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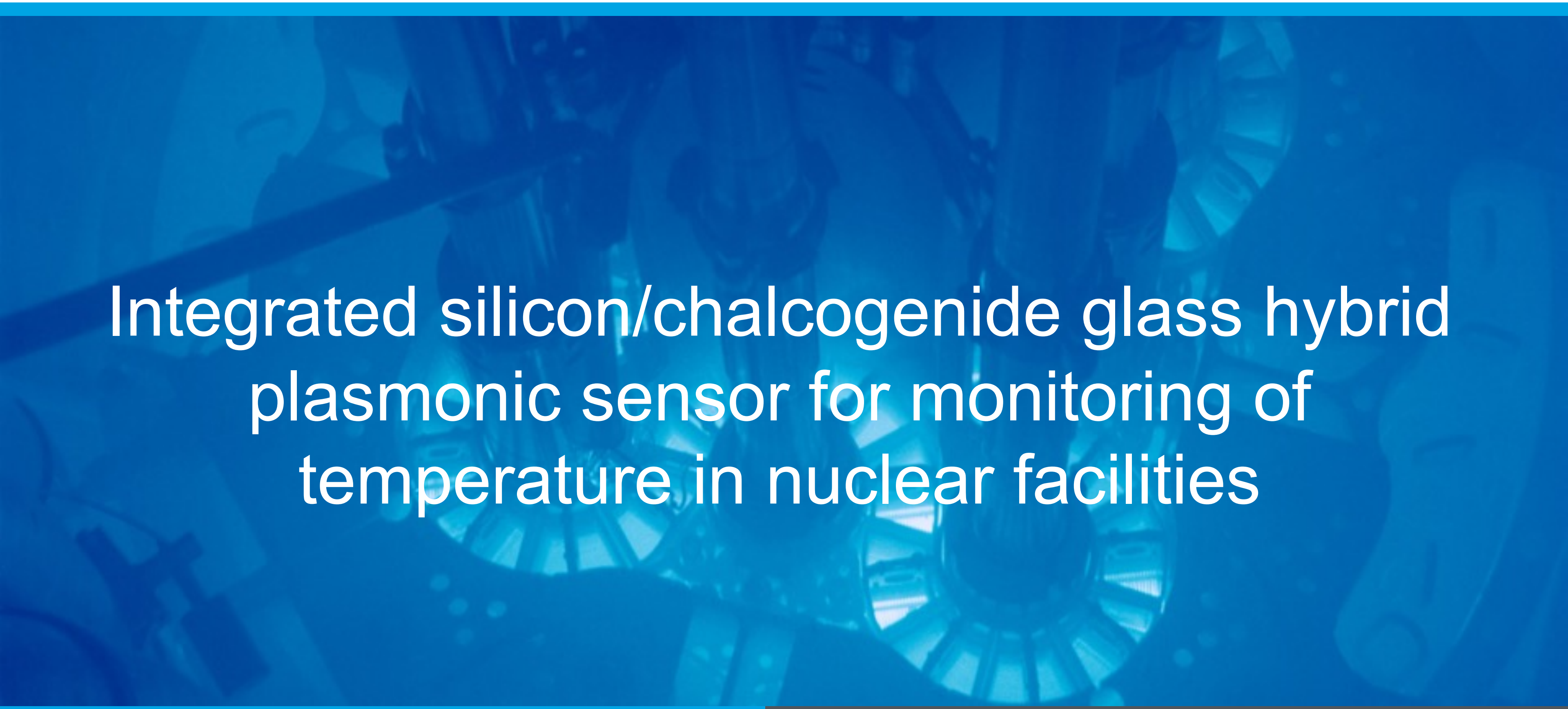

Integrated silicon/chalcogenide glass hybrid plasmonic sensor for monitoring of temperature in nuclear facilities

Advanced Sensors and Instrumentation Annual Webinar

October 31 – November 1, 2018

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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 (2018)**
  - Dr. Maria Mitkova – Principal Investigator
  - Dr. Harish Subbaraman – Co- PI
  - Mr. Al-Amin Ahmed Simon (Graduate Student)
  - Ms. Bahareh Badamchi (Graduate Student)
- **Schedule**

- Develop a chalcogenide glass material suite with variety crystallization temperatures

- Characterization of the synthesized glasses with Differential Scanning Calorimetry (DSC) and X-ray diffraction (XRD)

- Development of ink based on the synthesized chalcogenide glasses

- Design sensor architecture simulation using known chalcogenide glasses data

- Fabricate thin films of the chalcogenide glasses and obtain optical properties data at different temperatures

-Design sensor architecture simulation using obtained data for sinthesizedchalcogenide glasses

Accomplishments

✓ A suite of chalcogenide glasses (ChG) was synthesized, including systems with more than one crystallization temperatures

✓ Completed the design of integrated silicon photonic chip and silica optical fiber based hybrid plasmonic sensors for sensing temperature in a nuclear facility (Major Milestone met).

✓ Characterization of the thermal characteristics of the synthesized ChG by DSC and XRD. (level 2 milestone met)

✓ Optimization of ChG hybrid architecture, four design structures simulated. The effect of misalignment of ChG on the waveguide was studied; minimal effect on sensors performance.

✓ Designing grating couplers, power dividers, inverse taper couplers to account for packaging the sensor array.

✓ Dissolution of the synthesized ChG in amine solutions for ink formation and characterization of the ink by its viscosity and surface tension

✓ Milling of the synthesized ChG and mixing with solvents for ink formation; characterization of the ink by its viscosity and surface tension (level 3 milestone met).

✓ Spinning of the inks on Si wafers and study of their optical properties at different temperatures

✓ Optimization of the design parameters of the integrated silicon photonic and optical fiber temperature sensors based on the measured optical properties.

✓ Printed films based on powdered substances

✓ Obtained gold coated rad hard fibers and started setting up experiments for temperature sensing

✓ Simulated and experimented by coating the end facet of an optical fiber with ChG the changes in reflected intensity detectable at the phase transition temperature (crystallization of ChG)

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.

Two different device architectures.

Fused silica Optical Fiber + **Chalcogenide Glass (ChG)** = **Fiber Tip based Temperature Sensor**

Features: Elegant design, simple fabrication process, reversible Temperature sensor

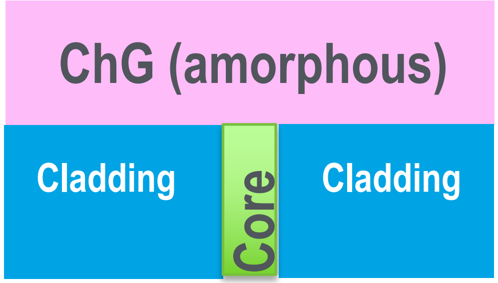
Nanophotonic Silicon Photonic Waveguide (SiPW) + **Chalcogenide Glass (ChG)** = **Hybrid Plasmonic Waveguide (HPW)**

Features: Real-time, reusable reversible Temperature sensor

Working Mechanism of the Fiber tip Sensor

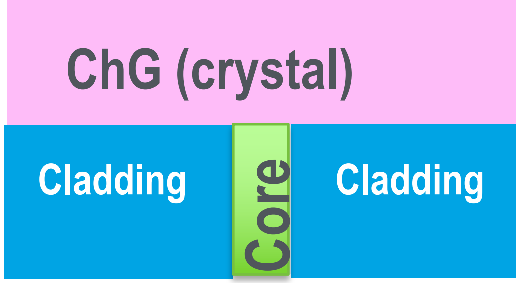
1- Temperature of the ambient is below the glass crystallization temperature of ChG

- ChG is transparent.
- Very low absorption loss in near infrared.
- Low reflected power.



