SEM Sample Prep 101

Elizabeth King

Nathan La Porte

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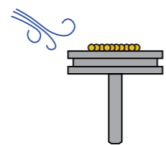*EXPLORING INNER SPACE*

New Members of the EPIC-SEM Team

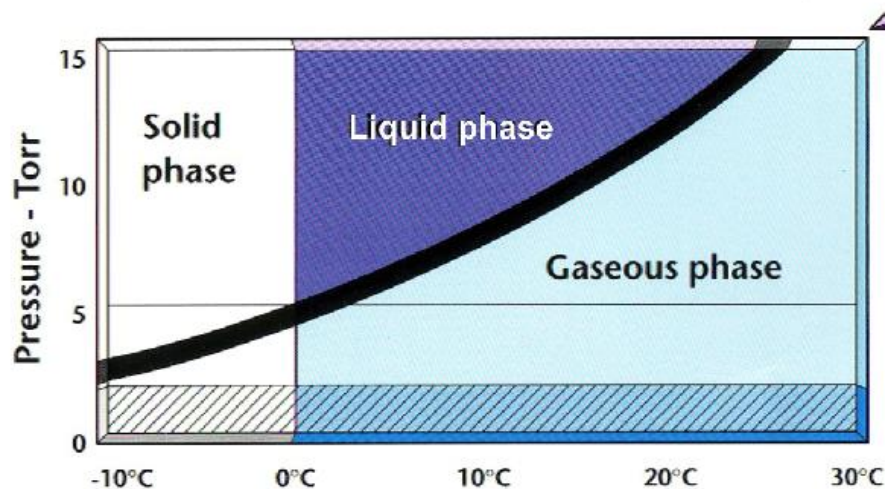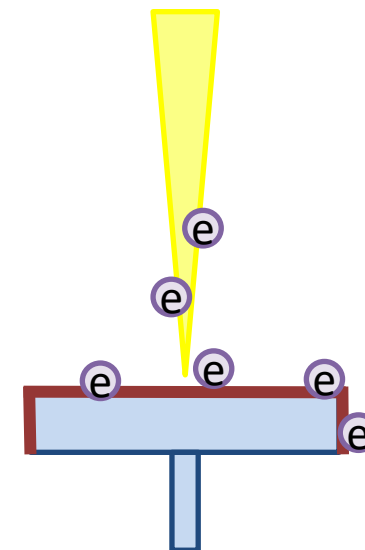**Elizabeth King**

