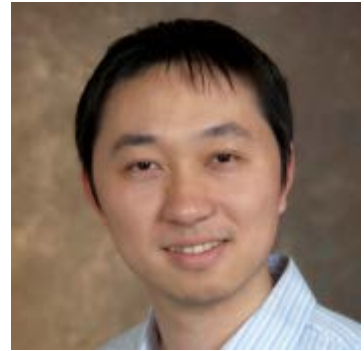


Deposition of a lateral gradient chalcogenide glass film via PVD thermal evaporator



Dr. Juejun Hu

BY: ALVIN CHANG, SAMUEL SERNA, JUEJUN HU

2018 MIT MRL SUMMER SCHOLARS REU



MIT MATERIALS
RESEARCH
LABORATORY

Oregon State University



Fun Fact: Corvallis is a hotspot for bird watching.



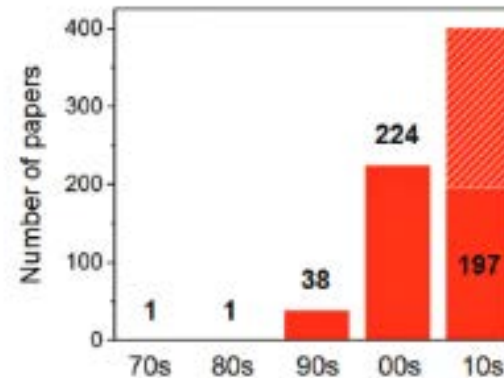
Background-Chalcogenide Glasses

Chalcogenide glasses are a family of inorganic amorphous materials containing chalcogen elements.

Chalcogenide glasses possess exceptional optical properties such as broadband IR transparency and large optical nonlinearity, making them particularly advantageous for microphotonics.

The periodic table highlights the chalcogen elements: Sulfur (S), Selenium (Se), and Tellurium (Te). A red box encloses these elements, with lines pointing to a detailed inset table for each.

16 S Sulfur 32.066
34 Se Selenium 78.96
52 Te Tellurium 127.60



<http://web.mit.edu/hujuejun/www/My%20Papers/Journal%20Papers/Chalcogenide%20glass%20microphotonics%20--%20Stepping%20into%20the%20spotlight.pdf>

Nonlinear optical functionalities

Supercontinuum generation

Wavelength-division multiplexing

λ conversion

Amplification and lasing

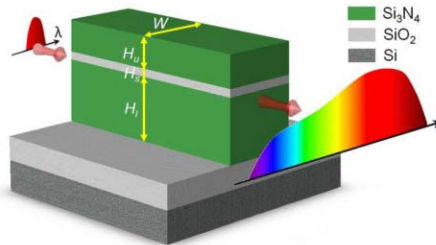
Frequency combs

Routing (switching/logic)

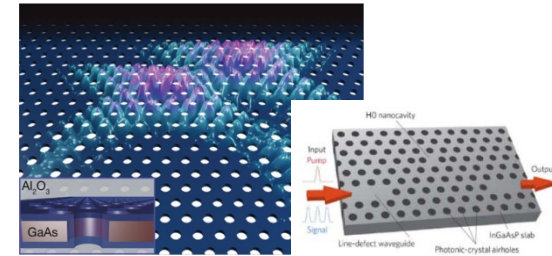
Modulation

Signal regeneration

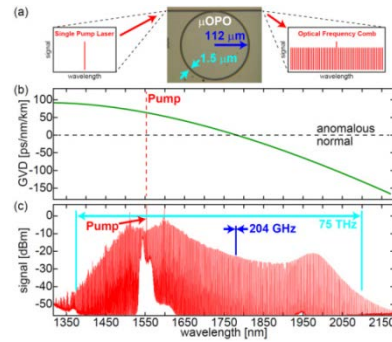
Integrated entangled photon sources



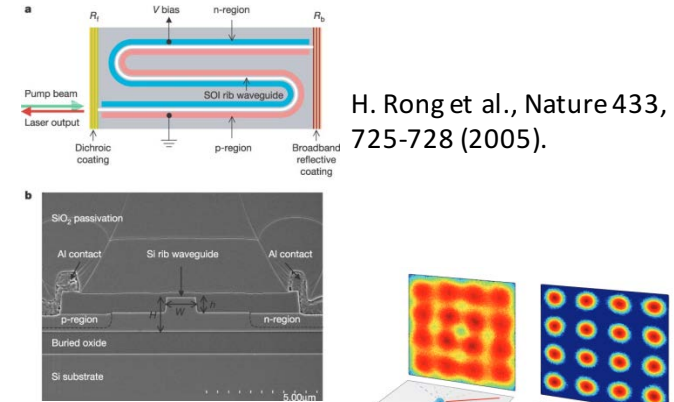
L. Zhang, et al., *Opt. Express* 19, 11584-11590 (2011).



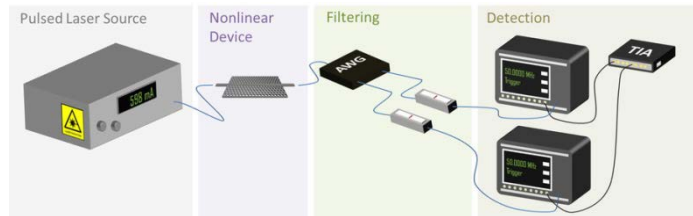
Moille et al., *Laser & Photonics Reviews* 10(3) (2016).
K. Nozaki, et al., *Nat. Photonics* 4, 477-483(2010).



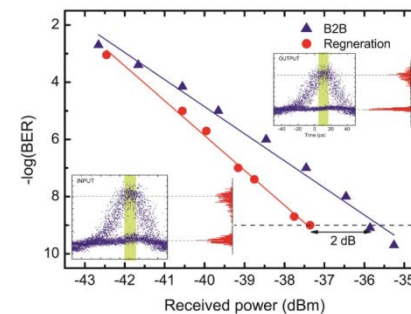
M. A. Foster, et al., *Opt. Express* 19, 14233-14239 (2011).



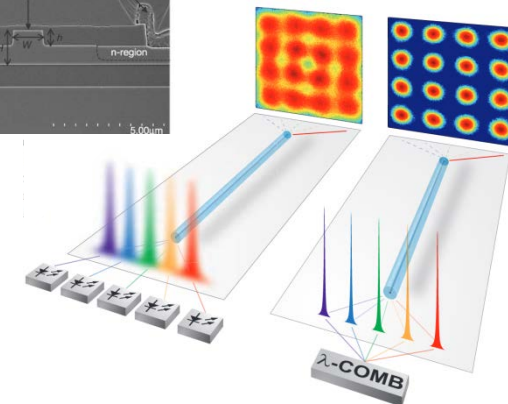
H. Rong et al., *Nature* 433, 725-728 (2005).



Husko et al., *Scientific Reports* 3, 3087 (2013)

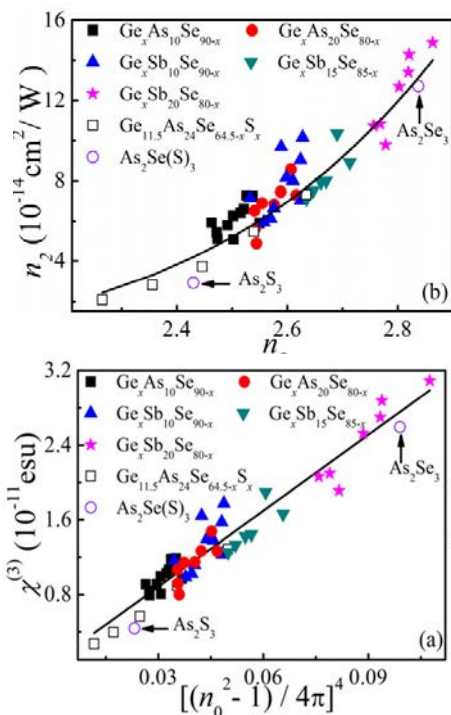


K.-Y. Wang, et al., *IEEE 9th GFP*(2012), pp. 308-310.

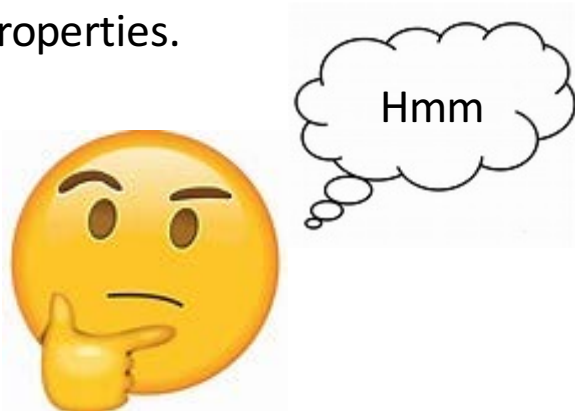


Temprana et al., *OPN*, March 2016, p. 30

Background-Chalcogenide Glasses



Chalcogenide glasses exhibit exceptional nonlinear properties.



