Optics and Photonics Research for Montana Economic Development - MREDI Project

Quarter 3 Report – May 06, 2016

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Introduction

This project is on schedule and within budget, and is continuing to enable important collaboration between the optics and photonics research community and the Montana optics and photonics industry. Substantial progress is reported here and activity is ramping up for a busy summer research season. In the following pages we report specific progress toward meeting the milestones for each subproject.

Subproject 1: Ultra-compact spectral imagers for precision agriculture and mapping of wildfires and natural resources (Joseph Shaw, joseph.shaw@montana.edu with NWB Sensors, Inc.). Development of ultra-compact imaging systems for weed mapping in precision agriculture, UAV mapping of wildfires, and a wide variety of ground-based and airborne remote sensing.

Milestones

a) September 30, 2015: Initial agricultural data collection completed
b) December 31, 2015: Initial weed maps complete
c) June 30, 2016: Prepare a refined imaging system and application-specific algorithm
d) December 31, 2016: Complete results of summer 2016 harvest experiment
e) June 30, 2017: Finish imaging system and algorithms and transfer to private partner

Activities to date

Weeds typically are managed with multiple herbicide applications throughout the year. One of these applications is post-harvest, usually with a focus on quack grass and other perennial weeds. In this project we are working toward a weed mapping solution using combine-mounted imagers to allow farmers to target specific regions of their field for herbicide application and to provide an annual record of problem regions. Our approach is based on the use of low-cost, multi-spectral cameras, connected to a GPS receiver to create geo-tagged images of the grain crop and weeds during harvest. Post processing of these images produces a map showing weed detections in the field. Figure 1 is a photograph of compact imaging systems mounted in a combine during the 2015 harvest.

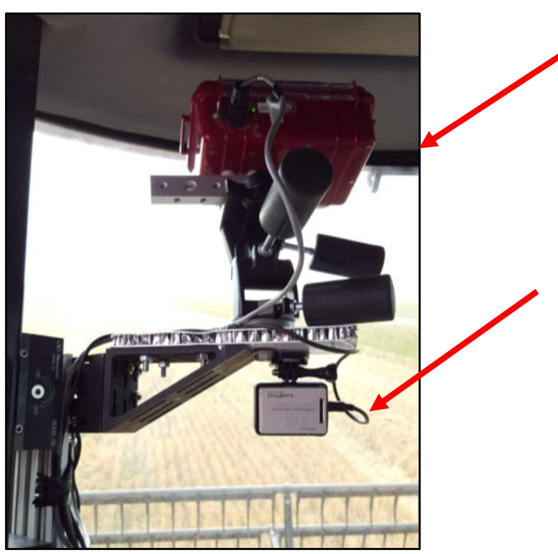

Figure 1. The Multi-spectral and GoPro cameras mounted in the combine during the August 2015 harvest.

The primary focus of the work during the third quarter of this project was refinement of the weed imaging and mapping system and the development of application-specific algorithms. The current application we are targeting is the precision application of the herbicide post-harvest in Montana grain crops. Our approach has been to use digital multi-spectral cameras connected to GPS receivers to auto-detect weeds and produce a map of the weed locations in the fields. To enable this application, our work has focused on both increasing the processing capabilities of these imagers and looking at lower-cost imaging systems.

Weed Detection with a RGB Camera

During the August 2015 barley and wheat harvest in Fairfield, MT a standard Red-Green-Blue (RGB) imager (a GoPro) was operated alongside the custom multi-spectral camera developed at Montana State University to provide photos of the field similar to what the operator would have seen during harvest. Following the initial success of the multi-spectral camera mapping, we decided to apply the same image-processing techniques to the RGB camera to determine if it also was capable of providing weed detection. The RGB cameras cost even less than our custom multi-spectral imagers, so if the machine vision algorithms could work with these cameras with similar performance to the multi-spectral camera, then they might be a promising path forward for a commercial product.

To enable weed detection with the RGB camera, a Support Vector Machine (SVM) algorithm was implemented (similar to the one used on the multi-spectral cameras). This SVM was based on standard color indices for RGB cameras and image texture features. The imagers triggered on many objects other than crop, such as people, vehicles, or the combine header. Therefore, to make the SVM classifier more robust, additional object classes were added into the training set, including lodged crop, green crop, side views of standing grain, grasses (quack grass), broad leaf weeds (milk weed), and manmade items. To make the classifier robust to natural lighting variations, the lighting conditions from cloudy skies (indirect lighting) and clear skies (direct lighting) were added to the training set. Initial analysis showed that even in two-dimensional projections of these data, separate clustering of the weeds from all other objects was observed (the weeds were identifiable and separable from the other objects). Figure 2 shows that this was consistent for clear and cloudy days.

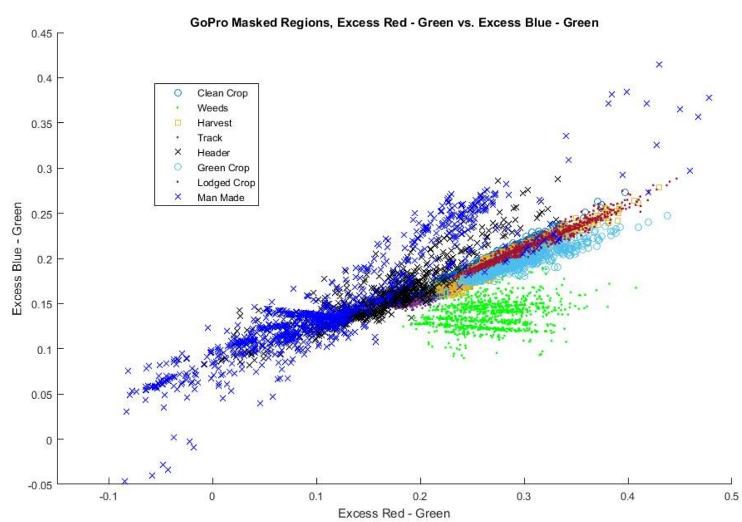

Figure 2. Two of the RGB color indices used as input parameters for the SVM used to process the GoPro images. This figure contains data from both clear and cloudy days.

Extensions to the SVM Processors

To provide the most useful post-harvest weed map, perennial grasses also needed to be identified from the annual plants. This required extensions of the SVM to not only detect weeds, but also to identify weed type. To implement this, we modified our image processing algorithms to also analyze image differences between quack grass and milk weed, two different types of weeds that often show up in grain fields. The weed detection SVM provides the information needed for our weed identification SVM to distinguish between two species of weeds.

Another modification was to identify the combine's header position so the system did not attempt to map the field when the header is raised. When the header is elevated, the harvester is generally not harvesting grain and most often is traveling either between fields or within the field over an already-harvested region. Therefore, there is no need to map the field when the header is raised.

The weed-detection, weed-classification, and header-detection SVMs are separate but analyze the images using the same set of indices (filters). This requires three separate SVMs to be applied to the data. Currently this can happen within 0.85 seconds. The output of this process for a specific image containing quack grass is shown in Figure 3.

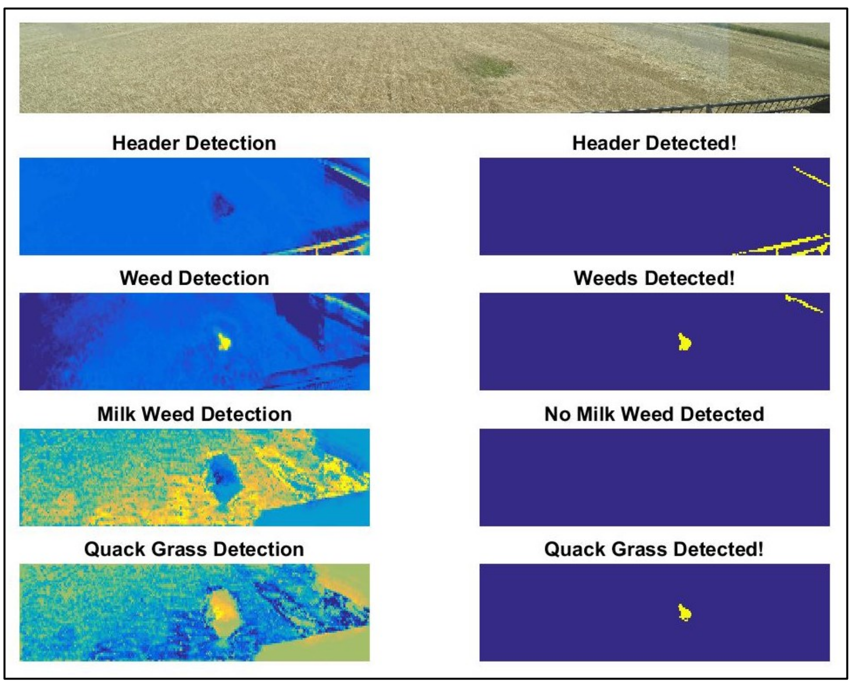

Figure 3. Output of the three SVMs, showing the detection of different species of weeds and the header in the corner of the image. In this image the weeds are correctly classified as quack grass.

Mapping with the Go-Pro Imagers and Multi-Class SVMs

Figure 4 shows the results of the multi-class SVM in the form of maps of the harvested grain fields. The maps show our ability to map weed locations in this particular field. We now have the ability to automatically determine when the header is elevated and detect and perform simple weed-type classification for milk weed and quack grass. The farmer can use these various maps to decide what the field boundaries are and also what herbicides to spray on certain weeds throughout his field.

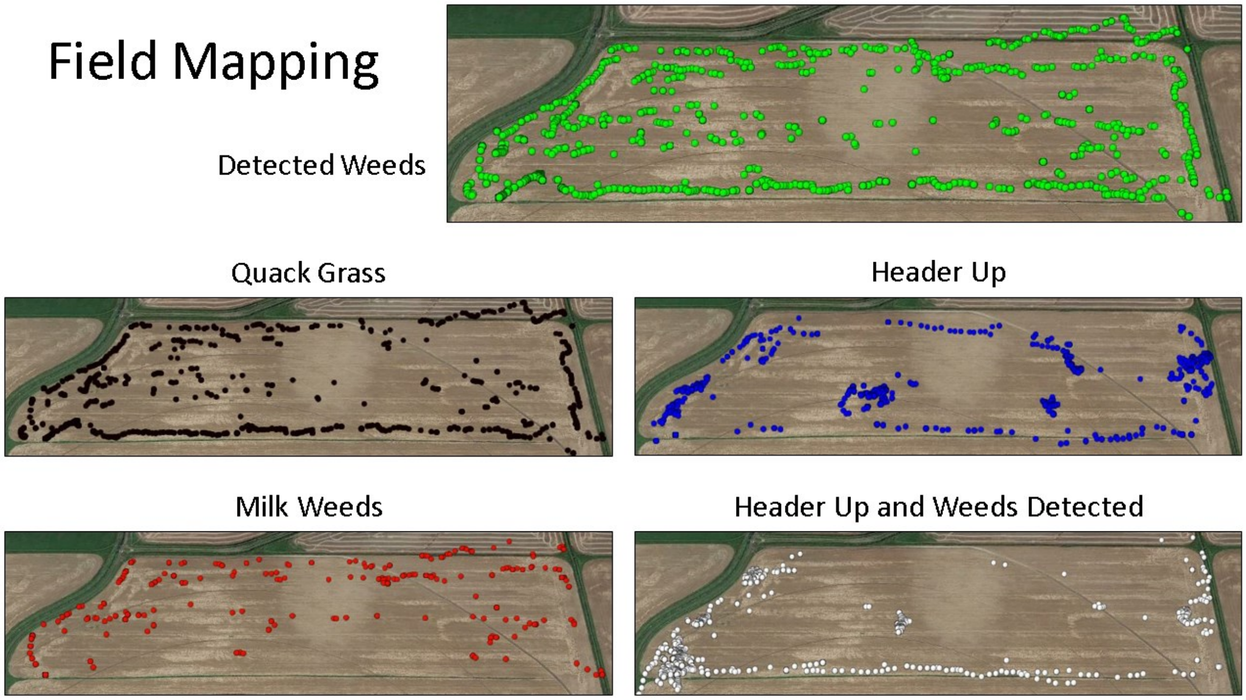

Figure 4. Maps of the harvested field, generated by the automated multi-class SVM image-processing algorithm applied to a images recorded with a commercial GoPro camera.

GPS-Enabled RGB Cameras

Following the success with applying our custom image-processing algorithms to images from the GoPro camera, we searched for a similar RGB camera that had a built-in GPS sensor. So far the best option was the Garmin VIRB XE camera. We purchased one of these cameras and have begun testing it to determine if it will provide similar classification capabilities. The Garmin camera is very similar to the GoPro in terms of its image capturing ability, but the most important additional characteristic is the GPS capability of the device. An image of this camera and GPS data are shown in Figure 5.

GPSVersionID	[2,2,0,0]
GPSLatitudeRef	'N'
GPSLatitude	[45,40,20.9425]
GPSLongitudeRef	'W'
GPSLongitude	[111,2,52.6946]
GPSAltitudeRef	0
GPSAltitude	1.4788e+03
GPSSpeedRef	'K'
GPSSpeed	35.3708
GPSTrackRef	'T'
GPSTrack	0.2307
GPSMapDatum	'WGS-84'

Figure 5. Garmin VIRB XE GPS-capable camera (left) and its GPS measurement recorded with the camera mounted on the dashboard of a car while driving at 35 km/h (right).

To ensure this camera will provide the required GPS accuracy, we performed stationary tests of the Garmin camera. The GPS accuracy needed to be less than the length of our header, which is approximately 10 m. These tests showed that this camera generated a stationary GPS accuracy of

±2.5 m, which is within our accuracy requirements. Figure 6 shows the results of these tests in latitude and longitude.