Nathan La Porte
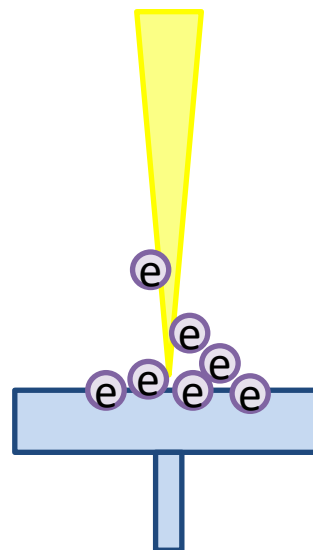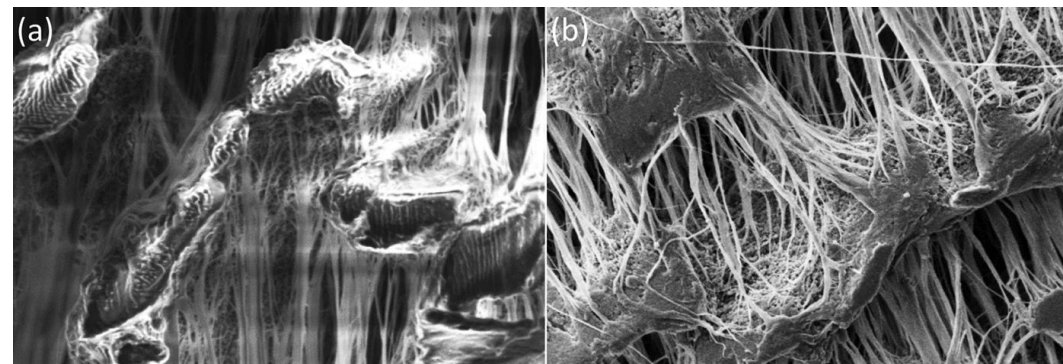
Nathaniel Kabat
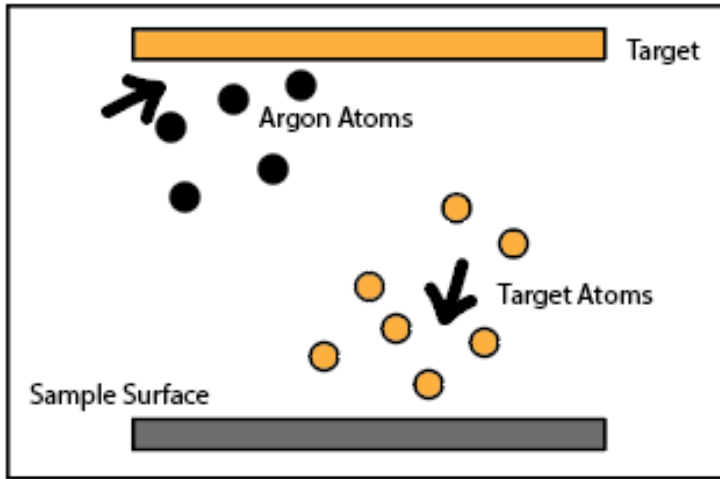
Sample Mounting
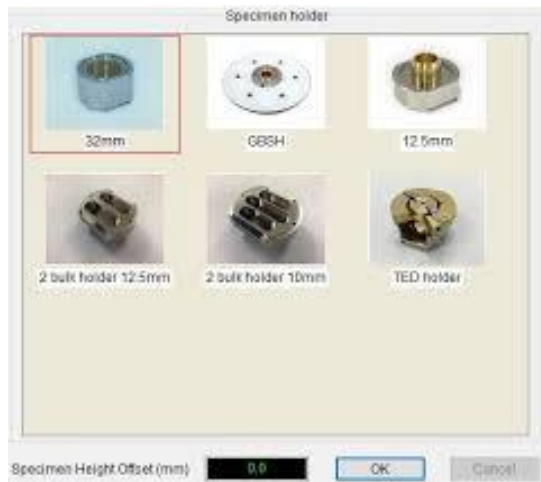
Things to Consider:
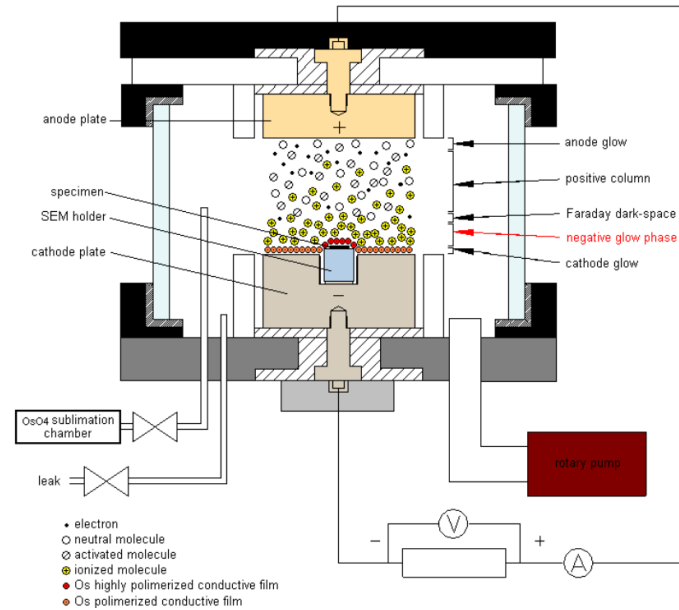
Vacuum

