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Metal Oxide Nanocomposite Nanoliter Reaction Chamber Fabrication and Applications for Harsh Environment Gas Sensing

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Metal Oxide Nanocomposite Nanoliter Reaction Chamber Fabrication and Applications for Harsh Environment Gas Sensing

Grant Proposal in partial fulfillment of requirements for graduation from the College of Nanoscale Science and Engineering at the State University at Albany with Honors in Nanoscale Engineering, B.S.

Michael C. Briggs

Research Mentor: Nicolas Joy

Research Advisor: Michael Carpenter, Ph.D.

May 2013

Abstract

In order to perform research at the College of Nanoscale Science and Engineering, funding for the desired research is required. Undergraduates get around that by working under a graduate student or professor who is currently funded. For those who seek funding, they receive it by submitting a grant proposal stating the motivation for research, the background, and previous research done to support this new endeavor, as well as future research plans that will be possible with funding. These grant proposals follow certain formats depending on where the principal investigator is seeking funding. As part of the honors requirement for the undergraduate B.S. through CNSE, a mock grant proposal is required.

This mock grant proposal is to be based on the Capstone Research done through the last 3 semesters of the program. My research was done under Dr. Michael Carpenter with the assistance of Nicolas Joy, who will be receiving his Ph.D. this May. Dr. Carpenter's research is funded by the Department of Energy [DOE], National Science Foundation [NSF] and industry partners.

This mock grant proposal in particular follows the National Institute of Health [NIH]. The NIH proposal outlines a problem that needs solving, as well as aims that are done to solve the problem. The problem in this proposal is the inability to sense harmful emissions in jet engine turbines. My solution is to fabricate a reaction chamber to assist in Dr. Carpenter's group metal-oxide nanocomposite harsh environment gas sensing research. The proposal also includes a detailed budget based on funds need to perform research for one year.

Acknowledgements

First and foremost, I would like to thank Dr. Carpenter for taking me under his wing in my sophomore year and letting me begin research with his group when few undergraduates had even thought about beginning research. I would also like to thank Nicolas Joy for putting up with me fellow undergraduate Brian Janiszewski when we had questions upon questions throughout these past two and a half years. In the past year, I have gotten a lot of assistance from two graduate students beside Nick. Prakash Dharmalingam has helped me with taking microscopy images from the scanning electron microscope as well as patterning samples with the e-beam tool in the cleanroom. James Williams has assisted me with all of the lithographic processes needed to fabricate my reactor components. He was patient and understanding of the fact that these lithographic processes were uncharted waters for us in the sensing group.

Through a different scope, I would like to thank my friends who supported me throughout these past 4 years. I am grateful not only for my friends in the nanoscale engineering program who stood by me through thick and thin, but for my other friends who I have met through the Honors College and other clubs and activities. Without an outlet to talk to and take my mind off course work, stress would have been much larger part of my life.

Lastly, and most importantly, I would like to thank my parents and siblings who always supported me through my academic career. As I grow up, I realize more and more how truly grateful I am for having the family I have. I never once with without, but I always knew the value of hard work and dedication. My parents never demanded perfection but they expected me to work my hardest in everything that I do. I attest that my success through my academic career. My family may not be scientists or engineers, but they are strong, dedicated, intelligent individuals who instilled those exactly qualities in me as I grew up.

Metal Oxide Nanocomposite Nanoliter Reaction Chamber Fabrications and Applications for Harsh Environment Gas Sensing

Principal Investigator: Michael Briggs
University at Albany, College of Nanoscale Science and Engineering
One-Year Project
06/01/2013 - 05/31/2014
$144,956.57 Requested for One-Year

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PROJECT SUMMARY

Problem: **Currently, gas sensors cannot perform reliably in harsh environments such as extremely high temperatures.** The materials that make up these sensors degrade at high temperatures and are unable to give accurate sensing measurements. Applications such as jet turbines need harsh environment sensors to measure the emissions released during combustion. Without these sensors, compliance with emission regulations are difficult since it is unknown how much harmful emissions are being put into the atmosphere during combustion.

