Pulsed Laser Deposition of a Silver Film For Laser-Induced Breakdown Spectroscopy Emission Enhancement of Bacteria

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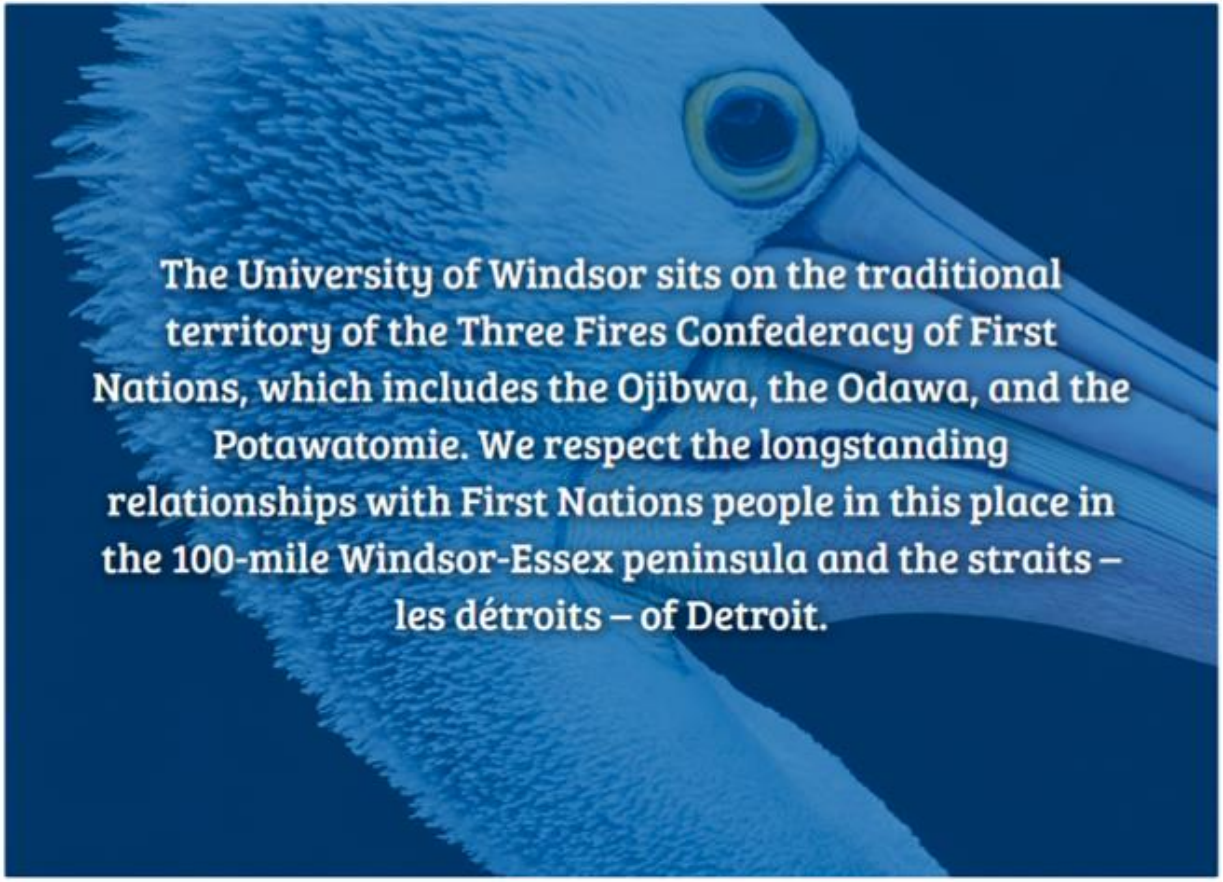

The University of Windsor sits on the traditional territory of the Three Fires Confederacy of First Nations, which includes the Ojibwa, the Odawa, and the Potawatomie. We respect the longstanding relationships with First Nations people in this place in the 100-mile Windsor-Essex peninsula and the straits – les détroits – of Detroit.

Motivation

Current methods of bacterial identification in clinical settings are contributing to the ongoing antibiotic resistance crisis:

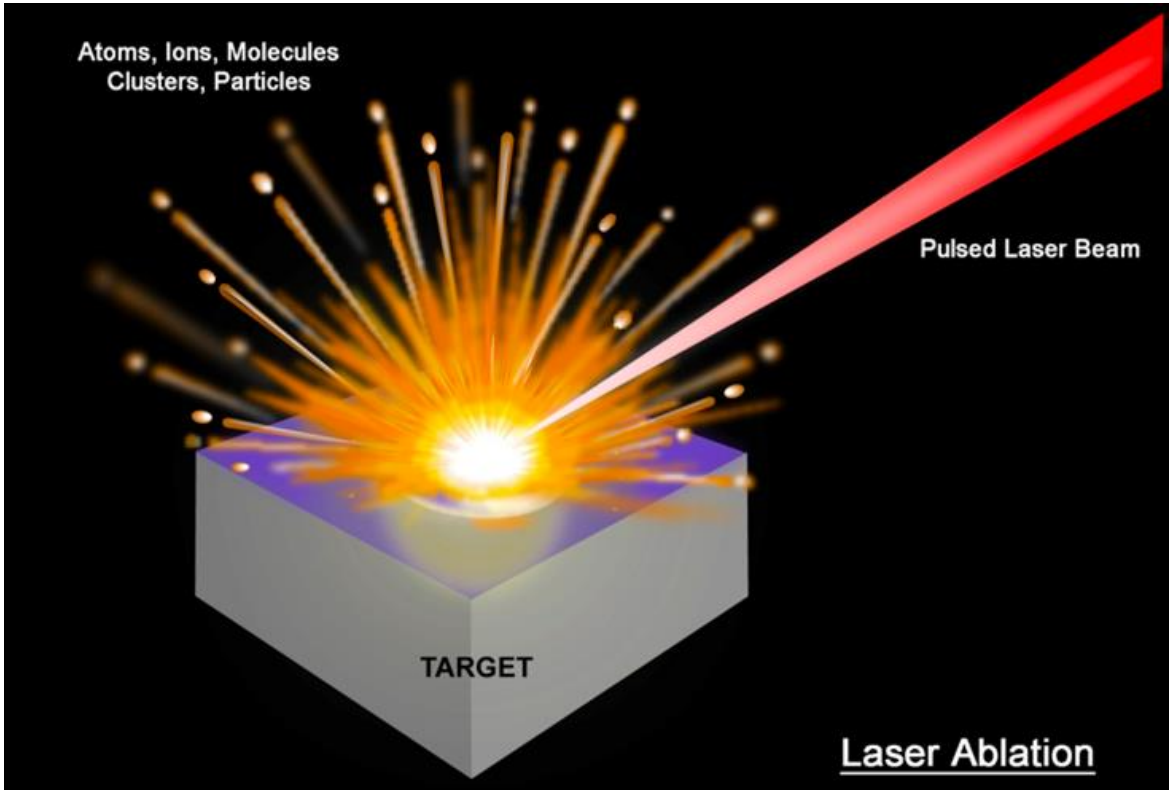
- ➔ They can be slow (take 2-3 days)