However, they also display considerable nonlinear losses

Compositions	E_g (eV)	Wavelength	n_2 (10^{-14} cm ² /W)	β_2 (10^{-9} cm/W)	FOM_{2PA}
As ₂ S ₃	2.22	1150nm	4.33	< 0.01	> 30
		1250nm	3.67	< 0.01	> 25
		1350nm	3.50	< 0.01	> 24
		1450nm	3.23	< 0.01	> 22
		1550nm	2.85	< 0.01	> 20
		1686nm	2.79	< 0.01	> 19
Ge _{11.5} As ₂₄ Se _{64.5}	1.75	1150nm	11.8	1.20	1
		1250nm	10.4	0.35	2
		1350nm	8.83	0.11	6
		1450nm	7.67	< 0.01	> 57
		1550nm	7.90	< 0.01	> 59
		1686nm	6.83	0.10	4
Ge ₁₅ Sb ₁₀ Se ₇₅	1.72	1150nm	12.5	1.27	1
		1250nm	9.00	0.35	2
		1350nm	7.67	0.12	5
		1450nm	8.30	0.05	11
		1550nm	7.50	< 0.01	> 52
		1686nm	7.33	< 0.01	> 51
Ge ₁₅ Sb ₁₅ Se ₇₀	1.62	1150nm	15.5	5.94	0.2
		1250nm	14.9	2.78	0.4
		1350nm	13.7	0.81	1
		1450nm	12.2	0.49	2
		1550nm	10.0	0.35	2
		1686nm	10.0	0.27	2
Ge _{12.5} Sb ₂₀ Se _{67.5}	1.57	1150nm	20.3	7.44	0.2
		1250nm	17.5	3.05	0.5
		1350nm	13.5	0.94	1
		1450nm	12.0	0.45	2
		1550nm	11.4	0.37	2
		1686nm	9.40	0.22	3

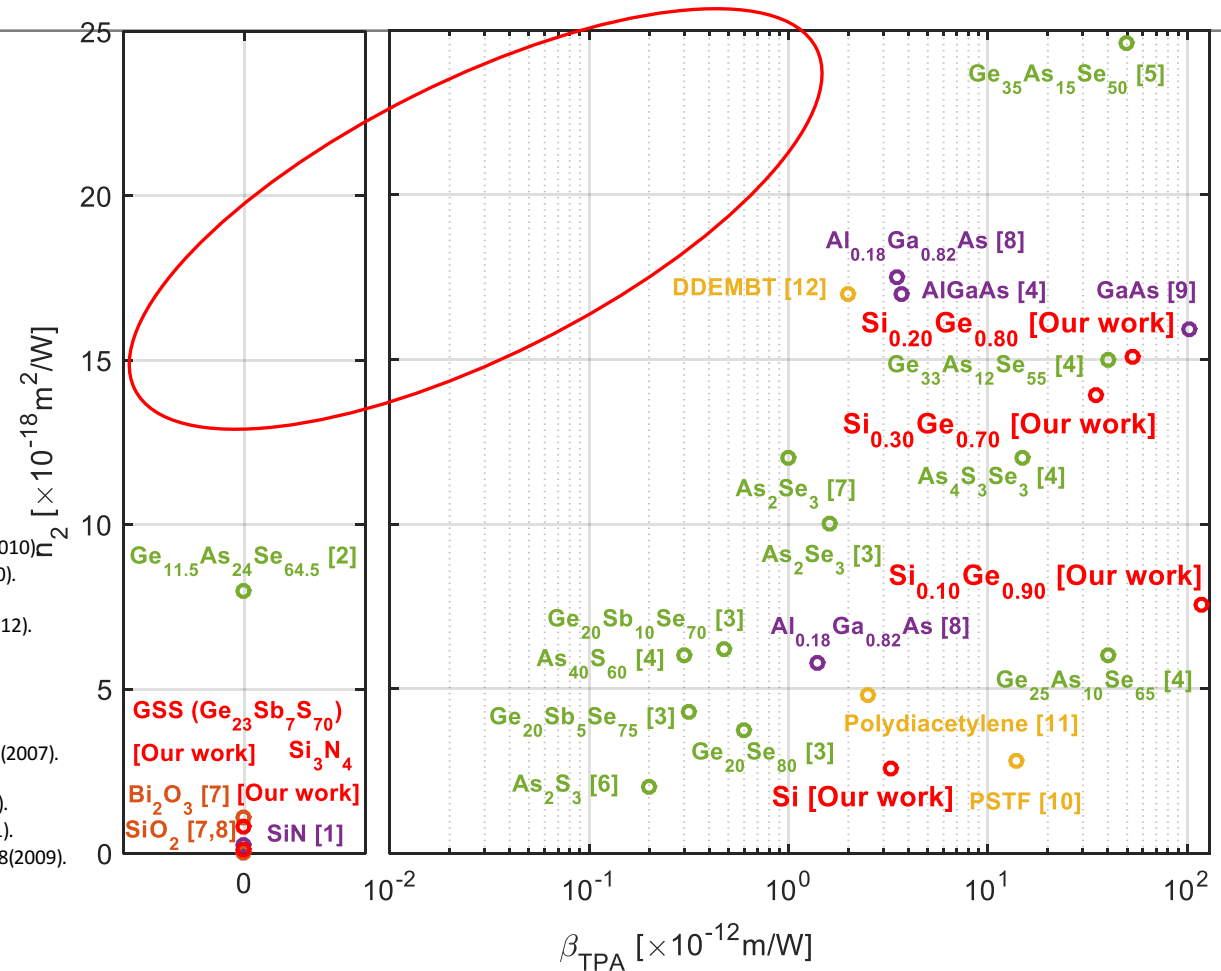
Wang, Ting, et al. "Systematic z-scan measurements of the third order nonlinearity of chalcogenide glasses." *Optical Materials Express* 4.5 (2014): 1011-1022.

Objectives

In this project, we aimed to fabricate a film that has both low nonlinear losses and high nonlinearities

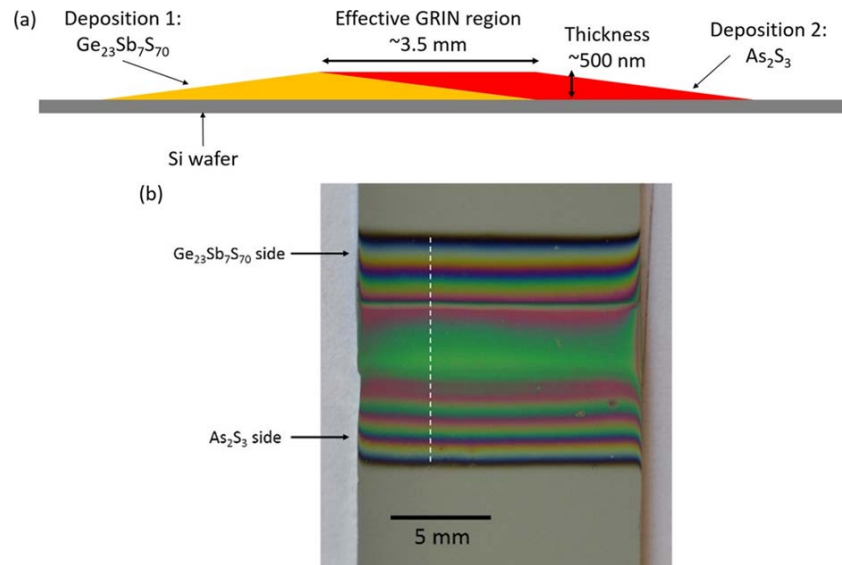
- Our work
- Chalcogenides
- III – V and SiN
- Polymers
- Oxides

[1] Tan et al., *Appl. Phys. Lett.*, 96, 061101–061103(2010)
 [2] Choi et al., *Opt. Express*, 18(18):18866–18874(2010).
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 [10] Asobe et al., *Appl. Phys. Lett.*, 67, 891-893(1995).
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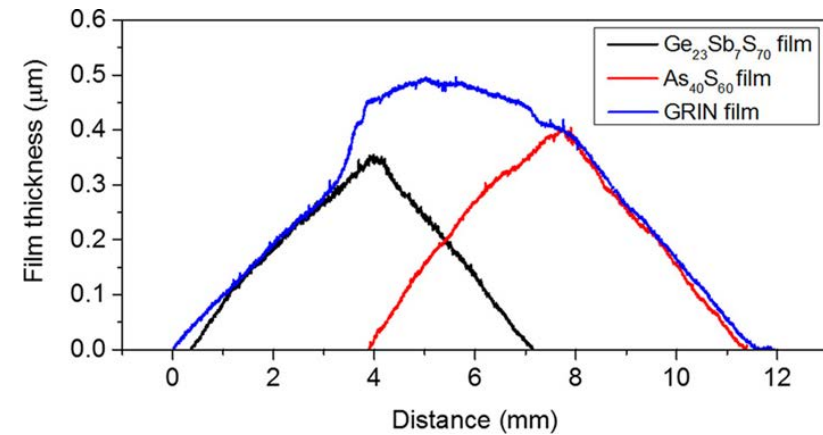


Direct Electropray Printing Example

Below is an electropray printing example of a lateral gradient chalcogenide film.

