Fabricating Dual-Sided Micro Lenses and High Frequency Talbot Diffraction Gratings Using Binary Masks

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Abstract and Introduction:
Binary photomasks are a way of approximating 3D structures when using photolithography. Multiple masks are used and the surface of a substrate is etched into a number of distinct heights.

A Fresnel lens was made employing this method, and was etched into both sides of a quartz substrate; the advantage of using a phase mask on both sides of a lens is that the diffraction effects that take place inside of the medium can be taken advantage of. This idea eventually was carried over to make a diffraction grating that used what is called the “Talbot Effect.” This effect describes the phenomenon where light that passes through a periodic grating repeats the grating pattern in the near field at certain distances termed the Talbot length ($Z_T$). The frequency of the pattern doubles at a quarter of $Z_T$. By creating relatively shifted diffraction gratings on either side of the substrate, we effectively created a higher frequency grating.

Experimental Procedure:
The Fresnel lens was simulated in MATLAB by first simulating the phase of a conventional lens [1] with a focal length of 50 cm; then the mod function was used on this phase to turn the lens into a Fresnel lens. Masks transferred into Clewin software would approximate the three dimensional surface in eight distinct heights. These binary masks were made on a single chromium photomask in the Heidelberg DWL 66FS and were used to expose our substrate in the SUSS MJB3 Maskaligner. Our substrate consisted of a fused silica slide with a 40 nm coating of chromium (Cr) on one side, which aided in alignment. Negative resist (NR9) was used in the photolithographic process to create the first Fresnel lens. First an aperture was created in Cr by etching it with a chemical known as CR-7S. The fused silica was etched in the Plasma-Therm reactive ion etch II (RIE II), which etched the surface using a plasma. A 3:1 mixture of tetrafluoromethane and trifluoromethane gases was used at a temperature of 25°C giving an etch rate of 12 nm per minute. For a material of refractive index $n(\lambda)$, the etch depth for a phase difference of $2\pi$ is given by $h_{2\pi} = \lambda/n(\lambda)-1$. The etch time for the $i$th photomask is given by $t_{2\pi}/2^i$, where $t_{2\pi}$ is the etching time for $h_{2\pi}$. This lens was made again on the other side of the substrate, opposite of the Cr coated side effectively creating a lens of focal length of 25 cm when using HeNe laser light.

Our Talbot grating was also simulated using MATLAB software. Our substrate for these gratings was a 1 mm thick quartz substrate, which also had a 40 nm coating of Cr on one side. We used the equation provided by Lord Rayleigh [2] in Figure 1 to determine our grating period so that the $Z_T$ would be four times the substrate thickness; this meant that at 1 mm, the intensity pattern of the grating would have twice the frequency as the original grating. The photolithographic process for making the Talbot grating was the same as earlier, except we only needed one etch into the quartz on each side to make the phase grating. The same grating was made four times on the Cr side of the one-inch square substrate in a 2x2 array. On the opposite side, two gratings were made; one grating was shifted by one fourth of a period, and the other was shifted by one half. This was so the effect on the diffraction pattern of the two could be compared against the grating etched on a single side.

\[ Z_T = \frac{\lambda}{1 - \sqrt{1 - \frac{\lambda^2}{\alpha^2}}} \]

Figure 1: Equation to determine $Z_T$. The letter $\alpha$ represents the grating period.

Figure 2: Top view of the MATLAB simulation of the Talbot grating.
Results and Conclusions:
To test the Fresnel lens made with NR9, we sent collimated HeNe light through the lens and measured the diameter of the central spot at several distances away; it should have been the smallest at the focal length. The diameter was converted from the number of pixels in the picture with one pixel corresponding to 3.7 µm. We found the focal length to be about ~ 25 cm, at which the central spot diameter matched the theoretical value (from Rayleigh’s Criterion). Moreover, the spot did not suffer from higher order aberrations such as astigmatism or coma showing that the alignment between the two sides of the mask was good. Thus, we had our successful proof of concept for aligning and etching a phase mask on both sides of a substrate. The Talbot gratings were tested with the same HeNe laser. The diffraction pattern we expected for both the single and double grating is shown by Figure 2. What we observed, however, is shown in Figures 3 and 4. The main difference observed in these patterns is that in the experiment, light shows up on the 0th order of light, which was not expected. It is hypothesized that this difference could be due to either an error in the simulation, inaccurate etch depths, or misalignment of the photomasks.

Future Work:
Future work would involve investigating the incongruences between simulation and experiment for the Talbot gratings that were tested. The accuracy of our alignment would also be measured. We would also explore using the Talbot effect for improving the resolution of photolithography.

Acknowledgments:
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References:
Infrared Filtering via Sub-Wavelength Gratings for Hyperspectral Imaging

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Abstract:
Fabry-Perot cavities are an attractive method of infrared filtering, provided they are made of broadband, lossless reflectors. A long wavelength infrared region (LWIR, 8-12 µm) broadband reflector is demonstrated using a high-index contrast sub-wavelength grating (HC-SWG) based on a suspended silicon system. The expected grating field response was simulated using COMSOL Multiphysics to exhibit greater than 90% reflectance in the LWIR, for transverse magnetic polarization. An un-switched CF$_4$/SF$_6$ reactive ion etch (RIE) was characterized, and showed improved sidewall profile compared to previously used etch chemistry. Fabricated gratings demonstrate greater than 85% reflectance between 8 µm and 14 µm, with the response agreeing well with simulations incorporating as-built dimensions.

Introduction:
Hyperspectral techniques are needed to advance to the next generation of thermal imaging systems. A hyperspectral image contains the electromagnetic spectrum for every point in an imaging plane, providing a “fingerprint” for different objects. These fingerprints improve object discrimination, which is useful in a wide array of applications including satellite analysis of climate change, military surveillance, and biological and chemical sensing. Hyperspectral images are formed from intensity acquired at each wavelength, which requires narrowband filters such as interference-based Fabry-Perot cavity filters. A Fabry-Perot cavity is composed of two parallel reflectors separated by a set distance, and transmits light when a standing wave can be established between the two mirrors. To ensure highly efficient and selective filters, the reflectors must exhibit very high reflectance and be non-absorbing. In this work we designed, fabricated and characterized a low loss reflector using a high-contrast grating.

