U.S. DEPARTMENT OF ENERGY | Office of NUCLEAR ENERGY

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# Integrated silicon/chalcogenide glass hybrid plasmonic sensor for monitoring of temperature in nuclear facilities

**Advanced Sensors and Instrumentation Annual Webinar**

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**Boise State University**

# Project Overview

- **Goal and Objective**

Applying additive technology to develop very low power consuming reversible and multiple time applicable devices, which can be deposited directly over the measured surface for real time temperature monitoring.

- **Participants (2019)**
  - Dr. Maria Mitkova – Principal Investigator
  - Dr. Harish Subbaraman – Co- PI
  - Mr. Al-Amin Ahmed Simon (Graduate Student)
  - Ms. Bahareh Badamchi (Graduate Student)
  - Mr. Henri Kunold (Undergraduate student)
- **Schedule**
- Obtain radiation hard gold coated fiber
- Characterize the fiber and its radiation stability
- Finish the design of waveguide sensor architecture and simulation
- Start fabrication of silicon-chalcogenide hybrid integrated plasmonic temperature sensor
- Start fabricating and testing chalcogenide coated rad hard fiber tip based temperature sensor
- Conduct experiments in the co-PIs laboratories

# Accomplishments

- ✓Optimization of the design parameters of the integrated silicon photonic and optical fiber temperature sensors, based on the measured optical properties.
- ✓Designing grating couplers, power dividers, inverse taper couplers to account for packaging the sensor array.
- ✓Printed films with powders based inks and characterized their surface and structure.
- ✓Obtained gold coated rad hard fibers, established procedure for their etching, deposited ChG films on their tips and started setting up experiments for temperature sensing
- ✓Successfully set up the THMSEL600 ellipsometry heating stage with the ellipsometer available at BSU.
- ✓Improved the sintering of the printed films and achieved very low surface roughness .
- ✓The printed films were characterized through their composition, structures and optical constants in situ at variety of temperatures.
- ✓ Built a waveguide coupling and testing system for characterizing the optical performance of silicon waveguides.
- ✓ Fabricated and tested the performance of silicon waveguide devices on an SOI chip. The waveguides demonstrated low fabrication yield and high insertion loss.
- ✓ Identified e-beam tool issues causing yield and high loss characteristics from fabricated waveguides.
- ✓Conducted DSC at different heating rates for devices calibration
- ✓Stitching method in EBL has been applied to pattern the whole sensor design.
- ✓Patterned slits parts in grating coupler with E-Beam Lithography (EBL
- ✓Fiber-based devices have been fabricated and tested in a controlled atmosphere

# Technology Impact

- ✓ *The state of the art for nuclear application will be advanced by introduction of new type of devices suitable for:*
  - Monitoring LWR, metallic or ceramic SFR reactors where the cladding temperature can reach 650° C.
  - Cohesive temperature monitoring using integration of photonic properties of radiation hard optical waveguides for nuclear application.

- ✓ *The DOE-NE research mission will benefit from generating knowledge about the performance of different components in the reactor environment.*

- ✓ *Obtaining knowledge about the performance of the radiation hard waveguides in the reactor environment will impact the nuclear industry by extending their application to much more important characterization methods like for light transmission for in situ in pile Raman spectroscopy.*

- ✓ *The project will develop the technological documentation which will enable its commercialization*

# Introduction

Environment inside nuclear reactor

- High temperatures
- High radiation doses
- Corrosive conditions
- etc

Material property changes:
- Chemical
- Mechanical
- Microstructure

- To monitor material performance in harsh environments, real-time sensors are critical to improve stability and functionality.

Methods to monitor temperature inside nuclear reactor

**Thermocouples**:
- unreliable after short time exposure to radiation
- lose accuracy quite fast.

**Melt wires techniques:**
- lower resolution
- real-time monitoring not possible.

**Optical waveguide sensors:**
- high accurate measurement, multiple time use.

# Introduction

## Objective

Design small size, highly accurate, real-time, reusable and reversible temperature sensor for use within a nuclear facility.

Three different device architectures.

**Fused silica Optical Fiber** + **Chalcogenide Glass (ChG)** = **Fiber Tip based Temperature Sensor <u>Features</u>:** Elegant design, simple fabrication process, reversible Temperature sensor

**Nanophotonic Silicon Photonic Waveguide (SiPW)** + **Chalcogenide Glass (ChG)** = **Hybrid Plasmonic Waveguide (HPW) <u>Features</u>:** Real-time, reusable reversible Temperature sensor

**Si wafer** + **Chalcogenide Glass (ChG)** = **Step-Resistive Temperature Detector <u>Features</u>:** Easy and cheap fabrication, Real-time, reusable reversible Temperature sensor

# Gold Coated Fiber Characterization

Gold coated fiber was obtained from Fiberguide.

The outer diameter of the fiber is around **150µm** (153.3 ± 2µm). Gold top layer **12.5µm**, Cladding **10µm**, Core **105µm.**