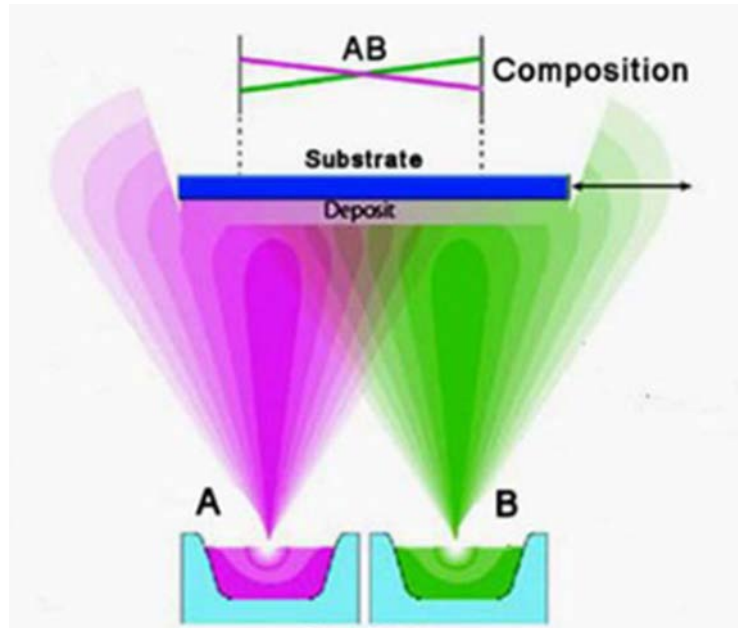


(a) Cross-sectional schematic and (b) photograph of a multilayer GRIN film



Individual film thickness profiles of $\text{Ge}_{23}\text{Sb}_7\text{S}_{70}$ and $\text{As}_{40}\text{S}_{60}$ and the resulting bilayer GRIN film thickness as a function of the spatial position. The peaks of the thickness profiles of the individual films are separated by 4 mm, the approximate distance between the films during fabrication of the GRIN film.

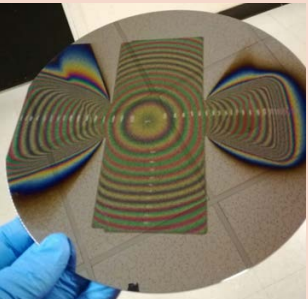
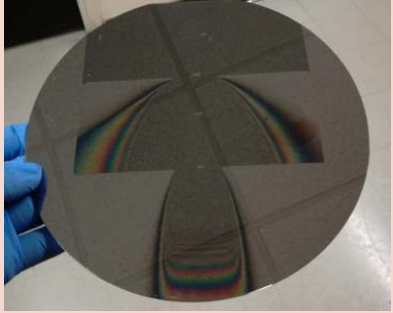
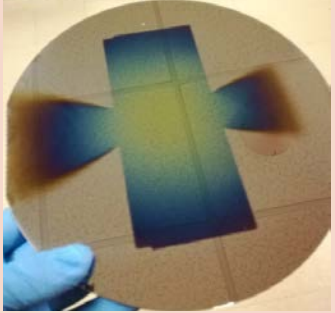
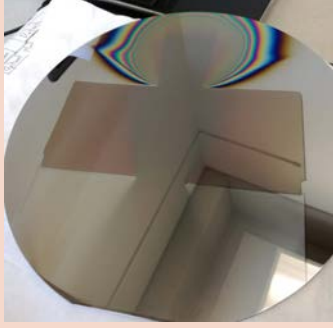
PVD



We hope to use the PVD thermal evaporator to deposit a lateral gradient film of $\text{Ge}_2\text{Sb}_2\text{Se}_5$ and $\text{Ge}_{23}\text{Sb}_7\text{S}_{70}$

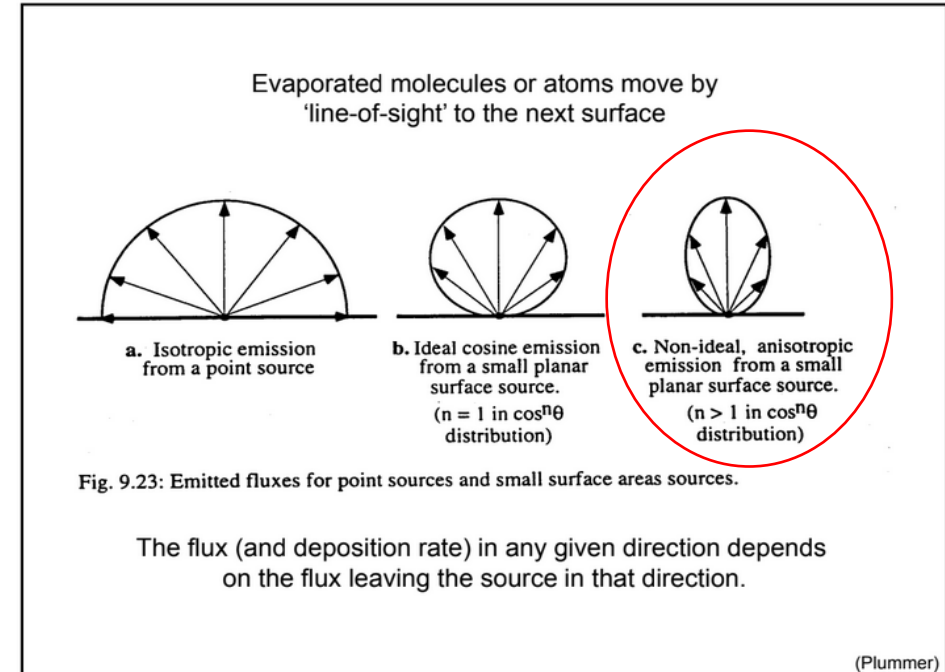
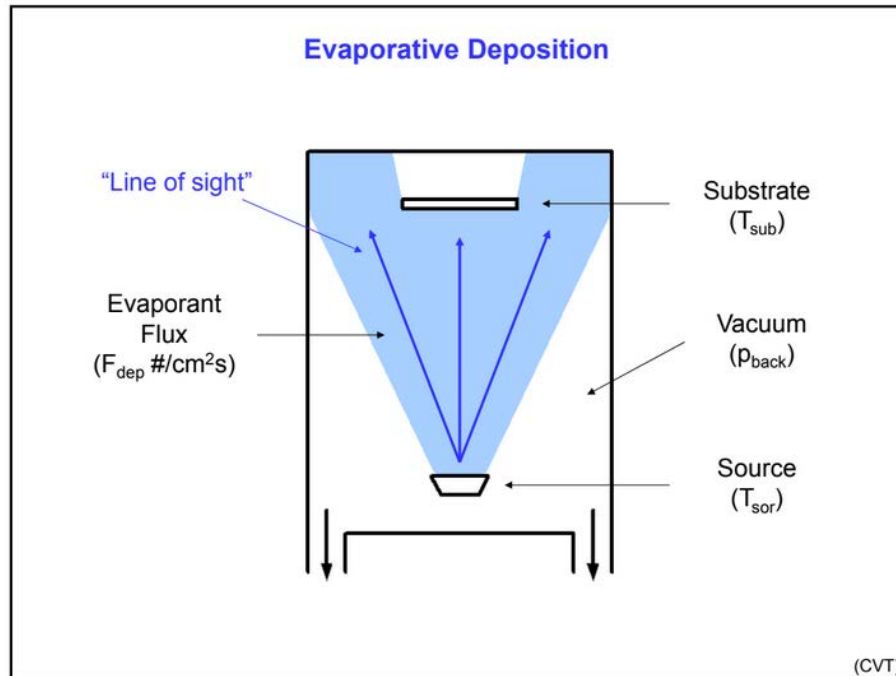
Early PVD runs

Many first PVD runs were unsuccessful. Because of the size of our substrates, we did not have access to any information from the crystals so we had to use trial and error with various time and power ramps.