It was first shown by Mateus [1] using a system of poly-Si gratings (n = 3.48) on top of SiO$_2$ (n = 1.4) that high-contrast-gratings could function as broadband and lossless reflectors. “High-index-contrast” refers to the large difference between the refractive indices of the grating and surrounding media. Figure 1 shows a schematic of a high-contrast-grating with dimensions and polarization defined as well as a corresponding fabricated grating. The parameters necessary to optimize the response are the grating period (A), fill factor (FF), which is the ratio of a grating’s width to the period, and thicknesses of the high and low index layers, (t$_h$ and t$_l$).

Experimental Procedure:
We optimized the design of a silicon (n = 3.4) grating suspended in air (n = 1) using commercial finite element analysis software for transverse magnetic (TM, electric field directed along the grating periodicity) polarized light. A single grating period was used as the computational domain assuming periodic boundary conditions and material optical properties from Palik [2]. Iterative optimization showed an expected reflectance greater than 90% between 8 µm and 12 µm, an important spectral range for thermal imaging.
We used a commercial silicon-on-insulator wafer as the platform for fabricating our suspended grating using photolithography and reactive ion etching (RIE). Subsequent wet etching of the sacrificial SiO$_2$ layer with hydrofluoric acid suspended the gratings as shown in Figure 1b. We characterized the grating response using polarization dependent Fourier transform infrared spectroscopy (FTIR) in transmission mode using an aperture to constrain the focused light.

Results:
We compared the sidewall profiles of a fluorine-based RIE chemistry ($\text{C}_4\text{F}_8/\text{SF}_6$) to a previously used bromide-based etch chemistry ($\text{H}_2/\text{HBr}$) using scanning electron microscopy (SEM). The fluorine-based plasma etching improved sidewall profiles over the bromide-based chemistry, as shown in the micrographs of Figure 2. The sidewalls produced by the $\text{C}_4\text{F}_8/\text{SF}_6$ RIE are more vertical and have minimal bowing compared to the $\text{H}_2/\text{HBr}$ profiles, both of which are expected to improve the structure response. We further characterized the etch rate dependence on open area of the $\text{C}_4\text{F}_8/\text{SF}_6$ RIE as shown in Figure 3, which shows larger periods, with larger open areas, exhibit higher etch rates. These results were used to optimize the grating fabrication process.

FTIR characterization of our gratings indicated less than 15% of incident light was transmitted in the spectral range of 8-14 $\mu$m. The expected transmittance, obtained from simulation with the as-built dimensions of the structure ($\Lambda = 5.3$ $\mu$m, FF = 0.7, $t_s = 3.3$ $\mu$m, and $t_l = 4.3$ $\mu$m) agreed well with the experimental transmittance, indicating the reflectance may be approximated as $R+\text{Loss} = 1-T$, where the Loss term is small.

Figure 4 shows the derived reflectance and simulated response of the gratings as well as the reflectance of a control silicon wafer. The derived reflectance agrees well with the simulation further suggesting loss in the materials does not significantly affect the response. The agreement between the expected reflectance and experimental data indicate the grating acts as a broadband reflector, with greater than 85% reflectance between 8 $\mu$m and 14 $\mu$m.

Conclusions and Future Work:
We fabricated and characterized a high-contrast grating reflector that exhibits greater than 85% reflectance between 8 $\mu$m and 14 $\mu$m. We improved the fabrication procedure using a $\text{C}_4\text{F}_8/\text{SF}_6$ etch chemistry, which improved sidewall profiles over a previously used $\text{H}_2/\text{HBr}$ etch. Future work includes design and fabrication of a polarization independent high-contrast-grating consisting of a two-dimensional layout. Initial design optimization shows a response with greater than 80% reflectance between 9 and 12 $\mu$m.

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References:
Silicon Nanophotonic Add-Drop Filter 
Based on High-Q Square Resonant Cavities

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Abstract and Introduction:
Silicon nanophotonic devices can process optical signals analogously to the way in which electronic devices process electrical signals. They have applications in telecommunications and the potential to be integrated within computer chips. One key device in photonic circuits is the add-drop filter, which can be used for wavelength-division multiplexing. This filter is commonly implemented with a microring resonator placed in between two parallel waveguides. When light passes through the transmitting bus, the ring resonates at a certain wavelength, based on its geometry. This mode propagates around the ring and into the receiving waveguide. The modal power fraction of light transmitted from the input “port” to each of the output ports, i.e. waveguides’ cross-sections, is of interest. Figure 1 shows the through and drop port responses of a microring resonator add-drop filter. A similar response can be achieved with a two-square cavity geometry [1]. In this design, the optical excitation alternates back and forth between the two square cavities in time. This “push pull” behavior means that these standing-wave cavities channel light as does a traveling-wave ring resonator. The goal of this project was to fabricate a two-square cavity add-drop filter.

Procedure:
A silicon-on-insulator wafer was used to fabricate the photonic structures. The wafer consisted of 220 nm of silicon atop 3 μm of silicon dioxide. A 190 nm layer of hydrogen silsesquioxane (HSQ), a negative tone resist, was spun onto the wafer at 1000 RPM for one minute. The wafer was dried in a vacuum oven for five minutes to remove the HSQ’s solvent. Electron-beam lithography (EBL) was performed using a JEOL 5910 LV scanning electron microscope (SEM) that interfaces with a computer program to write the desired pattern in a single writing field. The design was created in DesignCAD based on the results of two-dimensional finite-difference time-domain (FDTD) simulations.

Various electron-beam dosages were given for the different structures in the design. The sample was then developed in tetramethyl ammonium hydroxide (TMAH) heated in a 40°C water bath and then in a solution of 1:9 TMAH:water, also heated to 40°C in a water bath.
**Process Development:**

The HSQ development procedure was changed during this project to fabricate high-contrast structures. Previously, the HSQ was pre-baked at 90°C before EBL to dry the solvent from the solution. After EBL, the sample was developed in the two solutions of TMAH at room temperature. However, polymer chains within the HSQ cross-link when it is exposed to elevated temperatures [2], producing areas of partially developed HSQ near exposed areas. The procedure was improved by drying the solvent at room temperature and development at 40°C. Heating during development removed partially developed HSQ caused by electron beam backscattering.

Another challenge in fabrication were proximity effects from EBL, which are caused by backscattering of secondary electrons that bombard the sample. The proximity effects were mitigated by varying exposure dosages and by adding sidewalls near the gratings to create approximately “even loading” (average dosage) across structures. However, the equipment seemed to give inconsistent exposures, and exact numbers were not calibrated for the electron-beam dosage.