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

- ChG crystallizes, refractive index and extinction coefficient increase.
- Absorption increases.
- Reflected power increases.



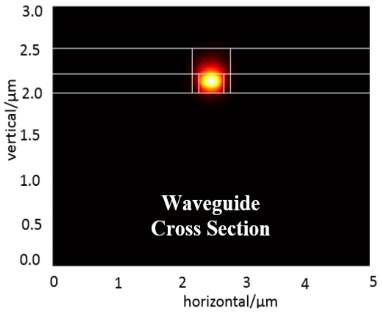
3.Application of a bundle of fibers topped with different compositions ChG and/or application of ChG with several crystallization temperatures opens the opportunity for a real time measurement of temperatures coinciding with the crystallization temperatures of the material

Working Mechanism of the Plasmonic Sensor

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

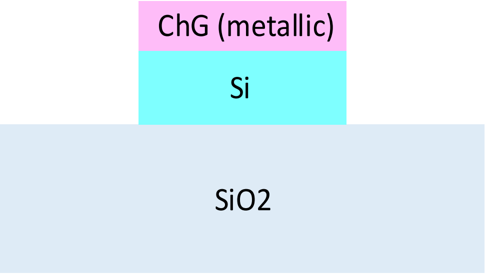
- Material acts like a dielectric.
- Very low absorption loss in near infrared.
- Fundamental mode is confined in the silicon waveguide.
- Mode propagates along the waveguide with nominal loss of ~2.5-3.0 dB/cm.

Intensity profile of TM mode

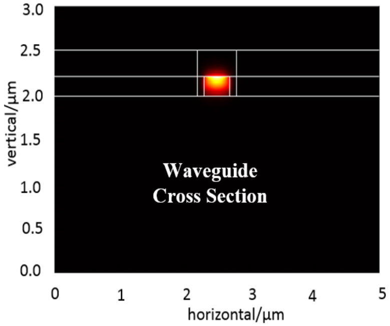


2- Temperature of the ambient is above the crystallization transition temperature of ChG

- 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



Designs and Simulations

- Different device structures have been investigated to provide an optimum design with the best performance.
- As a starting point, the team used well-characterized complex refractive indices of $Ge_2Sb_2Te_5$ (GST) ChG films from literature in the simulations [1].
- Simulations performed using FIMMWAVE Software, which uses Finite Element Method.

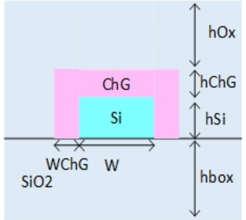
Complex Refractive index

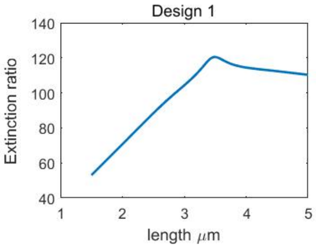
Material	Refractive index (n)	Extinction coefficient (k)
ChG (amorphous)	2.14	0.17
ChG (crystalline)	2.62	2.11
Si	3.47	0
SiO2	1.45	0

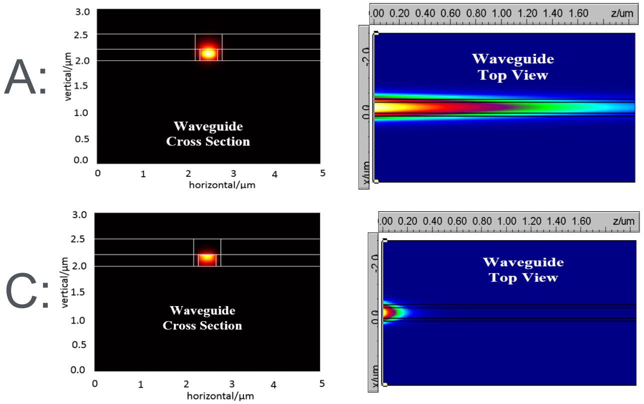
[1] Lee, Seung-Yeol. "Design of a Plasmonic Switch Using Ultrathin Chalcogenide Phase-change Material." Current Optics and Photonics 1, no. 3 (2017): 239-246.

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

Design # 1



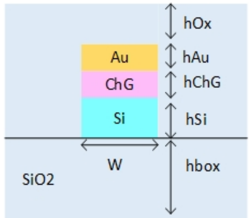


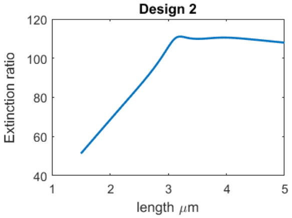


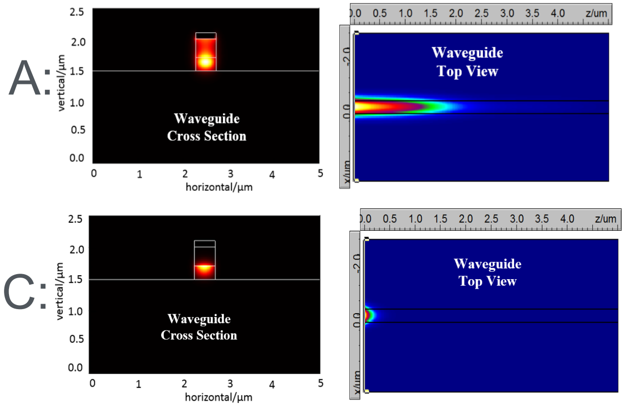
Optimum design parameters obtained for design 1.

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

Design # 2



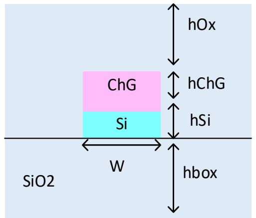


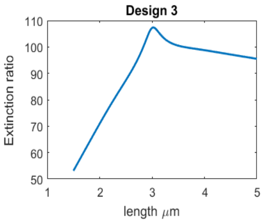


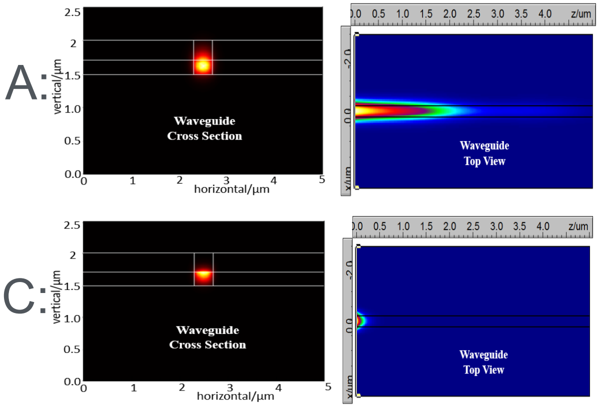
Optimum design parameters obtained for design 2.