Sample Conductivity

Coating Samples for SEM Imaging
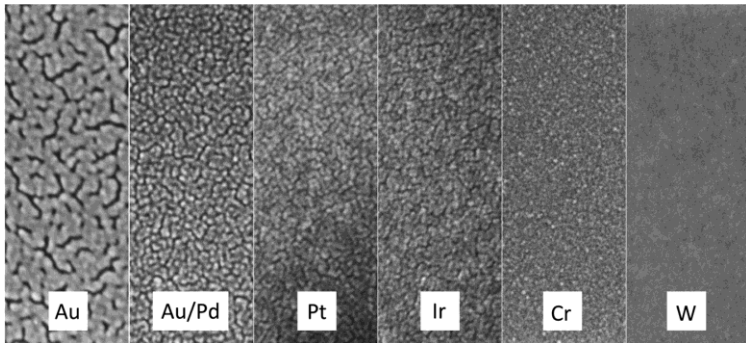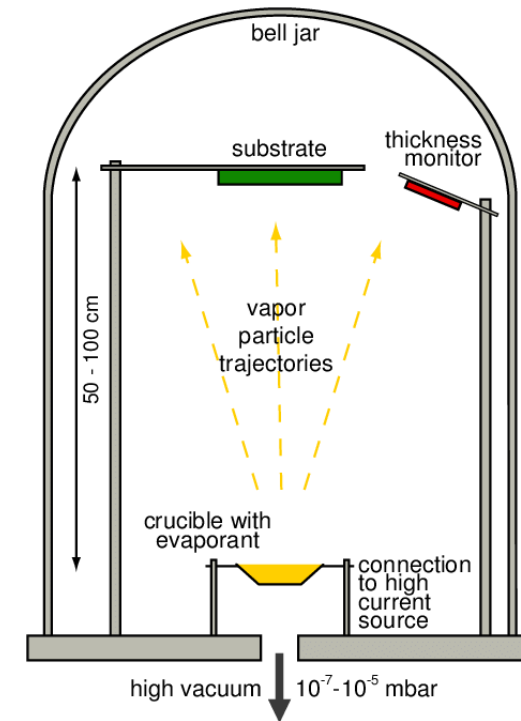
Sputter Coating

Osmium Coating

Thermal Evaporation

140nm

Selection of Materials

- **Gold**
- **Gold/Palladium**
- Silver
- **Platinum**

- Platinum/Palladium
- Iridium
- Chromium
- Tungsten

- Tantalum
- Palladium
- Nickel
- Copper

- Titanium
- **Osmium**
- **Carbon**

Material	Au	Au/Pd	Pt	C	Os
Grain size	5-10 nm	4-8 nm	2-3 nm	Amorphous	Amorphous
Low/Med Mag/Res	x	x	x	x	x
High Mag/Res			x	x	x
SE Signal Boost	x	x	x		
BSE Image				x	
EBSD(2-3nm)	x		x	x	
EDS				x	
Optically Transparent					x
Easily Removed				x	
Notes			Slow	Messy	Toxic!

