Preparation and characterization of laboratory samples

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UNIVERSITY OF HELSINKI

Outline

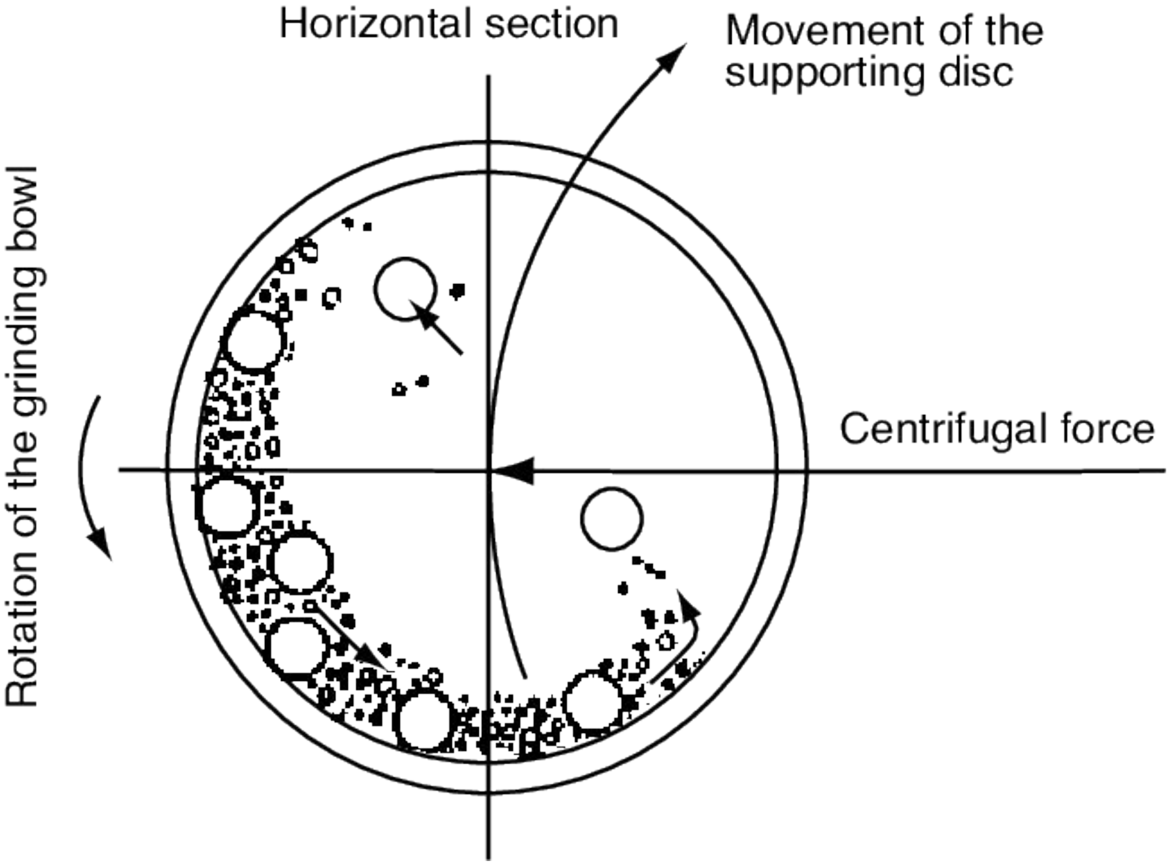
- Milling
- Sieving
- Measuring size distribution
- Characterizing the structure:SEM and FESEM
- Laboratory daily procedures (safety and cleaning)

Milling

Milling: ball milling

Ball mill

Milling: ball milling



Milling: ball milling

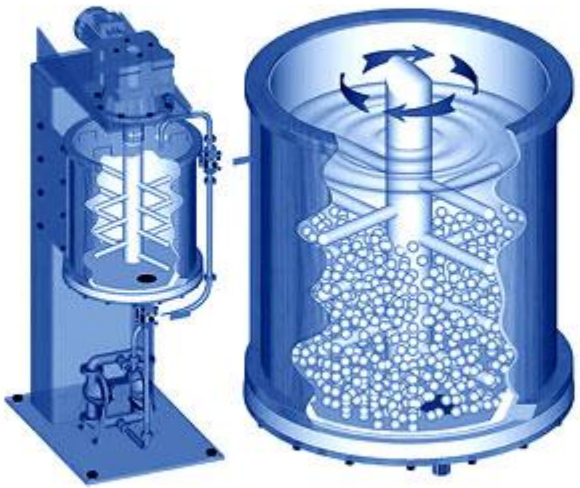
Advantages:

- It produces very fine powder (particle size less than or equal to 10 microns).
- It is suitable for milling toxic materials since it can be used in a completely enclosed form.
- Has a wide application.
- It can be used for continuous operation.
- It is used in milling highly abrasive materials.

Disadvantages:

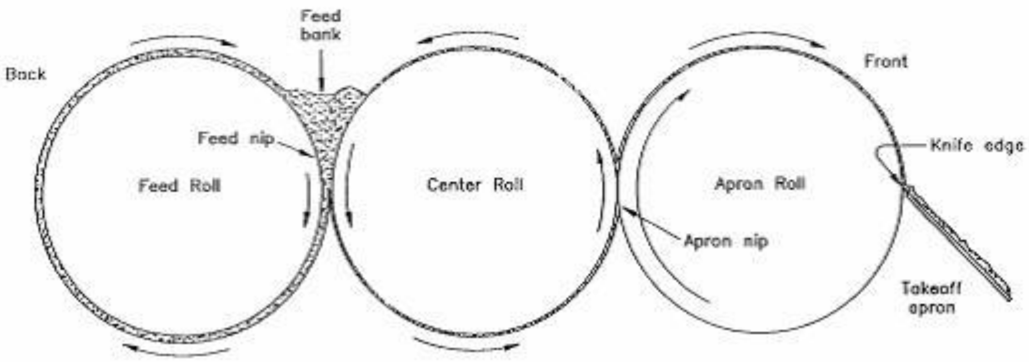
- Contamination of product may occur as a result of wear and tear which occurs principally from the balls and partially from the casing.
- High machine noise level especially if the hollow cylinder is mode of metal, but much less if rubber is used.
- Relatively long time of milling.
- It is difficult to clean the machine after use.

