Vacuum in electron microscopy

5th CEMM workshop

Maja Koblar, Sc. Eng. Physics

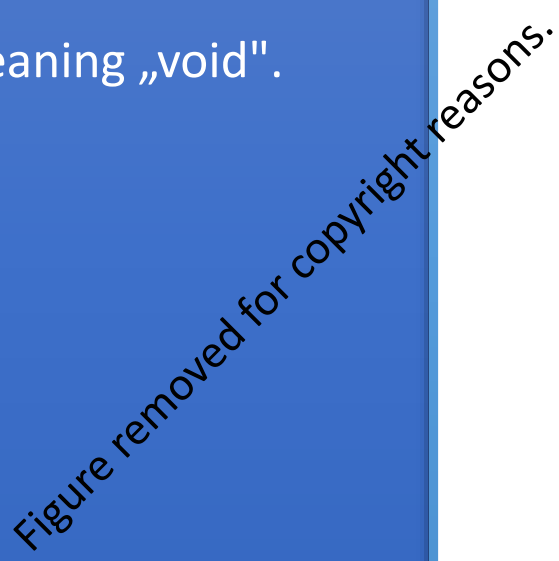
Outline

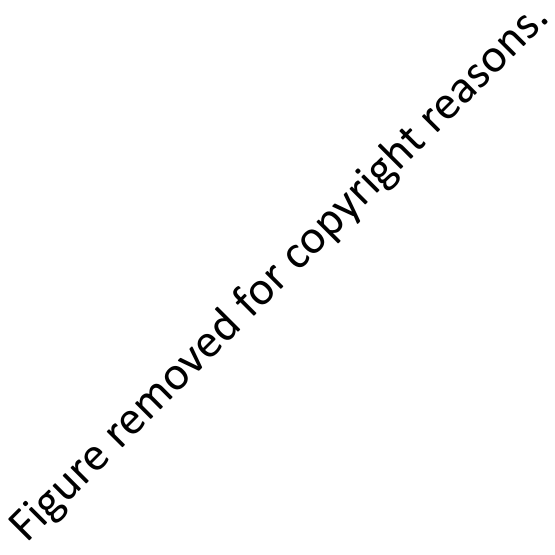
- What is vacuum?
- History
- Pressure ranges
- How do we create low pressure?
- Different pumps in EM
- Why do we need vacuum in EM?
- Samples and vacuum

What is vacuum?

Latin “vacuus”

meaning „void".





Aristotle

„Nature abhors a vacuum“

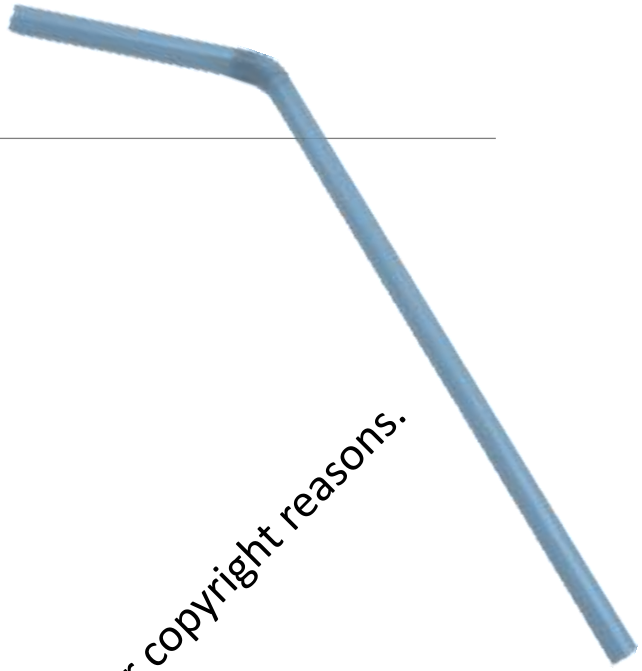


Figure removed for copyright reasons.

Figure removed for copyright reasons.

Image: davidperks.com

Evangelista Torricelli

„We live submerged at the bottom of an ocean of air.“

Figure removed for copyright reasons.

Figure removed for copyright reasons.

Image: etc.usf.edu

Blaise Pascal

performed several groundbreaking experiments using barometers and studying pressure:

- wine, cask –bursting, experiment
- atmospheric pressure changes with altitude

Image: sciencedemonstrations.fas.harvard.edu

Image: wiki

Figure removed for copyright reasons.

Figure removed for copyright reasons.

Figure removed for copyright reasons.

Figure removed for copyright reasons.

The need to understand vacuum

Magdeburg hemispheres, by Otto von Guericke

Figure removed for copyright reasons.

Figure removed for copyright reasons.

The need to understand vacuum

A ringing bell in vacuum

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

How does light travel through vacuum?

Vacuum isn’t empty space anymore!

It seemed that vacuum doesn't exist in nature.

