Vacuum in electron microscopy

6th CEMM workshop

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The use of vacuum gave us “new” technologies such as:

- lightbulb - electrical industry,
- vacuum packing – food industry,
- lyophilization – chemical industry,
- particle accelerator,
- thin film technology,
- plasma
- fusion reactors,

….

Image: pexels.com

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… and electron microscopy

Outline

- What is vacuum?
- How do we create vacuum?
- Why do we need vacuum in EM?
- How the samples and our handling affects the vacuum system?

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What is vacuum?

Latin “vacuus” meaning „void".

In engineering and applied physics, vacuum refers to **any space** in which the **pressure is lower** than atmospheric pressure.

Units of pressure:

SI unit: **Pa**

Other units: bar, atm, Torr,

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History

PECS and PIPS

Image: thefamouspeople.com

Image: wiki

Evangelista Torricelli

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Blaise Pascal

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Image: etc.usf.edu

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The need to understand vacuum

Magdeburg hemispheres, by Otto von Guericke

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The need to understand vacuum

A ringing bell in vacuum

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Link:
www.youtube.com/watch?v=ce7AMJdq0Gw

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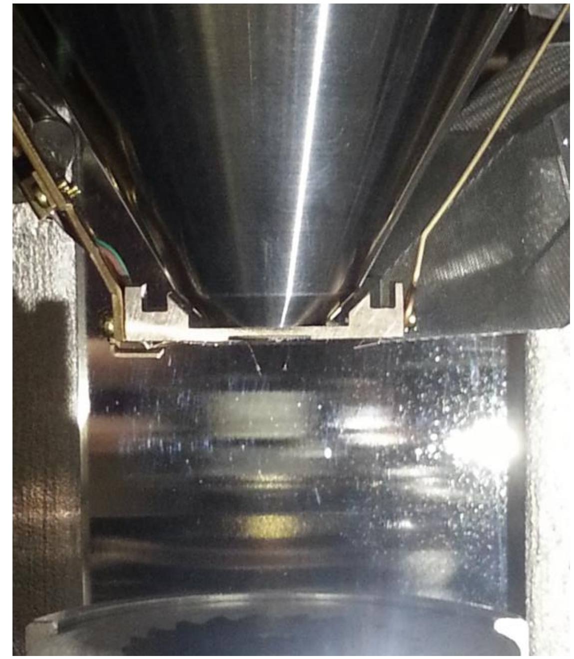

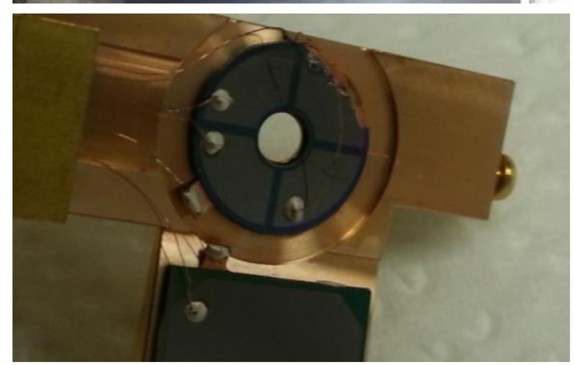

Revolution in physics

1895 **X-rays** (that we use in EDS, WDS) discovered by Wilhelm Conrad Roentgen.

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Revolution in physics

1897 **electron** (that we use) identified by Joseph John Thompson.

Figure removed for copyright reasons.

Revolution in physics

1909 Ernest Rutherford discovered that we are 99,999 % of vacuum…

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"It was almost as incredible as if you fired a 15-inch shell at a piece of tissue paper and it came back and hit you."

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Revolution in physics

In 1913 Henry Moseley **arranged the elements in the periodic table by their number of protons** rather than their atomic weights, the flaws in the periodic table that had been making scientists uncomfortable for decades simply disappeared.

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"We have here a proof that there is in the atom a fundamental quantity, which increases by regular steps as one passes from one element to the next. This quantity can only be the charge on the central positive nucleus, of the existence of which we already have definite proof."

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How do we create vacuum?

First – what are the areas of low pressure?

rough

abbreviation	Vacuum area	Pressure [mbar]	numerical density [particles/m³]
LV (sl: GV)	low vacuum	1000 - 1	10^{19} - 10^{16}
MV (sl. SV)	medium vacuum	1 - 10^{-3}	10^{16} - 10^{13}
HV (sl. VV)	high vacuum	10^{-3} - 10^{-7}	10^{13} – 10^{9}
UHV (sl. UVV)	ultra high vacuum	10^{-7} - 10^{-12}	10^{9} - 10^{4}
EXV (sl. EVV)	extremely high vacuum	under 10^{-12}	under 10^{4}

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The power of teamwork!