We use laser-induced breakdown spectroscopy (LIBS) on bacteria to identify and classify the bacteria species

- ➔ It's very fast (under 1 min) and requires minimal sample preparation

What is LIBS?

A spectrochemical technique used to rapidly determine elemental composition



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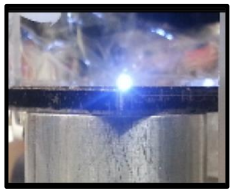
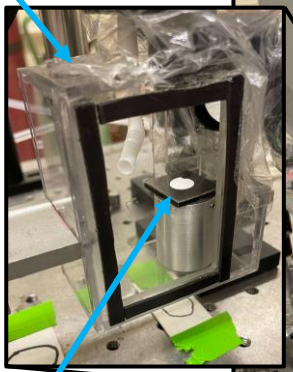
LIBS Experimental Setup

Nd:YAG 1064 nm laser, 10 ns pulse duration, 10 Hz pulse frequency

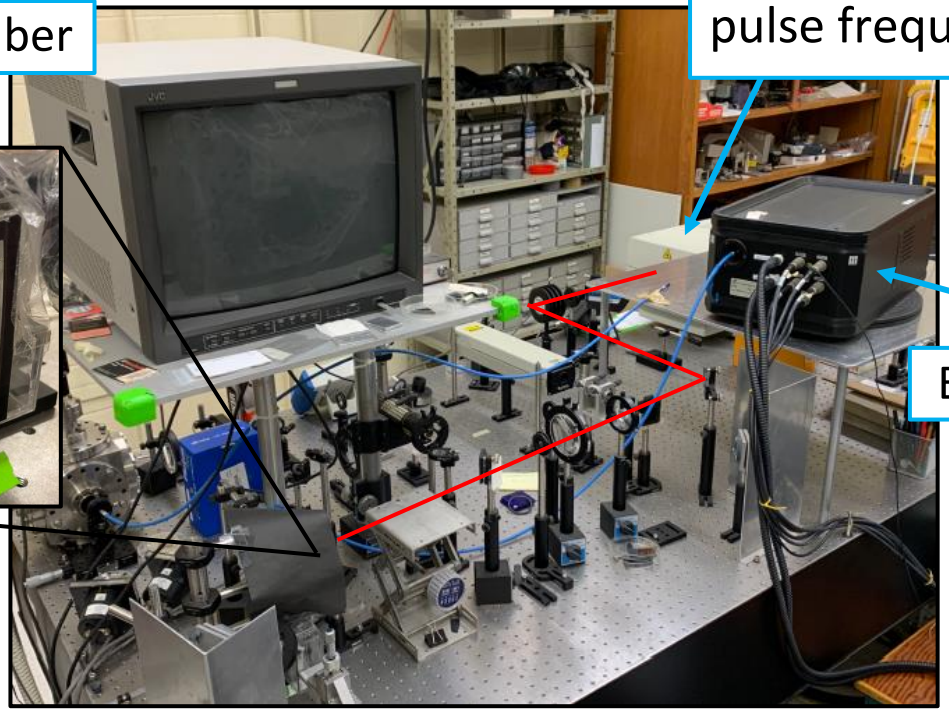
Argon purge chamber

Bacteria sample being ablated





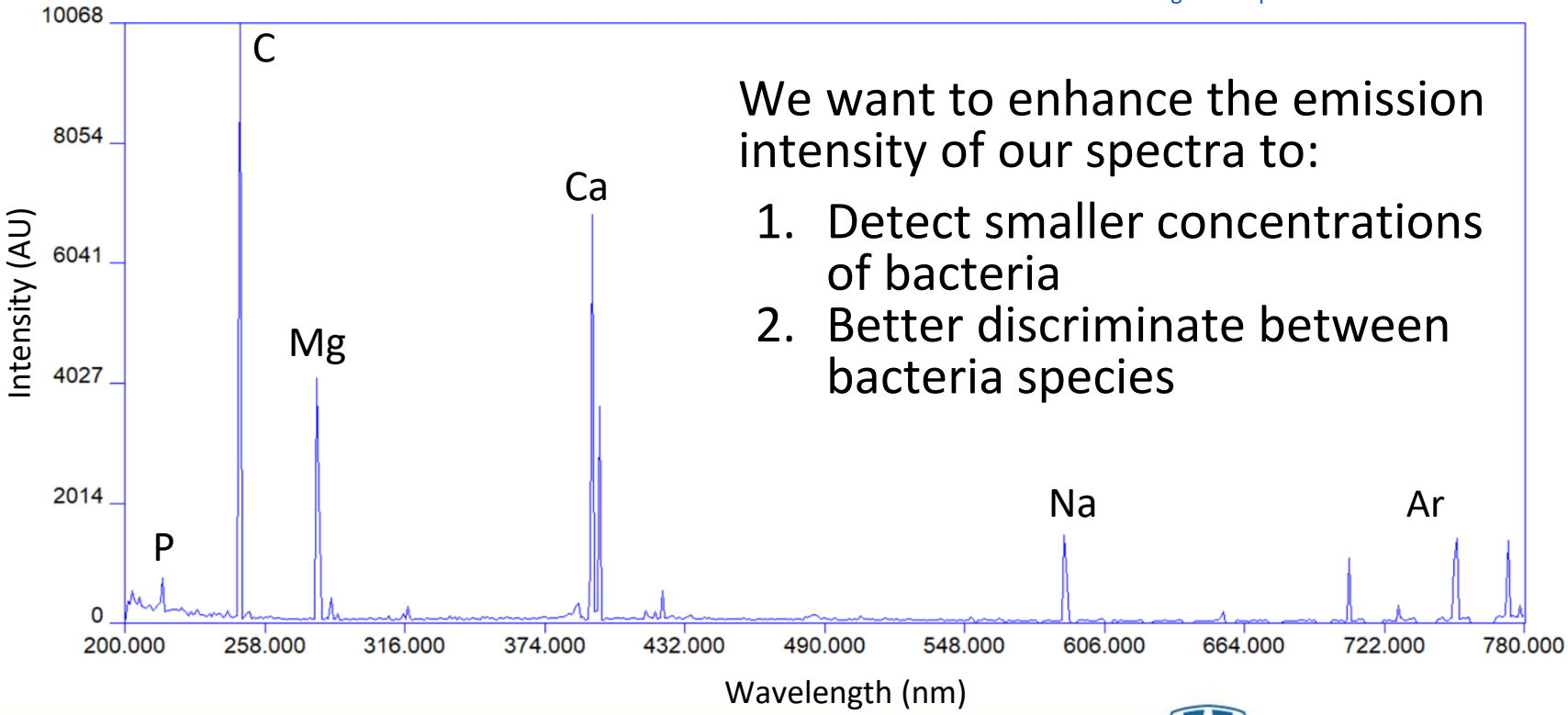
Bacteria deposited on nitrocellulose filter



