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DEVELOPMENT OF LEAD-CHALCOGENIDE MID-INFRARED LIGHT EMITTING
DEVICES

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in partial fulfillment of the requirements for the

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By

DEWALI RAY
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DEVELOPMENT OF LEAD-CHALCOGENIDE MID-INFRARED LIGHT EMITTING
DEVICES

A DISSERTATION APPROVED FOR THE
SCHOOL OF ELECTRICAL AND COMPUTER ENGINEERING

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ABSTRACT

The research described by this dissertation has focused on the fabrication and design, for the improvement of lead chalcogenide lasers. The ultimate goal was to develop TE-cooled single-mode continuous wave Mid-IR laser for high resolution spectroscopic applications. Processing techniques and procedures as well as theoretical design have been successfully developed that could lead to high temperature single-mode continuous wave (CW) laser operation. Light emitting Pb-salt structures on BaF₂ substrate has been examined. An antireflection coating material, for diode-pumped Pb-salt light emitters, that has enhanced room temperature continuous wave light emission has been described. A new surface preparation technology for epi-ready [110] oriented BaF₂ substrates and a novel mounting technique for the fabrication of cleaved cavity Pb-salt laser grown on {110} BaF₂ was developed. Lasing results from [110] oriented Pb-salt laser fabricated on PbSnSe substrate have been reported for the first time. Theoretical modeling of a Pb-Salt ridge waveguide laser for single lateral mode operation and the development of a fabrication process for the same has been described. A novel fabrication method of cleaved facet Pb-salt laser on as-grown metal substrate for high temperature operation of the device has been described.

Chapter 1

Introduction to Lead Chalcogenide Light Emitting Devices

1.1 Mid-Infrared Semiconductor Light Emitting Devices

In the recent years a worldwide increase in the research of mid infrared semiconductor light emitting devices has been observed due to various important applications in telecommunication and molecular spectroscopy. Spectroscopic applications mainly include Trace-Gas-Sensing systems based on Laser Absorption Spectroscopy, which in turn has applications in scientific research, automobile industry, power plants, medicine, environmental control, military base cleaning, chemical and biological weapon detection. Other than spectroscopic applications there are also potential applications like countermeasure, open path A-B Distance Wireless communication, differential absorption light detection and ranging systems etc. But most of the interest in mid infrared light emitters arouse from the prospects for ultrahigh sensitive chemical gas analysis and atmospheric pollution monitoring, using inexpensive and portable spectroscopic instruments¹.

Mid infrared section of optical spectrum lies between 2 to 30 μm wavelengths. And numerous absorption lines of many gaseous molecules lie in the range of mid infrared spectra, for example CO_2 (4.25 μm), CO (4.6 μm), CH_4 (3.3 μm), N_2O (4.5 μm), HF (2.52 μm), O_3 (4.73 μm) etc. So there is a need of thermally stable mid infrared sources with narrow line-widths to obtain high selectivity and sensitivity; single longitudinal mode operation for high selectivity and the elimination of inter-mode noise; rapid wavelength tunability for fast response and high data acquisition rates; high beam quality, i.e., small beam divergence, small astigmatism, and stable beam output direction for optimum coupling into and through a gas sampling cell; and sufficient output power to overcome inherent electronic detection noise and to obtain high laser signal-to-noise

ratios, for the Mid-IR absorption spectroscopy². An ideal source for such applications would be, narrow line width tunable lasers with continuous-wave (cw) emission at room temperature and with considerable output power (>1mW). Figure 1.1 shows bandgap energy vs. lattice periodicity of some semiconductor compounds and substrates including those used in Mid-IR laser fabrication.

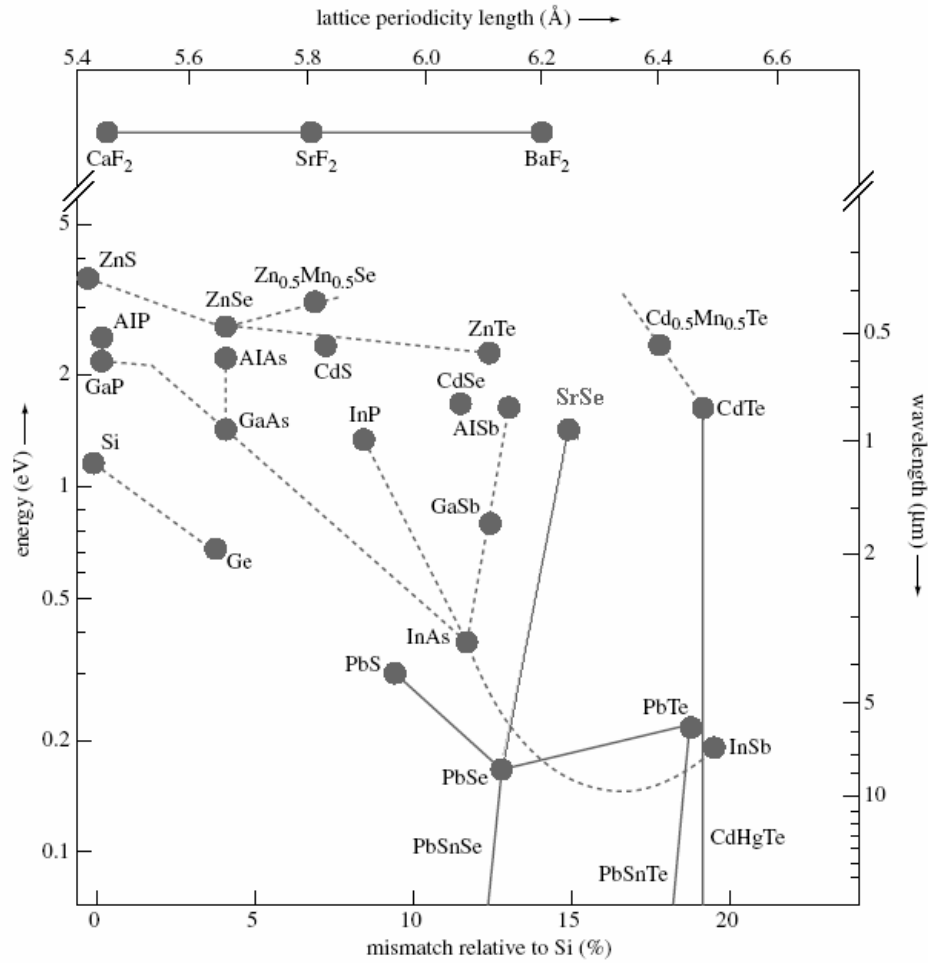


Fig 1.1 The bandgap energy versus lattice periodicity length of some semiconductor compounds. Adapted from Zogg *et al.* (1989).

1.2 Auger Recombination in Mid-Infrared Emitters

In the Mid-Infrared interband devices, the principal carrier loss mechanism is Auger recombination³. The Auger recombination rate per unit volume for electron auger type-1 process (CHCC), according to Beattie & Landsberg (1959) is expressed as⁴

$$R = \frac{2\pi}{\hbar} \left(\frac{1}{(2\pi)^3} \right) \iiint |M_{if}|^2 P(1,1',2,2') \delta(k_1 + k_2 - k_{1'} - k_{2'}) \delta(E_i - E_f) d^3k_1 d^3k_2 d^3k_{1'} d^3k_{2'}$$

where 1 and 1' indicate the electron and hole recombination pair respectively, 2 points to second electron excited to 2'. $P(1,1',2,2')$ is the net probability for the auger process.

Therefore, in the case of a CHCC process³,

$$P(1,1',2,2') = f_{e1} f_{h1} f_{e2} (1 - f_{e2'})$$

In the above expression of CHCC, f_e and f_h are the Fermi-Dirac distribution functions for electrons and holes respectively. These are actually the quasi-Fermi levels of conduction band and valence band. Considering $n = p$ and replacing the last term for empty state by Boltzmann-factor, the approximated probability can be written as³,

$$P \propto n^3 \exp\left(-\frac{E_{2'} - E_g}{k_B T}\right),$$

For highly degenerate states (lasers) this equation does not apply. However, it describes when P is largest then $E_{2'}$ is smallest. Therefore, the auger rate is basically controlled by the exponential by contributing at the threshold energy E_T (Beattie & Landsberg 1959), as there exists a minimum energy below which the δ -function no longer satisfies⁴.

Hence,

$$E_T = \frac{E_g}{1 + m_h/m_e},$$

For CHHH process E_T is described as,

$$E_T = \frac{E_g}{1 + m_e/m_h},$$

The auger coefficient C is derived from the approximation of R which is again derived from the above equations³. Hence, we have

$$R = \text{const.} \times n^3 |M|^2 \exp\left[-\frac{E_T}{kT}\right]$$

$$= Cn^3$$

Narrow bandgap lasers emitting in the range of 3-5 μm with interband transition gained much popularity due to low non-radiative transition rates. For these materials Auger suppression takes place when they are in the form of bulk materials. Figure below shows the Auger coefficient as a function of emission wavelengths for different materials used in the MIR regime as light sources.

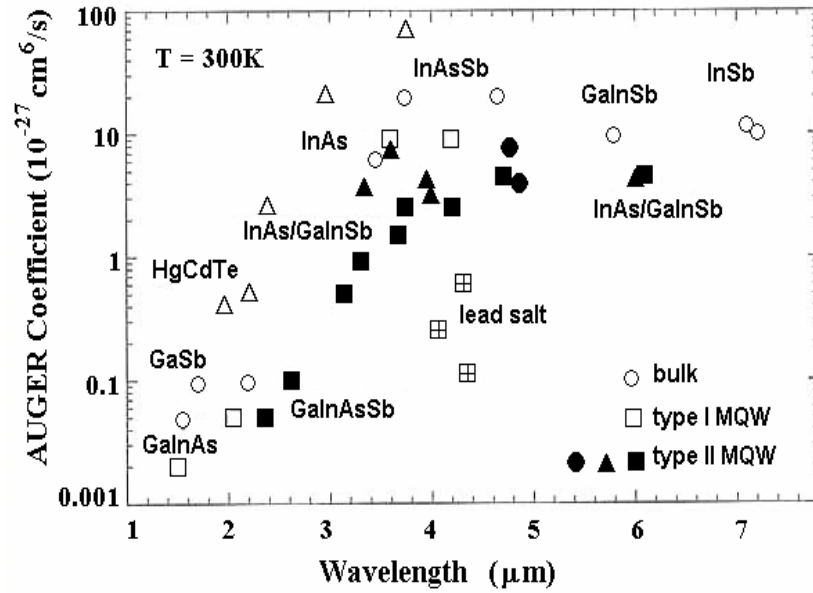


Fig 1.2 Auger coefficient of different materials used as MIR sources¹⁶.

Auger transition rate is mainly controlled by the δ function which is actually accountable for the momentum conservation in the Auger rate equation. It can be reduced by bandgap engineering so that it changes the range of momenta. This is possible by reducing heavy hole effective mass. Also, if the effective mass ratio is close to one, that lowers the Auger rate³.

Lead chalcogenides have symmetric valence and conduction bands. Therefore, they have a maximum value of E_T . Findlay *et al.* (1998)⁵ had performed Pump-probe transmission experiments on PbSe above the fundamental absorption edge near 4 μm in the temperature range 30 - 300 K. A Dutch picosecond free electron laser was used in this experiment. For carrier densities above threshold and at temperatures below 200 K, stimulated recombination represented the most efficient recombination mechanism with relatively fast kinetics in the 50-100 ps region, as was reported by them. Above this temperature although Auger recombination dominated but still it remained at a lower value. The Auger coefficient was approximately constant (about $8 \times 10^{-28} \text{cm}^6 \text{s}^{-1}$) in the temperature range of 300 and 70 K, and then dropped to a value of about $1 \times 10^{-28} \text{cm}^6 \text{s}^{-1}$ at 30 K, which was in good agreement with the theory for nonparabolic near-mirror bands and nondegenerate statistics, as was reported by them.

As Auger recombination is much lower in IV-VI semiconductors than III-V materials emitting in the 3 – 5 μm wavelength region, these materials have the potential to lase at and above room temperature¹¹.

1.3 State of the Art in Mid-Infrared Semiconductor Laser

Mid-infrared emissions were first demonstrated in 1963, from InAs and InSb p-n junction diode emitting at a wavelength of $3.1\ \mu\text{m}^6$ and $5.3\mu\text{m}^7$ respectively. In 1964, just after a year, laser emission from PbTe p-n junction diode at a higher wavelength was reported⁸. After this demonstration of first Pb-salt laser, standard MIR lasers were fabricated from narrow gap IV-VI semiconductors such as PbS, PbTe, PbSe, PbSSe, PbSnTe, and PbSnSe, in the next twenty years. These lasers were all diffused-diodes emitting in $4 - 30\ \mu\text{m}$ wavelength range with an operation temperature of $4 - 77\ \text{K}$. As the epitaxial growth techniques advanced, new growing techniques such as Liquid-phase-epitaxy (LPE), hot-wall-epitaxy (HPE), molecular-beam-epitaxy (MBE) were developed and double-hetrostructures (DH) were grown. DH-structure made revolution in IV-VI laser performances. Lead-salt DH lasers remained the standard of MIR lasers still 1990. These lasers were grown on PbS, PbSe or PbTe substrates, with active layers having PbEuSSe for $3 - 4\ \mu\text{m}^9$, PbEuSeTe or PbEuSe for $4 - 8\ \mu\text{m}$ range, and PbSnTe or PbSnSe for wavelengths beyond $8\ \mu\text{m}$. During that time DH laser based on III-V materials were becoming promising candidate for MIR light source as well. AlGaAsSb/GaInAsSb/AlGaAsSb DH lasers on GaSb substrate and InAsPSb/InAsSb/InAsPSb DH lasers on InAs substrates showed excellent performance at room temperature in the $2.0 - 2.5\ \mu\text{m}$ range^{10,11,12}. The InGaAsSb/AlGaAsSb strained multi-quantum-well (MQW) laser showed striking results in CW operation at and above room temperature and till date appears to be well-established technology for laser emission in the $2.0 - 2.7\ \mu\text{m}$ wavelength range¹³. Beyond $2.7\ \mu\text{m}$, type-II quantum well semiconductor lasers based on the InAs–GaSb system, III–V quantum cascade laser

and IV–VI lead salt semiconductor lasers are in use^{14 15 16}. To give an overview of the present state of the art in MIR semiconductor laser, Figure 1.3 shows the maximum operation temperature in the pulse and CW regime as a function of wavelength for various lasers in the 2-5 μm wavelength range.

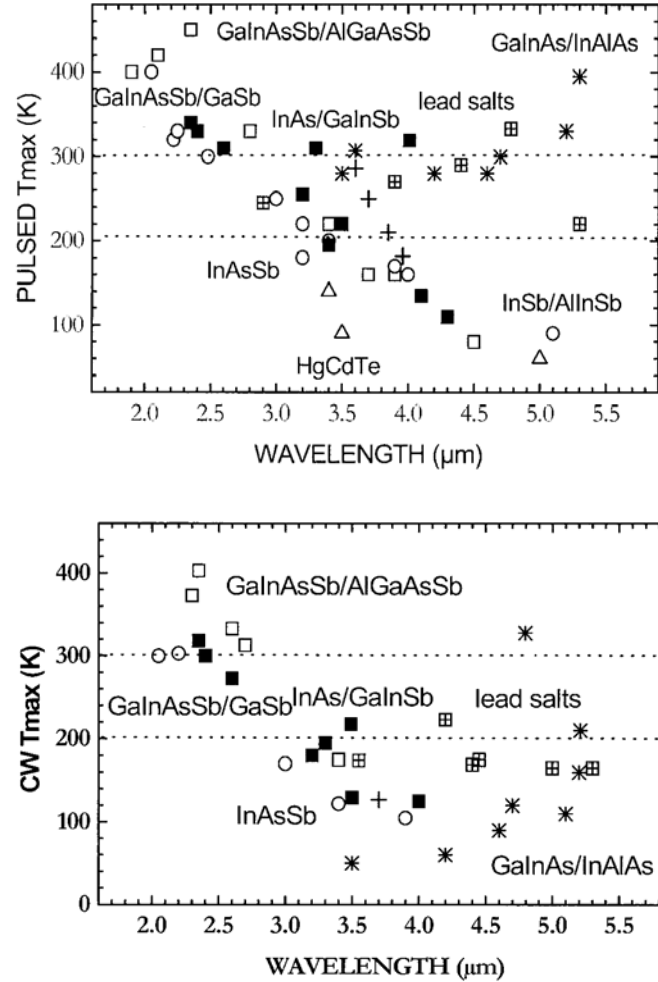


Fig 1.3 Maximum operating temperature of semiconductor laser diodes in the mid-IR; *open circles*: Sb-based DH lasers; *open boxes*: Sb-based type-I MQW lasers; *solid boxes*: Sb-based type-II MQW lasers; *+*: Sb-based type-II interband cascade lasers; ***: GaInAs/AlInAs inter sub-band quantum cascade lasers; *crossed box*: lead salt lasers; *open triangles*: HgCdTe lasers¹⁷.

1.4 Lead-Chalcogenide Diode Laser

Lead chalcogenide diode lasers emitting in the MIR region have been commercially available for more than two decades. Their emission covers the wavelength range from $3\mu\text{m}$ to more than $20\mu\text{m}$ ¹⁸. Electrically injected lead salt lasers have achieved 223K operation in continuous wave mode¹⁹ and above-room-temperature operations in pulsed mode²⁰. Earlier, there was misconception regarding the incapability of IV-VI lasers to produce high output power. However, in 1997 it was reported that even a diffused-junction laser can produce up to 24 mW of CW output power²¹.

Bandgap energies of IV-VI semiconductors increase with increasing temperature. Hence, they have very large temperature and current tuning ranges. The temperature induced bandgap energy change also helps in reducing facet heating. Facet heating is a great disadvantage of III-V lasers that suffer from thermal runaway problems associated with the decrease in bandgap energy with increasing temperature²². Absence of facet heating in IV-VI lasers make them more reliable for high temperature operation.

Among all the previously-mentioned state of the art MIR lasers, lead salt diode lasers have the advantages of large tuning range, easy current tuning and narrow linewidth. Group IV-VI materials have suppressed Auger non-radiative loss (by more than an order of magnitude over III-V quantum wells)^{23 24}. Also they possess much lighter electron and hole masses that lead to further reduction of the lasing thresholds. These are the properties that enabled lead salt lasers to set and maintain the earlier records for maximum operation temperatures for both pulsed and CW operation among all mid infrared semiconductor diodes.

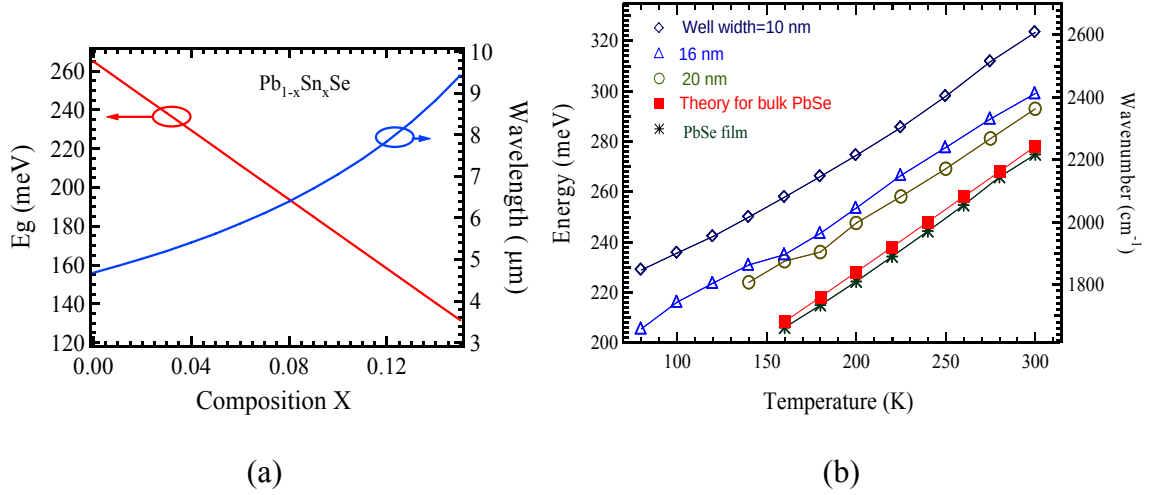


Fig 1.4 Energy band gap of PbSnSe at room temperature (a), Temperature dependant emission wavelengths of PbSe/PbSrSe QW structures (b)²⁵.

As can be seen in Figure 1.4(a), the wavelength of a MIR Pb-salt diode can be easily tuned by changing the composition from 3.5 μm to 10 μm with high reproducibility. It is clear from Figure 1.4(b) that by changing the QW width and temperature further fine-tuning of the device is possible. As can be seen in figure 1.4(b), the temperature tuning is about five times larger than III-V materials. It was mentioned before that the energy bandgap of IV-VI materials increase with the temperature. Therefore, an increment in the injection current will make a blue-shift of both the gain peak and the energy band gap by Joule-heating and thus the laser emission wavelength can be easily tuned.

For a number of years, lead salt diode lasers remained the only commercially available semiconductor laser emitting in the mid-infrared region. However, their performance remains far from that desired. Low thermal conductivity of IV-VI materials prevents room temperature CW operation of traditional IV-VI lasers. Also, there are issues with high dislocation density²⁶ ($\sim 10^4$ to 10^7 cm^{-2}) as well. Presently, commercially available Pb-salt lasers require cryogenic cooling.

For commercially available IV-VI mid-IR lasers, the lasing thresholds are significantly increased by the four-fold degeneracy of the L-valley conduction and valence band extrema. Quantum confinement does not lift the degeneracy in [100] oriented edge-emitting QW devices, since the four valleys remain symmetric for the (100) growth that was employed to allow the cleaving of laser cavities. This prevents the full exploitation of what is perhaps the greatest advantage of IV-VI laser materials for high-temperature and long-wavelength operation, namely the threshold reduction that results from a low non-radiative recombination rate. Therefore, a continued effort in the development of lasers based on this promising material system was needed.

In this research work, to solve the problems of higher dislocation density and poor heat dissipation of traditional IV-VI mid-IR lasers, fabrication of lasers using PbSe-based material on metal halide substrates, particularly BaF₂ was carried out. BaF₂ is lattice matched with PbSe. Epitaxial growth on (111) BaF₂ produces high quality material due to dislocation gliding. PbSe/PbSrSe quantum well grown on BaF₂ substrates showed very high power photoluminescence²⁷. BaF₂ has five times higher thermal conductivity than that of IV-VI materials and hence has the potential to improve heat dissipation from active region. It is more economic than the expensive Pb-salt substrates. In addition, refractive index of BaF₂ in the 3.5 to 4.5 μ m wavelength region is 1.4 while that of PbSe in the same wavelength range is 4.8, thus the combination makes the optical confinement very high. This is quite advantageous for optically pumped devices. Previously Vertical cavity surface emitting lasers on BaF₂ substrates with above-room-temperature operation was reported²⁸. However, several issues still remain for the fabrication of edge emitting devices with IV-VI epitaxy grown on BaF₂ substrate,

which is addressed in Chapter 3. In connection to optical pumping, an antireflection coating material, for diode-pumped Pb-salt light emitters, that enhanced room temperature CW light emission²⁹ was also reported as a part of this research.

To overcome the existing problems associated with the [100] orientation of the edge emitting devices, [110] orientation was further chosen. Compared with the conventional [100] orientated materials, [110]-orientation provides higher material quality, high modal gain and partially lifts the degeneracy. Based on the theoretical calculations and waveguide simulations, it was found that single lateral mode emission is possible from a Pb-salt ridge waveguide laser having a ridge of width 5 μm and height 1 μm . In the design of this ridge waveguide laser additional criterion that can produce selective losses for the higher order modes was included. Novel surface preparation technology for epi-ready [110] oriented Pb-salt and metal halide wafers and lasing results from {110} Pb-salt laser was also reported for the first time.

Despite the low thermal conductivity of Pb-salt materials (thermal conductivity $4.2 \text{ Wm}^{-1} \cdot \text{K}^{-1}$ @ 300 K), proper design of laser fabrication method on foreign substrates having higher thermal conductivities allow significant improvements in the operation temperature. A successful epitaxial transfer and cleaving of [110] oriented Pb-salt based epitaxy on GaAs wafer³⁰, having almost 11 times higher thermal conductivity ($46 \text{ Wm}^{-1} \cdot \text{K}^{-1}$ @ 298 K) than that of thermally resistive Pb-salt substrates, had been reported. In addition, a novel fabrication method of cleaved facet Pb-salt laser on as grown metal substrate for high temperature CW operation of the device had also been reported.

1.5 Fundamentals of Semiconductor Laser Operation

The basic description of a semiconductor is its band structure, i.e. the variation of energy E with wave vector k , which is shown in Figure 1.5. The valence band is the last completely filled band and the Conduction band is the first unfilled band at $T=0$. At higher temperatures some electrons in the valence band absorb enough energy to jump to the conduction band creating a vacant space or hole in the valence band. Such a transition is also possible by photon absorption or electrical excitation. When the electron in the higher energy conduction band jumps back to the empty space and thus combining with a hole in the lower energy conduction band then a photon is emitted. Thus the generation of a photon in this process may be written by the chemical equation, $e + h \rightarrow h\nu > E_g$.

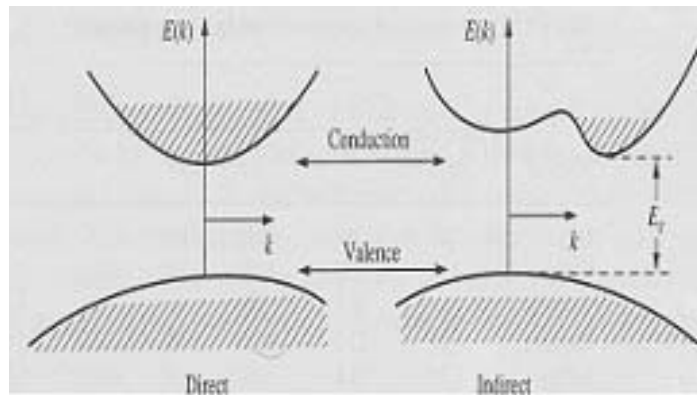


Fig 1.5 Band Diagram of Semiconductor ³¹

Now, direct band gap semiconductors strictly follow the above mentioned equation and for indirect band gap semiconductors some non-radiative processes compete for electron population. Figure 1.5 shows the occupied (shaded) and empty states (white) for direct and indirect band gap semiconductors as a function of the momentum of a carrier.

In the absorption process the electron absorbs energy of an incident photon, which is greater than E_g and jumps to a higher energy in the conduction band but by the phonon vibrations it settles down to the minimum of the conduction band. The hole does the opposite, it settles to the maximum of the valence band. Thus if enough electrons can be pumped to the conduction band, a population inversion may be created between the lowest energies of the conduction band and the highest energies of the valence band. This gives the possibility of creating a laser.

The density of states in either the conduction band or the valence band can be given by the equation

$$\rho_{cv}(E)dE = \left\{ \frac{1}{2\pi^2} \right\} \left\{ \frac{2m^*}{\hbar^2} \right\}^{3/2} \{E^{1/2} dE\} ,$$

where E is measured from the band edge, that is up to the conduction band or down to the valence band, and m^* is the effective mass of each of the carriers. It should be noted that since the effective mass of electrons are larger than the holes the density of states for holes per unit energy dE is much larger than for electrons. Suppose we wish to fill every available energy from E_c to $E_c + \Delta E$, the densities of electrons that must be created either by injection or by some other means are

$$N_e = \int_0^{\Delta E} \rho_{cv}(E)dE = \frac{1}{3\pi^2} \left[\frac{2m_e^* \Delta E}{\hbar^2} \right]^{3/2}$$

Since electrons are fermions the occupation probability of a state of energy ϵ at a temp T

is given by the Fermi function, $f(\epsilon) = \frac{1}{\exp[(\epsilon - E_f)/kT] + 1}$

and $1 - f(\epsilon)$ is the probability that it is empty.

Thus the density of electrons in the conduction band in the energy interval $d\epsilon$ is given by

$$n_c(\epsilon)d\epsilon = \frac{1}{3\pi^2} \left[\frac{2m_e^* \Delta E}{\hbar^2} \right]^{3/2} \frac{(\epsilon - E_c)^{1/2} d\epsilon}{\exp[(\epsilon - E_f)/kT] + 1}$$

And the density of holes in the interval $d\epsilon$ below E_v is given by,

$$p_v(\epsilon)d\epsilon = \frac{1}{2\pi^2} \left[\frac{2m_h^*}{\hbar^2} \right]^{3/2} \frac{(E_v - \epsilon)^{1/2} d\epsilon}{\exp[(F_p - \epsilon)/kT] + 1}$$

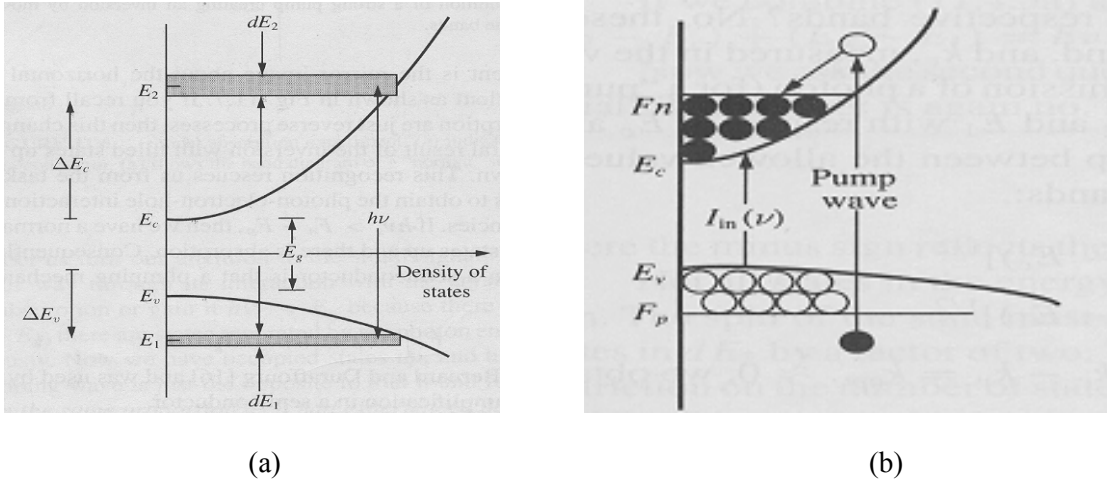


Fig 1.6 Optical transitions in a semiconductor (a) Band diagram showing strong pump creating an inversion by moving quasi-Fermi level into band (b)³¹.

Referring to the Figure 1.6(a),

$$\hbar k_c = [2m_e^*(E_2 - E_c)]^{1/2}$$

$$\hbar k_v = [2m_h^*(E_v - E_1)]^{1/2}$$

Since the transition must conserve momentum, $k_c - k_v = k_{\text{opt}} \approx 0$, we obtain the different energy spreads of the electrons and holes in the above figure, not equal but related by the equation,

$$dE_2 = \frac{m_h^*}{m_e^*} d(-E_1)$$

It should be noted that all the states in the energy interval dE_2 and dE_1 cannot participate in the optical transition. The spin of the state must be conserved, and this divides the density of states by two. The conservation of momentum should also be maintained, and there is also a restriction in the available appropriate empty states. The reduced joint density of states is defined to reflect the number of states at E_2 and E_1 within dE_2 and dE_1 which can participate in the transition at $h\nu$, and which conserve spin momentum.

Therefore, the joint density of state can be expressed as,

$$\rho_{jnt}(h\nu)dE = \frac{1}{4\pi^2\hbar^2} \left(\frac{2m_e^*m_h^*}{\hbar^2(m_e^* + m_h^*)} \right)^{3/2} (h\nu - E_g)^{1/2}$$

When light travels through such a medium the following processes may occur,

1. Stimulated emission: the photon strikes an electron in the conduction band and forces it to jump to the valence band releasing another photon of same wavelength, phase, and direction.
2. Absorption: An electron in the valence band absorbs the photon and jumps to the conduction band
3. Spontaneous emission: A spontaneous transition of the electron from conduction band to valence band occurs releasing a photon in the process.

The gain coefficient γ is defined as the rate of increase in intensity per unit distance as the light travels through it due to the above-mentioned processes.

$$\gamma(\nu) = A_{21} [f_2(1 - f_1)] \frac{\lambda_0^2}{8\pi n^2} \rho_{jnt}(\nu) \left\{ 1 - \exp \left[\frac{h\nu - (F_n - F_p)}{kT} \right] \right\}$$

It should be noted that we can get a positive gain coefficient when $F_n - F_p$ (as in Figure 1.6b) is greater than $h\nu$. This is possible when Fermi levels (rather pseudo Fermi levels) are made wider than the band-gap by carrier injection or by optical pumping. In a homojunction laser a p-n junction which when forward biased causes a Fermi level split because of injection of minority carriers and forms a region near the junction where exists simultaneously both a high density of holes and electrons. The electrons and holes in this region recombine releasing a photon. In a heterojunction laser there exists an undoped region between the p and n type material. This gives an advantage of large area where the recombination of electrons and holes might occur, thus increasing the output power.

Quantum size effects

Modern technologies have made it possible to grow thin layers of material whose dimension is of the order of deBroglie wavelengths. This modifies the density of states and makes it independent of energy.

$$\rho(E)dE = \frac{1}{2\pi^2} \left(\frac{2m^*}{\hbar^2} \right) \left(\frac{\pi}{L_z} \right) dE$$

If we consider quantum confinement to be in the z direction, the density of states becomes independent of the energy E, and can be controlled by the thickness L_z . Thus by choosing the dimension of L_z we can design the energy state and thus engineer the band-gap³¹.

1.6 Thesis Overview

The main goal of this research was to assist the development of an improved Pb-chalcogenide light source for Tunable Diode Laser Absorption Spectroscopy. In order to achieve such a goal, proper design of a laser structure and proper design of laser fabrication methods were needed that would preferentially make the device to operate in continuous wave single lateral mode and increase the operation temperature to at least the thermoelectric range, above 230K. At the initial stage, light emitting Pb-salt structures on BaF₂ substrate had been examined, owing to the advantages mentioned in the previous section. Then to overcome the existing problems associated with the [100] orientation of the edge emitting devices, the [110] orientation was chosen further. Compared with the conventional [100] orientated materials, [110]-orientation provides higher material quality, high modal gain and partially lifts the degeneracy. Based on the theoretical calculations and waveguide simulations, it was found that single lateral mode emission is possible from a Pb-salt ridge waveguide laser having a ridge of width 5 μm and height 1 μm . Also, despite the low thermal conductivity of IV-VI semiconductor materials (thermal conductivity $4.2 \text{ Wm}^{-1}\text{.K}^{-1}$ @300 K for PbSe), proper design of a laser fabrication method on foreign substrates having higher thermal conductivities allow significant improvements in the operation temperature. A successful epitaxial transfer and cleaving of [110] oriented IV-VI semiconductor based epitaxy on GaAs wafer³², which has almost 11 times higher thermal conductivity ($46 \text{ Wm}^{-1}\text{.K}^{-1}$ @298 K) than that of thermally resistive IV-VI semiconductor substrates, was reported. This dissertation describes all my experimental work towards the development of Pb-chalcogenide light emitting structures, both optically and electrically pumped and theoretical modeling of a

ridge waveguide laser for single lateral mode operation. The journey was full of challenges and the following chapters also describe how they were met and solved.

Chapter-2 describes the fabrication and measurement results of an optically pumped Pb-salt light emitting structure on [111] oriented BaF₂ substrate. Also discussed is the development of an antireflection coating material for diode-pumped Pb-salt light emitters that enhanced CW light emission up to 3-4 times from the structure. Chapter-3 describes the fabrication of a [110] oriented edge emitting laser grown on a BaF₂ substrate using both cleaved and etched facets and novel surface preparation technology of [110] oriented wafers for epitaxial growth. Chapter-4 presents [110] oriented Pb-salt laser fabricated on a PbSnSe substrate. Lasing results have been represented and fabrication issues have been discussed. Chapter-5 describes the modeling of a ridge waveguide laser and addresses the fabrication steps taken for the development of such a device. Chapter-6 introduces a novel fabrication method of cleaved facet Pb-salt laser on an as-grown metal substrate for high temperature operation of the device. This method uses electroplating technique to form a metal base or carrier substrate in contact with epitaxial layer and is followed by a growth substrate removal technique for complete transfer of the epitaxial layer to a metal. Chapter-7 details the future direction for this project and explains the issues needing further investigation and improvements.

Some material properties and outcomes thereafter were not clearly understood. Therefore, some questions remain unanswered. Despite this, many novel processes have been developed and challenges were overcome in an honest effort towards betterment. It is hoped that this dissertation provides a clear picture of the field of Pb-salt light emitting devices and inspires a continuation of this work at the University of Oklahoma.

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Chapter 2

Optically Pumped Lead-Salt Light Emitting Structures

2.1 Optically Pumped Lead-Salt Structure on [111] Oriented BaF₂ Substrate

In the recent years, in contrast to electrical excitation, a renewed interest has been observed in optical excitation to demonstrate lasing in the mid-infrared wavelength region^{1, 2}. This is due to the fact that optical pumping offers several advantages, first of all simple design or in other words the design freedom is much higher. This includes use of high resistance cladding layers like Pb_{1-x}Sr_xSe with comparatively larger Sr content and lower carrier concentration that in addition further diminishes Auger recombination increasing the chances of having a higher operating temperature device. Secondly, in an optically pumped device there is no need of the development of p-n junctions and low resistance ohmic contacts for the electrical connections. Therefore, as an initial step to demonstrate MIR emission from edge-emitting devices we choose optical pumping of Pb-salt based structure grown on [111] oriented BaF₂ substrate. Now, as it was mentioned earlier that IV-VI lasers, especially PbSe-based lasers on BaF₂ substrate, offer several advantages. PbSe is almost lattice matched with BaF₂ and they have a large difference in refractive index. PbSe has a refractive index of 4.8 where as BaF₂ has a refractive index of 1.4 in the 3.5 to 4.5 μm wavelength range, thus giving an excellent optical confinement. Third, the thermal conductivity of BaF₂ is five times larger than that of IV-VI materials. In addition, growth on [111] BaF₂ allows dislocation gliding and thus leads to superior material quality. Owing to the previous success of quantum well structures grown with PbSe and PbSrSe on [111] oriented BaF₂ substrates with high output power³ and above-room-temperature operation of vertical cavity surface emitting lasers (VCSEL)⁴, on [111] BaF₂, the [111] orientation was opted for the edge-emitting device

as well . Although PbSe-based VCSELs have been successful, edge-emitting lasers would have better tunability and much higher output power as are needed for TDLAS.

2.1.1 Edge Emitting Device Fabrication

In this experiment, a waveguide structure was grown on $[111]$ oriented BaF_2 by molecular beam epitaxy (MBE), using compound sources for PbSe and BaF_2 and elemental sources for Sr and Se. The structure consisted of seven pairs of PbSe (20 nm) / PbSrSe (30 nm) multi-quantum wells followed by a 100 nm BaF_2 top confinement layer. This BaF_2 layer also serves as a passivation layer. Ridge structures of dimension $470\text{ }\mu\text{m}$ in length and $40\text{ }\mu\text{m}$ in width were then fabricated by wet chemical etching. It can be seen from Figure 2.1 that parallel facets cannot be obtained by cleaving the Pb-salt- BaF_2 assembly where the growth took place along $[111]$ orientation of the substrate, and that is why wet chemical etching was used to obtain the mirror facets to form a Fabry-Perot cavity for lasing operation.

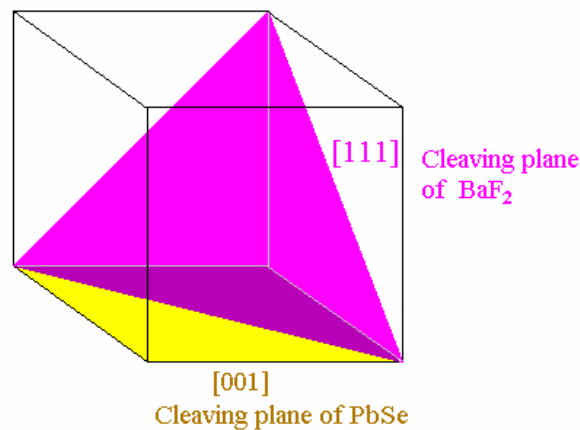
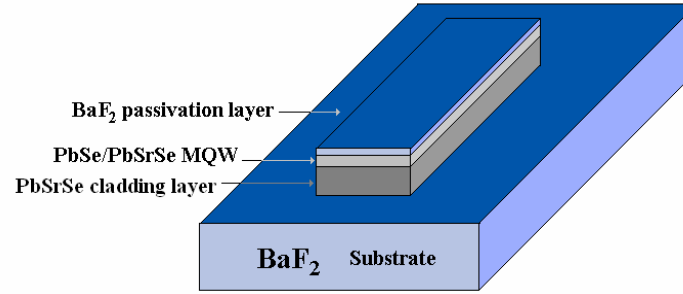
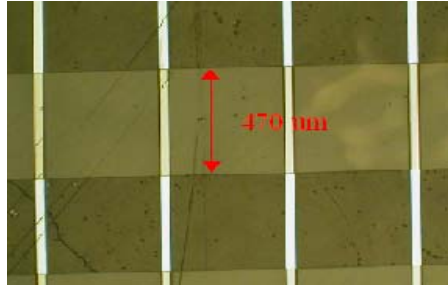


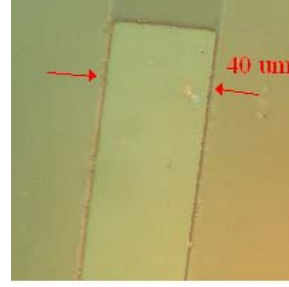
Fig 2.1 Crystal orientation showing $\{111\}$ and $\{100\}$ plane



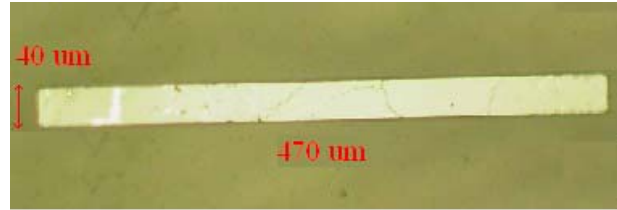
(a)



(b)



(c)



(d)

Fig 2.2 Schematic of the wet etched structure showing different layers (a); Nomarski image of the etched structure showing several active elements (b); Top view of a single etched element at a higher magnification (c); Nomarski image of a single ridge showing cavity length and width (d).

Two times photolithography was performed followed by the wet etching to form $470\mu\text{m}$ X $40\mu\text{m}$ ridge elements on 1cm^2 sample. The etching solutions are proprietary mixtures provided by an institution in Germany and are subject to a non-disclosure agreement (NDA) with the University of Oklahoma, so their exact recipes will not be described here. BaF_2 passivation layer was deposited on the whole etched structure after a thorough cleaning, by using molecular beam epitaxy method (MBE).

2.1.2 Measurement and Results

The sample was optically pumped with light incident normal to the substrate by a 1.064 μm Nd:YAG laser ($\tau_{\text{pulse}} \approx 23\text{ns}$, 10Hz) with a spot size of 2.6 mm in diameter. The emission from the side i.e from one facet of the sample was measured with an IFS 66/S spectrometer in step-scan mode using an InSb detector. The output power from the sample was calibrated using a standard blackbody reference source whose emission energy at certain temperature and blackbody field of view was provided by the manufacturer. Figure 2.3 shows the schematic of the sample showing direction of pumping and light emission from facet.

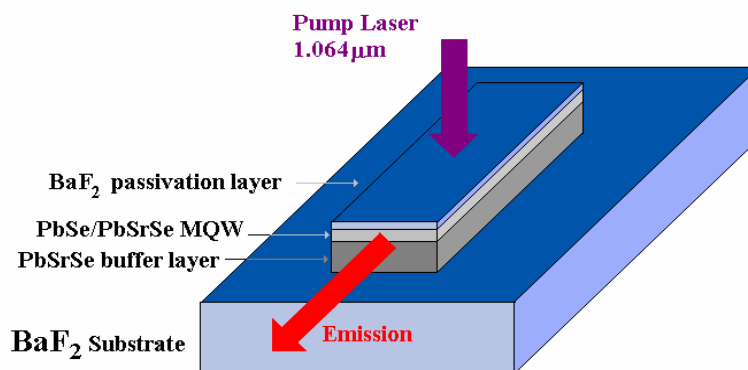
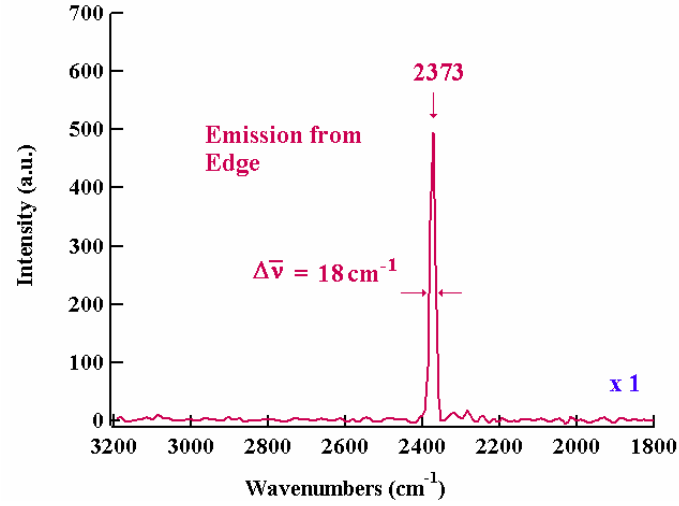
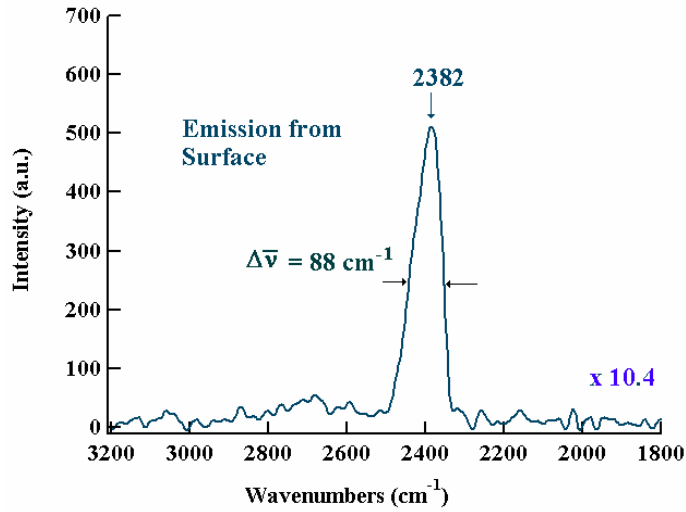


Fig 2.3 Schematic of etched structure showing pumping direction and light emission

Figure 2.4(a) shows room temperature emission spectrum measured from one facet of the edge emitting structure and 2.4(b) shows the room temperature photoluminescence (PL) measured from the surface of the sample before processing.



(a)

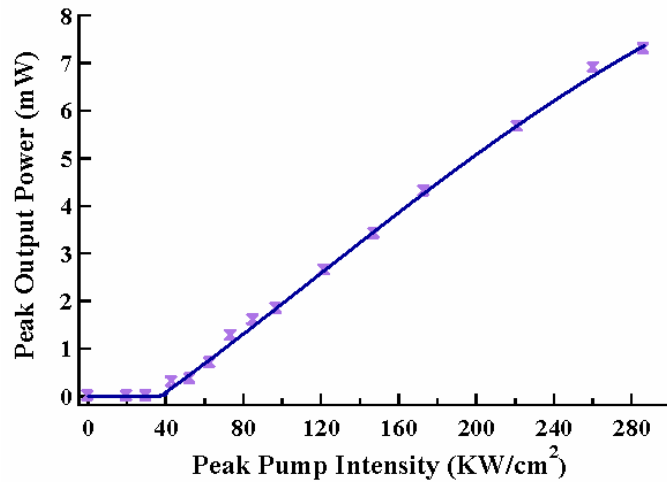


(b)

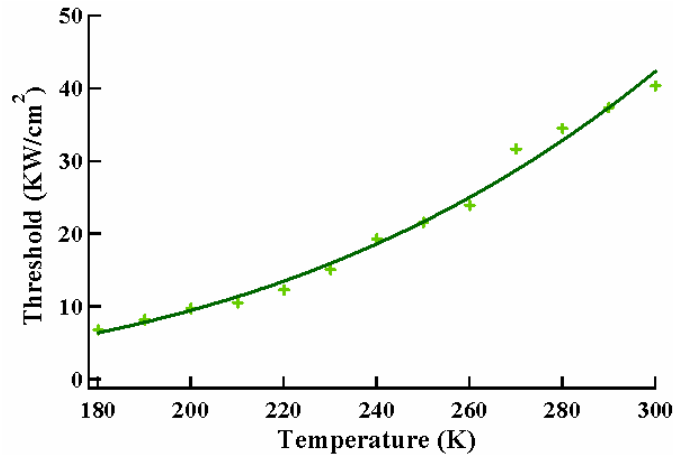
Fig 2.4 Side emission spectrum (a) and Surface emission spectrum (b) at room temperature from optically pumped Edge Emitting structure

As can be seen in the figure, the line-width of side emission is 18 cm^{-1} , which is much wider than cavity modes but it is much narrower than the line-width of surface emission, which is actually 88 cm^{-1} at room temperature. Also, emission intensity from the facet is almost 10 times higher than that of surface. Therefore, the results although do not indicate lasing but definitely points toward Amplified Spontaneous Emission (ASE).

The peak output power vs. peak pump intensity observed from the sample is shown in Figure 2.5(a). The numbers in the figure are as seen by the FTIR without considering far-field of the emission. As shown in Figure 2.5(b), the emission from the sample showed a threshold-like behavior, the possible cause of which is ASE, indicating high reflectivity of the facets. Also, peak output power as high as several milliwatts was obtained at room temperature.



(a)



(b)

Fig 2.5 Peak output power vs. peak pumping power intensity at room temperature (a) and Threshold behavior of the sample at different temperatures (b)

Figure 2.6 shows emission peaks at different temperatures and hence represents the tunability of the emission. It can be seen from the figure that the wavelength is tunable from 4.38 μm at room temperature to 4.72 μm at almost TE cooler temperature (240K). Temperature tunability is almost $5.7 \times 10^{-3} \mu\text{m/K}$. Considering the measured far field emission and the pumping efficiency (26.6%) in the calculation, internal quantum efficiency was found to be 2.4% at room temperature, and significantly higher at lower temperatures. However, internal reflection loss was not considered while calculating quantum efficiency.