“Ether” the carrier of light

Vacuum isn’t empty - Ether

Ether is static, but Earth moves

Figure removed for copyright reasons.

Albert Michelson in Edward Morley

Figure removed for copyright reasons.

The result was disappointing??

What is light?

Albert Einstein gave a talk: „ Ether and the Theory of Relativity“ on May 5, 1920 at the University of Leiden.

What do you see?

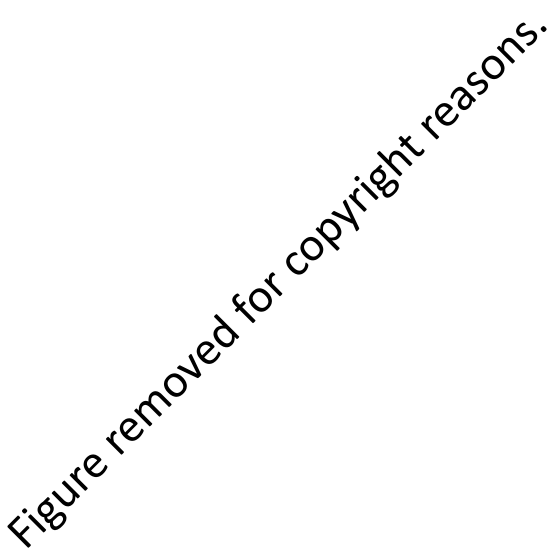


Image: hdfondos.eu

Light is a wave, but also acts as a particle!

Photons - light particles

Einstein explained the photoelectric effect and ultraviolet catastrophe.

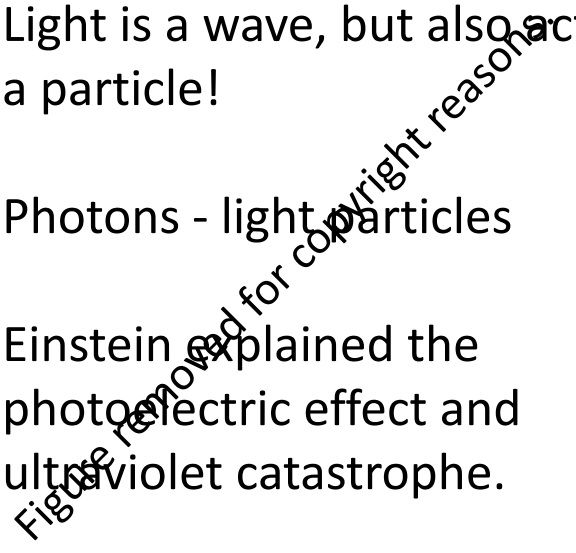


Image: getmedic.ru

Ether doesn’t exist!