- Nobody can do it alone

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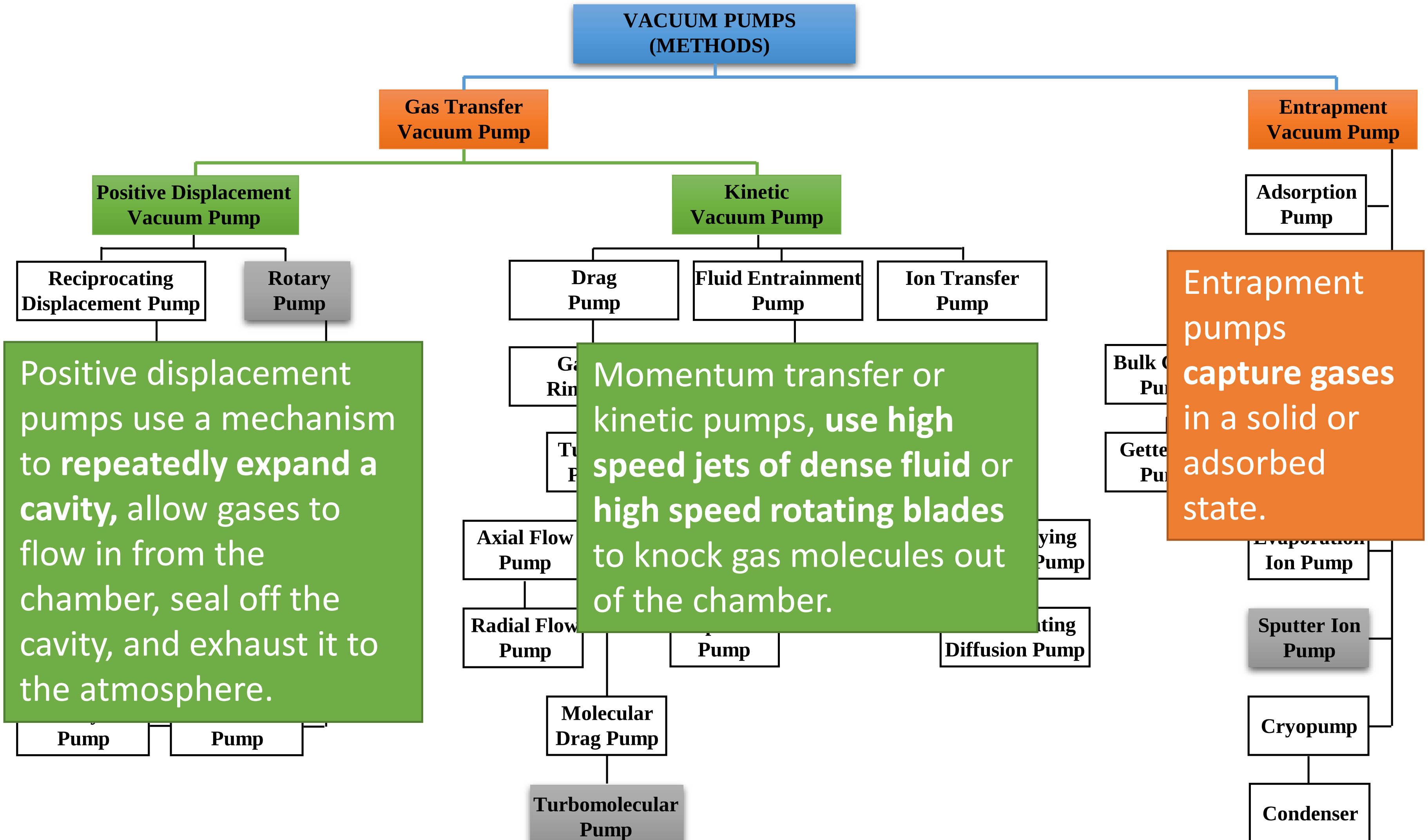
How do we create low pressure?



Positive displacement pumps use a mechanism to **repeatedly expand a cavity,** allow gases to flow in from the chamber, seal off the cavity, and exhaust it to the atmosphere.

Momentum transfer or kinetic pumps, **use high speed jets of dense fluid** or **high speed rotating blades** to knock gas molecules out of the chamber.

Entrapment pumps **capture gases** in a solid or adsorbed state.

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We need a pumping

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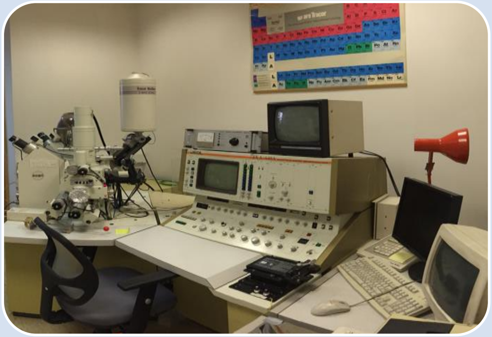		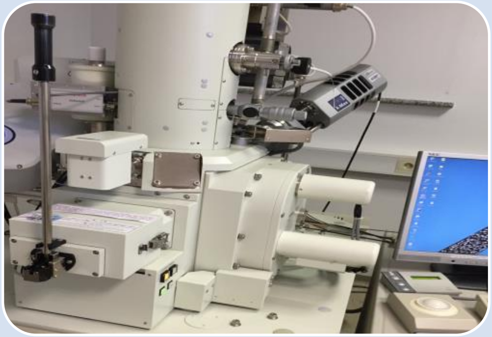
- Rotary pump - Diffusion pump	- Rotary pump - Diffusion pump	- Rotary pump - Turbomolecular - Sputter ion pump -Cold trap

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Rotary Pump

First pump.

Good efficiency for high pressure.

The oil is a lubricant and (to some degree) protects the pump (corrosive gases, particles).

Figure removed for copyright reasons.

(Video: YouTube)

Disadvantages:

- vibrations
- oil vapour

JXA-840A

JSM-7600F

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Diffusion Pump (DP)

Second pump, after the rotary pump.