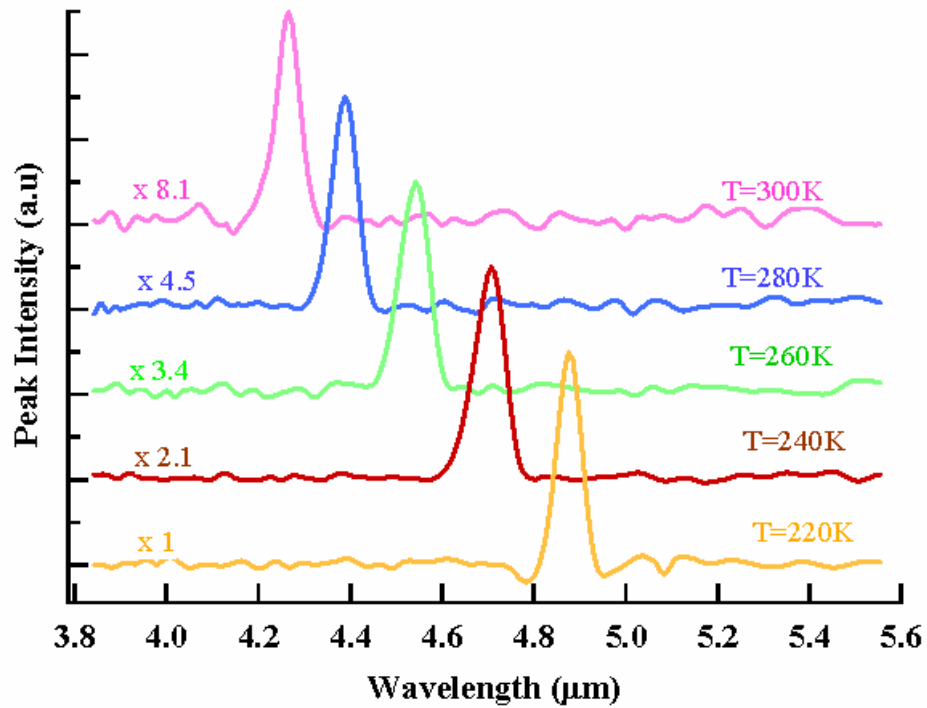


Fig 2.6 Emission peaks at different temperature from the Edge Emitting structure

2.1.3 Discussion

In a laser medium with large gain, the fluorescence from spontaneous emission can be amplified to high levels. This amplified fluorescence has low temporal coherence but possesses good spatial coherence. This is actually called amplified spontaneous emission (ASE). This emission of fluorescence can be relatively intense as it experiences significant optical gain within the emitting device. Due to the finite gain bandwidth, the bandwidth of the emitted light is usually smaller than that of the fluorescence itself. Although ASE is considered to be a noise parameter for a laser medium or in a large gain amplifier, but in our case at least it indicated the presence of a gain medium.

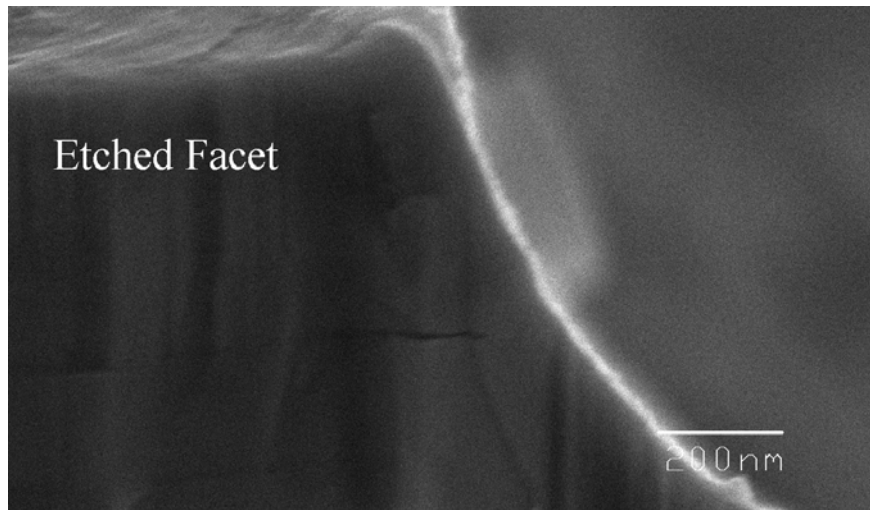


Fig 2.7 SEM image of wet etched facet of the Edge Emitting structure

From the SEM image of the facet we can see that the facets are concave in nature although the surface-roughness is less than $0.1\mu\text{m}$, which for $4\mu\text{m}$ emission is much better than the tolerance limit of $\lambda/10$ for lasing⁵. Therefore, it is clear that the reason for not getting cavity modes from this structure was actually the absence of a proper Fabry-

Perot cavity that is needed to achieve stimulated emission inside the gain medium. From the experiment it seemed that wet processing alone was not able to form parallel laser cavity. A combination of dry etching followed by wet processing could help achieve the cavity requirements. Considering the high quantum efficiency, tunability and ease of fabrication, light emitters using IV-VI materials grown on BaF₂ substrate generated lot of hope in us as a promising candidate for Mid-IR laser. Hence, to solve the existing problem of cavity formation, plasma etching of IV-VI material had been started in collaboration with a vendor, to form laser cavity. Figure 2.8 shows the first step towards plasma etching. Electroplated thick gold was used as a mask for the etching.

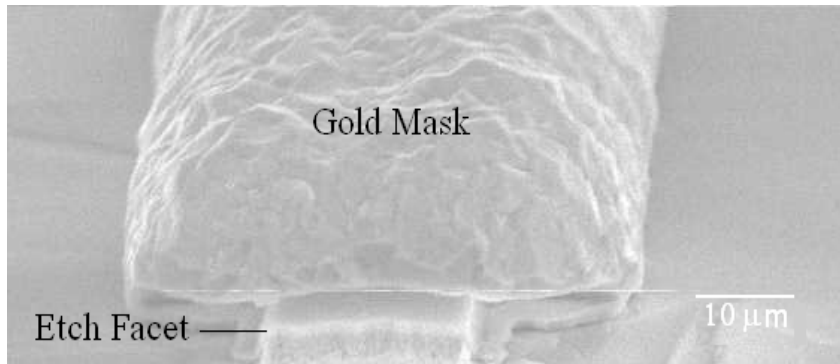


Fig 2.8 SEM image of dry etched cross-section of a PbSe layer

For masking, 40 μm wide stripes of plated gold was fabricated on a single layer PbSe sample grown on BaF₂ to check dry etching conditions and outcome thereafter. It can be seen from Figure 2.8 that etched facet was quite rough. This happened because of the roughness of mask material. Hence, the idea of using plated gold as mask and the first attempt towards dry etching was not quite successful. But the work on dry etching was continued. Details of dry etching results obtained afterwards are discussed in Chapter 3.

2.2 Antireflection Coating for Diode Pumped Lead-Salt Light Emitters

It was mentioned at the beginning of this chapter that a renewed interest has been observed in optical pumping. Optical pumping with low cost, compact, high power III-V diode lasers has recently been employed by several research groups to demonstrate emission and lasing in the mid-infrared wavelength region^{6 7}. The pumping source, mostly used for these applications are III-V diode lasers emitting in the 980 nm - 1 μ m wavelength region. Lead-chalcogenide semiconductor materials having higher refractive indices in this wavelength region reflects back almost 40-42 % of the incident pumping light allowing only a small fraction of pump-light to enter the high-index semiconductor. So there is a need to minimize the reflectance of the light-receiving surface in order to boost the pumping efficiency and increase the generation of photons in the semiconductor material. Thus, an antireflection coating material has been proposed to increase the pumping efficiency of the optically pumped lead-chalcogenide semiconductor light emitters⁸. The antireflection property of the coated film was investigated by FTIR–spectroscopic reflectance measurement. Room temperature continuous wave (CW) mid-infrared photoluminescence (PL) of the diode pumped IV-VI semiconductor multiple quantum well (MQW) structures was studied before and after coating the antireflection material. The mid-infrared photoluminescence from the coated structure was increased up to 4-times when compared with uncoated structure.

2.2.1 Zero reflectance condition for the proposed coating

The necessary and sufficient conditions for a single layer coating to produce zero reflectance are⁹:

$$n_1 = (n_0 n_s)^{\frac{1}{2}}, n_1 < n_s \quad (1)$$

and
$$\phi_1 = (2m - 1) \frac{\pi}{2}, m = 1, 2, 3, \dots, \quad (2)$$

where n_0 and n_s are the refractive indices of the surrounding medium and substrate respectively. The substrate is actually the epitaxial layer in our case. $\phi_1 = 2\pi n_1 d_1 / \lambda$ is the phase thickness of the coating, where d_1 and n_1 are the geometrical thickness and refractive index of the coating material. Here λ is the wavelength of the incident light. In practice m is usually chosen to be one, and the optical thickness $n_1 d_1$ is then one quarter wavelength. The reflectance of a quarter-wave coating is equal to zero at the wavelength corresponding to the optical thickness of quarter wavelength, if Eq. (1) is satisfied. If Eq. (2) is not satisfied, then the reflectance will indicate a minimum at the same wavelength. The position of the reflectance minimum of a surface coated with a quarter-wave coating depends on the optical thickness of the coating and shifts towards longer wavelengths as the optical thickness increases.

The average refractive index n_s of PbSe/PbSrSe QW in the wavelength region of 980-982 nm is between 4.4-4.48. Although the refractive index of $\text{Pb}_{0.97}\text{Sr}_{0.03}\text{Se}$ at this particular wavelength is not accurately known but an approximate value was calculated from the experimentally obtained transmission spectra by the methods described elsewhere¹⁰. Therefore, when the surrounding medium is air ($n_o=1.0$), the refractive index of the coating material to be used for a quarterwave antireflection coating should

have a value between 2 and 2.1, according to the index condition in Eq.(2). So we have chosen the binary material SrSe as the coating material, whose refractive index in this wavelength region is around 2.0¹¹.

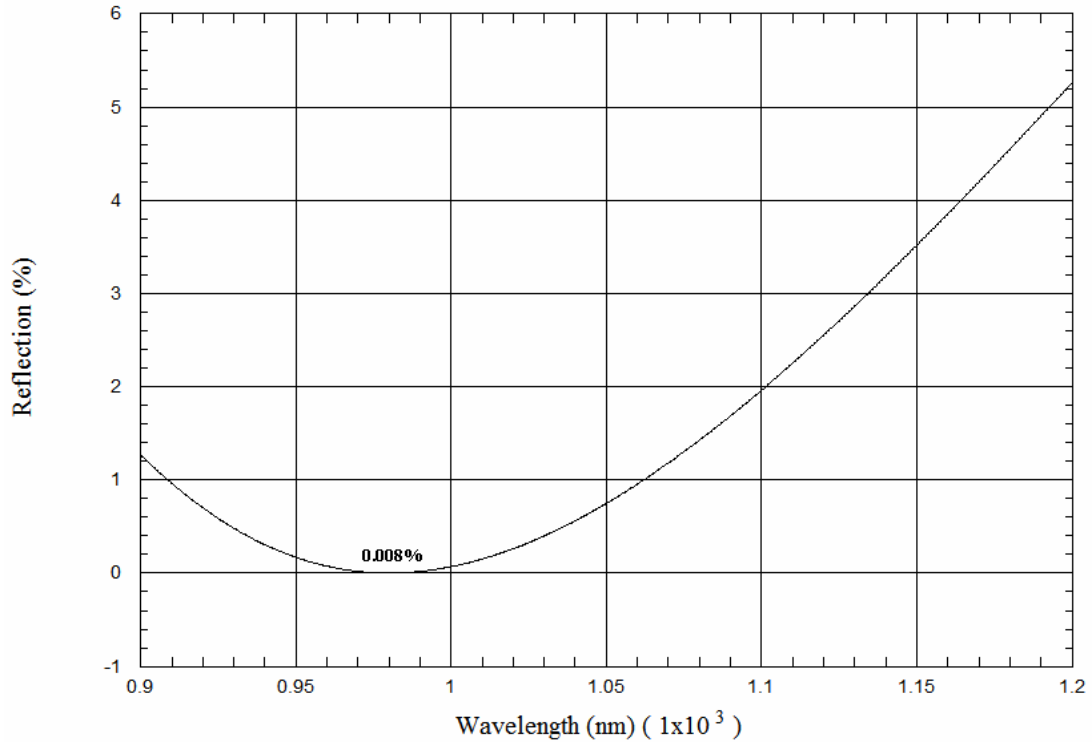


Fig 2.9 Simulated reflection spectrum of SrSe coated MQW sample as a function of pumping wavelength

The reflectance spectrum of the SrSe coated multi quantum well sample was simulated using FilmWizardTM and is shown in Figure 2.9. The figure shows clear minima in 970-990 nm wavelength region having a reflectivity of 0.008%.

2.2.2 Experiment

The $\text{PbSe}/\text{Pb}_{0.97}\text{Sr}_{0.03}\text{Se}$ multi quantum well structure was grown on [110] oriented Si substrate by molecular-beam epitaxy (MBE), using compound sources for PbSe and elemental sources for Sr and Se. The QW structure consisted of 18 pairs of 20nm PbSe well separated by 30nm thick $\text{Pb}_{0.97}\text{Sr}_{0.03}\text{Se}$ barrier. A CaF_2 buffer layer was grown on Si substrate before the growth of IV-VI semiconductor material. After a photoluminescence measurement these samples were transferred back into the MBE chamber for the growth of SrSe antireflection coating material, followed by a thorough ultrasonic cleaning using acetone, methanol, propanol and DI water. SrSe was grown on the MQW sample at a growth rate of $0.6\mu\text{m}/\text{h}$.

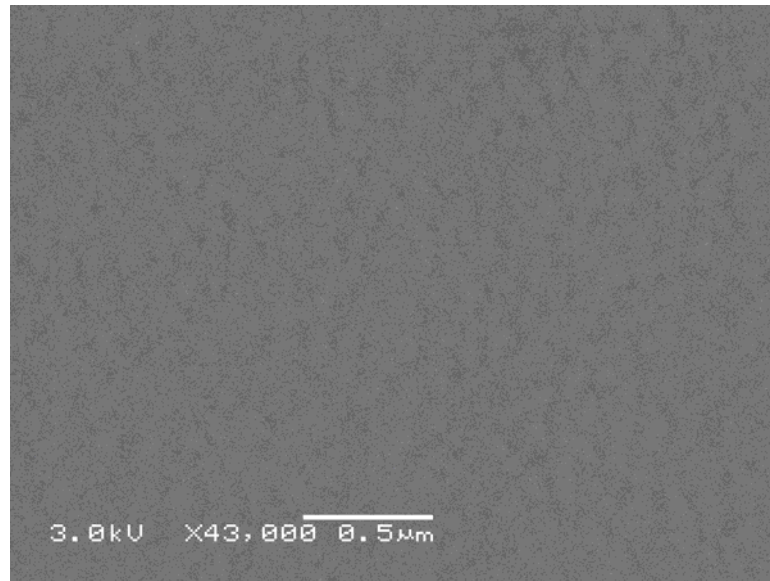


Fig 2.10 SEM image of the surface of SrSe coated MQW sample

The image above shows the surface morphology of the deposited material. Even after a CTE (coefficient of thermal expansion) mismatch between SrSe ($7.1 \times 10^{-6} \text{ K}^{-1}$) and PbSe ($19.4 \times 10^{-6} \text{ K}^{-1}$) no cracks has been observed on the surface. One of the technological

challenge for the AR coating of IV-VI material is thermal expansion coefficient difference between common coating materials and the IV-VI semiconductor which results crack on the surface of deposited material when cycled from higher temperature to ambient or much lower temperature to ambient and vice versa. But in our case most probably longer cooling time after growth helped to produce a crack free surface. A surface roughness of the order of 50 nm has been observed through the SEM image of the surface of the sample. After growth, the reflection spectrum of the sample was measured by a BRUKER IFS 66/S Fourier Transform Spectrometer using NIR source and a DTGS detector. The schematic of measurement set up is shown in Figure 2.11.

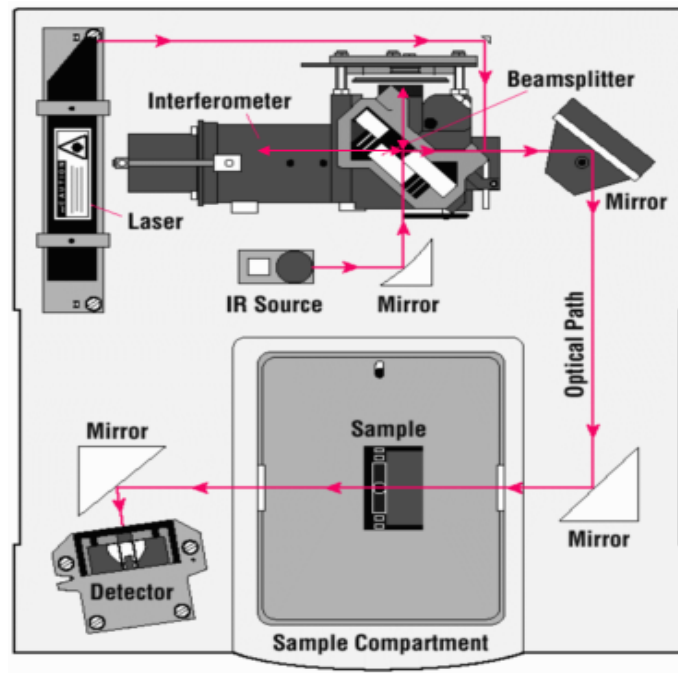


Fig 2.11 Schematic of reflection/transmission measurement set up

Before and after SrSe growth, the quantum well sample was mounted episcide up on a copper heat sink with silver epoxy and pumped by a 980 nm low-power CW (continuous wave) InGaAs diode laser to measure room temperature photoluminescence (PL) spectra from the quantum well sample. The laser beam was focused on to the surface of the sample with a spot size of 400 μm in diameter. The PL spectra were measured again with IFS 66/S spectrometer in single-channel mode at resolution of 1 cm^{-1} , using a LN_2 -cooled InSb detector having a rise time of 23 ns. Emission was measured at different intensities of pumping before and after the coating of SrSe on MQW sample, and the emitted power was calibrated by a standard blackbody reference source.

2.2.3 Results and discussion

Figure 2.12 shows the room temperature reflection spectrum, obtained experimentally from the multi quantum well sample coated with SrSe.

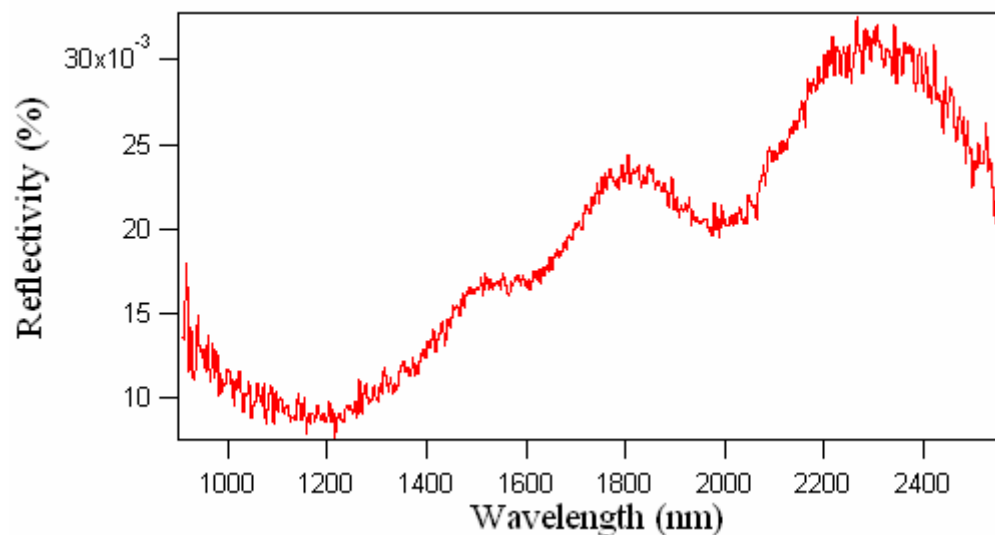


Fig 2.12 Room Temperature Reflection spectrum of SrSe coated MQW sample

The spectrum shows around 0.01% reflectivity in 980-990 nm wavelength region, which is in good agreement with the theoretically simulated reflectivity. But at this point I would like to mention that with such a low signal, the noise part in the FTIR becomes quite dominant, so the spectrum becomes noisy, which is also clear from the above figure and so it becomes inconvenient to consider this data as an absolute measure. Rather qualitatively it gives us the idea that the reflectivity is so low it almost approached the detection limit.

Figure 2.13 shows the continuous wave (CW) photoluminescence (PL) spectra of the SrSe coated PbSe/PbSrSe multi quantum well sample at room temperature. CW emission from the sample was recorded at different pump power. This figure shows the spectra where the pump power was increased gradually from 65 mW to 1.1 W.

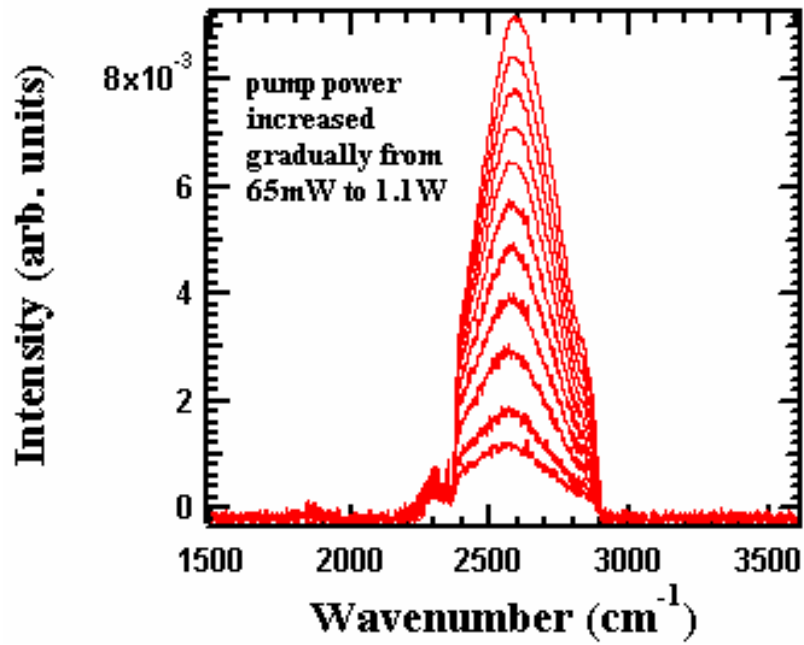


Fig 2.13 Room Temperature PL spectra of SrSe coated MQW sample

A blue-shift in the peak emission has been observed with the increased pump power. This is because of the heat generated and temperature induced band gap change in the epilayer.

Figure 2.14 shows the calibrated output power of PL emissions at room temperature as a function of pump power density, before and after coating SrSe on the sample. The output power increases monotonically with the increase in pump intensity, indicating the spontaneous nature of emission from the sample. Before coating of SrSe the maximum CW output power from the sample was $40\mu\text{W}$ for a pump power of 1W on a spot of $400\mu\text{m}$ diameter. But after the coating of SrSe the maximum CW output power from the same sample increased to $95\mu\text{W}$ for the same pumping condition.

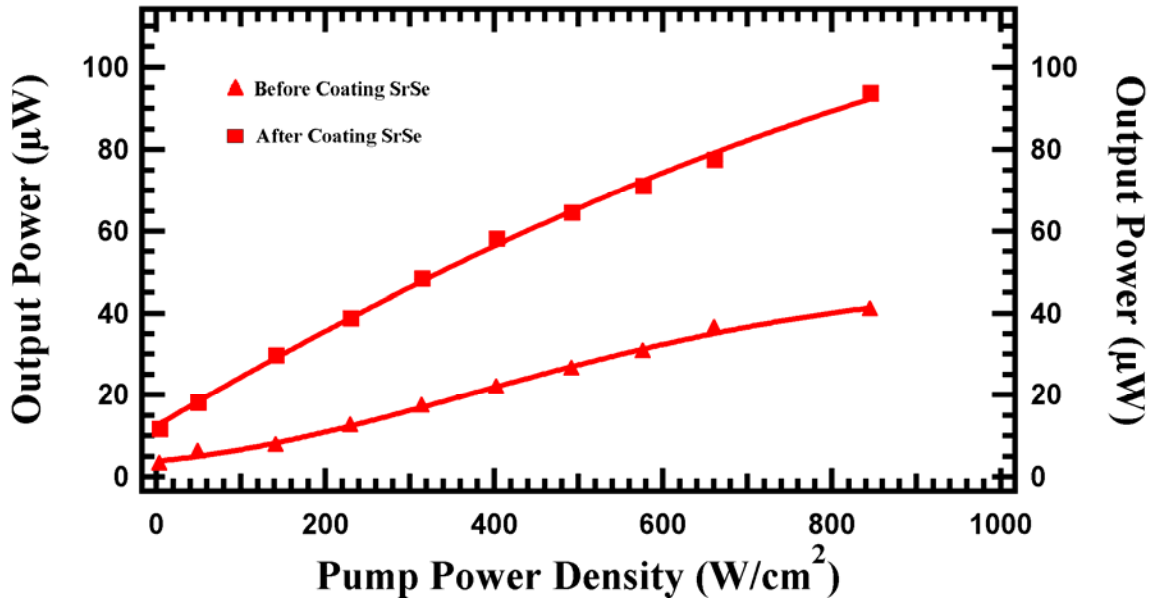


Fig 2.14 Room temperature CW output power from sample as a function of pump power density

Although further measurement of output power from the sample at a relatively higher pump power was limited by the low power of the pumping laser. Also, the collection optics of the spectrometer contains a parabolic mirror that collects only 4.63% of the total emission from the sample considering a conservative value that emission occurs through

an angle -75° to $+75^\circ$. Therefore, the total emission from the sample before coating can be estimated as $863\mu\text{W}$ and after SrSe coating it is 2 mW .

In conclusion, an antireflection coating material has been demonstrated for the optically pumped IV-VI semiconductor devices. Coating its surface with a quarter-wave single layer coating of SrSe reduced the reflectance of the IV-VI semiconductor surface in the studied spectral range covered by the pump light. The position of the minimum in the reflectance spectra obtained for single-layer coatings can be shifted to a desired wavelength by changing the optical thickness of the coating. Also, the minimum reflectance band obtained with this single-layer coating is sufficiently wide for practical optical pumping. The coating enhances the pumping efficiency thus by allowing more photons to be absorbed in the device material. Almost 3-4 times increment in the output power of the PbSe/PbSrSe light emitting structure has been demonstrated. The multiple-quantum-well sample used in the experiment is not the best one and epi-side-up mounting has been used during photoluminescence measurement. Therefore, it can be anticipated that better heat management could lead to an emission intensity of approximately 10 mW with this coating.

2.3 References

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Chapter 3

[110] Oriented Edge Emitting Lasers Grown on BaF₂ Substrate

3.1 Potential Advantages of [110] Orientation for IV-VI Pb-Salt Lasers

The first laser action of lead chalcogenides was obtained from PbTe in 1964¹; two year after the first semiconductor GaAs laser was made. These lead salt diode lasers were usually fabricated on [100] orientated lead salt wafers because of the availability of two parallel facets that can be cleaved along the easiest cleavage plane {100} to form a Fabry-Perot cavity. However, it has been theoretically demonstrated that [110] orientation is the best orientation for QW lead salt laser fabrication². Theoretical simulations for the [110] orientation showed higher gain, higher efficiency, compared to conventional [100] orientated lasers.

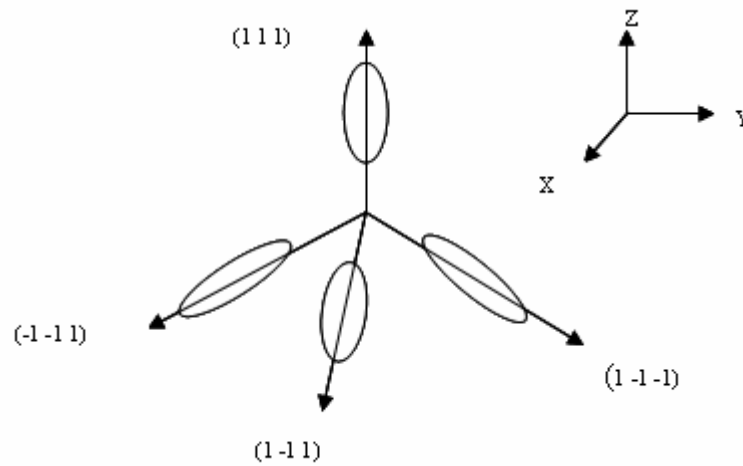


Fig 3.1 Illustration of PbSe L-valley energy minima

Figure 3.1 illustrates the configuration of L-valley constant energy surfaces in k space for IV-VI materials. The gain function due to a single L-point band extremum is proportional to the momentum matrix element³.

The momentum matrix element in terms of interband matrix element can be expressed as,

$$(p_x)_{vc}^2 = (P_l^2 \cos^2 \theta + P_l^2 \sin^2 \theta) \frac{m_0^2}{\hbar^2} \quad (3.1)$$

,where θ is the angle between a single [111] direction and the electric field vector of the radiation. Table 3.1 shows the effective masses for electrons and holes for different orientations for the energy minima shown in Figure 3.1.

Table 3.1 Effective mass along different orientations for L-valley energy minima ²

Surface Orientation	m_x	m_y	m_z	Degeneracy
[100]	m_t	$(m_t+2m_l)/3$	$3m_t m_l/(m_t+2m_l)$	4
[110]	m_t	$(2m_t+m_l)/3$	$3m_t m_l/(2m_t+m_l)$	2
	m_t	m_l	m_t	2
[111]	m_t	m_t	m_l	1
	m_t	$(m_t+8m_l)/3$	$9m_t m_l/(m_t+8m_l)$	3

For PbSe, $m_l \approx 2m_t$ (both electrons and holes). When growing QW structures along [111] orientation, in which the effective mass m_l in the longitudinal valley is heavier than that of the three transverse valleys, the sub-band energies of the oblique valley increase faster. Hence, 1 $\rightarrow$ 1 sub-band transition of the longitudinal valley becomes lowest. The degeneracy is then lifted.

Similarly, [110]-orientation partially lifts the degeneracy. However, four energy valleys in [100] orientation, stay degenerated. For QW structures when degeneracy is lifted off on [111] and [110] orientations, the gain is determined by the angle in equation (3.1) and the quasi Fermi energy levels.

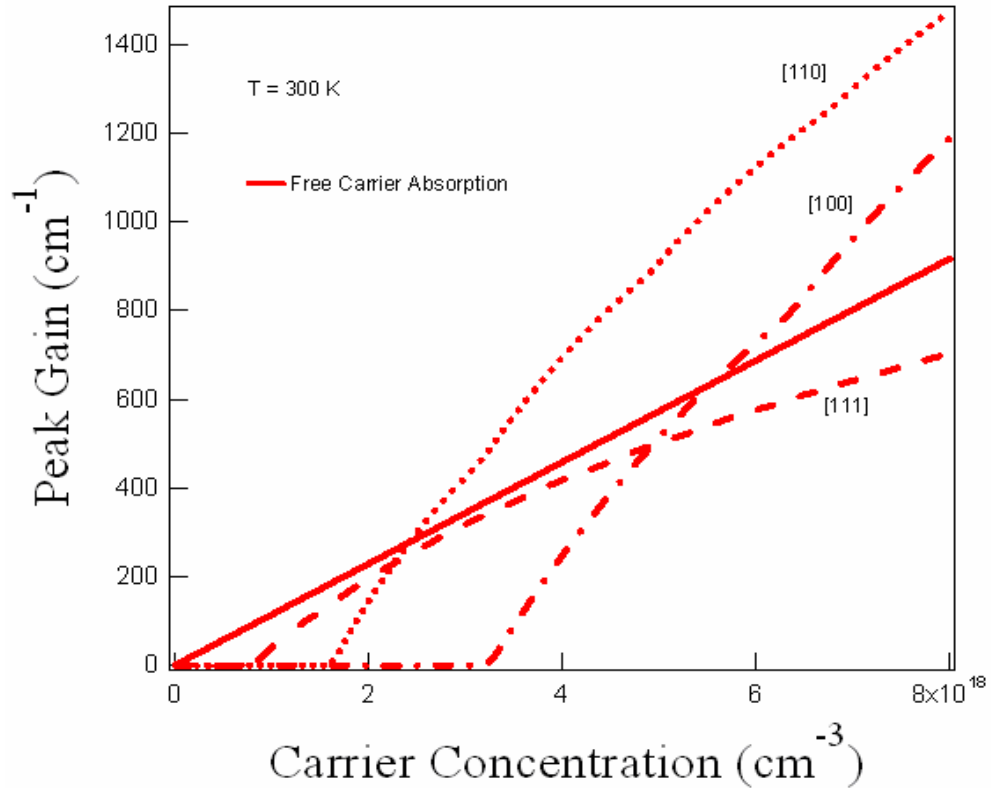


Fig 3.2 Calculated peak gain for [111], [100] and [110] QW structure at different injected carrier concentrations at 300 K. The free carrier absorption (solid line) is shown as well ².

Figure 3.2 shows the calculated peak gain along [111], [110] and [100] orientations together with the free carrier absorption loss $\sigma_F N$. As can be seen that [111] orientation has the lowest threshold concentration. For low loss laser devices such as VCSEL, [111]-orientation is best suited for low threshold operation. [111]-gain increases at a rate slower than the increase of free carrier absorption loss. Therefore, [111] orientation would tend

to have gain saturation for a relatively high loss device such as an edge emitting laser. For edge emitting lasers whose loss is significantly higher than VCSEL, higher gain is needed. [110] orientation provides the highest gain among all these three possible orientations for laser fabrication. For example, at 300K the [110]-gain is about twice as high as [100] gain. With the lifted degeneracy for structures on [111] and [110] orientation, less number of valleys along is filled. This causes the quasi-Fermi levels to separate more from the band edges. This in turn lowers the threshold and increases the gain, as the Fermi energies are in the exponential term of the gain equation. As the injected carrier concentration increases, the density of states increases proportional to the number of degenerate valleys. The combination determines the gain value. Although the calculations using the gain expression for bulk material is not as accurate as direct simulation of gain in a lead salt QW structure, however it should give some insights as to why different orientation could provide different gain. It is worthwhile mentioning that QW gain should be much larger than the bulk material gain because of the stronger electron-hole coupling.

Another significant advantage of [110] orientation is the low dislocation density. The mechanism of strain relaxation in IV-VI materials is by glide of dislocations. This mechanism will be quite clear from Figure 3.3, which is actually the schematic showing the geometry of dislocation gliding, on the next page.

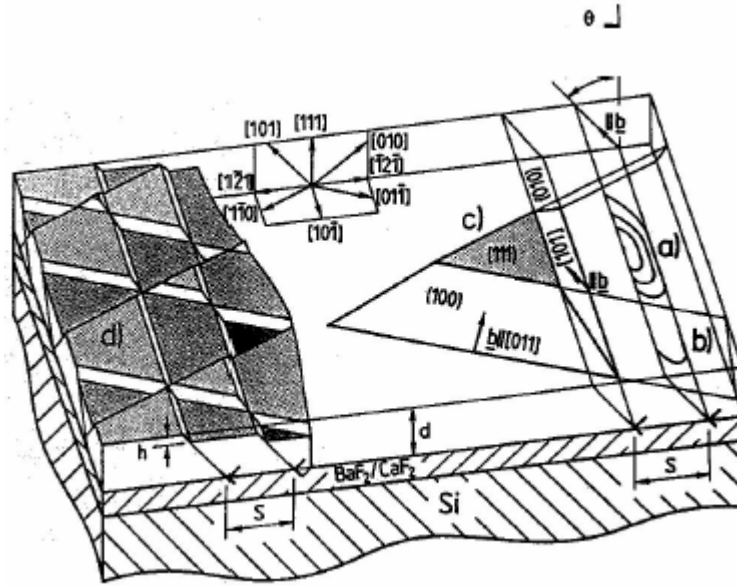


Fig 3.3 Schematic drawing of the arrangement of the $\{100\}\langle 110\rangle$ glide system for the NaCl type PbSe(111) layers⁴.

It is evident from Figure 3.3, the Burgers vectors are of type $a/2 \langle 110\rangle$. However, unlike the zinc-blende type semiconductors, the primary glide planes of IV-VI Pb-salt materials are $\{100\}$. The critical resolved shear stresses are low for these planes (while they are much higher in the second glide system with $\{110\}$ type glide planes). The $\{100\}$ planes are inclined to the $[111]$ oriented layer surface. Therefore, each time a mechanical strain tends to build up (e.g. during the MBE growth because of the lattice mismatch or due to temperature changes), this strain can relax by the glide of dislocations on the $\{100\}$ type planes since the Schmid factors are high for this growth orientation. In the contrary, for $[100]$ oriented layers, the Schmid factors for glide in the primary $\{100\}$ planes are zero, and therefore, no glide can occur in the main $\{100\}$ glide system. The misfit dislocations lie along $\langle 110\rangle$ directions, i.e., the intersection of the $\{100\}$ type glide planes with the

[111] oriented interface. They are arranged with three-fold symmetry. When a misfit dislocation is formed by glide of a threading dislocation no defect remains in the interior of the layer, instead, the misfit dislocation lies at the interface and a single atomic step remains at the surface. If more than one dislocation glides on the same slip plane, the surface step is higher.

3.2 Development of Novel Surface Preparation Technology of {110} BaF₂ Wafers for Epitaxial Growth

The first step towards the fabrication of [110] oriented laser on BaF₂ substrate was the development of surface preparation technology for [110] oriented wafers for the epitaxial growth of IV-VI semiconductor layers. Since [110] orientation is not the natural cleaving direction of BaF₂ crystal, {110} BaF₂ is obtained by cutting the crystal along [110] orientation. Therefore, the surface of an unpolished substrate is severely rough; contains surface defects and grooves. Figure 3.4 shows the SEM image of an unpolished {110} BaF₂ wafer, as bought from a commercial vendor.

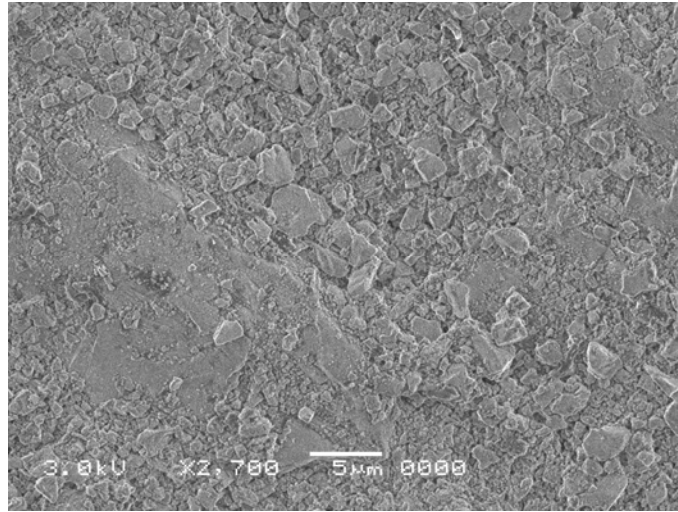


Fig 3.4 SEM image of the surface of unpolished {110} BaF₂ wafer

The rougher the surface of the wafer the smaller the chances of getting single crystalline quality of the epitaxial layer grown on that surface. But all these drawbacks can be eliminated by polishing the substrates. Polishing helps in achieving surface planarity, removes the surface roughness, and lowers the defect density on the surface as well.

Epilayers grown on a polished substrate should have a better quality and also at the same time enables easier processing of the device.

As a part of this research a novel chemical mechanical polishing recipe was developed to make epi-ready [110] oriented BaF₂ substrate for MBE growth. There were several challenges regarding this process. Epi-ready BaF₂ substrates are not commercially available. Also to date there is no report available on polishing of [110] oriented BaF₂ wafers. Metal halides are not common materials for epitaxial growth; rather they have wide applications in IR windows. For these applications the surface flatness needed is not comparable to the flatness required for an epi-ready wafer. While developing a polishing recipe for {110} BaF₂, the biggest of all the challenges was the extremely low Mohs' hardness value of BaF₂ crystal. Mohs' hardness is actually the physical property of a material that represents a materials resistance to scratching. And among all metal fluorides, BaF₂ has the lowest Mohs' hardness of 3. Diamond is the material available in nature having the maximum Mohs' hardness value of 10. Now all the abrasives and chemical slurries available commercially in the market for chemical mechanical polishing contain either Alumina (hardness: 9) or colloidal silica (hardness: 6 -7) that are much harder than BaF₂. Therefore, commercially available slurry materials for chemical mechanical polishing do not have the ability to produce scratchless surface of BaF₂, regardless of their particle size. In addition, most of the chemical polishing cloth available in the market was slightly harder and was found to scratch BaF₂. Therefore, a polishing method of BaF₂ that overcomes all these shortcomings needs to be developed. After a thorough research and rigorous experiments a novel polishing recipe was

developed in our lab, containing both mechanical and chemical material removal steps for $\{110\}$ BaF₂.

3.2.1 Fundamentals of Chemical Mechanical Polishing (CMP)

Chemical mechanical polishing has come to occupy the role of an enabling technology for the semiconductor industry in its drive toward gigabit chips and sub-quarter micron feature sizes. At the moment, it appears that the global planarization necessary for establishing reliable uniform interconnections or growth substrates can be achieved only by using CMP.

CMP is described as a combination of mechanical abrasion and chemical reaction process. According to M. Moinpour *et al.*, CMP depends on at least sixteen parameters; a major parameter among them is the composition of the slurry⁵. The development of an invariable CMP process needs consideration of many variables such as abrasive type and size; slurry chemicals including concentration and pH; percentage of solid fraction; surfactants; buffering agents.

According to Sun Hyuk *et al* wafer polishing can be divided in two steps- 1) stock or bulk material removal and 2) final polishing step. In the stock removal step, layers of wafer-materials from the wafer surface are effectively removed and in the final polishing step flatness of the wafer surface is achieved⁶. The material removal rate is in turn a function of the concentration of chemicals and abrasives in the slurry⁷. In a CMP process the polishing pad and the slurries play a vital role, as the formation of CMP by-product by slurry takes place between the interface of polishing pad and the wafer. The concentration of slurry between the wafer and the pad also controls the rate of formation of chemical byproduct in this process. Hence, a continuous flow of slurry is required to keep a consistency in the material removal process⁸.

Slurry stability is another great concern in a CMP process. Abrasive particles in the slurry agglomerate; thus by modifying the slurry compositions. Particle agglomeration causes hefty defects on the wafer surface as well as on the polishing pads, during CMP. As a matter of course, mechanical grinding or shearing during the polishing process can detach the agglomerates as they remain attached by weak Vander-Waals force ⁹.

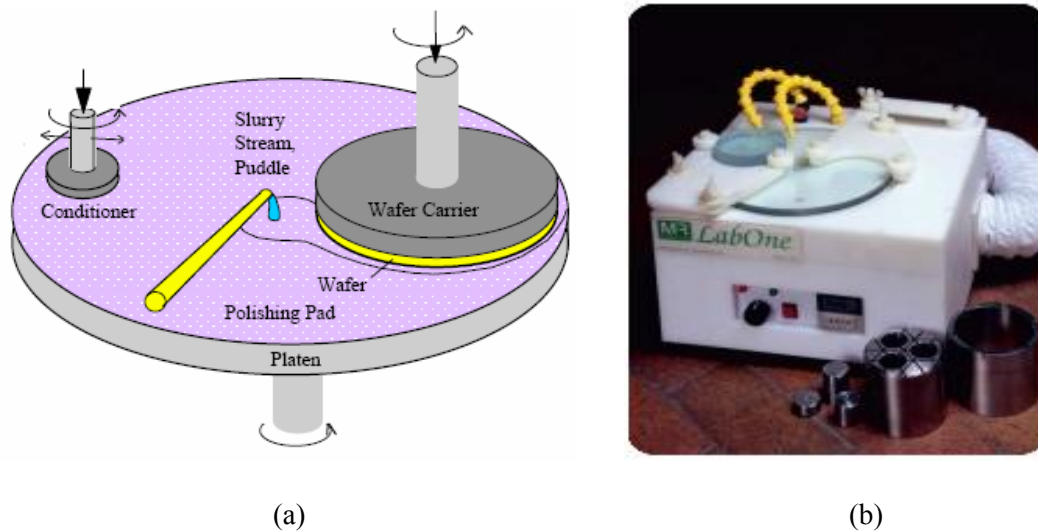


Fig 3.5 Schematic of a generic rotary polishing tool (a); Bench Top LabOne polisher used in optoelectronics laboratory of OU for Chemical Mechanical Polishing (b)

The substrates or wafers are mounted on a wafer-carrier during polishing. Depending on the design of the polisher, external weights or wafer carrier itself provides the pressure on the wafer needed for polishing. The schematic of a generic CMP tool and the picture of the CMP tool used by us in the optoelectronics laboratory of OU are shown in Figure 3.5. During the CMP process, the wafer and polishing pad rotate against each other keeping the slurry at the middle of pad and wafer. The speed of rotation of the wafer plays an important role in controlling material removal rates of wafer¹⁰. However, the removal

rate is actually function of the product of pad pressure and relative velocity between pad and wafer, and is given by Preston's Equation¹¹. The smoothness that can be attained after CMP can be approximated by the Hertzian penetration depth:

$$R_s = \frac{3}{4} \frac{\phi P}{2K_p E}$$

,where Φ is the abrasive particle diameter, P is polishing pressure, E is Young's modulus of wafer material and K_p is a constant related to density of particle. $K_p=1$ for closely packed particle.

“Polishing pads are considered to be the major consumables which affect the within-wafer and wafer-to-wafer non-uniformity (WIWNU and WTWNU)”¹². Hence, CMP performance is also get affected by the mechanical properties of polishing pads. The macrostructure of the polishing pad and the relative motion of the polishing pad and the wafer control the movement of the slurry between pad and wafer¹⁰. A polishing pad consists of peaks and valleys. The role of valley is to hold slurry and carry it to the contact region of pad and wafer by the relative movement between wafer and polishing pad and also carry by-products from the interface region of pad and wafer. And the role of peaks is to withstand the stress on the wafer. The quality of the polishing pad degrades with time because of this stress caused by abrasive particle. This also results in a sharp decrease in the material removal rate (MRR) with the increment in contact area and decrease in the contact pressure¹³. Deformations in the polishing pads such as dishing and thinning have a negative effect on wafer surface uniformity. Irregularities on the pad surface cause severe non-uniformities on wafer surface¹⁴. Hence, the pre-conditioning of the pad and conditioning during polishing is a key issue to a successful CMP process.

Several types of pad conditioning schemes are out there in the market. Appropriate wet and dry conditions should be carried out to prevent deterioration of the polishing pad. Conditioning also helps to improve the substrate surface and to keep a consistency in the polishing recipe.

3.2.2 CMP of {110} BaF₂

At the beginning of this development process, very few polishing recipes, that are available for [111] oriented BaF₂, were attempted and applied on [110] orientation as well. Below is a brief summary of methods used and results obtained from them.

Method I

Earlier R. F. Bis et al. introduced a polishing method of BaF₂ using a mixture of two solutions (a) 30% H₂SO₄ with 70% H₂O (b) 40% HCl with 60% H₂O. According to that process, the polishing pad is first soaked with DI water and the two solutions are introduced drop by drop in a ratio of 4:6 i.e., four drops of H₂SO₄ solution and six drops of HCl solution. They recommended continuing polishing with the mixture and adding only HCl solution in case the polishing slows down. The estimated time for this process is approximately 4 hrs but it varies according to the height of the cleavage steps on the substrate surface. After polishing, the substrates are cleaned in a solution of 80% aerosol and 20% acetic acid¹⁵.

This method was tried on [110]-oriented BaF₂ substrates. The process was initially started with 14 drops/min. The etch rate was observed to be 3µm/min. To increase the etch rate at initial stage, later on solution flow increased to 19 drops/min and finally to 30 drops/min and polished for an hour. It took about an hour to remove all surface irregularities that were initially there on the unpolished substrate. But after this one hour the surface of the substrate was found with deep scratches. In order to repair the scratched surface the substrates were further polished with a solution of HCl and DI water in the ratio 4:6 for approximately 17 minutes. It did not produce desirable results and a further increase in surface scratches was observed, which is shown in Figure 3.6.



Fig 3.6 Nomarski image of the substrate surface polished with the recipe of Method-I

Method II

In the second method proposed by James Harrington *et. al.* the substrates are first polished on sandpaper of decreasing coarseness followed by a mechanical polishing with selected polishing abrasives of decreasing particle size till a relatively scratch free surface is obtained¹⁶. After mechanical polishing, according to this process, the chemical cycle consists of an immersion cycle of mechanically polished substrates into a solution of concentrated sulfuric acid and acetic acid in the ratio of 4:9 for up to several hours. This cycle is supposed to remove fine scratches remained after mechanical polishing. The time of polishing can be varied based on observations. Several combinations of these variable factors were tried but the surface quality never improved. With this recipe each time the substrate immersed into the chemical solution after mechanical polishing, it instantly became smoky and severely damaged. Even after seven to eight cycles of mechanical and chemical polishing the situation did not improve. A scratch free substrate surface was not obtained with this method. Figure 3.7 shows the Nomarski image of the substrate surface after mechanical and chemical cycle following this method.

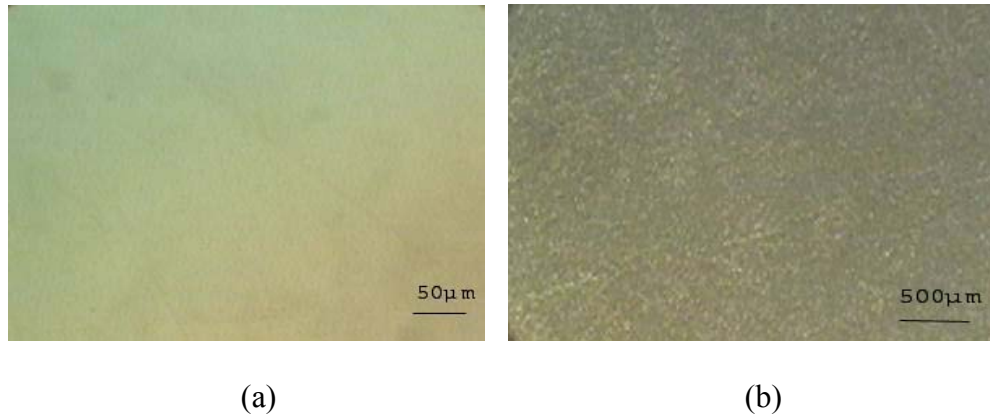


Fig 3.7 Nomarski image of the substrate surface polished with the recipe of Method-II. Surface after mechanical polish (a); surface after chemical cycle (b).

Method III

In this method a recipe proposed by Jr. Nordquist *et. al.* was followed¹⁷. The chemical polishing solution comprised of a mixture of 20 ml boric acid and 100 ml tartaric acid. This recipe produced better result than the previous ones but still surface quality was not acceptable for epitaxial growth. SEM image of the surface of the polished substrate obtained with this recipe is shown in Figure 3.8.