Parameters	Value (µm)
W	0.45
hAu	0.1
his	0.22
hChG	0.3
hOx	0.4
hbox	1.5

Design # 3



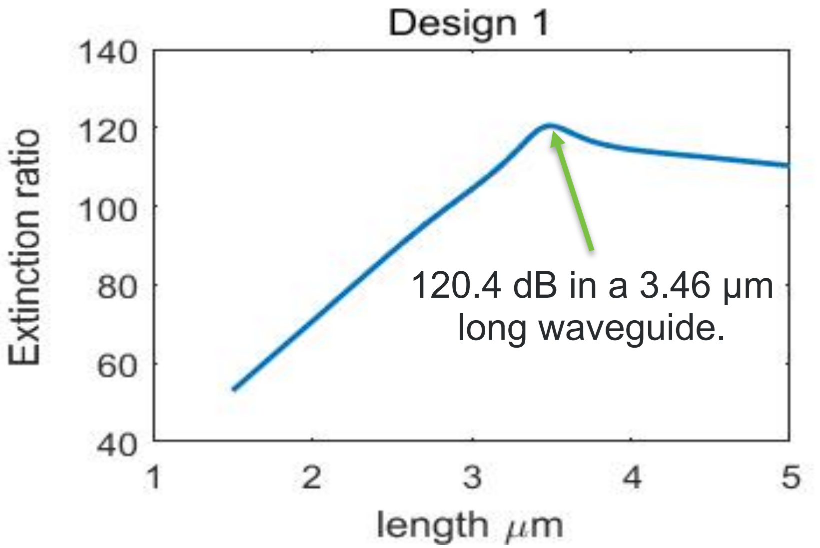




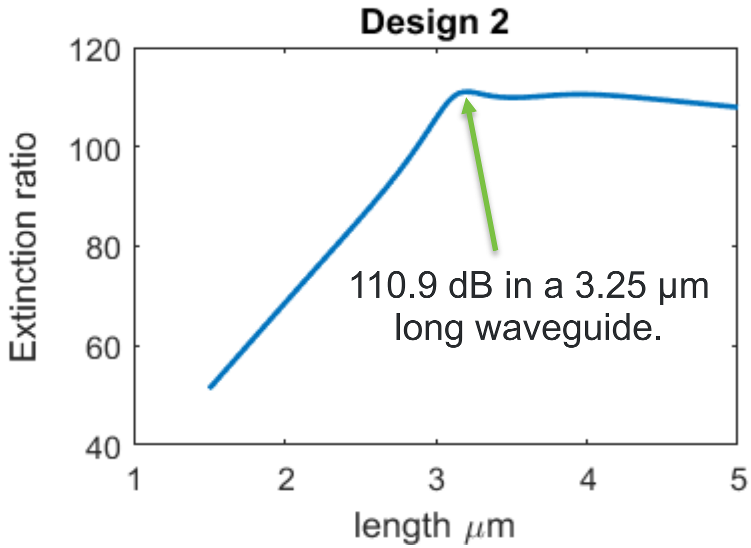
Optimum design parameters obtained for design 3.

Parameters	Value (µm)
W	0.4
hChG	0.3
hSi	0.22
hOx	0.5
hbox	1.5

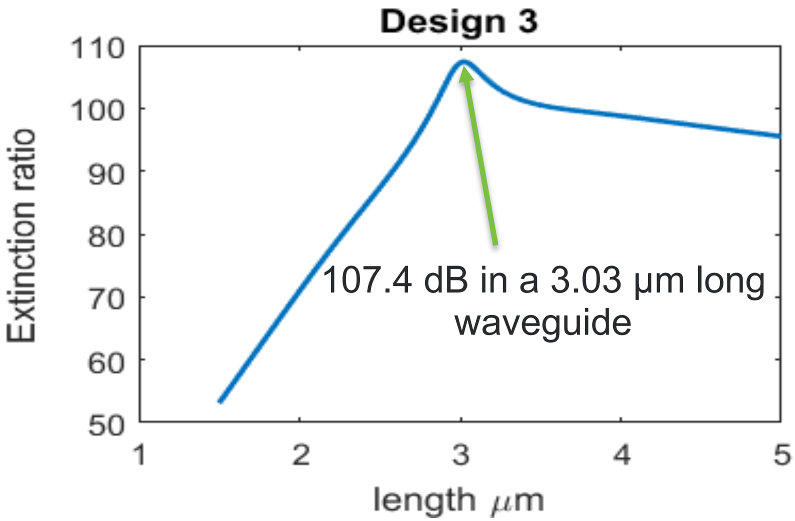
Extinction Ratio



120.4 dB in a 3.46 µm long waveguide.



110.9 dB in a 3.25 µm long waveguide.



107.4 dB in a 3.03 µm long waveguide

Effect of Misalignment on Sensor Performance

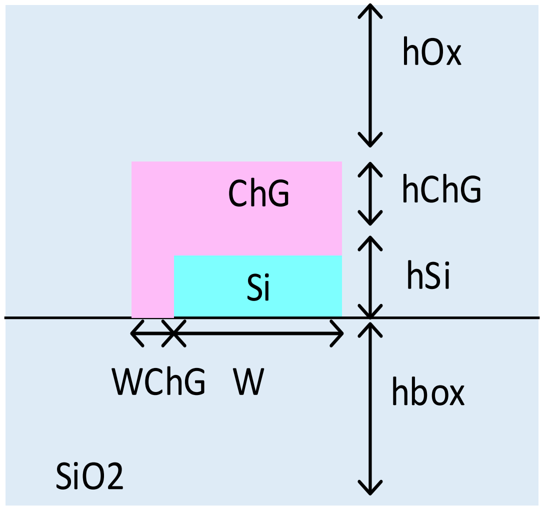
Case: ChG misaligned and covering surface and one side of silicon waveguide

- Example: ChG ink misalignment during printing of design 3.
- ChG ink covers top surface and one sidewall of Si waveguide.

