Low Vacuum Scanning Electron Microscopy and Microanalysis

Principles and Practice of Variable Pressure/Environmental Scanning Electron Microscopy (VP-ESEM), Debbie J Stokes,
John Wiley&Sons 2008
Several graphs courtesy of prof. Bradley Thiel

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- Conventional scanning electron microscopy (SEM) is a widely used as an analytical tool. However, there are several limitations on the type of samples, which may be observed.
  - When a specimen consists of a non-conductive material, or not properly grounded to the specimen stub, charging occurs. **Charging is the build-up of an excess of electrons on the surface of the specimen, which causes many undesirable artefacts.** Both secondary and backscattered electrons utilised for SEM observations are significantly affected.
- **The elimination of specimen charging is accomplished in the LowVac/ESEM scanning electron microscope by the introduction of a gas, water, nitrogen or air into the specimen chamber.**

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Outline

- Introduction to Variable Pressure (Environmental) Scanning Electron Microscopy
- Image formation in VP-SEM
- X-ray Microanalysis in VP-SEM
- Variable pressure method
- EBSD in VP-SEM
- Conclusions

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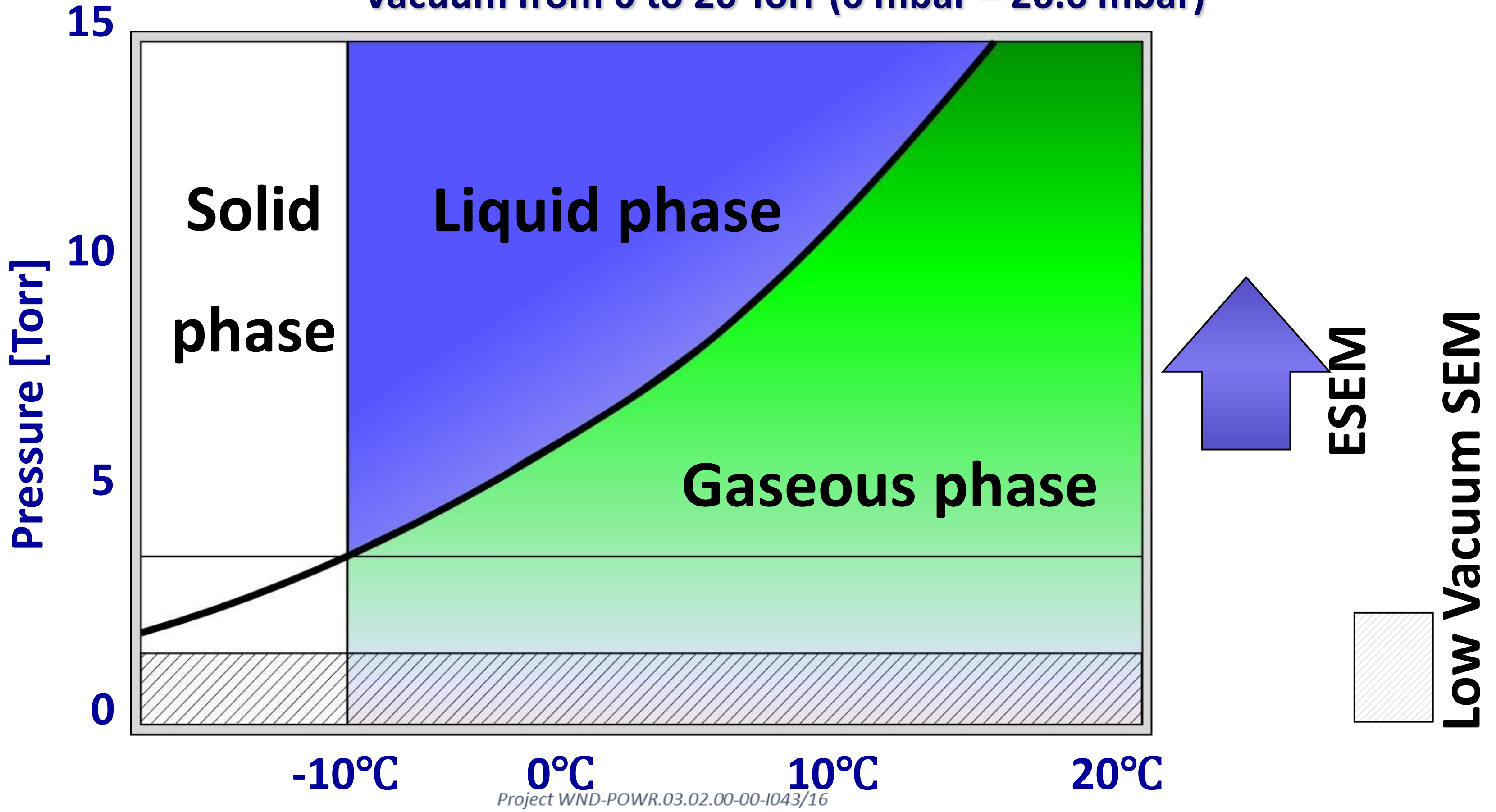
- **LV-SEM – Low Vacuum Scanning Electron Microscopy**

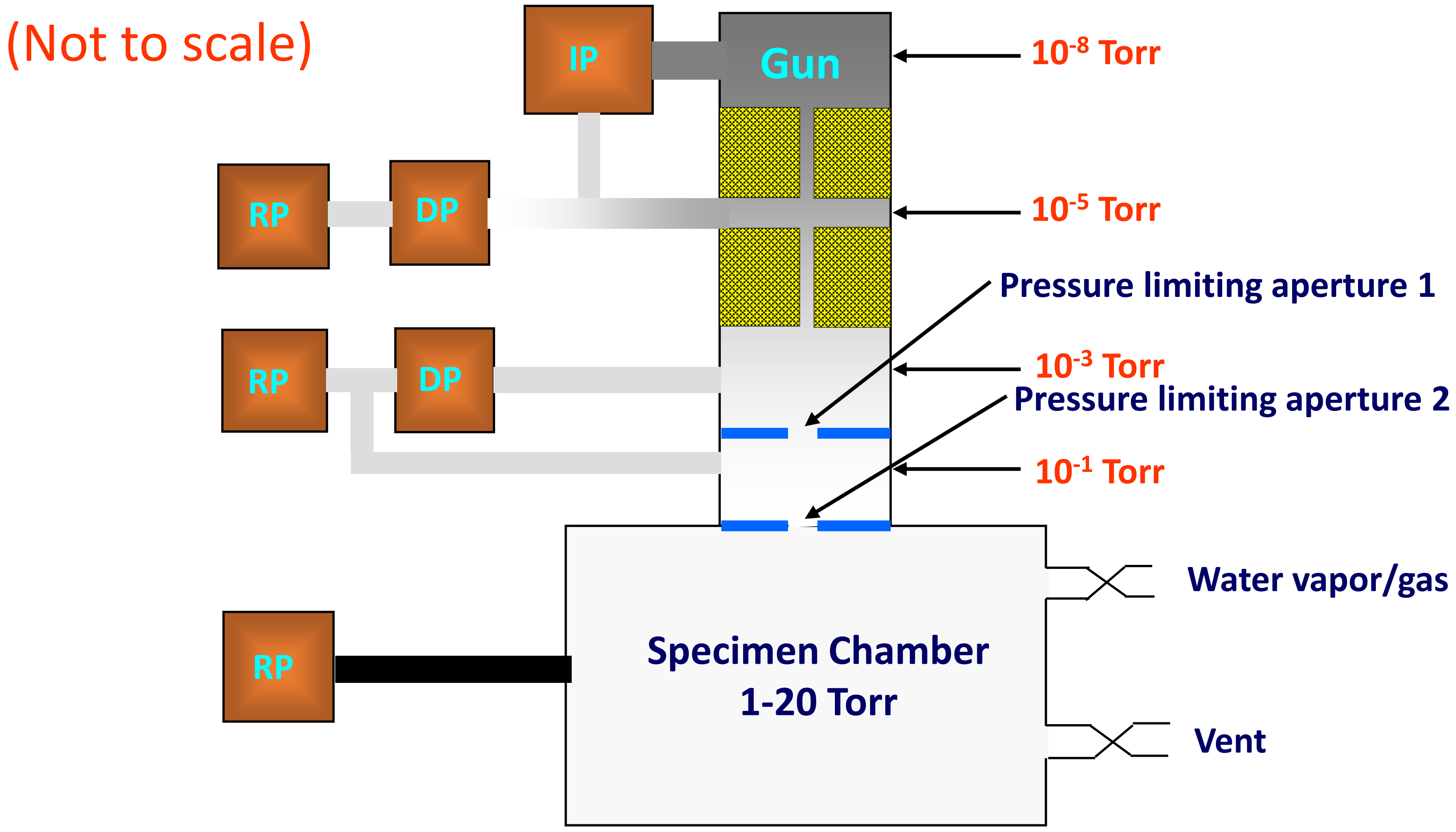
Vacuum from 0 to 1 Torr (0 mbar – 1.33 mbar)

- **VP-SEM or Environmental Scanning Electron Microscopy**

Vacuum from 0 to 20 Torr (0 mbar – 26.6 mbar)



Variable Pressure Scanning Electron Microscope



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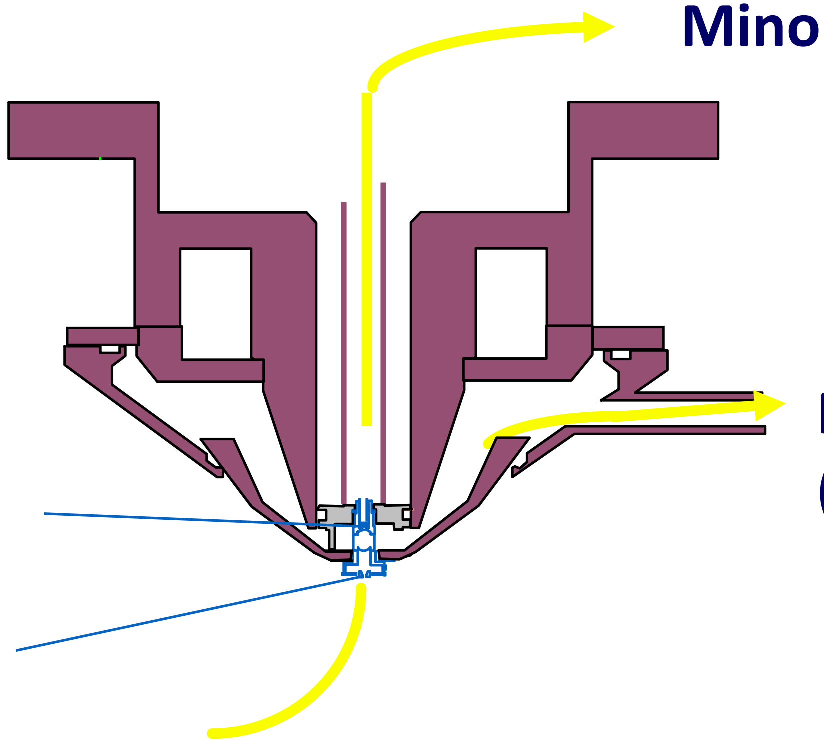
Pumping system of ESEM

Emission area: min. 10^{-5} Torr



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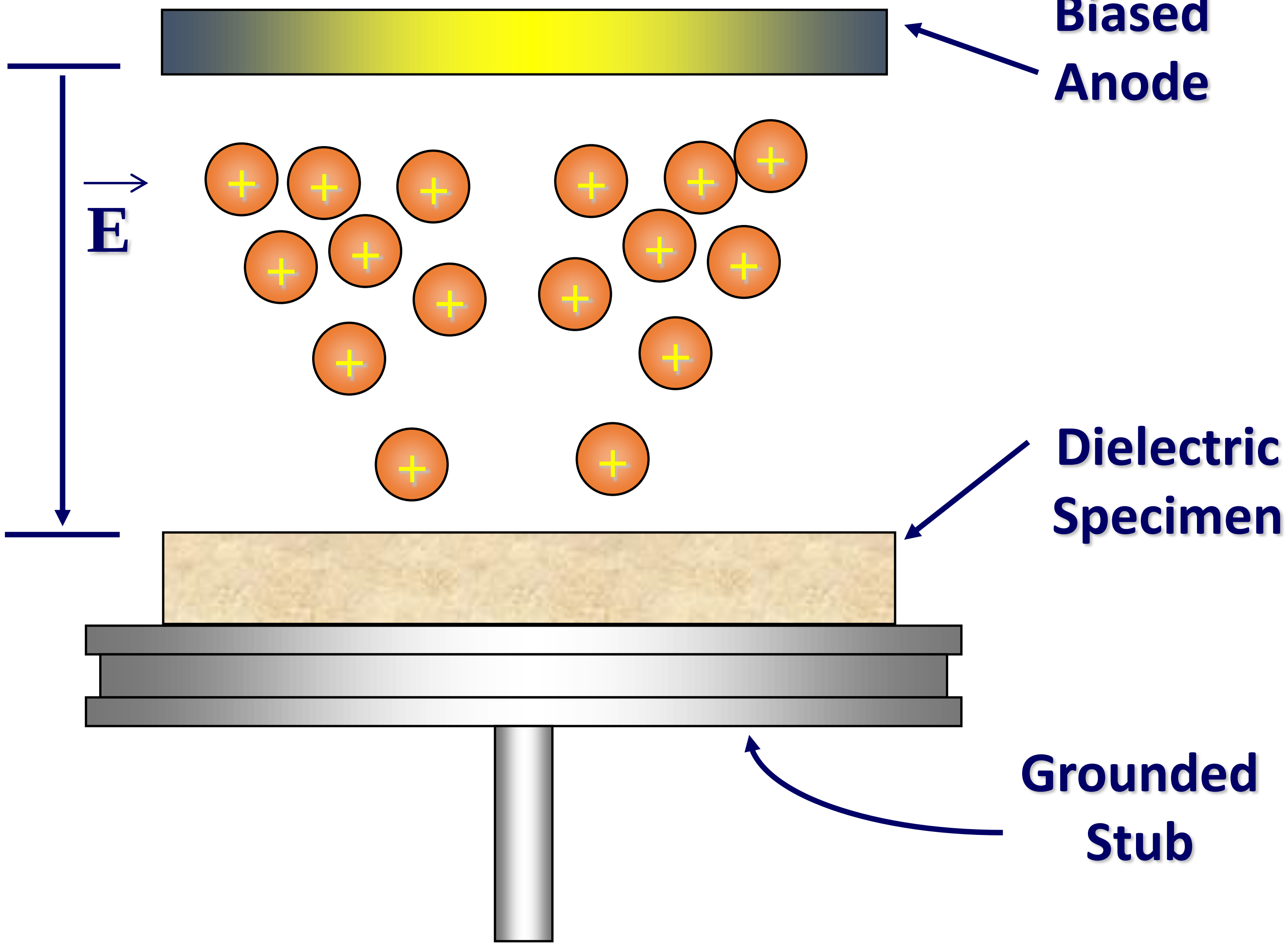
Cascade Amplification



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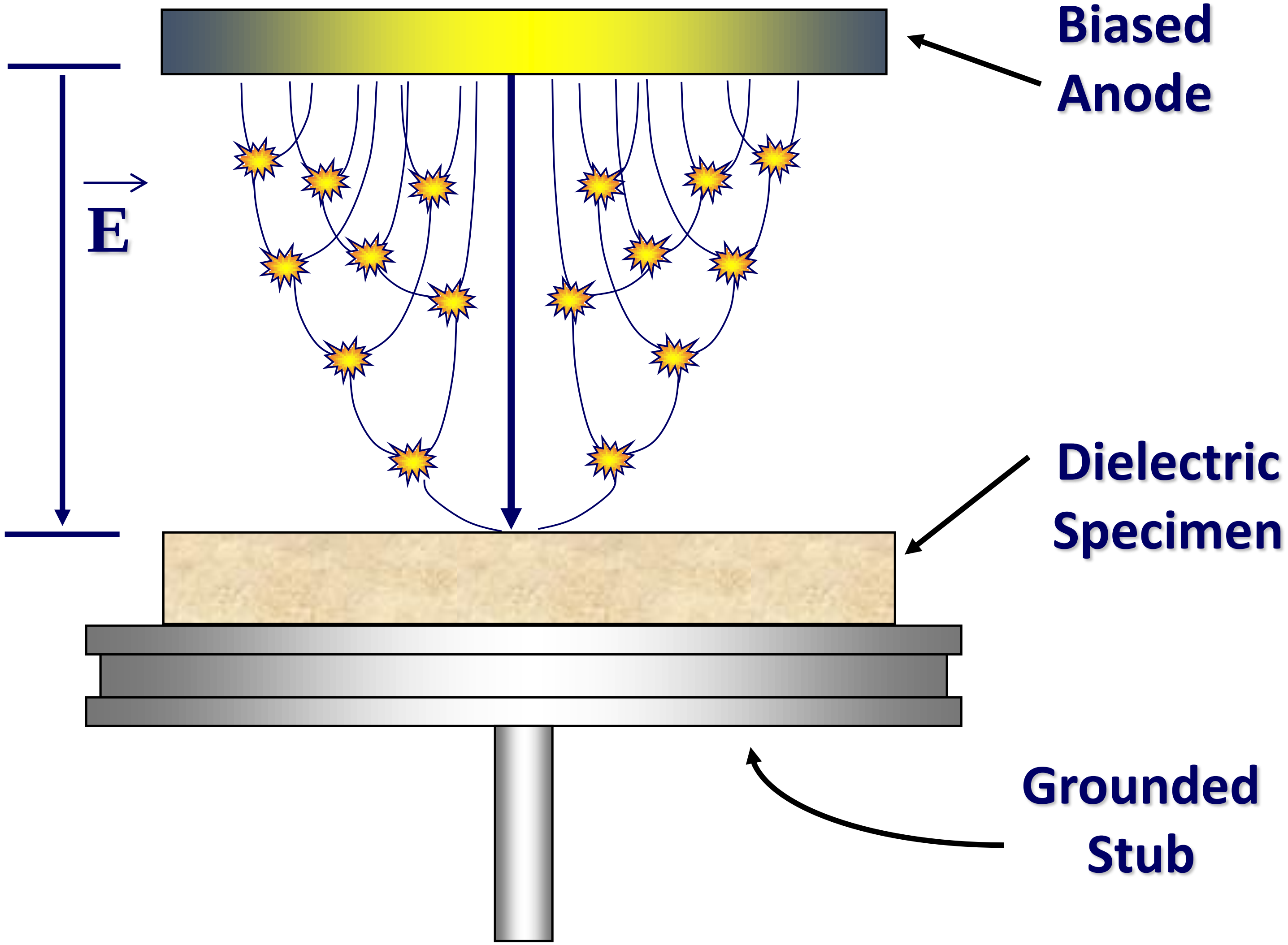
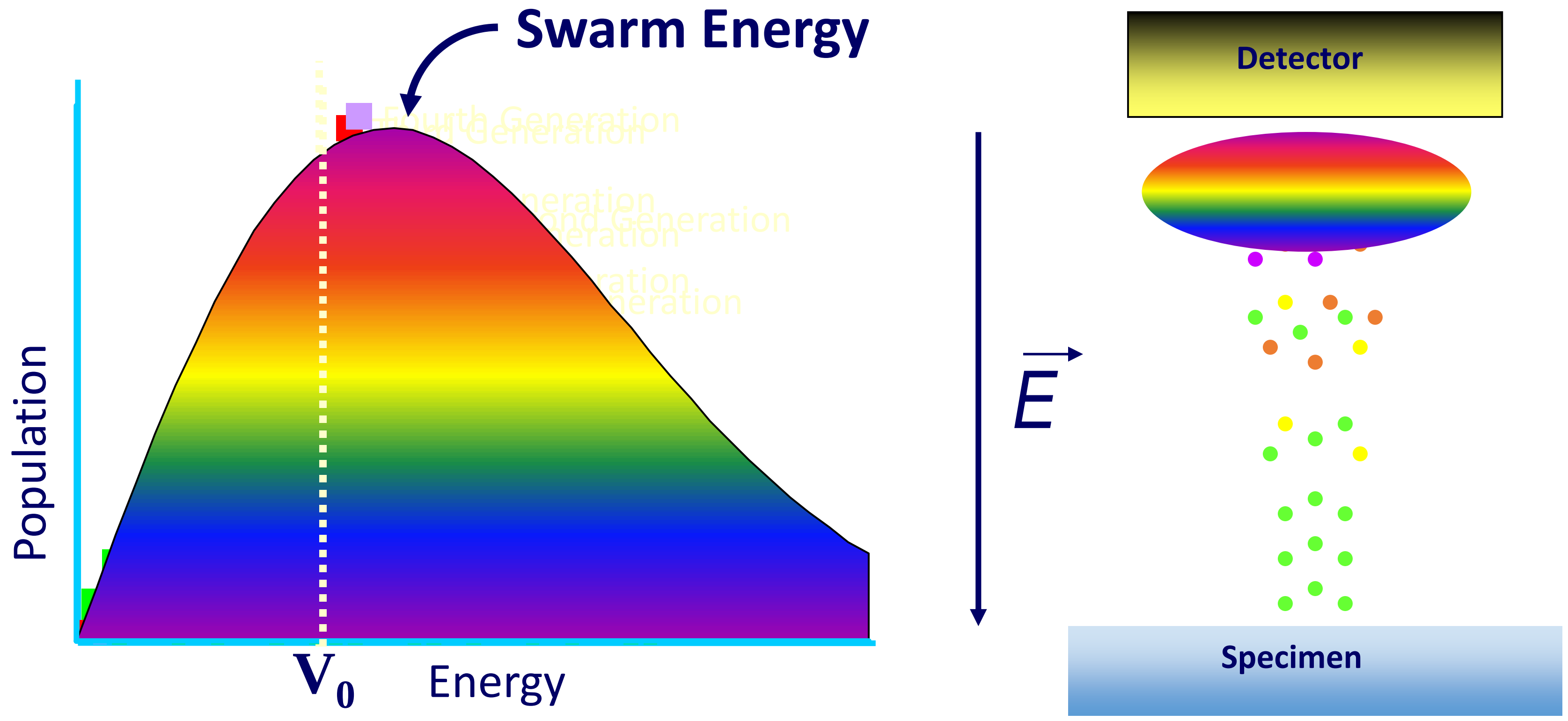
Cascade Amplification

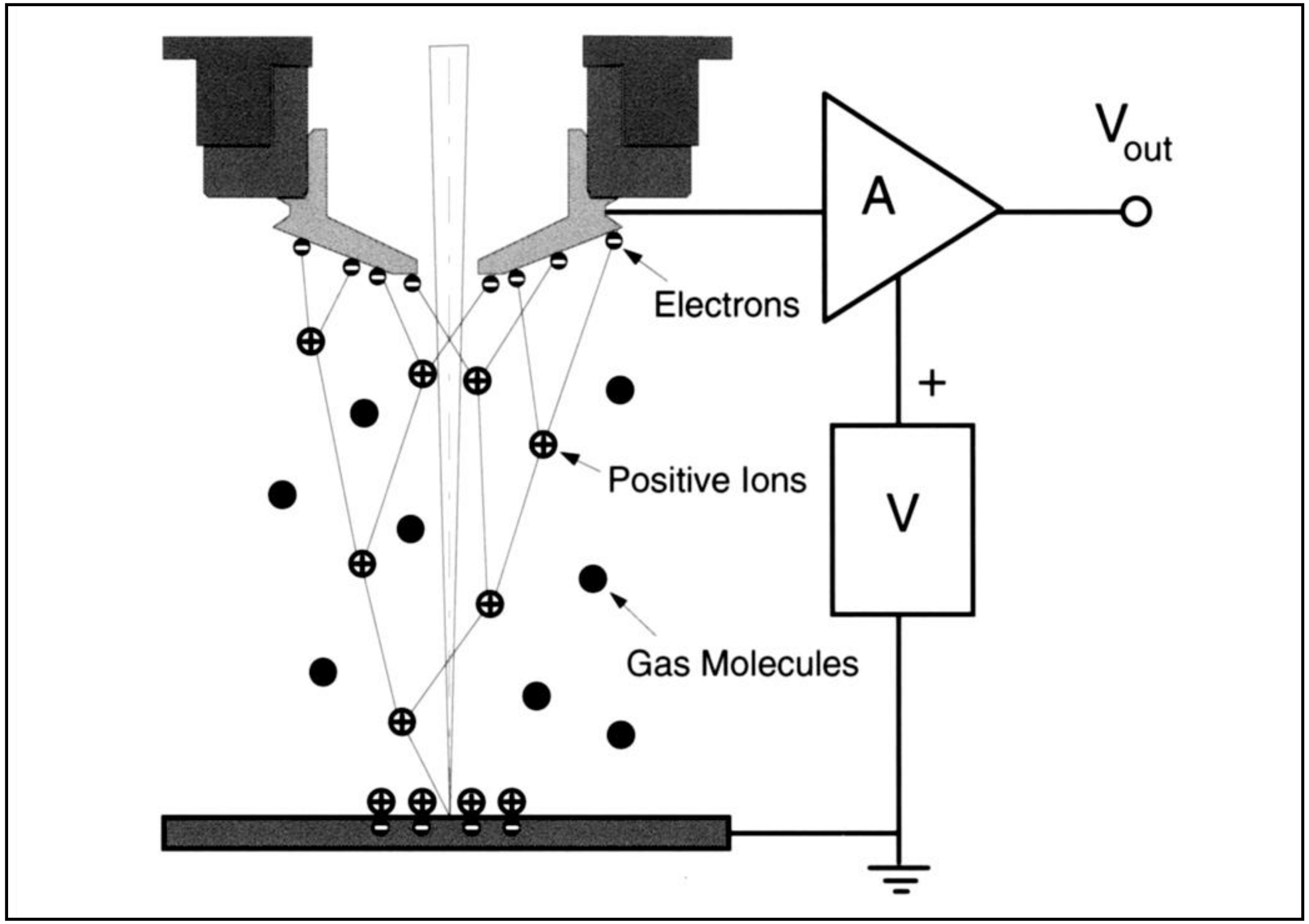
The SEs collide with the gas molecules in the chamber and create further electrons which are also accelerated in the electric field: a cascade or avalanche promotes amplification