Echelle spectrometer

Typical LIBS Bacteria Spectrum

E. coli
2 µs delay after plasma initiation
20 SCFH Argon environment
Single laser pulse



We want to enhance the emission intensity of our spectra to:

1. Detect smaller concentrations of bacteria
2. Better discriminate between bacteria species

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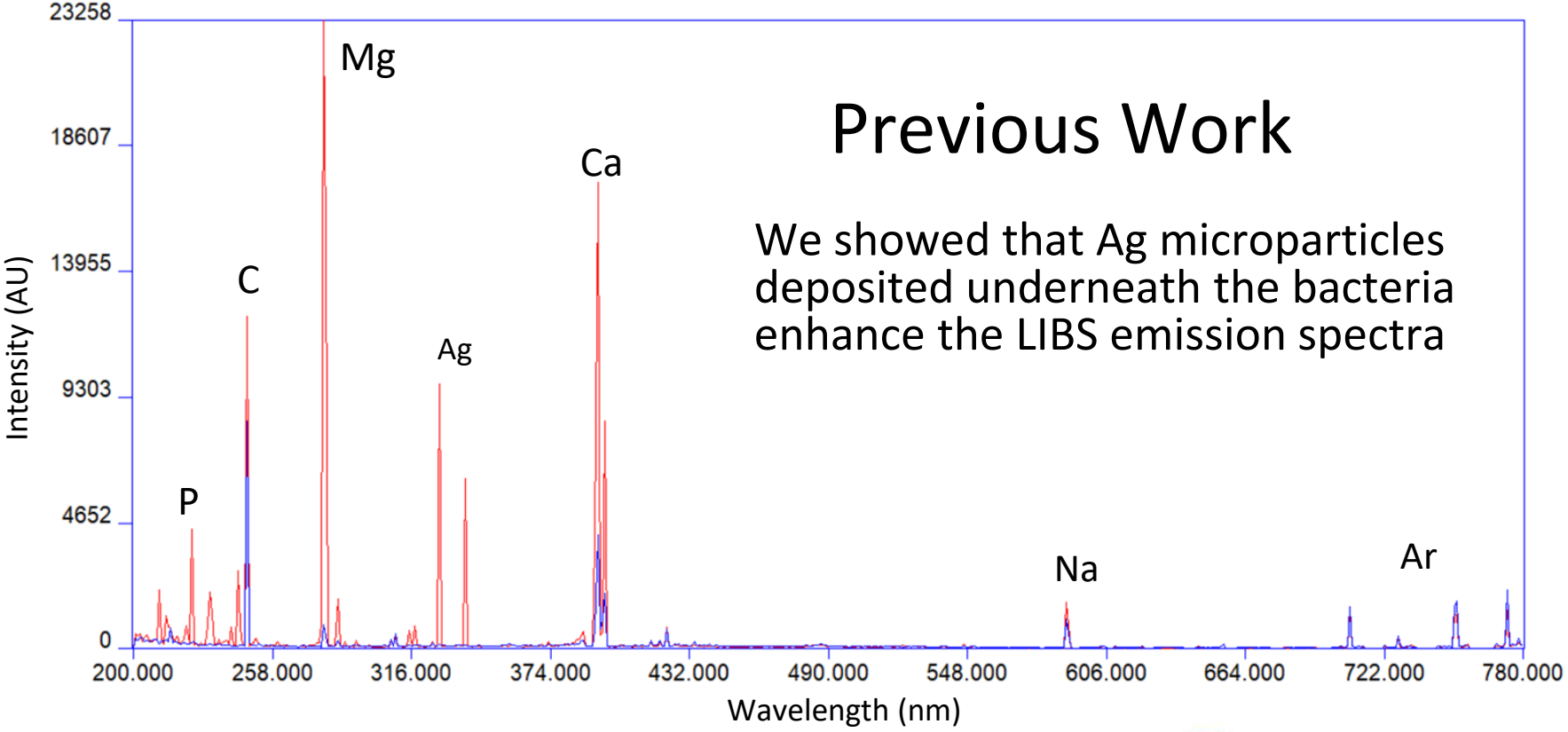
Red: with silver microparticles

Blue: without silver microparticles



Previous Work

We showed that Ag microparticles deposited underneath the bacteria enhance the LIBS emission spectra

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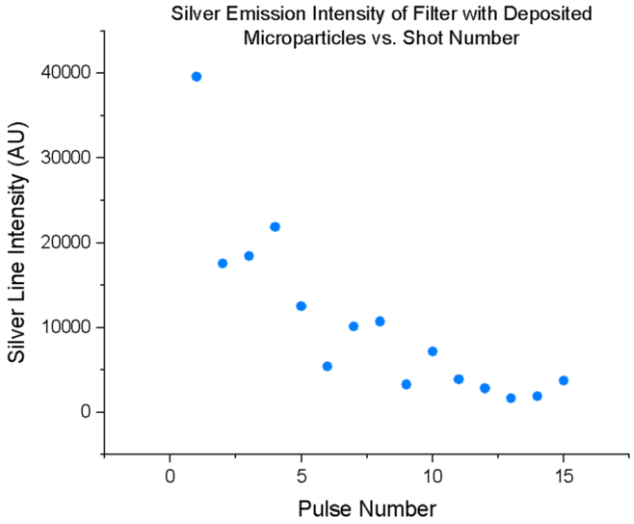
Complications:

The silver microparticles are being displaced by the plasma shockwave.