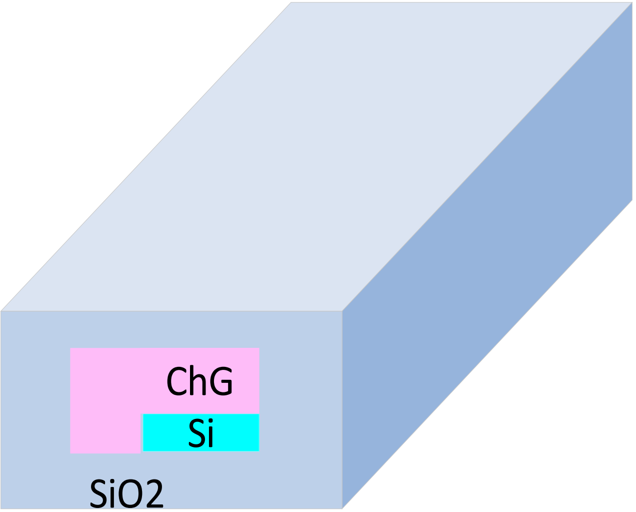
Optimum design parameters obtained for this case

Parameters	Value (µm)
W	0.5
WChG	0.3
hSi	0.22
hChG	0.3
hOx	1
hbox	2

Cross section



3D view of misaligned print

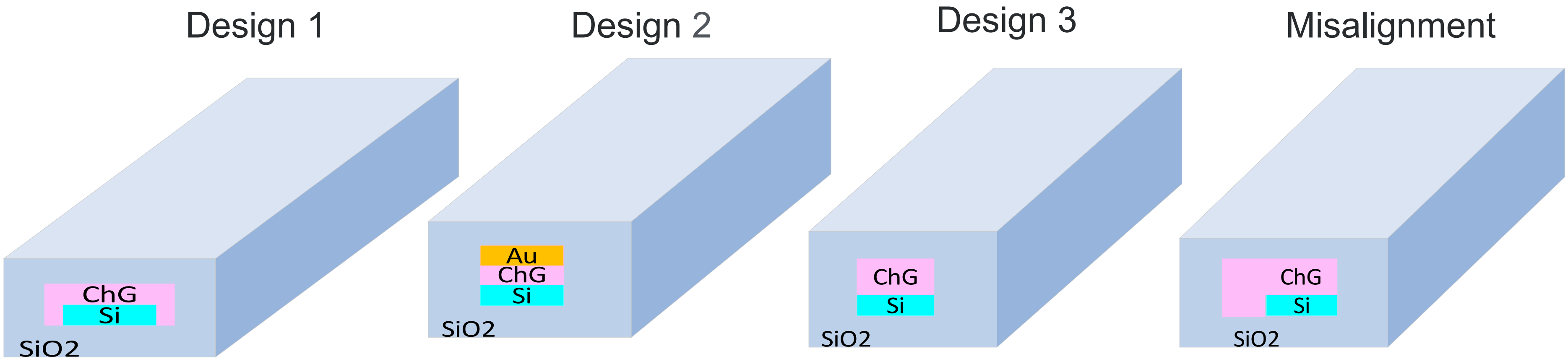


Extinction ratio: Compared to Design # 1 wherein the ChG fully covers the silicon waveguide, the misaligned case produces lower loss. An extinction ratio greater than 70dB is still available for successful device operation.

HPW Design Summary

Comparison of all waveguide design losses in fundamental TE and TM mode in amorphous and crystalline phases of ChG.

	Amorphous		Crystalline	
Design #	Loss in TE mode (dB/μm)	Loss in TM mode (dB/μm)	Loss in TE mode (dB/μm)	Loss in TM mode (dB/μm)
Design 1	-6.62	-13.4	-68.7	-134
Design 2	-2.45	-15.1	-18.8	-126
Design 3	-2.54	-10.6	-18.9	-118.8
Misalignment	-3.48	-10.2	-32.3	-81.4



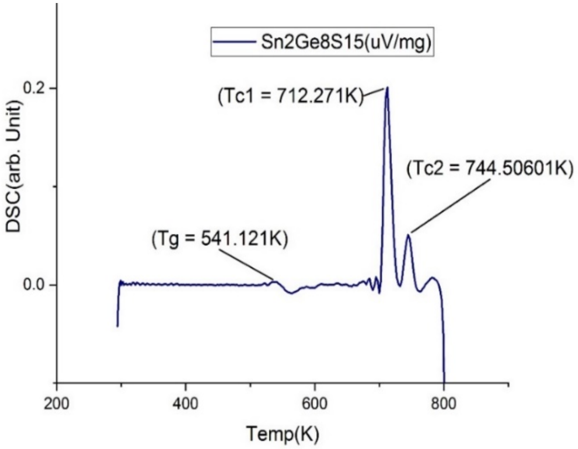
Device Fabrication: Chalcogenide Glass Synthesis

- A suite of Ge_xSe_{100-x}, Ge_xS_{100-x}, (x=20, 30, 33, 40), $Sn_2Ge_8S_{15}$ bulk glasses were synthesized by standard melt quenching technique to ensure availability of materials with variable thermal properties for devices production.
- The glass synthesis was carried out in a programmable tube furnace which was programmed (at different rates) to reach 1023K with holdings at the characteristic temperatures accordingly to the particular phase diagrams. The synthesis was carried out for 168 hours to achieve homogeneous glasses.

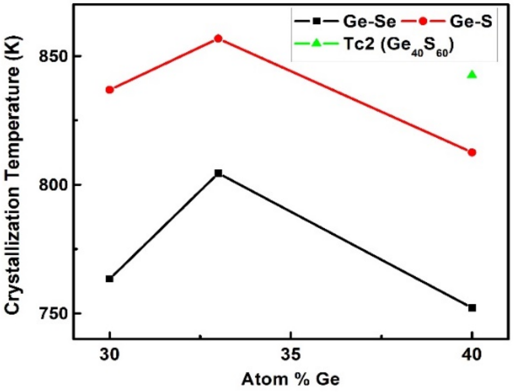
- Differential Scanning Calorimetry was done to obtain crystallization temperature of the bulk glasses.
- Ternary ChGs and $Ge_{40}S_{60}$ showed multiple crystallization which could be crucial as it demonstrates ability for measurement of various temperatures with one material.

Thermal Characterization of Chalcogenide Glasses

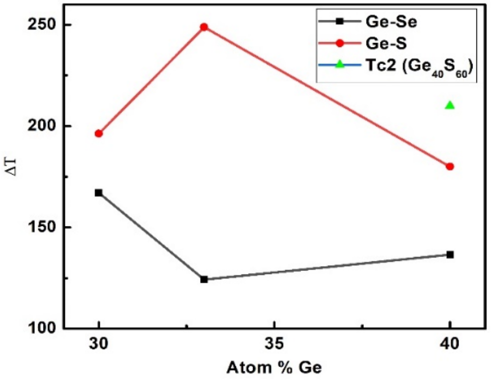
Crystallization Temperature and Thermal Stability



DSC curve illustrating 3 crystallization peaks for $Sn_2Ge_8S_{15}$



Crystallization Temperature vs atom % Ge in Ge-S and Ge-Se systems.



ΔT (= T_c-T_g) vs atom % Ge in Ge-S and Ge-Se systems.