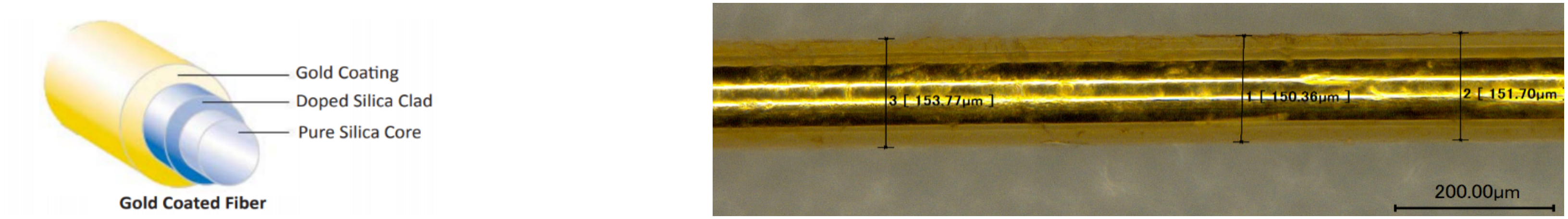



**Figure. Schematic of the gold coated fiber and actual fiber and geometry.**

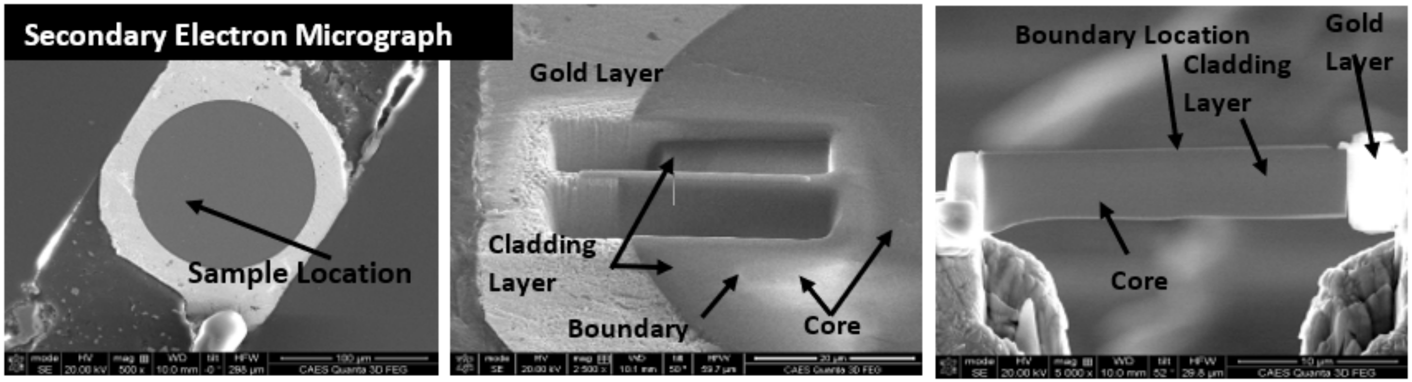



**Figure. Cross section of the fiber and FIB lamella.**

# Gold Coated Fiber Irradiation and Characterization

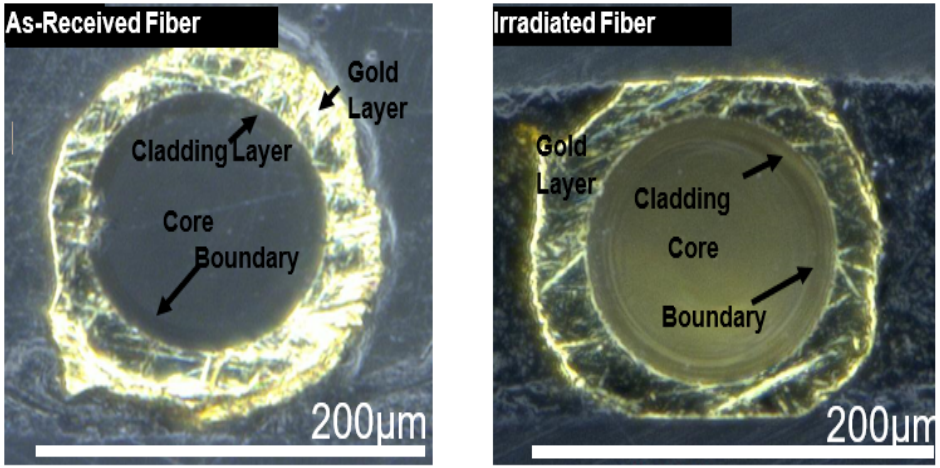



**Figure. Optical micrographs did not reveal any obvious changes in the structure or microstructure of the fiber after irradiation.**

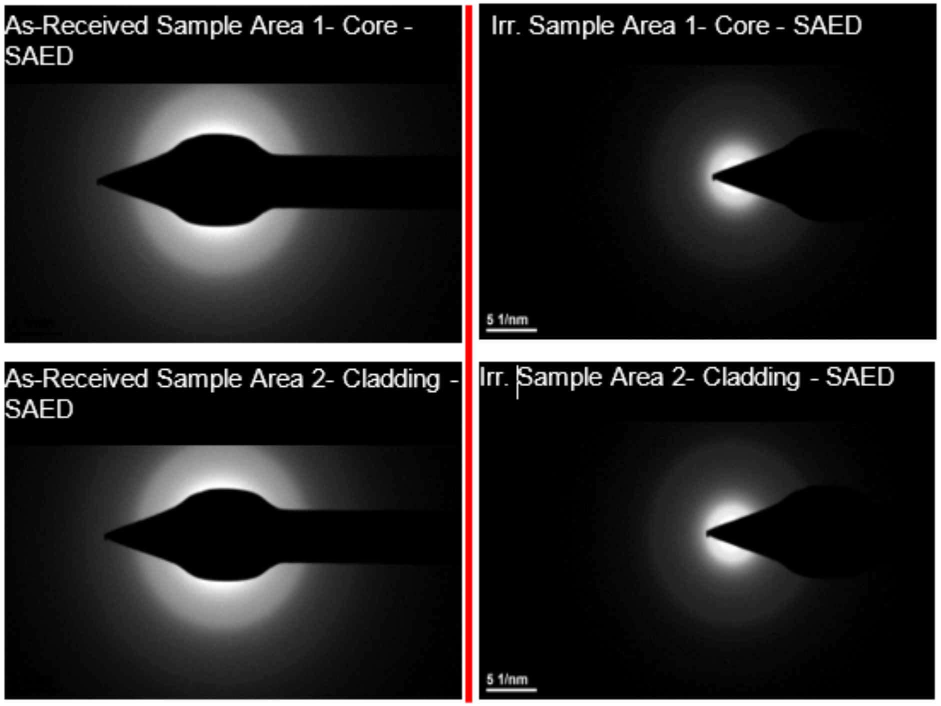



**Figure. Sample-Irradiated Characterization by SAED**

- Irradiation of a fiber was carried out in the Aerojet-General Nucleosis, AGN-201, nuclear reactor at Idaho State University (ISU).
- The reactor is critical at 3.2 Watts, has a maximum operating power of 5 Watts thermal, and an average and peak thermal fluxes of $1.5 \times 10^8$ and $2.5 \times 10^8$ n/(cm$^2$-s) at 5W.
- Only two of the isotopes produced, gold and silicon, are detectable with a High-Purity Germanium (HPGe) detector used for characterization at ISU.
- The low dose was not expected to produce any significant material damage in displacements per atom (dpa). The calculated dose for the fiber was $1.9353216 * 10^{-9}$ dpa.