Works by momentum transfer (not much to do with diffusion). The vaporized oil jets grab the gas molecules. When the oil condenses, the gas is free and pumped out.

Very **high pumping speed**, pumps also light gases.

Tolerant with particles and corrosive gases.

Figure removed for copyright reasons.

(Video: YouTube)

Disadvantages:

- needs warm up and special oil, which is evaporated
- needs cooling too
- can only work vertically

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(Image: pchemlabs.com*)*

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Turbomolecular Pump (TMP)

Second pump, after the rotary pump.

A jet turbine and works by momentum transfer (multiple stages of rotating blades (rotor) spaced between fixed blades (stator). The rotor hits the molecules and the stator moves the molecules down.

High pumping speed but not for light gases.

Clean, no warm up, oil free.

Disadvantages:

- relatively expensive
- not tolerant to particles
- can fail catastrophically
- high vacuum is pure hydrogen

Figure removed for copyright reasons.

(Video: YouTube)

Figure removed for copyright reasons.

(Image: wikipedia)

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Sputter Ion Pump

Third pump, after the TMP or DP.

No moving parts.

Ions from gas molecules are pulled on the cathode.

Cathode trapes the gas and as a result we get free e- and Ti ions.

Anode (5 000 V) pulls the free e-. Magnets make longer path for the gas molecules collision (ionization).

The sputtered Ti can also bury residual gases under a film.

Disadvantages:

- not very efficient for water
- low capacity

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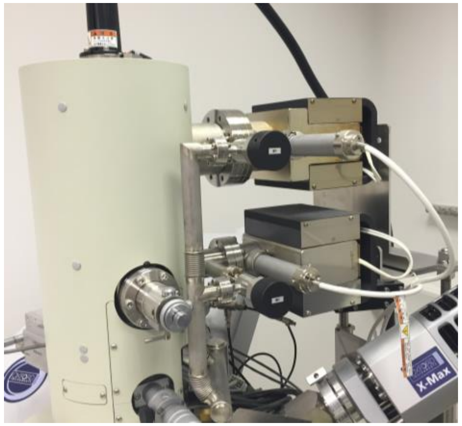
(Video: YouTube)



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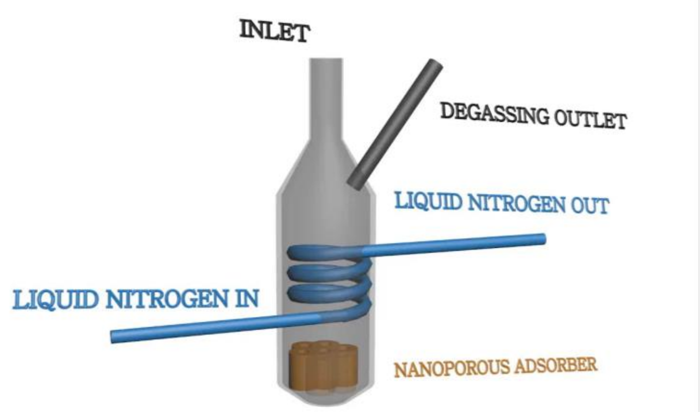
Cold trap (ACD-anti contamination device)

Fourth, used to improve the vacuum in the chamber (depending on the sample).

Molecules are trapped on the surface (sorption).

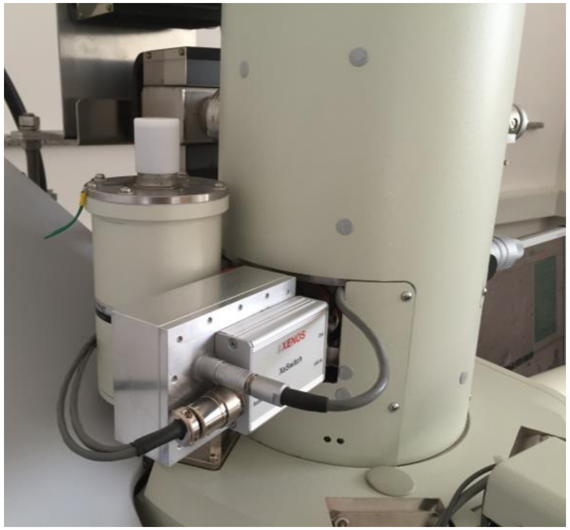
Good for water.



(Video: YouTube)

Disadvantages:

- frequently degassed
- gasses not permanently removed



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Why do we need vacuum in EM?

To move an e- in a (straight) line over a **large distance**.
To prevent **beam induced** chemical reactions.

Required for **stable emission** and for some **detectors** and **lenses**.

To provide a „**clean“ surface**.

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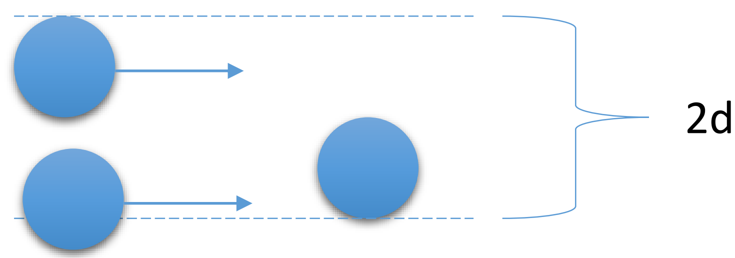
Kinetic teory for ideal gas

The kinetic theory of gases is the study of the **microscopic (x, v)** behavior of molecules and the interactions which led to **macroscopic (P, V, T)** relationships like the ideal gas law.

