SEM: Low Vacuum vs High Vacuum Imaging

A webinar for all SEM users.

Introduction

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Objectives

- Introduce the SEM and SEM sample prep
- Demonstrate the use cases for Low-Vacuum imaging
- Consider **time**, **cost**, and **analytical capability** tradeoffs

Agenda

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1. Why use SEM
2. How SEM works
3. Sample Preparation
4. Live Demo!
5. Wrap-up/Questions

Why use SEM?

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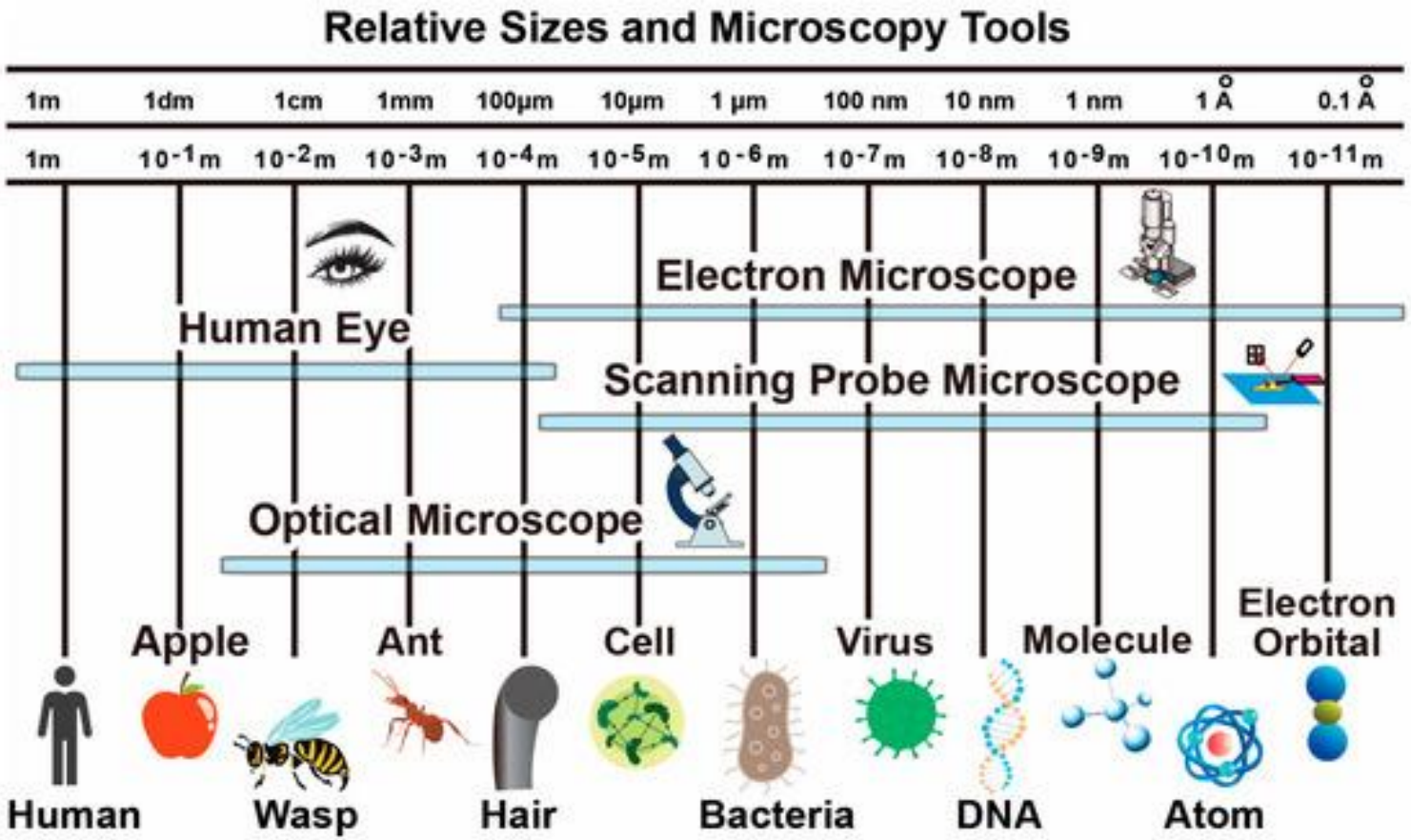

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Why Use SEM?

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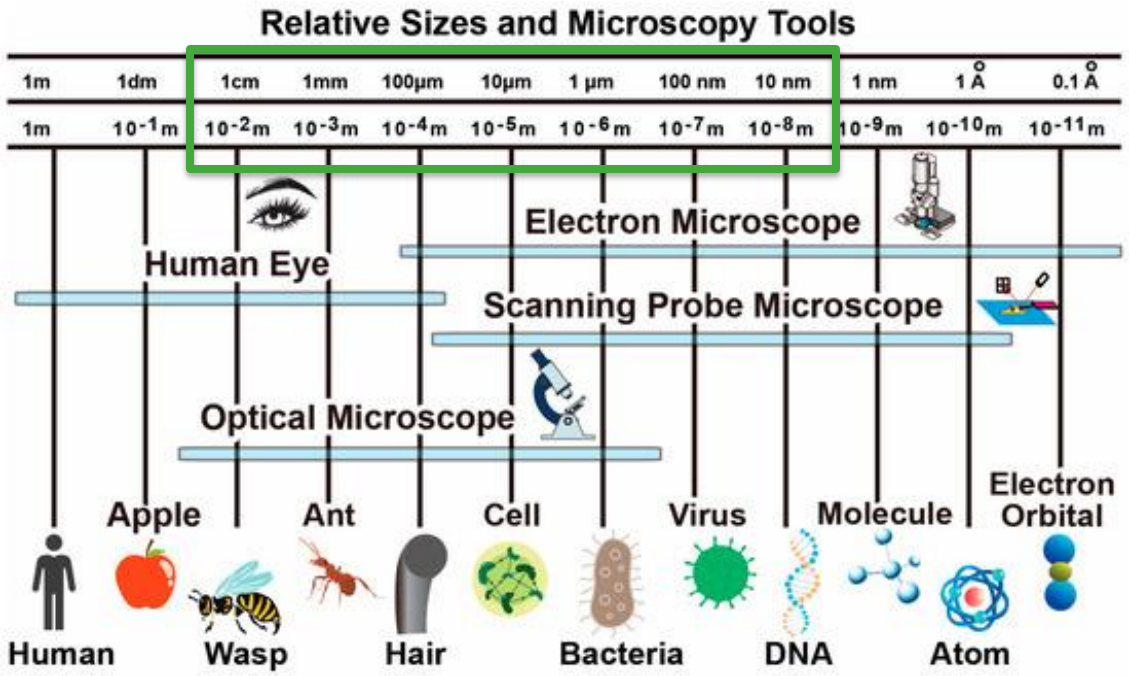

- High magnification and depth of field
- Microscale to nanoscale resolution
- Surface morphology
- Elemental analysis capability (EDS)

Who uses SEM?

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- Materials science, Biology, failure analysis (FA), quality assurance (QA)
- Education!

- SEM is a powerful technique, but it is more cost- and time-intensive than an optical microscope.

Why use SEM?

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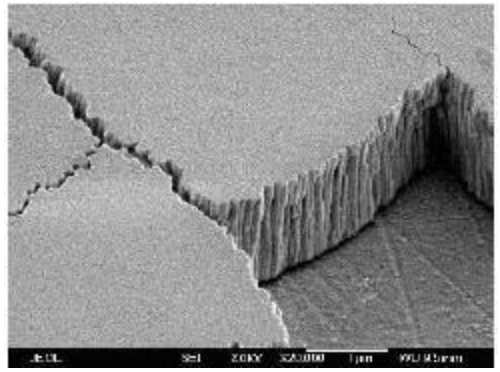
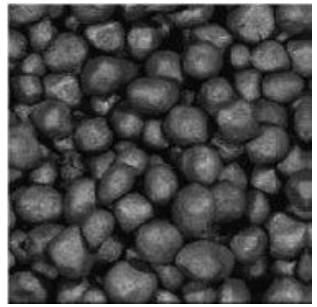
Topography

The surface features of an object or “how it looks”.
Scientists relate texture, shape, and structure to materials properties.



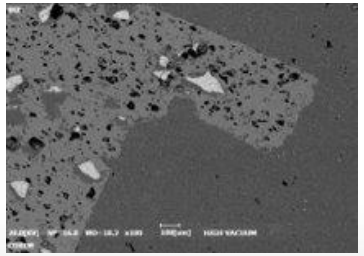
Morphology

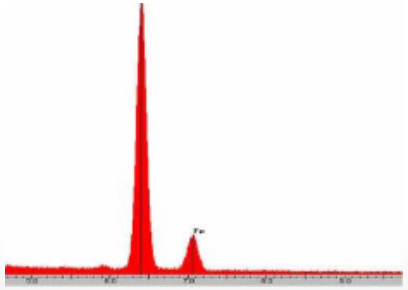
The shape and size of the particles making up the object.
Scientists relate texture, shape, and structure to materials properties.