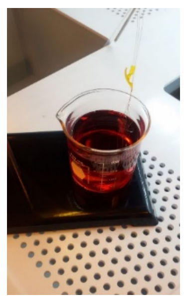
# Working Mechanism & Fabrication of the Fiber tip Sensor

## Working Mechanism

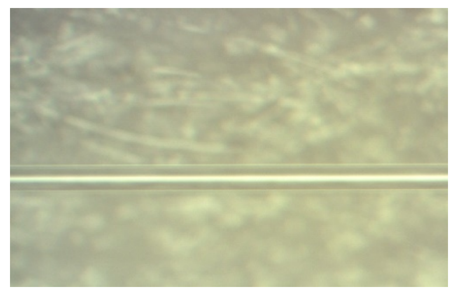
| Temperature < Crystallization temperature | Temperature > Crystallization temperature |
|---|---|
| ChG is transparent. | ChG crystallizes, refractive index and extinction coefficient increase. |
| Very low absorption loss in near infrared. | Absorption increases. |
| Low reflected power. | Reflected power changes. |

## Fabrication of the Fiber tip Sensor

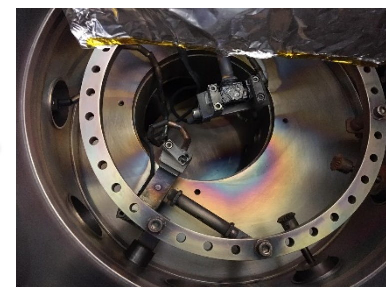
Gold coated fiber under microscope



Gold etching in Aqua Regia solution

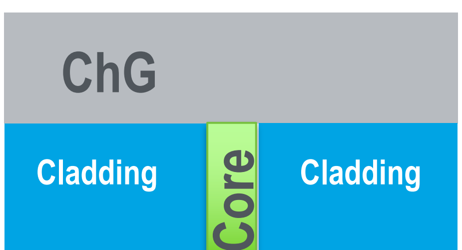


Gold etched fiber



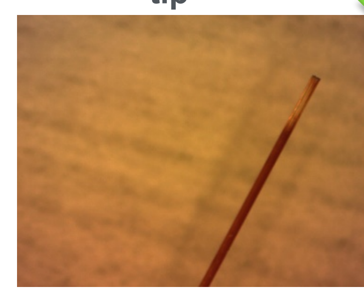
ChG deposition on fiber tip

Fiber with deposited chalcogenide glass on its tip




Schematic

Sample



# Characterization and Simulation of Fiber Tip Sensor

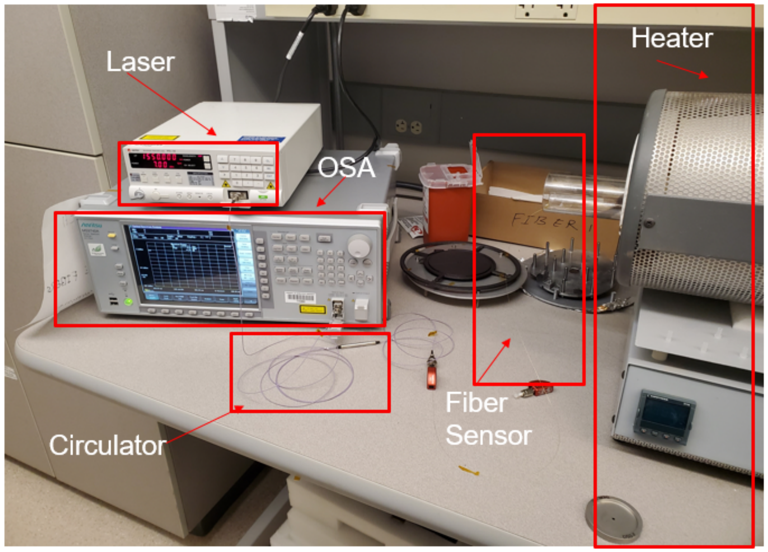



Figure. Fiber Sensor Testing Setup

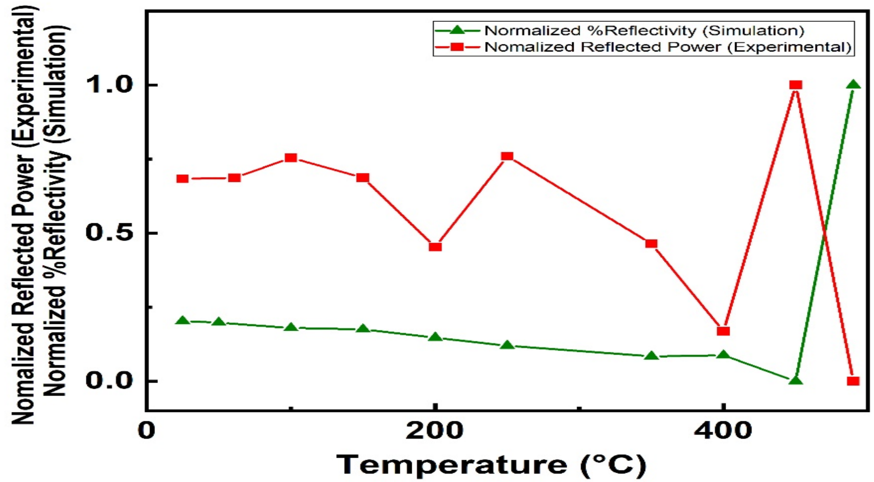



Figure. Normalized reflected optical power (from experiment) and % reflectivity (from simulation) as a function of temperature data. This is from a temperature sensor built on the tip of a radiation hard gold coated fiber ($Ge_{30}Se_{70}$).