Solution: **The solution we are proposing uses a material consisting of metal nanoparticles in metal-oxide nanocomposites to sense gas concentrations at high temperatures.** Yttria-stabilized Zirconia [YSZ] is a metal-oxide complex with an oxygen vacancy in its cubic lattice. The oxygen vacancy along with gold nanorods encapsulated by a YSZ film act as a sensor. Gold nanorods have a characteristic surface plasmon resonance [SPR], which has peaks in the visible or infrared light spectrum depending on their aspect ratio. These peak positions change in response to the environment they're exposed to.

Our proposal is to create a micro-liter reaction chamber made of gold nanorods encapsulated in YSZ. Gas inlets and outlets will be attached to the chamber allowing optical gas sensor testing. Heaters will be attached to the chamber to heat it to 500 C during exposure periods.

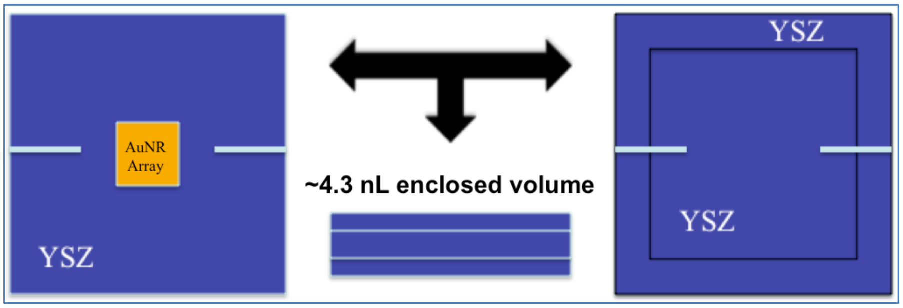

Figure 1 Both Components of the nanoliter reaction chamber bonded together

Aim 1: **Fabrication of micro-liter reaction chamber.** Gold nanorods will be patterned using an electron beam and YSZ films will be deposited using Radio Frequency Physical Vapor Deposition [RF PVD]. A die bond will mechanically bond two gold-YSZ samples to one another creating a chamber with two openings for gas line connections.

Aim 2: **Optically sense gas concentrations as a function of the change in SPR peak position.** The samples will be exposed to gases such as hydrogen, carbon monoxide and nitrogen dioxide of varying concentrations. The SPR peak positions will shift to higher or lower energies depending on the type of gas present in the environment as well the concentration.

Success Criteria: Success in phase 1 will be achieved by successfully fabricating the micro-liter reaction chamber with an airtight seal and nanorods stable at high temperatures, resistant to changes in shape. Success in phase 2 will be achieved by collecting reliable and repeatable changes in peak position unique to gas types and concentrations.

Future Use: This micro chamber system will make other experiments possible as well. Electrically based sensing can be investigated as well as gas-kinetics experiments. Leads can be attached to the thin film composites to measure the change in resistance as a function of gas compositions and concentrations. Also, a gas concentration mass spectrometer can be attached to the reaction chamber to measure exactly how much gas molecules go into the chamber, as how many come out. These experiments can not only help make better sensors but also further develop the sensing mechanism behind the charge exchange between the environment, the oxygen vacancies and the gold nanoparticles.

PROJECT DESCRIPTION

Motivation

Current harsh environment gas sensors are unreliable and costly to produce. With emission regulations becoming more and more strict, accurate and reliable high temperature gas sensors are necessary to regulate turbine emissions in jets and airplanes. Current technologies used include field effect sensors, oxide semiconductors, fluorescence, non-dispersive infrared, and chemiluminescence spectroscopy. [8] These technologies have reliability, selectivity and cost issues. Harsh environment sensors need to be able to detect turbine exhaust gases such as NO_2, CO and H_2 in the ppm and ppb range. [1][4][6][7] Our group has been able to demonstrate gas sensing in these ranges using Gold-Yttria Stabilized Zirconia [Au-YSZ] nanocomposite structures.[1] Using optical spectroscopy and the plasmon properties of gold, changes in the Localized Surface Plasmon Resonance [LSPR] peaks can be used as the sensing mechanism to detect these exhaust gases at high temperatures. [1][4][5][6][7]

Despite being able to sense gases in harsh environments using the nanocomposite structures, the sensing mechanism is not completely understood. A sealed reaction chamber with a small volume can serve as a multi-purpose tool to perform both kinetics and optical spectroscopy experiments to further investigate the sensing mechanism in the Au-YSZ nanocomposite materials system.