Composition

The elements and compounds that the object is composed of and the relative amounts of them.
Direct relationship between composition and materials properties.





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How SEM Works – Basics

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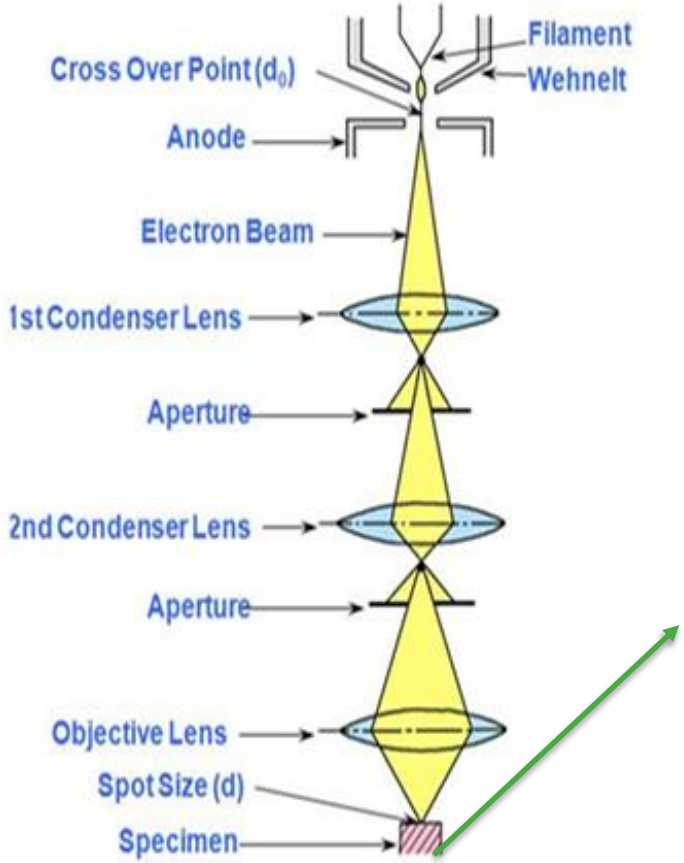
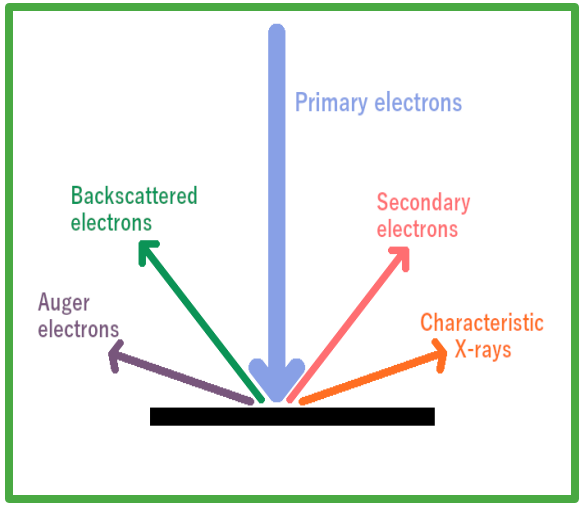
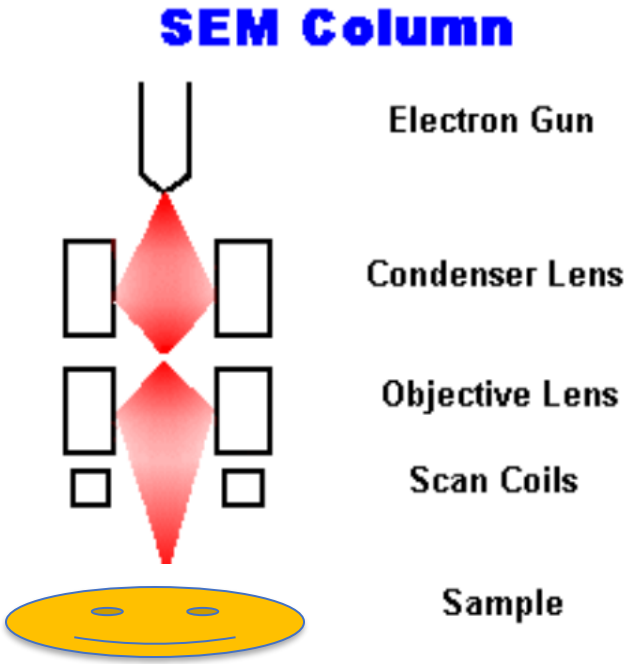
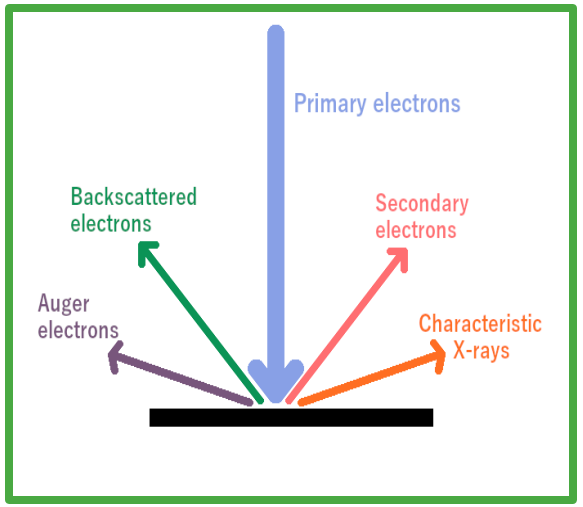
- Chamber under vacuum
- Electron gun generates focused electron beam
- Beam scans across sample surface
- Interaction Volume
- Electron interactions generate signals
- Detectors collect signals to form image
- Secondary Electrons (SE), Backscattered Electrons (BSE)





How SEM Works – Basics





How SEM Works – Gun and Vacuum

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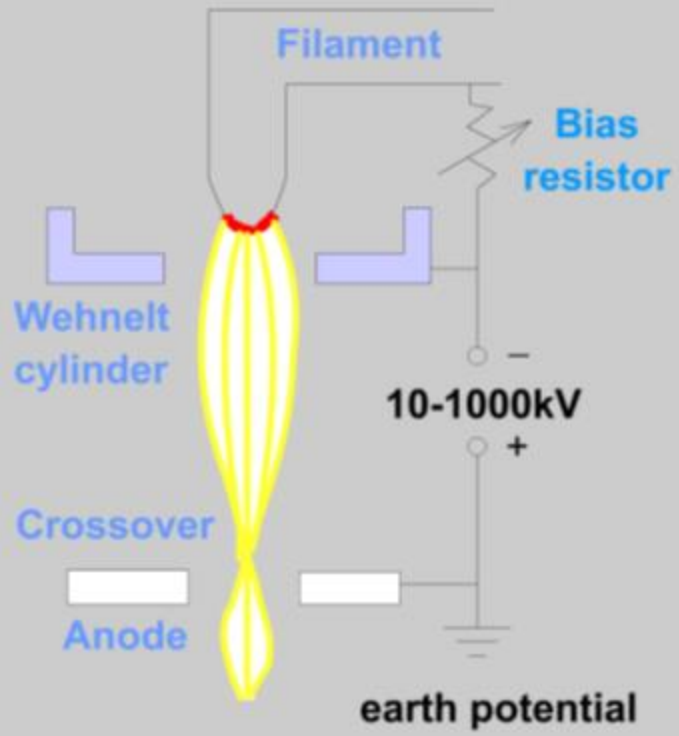

This is the type of electron gun used in most electron microscopes. It is robust, relatively cheap and does not require an ultra high vacuum.

In the thermionic electron gun, electrons are emitted from a heated filament and then accelerated towards an anode

A divergent beam of electrons emerges from the anode hole.

