Final Report: Mode-locking of an InAs Quantum Dot Based Vertical External Cavity Surface Emitting Laser Using Atomic Layer Graphene

ABSTRACT
The InAs quantum dot (QD) grown on GaAs substrates represents a highly performance active region in the 1 - 1.3 µm wavelength range. The use of such QDs in vertical-cavity devices has resulted in high-power and broad gain bandwidth VECSELs in the NIR. In this project we demonstrate growth and design of InAs QD based VECSELs in the 1200 -1300 nm wavelength range with record CW output powers. We utilize a resonant periodic gain (RPG) structure with a single QD layer per standing wave antinode to effectively increase the distance between strained layers to improve the quality of the active region. We compare the performance of a VECSEL with 12 separate QD layers.

SUBJECT TERMS
Semiconductor lasers, Vertical cavity surface emitting lasers, semiconductor epitaxy

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- Abstract: UU
- This Page: UU

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Final Report: Mode-locking of an InAs Quantum Dot Based Vertical External Cavity Surface Emitting Laser Using Atomic Layer Graphene

ABSTRACT
The InAs quantum dot (QD) grown on GaAs substrates represents a highly performance active region in the 1 - 1.3 \( \mu \)m wavelength range. The use of such QDs in vertical-cavity devices has resulted in high-power and broad gain bandwidth VECSELs in the NIR. In this project we demonstrate growth and design of InAs QD based VECSELs in the 1200 -1300 nm wavelength range with record CW output powers. We utilize a resonant periodic gain (RPG) structure with a single QD layer per standing wave antinode to effectively increase the distance between strained layers to improve the quality of the active region. We compare the performance of a VECSEL with 12 separate QD layers ("12x1" structure) to a more traditional design that uses 4 groups of 3 closely spaced QD layers ("4x3"). The experimental performance of the 12x1 device is superior to the 4x3 structure in terms of threshold pump power, differential efficiency, and maximum output power. For thermal management, the GaAs wafer of the 12x1 structure was thinned by mechanical polishing, and indium was used to mount the gain mirror on a thermal-grade chemical vapor deposition (CVD) diamond, allowing for over 6 W of CW output power, which is a record result for stranski-krastanov based quantum dots.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received Paper


08/31/2013  1.00 Pankaj Ahirwar, Thomas J. Rotter, Darryl Shima, Nahid A. Jahan, Stephen P. R. Clark, Sadhvikas J. Addamane, Ganesh Balakrishnan, Alexandre Laurain, Jorg Hader, Yi-Ying Lai, Jerome V. Moloney, Ikuo Suemune, Robert G. Bedford. Growth and Optimization of 2-\text{&x03BC;}m InGaSb/AlGaSb Quantum-Well-Based VECSELs on GaAs/AlGaAs DBRs, IEEE Journal of Selected Topics in Quantum Electronics, (07 2013): 0. doi: 10.1109/JSTQE.2013.2239615

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Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

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(c) Presentations


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Awards

- 2014 IEEE Albuquerque section outstanding Educator award.
- 2014 UNM SOE Regents’ Lecturer.
- 2013 UNM SOE Junior faculty research award.
- 2013 ECE department research award.
- 2012 IEEE Albuquerque section outstanding young engineer award.
- 2012 ASEE Air Force summer faculty fellowship.

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Sub Contractors (DD882)

Inventions (DD882)
Scientific Progress

The InAs Quantum Dot VECSELs research resulted in the demonstration of >6 Watts CW which is a world record for stranski-krastanov quantum dot based lasers. The results will be further improved by attempting a bottom emitting structure that is bonded to CVD diamond. Furthermore in the final year significant work was done towards the realization of InAs Quantum Dash based active regions for VECSELs at 2 microns emission wavelength. The active region was demonstrated as edge emitting lasers and resulted in some of the longest wavelength quantum dot/dash based lasers. The conversion of the lasers to vertical cavity structures will require metamorphic distributed Bragg reflectors and this has been successfully demonstrated. The details of the research can be found in the attachment section of this technical report.