Background

Gas Sensing: Noble metals are well known for their light absorption properties. Metals have a property in which they absorb light at a specific wavelength range and that absorption is amplified due to its geometry. [5] The localized surface plasmon resonance [LSPR] spectrum of a material is the collective oscillations of electrons due to the electromagnetic field associated with photons. Gold SPR spectra have a broad peak in the visible light. [1][4][5][6][7]

In our gas sensing, the gold-YSZ nanocomposite is exposed to a gaseous environment and a light source simultaneously. The LSPR peak position is extremely sensitive to the environment around it. Depending on the gases in the environment, the peak position of the LSPR [peak] will shift to higher or lower energies. The amount of shift in the peak position can determine the amount of gases, such as carbon monoxide or hydrogen, are in the environment. These sensors can detect gases in the parts per million ranges. Sensing tests are done normally at 500C. YSZ is a very stable material and harsh environment sensing is our aim.

The SPR spectra of gold particles are greatly affected by geometry. [5] Spherical gold nanoparticles have one broad absorption peak in the visible light. Gold nanorods have two peaks, which correspond to the lateral and longitudinal modes of the electron oscillations. It has been observed that the lateral and longitudinal peaks are sensitive to their environment, but they are also selective. This allow for another degree of sensitivity and selectivity in terms of gas sensing capabilities. [1]

SPR Peak Prediction: The SPR peak positions were predicted using a dipole approximation calculation. The peak positions are calculated by the emission and absorption properties of gold using Gans extension of Mie theory. Longitudinal peak positions for gold nanorods are a function of aspect ratio. The lateral peak position remains stationary, unaffected by the change in aspect ratio. The equation used to calculate the extinction of gold nanorods is the following:

$$\gamma = \frac{2\pi N V \epsilon_m^{3/2}}{3\lambda} \sum_j \frac{(1/P_j^2)\epsilon_2}{\left(\epsilon_1 + \frac{1-P_j}{P_j}\epsilon_m\right)^2 + {\epsilon_2}^2}$$

$$P_A = \frac{1-e^2}{e^2}\left[\frac{1}{2e} ln\left(\frac{1+e}{1-e}\right) - 1\right]$$

$$P_B = P_C = \frac{1-P_A}{2}$$

$$e = \sqrt{1-(\tfrac{b}{a})^2}$$

Figure 2 Extinction Equation derived from Gans extension of Mie Theory [2]

P_A, P_B and P_C are all functions, which are inversely proportional to the aspect ratio. The extinction (γ) is a directly proportional to the SPR spectrum so this is the perfect prediction for peak position as the gold nanorod dimensions change. [2] The complex refractive index of gold was used in the calculation and denoted as ε_1 and ε_2. [3]

Statement of Work

Sample Preparation: The substrates used to build the micro-liter reaction chambers are silicon and quartz. These substrates were chosen because they give a wide range of optical transparencies. Quartz is transparent in the visible spectrum as well as the near infrared frequencies. Silicon is transparent in the near to far infrared frequencies. The first step in fabrication is pre-cutting the 4cm^2 silicon and quartz samples into 1cm^2 squares using a diamond saw. This allows the substrate samples to be broken into exact sized pieces after deposition and anneal. The next step is to deposit 60nm of YSZ onto the substrates using Radio Frequency co-magnetron confocal Physical Vapor Deposition [RF PVD]. The PVD target used was 99.9% pure 5 wt% YSZ. Sputtering occurred in argon (5 mTorr) with the target at 200 W. The substrates are then annealed for 1 hour at 900 C in 2000 standard cubic centimeters [sccm] of Argon. The anneal step is done to crystallize the amorphous film formed by PVD deposition.