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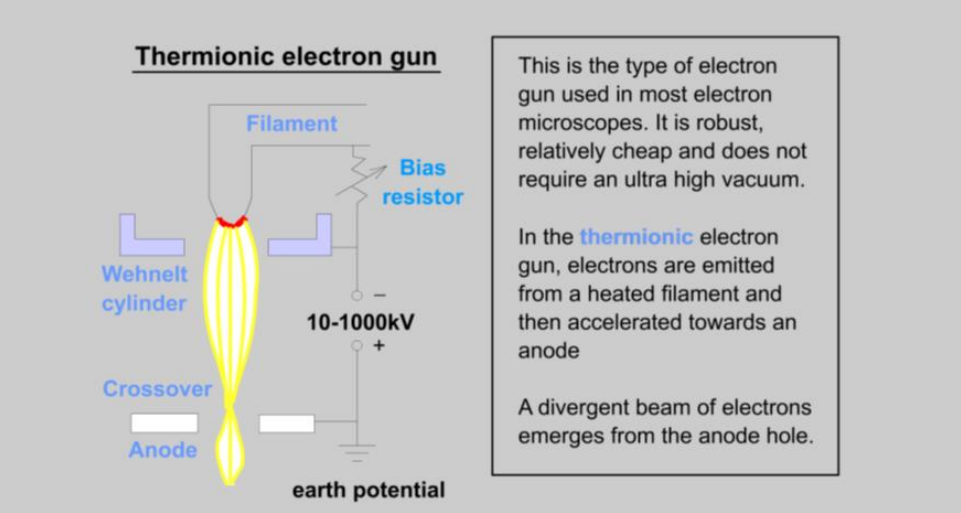
How SEM Works – Gun and Vacuum



Feature	High Vacuum (HV)	Low Vacuum (LV)
Chamber Pressure	10^-5 to 10^-7 Torr	10^-2 to 1 Torr
Sample Type	Conductive	Non-conductive allowed
Resolution	Highest	Moderate
Sample Prep	Often requires coating	Minimal coating

The electrons emitted from the filament are highly charged, and reactive to any material. They must be stabilized in a vacuum.

How SEM Works – Vacuum

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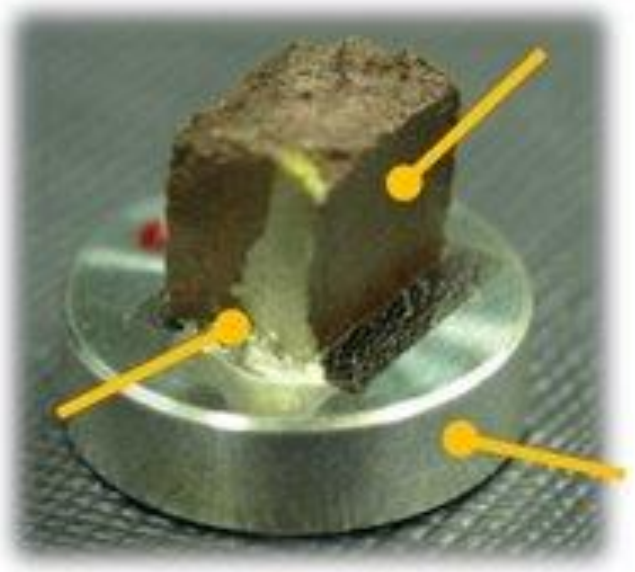
- **HV – High Vacuum**, most common: Maximum resolution, requires conductive samples
- **LV – Low Vacuum**: Reduced charging, minimal sample prep
- **Trade-off:** Resolution vs. sample flexibility

Sample Preparation – Mounting



Irregular, but conductive specimen on stub

Prepared cross section specimen on stub

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Sample Preparation – Ion Sputtering

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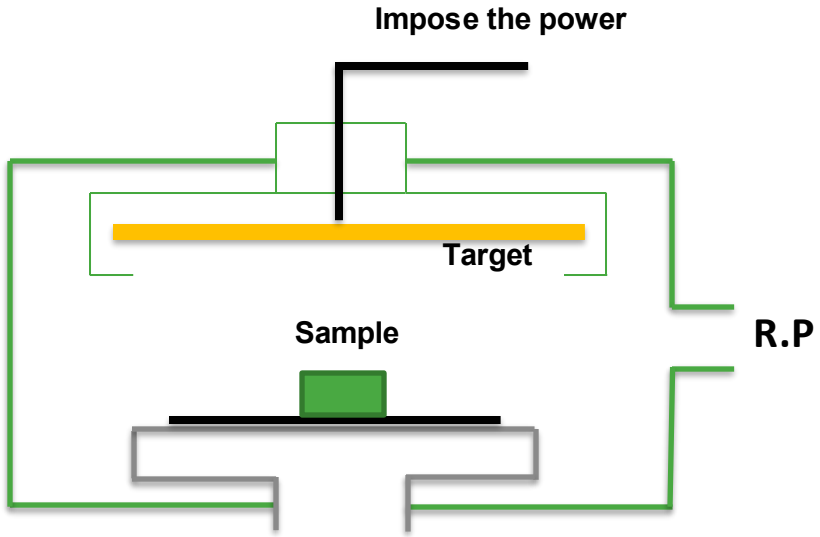

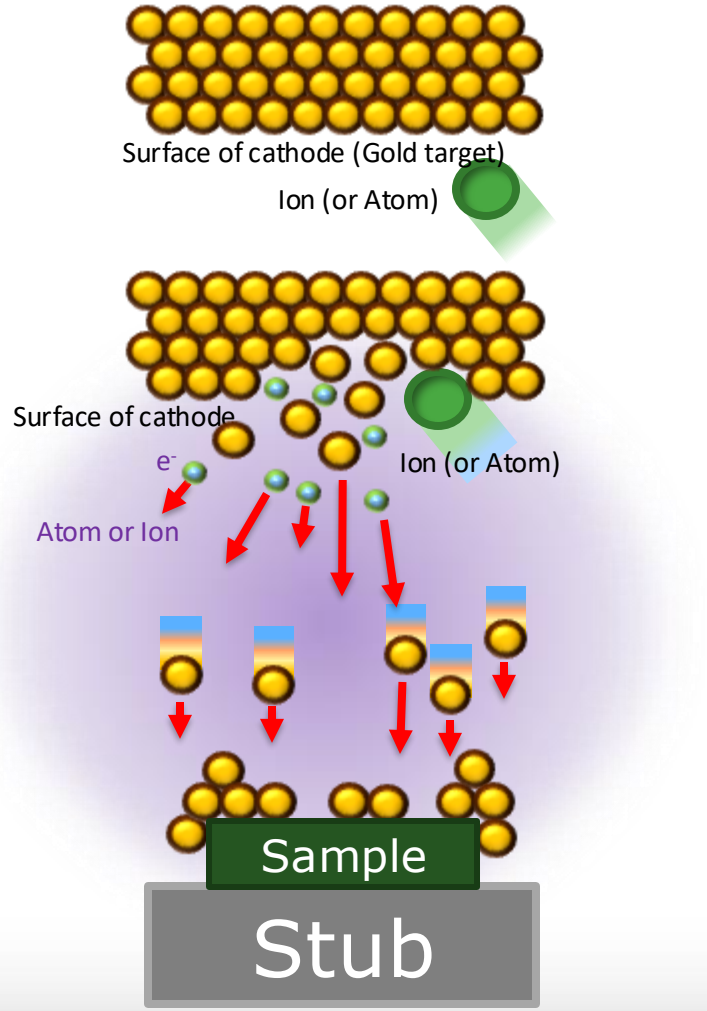

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Sample Preparation – Ion Sputtering

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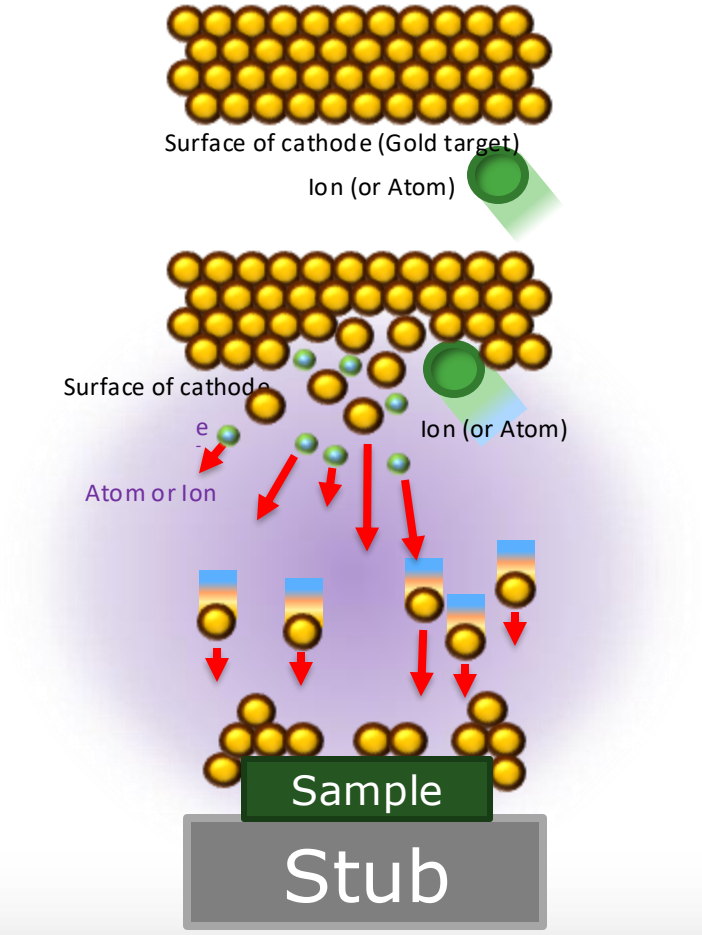
- Sputtering in gold, platinum, or other elements will deposit a nanometer-thin coating on the surface of the sample, improving conductivity, and therefore improving resolution.
- Sputtering requires a special machine, and uses consumable targets made of the material of choice.
- Pros: Best resolution possible
- Cons: Requires sputter coater, extra process time.