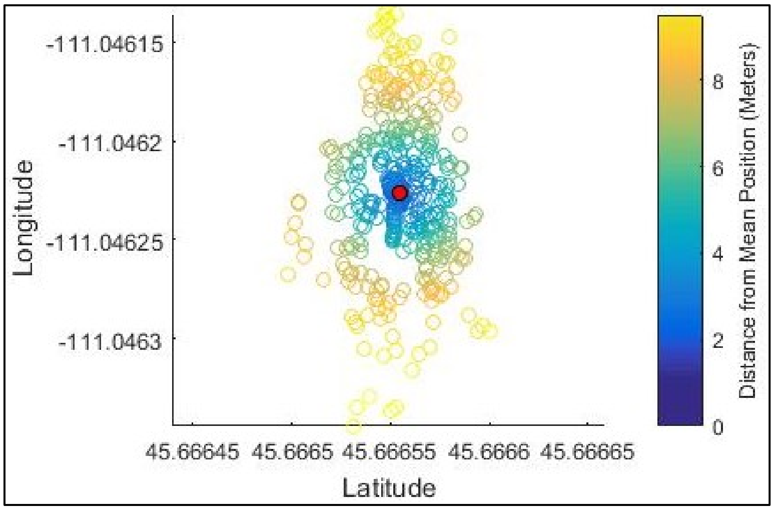

Figure 6. GPS accuracy of the Garmin VIRB XE determined in stationary camera tests.

Preparation for August 2016 Harvest Experiments.

We are now preparing for a second round of experiments during the August 2016 harvest. During the 2016 experiments we will deploy multiple systems incorporating the Garmin VIRB XE, a modified Montana State State University multispectral GPS-capable camera system, and an additional professional NDVI camera to collect data. We will use an antenna on our multispectral camera to improve the GPS accuracy during this year's harvest. For consistency, harvest and data collection will be done on some of the same fields where we collected data in 2015. For diversity, we also will collect data on some different fields as well. This will allow us to further understand the performance of these different imagers in preparation for the commercialization of the systems in the following year.

Expenditures to date:
Salary to date $56,685.01, Benefits to date $13,079.01 and Operations to date $63,500.08 total expenditures to date $133,264.10.

Subproject 2: High-performance, real-time image processing for hyperspectral imaging (Ross Snider, rksnider@ece.montana.edu with Resonon, Inc.). Design a high-speed hyperspectral waterfall sorting system to fuse object edge information with hyperspectral data to sort agricultural products quickly and efficiently using Resonon's Hyperspectral Imagers and remove rejected items via airjets. The goal is to perform the data fusion, accept/reject decision, and removal all in real-time using FPGA technology.

Milestones
a) February 1, 2016: Determination of center of mass of each food item in image/line scan
b) September 1, 2016: Determine trajectory of food item for precise timing removal
c) February 1, 2017: Integrate hyperspectral data within food item edge boundaries

d) June 31, 2017: Use hyperspectral data within food item edges to classify food item as accept/reject
e) June 31, 2017: Time air jets to remove rejected food items
f) June 31, 2017: Final report emphasizing commercial products and potential

<u>Activities to date</u>

- A senior design group that I advised and sponsored (Tyler de Caussin and Alex Matejunas) has developed a real-time algorithm to compute the center of mass of a falling object in real-time where data is coming from a line scan camera. This camera is capable of imaging 80,000 lines/second where each line is 8,000 pixels wide, which means there is 640 Mbytes of pixel data being generated each second, which must then be processed in real-time. They have tested and verified a VHDL design that can process this data in real-time that computes the center of masses of objects as they fall through the scan line. The design will be ultimately placed in the fabric of an Altera Arria V FPGA. The team presented their design at the MSU COE Engineering Design Fair on April 28, 2016 (Figure 7).

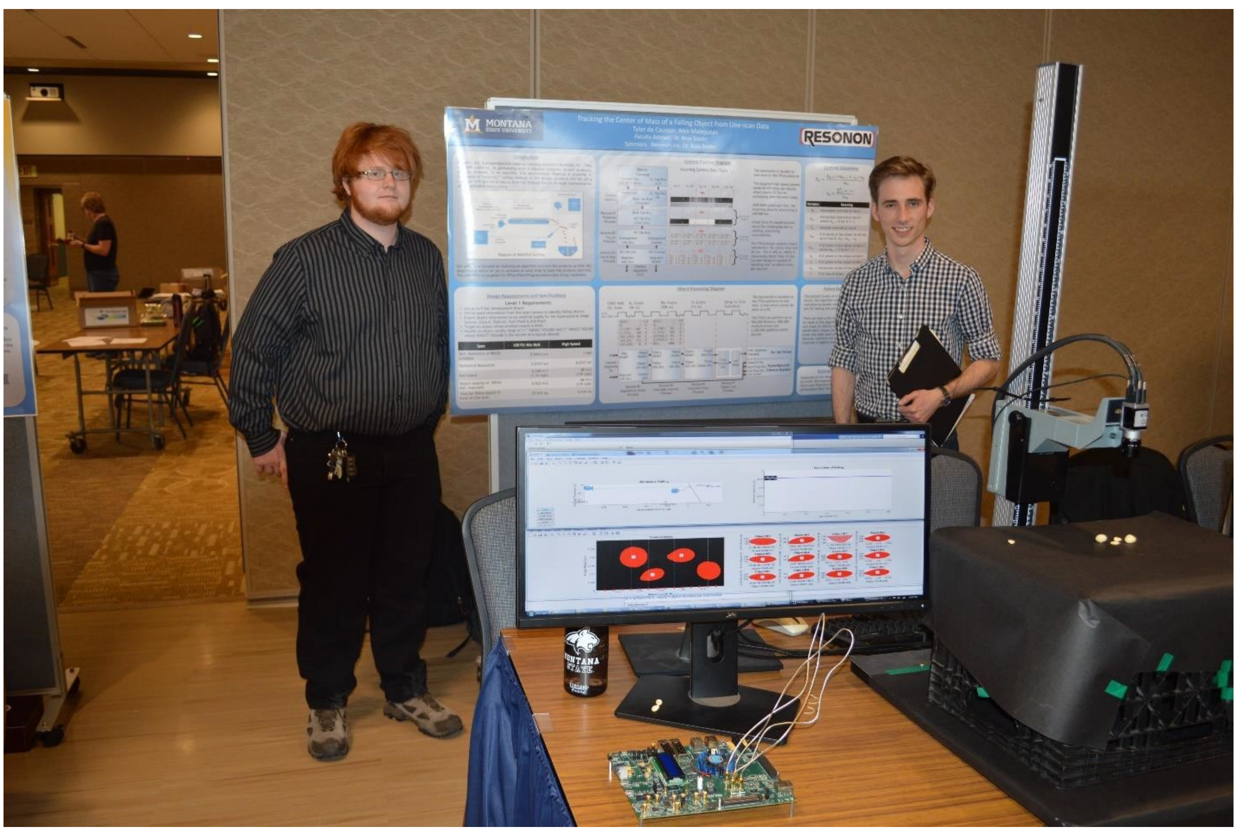

Figure 7. Student team presenting senior design project results for waterfall sorting at the Montana State University College of Engineering Design Fair in April 2016.

- The air jet subsystem is being put together by undergraduate student Sam Kyser, a Mechanical Engineering and Computer Engineering double major who also has machining skills. He has designed a test bed system that incorporates the manifolds and is currently machining the parts to assemble the test fixture that will be used to test the air jets. A primary goal is to characterize the 3D plume force profile as a function of time. We need a model of the air jet plume to know the exact time when sufficient force as built up at the target location. We are building our own instrumentation setup to characterize the 3D plume time-force profile. The design of the test setup can be seen at the end of this report and a picture of Sam Kyser is shown in Figure 8 with some of the parts that he has already designed and machined.

Figure 8. Montana State University undergraduate student Sam Kyser with parts he has designed and machined for a waterfall sorting system.

- Undergraduate student Nick Lapp has been working on the high speed transceivers that will be used as the SATA interface to store high bandwidth image data to mSATA flash drives.
- We are creating high speed serial interface links to transmit data between two FPGA boards, the Altera Arria V FPGA and Arria 10, which will be used for processing hyperspectral image data. This is how the edge information will be fused with the hyperspectral information. This has involved creating custom FMC connector boards that contain SMA connectors (see Figure 9). The high speed serial links will connect through the SMA connectors to the FMC connectors to the FPGA boards since the FPGA development boards contain high pin count FMC connectors.

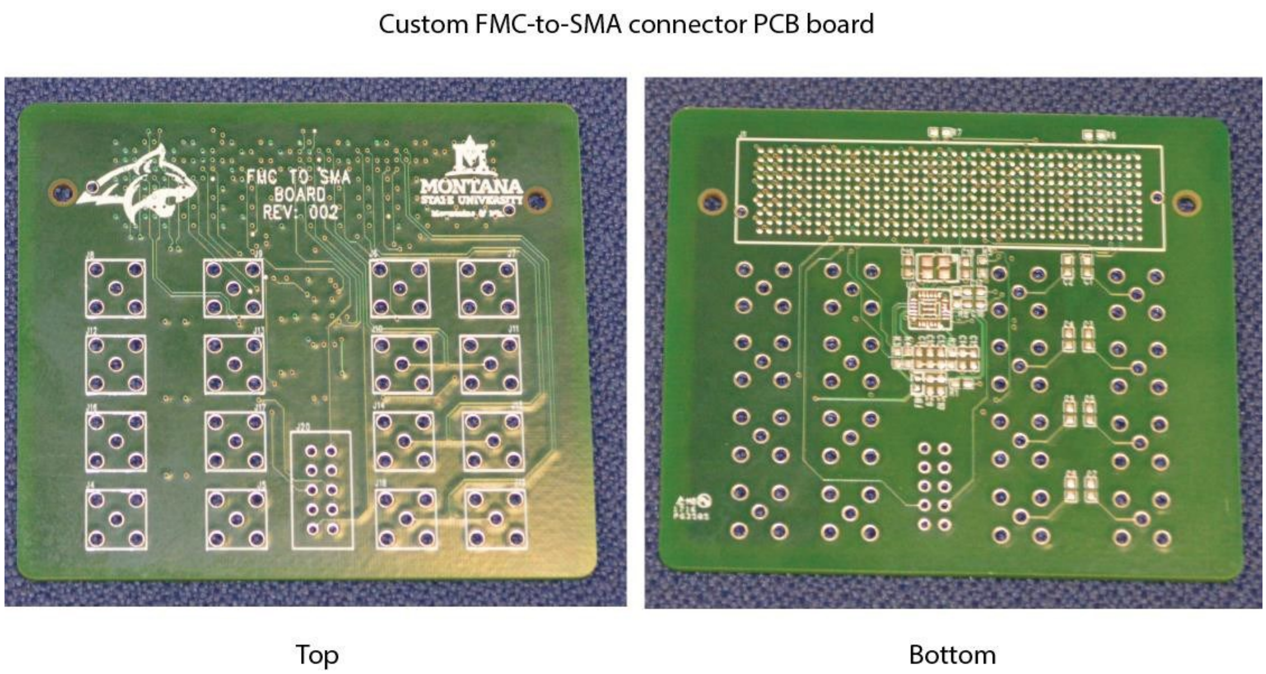

Figure 9. Custom FMC-to-SMA connector printed circuit boards custom designed and produced by students at Montana State University.

- In collaboration with Ed Dratz on the project titled: *Design a hyperspectral imaging system for monitoring the metabolic state of live cells in culture*, we are designing a platform that can automate lab equipment. This will ultimately be used to control the microscope stage and Cellasic microfluidics plate to grow cells and monitor cell growth using the hyperspectral camera. The framework to automate lab equipment was a senior design project (Trevor Vannoy and Hongchuan Wang) I advised and co-sponsored with the chemistry department to automate the gantry robot to spray matrix on slides for MALDI mass spec. The team presented their design at the MSU COE Engineering Design Fair on April 28, 2016 (Figure 10).

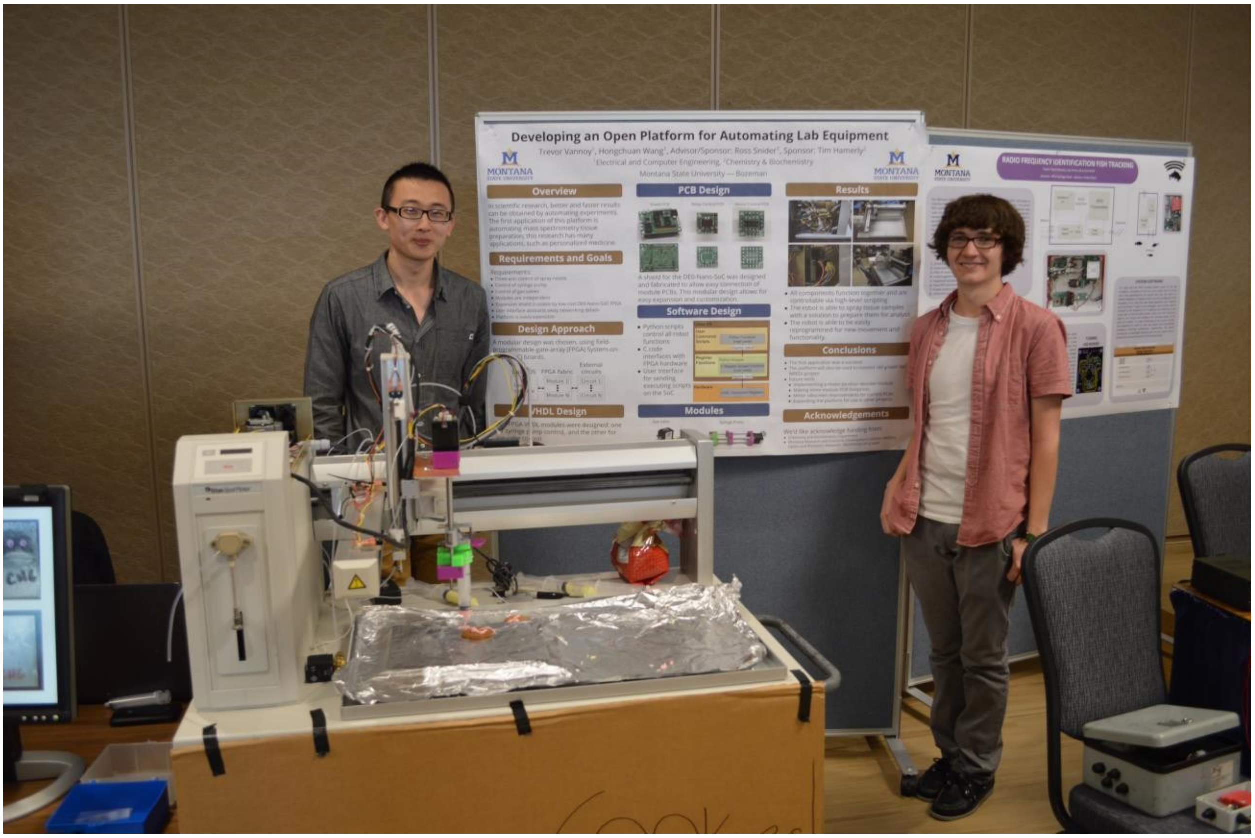

Figure 10. Montana State University undergraduate students presenting at the College of Engineering Design Fair in April 2016. These students are designing a system to control a microscope stage and microfluidics plate to grow cells and monitor cell growth using a hyperspectral camera.