Milling : attrition milling



- Commonly used grinding media types are stainless steel, chrome steel, tungsten carbide, ceramic, or zirconium oxide.
- The material and the media are then agitated by the shaft with cross arms, rotating at high speed.
- This causes the media to exert both shearing and impact forces on the material, resulting in optimum size reduction and dispersion.
- No premixing is necessary. The material can be directly fed into the jacketed grinding tank of the mill.
- The final result of the grinding action is a fine, even particle dispersion.
- Temperature control (either cooling or heating) can be maintained with the use of jacketed tanks.

Milling : roll mill



Sieving

Dry sieving

Different sieve sizes

Sieve shaker

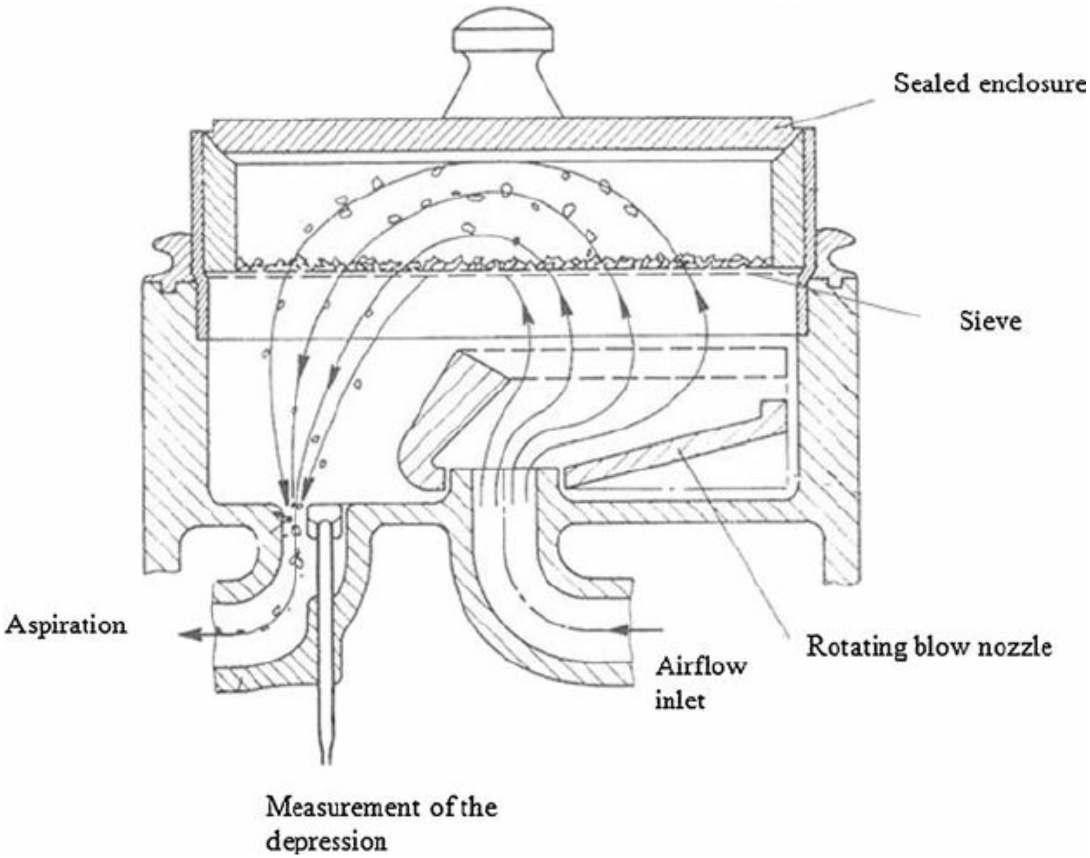
Wet sieving

It is used when the sample tends to aggregate. To recover the smallest particles we have to let dry the water remanent.

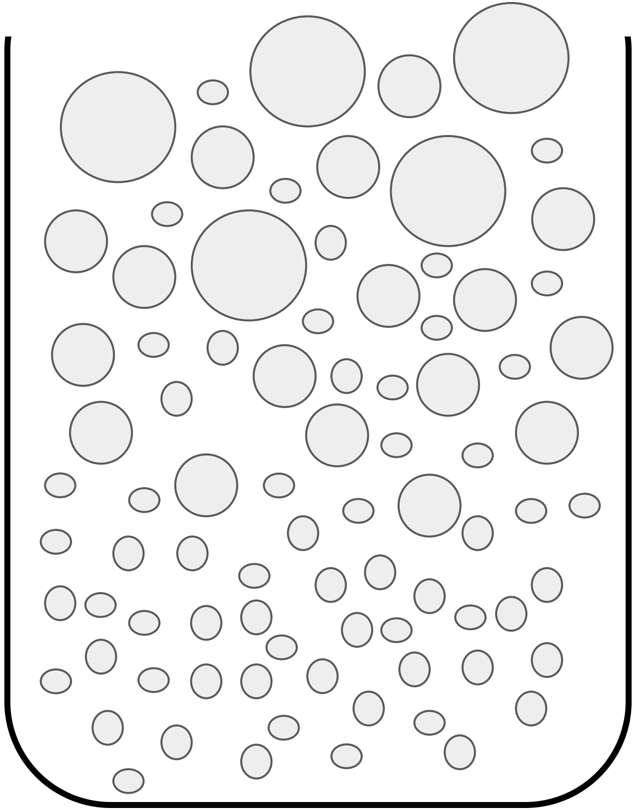
Not recommended if the sample is soluble in water.

Not recommended if the water reacts with water. Another solvent has to be considered.