Sample Preparation - Considerations

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- Non-conductive materials charge under electron beam
- Conventional High Vacuum requires conductive coating
- Ion sputter coating or Critical point dryer
- Biological / wet samples **impossible** in HV

Sample Preparation – Typical Process

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Sectioning → Mounting → Grinding and Polishing → Coating → Imaging

Non-conductive

Sample Preparation – Low Vac

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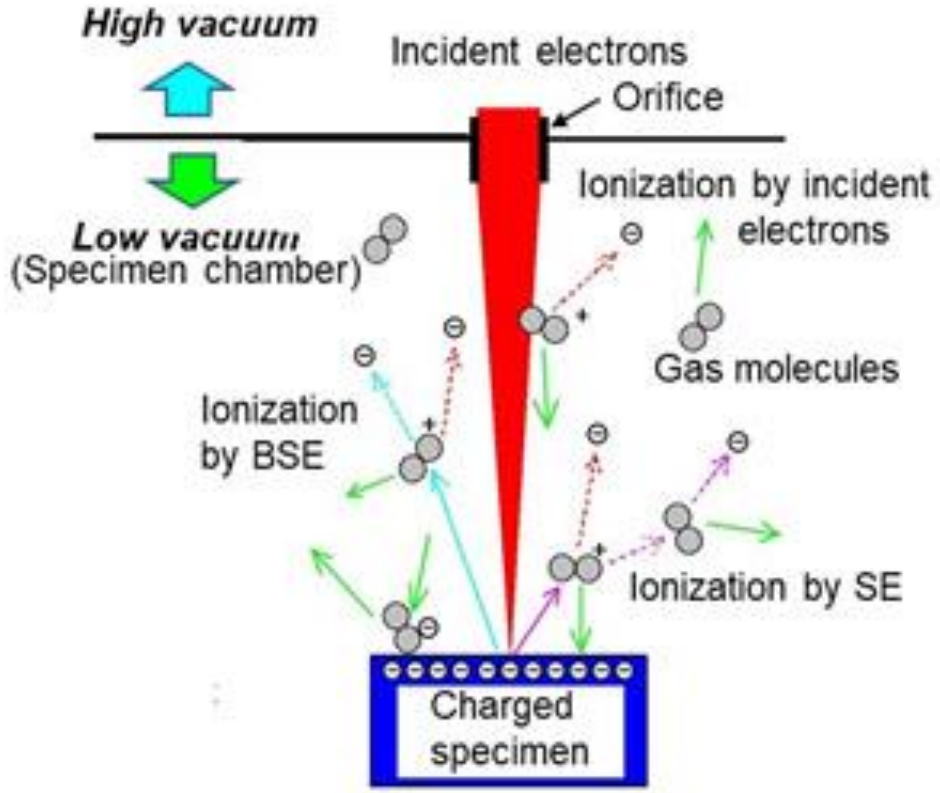

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Sample Preparation – Low Vac

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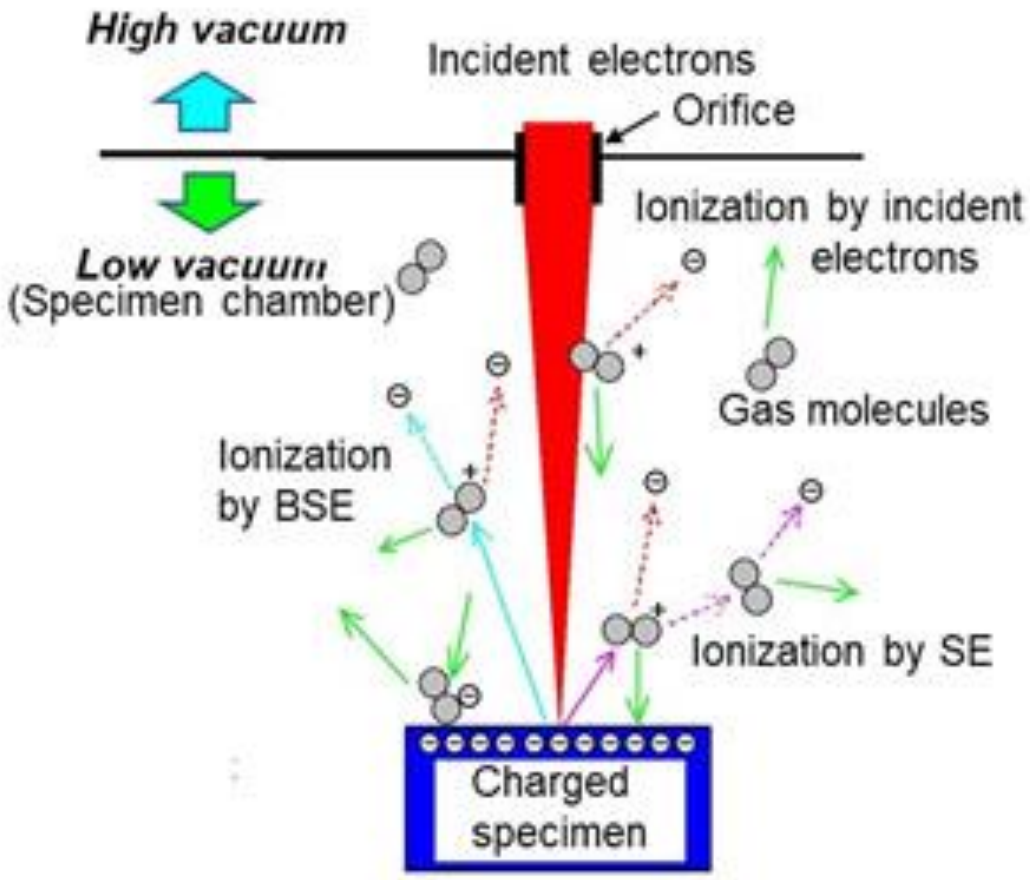
- Lo Vac is a technique where the pressure of the specimen chamber is increased, allowing in atmospheric gas for extra electrons to "ground out"
- Pros: Saves setup time and cost of consumables
- Cons: Resolution less than best, most SEMs can only do BSE



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Non-Conductive Specimens

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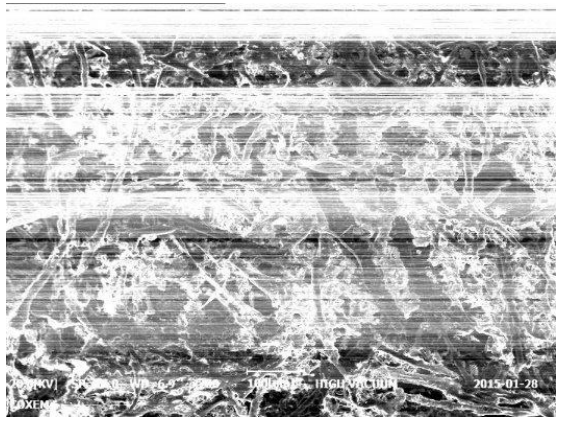
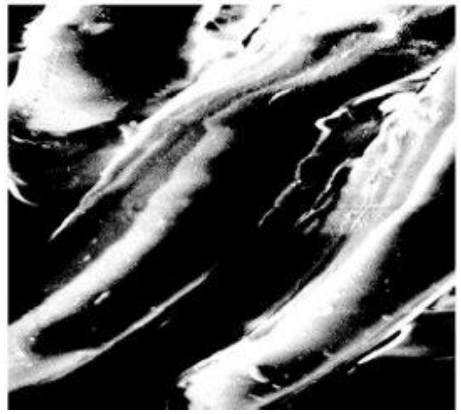
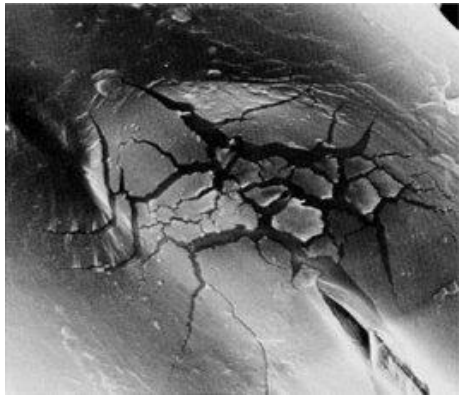