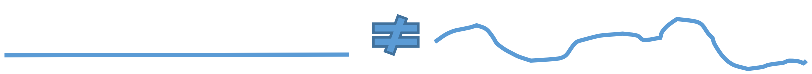
Mean free path $l_{mfp} = \dfrac{k_B T}{\sqrt{2}\,\pi\, d^2 p}$



Monolayer $t_{monolayer} = \dfrac{10^{15}\sqrt{2\pi m k_B T}}{p}$

1 monolayer ≈ 10^{15} particles/cm^2

p - pressure
T-temperature
k_B - Boltzmann constant
d –diameter of the atom



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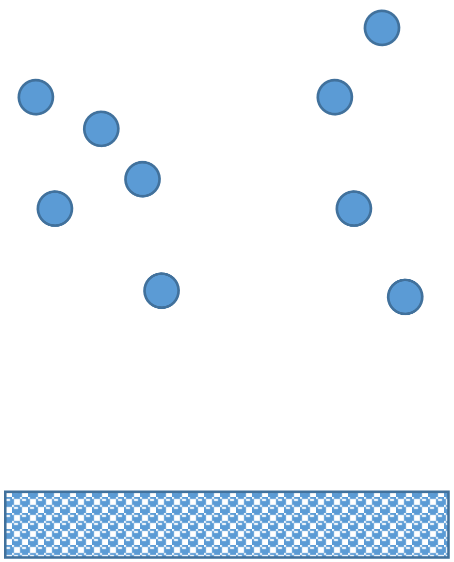
Is the surface really clean?

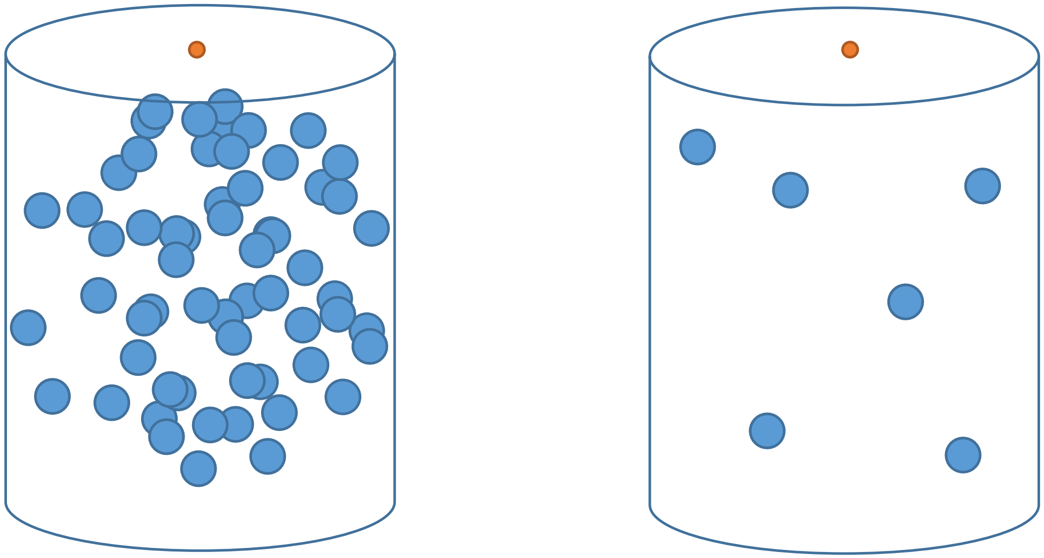
We can calculate how much time is needed to form one monolayer. Assumption – every particles that collides with the surface attaches on it.

For N_2 molecules at room temperature for different pressures:



p [mbar]	l_{mfp}	$t_{monolayer}$
1000	65 nm	3,5 ns
1	65 µm	3,5 ms
10^{-3}	65 mm	3,5 ms
10^{-5}	6,5 m	**0,35 s**
10^{-6}	65 m	3,5 s
10^{-9}	65 km	58 min

How do the e- move in vacuum?



Higher than the mean free path between atoms.

Better vacuum means less interactions between e- and gas.

