surface emitting design (VCSEL), with laser beam emission perpendicular from the top surface, contrary to conventional in-plane-emitting semiconductor lasers allow improvement Q -factor of the resonator. But the fabrication of these lasers is rather laboured, material-intensive and high-cost process.

In recent years, much attention has been focused on lasers with circular resonators (cavities) operating via the whispering-gallery modes (WGMs) [8]. The cavities of WGM-lasers are distinguished by a higher Q -factor and may compensate for the low gain, reduce the values of threshold current in comparison with the stripe resonator Fabry-Perot lasers, and help to achieve room temperature operation.

Referring to the acoustic phenomenon, which was observed by Lord Rayleigh under the cupola of St. Paul Cathedral in London the name “whispering gallery modes” was also coined for electromagnetic eigenmodes of circular resonators in which WGM effect exists [7].

The WGM-like modes in ring cavities and microdisks have been studied both theoretically and experimentally at shorter wave-lengths [3,4,9]. So, for visible and near-infrared semiconductor lasers WGM cavity not an optimal choice, but for the MID-IR wave range, where the nonradiative recombination is high, WGM lasers seem to be a promising devices [6].

WGM-effect may allow one to make a working room temperature laser even when the gain in the active region is small without requiring a long cavity or anti-reflection coating of the facets. The laser beam traveling close to the perimeter of the disk cavity reflects on the disk edges many times and has a great gain. Thus, it is possible to obtain laser emission from the structures with relatively small optical gain.

GaSb- and *InAs*-based heterostructures are promising materials for cavity fabrication since optoelectronic devices manufactured on such $A^{III}B^V$ compositions allow the coverage of the (2–4) μm wavelength range. Unfortunately, in comparison with the other semiconductor materials, narrow bandgap *III–V* materials are subjected to strong nonradiative recombination, which spoils their quantum efficiency, reduces net gain and prevents continuous room temperature operation. There is an opportunity to improve this situation due to the whispering gallery-effect.

It is crucial for WGM-laser disk cavity to have smooth and vertical sidewalls, free from lateral projections, cracks, and other irregularities. Therefore the purpose of our activity is to elaborate technology of cavity on $A^{III}B^V$ structure for MIR lasers. In this paper technology of disk-shaped cavity fabrication for *InAs*-based electrically pumped WGM lasers is presented.

7.2 Wet Etching Cavity Fabrication on the Base of *InAs(Sb)/InAsSbP* Heterostructure

For our experiments we chose wet-chemical etching as it is more preferable for application, especially if semiconductor material have to be removed without crystallographic damage to the surface as is the result with dry-chemical etching.

7.2.1 Wet Etching by Using $\text{CrO}_3\text{-HCl-HF-H}_2\text{O}$ Solution

A number of publications on wet etching for *InAs*-based materials are very limited and in most cases the main component in such etching solutions is the nitric acid [5].

Disadvantages of such etching are the follow: firstly, impossibility of treating multilayer heterostructures containing *InAs* and quaternary solid solutions, such as $\text{Ga}_x\text{In}_{1-x}\text{As}_{1-y}\text{Sb}_y$ and $\text{InAs}_{1-x-y}\text{Sb}_y\text{P}_x$, because of difference in rates of the red-ox reaction at the surface of layers which are different in composition, results in formation of a stepped mesa. Second, the active release of nitrogen oxides during etching results in partially blocked by gas bubbles surface and islands form at these sites, producing a rough surface.

For forming a cavity on the surface of *InAs*-based optoelectronic structure we developed an etching solution which can ensure isotropic etching of *InAs* and solid solutions (*GaInAsSb*, *InAsSbP*, and *InAsSb*) due to equal etching rates.

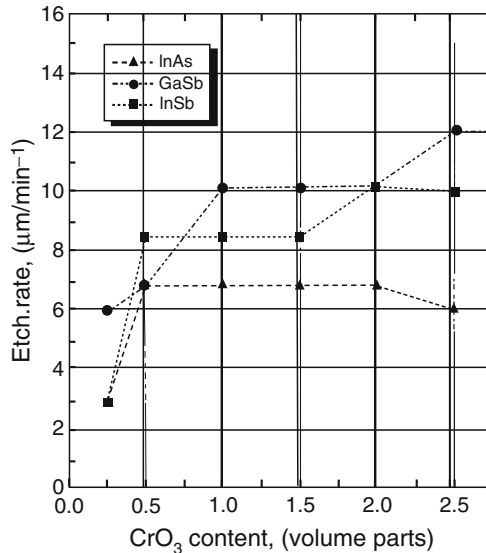


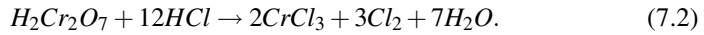
Fig. 7.1 Etching rates of binary compounds as a function of CrO_3 content in solution