Why is this a problem?

During data collection, several pulses must be taken on the filter.

The intensity of silver decreases after each subsequent pulse and therefore the enhancement due to the silver decreases as well.



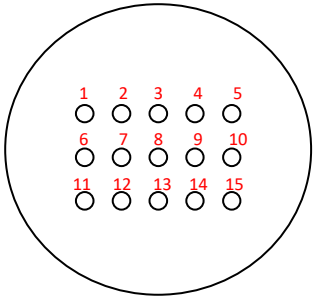


Illustration of 15 pulses on a filter

Proposed solution: Laser sputtering of a thin film

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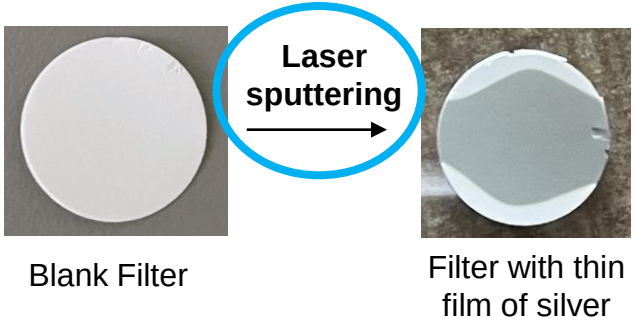
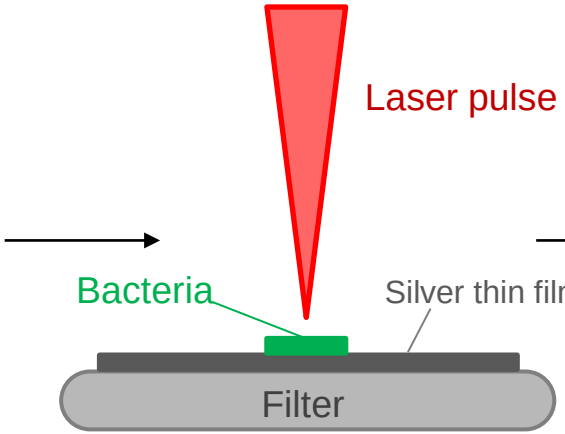
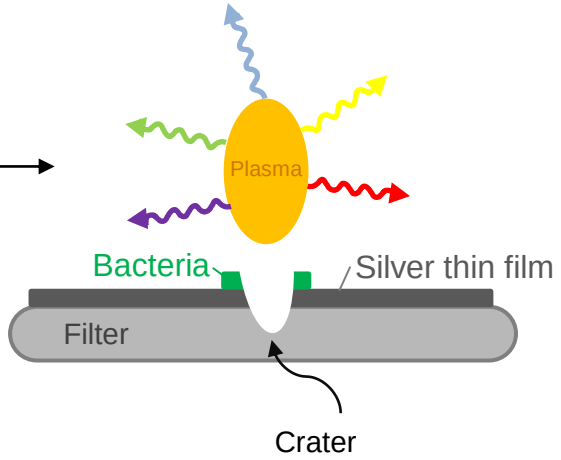
Method:



Blank Filter

Filter with thin film of silver





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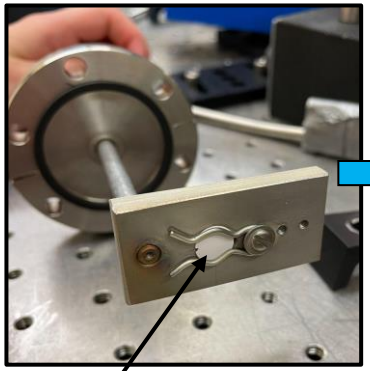
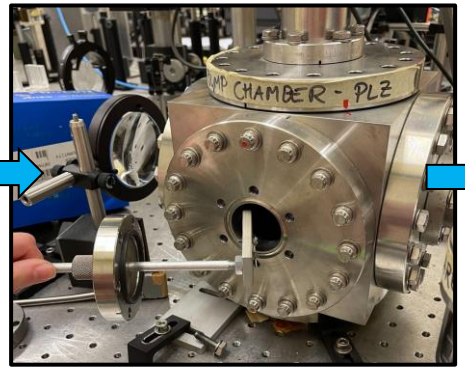
Pulsed Laser Sputtering

A 1064 nm pulsed laser (60 mJ per pulse) is focused onto a rotating silver target inside a vacuum chamber.

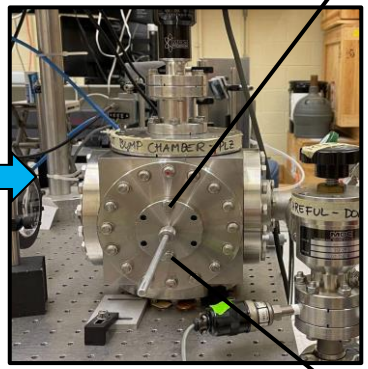
Experimental setup:



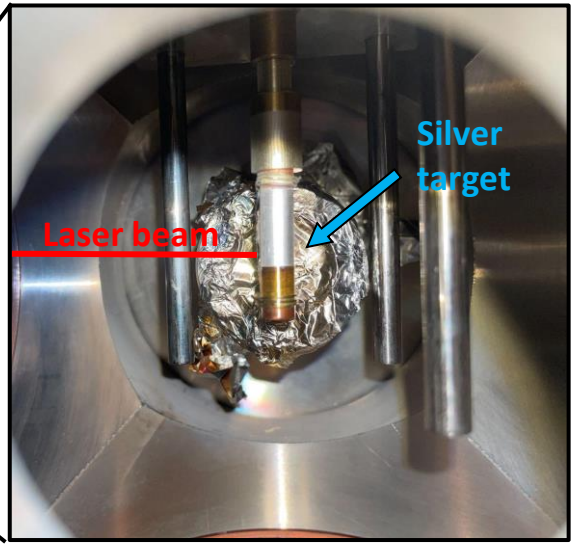
Filter on custom **aluminum filter holder**