The reaction chamber consists of two components. The first component is fabricated by first spin coating the substrates with a thin film of Standard Photoresist [Spr] 1813. The spin coat recipe is 45 seconds at 3000 RPM to result in a film of around 10 microns. Next, a soft bake at 90 C is done for 1 minute to set the photoresist. The samples are then placed in an exposure tool and exposed to ultraviolet light through a mask for 22 seconds. There are two separate masks for chamber fabrication. This step involves the mask to create the annular region [see Figure 3]. Both masks were designed using a program called L-Edit, printed on transparency paper and taped smoothly to a 7" piece of glass. After exposure, the Spr 1813 is developed using AZI 300 MIF developer until the exposed resist is removed from the sample.

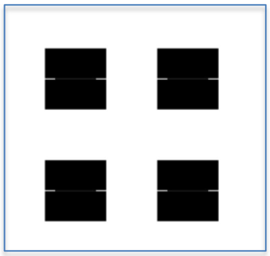

Figure 3 Exposure Mask 1

Next, the substrates are placed into the RF PVD system again and a 120nm YSZ film is deposited on top of the remaining Spr 1813 layer. The Spr 1813 layer is lifted off by placing the substrates into boiling acetone for 5 minutes. The YSZ on top of the remaining photoresist is removed leaving an annular region of YSZ on top of the already deposited 60nm YSZ layer from the first deposition. The YSZ film is once again annealed at 900C for 1 hour under 2000 sccm of Argon.

Gold Nanorod Patterning: The gold nanorod [AuNR] arrays are designed and saved to a .gds file using a program online called Layout Editor. The nanorod dimensions are designed specifically for the silicon and quartz substrates. Gold nanorods with lower aspect ratios will have surface plasmon resonance [SPR] peaks in the visible and near infrared [IR] frequencies. These will be patterned on the quartz substrates. AuNRs with higher aspect ratios will be patterned on silicon wafers since they will have SPR peaks in the IR wavelength range. The patterned areas are 2 mm^2 in size and have marks outside them so patterned areas can be seen with naked eye after patterning. Prior to patterning, the substrates with 60nm of deposited YSZ on them will be spun coat with PMMA. These are samples that have yet to undergo any lithographic processes. The samples are placed in the Vistec Electron Beam patterning tool. The .gds files for the AuNR arrays are input into the computer and electron beam prints out the areas where the AuNR are to be placed. Six nanometers of titanium are evaporated onto the substrate followed by a 30 nm film of gold. The Ti/Au film on top of the unpatterned PMMA is lifted off by placing the substrate in boiling acetone for 5 minutes.

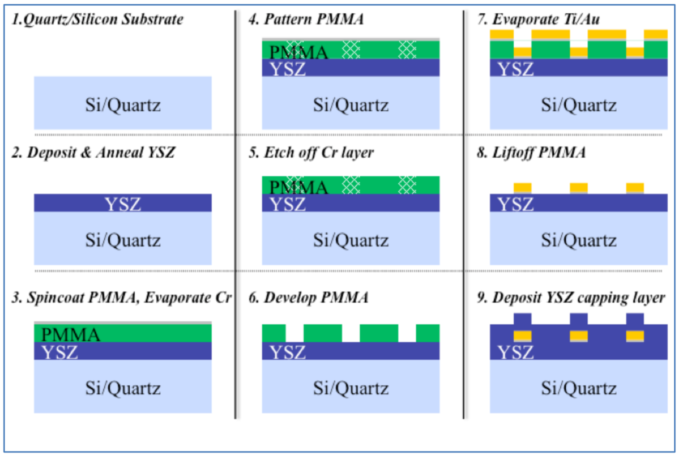

Figure 4 Nanorod Patterning Process Steps

Gas Line Trench Fabrication: Using the exact lithographic process as stated previously in ***Sample Preparation***, both the annular component and the AuNR array component are exposed using the second mask [See Figure 5]. Post exposure, a hard bake at 115C for 5 minutes is performed to set the photoresist, make it more durable. The samples are then mounted to a 200mm Si wafer using nonreactive adhesive tape and placed in a Reactive Ion Etch chamber [RIE]. After optimization of etching parameters, the YSZ/Si is etched to a depth of 50 microns. The YSZ/Si etches roughly 20 times faster than the photoresist. After the RIE step, the remaining Spr 1813 is removed by placing the substrates in boiling acetone as performed in previous steps. These trenches will house 100 micron diameter Quartz tubes which will flow gases through the reaction chamber when completed. Now both components of the nanoliter reaction chamber are fabricated and ready for bonding.