The use of vacuum

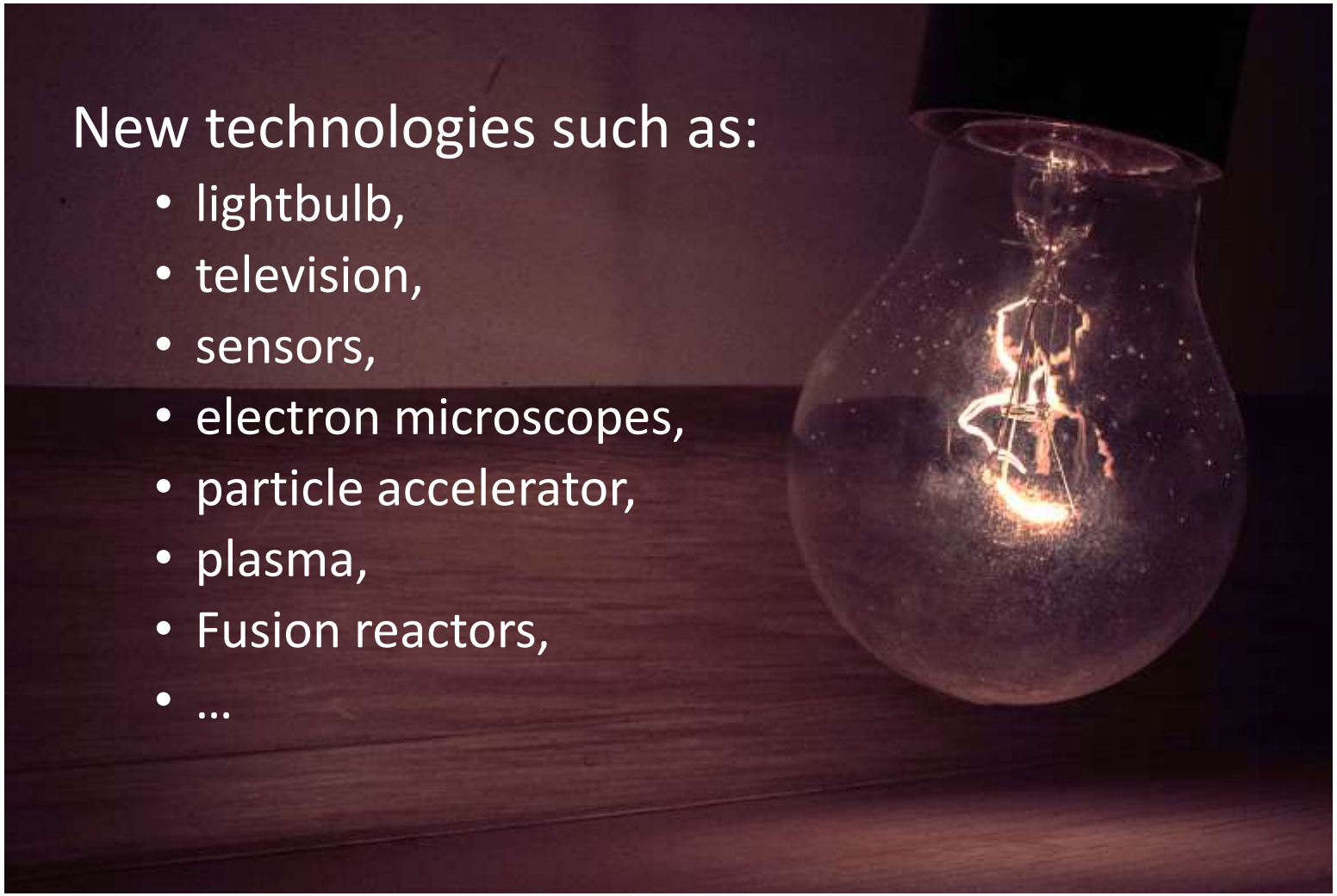
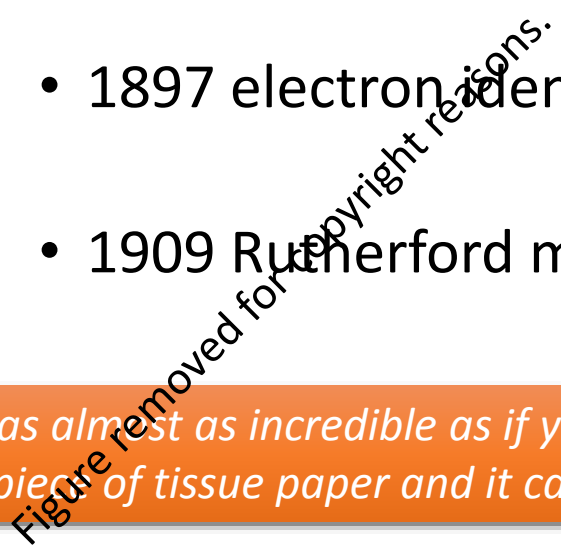


Image: pexels.com

The understanding of vacuum

Revolution in physics:

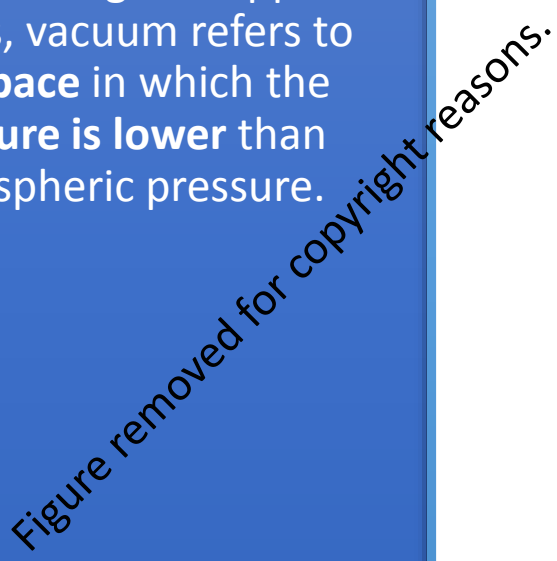
- 1895 x-rays discovered by W. C. Roentgen
- 1897 electron identified by J. J. Thompson
- 1909 Rutherford model of an atom



"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."

What is vacuum?