Filter holder being inserted into **10 mTorr evacuated chamber**



Closed vacuum chamber apparatus



Inside the chamber

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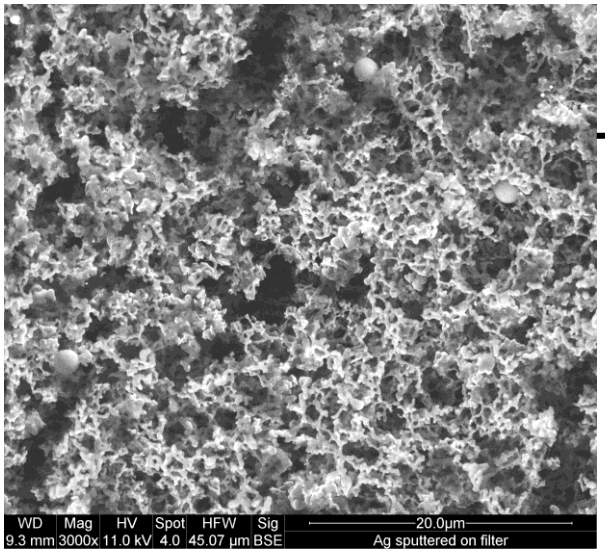
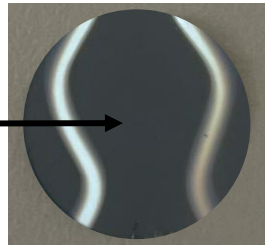
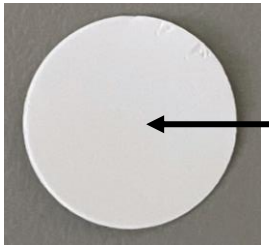
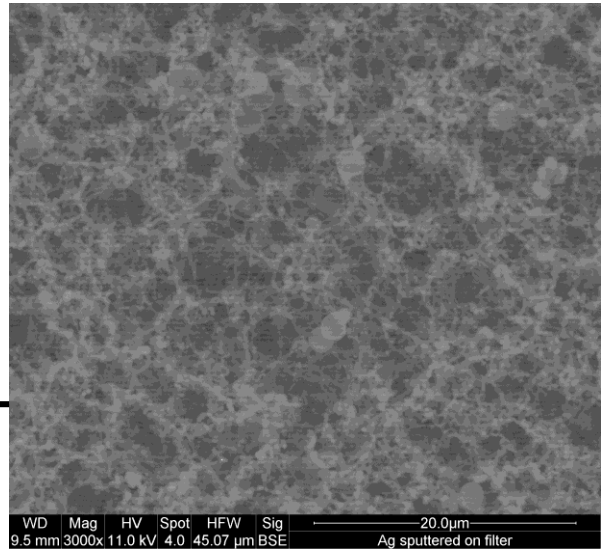
Results: Silver Film Analyzed with Scanning Electron Microscope (SEM)



Filter with 20 min Ag







Blank Filter

The structure of the filter is unchanged but it is clear that silver has coated the filter.

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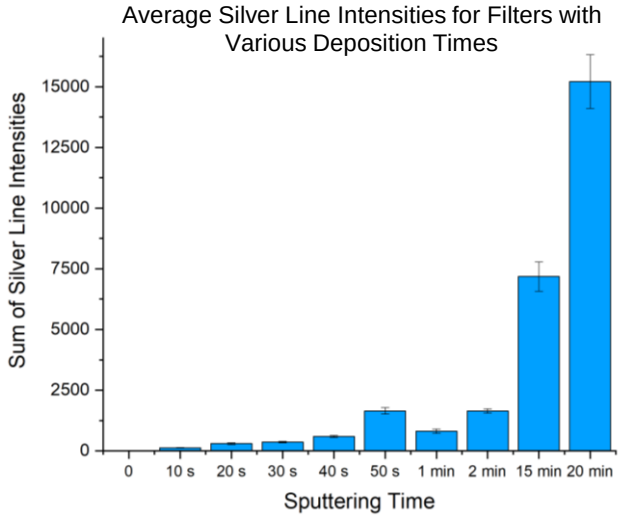
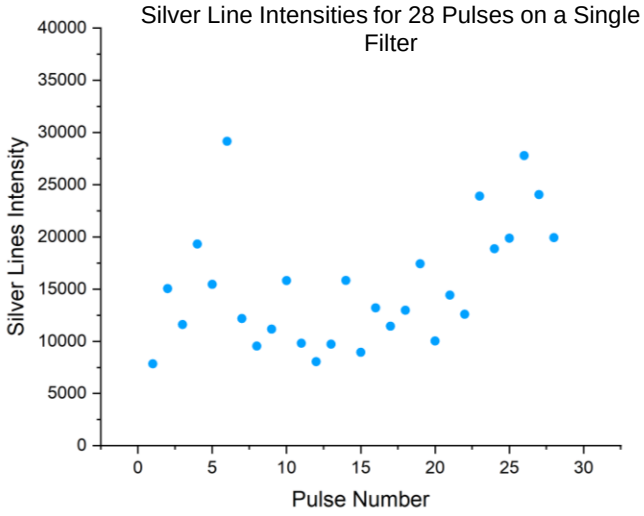
Results: Silver Film Analyzed with LIBS

Filters were coated with different amounts of silver by using different sputtering times. The filters were removed from the vacuum chamber and 10 single-shot LIBS spectra were acquired on each filter.





Silver line intensity increases with deposition time.