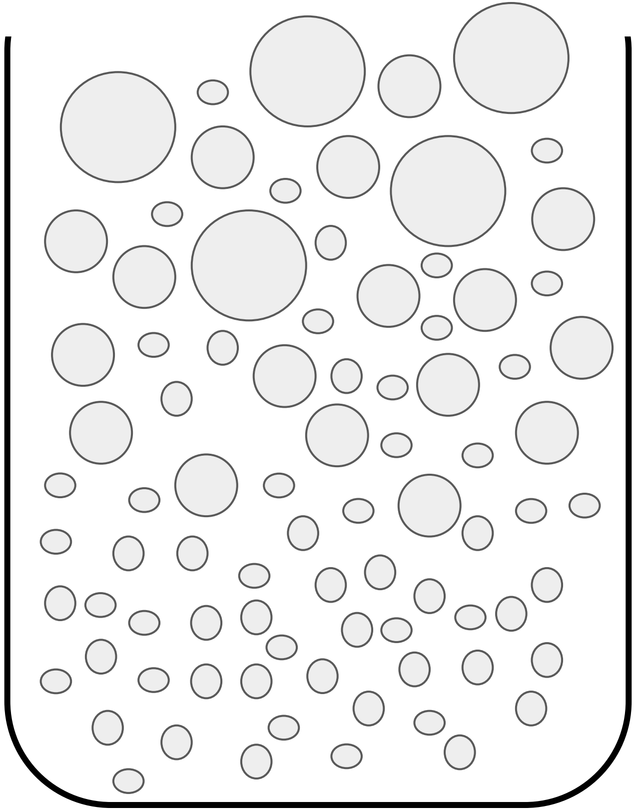
Air circulation jet sieving



Size migration in a container



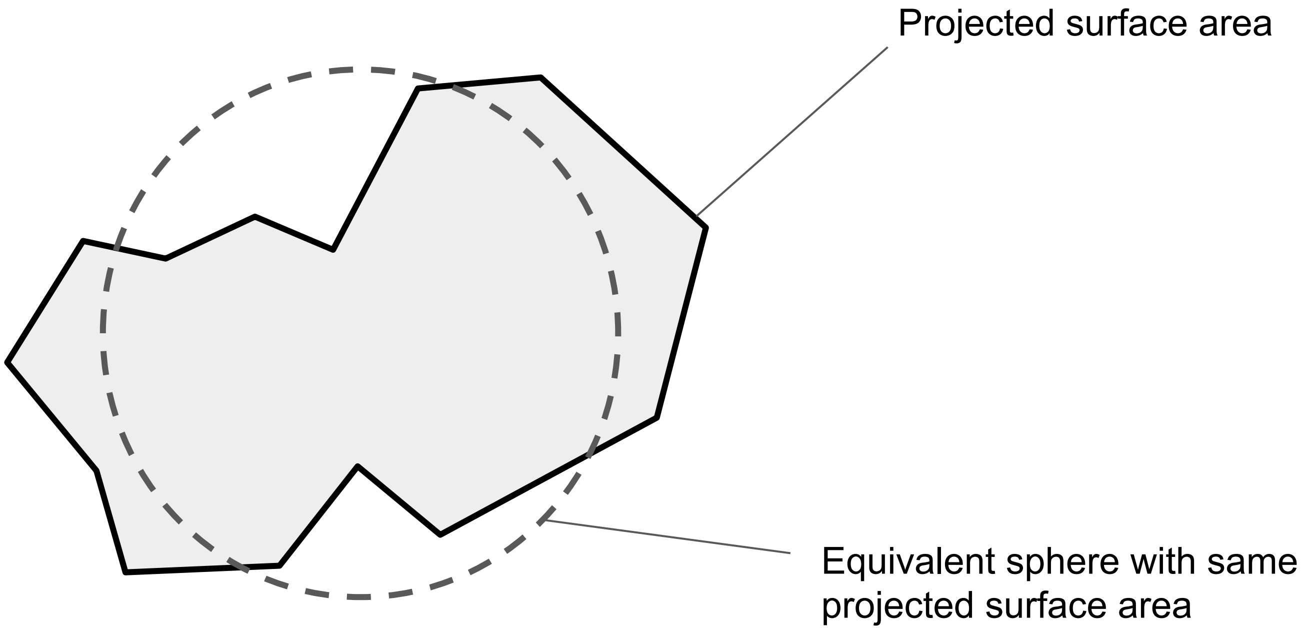
Size migration in a container



To mix it we flip the container 10 times.