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



Division of areas of low pressure

	abbreviation	Vacuum area	Pressure [mbar]	numerical density [particles/m^3]
rough	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}

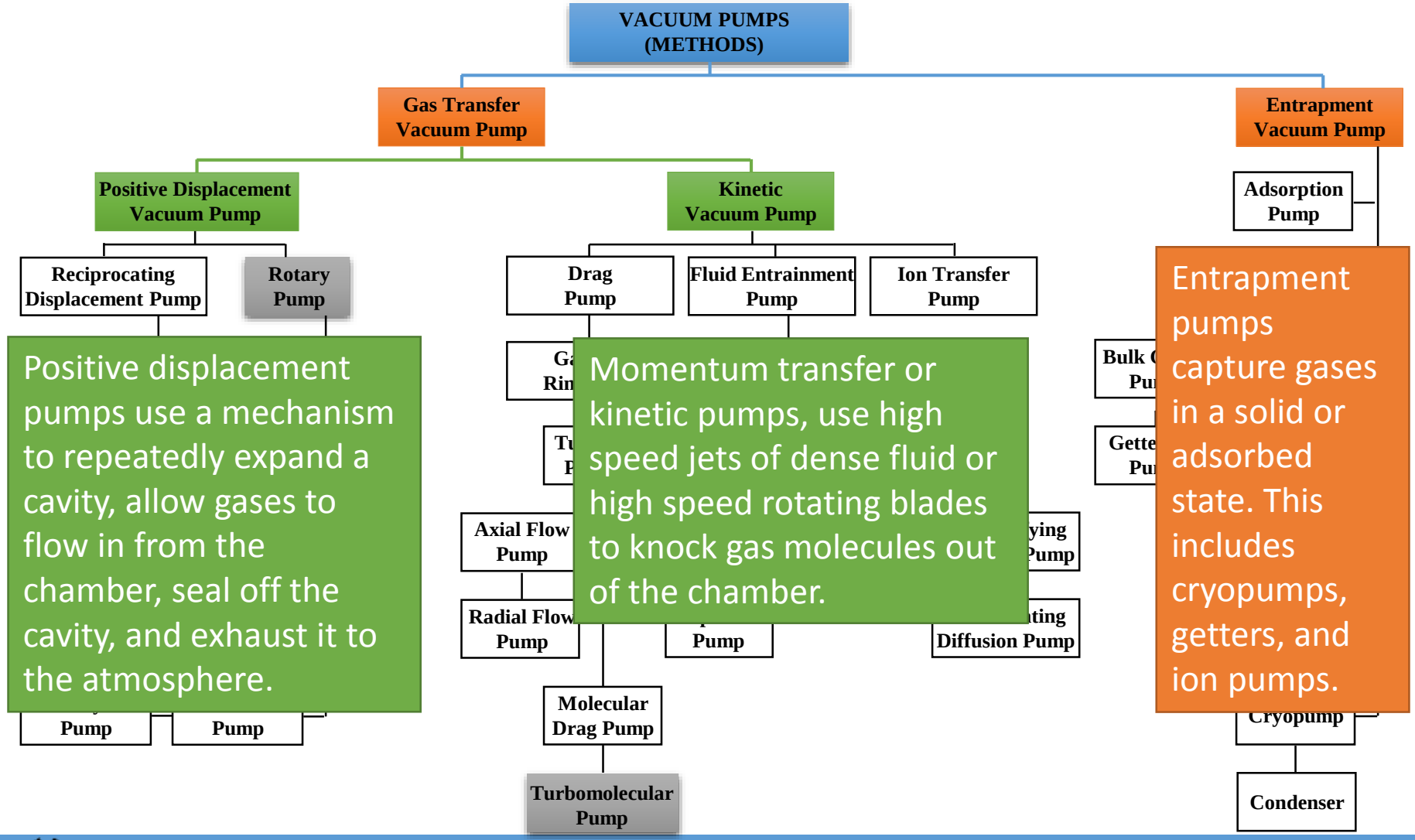
Atmospheric pressure: 760 Torr = 101.3 kPa = 1013 mbar

Pressure units:

Pa (SI unit),
mbar, bar,
Torr (USA)

Torr ~ mbar
1 bar = 10^5 Pa

How do we create low pressure?