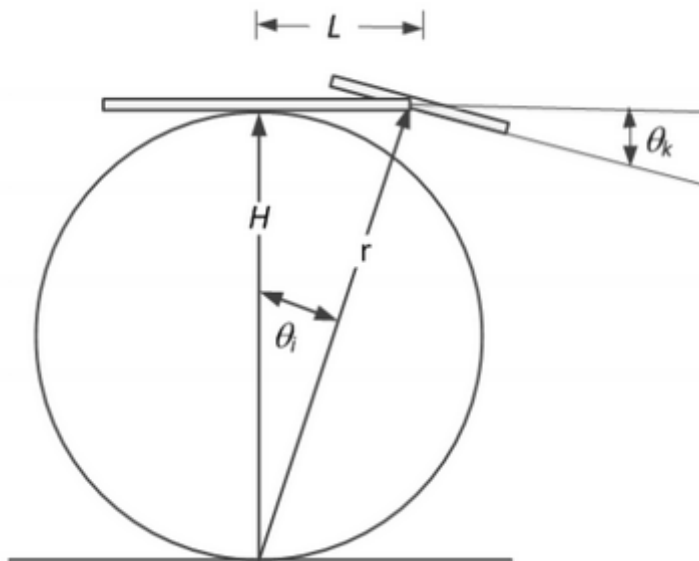
Single evaporation of GSS	Single evaporation of GSSe	First coevaporation	Second coevaporation
			
Too thick (1.6 μm average)	Too thick (16.5 μm average)	Too thin (40-80 nm)	Too thick on the GSSe side (4-5 μm) and GSS side (3-4.5 μm)

Modeling

Before we started modeling, we wanted to first verify that the evaporation followed a line of sight model.



Modeling



$$F_{dep,L} = \frac{N_e}{2\pi r^2} \cos^n(\theta_i) \cos(\theta_k)$$

Key Assumptions

($n > 1$ in $\cos^n\theta$ distribution)

$$\theta_i = \theta_k$$

$$\frac{H}{r} = \cos(\theta_i)$$

$$F_{dep,0} = \frac{N_e}{2\pi H^2}$$

Final Simplified Thickness Model

$$F_{dep,L} = F_{dep,0} \left(\frac{H}{r} \right)^{n+2}$$

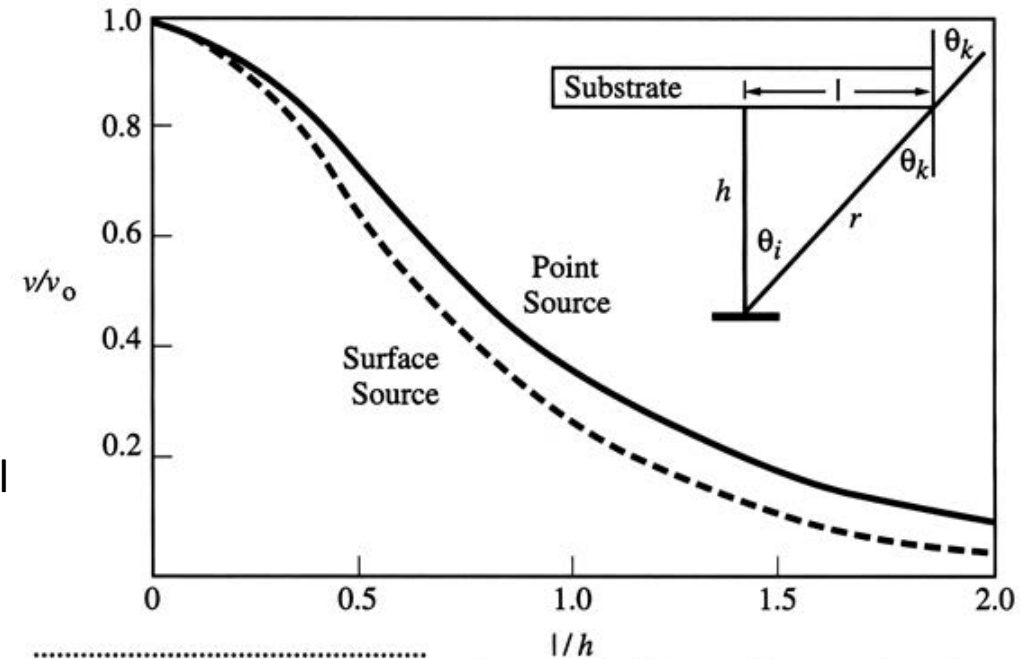


Figure 9-17 Deposition rate of evaporated film as a function of position on substrate for point and surface sources. $\theta_i = \theta_k$ in this configuration for both point and surface sources. (After [9.6].)

Modeling

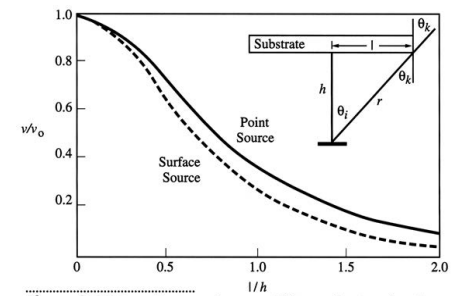
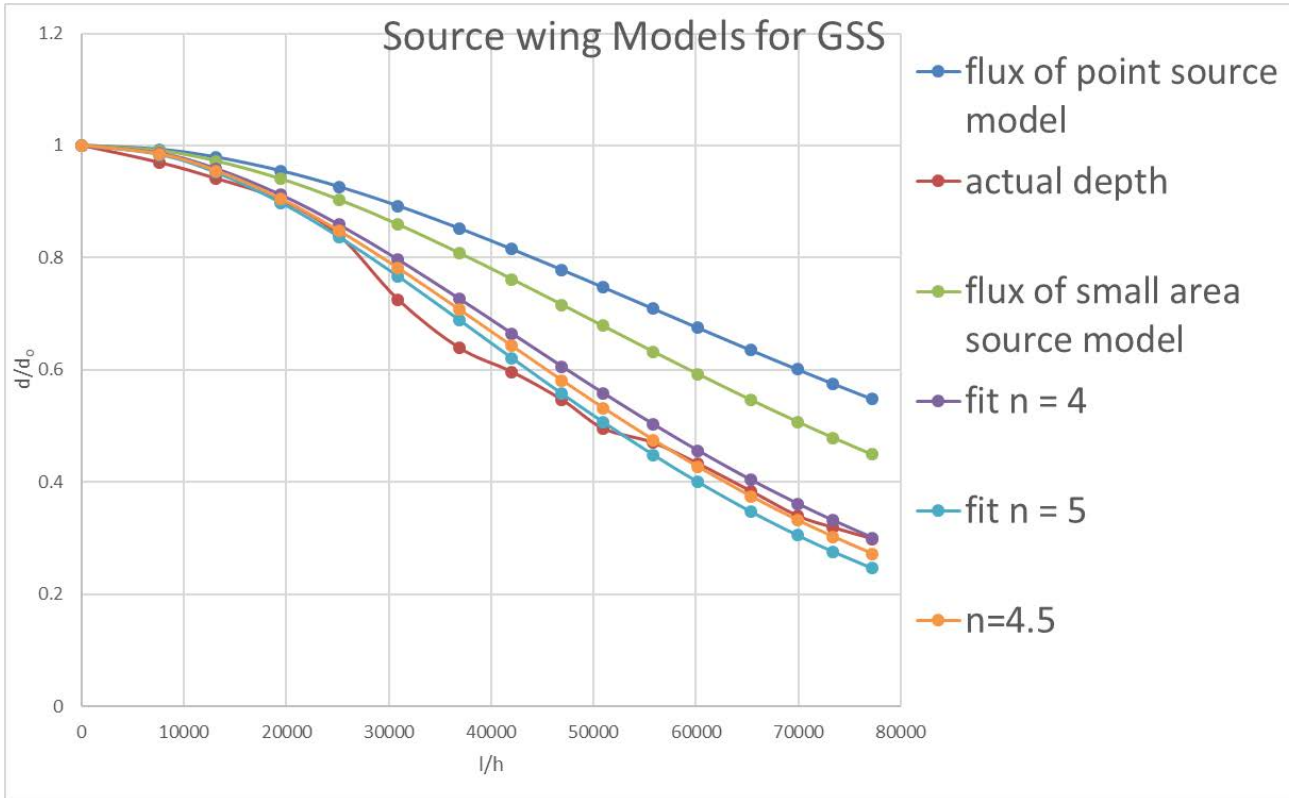
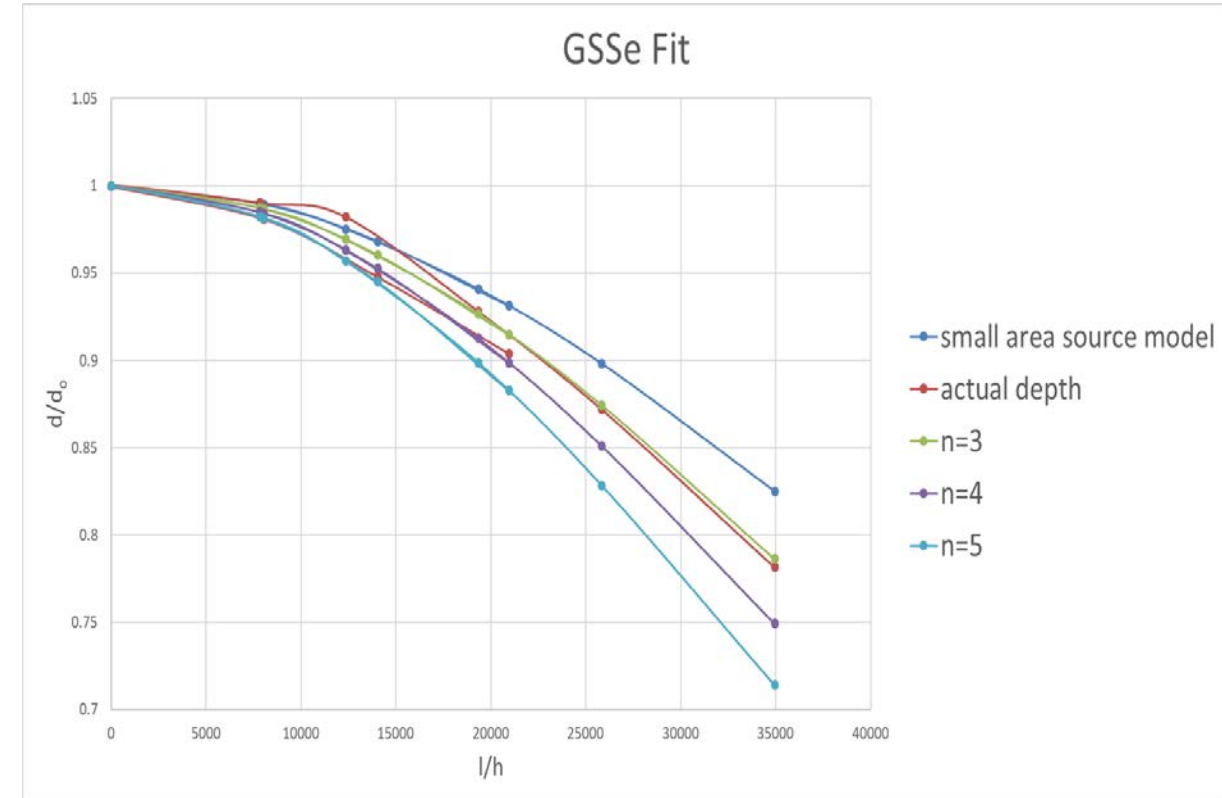