Technology Transfer
“Mode-locking of an InAs Quantum Dot Based Vertical External Cavity Surface Emitting Laser Using Atomic Layer Graphene”

Final Report

In the past three years the effort at UNM has resulted in the following key developments as a result of the DOD HBCU/MI funding.

1) Installation of state of the art molecular beam epitaxy (MBE) control and effusion cells on the existing V80 reactor for growth of high power VECSELs (Vertical External Cavity Surface Emitting Lasers).
2) Installation of a FTIR based temperature dependent reflectivity setup for characterizing VECSELs and SESAMs (Semiconductor Saturable Absorber Mirrors).
3) Demonstration of up to 6 Watts CW with InAs QD (Quantum Dot) VECSELs (1250 nm) and 15 Watts with InGaAs QW (Quantum Well) VECSELs (1040 nm).
4) Growth and characterization of AlGaAs DBR (Distributed Bragg Reflector)/Spacer structures for fabrication of InGaAs quantum well based SESAMs, resulting in mode locked VECSELs.
5) Development of transfer process for monolayer graphene for use in SESAMs.
6) Growth and characterization of AlGaAs DBR/Spacer structures for fabrication of graphene SESAMs.

1. Installation of state of the art MBE control system and effusion cells on V80 reactor.

One of the proposed tasks of this project was to bring the V80 solid source molecular beam epitaxy system at UNM up to spec for the demands and rigor of VECSEL and SESAM growth. While this reactor still holds numerous records for VECSELs including the highest power QD VECSEL and some of the highest power antimonide lasers, the control process for the reactor is based on LABVIEW® and this has made the epitaxial process very difficult to control since some of the lasers require as many as 5 to 8K shutter actions in a growth. Also of concern was the fact that a significant drift in the aluminum growth rate was observed during the growth of the lasers, which made the development of very thick VECSEL structures extremely difficult.

In the past three years both these issues have been addressed through the use of this HBCU/MI grant. The control system for the V80 has been replaced with a MBE control® Amber system along with a completely redesigned rack to accommodate the newer software. The design of the new rack is shown in figure 1. The control system for the V80 now includes 1KW Sorensen DC Power supplies for all the sources and a Dual Loop Eurotherm 3508 controllers in chassis with interlock circuitry and individual power switches, for a more precise flux calibration we have made use of Agilent XGS-600 controller for 3 ion gauges, we have also installed a new shutter controller and a PLC with status indicator for vacuum and water interlocks - multiple inputs for pressure. The software operating this system is AMBER, which has significant improvements to structure design and growth control.
We have currently installed the new control systems for the reactor and we switched systems in the middle of fall 2013. This has resulted in significant increase in the output of high quality VECSEL and SESAM wafers from our team. From initially growing SESAMs for the first time at the beginning of this project, we are now able to grow these structures with extreme precision resulting in both mode locked VECSEL demonstration at UNM and at DoD labs such as AFRL and at other university collaborators such as the University of Arizona.

2. Installation of a FTIR based temperature dependent reflectivity setup for characterizing VECSELs and SESAMs.

One of the key issues prior to the start of this program was that temperature dependent reflectivity has now become a key technology in the growth and troubleshooting of VECSELs. The conventional FTIRs are not able to accommodate robust heating and cooling stages and hence there is a significant limitation to the range of temperatures over which a scan can be done. Also of concern was the fact that characterization of graphene based SESAMs and the characterization of graphene layers in general would require a more sensitive FTIR.

We have worked with NICOLET to realize a FTIR that can accommodate our heating and cooling stages (-25ºC to +150ºC) and to image the reflectivity at with very high sensitivity and a very high resolution. This setup was commissioned in January 2013 and has resulted in unprecedented...
information about the growth and performance of VECSELs. For instance the data shown in figure 2 is that of a VECSEL being optimized and one can see that the shift of the gain peak and the distributed Bragg reflectors can be observed with unprecedented precision. This allows us to model and optimize the VECSEL designs with a significantly improved accuracy.

Fig. 2: High resolution TDR with extended temperature range.