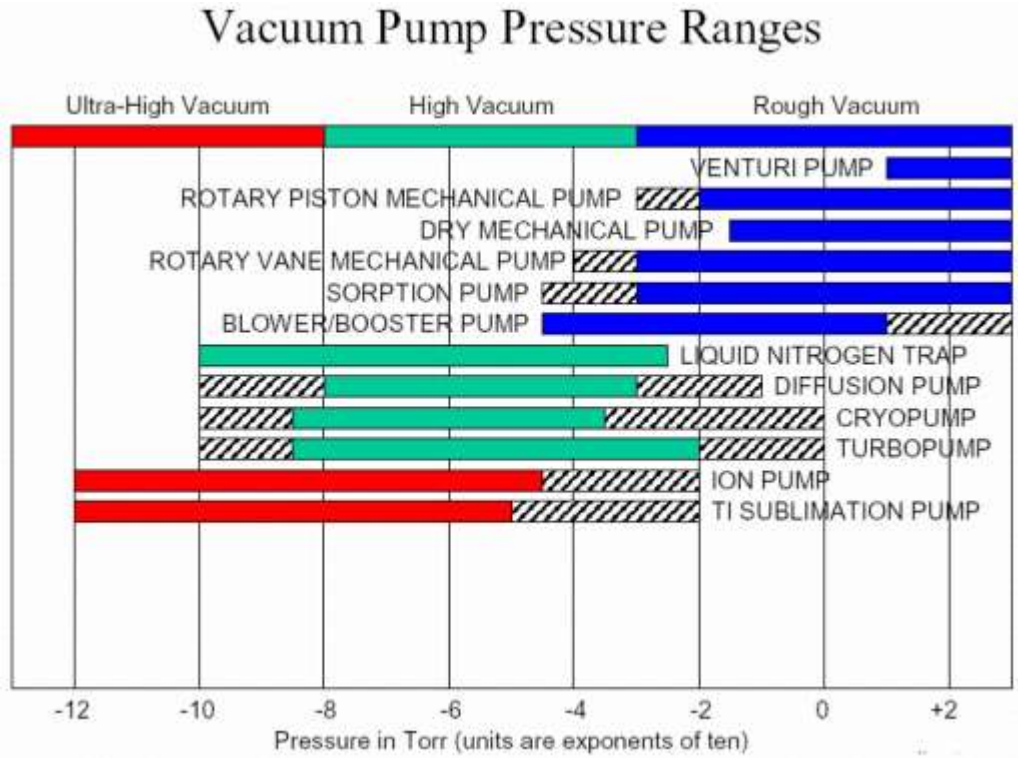
We need a pumping system!

Gas transfer vacuum pumps:

- Rotary pump
- Diffusion pump
- Turbomolecular pump

Entrapment vacuum pumps:

- Ion pump
- Cold trap



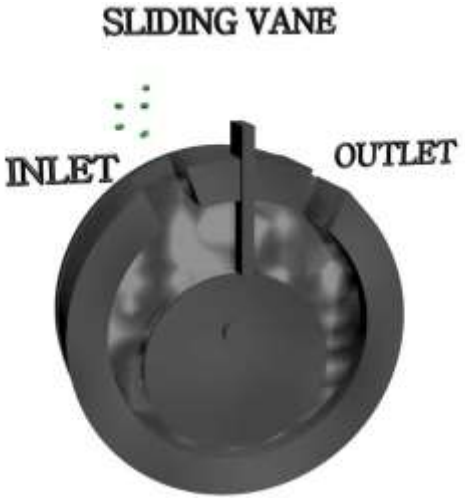
(Image: electronica.ugr.es)

Rotary Pump

Initial pump (sample exchange).

Good efficiency for high pressure.

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



(Video: YouTube)

Disadvantages:

- vibrations
- oil vapour
- maintenance

JXA-840A

JSM-7600F

Diffusion 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.

Disadvantages:

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

(Video: YouTube)

Figure removed for copyright reasons.

(Image: pchemlabs.com*)*

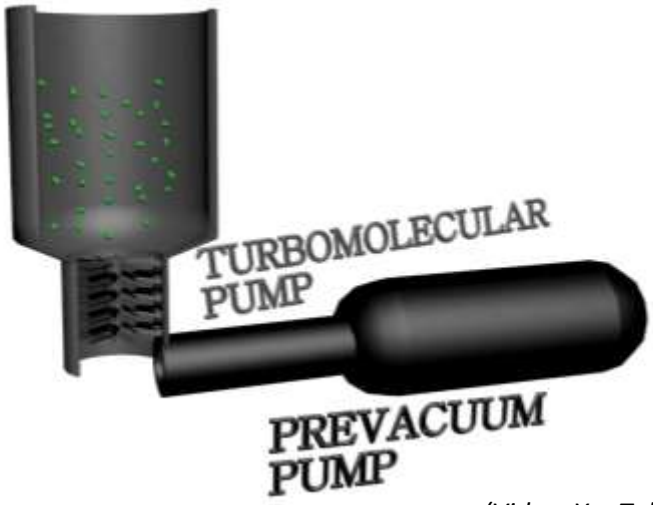
Turbomolecular Pump

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.



(Video: YouTube)

Figure removed for copyright reasons.

(Image: wikipedia)

Disadvantages:

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

Sputter Ion Pump

After the turbomolecular pump.

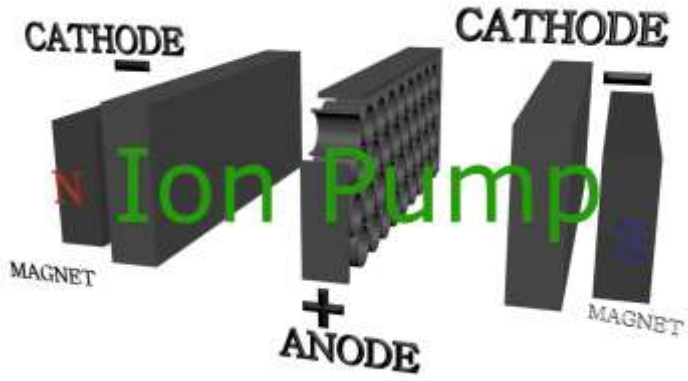
No moving parts.