**Results:**

A grating-to-grating connection via a waveguide was fabricated to obtain a baseline test for the optical fiber-to-fiber power loss. The device’s gratings were designed to couple 1550 nm wavelength light into the waveguide. The laser’s wavelength was swept from 1495-1640 nm, and the power transmission along this range is shown in Figure 3. Transmission of about -50 dB was achieved, but high coupling loss remains. The response shows a deep modulation with a 21 nm wavelength period, which corresponds to a free spectral range (FSR) of 2.6THz. This could be the result of coupling between the fundamental and third modes within the taper, coupling from a transverse electric (TE) to a transverse magnetic (TM) mode, or mode conversion from TE to TM in the waveguide bends. There is also a secondary ripple with a 0.5 nm period, which could be caused by a reflection between the input and output terminals, with sufficient resonant delay in reflection by the grating couplers.

A microring resonator was fabricated for testing, because its response is well known [3] (see Figure 4). This device gave no meaningful optical response. SEM images showed inconsistent structure heights, which would have caused high power loss if light had been coupled into the device.

**Conclusions:**

A photonic fiber-to-fiber connection, comprising two fiber-to-chip grating couplers and a silicon waveguide connection on a silicon chip was demonstrated.

To complete the sought goal, the next step would be to fine tune the fabrication process until a circuit with a working microring resonator add-drop filter is produced. Then, a three-dimensional mode solver software would be used to find the square resonant cavities’ dimensions with a high quality factor at a decided wavelength of light. The two-square add-drop filter would be tested optically, and these measurements would be compared with the theoretical response produced by FDTD simulation.

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**References:**


Voltage-Tunable Plasmonic Metamaterials Based on Stark Tunable Intersubband Polaritons

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Abstract:
This paper explores ways of producing voltage-tunable plasmonic metamaterials. By combining plasmonic metamaterials with intersubband transitions in In$_{0.53}$Ga$_{0.47}$As/Al$_{0.48}$In$_{0.52}$As coupled quantum well heterostructures, we fabricated and tested devices with optical response sensitive to applied bias voltage. The metamaterials consisted of an array of sub-wavelength plasmonic elements resonant in the mid-infrared. The plasmonic structures were fabricated using electron-beam lithography. The devices were characterized using Fourier transform infrared spectrometer (FTIR)-based reflection measurement. Resonance matching between intersubband transition and plasmonic resonances was confirmed by observing polaritonic splitting of absorption peaks in reflection spectrum. Experimentally, applying 5.5 volts of DC bias achieved 71 nm of wavelength tuning at 7 µm wavelength.

Introduction:
Plasmonic metamaterials are artificial materials constructed on the sub-wavelength scale to provide electromagnetic properties that cannot be obtained by naturally occurring materials. Our goal was to produce voltage-tunable plasmonic metamaterials by combining plasmonic metamaterials with artificial semiconductors designed for electro-optic effect. The tunability was based on the integration of intersubband transitions, which occurred from excitation of an electron between quantized energy levels within a multi-quantum-well (MQW) [1], and plasmonic absorption from surface plasmon polaritons (SPP’s), which occurred when infrared waves struck a metal and caused electron oscillations [2]. Intersubband transitions were designed to have diagonal transitions in real space in which sharp absorption allowed for changes in permittivity in the direction normal to the surface based on the Kramers-Kronig relation for transverse-magnetic (TM) polarization; see Figure 1(c). Bias voltage tunes the position of intersubband absorption lines; see Figure 1(a), (b). With proper planar configuration, we could apply bias through the MQW layer and utilize $\varepsilon_z$ for tuning. The bias voltage led to significant tuning of absorption peak position in the plasmonic absorption spectrum.

Our device confirmed the potential for spectral tuning in the mid-infrared range with bias voltages, known as the Stark Shift. We modeled a unit cell of our structure with Computer Simulation Technology (CST) Microwave Studio; see Figure 2(a). The structure consisted of a MQW layer positioned between bottom and top metal layers. The plasmonic resonance wavelength was based on the geometry of the elements and dimensions of the resonator. Band structure engineering determined the transition energies and operating wavelengths. Since the refractive index of the surrounding environment of the plasmonic metamaterial changed, there was a resultant shift in the resonance frequency; see Figure 2(b). The resonance frequency of the plasmonic metamaterial and multi-quantum-well structure must coincidently align to achieve maximal tuning of the plasmonic resonance.

Figure 1: Intersubband transition simulation when the device is biased at (a) 0V and (b) 1V. (c) Calculated real part of dielectric constant at different bias voltage.

Figure 2: (a) Device structure designed in CST Microwave Studio composed of complementary cross resonators. (b) Simulated plasmonic absorption tuning at different bias voltages.
Experimental Procedure:
We fabricated a 400 µm by 400 µm array of complementary cross-shaped resonators on a 200 nm-thick MQW layer. The MQW structure was grown by molecular beam epitaxy (MBE) on an n-doped InP substrate. After wafer bonding, polishing, and wet etching, electron beam lithography (EBL) was used to fabricate the plasmonic structures. The minimum feature was 214 nm, and the maximum was 1.544 µm. Titanium and gold were deposited onto the sample’s surface, and lift-off was performed.

Figure 2 (a-c) show scanning electron microscope (SEM) images of the cross-shaped pattern and finalized device. A mesa-structure was necessary to prevent current spreading away from the plasmonic metamaterial. The samples were cleaved and mounted to a copper carrier block. Using a manual tool, we created multiple wire connections to bottom and top metal contacts for current flow. We performed several tests to obtain a close approximation to the designed structure, including adjusting the dose factor of the electron beam and changing the dimension of the pattern file during e-beam writing.

The FTIR characterized the finished device with spectral measurements. A broadband mid-infrared light from a globar lamp inside the FTIR was focused onto the plasmonic array via objective lens, and the reflected signal was measured through a beam splitter and ZnSe lens with a liquid nitrogen cooled mercury cadmium telluride (MCT) photo-detector.