For the etching experiments we used *n*-type *InAs* [100], *GaSb* [100] *InSb* [100] and structures with *GaInAsSb*, *InAsSbP*, and *InAsSb* epitaxial layers of different composition. A positive photoresist was chosen for the photolithography. Our stated aim was satisfied by using an etching solution consists of $\text{CrO}_3 - \text{HCl} - \text{HF} - \text{H}_2\text{O}$. These components are contained independently in various etchants [5]. However, the role of hydrochloric acid is altered in the combination of components being discussed. In the others etching solutions hydrochloric acid created an acidic medium.

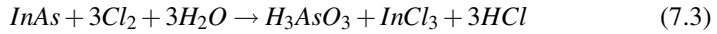
In our etching solution the hydrochloric acid participates in the reaction (7.2) to give a strong oxidant ($2Cl^0 \rightarrow Cl_2$) which can produce a high rate of red-ox reactions at the surface of the semiconductor.

Kinetics of the entire process may be very complicated and is determined by the slowest (controlling) stage. At moderate temperatures, etching is controlled by the chemical interaction stage, less frequently by the diffusion. At high temperatures the etching kinetics is usually determined by the diffusion rate. The presence of hydrochloric acid creates conditions for the rapid oxidation of the solid solution components so that layers of a quaternary solid solution having different chemical compositions can be etched at the same rate, producing a high-quality mesa. The hydrochloric acid performs two functions: it creates an acidic medium and forms an active Cl_2 oxidant (at the instant when a Cl^0 radical is released). The high oxidizing properties of the free Cl^0 radical are responsible for the high density of etching centers and can produce a polished mesa surface.

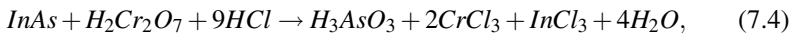
We shall analyze the red-ox reactions taking place in this particular etchant [2]:



Thus, the solution contains two strong oxidants, $Cr_2O_7^{2-}$ and Cl_2 . For *InAs* we analyze the oxidation of the solid-solution components:



(As^{-3} oxidizes to oxidation state As^{+3})



An additional process is also possible, deeper oxidation to As^{+5} :

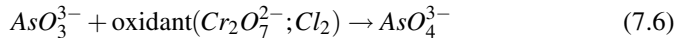


Table 7.1 *InAs*-based heterostructures etching rates in treating with $CrO_3 - HCl - HF - H_2O$ solution

Composition of structure	Etching rate, $\mu\text{m}/\text{min}$
<i>InAs</i>	6.9
<i>Ga</i> _{0.08} <i>In</i> _{0.92} <i>As</i> _{0.77} <i>Sb</i> _{0.23}	7.0
<i>Ga</i> _{0.12} <i>In</i> _{0.88} <i>As</i> _{0.81} <i>Sb</i> _{0.19}	7.0
<i>Ga</i> _{0.14} <i>In</i> _{0.86} <i>As</i> _{0.77} <i>Sb</i> _{0.23}	7.1
<i>In</i> _{1.0} <i>As</i> _{0.68} <i>Sb</i> _{0.12} <i>P</i> _{0.20}	7.0

The presence of a powerful oxidant results in deeper oxidation of the components and the limitation the etching rate by diffusion processes rather than by

red-ox processes, giving the same etching rates to layers with *InAs* and multicomponent solid solutions based on it: $In_{1-x}Ga_xSb_yAs_{1-y}$, (where $x \leq 0.2$, $y \leq 0.35$) and $InAs_{1-x-y}Sb_yP_x$ ($x \leq 0.32$, $y \leq 0.15$).

Oxidation is usually accompanied by the formation of barely soluble oxides which must be converted to solution using complexing agents. In this etching solution *HF* functions as the complexing agent.

The etching rates of binary compounds as a function of the CrO_3 content in solution $HCl - CrO_3 - HF - H_2O$ are shown in Fig. 7.1. It can be seen that the binary compounds were discussed are able to have a polished surface when the etching rates are similar and remain almost constant. The range of concentrations in which a polished surface can be obtained is indicated by the two vertical lines.

The etching rate of the quaternary solid solutions differs little from that of *InAs* (Table 7.1) and is almost independent of the composition of the solid solution, as is confirmed by a picture (Fig. 7.2(a)).