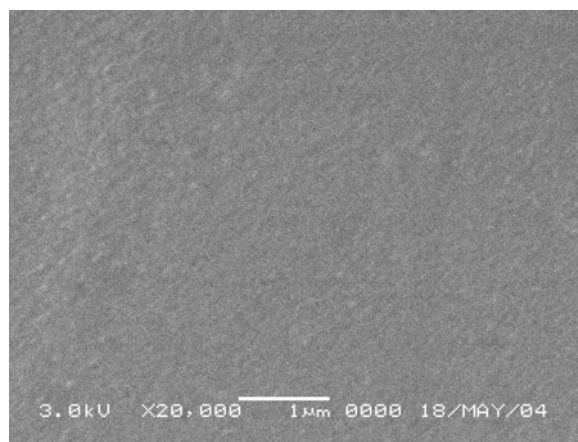


Fig 3.8 SEM image of the substrate surface polished with the recipe of Method-III

CMP with ChemPol

After experimenting with several polishing solution ultimately a novel polishing recipe that was developed for {110} BaF₂ was a multistage polishing. Each stages of polishing were carried out with different polishing solutions that are named as ChemPol I, ChemPol II and ChemPol IV. Smooth epi-ready wafer surfaces were achieved with this method of polishing. The recipes for ChemPol I to ChemPol IV are proprietary and subject to a non-disclosure agreement (NDA). The three step polishing method is sequentially written below:

- ❖ Reduction of surface roughness on back and front side of wafer with ChemPol I
- ❖ Rough polishing with ChemPol II
- ❖ Final chemical polishing with ChemPol IV

Step I: In the first step Chem Pol I is used as the polishing solution on a 1200 grit size SiC cloth till the substrate become transparent and blisters on the surface get removed. Below are the images at different stages of polishing with Chem Pol I.

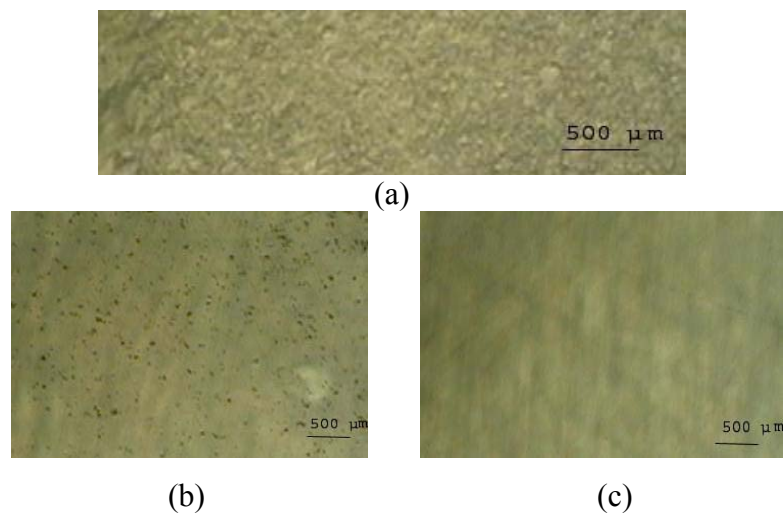


Fig 3.9 Nomarski image of unpolished substrate surface (a); substrate surface after 11 min (b); after 17 min (c) polishing with ChemPol I.

Step II: Once the substrate reaches the above stage, the next step is polishing with ChemPol II till no scratches are visible under 50X magnification of the Nomarski Microscope. ChemPol II is an alkaline solution containing high purity nano-abrasives. The cloth initially moistened using DI water and the polishing solution was added at a rate of 1 ml/min. The substrates were polished for 10 minutes and checked for scratches. After every run, the cloth was conditioned with DI water and the substrates were also cleaned with DI water as well. Cleaning the cloth at regular interval with DI water is important as the silica particles in the polishing slurry solidify quickly in air-contact on the cloths thus causing deterioration of the cloth and the substrate surface.

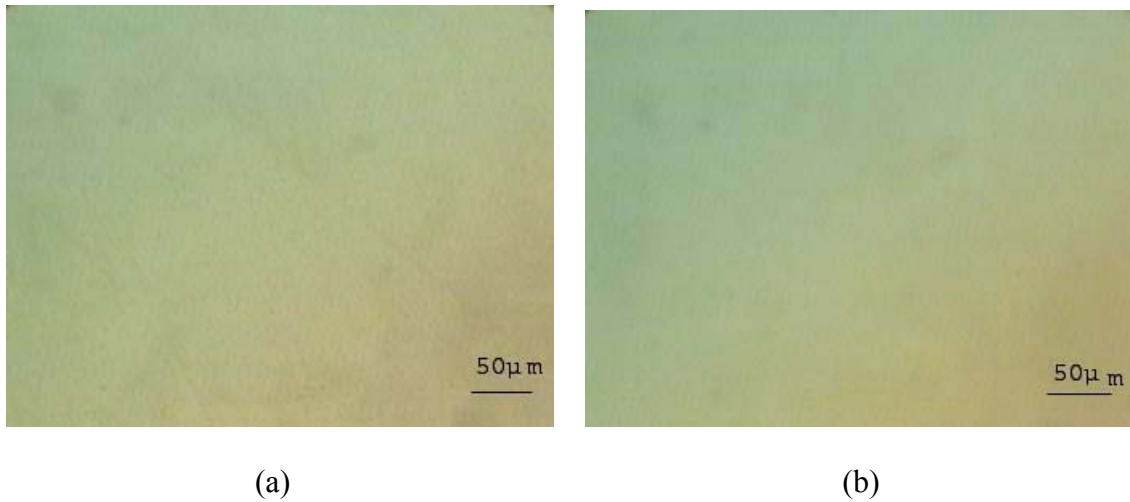


Fig 3.10 Nomarski image of polished substrate surface after 15 min (a); after 25 min (b) polishing with ChemPol II.

It can be seen in the above figure that after 15 min of polishing with ChemPol II still one or two fine scratches remain visible on the substrate. But after polishing 25 mins scratches become invisible at higher magnification of Nomarski Microscope.

Step III: The next stage involves final polishing using a purely chemical solution without any abrasive to attain a completely uniform surface with no scratches at the highest possible magnification of Nomarski and SEM. After polishing with the ChemPol II, although a scratch-free surface was observed under Nomarski but the SEM images showed light scratches and roughness on the surface. Figure 3.11 shows the SEM image of [110]-oriented substrate, after polishing with ChemPol II and before the final polishing stage.

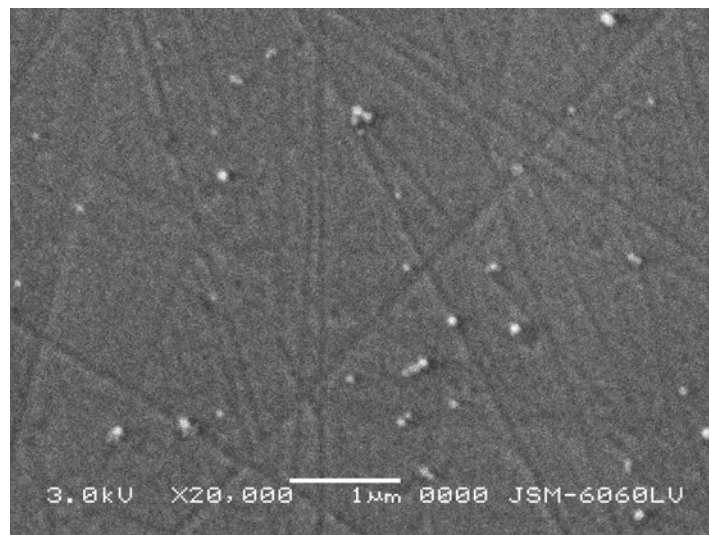


Fig 3.11 SEM image of the substrate surface polished with ChemPol II

It is observed from the above SEM that ChemPol II was not sufficient enough to produce epi-ready surface. Therefore, final polishing was carried out using yet another fresh solution, ChemPol IV for 30 seconds. The cloth was initially moistened using the same solution and the solution was added continuously at a rate of 1 ml/sec. The SEM pictures showed no surface non-uniformity or any scratches. Figure 3.12 shows the surface of a [110]-oriented substrate after final polishing with this new solution.

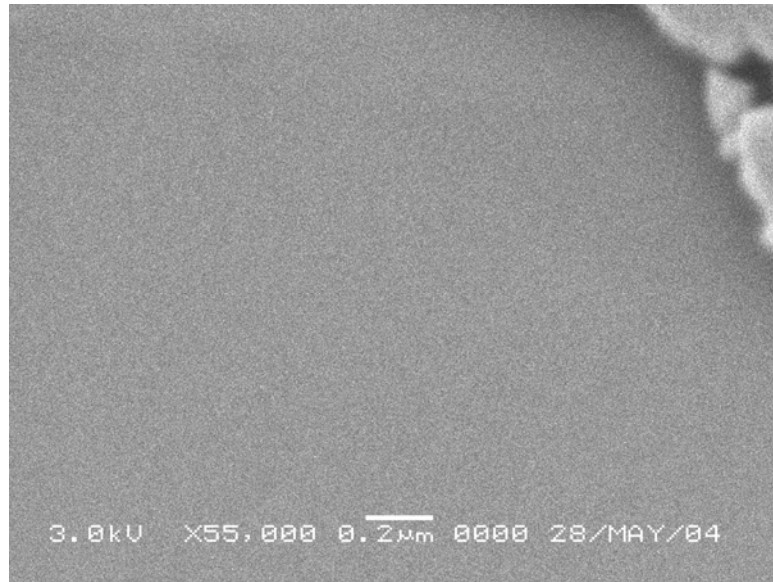


Fig 3.12 SEM image of the substrate surface polished with ChemPol IV

The image was taken at quite high magnification and no surface non uniformity was observed. Figure 3.13 shows the Nomarski image of a single layer PbSe sample that was grown on polished {110} BaF₂ wafer.

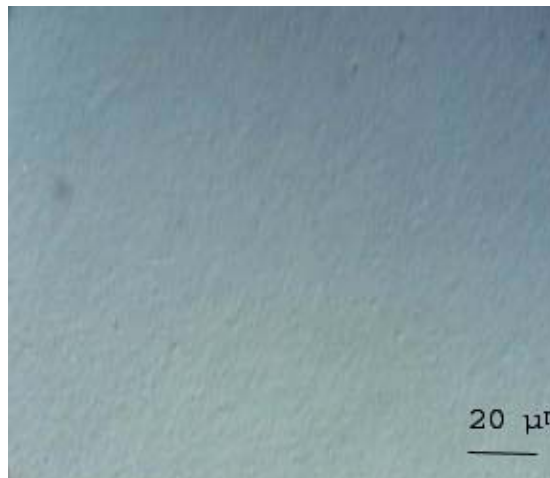
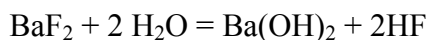


Fig 3.13 Nomarski image of PbSe epitaxy grown on polished {110} BaF₂ substrate

Chemistry behind the process

The principal etching solution used in this chemical process is de-ionized (DI) water. Hydrated surface of BaF₂ forms a thin layer of barium hydroxide Ba(OH)₂ by reacting with H₂O (basically the OH⁻ ion). The chemical reaction is written below:



Thus to remove few mono layers of barium fluoride materials from the surface of a BaF₂ wafer by forming metal-hydroxide at the interface of wafer surface and polishing pads, strict process control of several parameters including the etch rate, pressure on polishing pad , relative velocity between pad and wafer, was needed. Each parameter was optimized after a vivid research and chain of experiments. Control of etch rate was accomplished by adding appropriate amount of a moderator in the polishing solution. Pressure on Pad and the relative velocity was optimized by using design of experiment (DOE) technique. Also since the final polishing was completely chemical without any abrasive, the abrasion of reaction by-products was accomplished by the polishing pad only. Hence, after a thorough research a polishing cloth having large valley area but at the same time having less hardness and short nap was chosen.

3.2.3 Characterization of polished substrate after MBE growth

MBE growth of Pb-salt epitaxial layers on polished $[110]$ oriented BaF_2 substrate showed promising results¹⁸. During growth of PbSe/PbSrSe QW on polished BaF_2 substrate, *In situ* RHEED showed two dimensional growth modes. Figure 3.14 shows the RHEED patterns from $[11\bar{2}]$ BaF_2 orientation and of PbSe after one minute growth (33 nm).

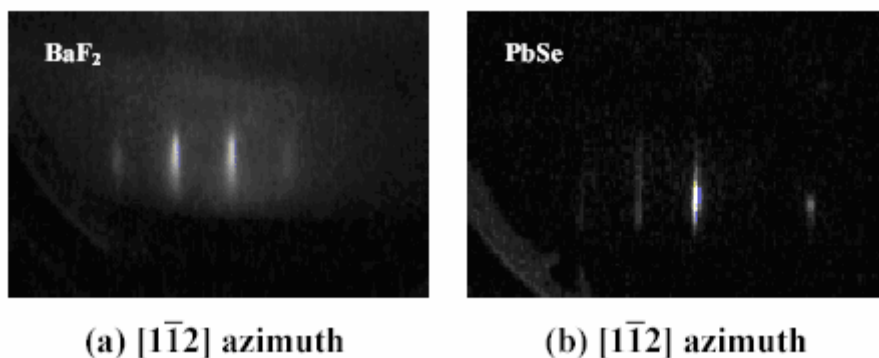
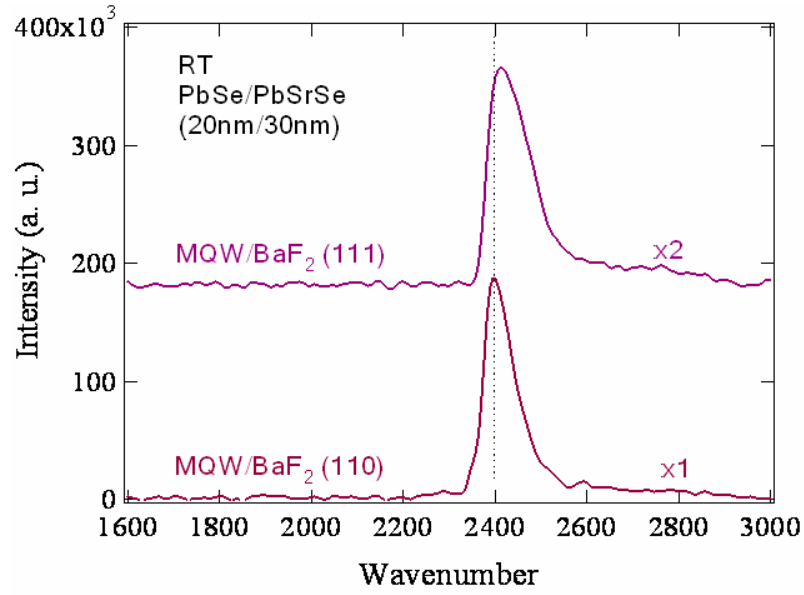
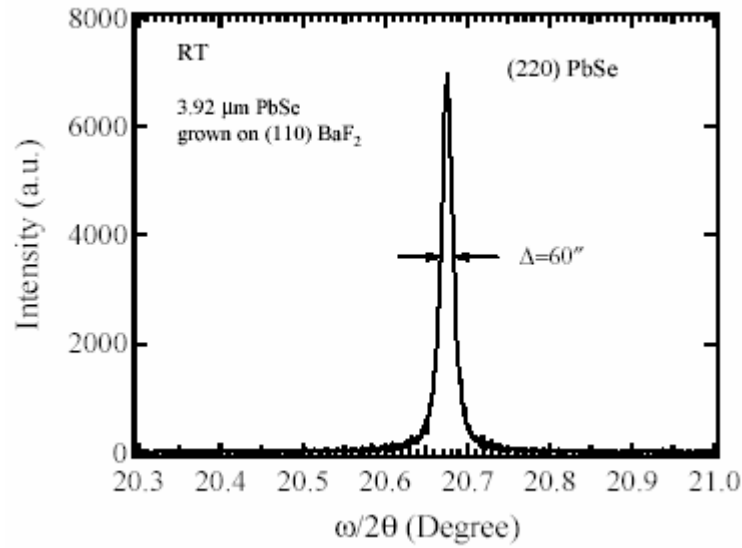


Fig 3.14 RHEED patterns from BaF_2 substrate (a) grown PbSe layer (b)

The 2D nature of the RHEED images depicts improved surface quality and single crystalline nature of grown epitaxial layer. The RHEED of the PbSe film has an equal spacing identical to that of the BaF_2 substrate which suggests the idea of same lattice registrations and better epilayer quality. After MBE growth, photoluminescence and HRXRD measurements were carried out to know the optical performance and material quality of the grown layers on polished wafer. PL intensity of a seven pair PbSe (20 nm)/PbSrSe (30nm) QW structure grown on $[110]$ sample was about 2 times higher than that of cleaved $[111]$ sample grown in the same MBE run¹⁸.



(a)



(b)

Fig 3.15 Comparison of room temperature photoluminescence on cleaved [111] and polished [110] oriented BaF₂ substrate (a) and HRXRD spectrum of the (220) reflection from a 3.9 μm PbSe thin film on [110] oriented BaF₂ substrate (b) ¹⁸.

Figure shows, the PL intensity of [110] sample is about 2 times higher than that of [111] sample. Figure 3.16 (b) shows (220) rocking curve of PbSe thin film which has a FWHM of 60 arcsec. All these results indicate high material quality. The dislocation density of PbSe thin film was estimated to be $1 \times 10^7 \text{ cm}^{-2}$ from rocking-curve measurements ¹⁹.

3.3 Development of [110] Oriented Edge Emitting Laser

It was mentioned before that commercially available lead salt diode lasers are fabricated on [100] orientated lead salt wafers because of the availability of two parallel facets that can be cleaved along $\{100\}$ to form a Fabry-Perot cavity. Now for fabrication of lasers grown on [110] oriented BaF_2 , Fabry-Perot cavities can be obtained by cleaving the structure along $\{111\}$ planes, which is the natural cleavage plane of BaF_2 . Figure 3.16 shows the orientation of $\{110\}$, $\{111\}$, $\{100\}$ planes. From the figure it is clear that for growth along [110] orientation, there exists parallel $\{111\}$ planes that can lead to the formation of cavity for a cleaved cavity laser.

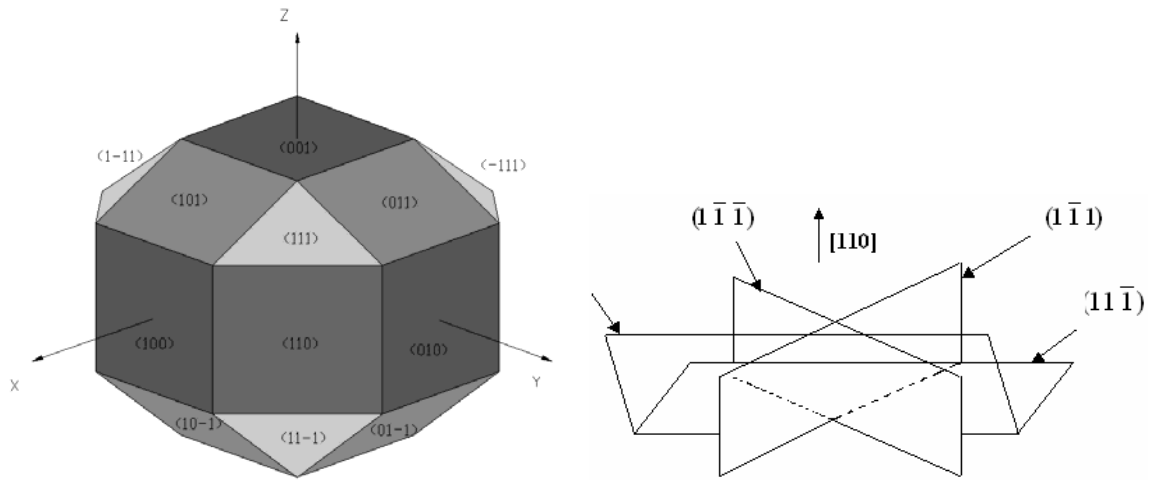


Fig 3.16 Crystal orientation showing different planes and their intersection

Another approach of having Fabry-Perot cavity for lasing is etching. With etching, parallel facets can be obtained along any direction. Wet etching and a step toward dry etching has already been discussed in Chapter 2. In this chapter, other than cleaved facet, progress made afterwards toward dry etched structures will also be discussed.

3.3.1 Cleaved facet laser

As a first step toward cleaved facet [110] oriented edge emitting laser (EEL), 7 pair PbSe/PbSrSe multi quantum well followed by 50 nm $\text{Pb}_{0.96}\text{Sr}_{0.04}\text{Se}$ optical confinement layer was grown on {110} BaF_2 for optical pumping of the device. A ridge waveguide structure with thick gold coating on top surface, for better heat dissipation, was planned for this laser. Figure 3.17 shows the schematic of layer and device structure.

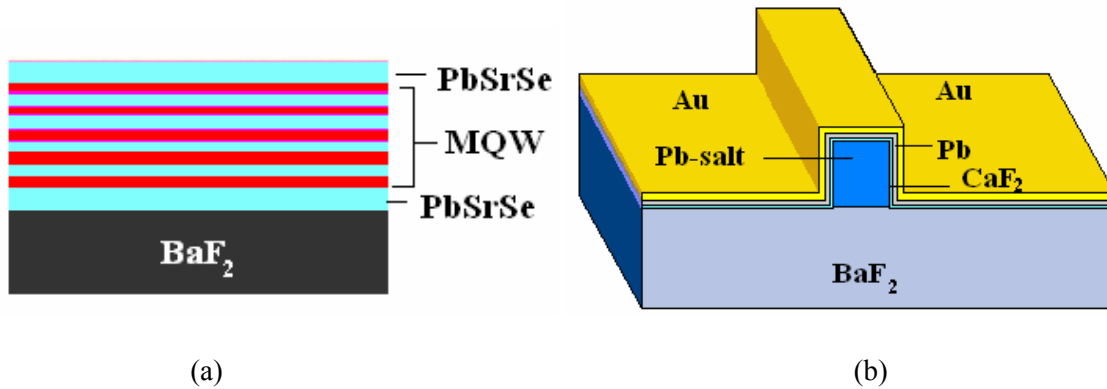


Fig 3.17 Schematic of layer structure (a) device structure (b) for optically pumped EEL on BaF_2

In the device structure, a thin layer of CaF_2 on top of ridge and entire sample served as passivation layer. Pb served as a seed layer for the electro deposition of Au. To form a ridge waveguide, the epitaxial layer was etched followed by a photolithography step. Before lithography the entire sample was cleaved along [111] orientation and then $9\text{ }\mu\text{m}$ wide photoresist stripes were fabricated perpendicular to the cleaving plane. Wet etching was performed to obtain the ridge. Because of isotropic etching, the width of the top portion of ridge narrowed down to $5\text{ }\mu\text{m}$ after etching. After the formation of the ridge the sample was cleaned thoroughly and transferred into the MBE chamber for the growth of CaF_2 and Pb layer on the sample. After this deposition, a $1.5\text{ }\mu\text{m}$ thick layer of

Au was electrodeposited on the entire sample. The figure below shows the Nomarski image of the sample at different stages of fabrication.

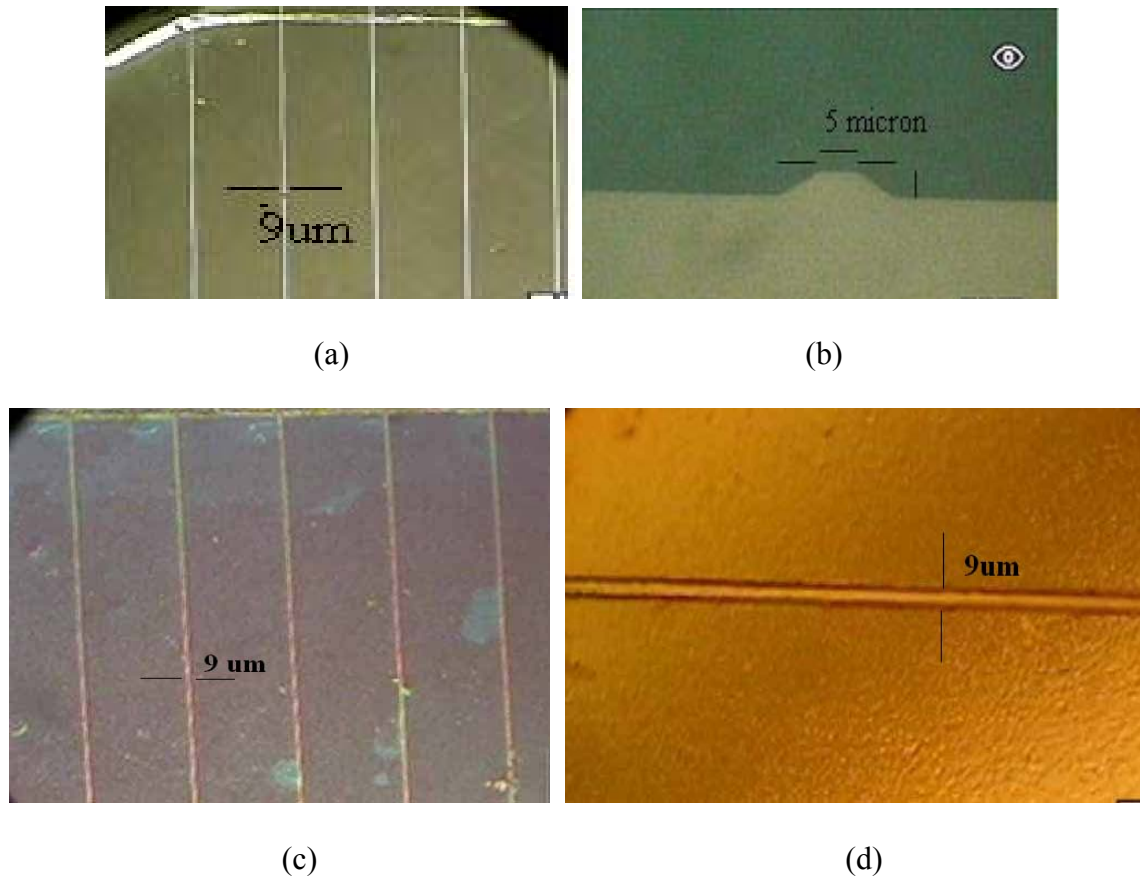


Fig 3.18 Nomarski image of top view of the EEL sample after photolithography (a); image of cross-section of the ridge after wet etching (b); image of top view of sample after depositing CaF₂ and Pb consecutively (c); image of top view after depositing gold on entire sample.

After the deposition of gold, BaF₂ substrate was thinned down from the back, to 200 μm from the original thickness of 500 μm for the ease of cleaving. The entire sample was mounted upside down on a thinning holder and wafer thinning was carried out.

Problem faced and probable solution

After the fabrication of the device the next step was cleaving the assembly along $\{111\}$ to form facet and or cavity. But during cleaving we encountered problem. It was found that the epitaxial layer was not cleaving along with the growth substrate BaF_2 . At a first glance we assumed that this happened because of thick gold on top of the sample. Therefore, to clarify this point we fabricated another piece of the same sample; omitted the gold deposition step and performed cleaving again. The results obtained from both the cases are shown in Figure 3.19.

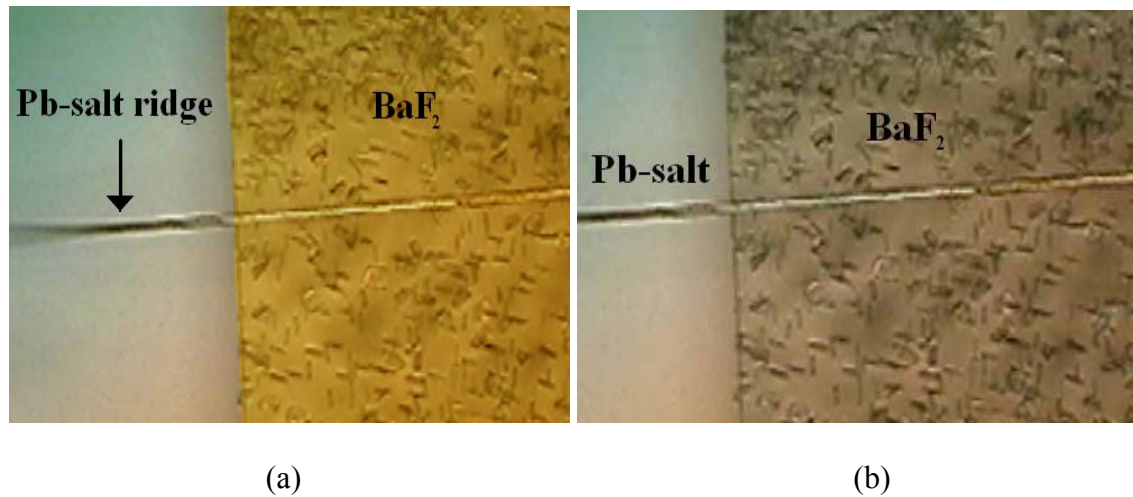


Fig 3.19 Nomarski image of top view of the gold deposited EEL sample after cleaving (a); image of top view of same sample without gold after cleaving (b).

From the above figure it can be seen that with or without gold, in both the cases Pb-salt layer did not cleave along with BaF_2 . In both the cases the Pb-salt ridge was hanging from the edge of cleaved BaF_2 . Hence it was confirmed that thick metal did not play any role in this uneven cleaving.

To solve the problem of this uneven cleaving a new structure was designed where the Pb-salt epitaxial layer was sandwiched between the BaF₂ growth substrate and another layer of BaF₂ deposited on top of epitaxial layer. The schematic of this new ridge structure is shown in Figure 3.20.

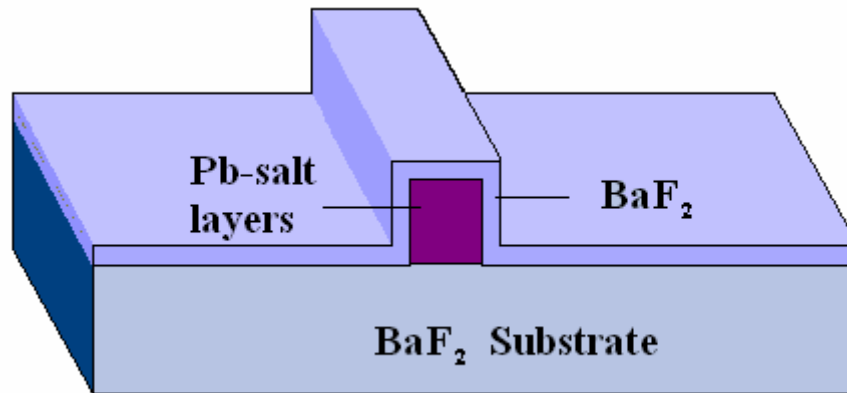
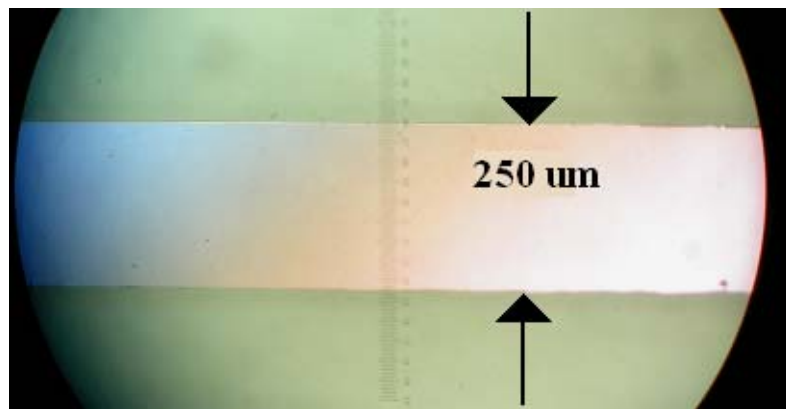


Fig 3.20 Schematic of modified device structure for optically pumped EEL on BaF₂

With this structure it was expected that the cleaving force would be transferred to the epitaxial layer both from top and bottom by the top and bottom BaF₂ and preferentially that would cleave the Pb-salt epitaxy. To verify this fact we grew a 500 nm thick layer of BaF₂ on top of the same sample whose layer structure was shown in Figure 3.17. After deposition of BaF₂, the substrate was thinned from the back as before, for the ease of cleaving. After thinning the sample was cleaved along [111] direction. The Nomarski image of the cleaved sample is shown in Figure 3.21.



(a)



(b)

Fig 3.21 Nomarski image of top view of the modified EEL sample after cleaving (a); image of cross-section of the sample after cleaving (b).

From these images it is quite clear that for the second structure, Pb-salt epilayer cleaved with the growth substrate. No hanging Pb-salt layer was found after cleaving for the second structure, as it was the case in the first structure. Therefore, we did proceed to measurement with this sample.

Characterization of the EEL structure

The sample was optically pumped normal to the substrate, by a 1.064 μm Nd:YAG laser ($\tau_{\text{pulse}} \approx 23\text{ns}$, 10Hz) with a rectangular spot having a dimension of 100X600 μm^2 . The emission from the side i.e from one facet of the sample was measured with an IFS 66/S spectrometer in step-scan mode using an InSb detector, in 1 cm^{-1} resolution of spectrometer. Peak pumping intensity was 5 kW/cm^2 . Figure 3.22 shows the room temperature emission spectrum measured from one facet of this edge emitting structure.

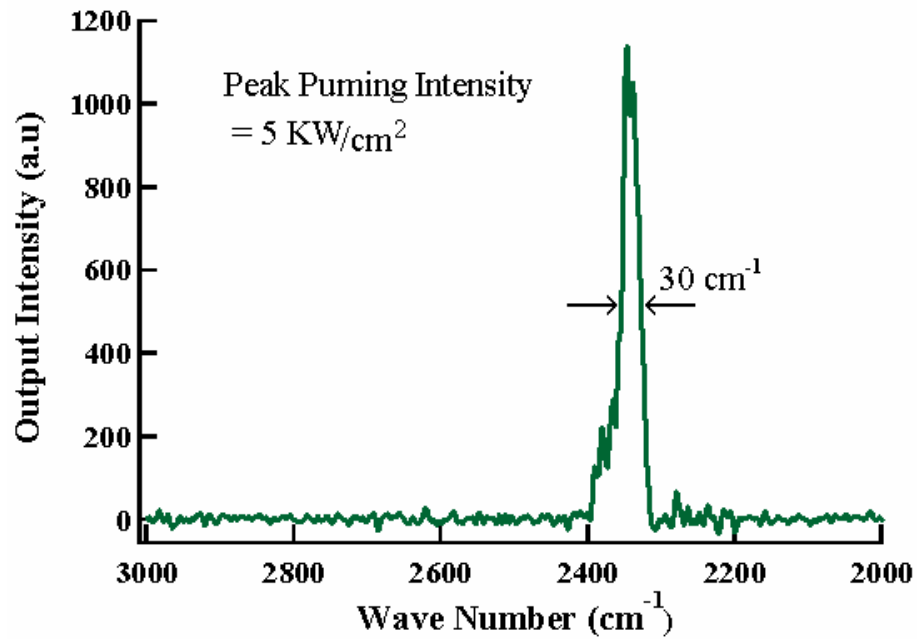


Fig 3.22 Side emission spectrum at room temperature from optically pumped Edge Emitting structure on [110] oriented BaF₂ substrate

As can be seen in the figure, strong emission was observed at room temperature, but the line-width of side emission was 30 cm^{-1} , which is much wider than cavity modes. Therefore, the results do not indicate lasing.

Discussion on the result

The reason for not getting cavity modes or lasing from this structure became clear afterwards from the SEM image of the cross-sections. Although with the 2nd structure the epilayer did cleave or in other words we should say broke along with the BaF₂ but it did not cleave along its natural cleaving direction, which is actually [100] for Pb-salt based crystals. And therefore, it was not actually cleaved but broken perpendicular to the [111] direction. Hence, the absence of a proper Fabry-Perot cavity that is needed to achieve stimulated emission prevented lasing. Figure 3.23 shows an SEM cross section of the modified EEL sample obtained after cleaving.

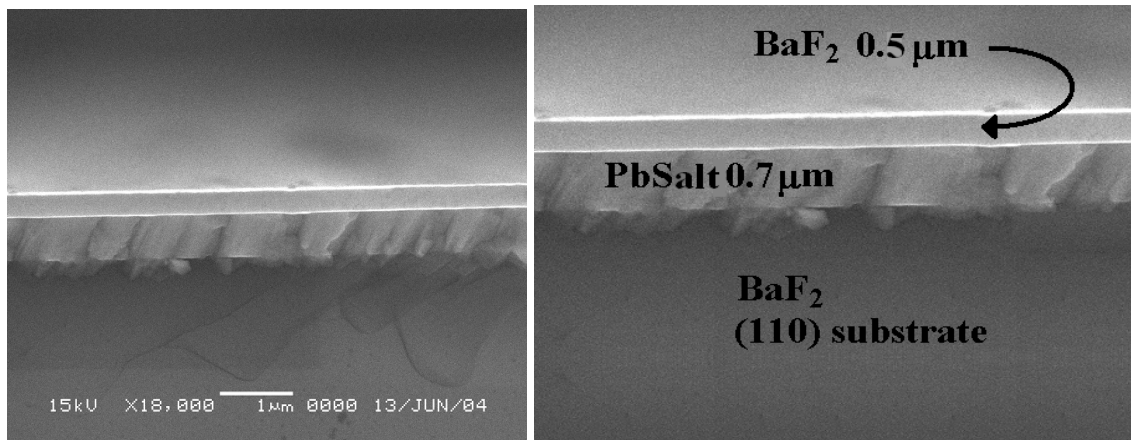


Fig 3.23 SEM image of cleaved cross-section of the modified EEL sample, lower magnification (a) higher magnification (b)

From the above figure, it can be seen that both BaF₂ substrate and top BaF₂ layer were nicely cleaved along [111] orientation. But the Pb-salt layer did not cleave, rather broke.

3.3.2 Plasma etching for edge-emitting laser

To obtain Fabry-Perot cavity for edge emitting structure, grown on {110} BaF₂, plasma etching of PbSe and PbSrSe based material was investigated in collaboration with a commercial vendor. My role in this collaboration was researching on the process; proposing the recipe; also helping to optimize the recipe and producing feedback. Plasma etching of IV-VI material has been reported, to obtain mirror facet for the EEL, by Zogg. et. al.²⁰. Also, dry etching of PbTe/ Pb_{1-x}Eu_xTe to form nano-structures using CH₄/H₂ plasma was reported by Schwarzl et. al.²¹. In particular, CH₄/H₂ dry etching has been proven to cause the least damage to III-V semiconductors as well²². Therefore, for our PbSe/PbSrSe material system we decided to start with CH₄/H₂ chemistry. As it was discussed in Chapter 2, for the first dry etch sample, plated Au was used as mask and that did not work because of huge roughness of plated gold, thus during second trial, a photoresist mask, which is quite smooth, was used. For etching trials, PbSe/PbSrSe multiple quantum well (MQW) layers grown on BaF₂ substrate was used. The etching recipe that was used for this etching is shown in Table 3.2.

Table 3.2 Etching recipe of PbSe/PbSrSe material system with CH₄/H₂ chemistry

CH ₄	H ₂	Ar	ICP	RIE	Pressure	Rate
8 sccm	32 sccm	10 sccm	300 W	65 W	20 mtorr	45 nm/min
4 sccm	32 sccm	10 sccm	0 W	170 W	20 mtorr	45 nm/min

The SEM cross sectional image of the etched facet of the sample is shown in Figure 3.24.

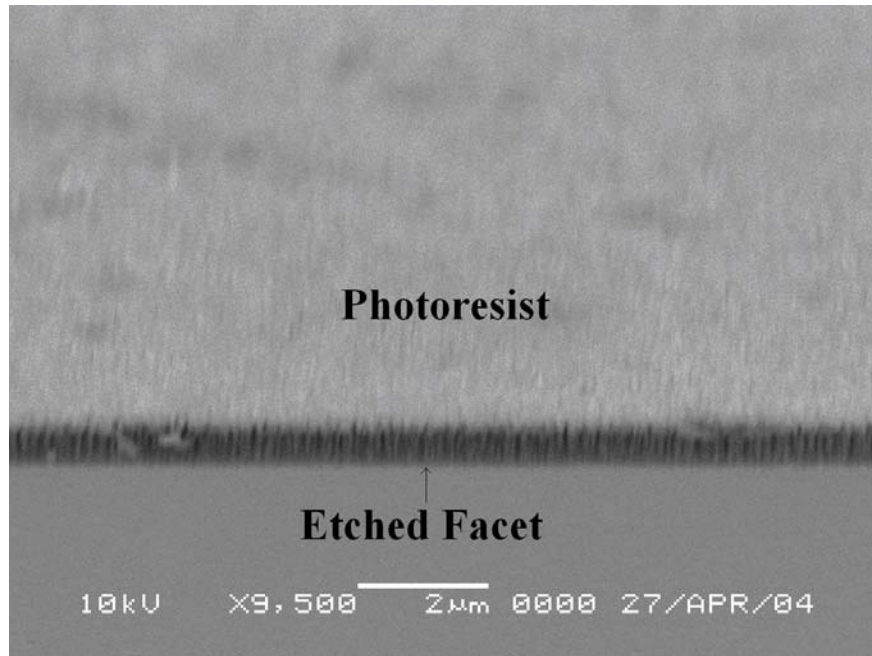


Fig 3.24 SEM image of dry-etched cross-section of PbSe/PbSrSe sample, with photoresist mask

From the above figure, it can be seen that during etching the etchant gases did react with photoresist making it rough. And hence, the etched facet also became rough. The photoresist that we used for this purpose was AZ-4110, which did not work with CH_4/H_2 chemistry. The reason for using AZ-4110 was easy processing and easy post-etching removal process. The best option with photoresist would be use of SU-8. But once this photoresist gets cured, the only way to remove it is through plasma-ashing and this was not possible in our laboratory facility.

To interpret the tolerance limit of facet roughness, a calculation was carried out to analyze reflectivity of the facet as a function of facet roughness based on the formula provided by Stocker et. al.²³.

The normalized power reflectivity[R(d)], i.e the ratio of the actual power reflectivity (R) to the power reflectivity for a perfectly smooth facet (R₀) is given by,

$$\frac{R(\Delta d)}{R_0} = e^{-16\pi^2(n\Delta d / \lambda_0)^2}$$

For an uncoated surface, $R_0 = (n_1 - n_2)^2 / (n_1 + n_2)^2$. For our case, by putting $\lambda_0 = 4 \mu\text{m}$ and $n=4.8$, the plot of normalized power reflectivity[R(d)] and surface roughness 'd' can be obtained as shown below,

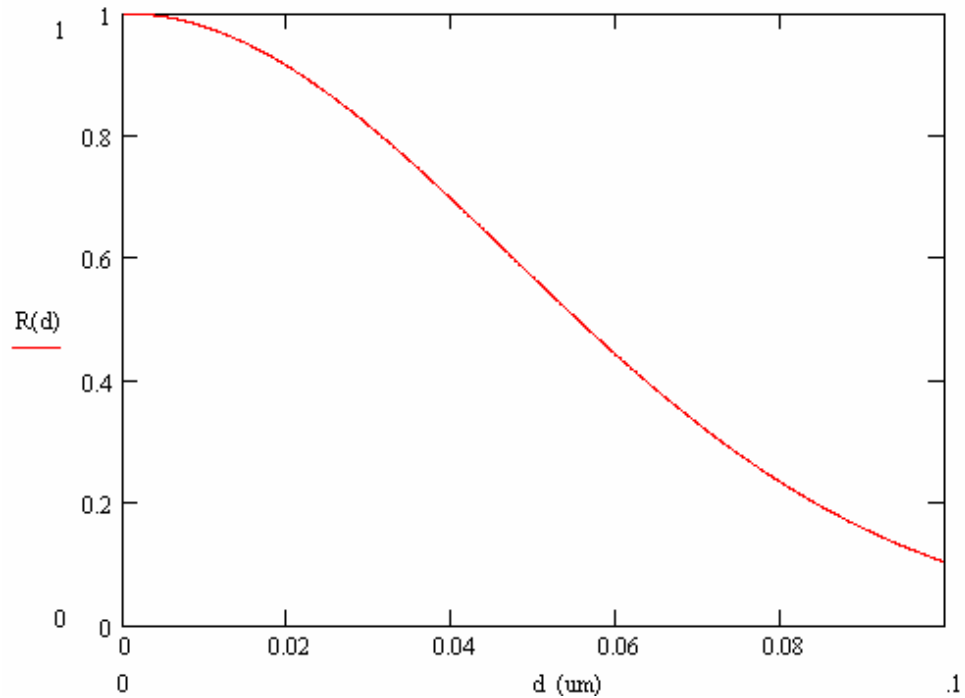


Fig 3.25 Plot of normalized facet reflectivity R(d) vs facet roughness d (μm)

Literature review revealed that with CH_4/H_2 chemistry, a 1 μm thick Si_3N_4 layer could be the better etch mask. Hence, we used sputter deposited Si_3N_4 as the mask material for our next trial. Although due to very slow deposition rate of Si_3N_4 in our sputtering chamber, we were able to deposit only 0.3 μm of this material. Figure 3.26 shows the etched cross-section of a 2 μm thick PbSe/PbSrSe layer obtained with Si_3N_4 masking.

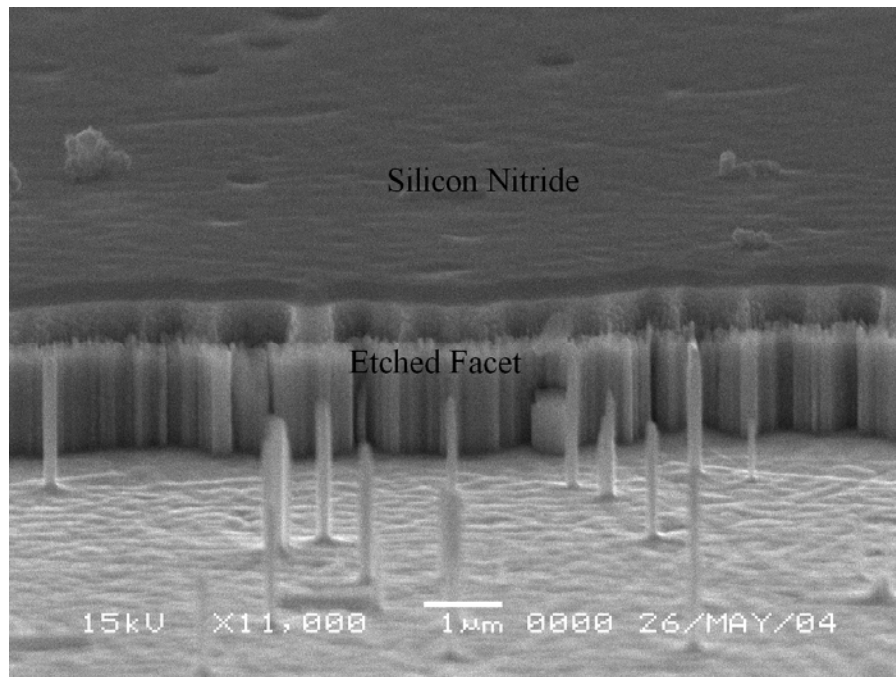


Fig 3.26 SEM image of dry-etched cross-section of PbSe/PbSrSe sample, with Si_3N_4 mask

From the above figure, it is clear that from many portion of Si_3N_4 layer the material was etched away during RIE of IV-VI material. In this particular case the reason of this etching was very low thickness of Si_3N_4 and above all poor quality of sputtered material. Although in the Si industry it is commonly known that sputtered Si_3N_4 always has a very

poor quality and for that reason the recommended deposition technique for this material is CVD, but since in our lab we did not have the facility to do that, we had to go ahead with sputtering.

Later on, during collaboration with Penn State University some promising results were obtained from dry etching of PbSe/PbSrSe based material using Ni mask. However, a plasma etcher was not available at OU and hence, the whole process of dry etching by collaborating with others at a remote facility was found to be very slow. Therefore, further research was carried out on the development of a substrate removal method that would preferentially cleave Pb-salt material along [100] direction, for the growth on [110] oriented BaF₂ substrate.

3.3.3 Gold-Indium bonded laser on GaAs wafer

It was already discussed in the previous sections that parallel natural cleavage {100} planes also exist for [110] orientated structure that allows the formation of Fabry-Perot cavity. However, from the results shown in section 3.3.1, it is clear that the challenge still remained in fabricating the cleaved cavity laser structure on BaF₂ substrate because of the dissimilar natural cleavage plane of epilayers and the BaF₂ substrate that has natural {111} cleavage plane. Therefore, development of a proper cleaving method by removing the growth substrate became necessary. Epitaxial transfer and the removal of growth substrate have already been developed for various material systems^{24 25 26}.

In this section a novel mounting technique will be described with epitaxial transfer of Pb-salt based structure onto {100} GaAs wafers for the fabrication of cleaved cavity Pb-salt laser grown on BaF₂²⁷. In this process the cleavage plane of PbSe in the [100] direction is aligned with GaAs (110) plane. The advantage of this approach is that BaF₂ growth substrate is commercially available and has high material quality. The use of BaF₂ as a growth substrate is much more cost effective and economic than the use of expensive PbSe substrates. Again, the GaAs carrier wafers not only facilitate the cleaving technique but also have the advantage of better thermal conductivity (46 Wm⁻¹.K⁻¹@298 K) over BaF₂ substrates (11.7Wm⁻¹.K⁻¹@286K). Conductive GaAs wafers also overcome the disadvantages of insulating BaF₂ substrate and ease the fabrication of electrically injected devices.

Experimental Procedure of transferring Pb-salt epitaxy on {100} GaAs

PbSe/ PbSrSe epitaxial layers were grown on [110] orientated BaF₂ substrates by molecular beam epitaxy (MBE) as described in reference-18. Epilayers were bonded to the GaAs wafers by using a metallic Gold-Indium alloy having very high melting temperature²⁸. The GaAs wafers used for these experiments were n-type doped. Figure 3.27 shows a schematic of bonded epilayer on GaAs, before and after growth substrate removal. A layer of Ni/Au/Ge alloy on both sides of GaAs wafers serves the purpose of ohmic contact on GaAs.

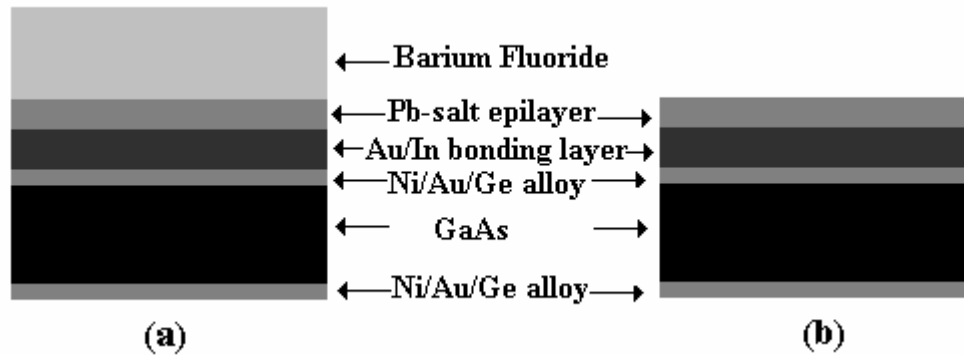


Fig 3.27 Schematic of bonded cross-section before BaF₂ substrate removal. Pb-salt sample mounted episode down on GaAs (a); The sample after BaF₂ substrate removal (b).

Prior to bonding, the GaAs wafers (2"dia) were mechanically thinned to almost 170 μm from an initial thickness of 600 μm for the ease of cleaving. Then they were polished using a sodium hypochlorite based commercially available polishing fluid, Chemlox. After polishing, the wafers were subject to ultrasonic cleaning in acetone, propanol and methanol bath respectively, followed by an oxide removal technique in which the wafers were boiled in 1:1 HCL, De-Ionized water solution until they become hygroscopic.

Figure 3.28 shows the SEM image of the polished GaAs wafer, revealing the surface morphology.