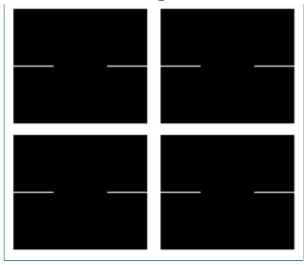

Figure 5 Exposure Mask 2

A fabrication process overview for the two reaction chamber components can be found in Figure 6. NOTE: Step 9 which repeats steps 2-6 involves exposure mask 2 found in Figure5.

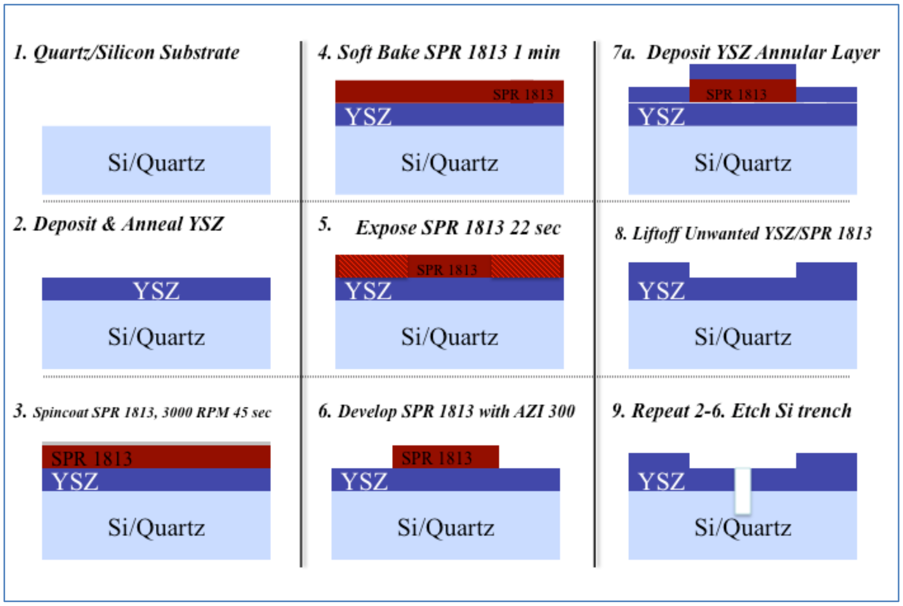

Figure 6 Chamber Component Fabrication Process Layout

Die Bonding: The YSZ coated substrates will be broken up into their 1cm^2 pieces by breaking along the pre-cuts. Next the samples will be placed into a die bonder owned by SEMATECH to bond the samples with the patterned rods to the samples with the annular regions. The tool is a SET FC-300 High Precision Die/ Flip Chip bonder.

Future Work

Fabrication Optimization: Two main regions where optimization is needed is in regards to the anisotropic RIE of the YSZ/Si as well as direct bonding of YSZ-YSZ. Since YSZ is mainly only used in Solid Oxide Fuel Cell applications, little research has been done in regards to direct bonding and YSZ etching. The etch rate of YSZ is unknown but it can be assumed that it etches similarly to Si and SiO_2 [Quartz]. Tests need to be performed to investigate this. Metrology techniques such as Scanning Electron Microscopy will be used to determine etch depth.

Die Bonding tests need to be performed to determine the appropriate parameters needed for successful YSZ-YSZ bonding. The main parameters that need to be optimized are temperature, length, mechanical pressure and, in relation to both duration and pressure, stress profile.

Preliminary Tests: Assuming successful bonding parameters are discovered, initial tests can be done with enclosed chambers, those being samples which were bonded without the RIE etch step. These experiments with the bonded samples will consist of temperature dependence measurements of Raman spectroscopy spectra. Realistically, there will be trapped hydrocarbons in the enclosed reaction chamber. These hydrocarbons have a signature Raman peak whose intensity will be dependent on temperature. This temperature dependence can be observed and help with our sensing work since hydrocarbons are one of the unwanted emissions in jet turbines.