Figure 9-17 Deposition rate of evaporated film as a function of position on substrate for point and surface sources. $\theta_i = \theta_k$ in this configuration for both point and surface sources. (After [9.6].)



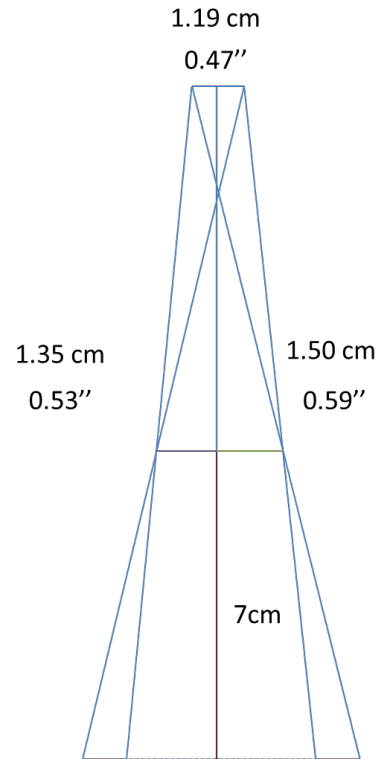
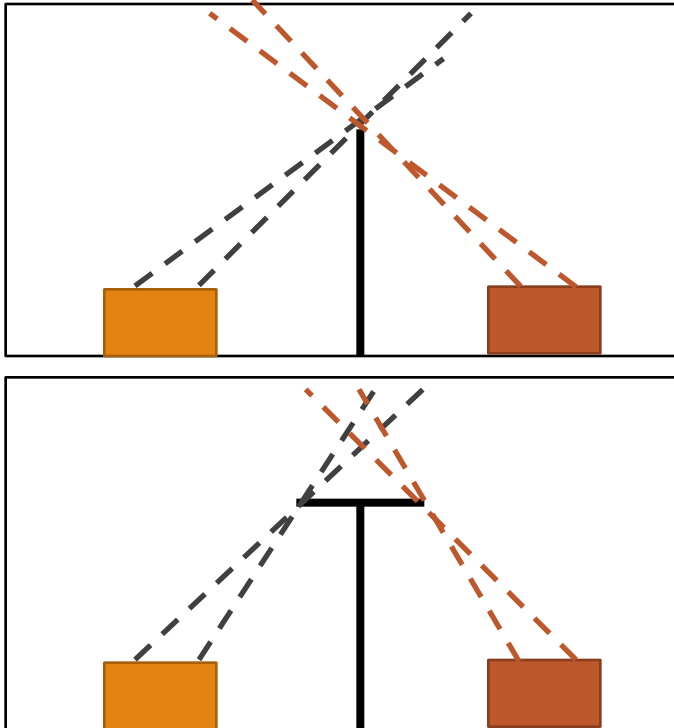
The GSS model shows a fit of around $n=4.5$ to $n=5$



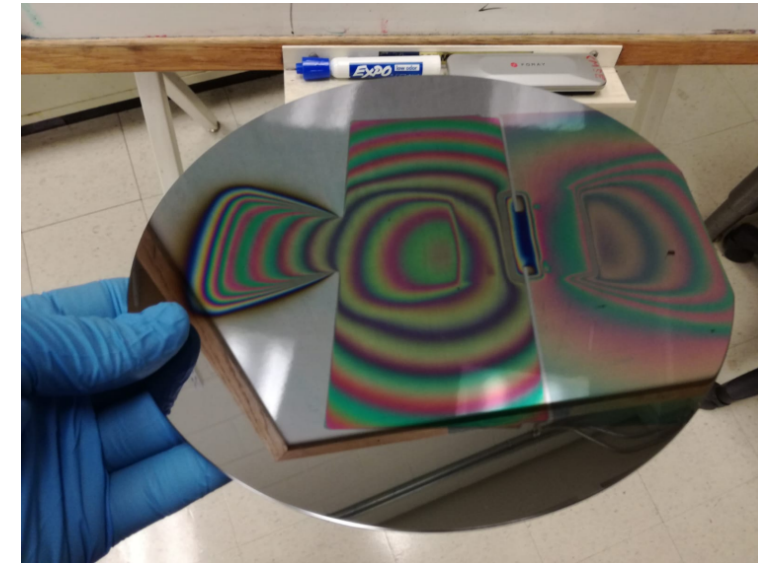
The GSSE model shows a fit of around $n=3$

Creating Overlap

We needed to add an obstacle to create overlap between the two line of sight models.



According to our geometrical calculations, we can get a 1.19 cm overlap of overlap using a 2.85cm obstacle.

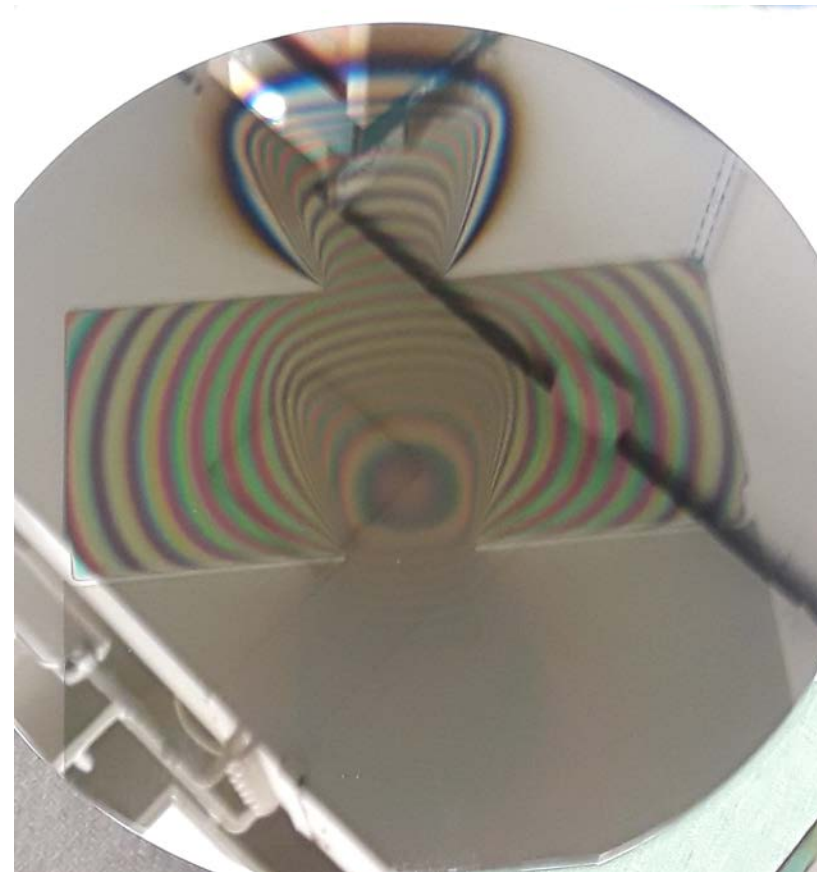


However, our film turned out to have an odd gradient—caused by leaking from the sides of the obstacle!