Projected next quarter activities

- Conveyor belt system with recirculator.
- Air jet plume characterization
- Continue with physically integrate line scan camera to Arria V FPGA board.
- Get high speed serial link communication working between FPGA boards (3 Gbps).

<u>Expenditures to date:</u>
Salary to date $8517.04, Benefits to date $327.89, Operations to date $27,336.47, Capital to date $4,845.00, Total Expenditures to date $41,026.40

Subproject 3: Remote Sensing Algorithms for Precision Agriculture (Rick Lawrence with Resonon, Inc.) Develop and apply a methodology using hyperspectral imagery for determining optimal narrow spectral band combinations for identifying targeted invasive weeds in specific crops.

Milestones

a) July 31, 2016: Collect invasive weed field data
b) August 31, 2016: Collect hyperspectral image data
c) October 31, 2016: Complete image preprocessing
d) January 31, 2017: Complete analysis of spectral band optimization and weed species mapping
e) June 30, 2017: Final report, including applications for commercial site-specific agriculture

Activities to date:

- Potential farm field sites were identified and contacts made with landowners. Several sites had to be rejected, because weeds were sprayed prior to acceptable weather conditions for conducting hyperspectral flights and conducting ground surveys.
- Methods were developed for ground data collection and arrangements were made with a pilot for conducting flights.
- Previously preprocessed Pika hyperspectral imagery was used to conduct initial testing of spectral band optimization methods.

Initial band optimization tests were evaluated using the Bandmax tool in the hyperspectral image processing software, ENVI. This, in turn, enabled us to evaluate the effect of using the full suite of Pika's 80 bands vs two optimized bands. Weed data was unavailable for these data, rather this was a developmental test to prepare to the 2016 data collection, and the classifications were thus conducted for general land cover types to develop our methodology. The base classification demonstrated that this particular approach to band optimization and classification was not able to maintain the level of accuracy that was possible with the full range of bands, with overall accuracies dropping from 74% with 80 bands to 52% with two optimized bands. The images below demonstrate that the use of 80 bands was able to capture substantially more spatial variability than the two band analysis. This will direct us to more robust methods available in R for the 2016 data collection.

Expenditures to date:

Salary to date $7872.50, Benefits to date $234.07, Operations to date $3,424.55, Total Expenditures to date $11,531.12

Subproject 4: Machine Vision Algorithms for Precision Agriculture (Neda Nategh with Resonon and NWB Sensors, Inc.) Develop machine vision algorithms for weed detection and food sorting using spectral imaging data.

Milestones

- May 31, 2016 Initial machine vision algorithms developed.
- Sep. 30, 2016 Initial testing of machine vision algorithms complete.
- May 31, 2017 final testing and development complete.
- June 30, 2017 Final report completed

Activities to date

- Students were advised on the analysis methods for hyperspectral image data and the results are being used for weed mapping.

Expenditures to date

Salaries $30,123.05, Benefits $1823.48, Operations $5286.00, Total Expenditures to date $37,232.53.

Subproject 5: Microcavity sensors for hyperspectral imaging (Zeb Barber with Advanced Microcavity Sensors LLC). Advance MSU/Advanced Microcavity Sensors LLC (AMS) technology on microcavity hyperspectral imaging sensors toward commercial applications in agriculture and engineering tests to determine feasibility of mounting sensor technology on UAV; secondary objective solving MT problems in agriculture and biomedical (skin cancer). The primary objective focused on MREDI goal #2: creating private sector jobs.

Milestones

a) June 1, 2016: Investigate non-circular symmetric micro-cavity mirrors for transverse mode manipulation
b) September 1, 2016: Evaluate Microcavity Hyperspectral Imaging prototype system for early crop disease/weed detection
c) December 30, 2016: Determine engineering specifications for use of Hyperspectral Sensor on UAV
d) June 30, 2017: Submit final report specifying technical accomplishments and outlining commercial potential.

Activities to date

- MSU Spectrum Lab has hired Dr. Caleb Stoltzfus a recent Physics PhD from Prof. Rebane's group as a Post-Doc on the microcavity technology. His PhD emphasis in biophotonics will be an asset to the project.

- MSU Spectrum Lab, Spectral Molecular Imaging Inc. (SMI), and AMS have been informed that they will be awarded a Phase II STTR contract from the Air Force to advanced commercialization of the microcavity technology. The contract will be in the amount of $750,000 with $230,000 of that as a subcontract to MSU and $100,000 as a subcontract to AMS.

During this quarter a new proposal on multi-wavelength, independently tunable micro lasers has been submitted to MBRCT. This new proposal by Spectrum Lab at Montana State University (MSU-SL) and Advanced Microcavity Sensors (AMS) proposes to design, build and test a revolutionary new microscopic liquid crystal laser system that will have significant impact on many industries in Montana. This project will focus on addressing the urgent need in many industries including biomedical spectral imaging for a bright, tunable, compact and low cost illumination source and the current broadband white light sources. High spectral resolution imaging for biomedical imaging applications will be enabled by our new laser array system, which will enable new techniques in identifying cancer cells and offer much higher diagnostic specificity for other serious diseases.

This laser array proposal leverages the technology behind the Liquid Crystal filled Arrays of optical Microcavities (LCAM) that is currently supported by the M-REDI project. The basic concept is remarkably simple: construct microscopic laser cavities that use liquid crystal to enable wavelength tuning, conventional absorbers to enable lasing gain and utilize the core optical technology developed by AMS to confine the 'seed' light to enable remarkably low threshold lasing.

Key value propositions for the laser array concept are:

1) Ultra-low lasing threshold reduces power requirements: 2) Physical size and portability: and 3) Scalability of the array architecture: The final device has the potential to encompass >1000's of independently, high power, tunable microscopic lasers within an area the size of a quarter. This enables applications (e.g. display technology) that are not practical using conventional lasers (primarily due to the prohibitive cost, fabrication complexity and size of such a scheme).

The proposed effort will result in a new laser technology that has the potential to revolutionize the fields of spectral imaging, display technology and many other local photonic industry applications.

This will be achieved by providing a low-cost, compact, low-power-consumption laser array for illuminating targets such as the skin, cells or other biological samples that are subject to spectral imaging. It is anticipated that the laser array will enable important measurements such as Raman imaging (especially in conjunction with our hyperspectral imaging sensor) that is not feasible in current spectral imaging systems due to lack of a practical source that allows efficient illumination. This new product will be an excellent complementary product line to the hyperspectral imaging sensor currently under development at AMS.

Although the initial applications for this project are likely to biomedical, the new technology would provide an ideal solution for many applications which will add significant value to these industries and to the Montana optics industry. Our new miniature tunable laser source will significantly enable many hyperspectral imaging applications for example in collaboration with Resonon or Bridger Photonics laser ranging applications.

The project has significant potential to further a state-of-the-art spectral imaging technology towards commercialization in Bozeman, MT. The hyperspectral imaging technology being developed by AMS in collaboration with MSU Spectrum Lab, will benefit greatly from this new highly configurable laser array and allow us to explore more commercial markets (particularly portable, in-field detection of disease such as Tuberculosis and automated bacterial identification). This will further transfer research that originated at MSU-SL to technologies at a local company (AMS).

Technical Progress toward Objectives:

a) AMS has greatly improved the laser ablation process for creating the curved mirror template using active feedback to the ablation laser. This makes the crater depth consistent and controllable. This improve system will be used to create elongated craters to improve transverse and longitudinal mode separation.

b) Spectrum Lab staff have investigated concepts for use of the LCAM technology for agricultural applications. Through discussions with Paul Nugent (a PhD student in Prof. Joe Shaw's group working on other agricultural spectral imaging projects), we have determined that for basic weed detection while the potential low cost of the LCAM technology is attractive these applications most often do not require the high spectral resolution of the LCAM technology which is one of its main performance advantages. Spectrum Lab staff also re-evaluated prior concepts for agricultural weed detection based on inserting fluorescent markers in the desired crop to provide a clear marker separating crop from weeds. This next quarter we will be using recently designed LCAM coatings (see Figure 11) to investigate the spectral measurement of fluorescent markers. This takes better advantage of the spectral resolution of the LCAM, but requires special genetically modified seed.

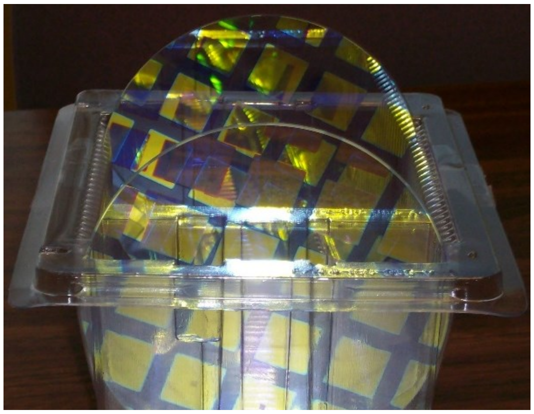

Figure 11. Four-inch wafers with microcavity craters and mirror coatings at 580 nm designed to measure common fluorescent protein markers. The wafers were designed and purchased on the AF STTR. The cross-hatch pattern allows access to a base ITO layer which provided electrical tuning of the liquid crystal layer. This next quarter they will be constructed into tunable LCAM cells and used to measure two fluorescent protein markers.

c) No progress to report this quarter.

Expenditures to date
Salaries $8194.14, Benefits $2888.35, Operations $212.63, total expenditures $11,294.12

Subproject 6: Hyperspectral imaging for monitoring cell growth (Ed Dratz, dratz@chemistry.montana.edu with Resonon, Inc.). Design a hyperspectral imaging system for monitoring the metabolic state of live cells in culture. Applications to stem cells for understanding disease mechanisms in individuals, drug testing in cells from individuals, potentially optimize personal nutrition, and solve Montanan's health problems.

Milestones

a) February 1, 2016: Complete design and testing of proof of principle prototype hyperspectral imager with improved cost/benefit, prototype interface for cell hyperspectral analysis, and development of stem cell labeling
b) May 1, 2016: Integrate the prototype systems for advanced analysis of stem cell metabolism with hardware and software control. Test for evaluation of optimization of selected nutrients
c) October 1, 2016: Refine and improve software and operating conditions of real time hardware and software for variations of metabolic state for culture optimization
d) February 1, 2017: Enhance user interface to control system and software to control and optimize nutrient composition; evaluate possible changes in microscope system for improved performance

e) June 30, 2017: Proof of principle for feedback control of nutrient optimization with nutrient dosing control system. Investigate biochemical individuality in pilot experiment
f) June 30, 2017: Submit grant proposals to leverage additional support. Final report to MUS that summarizes accomplishments and commercial potential

Activities to date:

The inverted epifluorescence microscope optical system has been designed, optical vendors contacted for providing optical modules, the optical system has been configured, and all the microscope components ordered. We were able to find a single vendor that is able to provide an integrated inverted microscope solution, where all the components are sure to fit together with appropriate focal lengths and where the specialized hyperspectral components can be fitted. We expect that the hyperspectral components in hand can be integrated with the modular microscope as soon as the parts arrive in about two weeks. We expect that this system will offer high performance and these selected components will also be affordable as parts of a final hyperspectral product for sale.

One particularly critical component is the computer-controlled XY sample stage, which has to be able to scan in small, very high resolution steps for maximum hyperspectral resolution (22 nm in this case) along with rapid movement speed (7mm/sec in this case). The microscope will do an initial rapid scan of the image field, the software will locate cells, and the cells will then be scanned at high resolution repeatedly during the course of the experiments. Thus, the stage has to be able to scan rapidly between cells and then switch to much slower, small step sizes for high resolution imaging. The software control for the XY stage and Z automatic focusing with programmable nose piece for changing objectives for different magnifications has been designed. The upright epifluorescence microscope test system from the Dratz Lab that was moved to the new Resonon facility is being used for initial testing of the excitation laser system for hyperspectral imaging.

The Onix CellAsic microfluidic cell culture control and observation system has been installed on the refurbished laser-excited confocal inverted epifluorescence microscope system. The Zeiss LSM 510 meta is a high performance confocal, with the latest image analysis software, that is important for providing benchmarks for the new hyperspectral system. The Onix CellAsic microfluidic system is being used for controlling and optimizing the nutrient media on the human inducible stem cell (hiPSC). Prof. Snider’s team is working on the software to integrate the Onix CellAsic microfluidic culture control with the hyperspectral data acquisition and processing.