Measuring size distribution

Size distribution characterization



Size distribution characterization

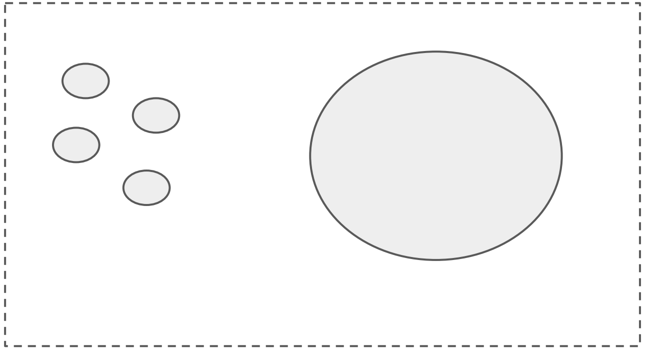
Number distributions

$$N = \int_0^\infty \nu(r)\, dr$$

Number of spheres per unit volume having a size between *r* and *r*+dr

Number distribution

Total number of spheres per unit volume



$$n(r) = \nu(r)/N \rightarrow \int_0^\infty n(r)\, dr = 1$$

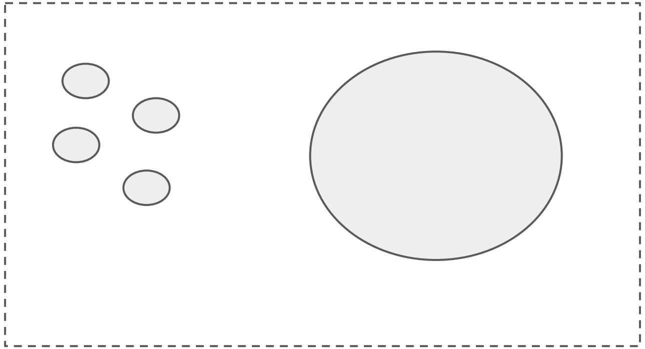
Normalized number distribution

Size distribution characterization

Volume distributions

$$\mathtt{V} = \int_0^\infty \nu(r)(\frac{4}{3}\pi r^3)\,\mathrm{d}r$$

Total volume occupied by the sample



$$v(r) = \frac{\nu(r)\frac{4}{3}\pi r^3}{\mathtt{V}}$$

$$\longrightarrow \quad \int_0^\infty v(r)\,\mathrm{d}r = 1$$

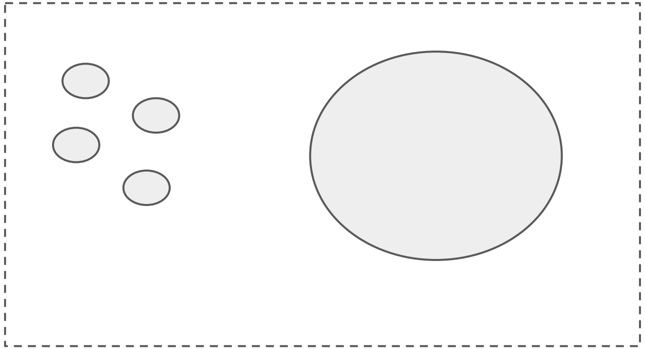
Normalized volume distribution

Size distribution characterization

Projected-surface-area distributions

$$\frac{\int_{r_1}^{r_2} \nu(r)\pi r^2\,\mathrm{d}r}{\int_0^{\infty} \nu(r)\pi r^2\,\mathrm{d}r} = \frac{\int_{r_1}^{r_2} \nu(r)\pi r^2\,\mathrm{d}r}{\mathsf{S}} = \int_{r_1}^{r_2} s(r)\,\mathrm{d}r$$

Projected-surface-area distribution



$$s(r) = \frac{\nu(r)\pi r^2}{\mathsf{S}} = \frac{\nu(r) r^2}{\int_0^{\infty} \nu(r) r^2\,\mathrm{d}r} \longrightarrow \int_0^{\infty} s(r)\,\mathrm{d}r = 1$$

Normalized projected-surface-area distribution

Size distribution characterization

Used as weighting function

$$r_{\text{eff}} = \frac{\int_0^\infty r \pi r^2 n(r)\,\mathrm{d}r}{\int_0^\infty \pi r^2 n(r)\,\mathrm{d}r} = \frac{3}{4}\frac{\mathtt{V}}{\mathtt{S}} = \int_0^\infty r s(r)\,\mathrm{d}r$$

$$\sigma_{\text{eff}} = \sqrt{\frac{\int_0^\infty (r - r_{\text{eff}})^2 \pi r^2 n(r)\,\mathrm{d}r}{r_{\text{eff}}^2 \int_0^\infty \pi r^2 n(r)\,\mathrm{d}r}} = \sqrt{\frac{\int_0^\infty (r - r_{\text{eff}})^2 s(r)\,\mathrm{d}r}{r_{\text{eff}}^2 \int_0^\infty s(r)\,\mathrm{d}r}}$$

Size distribution characterization

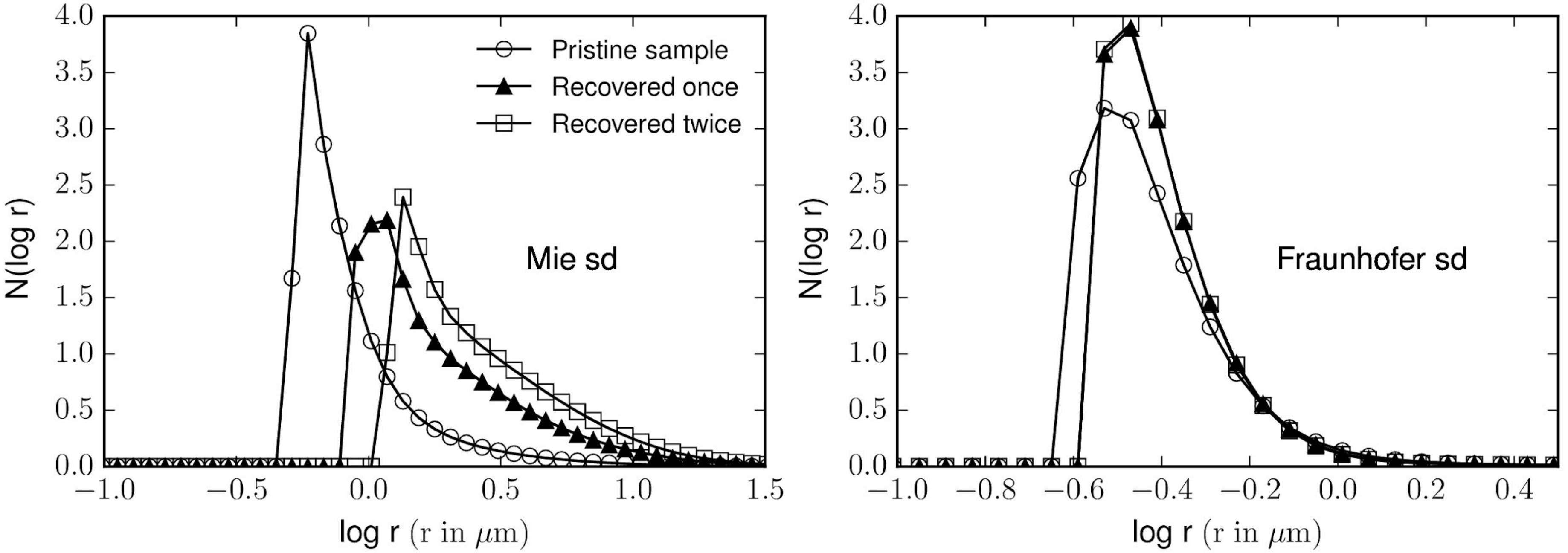
$$\int_{r_1}^{r_2} n(r)\,\mathrm{d}r = \int_{\log r_1}^{\log r_2} N(\log r)\,\mathrm{d}\log r = \int_{r_1}^{r_2} [N(\log r)\frac{\mathrm{d}\log r}{\mathrm{d}r}]\,\mathrm{d}r = \int_{r_1}^{r_2} \frac{N(\log r)}{r\ln 10}\,\mathrm{d}r$$