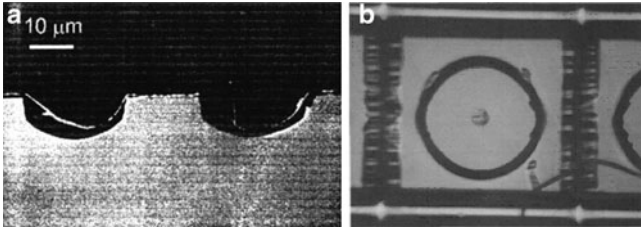


Fig. 7.2 (a) Picture of a cleaved strip-mesa section of an *InAsSbP/InAsSb/InAsSbP/InAs* heterostructure; (b) Anisotropic etching in $CrO_3 - HCl - HF - H_2O$ solution. Picture of the ellipse-shaped cavities of 100 μm in diameter

This suggests that solution composed of $CrO_3 - HCl - HF - H_2O$ allows to obtain a smooth side-wall and a polish surface for the circular cavities of 300 μm and bigger in diameter from structure containing layers of different composition solid solutions due to the equal etching rates.

However, in using this etching composition for fabrication cavities of 200 μm and below in diameter we faced with the evidence for anisotropic etching, whereby the etching rate depended on the crystallographic direction and the mesas finally acquired an ellipse-like shape (Fig. 7.2(b)).

7.2.2 Electro-Chemical Etching by Using $HClO_4-CH_3COOH$ Solution

In search of isotropic etching *InAs*-based multilayer heterostructures to obtain circular cavities 50–270 μm in diameter we have employed an electro-chemical etching technique using $HClO_4 - CH_3COOH$ mixture as a working medium. The

experiments were performed with plates cut from a structure grown by metalorganic chemical vapor deposition (MOCVD). In these laser structures, the active *InAs* region with a thickness of $0.5\ \mu\text{m}$ was confined between $2.7\ \mu\text{m}$ thick broadband *p*- and *n*-type *InAsSb*_{0.14}*P*_{0.3} emitters ($p = 1 \times 10^{18}\ \text{cm}^{-3}$; $n = 5 \times 10^{18}\ \text{cm}^{-3}$). A mask pattern of circles with various diameters ranging from 80 to $300\ \mu\text{m}$ was formed on the surface of plates. Then, the plates were electrochemically etched in an $\text{HClO}_4 - \text{CH}_3\text{COOH}$ mixture.

We have studied the dependence of the cavity's quality and depth on the density of current and the etching time. For this purpose, the depth of etched figures was measured, the character of the side wall profile was studied, and surface quality was evaluated after the removal of the residual photoresist.

In Fig. 7.3 (curve 1) the dependence of the etching depth of the *InAs*-based heterostructure on the density of current passing through the crystal for a fixed time (60 s) is shown. The current density was calculated as the ratio of the total current to the crystal area. As can be seen from these data the current density increasing from 0.3 to $1.6\ \text{mA}/\text{mm}^2$ is accompanied by the slight enlarging of the etching depth. The maximum depth was about $15\ \mu\text{m}$ (at a current density of $1.6\ \text{mA}/\text{mm}^2$). A further current density increasing did not lead to an additional etching depth's enlarging.

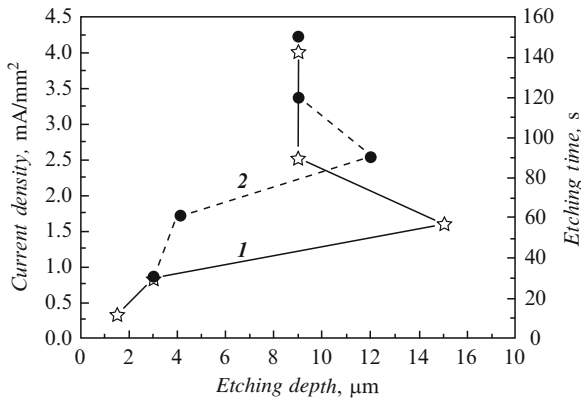


Fig. 7.3 Plots of the maximum etching depth (1) versus current density (for the etching time as long as 60 s) and (2) versus etching duration (for the current density as large as $1\ \text{mA}/\text{mm}^2$) for the electrochemical etching of *InAs(Sb)/InAsSb*_{0.14}*P*_{0.3} heterostructure by using $\text{HClO}_4 - \text{CH}_3\text{COOH}$ mixture

The dependence of the etching depth on time is illustrated by curve 2 in Fig. 7.3, which was obtained for a fixed current density as large as $1\ \text{mA}/\text{mm}^2$. For the etching periods as long as 30 and 60 s, the etching depth was 3 and $4\ \mu\text{m}$, respectively. As the etching time was increased to 100 s, the depth reached $12\ \mu\text{m}$, but a further etching did not lead to the formation of a deeper mesa with vertical side walls.