Further progress has been made in the Dratz lab (PI) on introducing optogenetic probes of the oxidation/reduction state into human adult stem cells in culture. The probes have been transferred to efficient carrier vectors that are providing improved, more facile optical probe introduction. A human-inducible pluripotent stem cell (hiPSC) line was transfected with a modified green fluorescent protein (GFP) that was fused with the ORP1 protein. The ORP1 protein causes the GFP to greatly increase fluorescence in the presence of hydrogen peroxide (H2O2). The GFP was also fused to a mitochondria targeting protein sequence to form the complete protein: Mito ro GFP-ORP1. Hydrogen peroxide is a common driver of oxidative stress in animal cells. Referring to Figure 12, the very low fluorescent cells on the right contained GFP-ORP1 but were not stressed with H2O2 and the cells on the left were stressed with H2O2. The cytoplasm of the cells are not fluorescent and the smaller punctate fluorescent zones are the mitochondria, as expected.

hiPSCs + Mito ro GFP-ORP1, targeted to mitochondria

Plus H2O2 oxidation

Minus H2O2

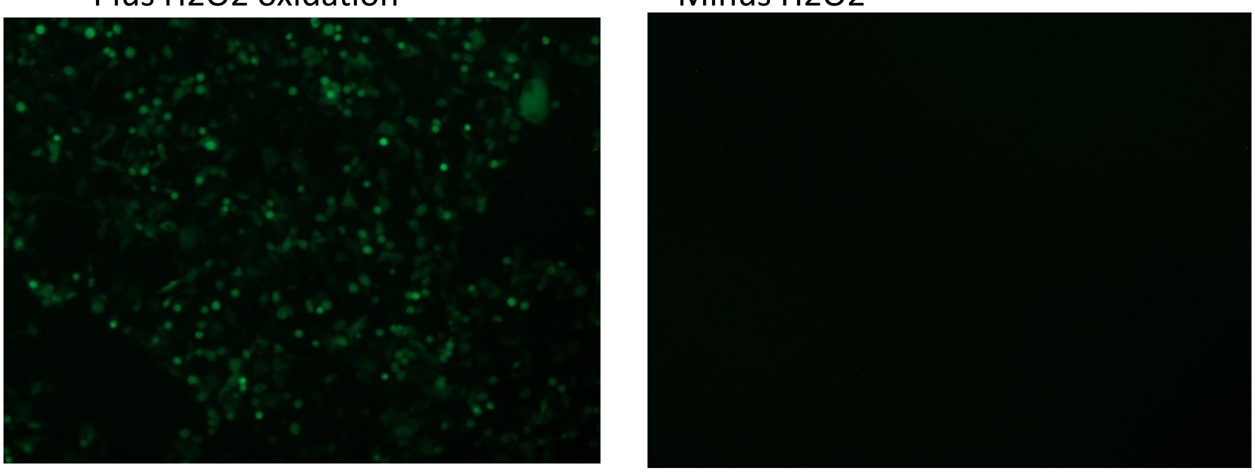

Figure 12. Very low fluorescent cells on the right contained GFP-ORP1 but were not stressed with H2O2 and the cells on the left were stressed with H2O2. The cell cytoplasm are not fluorescent and the smaller punctate fluorescent zones are the mitochondria, as expected.

A graduate student in the Dratz lab is devoting full effort to working with the optogenetic probes, assisted by a research undergraduate, two post-doctorals in the Reijo Pera lab, and a Research Assistant Professor in the Singel lab, all in the Chemistry and Biochemistry Department. An advanced undergraduate Electrical and Computer Engineering (ECE) design team in the Snider lab in ECE is continuing to design the microscope stage controller system and the controller for the cell culture environmental control system. A graduate student in the

Snider lab is devoting full effort to the high speed hyperspectral imaging analysis software and will be working on this crucial aspect of this project into the next year. The personnel include two graduate students devoting full effort to the project, two advanced undergraduates on an ECE Design team.

Expenditures to date

Salaries $22,698.17, Benefits $1177.29, Operations $15,282.25, Capital $29,261.20, Total expenditures to date $68,418.91.

Subproject 7: Translational research to commercialize micro-mirror technology (Arrasmith at Revibro Optics). Translate MSU-developed deformable mirror technology to a commercially sustainable product.

Milestones

a) Refine production to achieve a repeatable fabrication process. This milestone will involve a redesign of fabrication masks, purchase of new wafer bonding equipment, and refinement of wafer bonding process (June 30, 2016).
b) Obtain funding from another source. Revibro will pursue funding through commercial sales and commercial R&D efforts (June 2016), and through SBIR/STTR or similar government funding (June 2017).
c) Create 2 full time Montana jobs: One job will be created immediately to sustain the founder of Revibro (August 2015); Technical and/or sales and marketing hire (December 2015).

Activities to date

- Characterized mirror step response as a function of air damping.
- Developed a mirror electrode layout and damping design for new fabrication.
- Measured mirror performance under illumination with high power femtosecond laser illumination.
- Created a white paper on mirror operation under high power conditions to satisfy customer requests.
- Participated in Photonics West trade show with over 26,000 people attending.
- Prepared for Hannover Messe trade show in Germany. This trade show is the largest industrial automation trade show in the world, and will have over 120,000 people attending.

During Q3 Revibro Optics completed additional measurements of the frequency performance of our mirrors. Because our mirrors are damped by the air under the membrane, the rate that the air can escape from underneath the membrane has a direct effect on high speed performance.

The mirrors are engineered with a series of air holes to allow air to escape from the gap space. The number of air holes has a direct effect on the membrane deflection response – having the possibility of producing underdamped or overdamped deflection behavior. During Q3 the step response performance for four different air hole patterns ranging from 24 to 64 holes was compared. Based on the results we are were able to choose an appropriate number of holes for a new fabrication mask design. The results are shown in Figure 13.

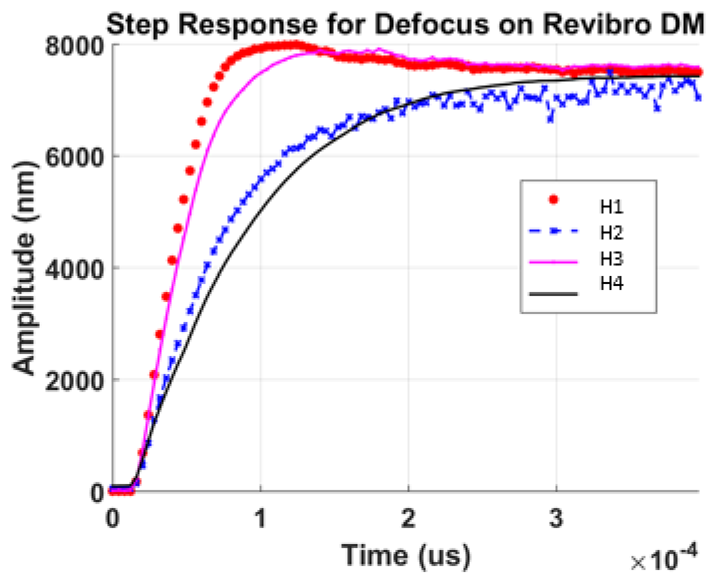

Figure 13. High-speed defocus step response for Revibro deformable mirrors with different hole patterns.

For the new fabrication masks we will produce mirrors with four electrodes. Having chosen the optimal number of air holes resulting in work from Q3, we will be design fabrication masks in Q4 and begin the actual fabrication process to produce new devices. We hope to begin work in the MMF fabricating new mirrors in June.

In addition to step response testing, we worked to define the power handling capability of our mirrors during Q3. We were able to use a Spectra Physics Deep See femtosecond laser for this testing. The use of this laser was important, as many of our potential customers in the multi photon microscopy markets use this laser, or one very similar in their experiments. The results were very promising. We were able to expose our mirrors to 0.6 watts of laser power across the 4 mm diameter membrane without any damage. Though no damage took place, there is a heating of the membrane due to thermal absorption of laser light that causes the polymer membrane to soften, meaning that less voltage is required to achieve the same deflection as measured with no high power laser illumination. Because our mirrors currently operate with open loop control, it is very important to have reproducible voltage versus deflection (V-D) performance. We found through our testing that it is possible to develop reliable deflection look-up-tables for different illumination powers. In a typical setup, the laser power is not adjusted during an imaging test, so using a preset deflection table for the given laser power should be able to produce the necessary focusing precision.

This testing was performed in response to requests from several potential customers. We produced a white paper summarizing our findings and provided this to the interested parties. This white paper will be an important support document for future sales. The results are summarized in Figure 14.

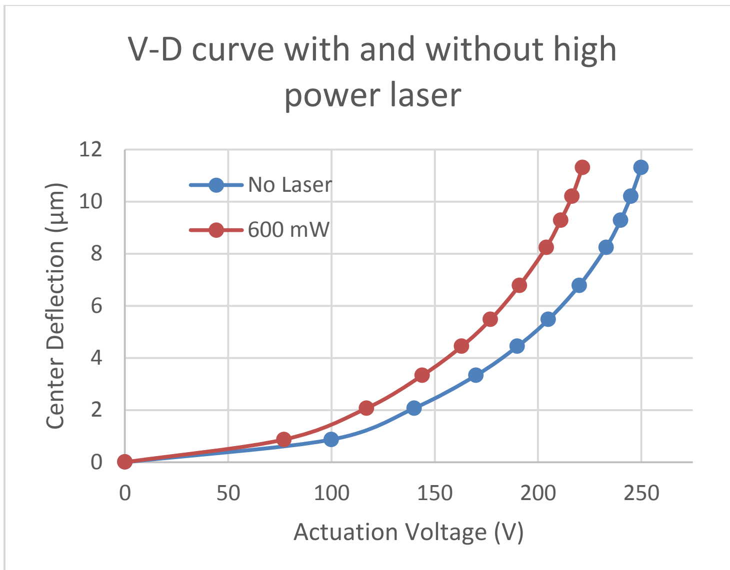

Figure 14. V-D curves for mirror operation with and without 0.6 W 930 nm laser illumination.

Revibro Optics is actively in pursuit of commercial income through product sales. To help accomplish this, we participated in Photonics West, the largest optics trade show in the US (see Figure 15). We shared a booth with MSU's OPTEC and the Montana Photonics Industry Alliance (MPIA). MPIA helped greatly by organizing the booth space and taking care of design and purchasing of our booth graphics. Our booth attracted many people interested in our mirrors, and we retained several promising sales leads. Because our technology is in its early product stage, we are working with interested parties to secure initial sales while being clear that this is a newly developed product. We hope to have several commercial sales by the end of the year, and start getting very valuable usage input from these early adopters.

Figure 15. Revibro Optics shared a booth at Photonics West in February 2016 with Montana State University/OpTeC and the Montana Photonics Industry Alliance (MPIA).

Through help from the Department of Commerce, we will be taking advantage of an opportunity to exhibit at the Hannover Messe trade show at the end of April 2016. Taking place in Hannover Germany, this is the world's largest trade show for industrial automation. Revibro Optics will share a booth with three other Montana companies. This show will provide an important opportunity for finding additional customers for early sales and possible development projects. We will also stay for the week after the show to visit a very important customer and microscope manufacturer that we have done development projects with in the past.

Total Expenditures

Salary and Benefits to date $41650.67, Operations to date $1874.04, Total Expenditures to date $43,524.71.

Subproject 8: Active waveguides and integrated optical circuits (Rufus Cone, cone@physics.montana.edu, collaborating with Babbitt, Nakagawa, Barber, Himmer, Avci, and Thiel with S2 Corp., AdvR, FLIR/Scientific Materials, and Montana Instruments). Integrate Montana products, expertise, and capabilities to improve marketability, performance, and enable additional products: Build interdisciplinary connections among MUS and Montana optics industries to integrate (a) optical crystals by FLIR/Scientific Materials Corp. (SMC); (b) waveguide photonic components of AdvR, Inc.; (c) Montana Instruments (MI) cryogenic systems; and (d) S2 Corp. (S2C) signal processing devices.

Milestones

a) Fall 2015: Fabrication of rare earth doped optical waveguide suitable for optical signal processing applications
b) Summer 2016: Integration of an optical waveguide into a cryostat
c) Spring 2017: Demonstration of SSH processing in a cryogenic waveguide
d) June 2017: Final report summarizing technical results and emphasizing commercial potential.

Activities to date

During this third reporting period, significant progress continued on all project milestones. This included installation, testing, and improvement of new facilities for characterization of the rare-earth-activated waveguides that have been developed as part of the milestone achieved during the previous quarter. Work has advanced on both theoretical modeling of the dielectric micro-structures and optical interfaces required for commercialization of Montana photonic signal processing technologies as well as fabrication and demonstrations of the components being developed as part of this MREDI effort. Furthermore, MSU personnel have been working closely with several of the local companies in the optics industry (S2 Corp., FLIR / Scientific Materials Corp., Montana Instruments, and AdvR Inc.) to transfer technology and expertise both from the university to the companies and from the companies to the university.

All project activities are on schedule to successfully meet our objectives and milestones. The Montana Instruments (Bozeman, MT) cryostation has been received, set up, and tested, with efforts beginning on integration of rare-earth waveguides into the cryogenic environment. Development of cryogenic optical fiber interfaces for photonic technologies is underway and MSU personnel are working in close collaboration with AdvR (Bozeman, MT) on improving local capabilities for waveguide fabrication and lithography of a wider range of optical materials. This work is closely supporting integrated photonic signal processing efforts between the MREDI project and the significant new funding brought into the state by S2 Corp and MSU Spectrum Lab, as recently highlighted on the southwestern news television station KBZK (news video at www.kbzk.com/story/31588503/msu-bozeman-corporation-land-45m-contract) and in the Bozeman Daily Chronicle newspaper (the news article is available online at the following link: www.bozemandailychronicle.com/news/montana_state_university/msu-bozeman-based-s-corp-announce-m-contract-to-provide/article_f97ebf15-e82b-54ef-817a-f719481681fc.html).

Some of the specific efforts and outcomes during this quarter are briefly outlined below.

<u>Progress on Technical/Educational Objectives:</u>

- Toward the objective of constructing optically active waveguide circuits, Spectrum Lab Research Engineer Tia Sharpe has been acquiring training in microfabrication, lithography, and optical waveguide process techniques. In addition to completing training on process tools at the Montana Microfabrication Facility (MMF), project partner company AdvR has allowed Tia to shadow their engineers in the MMF to learn and study the processes the AdvR uses to fabricate waveguides on $LiNbO_3$ (LN) substrates (see Figure 16). The fabrication processes include photoresist deposition, lithography, acid and plasma etching, sputter and e-beam deposition. A contracted services agreement and Confidentiality Agreement with AdvR has been prepared to provide this training. In addition to the training goal MSU will be monitoring and studying AdvR's process for changes toward improving yields and developing techniques to characterize the performance of the waveguides.