3. Demonstration of upto 6 Watts CW with InAs QD VECSELs (1250 nm) and 15 Watts with InGaAs QW VECSELs (1040 nm).

In the following paragraphs we explain the novel and innovative techniques developed to achieve record QD and QW VECSEL performance. This effort provides us with sufficient laser material and processed devices to conduct the mode locking studies in the rest of the program. The key achievements have been:

1) >6 Watts from InAs QD VECSELs
2) ~ 20 Watts CW from InGaAs QW VECSELs.
1. Quantum Dot VECSEL design and optimization.

In the following paragraphs we explain the novel and innovative techniques developed to achieve record QD-VECSEL performance and we shall also explain the experiments conducted in this area during the course of the performance period.

The key challenge for all quantum dot VECSEL research at present is the accommodation of large number of quantum dot layers in a vertical cavity structure. The lack of gain in the nano-scale ensembles can require as many as 30 quantum dot layers in the structure. The issue however is that, in adding these many layers, threading dislocations form in the active region which are highly detrimental to the laser’s performance. Thus, to resolve this issue, we have investigated two techniques:

1) The use of resonant periodic structures which can space the QD layers out significantly thus reducing strain in the active region.
2) QD optimization specifically for mode-locked VECSELs.

Perhaps the most important innovation from the PI’s group in developing world-record quantum dot lasers is the introduction of single dot-layer per antinode resonant periodic gain designs. We have developed a resonant periodic gain (RPG) structure with a single QD layer per standing wave antinode to effectively increase the distance between strained layers to improve the quality of the active region. We have already compared the performance of a VECSEL with 12 separate QD layers ("12x1" structure) to a more traditional design that uses 4 groups of 3 closely spaced QD layers ("4x3"). The experimental performance of the 12x1 device is superior to the 4x3 structure in terms of threshold pump power, differential efficiency, and maximum output power.

![Fig. 3: Two different resonant periodic gain designs for InAs QD based VECSELs. The design on the left has three dot layers per antinode (4x3 design) of the E-field standing wave and the design on the right has a single dot layer per antinode (12x1 design).](image)

The VECSEL structures are grown in the Vacuum-Generators V80 molecular beam epitaxy (MBE) reactor on 3-inch diameter GaAs substrates. After oxide desorption, the DBR consisting of 30 pairs of quarter-wave optical thickness AlAs and GaAs layers was grown. The InAs QDs were formed by depositing 1.68 monolayers of InAs inside 7 nm thick In$_{0.15}$Ga$_{0.85}$As quantum wells grown at a substrate temperature of 480ºC as determined by optical pyrometry. For the 4x3 structure, 3 QD layers are separated by 15 nm GaAs barriers, and repeated 4 times at adjacent standing wave antinodes, resulting in a total GaAs barrier/subcavity thickness of 0.9 µm. In the case of the 12x1 RPG design, a single QD layer is placed at 12 consecutive antinodes, resulting in a subcavity thickness of almost 2.4 µm. These designs are shown in Figure 1. While this should be beneficial due to a more complete absorption of incident pump light, it also results in larger pump non-uniformity across the QD layers.
Both VECSEL structures are capped with one DBR pair consisting of AlAs and Al_{0.3}Ga_{0.7}As (transparent to the 808 nm pump light) to both prevent surface recombination and reduce lasing threshold.

**Fig. 4: L-L curves for the two VECSEL designs at different heat sink temperatures.**

The VECSEL output power as function of the incident pump power for different heatsink temperatures has been measured for both designs and is shown in Figure 4. By examining these curves, a number of observations can be made to compare the performance of the two active region designs: The 4x3 structure consistently has a higher threshold pump power compared to the 12x1 sample, as is shown in Figure 5 (a). At heatsink temperatures above 20ºC, lasing threshold could not be reached for the 4x3 VECSEL. Figure 5 (b) shows the differential efficiency of both structures, as determined by a linear fit to the output power curves above threshold but below thermal roll-over. The thermally limited output powers as function of the heatsink temperature are compared in Figure 5 (c).