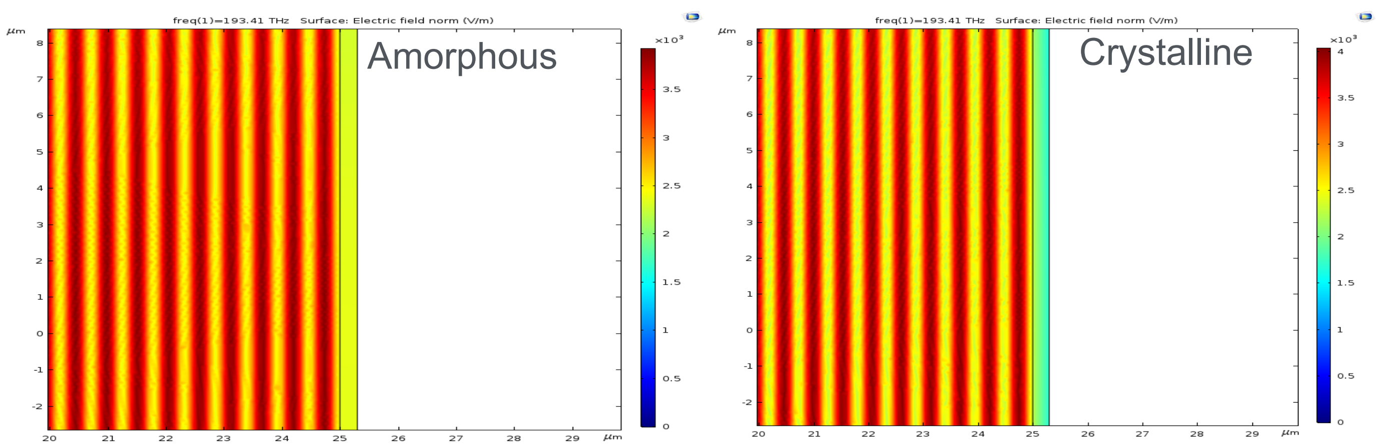



Figure. COMSOL Multiphysics simulation of Fiber Based Device ($Ge_{30}Se_{70}$).

# Step-Resistive Temperature Detector

**Working Mechanism**
Amorphous to crystalline phase transition decreases materials resistivity.

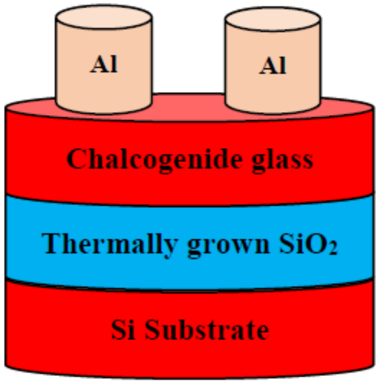



**Figure. Schematic of Device Structure**

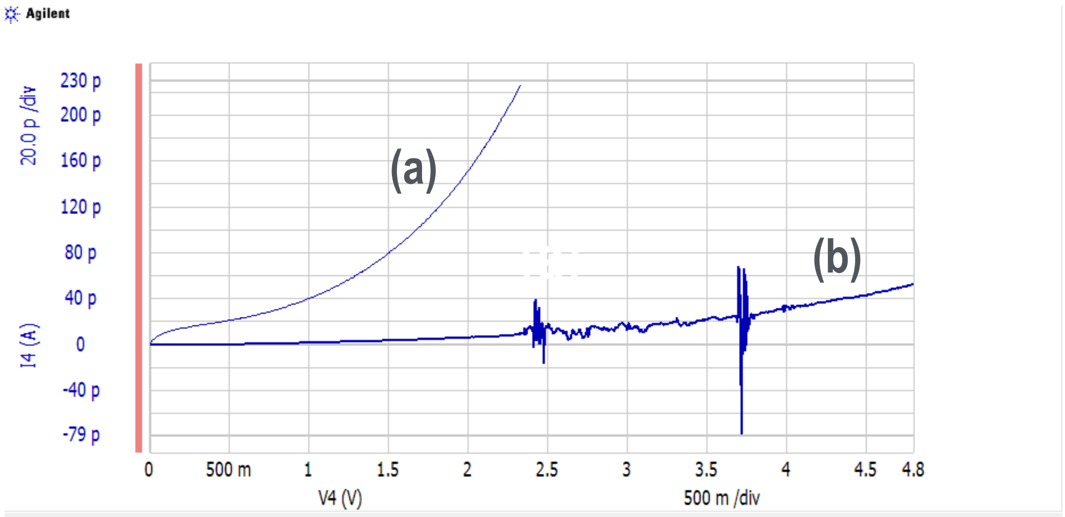



**Figure. I-V characteristics in crystalline and amorphous (bold) phase.**

- Initially high resistive material has become conductive due to its crystallization by increasing the device temperature to T cryst. (a)
- The high resistive state was regained by using electrical pulse at room temperature, causing materials' amorphization (b) .

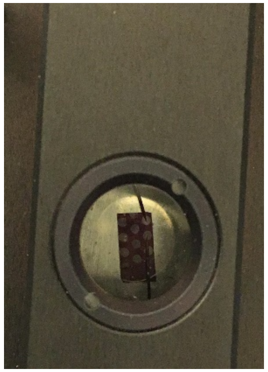
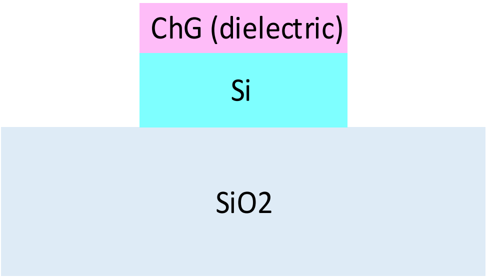


**Figure. Device Under Testing**

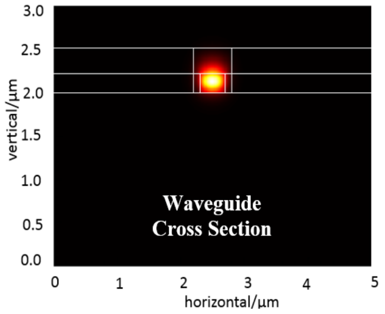
# Working Mechanism of the Hybrid Plasmonic Waveguide Sensor

## 1- Temperature of the ambient is below the glass crystallization temperature of ChG

- Material acts like a dielectric.
- Very low absorption loss in near infrared.
- Fundamental mode is confined in the silicon waveguide.
- Mode propagates along the waveguide with nominal loss of ~2.5-3.0 dB/cm.