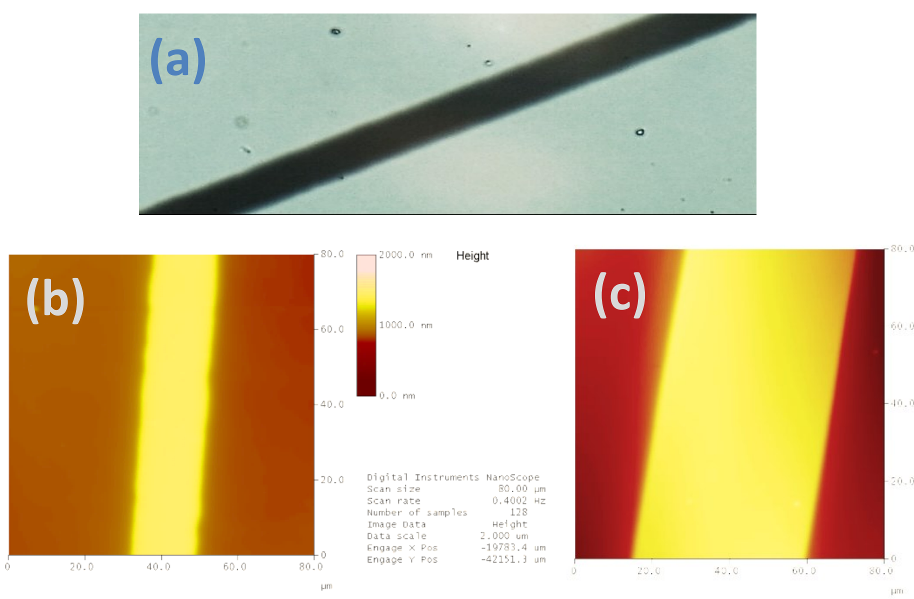

Figure 16. Examples of micro-structures of rare-earth metals fabricated onto $LiNbO_3$ optical substrates as a precursor step in ion-diffused waveguide production. (a) Optical microscope image of a portion of a 20 micron wide and 1 cm long Thulium metal strip on a $LiNbO_3$ wafer. (b) and (c) are examples of Atomic Force Microscopy images and profiles of fabricated rare-earth strips with different dimensions.

- Work has progressed on structuring rare-earth chemical composition of optical materials for photonic waveguides. Metallic stripes of rare-earth elements have been produced with initial dimension targets and then diffused into the crystal structure to modify the optical properties using the new processing capabilities developed on this effort during the previous quarter.

- The Montana Instruments cryostation system (Figure 17) was delivered during this quarter, enabling work towards our key objectives of developing and demonstrating cryogenic optical waveguide technology. System testing and evaluation was carried out and a range of potential collaborative research and development efforts enabled by this system were discussed with Montana Instruments. In addition to enabling sustainable research in this area at MSU, this system will also greatly expand the access and training of undergraduate and graduate students with high-end commercial cryogenic systems, an area of specific importance for the local optics industry.

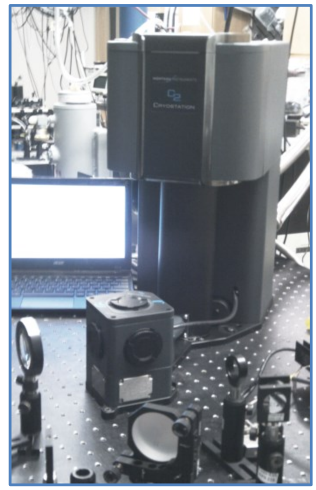

Figure 17. Installed Montana Instruments cryostation to enable planned cryogenic waveguide implementations and support collaborative efforts with Montana Instruments.

- MSU Spectrum Lab has continued investigations of incorporating optics into the cryogenic environment to provide more compact and robust spatial-spectral processing systems. Ray trace modeling of an optical system based on optical fibers being collimated/focused using gradient index (GRIN) lenses through a bulk spectral hole burning crystal and recollected into fiber with a second GRIN lens has been started. In addition, Spectrum Lab personnel have been interacting with engineers at optical fiber component vendors to identify components and parts.

- We have continued to make significant progress on developing and expanding our new rare-earth-doped waveguide fabrication and characterization capabilities. A range of new analysis capabilities have been implemented and applied to this effort. Among these, we set up a stylus profilometer with vertical resolution of 0.1 nm for measuring chemical deposition and lithography profiles of micro- and nano-fabricated features (Figure 18). We also further refined the optical ellipsometer apparatus reported in the previous quarter. A unique quartz-crystal microbalance measurement system was developed and implemented to monitor deposition of thin films of rare-earth elements and metal electrodes onto opto-electronic crystals. Furthermore, electron backscatter diffraction techniques were developed for studying the structure, composition and orientation of

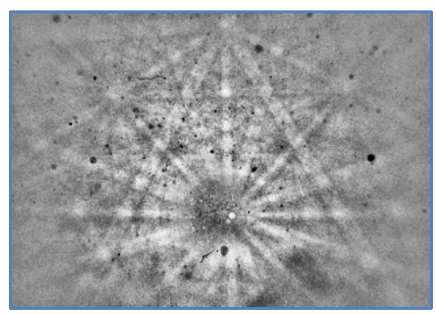

Figure 18. Example electron backscatter measurements of the crystal structure of chemically modified $LiNbO_3$ optical materials for waveguides.

optical crystal surfaces using the Field-emission Scanning Electron Microscope in the Imaging and Chemical Analysis Laboratory. This new capability and expertise will greatly benefit the waveguide development efforts, as well as broader research at MSU and product development and characterization at Scientific Materials Corp.

- Development of suitable waveguide models using the COMSOL Multiphysics software with Wave Optics Package continues. Models of wave propagation in guiding structures similar to those in development in other areas of this project are in progress. Moreover, computational time and memory required for various scale models are being evaluated. Training of graduate students in the use and applications of this tool also continues. A new dedicated computer workstation was purchased and installed to meet the extreme computational demands of this sophisticated software package. Furthermore, discussions are underway with local companies regarding potential joint research projects that may be enabled by this new capability at MSU.

- We continue efforts on the new concept developed under this effort for fabricating rare-earth-activated waveguide structures through micro- and nano-fabrication of heterogeneous dielectric structures or “strips” onto the surface of bulk transparent optical crystals. This work includes fabrication of micro-structured features on optical crystals, production of high-quality thin crystal wafer substrates, and simulations of device design and performance using the COMSOL software (see Figure 19).

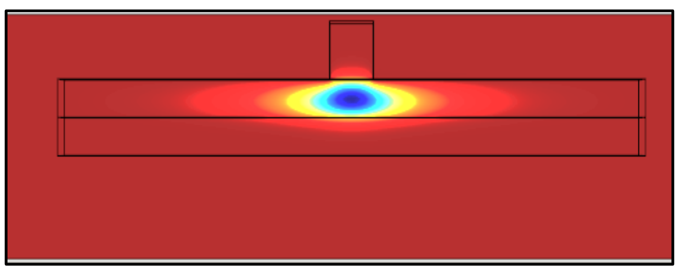

Figure 19. Cross-sectional image of a “strip-loaded” optical waveguide modeled using COMSOL theoretical simulations and analysis. The color map indicates the distribution of light intensity confined by the micro-structure dielectric design, indicating the control and manipulation of the light. Simulations such as these allow the material composition, mechanical design, and required fabrication precision to be determined and optimized.

- Research Engineer Tia Sharpe has enrolled in an online course on silicon photonics called ‘edX UBC Photonics 101 course Silicon Photonics Design, Fabrication and Data Analysis’ coordinated by IEEE Photonics. While silicon is unsuitable for rare earth doped devices many of the design and analysis tools learned in the course can be directly adapted LN or other materials for MREDI project objectives.

- MSU Physics graduate student Jacob Braunberger is working on Maxwell-Bloch simulations of the optical interactions with the rare-earth doped materials to predict interactions and propagation effects in optically thick media. One dimensional plane wave models have been previously developed at MSU Spectrum Lab this model will attempt to incorporate full 2D modeling. The initial simulations will focus on propagation in bulk materials with an end goal of including propagation in waveguide structures.

- As determined by the needs of the project, a contract has been successfully established with Scientific Materials for custom crystal growths with a wide range of rare earth dopants. This effort includes both lithium niobate and lithium tantalate optical materials doped with rare-earth ions, as well as special chemical composition that can increase the optical damage thresholds and power handling capabilities of the crystals—critical needs for applications at both S2 Corp and AdvR Inc.

- The sophisticated commercial software package Comsol Multiphyics, including the Wave Optics Package, was purchased and installed at MSU during this quarter. This MREDI effort enables opportunities for student training, device design and engineering, and scientific research and development in Physics, Electrical and Computer Engineering, and Spectrum Lab. Currently, two PhD students (one in ECE, one in Physics) are being trained to use this software, and this number is expected to grow over the course of the MREDI effort. This know-how and experience, in addition supporting their current research and progress towards the doctoral thesis, will be an important competitive skill after these students graduate and pursue a professional career.

- Undergraduate student Brett Wilkins carried out a series of measurements and subsequent analysis of index of refraction (speed of light) variations in garnet crystals caused by the addition of rare-earth ions to the chemical composition. This work is a key step in producing optical waveguides in this important system. Brett is graduating with a BS in Physics this semester and anticipates working in the local optics industry this summer before entering graduate school at MSU in the fall.

- The effects of applied electric fields and electric field gradients on rare-earth-activated crystals were studied for different electrode configurations by both experimental optical coherence measurements and theoretical simulations. The material Tm:YAG used by S2 Corp. for photonic signal processing was characterized, resulting in new insights into the microscopic material dynamics that determine some aspects of performance in those applications. The theoretical modeling was also applied to understand basic electrode configurations to be considered for initial demonstrations with the waveguides developed on this effort. Example simulations are shown in Figure 20.

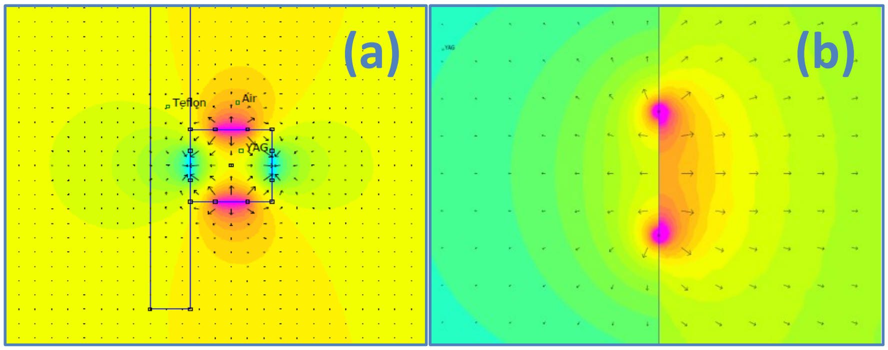

Figure 20. (left) Example simulations of electric potentials (voltage) and fields produced by complex electrode configurations used to manipulate and control the optical properties of a rare-earth-activated crystal. (right) Simulation of the electric fields produced by a voltage applied to a strip metal electrode on the surface of a crystal wafer, as required for some applications of the optical waveguides.

- Visiting researchers from the University of Calgary, Australian National University, the Army Research Lab, and IBM came to MSU during this quarter to participate in work directly related to our MREDI efforts. Some of the visiting researchers also provided their own research funds to carry out research in support of our collaborative efforts. This work ranged from studies of new rare-earth-activated crystals for quantum memory and quantum computing applications to materials for photonic signal processing. See Figure 21.

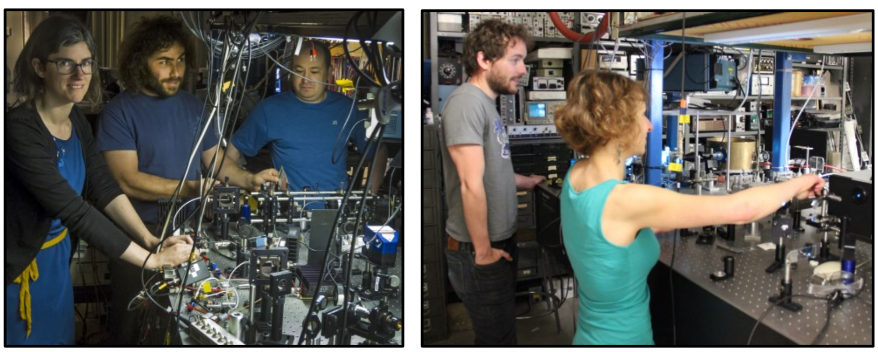

Figure 21. Researchers and students working on MREDI efforts in the MSU Physics Department (left). A visiting scientist and graduate student from University of Calgary working in the MSU Physics Department on characterizing new rare-earth materials for signal processing applications related to the MREDI effort (right).

Progress on Economic Objectives:

- MSU and S2 Corporation announced with a press release a $4.5 Million joint project funded by the Office of Naval Research in partnership with BAE Systems or Nashua, NH. The MSU press release can be found at http://www.montana.edu/news/16038/s2-corporation-and-msu-jointly-announce-contract-to-provide-wideband-sensor-capability-to-u-s-navy and a TV segment filmed and produced by KXLF TV in Bozeman, MT can be viewed at https://www.youtube.com/watch?v=k9TnY49tvyc&feature=youtu.be.

- With the delivery and installation of the new Montana Instruments cryostation system at MSU, work has begun on integrating MSU photonics technologies into the system as well as developing new product options in collaboration with Montana Instruments. Several new potential technologies have been discussed and evaluated, and plans have been made for joint testing of the most promising ideas. More exploratory work on particularly novel cryogenic components is also underway at MSU as part of this effort.

- As part of the initiative to spur economic development, one of our goals is to enable new optical crystals designed by MSU to be produced by *FLIR/Scientific Materials Corp*. (Bozeman, MT), a company that specializes in the growth of high-quality crystalline materials for lasers and other related opto-electronic technologies. As part of this, a contract was put in place with Scientific Materials to develop a series of new crystal growths for materials that are leading commercial products in the opto-electronic market. Materials activated by rare-earth ions as well as other elements that improve resistance to optical damage are being developed and studied under this effort, with direct impact on our specific project goals as well as being expected to lead to new Montana products for Scientific Materials. In addition, the high consistency and quality of the materials produced by Scientific Materials are being studied at MSU to understand how the material quality may be even further engineered to meet specific needs of different optical applications.