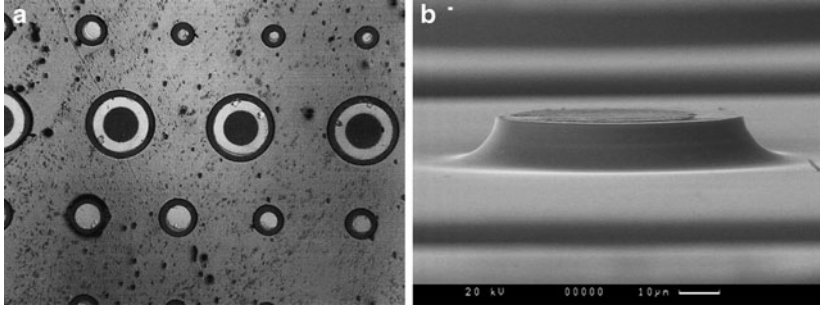


Fig. 7.4 The optical scoped picture of the circular mesas (a) and the scanning electron microscopy (SEM) picture of the circular cavity (b) on the surface of electrochemically etched in $HClO_4 - CH_3COOH$ mixture $InAs(Sb)/InAsSb_{0.14}P_{0.3}$ heterostructure

The optical scoped and scanning electron microscopy (SEM) pictures of the cavity is shown in Fig. 7.4. As can be seen in Fig. 7.4 fabricated by using electrochemical etching in the $HClO_4 - CH_3COOH$ solution cavity has perfect circular shape in contrast to the elliptic shape obtained by etching with $CrO_3 - HCl - HF - H_2O$ solution. Thus, using $HClO_4 - CH_3COOH$ mixture for the electrochemical etching of $InAs(Sb)/InAsSb_{0.14}P_{0.3}$ heterostructures allowed us to fabricate disk cavities with smooth sidewalls, free from roughness and other irregularities.

The maximum etching depth reached $12\ \mu m$ with the vertical part as long as $5\ \mu m$. In these samples, the etching was isotropic and the obtained mesas had the shape of perfect circles with diameters variable from 270 to $50\ \mu m$. These structures were successfully used as disk cavities for WGM lasers.

However, the length of the cavity's sidewall vertical part ($\approx 5\ \mu m$) was not sufficient for the stability due to the fact that disk cavities in the vicinity of the active region had a nearly cone shape, which led to the mode leakage toward the substrate, significantly decreased the Q value, and accordingly increased the threshold current [5].

We have also performed experiments in attempts to increase the etching depth by increasing either the current density or the duration of etching. It was established that the etching depth could be increased further, but this was accompanied by a significant growth in the lateral component of the etching rate. As a result, the desired mesa profile could not be obtained because the side wall deviated strongly from vertical direction.

7.2.3 Wet-Etching by Using $HBr-H_2Cr_2O_7-H_3PO_4$ Solution

In the case of wet-chemical etching, a vertical part of the cavity sidewall amounts approximately to one-third of the total mesa height. In order to increase the length of this part, it is necessary to increase the total depth of etching.

We have studied the results of etching as dependent on the temperature, stirring and concentrations of the components.

The stirring led to deterioration of the surface quality, which was probably related to a change in the viscosity of a solution layer contacting with the semiconductor surface.

The experiments show that not optimal etchant composition results in cavity sidewall roughness of a considerable degree (inset in Fig. 7.5). Based on the obtained disk cavities, WGM lasers show the spontaneous emission spectrum (Fig. 7.5).

The best results were achieved by etching samples at room temperature in a mixture with the composition $HBr - H_2Cr_2O_7 - H_3PO_4$ (4:1:0.5) without stirring. The InAs(Sb)/InAsSbP heterostructure-based disk cavities had a smooth polished sidewalls without any projections or pits. Diameters variable from 270 to 50 μm and at a total cavity height reaching 30 μm , the vertical part of the sidewalls was about 10 μm . A cross-sectional micrograph of the disk is shown on the inset in Fig. 7.6.

Based on the obtained disk cavities, WGM lasers show the both spontaneous and the coherent- emission spectrum (Fig. 7.6).

The emitted light was collected by a parabolic reflector. A number of laser diodes were fabricated and measured; all of these had diode characteristics with a cutoff voltage of -0.3 V at $T = 77\text{ K}$ and a differential resistance of 1.2–1.4 Ω . Electroluminescence spectra of the laser diodes were studied in the pulsed and quasi-continuous modes. In the pulsed mode, measurements were performed at different pulse durations of 50 ns to 20 μs and pulse repetition frequencies of 1 to 32 kHz. A current of 0.02–5 A was passed through the diodes. The measurements were conducted in the temperature range 77–125 K.