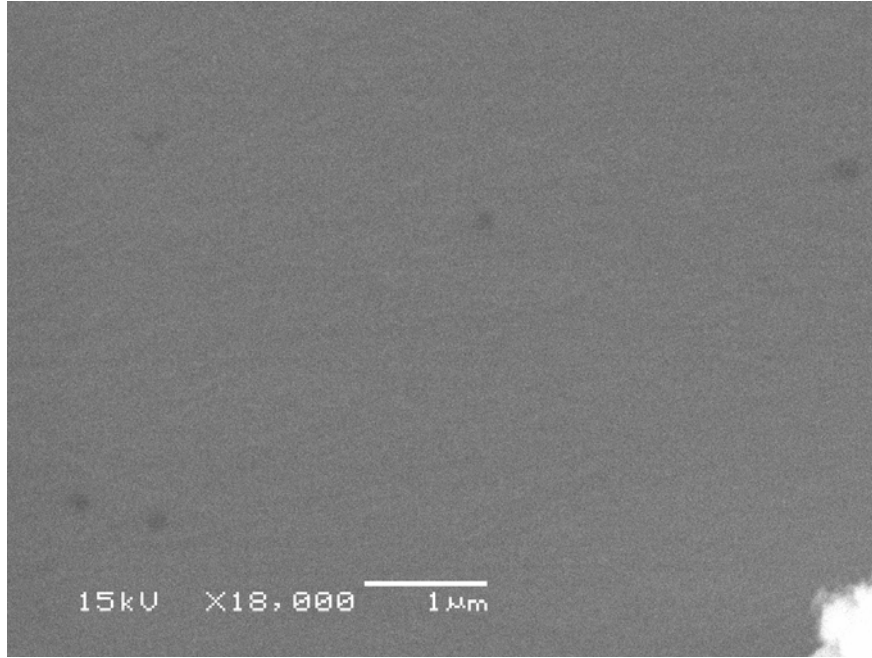


Fig 3.28 SEM image of polished GaAs wafer, revealing surface morphology

After polishing and cleaning, these wafers were sputter-coated with a thin layer of Au ($\sim 0.5\mu\text{m}$) and cleaved into pieces to get two mutually perpendicular $\{110\}$ planes. At this stage I would like to mention, to verify the bond ability and cleaving of Pb-salt epitaxy on GaAs, initially no Ni/Au/Ge alloy was deposited. But during the fabrication of diode laser, we did deposit this layer for ohmic contact formation.

To cleave the Pb-salt layers in the $[100]$ direction after bonding it onto GaAs, a proper alignment of epilayers along with growth substrate on the GaAs wafer was needed before bonding.

For this purpose at first the Pb-salt sample together with BaF₂ substrate was cleaved twice to obtain two cleaved {111} planes as shown in Figure 3.29 (a) and (b).

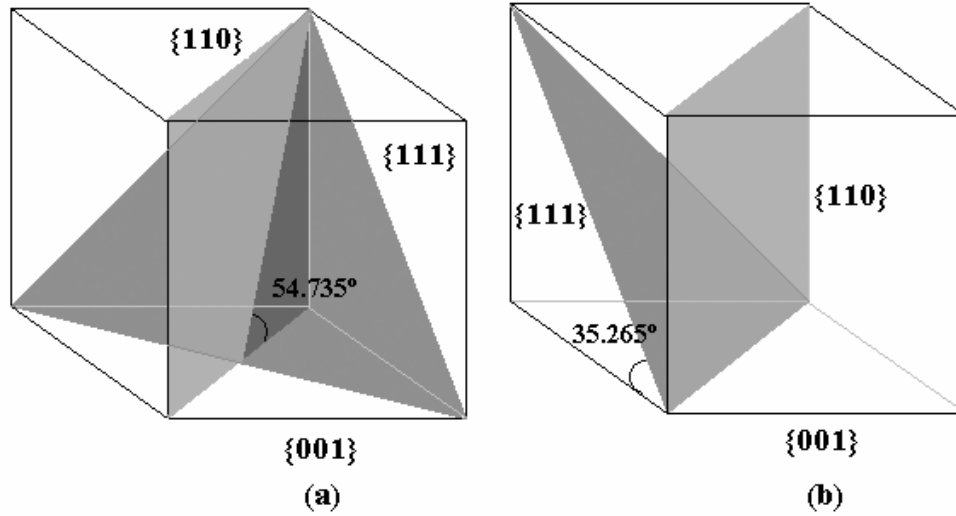


Fig 3.29 Schematic of crystal orientation, showing the intersection of {111} and {110} for different orientation of {111} plane and the correspondingly angle between {111} and {100}.

As it can be seen that the angle between {111} and {100} is 54.735° and 35.265° respectively, depending upon two different orientations of the {111} planes. When two {111} planes intersect the {110} plane and meet at an acute angle, the angle between {111} and {100} is 54.735° and when they are obtuse this angle becomes 35.265°. Now for this bonding process we made a sample holder made of brass with a mark of 54.735° angle on it. Now after cleaving the Pb-salt sample twice along with BaF₂, they were mounted on this holder in a specific manner depending on the angle between two {111} planes. This mounting is shown in Figure 3.30.

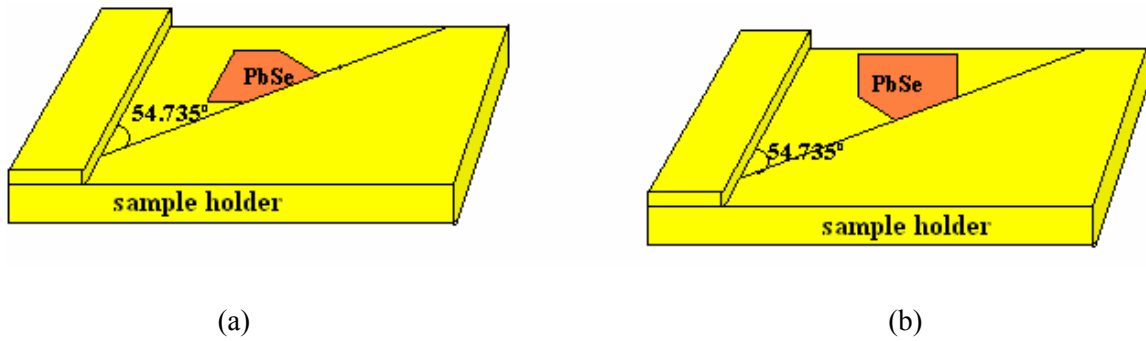


Fig 3.30 Schematic of the mounting of Pb-salt sample on holder. Aligned Pb-salt sample when the angle between two $\{111\}$ plane is acute (a); Aligned Pb-salt sample when the angle between two $\{111\}$ plane is obtuse (b).

Before mounting the Pb-salt sample on the holder, about $1.5\ \mu\text{m}$ of Au was electroplated on the Pb-salt sample. This gold layer also forms an ohmic contact to the Pb-salt layer. After the mounting of Pb-salt sample, about $5\ \mu\text{m}$ of 'In' was electroplated on the Au-sputtered GaAs wafers. Pb-salt epilayer and GaAs wafer were then aligned quickly under an optical microscope and bonded together by the 'In' layer. Figure 3.31 shows the alignment with GaAs and the direction of cleaving.

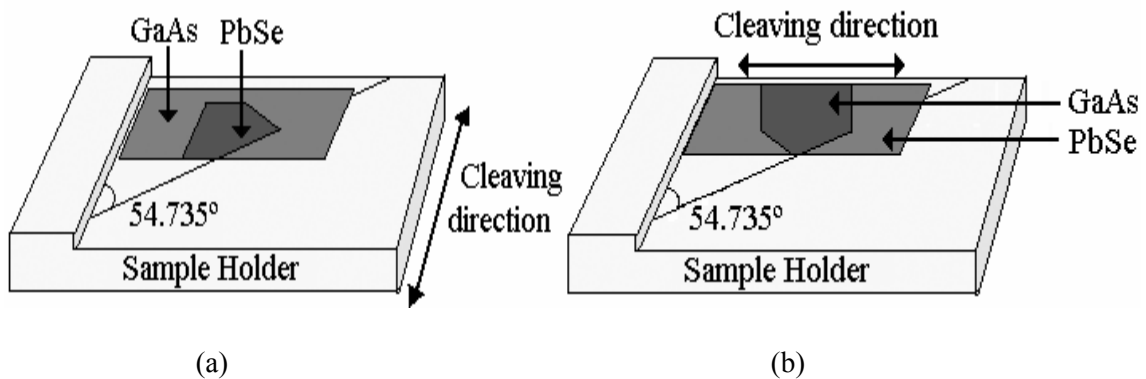
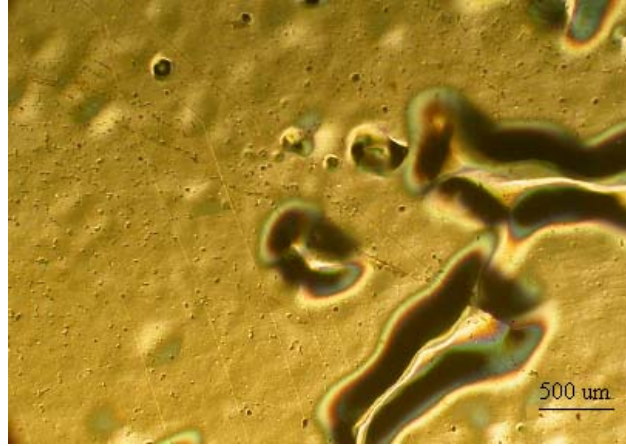


Fig 3.31 Schematic of aligned GaAs and Pb-salt before bonding, when the angle between two $\{111\}$ plane is acute (a); when the angle is obtuse (b). Arrows show the direction of cleaving after growth substrate removal.

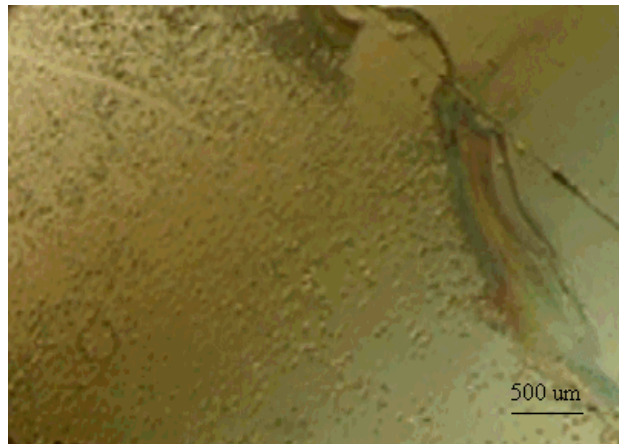
The alignment process that is shown in the above two figures allows natural cleavage of {100} planes of [110] orientated Pb-salt epilayer together with {110} planes of GaAs after the BaF₂ substrate is removed. After securing the In plated GaAs wafer on mounted Pb-salt sample on the brass holder, they were transferred in a small furnace and heated under vacuum ($\sim 10^{-3}$ torr). To ensure good adhesion between the samples they were kept under a static pressure by providing a weight of 500 gm/cm² by mechanical means. The furnace temperature was raised to 290°C in 1 hour and stayed at that temperature for 8 hour. Then the furnace was turned off and allowed to cool at room temperature. Experimentally it was found that when the furnace temperature is 290°C the temperature at the interface of Pb-salt and GaAs is around 200 °C. However, this temperature gradient may change with the change in metallic weights that are usually kept on top of this assembly, during bonding, to apply pressure on the sample. Also during initial experiments the bonding time was 8 hours, but later on it was reduced to 2 hr and same bonding strength was obtained. Upon completion of the bonding process, the sample was taken out from the chamber and mounted on another holder keeping GaAs side down and the BaF₂ side up and then BaF₂ substrate was thinned down to ~ 10 -20 μ m by a chemo-mechanical thinning process. The remaining BaF₂ was removed by immersing the sample in DI water to avoid any mechanical damage to Pb-salt epi-layer. Now, during this removal process several obstacles were encountered. The etch rate of BaF₂ substrate in DI water at room temperature was found quite low. After thinning, the removal of substrate in this method used to take 2-3 days. Therefore, substrate removal became quite a time consuming process. To make this process faster, substrate removal had been tested with i) DI water at 50 °C; and ii) a mixture of DI water with 30% filtered city water.

Time of etching came down to several hours with these processes, but in both cases the surface of the epitaxial layer got damaged and contaminated.

Figure 3.32 (a) shows the Nomarski image of the surface of epitaxial layer, after removing BaF_2 substrate by above mentioned processes.



(a)



(b)

Fig 3.32 Nomarski image of the surface of epitaxial layer, after BaF_2 removal by DI water at 50 °C (a); image of the surface of epitaxial layer, after BaF_2 removal by a mixture of DI water and 30% filtered city water (acidic in nature) (b).

During removal of BaF₂ in hot DI water, air bubbles created in water with enormous vapor pressure inside, and made the interface of epitaxial layer bonded on GaAs very weak and enhanced water getting under the epitaxial layer, which made the epitaxial layer rough. In the second method even after using filtered water, lot of precipitation from water was found on the surface thus by making it severely contaminated. To solve these problems but at the same time to increase the etch rate of BaF₂, a method was developed in which the BaF₂ substrate was etched in DI water at room temperature while stirring the water by a magnetic stirrer at a speed of 100 rpm. And the sample was hanged inside the solution with the BaF₂ side parallel to the direction of rotation of water. In this method the speed of rotation is a critical parameter. Very high speed creates vortex inside water and makes the bonded interface weak with time. Also very slow speed makes the etch rate slow. By this process, even 50 μm thick substrate was removed completely in 3 hrs. A point to be noted here is that the water was changed at an interval of 1 hr to prevent ionic saturation and enhance dissolution of more BaF₂ in the de-ionized water. By using this method a mirror like surface of epitaxial layer was obtained. The SEM surface morphology of the epilayer after growth substrate removal is shown in Figure 3.33. To check any degradation of material properties during this process photoluminescence measurement was carried out before and after bonding of the epitaxial layer. No change in PL intensity from the epitaxial layer was observed after bonding, however a peak shift of 38 cm^{-1} was observed from a single layer PbSe sample after bonding and substrate removal. Figure 3.34 shows the PL spectrum of that sample before and after bonding. The pumping power and position of the sample was same for both the measurements.

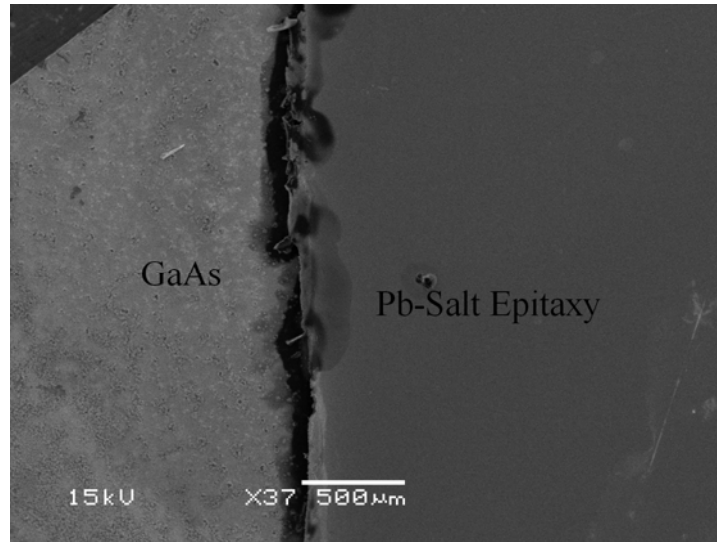


Fig 3.33 SEM top view of the bonded epilayer on GaAs, after BaF₂ substrate removal

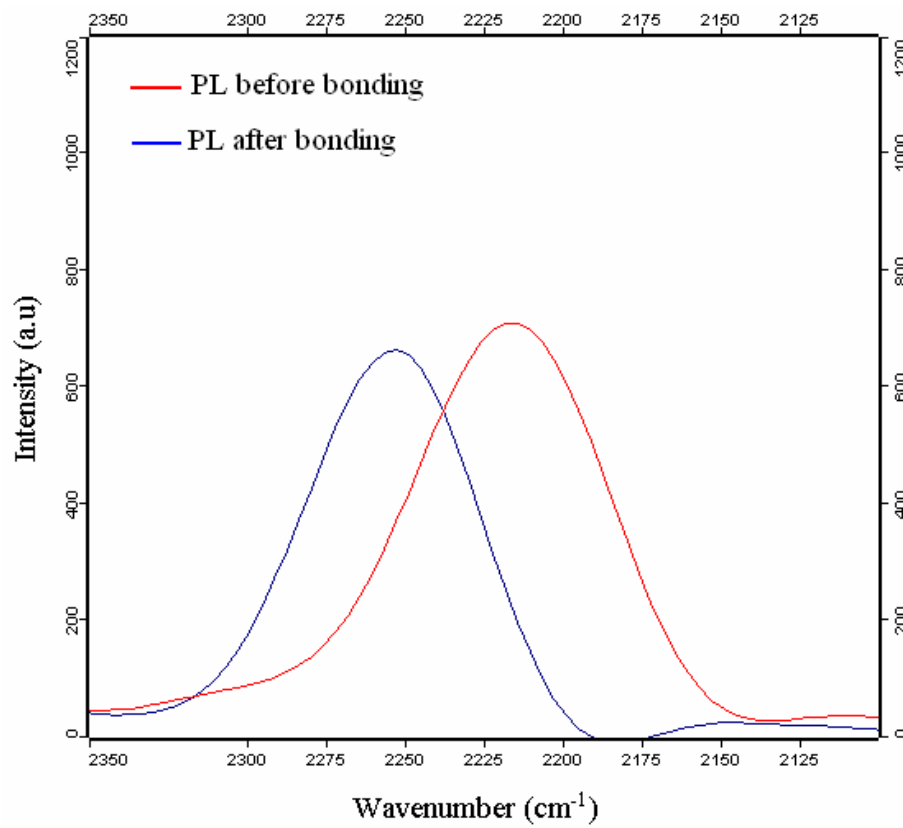
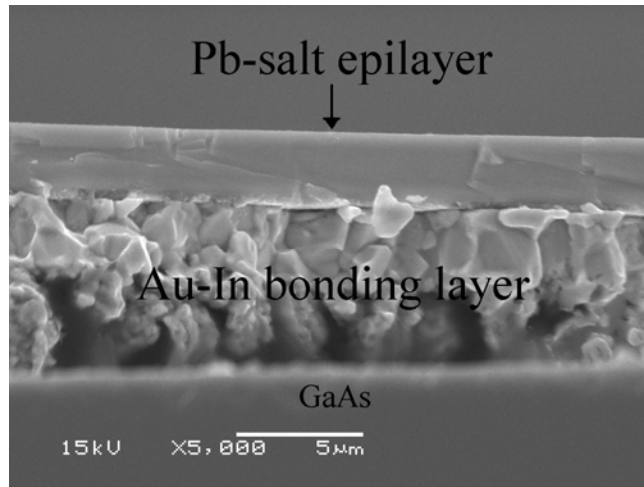
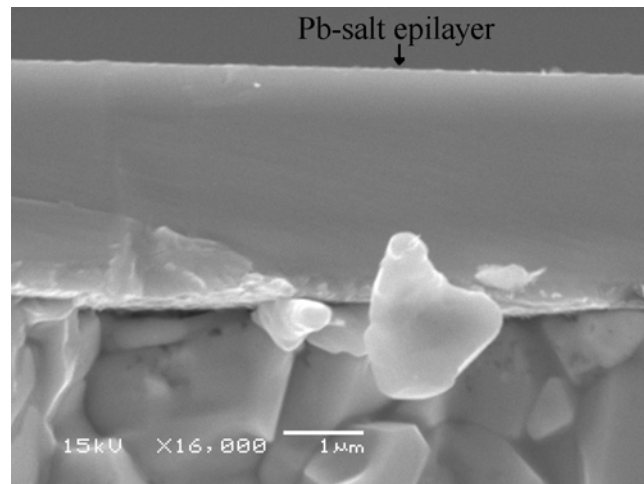


Fig 3.34 PL spectrum from a single layer PbSe sample before and after bonding on GaAs and after BaF₂ substrate removal. The intensity showed here is 10% of actual intensity.

After the removal of BaF₂ substrate the GaAs host substrates were cleaved in the directions as shown in Figure 3.31, this preferably cleaves Pb-salt material in the [100] direction. The quality of the cleaved plane and bonded cross-section was observed by scanning electron microscope (SEM); also the bonded cross-section was examined using energy dispersive x-ray spectroscopy (EDX).



(a)



(b)

Fig 3.35 Scanning electron micrograph of bonded cross section (a); {100} cleaved facet of Pb-salt epilayer after bonding and BaF₂ substrate removal (b).

Cleaved facet of a PbSe epilayer bonded onto GaAs is shown in Figure 3.35. The figure shows a smooth {100} cleaved facet, and that proves the validity of the mounting technique. The bonding medium showed a total thickness of 6.95 μm , which is consistent with the total thickness of the deposited metal before alloy formation. Proper bonding of the epilayer on GaAs happened because of the formation of high melting temperature Au-In alloy (AuIn₂-540.7°C, AuIn-509.6°C) in the bonding medium.

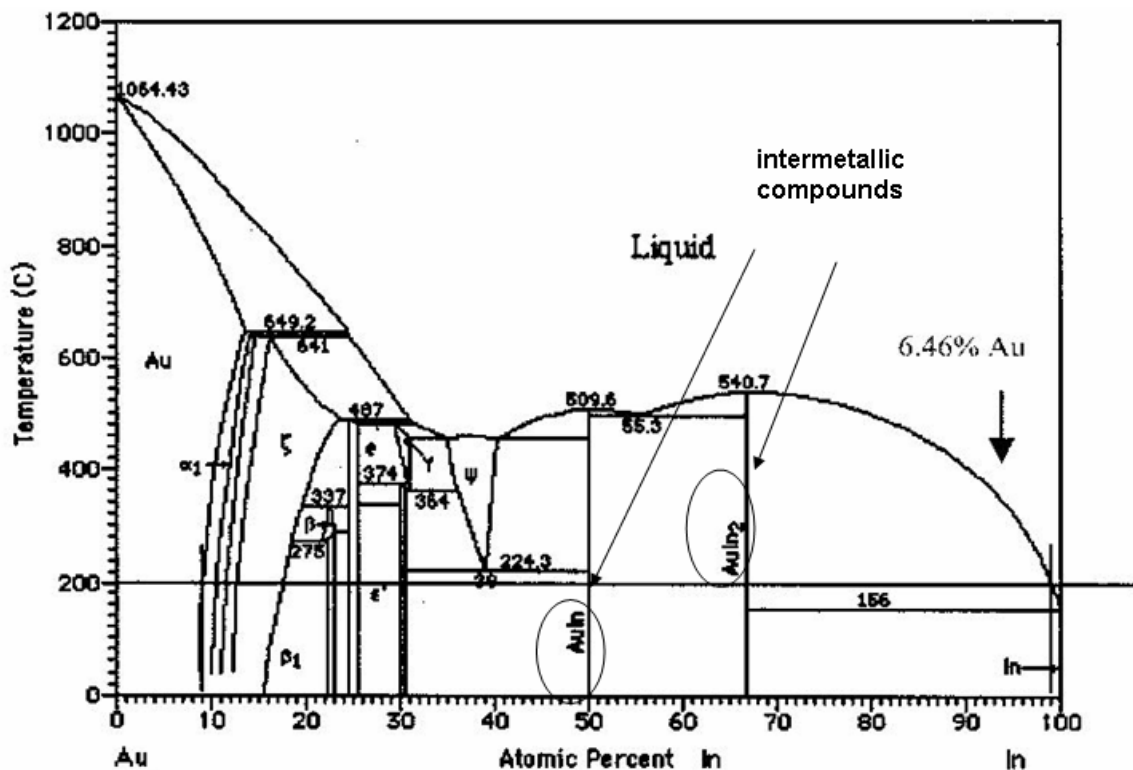


Fig 3.36 Au-In Phase Diagram²⁶

Figure 3.36 shows the Au-In phase diagram. AuIn₂, AuIn alloys basically form through a solid-liquid diffusion process. When 'In' melts AuIn₂ is formed at the interface of solid Au and liquid 'In', as the temperature rises above the melting point of 'In', the mixture dissolves Au to form more AuIn₂. This solid-liquid diffusion continues until the mixture

solidifies. With higher concentration of gold, AuIn_2 eventually results in AuIn , Au_3In (γ) and Au_4In (β) phases^{26, 29}.

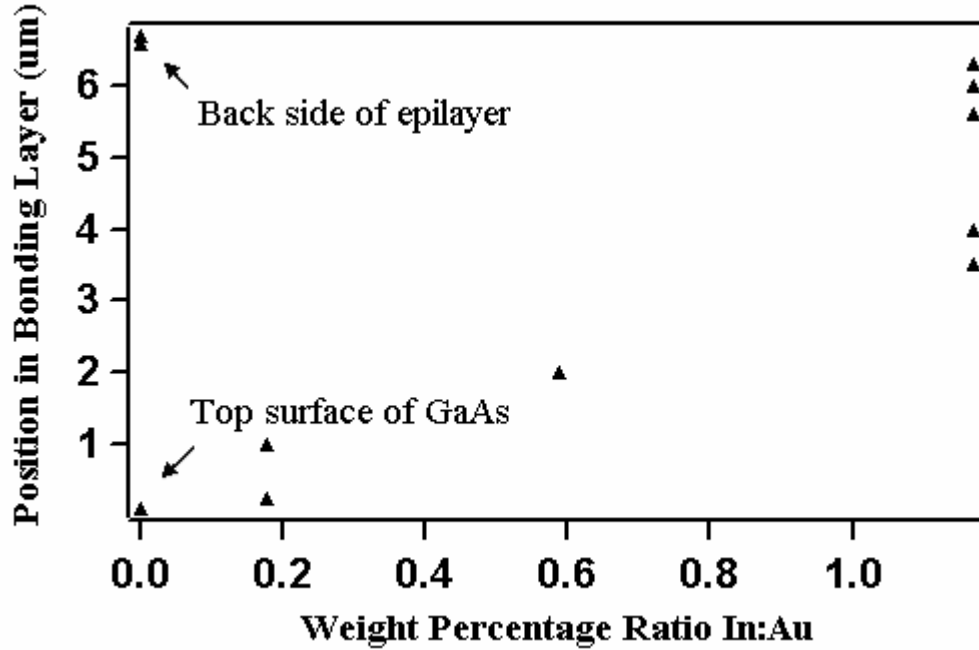


Fig 3.37 Weight percentage ratio of In-Au along bonding cross-section starting from the top surface of GaAs wafer to the backside of epilayer.

Figure 3.37 shows the line-scan measurement results of EDX measurement. The ratio of In and Au weight percentages throughout the bonding layer extending from GaAs side to the PbSe side was calculated and shown in the figure. Several sets of data were obtained by measuring along different trace lines across the bonding layer during EDX measurement. Measurement along different traces gave almost similar results. From the EDX data we observed that the bonding layer has three distinct regions: almost $0.4\mu\text{m}$ thick region with much higher wt% of Au and much less wt% of In on the GaAs side; then approximately $6.2\mu\text{m}$ thick region at the middle portion of the bonding medium

where the ratio of concentration of In and Au in wt% was variable and showed typical values like 1.17, 0.587, 0.176 indicating the formation AuIn_2 , AuIn , Au_4In phases in the alloy and at the end an almost $0.35\text{ }\mu\text{m}$ thick region with $\sim 90\text{ wt\%}$ Au and no In on the PbSe side. But it can be seen from Figure 3.37 that the dominant intermetallic compound in the alloy was AuIn_2 . These data were consistent with the desired result. Initially the thickness of Au and Indium were chosen to give $51.5\text{ wt\%}$ of Au and $48.5\text{ wt\%}$ of In. Although for the formation of AuIn_2 intermetallic compound the required composition was $46\text{ wt\%}$ of Au and $54\text{ wt\%}$ of In, but we preferred some extra gold so that even after alloy formation a layer of pure Au remain on the PbSe side and the Au-PbSe ohmic contact resistance remains unaffected. The formation of AuIn and Au_4In alloy (shown as β phase in the phase diagram) in the bonding medium suggests that Au and Indium atoms continue to interdiffuse by solid-state diffusion as the time passes at higher temperature and form a mixture of AuIn_2 , AuIn , Au_4In having much higher melting point. But among these three intermetallics the melting point of Au_4In is comparatively lower than other two. In this particular experiment, the formation of Au_4In was mainly because of prolonged bonding time. By reducing this time, one can eliminate the presence of this phase in the bonding intermetallics.

Fabrication of electrically pumped diode laser from bonded epitaxy on GaAs

For the fabrication of diode laser first of all we grew epitaxial layers forming active region, as well as electrical and optical confinement region on {110} BaF₂ substrate. The active region consisted of a 7 pair MQW with 20 nm PbSe well and 30 nm Pb_{0.97}Sr_{0.03}Se barrier. The n-cladding layer consisted of a 1.67 μ m layer of Bi₂Se₃ doped Pb_{0.96}Sr_{0.04}Se and p-cladding layer consisted of 1.3 μ m of Ag doped Pb_{0.96}Sr_{0.04}Se. Before fabricating laser structure the Pb-salt based epitaxy was transferred on GaAs wafer following the method discussed above. The surface morphology of the Pb-salt epitaxial layer after bonding it on GaAs is shown in the figure below.

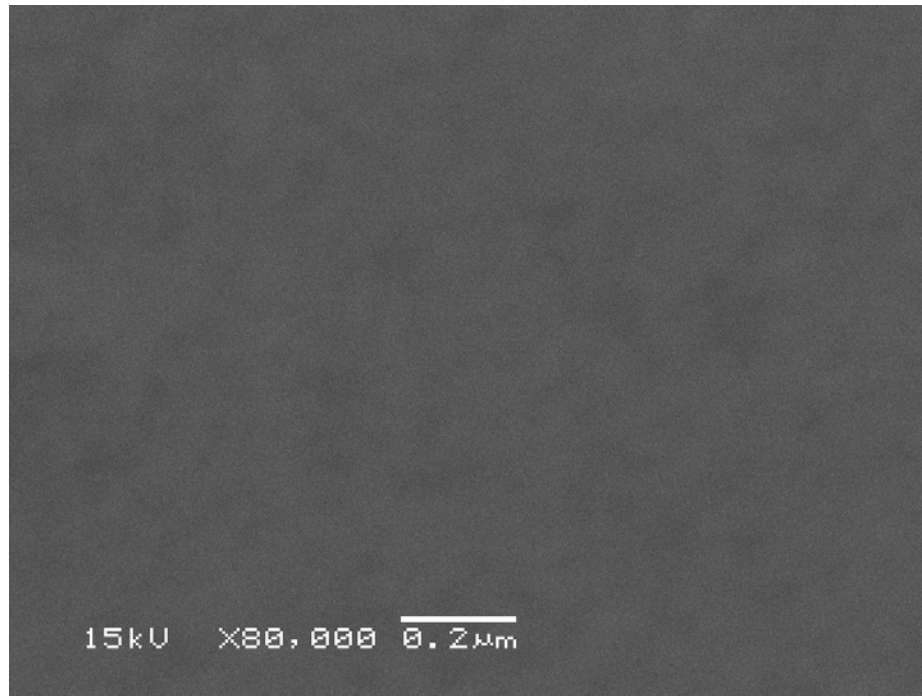


Fig 3.38 Scanning electron micrograph of the bonded epilayer on GaAs carrier wafer, after BaF₂ growth substrate removal

Stripe geometry was followed for the diode laser fabrication. After the BaF_2 substrate removal the Pb-salt epitaxy was cleaved along with GaAs to obtain the $\{100\}$ plane. 40 μm wide openings were then patterned perpendicular to this $\{100\}$ plane by photolithography. An SEM cross-section of the bonded epitaxy after cleaving is shown in Figure 3.39.

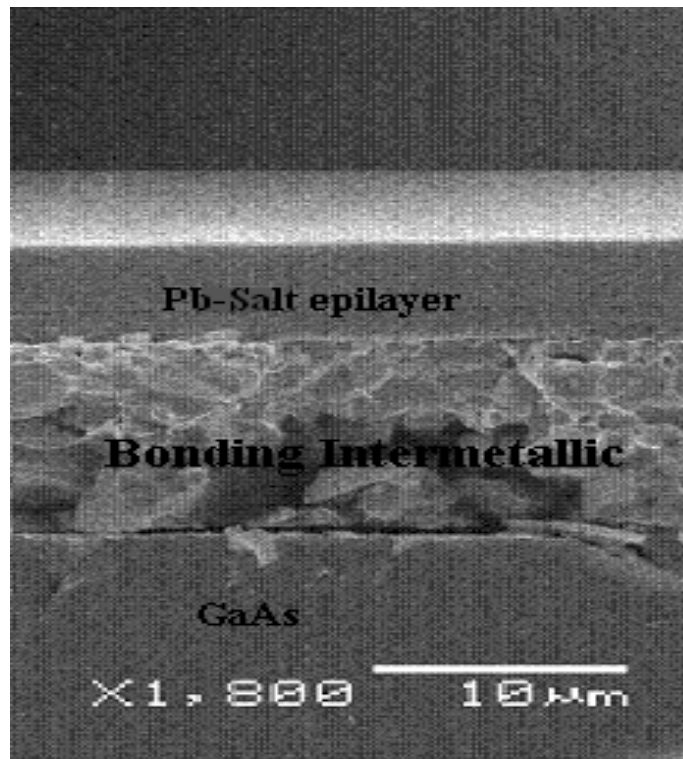
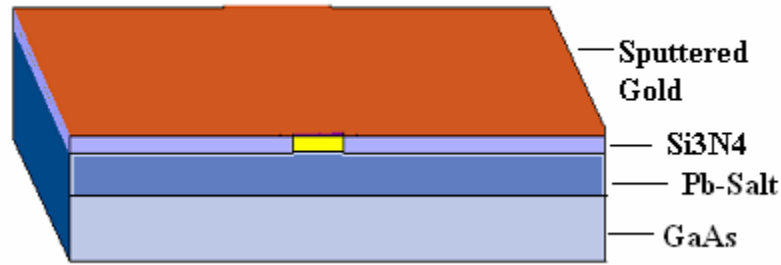
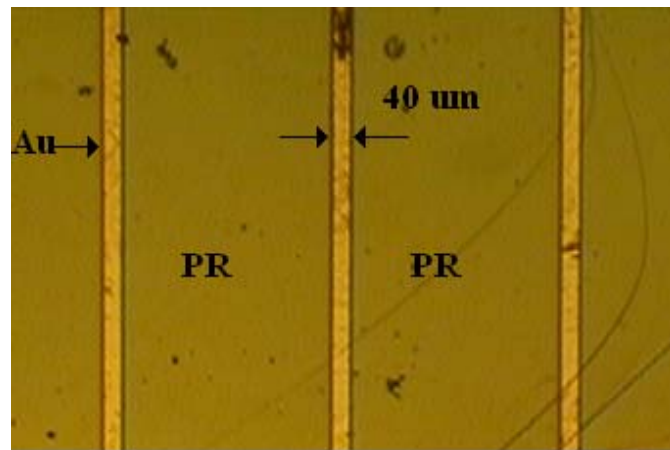


Fig 3.39 SEM cross-section of the bonded epilayer on GaAs carrier wafer, after BaF_2 growth substrate removal. $\{100\}$ plane of Pb-salt epitaxy is visible in the micrograph.

After forming 40 μm wide openings on the epilayer, 1 μm thick Au layer was electrodeposited on those openings for the formation of ohmic contacts to the Pb-salt layer. A schematic of this laser structure and Nomarski image of the plated gold stripes is shown in Figure 3.40 below.



(a)

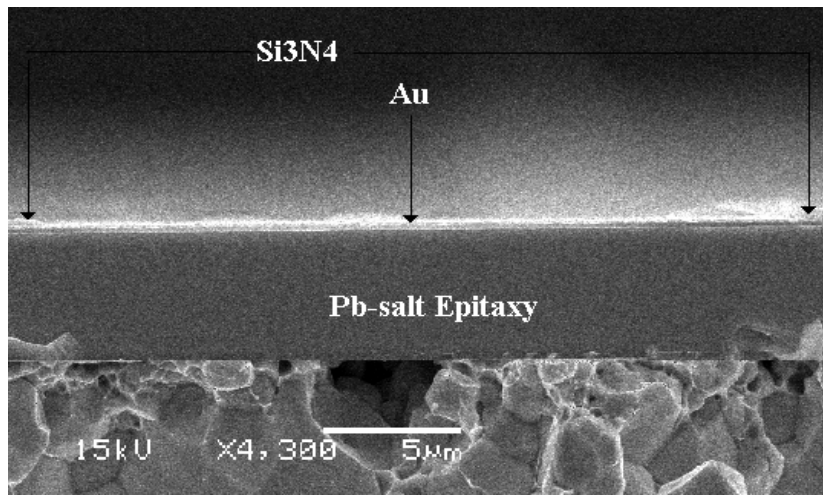


(b)

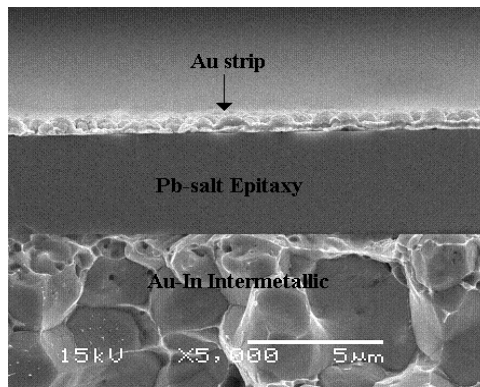
Fig 3.40 Schematic of the stripe geometry laser structure (a); Nomarski image of the plated gold stripes on Pb-salt epitaxial layer (b)

It can be seen in the schematic that Si₃N₄ had been used as the insulating layer for epitaxial down mounting on laser head and a thin layer of sputtered gold was used on top for better heat dissipation. After Au electrodeposition for stripe formation, photoresist

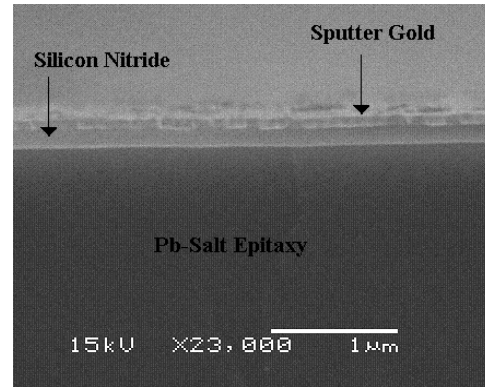
was removed and 0.2 μm thick silicon nitride was sputter deposited on the whole structure. Next Si_3N_4 was etched from the top of the gold stripe to open 25 μm window on gold stripes and hence exposing the stripes for electrical connection. Finally a 0.1 μm thin layer of Au was sputter coated to form the top Au layer. After this step, the structure was cleaved into 300-500 μm long rectangular pieces to form the cavity and individual diode lasers. The figure below shows the SEM cross section of a single diode laser.



(a)



(b)



(c)

Fig 3.41 SEM cross sections of a single diode laser (a); magnified image of gold stripe region (b); magnified image of sputter gold/ Si_3N_4 region (c).

Device Characterization

The first step in device characterization started with measuring the I-V characteristics of the fabricated edge emitting structure. The following figure shows the I-V curve of the above fabricated structure in forward bias.

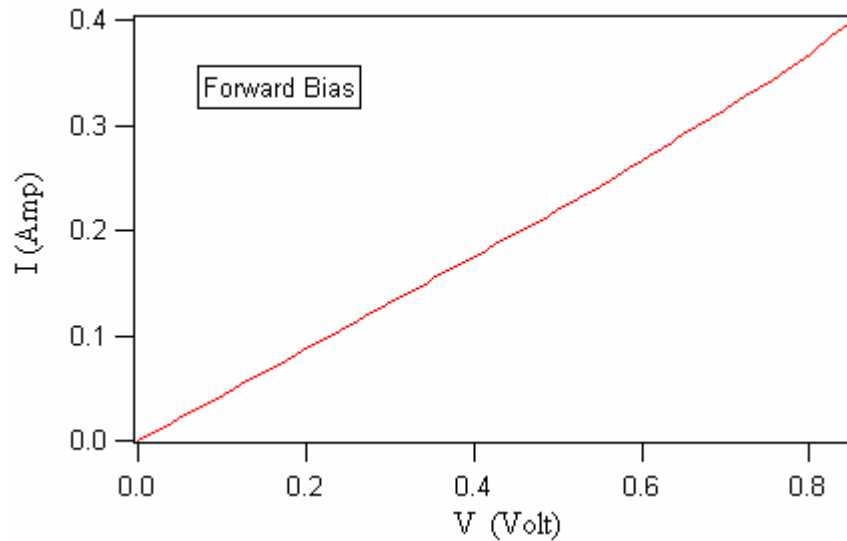


Fig 3.42 Current –Voltage relationship of the fabricated structure in forward bias

From the I-V characteristics the existence of a p-n junction was not observed in this sample. Therefore, lasing could not be expected from this structure.

The probable reason of not having a p-n junction in the sample could be longer post growth annealing which might cause dopant diffusion. During the growth of this sample, after the growth of n-PbSrSe layer it was annealed overnight at 250 °C, then again after growing p-PbSrSe layer and p+ PbSe cap layer, it was annealed overnight at 250 °C. Also, [110] orientation that provides dislocation gliding might lead to the gliding of dopants as well during the growth of epitaxial layer. To our knowledge, the diffusion properties of both p- and n-dopants in Pb-salt materials on BaF₂ substrates have not been

studied. Since, only three p-n junction laser structures on $\{110\}$ BaF₂ have been fabricated till now, more experiments are needed for conclusive suggestion.

In sum, a novel fabrication method has been developed for electrically pumped Pb-salt laser grown on $[110]$ oriented BaF₂ substrates. Once the junction characteristics are improved, electrically pumped Mid-IR lasers would be anticipated.

3.4 References

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Chapter 4

[110] Oriented Edge Emitting Laser on PbSnSe Substrate

4.1 Surface Preparation of {110} PbSnSe Wafers for MBE Growth

MBE epitaxial growth of Pb-salt based material on [110] oriented PbSnSe substrates has not been performed before the research work of this dissertation. Therefore, there was a need to develop a polishing recipe for such substrates. PbSnSe substrate polishing also consisted of both mechanical and chemical cycles. Chemical cycle comprised of two parts with two different sets of chemical solution, SOLN1 and SOLN2. First solution had much higher etch-rate than the second. This was to get rid of the higher order surface non uniformities at a much faster rate, with the first solution. The chemicals chosen for the chemical cycles were kept same as the one used for [100] oriented Pb-salt wafers, but the composition was changed for [110] oriented substrates because of etch rate difference between two orientations. After a series of experiments, a chemical mechanical polishing recipe was optimized for [110] oriented epi-ready PbSnSe substrate. Again, recipes of SOLN1 and SOLN2 are proprietary and subject to a NDA (Non-disclosure agreement).

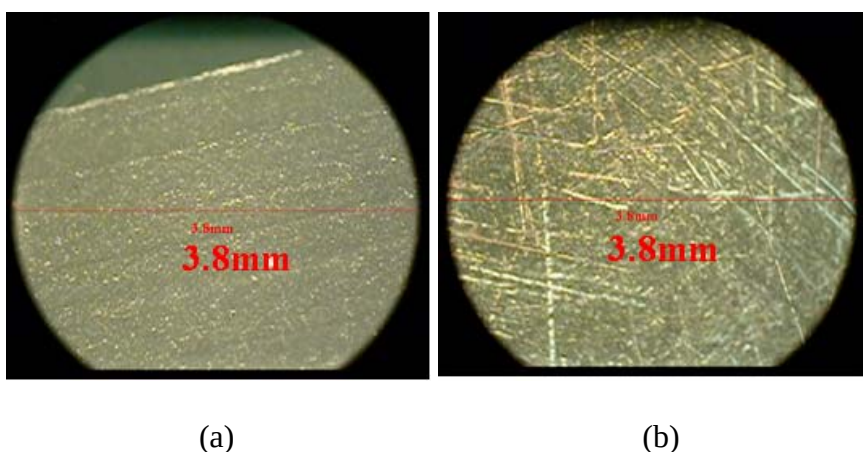


Fig 4.1 Nomarski image of the PbSnSe wafer surface before polishing (a); image of same wafer surface after mechanical polishing (b).

The figure above shows PbSnSe wafer surface before and after mechanical polishing. Unpolished wafers had several deep grooves and saw marks. Therefore, at first they were mechanically polished on a glass plate by hand with 5 μ m alumina suspension. This removes the deep saw marks but leaves scratches in the range of (5- 40) μ m. For the faster removal of these scratches a chemical cycle followed the mechanical. The first solution, SOLN1, used for chemical polishing has an etch rate of around 4 μ m/min. Figure 4.2 shows the Nomarski image of the wafer surface after chemical polishing with this solution.

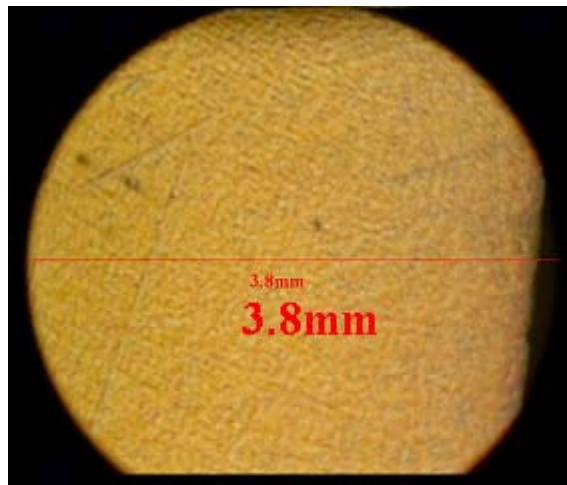


Fig 4.2 Nomarski image of the PbSnSe wafer surface after chemical polishing with SOLN1.

As can be seen from the figure, other than one or two light scratches, most of the deep scratches occurred during mechanical polishing were being removed by this solution. However, there still remained some surface non-uniformities and light scratches after polishing with SOLN1. The purpose of final polishing solution SOLN2 is actually to remove these surface non-uniformities that still left after polishing with SOLN1 and to produce a smooth surface.

As it was mentioned before that chemicals chosen for the chemical cycles were kept same as the one used for [100] oriented wafers, but the composition was changed for [110] oriented substrates because of difference in the outcome, Figure 4.3 is evidence of this fact. Figure 4.3 shows the SEM surface morphology of PbSnSe wafer polished with final polishing solution SOLN2 having the same composition of chemicals used for [100] oriented Pb-salt wafers.

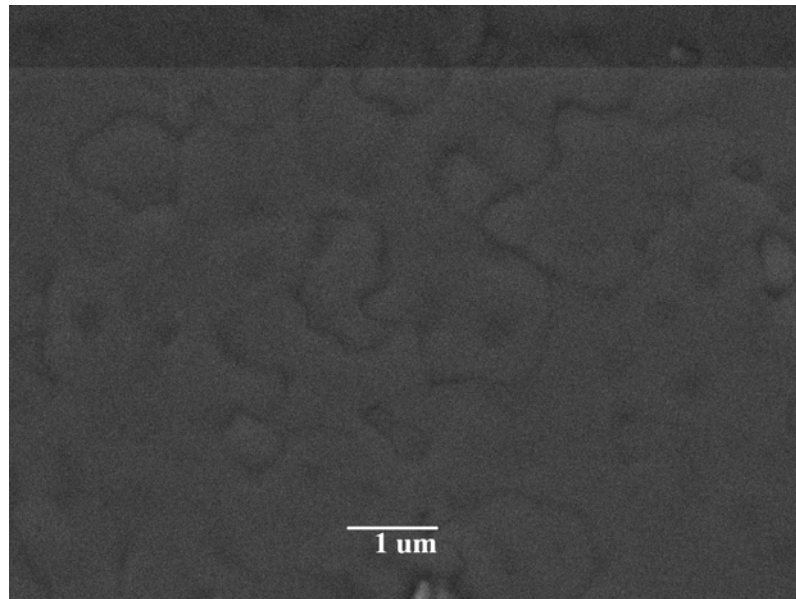
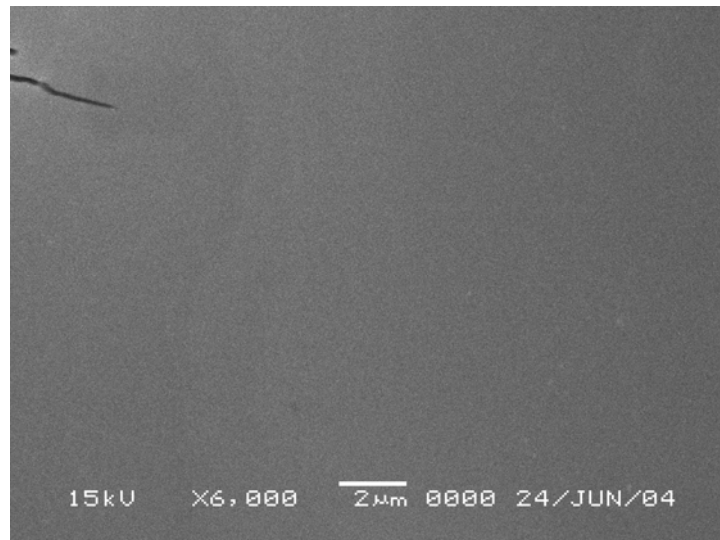


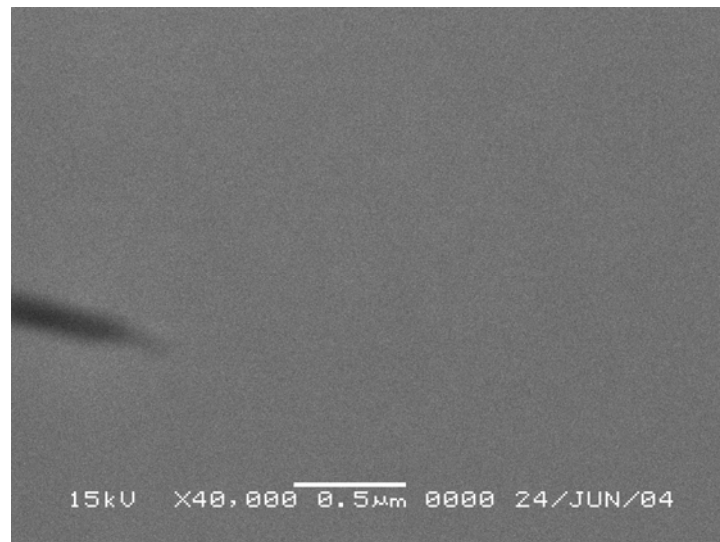
Fig 4.3 SEM of the PbSnSe wafer surface after polishing with final polishing chemicals having same composition as the one used for polishing {100} Pb-salt wafers.

From the SEM image it is quite clear that final polishing using the same composition of chemicals used for {100} wafers did not produce a defect free smooth surface for the [110] orientation. Therefore, the composition of the final polishing solution SOLN2 was changed for [110] oriented wafers.

SOLN2 with changed composition has an etch rate of around 1 $\mu\text{m}/\text{min}$. Figure 4.4 shows the SEM surface morphology of the PbSnSe wafer after polishing with a changed composition of SOLN2.



(a)



(b)

Fig 4.4 SEM of the PbSnSe wafer surface after polishing with changed composition of SOLN2 (a); same image at a much higher magnification of the microscope (b).