SE Amplification



A simplified schematic diagram of gaseous signal amplification via ionisation



- **The primary electron beam impinges on the sample, leading to the production of backscattered and secondary electrons.**
- **The electric field between the positively biased anode and the grounded specimen stage accelerates secondary electrons towards the anode.**
- **Secondary electrons collide with and ionise gas molecules in their path and amplify the secondary signal.**
- **Positive ions drift towards the sample surface to compensate negative charge build up.**

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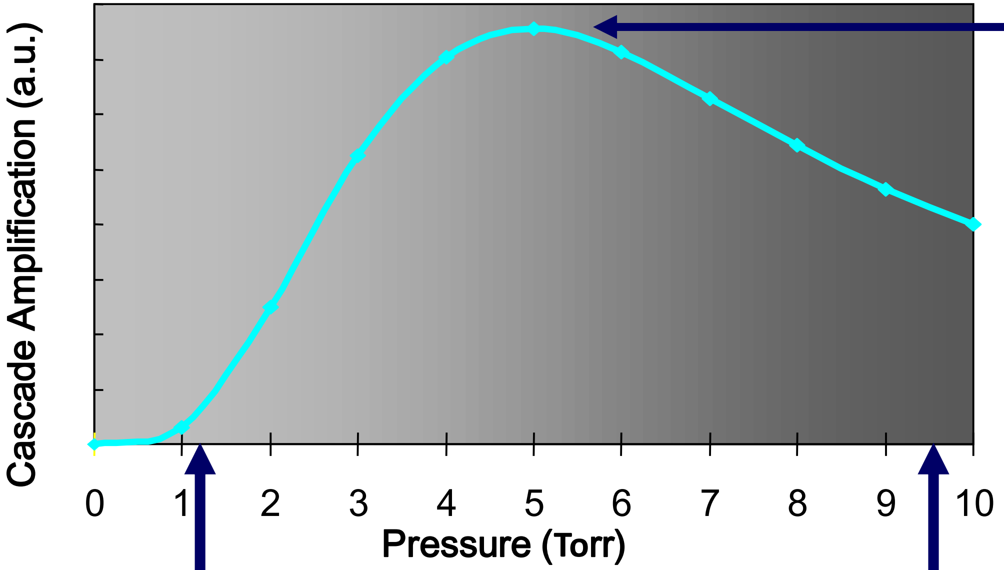
General Shape of Amplification Curve



The most efficient amplification

If the pressure is too small (concentration of gas molecules is small too), MFP is large and amplification events are few and the secondary electron signal is weak

If the pressure is too high (concentration of gas molecules is large), MFP is short and significant fraction of secondary electrons is scattered before they acquire sufficient energy to cause ionisation

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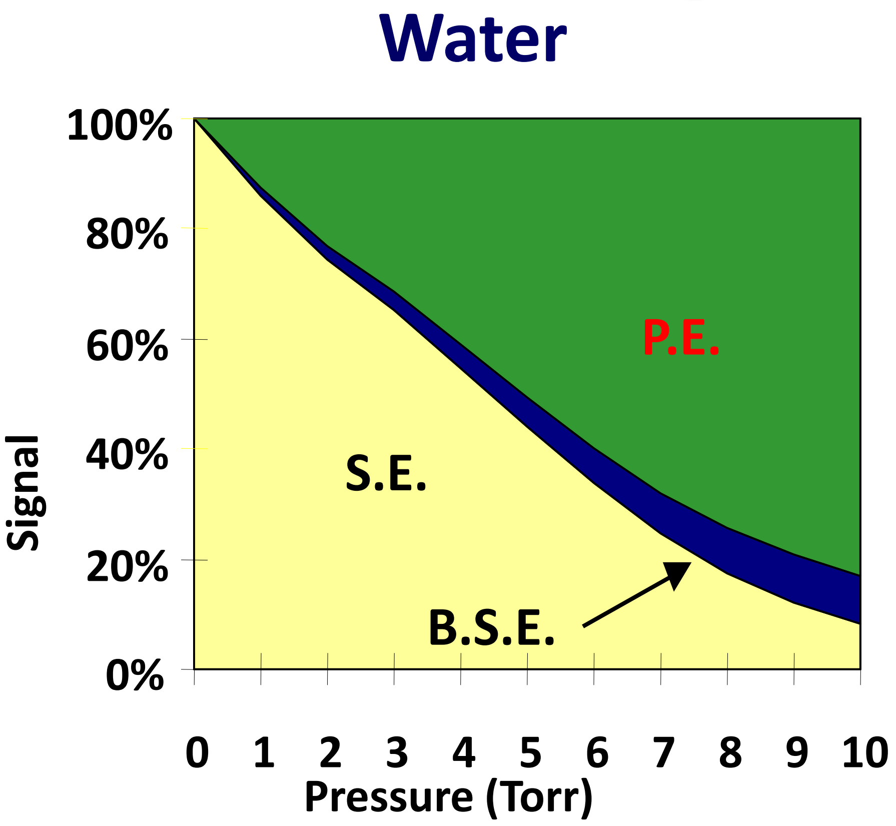
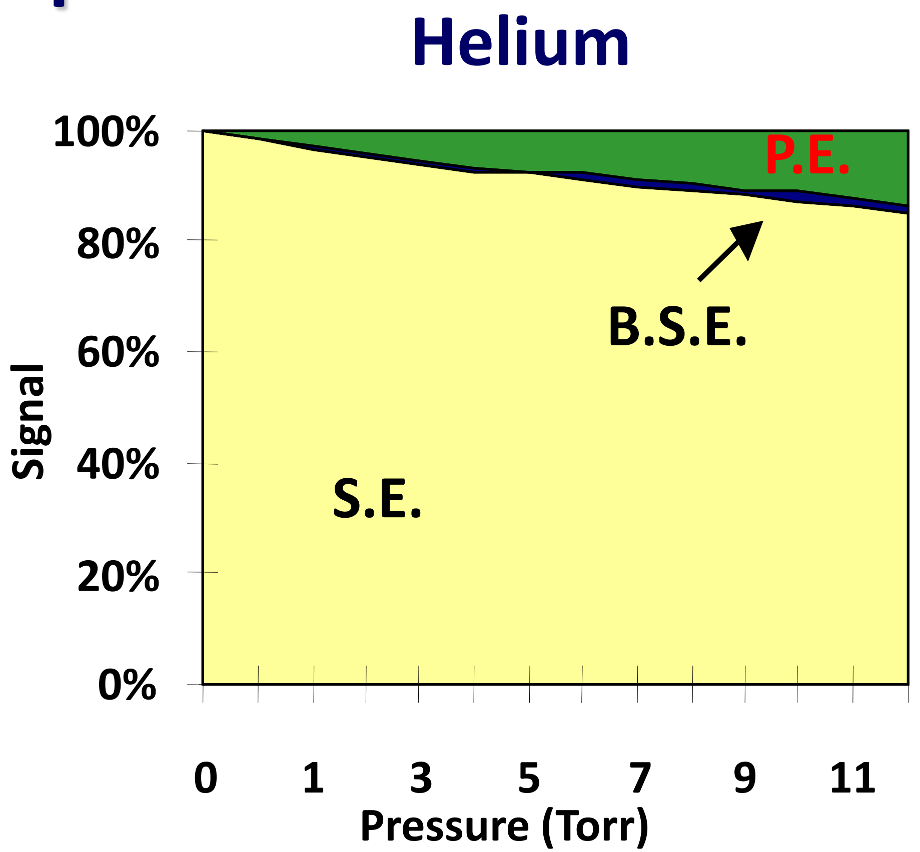
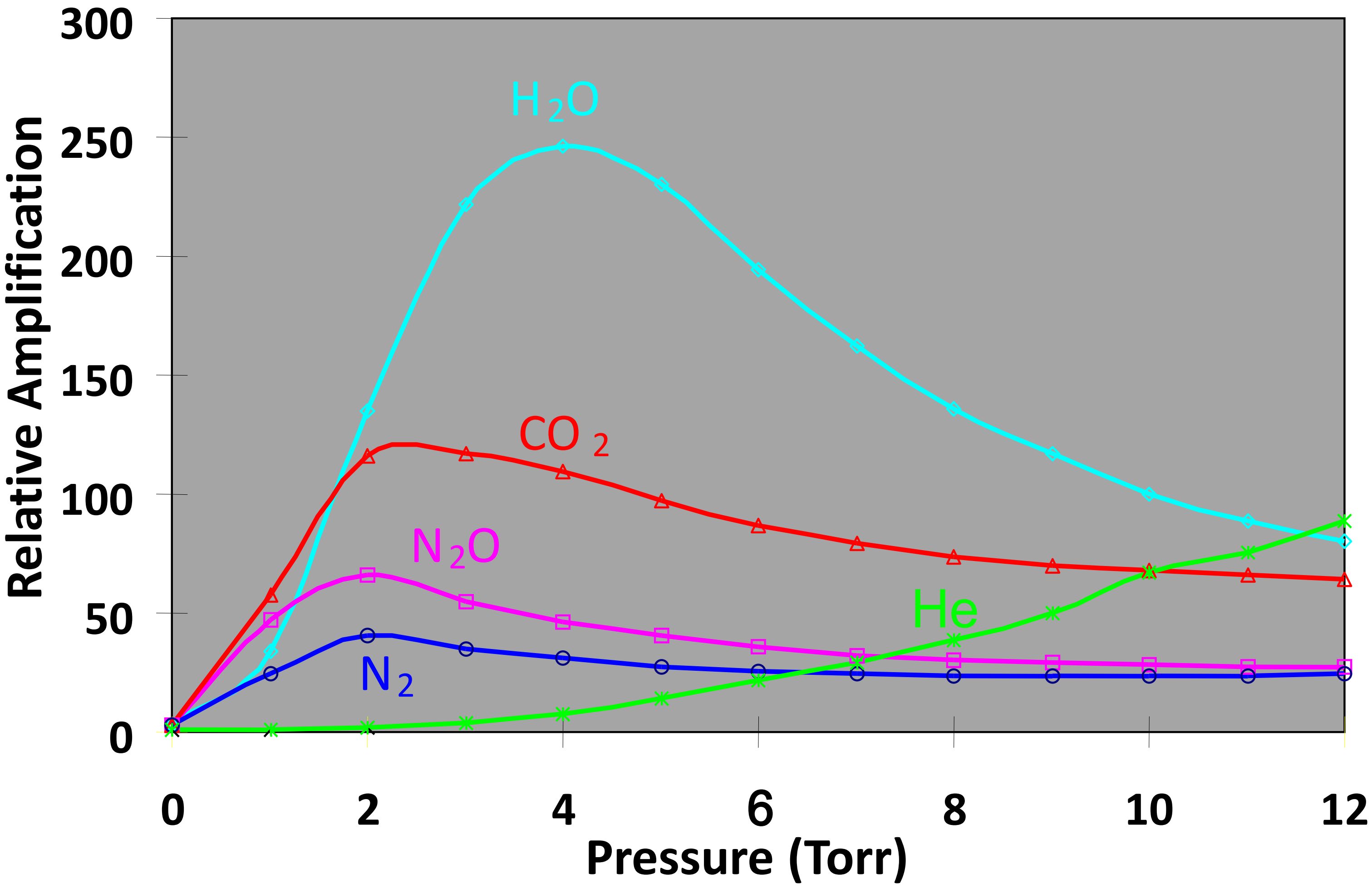
Signal Components





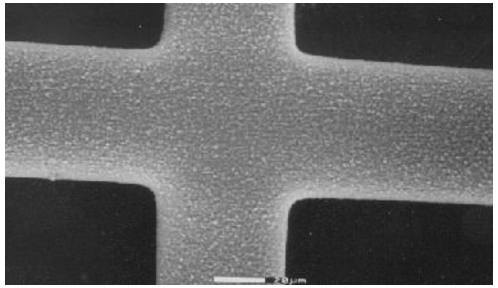
PE – signal due to ionisation from primary electrons
BSE – signal due to ionisation from backscatter electrons
SE – signal due to ionisation from secondary electrons (most desired!)

Alternative Gases

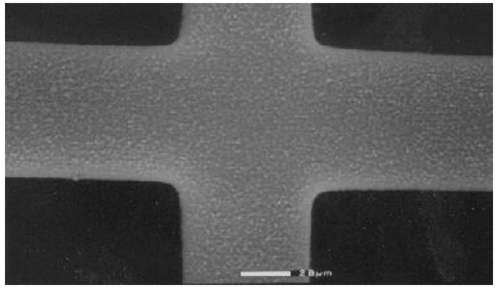


Copper grid on Carbon

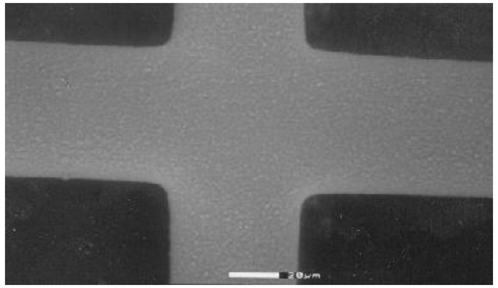
H_2O



4 torr

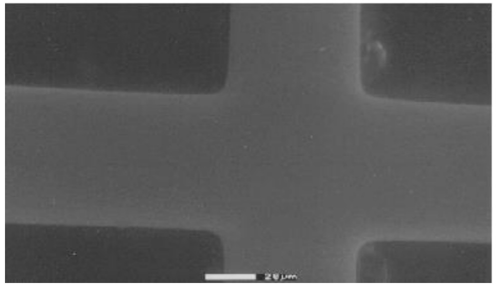


6 torr

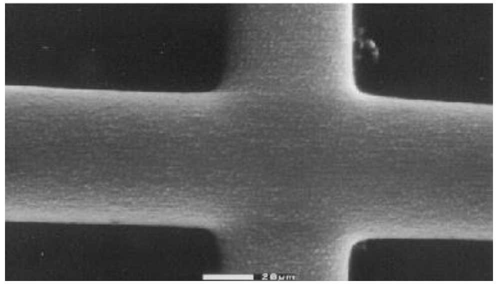


8 torr

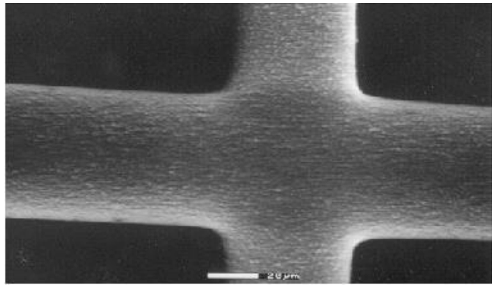
He



2 torr



5 torr



12 torr

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$$I_c = I_o \exp^{\alpha d}\left\{\delta + \frac{S_{PE}}{\alpha d} + \eta \frac{S_{BSE}}{\alpha d}\right\}$$

I_c – amplified gaseous electron current
I_o – primary beam current
S_{PE} – ionisation efficiency of primary electrons
S_{BSE} – ionisation efficiency of backscatter electrons
δ – secondary electron coefficient
η - backscatter electron coefficient
α – ionisation efficiency of the gas
d – sample-anode gap

The factor α is called Townsend's first ionisation coefficient

$$\alpha = APe^{-BPd/V_0}$$

P – pressure, V_0 – anode bias, A & B - gas specific constants

$$G = e^{\alpha d}$$

Signal gain G is a function of the ionisation coefficient α and the specimen-anode distance d

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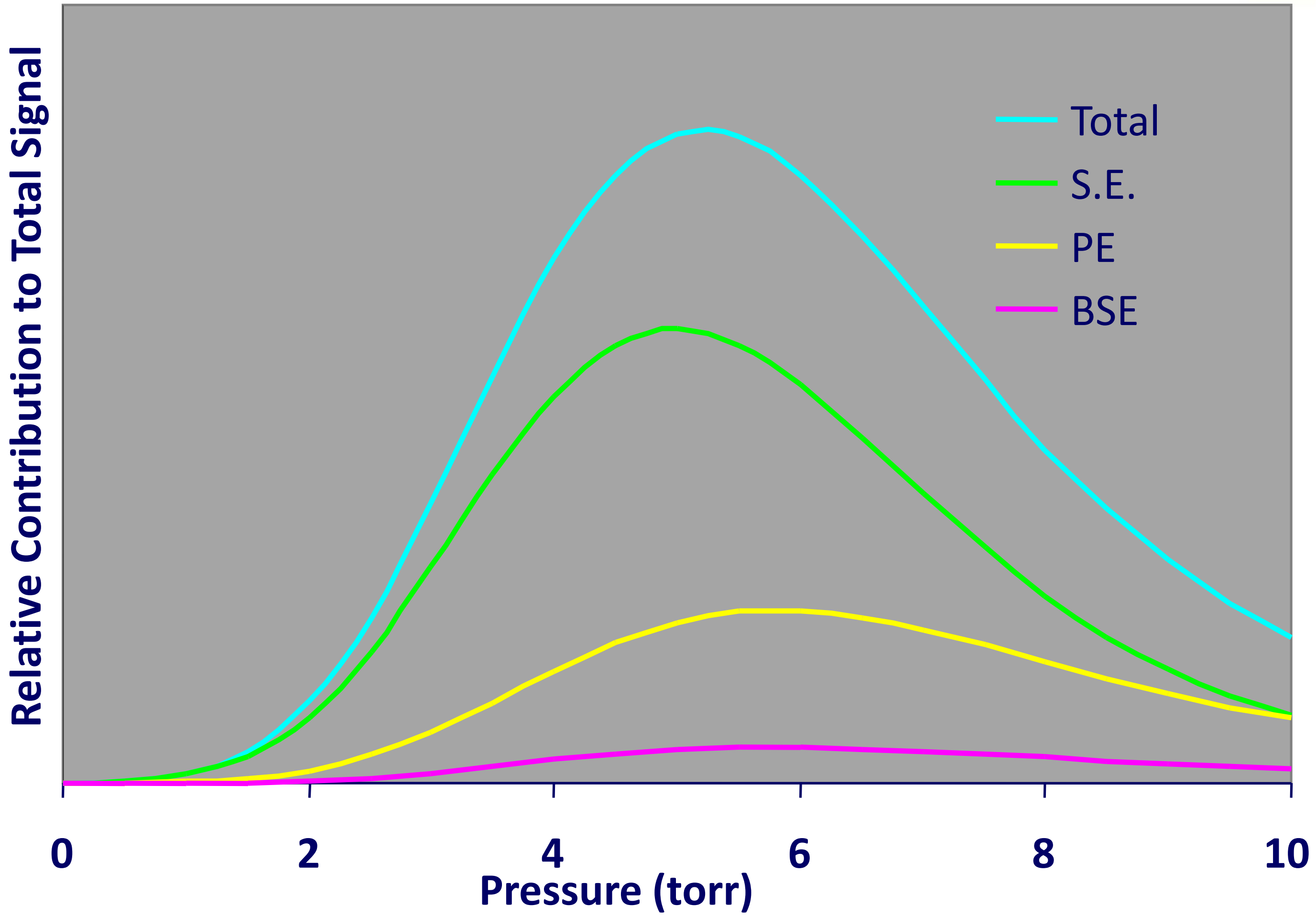

Graph of signal contributions as a function of chamber gas pressure (for water vapour). The maximum total amplification occurs at slightly higher pressure than the secondary electron maximum. At high pressures, the signal is dominated by background signals such as ionisation of gas molecules by primary electrons

Cascade Signal Components

The cascade signal is then composed of:

- **Amplified SE**
- **BSE derived signal**
- **PE derived signal**
- **All of the above from the "skirt region"**
- **Detector noise**

Of these, only the first is desired!

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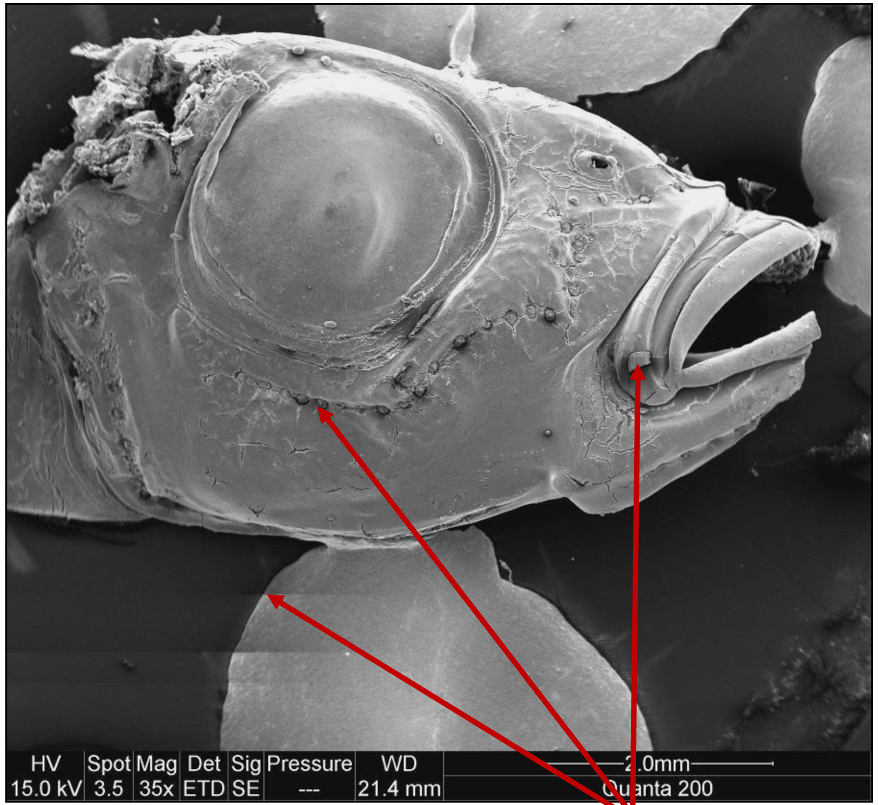
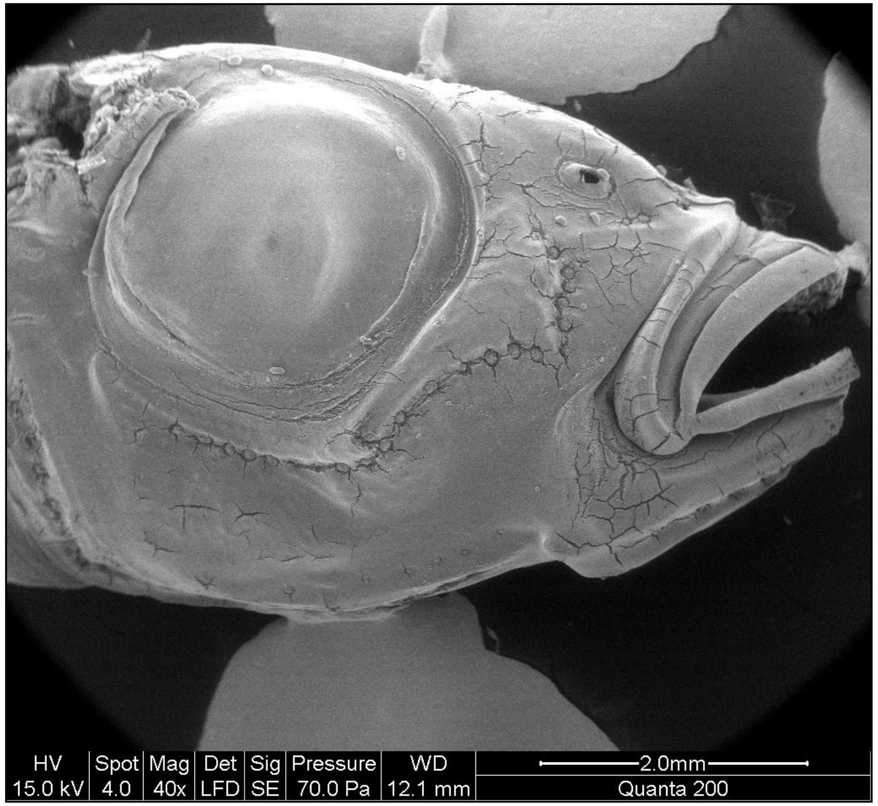
High vacuum versus low vacuum mode



SE images of Au coated Trich:
despite the coating, the image still shows some charging effects



SE image of Au coated Trich made in low vacuum mode (without charging)

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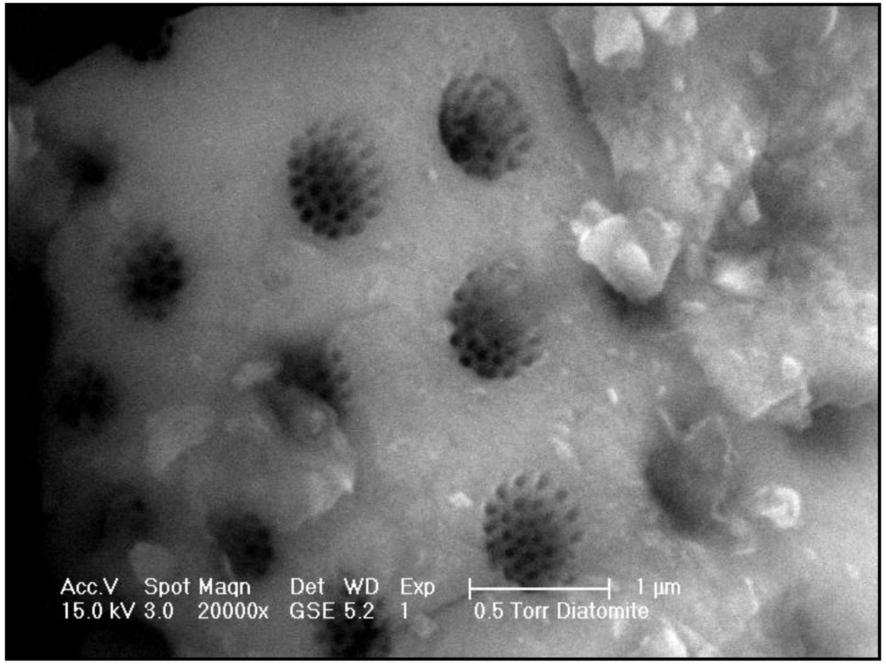
Microstructure