$$N(\log r) = \ln 10 r n(r) = 2.303 r n(r)$$

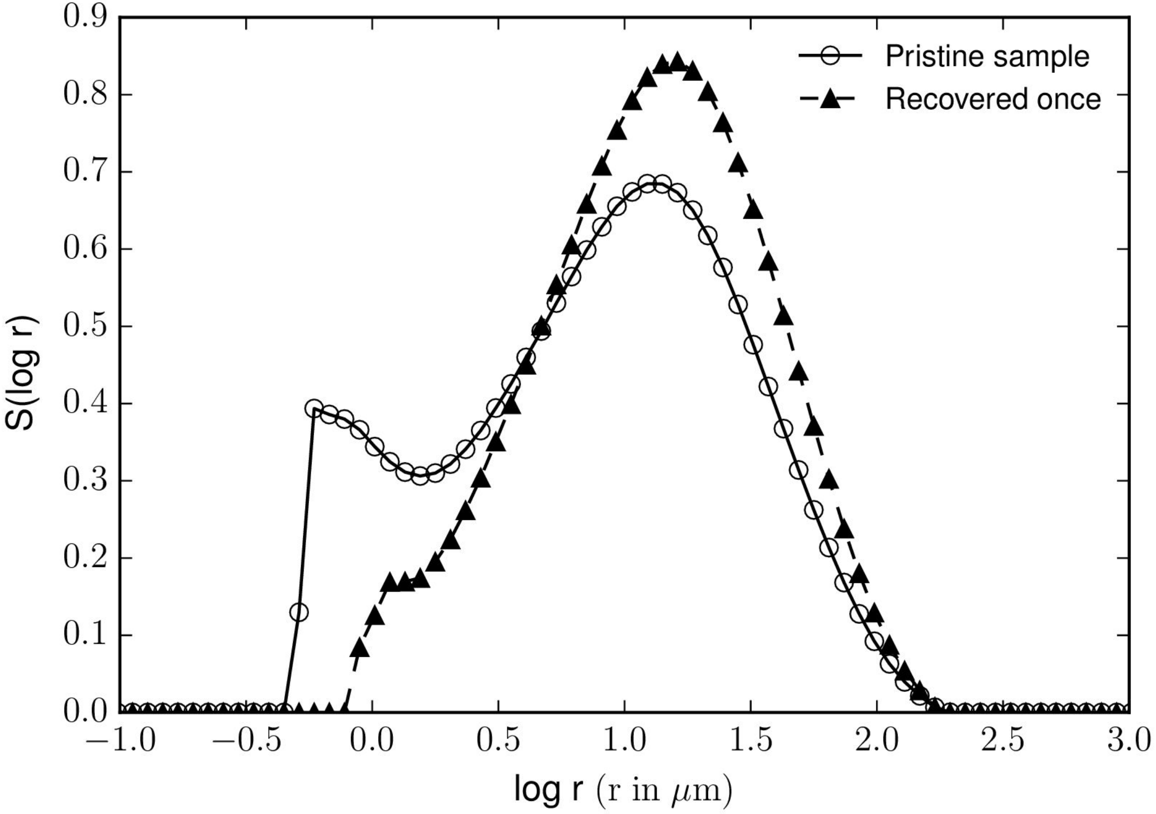
$$S(\log r) = \ln 10 r s(r) = 2.303 r s(r)$$