As can be seen from the above figure, a smooth defect free surface for {110} PbSnSe was obtained after polishing with an optimized chemical solution. Based on the image, a convenient standard for the end point detection of PbSnSe wafer was also developed. When the light scattering of a narrow intense spot (focused by a lens of Nomarski optical microscope) from the wafer is invisible by the naked eye, the surface morphology of the wafer is comparable to the surface morphology shown in Figure 4.4. This method was therefore accepted as the standard end point detection for epi-ready wafer.

Since most of the Pb-salts are extremely soft, this poses practical difficulties when handling bulk single crystals. Extreme care is needed and samples must not be treated with regular tweezers¹. Therefore, throughout experimentation with Pb-salt substrate, filter papers and chemically resistant sample holders were used to handle the sample for complex processing. After chemical polishing cycle the samples were cleaned under flowing DI water for about 30 minutes. Before MBE growth the wafers were cleaned consecutively in electronic grade organic solvents, such as acetone, propanol and methanol. During growth of diode laser structure on polished {110} PbSnSe wafers, two dimensional RHEED pattern was observed from the substrate surface.

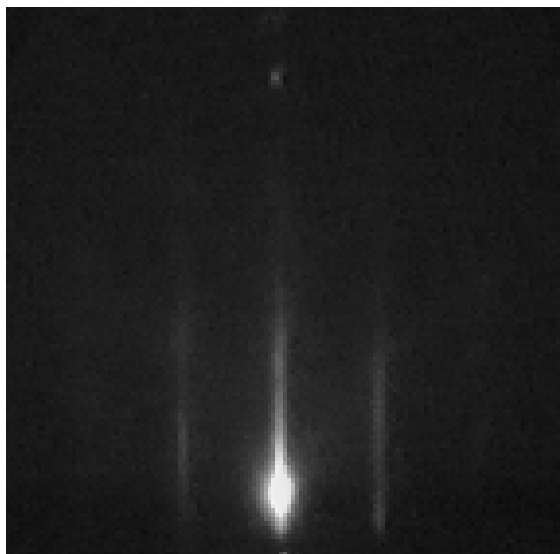


Fig 4.5 RHEED pattern from {110} PbSnSe substrate surface before epitaxial growth

Figure 4.5 shows the RHEED pattern from such a substrate before starting epitaxial growth. Streaky RHEED pattern as it can be seen in the figure, confirms a defect free smooth substrate surface for 2-dimensional epitaxial growth.

4.2 Electrically Pumped Diode Laser with Si₃N₄ Insulating Layer

PbSe/PbSrSe double heterostructure lasers on Pb-salt substrate were first developed by Spanger *et al.* in 1988². In PbSe-based lasers most commonly PbSSe, PbEuSe and PbSrSe are used as cladding materials; but among all these PbSrSe has some advantages. It was reported by Zogg *et. al.*, “in PbEuTe, high carrier-concentration p-type films are obtained up to 600 meV of band gap while in PbEuSe band gap cannot exceed 450 meV in order to grow such film. In PbSrSe-system p- and n-type films with higher carrier concentration are readily obtained even for band gaps above 600 meV”³. Commercially available Pb-salt lasers are fabricated with stripe geometry. Therefore, initially for the fabrication of diode laser on PbSnSe substrate the stripe geometry was followed with some modifications for better heat dissipation. The schematic of the structure of a commercial Pb-salt laser chip is shown in the figure below for reference.

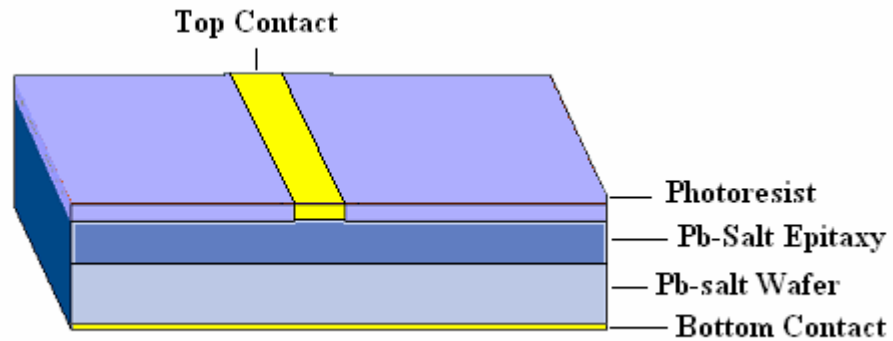


Fig 4.6 Schematic of a commercial Pb-salt diode laser chip

In our diode laser structure, the photoresist layer as shown in Figure 4.6 was replaced by a layer of silicon nitride.

4.2.1 Device structure and fabrication

PbSe/PbSrSe double heterostructure was grown on {110} PbSnSe substrate for the cleaved cavity diode laser fabrication. The laser fabrication process flow is shown schematically in Figure 4.7.

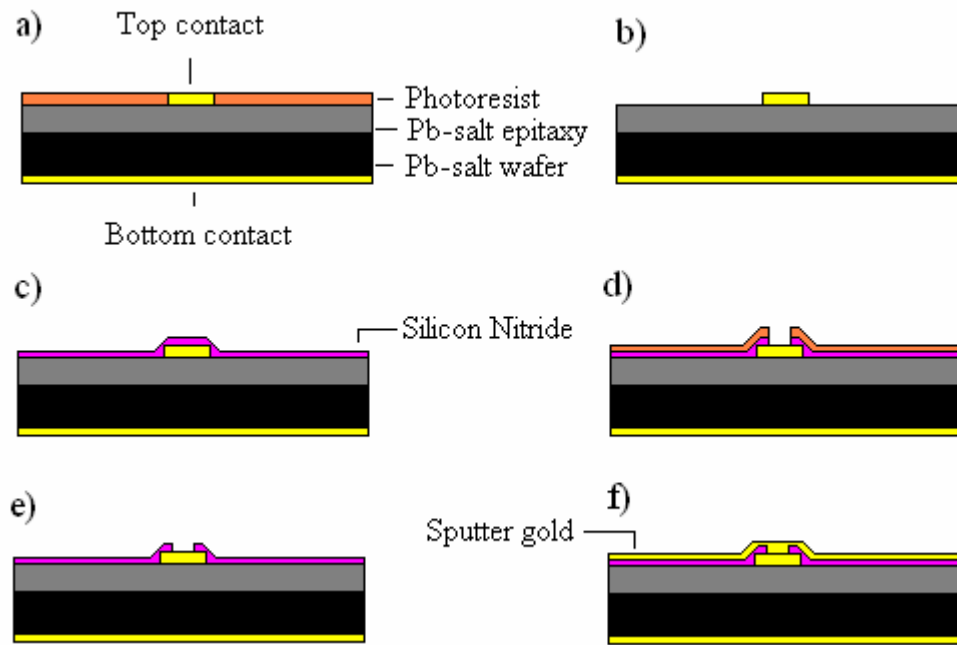


Fig 4.7 Schematic of processing steps for the fabrication of cleaved cavity PbSe/PbSrSe DH laser on {110} PbSnSe substrate.

Results from two samples processed according to the above mentioned processing steps, will be presented in this section. The active region of the first sample LD1 consisted of 1.03 μm of PbSe/PbSrSe double heterostructure, having 0.72 μm undoped PbSe and 0.11 μm of undoped PbSrSe. The PbSrSe p-confinement layer was 2 μm thick and the PbSrSe n-confinement layer was 1.5 μm thick. The p- and n-confinement layers were followed by two graded p- and n- PbSrSe layers of 0.06 μm thickness, where the Sr composition was graded between 3% and 4%. And the top most layer was a 0.2 μm thick

n+-PbSe cap layer. The second sample, i.e LD2 had almost same structure, except p-confinement layer, which was 1.48 μm thick and n-confinement layer, which was 1 μm thick. Also there was no graded layer in this structure. The {110} PbSnSe substrates used in these fabrications are p-type doped and have hole concentrations at around $5\text{-}8 \times 10^{18} / \text{cm}^3$.

Initially during MBE growth on {110} PbSnSe, [100] oriented PbSe wafers were also mounted together with {110} wafers for epitaxial growth to compare lasing results from two different orientations. But after one or two runs of growth it was found that there are some differences in the growth condition for the two different orientations. Growth conditions that produced satisfactory results on {110}, did not produce high quality epilayers on {100} substrates. Surface morphology of the epitaxial layer on {100} PbSe wafer, grown at the same run along with LD1 sample on {110} PbSnSe, is shown in Figure 4.8 below.

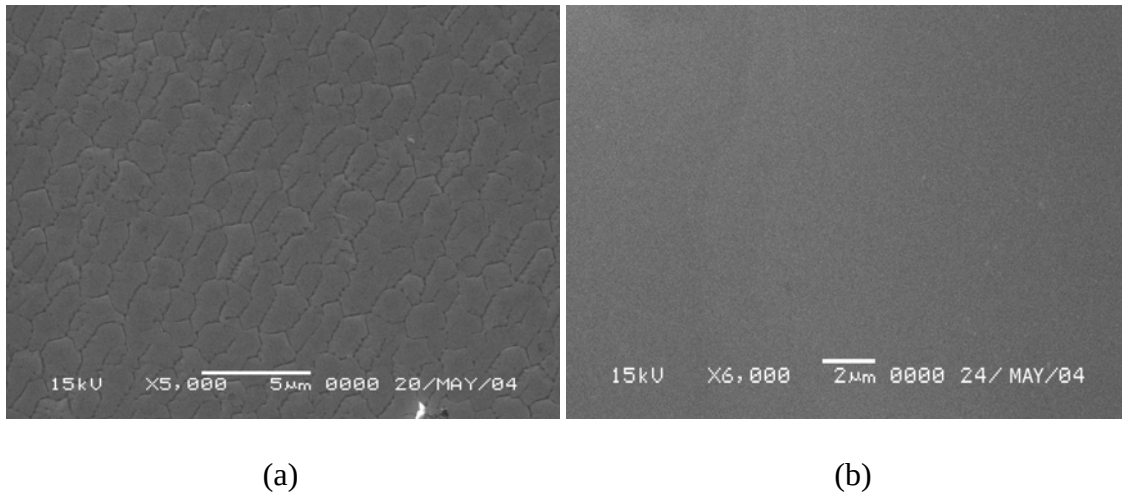


Fig 4.8 SEM surface morphology of the epitaxial layer on {100} PbSe wafer (a), grown at the same run along with sample on {110} PbSnSe wafer (b).

It is clear from the SEM images that epitaxial layer on {100} wafer has a rougher surface morphology. Hence, comparison could not be done for the epitaxial layers grown at the same run for these two orientations.

After MBE growth of the epitaxial layer, the sample was thinned from the back side, i.e the substrate side, from an original thickness of 500 μm to 250 μm for facilitating cleaving. After thinning, the back side of the substrate was polished with a chemical solution for the better adhesion of electrodeposited gold during laser fabrication.

After thinning, the sample was cleaved along the {100} plane that is perpendicular to {110} plane. Then 40 μm wide stripe patterns were made perpendicular to the {100} plane by a photolithographic technique. The microscope attached with the camera of our mask aligner has a maximum magnification of 20X. Therefore, to avoid any miss-alignment of the stripe contact with the {100} cleaved facet due to lower magnification of microscope, wider contact stripe was preferred. From a simple geometrical calculation it can be inferred that 1° miss-alignment can create a 5 μm deviation from perpendicular to obliqueness of the stripe to facet, for a 300 μm long cavity. After analyzing several stripe geometry lasers fabricated in our lab by SEM, it was found that maximum alignment error ranged from 0.1° to 0.3°. For a 300 μm long cavity laser this could lead to a shift of 0.52 μm to 1.57 μm . Therefore, the reason for choosing a large contact width of 40 μm was to make the alignment error negligible. Because of the large contact width, multi mode behavior, higher threshold current and joule heating and therefore lower CW operation temperature was anticipated. However,

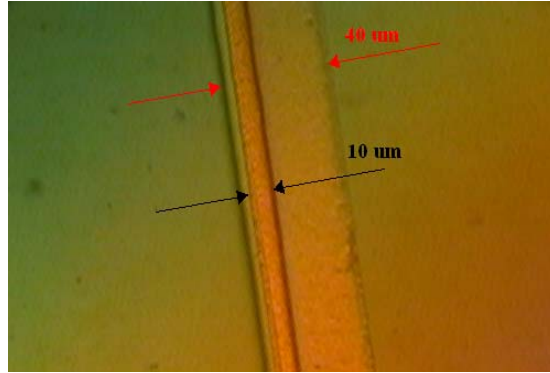
the goal of such laser structure is to demonstrate the feasibility of the [110] oriented lead salt laser.

After photolithography a 0.3 μm thick Au layer was electroplated on the stripe and on the back side of the substrate to form both top and bottom contact as shown in Figure 4.7a. The photoresist was then completely removed from the sample as shown in Figure 4.7b and 0.2 μm thick Si_3N_4 layer was sputtered on top of the sample as shown in Figure 4.7c.

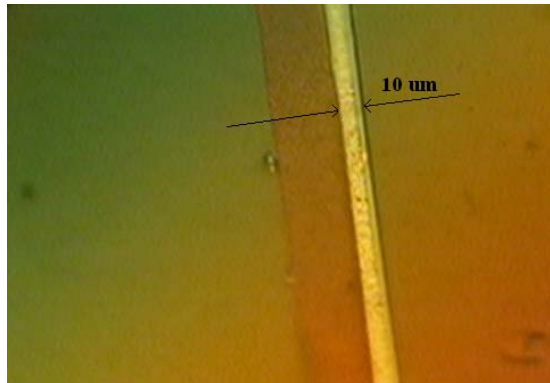
Following the sputtering, a second time photolithography was performed on the sample to open a 10 μm wide window on gold stripe and etch the Si_3N_4 from the window to expose the gold stripe underneath for contact. This step is shown in Figure 4.7d.

Opening window on gold stripe was another challenge. In our mask-aligner the camera for alignment is on one side and the exposure facility is on other side of the machine. And sample stage on a rail moves from one side to the other for exposure after alignment, and this is the time when there is a chance of a shift of the sample from the aligned position. The problem with mis-alignment is that if the window is opened even slightly outside the gold stripe, then the area of current spreading region will change and device performance will also change. Therefore, after the photolithography step for window opening, each time the sample was checked under microscope to find out the position of the window on the stripe. If there was an alignment error the photolithography step was repeated.

Figure 4.9 shows the Nomarski image of the sample after having improper alignment of the window in the Si_3N_4 deposited on the gold stripe.



(a)



(b)

Fig 4.9 Nomarski image of the sample after having improper alignment of the window in the Si_3N_4 deposited gold stripe; image before etching Si_3N_4 (a); image after etching Si_3N_4 (b) from window.

Figure 4.9 shows the Nomarski image of the sample with mis-alignment of the window.

Figure 4.10 shows the Nomarski image of a properly aligned window. Si_3N_4 wet etching was carried out by 7:1 BHF solution at room temperature. The etch rate was around 0.04 $\mu\text{m}/\text{min}$.

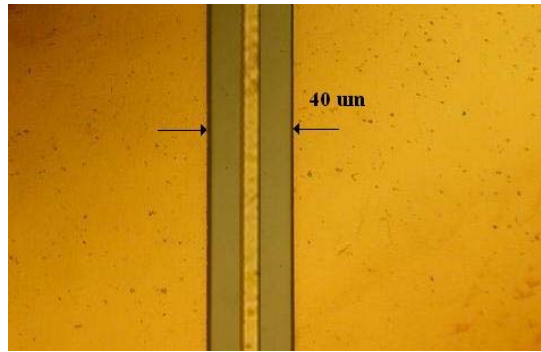


Fig 4.10 Nomarski image of the sample after having proper alignment of the window over Si_3N_4 on top of the gold stripe. Si_3N_4 had not been etched from window region yet.

After etching Si_3N_4 from the window the next step was sputtering a layer of gold on top of the whole sample, as shown in Figure 4.7f. The purpose of this layer was better heat dissipation through broad metal sheet. It also makes the epispide down mounting easier. This is the last step in the laser fabrication process, after this step the sample becomes ready for cleaving and mounting on a laser head for measurements.

The cavity and mirror facets for lasing were formed by cleaving. Cleaving of a single laser chip is a manual process. Cavity length usually ranged from 300-500 μm . Figure 4.11 shows a scanning electron micrograph of the cleaved cross section of a single laser chip after cleaving.

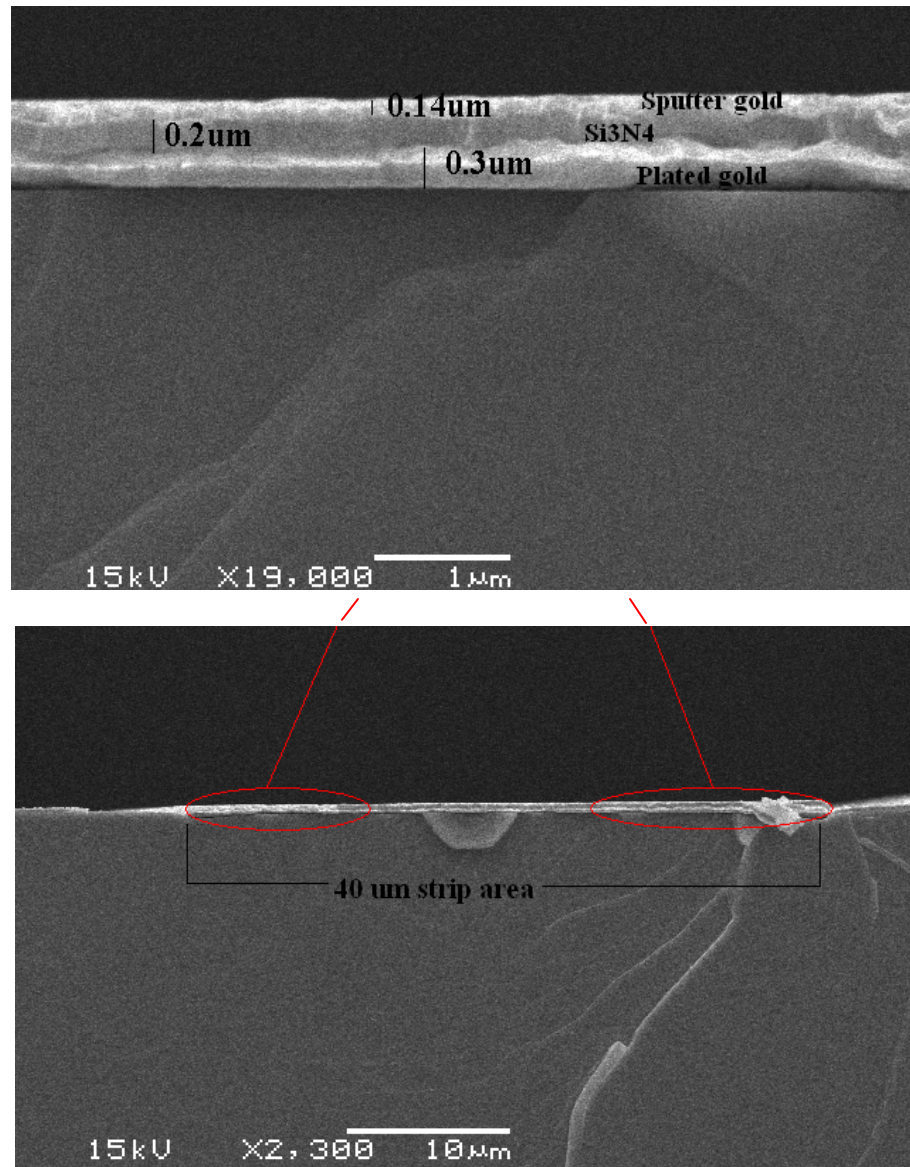


Fig 4.11 SEM image of cleaved cross section of a single laser chip. The image at the top is the magnified version of the region beside the window region on gold stripe. On two sides of the window Si_3N_4 was not etched. Hence, at that region gold is sputtered on Si_3N_4 .

Figure 4.12 shows a scanning electron micrograph of the window region at a higher magnification, where Si_3N_4 was etched and gold got sputtered directly on the plated gold stripe.

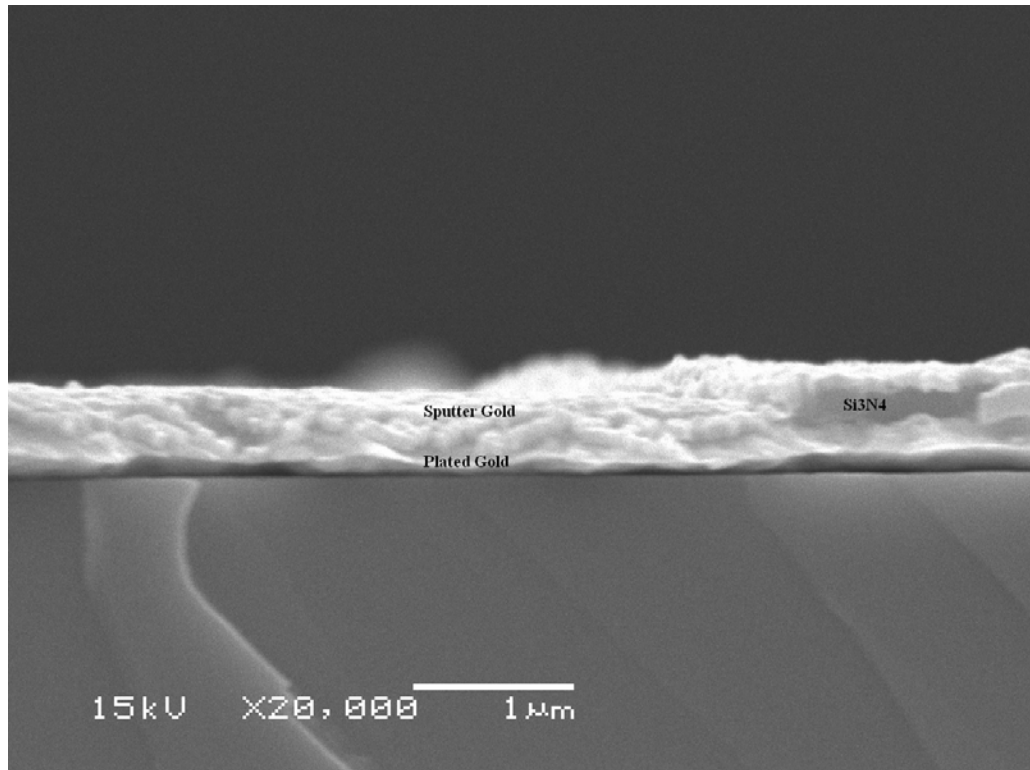


Fig 4.12 Scanning electron micrograph of the window region at a higher magnification

The sample that is shown in the images is not the best cleaved sample. It is difficult to see layer details from the mounted sample that are usually kept for measurement, therefore a different piece had been used to check the fabrication detail.

After cleaving, the next step was the mounting of the single laser chip on the laser head, a small pillar made of copper, which is a part of the laser housing. To mount the laser chip on this pillar, Indium is electroplated on top surface of the pillar. Figure 4.13 shows the picture of the laser housing with the chip mounted on it and SEM image of a mounted chip.

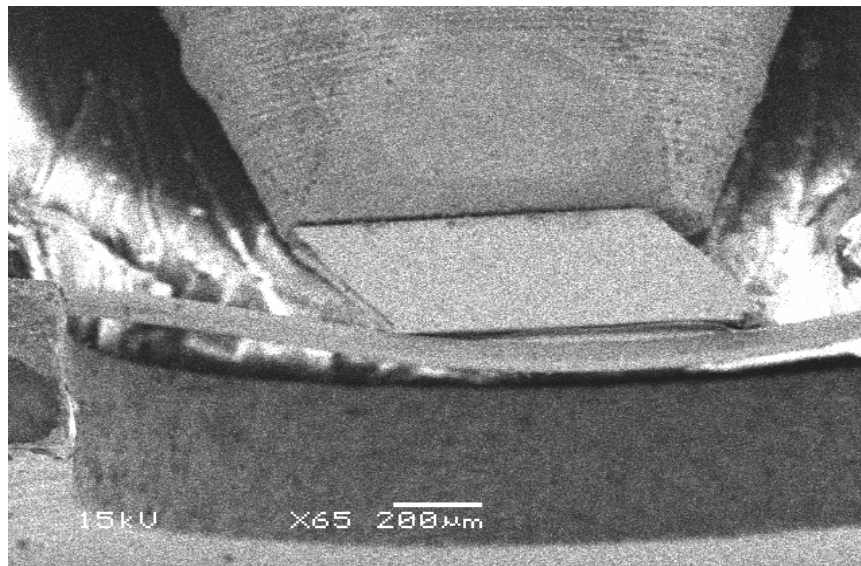
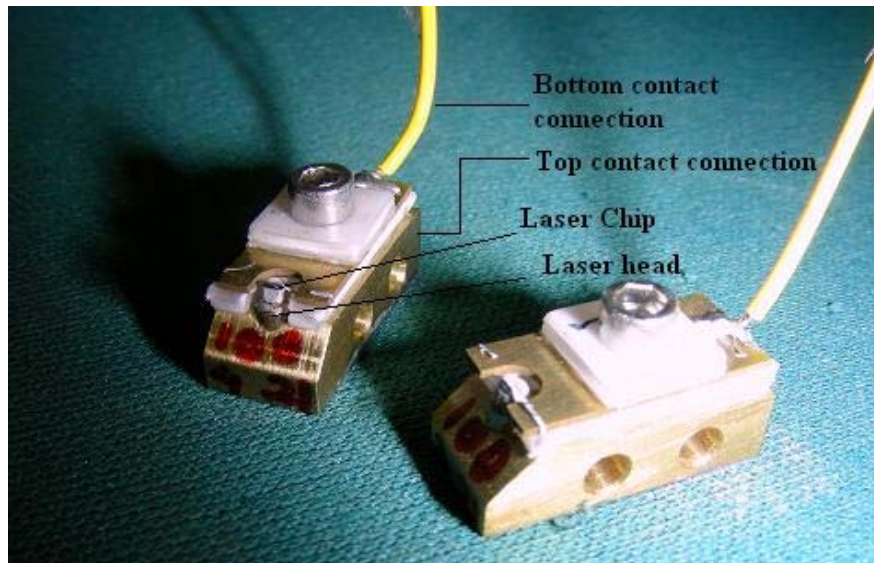


Fig 4.13 Picture of laser housing with the chip mounted (a); Scanning electron micrograph of the mounted chip, showing leaser head pillar on the top and chip at the bottom.

Since, 'In' metal has a quite low melting temperature (157°C), freshly plated Indium on laser head, acts as an adhesive to stick the laser chip even at room temperature. The chip was placed episeide down by vacuum tweezers on freshly 'In' plated pillar while observing under a microscope. After placement of the chip on the pillar, a thin freshly

'In' plated copper wire secures the chip on the pillar tightly. This wire also carries current to the bottom contact. This Cu wire is visible in the SEM image, shown in Figure 4.13, at the bottom of the chip.

4.2.2 Device characterization

After mounting the laser diodes, typical tests were performed that indicated that the device lase: device showed threshold characteristics, spectral narrowing upon FTIR spectrum analysis. This section details the characteristics of the fabricated device and interprets the results.

At first to check the diode characteristics, the current versus voltage relationship of the diodes were measured in forward bias. Due to variation in material characteristics over the entire wafer, the forward bias resistance and I-V characteristics varied from chip to chip, selected from different portions of the wafer. Figure 4.14 below shows the I-V relationship at 77K of several chips cleaved from different portions of sample LD1.

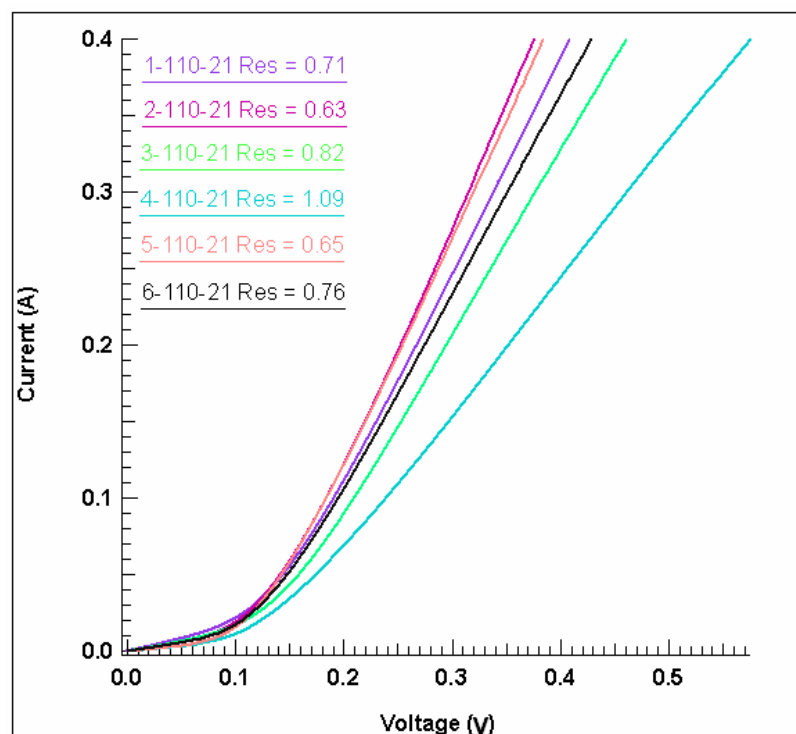


Fig 4.14 I-V characteristics of cleaved facet diodes selected from different regions of the same wafer, measured at 77K. Forward bias series resistance values in Ohms, for different chips are appended on the graph.

The data from the chip which had narrower linewidth will be presented here. The I-V curve of that typical chip at 77K is shown in the Figure 4.15 below.

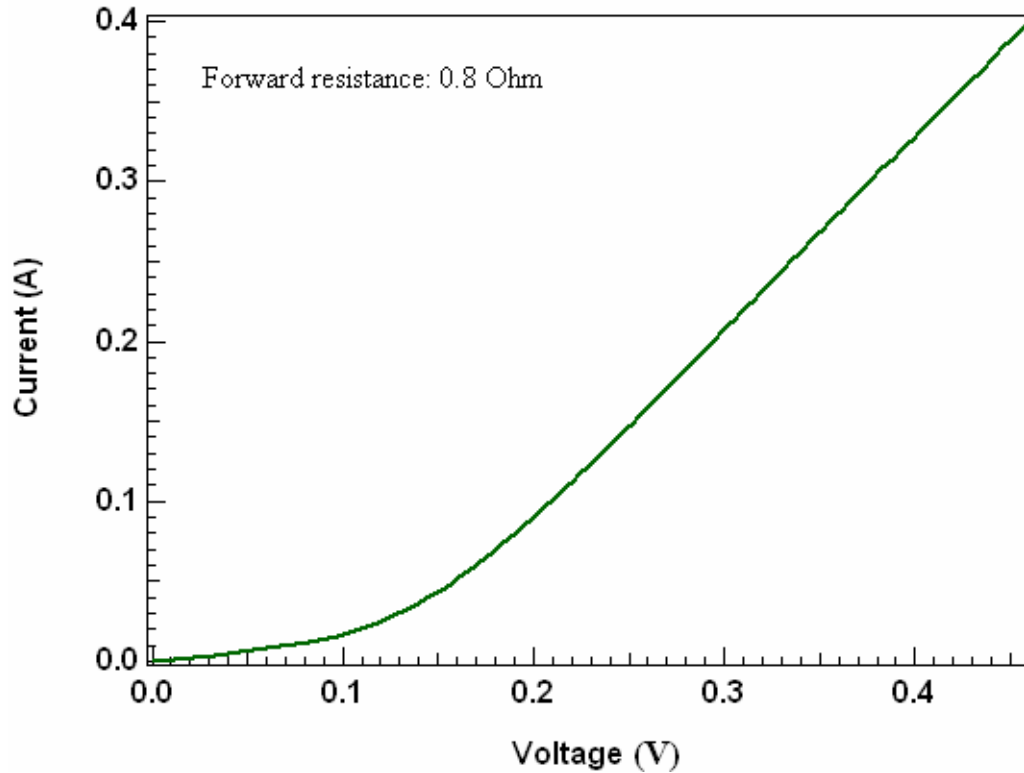


Fig 4.15 I-V characteristics at 77K of a typical diode; Forward bias series resistance is appended on the graph.

The emission spectrum from one facet of this sample was measured at liquid nitrogen temperature and higher temperature with an IFS 66/S spectrometer in step-scan mode using a liquid nitrogen cooled MCT detector having rise time of 500 ns. The MCT detector had a pre-amplifier connected with it. Device testing was performed with 500 ns pulses with pulse repetition frequency of 100 kHz (5% duty cycle), provided by a DEI – PCX-7410 laser diode driver. For this sample, CW pumping and pumping with higher pulse-width had been tried, but no emission was observed.

The light collection optics and the measurement set up for the spectrum analysis of the diode laser chip using FTIR is shown in Figure 4.16.

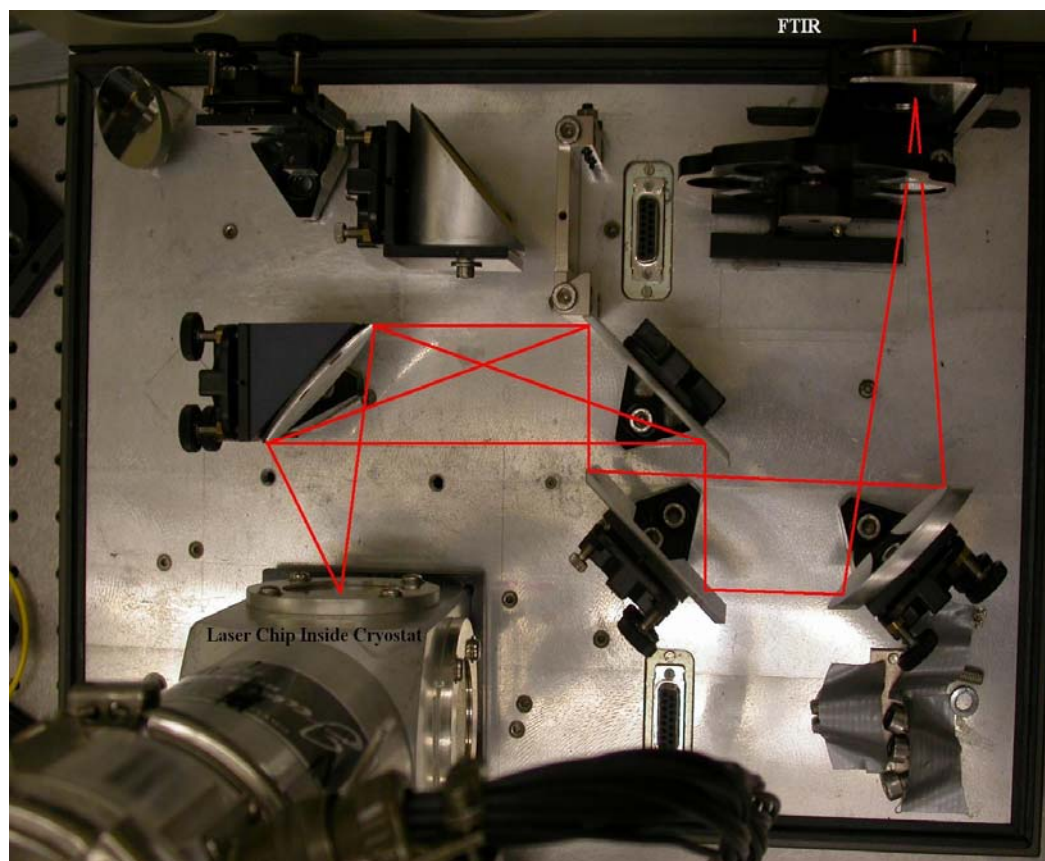


Fig 4.16 Light collection optics for the FTIR spectrum analysis with superimposed optical traces

The measured emission spectrum of the diode laser above threshold at the heat sink temperature of 80K is shown in Figure 4.17.

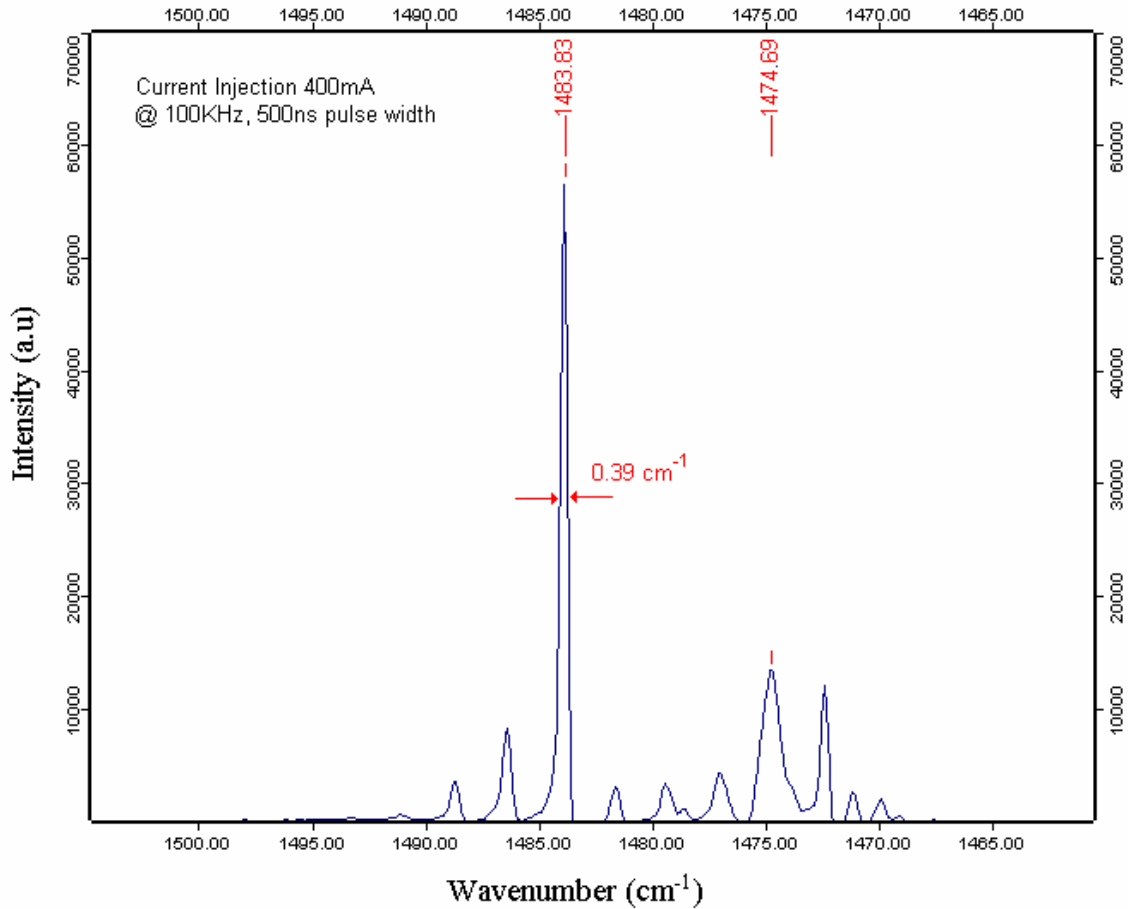


Fig 4.17 Laser emission spectrum above threshold from sample LD1 at a temperature of 80K.

As seen in the figure, the peak laser emission wavelength was 6.7 μm at 400 mA injection currents for a heat sink temperature of 80K. The spectrum was measured with a 0.25 cm^{-1} resolution setting of the FTIR. The line width of the spectrum measured in wavenumber scale was 0.39 cm^{-1} even at this low current. Higher resolution of the

spectrometer could have lead to narrower linewidth; however the FTIR does have some limitations in terms of resolution.

Figure 4.18 shows the spectra or emission peaks at different injection currents at the heat sink temperature of 80K and hence represents the current tunability of the emission spectrum.

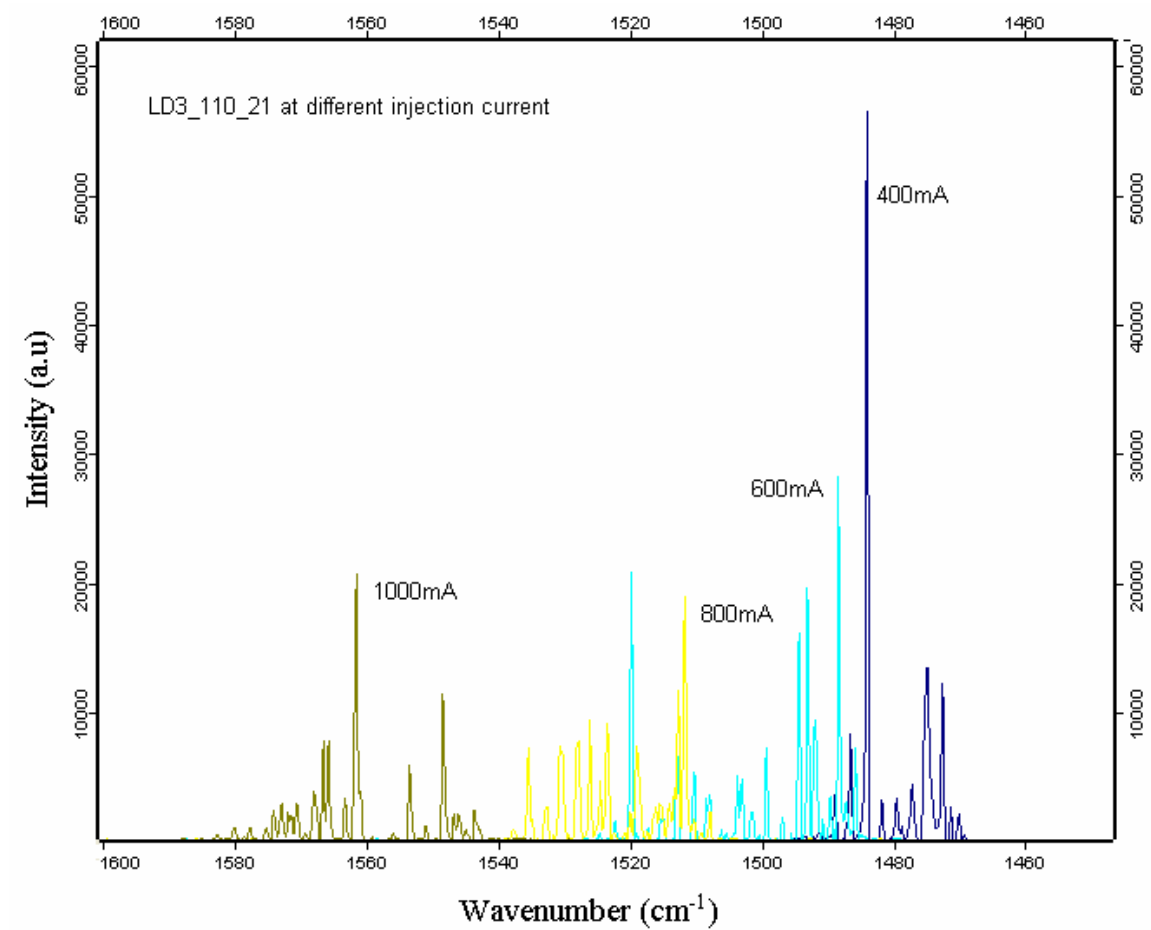


Fig 4.18 Laser emission spectra above threshold from sample LD1 at different injection currents at a heat sink temperature of 80K

From the figure, it can be seen that the current tuning range increases at higher injection current. From 400 to 800 mA current range, the tunability of peak emission in

wavenumber scale is almost $0.0675 \text{ cm}^{-1}/\text{mA}$. Above 800 mA the tunability is almost $0.25 \text{ cm}^{-1}/\text{mA}$.

The L-I characteristics or the light output versus injection current data of this laser chip were collected at different heat sink temperature. The light output was expressed in terms of detector output voltage. The measurement set up that was used to collect these data is schematically shown in Figure 4.19.

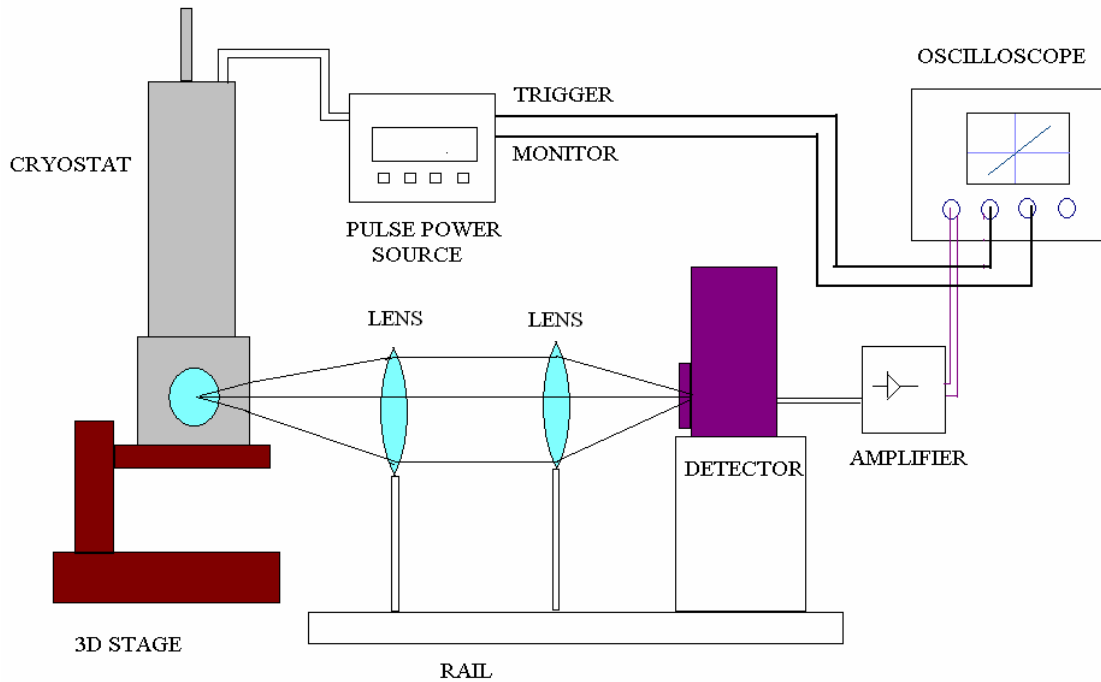


Fig 4.19 Schematic of the measurement set up used for collecting L-I data for the sample LD1

In this set up, the emitted laser light from the sample was focused by two CaF_2 lenses on to the MCT detector and the amplified signal from the detector was measured by the oscilloscope. This is a very fast method to measure and obtain the L-I curve of a sample. However, this set up was used for measuring the sample LD1 only. Later on for other

samples, the light output power at different injection current was calibrated using a standard blackbody reference source directly from the emission spectrum.

Figure 4.20 shows the L-I characteristics, i.e the light output versus injection current data of the sample LD1 at different heat sink temperature.

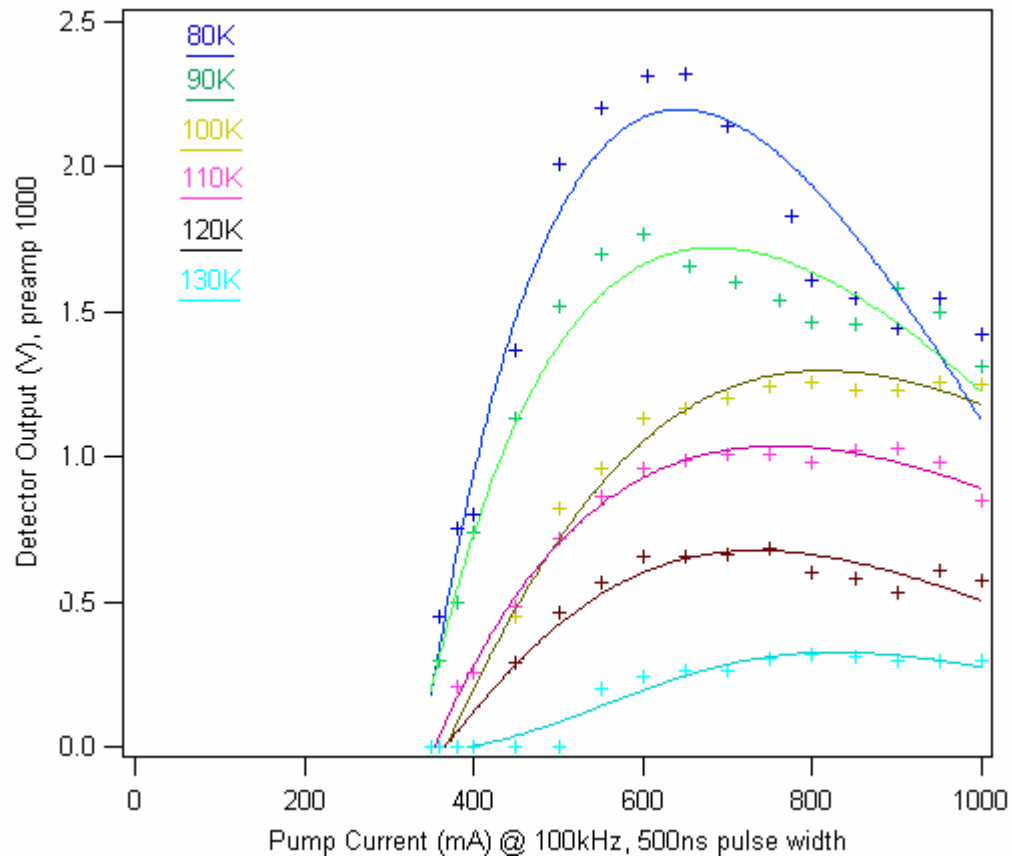


Fig 4.20 Light output vs. Injection current (L-I) curve of LD1 at different operation temperature

In the above curve the light output has been expressed in terms of amplified detector output voltage. Detector output signal was 1000 times amplified by a preamplifier. From the L-I curve it can be seen that above threshold at a particular operation temperature, initially as the injection current increases, the laser output power also increases but after

a certain injection current the output decreases. The reason behind this is the Joule heating of the device at higher current values. Threshold current of the device also increases with increasing operation temperature.

Figure 4.21 shows the I-V curve of a typical laser chip at 77K from the sample LD2 that was processed the same way as LD1, but had slightly different layer thicknesses as mentioned before.