**Fig. 5: Threshold, slope efficiency and output power as a function of temperatures.**

The above body of the work is perhaps the most mature set of results for QD-VECSELs published to date. However these results are based on a very limited number of epitaxial runs. We have recently also realized that in the structures grown above the micro-cavity resonance and the gain are not detuned optimally for high power operation. We believe that achieving the correct level of detuning could take the CW output power from the VECSEL from the current best of 5 Watts CW to as much as 20 Watts CW. To achieve the precise detuning we shall be making use of a technique called temperature dependent reflectivity, which looks at the depth of the microcavity resonance as a function of temperature. The deepest dip typically corresponds to the point at which the gain is perfectly slotted into the microcavity resonance. This alignment technique has been very effective in other active
regions grown by us and will be instrumental in achieving tens of watts CW from the QD VECSELs. Such a setup is installed in the PI’s lab.

![Graph](image)

**Fig. 6: High power QD VECSEL demonstration. (top) LL curve and (bottom) spectrum.**

The maximum power from these devices were observed to be a little greater than 6 Watts CW. We expect significant improvements to the performance of these lasers with a move to bottom emitting configurations. However for the purposes of this project we believe that this world record result will suffice. We have similarly also grown InGaAs VECSELs based on InGaAs QWs. These lasers have yielded impressive 20 Watts CW results and the power from these wafers can be scaled to reach upto 100 Watts CW if required.
Figure 6 shows the LL curve and the spectrum for the 1250 nm InAs QD VECSEL. While this result stops at a maximum power of 5 W CW, we have recently been able to push this result to > 6 Watts CW.

Figure 7 shows the LL curve and the spectrum for the high power InGaAs QW lasers. Figure 8 shows the effect of using different output couplers on the efficiency of the lasers.

Fig. 7: High power QW VECSEL demonstration. (top) LL curve and (bottom) spectrum.
Fig. 8: QW VECSEL wall plug efficiency.

3. Growth and characterization of AlGaAs DBR/Spacer structures for fabrication of InGaAs QW and graphene SESAMs.

A series of SESAMs and graphene SESAM bases were grown for use with the 1020nm VECSELs. Fig. 9 shows the reflectivity measurements of resonant and anti-resonant QW based SESAMs (note the R values >100% are due to artifacts of the measurement). The designs were used from Keller et al.’s group and involved the growth of a single InGaAs QW as the absorber.

Fig. 10 shows the DBR/spacer base for a resonant graphene SESAM. This does not exhibit the dip of the resonant SESAM in Fig. 10 as there is no absorber present until the graphene is deposited.
Fig. 9: Reflectivity of 1020nm QW based SESAMs

Fig. 10: Reflectivity DBR/Spacer for Graphene SESAMs


Central to the project objectives is the use of graphene as the saturable absorber layer in SESAMs for the modelocking of VECSELs. Currently high grade monolayer graphene is available from many commercial vendors. This graphene is catalyzed on either a copper foil or on nickel coated substrates.
The graphene must be transferred off of these substrates and then on to the DBR/spacer to form a completed SESAM.

2. Graphene III-V integration study

The starting point for this body of research will be to conduct a comprehensive study of graphene transfer and growth techniques. We are currently conducting experiments with chemical synthesis techniques, exfoliation techniques and epitaxial techniques. The emphasis in the coming performance period will be to try these established techniques on a variety of optical, dielectric and semiconductor materials and to characterize them.

I. Transfer of graphene on III-Vs:
The state of art in transferring graphene onto various surfaces is very comprehensive and we have established the ability to synthesize graphene through some select techniques. The difference between a more traditional graphene synthesis process for electronics and one for optical applications such as mode-locking is that the process has to be very sensitive to three key parameters when graphene is used for mode-locking.

1) Optical quality.
2) Graphene size and uniformity.
3) Ability to achieve single – multi layer graphene.