Sample Contamination

Sources of contamination:
- Naturally occurring hydrocarbon contamination
- Sample outgassing
- Gases in the sample chamber
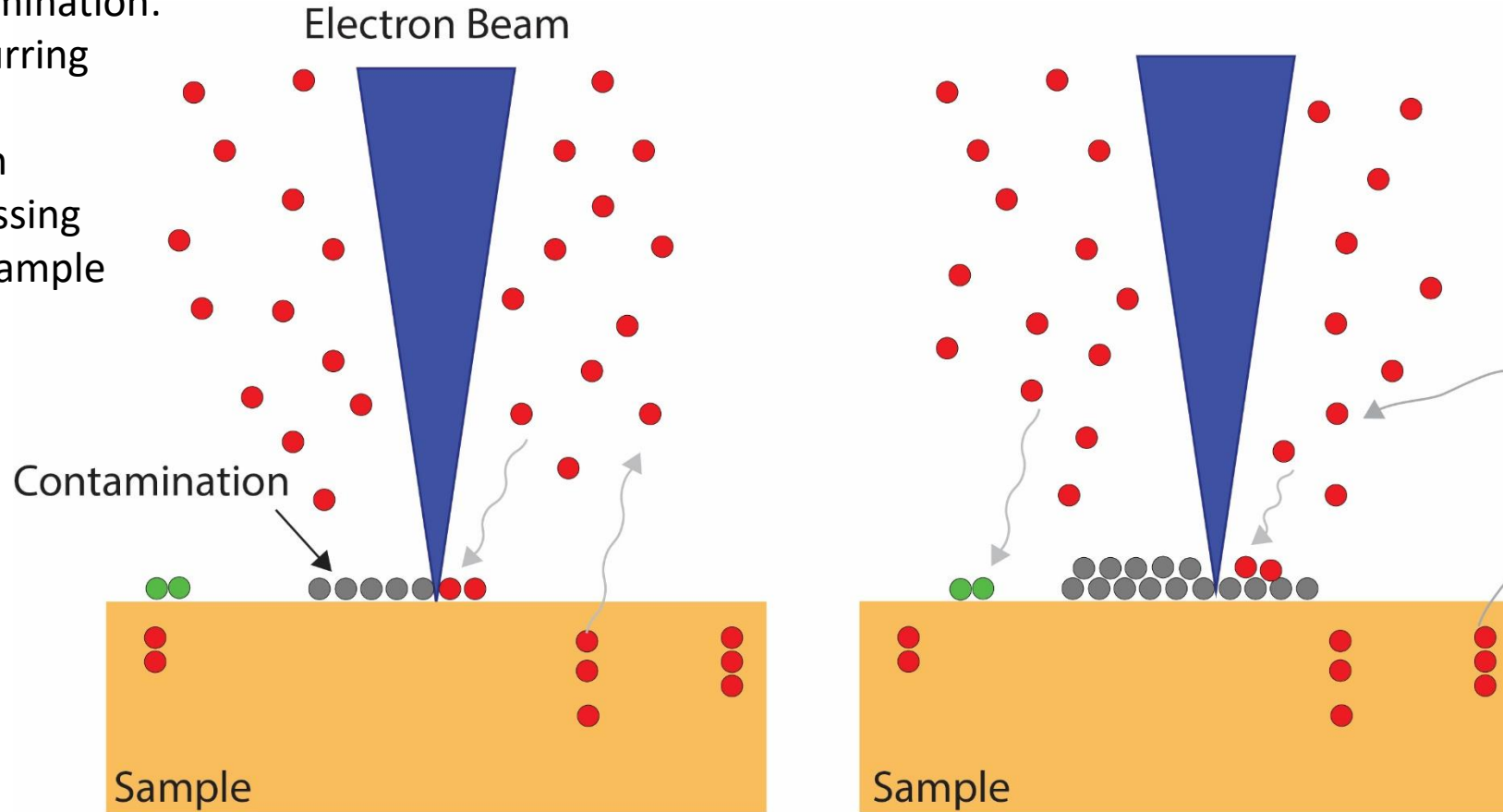
Basic Process: Hydrocarbons present around the sample are irradiated by electron beam

This process forms a contamination layer on the surface of the sample
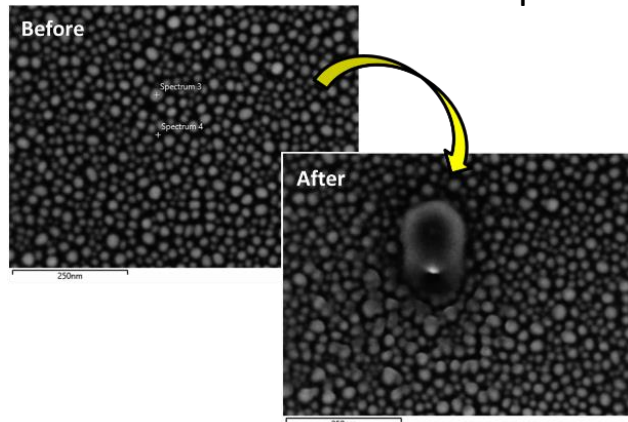
Sample Contamination: What does it look like?