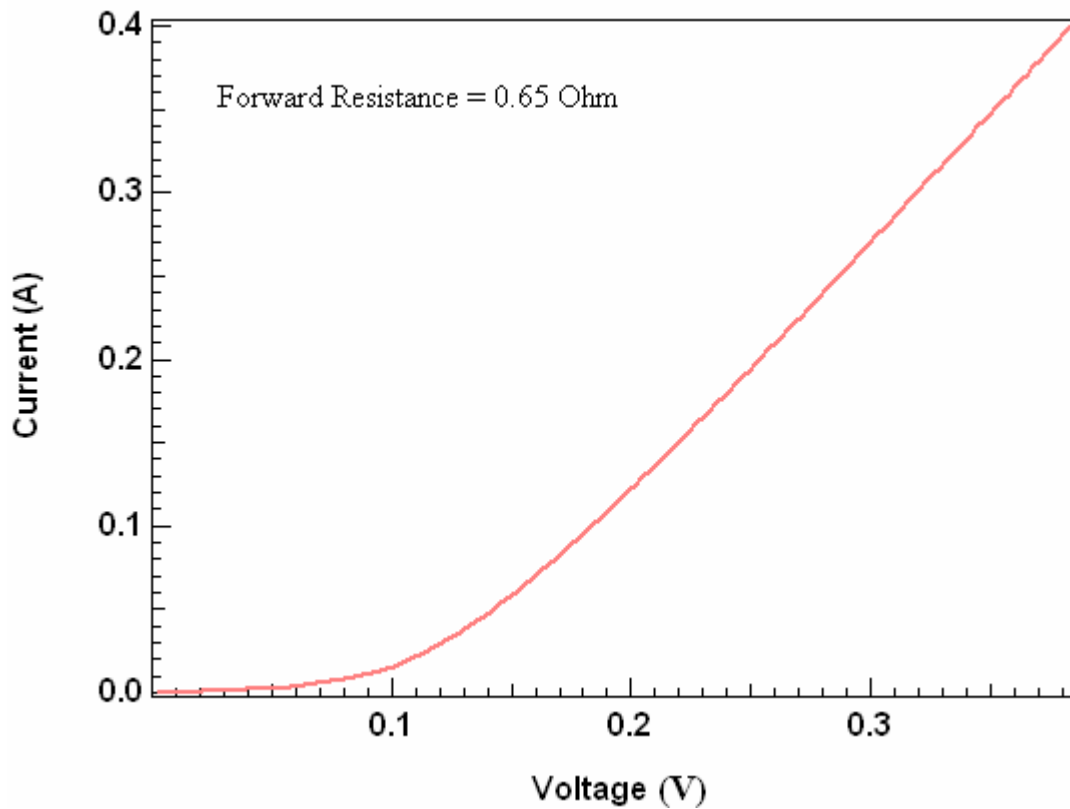


Fig 4.21 I-V characteristics at 77K of a typical diode laser from sample LD2. Forward bias series resistance is appended on the graph.

The laser emission from this sample was also measured at different heat sink temperature by the FTIR spectrometer. This sample also lased at a pump pulse width of 500 ns, with 100 kHz repetition frequency.

The measured emission spectrum of this diode laser above threshold at the heat sink temperature of 77K is shown in Figure 4.22.

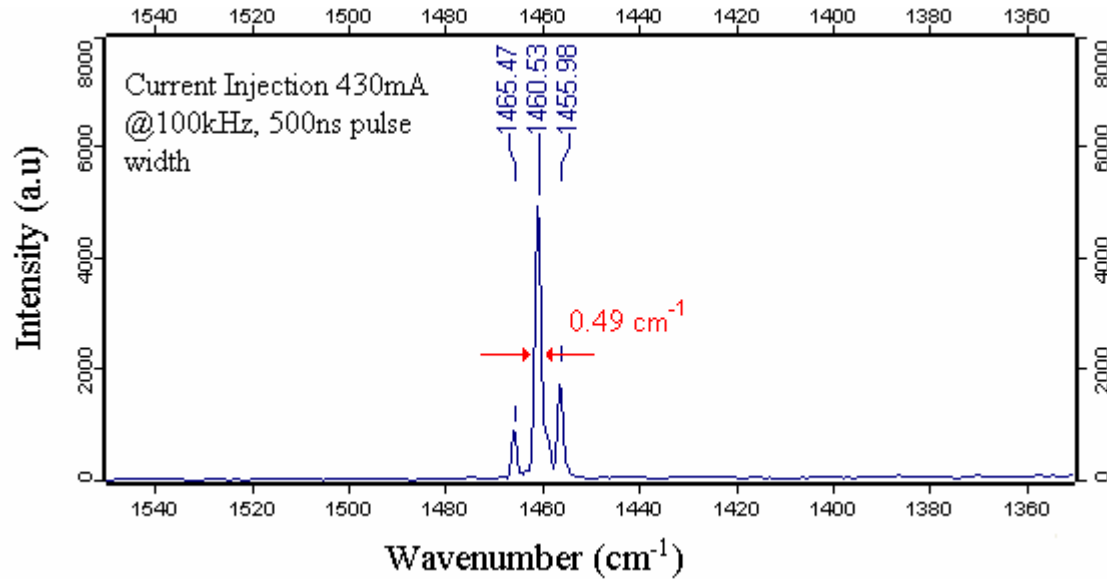


Fig 4.22 Laser emission spectrum above threshold from sample LD2 at temperature 77K

As seen in the figure, the emission is multimode with a modal spacing of approximately 5 cm^{-1} . Peak laser emission wavelength was $6.85 \text{ }\mu\text{m}$ at 430 mA injection current at 77K. This spectrum was also measured with a 0.25 cm^{-1} resolution setting of the FTIR. The line width of the peak emission as can be seen in the figure is 0.49 cm^{-1} which is a little bit higher than the linewidth obtained from LD1. Also, the measured threshold current density of LD1 at 80K was approximately 1.8 kA/cm^2 , whereas for LD2 the measured threshold current density at 77K is about 2.05 kA/cm^2 . The difference in the emission characteristics of these two samples is not quite obvious, but a probable reason could be a difference in the material quality as well as cleaving and mounting, as the latter two processes are completely manual. Also, this sample was processed after almost one

month from the day of completion of MBE growth. Therefore, to some extent there could be a probability of material degradation upon aging. This interpretation is based on the observation of reduced PL or EL intensity from some sample with time, although the reason behind this is not exactly known.

Figure 4.23 shows the spectra at different injection currents at a heat sink temperature of 77K and hence represents the current tunability of the emission for LD2.

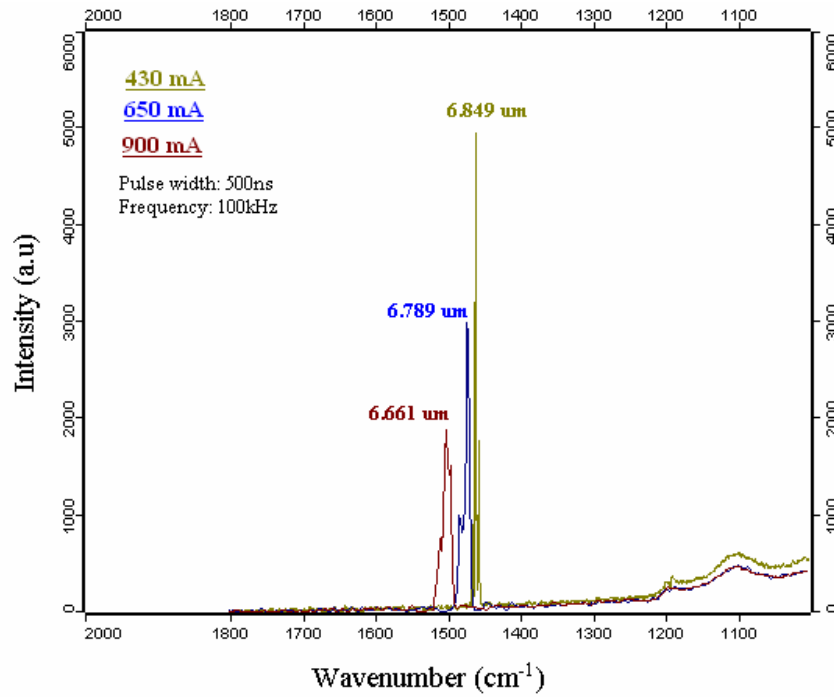


Fig 4.23 Laser emission spectra above threshold from sample LD2 at different injection current at a heat sink temperature of 77K

From the figure, it can be seen that by increasing current from 430 to 650 mA, the peak emission wavelength can be tuned by almost 17 cm^{-1} , in wavenumber scale. Hence, the tunability of peak emission is almost $0.077 \text{ cm}^{-1}/\text{mA}$, from 430 to 650 mA current range.

Figure 4.24 shows the L-I characteristics, the light output versus injection current data of the sample LD2 at different heat sink temperatures. The output power from one facet of the sample was calibrated using a standard blackbody reference source whose emission energy at a certain temperature and blackbody field of view was provided by the manufacturer.

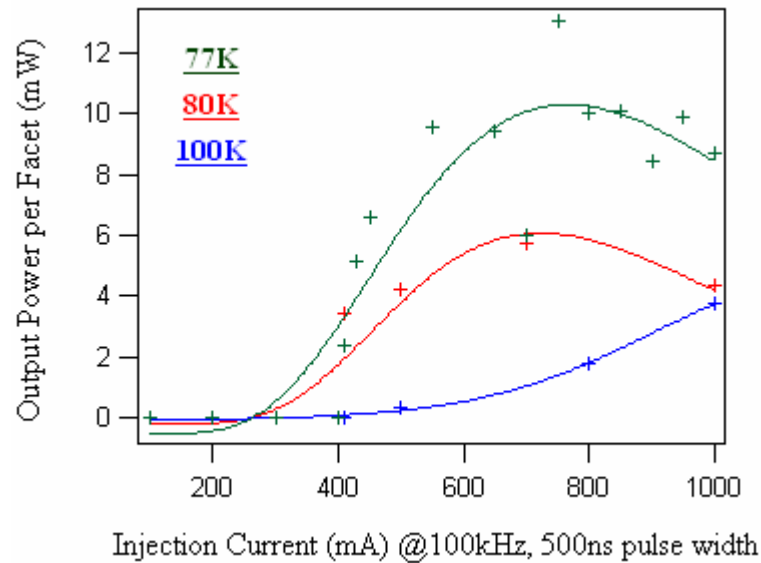


Fig 4.24 Light output vs. Injection current (L-I) curve of LD2 at different operation temperature

As can be seen in the figure, maximum peak output power of more than 10 mW was obtained at a heat sink temperature of 77K. However, as the operation temperature increases the output power decreases and the threshold current density increases. With increase in temperature, phonon assisted loss mechanisms become dominant and that deteriorates the laser performance. Also initially as the injection current increases, the laser output power increases but after a certain injection current the output decreases because of the Joule heating at higher current.

4.3 Electrically Pumped Diode Laser with Polymer Insulating Layer

Continuous wave operation of PbTe/PbSnTe double heterostructure laser at 77K was obtained in 1974, from a simple stripe geometry structure⁴. Our primary aim for PbSe/PbSrSe based laser was also obtaining CW operation from the structure. Hence, for the further fabrication of laser structures on PbSnSe substrate we chose to proceed with the commonly used photoresist or polymer insulating layer in accordance with the stripe geometry, avoiding the time consuming fabrication process mentioned in the previous section. The schematic of this simple stripe geometry structure is shown in the figure below.

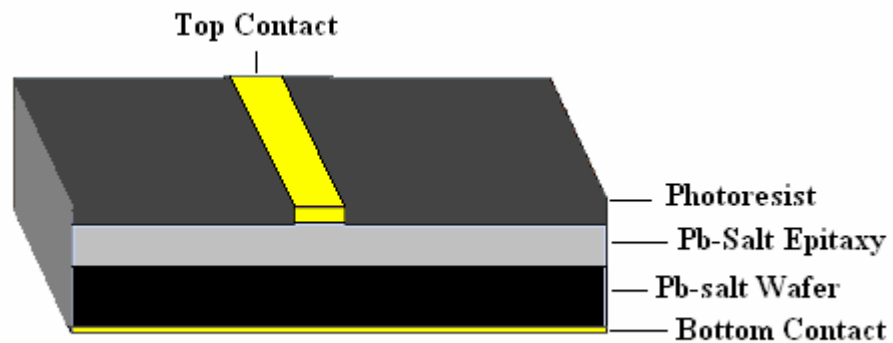


Fig 4.25 Schematic of a Pb-salt diode laser chip with photoresist insulation

4.3.1 Device structure and fabrication

PbSe/PbSrSe double heterostructure was grown on {110} PbSnSe substrate for the cleaved cavity diode laser fabrication. The active region of the sample consisted of 0.616 μm of undoped PbSe. PbSrSe p-confinement layer was 1.44 μm and PbSrSe n-confinement layer was 1.32 μm thick. The p- and n-confinement layers were followed by two graded p- and n- PbSrSe layers of 0.24 μm and 0.15 μm thickness, where the Sr composition was graded between 3% and 4%. And the top most layer was 0.15 μm n+-PbSe cap layer. The laser fabrication process flow is shown schematically in Figure 4.26.

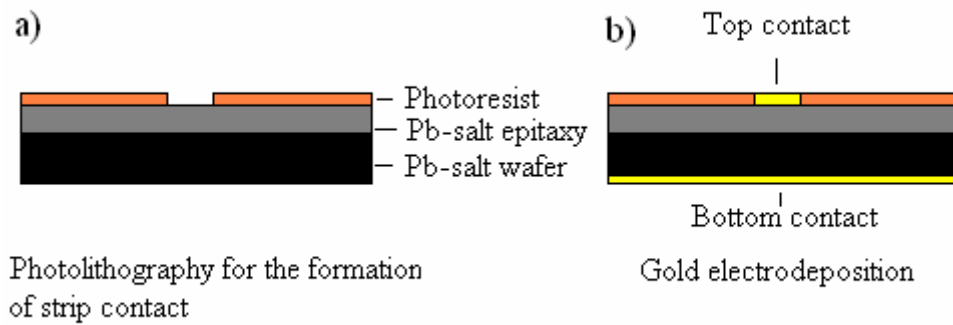


Fig 4.26 Schematic of processing steps for the fabrication of cleaved cavity PbSe/PbSrSe DH laser on {110} PbSnSe substrate, with polymer insulating layer.

After MBE growth of the epitaxial layer, the sample was thinned from the back side, i.e the substrate side, to 250 μm for the ease of cleaving. After thinning, the back side of the substrate was polished with a chemical solution for the better adhesion of electrodeposited gold during laser fabrication.

After thinning, the sample was cleaved to get the {100} plane that is perpendicular to the {110} plane. Then 40 μ m wide stripe patterns were made perpendicular to the {100} plane by photolithography. Electrodeposition of 1 μ m gold followed the photolithography for the formation of top and bottom contact.

The cavity and mirror facets for lasing were formed by cleaving, as was mentioned in the previous section. Cavity length usually ranged from 300-500 μ m. Figure 4.27 shows a scanning electron micrograph of the cleaved cross section of a single laser chip with photoresist insulation.

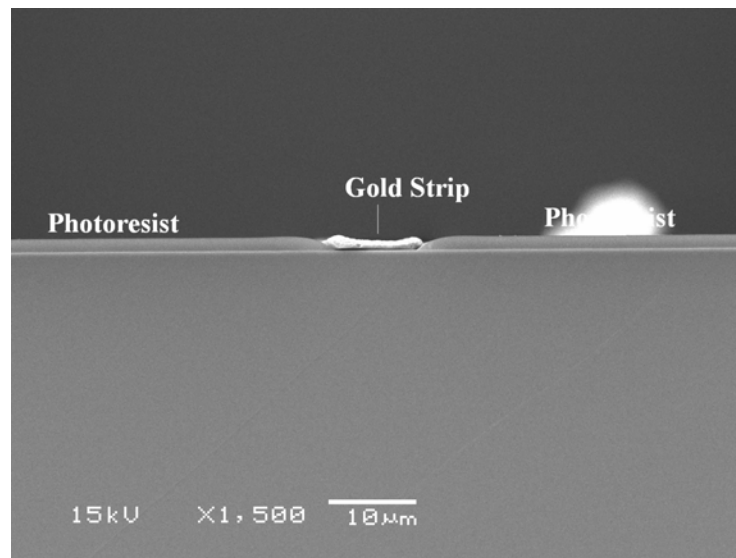


Fig 4.27 SEM image of cleaved cross section of a single laser chip with photoresist insulation.

The SEM image shows the process technology. However, this is not the same sample used for measurement. As it was mentioned before, measured samples are all mounted and packaged in laser housing, therefore SEM cross-section of those samples do not reveal process details. Mounting of these samples were also done by the same way as previously mentioned.

4.3.2 Device characterization

At first to check the diode characteristics, the current versus voltage relationship of the diodes was measured in forward bias. Due to variation in material characteristics over the entire wafer, the forward bias resistance and I-V characteristics varied from chip to chip, selected from different portion of the same wafer. The data from the chip which had narrower linewidth will be presented here. I-V curve of that typical chip at 77K is shown in the Figure 4.28 below.

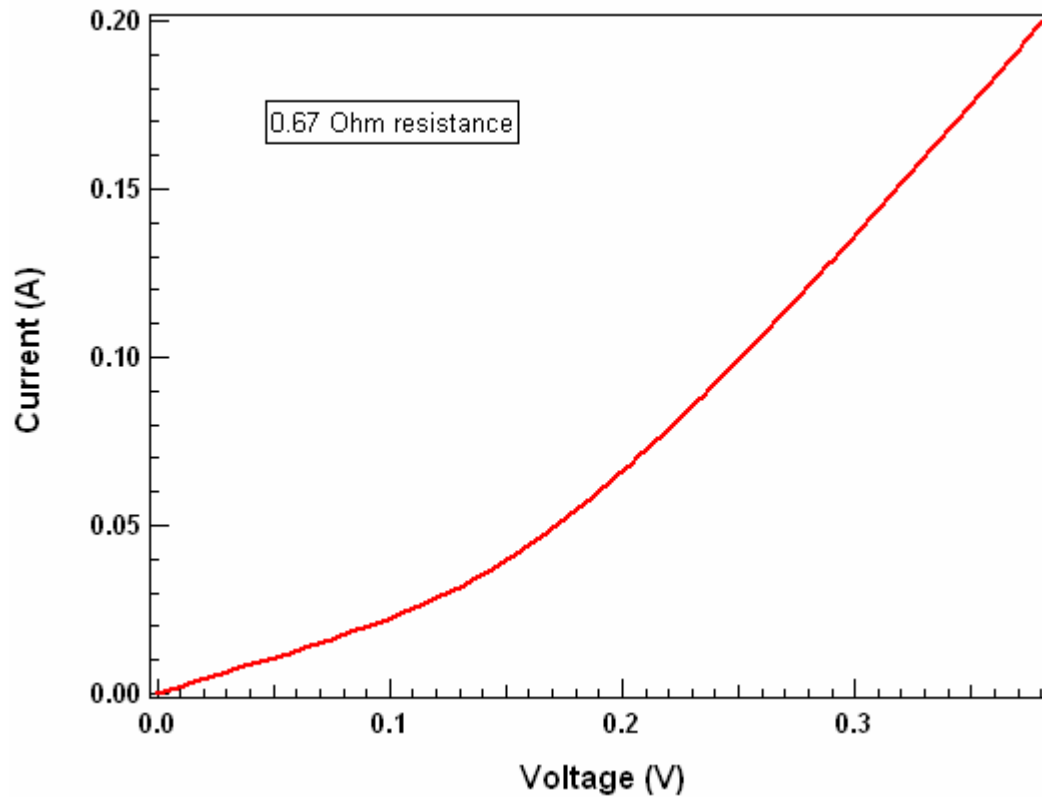


Fig 4.28 I-V characteristics at 77K of a typical diode; Forward bias series resistance is appended on the graph.

The laser emission from this sample was also measured at different heat sink temperatures using FTIR spectrometer. This sample lased at a pump pulse width of 3 μ s, with 100 kHz repetition frequency (30% duty cycle), which is much larger than the pump pulse width of previously measured samples. Therefore, positive improvement in the laser performance was being observed. However, CW emission was not observed from any of these samples. The measured emission spectrum of this diode laser above threshold at the heat sink temperature of 77K is shown in Figure 4.29 and Figure 4.30.

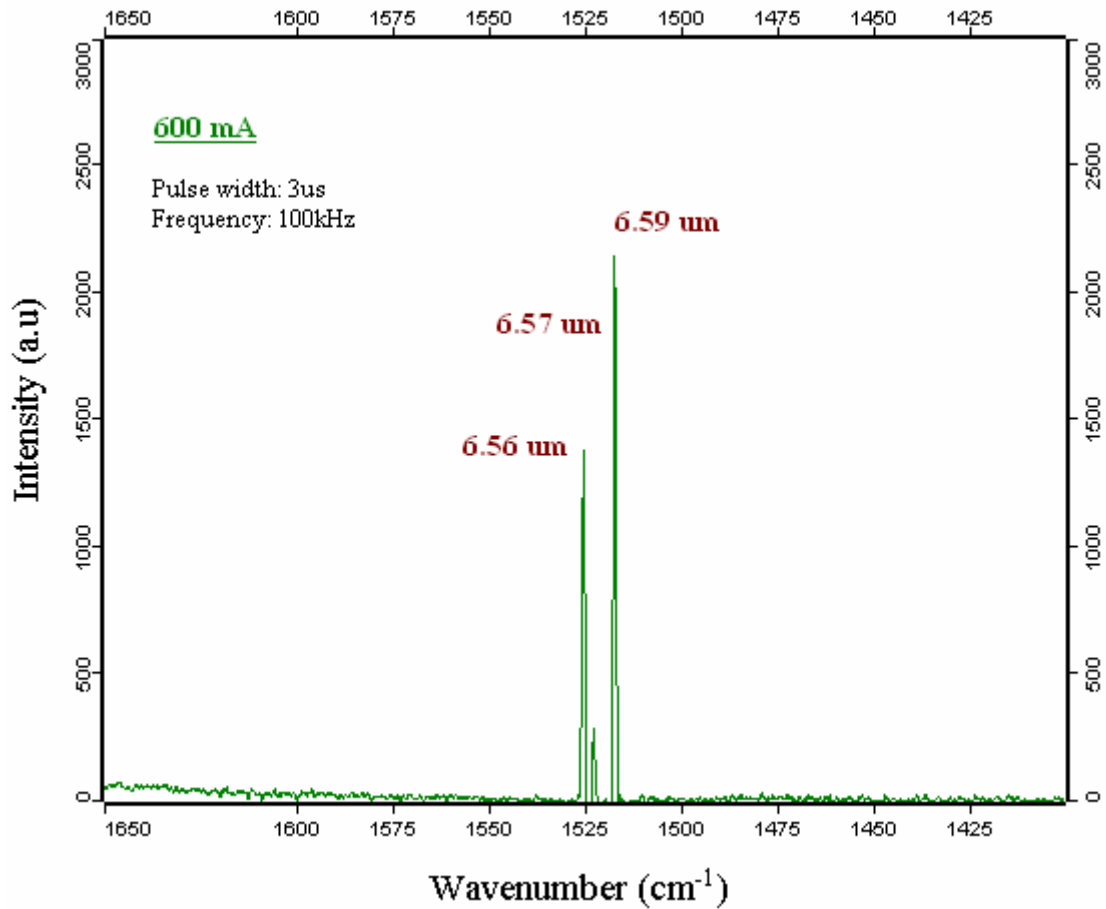


Fig 4.29 Laser emission spectrum above threshold at 600 mA injection current at temperature 77K

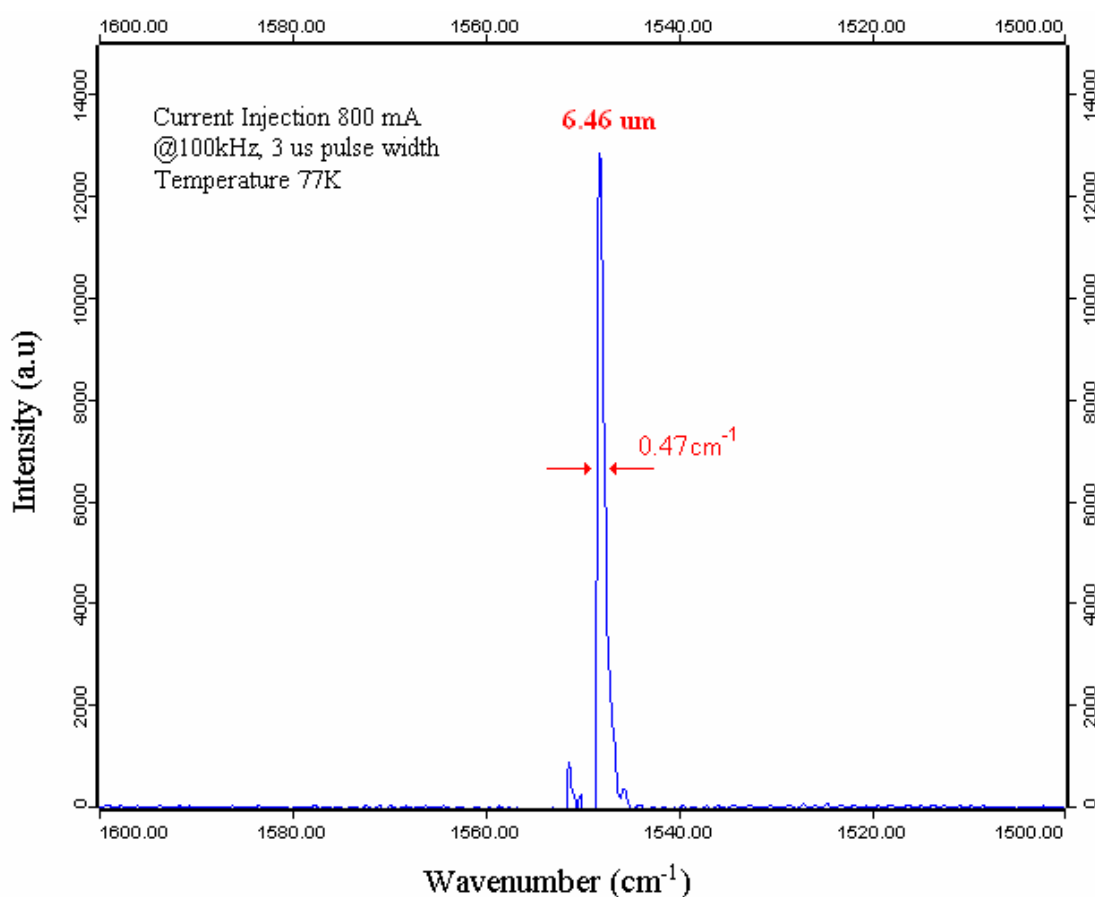


Fig 4.30 Laser emission spectrum above threshold at 800 mA injection current at temperature 77K

Peak laser emission wavelength was 6.59 μm at 600 mA and 6.46 μm at 800 mA injection current at 77K. At 600 mA three modes were observed with intermodal spacing of 5 cm^{-1} . These spectra were also measured with a 0.25 cm^{-1} resolution setting of the FTIR. The line width of the peak emission as can be seen in the Figure 4.30 is 0.47 cm^{-1} and was measured in wavenumber scale. Also the measured threshold current density was approximately 2.5 kA/cm^2 .

Figure 4.31 shows the spectra at different injection current at the heat sink temperature of 77K and hence represents the current tunability of the emission spectrum.

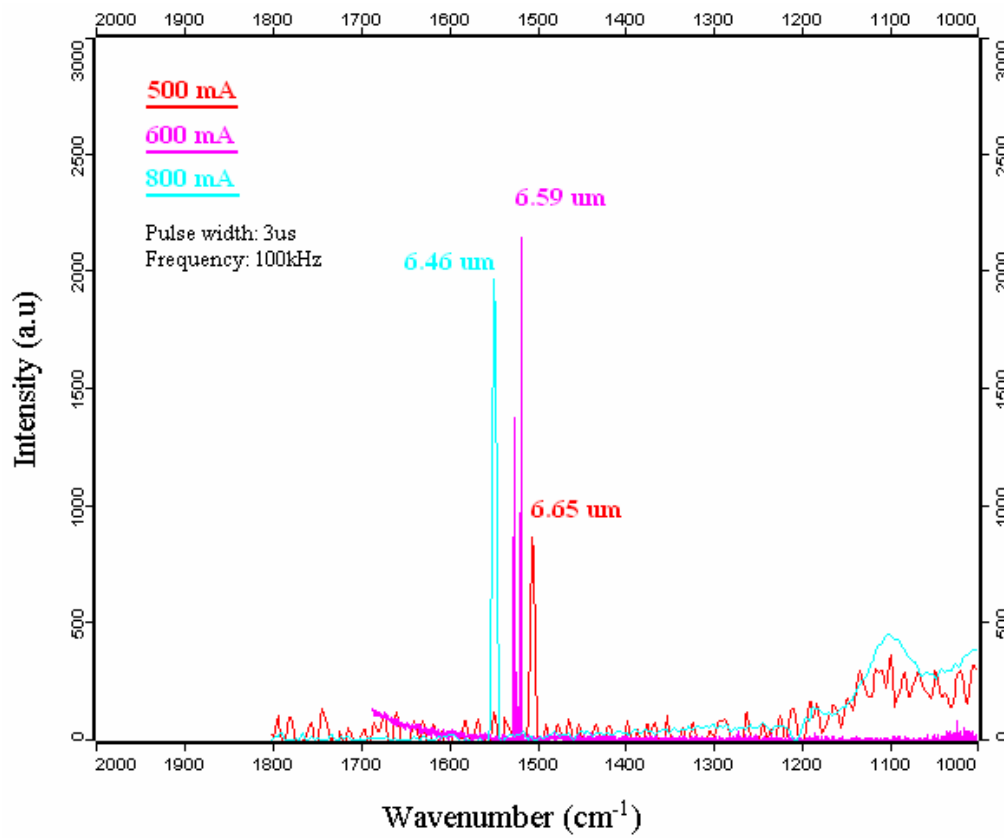


Fig 4.31 Laser emission spectra above threshold at different injection current at the heat sink temperature of 77K

From the figure it can be seen, by increasing current from 500 to 600 mA, the peak emission wavelength can be tuned almost 14 cm^{-1} (in wavenumber scale). Similarly in the above mentioned current ranges, the tunability of peak emission is almost $0.14 - 0.15 \text{ cm}^{-1}/\text{mA}$, measuring in wavenumber scale.

Figure 4.32 shows the L-I characteristics, the light output versus injection current data at different heat sink temperatures. The output power from one facet of the sample was calibrated using a standard blackbody reference source as mentioned in the previous section as well.

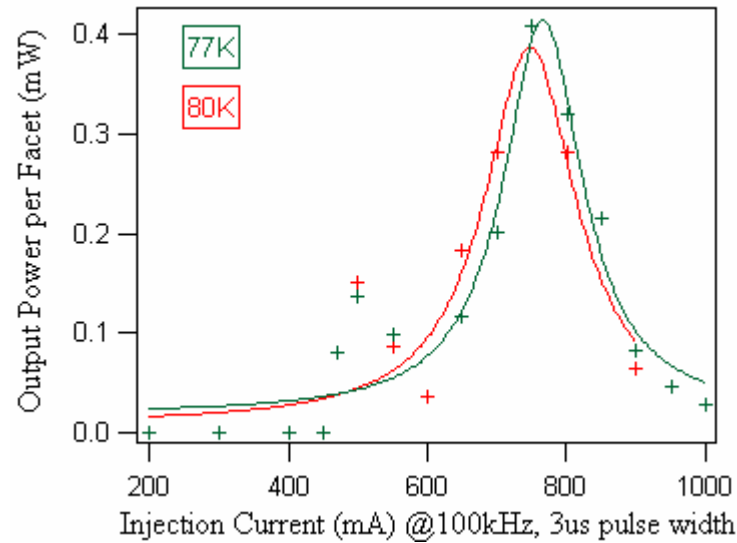


Fig 4.32 Light output vs. Injection current (L-I) curve at different operation temperature

As seen in the figure, maximum peak output power of 0.4 mW was obtained at a heat sink temperature of 77K, at 3 μ s pulse width. As the operation temperature increases the output power decreases and the threshold current density increases, due to the domination of phonon assisted losses. Lead salt materials have low thermal conductivity. At threshold, there is a large difference between lattice and heat sink temperature. As the injection current or the heat sink temperature increases, this temperature difference becomes higher and higher, which in turn lead to thermal rollover. Hence, a structure having better heat management could lead to much better performance from this device.

4.4 Conclusion

Conclusively, pulsed operation of PbSe/PbSrSe diode laser based on double heterostructure active region on {110} PbSnSe substrate has been reported. Threshold current density, as low as 1.8 kA/cm^2 , was obtained from the laser chips with broad stripe contact of width $40\mu\text{m}$. However, with broader stripe contact maximum operation temperature remained 130K. Instead of average power, peak output power from the laser chips were calculated as the pulse repetition frequency during pumping were kept very low in order to provide enough cooling time to the device. MBE growth on [110] oriented PbSnSe substrate has been performed for the first time. More experiments are needed for the optimization of growth condition for best lasing results. Material quality has been improved at each run of sample growth as is quite evident from the above mentioned results. As was interpreted from theoretical simulation on [110] oriented lasers, QW active region has the highest gain along [110] orientation in comparison to [100] or [111]⁵, replacement of double heterostructure with quantum wells could further improve the performance of these lasers.

4.5 References

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- ² B. Spanger, U. Schiessl, A. Lambrecht, H. Böttner, and M. Tacke *Appl. Phys. Lett.*, 53, 2582 (1988).
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Chapter 5

Design and Fabrication Process of Single Mode Lead Chalcogenide Laser

5.1 Single Mode Operation of Pb-Salt Edge Emitting Laser

Single mode tunable laser operation in the mid infrared spectral region is of great interest in high resolution IR spectroscopy, heterodyne detection and also in MIR-fiber transmission¹. A number of techniques have been employed to obtain single mode emission from lead chalcogenide lasers, among them are buried-heterostructure (BH) lasers², distributed feedback (DFB) lasers³ and distributed Bragg reflectors (DBR)⁴. Other than the buried-heterostructure, which operated in CW mode at 120K, DFB or DBR structures all operated below 77K. In the recent time in PbSnTe materials system, Nishijima *et al.*⁵ reported a BH laser with a single mode CW optical output power exceeding 1 mW. Figure 5.1 shows the wavelength coverage of a PbTeSe/PbSnTe buried heterostructure laser. Bold lines in the figure show the single mode region.

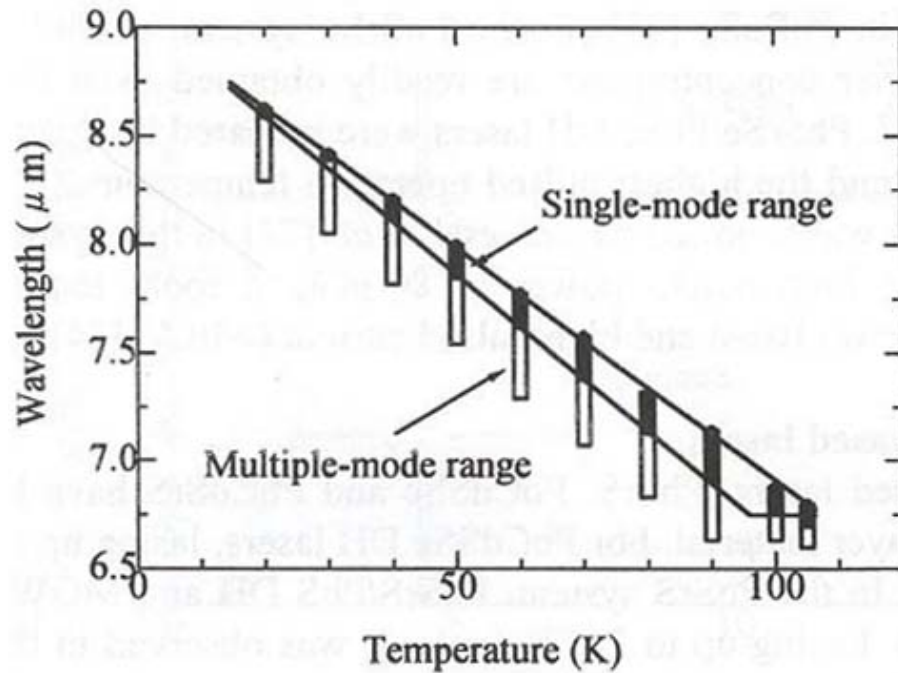


Fig 5.1 Wavelength coverage of PbTeSe/PbSnTe buried heterostructure laser ⁶

Among the three structures mentioned above, BH lasers structure in case of Pb-salt material showed some potential. PbEuTeSe/PbTe BH laser was reported by Feit *et al* and CW emission up to 223K was observed^{7 8}. From all these information, it was expected that a ridge waveguide (RW) structure would perform better for the single mode operation of the device. The advantage of this structure over BH structure is that, it is not as process sensitive as a BH structure. The fabrication of BH laser diodes require multiple epitaxial steps, interrupted by etching processes to build the active stripe, therefore any process defects occurred during fabrication affects its performance. Also the device technology is complicated and the reliability may be degraded. Whereas in ridge waveguide structure the whole epitaxial growth is finished in one run and etching takes place afterwards. The active region is not processed between epitaxial growth steps, and the lateral optical confinement is dominated by the index guiding. However, in the RW structure taking electrical carrier confinement into consideration, the effective active region width equals the stripe width w plus approximately one diffusion length on each side.

This chapter will describe the simulation of a ridge waveguide laser for single lateral mode operation for the PbSe/PbSrSe based material system. A method has been proposed where selective losses can be introduced for the higher order modes allowing the device to oscillate in single lateral mode. Also the fabrication process development for this ridge laser structure has been discussed.

5.2 Principles of Modeling Guided Waves

By definition, the field intensity of a guided mode decays monotonically in the transverse direction everywhere external to the core without losing power to radiation.

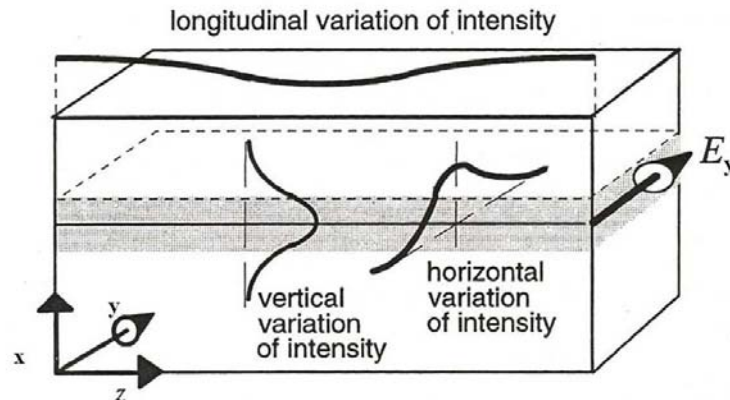


Fig 5.2 Schematic of vertical (Transverse) and horizontal (Lateral) optical field intensity⁹

There are formal distinctions between transverse and lateral guiding as shown in figure 5.2. The light usually propagates along the ‘longitudinal’ z-direction and the transverse and lateral terms refers to the directions (towards X and Y) perpendicular to z-direction. Lateral variation is described by the changes in the y-direction, which is perpendicular to the junction or layer interfaces. Vertical or transverse variation refers to changes in x-direction. X, Y, Z follows right handed set of axes. Longitudinal variations are usually taken to be forward and reverse traveling waves of constant amplitude. The forward and reverse traveling waves beat together to form standing waves. However, their average intensity remains uniform⁹.

5.2.1 Transverse wave guiding for slab-waveguide

A three layer slab waveguide is formed with three slabs where the material of lower refractive index embraces material with highest refractive index. The E and H fields of a plane wave propagating in one direction form a right handed set with,

$$\left| \frac{E}{H} \right| = \sqrt{\frac{\mu_r \mu_0}{\epsilon_r \epsilon_0}}, \text{ where } \epsilon_r \text{ and } \mu_r \text{ are the real part of material permittivity and permeability}$$

and ϵ_0 and μ_0 are the free space permittivity and permeability.

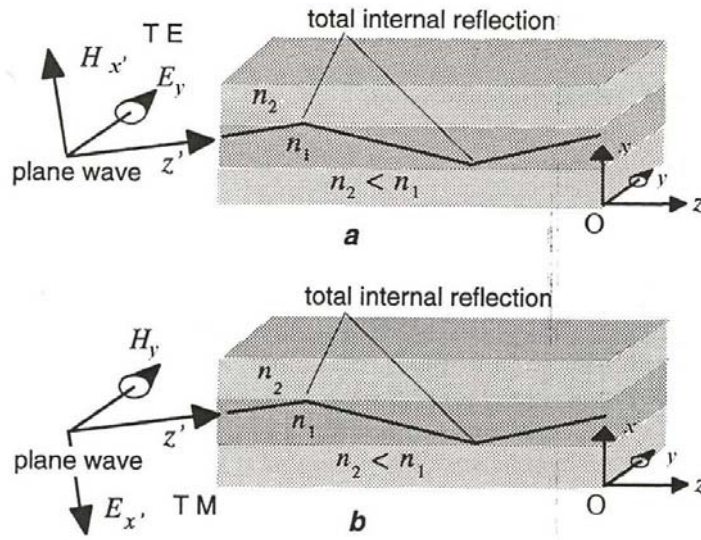


Fig 5.3 Schematic of Transverse Electric (a) and Transverse Magnetic mode (b)⁹

The orientation of E-field is considered to be along the plane of the junction as shown in Figure 5.3(a). Hence, the E-field has a component only in the horizontal direction, i.e. transverse to the junction; thus the nomenclature TE mode. When a plane wave is launched into this slab at an angle to O_z axis so that $O_{z'}$ is not exactly parallel to the O_z axis, then-only the entire structure acts as a guide. This plane wave is reflected through Total Internal Reflection at the boundaries when the angle of launching into the guide is

smaller than a certain critical value. The only difference between TM and TE mode is that, for TM mode, the H_y field is transverse to junction⁹.

In practice, the modes that are usually excited in semiconductor laser because of strong guiding by the di-electric waveguide are TE-modes. Thus they also exhibit higher modal gain. Moreover, the reflectivity of the end facet is higher for TE than for TM modes of the same order. So our calculation will be mainly based on TE modes. Later on we will show that each propagating mode can be described by a propagation constant β , where $\beta = n_{\text{eff}} k_0$. n_{eff} is close to n_2 for small thickness of core layer and close to n_1 for large thickness of core layer.

Gain or loss in the active layer is described by considering n_1 to be complex:

$$n_1 = n_1' + jn_1''$$

,where the active layer gain g_a is related to the imaginary part of n_1 by the following equation :

$$g_a = 2k_0 n_1''$$

The modal gain of a particular mode is given by,

$$g_{\text{eff}} = 2 \text{Im}\{\beta\}$$

The transverse structure of the laser diodes is designed in such a way that only fundamental mode is guided. In the symmetric dielectric slab waveguide as shown in Figure 5.3, this is the case for

$$d < \frac{\lambda}{2\sqrt{n_1^2 - n_2^2}},$$

where d is thickness of core layer¹¹.

5.2.2 Lateral wave guiding for slab-waveguide

The laser structures are described as either index guided (IG) or quasi-index guided (QIG), depending on how the lateral index guiding is accomplished. Two dimensional waveguides can be treated by the effective refractive index method¹⁰.

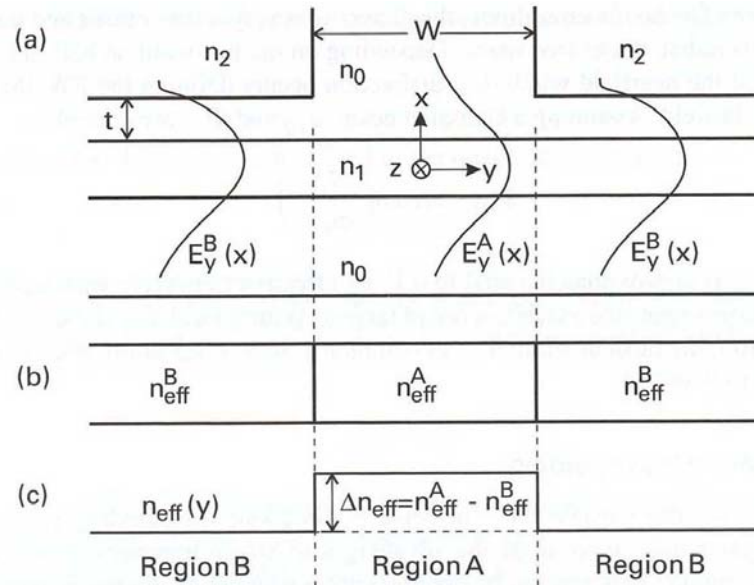


Fig 5.4 The effective refractive index method applied to a ridge waveguide (a) From the solution of transverse wave equation in each lateral region, an effective lateral slab waveguide is deduced (b) with effective refractive index step Δn_{eff} (c)¹¹.

The illustration of effective refractive index method is shown in Figure 5.4 for a QIG ridge waveguide (RW). As shown in figure 5.4 the RW consists of a laterally modified transverse dielectric slab, where part of the upper confinement layer has been replaced with a material of lower refractive index so that $n_0 > n_2$. The evanescent optical field becomes affected when the distance 't' to the active region is extremely small. The effective refractive index of the transverse mode outside the active stripe gets reduced depending on the confinement inside the lower index region. Accordingly, an effective lateral-slab waveguide can be made as illustrated in Figure 5.4(b) with effective

refractive indices n_{eff}^A and n_{eff}^B for the middle-stripe region and side-wing regions, respectively. When the effective refractive index difference $\Delta n_{eff} = n_{eff}^A - n_{eff}^B$, becomes positive, lateral wave guiding takes place.

Therefore, the 2D waveguiding problem can be solved with effective refractive index method by first solving the 1D wave equation for the slab waveguides along the transverse direction in the stripe or ridge region and the side or wing region and then using the effective refractive indices to build another effective lateral slab waveguide, and again solving the 1D lateral wave equation for this effective lateral slab waveguide.

As, $\Delta n = n_1 - n_0 \gg \Delta n_{eff}$, single mode condition ($d < \frac{\lambda}{2\sqrt{n_1^2 - n_2^2}}$) for the lateral direction allows $w \gg d$ ^{10,11}.

5.2.3 Solution of wave equation for modal analysis

To analyze the modal behavior in certain waveguide structure a we need to calculate the eigenstate for an optical TE or TM mode. By definition, a guided optical beam and a confined eigenstate for a quantum well have the boundary condition that as x approaches positive or negative infinity, the electric field (or wave-functions for the QW case) must approach zero.

Propagation of a plane wave is described by Equation 5.1.

$$\mu\epsilon \frac{d^2}{dt^2} E = \nabla^2 E \quad (5.1)$$

This equation can be simplified in the form,

$$E = E_0 e^{-j(\omega t - K(r) \cdot r)} \quad (5.2)$$

Considering a plane wave propagating in the transverse direction in a slab waveguide, the gradient can be described using Cartesian Co-ordinate like this,

$$\nabla^2 E = \frac{d^2 E}{dx^2} + \frac{d^2 E}{dy^2} + \frac{d^2 E}{dz^2} = \frac{d^2 E}{dx^2} + 0 + (-k_z^2)E \quad (5.3)$$

$$\mu\epsilon \frac{d^2}{dt^2} E + k_z^2 E = \frac{d^2 E}{dx^2} \quad (5.4)$$

$$\mu\epsilon \frac{d^2}{dt^2} E = \mu\epsilon(r)\omega^2 E = -|K(r)|^2 E = -n^2(r)k_0^2 E \quad (5.5)$$

In these equations, E is the electric field profile, k_z is the eigenvalue for the mode, $n(r)$ is the index as a function of position, and $K(r)$ is the wave function. The modal index or the effective refractive index for a particular mode is expressed as $n = k_z/k_0$, where $k_0 = 2\pi/\lambda$.

For uniform propagation in the z direction, we want to describe the mode shape in the x direction. K is proportional to the index of refraction in each section and so it varies across the waveguide. For a single mode, k_z remains the same across the entire waveguide. For this reason, the second derivative of “ E ” with respect to “ x ” position must change to compensate for the change in index in each layer of the waveguide.

$$\frac{d^2 E}{dx^2} = -(n^2(r)k_0^2 - k_z^2)E \quad (5.6)$$

For the TE and TM cases, Maxwell’s equations require that the transverse electric and magnetic field be continuous across a dielectric boundary. The requirements of Maxwell’s equations on the TE mode yield boundary conditions at an index step,

$$E_1 = E_2 \text{ and } \frac{dE_1}{dx} = \frac{dE_2}{dx} \quad (5.7)$$

Now there are several methods to solve the eigenmodes of the system.

Transfer matrix method

This method is significantly faster. The transfer matrix method represents each boundary using a 2x2 matrix. Assuming for the plane wave and from Maxwell’s equations, we can get a scalar wave equation for each i^{th} layer as follows:

$$\frac{\partial^2}{\partial x^2} \Psi_i + (k_0^2 \epsilon_i + \gamma^2) \varphi_i = 0 \quad (5.8)$$

Here φ_i is E_y for TE mode at i th layer and H_y for TM mode, γ is the modal propagation constant ($= \alpha + j\beta$), and k_0^2 is $\omega^2 \mu_0 \epsilon_0$. ‘ k_z ’ of Equation (5.7) is replaced by ‘ γ ’ in Equation (5.8).

Also, n^2 is replaced by ϵ_i , where ϵ is the permittivity of the material and $\epsilon = n^2$. For TE modes, the boundaries need to have ϕ_i and $\partial\phi_i/\partial x$ be continuous over different layers. If we consider the bounded mode and no loss or gain, which means ϵ_i and γ are real, for a three-layer waveguide, we can get a coupled equation in matrix form for the inner layer:

$$\begin{bmatrix} \phi_i \\ \frac{\partial \phi_i}{\partial x} \end{bmatrix} = \begin{bmatrix} \cos(h_i x) & \sin(h_i x) \\ -h_i \sin(h_i x) & h_i \cos(h_i x) \end{bmatrix} \begin{bmatrix} A_i \\ B_i \end{bmatrix} \quad (5.9)$$

where h_i is, $\sqrt{k_0^2 \epsilon_i - \gamma^2}$ and $|\gamma^2| > |k_0^2 \epsilon|$

The outer layer has the matrix form:

$$\begin{bmatrix} \phi_i \\ \frac{\partial \phi_i}{\partial x} \end{bmatrix} = [h_{1orL}] \begin{bmatrix} A_0 \\ A_0 \end{bmatrix} \quad (5.10)$$

where h_{1orL} is $\sqrt{\gamma^2 - k_0^2 \epsilon_{1orL}}$, h_1 is for 1st layer and negative ($-h_L$) for Lth layer, A_0 is A_1 for 1st layer and A_L for the last layer.

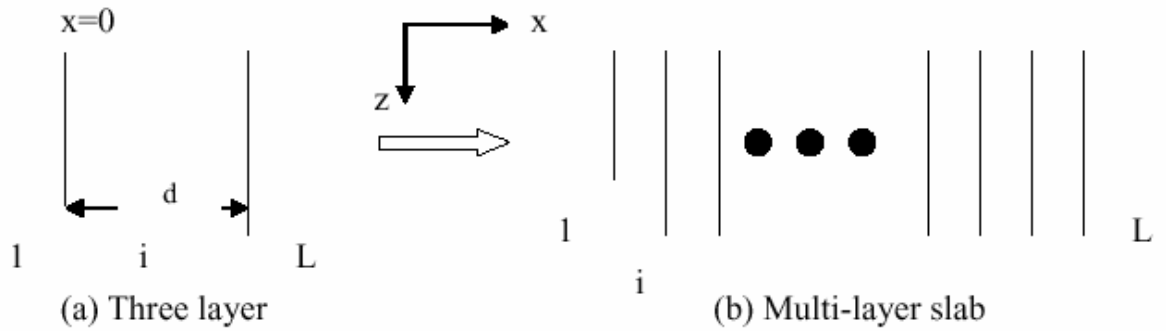


Fig 5.5 Slab Waveguide model, three layer (a) and multi-layer (b)

After matching the boundary conditions at each layer, we can get the following matrix form for the whole waveguide structure:

$$\begin{bmatrix} A_L \\ -h_L A_L \end{bmatrix} = T \begin{bmatrix} A_1 \\ A_1 h_1 \end{bmatrix} \quad (5.11)$$

$$\text{where, } T = \begin{bmatrix} T_{11} & T_{12} \\ T_{21} & T_{22} \end{bmatrix} = \begin{bmatrix} \cos(h_i d) & \frac{\sin(h_i d)}{h_i} \\ -h_i \sin(h_i d) \cos(h_i d) & \end{bmatrix}$$

From this equation, we can further get the characteristic equation as the following:

$$T_{21} + T_{22} h_1 + h_L (T_{11} + T_{12} h_1) = 0 \quad (5.12)$$

For multi-layer waveguides the overall T will become a multiplication of matrices T with different h and d in the matrix entries. However, the final characteristic equation will be the same as equation 5.12^{12 13}.