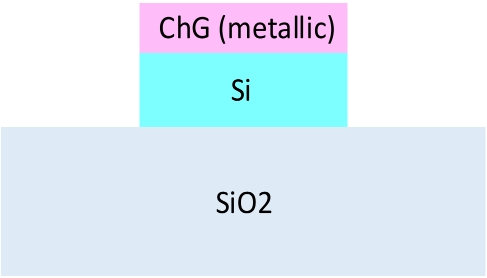



Intensity profile of TM mode

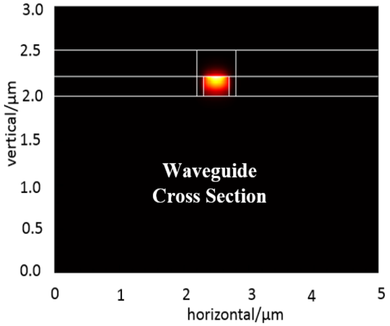



## 2- Temperature of the ambient is above the crystallization transition temperature of ChG

- Material crystallizes and exhibits conductive characteristics.
- Fundamental mode disappears.
- Tightly confined plasmon modes appear at the interface between silicon and the conductive material.
- The plasmonic modes provide confinement beyond the diffraction limit.
- Characterized by a very large propagation loss (~1-10dB/μm).

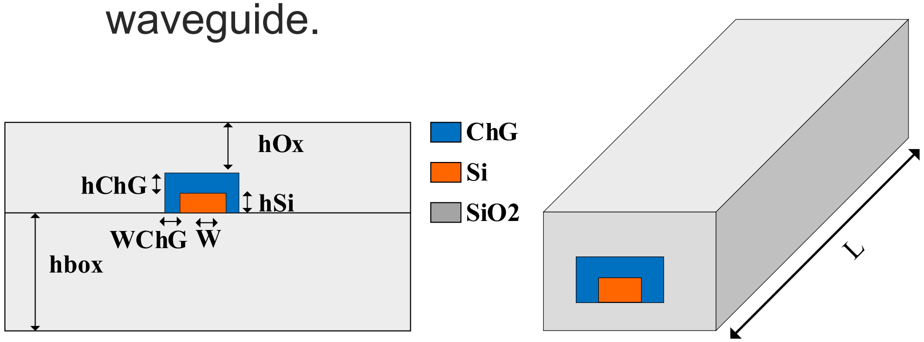


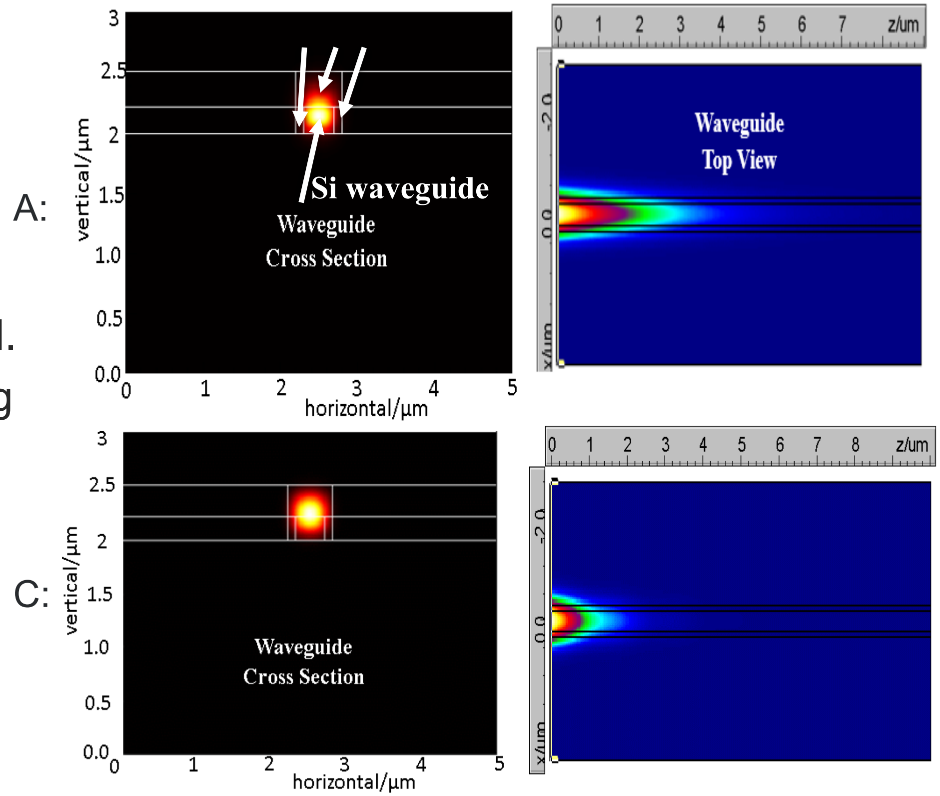

Intensity profile of TM mode



# Si:ChG Hybrid Plasmonic Waveguide (HPW) Temperature Sensor Design

- The team used well-characterized complex refractive indices of $Ge_{40}S_{60}$ chalcogenide glass.
- The complex refractive index is measured using ellipsometry.
- Simulations performed using FIMMWAVE Software, which uses Finite Element Method.
- High extinction ratio of 70 dB in a 22 µm long waveguide.