Gas Sensing Tests: With fully fabricated metal-oxide nanocomposite nanoliter reaction chambers, optical spectroscopy tests can be performed. The chambers will be heated to 500 C and exposed to different gases at varying concentrations. During testing, the SPR spectra will be collected and the change in peak position for both the transverse and longitudinal peak can be calculated using a computer automated python program. Nanoliter reaction chambers made of quartz will be optically exposed to visible light and silicon nanoliter reaction chambers will be optically exposed to IR light and only the longitudinal peak can be collected using Fourier Transform Infrared [FTIR] spectroscopy since the transverse peak is found in the visible wavelength range.

Further out, the nanoliter reaction chambers can be coupled to a Gas Chromatography Mass Spectrometer, which will measure the gases going into the chamber as well as the gases coming out, providing useful kinetics information. An ideal test would be to perform optical spectroscopy side-by-side with a GC Mass Spectrometer to get real-time kinetics data to couple to observed shifts in SPR peak position. This data would prove to be most useful in determining the sensing mechanism of the Au-YSZ matrix.

Proposed Research Timeline

Month 1-2
- Si/SiO_2 RIE Optimization
- Die Bonding Parameter Investigation

Months 3-4
- Further Fabrication of Reaction Chambers
- Prelimenary Temperature Dependent Raman Experiments

Month 5-6
- Optical Gas Sensing Experiments
- GC Mass Spectrometer set-up

Month 7
- GC Mass Spectrometer Gas Exposure Kinetics Experiments
- Write Program to simultaneously collect and analyze GC Mass Spec and Optical Spectroscopy data

Month 8-9
- Simultaneaous GC-Mass Spec/Opticacal Spectroscopy Gas Sensing Experiments

Month 10-12
- Compile Collected Gas Sensing data to be used in publications
- Investigate Possible Future Electronic-Based Gas Sensing Possibilities

REFERENCES

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[5] P. H. Rogers, “Particle Size Sensitivity Dependence of Nanocomposites for Plasmonic-Based All-Optical Sensing Applications,” *The Journal of Physical Chemistry C*, vol. 114, no. 25, pp. 11033–11039, 2010.

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BIOLOGICAL-SKETCH

Name: Michael C. Briggs
Position Title: Project Investigator
Education: Undergraduate
  Institution: University at Albany, College of Nanoscale Science and Engineering [CNSE]
  Degree: Bachelor of Science
  MM/YY: 05/2013
  Field of Study: Nanoscale Engineering Major, Mathematic Minor

A. Personal Statement:

I am currently a graduating senior at CNSE and have been a part of the undergraduate nanoscale engineering program since its establishment in the spring of 2010. I have worked with Dr. Carpenters sensing group for two and a half years and am very comfortable with all of the deposition processes and exposure benches. These past 12 months I have been working almost independently on taking this reaction chamber fabrication from an idea to a reality. With the assistance from other groups here at CNSE as well as advisement from Dr. Carpenter, I am confident that this research endeavor will be a success. The idea of one day being able to track data and relate a change in peak position to a gas composition read-out is very exciting to me. That capability alone may be the answer to the ongoing question of what the Au-YSZ sensing mechanism really is. I intend on personally revealing that mystery in due time.

B. Positions and Honors:

Positions

August 2012 – May 2013	Research Assistant, CNSE
May 2012 – August 2012	Intern, National Institute of Standards and Technology [NIST]
July 2011 – May 2012	Research Assistant, CNSE
June 2011 – August 2011	Intern, CNSE

Memberships

2009 – 2013	Presidential Honors Society
2009 – 2013	Honors College of SUNY Albany
2009 – 2013	Albany Business Leaders Emerging [ABLE]
2011 – 2013	Omicron Delta Kappa National Leadership Fraternity

Honors

Fall 2008	Dean's List of Extinguished Students
Spring 2009	Dean's List of Extinguished Students
Fall 2009	Dean's List of Extinguished Students

Spring 2010	Dean's List of Extinguished Students
Fall 2010	Dean's List of Extinguished Students
Spring 2011	Dean's List of Extinguished Students
Fall 2011	Dean's List of Extinguished Students
Spring 2012	Dean's List of Extinguished Students
Fall 2012	Dean's List of Extinguished Students