- MSU personnel have been working closely with Advr Inc. (Bozeman, MT) on evaluating and improving the consistency and quality of lithographic methods for fabricating optical waveguide structures at the Montana Microfabrication Facility in MSU. In particular, effects of chemical contamination and practical limitations of lithography are being investigated using the unique capabilities of the Imaging and Chemical Analysis Laboratory user facility at MSU (see Figure 22).

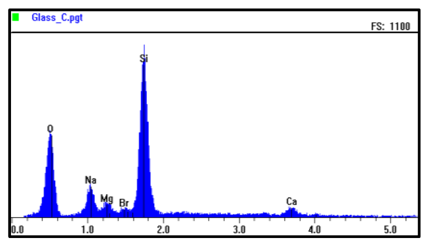

Figure 22. Example chemical analysis of waveguide components by MSU personnel using ICAL facilities.

- Rufus Cone supported Montana Photonics Industry Association and MSU Optical Technology Center at the Photonics West Trade show in San Francisco February 16-18, 2016. In his role as Deputy Director of the MSU Optical Technology Center – OpTeC, he presented a booth on optics and photonics for MSU and Montana during the massive commercial trade show that is part of this conference, providing critical exposure and promotion for the Montana high-tech economy. Cone regularly participates in Montana Photonics Industry Association activities. He also was called on by the Montana Office of Trade and International Relations to promote MSU technology transfer.

- Rufus Cone helped with successful recruiting of a new MSU engineering faculty member for the Gilhousen Telecommunications Chair in the MSU Electrical and Computer Engineering Department. This appointment is expected to provide additional theoretical support for our MREDI project.

- Rufus Cone Hosted the following visitors, all of whom presented public lectures at MSU in addition interacting with us on MREDI-related research projects.
  - Professor Erik Grumstrup, MSU Department of Chemistry, Grumstrup Research Group, and Materials Science Ph.D. Program. Lecture: *Optoelectronics at the Nanoscale: Ultrafast Electron Dynamics of Complex Materials*
  - Professor James Skinner, Joseph O. Hirschfelder Professor of Chemistry, University of Wisconsin-Madison, and Member of the National Academy of Sciences. Lecture: *The mystery of water and its condensed phases*
  - Professor Stephen Hill, Department of Physics, Florida State University and Fellow of the American Physical Society, Director of the National High Magnetic Field Laboratory Electron Magnetic Resonance Program. Lecture: *High-Field Electron Paramagnetic Resonance Studies of Molecular Nanomagnets*
  - Dr. Elizabeth Goldschmidt, Quantum Information Science, U.S. Army Research Lab. Lecture: *Single Photons for Quantum Information*
  - Professor David Dickensheets, MSU Department of Electrical and Computer Engineering and Director of Montana Microfabrication Facility with a focus on optical instrument and microdevice development. Lecture: *From Microfluidics and MEMS to Nanostructured and Bio-derived Materials: What's New in Nanotechnology at Montana State University*

- New discussions motivated by the MREDI effort have been underway with a group at the Navy Research Lab regarding potential collaborations and joint funding to investigate advanced electrode design and fabrication for optical modulators for photonics and quantum information applications.

- Outreach to local Montana schools to promote science, technology, engineering, and mathematics (STEM) was carried out by MREDI-supported graduate student Tino Woodburn. This volunteer work involved presentations, discussions, and science activities with school children at Hawthorne Elementary School in Bozeman (Jan 21) and at Mindbenders Preschool at the Lone Mountain Gym in Bozeman (February 11 & 12, and April 27 & 28). See Figure 23.

Figure 23. STEM outreach at local Montana schools.

- Dr. Zeb Barber has been invited to speak at an IEEE Summer Topical Meeting on Photonic and Analog Hardware Accelerators for Energy Efficient Computing July 11-13th in Newport

Beach, CA. He will present recent collaborative work with S2 Corporation on high bandwidth real-time matched filtering.

- Charles Thiel (Senior Research Scientist, MSU Spectrum Lab and Physics) was accepted to give a presentation on work related to our MREDI efforts for the 19th International Conference on Dynamical Processes in Excited States of Solids (DPC'16), which will be held at Chimie ParisTech, Paris, France on July 17-22, 2016. The topic of this talk will be to explain and promote "Rare-earth-activated Materials for Optical Frequency References," a technology patented by MSU and licensed to the Montana company S2 Corp. All travel expenses to give this presentation will be paid for by the National Science Foundation.

Expenditures to date

Salaries $86,071.89, Benefits $20,949.54, Operations $61,608.91, Capital $127,200.00, Total expenditures to date $295,830.34.

Subproject 9: Optical Parametric Oscillator for Tunable Lasers (Kevin Repasky, repasky@ece.montana.edu, with AdvR, Inc.). Investigate optical parametric oscillator performance in support of characterizing large aperture periodically poled non-linear optical crystals and in support of continued development of large area methane detection.

Milestones

a) December 2016: Model optical parametric oscillator performance using SNLO modeling tools
b) June 30, 2017: Demonstrate singly resonant optical parametric oscillator pumped at 1064 nm and seeded at 1650 nm
c) June 30, 2017: Final report including scientific merit and commercial products or potential

Activities to date

A setup for an OPG and OPA is completed with a schematic shown in figure 24. A 10 ns pulsed laser with 1064 nm wavelength (shown with the green line) is used as a pump source. A CW DFB laser at a wavelength of 1571 nm being amplified through an optical amplifier is used as a seed laser (shown with the red line). A half wave plate and a polarizer are used for controlling the laser energy without changing its Gaussian beam properties. A dichroic mirror is used to combine both beams and make them collinear. A magnesium-doped lithium niobate crystal is used in the nonlinear process. Two Pellin-Broca prisms are used to separate the spectrum. Two Pellin-broca prisms are used to separate the spectrum.

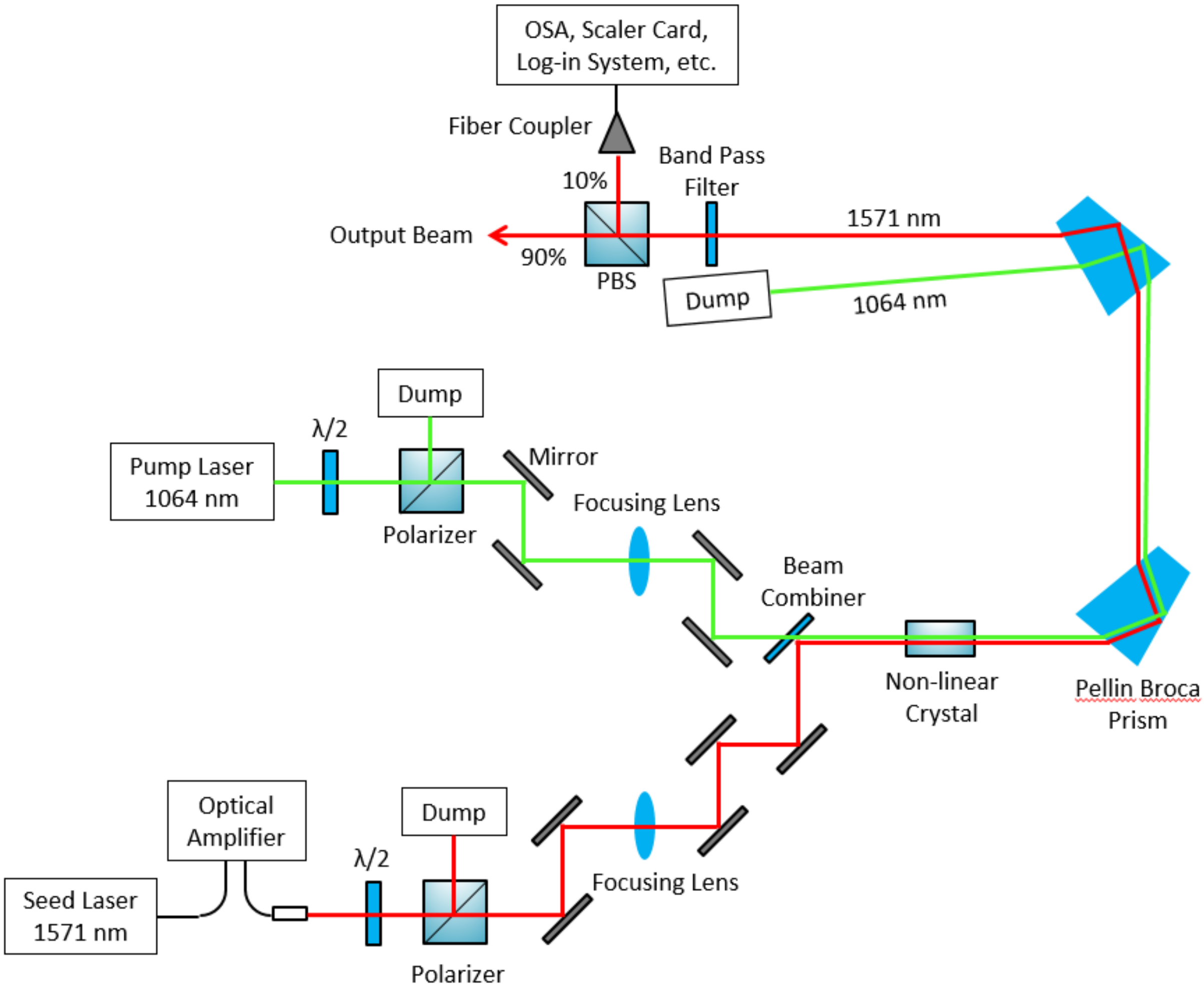

Figure 24. Schematic of the setup for the OPO laser.

For the best result, both the pump and the seed beam were carefully aligned parallel to the optics table. As an example, the measure of the centroid location of the pump beam is shown in figure 25. Careful alignment of the infrared beams is critical for maximizing the laser performance.

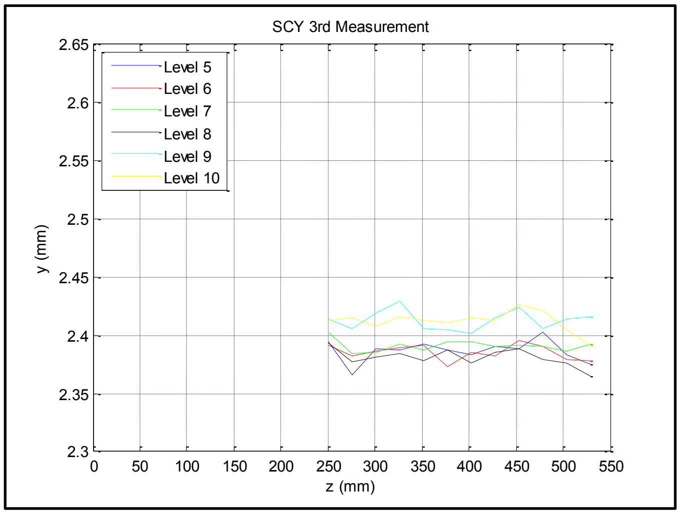

Figure 25. Pump beam centroid as a function of the pump power at the crystal location.

The pump beam size and the Gaussian fit for each energy level setting are shown in figure 26. The calculated beam waist, waist location, and Rayleigh range are shown in the label. The location of the focus changes significantly due to thermal lensing and can greatly affect the performance of the OPO laser. Documenting this and designing the attenuation scheme shown in Figure 24 will minimize thermal lensing, help avoid damage, and will be important when comparing the completed SNLO modeling with performance of the actual OPO laser.

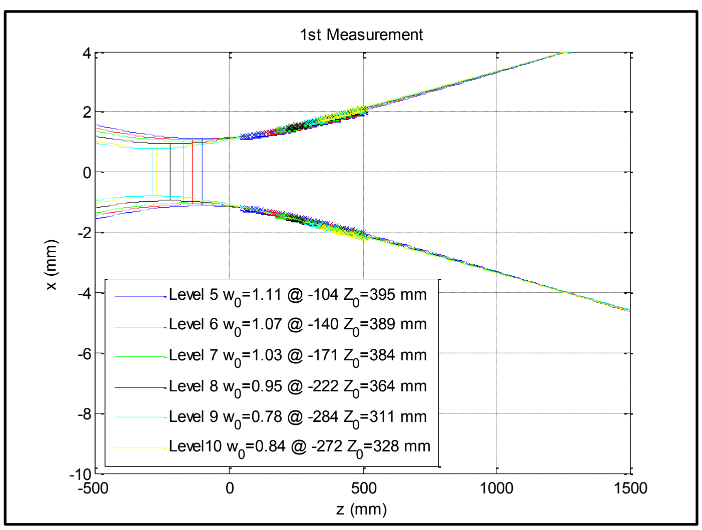

Figure 26. Beam waist and Rayleigh range as a function of pump power for the pump laser.

The focal length and the location where to position the focusing lens for the pump beam was optimized for the best result. Both one lens and two lenses calculations with various combinations of all commercially available focal length and location were considered. As an example, the figure below shows the result of a custom Matlab program indicating the focus location and spot size when scanning the location for a fixed focal length at 250 mm lens. This modeling was completed for all the important optical elements used in the OPO laser setup.