"Scan line" effect

"Glowing" or Charging" effect



"Moving image"effect



"edge"effect

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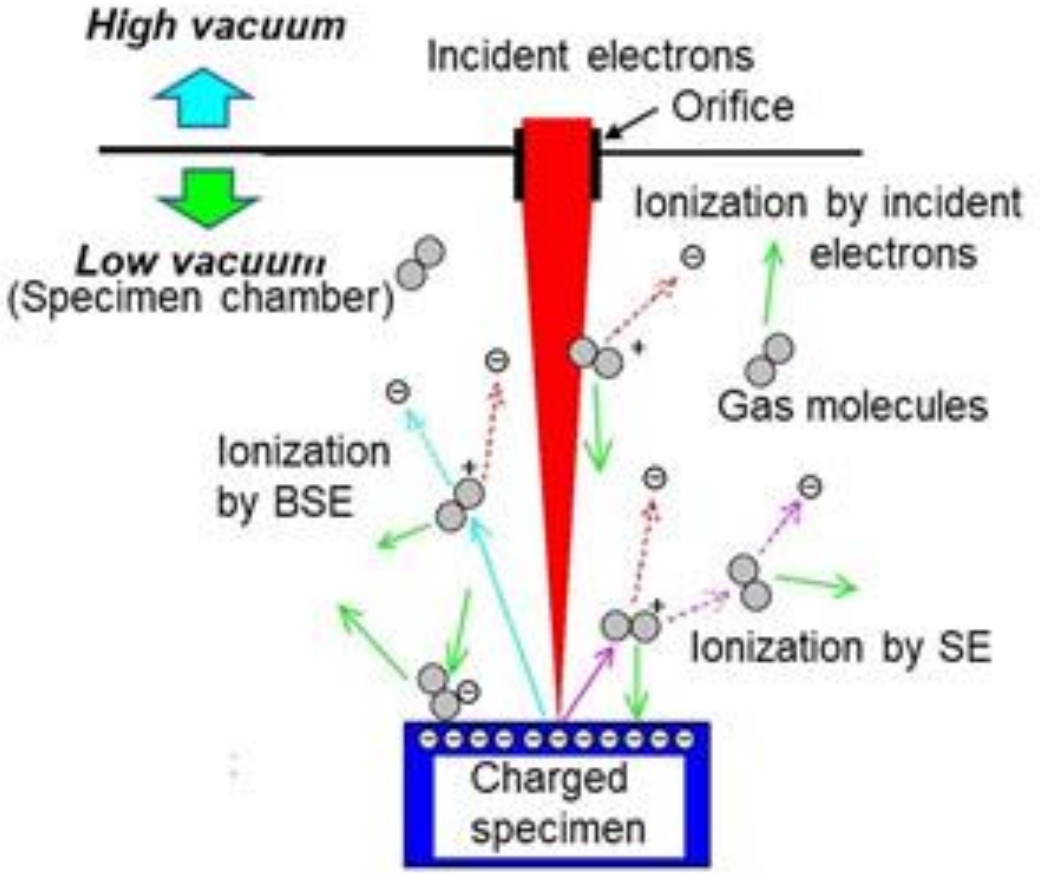
Material Types

- Low Vac is needed for organics and other non-conductives such as polymers.



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Sample Preparation – Lo Vac Process

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Sectioning

Mounting

Grinding and Polishing

Coating

Imaging

Takeaways

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- Consider typical sputter coating for a non-conductive sample
- When coating is not possible, Low Vac is an option

Low Vac on COXEM EM-40 SEM

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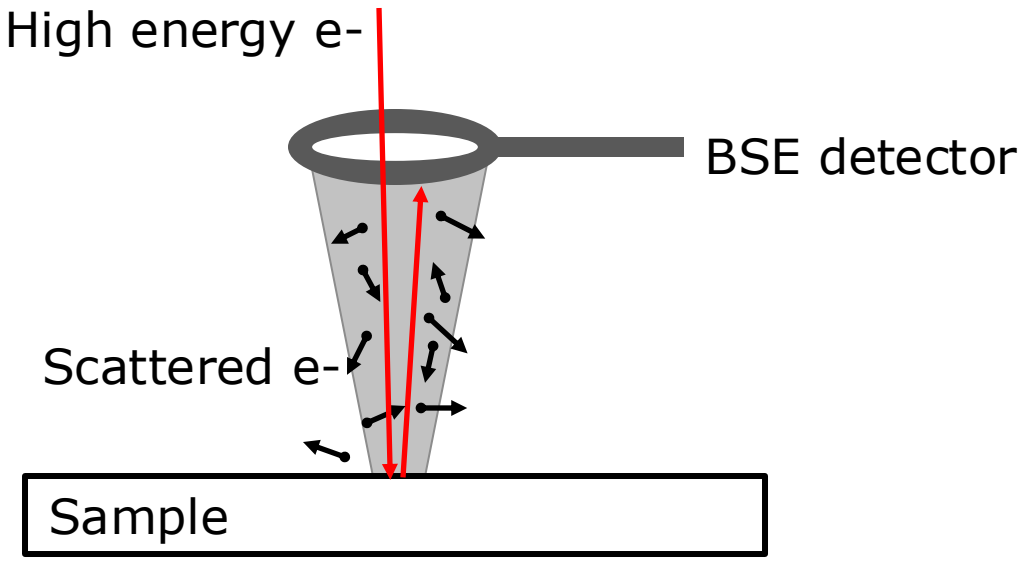
- Low vacuum chamber is achieved via a chamber valve module
- Most of the electrons are scattered by gas molecules
- High energy electrons can be collected using BSE detector for imaging
- Save space/expense on extra low vac detectors
- Ionization of the gas molecules neutralized the charging on the sample



Cases - High Vac vs Low Vac

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Paper towel

Hair

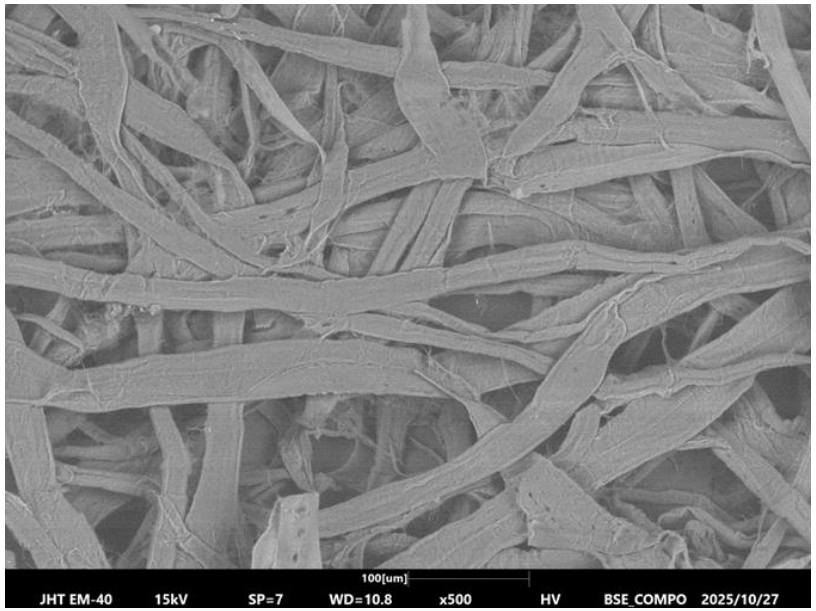
Diatom algae

Epoxy-mounted cross-section

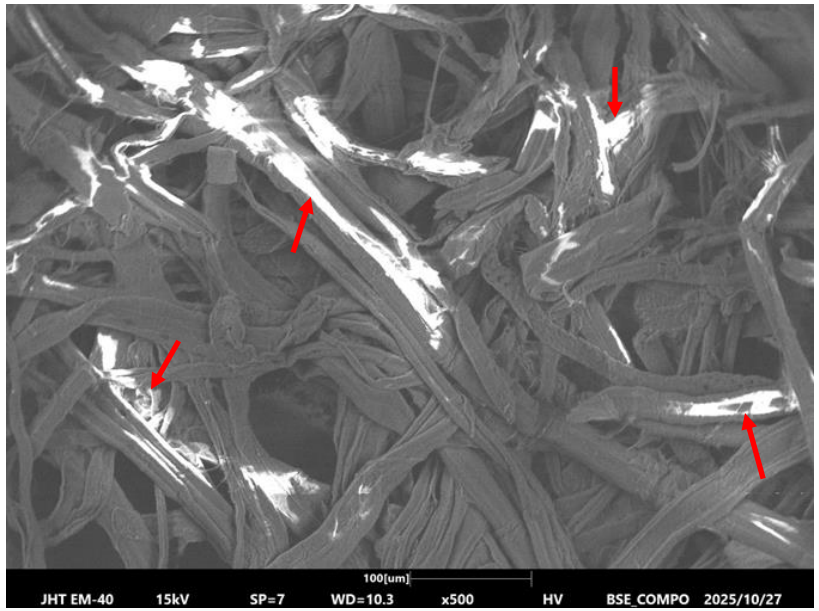
Cases - High Vac vs Low Vac

Paper towel (polymer)