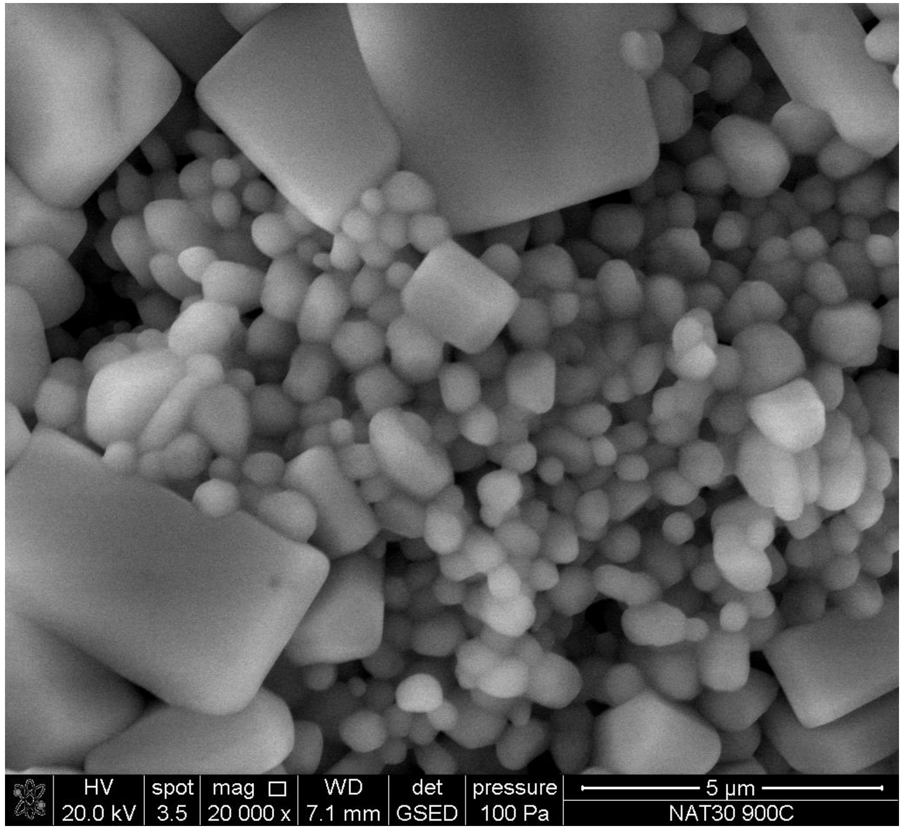
Diatomite

(a powdery mineral composed of the fossilised skeletal remains of microscopic single-celled aquatic plants called diatoms)



E-SEM with W hair-pin filament

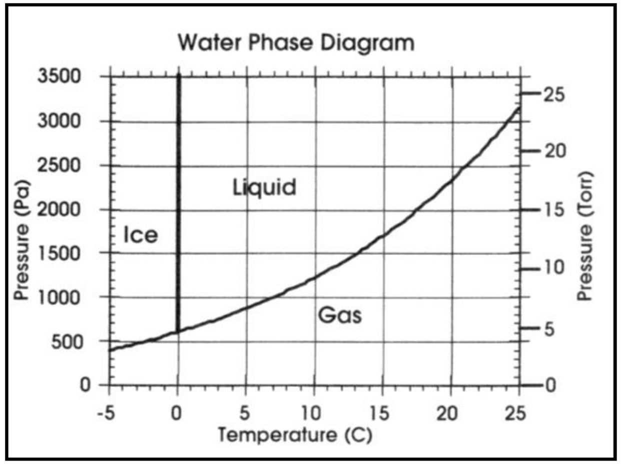
Natural HAp/bioglass composite

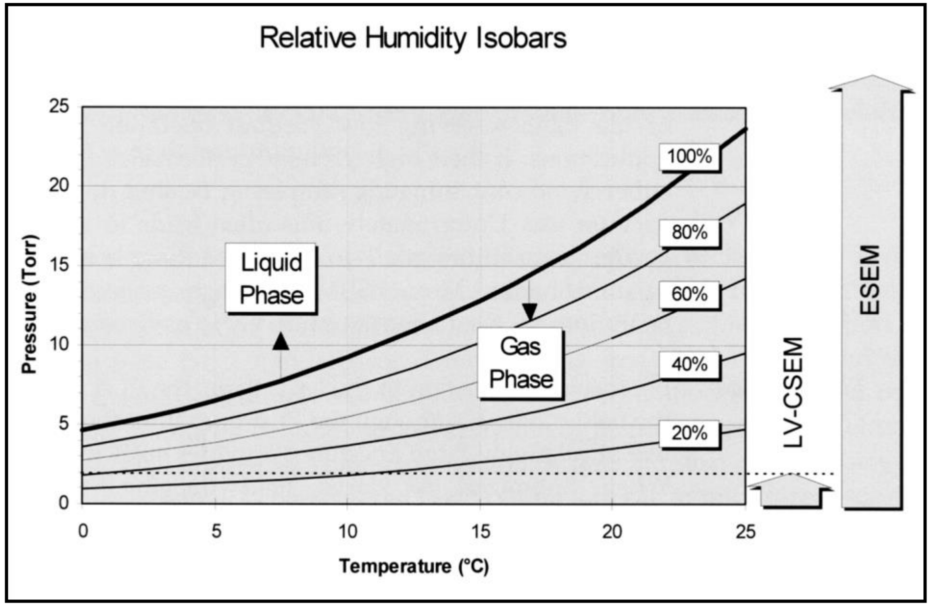


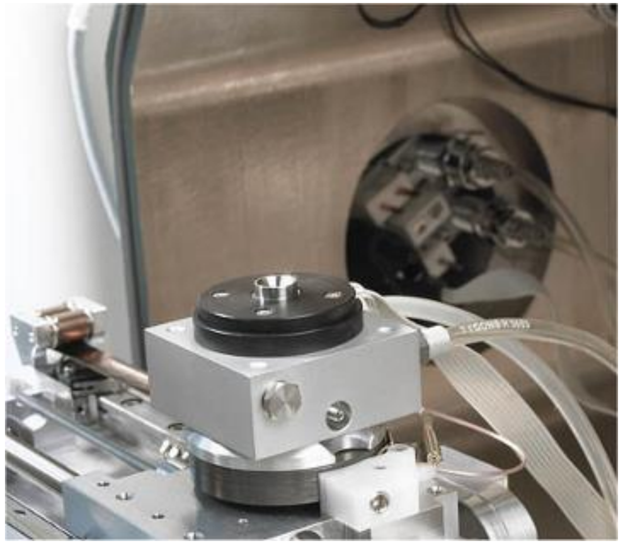
E-SEM with Shottky FEG

The minimum pressure that can sustain water in the liquid phase is about 4.6 Torr at 0°C.

Higher temperatures require higher pressures







To maintain a sample in its natural hydrated state it is necessary to drop the temperature below room temperature,

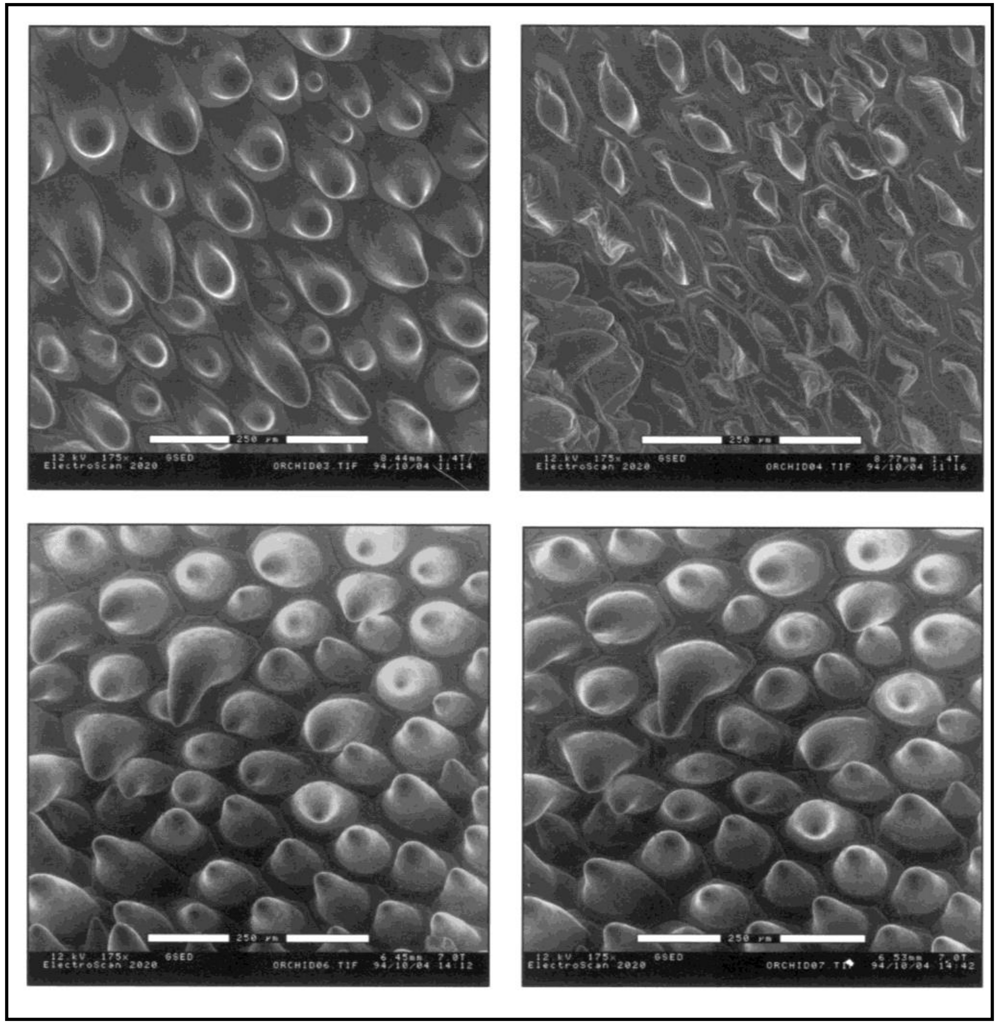
Peltier cooling stage -25°C to +50°C

Hydrated sample orchid petal

before 2 min. exposure | after 2 min. exposure

1.4 Torr H_2O vapor at 6°C

dehydration is obvious



7 Torr H_2O vapor at 6°C

H_2O environment becomes saturated and dehydration does not occur

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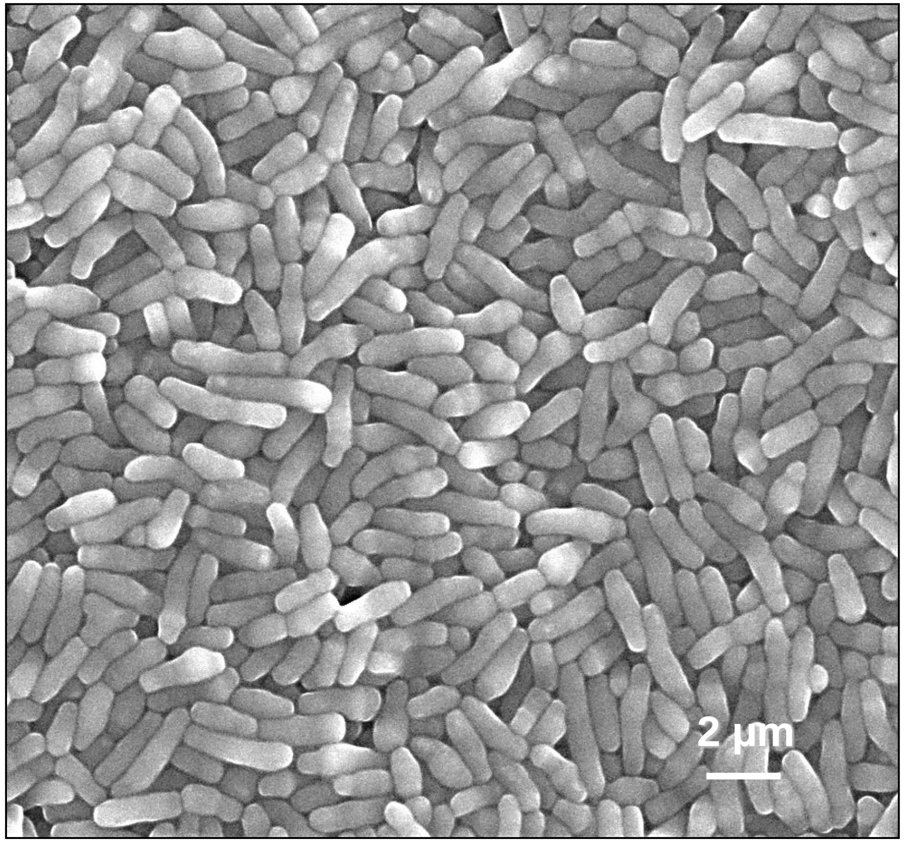
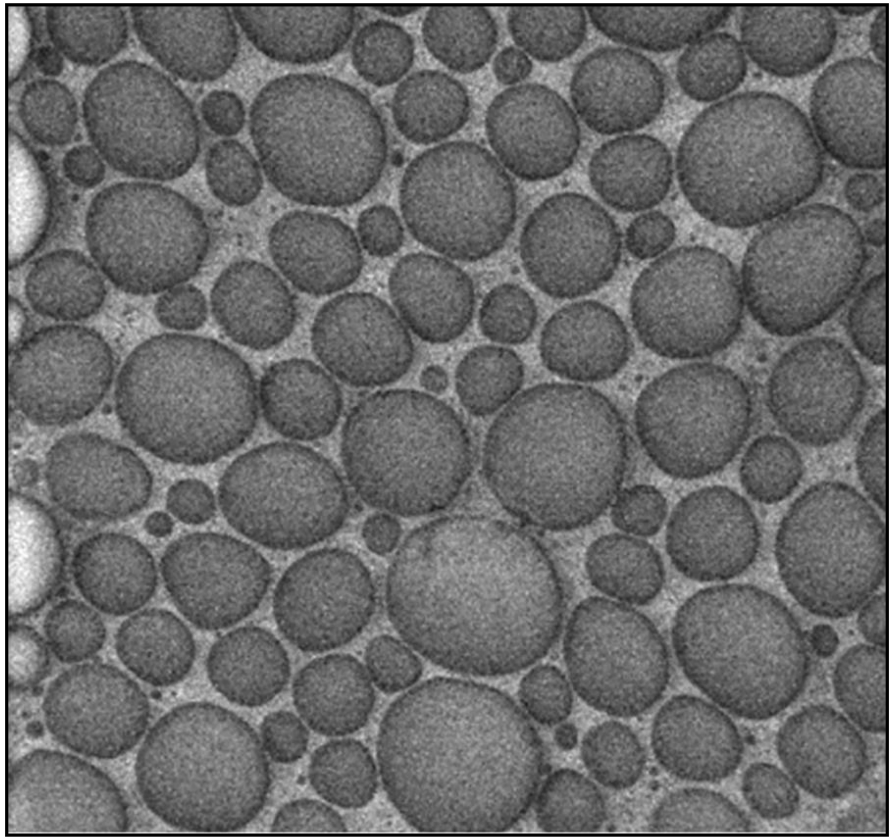
ESEM ‘wet’ mode



Fresh bacteria



Oil-in-water emulsion

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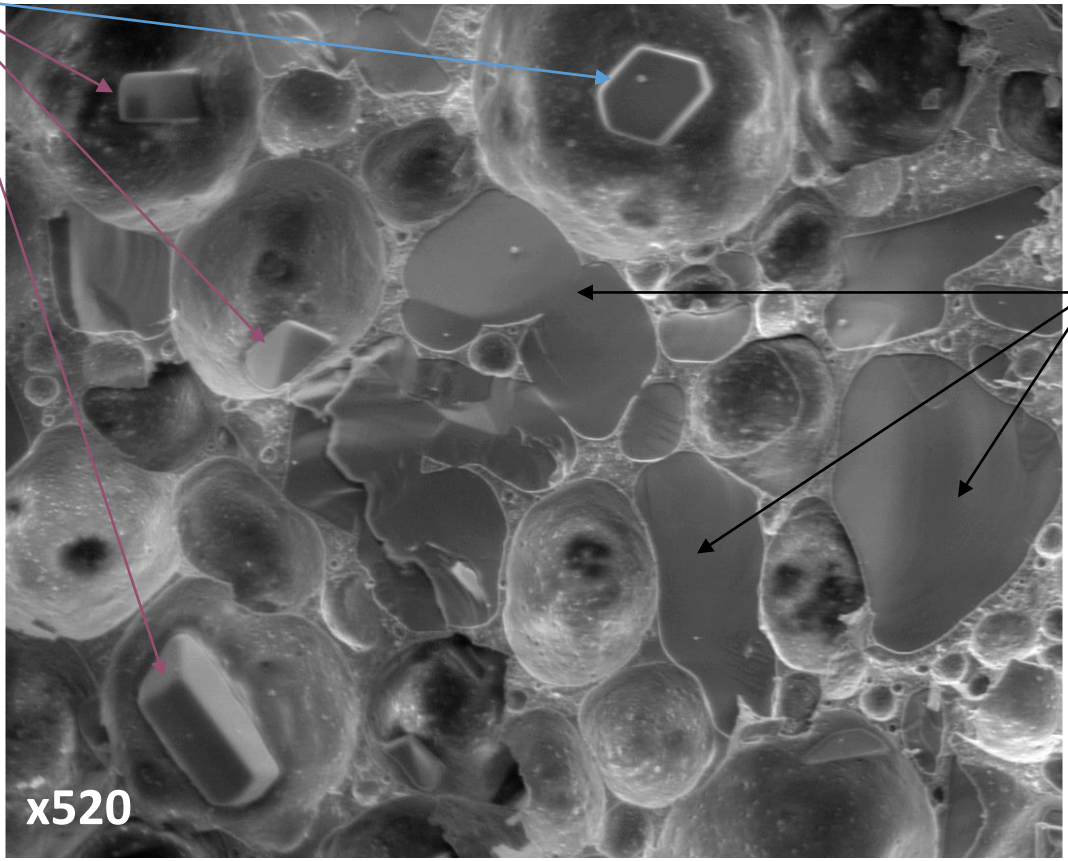
Interfacial ice T = -120°C P = 0.7 Torr N_2

Matrix ice



Stokes, Mugnier & Clarke (2004) J Microscopy 213(Pt 3)

NaCl.avi

Microanalysis
under low vacuum conditions

- Low vacuum SEM: charge is eliminated by a gas (water, air or N_2)
- Two major problems :

  - beam damage (heating of sample)

  - beam spread (skirt effect)

Beam spread

- Electrons are scattered due to collisions with gas atoms
- X-ray is generated outside the probe
- X-ray information comes from up to 500 micron from central spot (skirt area)

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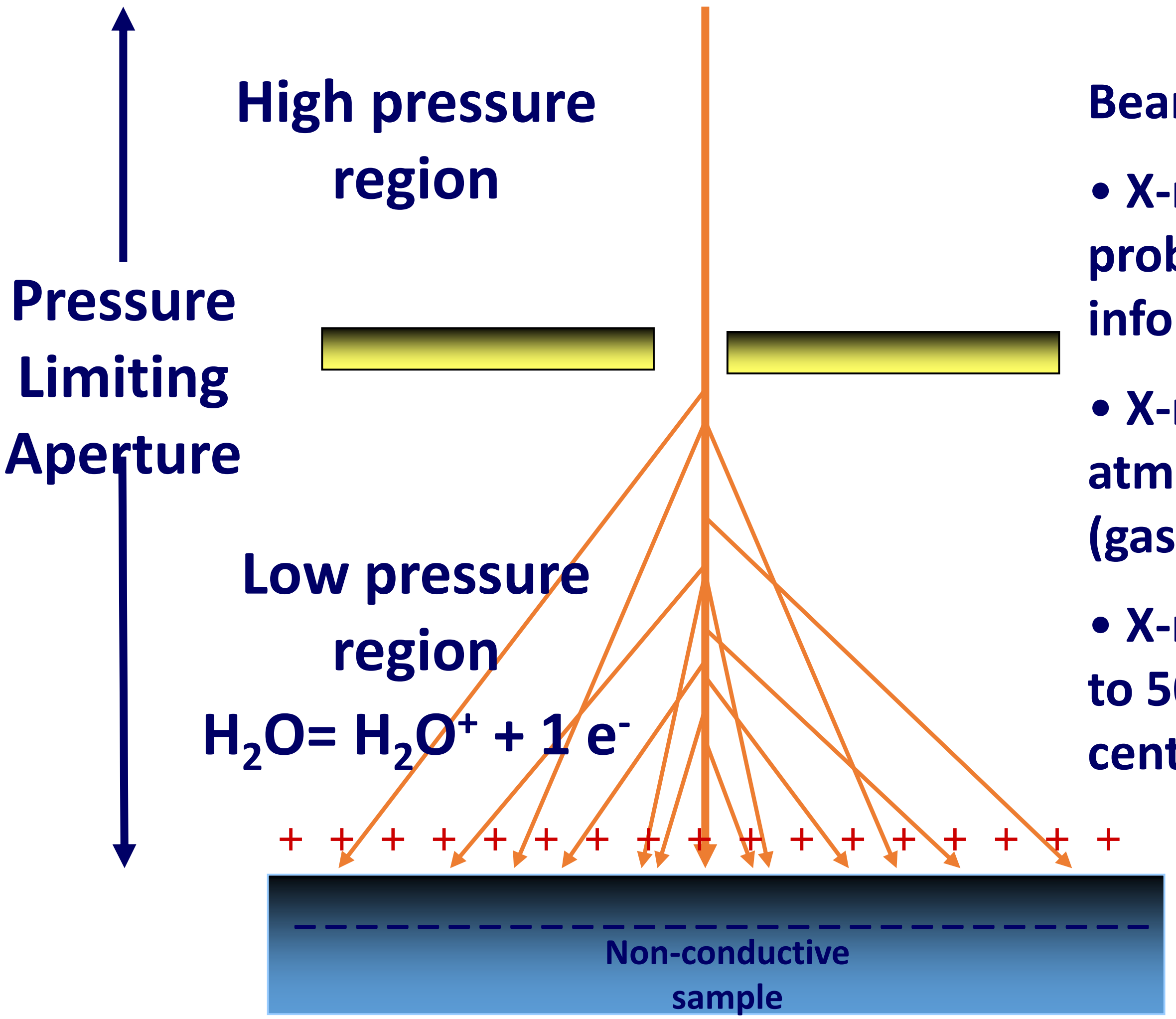
Elastic scattering – Skirt formation



Beam spread :

- X-rays from the probe spot (actual information)
- X-rays due to atmospheric effect (gas)
- X-rays information up to 500 micron from central spot (skirt)