Electron Beam

Contamination

Sample

Sample

ITO Film on Glass

SU8200 1.0kV-D 2.1mm x150k SE+BSE(U)   300nm

Contamination from Spot EDS

Before

After

250nm

250nm
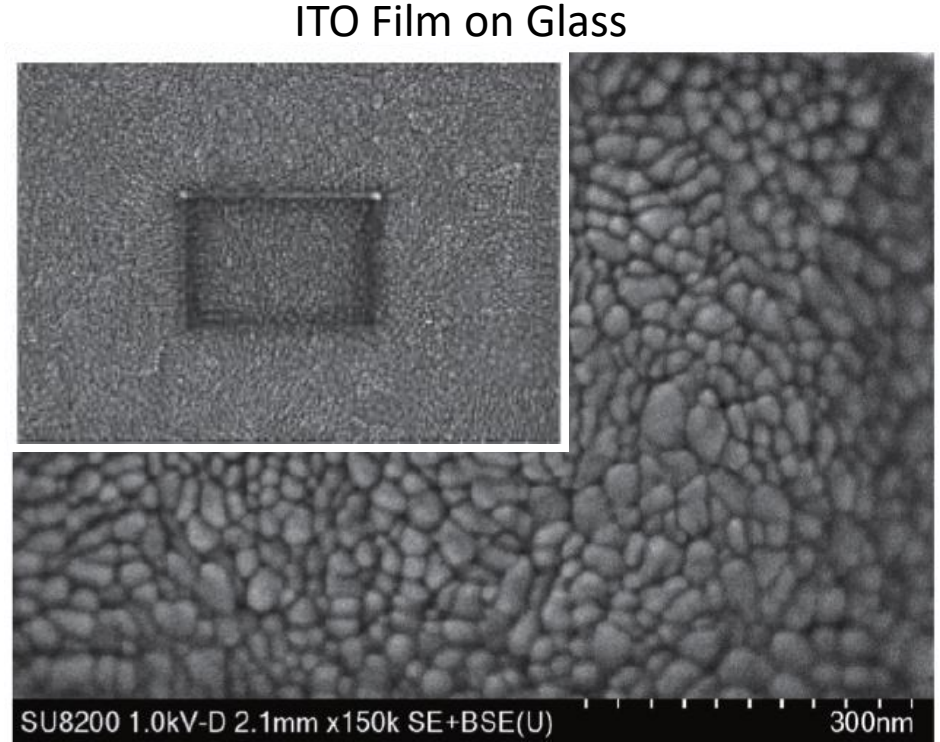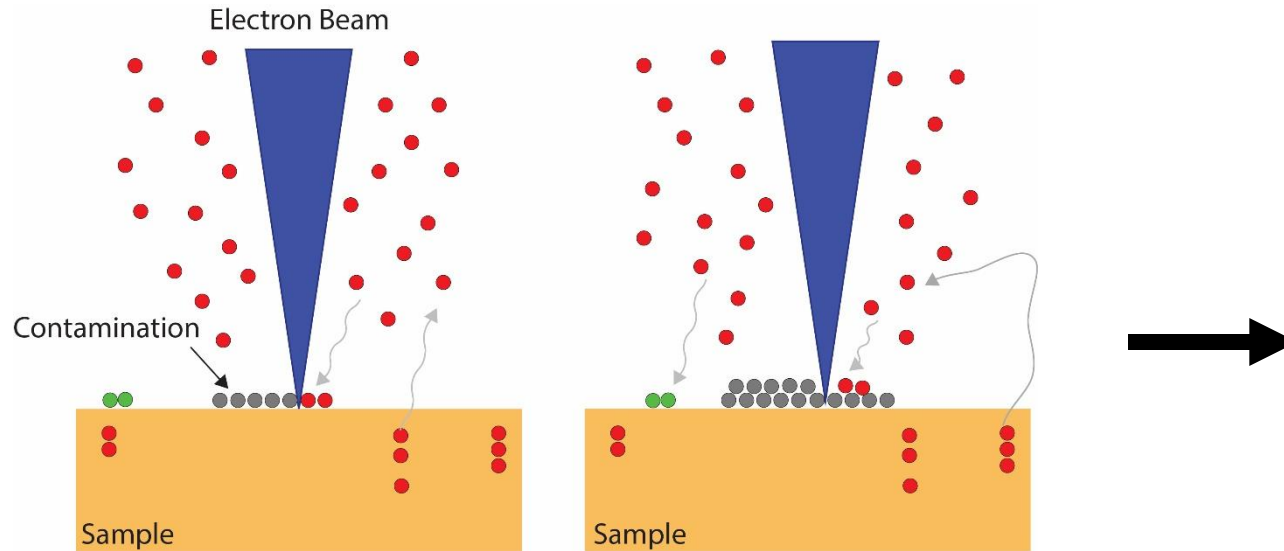
Patterned Silicon