Complex Refractive index

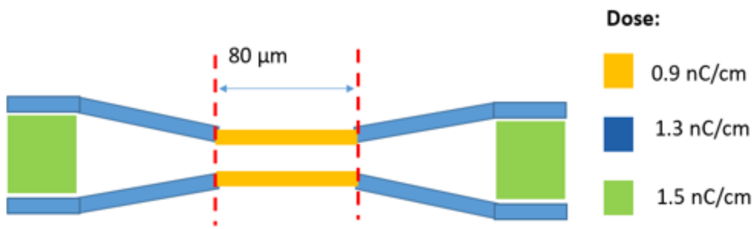
| Refractive index | Refractive index (n) | Extinction coefficient (k) |
|---|---|---|
| $SiO_2$ | 1.45 | - |
| Si | 3.47 | - |
| ChG (amorphous) | 2.45 | 0.10 |
| ChG (crystalline) | 3.39 | 0.17 |

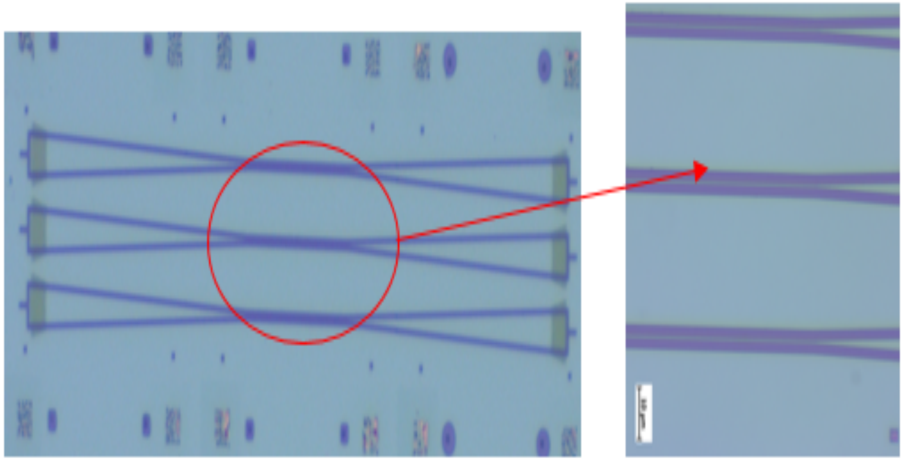
Optimum design parameters

| Parameters | Value (µm) |
|---|---|
| W | 0.4 |
| WChG | 0.1 |
| hChG | 0.3 |
| hSi | 0.22 |
| hOx | 0.5 |
| hbox | 2 |
| L | 5 |

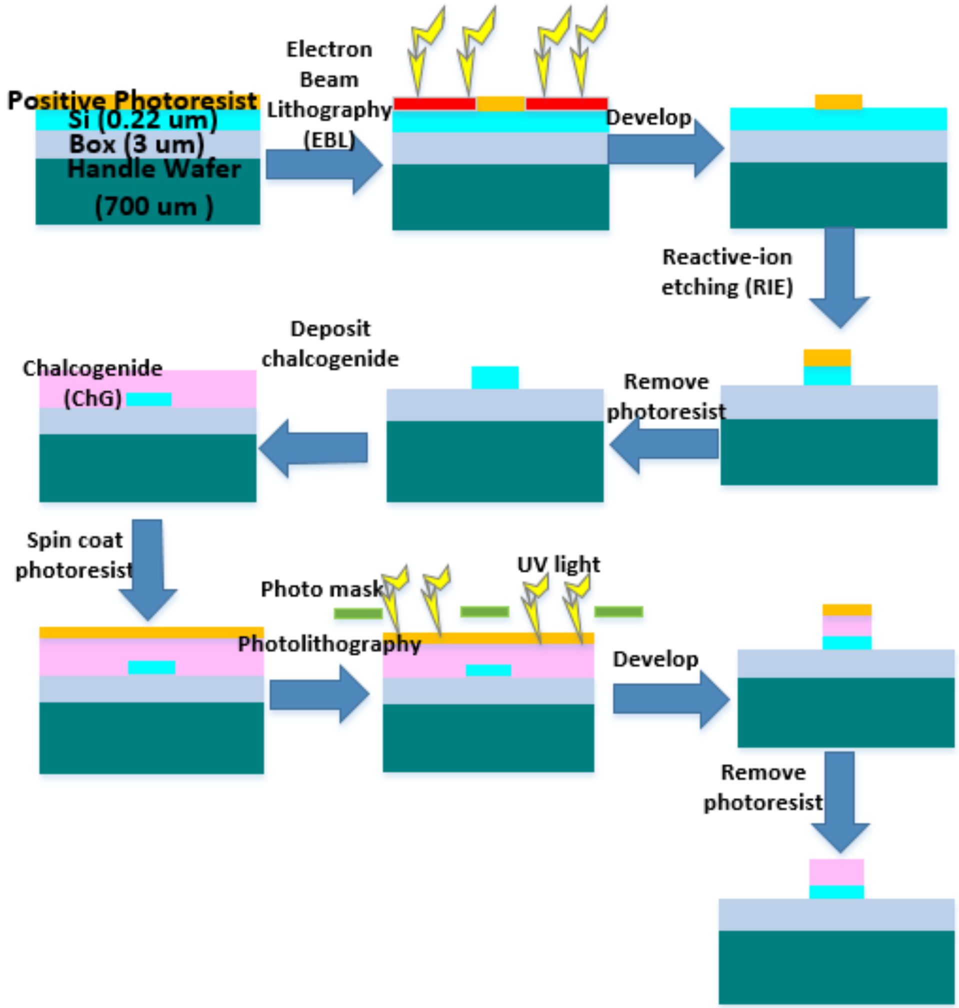
# Fabrication of Si:ChG Hybrid Plasmonic Waveguide (HPW) Temperature Sensor Design

- For the fabrication of silicon waveguides terminated with grating couplers, we used Electron-Beam Lithography (EBL).