- ➢ 6 compositions show 7 distinct crystallization temperatures ranging from 750K to 860K.
- ➢ High value of ΔT (= Tc-Tg) suggests that the glasses are thermally stable.

Chalcogenide Glass Ink Formation & Characterization

Dissolution based ink

1.Chalcogenide Glass Synthesis

2.Crush the ChGs into powder using mortar and pestle.

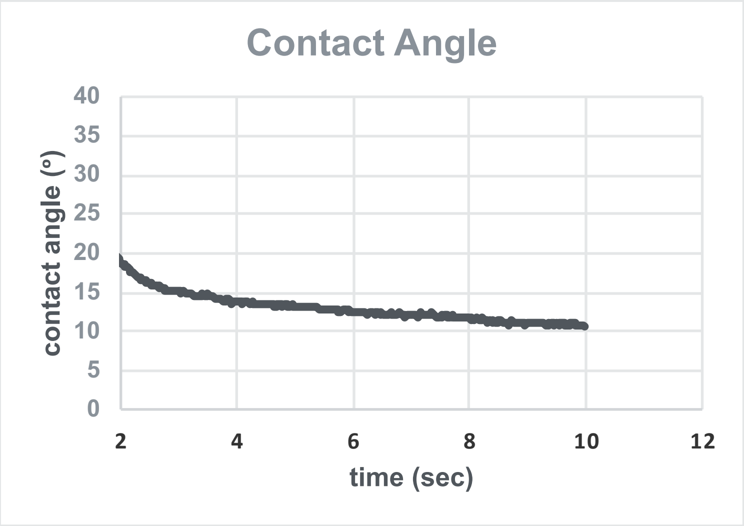
3.Mix the powder with solvent at 40°C with magnetic stirrer.

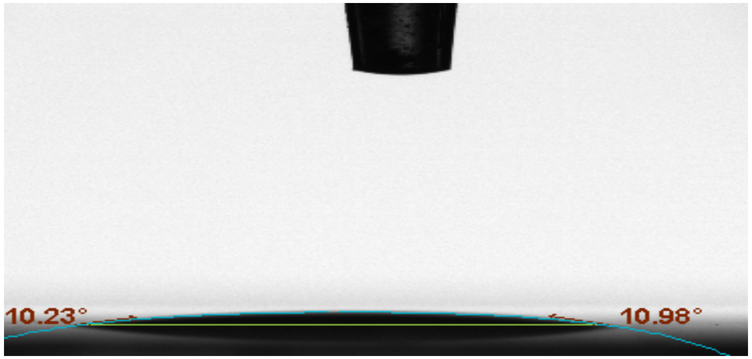
4. Ultra sonicate the solution

5.Centrifugation of the solution.

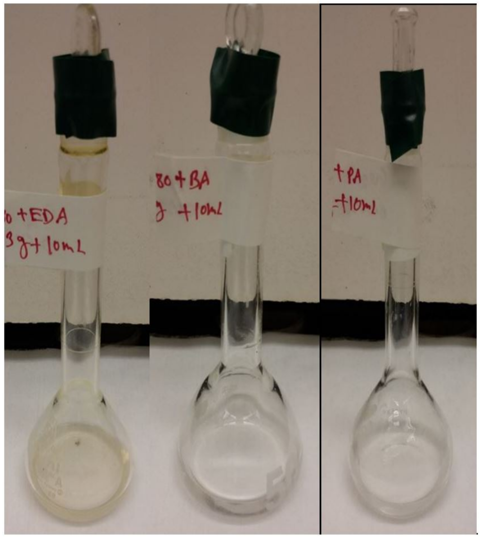
6.Use glass syringe to take out half the solution from the top and then filter the solution.

Ink is ready for characterization!





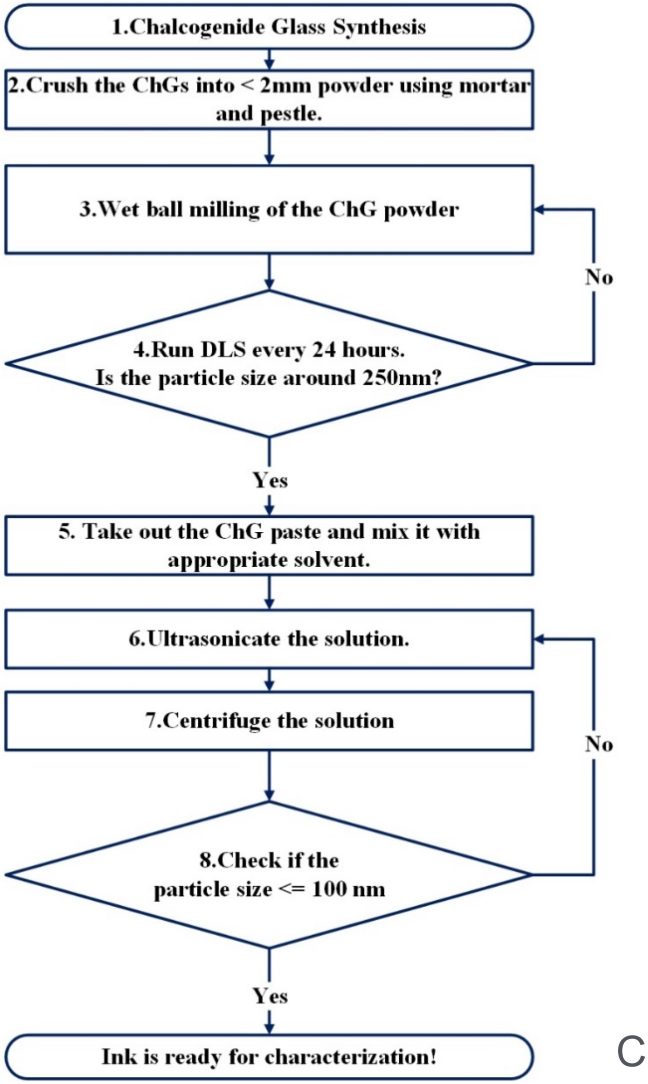
Contact angle of $Ge_{33}S_{67}$ dissolved in basic solvent on Si substrate.



ChG solutions

Chalcogenide Glass Ink Formation & Characterization

Nanoparticle ink



1.Chalcogenide Glass Synthesis

2.Crush the ChGs into < 2mm powder using mortar and pestle.

3.Wet ball milling of the ChG powder

4.Run DLS every 24 hours. Is the particle size around 250nm?

No

Yes

5. Take out the ChG paste and mix it with appropriate solvent.

6.Ultrasonicate the solution.