Results and Conclusions:
Resonance matching between intersubband transitions in the MQW and plasmonic resonances was confirmed by observing polaritonic splitting of absorption peaks, which occurs when two new energy states are effectively constructed as shown in Figure 4(a). The evident trend proved that as the period of the unit cell increased, the resonance wavelength increased. We were able to see that the polaritonic splitting of the absorption occurred with a period around 1.8 µm. We applied tuning to the structure with matched resonance of period 1.75 µm. We achieved 71 nm of wavelength tuning at 7 µm wavelength with 5.5 volts of DC bias. We observed plasmonic absorption tuning through a quantum-confined Stark Shift by applying bias through the MQW; as the voltage increased, the absorption peak spectrum shifted to longer wavelengths as shown in Figure 4(b).

Future Work:
Adjusting the structure to have a plasmonic absorption line closer to the intersubband absorption line of the MQW may broaden the tuning range. Technologies including spectrometry, photo-detectors, and quantum cascade lasers immensely benefit from such insights.

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References:
Silicon Nanowires for Optical Light Trapping in Solar Cells

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Introduction:
The advanced development of the semiconductor industry has placed crystalline silicon solar cells at the forefront of solar cell research. Currently, crystalline silicon solar cells (c:Si SCs) average around 20% efficiency in the conversion of sunlight into electricity [1]. Control of the surface morphology of solar cells is key to boosting the conversion efficiency beyond the current average through the reduction of reflectance and the enhancement of electrical performance. Inexpensive surface texturing is commonly used to minimize reflection losses; however, this method is dependent on the refractive index of the semiconductor and quickly reaches a maximum. Nanowires with sub-wavelength dimensions (~ 100 nm) offer great potential for decreasing reflectance beyond the bulk limit. These attributes are shared with other sub-wavelength scaled features, but nanowires offer the unique ability for the separation of light absorption and carrier transport through the confinement of electrons in two dimensions [2].

The approach used in this experiment to fabricate nanowires on silicon devices allowed for well-controlled nanowire structures. Using electron beam lithography (EBL) and deep reactive ion etching (DRIE), highly ordered nanowire patterns were produced on doped c:Si wafers. With additional processing, the resulting silicon nanowire solar cells were characterized optically and electrically. A schematic diagram of the finalized device structure is shown in Figure 1.

Experimental Procedure:
Nanowires were fabricated on back-side junction c:Si SCs with amorphous silicon (a:Si) passivation and aluminum contacts. To begin the fabrication process, a backside junction was formed on p-type c:Si wafers. This was done using backside diffusion of phosphorus at 1000°C for one hour. A drive in stage was performed after diffusion for seven hours at 1000°C. The front side of the wafers was coated with PMMA photoresist and patterned using EBL, forming exposed circular areas of 70 nm and 100 nm diameter after development. To form a hard mask for etching, 30 nm of silicon dioxide (SiO2) was deposited with electron-beam evaporation, leaving a grid pattern of SiO2 islands with 700 nm spacing.

DRIE etching was done using a Surface Technology Systems inductively coupled plasma (STS ICP) system. Highly anisotropic nanowires of diameters 70 nm and 100 nm, and a height of around 600 nm, were formed using the Bosch process (shown in Figure 2), which consists of alternating cycles of etchant sulfur hexafluoride (SF6) and deposition polymer octafluorocyclobutane (C8F8). This method inherently creates scalloping on the sides of the nanowires.

A passivation layer was deposited using PECVD, consisting of a 10 nm intrinsic layer of a:Si, followed by 30 nm of p⁺ a:Si. This increased the diameters of the nanowires to 150 nm and 180 nm. Electron-beam evaporation was used to deposit 200 nm of aluminum (Al) on the front and back sides of the wafer. A shadow mask was aligned on...
the front side of the wafer during deposition to avoid contact with the nanowires. Finally the wafers were placed in an oven at 300°C for 20 minutes to allow the Al to diffuse through the passivation layer without compromising the amorphous nature of the layer.

Results and Conclusions:

Figure 3 shows the current-voltage (IV) characteristics of the nanowire devices under AM1.5 illumination. Under these conditions, the best device showed an open circuit voltage ($V_{oc}$) of 0.44 V, a short circuit current density ($J_{sc}$) of 21.6 mA/cm$^2$, a fill factor (FF) of 0.664, and an efficiency ($\eta$) of 6.43%. The control device used was a planar c:Si device located on the same wafer as the nanowire devices. The control device showed a $V_{oc}$ of 0.42 V, $J_{sc}$ of 14.1 mA/cm$^2$, FF of 0.555, and $\eta$ of 3.48%. Light trapping in the nanowire structures increased the absorption of incident photons over the planar device.

Reflectance measurements were taken of 150 nm and 180 nm diameter nanowire patterns, and textured Si using a Filmetrics F40 microscope attachment, and a reference was used for planar Si. The results are shown in Figure 4. Nanowire devices showed higher absorption than planar Si, but textured Si showed a higher total absorption than the nanowire devices. Absorption peaks were observed at 700 nm wavelength for 150 nm diameter nanowires and at 800 nm wavelength for 180 nm diameter nanowires. The nanowire structures showed higher absorption at the observed peaks than textured Si. A red shift in absorption peaks with increased diameter shows tunability of the absorption peak through the diameter of the nanowires. Similar results were observed in indium arsenide and indium phosphide nanowires by Kupec [3].

Future Work:

Further research is needed to optimize the fabrication process so that control devices perform at a higher efficiency. Studies on the effects of nanowire diameter, spacing, and structure on absorbance must be performed to allow for optimization of nanowire structures. Eventual applications may focus on radial p-n junction nanowires and nanowire thin film devices using similar fabrication methods.

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References:

Abstract and Introduction:
We explored the tunable optical properties of III-V compound semiconductor films grown by molecular beam epitaxy (MBE) for infrared optoelectronics and integrated plasmonics. This project has been twofold. (1) Investigate the optical tunability of MBE-grown rare-earth monopnictide (RE-V) nanoparticle superlattices by adjusting superlattice period and composition to demonstrate intermediate energy states that enable sub-bandgap tunability of gallium arsenide (GaAs). (2) Study the absorption blueshift in highly-doped indium arsenide (InAs) to demonstrate an approximately three-fold increase in the effective bandgap range and the divergence from existing non-parabolic InAs electronic band structure models. Both systems studied demonstrate two separate methods to tune III-V materials to provide greater material flexibility for optical engineering applications.