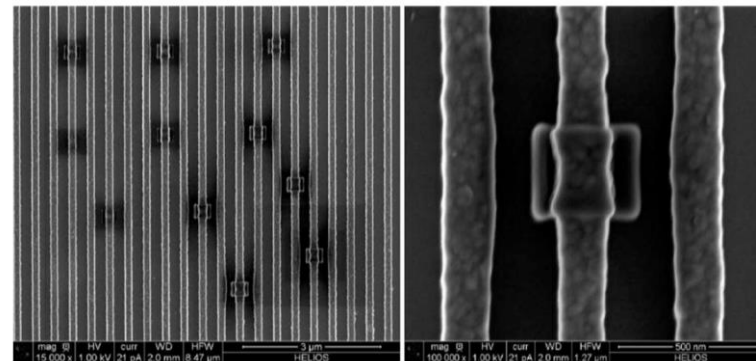- Images might appear fine initially
- If you lower the magnification, there are will be signs of contamination

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Sample Contamination: What to do about it?

While using an SEM:

- If seeing signs of contamination, try different mitigation strategies:
  - Different area to focus and save image
  - Use faster scan speed to limit carbon build up
- Use a cold trap to help collect contamination in the chamber (only available on the SU8030 SEM)
- In-situ plasma cleaning

Preventative:

- Store your samples in a desiccator or glovebox (vacuum, gas purged)
- Clean your samples immediately prior to SEM imaging
  - Plasma Cleaning
  - UV Cleaning
  - Other methods (not covered): Ion milling, polishing, heating

Soong, C., Woo, P., & Hoyle, D. (2012). Contamination Cleaning of TEM/SEM Samples with the ZONE Cleaner. *Microscopy Today, 20*(6), 44-48.
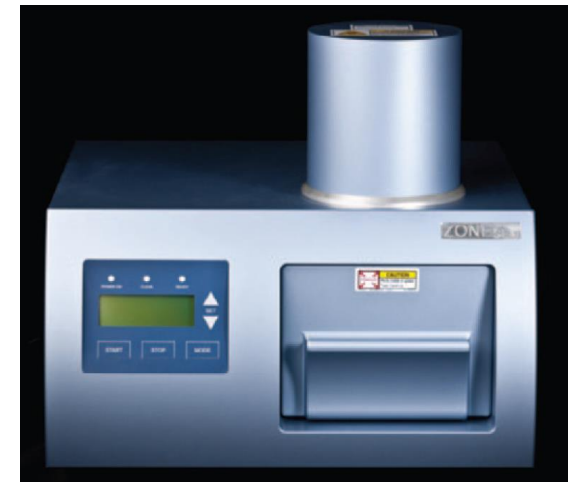
Sample Cleaning