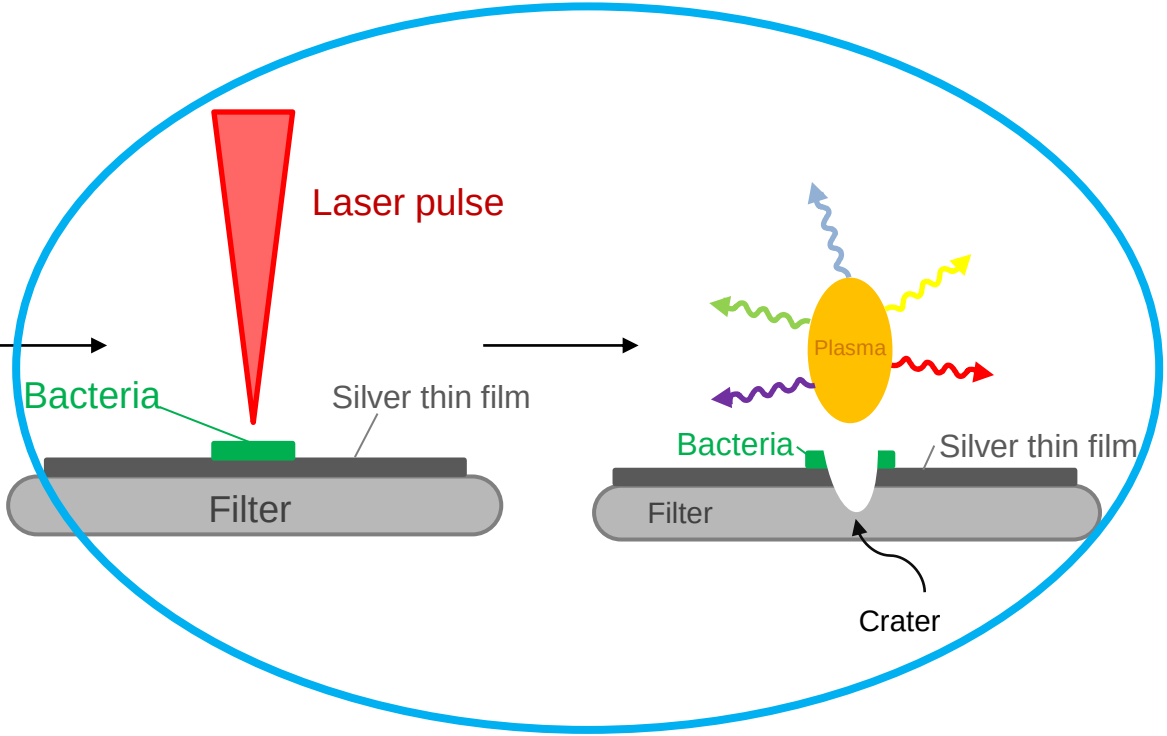
Silver line intensity shows no decrease with subsequent pulses.

Method:

Blank Filter

Laser sputtering

Filter with thin film of silver



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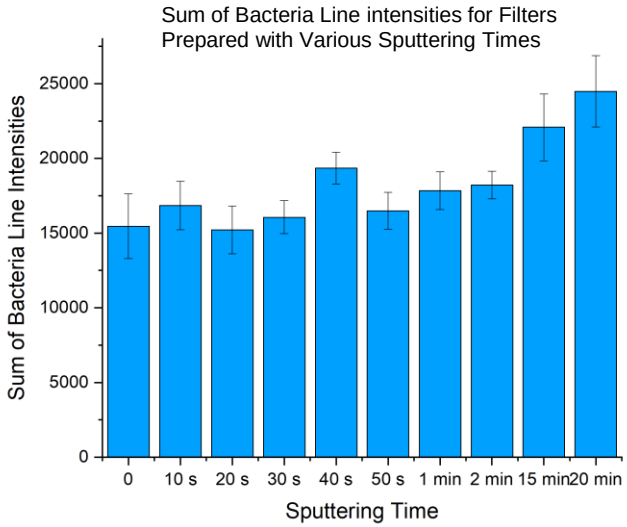
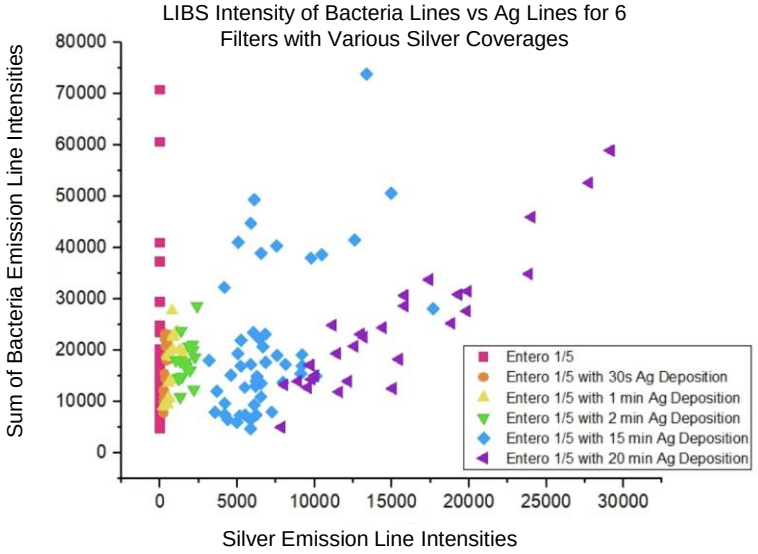
Results: Enhancement of Bacteria Emission Lines