Material Systems Studied:
RE-V/III-V superlattice structures consist of periodically repeating layers with a 5 to 20 nm thick GaAs layer and a 0.25 to one monolayer (ML) deposition of RE-V, where the fractional volume of RE-V is equivalent in each structure. We investigated erbium arsenide (ErAs), lanthanum lutetium arsenide (LaLuAs), and lutetium arsenide (LuAs) RE-V nanocomposites. Work by Hanson et al. [1] on similar RE-V/III-V superlattice structures demonstrated strong sub-bandgap absorption in gallium antimonide (GaSb). We sought similar absorption changes in GaAs as our first method to optically engineer III-V materials.

InAs is a direct, narrow-bandgap semiconductor with a room temperature bandgap of 0.33 eV. We studied 500 nm thick films doped with silicon (n-type) and beryllium (p-type) with high active carrier concentrations of $6 \times 10^{19}$ to $9 \times 10^{20}$ cm$^{-3}$. High n-type carrier concentrations, $n$, resulted in a strong absorption blueshift, which is attributable to band-filling, as described by Burstein [2] and Moss [3]. As $n$ increases, the Fermi energy increases and more states in the conduction band, Ec, become filled with electrons. Since photoelectric absorption from the valence band, Ev, requires an unfilled state in the conduction band, the effective bandgap, $E_{gap,eff}$, increases with the Fermi energy, as shown in Figure 1. This study investigates unexplored high $n$ values to tune $E_{gap,eff}$ in InAs as our second method to optically engineer III-V materials.

Experimental Procedure:
Near- and mid-infrared reflection and transmission spectra were collected with a 0.5 m grating spectrometer, using an InGaAs detector and a tungsten-filament source for the near-infrared, and an indium antimonide detector and carbon-rod source for the mid-infrared. Overlapping wavelength spectra were collected using LabVIEW and spliced using MATLAB, resulting in single spectra spanning wavelengths from 800 to 5000 nm (photon energies from 0.2 to 1.4 eV). The $E_{gap,eff}$ for different materials was found by using a linear extrapolation of the square of the absorption coefficient for a thin-film material, as suggested by Wu et al. [4].
Results and Conclusions:

RE-V/III-V Nanoparticle Superlattices. An aggregate transmission spectrum of several representative RE-V/III-V samples is shown in Figure 2. From 0.2 to 1.4 eV, GaAs exhibited a constant reflection value, thus any change in the transmission spectrum produced an opposite change in the absorption spectrum (e.g., decreased transmission produces increased absorption). Nanocomposites with Er and LaLu, small nanoparticles (small ML), and a small number of periods showed a small dip in transmission for incident photonic energy greater than 0.75 eV. Nanocomposites with Lu, large ML, and many periods showed a more pronounced transmission spectrum dip near 0.8 eV, which is far below 1.4 eV bandgap for intrinsic GaAs. RE-V nanoparticles create sub-bandgap energy states (Figure 3) that allow for sub-bandgap photoelectric absorption [1]. By adjusting the composition and structure of superlattices we can engineer GaAs for absorption near 0.8 eV. This value is critical for fiber optic systems, because 0.8 eV (1550 nm) is used by the existing fiber optic infrastructure for optimal signal relay and acquisition; III-V-based detectors using RE-V nanocomposites may be quickly adopted to work with existing technology. Future work will explore other RE materials and adjusted MBE growth techniques to refine the tunable properties of RE-V/III-V nanocomposites.

Heavily-Doped InAs. A plot of $E_{\text{gap,eff}}$ versus $n$ is given in Figure 4. We observed a tunable $E_{\text{gap,eff}}$ range of 0.64 to 1.16 eV, which is several times larger than the intrinsic bandgap of InAs of 0.33 eV and indicates InAs absorption may be engineered by controlling $n$. The data agreed well with previously published results at lower carrier concentrations and diverged noticeably from the best fit non-parabolic band model available [5, 6]. Although non-parabolic behavior is expected for large $n$, modeling such behavior is limited by low $n$ values previously explored. Our larger-$n$ trend describes non-parabolic behavior more completely and can be used to study and optically engineer narrow-bandgap materials. Future work will concentrate on quantifying a new non-parabolic model and exploring even higher $n$.

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References:
Stabilizing Fiber Optic Pressure Sensor Measurements by Fabricating an Enclosed Photonic Crystal Cavity

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Abstract:
Fiber optic pressure sensors are optical microelectromechanical systems (MEMS) devices used in a variety of industrial applications. Current sensors are assembled by affixing a silicon photonic crystal (PC) chip near an optical fiber-tip with a glass ferrule, which forms a Fabry-Perot (FP) cavity. However, environmental factors beyond human control decrease the sensor’s overall measurement stability by inducing fluctuations in the cavity length. A potential method to reduce this variability is to incorporate the FP cavity construction into the fabrication stage of the PC itself. To accomplish this, a silicon wafer was patterned and etched downward to form PCs, which were etched radially outward to create overlapping spherical cavities. Silicon oxide was deposited by low pressure chemical vapor deposition (LPCVD) to seal off the air cavities, after which their reflectivity was measured. Many PCs were fabricated with different diameters to determine the optimal parameters that will yield the highest PC reflectivity. Based on our measurements, PCs with higher photolithographic exposure (larger diameters) are optimal. By successfully implementing this design into the fabrication process, this compact pressure sensor’s measurement reproducibility will significantly improve for its industrial applications.

Introduction:
Fiber optic pressure sensors are used for their compactness and measurement sensitivity. Current sensors are comprised of a silicon PC chip near an optical fiber-tip, which forms the FP cavity, a cavity between two highly reflective mirrors (see Figure 1(a)). These sensors function by propagating light through the fiber, which is partially reflected from both mirrors. These reflections interfere to create the total optical reflected power as a function of wavelength (see Figure 1(b)).

However, because the FP cavity is usually assembled after fabrication, environmental factors decrease the sensor’s overall measurement stability by fluctuating the cavity length during periods of constant applied pressure. To reduce measurement variability and increase measurement stability, a procedural modification was developed and implemented where an external FP cavity is constructed and sealed during the fabrication stage of the PC itself, which is confirmed by the scanning electron microscope (SEM) and PC characterization.