$$V(\log r) = \ln 10 r v(r) = 2.303 r v(r)$$

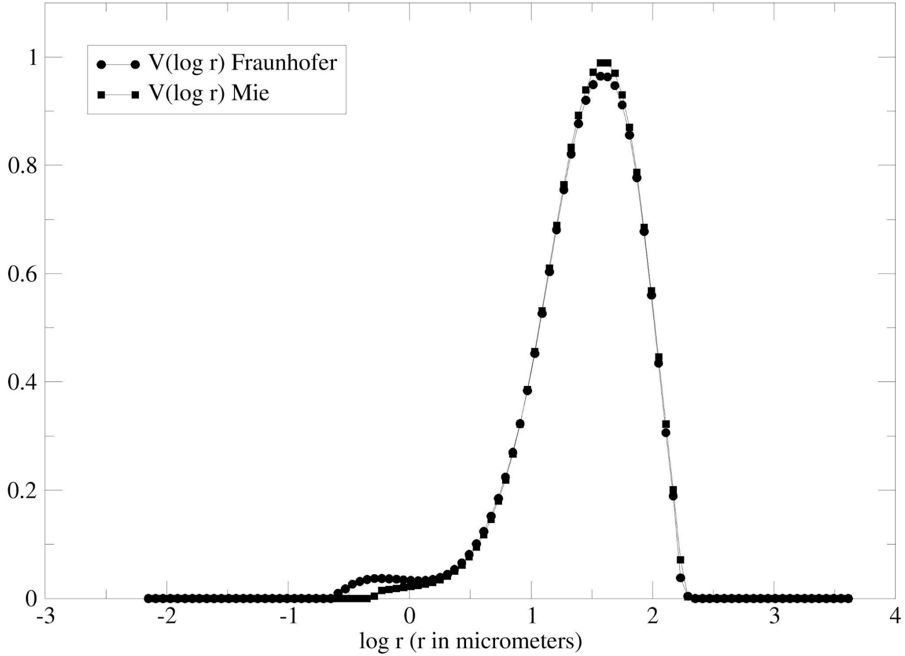
Size distribution characterization



Size distribution characterization



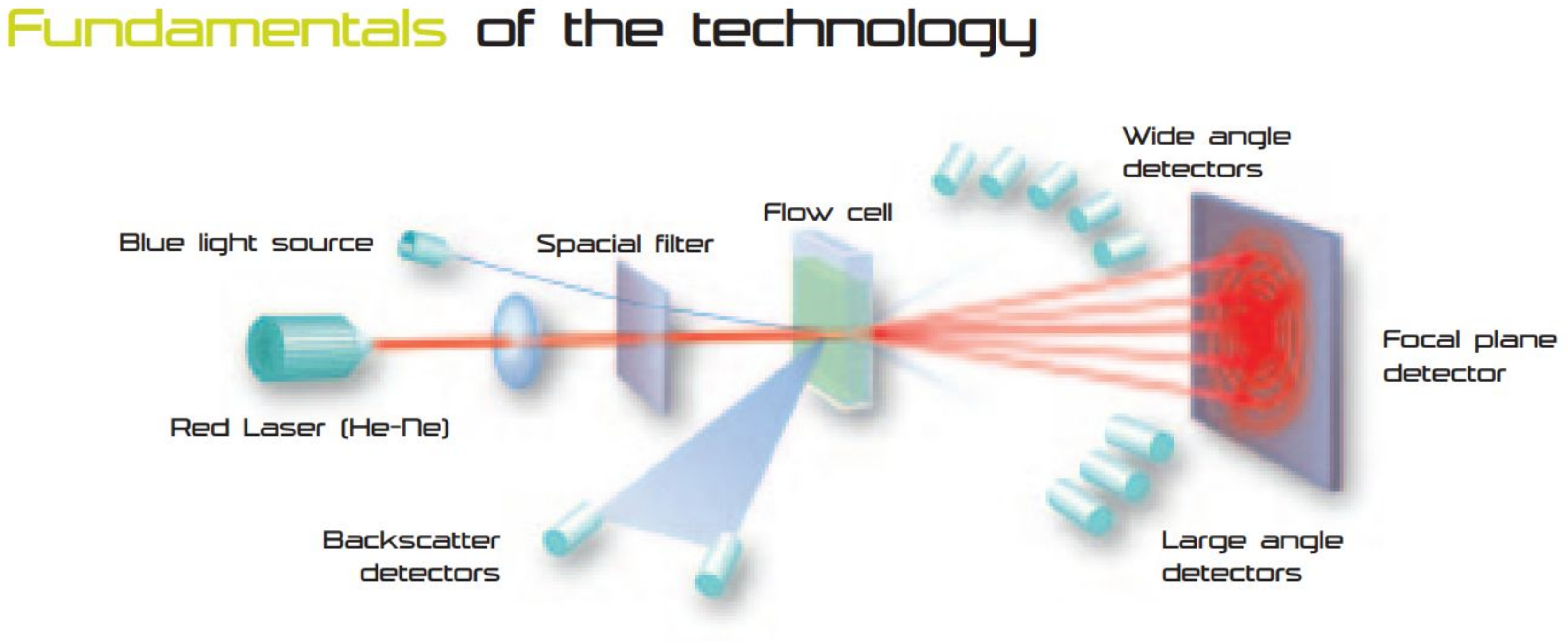
Size distribution characterization



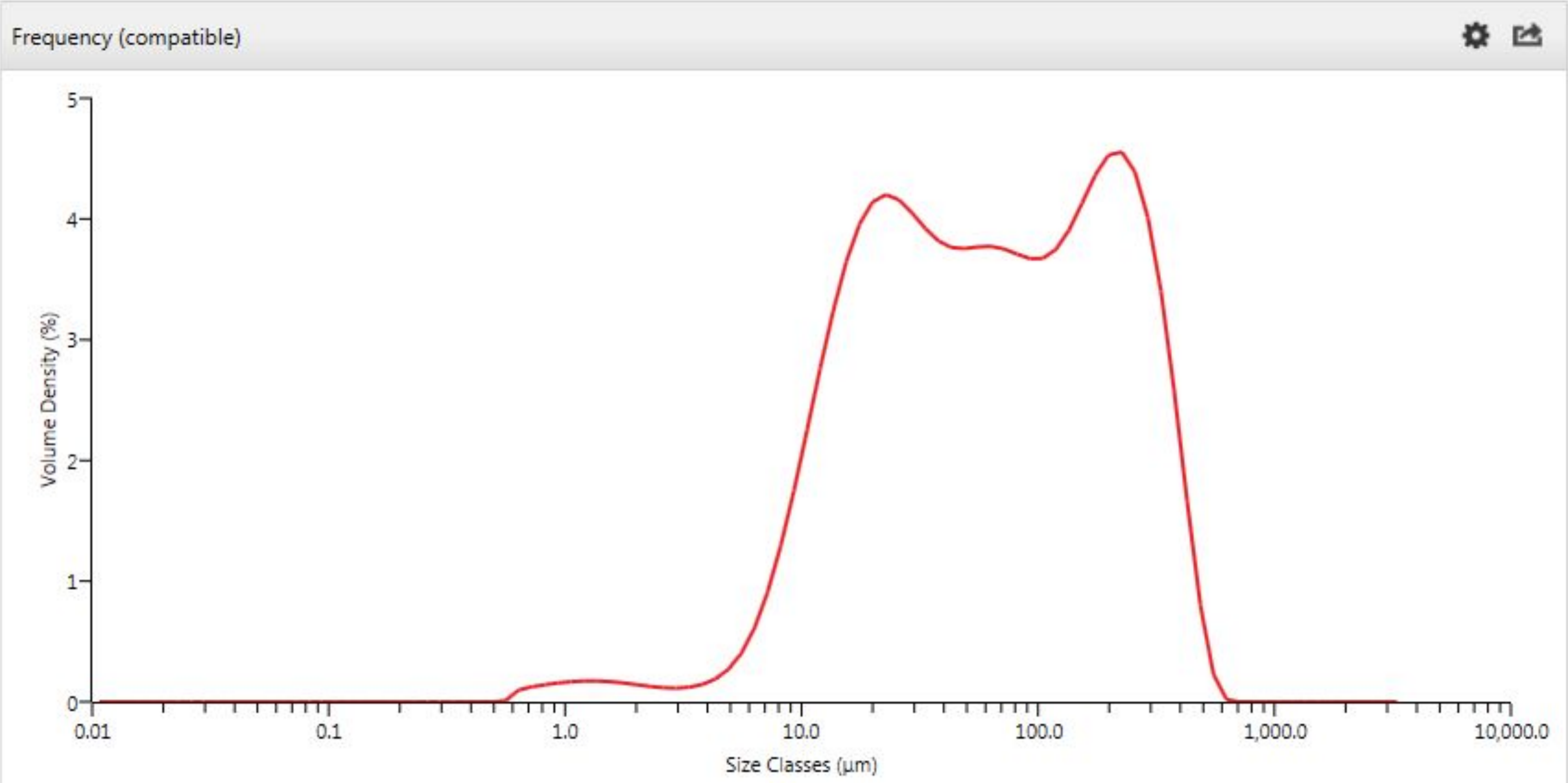
Size distribution characterization

Mastersizer 2000 from Malvern Instruments

Size distribution characterization



Size distribution characterization



Characterizing the structure: SEM and FESEM

SEM and FESEM