5.2.4 Calculation of near-field and far-field

Near field and far field are two of the important characteristics of an emitted optical field. Near field refers to the spatial intensity distribution of the emitted light near the waveguide end face. And the angular intensity distribution far from the end-face is known as the far field¹⁴. Far field is important in determining the coupling efficiency. Mathematically, the far-field pattern can be approximated by taking the Fourier transform of the near field intensity distribution. However, this method is not very accurate for a dielectric waveguide, though it is applicable for antennas. The idea of “obliquity factor” during the calculation of far field is applied in the present case¹⁵. Here, we can use a 3-layer waveguide to exemplify the calculation process. We can assume the mode fields to be independent of y direction (lateral direction) if the width of the waveguide is large in comparison to the thickness of the waveguide. Then the spatial distribution of the optical field is just associated with the transverse mode $\psi(x)$ that can be solved from the wave equation and boundary condition. For TE mode, the electric field can be written in the following form^{14 15}:

$$E_y = F\psi(x)e^{j(\alpha x - \beta z)} \quad (5.13)$$

And the modal distribution $\psi(x)$ that propagates along z direction has different form at each of the 3 layers:

$$\begin{aligned} \psi(x) &= Ce^{-px} & (x \geq d) \\ \psi(x) &= A\cos(qx) + B\sin(qx) & (d \geq x \geq 0) \\ \psi(x) &= De^{r(x+d)} & (x \leq 0) \end{aligned} \quad (5.14)$$

Where, p , q and r are wave-numbers at each layer and they satisfy the following conditions that relate with propagation constant at z direction:

$$\begin{aligned} p^2 + \varepsilon_1 k_0^2 &= \beta^2 \\ -q^2 + \varepsilon_2 k_0^2 &= \beta^2 \\ r^2 + \varepsilon_3 k_0^2 &= \beta^2 \end{aligned} \quad (5.15)$$

When combining with boundary condition, (i.e. tangential fields and their derivatives are continuous at the interface) and normalizing them, we can solve out the values of p , q , r and coefficients A , B , C , D . Thus $\psi(x)$ can be known.

Writing $\psi(x)$ in terms of plane waves by Fourier Transform theory,

$$\bar{\psi}(s) = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{+\infty} \psi(x) e^{jsx} dx \quad (5.16)$$

Where s is the propagation constant at x direction.

Then electric field is in the following form:

$$E_y(x) = \frac{1}{\sqrt{2\pi}} F \int_{-\infty}^{+\infty} \bar{\psi}(s) e^{j(\omega t - sx - \beta z)} ds \quad (5.17)$$

When the light radiates from the end-face of the waveguide, both reflection and transmission happen and the transmitted field is:

$$E_y(x)^{trans} = \tau F' \int_{-\infty}^{\infty} \bar{\psi}(s) e^{j(\omega t - sx - \beta z)} ds \quad (5.18)$$

Where τ is the transmission coefficient.

$$\tau = \frac{2\eta_{air}}{\eta_{air} + \eta_{waveguide}} = \frac{2\beta}{(k_0^2 - s^2)^{1/2} + \beta} \quad (5.19)$$

Considering the coordinate system shown in Figure 5.6, where the electromagnetic field is radiated into the air, we have the following relationships:

$$\begin{aligned} x &= r \sin\theta; & z &= r \cos\theta \\ s &= k_0 \sin\phi; & ds &= k_0 \cos\phi d\phi \end{aligned} \quad (5.20)$$

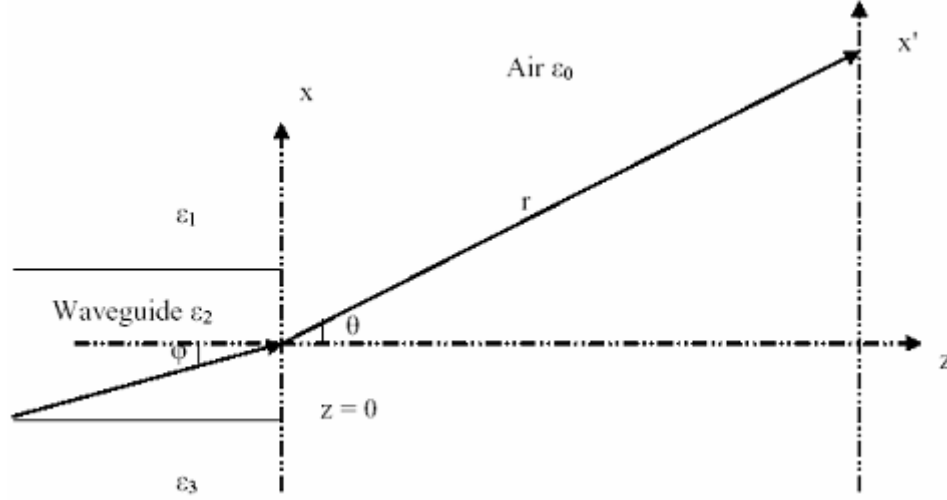


Fig 5.6 Schematic coordinate system of the far field of 3-layer waveguide

Then, the total field intensity at (r, θ) changes to: (ignoring the time term $e^{j\omega t}$)

$$E(r, \theta) = F' \int_{-\infty}^{\infty} \frac{2\beta}{k_0 \cos\phi + \beta} \bar{\psi}(k_0 \sin\phi) e^{-jk_0 r \cos(\theta - \phi)} k_0 \cos\phi d\phi \quad (5.21)$$

And then the “saddle point method”¹⁶ is used to approximate the value of $E(r, \theta)$ for large distance. The “saddle point method” is shown below:

$$\int_{-\infty}^{\infty} g(x) e^{kh(x)} dx \approx g(a) e^{kh(a)} \sqrt{\frac{-2\pi}{kh''(a)}} \quad (5.22)$$

Where ‘a’ is from,

$$h'(x)=0, \text{ in this case, } \sin(\theta - \phi)=0 \Rightarrow \theta = \phi \text{ and}$$

$$\sqrt{\frac{-2\pi}{kh''(a)}} = \sqrt{\frac{-2\pi}{-jk_0r(-\cos(\theta - \varphi))}} = \sqrt{\frac{2\pi}{k_0r}} \quad (5.23)$$

Therefore, the intensity of the far field is

$$I(\theta) = |E(r, \theta)|^2 = I_0 \frac{\cos^2 \theta}{|k_0 \cos \theta + \beta|^2} |\bar{\psi}(k_0 \sin \theta)|^2 \quad (5.24)$$

$\cos \theta$ is known as “Huygen obliquity factor” and $\cos^2 \theta$ the “intensity obliquity factor”.

In the present work, a coefficient $g(\theta)$ ^{xxvi} is used to combine all Huygen obliquity factor terms and the effective index of refraction calculated from previous steps contributes here to find the value of $g(\theta)$.

$$g(\theta) = 2 \cos \theta \frac{\frac{\beta}{k_0} + \sqrt{n_{eff}^2 - \sin^2 \theta}}{\cos \theta + \sqrt{n_{eff}^2 - \sin^2 \theta}} \quad (5.25)$$

Then, the far field intensity is calculated as:

$$I(\theta) = I_0 |\bar{\psi}(k_0 \sin \theta)|^2 g(\theta)^2 \quad (5.26)$$

5.2.5 Calculation of confinement factor

Physically the confinement factor or filling factor Γ accounts for the reduction in gain that occurs because of the spreading of the optical mode beyond the active region, it represents the fraction of the mode energy contained in the active region.

From the Maxwell's equations, we can get the time-independent wave equation^{12 13}:

$$\nabla^2 E + \epsilon k_0^2 E = 0 \quad (5.27)$$

Where ϵ is the complex dielectric constant:

$$\epsilon = \epsilon' + \epsilon'' \quad (5.28)$$

Where k_0 is the vacuum wave number:

$$k_0 = \omega / c = 2\pi / \lambda \quad (5.29)$$

Where E is the electric field:

$$E \cong \hat{e} \phi(y; x) \psi(x) \exp(i\beta z) \quad (5.30)$$

Here β is the propagation constant, e is the unit vector and z is the propagation direction.

By substituting Eq. (5.30) in Eq. (5.27), we obtain:

$$\frac{1}{\psi} \frac{\partial^2 \psi}{\partial x^2} + \frac{1}{\phi} \frac{\partial^2 \phi}{\partial y^2} + [\epsilon(x, y) k_0^2 - \beta^2] = 0 \quad (5.31)$$

From the above Equation (5.31), we can get the transverse field distribution $\phi(y; x)$ by solving:

$$\frac{\partial^2 \phi}{\partial y^2} + [\epsilon(x, y) k_0^2 - \beta_{eff}^2(x)] \phi = 0 \quad (5.32)$$

where $\beta_{eff}(x)$ is the effective propagation constant for a fixed value of x .

From the Equation (5.31), we can also get the lateral field distribution $\psi(x)$ by solving:

$$\frac{\partial^2 \psi}{\partial x^2} + [\beta_{eff}^2(x) - \beta^2] \psi = 0 \quad (5.33)$$

Confinement Factor of Transverse Modes:

The transverse modes depend on the thicknesses and refractive indices of the various layers used to fabricate a semiconductor lasers.

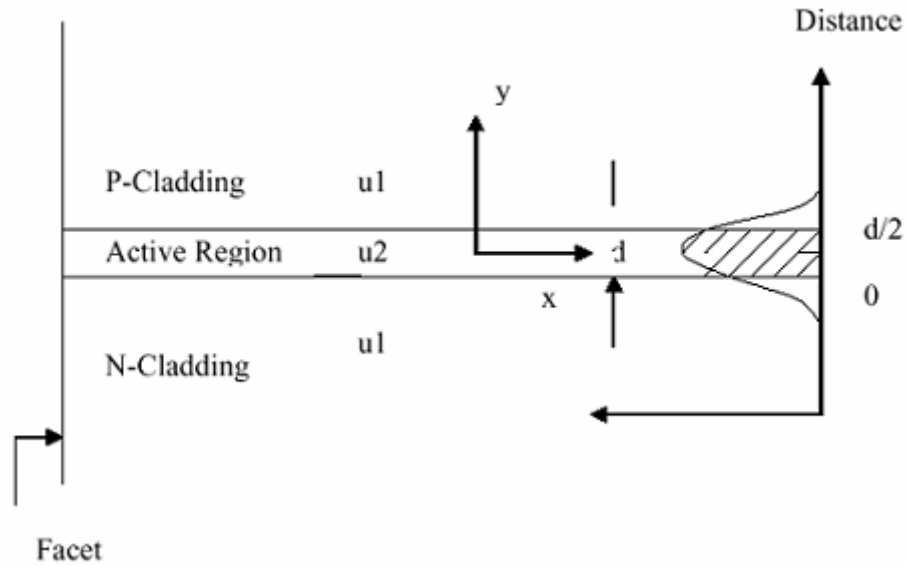


Fig 5.7 Three layer slab waveguide model

This Figure 5.7 shows the three-layer slab-waveguide model of a semiconductor laser with refractive indices such that $u_2 > u_1$. The intensity distribution of the fundamental waveguide mode is also shown. The hatched region represents the fraction of the mode within the active region.

Now, the transverse confinement factor can be given as:

$$\Gamma_T = \frac{\int_{-d/2}^{d/2} |\phi^2(y)| dy}{\int_{-\infty}^{+\infty} |\phi^2(y)| dy}.$$

Where, Γ_T is transverse confinement factor; $\phi(y)$ is the transverse field distribution, which can be solved from the Equation (5.32) and d is the active layer thickness.

Confinement Factor of Lateral Modes:

The lateral-mode behavior in semiconductor lasers is different depending on whether gain guiding or index guiding is used to confine the lateral modes.

$$\Gamma_L = \frac{\int_{-w/2}^{w/2} |\psi^2(x)| dy}{\int_{-\infty}^{+\infty} |\psi^2(x)| dy}$$

Where Γ_L is lateral confinement factor, $\psi(x)$ is lateral field distribution, can be solved from the Equation (5.33) and w is the central region width, i.e the ridge width.

Total confinement factor:

In the waveguide description,

$$\Gamma = \Gamma_T * \Gamma_L$$

5.3 Design of a Single Mode PbSe/PbSrSe Ridge Waveguide

The design of a ridge waveguide laser comprises of several steps. The first step starts with a basic waveguide design in the transverse direction. In the second step the height of the ridge is optimized to achieve the required lateral effective refractive index difference. Third, ridge width was designed to maintain single-lateral-mode and low loss, which is discussed. The effective index waveguide modeling software used in this work is *Waveguide BetaRelease 0.93*. The software handles complex modes in the presence of gain or loss. The mode characteristics, the phase propagation and exponential behavior can be determined from the transverse propagation constant plane. For bounded modes, the eigenvalue problem is well defined and all the roots are physically acceptable. For complex mode search, *Waveguide* uses an arbitrary branch cut in the square of modal propagation constant plane instead of a real axis line for proper mode search. The Muller-Traub method for complex plane is used in *Waveguide* to find a complex root. The program uses three kinds of convergence tests. If any one test is satisfied, the last iteration is accepted. Users can set up the maximum iteration number.

5.3.1 Design of a basic waveguide structure

The basic waveguide laser is composed of active layers, including quantum wells and barriers, and passive layers, including p-cladding layers, p-buffer layer, p-substrate, and n-cladding layer, n-cap layer. The basic design comprises optimization of the thickness of active layer for higher confinement and single transverse mode operation and optimization of the thickness of cladding layers for optimum confinement, lower modal intensity loss and narrow far-field divergence.

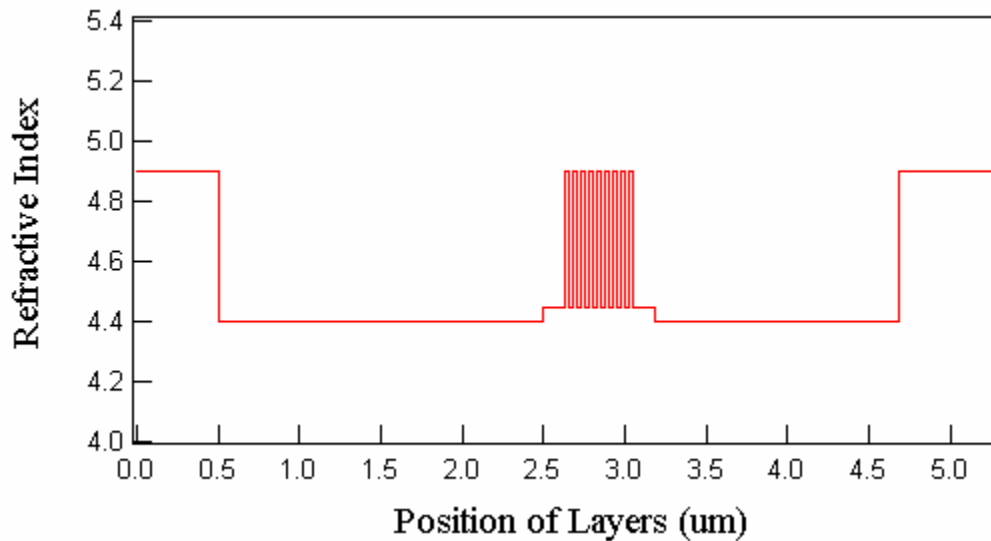


Fig 5.8 Refractive index profile of the layer structure in the transverse (vertical) direction

Figure 5.8 shows the refractive index profile of a basic waveguide laser in x direction.

Optimization of active region thickness

During the optimization of active layer thickness the fact that has been taken in consideration is maximizing the confinement factor but at the same time keeping the thickness in the limit which is needed for single transverse mode operation owing to the

condition $d < \frac{\lambda}{2\sqrt{n_1^2 - n_2^2}}$, where d is the thickness of active region. The detail of this

condition has already been described in the previous chapter. In our case the active region consists of multiple quantum well structure. PbSe (n= 4.9) well and Pb_{0.97}Sr_{0.03}Se (n= 4.45) barrier.

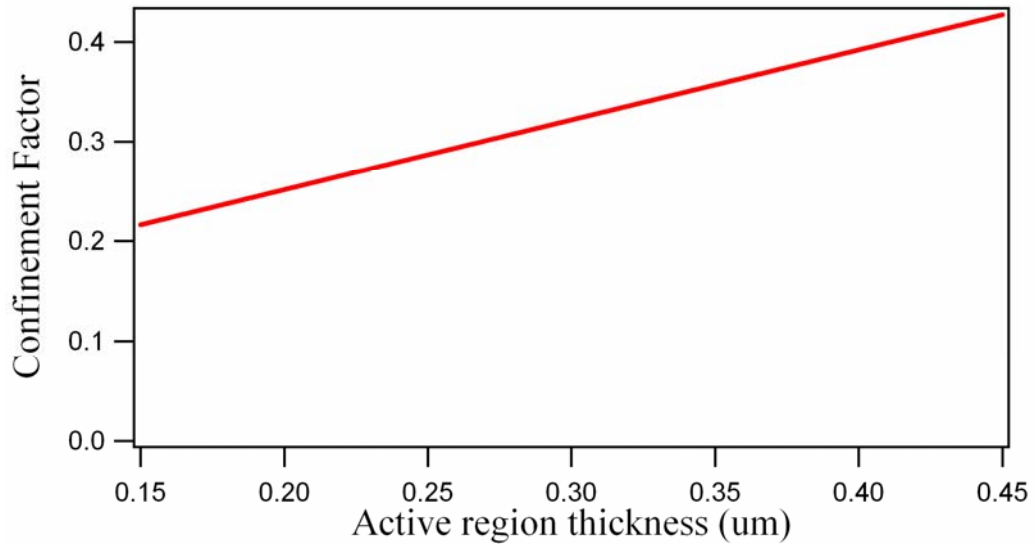


Fig 5.9 Plot of confinement factor of fundamental mode vs. active region thickness

Figure 5.9 shows the confinement factor of the fundamental mode in the active region as a function of active region thickness. It is clear that as the number of pairs of wells and barriers increases, confinement increases. So for our structure 9-pair of well and barrier was selected. For the single transverse mode operation the thickness should be less than 1.91 μm according to the formula mentioned above. So the thickness of a 9-pair active region is in perfect agreement with this, since our well and barrier have a thickness of 20 nm and 30 nm respectively. With 9-pairs the confinement factor in the active region is 0.43.

Optimization of cladding layer thickness

After optimizing the thickness of active region there was need to optimize the thickness of n and p cladding regions by plotting confinement factor, far-field divergence and modal loss as a function of thickness of these layers. In our case, $\text{Pb}_{0.96}\text{Sr}_{0.04}\text{Se}$ (absorption at 4000cm^{-1}) having refractive index 4.4 was chosen as the cladding layer. For the doped layers, doping concentration was $2 \times 10^{18} \text{ cm}^{-3}$.

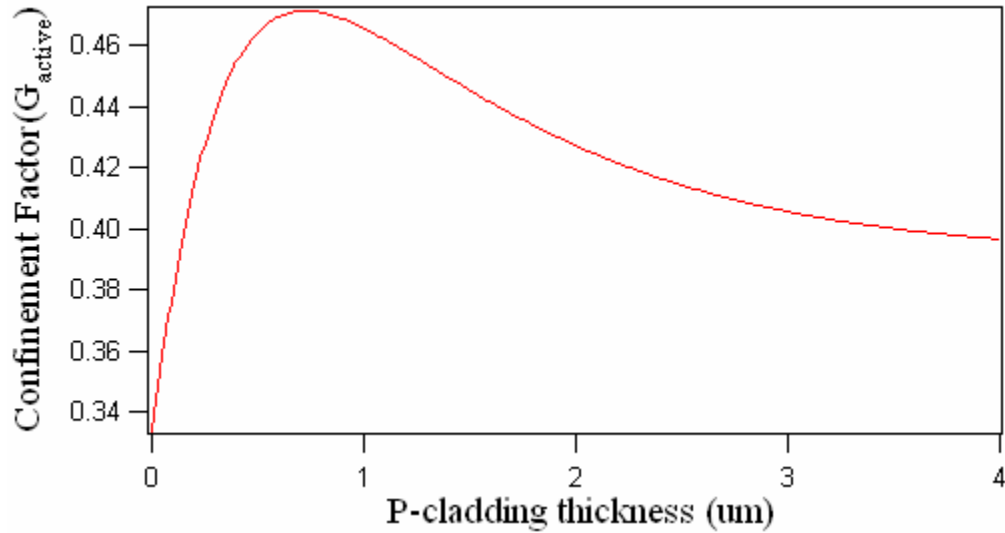


Fig 5.10 Plot of confinement factor of fundamental mode vs p-cladding thickness

From Figure 5.10, it can be seen that as the thickness of p-cladding region increases initially the confinement factor increases but then as thickness increases after $1\mu\text{m}$ it decreases little bit and becomes almost constant afterwards. The outcome is quite obvious. Initially, when the thickness increases, more field gets confined because of refractive index difference, but after a certain thickness a considerable amount of field propagates inside the cladding region itself and gets some confinement there, as a result,

confinement in the active region decreases a little bit. And when the thickness approaches a large value the field almost dies out in that region so the confinement factor becomes almost constant.

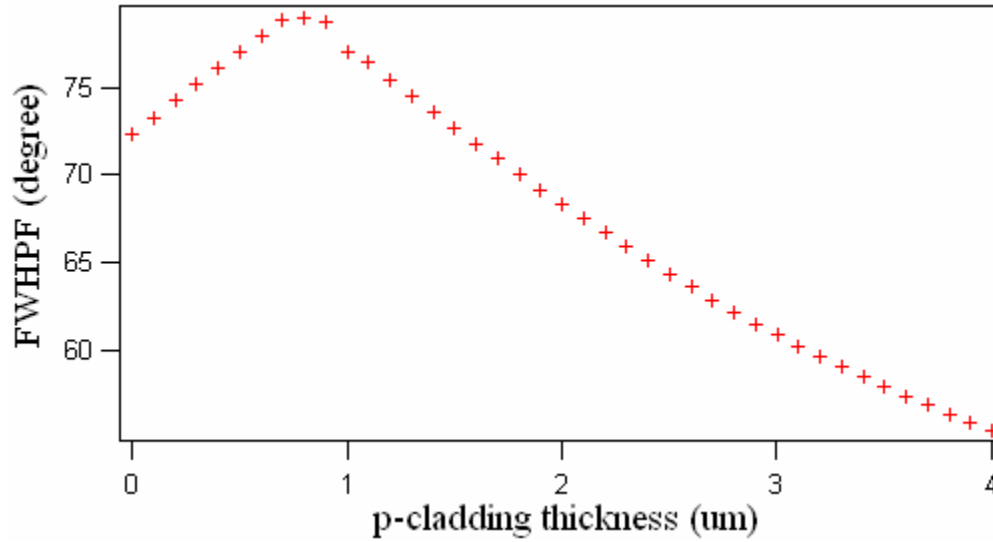


Fig 5.11 Farfield divergence (FullWidthHalfPower of farfield) vs p-cladding thickness

Now, as confinement of near field increases, divergence of farfield also increases. This effect is reflected in Figure 5.11. So we have to choose the thickness of cladding layer at which the confinement is also good enough and farfield divergence also remains at an acceptable value.

Figure 5.12 on the next page shows the plot of modal intensity loss as a function of p-cladding layer thickness. The modal loss was calculated from the formula,

$$\text{Modal Intensity loss} = \text{Im}(n_{\text{eff}}) \times (4\pi / \lambda) \times 10^4 / \text{cm}$$

During modal analysis we have also entered the free carrier loss values of the doped layers. Free carrier loss ($\sigma_F N$) was calculated from the equation below¹⁷:

$$\sigma_F N = \frac{q^3 N}{\sqrt{\epsilon_\infty} c \epsilon_0 \mu m_c^{*2} \omega^2}$$

Here, N is the carrier density, μ is the mobility, m_c is the conductivity effective mass.

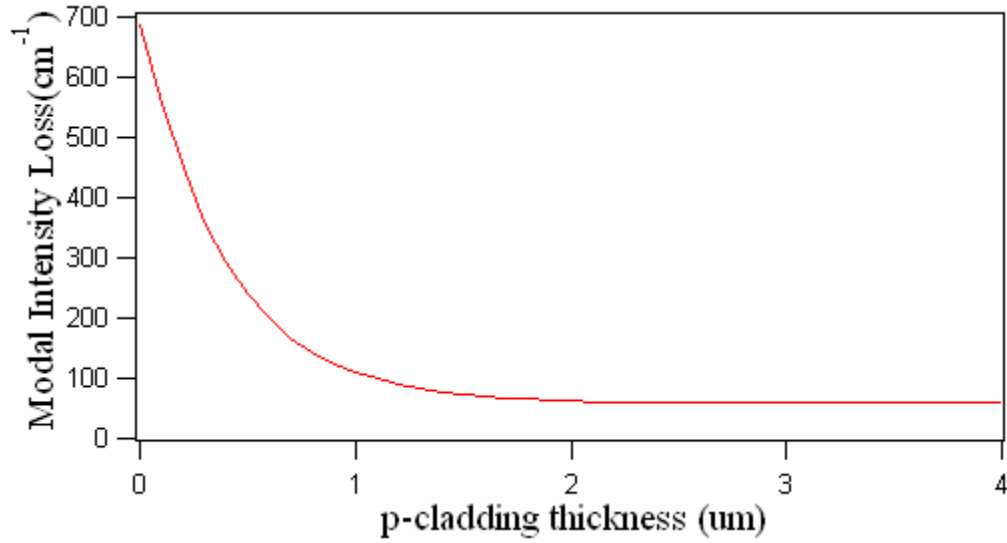
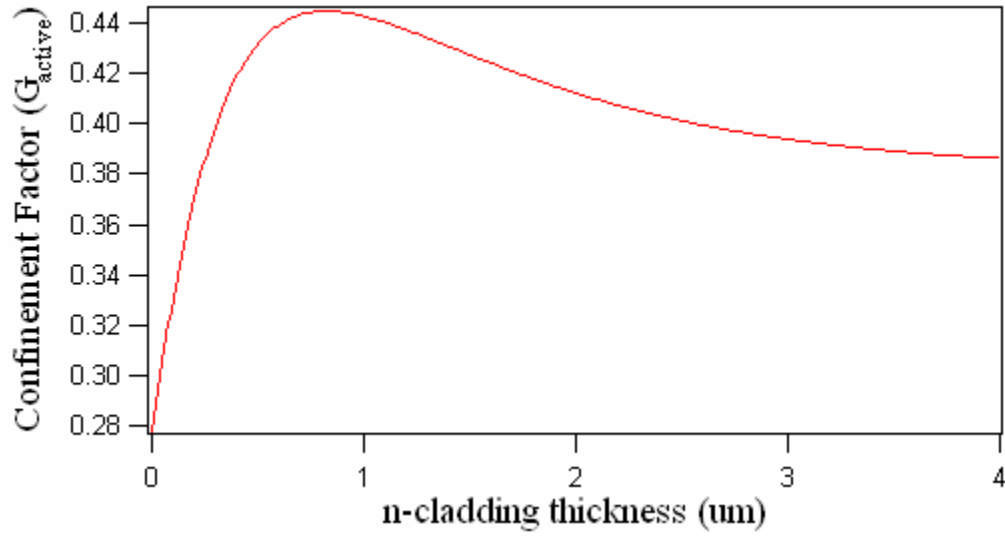


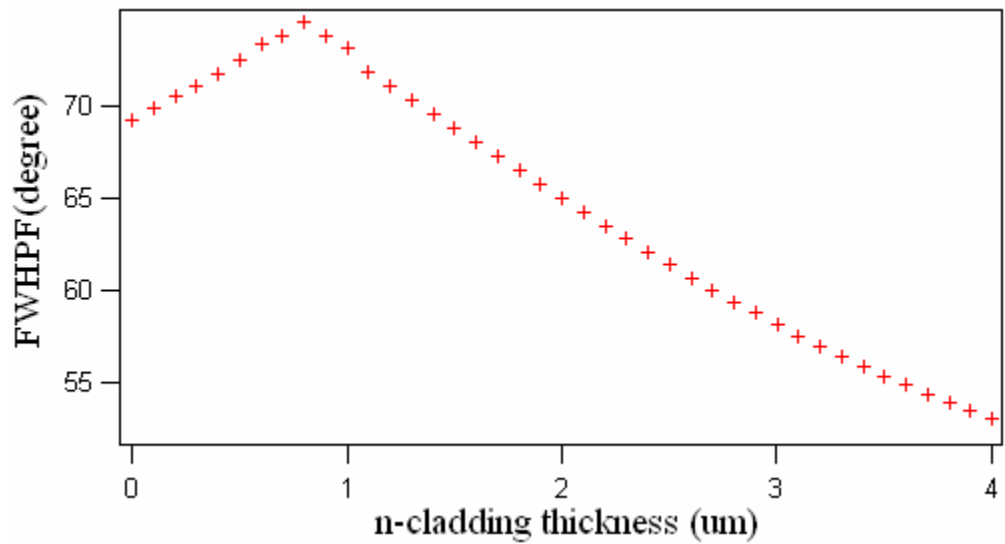
Fig 5.12 Plot of modal intensity loss of fundamental mode vs p-cladding thickness

In the absence of p-cladding thickness loss is very high because in that case most of the field penetrates into the lossy p-type PbSe substrate. Therefore, as the thickness increases, intensity loss decreases. However, after a certain thickness the field almost dies out in the cladding layer, hence modal loss becomes almost constant.

So, by analyzing all the above curves we choose p-cladding thickness to be 2 μm . At this thickness the confinement is 0.435 and far-field divergence is 66.5°.



(a)



(b)

Fig 5.13 Plot of confinement factor of fundamental mode vs. n-cladding thickness (a); far-field divergence (Full Width Half Power of far-field) vs n-cladding thickness (b).

From Figure 5.13, we can see that as the thickness of n-cladding region increases, initially the confinement factor increases but then as thickness increases after 1 μm it decreases little bit and becomes almost constant afterwards. Therefore, same explanation is valid here as well. Hence, we have to choose the thickness of n-cladding layer at which

the confinement is good enough and divergence of far-field also remain at an acceptable value.

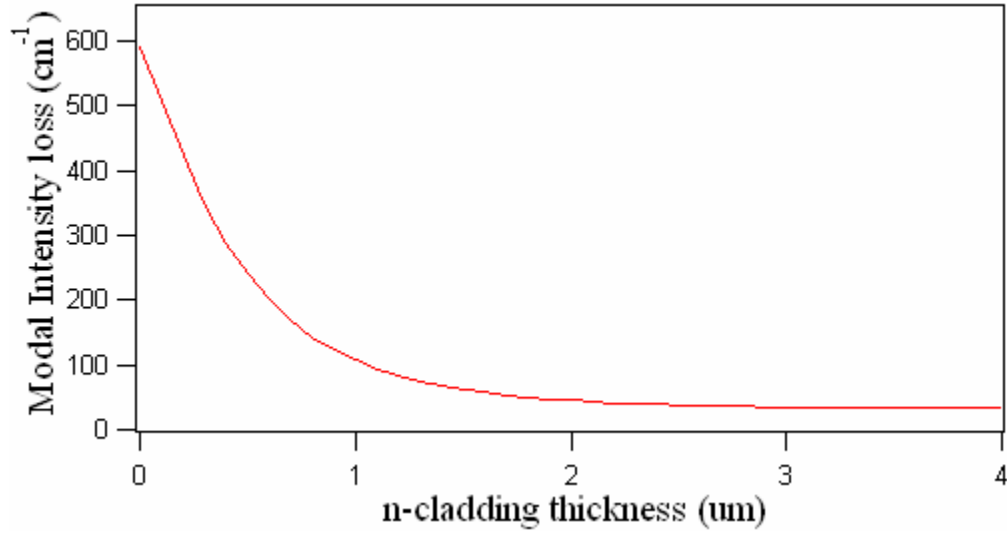
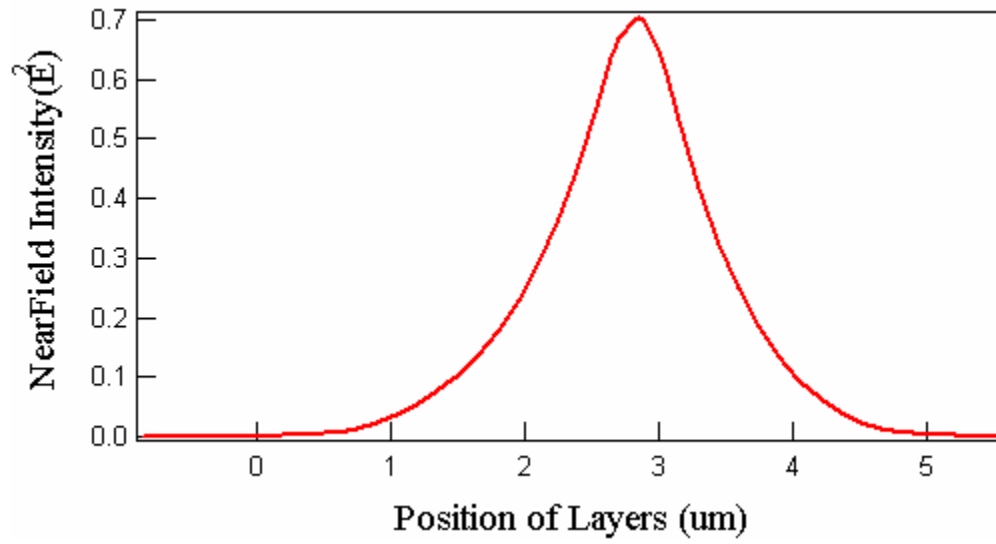


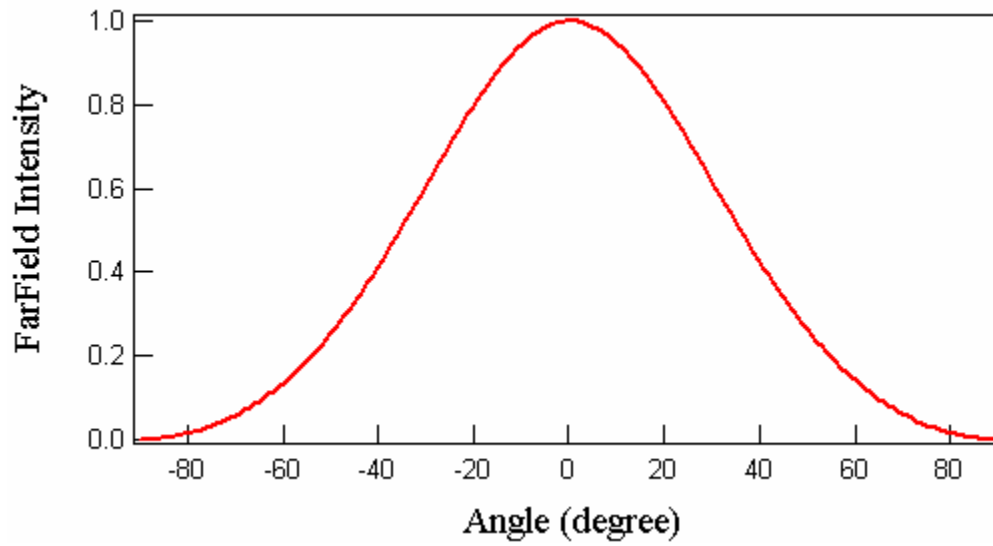
Fig 5.14 Plot of modal intensity loss of fundamental mode vs. n-cladding thickness

Figure 5.14 shows the plot of modal intensity loss as a function of n-cladding layer thickness. In the absence of p-cladding thickness loss is very high because in that case most of the field penetrates into the lossy and absorptive n-cap PbSe layer. Therefore, as the thickness increases intensity loss decreases, however after a certain thickness the field almost dies out in the cladding layer, hence modal loss becomes almost constant.

By analyzing all the above curves we chose n-cladding thickness to be 1.5 μm . At this thickness confinement is 0.425 and far-field divergence is 67° .



(a)



(b)

Fig 5.15 Plot of Near-field intensity (a); Far field intensity (b) of the fundamental mode in the transverse direction.

Figure 5.15 shows the near-field intensity and far-field intensity of the fundamental mode in transverse direction from the final optimized structure in the transverse or vertical direction.

The schematic of final structure in the transverse direction is shown in Figure 5.16.

n+-PbSe cap 0.1um
n-PbSrSe cladding 1.5um
MQW
p-PbSrSe cladding 2um
p-PbSe Buffer 0.5 um
PbSe Substrate

Fig 5.16 Schematic of final structure in transverse direction.

5.3.2 Design of ridge width and height for single lateral mode operation

As we know, by solving Maxwell's equations and matching boundary conditions of the three-layer dielectric slab waveguide, the graphical solutions can be found for Equation 5.34a, 5.34b, and 5.34c for TE polarization.

$$\alpha d/2 = (\mu/\mu_1)(k_x d/2) \tan(k_x d/2) \quad \text{for even modes (Equation 5.34a)}$$

$$\alpha d/2 = -(\mu/\mu_1)(k_x d/2) \cot(k_x d/2) \quad \text{for odd modes (Equation 5.34b)}$$

$$(\alpha d/2)^2 + (k_x d/2)^2 = \omega^2 (\mu_1 \epsilon_1 - \mu \epsilon)(d/2)^2 \quad \text{(Equation 5.34c)}$$

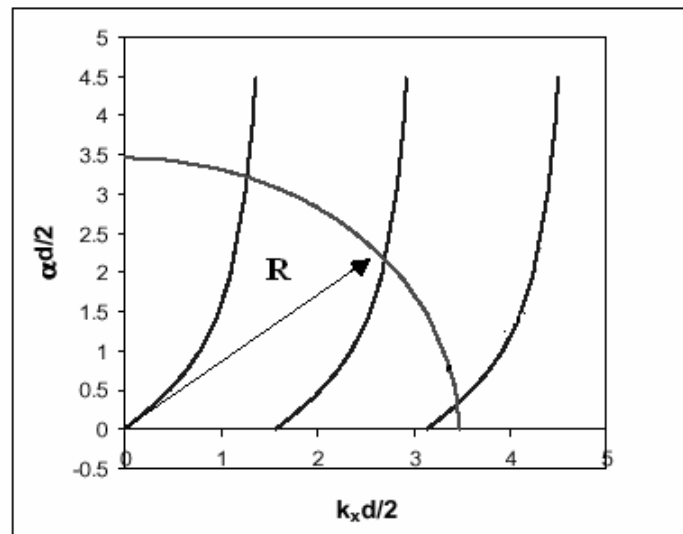
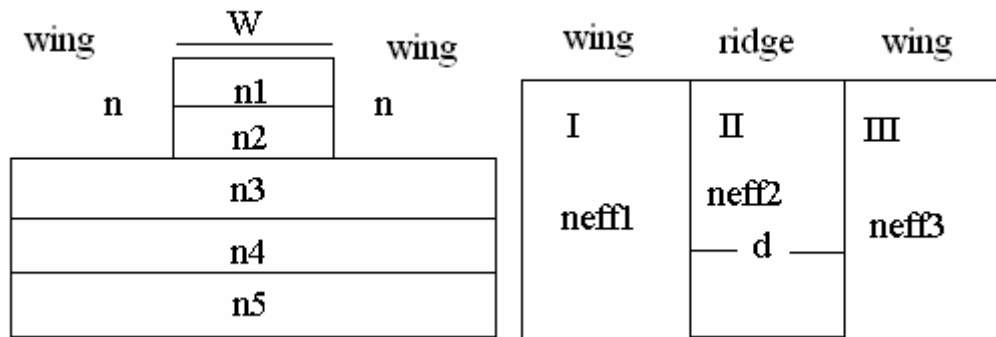


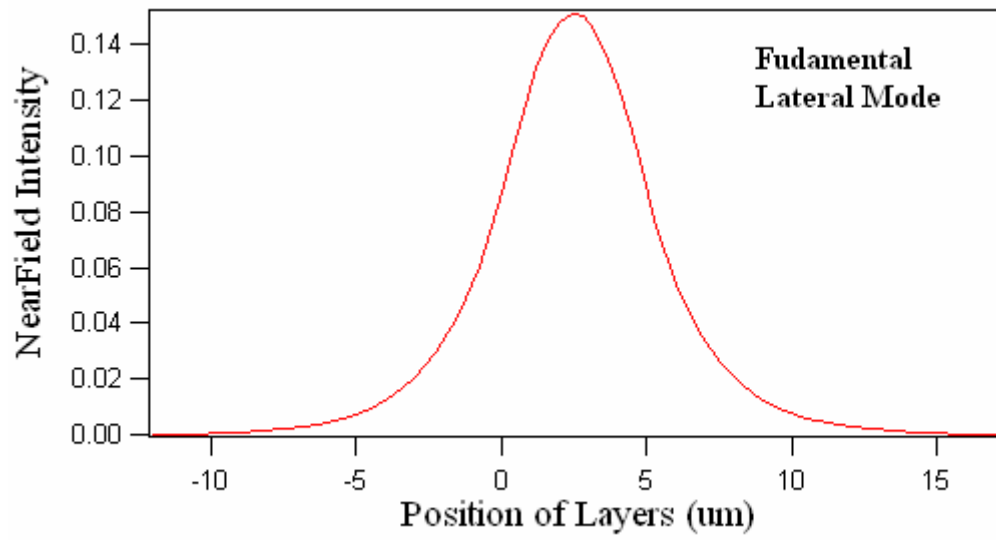
Fig 5.17 Graphical solution of Equations 5.34 - a, b, c for a three-layer dielectric slab waveguide.

In the above equations, α is the decay constant in the cladding layer I and III, this is shown in Figure 5.17. k_x is the x component of k in the core layer II, d is the thickness of the core layer II, μ and ε are the permeability and permittivity of layer I and III, and μ_1 and ε_1 are the permeability and permittivity of layer II, ω is the angular momentum of the modes in layer II. As shown in figure 23, the number of modes allowed to propagate in the three-layer dielectric slab waveguide is determined by the radius of the quarter circle R, which is $R = (k_x d / 2) (n_1^2 - n^2)^{1/2} < \pi / 2$. Therefore, the cutoff condition for the 1st TE mode TE₁ is derived as $d < \lambda / 2 (n_1^2 - n^2)^{1/2}$. In other words, only one mode, the TE₀ mode can propagate for the 3-layer dielectric waveguide with the core thickness (ridge width) that meets the condition set by above formula. Here n_1 and n are the effective refractive indices of ridge and wing (or side) region respectively.

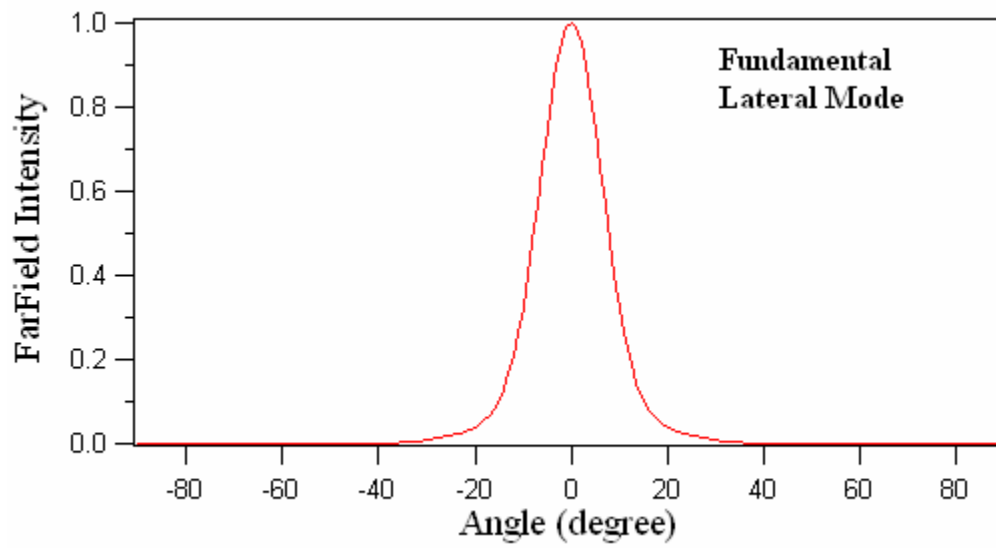
Therefore, after calculating the basic waveguide we found when the etch depth or ridge height is 1 μm the effective refractive index of wing region is 4.40.

Effective index in the ridge region is 4.42. Hence, if we put these values in place of n and n_1 in the formula $d < \lambda / 2 (n_1^2 - n^2)^{1/2}$, the inequality equation for d becomes $d < 5.85$. Then, putting $d=5$ if we simulate the structure again we find only one mode having modal index 4.42 is propagating in the structure.

Near-field and Far-Field intensity of this fundamental mode in lateral direction are shown in Figure 5.18.



(a)



(b)

Fig 5.18 Plot of Near-field intensity (a); Far-field intensity (b) of the fundamental mode in the lateral direction.

5.3.3 Design of waveguide for selective modal loss

In this section, design of a waveguide structure to produce selective losses for higher order modes will be discussed. A ridge structure has been designed that will allow selective losses for the higher order modes with respect to the fundamental mode by changing the thickness of the deposited insulating layer outside the ridge and coupling the higher order optical field to metal layer and thus allowing only the fundamental mode to propagate within the structure. Introduction of selective losses to the higher order modes has already been applied through partial implantation outside ridge area in III-V lasers by several researchers^{18 19}. In our case, if we deposit a layer of Al after the BaF₂ insulating layer outside the ridge, in that case the first order mode suffers a huge loss since its modal index is less than the refractive index of Al at our emission wavelength. Figure 5.19 represents a schematic of this structure and Figure 5.20 shows the theoretical simulation results indicating the comparative losses for the first order and fundamental mode.

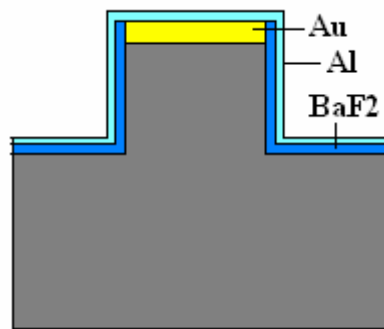
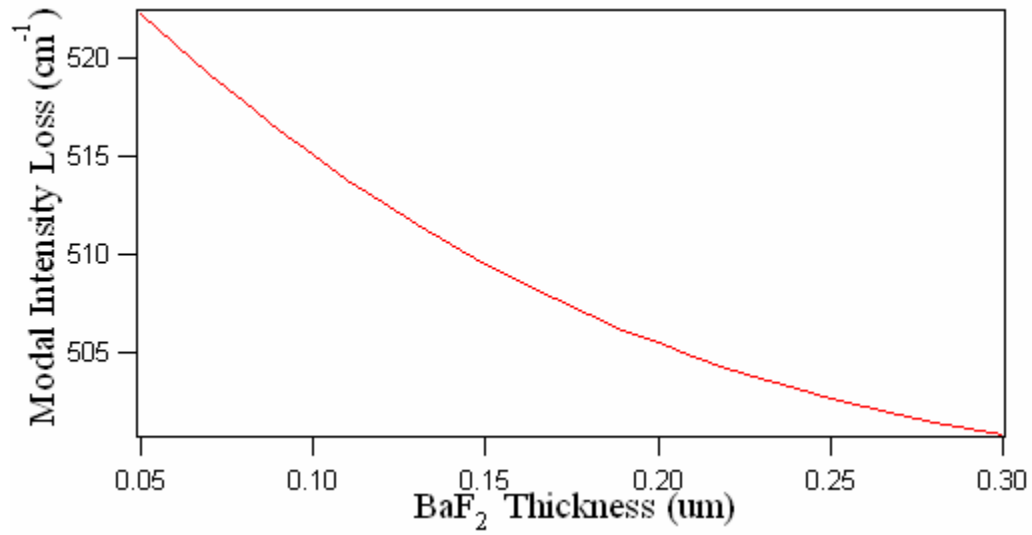
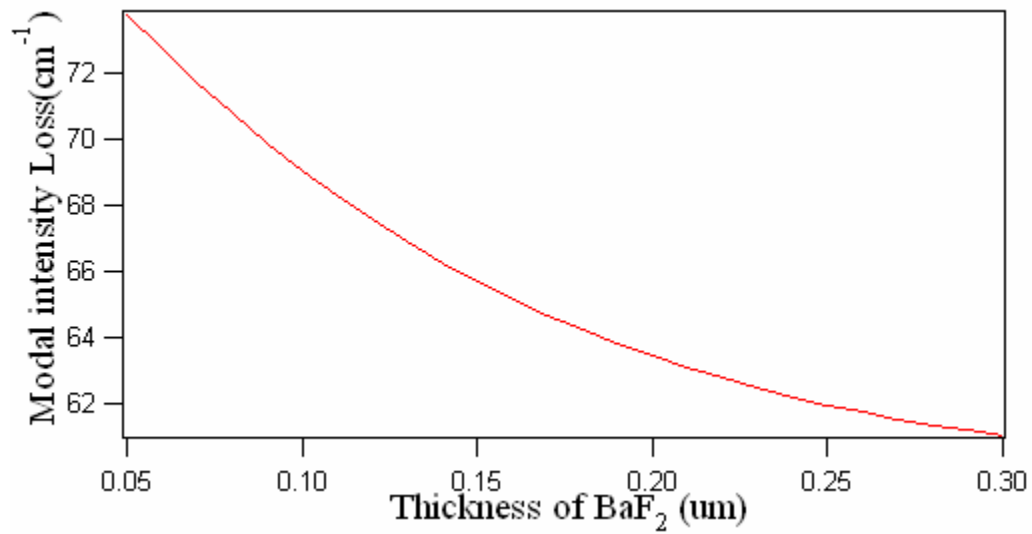


Fig 5.19 Schematic of selective modal loss ridge waveguide structure.



(a)



(b)

Fig 5.20 Modal Intensity loss of 1st order mode (a) and fundamental mode (b), with the change of thickness of insulating BaF₂ layer.

If the thickness of BaF₂ has been chosen to be 0.20 μm - 0.23 μm, in that case the 1st order mode experiences a huge loss; whereas the fundamental mode has a negligible loss at this thickness.

5.4 Fabrication Process Development of Ridge Waveguide Laser

Fabrication procedure has been developed to prove the feasibility of ridge waveguide structure. The schematic of the process flow is shown in Figure 5.21.