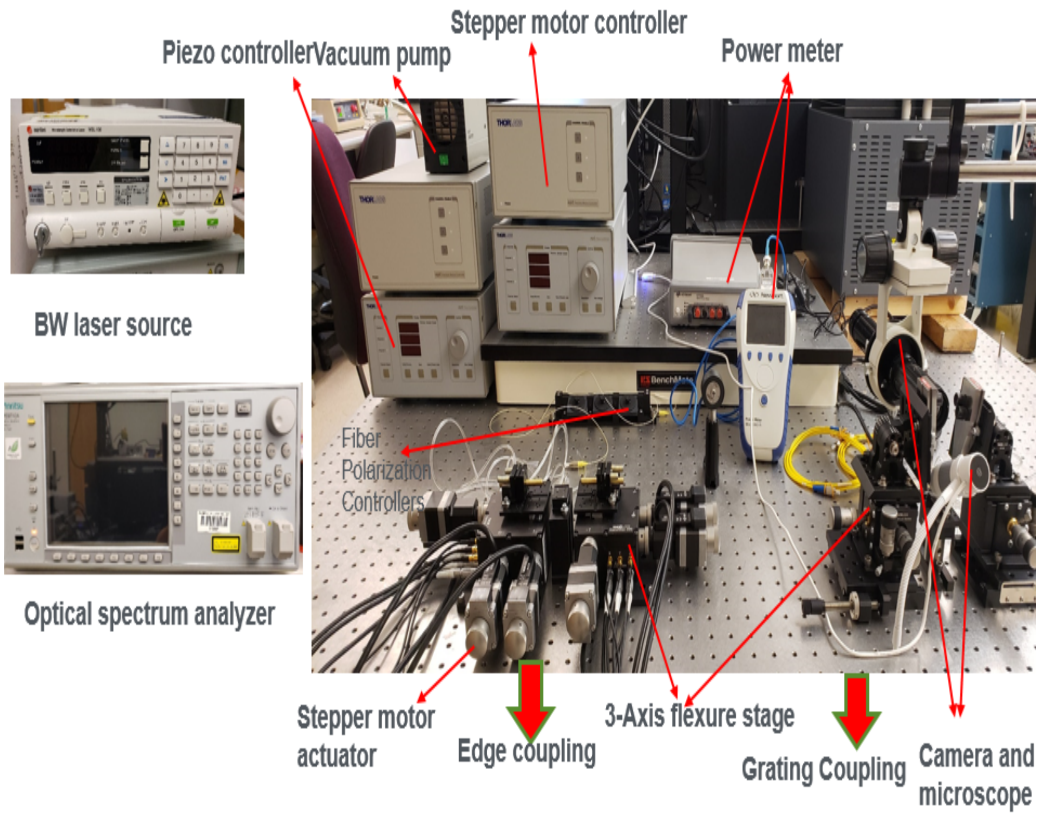
Fabricated device



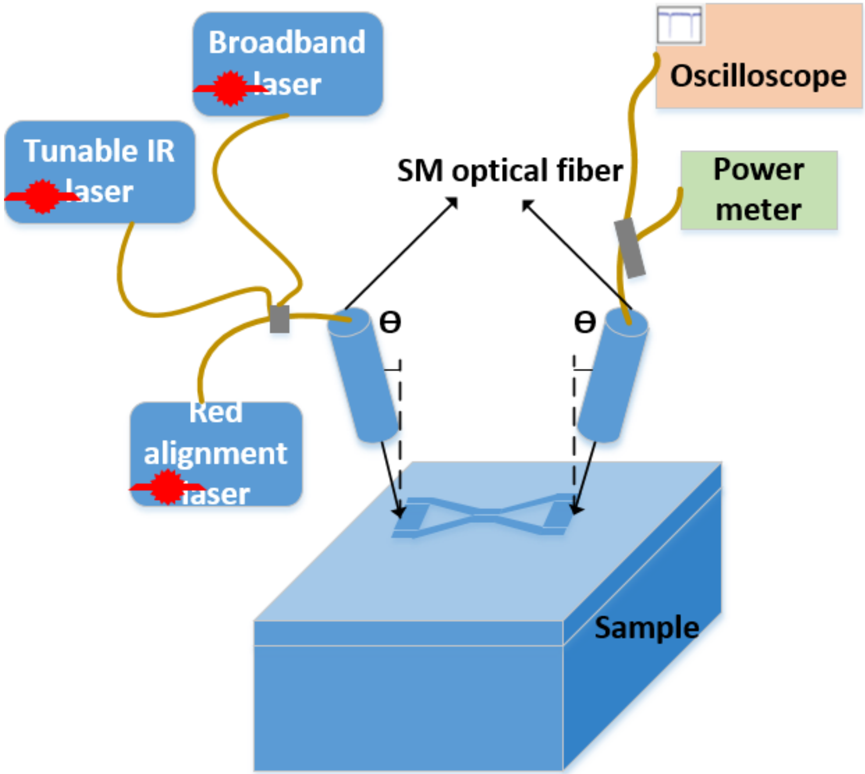

Process flow for fabricating Hybrid Silicon:ChG Waveguide based Plasmonic Temperature Sensors

# Testing of Si:ChG Hybrid Plasmonic Waveguide (HPW) Temperature Sensor Design

- The device is characterized with the grating coupler set up at Boise State University.