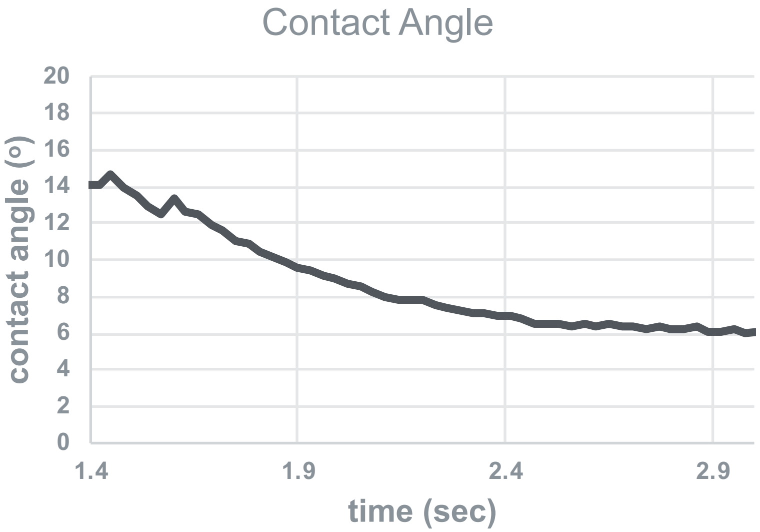
7.Centrifuge the solution

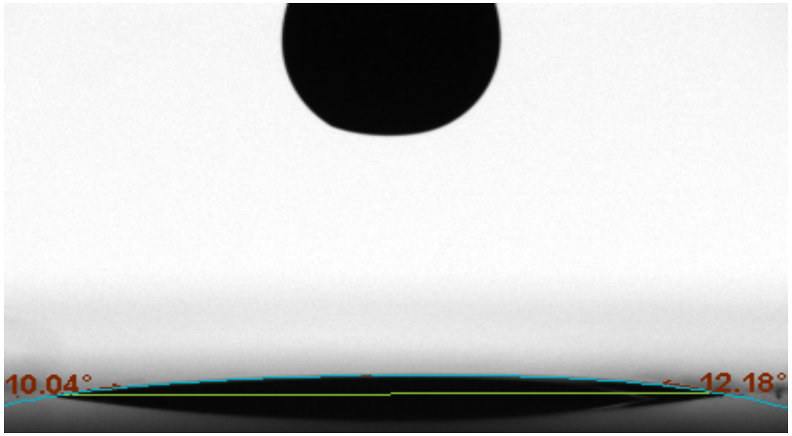
8.Check if the particle size <= 100 nm

No

Yes

Ink is ready for characterization!



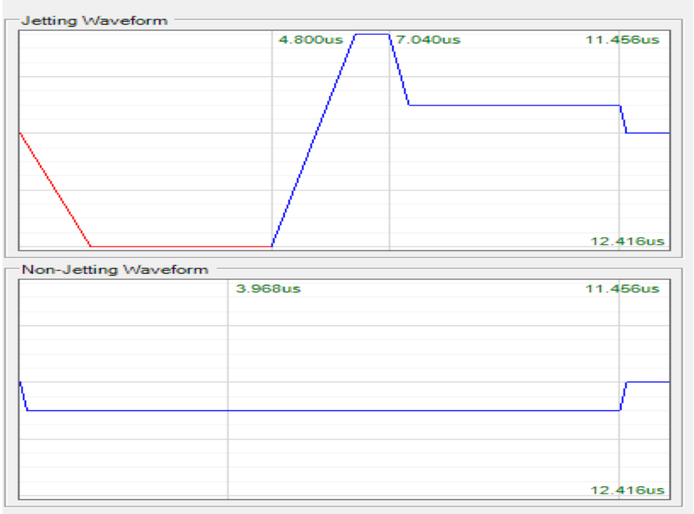


Contact angle of $Ge_{20}Se_{80}$ nanoparticle ink on a Si substrate

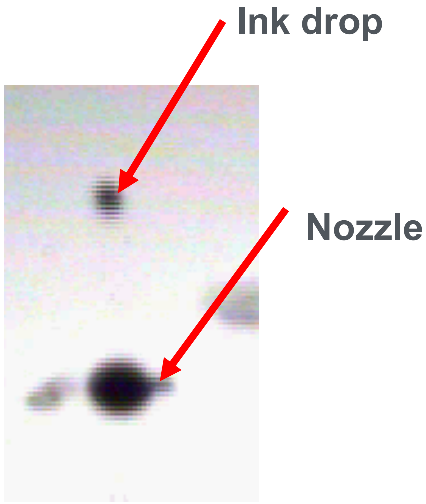
$Ge_{20}Se_{80}$ nanoparticle ink

Inkjet Printing Using Nanoparticle Ink

- Printing was done in a Dimatix inkjet printer. It uses a piezoelectric nozzle to disperse liquid.
- Optimization of the waveform is essential to have uniform thin film.
- Optimization was done for the produced $Ge_{20}Se_{80}$ nanoparticle ink.
- Individual ink drop, uniform velocity of the droplets, clogging during printing are some of the criteria that need to be checked during waveform optimization.

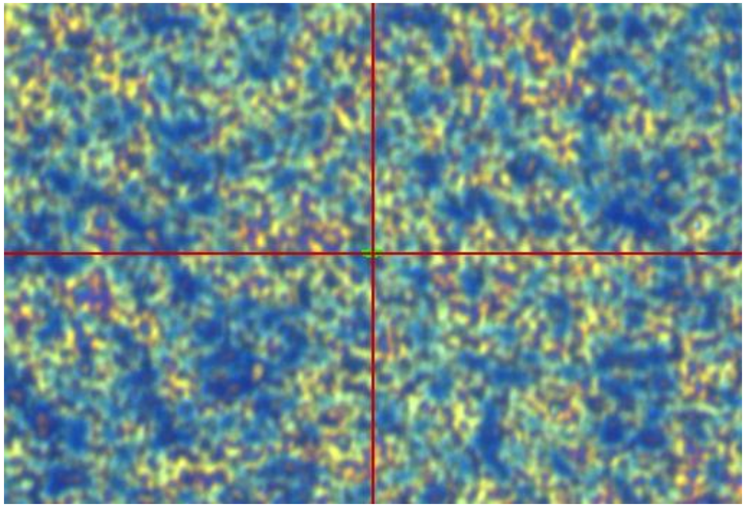


Waveform for jetting.

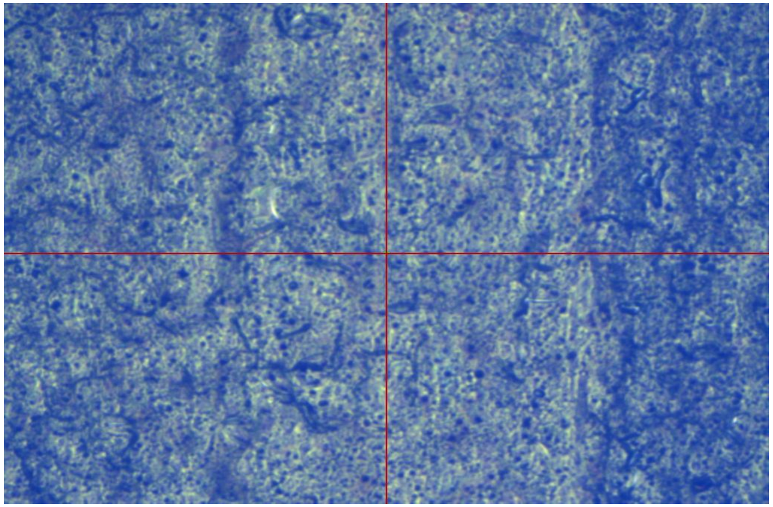


Individual ink drop from nozzle.