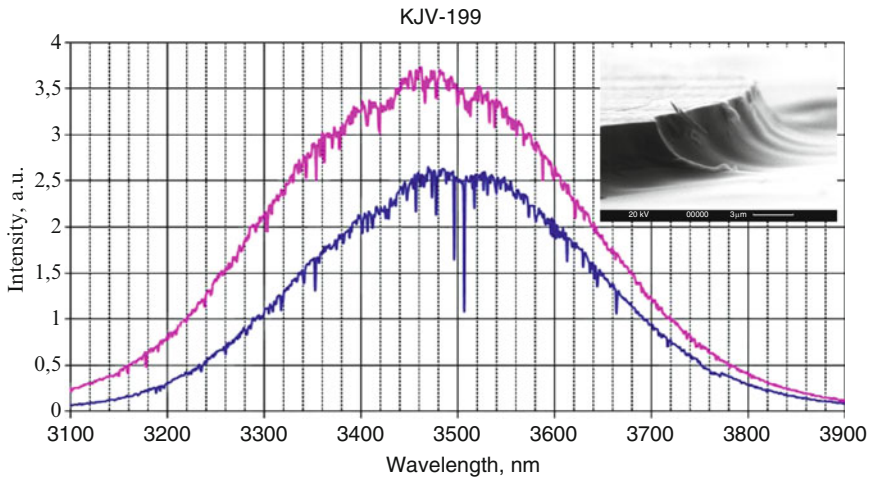


Fig. 7.5 Spontaneous-emission spectrum of the laser based on InAs(Sb)/InAsSbP cavity with rough sidewall (spectral resolution 15 \AA), measured in the quasi-continuous mode at 77 K for various pumping currents. The cavity was created by using not optimal etching solution, and is shown as the inset)

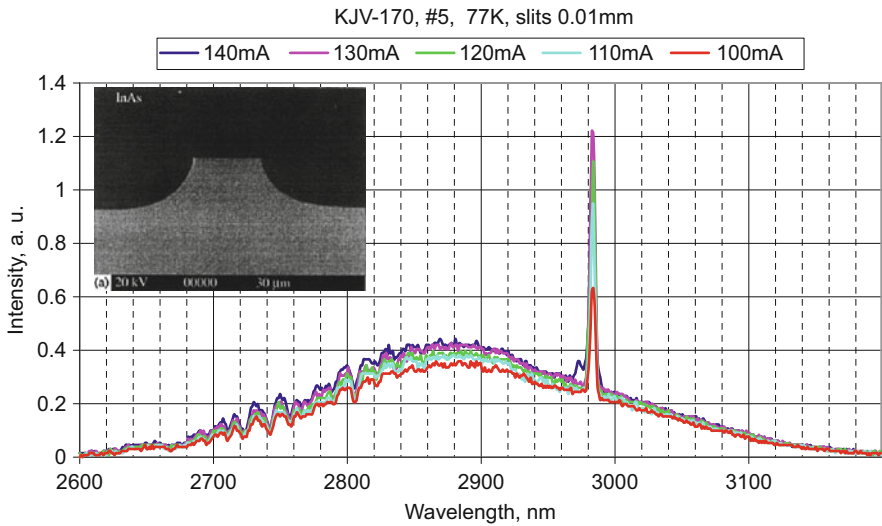


Fig. 7.6 The both spontaneous and coherent-emission spectrum of the laser based on *InAs(Sb)/InAsSbP* cavity with rough sidewall (spectral resolution 15 \AA), measured in the quasi-continuous mode at 77 K for various pumping currents. Cavity with smooth polished sidewalls is shown as inset

7.3 Conclusions

Different wet-etching solutions based on CrO_3 ; HClO_4 and HBr for materials lattice-matched to *InAs* for whispering gallery modes (WGM) lasers ($3\text{--}4 \mu\text{m}$) disk cavities fabrication were studied.

The best results were obtained by using *HBr*-based solution. Diameters of the circular disk cavities variable from 270 to $50 \mu\text{m}$ and at a total cavity height reaching $30 \mu\text{m}$, the vertical part of the sidewalls was about $10 \mu\text{m}$.

Based on the obtained disk cavities, WGM lasers are created that produce coherent radiation with a wavelength of $\lambda \approx (3.0\text{--}3.5) \mu\text{m}$ in a continuous regime at 77 K and in a pulsed regime up to 125 K.

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