Photonic Crystal Fabrication Process:
To investigate the parameters that affect the reflectance performance of the FP cavity, three initial silicon wafers were prepared by undergoing oxidation. They went through photolithography, which involved patterning and developing the mask design of 2D PCs. For each PC, a square lattice of circular holes spans over 500 µm by 500 µm. The three wafers were uniformly exposed to 180 mJ/cm², 210 mJ/cm² and 240 mJ/cm², respectively. Once patterned and developed, they were anisotropically and isotropically etched to form overlapping spherical cavities that form the PC cavity. To seal off the finished cavity, silicon oxide is deposited via LPCVD (see Figure 2).

Results and Discussion:
In Figure 3(a), the oxide deposition mostly sealed the 240 mJ/cm² PC holes from the 710 nm pre-deposition diameter to the 95 nm remaining hole, but is capable of completely sealing the holes given a longer deposition. The low exposure post-
deposition image (not displayed) had a remaining blur, suggesting that those holes were successfully sealed with a substantial oxide layer on top. This is confirmed in the circled portion of Figure 3(c) that indicates the 180 mJ/cm² holes closed as opposed to the 240 mJ/cm² holes. The stress that the silicon PC chip endured from oxide deposition caused the evident “buckling” (i.e. bending) effect in Figure 3(d) because of the compressive stress that the silicon oxide imparts on the silicon. The formed FP cavity has varying lengths across the entire cavity, which made the light behavior and ultimately the reflectance power spectrum inconsistent.

The freespace reflectances were measured by focusing a laser onto the PC chips and coupling the reflection into an optical spectrum analyzer. In Figure 4, each of the four curves is the reflectance recorded from four different locations on a single photonic crystal, which illustrates the inconsistent reflectance measurements observed for any PC at any of the given exposure levels by the evident dissimilar curves and reflectance values across each curve. Even though there is high measurement variability amongst the reflectances for a given photonic crystal, the higher exposure PCs have better reflectances.

Comparing the lowest and highest exposure photonic crystals (180 mJ/cm² and 240 mJ/cm²), their reflectances reached up to 21% and 80%, respectively. The higher photolithographic exposure produced a better photonic crystal reflectance most likely due to the larger holes.

Conclusions:

A fabrication method was developed that successfully seals the FP cavity, as indicated by SEM imaging. We found there is a direct relationship between the PC’s reflectance and the photolithographic exposure, despite the “buckling” effect. While the low exposure PCs exhibit higher sealing capability, the high exposure PCs exhibit higher reflectance, which allows for sensors with higher sensitivity. For future work, we recommend performing pressure sensing measurements and finding methods to handle the silicon oxide “buckling.”

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References:

Abstract:

Stacked bioreactors are built using slab-waveguides. Light is directed into the side of the waveguide and inside the light totally-externally reflects. Using photolithography, pillars of SU-8 photoresist, with a higher index than glass, were patterned on a waveguide. SU-8 pillars acted as a mechanism to scatter light into the reactor [1]. Experiments using florescent dyes, simulating a thin biofilm, were conducted to determine the rate at which the light intensity decreases at maximum SU-8 patterning density. The decrease was fit to an exponential and used to generate a gradient of coverage along the length of the reactor required for uniform scattering light distribution. 

Experimental Procedure:

Waveguides were made by applying SU-8 2002 photoresist (Microchem) on the surface of borosilicate glass slides with dimensions of 75 mm × 50 mm × 1 mm or 60 mm × 24 mm × 0.15 mm. The 25% uniform coverage waveguides were made by patterning an array of 5 µm by 5 µm pillars spaced 5.0 µm apart on the glass (Figure 1). Gradients of different percent coverage at each mm along the glass were constructed by changing the spacing between the pillars.

Introduction:

Cultures of algae, such as the cyanobacteria Synechococcus elongates, grown in bioreactors are a promising source of renewable energy [2]. However, algae growth is highly dependent on light intensity and standard bioreactors do a poor job at distributing light uniformly for algae utilization due to shading [3]. The goal of a stacked bioreactor is to uniformly distribute the optimum amount of light to all of the algae growing inside the reactor.
using $k$ (Figure 2). The gradients for the 0.15 mm and 1 mm thick waveguides started at 6.9 and 17.8 percent coverage, respectively, and increased exponentially every 1 mm, reaching a maximum of 50% coverage at the end. The highest percent coverage used was 50%; spacing the pillars less than 2.1 µm apart resulted in pillars overlapping and forming a film.

Results and Conclusions:
With 50% uniform coverage of SU-8, the maximum decrease coefficient ($k_{max}$) was -0.156 for a 0.15 mm thick waveguide (Figure 3) and -0.028 for a 1 mm thick waveguide. These negative $k_{max}$ values indicate a large exponential decrease in intensity. Our gradient of SU-8 coverage for the 0.15 mm thick waveguide improved the decrease coefficient $k_{max}$ from -0.156 to 0.001. This low absolute value of $k$ indicates no substantial change in light intensity along length of chamber. The gradient for the 1 mm thick waveguide was also successful, improving the decrease coefficient from -0.028 to -0.0001 (Figure 4). We have demonstrated a successful design for uniformly distributing light into a bioreactor.

Future Work:
Algae have been grown inside these waveguide chambers to measure growth. Florescent pictures of algae growing inside the reactor have been taken and will be analyzed to confirm uniform growth. The next step in our process will be to find a way to upscale our waveguide to the reactor scale. These manufacturing methods should allow for the waveguide to be mass produced. Once a manufacturing process has been selected, combining waveguide technology with nutrient delivery technology would result in a high efficiency bioreactor.

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References:
XPS Analysis of Sulfide and Aluminum Nitride Based Buffer Layers for Epitaxial Zinc Oxide Growth

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Abstract:
Thin films of non-polar MnS and AlN/MnS were grown on Si <100> by PLD. The chemical compositions of the samples were investigated by angle-resolved XPS. It was found that AlN serves as a barrier to preserve MnS films from oxidation. It was also found that Mn-Si bonds existed on the surface of Si, while Si-S bonding was not observed. Finally, a native SiO_x layer was observed to diffuse to the surface of the sample, residing on top of epitaxial AlN. It is also likely that a thin Al_2O_3 layer existed alongside SiO_x.

Introduction:
Group III-V nitrides have become vital in the advancement of short wavelength opto-electronic devices. Using these materials, devices like the blue-green light-emitting diode (LED) have seen rapid development in recent years. For these devices to function at their maximum potential, epitaxial gallium nitride (GaN) films are necessary. More specifically, epitaxial growth of GaN on silicon (Si) <100> is necessary for integration of these devices in widespread technology. Unfortunately, there has been some difficulty in growing polar GaN <0001> on Si <100> substrates. In addition, the polar nature of GaN <0001> reduces the quantum efficiency through spontaneous and piezoelectric polarizations. Electrostatic fields that arise from these effects located at the interfaces of the heterostructure lead to band bending and separation of electron-hole pairs, as shown in Figure 1.