Creating Overlap

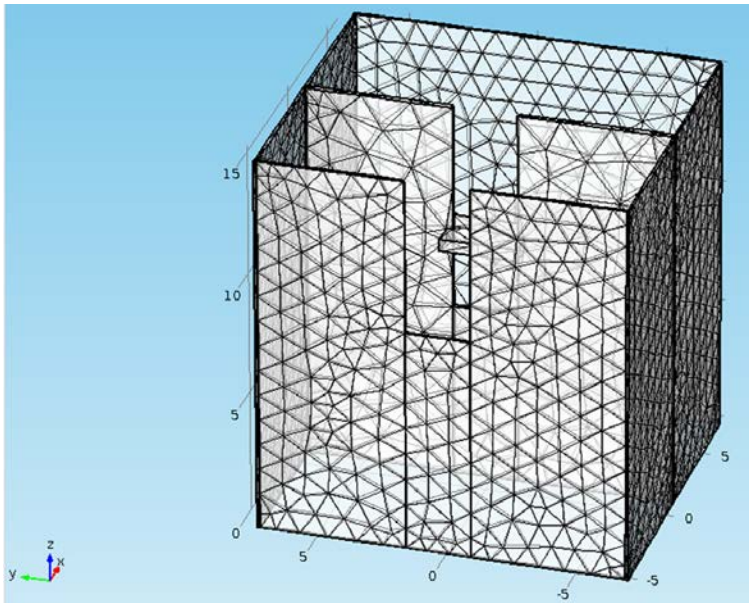


We made a new overlap using aluminum foil that wouldn't have leaks.

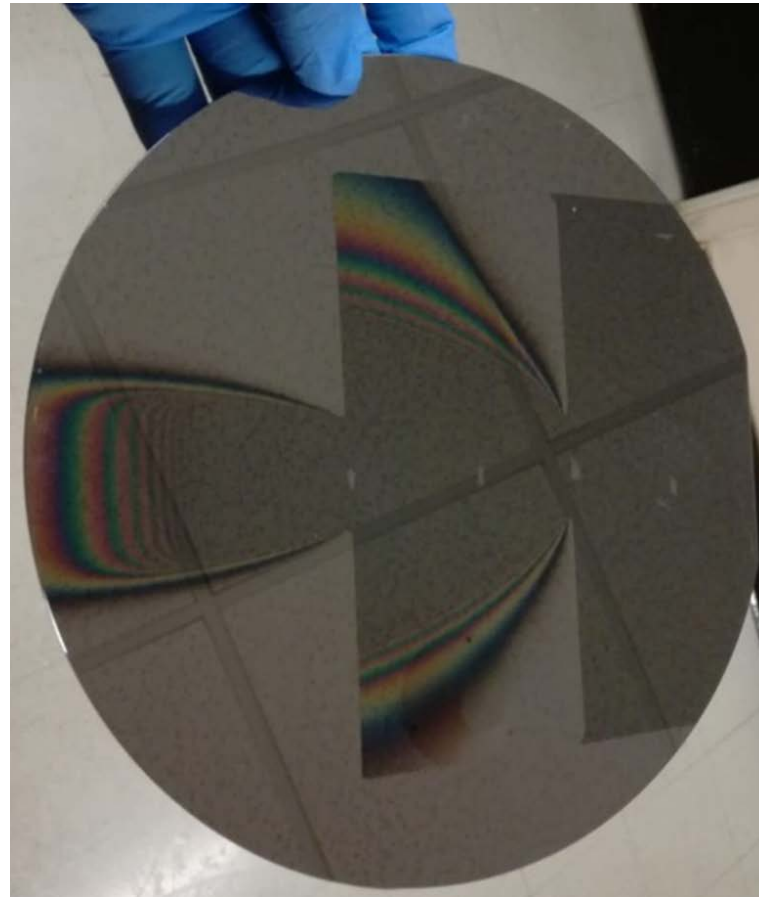


Our film ended up with a nice region of overlap and without the unusual super thick portion in the middle.

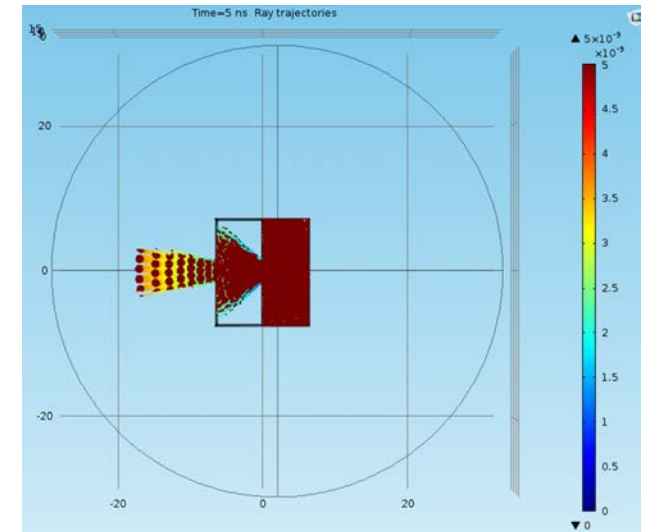
Modeling (COMSOL)



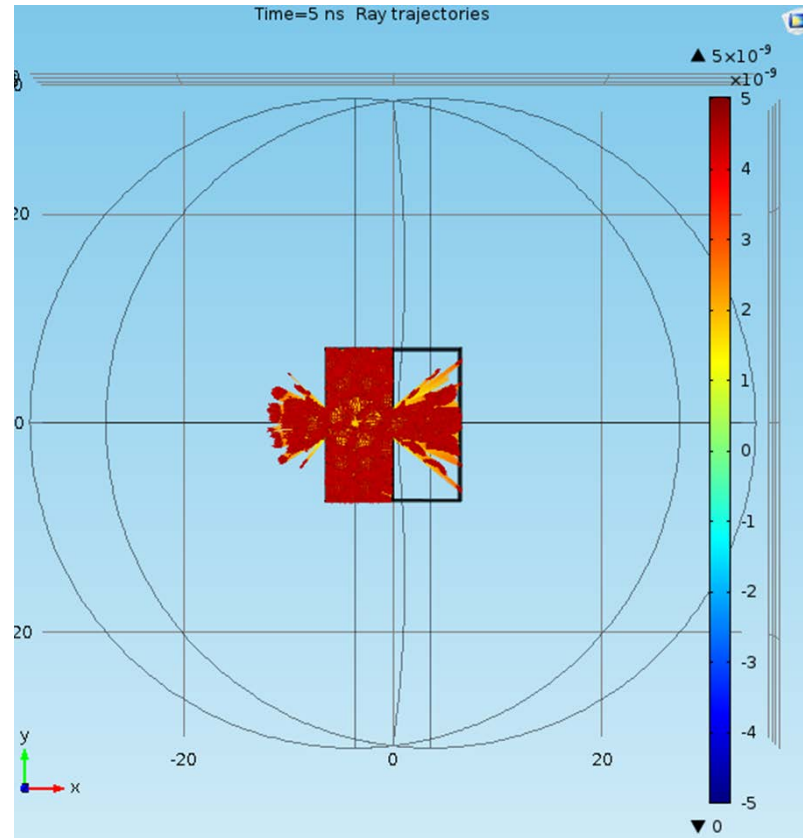
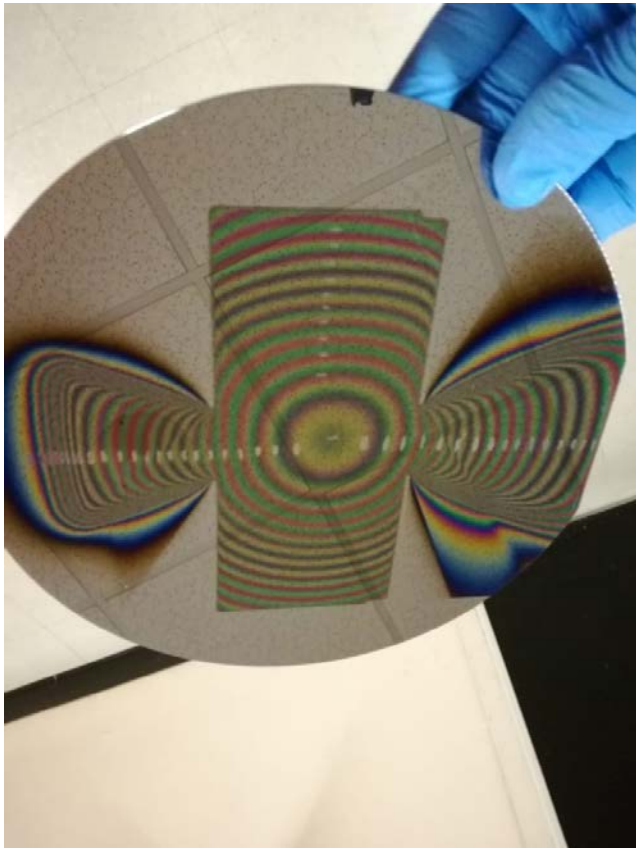
We created a representation of our setup in COMSOL using the ray optics geometric optics module.