Printed and Spin Coated Thin Film



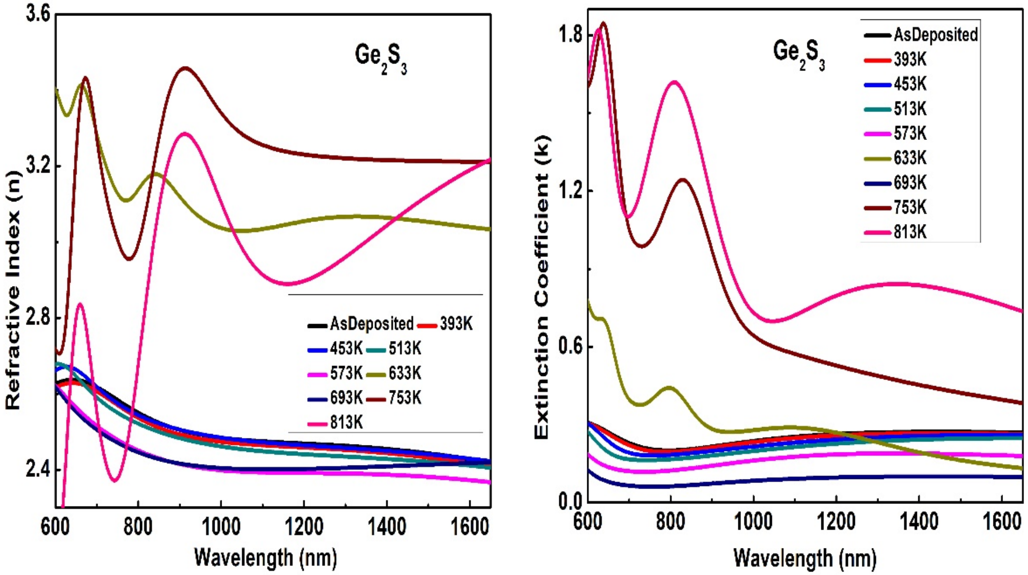
Uncured spin coated thin film of $Ge_{20}Se_{80}$ (dissolution based ink)



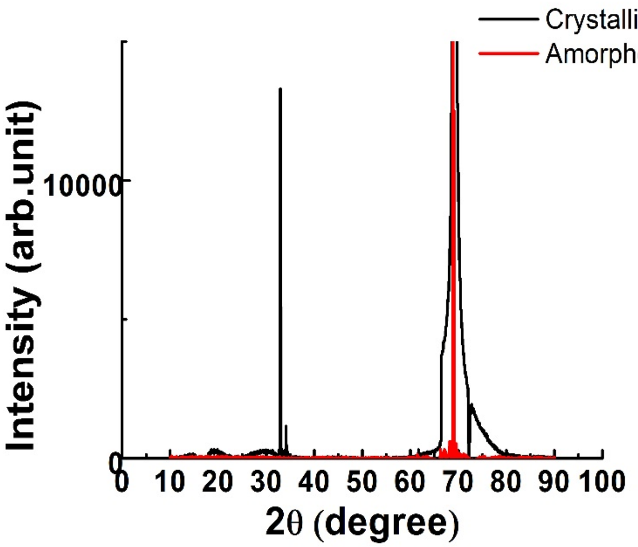
Uncured printed thin film of $Ge_{20}Se_{80}$ (Nanoparticle ink)

- We are optimizing the curing/sintering process.
- After sintering, ellipsometry will be done to obtain refractive index and extinction coefficient.
- As a proof-of-concept, crystallization in thermally evaporated thin films is studied.
- It shows that crystallization induces huge difference in optical properties.

Crystallization in Evaporated Thin Film ($Ge_{40}S_{60}$)



Refractive index (*n*) and Extinction coefficient (*k*) of $Ge_{40}S_{60}$



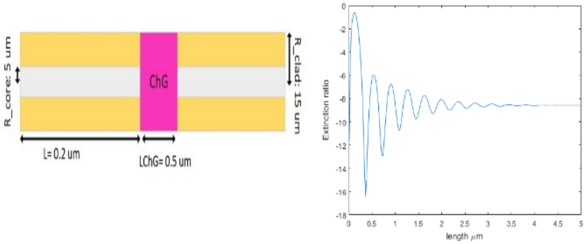
XRD of $Ge_{40}S_{60}$ thin films

- XRD shows that $Ge_{40}S_{60}$ thin films form crystals if heated at 753K.
- Ellipsometry shows that thin films which are heated up to 753K, have considerably higher refractive index and extinction coefficient.
- These ellipsometric data are successfully used in optical fiber-tip coated device simulation.

ChG coated Optical Fiber Tip Based Temperature Sensor

SMF 28 with literature data:
- Core refractive index: 1.4494
- Cladding refractive index: 1.4444

ChG (amorphous)	2.14	0.17
ChG (crystalline)	2.62	2.11



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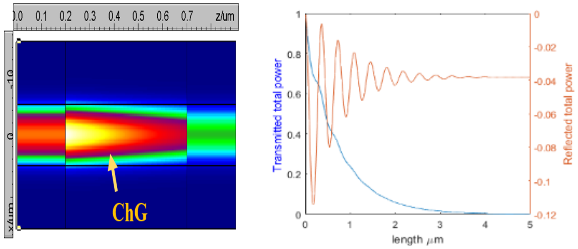

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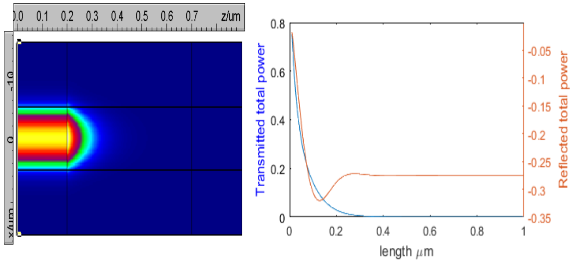
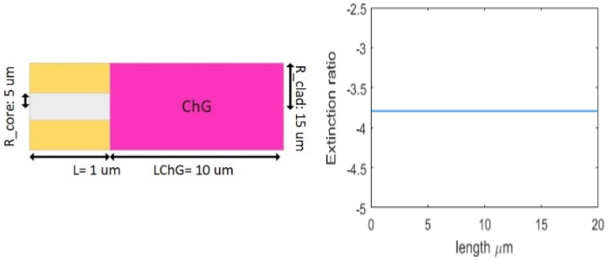