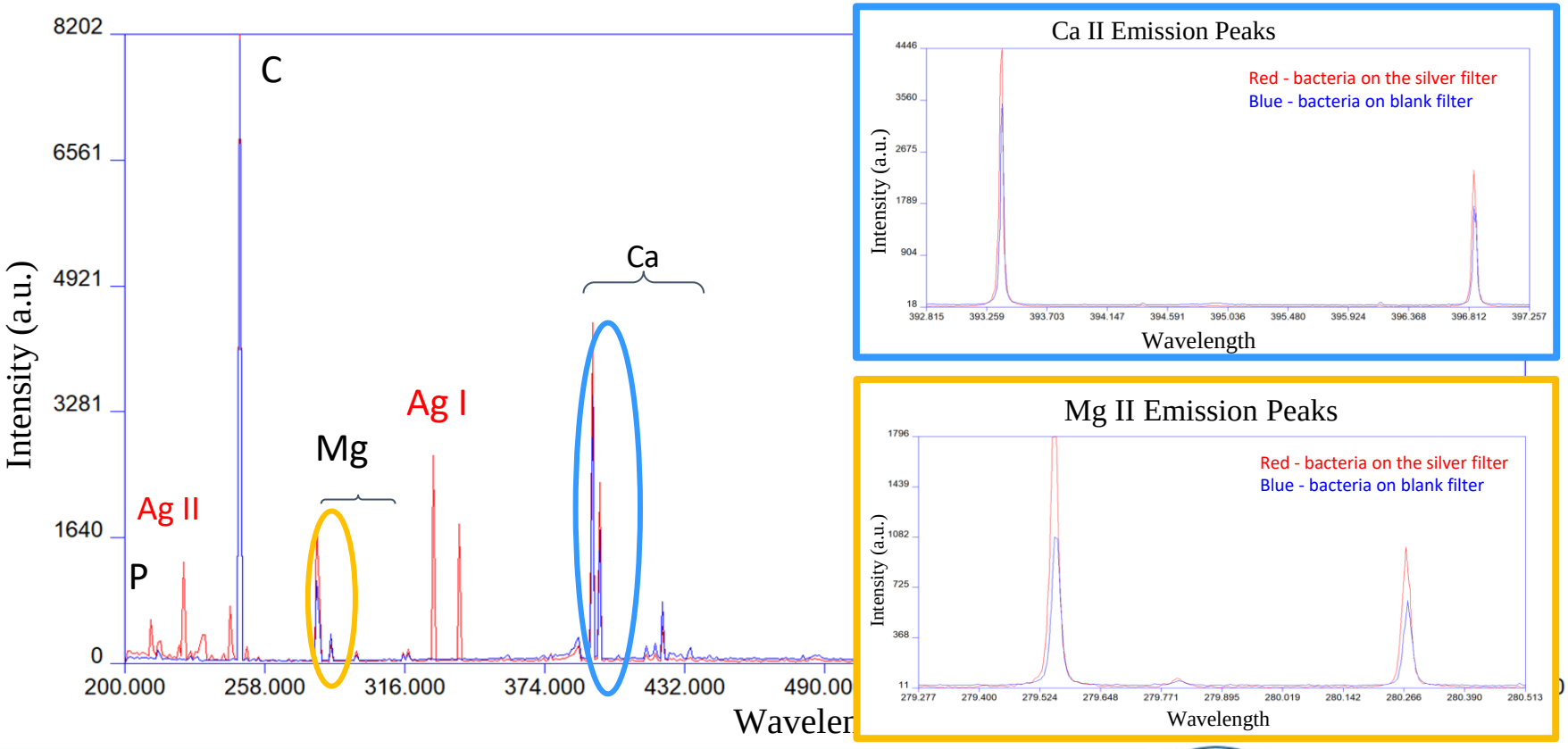
Filters prepared with various sputtering times were removed from the vacuum chamber, bacteria were deposited, and LIBS was performed on the depositions. 20-30 single-shot LIBS spectra were acquired from each filter.





Slight enhancement was observed for deposition times of 15 min or greater.

Results: Enhancement of Individual Peaks



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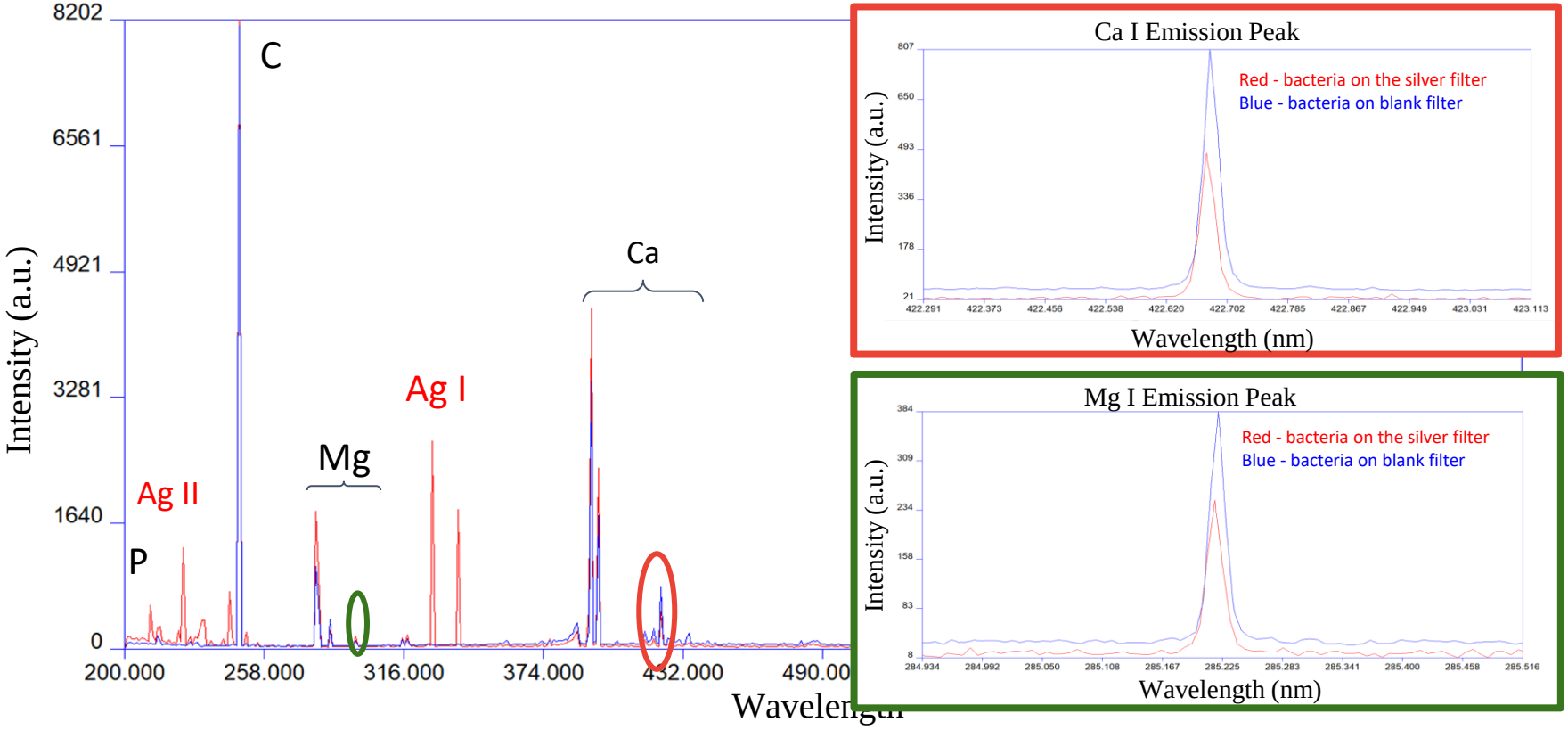
Results: Enhancement of Individual Peaks



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Plasma Temperature

The fraction of singly-ionized emission has increased indicating an increase in the temperature of the plasma.