HV coated

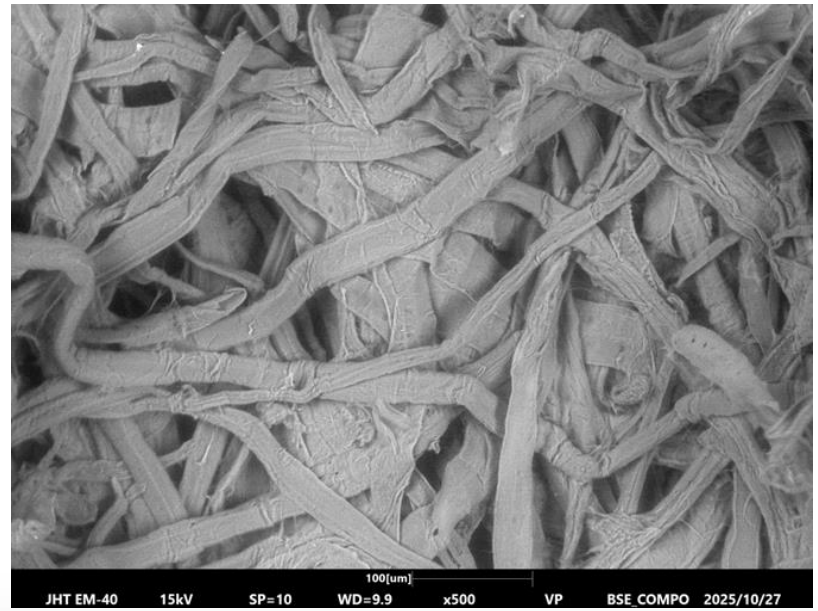


HV non-coated



Charging

LV non-coated



LV SEM avoids potential morphology change on porous polymer.

Cases - High Vac vs Low Vac

Hair

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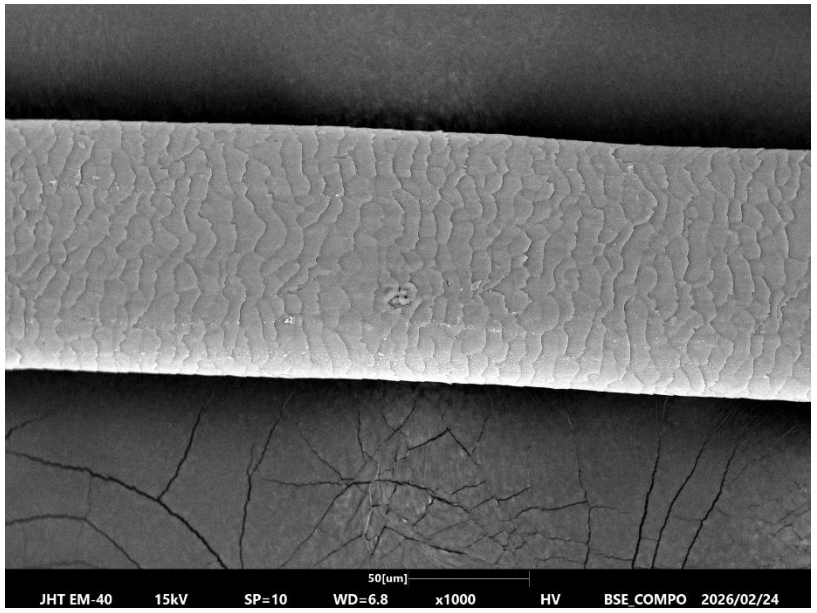
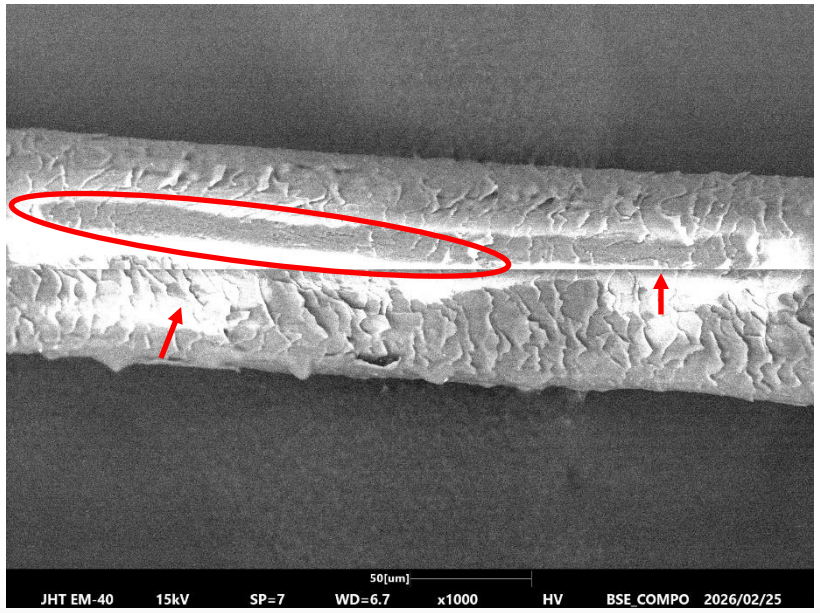
HV coated

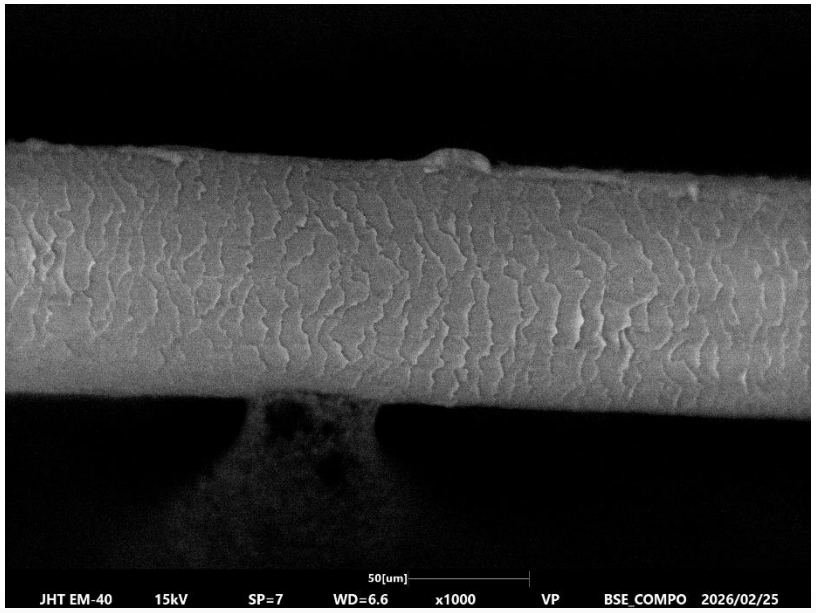


HV non-coated



Beam damage, charging, striping

LV non-coated

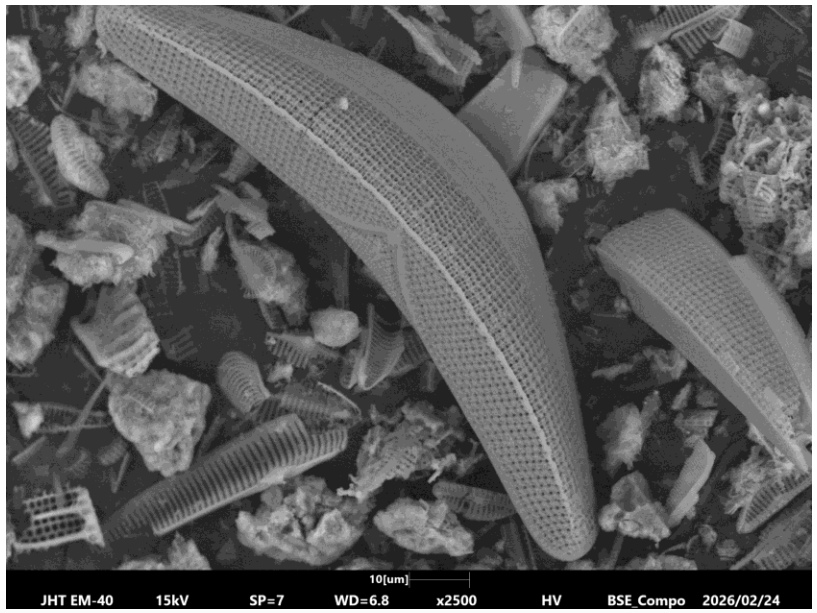


LV SEM avoids beam damage.