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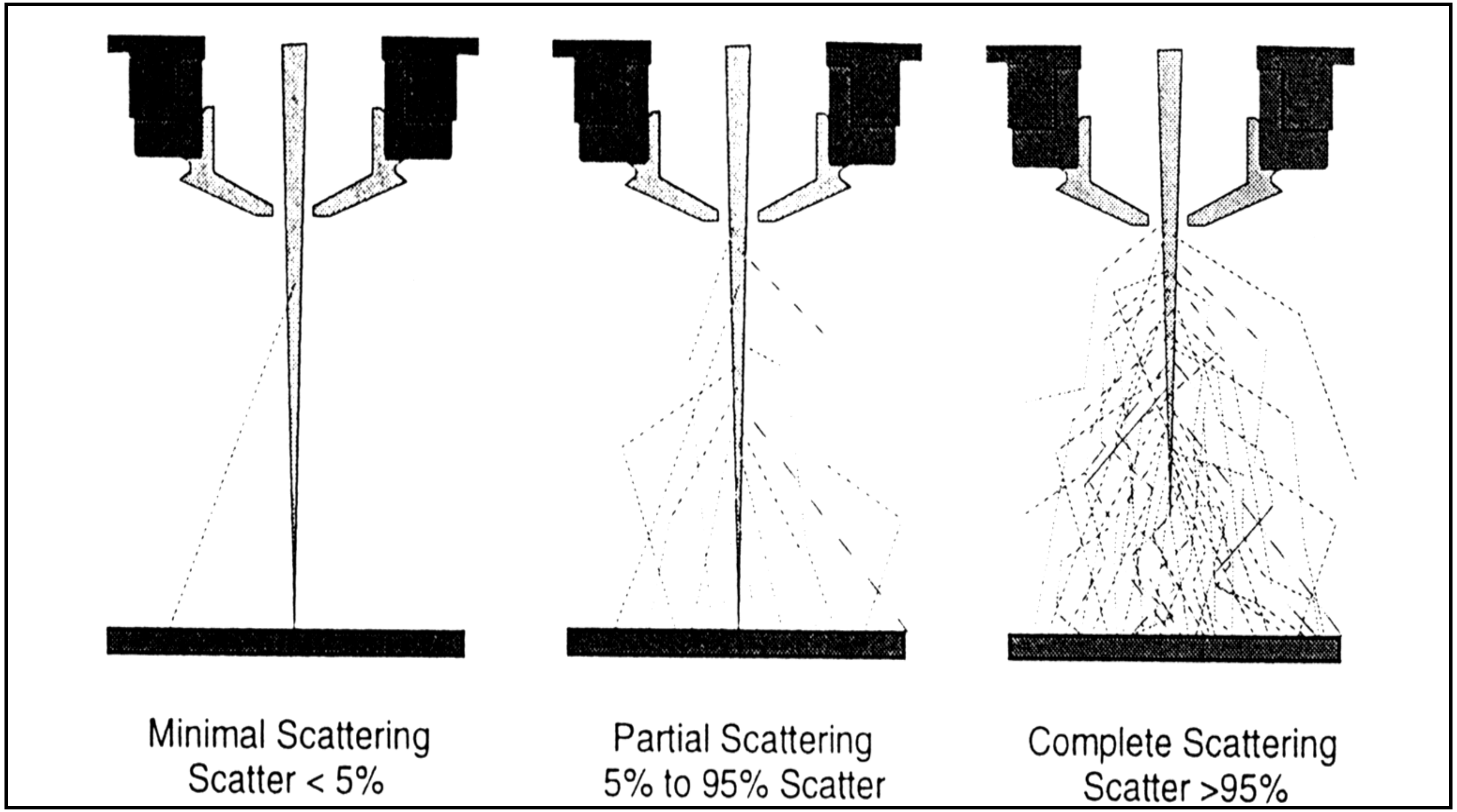
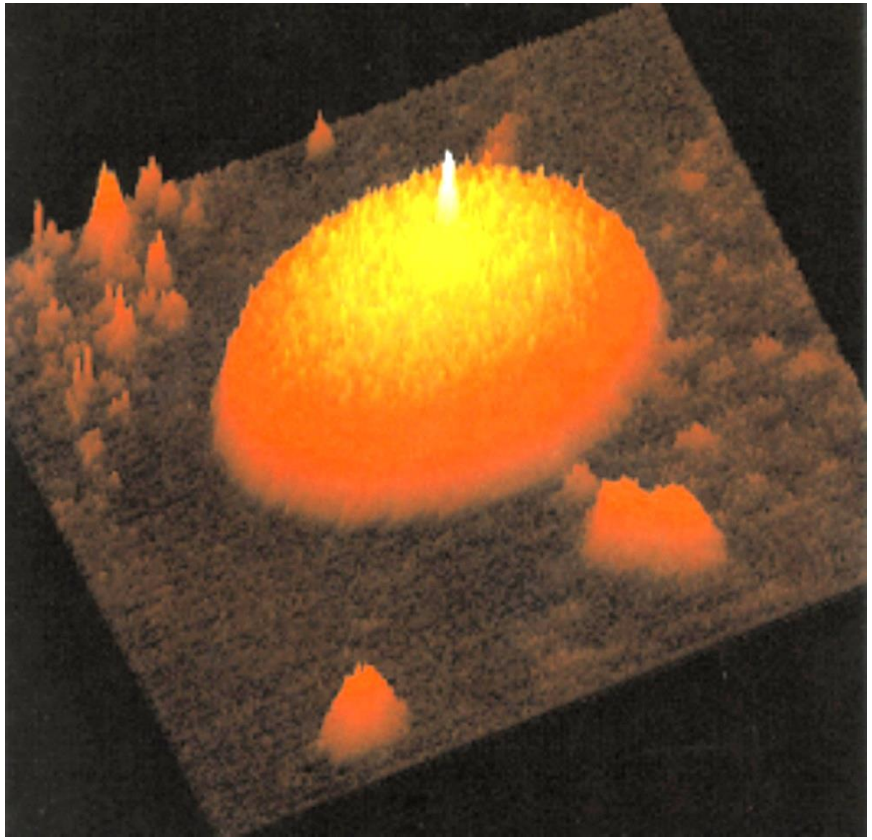
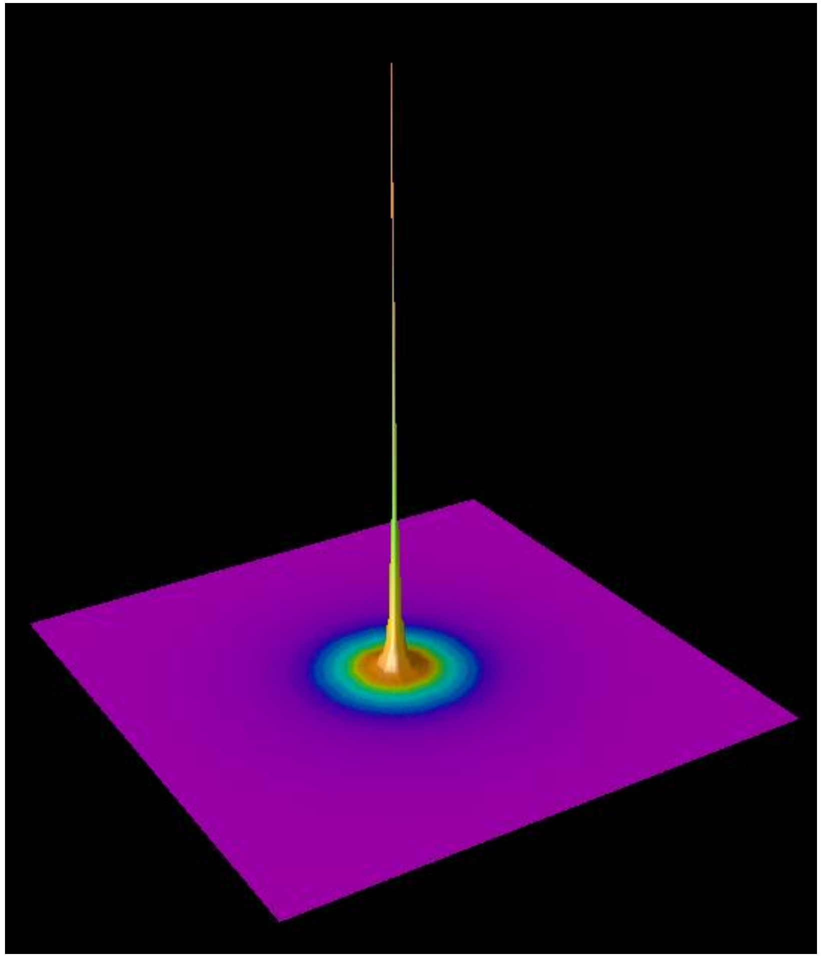

Minimal Scattering
Scatter < 5%
Partial Scattering
5% to 95% Scatter
Complete Scattering
Scatter >95%





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Electrons in the Skirt

Fraction of electrons remaining in central probe:

$$f = e^{-\sigma P d / RT}$$

where:

σ – scattering cross section

P – pressure

R – gas constant

d – gas path length

T – temperature

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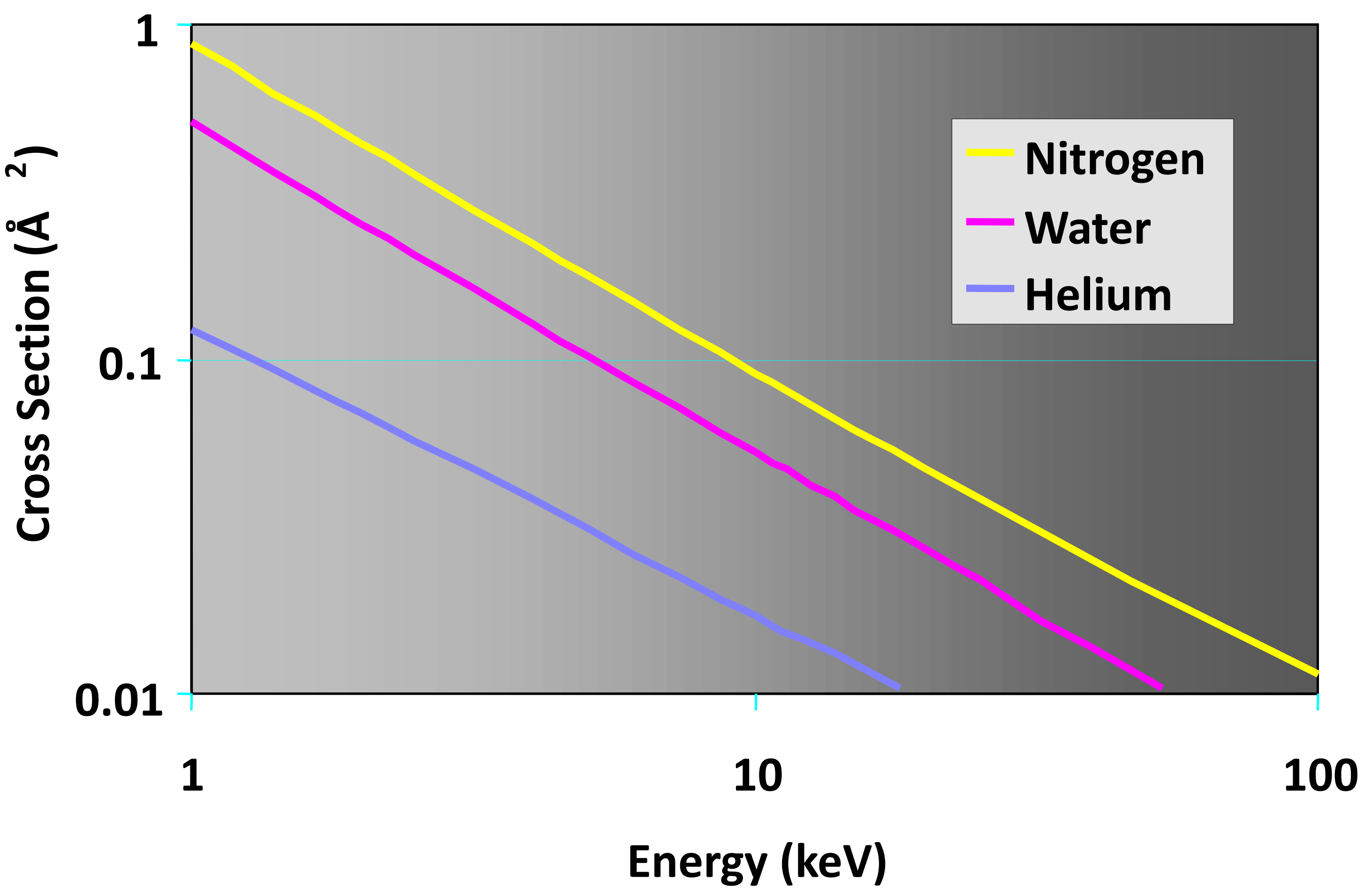
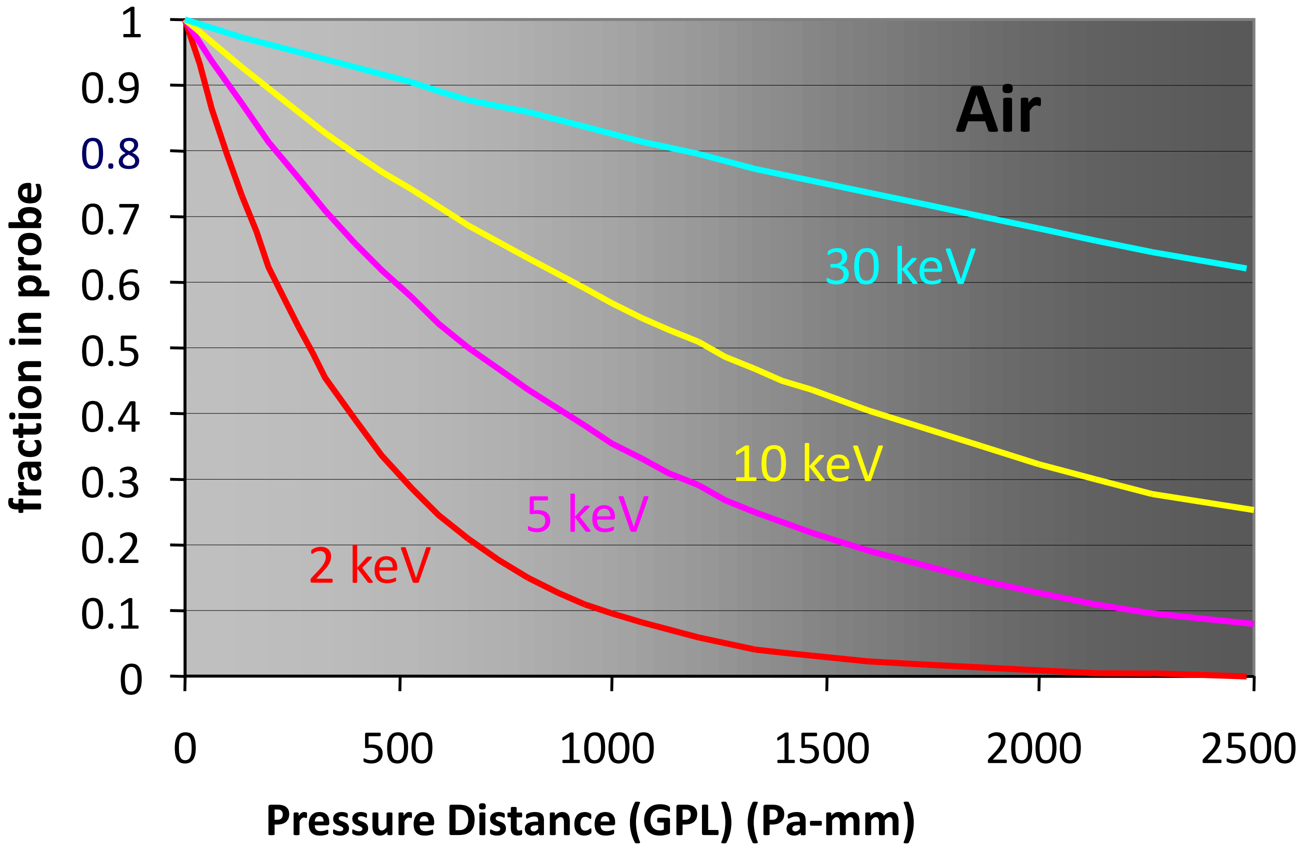
Elastic Cross Sections



Scattering out of the Probe



Use as low a pressure and as short a Gas Path Length as possible!

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Which gas?

Influence of atomic number on primary electron mean free path λ as a function of primary beam energy

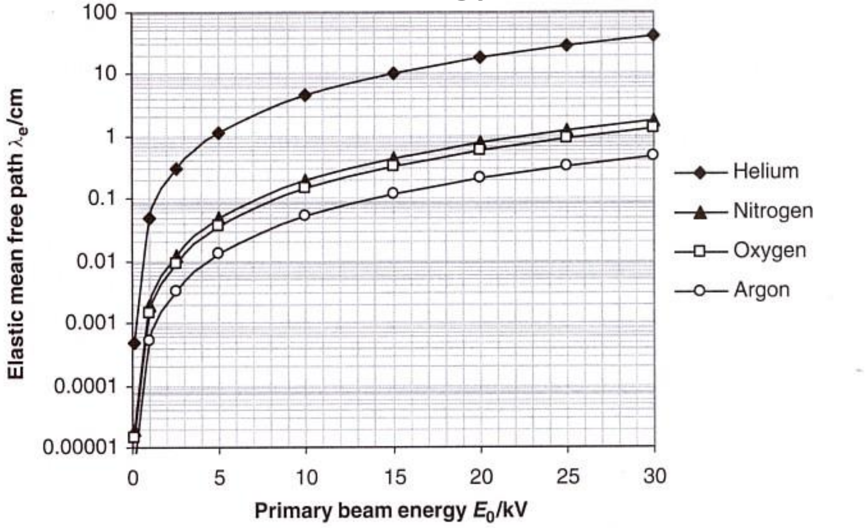


Figure 4.2 Log-linear plot of primary electron mean free paths as a function of atomic number for a range of primary beam energies. The data points are for helium ($Z = 2$), nitrogen ($Z = 7$), oxygen ($Z = 8$) and argon ($Z = 18$). Pressure $p = 100$ Pa

Influence of atomic number on skirt radius r_s as a function of primary beam energy

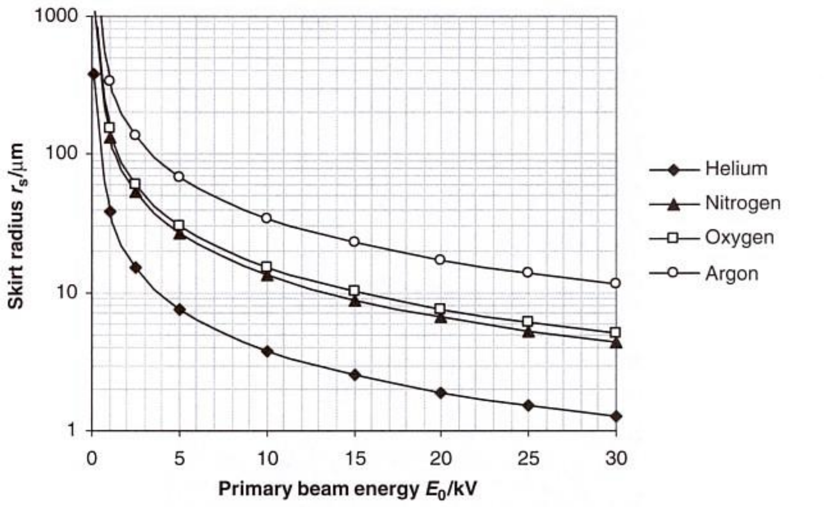


Figure 4.3 Log-linear plot of skirt radius r_s as a function of primary beam energy for a range of gases having atomic numbers $Z = 2$ (helium), $Z = 7$ (nitrogen), $Z = 8$ (oxygen) and $Z = 18$ (argon). Thickness of the gas layer (gas path length) = 2 mm. The temperature is assumed to be $T = 293$ K (20 °C) and pressure $p = 100$ Pa (0.75 torr)

Influence of atomic number on the percentage of primary electrons remaining in the focused probe as a function of primary beam energy

Helium – theoretically the best gas. However it is the most difficult gas to ionize and the small size of helium atoms makes this gas notoriously difficult to handle with a typical vacuum pump. Prolonged use of helium is definitely not recommended.

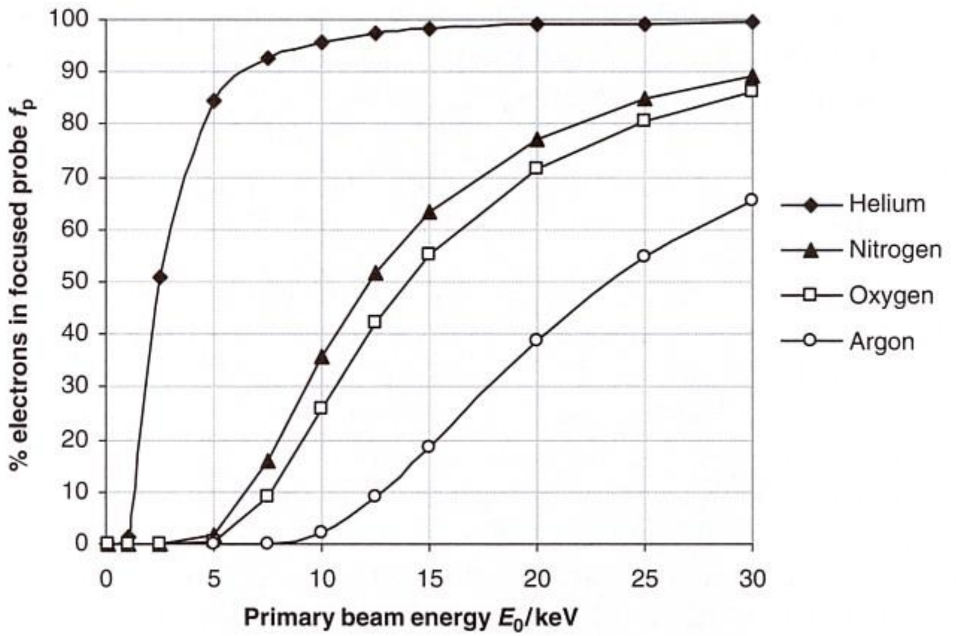
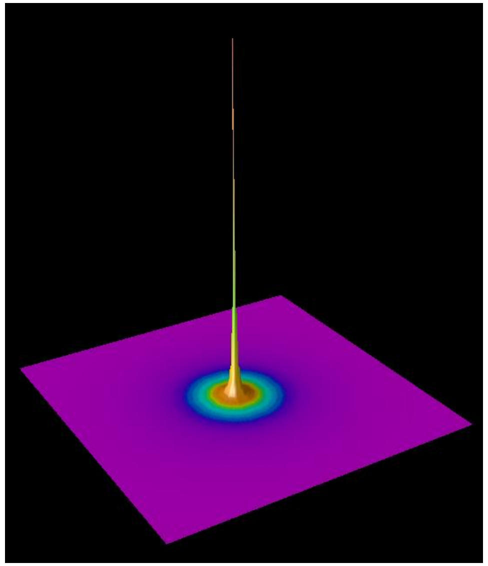
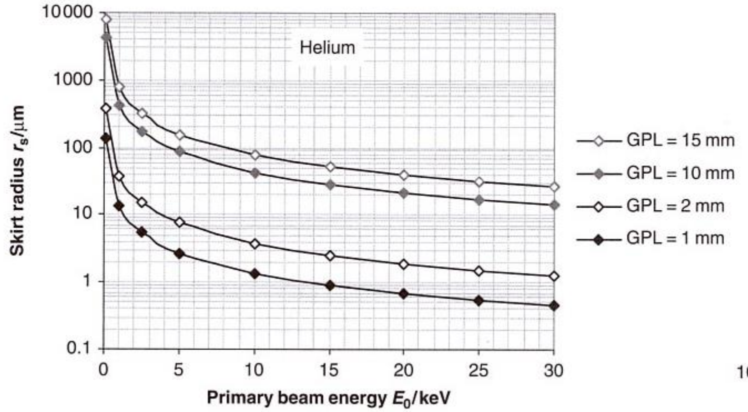


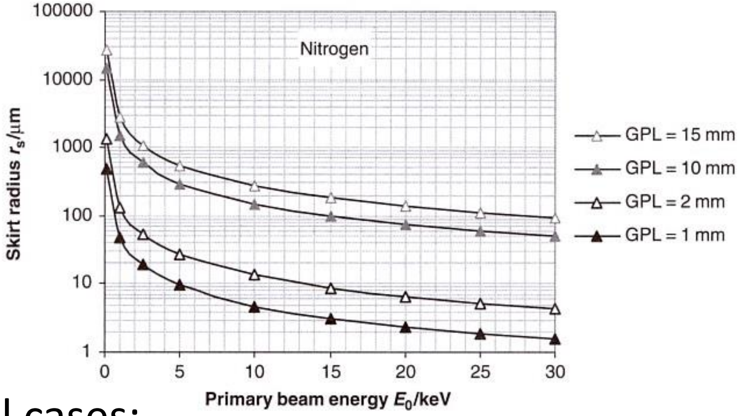
Figure 4.5 Plot of the percentage of primary electrons remaining in the focused probe to form a useful beam current, as a function of primary electron beam energy E_0 for a range of gases with atomic numbers Z = 2 (helium), Z = 7 (nitrogen), Z = 8 (oxygen) and Z = 18 (argon). Thickness of gas layer (gas path length) = 2 mm, pressure p = 100 Pa



Exploring the Gas Path Length



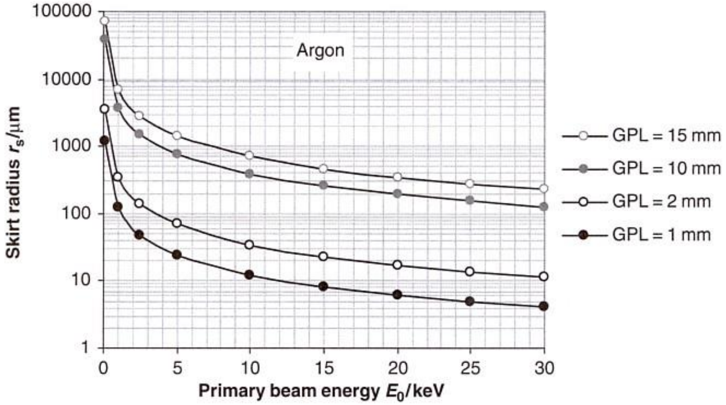
Helium



Nitrogen

The general trends can be seen in all cases: **there is a sharp rise in the radius of the skirt as the primary beam energy decreases below about 5 keV and almost two orders of magnitude difference between radii for GPL = 1 mm compared to 15 = mm**

Argon



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Exploring the Gas Path Length

With the exception of helium, the percentage of electrons remaining in the focused probe falls sharply as the GPL increases. That brings with it a reduction in electron beam current.
For argon, it would be advisable to maintain gas path length of a millimeter or two.
For nitrogen and oxygen the GPL of the order a few millimeters is advisable.

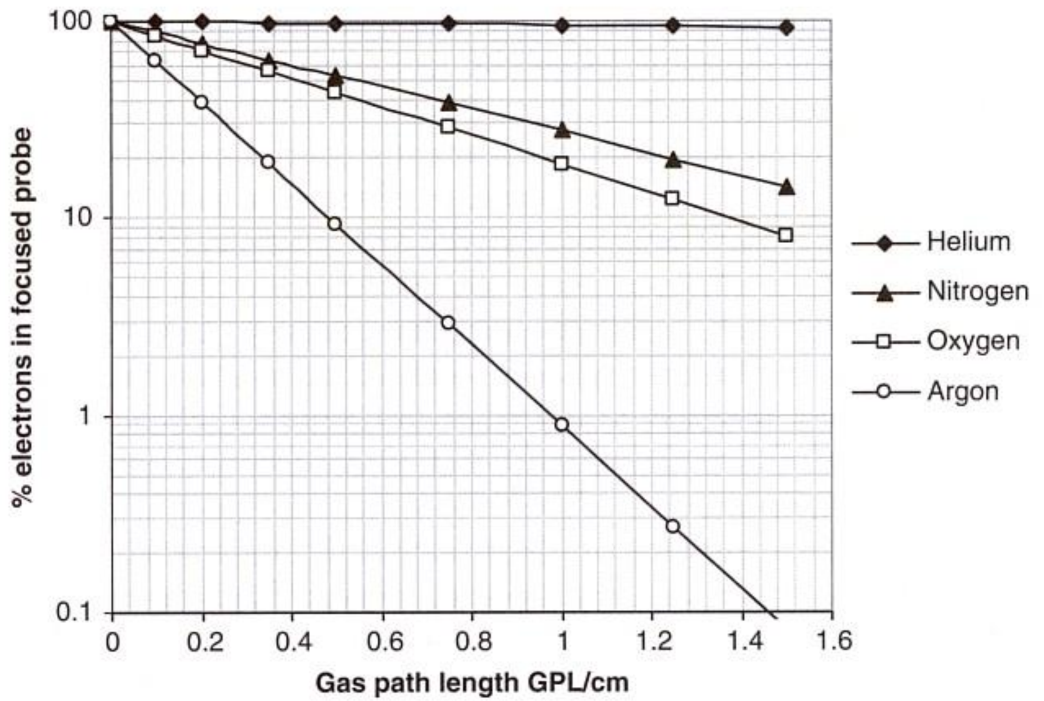

Figure 4.9 Log-linear plot to show the percentage of electrons remaining in the focused probe to form the useful primary current, as a function of atomic number of the gas for a primary beam energy $E_0 = 20\,\text{keV}$. Pressure $p = 100$ Pa

How much gas?