The temperature increase was quantified using Saha-Boltzmann calculations:

$$\frac{I_{ion}}{I_{atom}} = 2\,\frac{(2\pi m_e kT)^{3/2}}{N_e h^3}\left(\frac{gA}{\lambda}\right)_{ion}\left(\frac{\lambda}{gA}\right)_{atom} e^{\frac{-(V^{+}+E_{ion}+E_{atom})}{kT_{ion}}}$$

Results	Calcium	Magnesium
No Silver Temperature	6010 K	7167 K
With Silver Temperature	6402 K	7602 K
Percent Change	+6.13%	+5.72

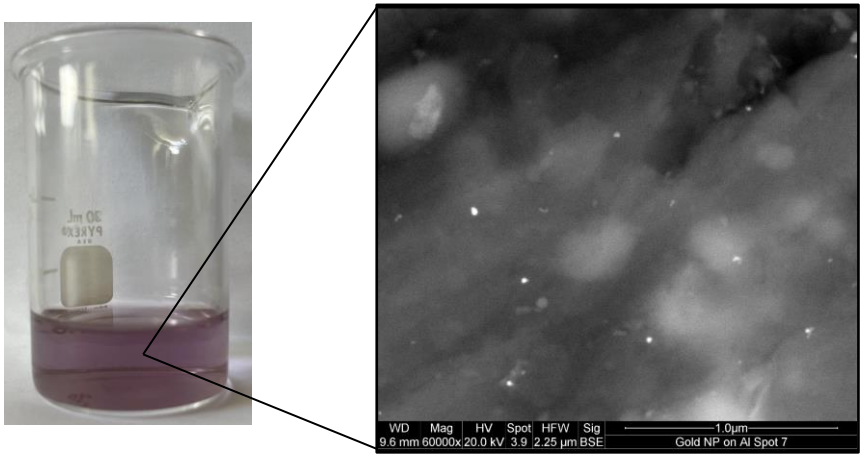
Average temperature increase of ~ 6%

Conclusions

- ➔ A uniform silver film was deposited as shown by SEM and LIBS that is not disrupted by subsequent laser pulses
- ➔ Slight bacterial enhancement was observed for deposition times 15 min or greater
- ➔ There was an increase in the fraction of singly-ionized emission
- ➔ A temperature increase of 6% was observed for deposition times of 15 min and 20 min

Future Work

- ➔ Use nano-particle enhanced LIBS (NELIBS) to increase the emission intensity of our bacteria
- ➔ We have successfully produced a nanoparticle suspension using pulsed laser ablation in liquid (PLAL)



Au Nanoparticle suspension

Next Steps:

Investigate various parameters' (laser pulse energy, volume of liquid, sputtering time) effect on size and spacing of the nanoparticles to observe and optimize enhancement.

Acknowledgements

- Advisor: Dr. Steven Rehse
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  - Grace Johnson
  - George Kobrossy
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  - NSERC USRA
  - University of Windsor Outstanding Scholars Program

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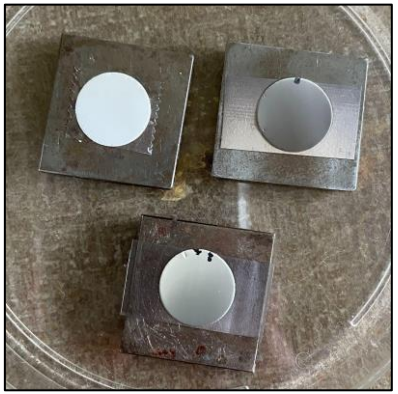
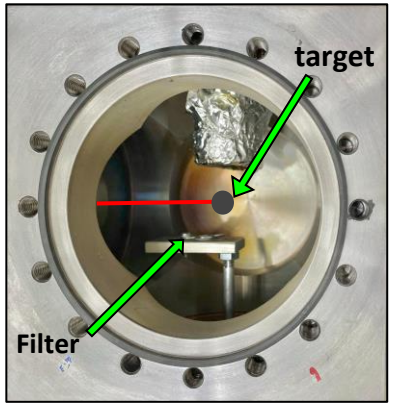
Determining Filter Location and Deposition Time

Filter location:

Brass rod



Brass coverage in various locations



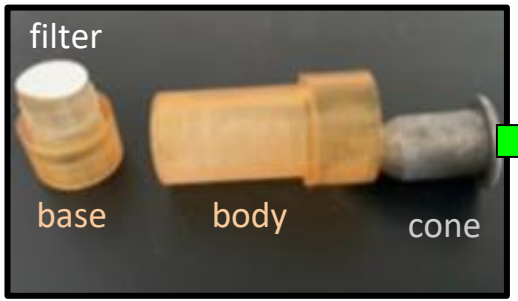
Top view of chamber

2 min deposition of silver

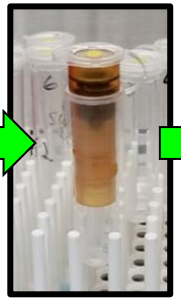
Deposition time:

- 2 min, 4 min, and 8 min sputtering times were investigated
  - ↪ Appeared to be too long, under 1 min would be investigated further with silver

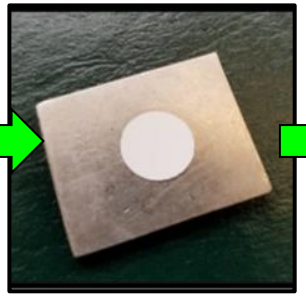
Bacterial Deposition



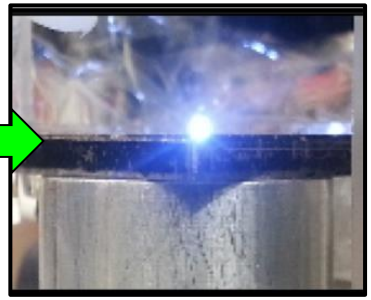
Centrifuge insert piece



Centrifugation



Sample mounted on steel piece



Sample being irradiated by laser pulse

Electron Density from Stark–broadened hydrogen line:

$$N_e = C(N_e, T_e)(\Delta\lambda_{FWHM})^{3/2}$$