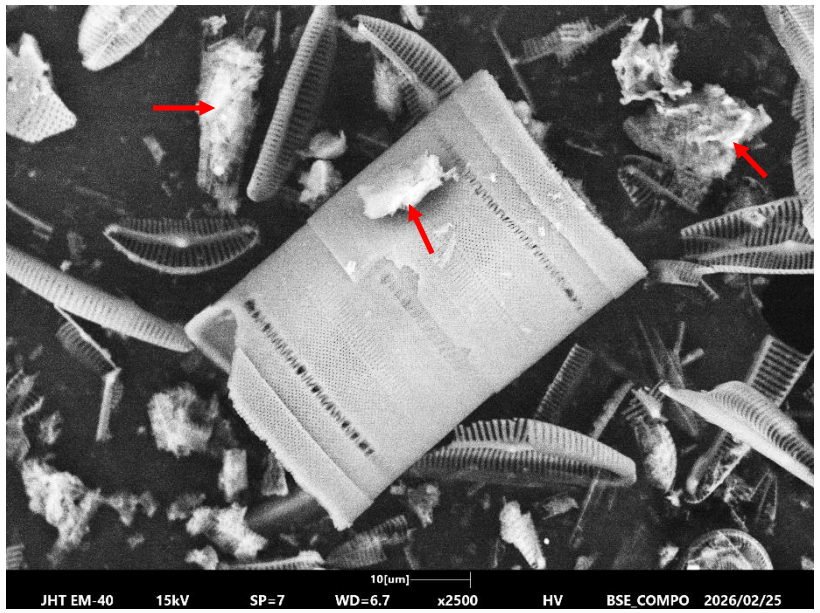
Cases - High Vac vs Low Vac

Diatom algae

HV coated

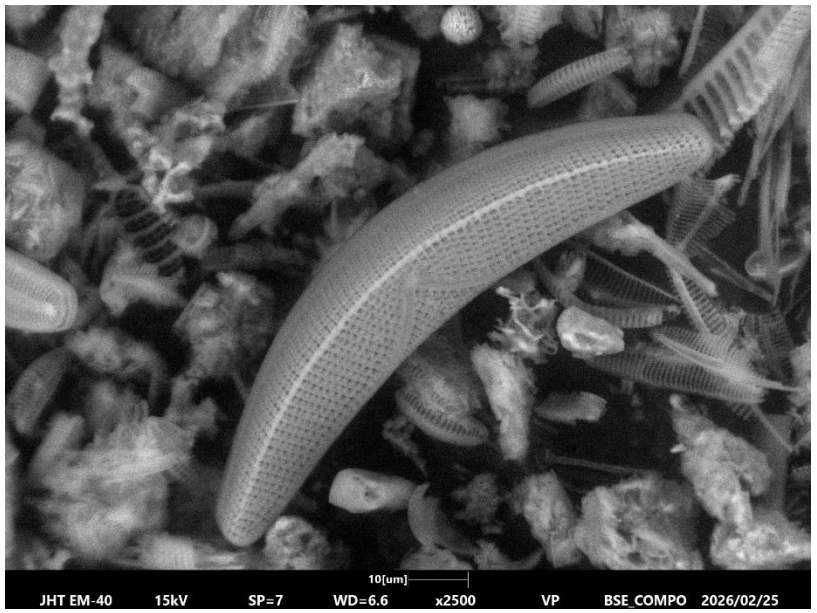


HV non-coated



Charging

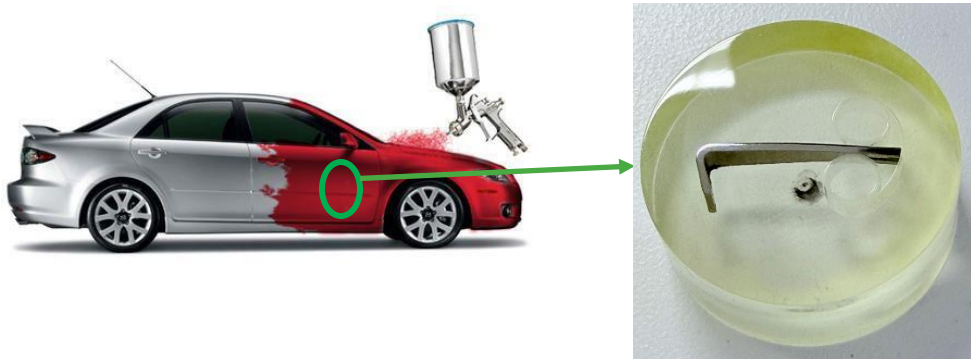
LV non-coated



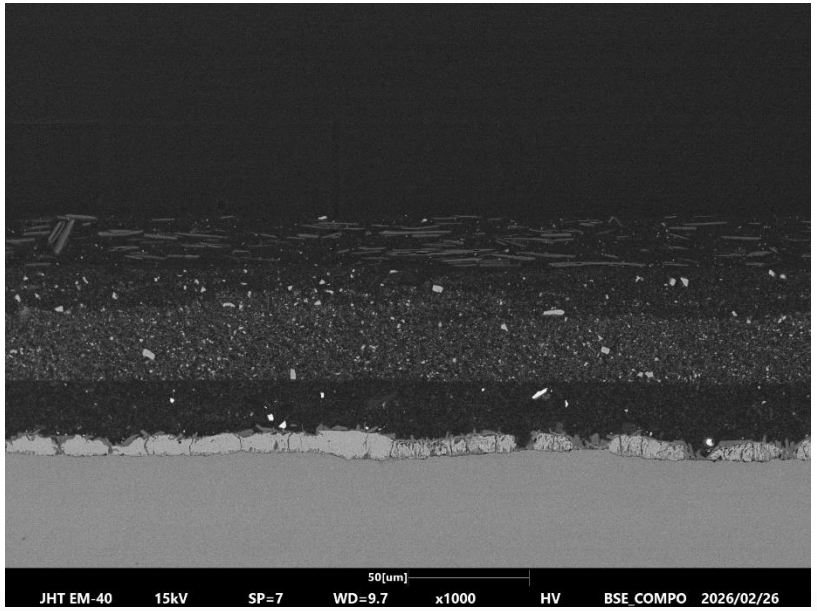
LV SEM preserves original surface features, allowing follow up testing.