Ions from gas molecules are pulled on the cathode.

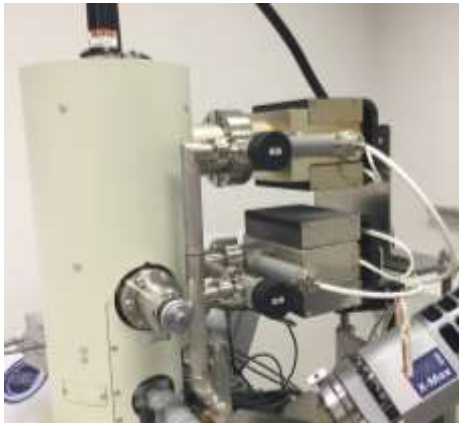
Cathode trapes the gas plus we get free e- and Ti ions.

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

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



(Video: YouTube)



JSM-7600F

Disadvantages:

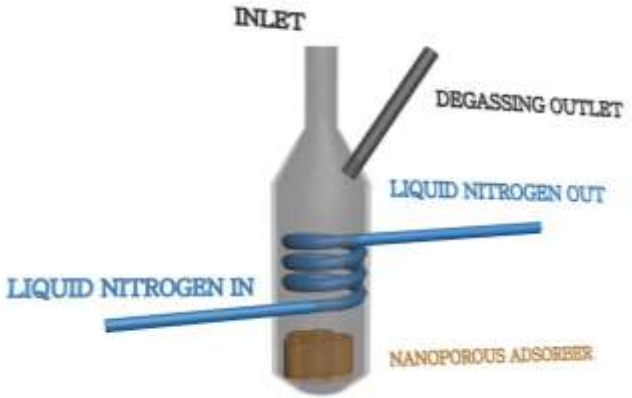
- not very efficient for water
- low capacity

Liquid nitrogen trap

Used to improve the vacuum in the chamber (depending on the sample).

Molecules are trapped on the surface (sorption).

Good for water.



(Video: YouTube)

JSM-7600F

Disadvantages:

- frequently degassed
- gasses not permanently removed

Why do we need vacuum in EM?

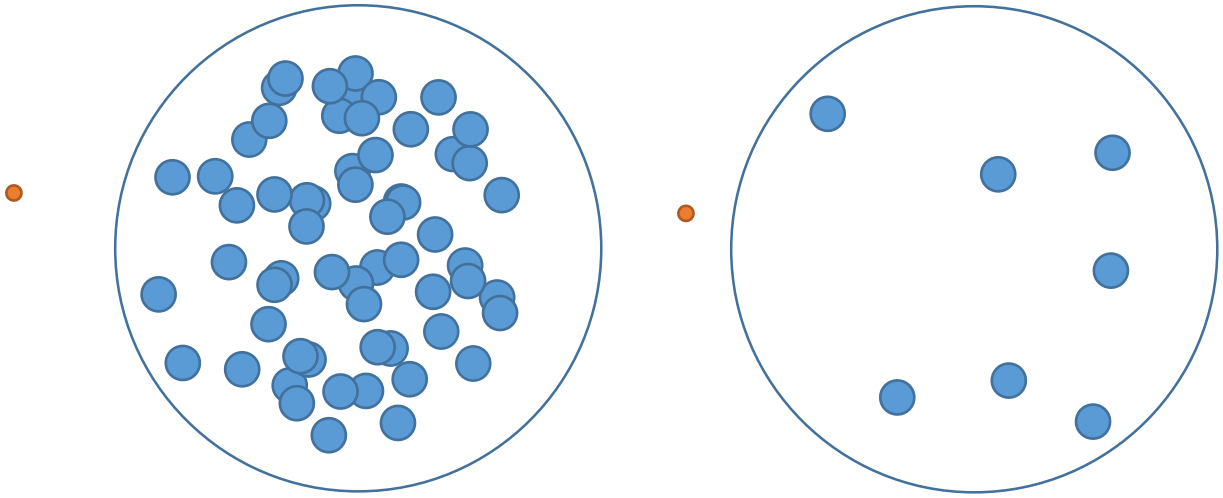
Why do we need vacuum in EM?

To move a particle in a (straight) line over a **large distance**.

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

To provide a **clean surface**.

To prevent **beam induced** chemical reactions.



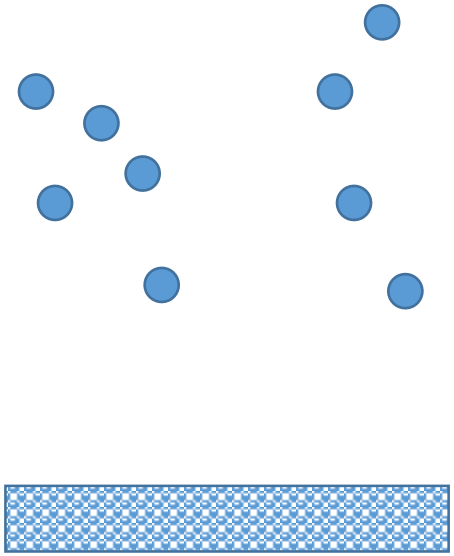
Why do we need vacuum in EM?