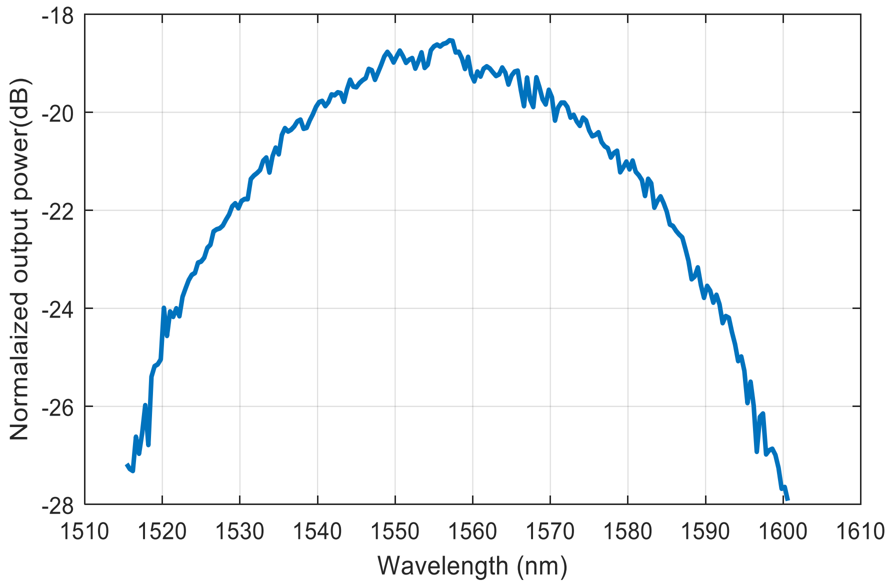

Waveguide characterization setup at Boise State University




Waveguide testing setup schematic

# Waveguide Fabrication Results

- Measured maximum transmission is -18.53 dB.
- Each grating coupler has ~8 dB loss by considering negligible loss in taper and waveguide loss.
- The 1 dB and 3 dB bandwidths are 24 nm and 51 nm, respectively
- Peak wavelength is shifted from 1550 nm wavelength to 1557 nm due to fabrication error during patterning with EBL.
- The fabrication yield is very low due to random spots at different locations (beam blanker leakage) and some devices shows very high loss.



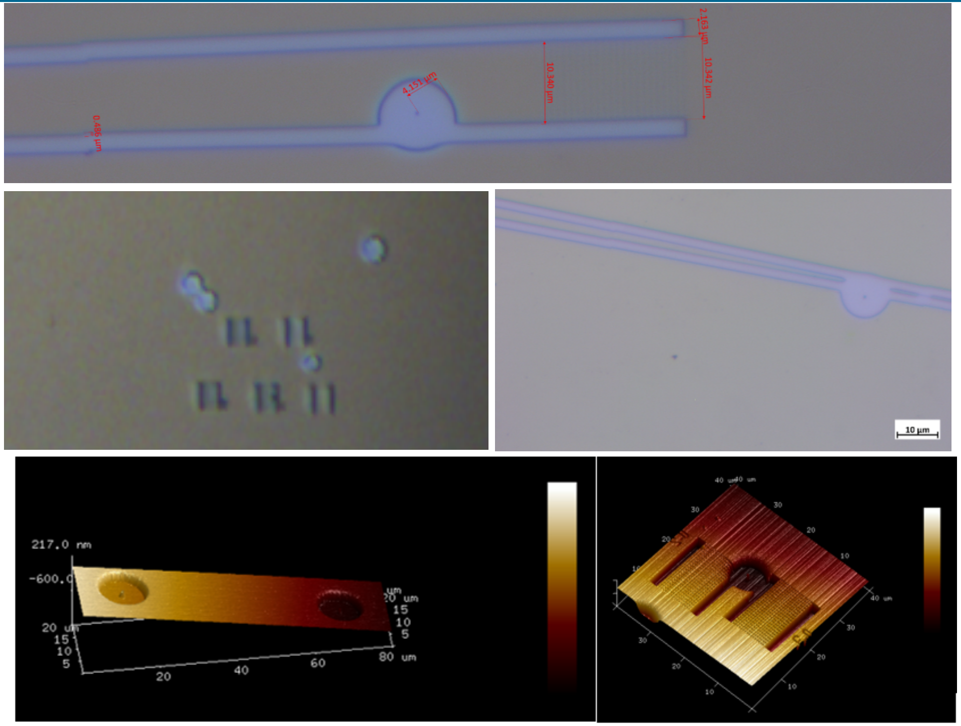

Normalized measured transmission spectrum

# Current problems with electron beam lithography

- Beam blanker leakage

\- Since our SEM did not have a fast blanker, the idle time during exposures caused extra exposure dots at the endpoints of every pattern.

\- During patterning, unwanted beam spots occurred on the pattern as shown in AFM image.

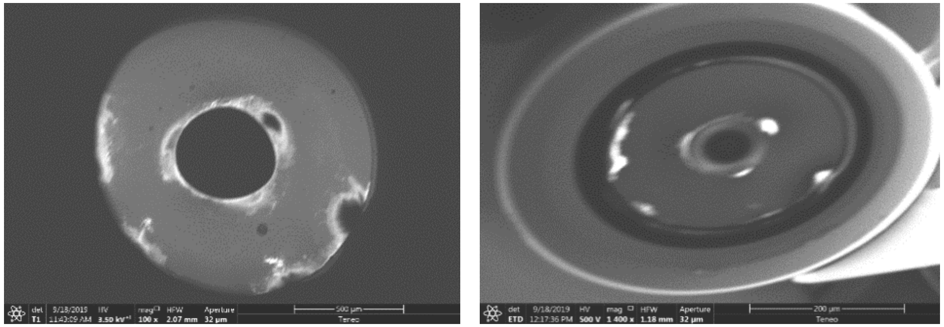


Beam blanker causes extra beam spots during idle time of the exposure.

- Astigmatism problem

\- Heavy contamination/corrosion of the T1 and T2 detectors in the pole piece in FEI TENEO.

\- Contaminations will shift the beam and affect the fields in pole pieces.



Heavy contamination in T1 and T2 detectors

# Conclusion

*With the conducted research throughout the second year of the project accomplishment we met the planned milestones in: (i) obtaining radiation hard gold coated fiber; (ii) characterizing the fiber and its radiation stability; (iii) finishing the design of waveguide sensor architecture and simulation; (iv) starting fabrication of silicon-chalcogenide hybrid integrated plasmonic temperature sensor; (v) starting fabricating and testing chalcogenide coated rad hard fiber tip based temperature sensor; (vi) conducting experiments in the co-PI,s laboratories*

*These accomplishments trace the avenue to formation of a very low power consuming reversible and multiple time applicable devices, which can be deposited directly over the measured surface for real time temperature monitoring in nuclear facilities. This is a new advanced cost effective sensor solution which will make the nuclear systems more reliable and effective*

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