Cases - High Vac vs Low Vac

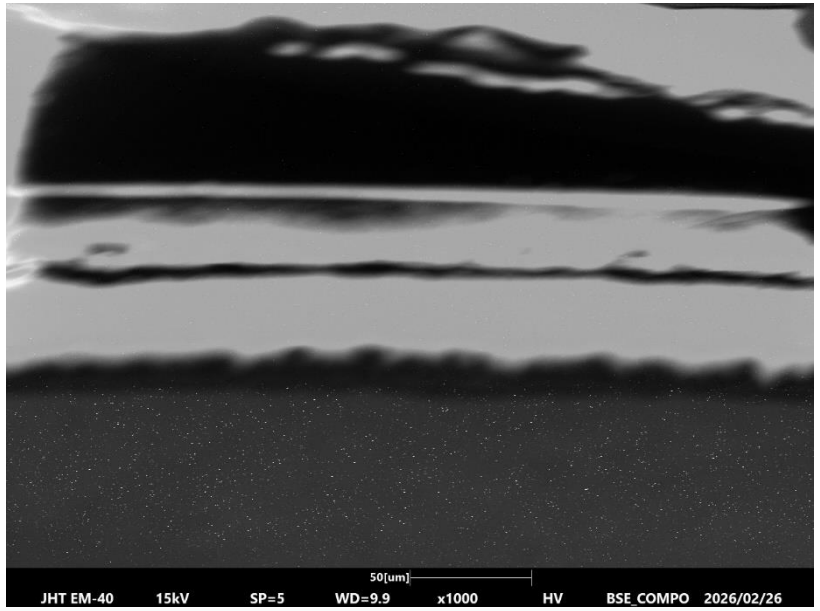
Epoxy-mounted car body with paint



HV coated

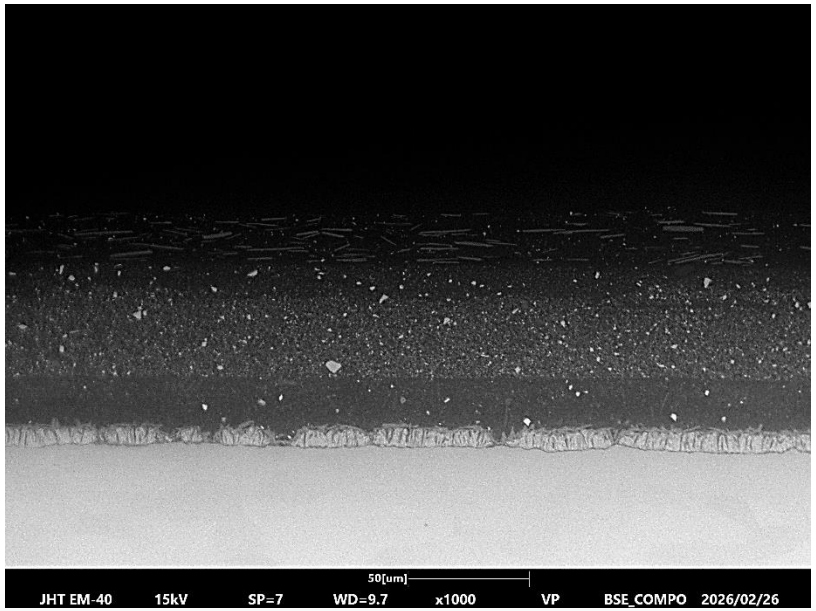


HV non-coated



Charging, cannot focus

LV non-coated



LV SEM allows faster turn-around for quick analysis.

Cases - High Vac vs Low Vac

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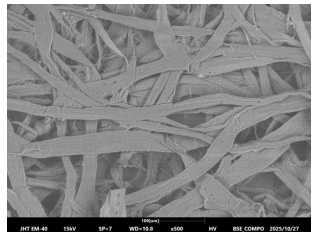
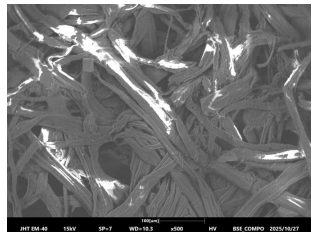
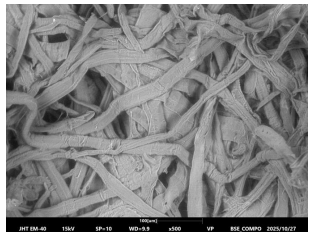
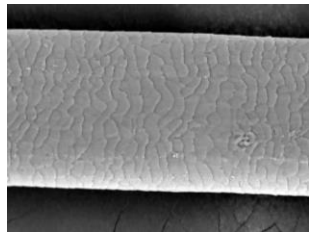
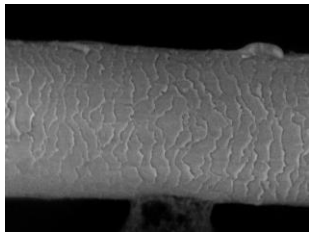
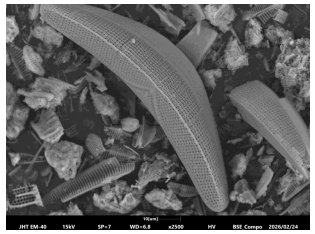
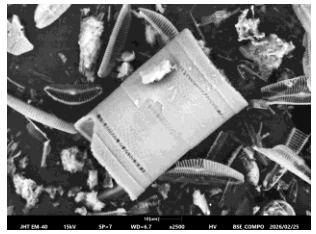
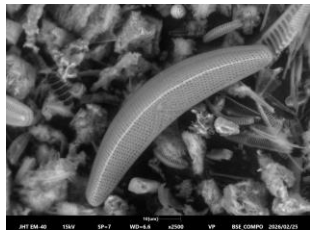
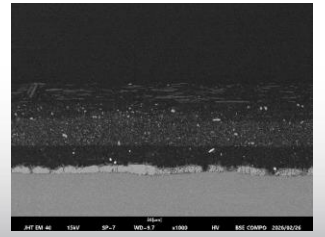
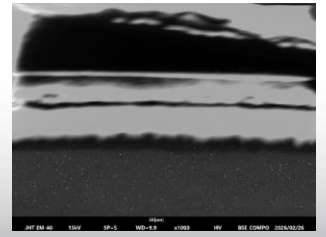
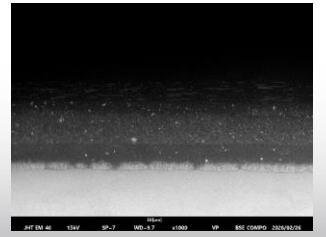
		HV coated by Au/Pd	HV non-coated	LV non-coated
Paper towel				
Hair				
Diatom algae				
Epoxy-mounted cross-section				

Benefits of Low Vacuum Imaging

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For non-conductive materials

- Avoid charging
- Avoid beam damage
- Preserve original surface features
- Allow faster turn-around for quick analysis

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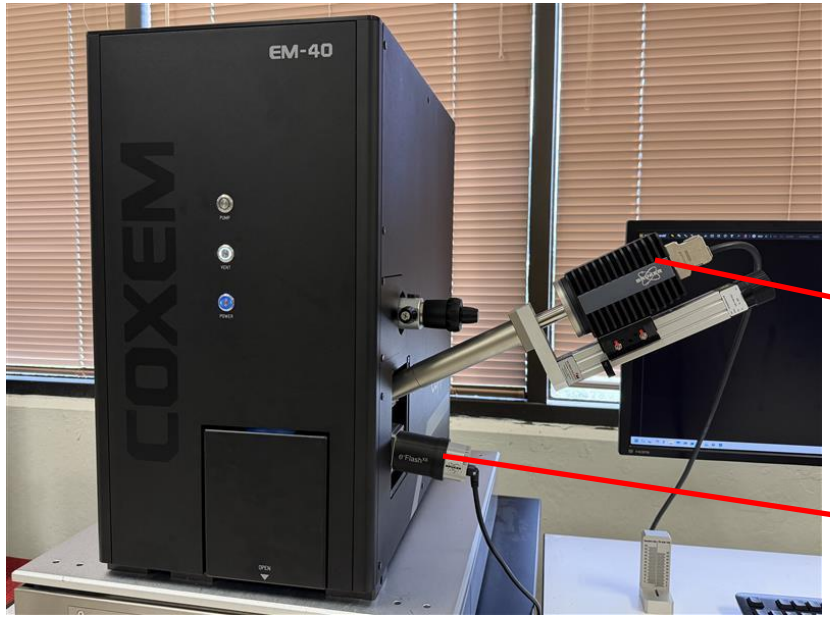
Product showcase: Coxem EM-40

COXEM EM40

Tungsten filament



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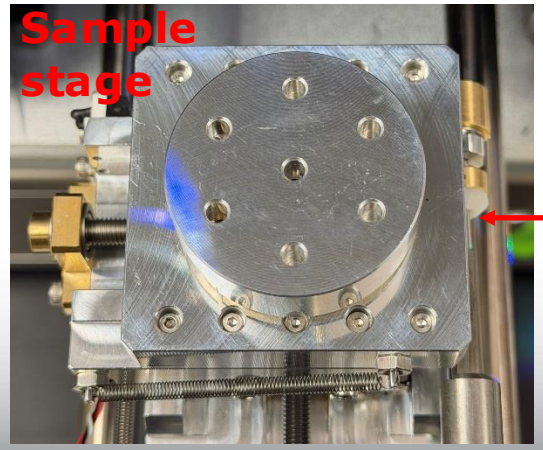
Bruker QUANTAX ED-XS system

EDS

EBSD



	EM-40
ELECTRON GUN	Tungsten filament (W)
SPATIAL RESOLUTION	5nm @ 30kV
MAGNIFICATION	13x to 250,000x
ACCELERATING VOLTAGE	1 ~ 30kV (1kV increments)
SAMPLE STAGE	X : 40mm, Y : 40mm, Z : 40mm, 3-axis motorized
DETECTOR	SE, BSE
VACUUM MODE	HV, VP

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Live Demo: Comparing Hi Vac and Lo Vac

Wrap Up: did we meet our Objectives?

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1. Introduce the SEM and SEM sample prep
2. Demonstrate the use cases for Low-Vacuum imaging
3. Consider **time**, **cost**, and **analytical capability** tradeoff

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Questions?

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Thank You!