Influence of energy on elastic mean free path λ as a function of pressure for nitrogen

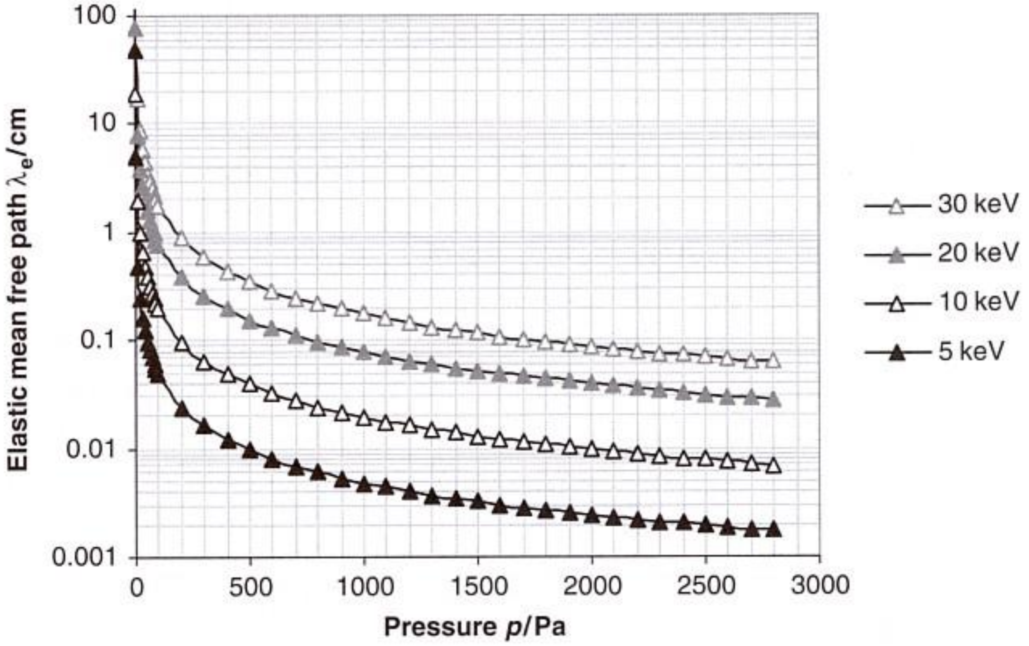

Figure 4.14 Log-linear plot to show the variation in elastic mean free path λ_e fo primary electrons in nitrogen gas over the wide range of pressures available in th VP-ESEM and for several primary beam energies E_0

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How much gas?

Influence of atomic number on skirt radius r_s as a function of pressure

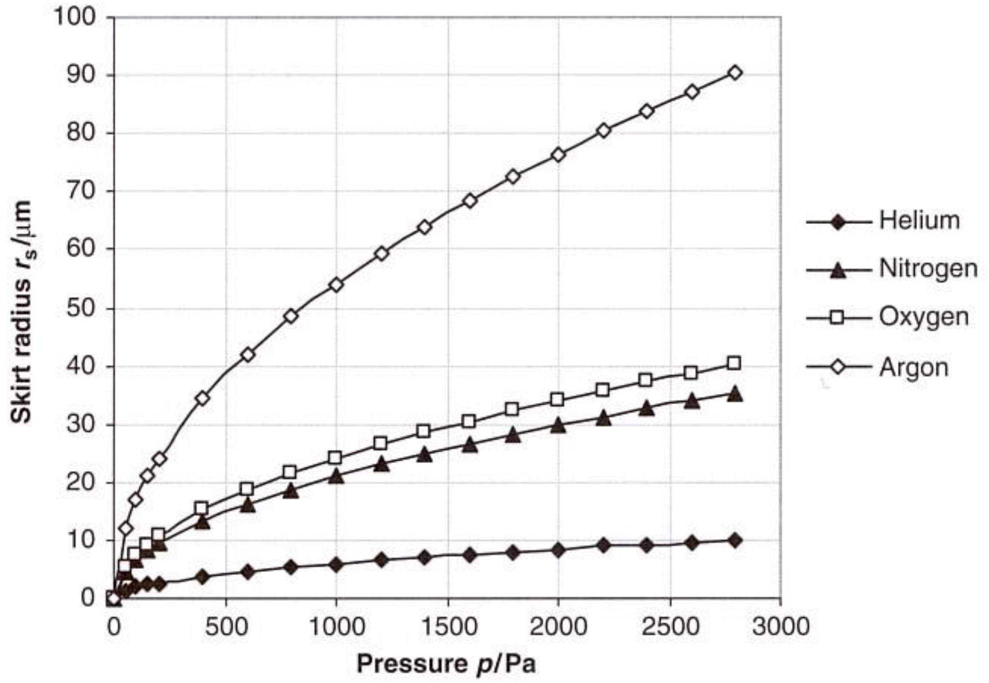


Figure 4.16 Plot of skirt radii r_s over the pressure range extending to 2.8 kPa for gases having atomic numbers Z = 2 (helium), Z = 7 (nitrogen), Z = 8 (oxygen) and Z = 18 (argon). Primary beam energy E_0 = 20 keV and gas path length GPL = 2 mm

How much gas?

Influence of pressure on skirt radius r_s for several GPLs for nitrogen

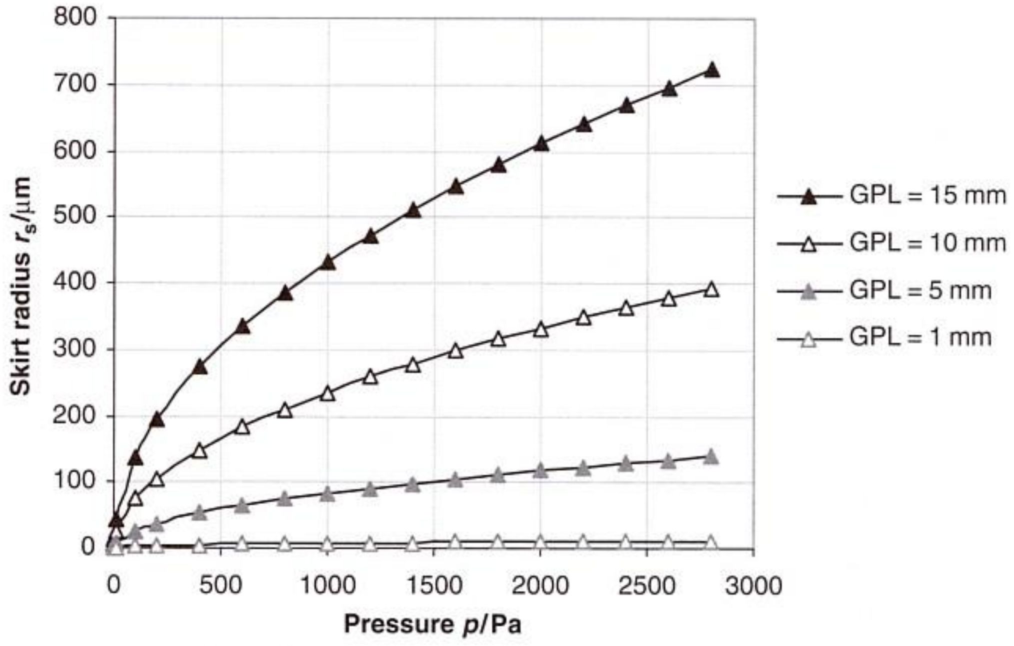
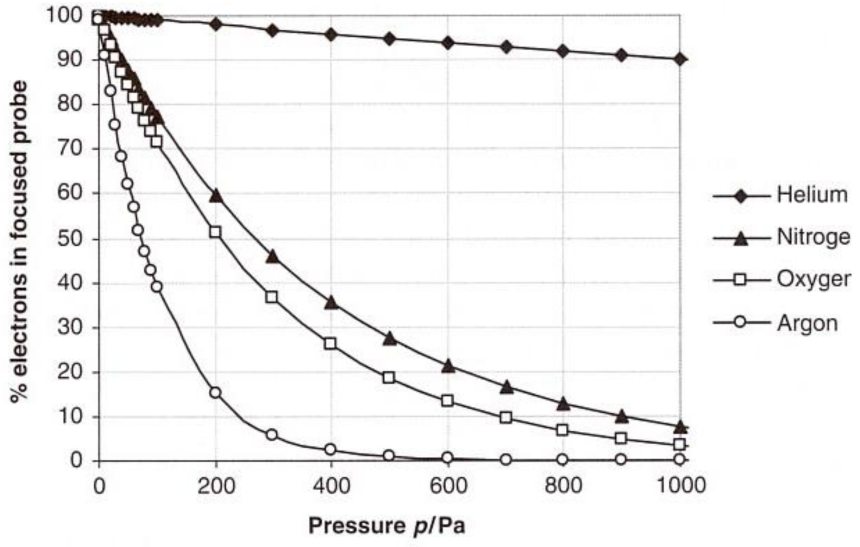


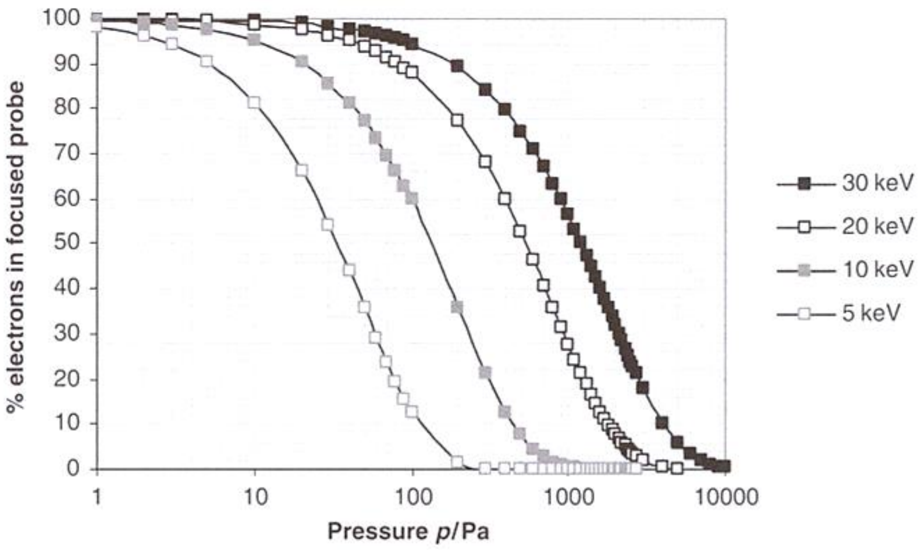
Figure 4.17 Plot of primary beam skirt radii r_s over the pressure range extending to 2.8 kPa for several gas path lengths and in nitrogen gas, $Z = 7$. Primary beam energy $E_0 = 20\,\text{keV}$

p = 100 Pa
R_s = 2.4 μm for GPL = 1 mm
r_s = 26 μm for GPL = 5 mm

p = 2.8 kPa
r_s = 12.5 μm for GPL = 1 mm
r_s = 139 μm for GPL = 5 mm

How much gas?





Percentage of electron remaining in the focused probe as a function of pressure and atomic number

GPL = 2 mm, E_0 = 20 keV

Percentage of electron remaining in the focused probe as a function of pressure and a range of primary beam energy

GPL = 1 mm, nitrogen

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Microanalysis under low vacuum conditions

- Low vacuum SEM: charge is eliminated by a gas (water, air or N_2)
- Two major problems :

  - beam damage (heating of sample)

  - beam spread (skirt effect)

Beam spread

- Electrons are scattered due to collisions with gas atoms
- X-ray is generated outside the probe
- X-ray information comes from up to 500 micron from central spot (skirt area)

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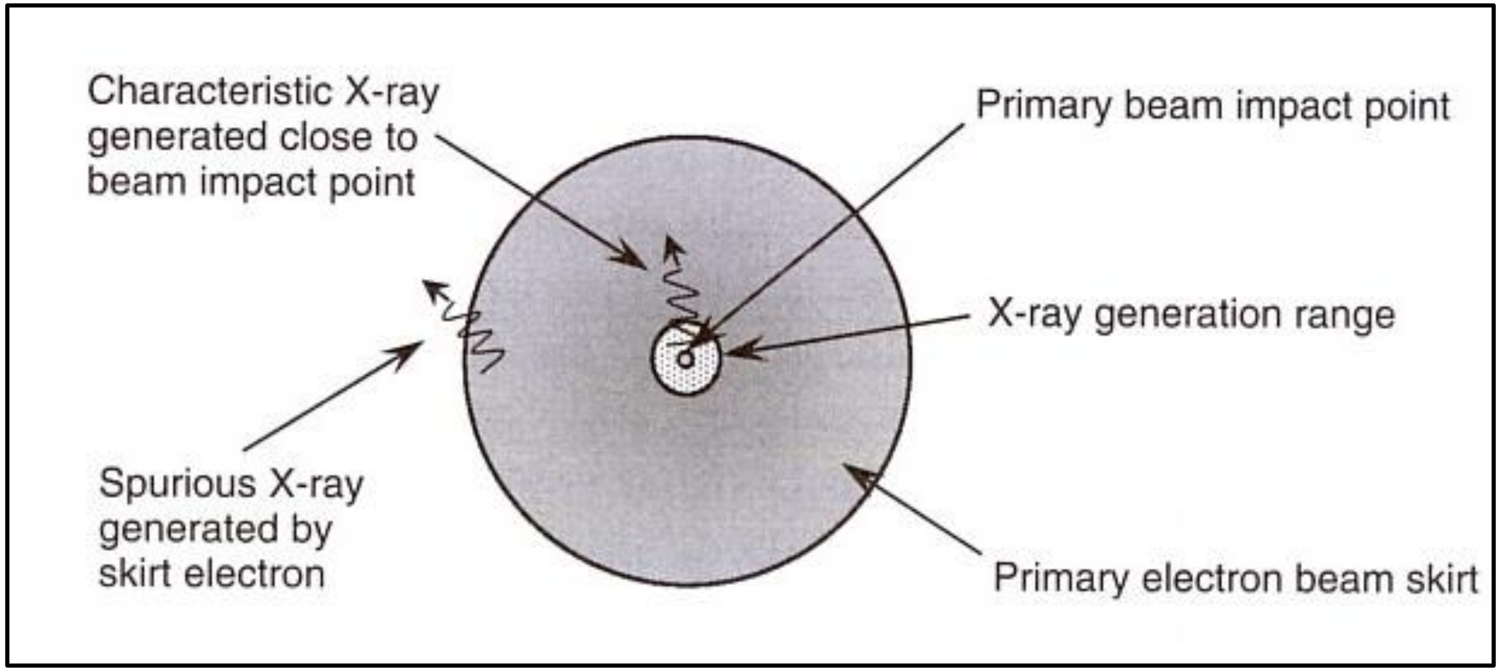

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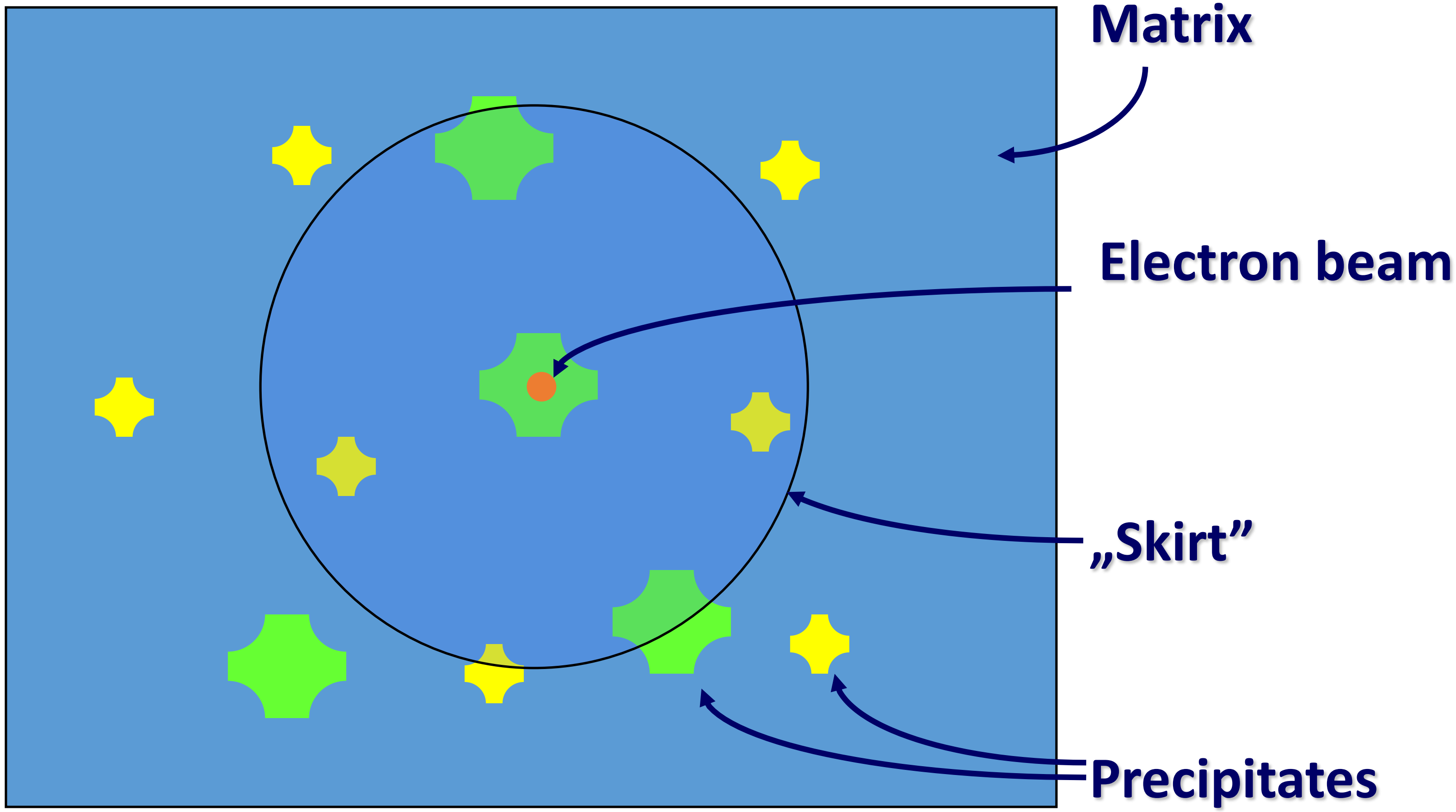
X-ray Skirt



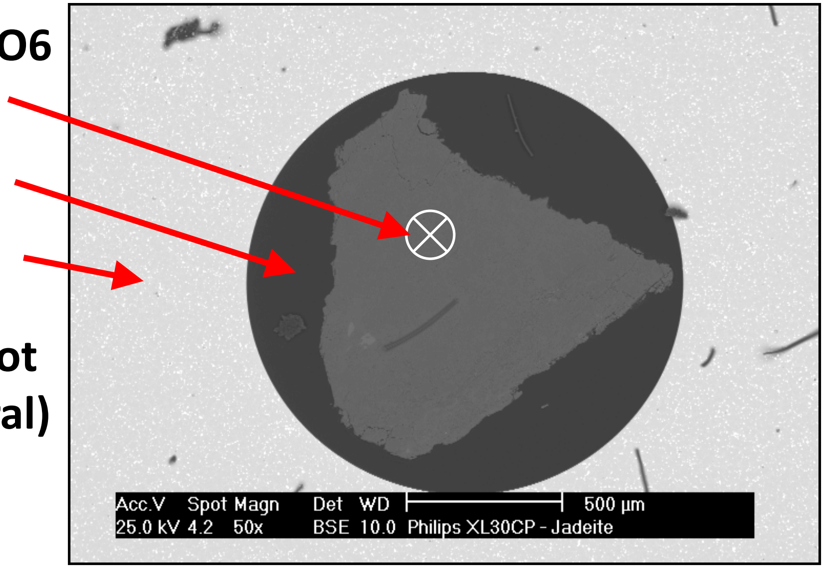
Large sample in homogeneous matrix

Sample : Jadeite $NaAlSi_2O_6$

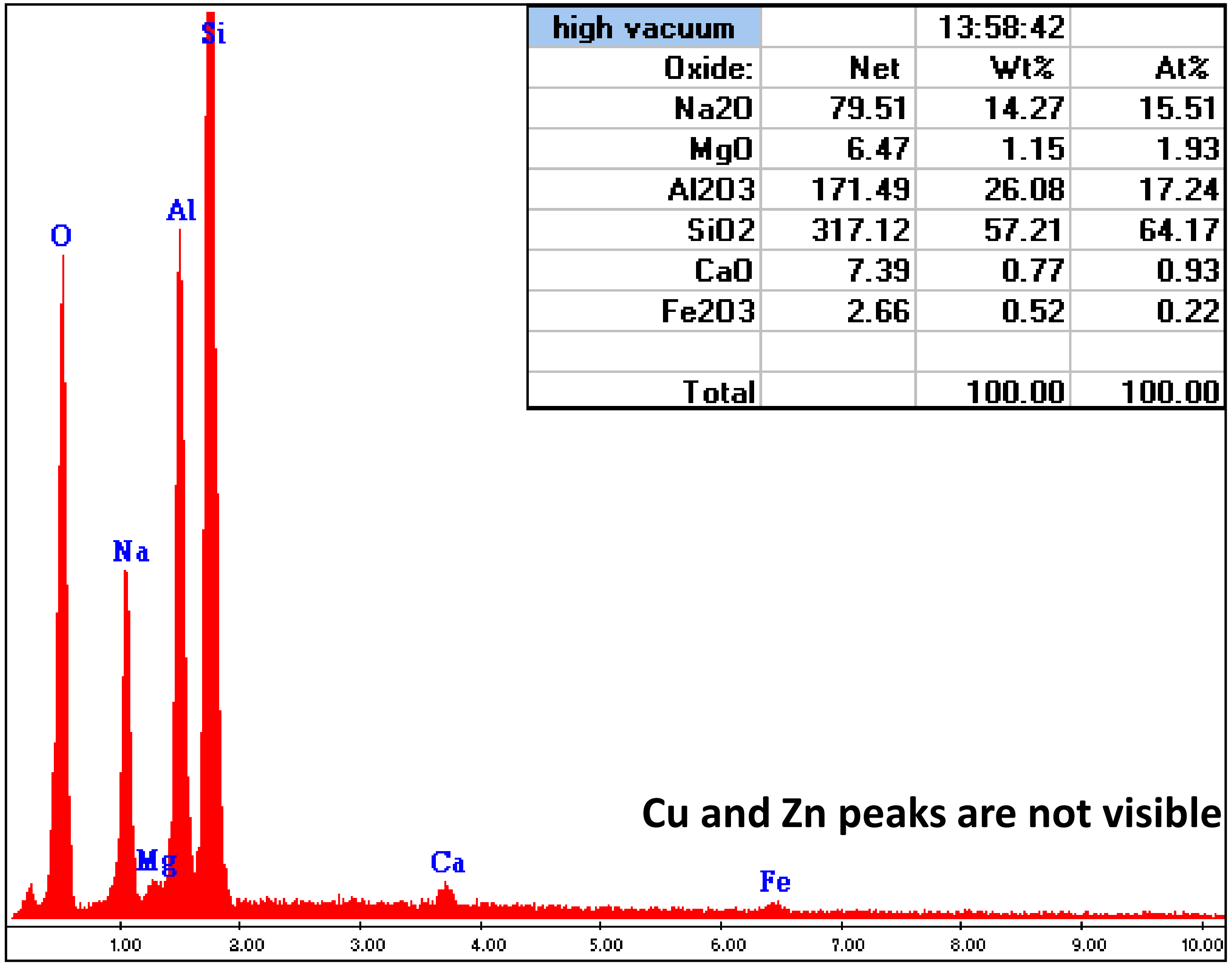
Matrix 1 : polymer

Matrix 2 : Cu/Zn block

Distance between the spot position (centre of mineral) and the Cu/Zn block is more than 500 micron



High-vacuum spectrum - Jadeite 25kV



high vacuum		13:58:42	
Oxide:	Net	Wt%	At%
Na2O	79.51	14.27	15.51
MgO	6.47	1.15	1.93
Al2O3	171.49	26.08	17.24
SiO2	317.12	57.21	64.17
CaO	7.39	0.77	0.93
Fe2O3	2.66	0.52	0.22
Total		100.00	100.00

Cu and Zn peaks are not visible

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Microanalysis
under low vacuum conditions

VP gas compensated technique
(with 2 spectra measured at different pressures)
developed by Wernisch and Dijkstra

To reduce the skirt effect:

- use short gas path (a special cone attached to pole piece)
- use high acceleration voltage (25kV)
- use low pressure (0.1 - 0.3 mbar)

A pressure variation method

involves collecting spectra at two different pressures (with other parameters constant) and uses the difference spectrum to extrapolate back to zero-scattering case.

This method assumes that the composition does not change with pressure!!!
It is only X-ray intensity that changes!