Plasma cleaning (RF)

- Can use Argon, Oxygen, or a mixture
- Argon uses a physical sputtering process to clean surface
- Oxygen uses a chemical process and reacts with hydrocarbon
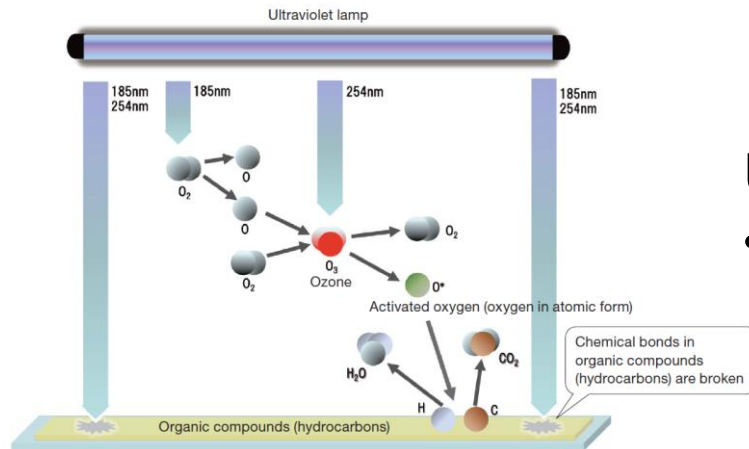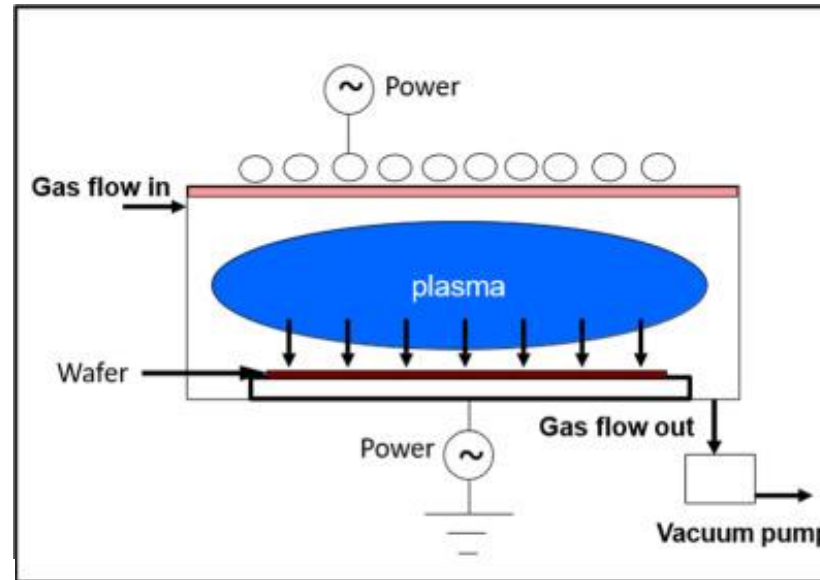
UV Cleaning
- UV light breaks up oxygen gas providing a similar cleaning process