Example of a COMSOL model compared to the actual PVD experiment.

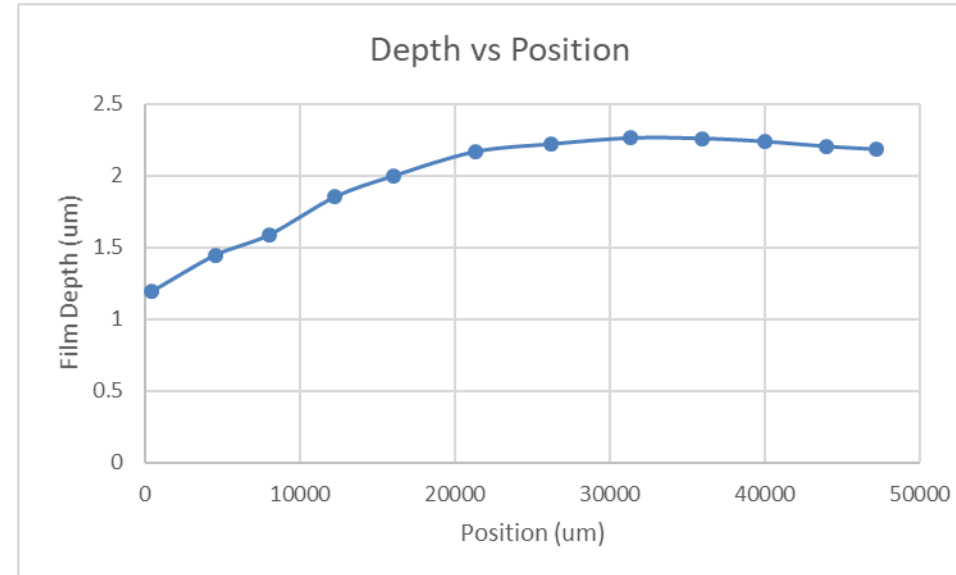
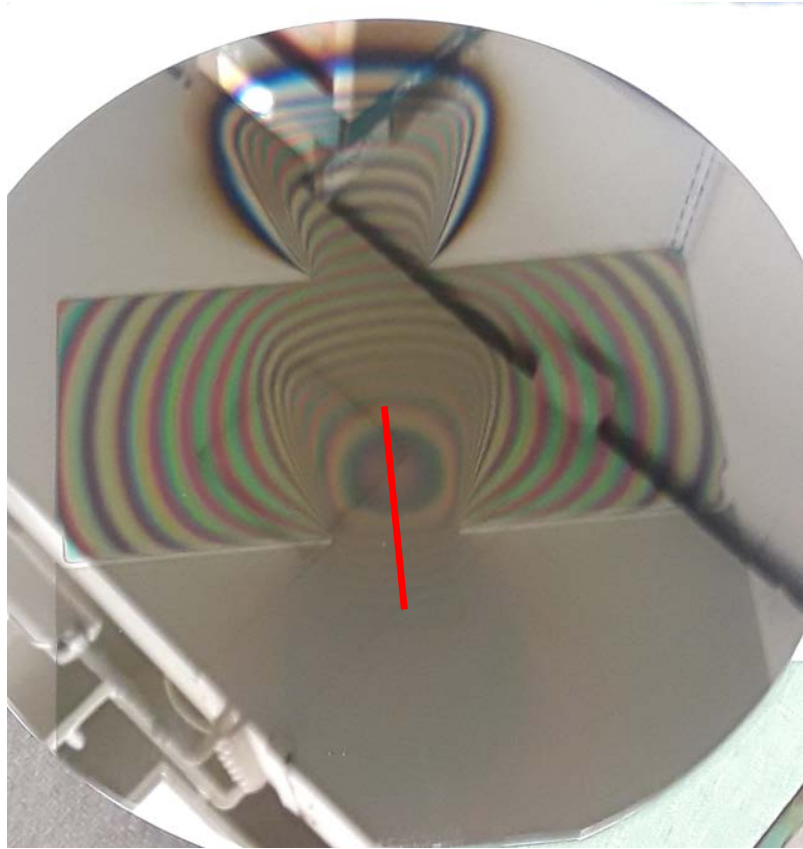


COMSOL modeling



Another example of a COMSOL model compared to the actual run. It gives a good prediction of the shape of the film but not of the intensity.

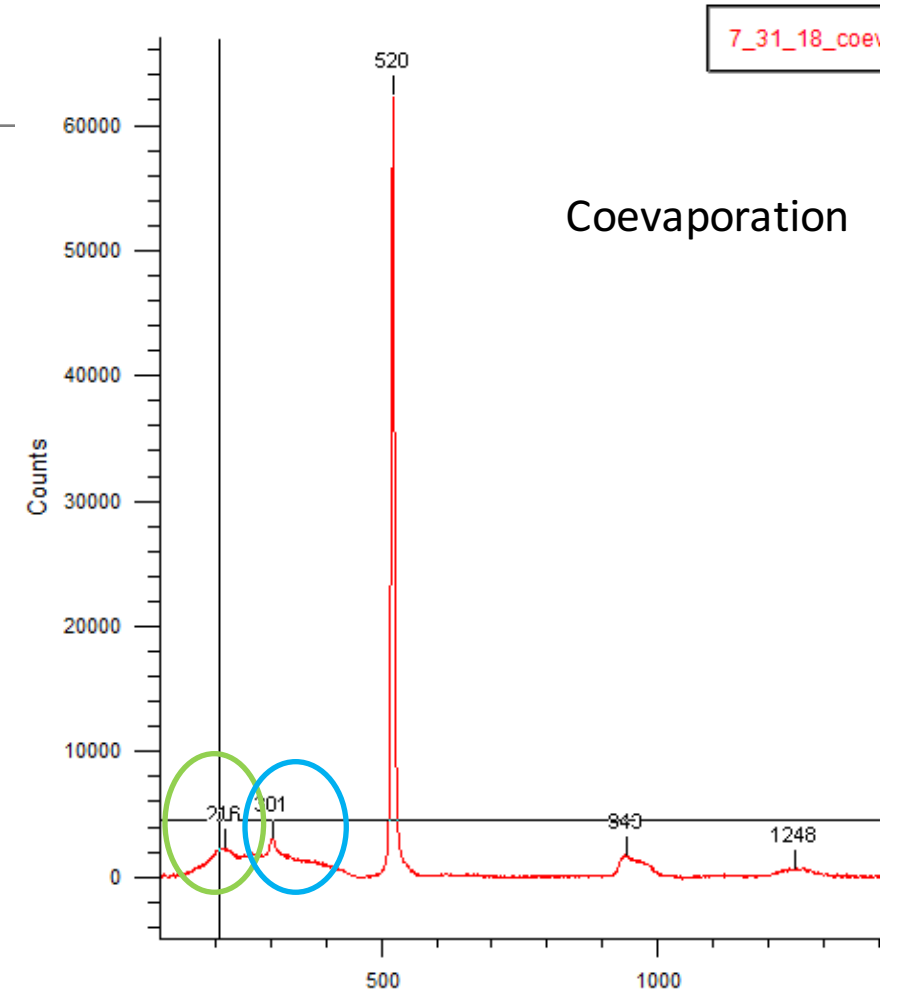
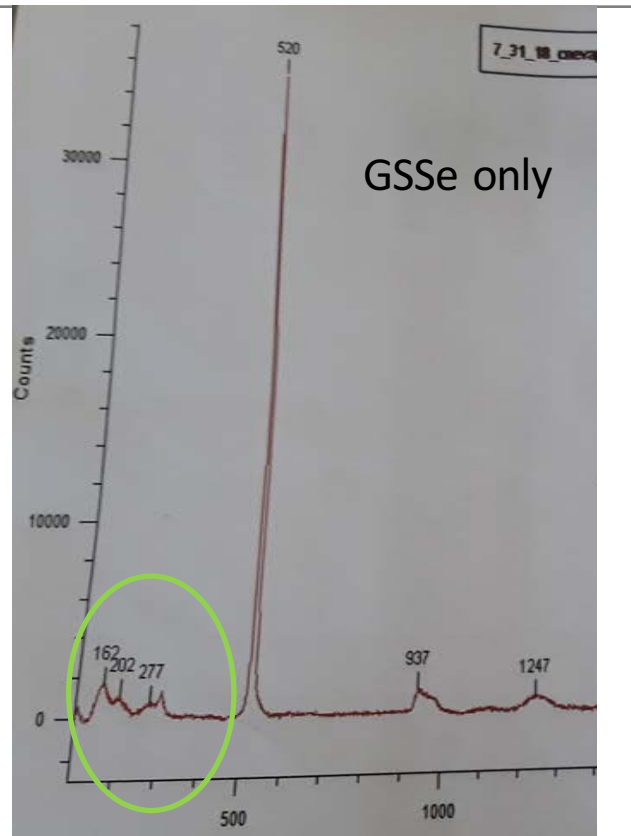
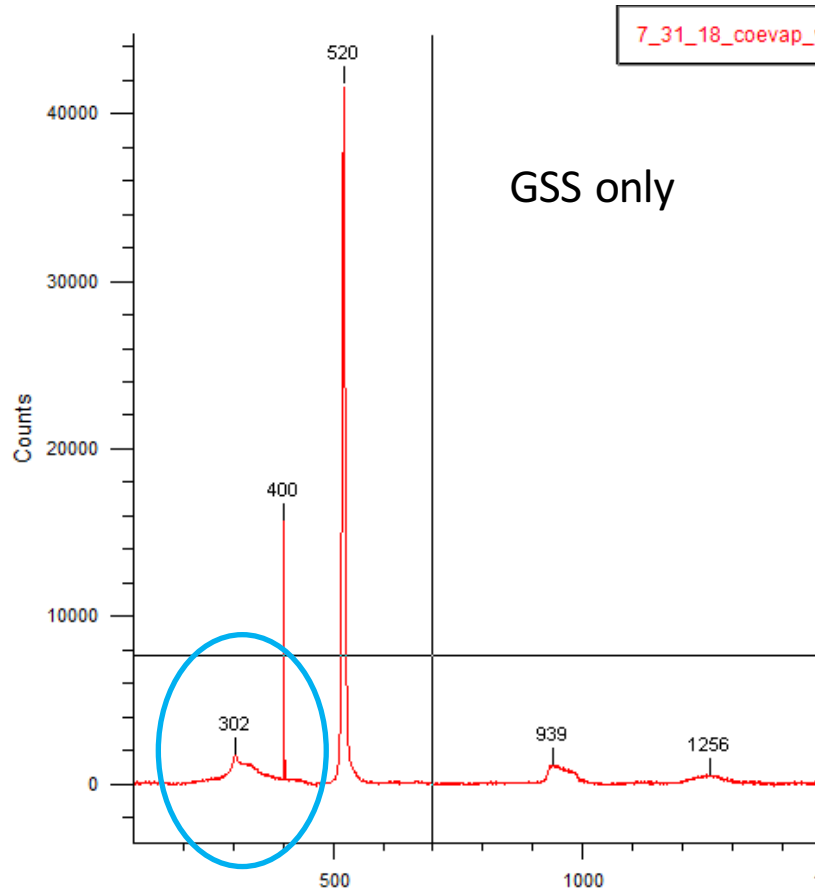
Current Work-Profilometer