To move a particle in a (straight) line over a **large distance**.

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

To provide a **clean surface**.

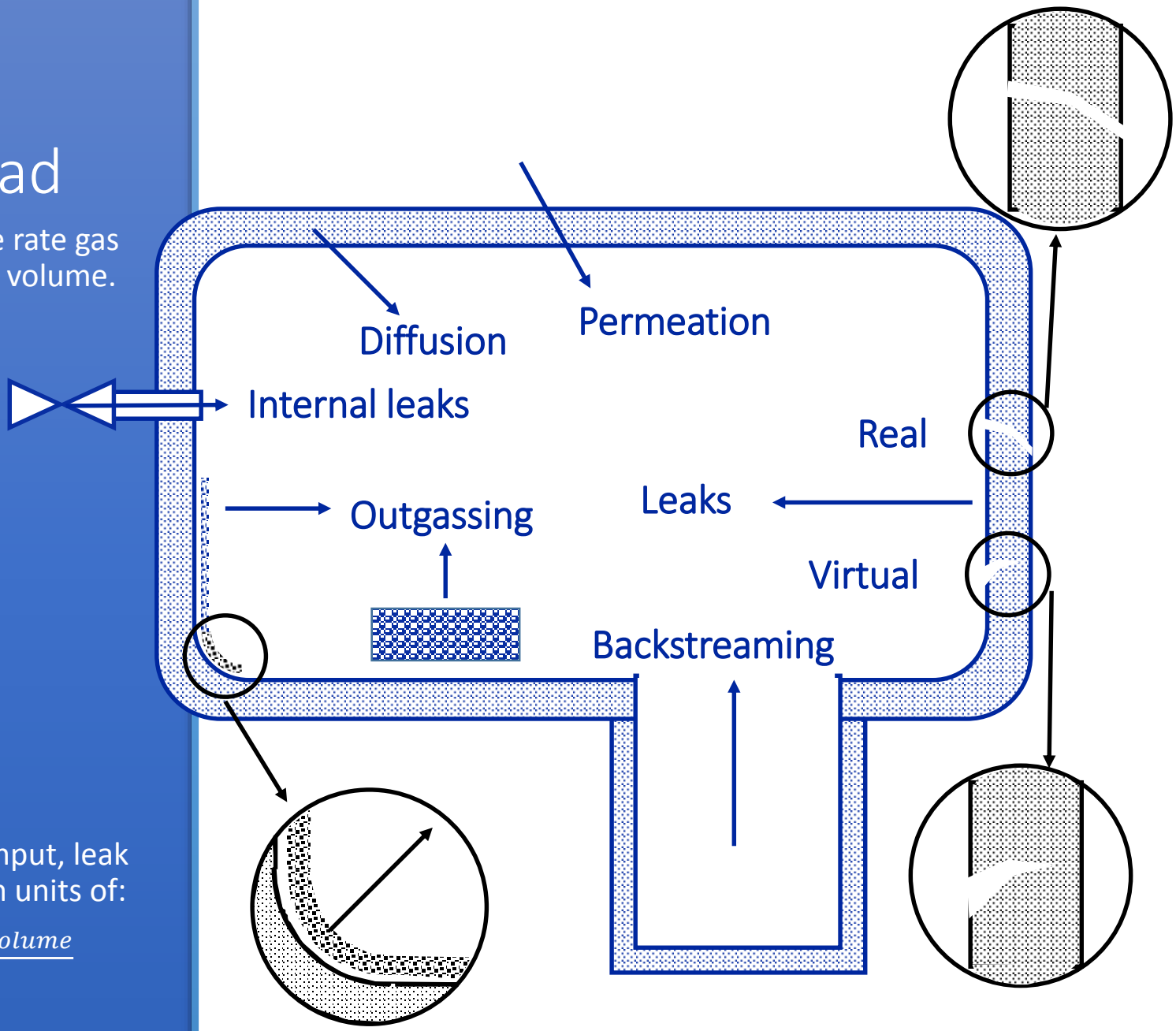
To prevent **beam induced** chemical reactions.



Pressure (mbar)	Time to form a monolayer (s)
10^{-3}	0,002 s = 2 ms
10^{-6}	2 s
10^{-9}	2000 s = 33 min

Gas Load

The gas load is the rate gas enters the system volume.