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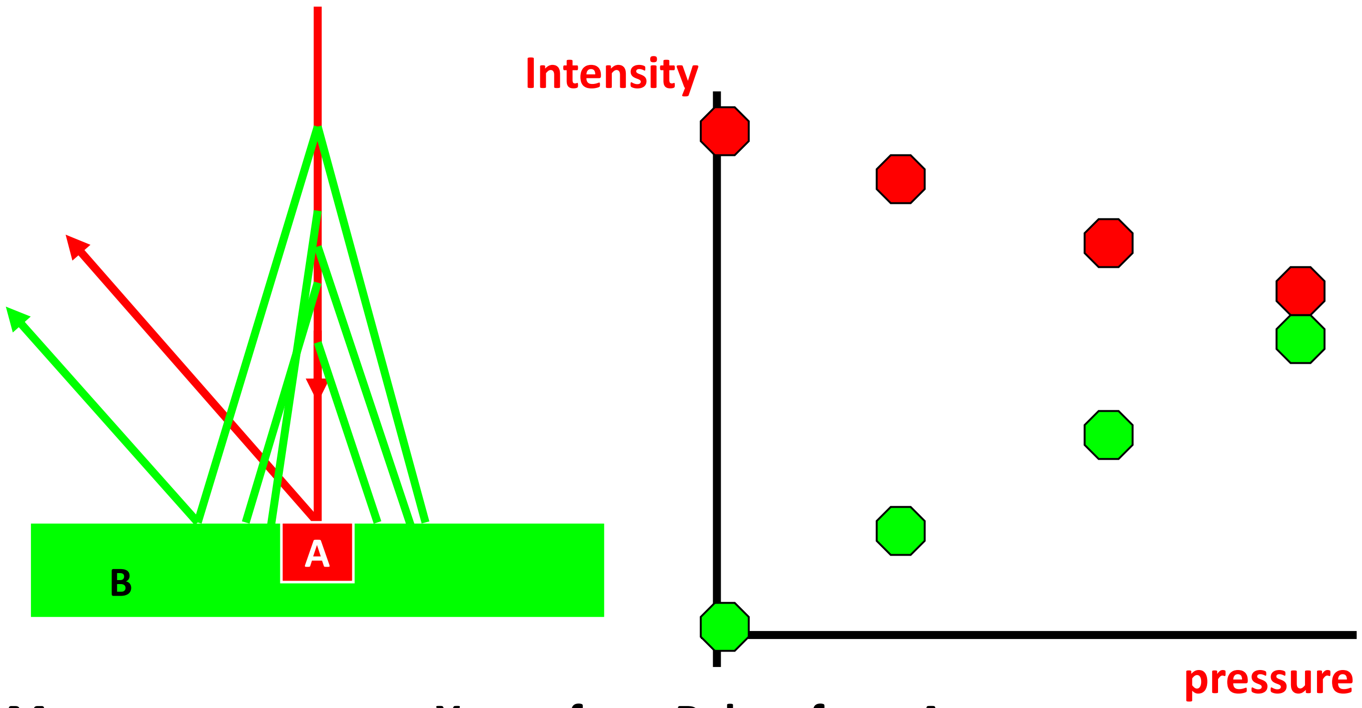
Pressure variation method



More pressure: more X-rays from B, less from A

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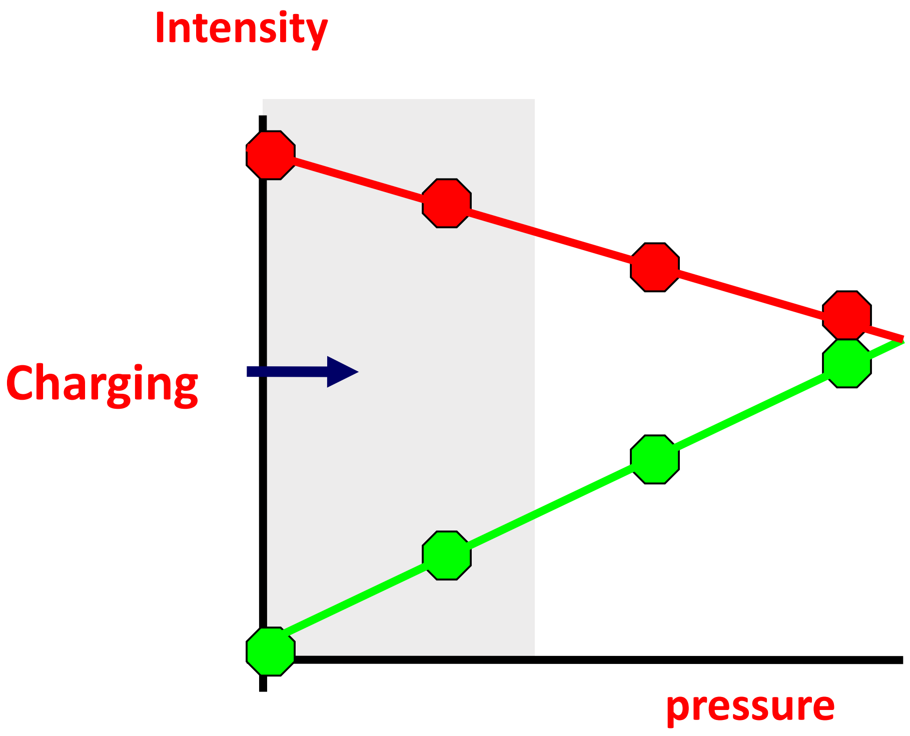
Pressure variation method



Take 2 measurements
at 2 different pressures

Extrapolate the intensities
to pressure 0

Apply matrix correction to
extrapolated intensities
to get composition

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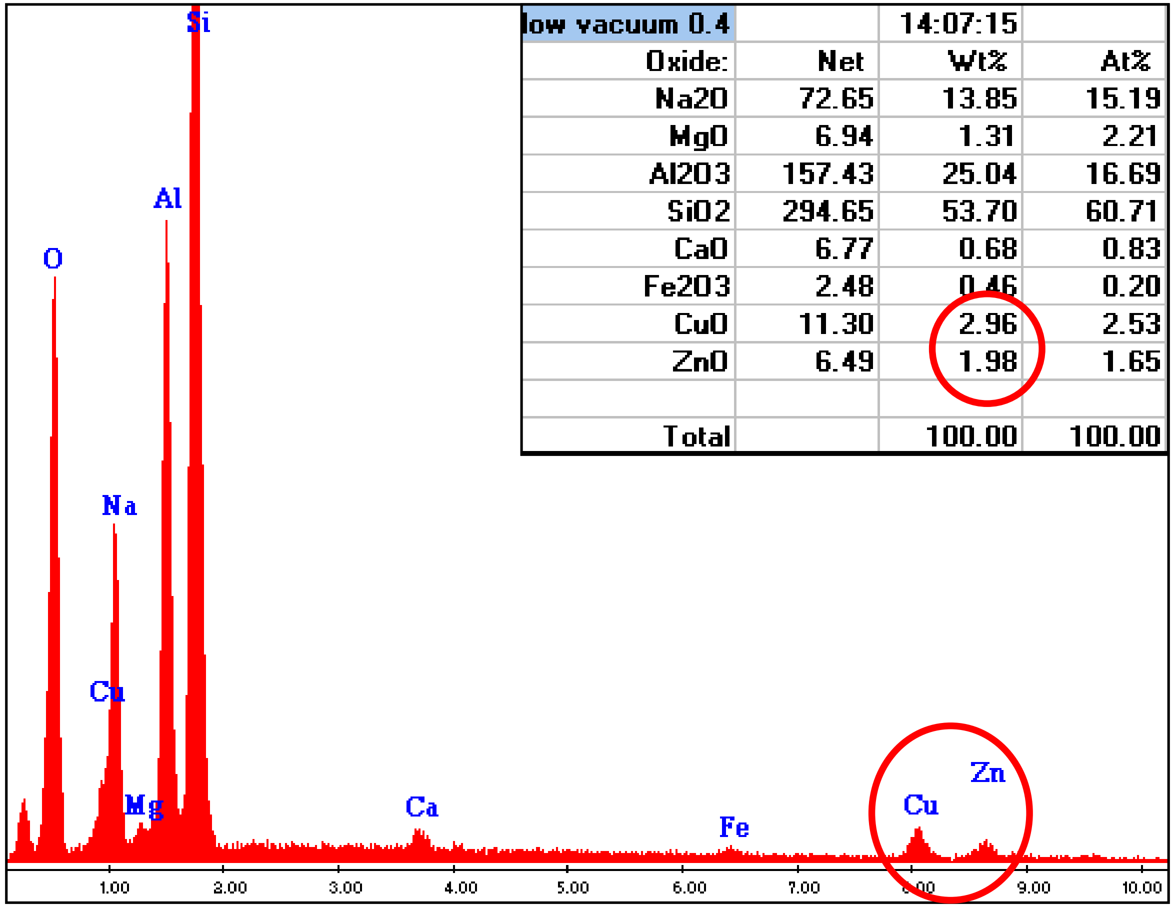
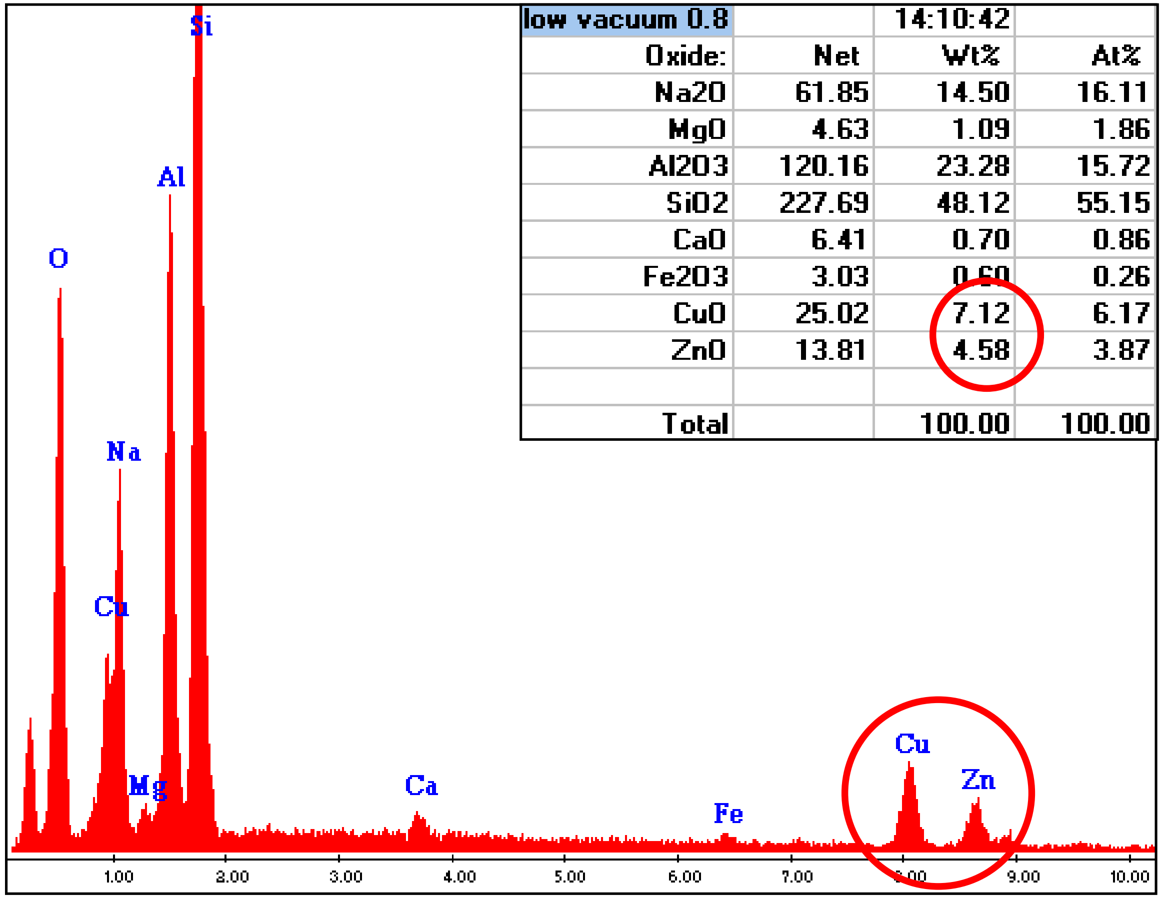
Low-vacuum spectrum 0.4 mbar 25kV



low vacuum 0.4		14:07:15	
Oxide:	Net	Wt%	At%
Na2O	72.65	13.85	15.19
MgO	6.94	1.31	2.21
Al2O3	157.43	25.04	16.69
SiO2	294.65	53.70	60.71
CaO	6.77	0.68	0.83
Fe2O3	2.48	0.46	0.20
CuO	11.30	2.96	2.53
ZnO	6.49	1.98	1.65
Total		100.00	100.00

Low-vacuum spectrum 0.8 mbar 25kV



low vacuum 0.8		14:10:42	
Oxide:	Net	Wt%	At%
Na2O	61.85	14.50	16.11
MgO	4.63	1.09	1.86
Al2O3	120.16	23.28	15.72
SiO2	227.69	48.12	55.15
CaO	6.41	0.70	0.86
Fe2O3	3.03	0.60	0.26
CuO	25.02	7.12	6.17
ZnO	13.81	4.58	3.87
Total		100.00	100.00

Contribution of the matrix (brass holder) is clearly visible

Results

Low Vac

(0.2 - 0.4 mbar)

corrected		14:08:31	
Oxide:	Net	Wt%	At%
Na2O	78.35	14.33	15.58
MgO	7.32	1.33	2.23
Al2O3	168.07	26.12	17.27
SiO2	308.47	56.83	63.76
CaO	6.99	0.74	0.89
Fe2O3	3.18	0.64	0.27
CuO	0.00	0.00	0.00
ZnO	0.00	0.00	0.00
Total		100.00	100.00

High Vac

high vacuum		13:58:42	
Oxide:	Net	Wt%	At%
Na2O	79.51	14.27	15.51
MgO	6.47	1.15	1.93
Al2O3	171.49	26.08	17.24
SiO2	317.12	57.21	64.17
CaO	7.39	0.77	0.93
Fe2O3	2.66	0.52	0.22
Total		100.00	100.00

Low Vac results (gas compensated) are close to those of the HV analysis.

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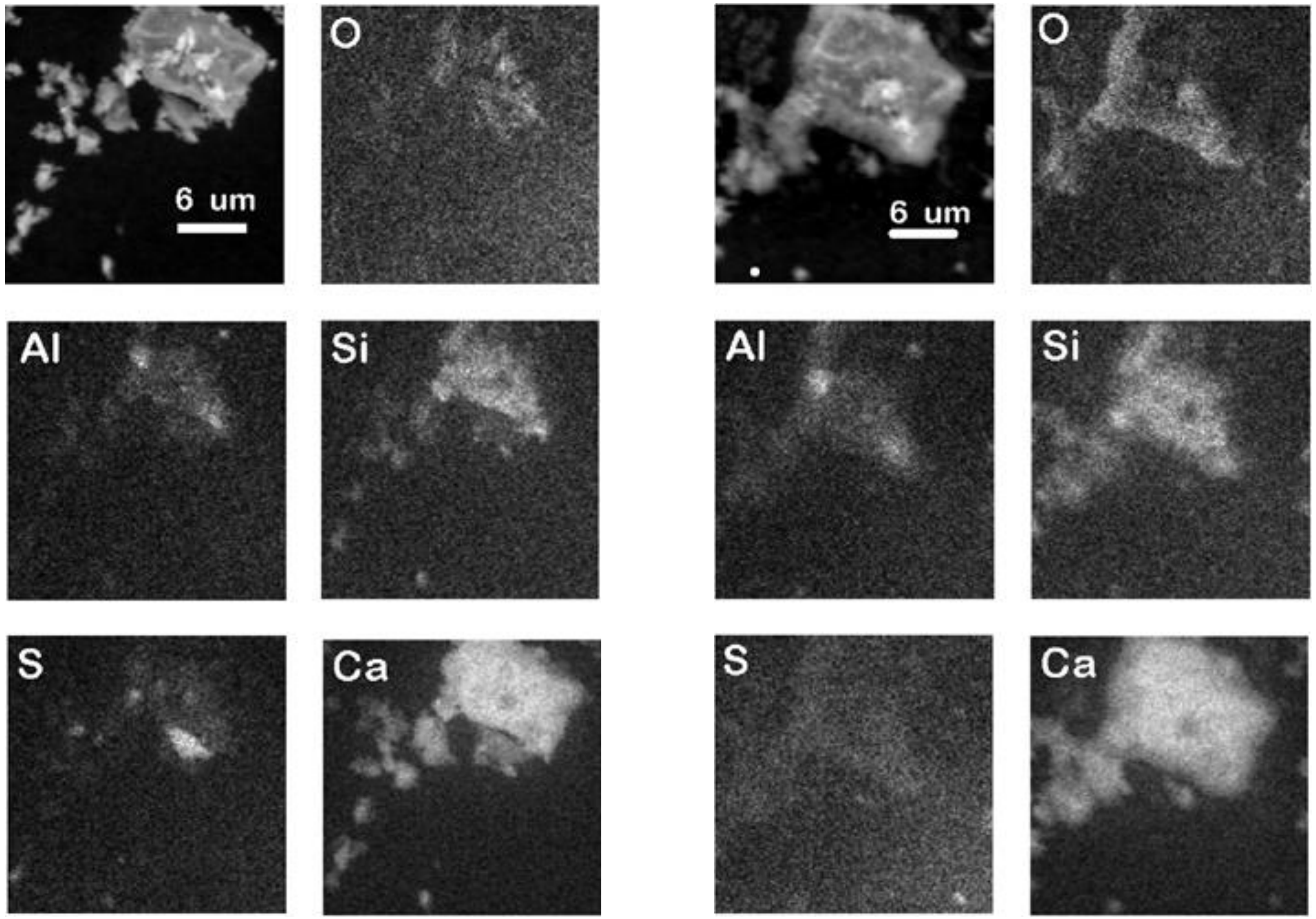
X-ray Mapping: Curing cement

Dry

Wet



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Summary - Skirt Effect

- **Imaging:**
  - **No problem; skirt adds a nearly uniform background**
  - **Resolution is defined by central probe**
- **X-ray analysis:**
  - **quantification is difficult but possible**
  - **mapping stillz possible**

To reduce skirt effect:

- **use short gas path (a special cone attached to pole piece)**
- **use high acceleration voltage**
- **use low pressure (0.3 - 0.4 torr)**

Trade-offs between variables:

- **Pressure**
- **Working distance**
- **Type of gas**
- **Energy of primary electrons**
- **Resolution**
- **Charge control**

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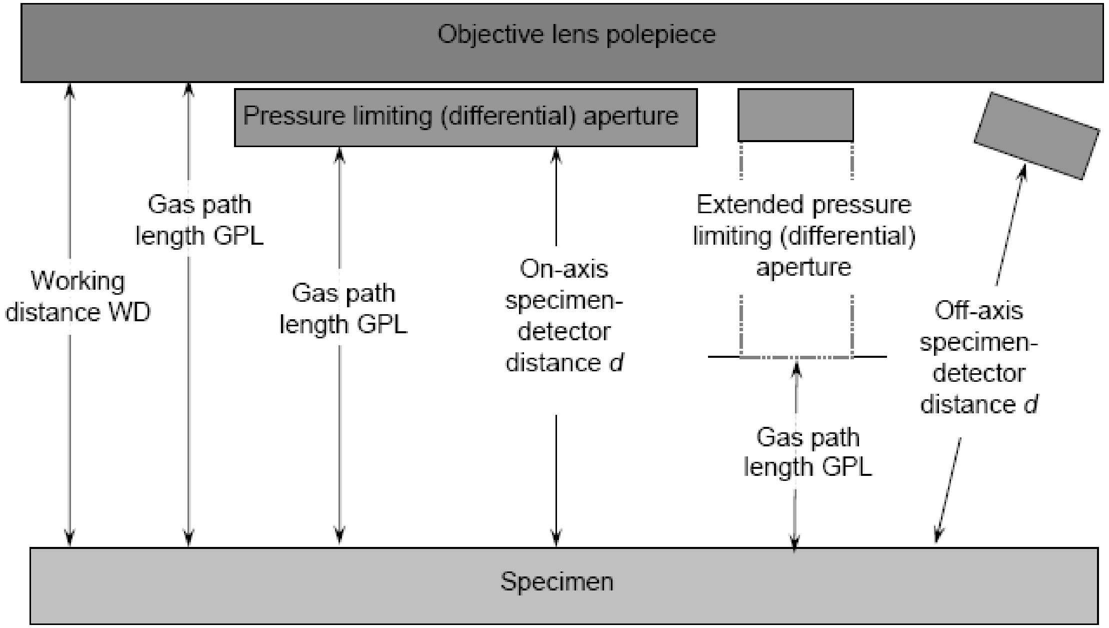
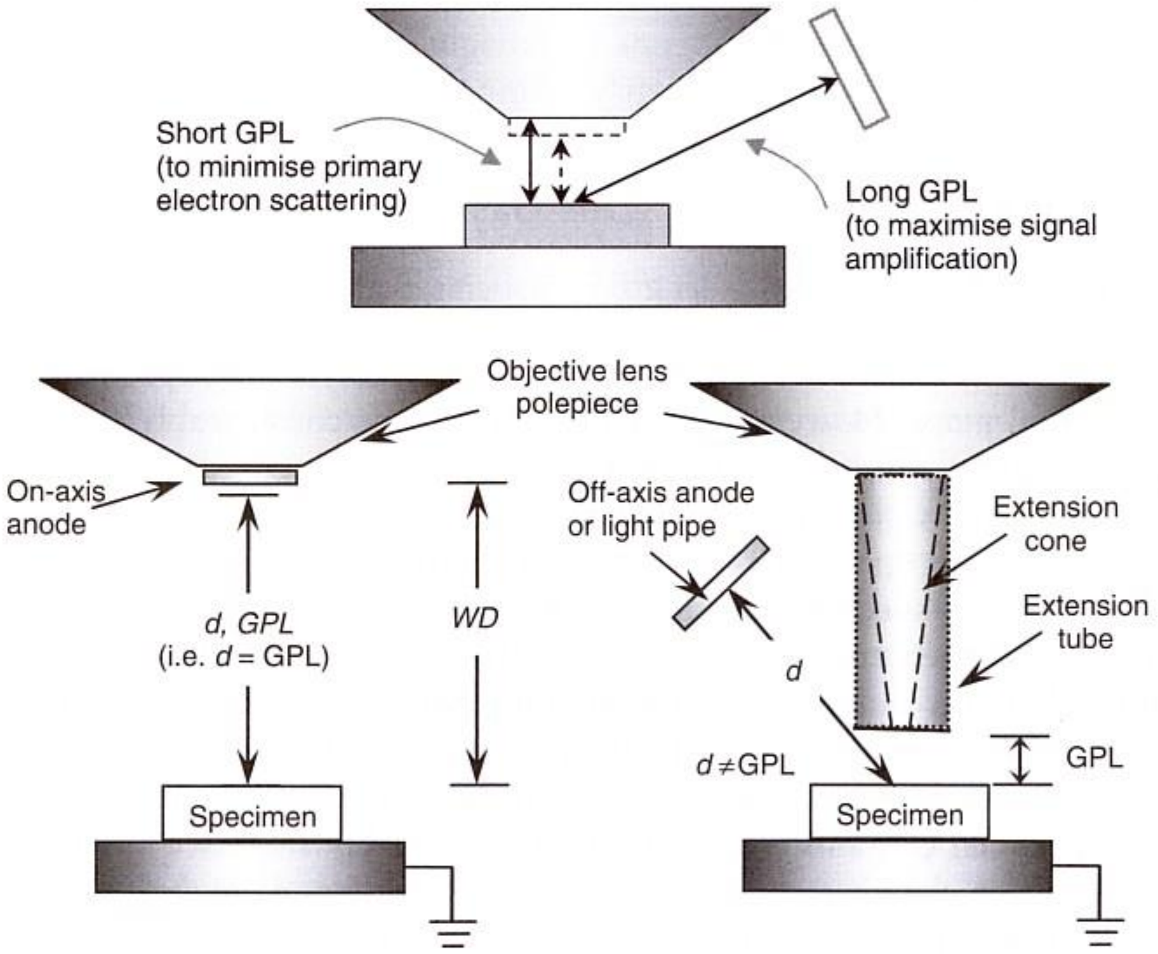
Primary electron scattering - definition of WD, *d* and GPL





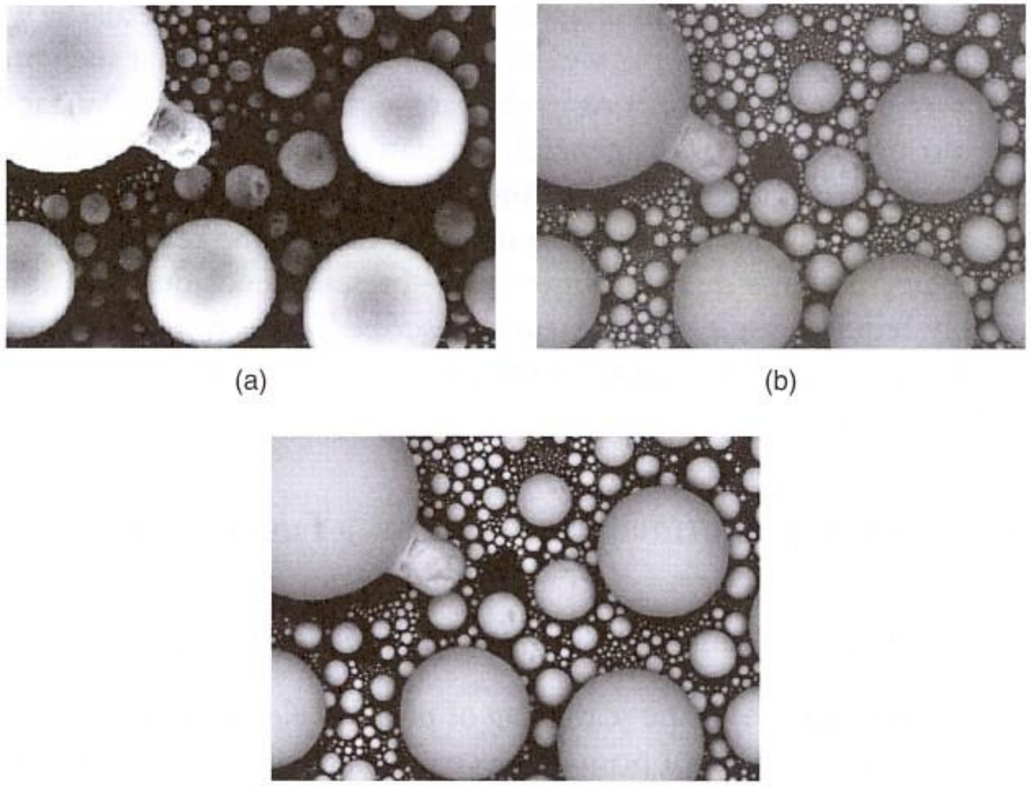
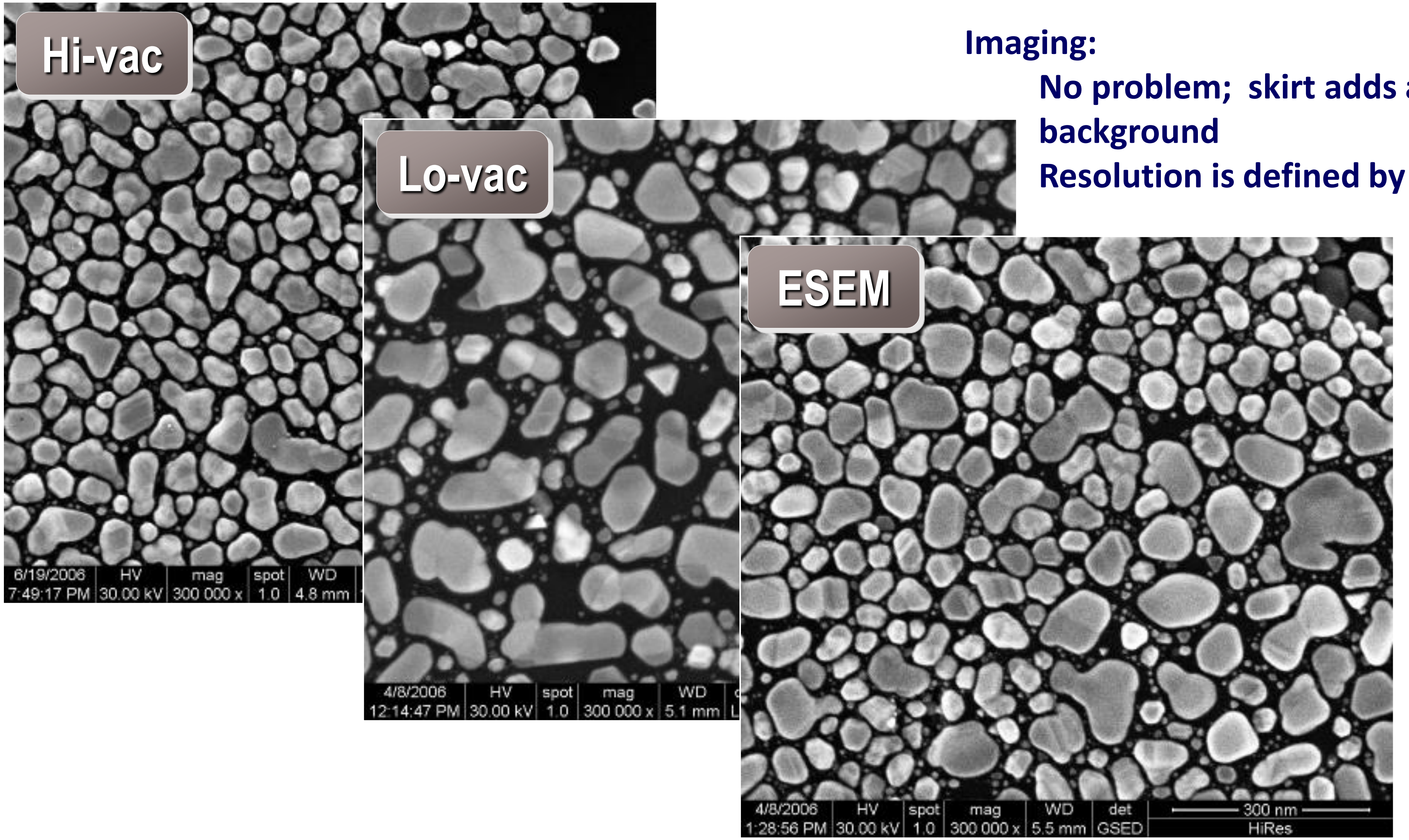