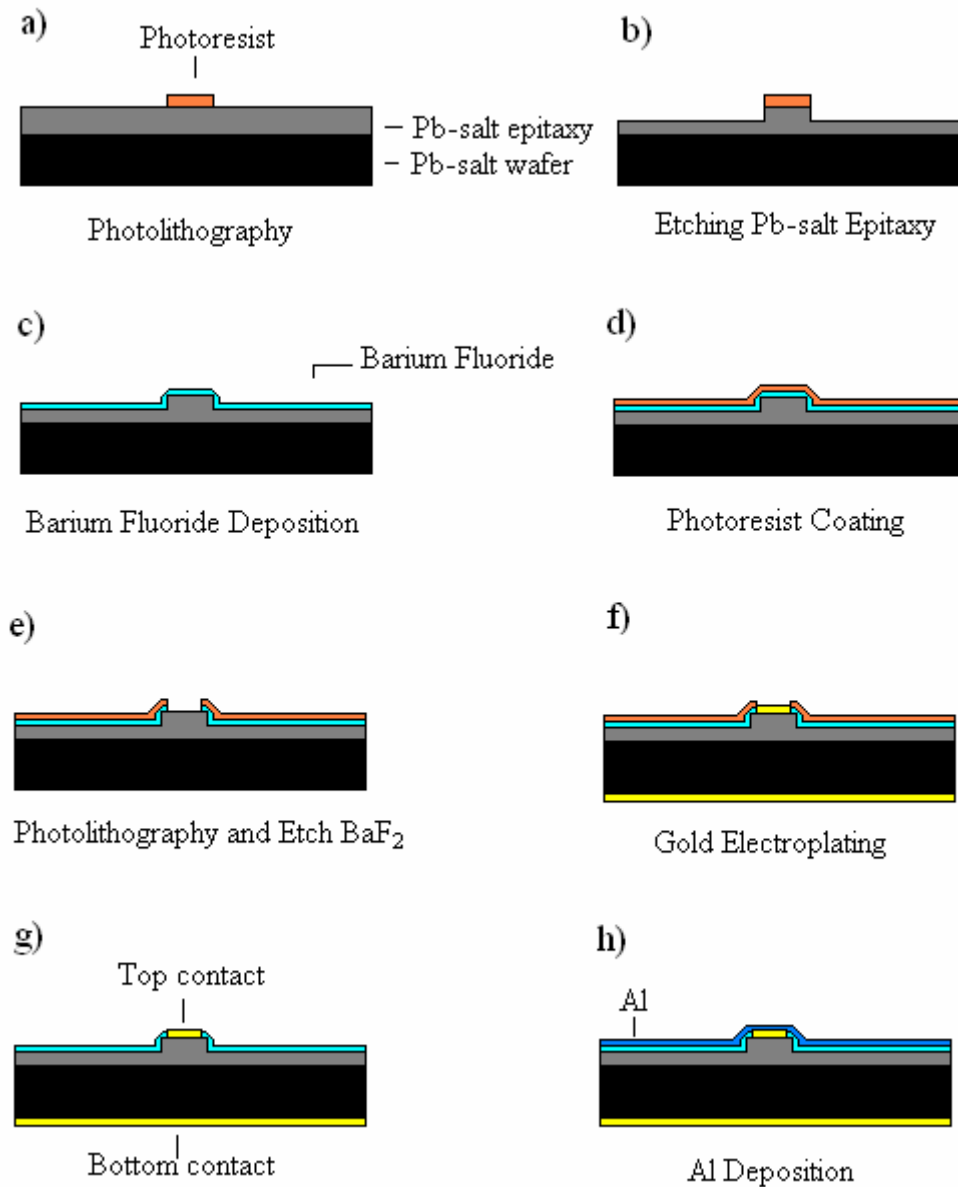


Fig 5.21 Schematic of the process flow for the fabrication of single mode ridge waveguide laser

The first step in the fabrication process started with photolithography for ridge formation. During the process development, masks having the exact dimension as per design were not available. Hence, masks having higher or lower dimension had been used to check the feasibility of the process. During the first photolithography step, stripe patterns were made perpendicular to the cleaving direction of epitaxial layer as in Figure 5.21a. Etching of the epitaxial layer was then performed with the protection of resist to form a ridge as shown in Figure 5.21b.

After the formation of the ridge, photoresist was completely removed from the sample and the sample was transferred into the MBE chamber for the deposition of a thin layer of BaF_2 , followed by a thorough cleaning in organic solvents and DI water. This step is shown in Figure 5.21c.

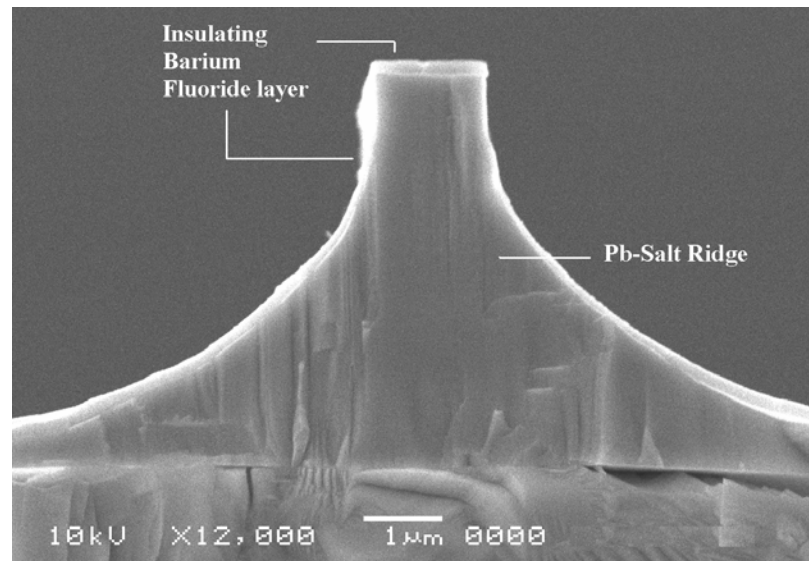


Fig 5.22 SEM cross-sectional image of a Pb-salt ridge after deposition of BaF_2 on the whole sample.

Figure 5.22 shows the cross-sectional image of a ridge after the deposition of BaF_2 .

The next step in the fabrication process is etching BaF_2 only from the top of the ridge for the formation of the metal contact. This step needs a photolithography that opens window on photoresist on top of the ridge so that BaF_2 gets etched only from that window. Now if the ridge width is only of the order of 2-3 μm , then opening a window smaller than that needs sub micron photolithographic system, which was not available to us. Therefore, for this step a technique called flood exposure was used. This is the most challenging part of this fabrication in the whole process. And it will be visible from next couple of SEM images shown below. In the flood exposure technique due to the variation of thickness of the photoresist on top and bottom of the ridge, a certain exposure helps to remove photoresist only from top of the ridge. However, a strict process control is needed on the exposure time, which is again a function of ridge height and a change in the ridge height changes the exposure time needed to completely remove photoresist from ridge top.

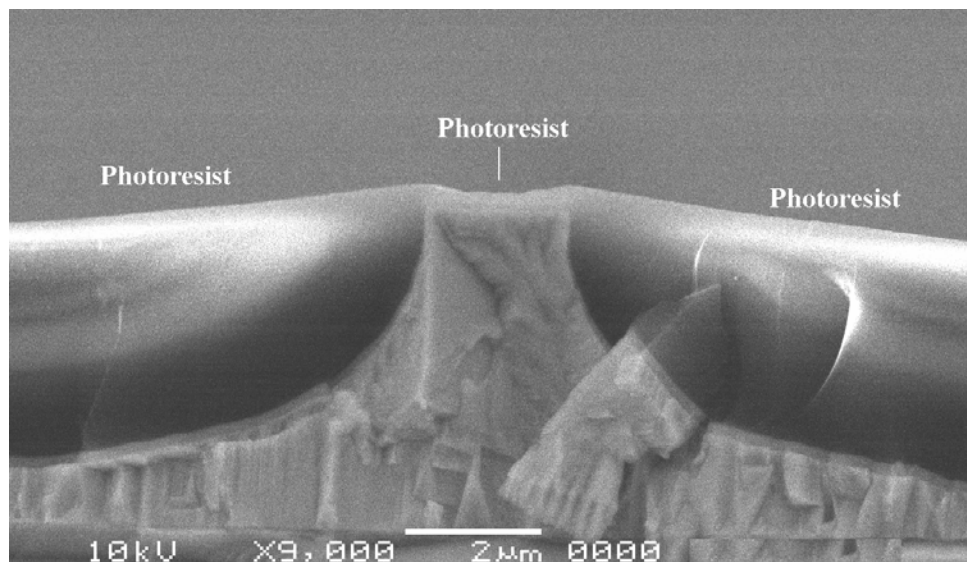


Fig 5.23 SEM cross-sectional image of a Pb-salt ridge after 2nd photolithography step to remove photoresist from ridge top. Photoresist had not been removed fully due to insufficient exposure.

It can be seen in above shown SEM image that the exposure was insufficient to remove completely the resist from ridge top. However, previously with the same exposure complete removal was achieved for a ridge that was $0.4\text{ }\mu\text{m}$ taller than this one. Figure 5.24 shows the cross-sectional SEM image, where, due longer exposure time, the removal was more than what was needed.

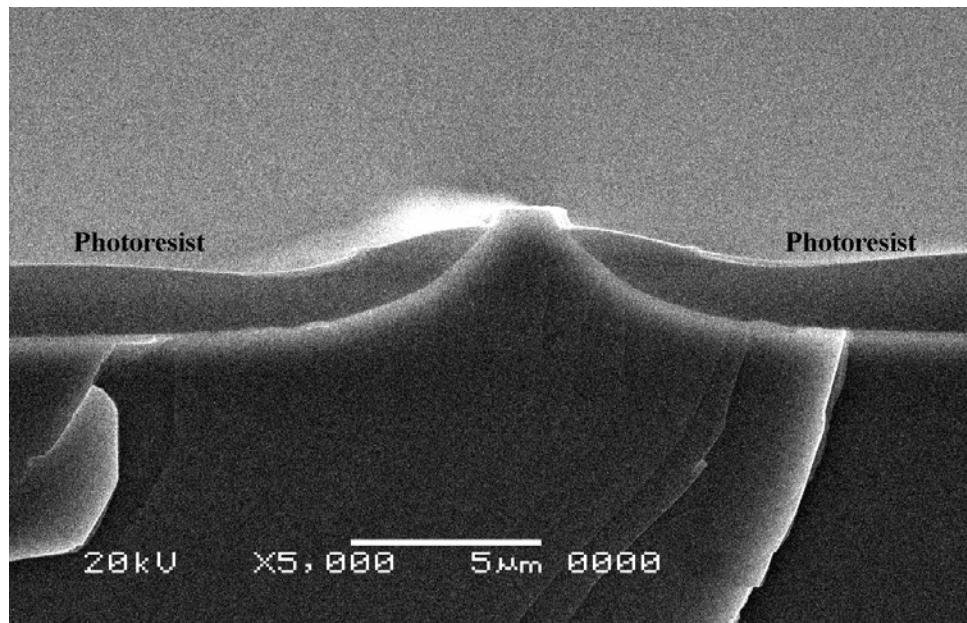


Fig 5.24 SEM cross-sectional image of a Pb-salt ridge after 2nd photolithography step to remove photoresist from ridge top. Photoresist had been removed also from side due to over exposure.

The problem with the profile shown in Figure 5.24 is that during BaF_2 etching the insulating material will also be removed from the side of ridge and therefore during gold electroplating, gold will be deposited on the side of the ridge thus changing the dimension of current spreading region and current conduction profile. Figure 5.25 shows the SEM cross-sectional image of a ridge with proper removal of photoresist from the top of the ridge. Experimentally, it was found that the ridge height tolerance level of flood exposure is approximately $0.2\text{-}0.3\text{ }\mu\text{m}$.

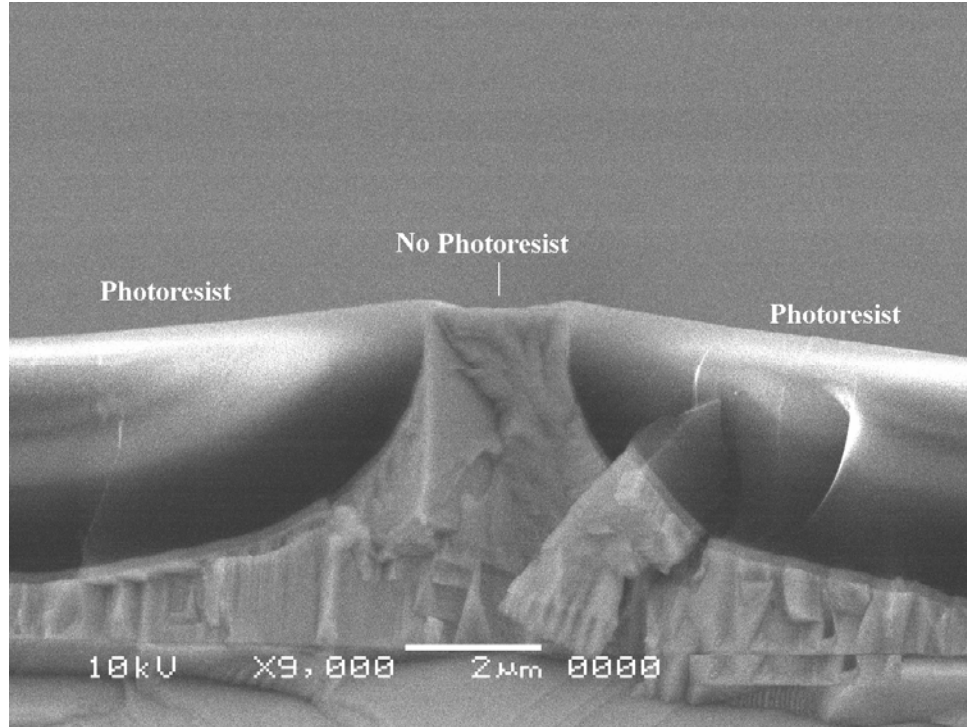


Fig 5.25 SEM cross-sectional image of a Pb-salt ridge after 2nd photolithography step to remove photoresist from ridge top. Photoresist had been removed completely from ridge top.

In the next step, BaF_2 was etched from ridge top and gold was electroplated as is shown in schematically in Figure 5.21f for the formation of top and bottom contact. Plating nearly $1\text{ }\mu\text{m}$ thick gold on a $2\text{ }\mu\text{m}$ wide ridge was also quite challenging. Due to fluidic friction at the sharp edges of the ridge, gold was deposited on photoresist on the ridge side. However, this does not create a major problem since the ridge side already remains insulated by the BaF_2 layer. The SEM cross sectional image of the ridge after gold plating and contact formation is shown in Figure 5.26 on the next page. All the cross-sectional images shown in this section so far are not cleaved but cut using diamond scriber.

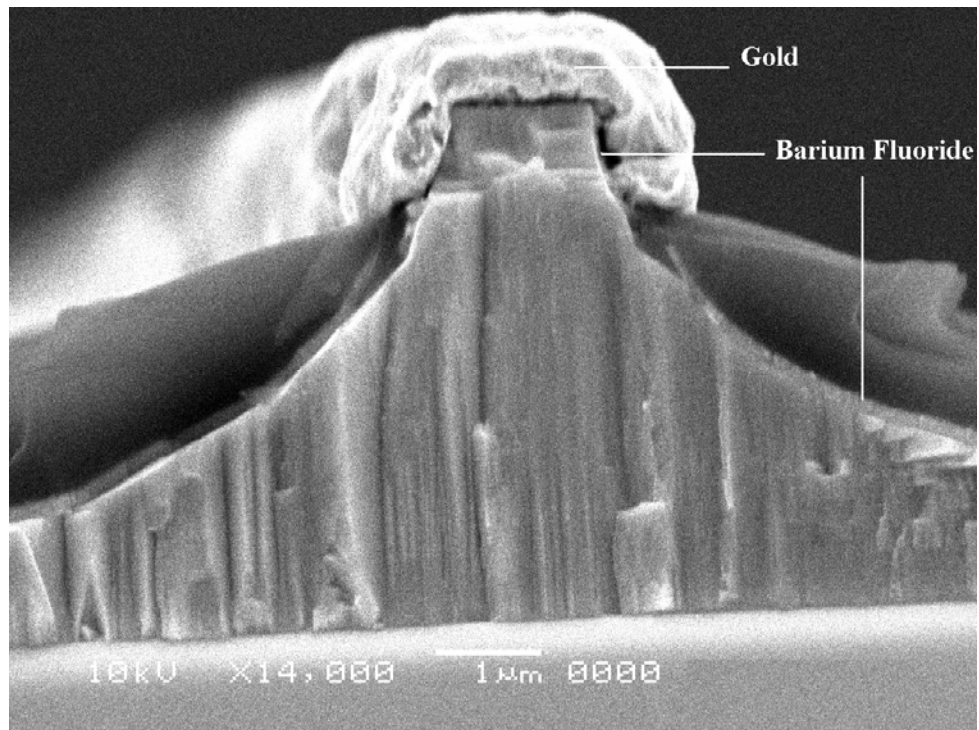
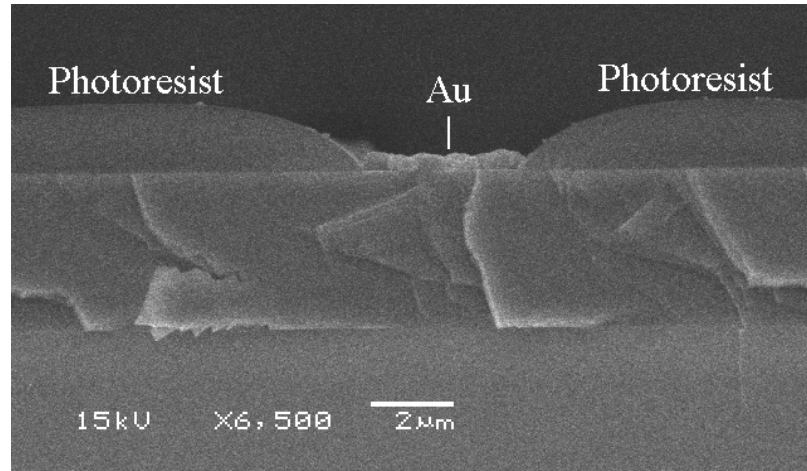
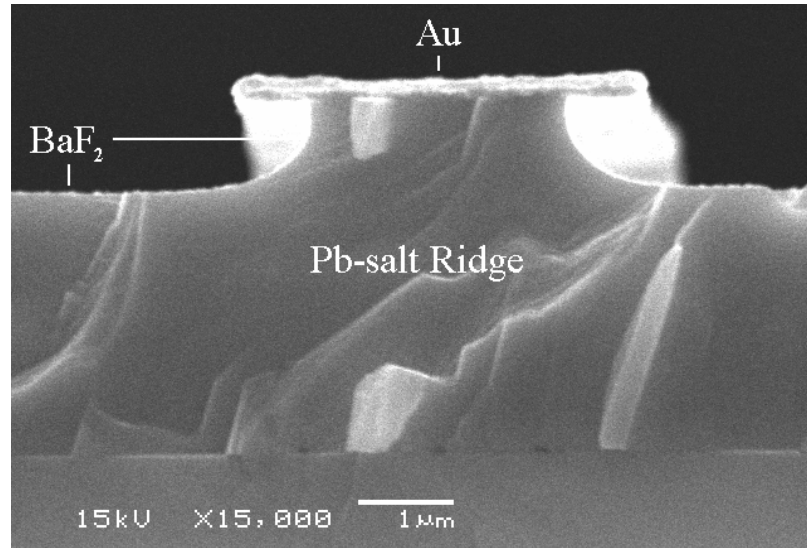


Fig 5.26 SEM cross-sectional image of a Pb-salt ridge after gold electroplating for contact formation.

The SEM images below show a ridge structure where the plated gold for contact has been used as a mask, to form a 3 μm wide 1 μm high ridge waveguide. All other fabrication processes remain the same as is shown schematically in Figure 5.21.



(a)



(b)

Fig 5.27 SEM cross-sectional image of a Pb-salt epitaxial layer after plating a 5 μm wide gold stripe, followed by a photolithography (a); Cleaved cross-section of ridge showing BaF_2 on ridge side. BaF_2 was etched from top of gold contact (b). Ridge was formed by etching the Pb-salt layer from all other areas of the sample except the area with a protection of gold.

5.5 Conclusion

The design and a fabrication process have been developed for the formation of a single mode ridge waveguide laser. The entire process is generic and can be applied to Pb-salt epitaxial layers grown on any substrate. Facets for the formation of Fabry Perot cavity can be formed either by cleaving or dry etching, as is discussed in the previous chapters.

After developing the entire process we were not able to grow a Pb-salt laser sample according to the design because of the multiple MBE downtimes in our lab, during my period of stay at OU. At the time of this writing, the MBE is up and running again. Once a laser structure is grown, a ridge waveguide laser with a single lateral mode could be expected.

5.6 References

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Chapter 6

Lead Chalcogenide on In-built Metal Heat Sink

6.1 Motivation

Currently, the main challenge for lead salt diode lasers is increasing maximum operation temperature in continuous wave mode. For high resolution spectroscopy continuous wave operation is always preferred over pulsed mode operation. However, highest operation temperature reported so far from a lead salt laser operating in CW mode and emitting around $3.5\mu\text{m}$ is 223K ¹. Longer wavelength devices have even lower operation temperatures, requiring the use of cryogenic cooling system. Thus any technique that will increase the operation temperature of the device is greatly needed.

In this chapter, a method of fabricating Pb-salt semiconductor light-emitting devices that may lead to higher operation temperature will be described. In this method, a metallic base was grown in contact with the semiconductor epitaxial layer, which serves as a supporting structure after the substrate removal and a heat sink that improves the heat dissipation from active region. To obtain the two parallel facets for the laser structures the semiconductor epitaxial layer can either be cleaved or dry etched. The cleaving scheme developed thereon allows the cleaving of the epitaxial layer in the natural cleaving plane after the removal of the growth substrate. The objective is a semiconductor laser that can be cooled using a thermoelectric cooler.

An efficient removal of heat from active region can increase the operation temperature but low thermal conductivity lead salt substrate hinders this process. Removal of growth substrate and transfer of epitaxial layer to carrier wafers have already been developed for various material systems, including IV-VI materials as well^{2 3 4 5}. An epitaxial transfer of IV-VI epitaxy on GaAs carrier wafer that was developed in our lab has already been discussed in chapter-3. However, all of these methods are based on

intermetallic bonding, that needs strict control of process conditions and above all, success of the process depends on several process parameters; if any one of them is not met, the process has a high chance of failure. Therefore, a method of growing a metal base or carrier substrate on epitaxial layer was developed followed by growth substrate removal.

By replacing the growth substrate completely with a metallic base or carrier substrate, heat dissipation increases several times. Considering the work of Kevin R. Lewelling and Patrick J. McCann⁶, thermoelectrically cooled IV–VI semiconductor diode laser can be obtained if the device is transferred from its thermally resistive IV-VI substrate to a more thermally conductive copper substrate. It was reported that the thermal models revealed a 63 K decrease in active region temperature under normal operating condition when copper replaces a conventional PbTe substrate.

Therefore, in the present method, copper layer of thickness 100 – 500 μm , forming a base was grown by an electroplating method in contact with the epitaxial layer. The Pb-salt epitaxial layer was grown by MBE on a BaF_2 substrate followed by growth of a BaF_2 sacrificial layer, which helps in removing the growth substrate by getting dissolved in water.

6.2 Optically and Electrically Pumped Device Structure

Figure 6.1 shows the fabrication steps for an optically-pumped laser with an as-grown metallic substrate deposited sequentially.

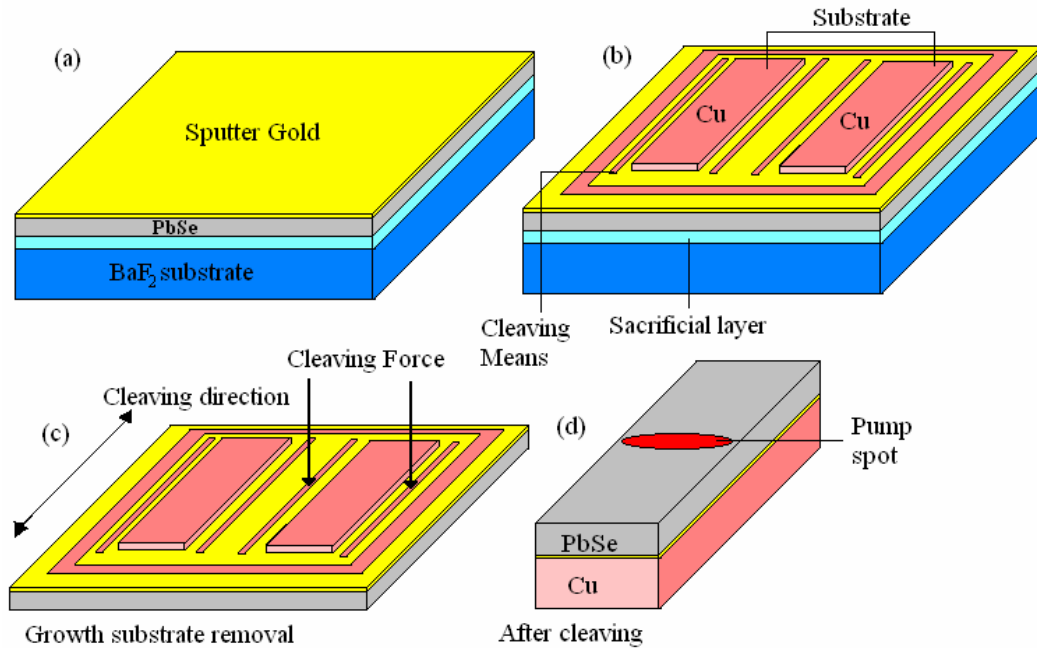


Fig 6.1 Schematic of process steps for the fabrication of a photo-pumped laser with an as-grown metallic substrate.

Referring to Figure 6.1a, in the very first step a layer of gold had been deposited over the surface of the MBE grown semiconductor epitaxial layer by standard sputtering technique. The thickness of this gold was approximately 100 nanometers to 700 nanometers. This was done to protect the epitaxial layer from the acidic copper plating solution. After this step, as shown in Figure 6.1b, thick layer of copper was electroplated, followed by a photolithographic technique, to define patterned structures. The result is a copper-carrier substrate, metallic cleaving-means and a metallic frame to hold the epitaxial layer after growth-substrate removal. The metallic-carrier substrate had a

thickness of 80 micrometers to 200 micrometers and width from about 200 micrometers to 500 micrometers. The width of these metal substrates actually forms the cavity length for lasing operation. Referring to Figure 6.1c, shown therein is a metal semiconductor structure, which was obtained after the removal of BaF₂ growth substrate. The growth substrate was removed by etching the BaF₂ sacrificial layer. The arrows in Figure 6.1c indicates the direction of cleaving force on the cleaving means, which is needed to obtain the two parallel facets for the formation of Fabry-Perot cavity for lasing. The cleaving means were aligned with the natural cleaving direction of the epitaxial layer. Upon application of the cleaving force along the directions shown in Figure 6.1c, the epitaxial layer gets cleaved along the directions parallel to the cleaving means.

Figure 6.1d shows a photo-pumped laser structure, which can be obtained after the cleaving of epitaxial layer and after separation from the metal semiconductor assembly as shown in Figure 6.1c. The advantage for optically pumped devices is that they do not need a p-n junction or electrical contacts. Also the absence of Joule heating increases the chance of lasing at higher operation temperatures. In Figure 6.1d, the pump spot from a diode laser, which can be used to pump the photo-pumped laser structure, is also shown.

The fabrication process described in Figure 6.1 can be modified for an electrically pumped laser as well. Figure 6.2 schematically describes the fabrication of an electrically pumped laser.

Referring to Figure 6.2a, shown therein is a semiconductor structure to which stripe patterns of gold had been made over the surface of a semiconductor epitaxial layer by standard photolithography and electroplating techniques. Gold stripes were made

perpendicular to the direction of the natural cleaving plane of the semiconductor. Stripe patterns have a length which is in the range from about 100 to 1000 microns, more preferably between 200 and 500 microns and the width, which is in the range from about 4 to 60 micron, more preferably between 5 and 20 microns.

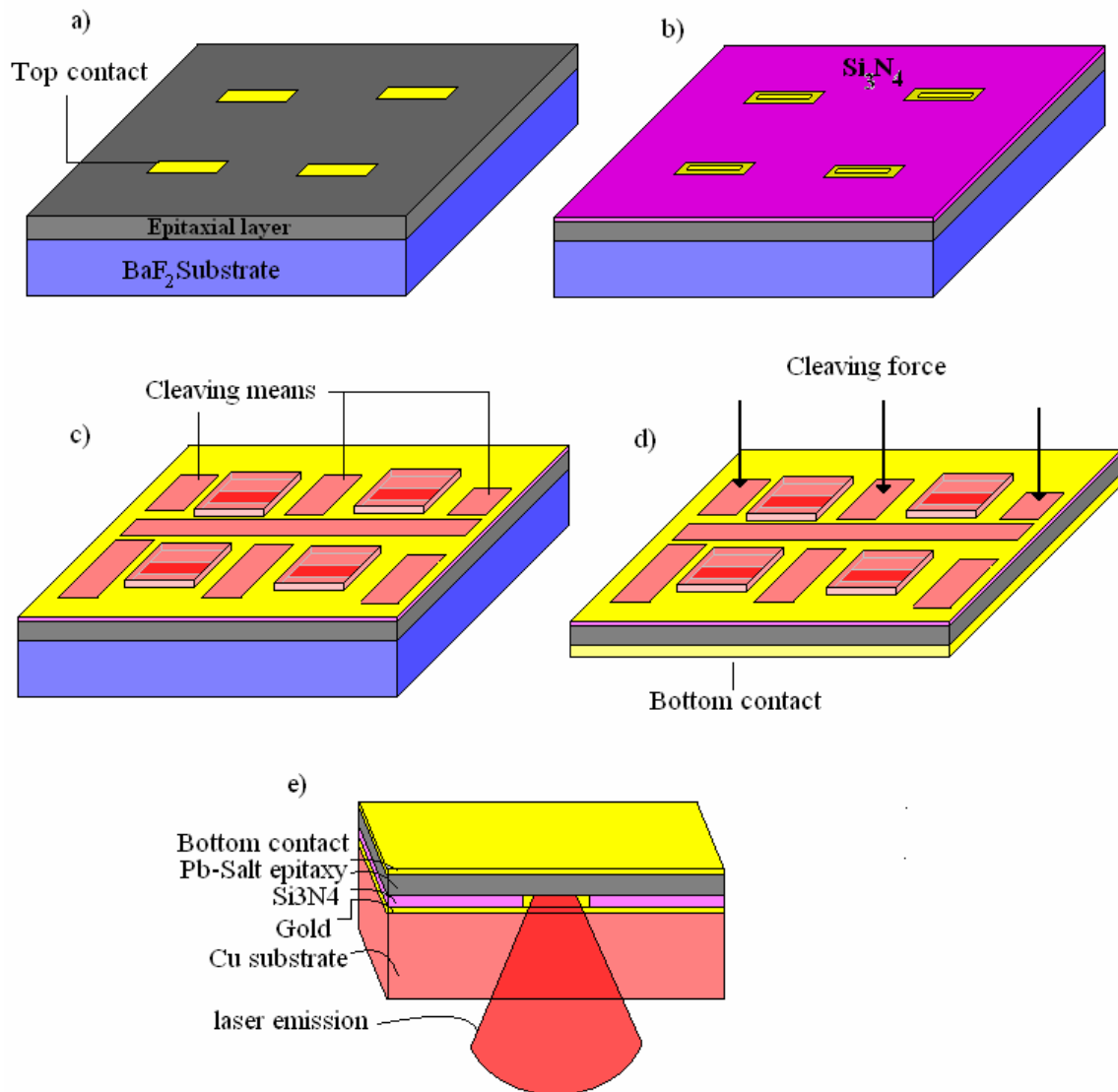


Fig 6.2 Schematic of process steps for the fabrication of a electrically-pumped laser with an as-grown metallic substrate

As shown in Figure 6.2b, an insulating layer of Si_3N_4 had been applied over the surface of the patterned epitaxial layer, by standard sputtering technique. The Si_3N_4 layer has a thickness of around 100 nanometers to 200 nanometers. The Si_3N_4 layer deposition was followed by a standard photolithographic technique in which small windows were opened on top of metal stripes and Si_3N_4 was etched away from those portions leaving a metallic gold underneath. After this step, a layer of gold was applied to the resulting surface of the semiconductor structure by a standard sputtering technique. This layer served the purpose of a seed layer for Cu electroplating. The layer has a thickness of 100-300 nm.

After this step, as shown in Figure 6.2c, a layer of copper was electroplated, followed by a photolithographic technique, to grow patterned structures, forming a copper-carrier substrate, metallic cleaving-means and a metallic frame to hold the epitaxial layer after growth substrate removal.

The metallic-carrier substrate has a thickness 80 micrometers to 200 micrometers and width from about 200 micrometers to 500 micrometers. The width of these metal substrates actually forms the cavity length for laser. Referring to Figure 6.2d is a metal semiconductor structure, which was obtained after the removal of BaF_2 growth substrate. The growth substrate was removed by etching the BaF_2 sacrificial layer. The arrows in Figure 6.2d indicate the direction of the cleaving force on the cleaving means, which is needed to obtain the two parallel facets for laser operation. The cleaving means are aligned with the natural cleaving direction of the epitaxial layer. Figure 6.2e shows an individual electrically pumped structure schematically.

6.3 Device Fabrication for Optical Pumping

An optically pumped device had been fabricated to check the feasibility of the entire fabrication process with a PbSe/PbSrSe quantum well sample grown on {110} BaF₂, having a thin BaF₂ sacrificial layer. The sample was processed according to the fabrication steps mentioned in the previous section and shown schematically in Figure 6.1. While performing photolithography for the formation of copper substrate, alignment of the patterns (to be made for copper deposition), with the [100] cleaving direction was done by following the same alignment technique described in section 3.3.3 of Chapter 3. The only difference in this particular case was, in place of metal holder with 54.735° angle mark, a mask was used with a pattern having the same 54.735° angle marking. Also the sample was cleaved twice before photolithography to obtain two {111} planes, as a part of this alignment process.

Figure 6.3 shows the SEM top view of a single Cu base formed on this quantum well sample. The BaF₂ growth substrate had already been removed from the metal-semiconductor structure.

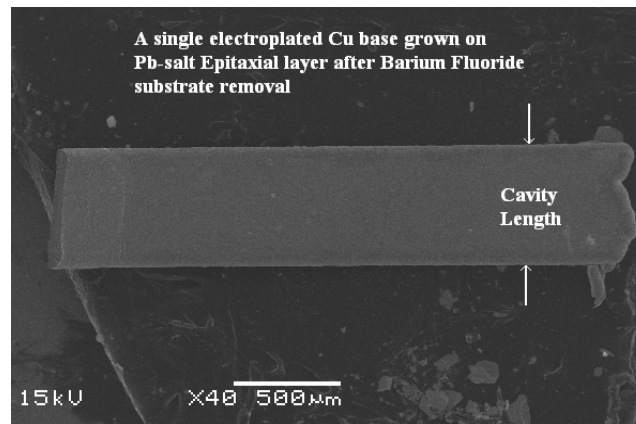


Fig 6.3 SEM top view of a single Cu substrate grown by electroplating on PbSe/PbSrSe quantum well sample. Image was taken after growth substrate removal and cleaving.

The SEM cross-sectional image of the cleaved epilayer after growth substrate removal on electroplated copper substrate is shown in Figure 6.4.

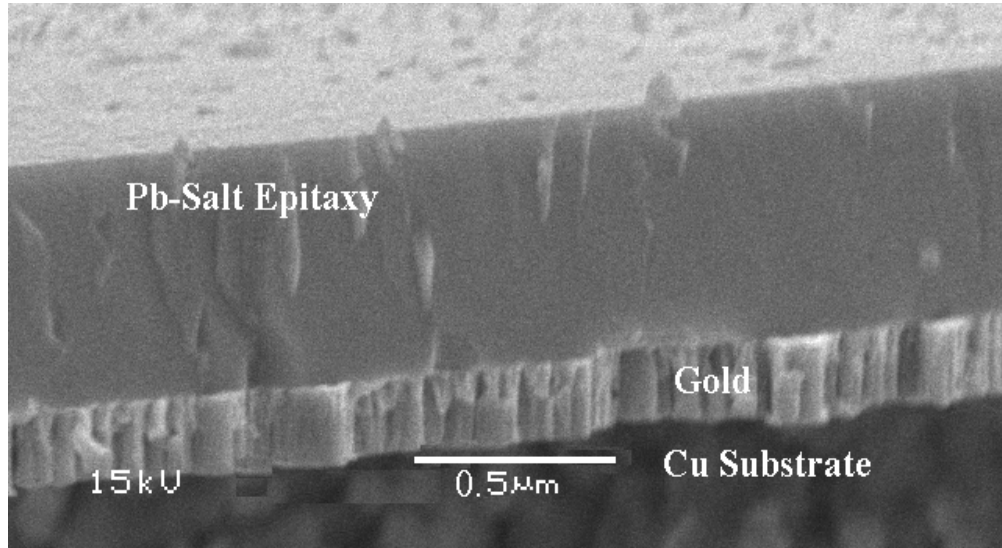


Fig 6.4 SEM cross-sectional image of the cleaved epilayer with multiple quantum well, after growth substrate removal, on as grown copper carrier substrate.

It was mentioned before, to protect the epilayer from acidic copper plating solution, a thin layer of gold was first sputtered on the sample. And then copper plating was done to make a copper substrate on the patterned sample. Patterns were made by photolithography.

The BaF₂ growth substrate was removed from the sample by etching the BaF₂ sacrificial layer in DI water at a temperature of 70°C.

Figure 6.5 also shows the SEM image of a cleaved epilayer after growth substrate removal of a dummy Pb-salt sample grown on {110} BaF₂. This sample was processed the same way as before, just to check the repeatability of the process. The epilayer was also thicker than the previous sample.

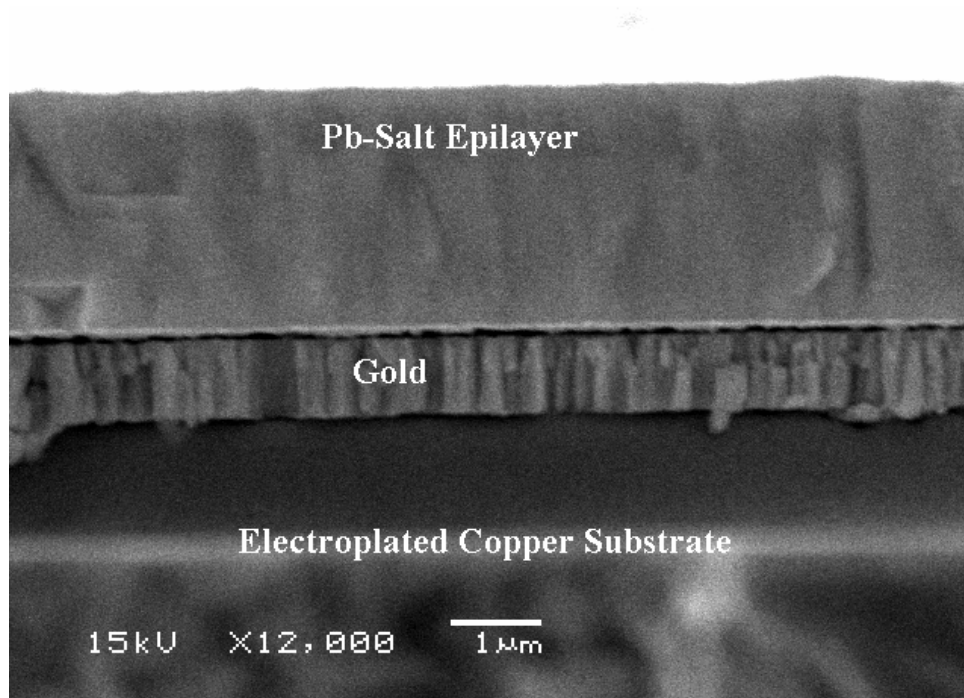


Fig 6.5 SEM cross-sectional image of a cleaved, thick epilayer, after growth substrate removal, on as grown copper carrier substrate.

6.4 Initial Results

Two samples had been processed according to this fabrication process, including a dummy one. However, as can be seen from the SEM images promising cleaving results were obtained from the Pb-salt epitaxy. Side emission was checked from the MQW sample by optical pumping with a 1.064 μm Nd:YAG laser ($\tau_{\text{pulse}} \approx 23\text{ns}$, 10Hz) having a rectangular spot width of 60 μm , focused by a cylindrical lens.

Even after nice cleaving, no side emission was observed from the MQW sample. There could be several reasons for that. First of all, other than air there was no optical confinement layer. Secondly the quality of this particular sample was much inferior to our average quantum well samples; the photoluminescence (PL) from the surface was several times lower than the photoluminescence from an average quantum sample.

Figure 6.6 shows the photoluminescence from the surface of the quantum well sample used in the fabrication process for optical pumping and photoluminescence from an average quantum well sample grown in our lab. Both of them were measured with same measurement set up and were also grown on $\{110\}$ BaF₂. The figure shows the difference in PL intensity from surface of both the sample.

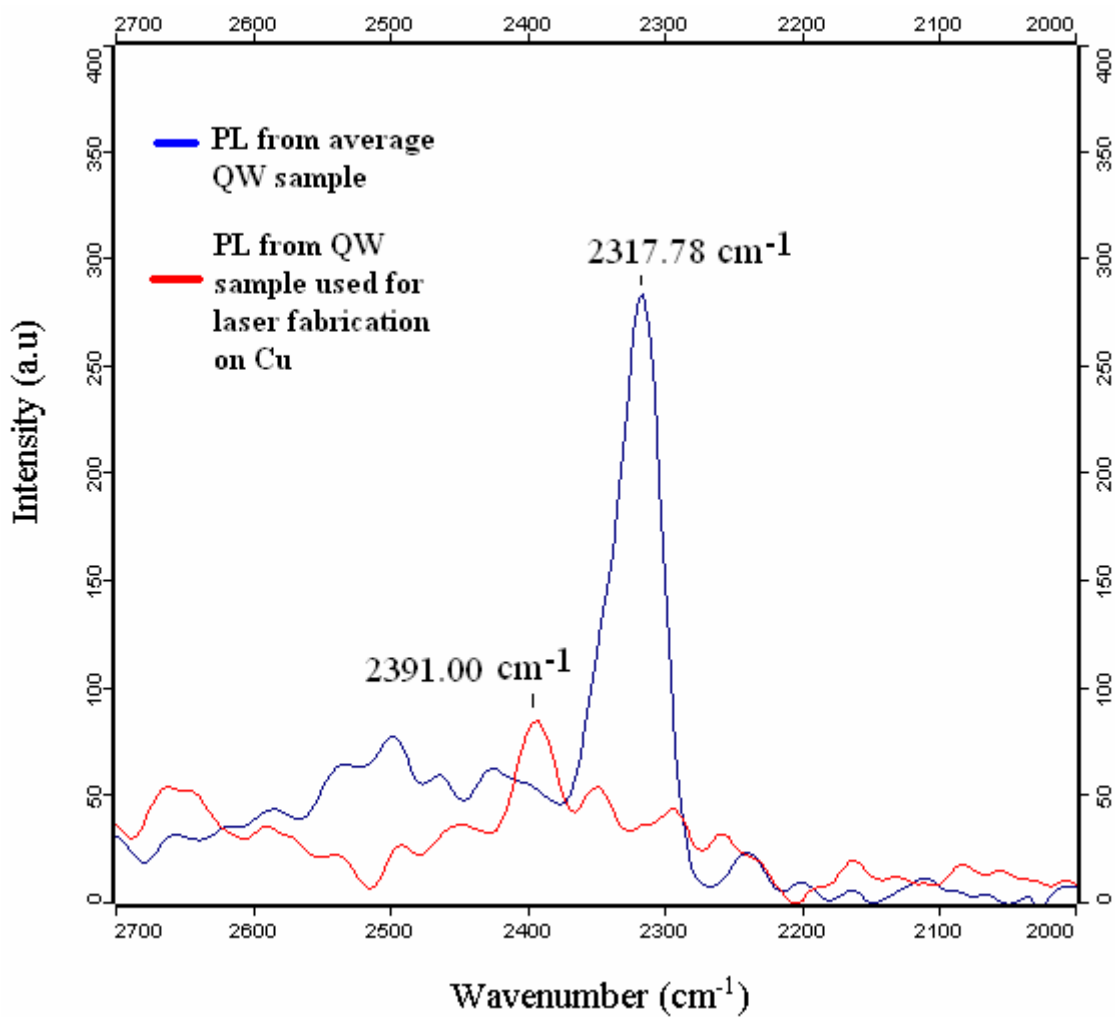


Fig 6.6 PL spectrum from quantum well sample used for fabricating optically pumped laser on Cu substrate and PL spectrum from an average quantum well sample grown in our lab. The intensity shown here from both the sample is actually 3.5% of actual intensity.

The most encouraging part of this process was the observation of continuous wave (CW) photoluminescence (PL) from the surface of a quantum well sample grown on BaF₂ at room temperature after plating gold and then forming copper base on the other side of the epilayer. The sample was optically pumped with a 980 nm InGaAs diode laser, at a pump power of 1.1W. The pump spot size was 400 μm in diameter. The PL spectrum was measured with IFS 66/S spectrometer in single-channel mode at resolution of 1 cm^{-1} , using a LN₂-cooled InSb detector. Ability to obtain CW PL from IV-VI semiconductor mid-IR laser materials transferred from growth substrates to more conductive heat sink materials is important since it provides a way to assess quantitatively the expected improvement in active region heat dissipation using methods similar to those described by Li et al.⁷.

Figure 6.7 shows the room temperature CW-PL spectrum of a quantum well sample after forming Cu substrate or base on the other side of the epilayer.

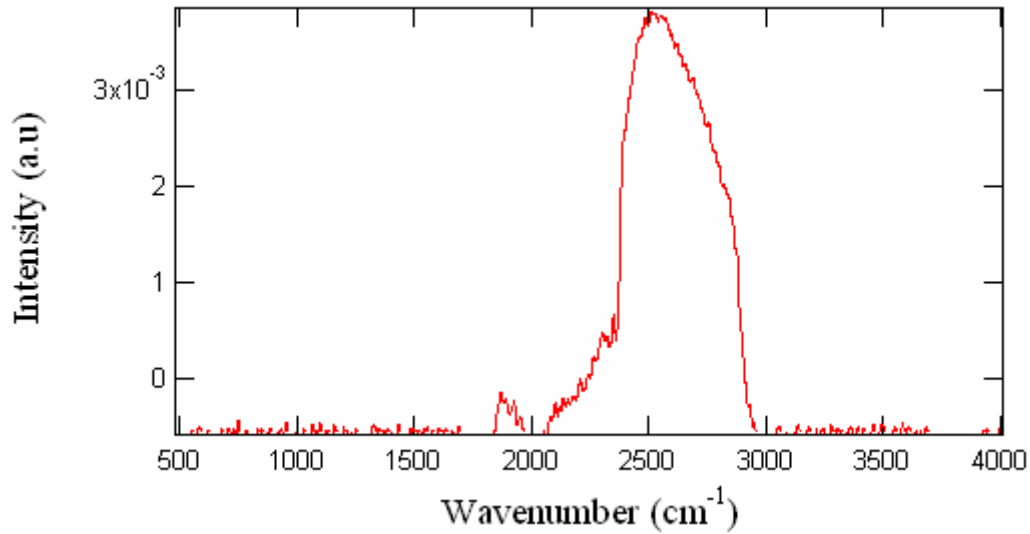


Fig 6.7 Room temperature CW-PL spectrum of a quantum well sample grown on {110} BaF₂, after forming Cu substrate on the other side of the epilayer.

6.5 Conclusion

Initial results including cleaved cavity and room temperature CW emission, obtained from the Pb-salt epitaxial layer on the inbuilt copper base or copper carrier substrate are promising and encouraging. However, further experiments with MBE grown Pb-salt epitaxial layer having either optical or electrical confinement layers are needed for the demonstration of lasing from these structures.

6.6 References

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Chapter 7

Summary and Future Work

7.1 Summary

The research described by this dissertation has focused on the fabrication and design, for the improvement of lead chalcogenide lasers. Several novel approaches that have not been tried previously were reported in this dissertation. The ultimate goal is to develop a TE-cooled single-mode CW Mid-IR laser for high resolution spectroscopic applications. Processing techniques and procedures, as well as theoretical design have been successfully developed that could lead to high temperature single-mode CW laser operation.

The second chapter focused on an antireflection coating material for diode-pumped Pb-salt light emitters that has enhanced room temperature CW light emission¹. The third and fourth chapters described in detail the development of [110] oriented laser fabrication. Chapter Three focused on the laser fabrication grown on {110} BaF₂ substrate. A new surface preparation technology for epi-ready [110] oriented BaF₂ substrates and a novel mounting technique for the fabrication of cleaved cavity Pb-salt laser grown on {110} BaF₂² was developed. In Chapter Four, lasing results from [110] oriented PbSe/PbSrSe double heterostructure laser fabricated on PbSnSe substrate have been reported for the first time. The fifth chapter described the design and fabrication of a ridge waveguide laser for single lateral mode operation. The sixth chapter introduced a novel fabrication method of cleaved facet Pb-salt laser on as-grown metal substrate for high temperature operation of the device.

7.2 Recommendations for Future Work

Formation of a cleaved cavity for a Pb-salt laser grown on [110] oriented BaF₂ substrate has been demonstrated. However, more research on the formation of p-n junctions and more studies are needed on the diffusion properties of both p- and n-dopants in Pb-salt material on BaF₂ substrates to demonstrate lasing.

Lasing results were achieved from the PbSe/PbSrSe based materials grown on a [110] oriented PbSnSe substrates. Although low threshold current densities were obtained even from broad contact stripe lasers, higher injection current increased the Joule heating, thus operation temperature remained below 150K. Therefore, a narrower stripe contact in the range of 5-20 μm should be tried to raise the operation temperature in the continuous wave mode.

The design and a fabrication process have been developed for the formation of a single lateral mode ridge waveguide laser. However, the laser beam from an edge emitting laser is elliptic in shape and therefore not very convenient for optimum coupling into and through a gas sampling cell for their applications in diode laser spectroscopy. Hence, more simulations on laser design to get small beam divergence and small astigmatism are needed.

Initial results including cleaved cavity and room temperature CW emission, obtained from Pb-salt epitaxy on the proposed inbuilt copper base or copper carrier substrate are promising and encouraging. Therefore, further experiments with MBE grown Pb-salt epitaxy having either optical or electrical confinement layers are needed for the demonstration of lasing from these structures.

7.3 Conclusion

Lead chalcogenides have been known for more than a century³. Unfortunately, they have been left out of the mainstream research in recent years. The modern epitaxial technology that allows high quality alternating material growth and advanced laser concepts can make a revitalization of IV-VI mid-IR lasers. Nevertheless, fabricating a CW, tunable, single mode near room temperature MIR device requires improved fabrication techniques and more accurate physical model. The physical model described in this research for single mode emission, along with [110] oriented QW structures and novel fabrication methods of devices on carrier substrates having much improved substrate thermal conductivity combined with the low Auger recombination promise the success of a MIR light emitting device.

Throughout this research several novel processes have been developed. All the developed processes are generic and can be applied to Pb-salt materials grown on any substrate. Nevertheless the challenges, which are not met yet and further experiments that are recommended in the previous section, present opportunities for ongoing research. Conclusively, the work presented in this dissertation holds the promise and made a step ahead towards a compact mid-infrared laser system. Such light sources will eventually lead to many applications such as a high efficiency, field-portable, mid-IR laser spectroscopy systems.

7.4 References

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