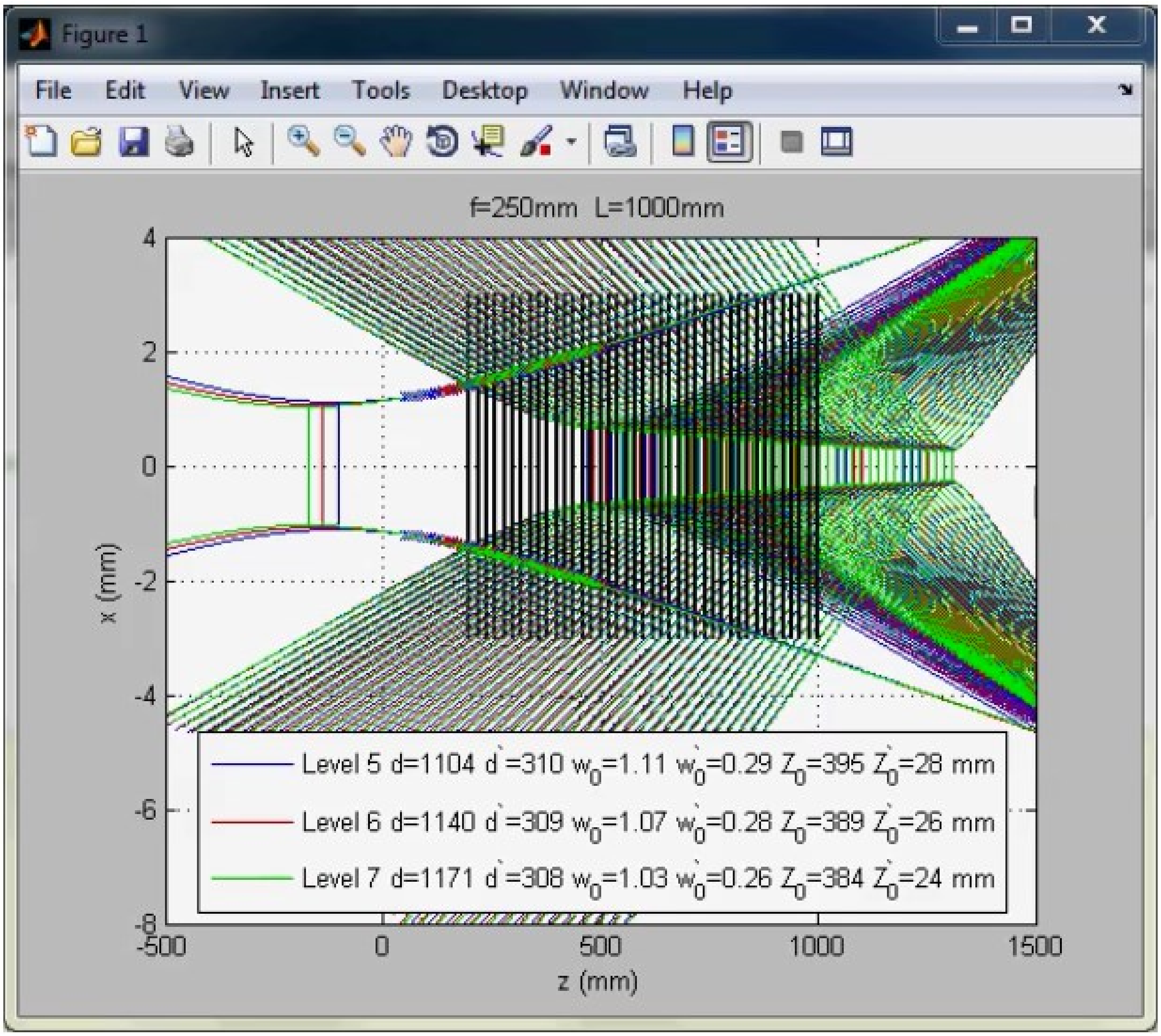

Figure 27. Custom Matlab programs developed for choosing and locating optical elements.

The beam waist, the waist location, the new Rayleigh range, and the beam size at the crystal face versus the lens location for various lens focal lengths are calculated to achieve the best overlap within the gain region for the OPO laser. An example of a calculation plots for a 250 mm focal length are shown in Figure 28.

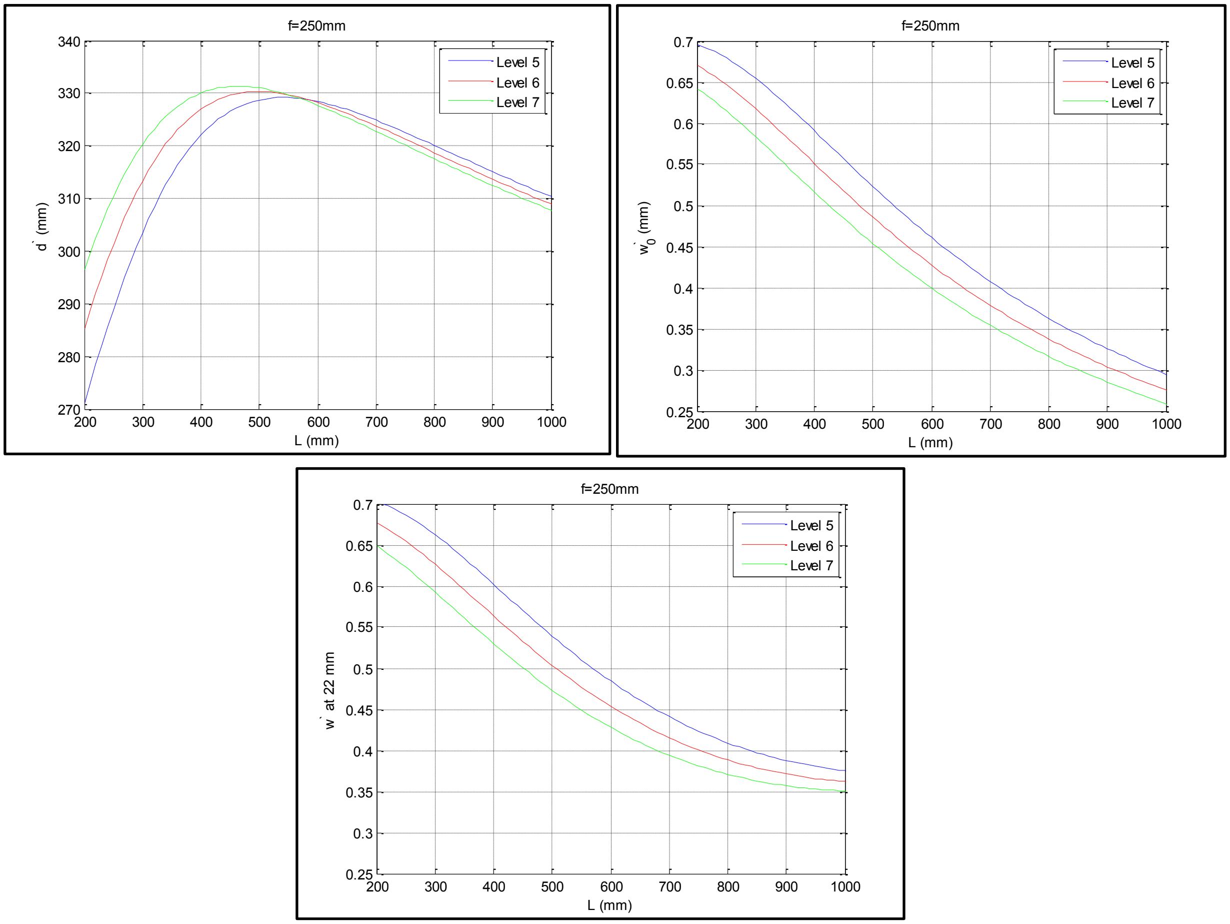

Figure 28. Optimizing the beam size and Rayleigh range for the OPO gain region.

Once the optimization for the focusing lens of the pump beam is finished, the selected lens had been placed at the selected location and aligned. The crystal was then carefully placed and aligned at the new waist location or the focal point. The same optimization process was used again to back calculate the focal length and the location of the focusing lens for the seed beam. Another focusing lens after the crystal was also optimized to refocus the beam at the fiber coupler. After several attempts of a very careful alignment, the measurement and Gaussian fit of the pump beam and seed beam through the crystal are shown below in Figure 29. The waist locations between the pump and seed beams are only 1 mm apart on the x-axis and only 2 mm apart in the y-axis. The black box on the plot indicates the crystal and it shows that both beams are completely within the crystal.

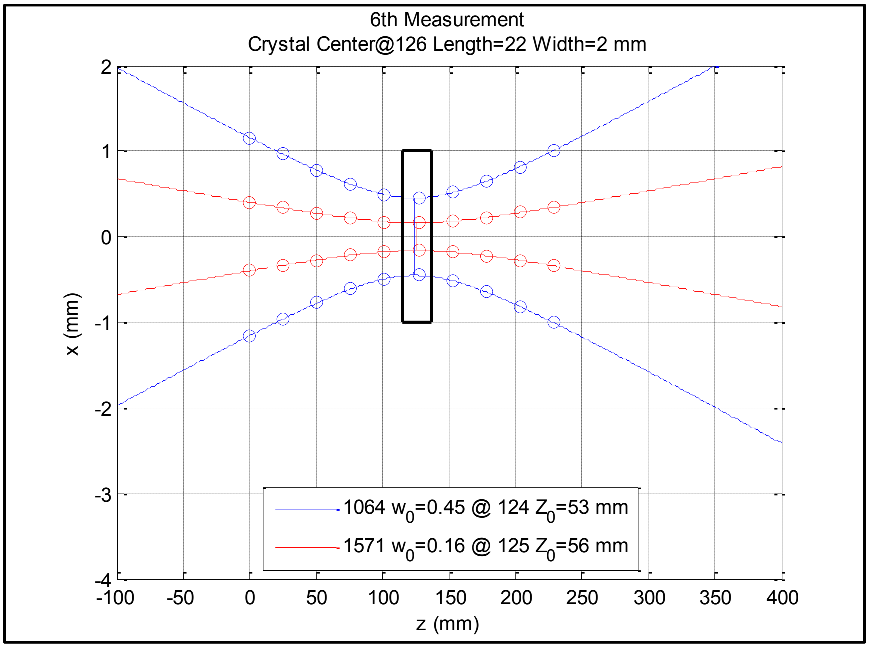

Figure 29. The pump and seed beam profiles measured through the location of the crystal.

Next, focus was turned to the OPO crystal housing. The crystal's poling period can be adjusted via thermal expansion. The thermal housing crystal was designed and made as shown in Figure 30.

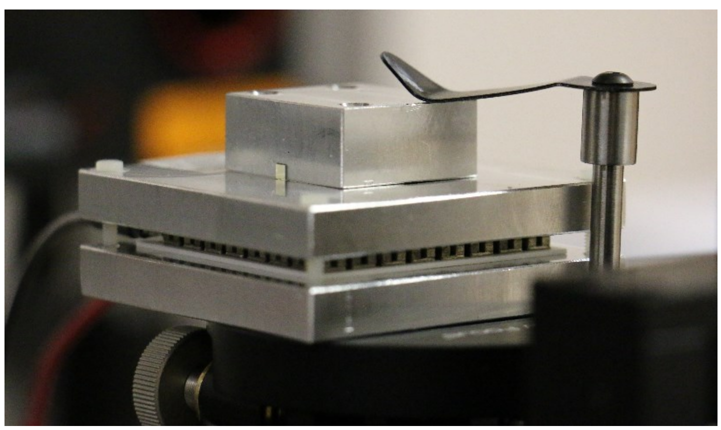

Figure 30. Custom thermal housing for the OPO crystal.

A plot shown in Figure 31 shows how the crystal temperature affects the output peak wavelength in the OPG case. The legend indicates the crystal's poling period in microns.

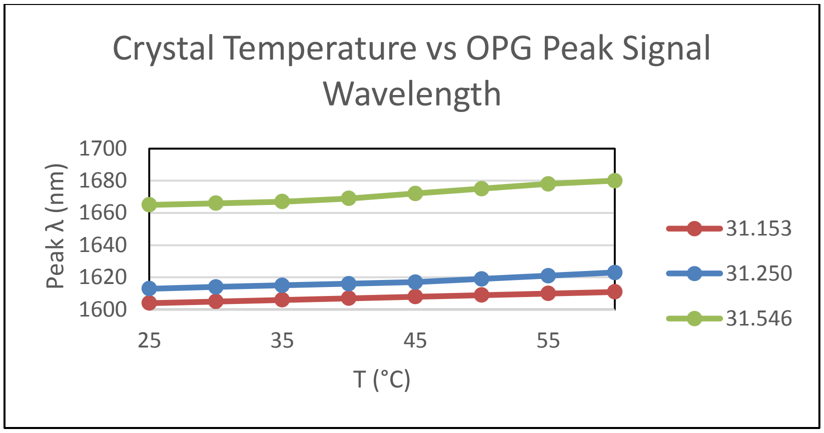

Figure 31. The wavelength for phase matching, as measured using optical parametric generation, as a function of temperature and poling period.

The peak wavelengths for each crystal were averaged and plotted against their poling period as shown below in Figure 32. To get a peak wavelength at 1571 nm, the quadratic fit estimates the poling period of 30.677 microns and the linear fit estimates the poling period of 30.943 microns. The appropriate crystal has been ordered. Once this crystal arrives it will be used in the OPO setup shown in Figure 33.

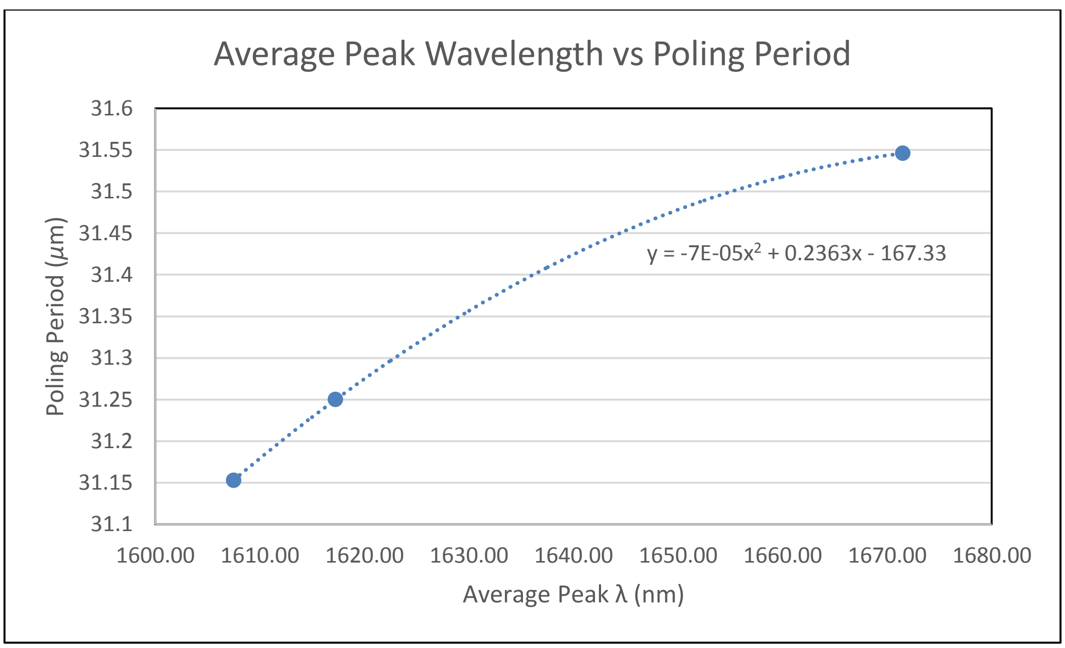

Figure 32. The poling period needed for the operating wavelength.