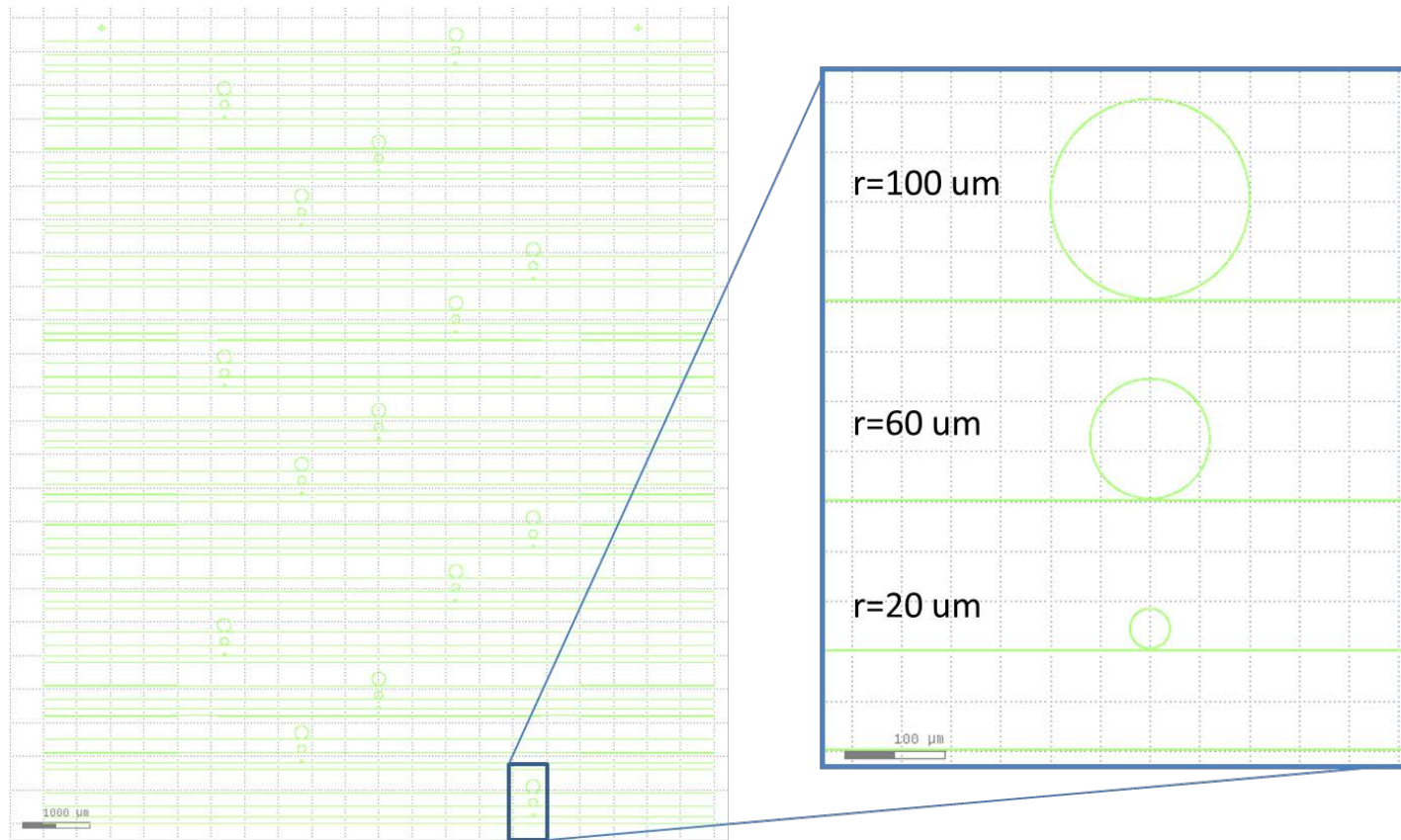
Around 3cm of uniformity ($\sim 2\mu\text{m}$ thick). The highest variation in this region is 2.5% from the average thickness.

Our resulting film shows a very similar depth profile to the GRIN film made in previous works. The red line shows where the profilometer measurements were taken.

Current Work-Raman



Ring Resonators



We hope to use ring resonators fabricated in a double layer liftoff approach of various lengths to determine the refractive index of our material.

We also plan to simulate this using Lumerical mode solver.

Free Spectral Range

RR Radius	RR length	lambda_0	n_max	n_min	FSR_min (nm)	FSR_max
100	628.318	1.55	2.6	2.18	1.47065413	1.753991164
60	376.9908	1.55	2.6	2.18	2.451090216	2.923318607
20	125.6636	1.55	2.6	2.18	7.353270649	8.76995582

Free Spectral Range calculations for our material.

Note: the group index is not the material refractive index but we can still use it to quantify the gradient.

$$FSR = \frac{\lambda_0^2}{n_g L}$$

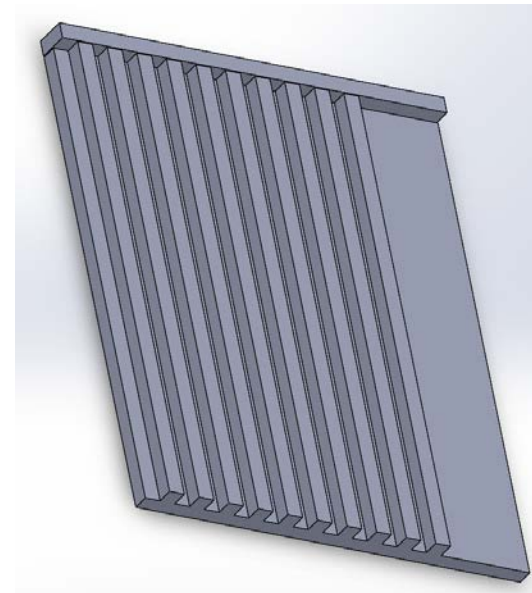
Future work

3-d print a solidworks holder to further increase sample height.

Other measurements = microTA analysis, EDS (can get a map of the composition of the film but we were unable to get training on it), optical properties (refractive index with RRs), measure NL with D-scan measurements.

Ring Resonator work = shoutout to Skylar

Optimizing of COMSOL model and development of an optimal obstacle.



Current solidworks holder design

Acknowledgments/What I learned

OPTICS

PVD

Picwriter (shoutout to Derek)

Lumerical

Basic Solidworks

Basic Python

Big shoutouts to Samuel for being a good mentor throughout the entire project and to JJ for the opportunity