SMF 28 with measured data:
- Core refractive index: 1.4494
- Cladding refractive index: 1.4444

ChG (amorphous)	2.45	0.10
ChG (crystalline)	3.39	0.17



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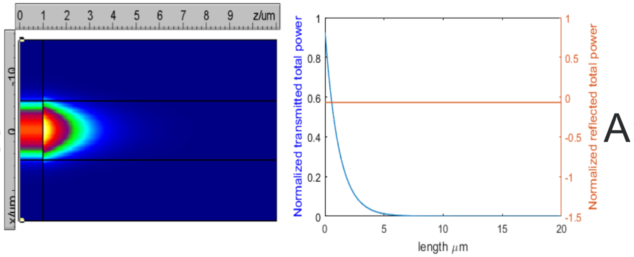

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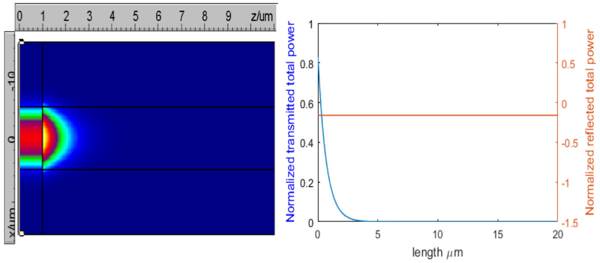
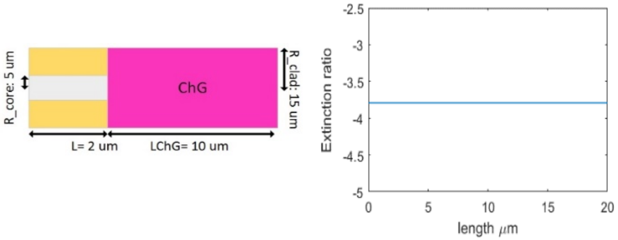


Rad Hard optical (Nufern (S1550-HTA)) fiber with measured data:
- Core refractive index: 1.45735
- Cladding refractive index: 1.44715

ChG (amorphous)	2.45	0.10
ChG (crystalline)	3.39	0.17



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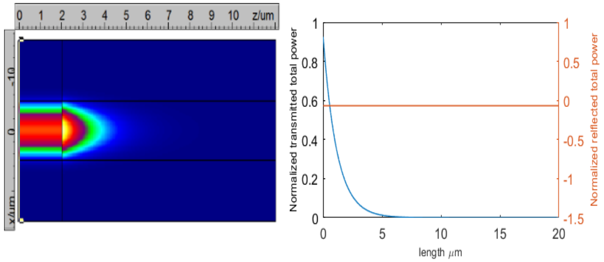

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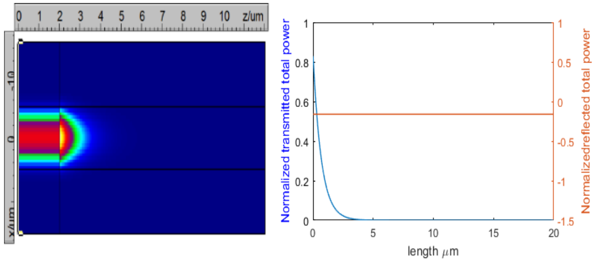
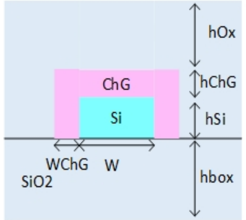


Design and Simulation with Measured Data

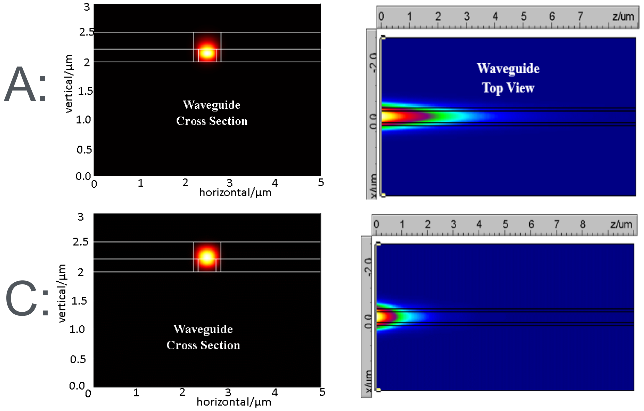
- We optimized our designs with a synthesized $Ge_{40}S_{60}$ chalcogenide glass.
- The complex refractive index is measured using ellipsometry.
- The operating wavelength is set at λ = 1550 nm.

Material	Refractive index (n)	Extinction coefficient (k)
ChG (amorphous)	2.45	0.10
ChG (crystalline)	3.39	0.17
Si	3.47	0
SiO2	1.45	0

Design # 1a



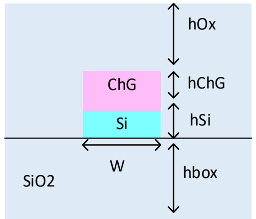
High extinction ratio of 70 dB in a 22 µm long waveguide.



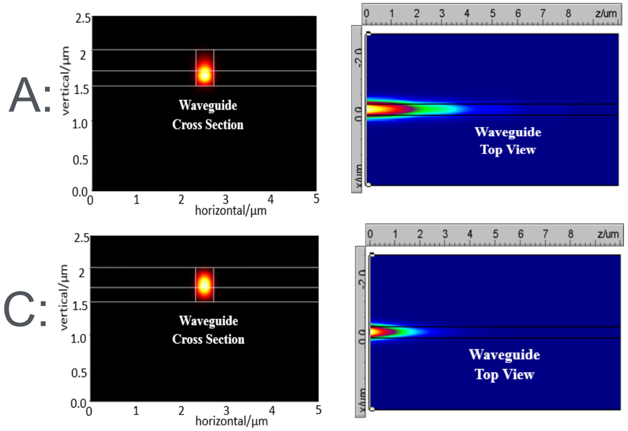
Optimum design parameters obtained for design 1a.

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

Design # 3a



High extinction ratio of 70 dB in a 22 µm long waveguide.



Optimum design parameters obtained for design 3a.

Parameters	Value (µm)
W	0.4
hChG	0.3
hSi	0.22
hOx	0.5
hbox	1.5

Conclusion

With the conducted research throughout the first year of the project accomplishment we met the planned milestones in (i)synthesis of chalcogenide glasses with different crystallization temperatures, (ii)the characterization of their thermal and optical properties of importance for the hybrid and fiber based plasmonic sensors; (iii)accomplished design simulation for establishment of devices architecture; (iv)developed ink based on synthesized materials;(v)conducted design optimization for the hybrid plasmonic sensors, based on measured optical properties of the materials;(vi) measured and simulated fiber optic based plasmonic sensors.

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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Clean. **Reliable.** **Nuclear.**