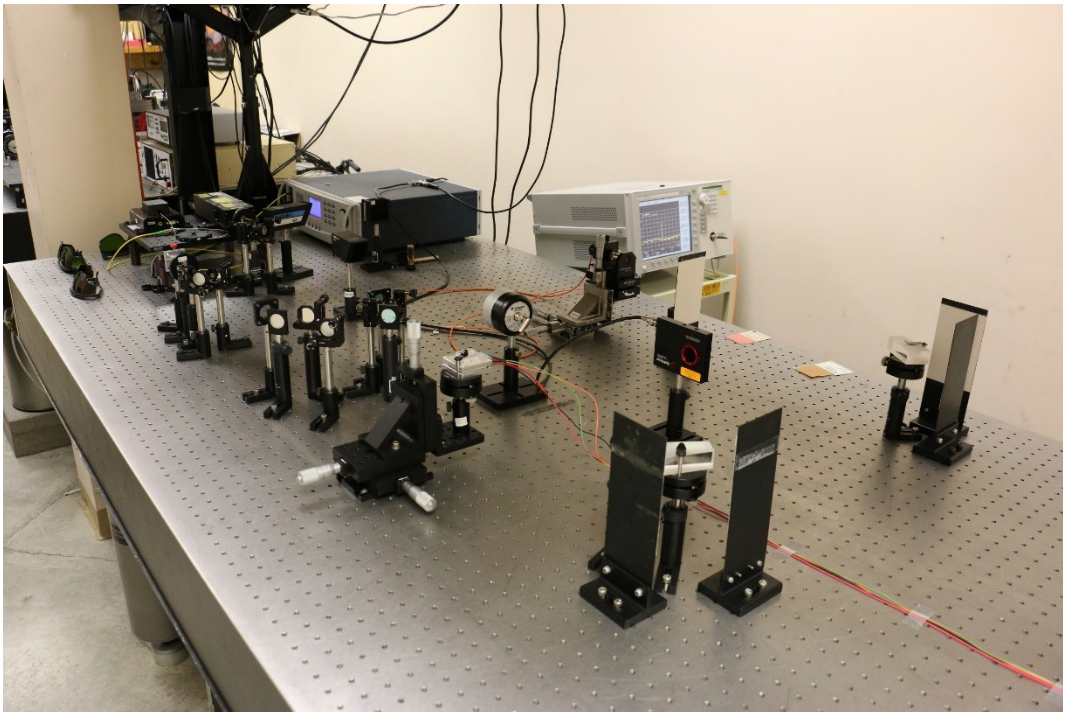

Figure 33. The final OPA/OPO setup.

At this point in the project, the SNLO modeling has been completed along with optical modeling and measurement allowing the setup of the OPO laser system shown in Figure 33. With the arrival of the crystal, the optical parametric amplification (OPA) and optical parametric oscillation (OPO) will begin to be characterized next quarter.

Expenditures to date

Salaries $19,144.40, Benefits $1005.44, Operations $16,646.19, total expenditures $36,796.03.

Subproject 10: Nonlinear Optical Detection of Surface Contaminants (Rob Walker, rawalker@chemistry.montana.edu, with Altos Photonics). Develop a new method for detecting organic contaminants that accumulate on the surface of water based on nonlinear vibrational overtone spectroscopy (NVOS).

Milestones

a) December 2015: Demonstrate feasibility of using new spectroscopic method for surface detection of adsorbed species
b) June 2016: Submit SBIR application with Altos to develop detection and monitoring instrument based on NVOS
c) December 2016: Successful application of NVOS to environmentally relevant systems including contaminants on water surfaces and solid substrates
d) June 2017: Final report summarizing technical accomplishments and commercial potential.

Activities to date

This project's goal is to develop new surface specific, optical methods capable of detecting adsorbed molecules. Specifically, our efforts are focused on exploiting the advantages of nonlinear optical spectroscopy to create a simple, sensitive technique that can identify the presence of organic contaminants at water/air and solid/liquid interfaces. Our ultimate objective is to use discoveries from our seminal studies to guide the development of portable devices capable of being used for field measurements.

The first quarter of 2016 began slowly but with several notable advances at the end of March. For the first two months of 2016, we worked to repair the Ti:sapphire regen amplifier and OPA that had suffered mechanical/optical damage at the end of 2015. At the end of February, we arranged for a visit from the Coherent service engineer who replaced several optics and retuned the regen cavity as well as the stretcher/compressor unit. Power was back to 3.8 W (at 800 nm, 1 kHz, 85 fs pulse duration) out of the regen and ~120 mW of power in the idler signal from the OPA (at ω_{idler} = 1.75 μm). These levels were similar to performance at the beginning of December, 2015, so we set about realigning optical paths on the table and rebuilding our NLO spectrometer assembly.

The proposed NLO methods to be developed in this project build off of conventional surface specific, 2nd order NLO (or $\chi^{(2)}$) techniques. Second order NLO spectroscopies, including second harmonic generation (SHG) and sum frequency generation (SFG), are techniques used to investigate the behavior of molecules at interfaces. Due to their inherent selectivity for the molecules at the surface, they allow detection of spectroscopic signatures from molecules influenced by surface anisotropy without interference from the molecules that make up the isotropic bulk materials on either side of the interface. However, the interfaces that can be examined by these techniques are limited by the need for complicated instrumental set-ups and, in the case of buried interfaces between two condensed phases, the limited transparency of many bulk materials in the visible and infrared (IR) regions of the electromagnetic spectrum, where SFG and SHG provide the most information.

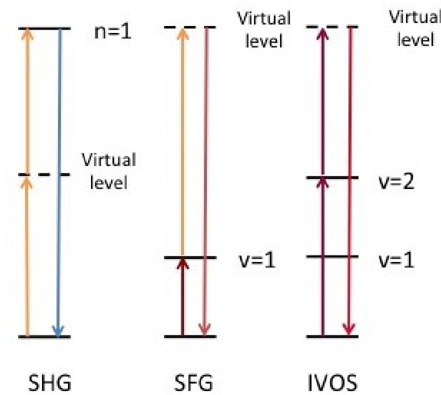

Figure 34: Energy level diagrams for relevant nonlinear optical spectroscopies.

Second harmonic generation spectroscopy (SHG) uses two incident photons of frequency ω to generate a single resultant second harmonic (SH) photon of frequency 2ω, and is typically performed with incident photons in the visible region, generating photons in the ultraviolet. When either the incident or the generated second harmonic light is resonant with an electronic transition of the interfacial molecules, the SH signal experiences a large resonant enhancement, making SHG well-suited to study the electronic structure of interfacial molecules and materials. Figure 34 shows the relevant energy level diagram.

Sum frequency generation (SFG) is similar in principle to SHG. In SFG, 2 photons of frequencies ω_1 and ω_2 combine to give a single photon of frequency ω_3, where $\omega_3 = \omega_1 + \omega_2$. A typical SFG experiment uses incident beams in the visible and infrared (IR) regions to generate a sum frequency (SF) photon. When the IR frequency is resonant with a vibrational transition of the interfacial molecules, enhancement of the SF signal will occur, allowing detailed structural information about the interfacial molecules to be gathered. SFG requires that 2 pulsed laser beams overlap at the sample in both time and space, as well as necessitating that at least one medium making up the interface be transparent to both visible and IR light. These technical constraints limit the utility of SFG when studying buried interfaces.

The method being developed by this project intends to use the vibrational overtones of molecules at surfaces to generate a 2nd harmonic field capable of being detected by conventional photomultiplier tube and Si array detectors. This new technique – dubbed IVOS or 'Interfacial Vibrational Overtone Spectroscopy' – will provide the vibrational specificity of SFG but with the ease of a single field in-single field out SHG experiment. Furthermore, by using light in the near infrared, this technique should be able to probe buried interfaces that are inaccessible to mid-IR frequencies (and conventional SFG experiments).

In order to fully develop IVOS, we needed to once again re-establish our ability to make conventional vibrational SFG measurements. This task was accomplished first by measuring the

SFG spectrum off of a plain Au surface followed by a vibrational SFG spectrum acquired from the dimethylsulfoxide (DMSO)/air liquid/vapor interface. These data are shown in Figure 35.

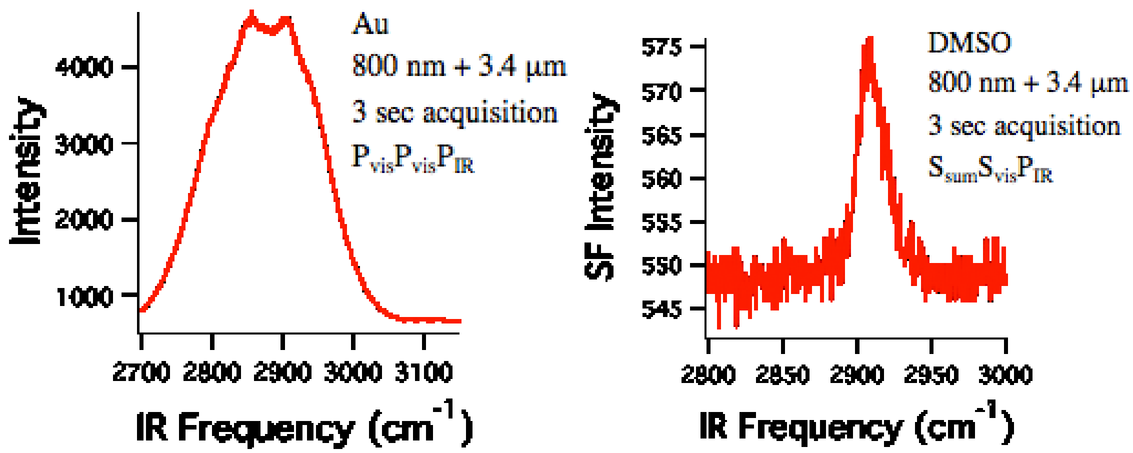

Figure 35: SFG spectrum of a plain Au surface (left) and vibrational SFG spectrum acquired from the dimethylsulfoxide (DMSO)/air liquid/vapor interface (right).

These measurements compare favorably to standard, benchmark data acquired in Fall, 2015 and inspired confidence that we could begin to develop the IVOS technique. To do so, we needed to order several optics and optical filters in order to deliver ω_{idler} from the OPA to the sample platform, free of any other higher order optical fields and we needed to re-set beam paths for the new field. Our first measurement (made at the end of March) used ω_{idler} (at 1.75 μm) and 800 nm light to measure a nonresonant SFG spectrum from gold. This spectrum is shown in Figure 36. To the best of our knowledge, this spectrum represents the first SFG spectrum acquired using near-IR output from an OPA.

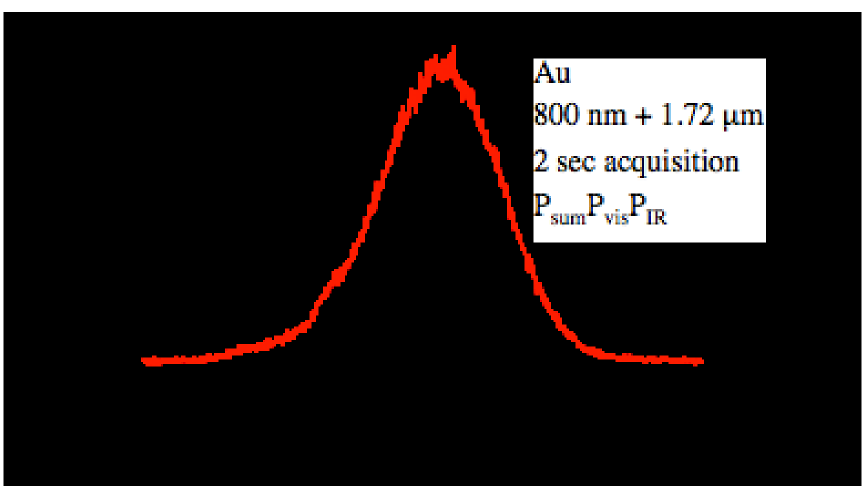

Figure 36: Nonresonant SFG spectrum of gold measured with ω_{idler} at 1.75 μm and 800 nm light.

In Q4, we will begin developing IVOS to probe *molecular* resonances rather than measuring the non-resonant response from metal surfaces. In order to develop IVOS without the benefit of the non-resonant signal from gold, we will use a molecularly similar system to the thiol monolayer on Au. Solutions of lipids deposited on an aqueous surface form self-assembled monolayers (SAMs), which have already been studied by SFG in several research groups including our own. Since surface order increases the second order non-linear signal, highly ordered SAMs are a good candidate for exploration of IVOS without the non-resonant signal enhancement from the gold surface, although the level of ordering in insoluble monolayers at aqueous interfaces is dependent on the lipid concentration at the surface. Thus, the lipid concentration, as well as chain length, level of branching and saturation will be varied to determine the effect of those variables on the IVOS signal.

The last experiments to characterize the IVOS signal will focus on less-ordered liquid/vapor interfaces. After lipids, the next surfaces to be characterized will be the liquid/vapor interfaces of common laboratory solvents, beginning with dimethylsulfoxide (DMSO), as its SFG response for the fundamental CH vibration is very strong. Other solvent interfaces to be explored will include acetonitrile (MeCN) and methanol (MeOH), because our group and others have carefully examined the behavior of those molecules at interfaces in the past.

<u>Expenditures to date</u>
Salaries $8,580.33, Benefits $663.62, Operations $8,562.83, total expenditures $17,806.78