In order to minimize these effects, work has been done to grow epitaxial non-polar <11-20> GaN on Si substrates. This goal was realized using manganese sulfide (MnS) <100> and aluminum nitride (AlN) <11-20> as buffer layers [1]. Work was done in this study to examine the chemical states that exist in these buffer layers.

Experimental Procedure:
Si <100> substrates were first cleaned by hydrogen fluoride (HF) etching to remove the native silicon dioxide (SiO_2) layer. MnS was deposited using pulsed laser deposition (PLD) using a substrate temperature of 700°C and P = 3 × 10^-7 torr to ensure clean MnS films. The film was then post-annealed for 30 minutes at 700°C and P = 2 × 10^-7 torr to further improve the film quality. AlN was then deposited using PLD at a temperature of 700°C and P(N_2) = 1 × 10^-4 torr, with a post anneal time of five minutes at 700°C. Analysis of the films was performed using angle-resolved x-ray photoelectron spectroscopy (XPS). Due to the shallow penetration depth of XPS, the thickness of MnS and AlN films were kept between 2.5-5.0 nm.

Results:
Figure 2 shows XPS results of the Sulfur 2p peak. Comparison of the left graph (2.5 nm MnS / Si) to the right graph (2.5 nm AlN / 2.5 nm MnS / Si) demonstrates that AlN acts as a protective layer for MnS against oxidation and allows for epitaxial MnS growth. Figure 3 shows XPS results of the Manganese 2p peak. The shoulder in the right graph is strongly indicative of Mn-Si bonding. In XPS larger angles correspond to information about deeper in the sample, so the sharp 90-degree peak and absence of a 30-degree peak means that Mn-Si bonding is located against the substrate.

Figure 1: Electrostatic fields reduced in non-polar structure, resulting in reduction of bend bending and higher quantum efficiency [2].
This result demonstrates that only Mn interacts with the Si substrate, while sulfur does not. While it is unclear whether there is a MnSi layer or if Si is simply bonding to Mn at the Mn/Si interface, Mn-Si bonding was clearly shown.

In addition, XPS analysis of the Silicon 2p peak (not shown) provided evidence that SiO$_x$ is present in the structure. The angular resolution of the measurements showed that SiO$_x$ diffuses to the surface of the thin-film structure, along with a probable monolayer of Al$_2$O$_3$. Examination of the aluminum and nitrogen peaks showed that AlN was epitaxial. These findings led us to model the buffer layer structure as shown in Figure 4.

**Conclusions:**

It was found that AlN serves as protective barrier for MnS against oxidation and that a thin layer of AlN allows for epitaxial MnS growth. From the Manganese 2p spectra, it was found that Mn-Si bonding is formed at Si/MnS interface while no S-Si bonding was formed. Lastly, SiO$_x$ and Al$_2$O$_3$ (likely) exist at surface of structure. It was found that SiO$_x$ diffuses to the surface of AlN regardless of the thickness of AlN.

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**References:**


Influence of the Type of Wetting Layer on the Contact Resistance in Metal-Graphene Ohmic Contacts

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Abstract:
In recent years, graphene has received a lot of attention because of its unique features, such as its high mechanical strength, its unique optical properties and the ultrahigh electron mobility. These unique properties promise a wealth of novel applications and devices. For example, graphene-based ultrathin and ultra-broadband electro-optic modulators, which employ the electrostatic-doping-induced transparency of graphene to create a voltage-controlled optical absorption, have enabled a variety of new applications. The ultra-high carrier mobility also promises transistors with switching speeds well in the terahertz range. However, we must take the evil with the good, as fabricating devices that rely on a single molecular organic layer can be very challenging. One of the main challenges is to fabricate ohmic metal contacts on graphene, as the density of states near the Dirac point is low. High contact resistance in such thin devices can severely degrade the performance. In this study, we focused on the influences of wetting metal layers and doping of graphene on the metal-graphene contact resistance.

Introduction:
Electronic and optoelectronic devices exhibit parasitic capacitance, which, if paired with a series resistance, limits the useful bandwidth of a device. To fabricate ohmic contact on semiconducting materials, it is necessary to avoid a Schottky potential barrier by choosing metal that allows the majority carriers to flow freely in and out of the semiconductor. For graphene, which is typically hole-doped in ambient conditions, this implies that the work function of metal should be higher than the energy gap between vacuum and graphene’s Fermi level. In this work we investigated the influence of the contacting metal on the series resistance in graphene devices.

Experimental Procedure:
As shown in Figure 1, silicon wafers were used as both the substrate and the back-gate electrode. A 260-nm-thick aluminum oxide (Al$_2$O$_3$) layer was deposited on the wafer by DC reactive sputtering (Target: Al metal, Pressure: 3 mTorr, Argon flow: 32 sccm, Oxygen flow: 1.6 sccm, Deposition rate: 9 nm/min). The breakdown voltage of the Al$_2$O$_3$ was measured to determine the allowable range of gating voltages. Monolayer graphene — synthesized by chemical vapor deposition on copper — was then transferred onto the dielectric and patterned by a reactive ion etch. To investigate the effects of adhesion layer on the contact resistance, three metals (10 nm Cr, Ni, and Ti) were used in combination with Al (100 nm). These metal electrodes were thermally evaporated and patterned by a lift-off process.

To measure the contact resistance, a four-point probe method was employed: four electrodes were used to measure the sheet...
resistance of the graphene between the inner electrodes (see Figure 2, B and C). Then the two probes were moved from A and D to the inner two contacts (B and C), to measure the total resistance between the metal and the graphene at the contacts. B and C was then given by the difference between these two measured values. Compared to a transmission line method, our method was much less subject to cracks in graphene, and individual pairs of contact resistance could be measured instead of an averaged value.

Results and Discussion:
Without annealing, the DC sputtered Al₂O₃ layer showed very high leakage currents (see Figure 3a). After annealing the oxide layer at 500°C in H₂ and N₂ atmosphere, the leakage current was significantly reduced and the breakdown voltage was measured to be ~280 MV/m.