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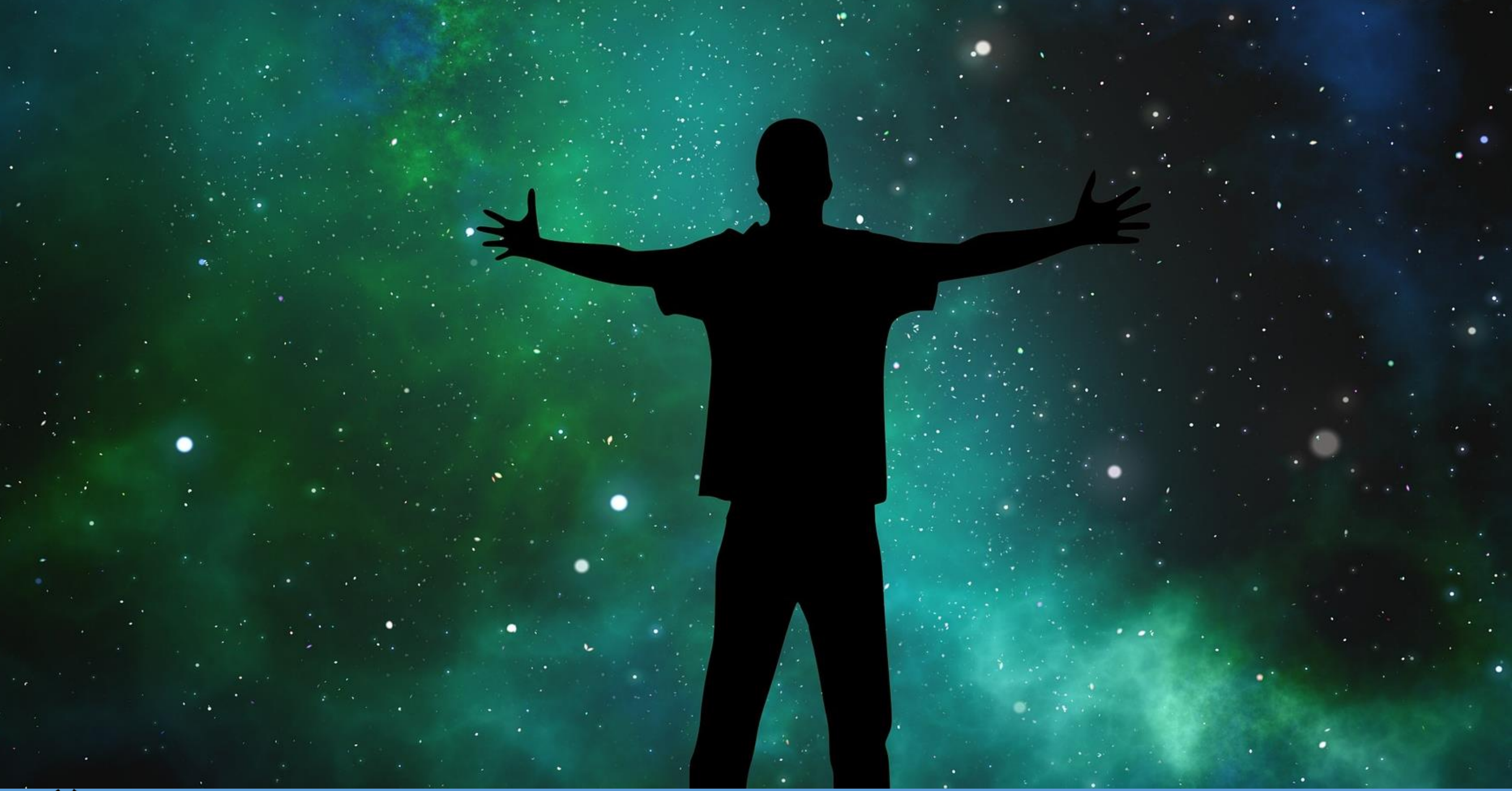
How the samples and our handling affect the vacuum system?