The dimension of the graphene layer is key to effective mode locking of the lasers. In case of a fiber integrated graphene absorber, the graphene has to uniformly encompass the core of the fiber. This is to ensure that the mode in the fiber-core is interacting with the graphene in a uniform manner. The issue is a little more complicated with free-space mode-locking such as in the case of graphene saturable absorber mirrors since the mode profile has to overlap with the graphene. In case the graphene is not large-area, it complicates the integration since the mode-graphene alignment will have to be performed and this is not a trivial task. This same issue will exist if the graphene is incorporated into the semiconductor matrix. Thus the emphasis of this research task was to make sure that the graphene size is optimal for the mode profile overlap. This issue will be elaborated upon in the following sections. To achieve optical quality graphene and to achieve single to multi-layer graphene we have established two fabrication processes that have demonstrated good success in our laboratory.

A) Polymer host technique: One such technique that is showing good results in our lab is the incorporation of the graphene in a into a host polymer. This can be done by extracting the graphene without functionalization in water under mild sonication. This technique demonstrated by Sun et al. allows the retention of single layer type properties of graphene in both the exfoliated single layer graphene (SLG) and few layer graphene (FLG) flakes. Sun et al. make use of bile salts to obtain a stable, higher concentration of SLG and FLGs. These amphiphilic molecules i.e molecules with a hydrophobic and a hydrophilic part are able to disperse the graphene by physical adsorption on its surface. The dispersions are mixed with poly vinyl alcohol (PVA) in a polymer mixer. The evaporation of the solvents leads to graphene composites. We hope to achieve these layers of graphene on various optics such as the output couplers or semiconductors to mode-lock the laser.

B) PMMA stamp technique - The second process that we are currently using for integration of the graphene with semiconductors and dielectrics is the transfer of CVD graphene grown on copper foils. The graphene is grown on thin Cu-foils by CVD using methane and hydrogen precursors. The grown graphene film is then coated with poly-methyl-meth-acrylate (PMMA). The Cu substrate is then selectively removed by a Marble’s reagent solution (distilled water (50 ml), hydrochloric acid (50 ml))
and copper sulphate (10 g)). The graphene attached to the PMMA stamp is now transferred to the semiconductor, the dielectric, the fiber or the optical element. The final step involves dissolving the PMMA resulting in a graphene layer on the required surface. We have already conducted out-gassing studies with the graphene on GaAs substrates in a vacuum chamber with the wafer heated to ~ 700 ºC monitored with a residual gas analyzer and have not seen any untoward species in the chamber. Thus the Copper-PMMA-semiconductor technique also seems to be UHV crystal growth equipment friendly and can be used with molecular beam epitaxy (MBE) for our graphene encapsulation studies.

II. Characterization studies for graphene III-V integration: The characterization of the graphene on various surfaces is done through Raman spectroscopy. The use of Raman spectroscopy with optical contrast microscopy will be used to determine the thickness of the graphene layer covering the surfaces. Apart from the Raman spectroscopy studies the investigation of the graphene will also employ techniques to study various properties of the graphene as a function of the number of layers including absorbance, modulation depth and saturated carrier density. We shall perform these studies as a method of qualifying and comparing the graphene absorbers. Apart from optical studies, we shall also employ high-resolution microscopy techniques including transmission electron microscopy and scanning tunneling microscopy to study the quality of the graphene. In particular, the TEM study will look into the effects of integrating the graphene in a semi-conductor matrix, the benefits of which will be explained in the following sections. The study will look into the effect of the matrix on the graphene layer and also into the effect of the graphene on the semiconductor epitaxial capping layer.

Numerous methods for the transfer of graphene from these substrates were investigated. The transfer development involved removing the graphene from the substrate with a carrier and then transferring it from the carrier to a glass slide, used as a stand in for the DBR/Spacer during process development. The use of graphene grown on Ni was abandoned as removal techniques proved to be unreliable. For graphene on copper a reliable process was developed. The final process consisted of adhering a polymer (PMMA) to the graphene-on-copper; dissolving the copper, leaving the graphene adhered to the PMMA; attaching the graphene to a glass slide; dissolving the PMMA leaving behind the graphene on glass. Figure 11 shows, on the left, a macroscopic image of graphene transferred to a glass slide. It shows as a ‘haze’ on the slide and is circled in blue for easy identification. The image on the right in Figure 11 shows an optical Nomarski image of the same sample. It can be seen that the graphene has transferred uniformly, save for a few tears, which can be minimized via more careful handling during the transfer process.