Figure 3b shows the dependence of the sheet resistance on the length/width ratio of the graphene. From the slope of the line-fit to the data one can infer that the sheet resistance of the un-gated graphene is 12.8 ± 0.5 kΩ/µm.

Figure 3(c) shows the dependence of the sheet resistance on the gating voltage. Field doping effectively changes the sheet resistance between 5 and 28 kΩ/µm, for gate voltages of the order of ±50 V, respectively. The carrier density was calculated to be 9.03 × 10¹² cm⁻², assuming that the Dirac point is near 55 V.

Figure 3(d) shows the influence of the choice of the metal adhesion layer on the contact resistance. Nickel showed the lowest contact resistance (8.7 ± 1.8 kΩ•µm) of the three, while titanium lead to the highest contact resistance (~36.6 ± 4.7 kΩ•µm). Our result is in agreement with the result reported by Giovannetti et al. [1].

Conclusions:
We have shown that aluminum oxide can be deposited by reactive sputtering and, when annealed, can serve as a good dielectric for electric field strength up to 280 MV/m. There was a clear influence of the wetting metal on the contact resistance in the fabricated devices. In particular we found that nickel (ϕ ~ 5.2 eV) provides the lowest contact resistance (~ 8.7 ± 1.8 kΩ•µm) and titanium (ϕ ~ 3.8 eV) led to the highest contact resistance (~36.6 ± 4.7 kΩ•µm). Our result is in agreement with the result reported by Giovannetti et al. [1].

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References:
Graphene-Based Electro-Optic Modulator Fabrication

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Abstract:
Graphene exhibits many useful properties including high carrier mobility, fast relaxation of photo-excited carriers, and carrier-density-dependent optical absorption. Because of graphene’s unique band structure, an external electric field can be used to change its carrier density and thus its optical absorption. Graphene modulators fabricated based on this principle have been employed as fast actuators to stabilize unwanted noise in lasers, paving the way to a new generation of ultra-low-noise optical frequency synthesizers. Although graphene modulators provide the above-mentioned benefits, during their fabrication it remains challenging to prevent defects introduced by chemicals, such as photoresists and developers, or processes such as sputtering and UV light exposure. In this work, we established a wet-etch fabrication procedure for patterning the metallic contact on graphene in graphene-based electro-optic modulators. Different approaches of wet-etching were investigated, which included the use of different metals.

Introduction:
Graphene exhibits strong interband absorption (~2.3%) for visible and near-infrared light. The interband transition is highly sensitive to the density of charge carriers, due to graphene’s cone-like band structure. It is possible to prohibit the interband transition at low photon energies by increasing the carrier density through electrostatic doping. To achieve this, we employed a gated graphene structure with a dielectric. By applying a voltage between the gates, we induced an electric field in the dielectric.

The thickness of such a dielectric layer was selected to have an optical thickness that was a quarter wavelength for light at 1550 nm. This maximized the interaction between incident light and the graphene on the dielectric. Modulators with a ring shape were chosen to provide a more uniform modulation over the active area. The backgate served a second function as a mirror, in our modulators.

Previously, a liftoff method was used to produce metal contacts on graphene. In this new procedure, metal covering the whole graphene surface was wet-etched. As Figure 1 shows, by using this wet-etch fabrication procedure, sources of doping and defects could be better controlled, and UV and photoresist contamination was completely eliminated. Exposing graphene to etchants, rather than photoresists and developers offered more control over potential sources of chemical contamination.

Etchants offer a wide variety of options, whereas photoresists and developers are limited to a few suitable products.

Experimental Procedure:
A 100 nm layer of aluminum (Al) was evaporated onto a silicon substrate, and subsequently patterned via wet-etch. Tantalum pentoxide (Ta$_2$O$_5$, 185 nm) was deposited via DC reactive sputtering. Ta$_2$O$_5$ features a high permittivity, transparency, and breakdown voltage. Monolayer graphene sheets were prepared via chemical vapor deposition (CVD) and transferred onto the Ta$_2$O$_5$ layer. Several different metals, of varying thicknesses, were evaporated onto the graphene, and subsequently patterned via wet-etch.

Figure 1: Differences between the previous and new fabrication process.
Results and Discussion:

There were also drawbacks to using wet-etch fabrication procedures. Photoresist adhesion to metal was a large issue. Certain etchants lifted off resist around the edge of metal structures. This effectively caused a lateral etching in excess of 30 µm in the worst cases. Adhesion to graphene was another issue; some chemical etchants caused the metal to lift off of the graphene or the graphene off of the Ta₂O₅. Backgate liftoff also occurred, as the porosity of the sputtered Ta₂O₅ layer allowed the etchant to penetrate and lift the Al backgate off of the substrate. Careful selection of metals and metal etchants, as well as annealing of the Ta₂O₅ effectively improved the quality of the devices.

Figure 2 shows functioning devices that were fabricated using this new method. The three top gates consisted of 35 nm nickel (Ni), a 10 nm Ni adhesion layer with 100 nm Al on top, and a 10 nm Ni adhesion layer with 85 nm gold (Au). The addition of the Ni layer improved the adhesion of the top gate to the graphene layer. Ni was etched with low concentration nitric acid (HNO₃), Al with Transene Al Etchant Type A, and Au with Gold Etchant Type TFA.

Conclusions and Future Work:

We were able to establish a wet-etch fabrication procedure for electric contacts on graphene without deteriorating the electronic properties of graphene. With this new procedure we fabricated functioning graphene-based electro-optic modulators. Further studies are needed to show if this wet-etch fabrication procedure really offers improvements in the electrical and optical performance of the modulators. Raman spectroscopy performed on both sets of graphene, would for instance allow one to quantify the defects created in graphene by either process. The results of such a study may be significant for any graphene-based electronic devices. These initial results, however, already show a lot of promise.

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Figure 3: Modulation depth measurement setup.

We examined the performance of the modulators using the setup shown in Figure 3. A continuous wave laser illuminated the modulator, while a sinusoidal voltage applied between the graphene and the metal mirror provoked the optical modulation. A computer-controlled x-y stage was employed to raster scan and produce a modulation heat map of the devices.

Figure 4 shows that although there were some breaks in the graphene, we were able to achieve a measurable modulation depth across a large area of nearly 1 mm in diameter. Outside of the contacting ring, no modulation was observed as the graphene was removed to reduce device capacitance.