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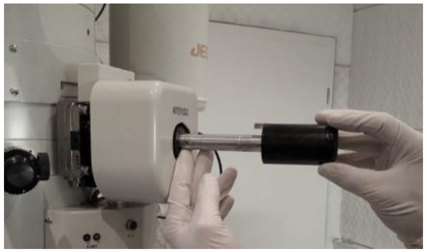
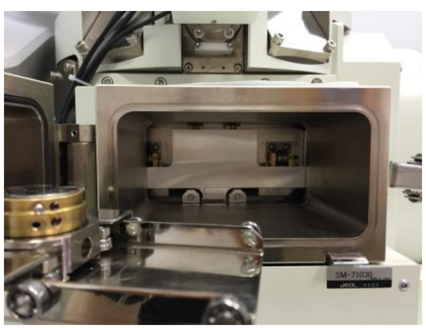
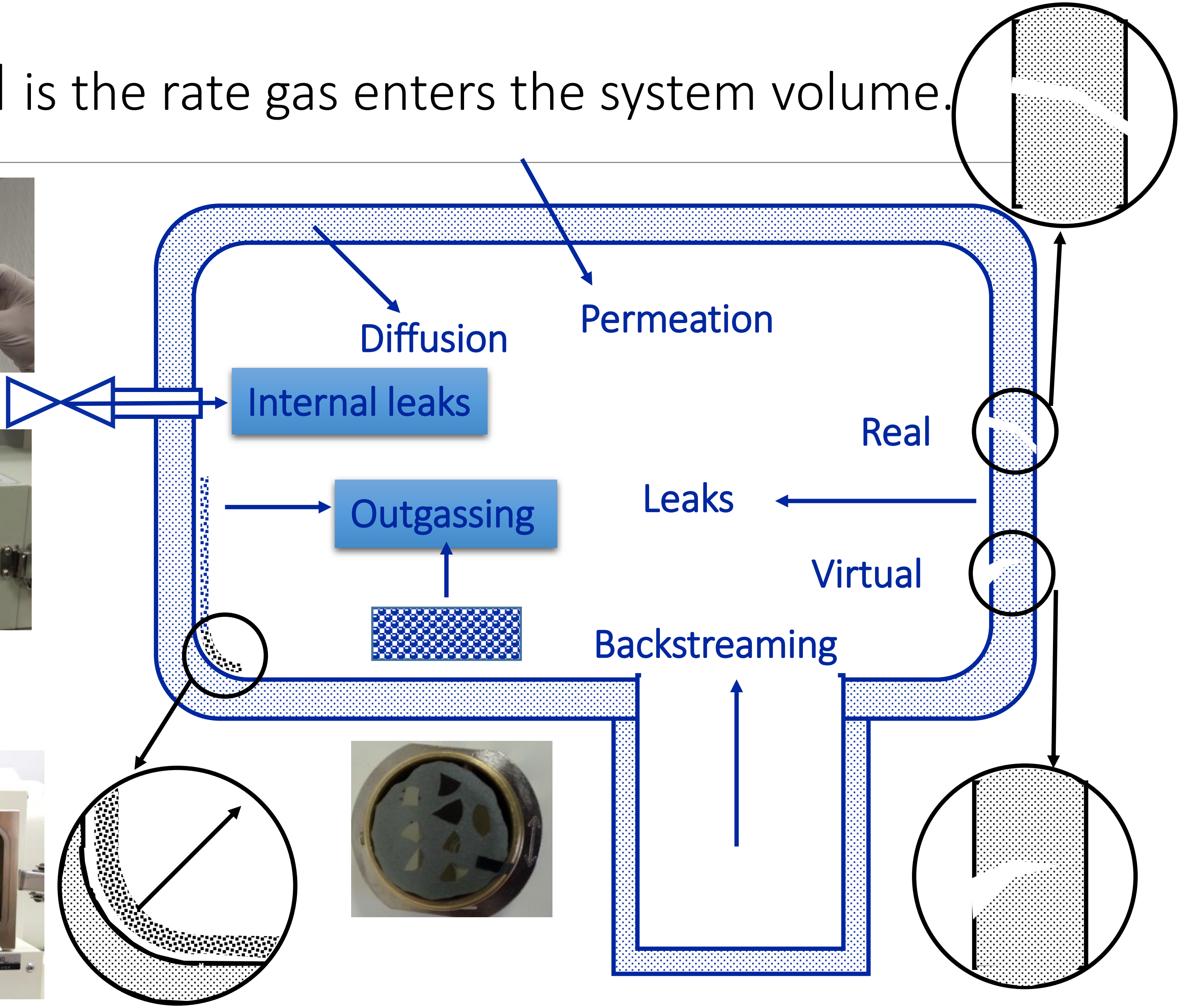
Gas Load is the rate gas enters the system volume.



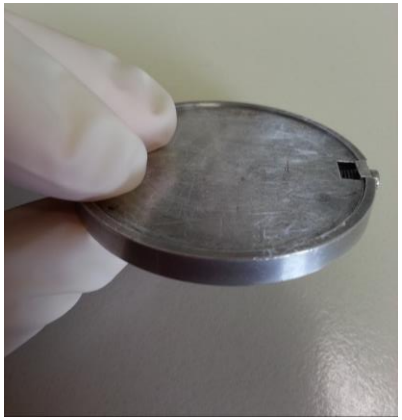




Clean and dry sample (no outgassing)

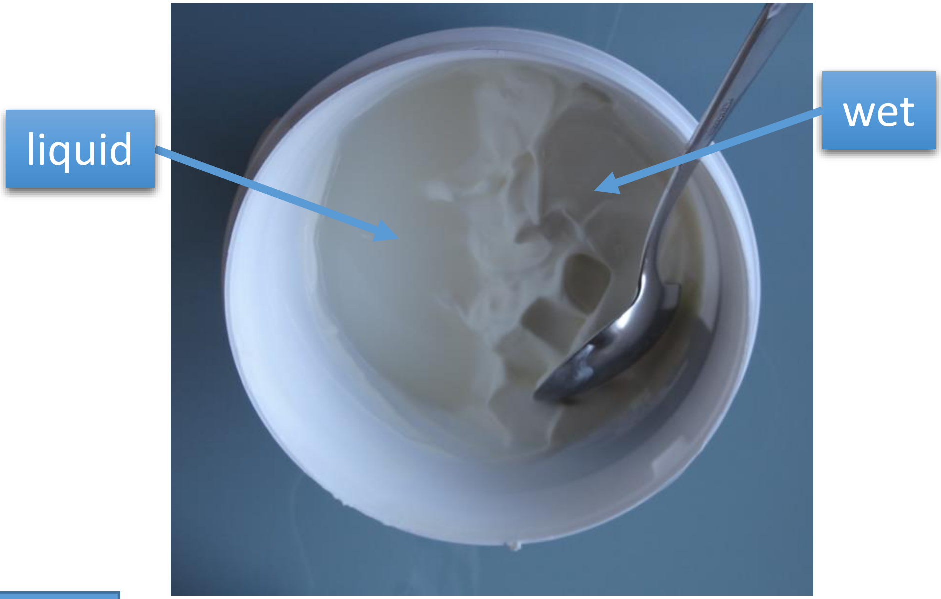
Definition of a clean sample

Clean with acetone, ethanol
Use gloves – hand fat…



Always check recent published research papers to check on current techniques being used.

Definition of a dry sample



Liquid: paint, milk, mud

Wet: fresh leaf, fresh concreate, a fly

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Can I image powder?

Is it dry and low rates of outgassing?

Let's assume it is stable under the e- beam.

Let's assume we have small amount!