SEM: Scanning Electron Microscopy

FESEM: Field Emission Scanning Electron Microscopy

SEM and FESEM

Here in the University of Helsinki you can find these services at:

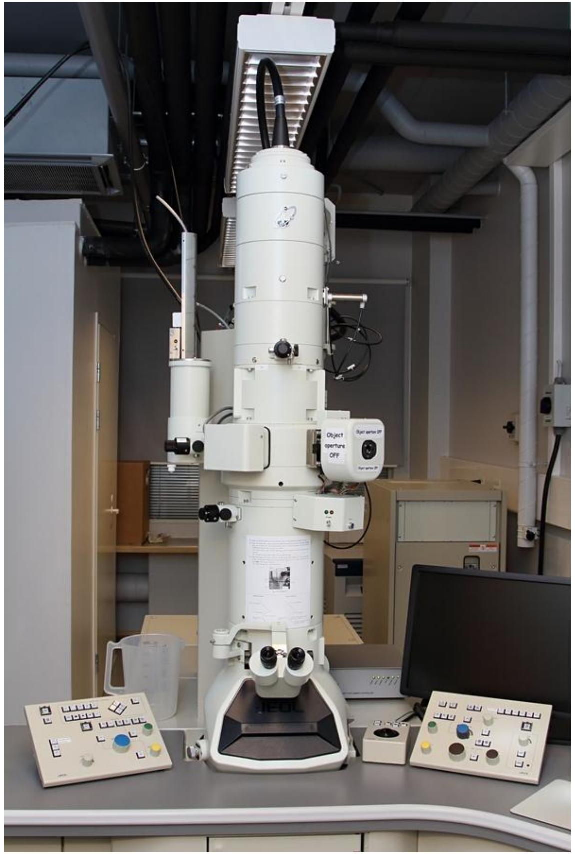
- EMBI: Electron Microscopy Institute of Biotechnology at Viikki Campus offers both SEM and FESEM

- In the laboratory of nanomaterials (Helsinki Accelerator Laboratory, Kumpula Campus) they offer Atomic Force Microscopy (AFM) and Scanning Tunneling Microscopy (STM) just to mention a couple of examples.