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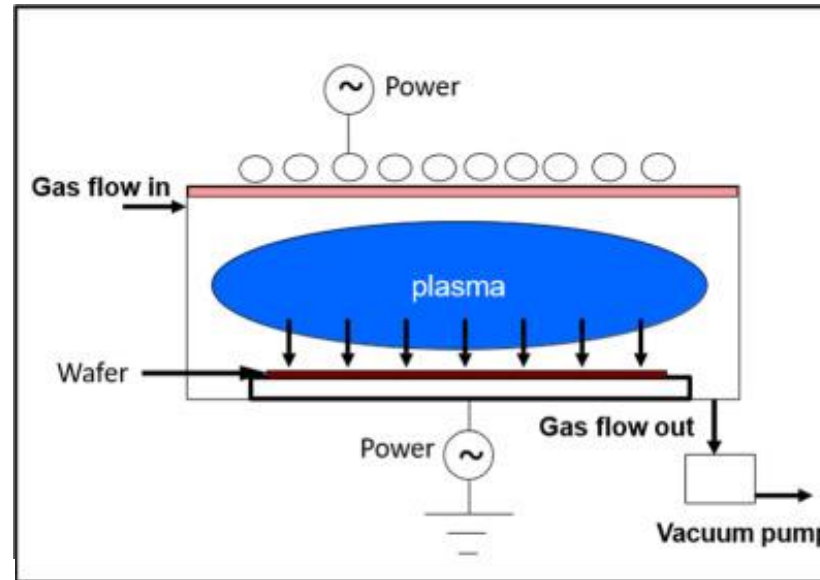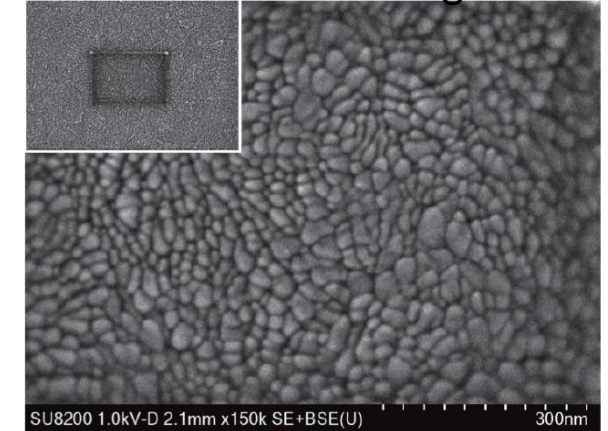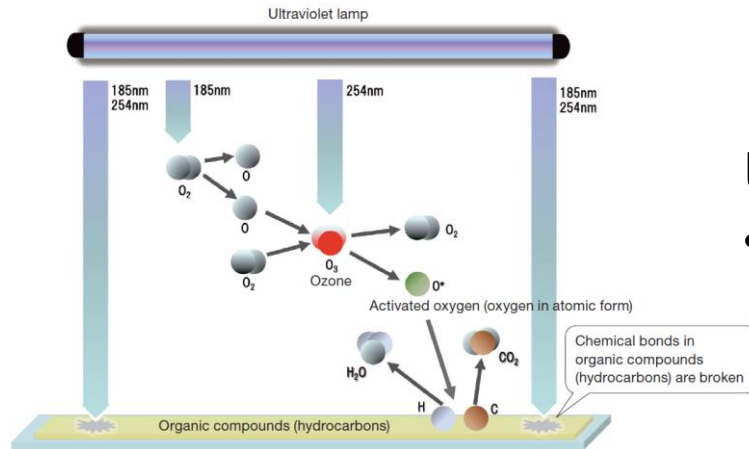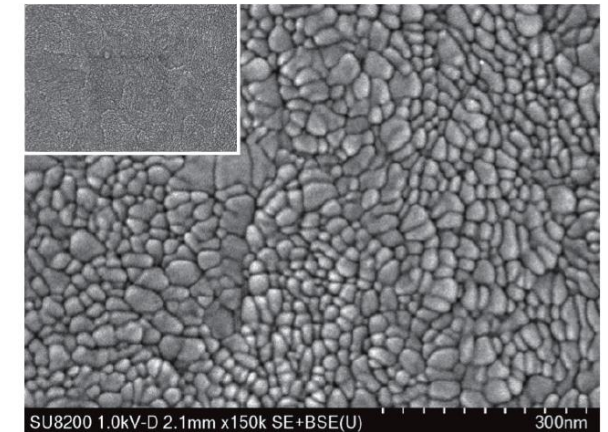- Oxygen uses a chemical process and reacts with hydrocarbon

Before Cleaning

After Cleaning

UV Cleaning

- UV light breaks up oxygen gas providing a similar cleaning process

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Questions?