NO → **NO SEM**

Sample can damage the pumping system.

YES → **Is it well attached?**

NO → **NO SEM**

Particles can damage the pumping system and the SEM.

YES → **Is it magnetic?**

Figure removed for copyright reasons.

NO → **YES, you may go on the SEM**

YES → **YES, but…**

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Can I image bulk?

Is it **dry** and **low rates of outgassing**?

Let's assume it is stable under the e- beam.

NO

NO SEM

Sample can damage the pumping system.

YES

Let's assume it is firmly attached!

Is it small?

NO

NO SEM

Long pumping time.

YES

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Is it magnetic?

NO

YES, you may go on the SEM

YES

YES, but…

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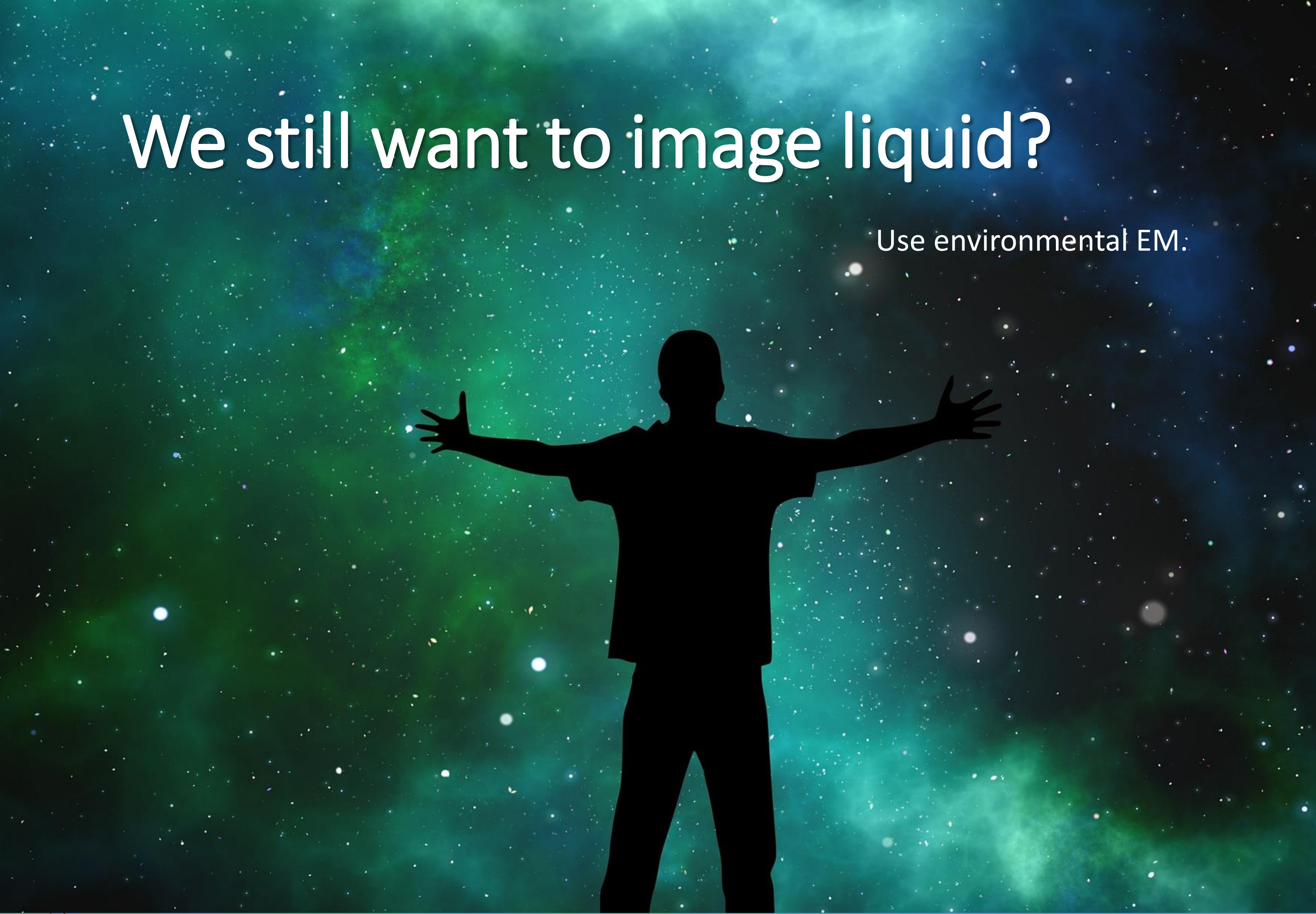
We still want to image liquid?

Use environmental EM.