C. Research Support

Commented [MB1]: Ask Carpenter who this is funded by and what the official title is

BUDGET AND JUSTIFICATION

Budget: College of Nanoscale Science and Engineering

Sponsor: SUNY RF			Title: Metal-Oxide Nanocomposite Nanoliter Reaction Chamber and Applications for Harsh Environment Gas Sensing		
Project Investigator: Michael Briggs			Co- Project Investigator :		
Period of Time: 1 YRS					
YEAR 1					
Salaries	**% Effort**	**Annual Salary**	**Total Cost**	**Requested Funding**	**Cost Share**
Faculty					
N/A			$0	$0	$0
			$0	$0	$0
			$0	$0	$0
Total State Paid employees			**$0**	**$0**	**$0**
RF Paid Employess					
Summer Intern [TBD]	100	$3,500.00	$3500.00	$3500.00	$0
			$0	$0	$0
Total RF Paid Employees	**100%**		**$3,500.00**	**$3500 .00**	**$0**
Graduate Students	**FTE**				
Name: Michael Briggs	**100%**	**$21,840.00**	$21,840.00	$21,840.00	$0
Name: TBD	**100%**	**$21,840.00**	$21,840.00	$21,840.00	$0
Total Graduate Students	100%		**$43,680.00**	**$43,680.00**	**$0**
Summer					
Michael Briggs	**100%**	**$6,000.00**	$6,000.00	$6,000.00	$0
			$0	$0	$0
Total Summer Salaries	0%		**$6,000.00**	**$6,000.00**	**$0**
Total Salaries			$53,180.00	$53,180.00	$0
Fringe Benefits	**Rate %**	**$ Base**			
State Employees	50.81%	$0	$0	$0	$0
RF Employees *	45.00%	$3500.00	$1,575.00	$1,575.00	$0
Graduate Students *	16.00%	$21,840.00	$3,494.00	$3,494.00	$0
Summer	17.00%	$1,020.00	$1,020.00	$1,020.00	$0
Total Fringe Benefits			**$6,089.00**	**$6,089.00**	**$0**
Total Salaries and Fringe Benefits			**$59,269.00**	**$59,269.00**	**$0**
Other Direct Costs - Attach Details for any item listed below					
Equipment			$15,000.00	$15,000.00	$0
*Tuition			$27,800.00	$27,800.00	$0
Travel			$2,500.00	$2,500.00	$0
Materials & Supplies			$5,000.00	$5,000.00	$0
Publications			$0	$0	$0
Total Other Direct Costs			**$50,300.00**	**$50,300.00**	**$0**
TOTAL DIRECT COSTS			**$109,569.00**	**$109,569.00**	**$0**
Facilities and Administrative Expense	Rate %	Base MTDC			
Does not incl:equip, install,tuit	53.00%	$66,769.00	**$35,387.57**	**$35,387.57**	**$0.00**
Total Estimated Project Cost			**$144,956.57**	**$144,956.57**	**$0**

Justification: Michael Briggs will be paid as per budget outlined as a graduate student working under Dr. Michael Carpenter. Another graduate student, to be determined, is budgeted to join the group in the Fall of 2013. A summer intern will be hired through the CNSE Summer Undergraduate Internship Program with payment provided through the SUNY Research Foundation.

There will be no necessary costs for equipment, as almost all tools necessary to complete our research are available to us through partnering CNSE laboratories. The only equipment budget of $15,000 is for e-beam lithography tool time on the Vistec in the NFS clean room. This tool time is a necessity to pattern our gold nanorod arrays used for sensing in multiple experiments.

We will require $5,000 for various materials and supplies such as gold, substrates, optics, gas cylinders etc. The travel budget of $2500 is allotted for the transportation of two graduate students to a yet to be determined conference. There they will present a poster and/or give a presentation and stay in a hotel for one night. Any travel funds not used will be left for possible costs that are unforeseen, such as repairs, new equipment and equipment upkeep.

The publications submitted by Dr. Carpenter are almost always submitted to journals in which there is no publication cost so a publication budget is not needed for our research.