Q (gas load, throughput, leak rate) is expressed in units of:

$$Q = \frac{pressure \cdot volume}{time}$$

Sample preparation for vacuum

- Suitable for vacuum
  - No water, dry,
  - Very low rates of outgassing
- Samples have to be well-attached
  - Particles can damage the pumping system
  - Magnetic particles can damage the SEM column

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 samples
  - Longer pumping time, outgassing,
  - Gases can be released from porous materials or cracks
  - If magnetic one can damage the SEM
- Small samples
  - Can fly from the holder, problems on the column, apertures, electron source
  - If magnetic one can damage the SEM!
- Use gloves and clean everything
  - Traces of lubricants and residues from machining may be present on surfaces
  - Hand fat…

Društvo za vakuumsko tehniko Slovenije DVTS

Learn more on:

Vacuum Fittings and Accessories,

Different pumps and gauges,

Physics of vacuum,

The use of vacuum,

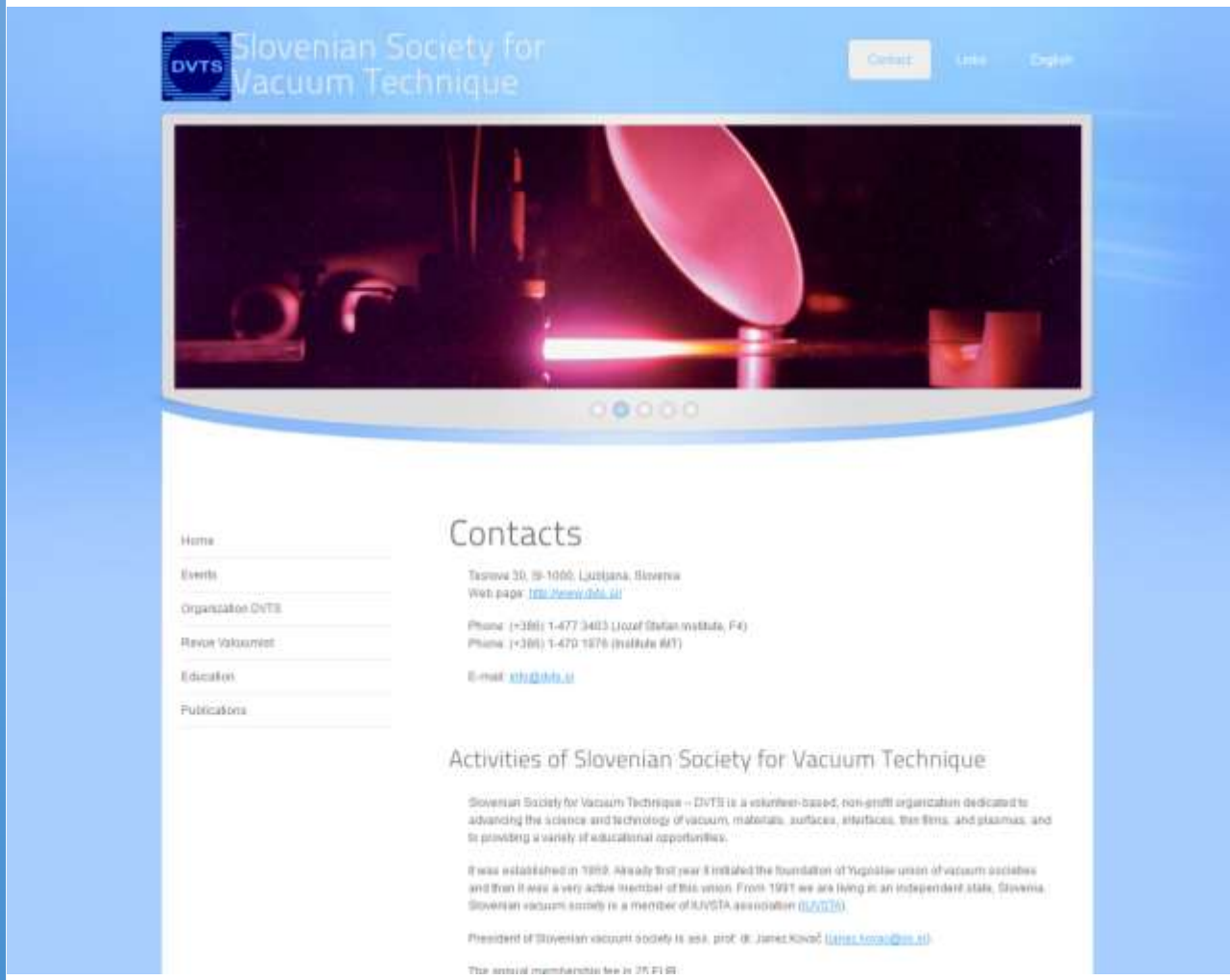
Symbols,

Leak detection technique,

…

The vacuum coarse will be organized in 2018, contact:

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




QUESTIONS?
COMMENTS?
CONCERNS?