Figure 4.12 Backscattered electron images to show the effect of (a) short working distance, short gas path length (3 mm), (b) long working distance, long gas path length (10.5 mm) and (c) long working distance (10.5 mm), short gas path length (3 mm). Notice how in (c) the contrast and signal-to-noise have improved. Imaged in nitrogen gas with primary beam energy $E_0 = 20$ keV. Horizontal field width = 255 µm. Images courtesy of Ken Robinson, Carl Zeiss SMT Ltd

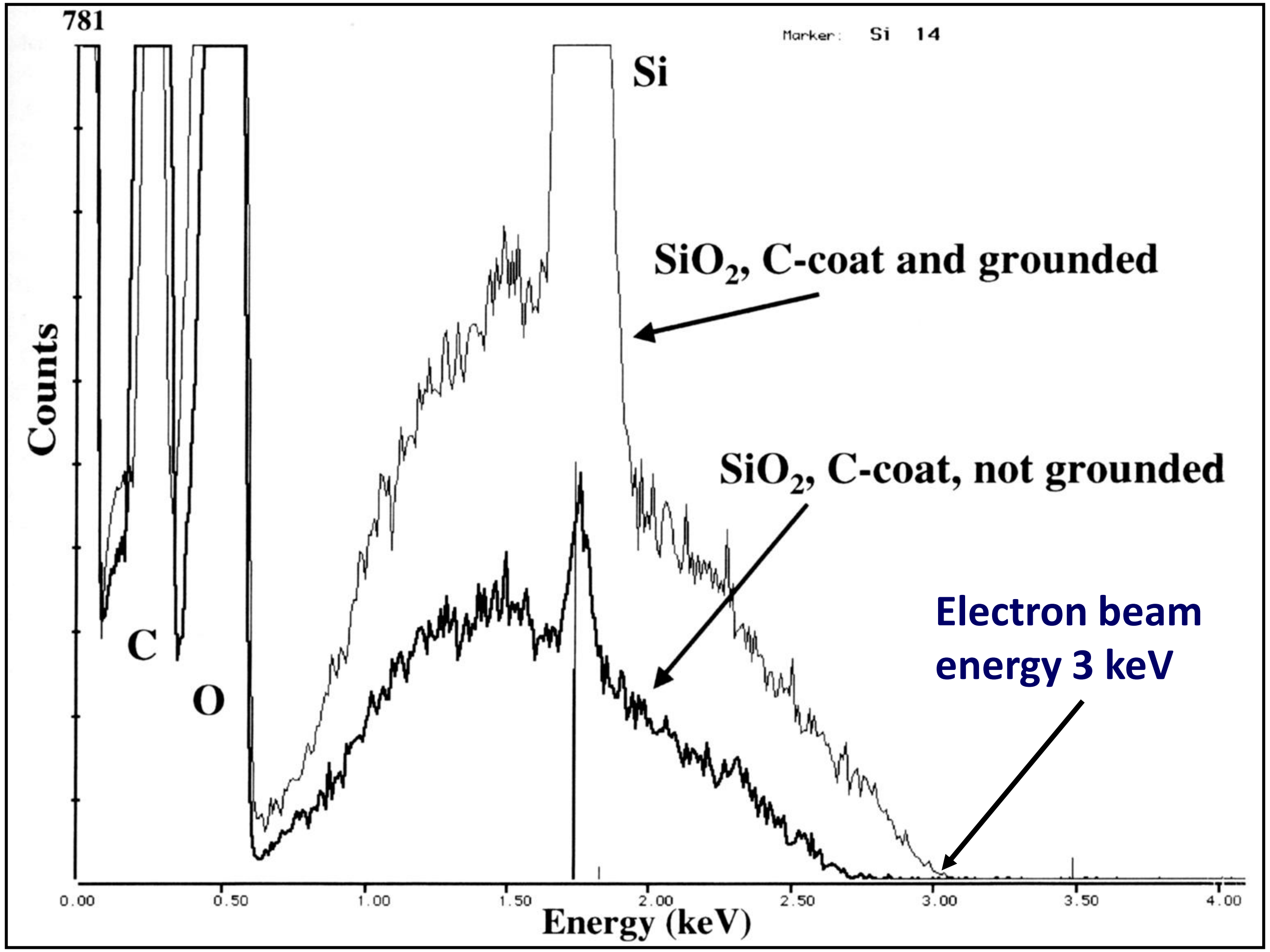
Imaging:

No problem; skirt adds a nearly uniform background

Resolution is defined by central probe



Charge control by Duane –Hunt limit



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EBSD from non-conductive samples

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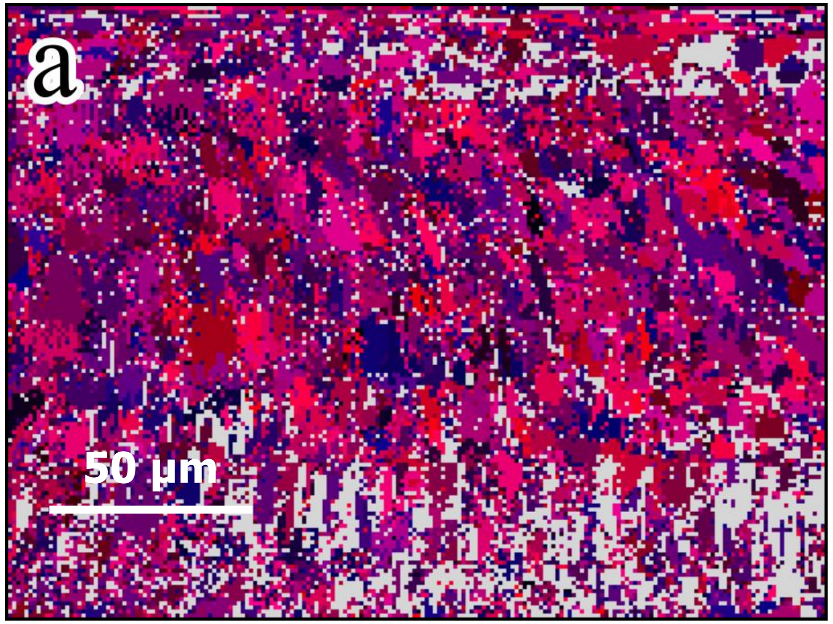
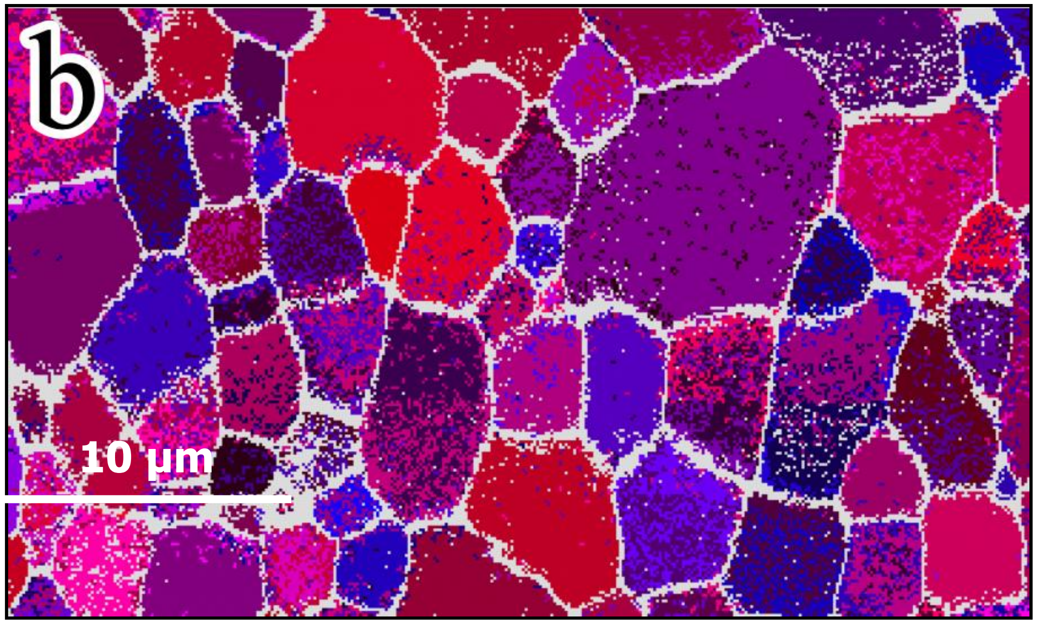
Orientation maps acquired from non-conductive smaple (cubic ZrO_2)





a) C-SEM – electric charge
non compensated
All Euler map

b) VP-SEM – electric charge
compensated
All Euler map

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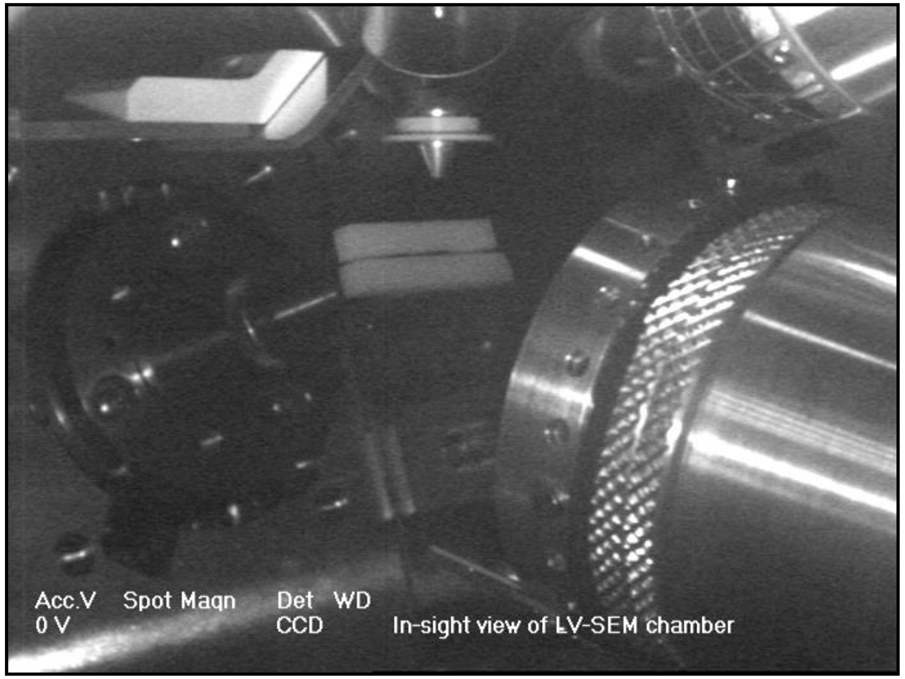
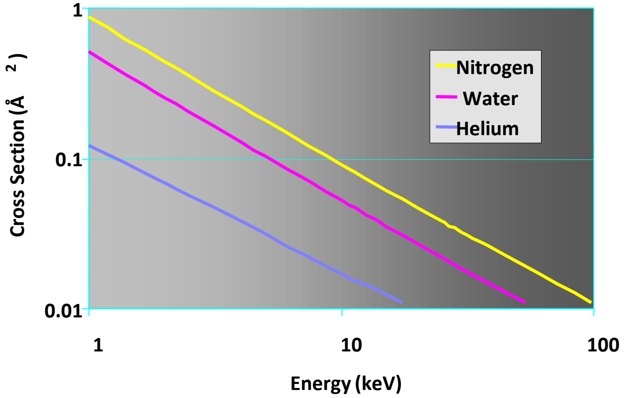
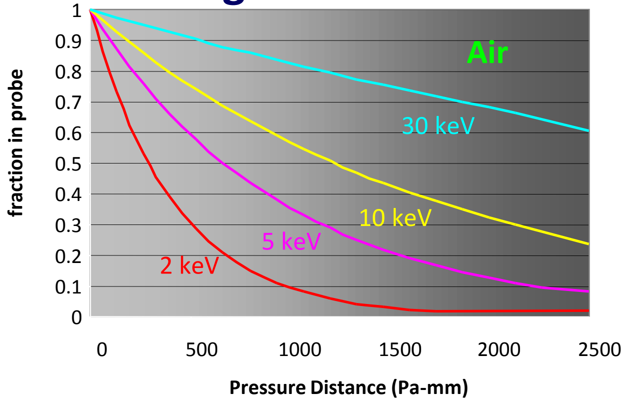


Elastic Cross Section



Scattering out of the Probe



Trade-offs between several variables:

- Pressure
- Working distance
- Type of gas
- Energy of primary electrons
- Resolution
- Charge control

Rule of thumb:
Use as low pressure and small polepiece-specimen distance as possible!

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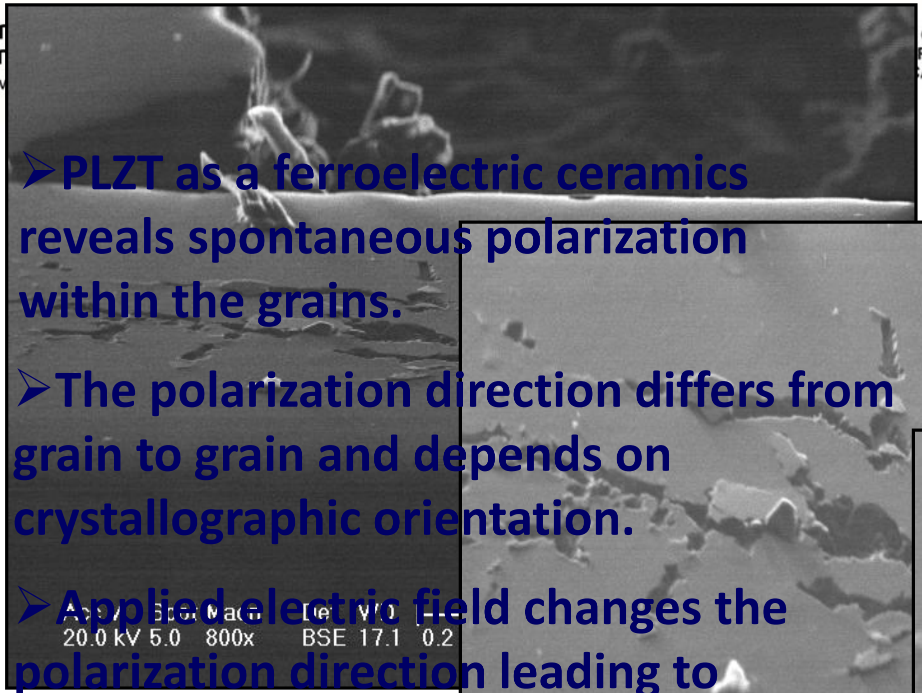
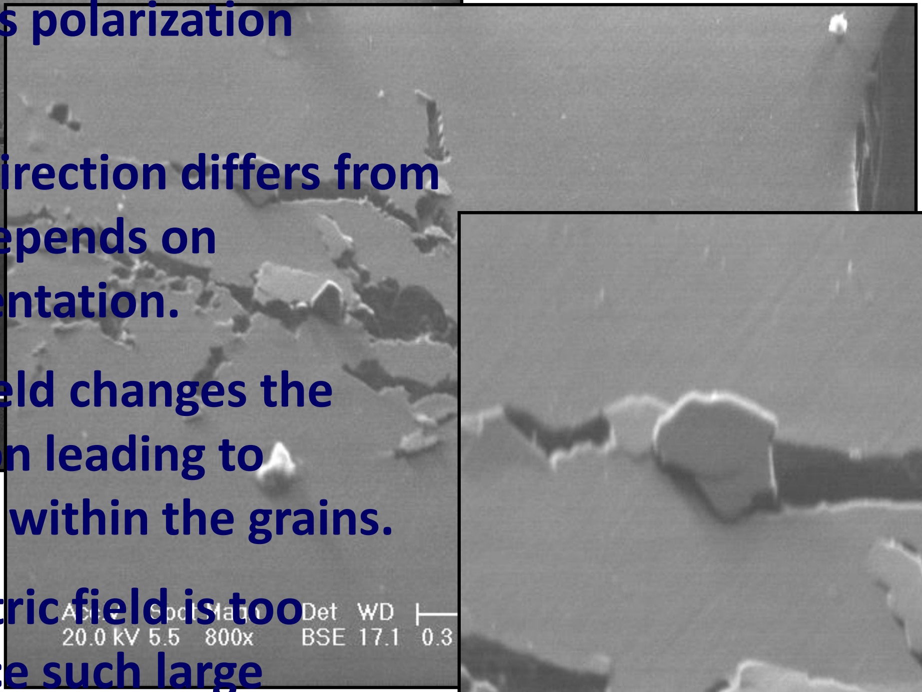
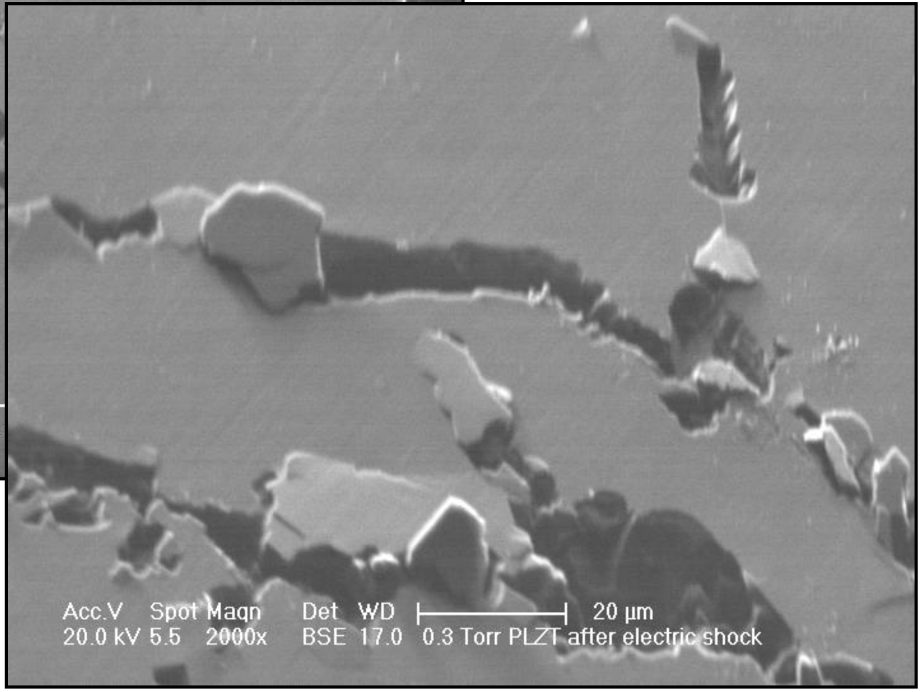
What happens when the charge is not compensated

- PLZT as a ferroelectric ceramics reveals spontaneous polarization within the grains.
- The polarization direction differs from grain to grain and depends on crystallographic orientation.
- Applied electric field changes the polarization direction leading to mechanical stresses within the grains.
- If the applied electric field is too strong, it can produce such large mechanical stress and corresponding strain within the grains that they can be pulled out and cracks are formed.







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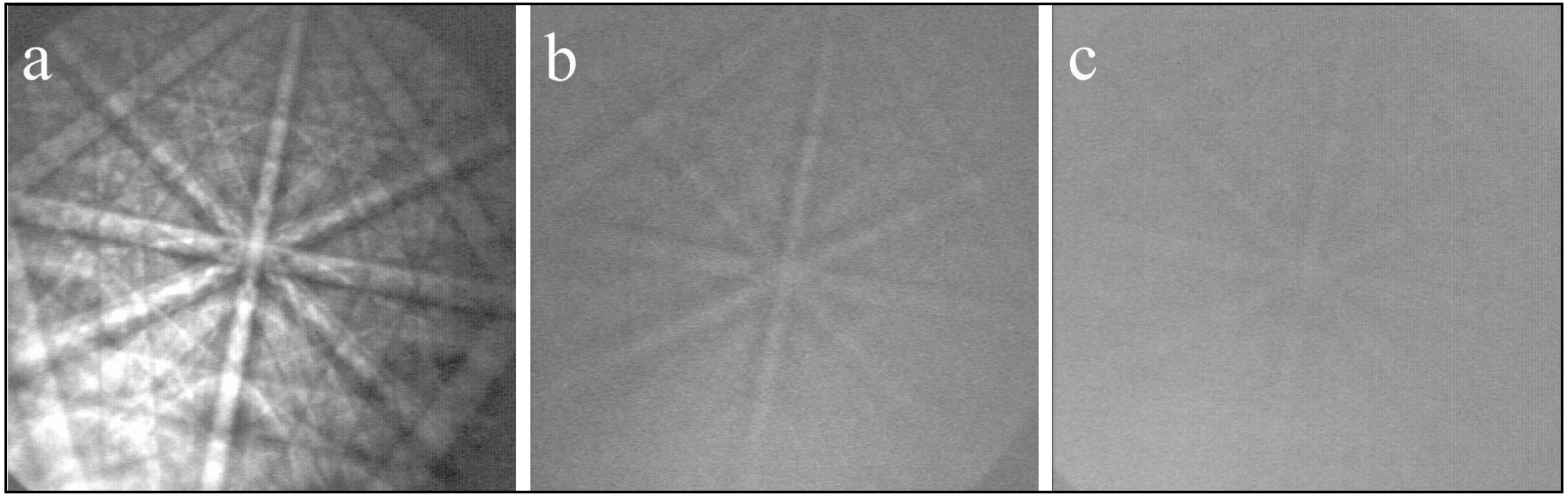

Effect of coating on diffraction pattern from NiO monocrystal
a) no coating, b) carbon coating, c) gold coating

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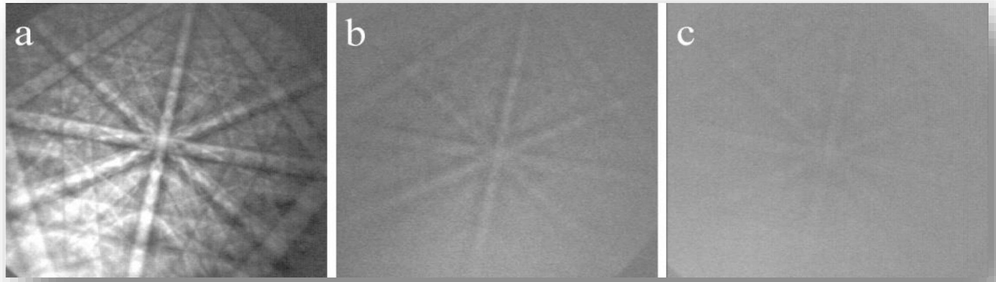
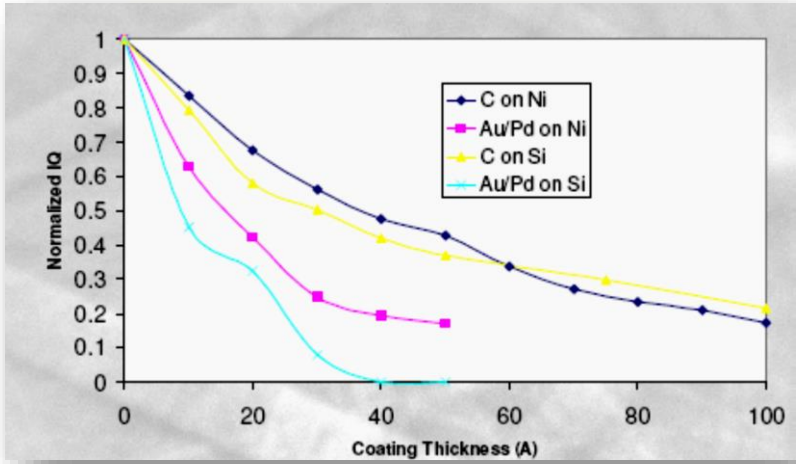
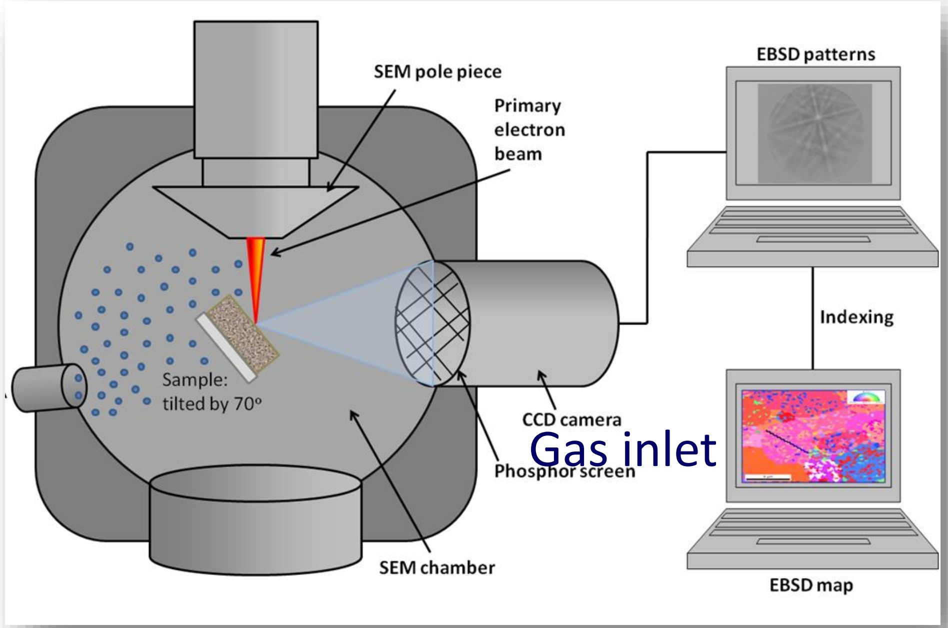


Effect of coating on diffraction patter quality
a) no coating – VP-SEM, b) carbon coating, c) gold coating



Courtesy of EDAX

Low backscatter yield of the ceramics due to low average atomic number !!!