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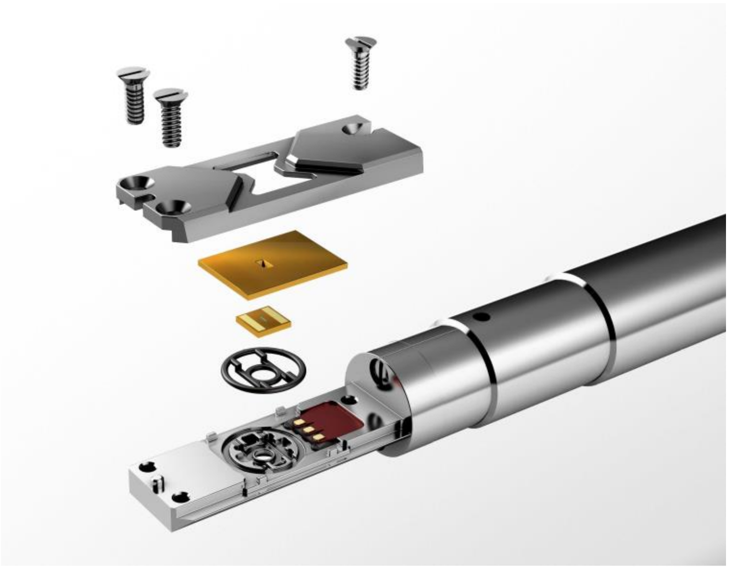
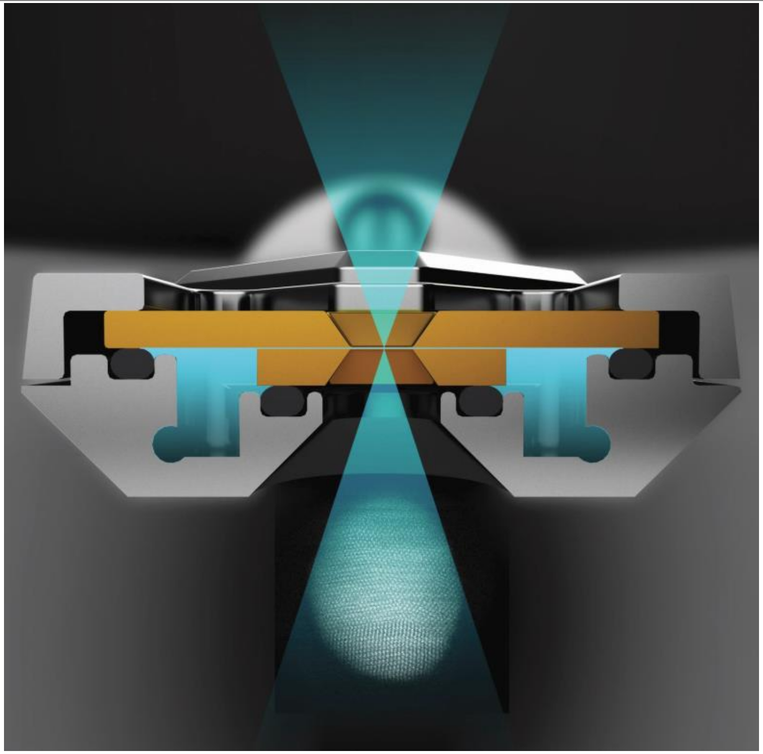
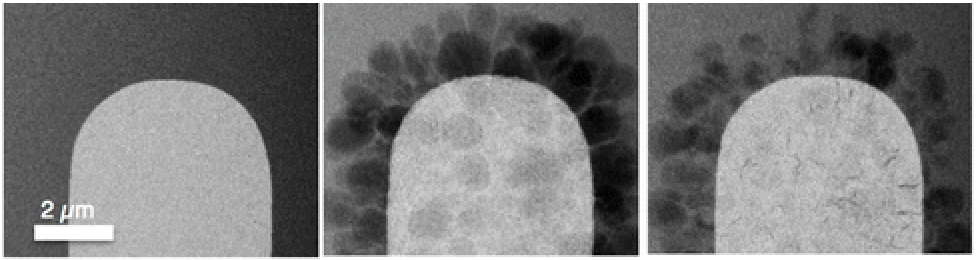
Environmental (holders) for TEM







Slika:protochips

Take home information

- Sound doesn’t travel through vacuum (vibrations)
  - **Use the camera** if you have one! One can damage detectors, column… If you have big magnetic samples you won´t hear when it will get stuck on the pole piece.
- Chamber shouldn't be opened long time:
  - Longer evacuation time.
  - If you have and SiLi EDS, that can cause ice formation on the window.
- Big bulk samples
  - Longer pumping time, outgassing,
  - Gases can be released from porous materials or cracks
  - If magnetic one can damage the SEM
- Small powders
  - Can fly from the holder, problems on the column, apertures, electron source
  - If magnetic one can damage the SEM!
  - **Never open** the V1 valve (ON/OFF button), if you are **closer than 8 mm** (WD) on 7600F – the difference in pressure is to **1000**.
- **Use gloves** and clean everything
  - Traces of lubricants and residues from machining may be present on surfaces
  - Hand fat…

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Društvo za vakuumsko tehniko Slovenije DVTS

Learn more on:

Vacuum Fittings and Accessories,

Different pumps and gauges,

Physics of vacuum,

The use of vacuum,

Symbols,

Leak detection technique,

…

The vacuum coarse will be organized in 2018, contact:

info@dvts.si, janez.kovac@ijs.si

Društvo za vakuumsko tehniko Slovenije - DVTS bo
27. in 28. marca 2018
organiziralo dvodnevni
tečaj »Osnove vakuumske tehnike«,
ki je namenjen mladim raziskovalcem in vsem tistim, ki delajo na področju vakuumskih tehnologij.
Tečaj bo potekal na Institutu »Jožef Stefan".
Obsegal bo teoretični in praktični del.
Cena tečaja je 400 EUR.
V ceni je všteta literatura (knjiga Vakuumska znanost in tehnika) in dve kosili.

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