**Fig. 11:** Graphene on glass (left) Nomarski of graphene(right)

The mode locking experiments were done with InGaAs QW SESAMs and then repeated with Graphene SESAMs. In the beginning both SESAMs did not mode-lock and upon further inspection it was determined that this was due to oxidation/ablation of the absorber. In the case of the InGaAs SESAMs the burial of the quantum wells deeper into the structures (at ~ 5 nm) seemed to solve the issue. **The mode locked lasers using InGaAs SESAMs resulted in pulse widths of a few pico seconds at repetition rates of 2 GHz with an average power of a few milli-watts.**

However the same experiment when tried with a GSAM did not succeed. There are a few issues that we have faced with the GSAMs. The location of the graphene layer in the SESAM left us with very few options. The only way to realize and verify the presence of the graphene layer on the DBR was to incorporate the graphene on the top of the DBR. Alternately we could also incorporate the graphene inside the VECSEL structure and not include a separate GSAM. The incorporation of the graphene into a VECSEL cavity has a few options. We attempted the use the GSAMs for mode locking the VECSELs and the schematic is shown in figure 6. The advantage of this technique is that we will be able to adjust the cavity design to have the mode size at a minimal value on the graphene and through inspection we can align the spot exactly onto the graphene layer.

![Graphene based Mode locking schemes currently investigated.](image)

The GSAMs would not mode lock despite numerous attempts and the primary reason for this was established as a lack of uniform monolayer converge of graphene on the GSAM surface. While we were able to realize single mono-layer graphene on the GSAM DBRs the fraction of the surface that was covered with graphene was less than a percent. Furthermore when used as GSAM in a V-cavity, the graphene further ablated. The area of the GSAM when inspected after a mode-locking attempt showed significant lack of graphene when compared before and after the attempt.
Fig. 13: Effect of fluence on the graphene on DBR. (Top) the graphene is realized on the DBR with the monolayer graphene showing up as translucent and the multilayer graphene showing up as the bright region. (Bottom) the graphene is ablating before saturation fluence can be reached. This results in the disappearance of the graphene in the area where the beam interacts with the GSAM.
Fig. 13 shows the effect of the VECSEL on the GSAM. The top image is a graphene incorporated DBR at 1050 nm showing the presence of graphene layers. The single layer graphene is translucent while the multi-layer graphene is a more bring color. The bottom structure shows the image of the structure after the incorporation of the GSAM in the VECSEL cavity. The single layer graphene is clearly gone with some remnants of the multi-layer graphene clearly visible. The immediate ablation of the graphene is the principle reason for not being able to achieve effective mode-locking of the GSAM VECSELs.

**Incorporating the Graphene inside the epitaxial structure:** The difficulty in using surface graphene for GSAM VECSELs lead us to attempt using graphene incorporated inside the GaAs matrix to mode-lock the VECSELs. These include both the use of traditional GSAMs very similar to the InGaAs QW structures with the graphene layer buried 5 nm inside the structure. This effort has to date not resulted in GSAM like behavior with no delectable absorption.

### 5. Summary

During the course of this project, we have demonstrated up to 6 Watts CW with InAs QD (Quantum Dot) VECSELs (1250 nm) and 15 Watts with InGaAs QW (Quantum Well) VECSELs (1040 nm). We have shown the growth and characterization of AlGaAs DBR (Distributed Bragg Reflector)/Spacer structures for fabrication of InGaAs quantum well based SESAMs, resulting in mode locked VECSELs. We have also been able to develop a transfer process for monolayer graphene for use in SESAMs. The QW SESAMs successfully mode-locked the VECSELs however the graphene SESAM has yet to demonstrate this result on account of non-uniform graphene distribution on the DBR.