Variable Pressure SEM seems to be a good solution to overcome charging problems!

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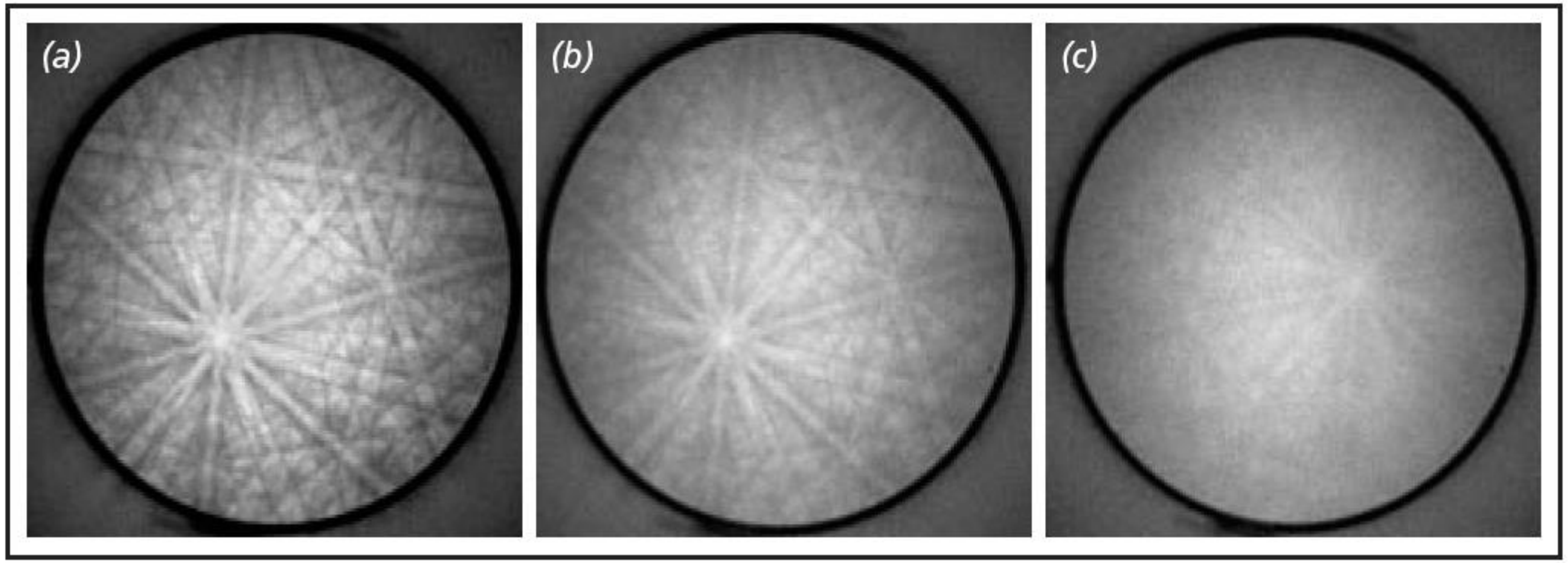
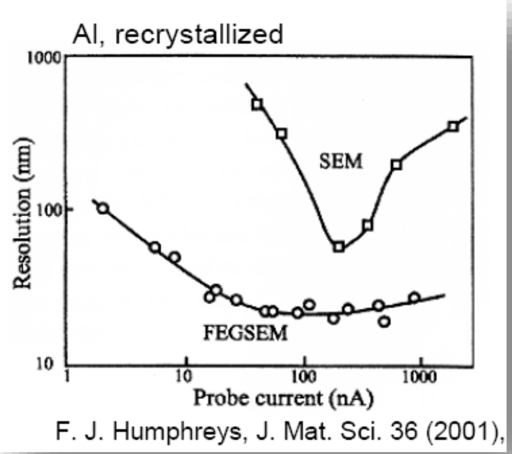
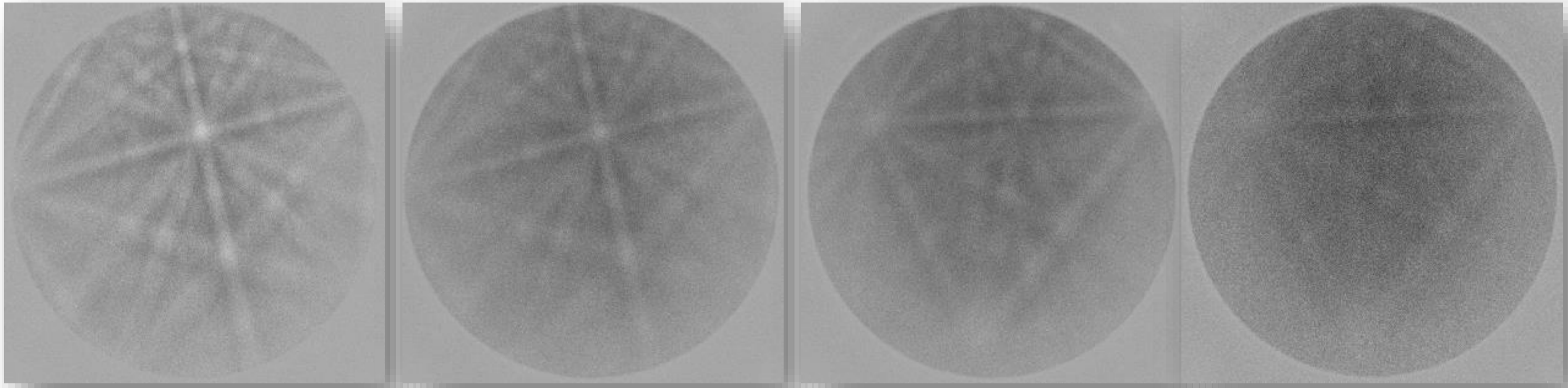


Effect of vacuum in SEM chamber on diffraction pattern from Pt
a) 0.05 Torr, b) 0.5 Torr, c) 1.0 Torr

FEGSEM

- a huge electrical charging due high beam current
- mechanical and electron beam drift





EBSD pattern recorded from the PLZT ceramics in FEGSEM at different pressures:
a) 50 Pa, b) 70 Pa, c) 90, d) 130 Pa

In order to achieve the best electron backscatter diffraction quality in low vacuum conditions, there are some parameters in the SEM which need to be adjusted, mainly:

- beam energy and current,
- gas path length,
- gas pressure,
- dwell time.

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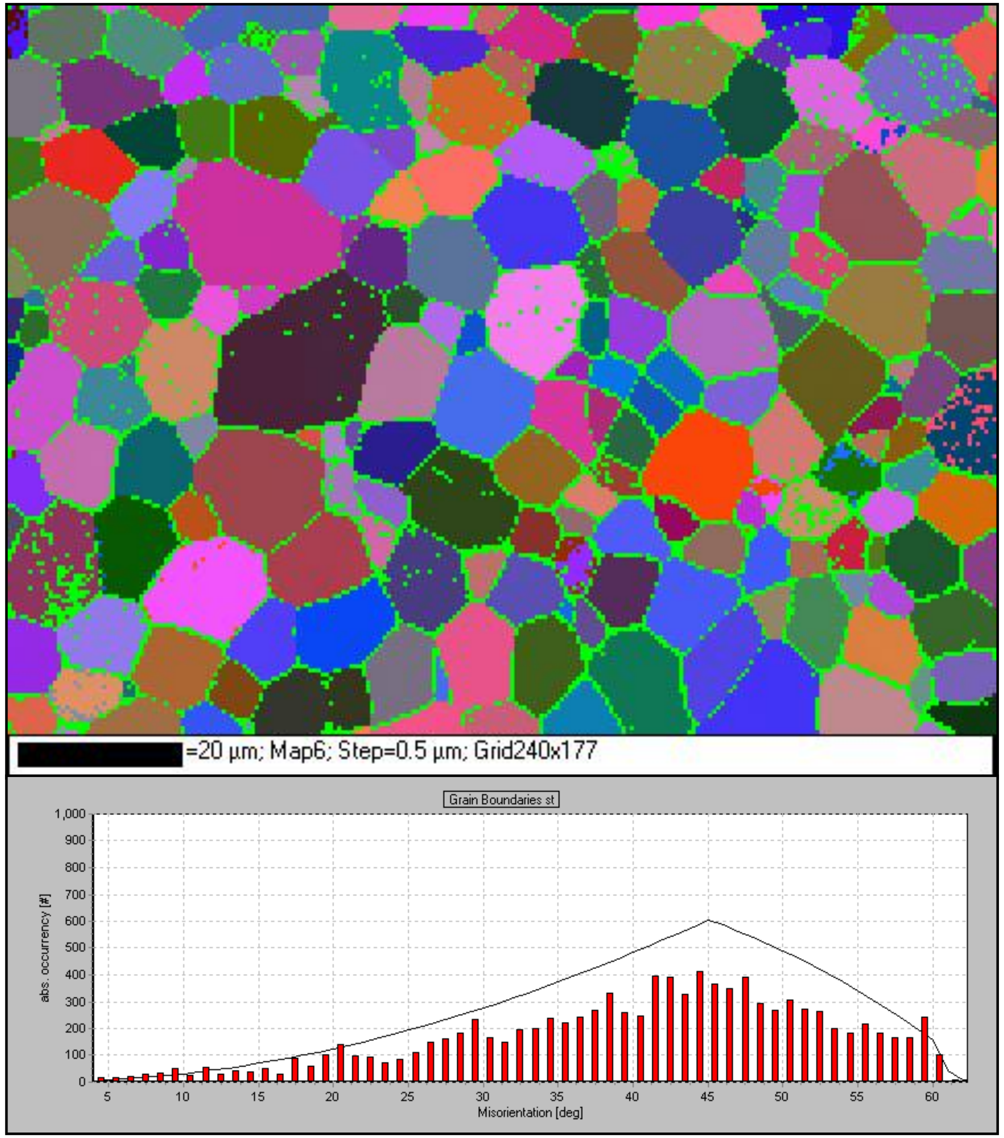
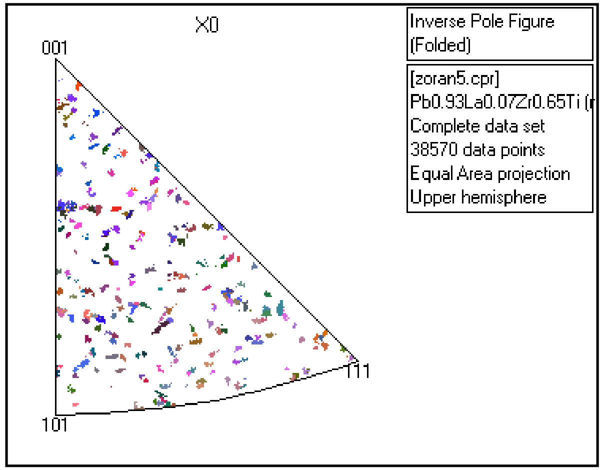
PLZT ceramics

$Pb_{1-3x/2}La_xZr_{0.65}Ti_{0.35}O_3$ for x = 0.08
(denoted as PLZT 8/65/35)

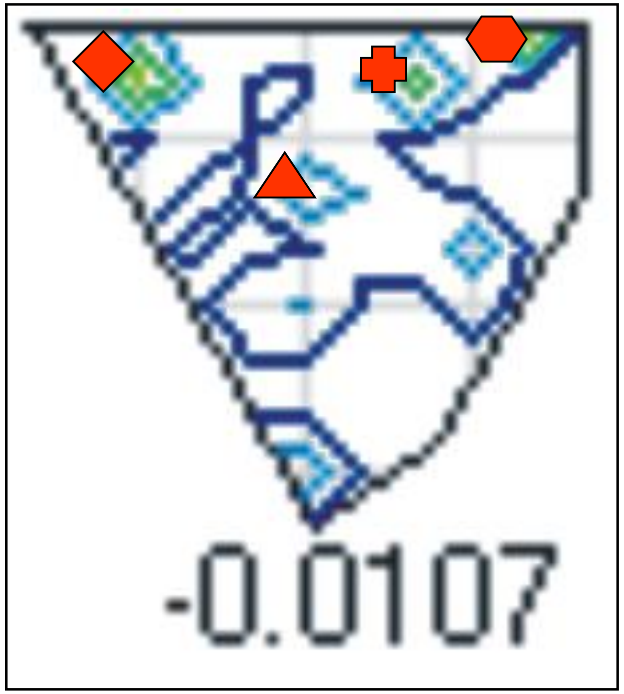
91% of diffraction patterns solved!!! All Euler map





Inverse pole figure shows randomly oriented grains

Misorientation Distribution Function (Al_2O_3/WC)



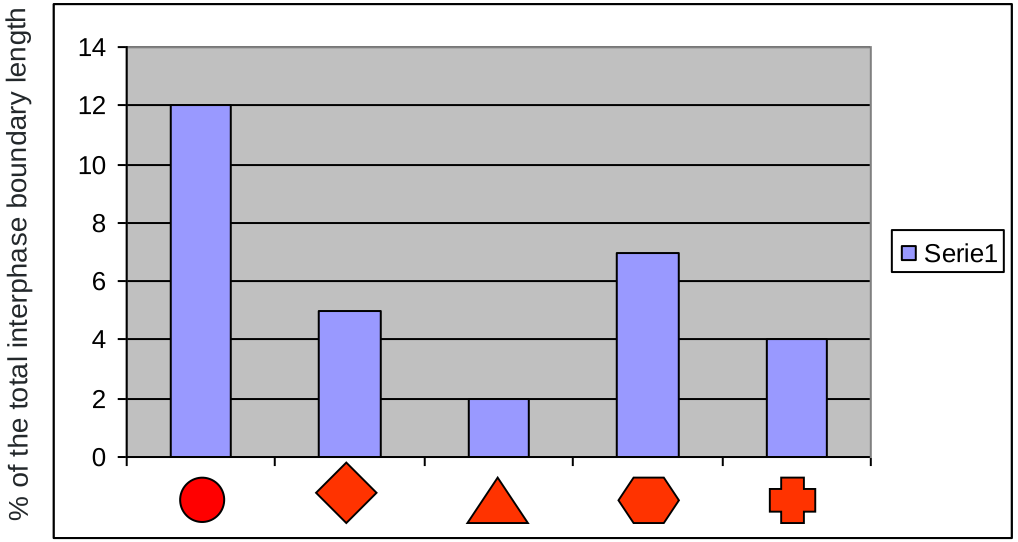
◆ $(1\,0\,\bar{1}\,0)\,WC \parallel (1\,0\,\bar{1}\,0)\,Al_2O_3$
$[0\,0\,0\,\bar{1}]\,WC \parallel [\bar{1}\,3\,\bar{2}\,\bar{2}]\,Al_2O_3$

▲ $(1\,0\,\bar{1}\,0)\,WC \parallel (1\,0\,\bar{1}\,0)\,Al_2O_3$
$[0\,0\,0\,\bar{1}]\,WC \parallel [\bar{4}\,9\,\bar{5}\,2]\,Al_2O_3$

⬢ $(2\,\bar{1}\,\bar{1}\,0)\,WC \parallel (2\,\bar{1}\,\bar{1}\,0)\,Al_2O_3$
$[0\,0\,0\,\bar{1}]\,WC \parallel [0\,1\,\bar{1}\,0]\,Al_2O_3$

✚ $(3\,\bar{1}\,\bar{2}\,0)\,WC \parallel (3\,\bar{1}\,\bar{2}\,0)\,Al_2O_3$
$[0\,1\,\bar{1}\,\overline{12}]\,WC \parallel [\bar{1}\,9\,\bar{8}\,\bar{1}]\,Al_2O_3$

Misorientation Distribution Function (Al_2O_3/ WC)



The crystallographic relationships correspond respectively to: 12%, 5%, 2%, 7% and 4% of the total Al_2O_3/WC interphase boundary length.

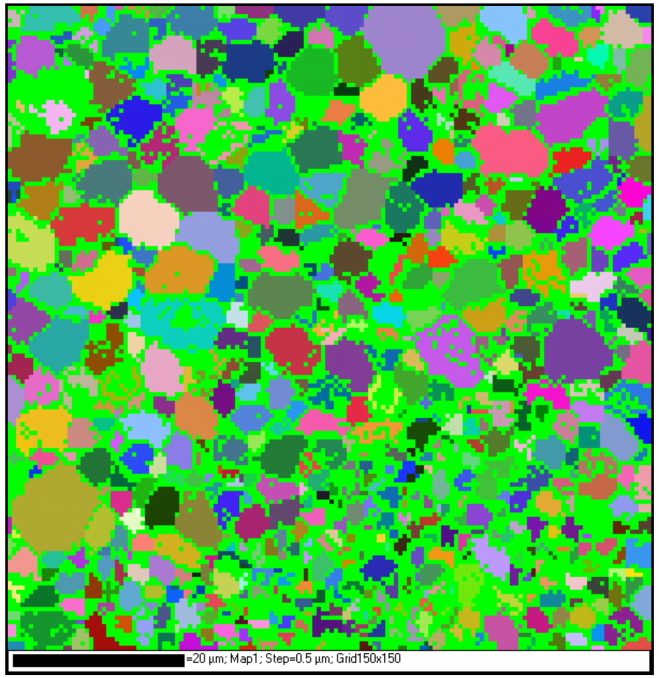
$$(0001)\,WC \parallel (0001)\,Al_2O_3 \quad [11\bar{2}0]\,WC \parallel [10\bar{1}0]\,Al_2O_3$$

$$(10\bar{1}0)\,WC \parallel (10\bar{1}0)\,Al_2O_3 \quad [000\bar{1}]\,WC \parallel [\bar{1}3\bar{2}\bar{2}]\,Al_2O_3$$

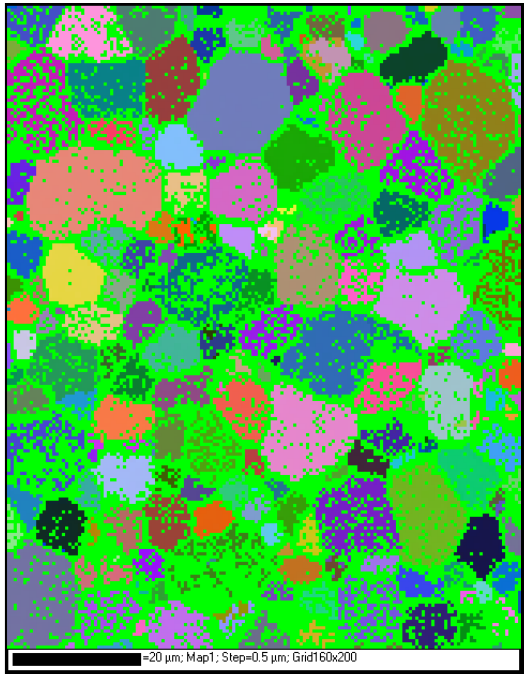
$$(10\bar{1}0)\,WC \parallel (10\bar{1}0)\,Al_2O_3 \quad [000\bar{1}]\,WC \parallel [\bar{4}9\bar{5}2]\,Al_2O_3$$

$$(2\bar{1}\bar{1}0)\,WC \parallel (2\bar{1}\bar{1}0)\,Al_2O_3 \quad [000\bar{1}]\,WC \parallel [01\bar{1}0]\,Al_2O_3$$

$$(3\bar{1}\bar{2}0)\,WC \parallel (3\bar{1}\bar{2}0)\,Al_2O_3 \quad [01\bar{1}\bar{1}2]\,WC \parallel [\bar{1}98\bar{1}]\,Al_2O_3$$



Al_2O_3 (H_2O pressure - <u>0.4 mbar</u>)



Al_2O_3 (pressure **H_2O - <u>1.33 mbar</u>)**

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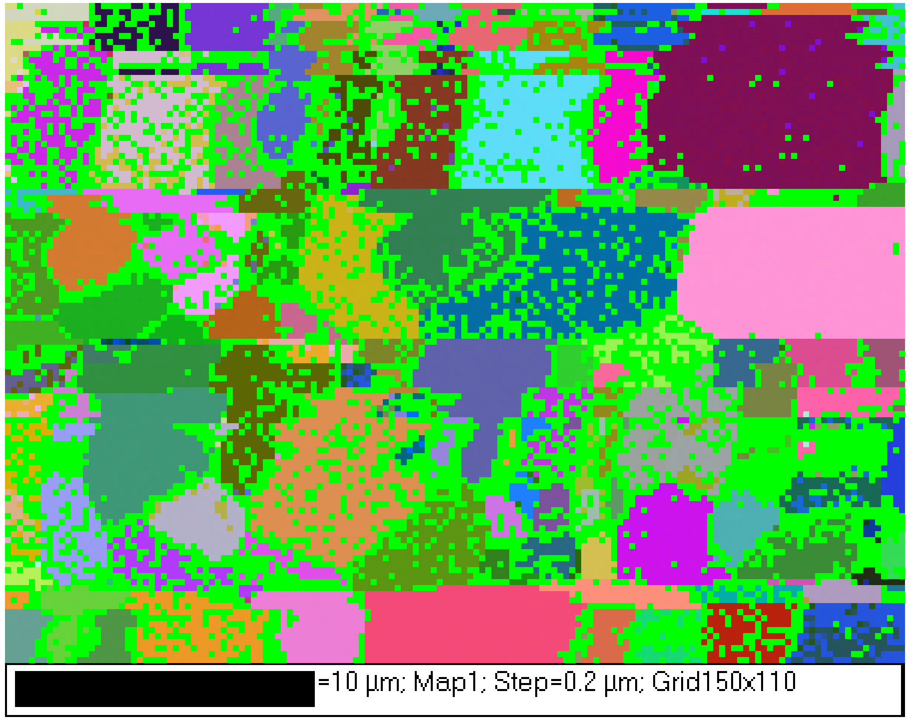
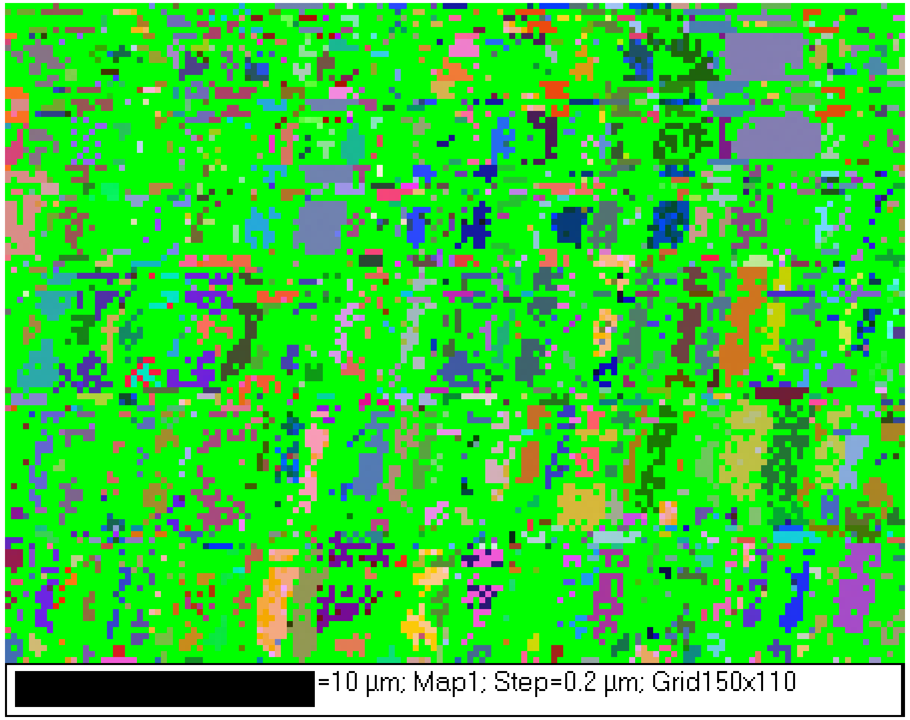
When the pressure is too low…



Coarse grained Al_2O_3



Fine grained Al_2O_3

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Thank you for attention

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