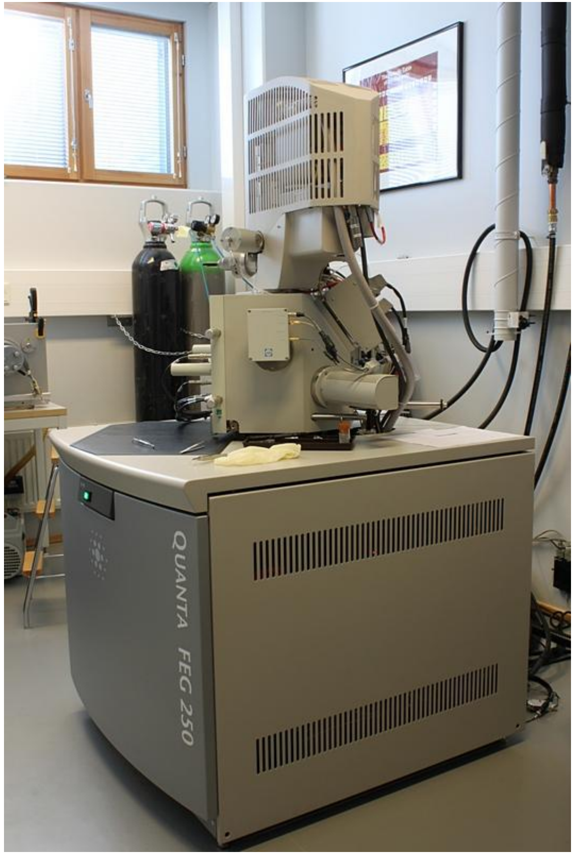
SEM and FESEM

Hitachi S-510 SEM

SEM and FESEM



Jeol JEM-1400 Transmission Electron Microscope



FEI Quanta 250 Field Emission Gun Scanning Electron Microscope

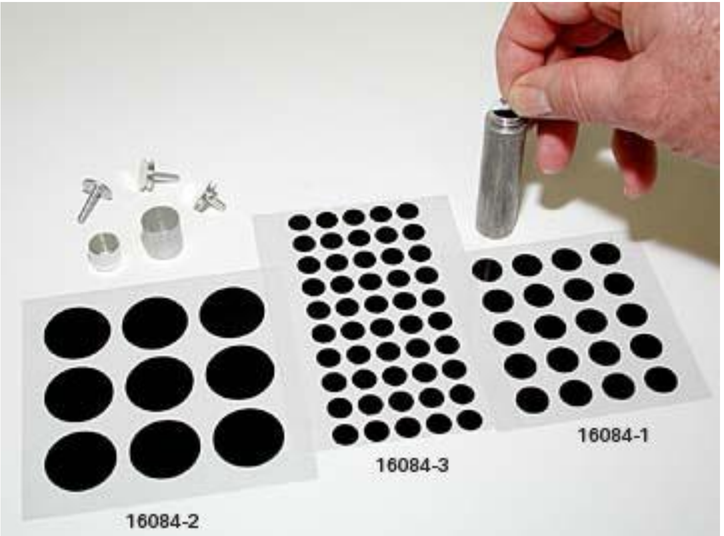
SEM and FESEM

SEM stubs

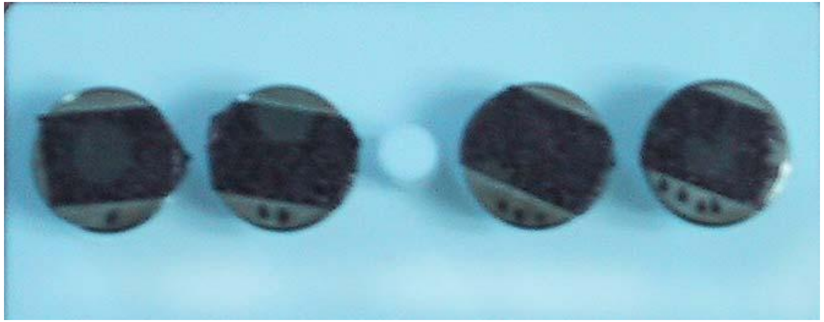
SEM and FESEM

SEM stubs

SEM and FESEM



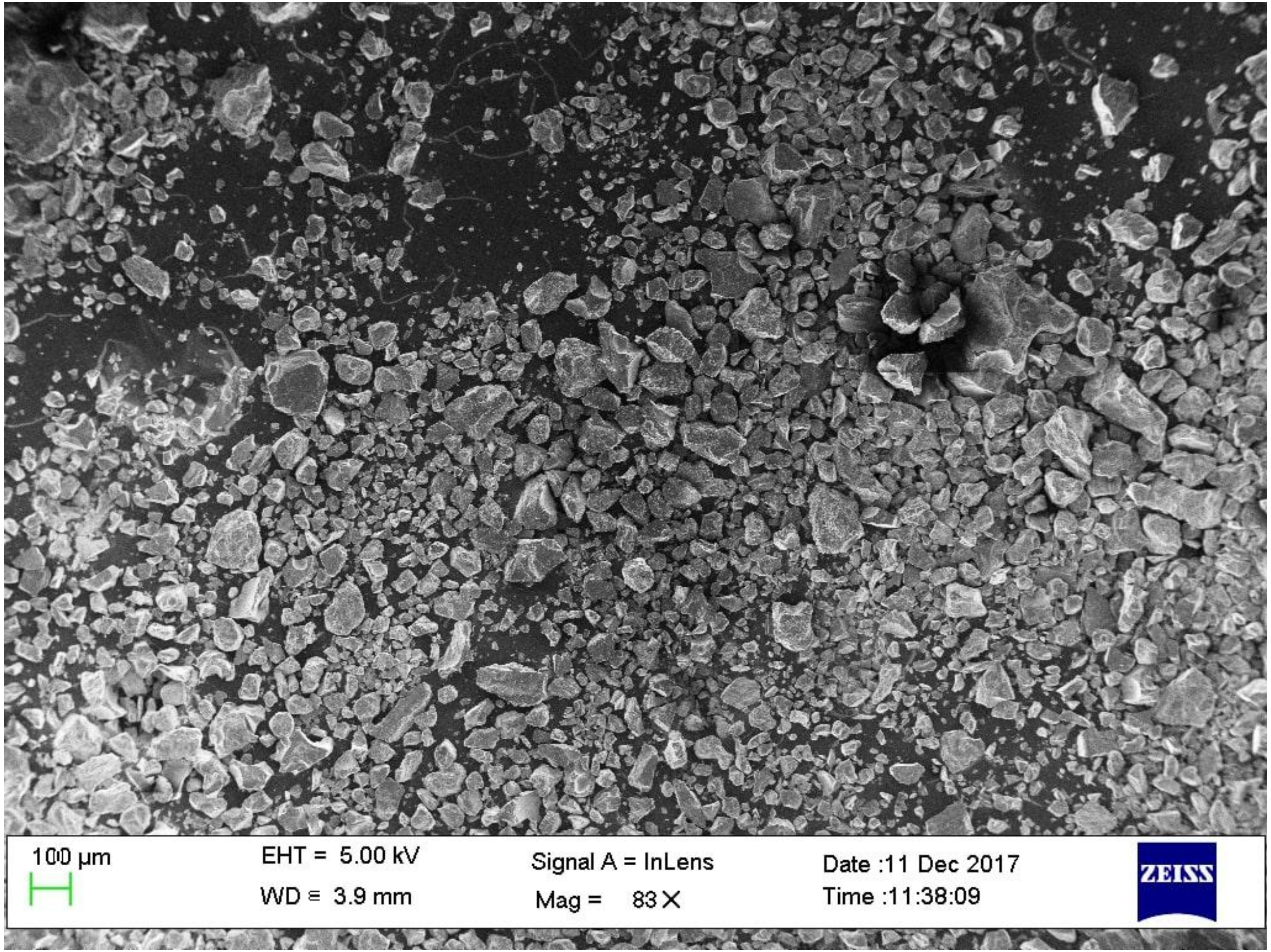
Carbon conductive tape



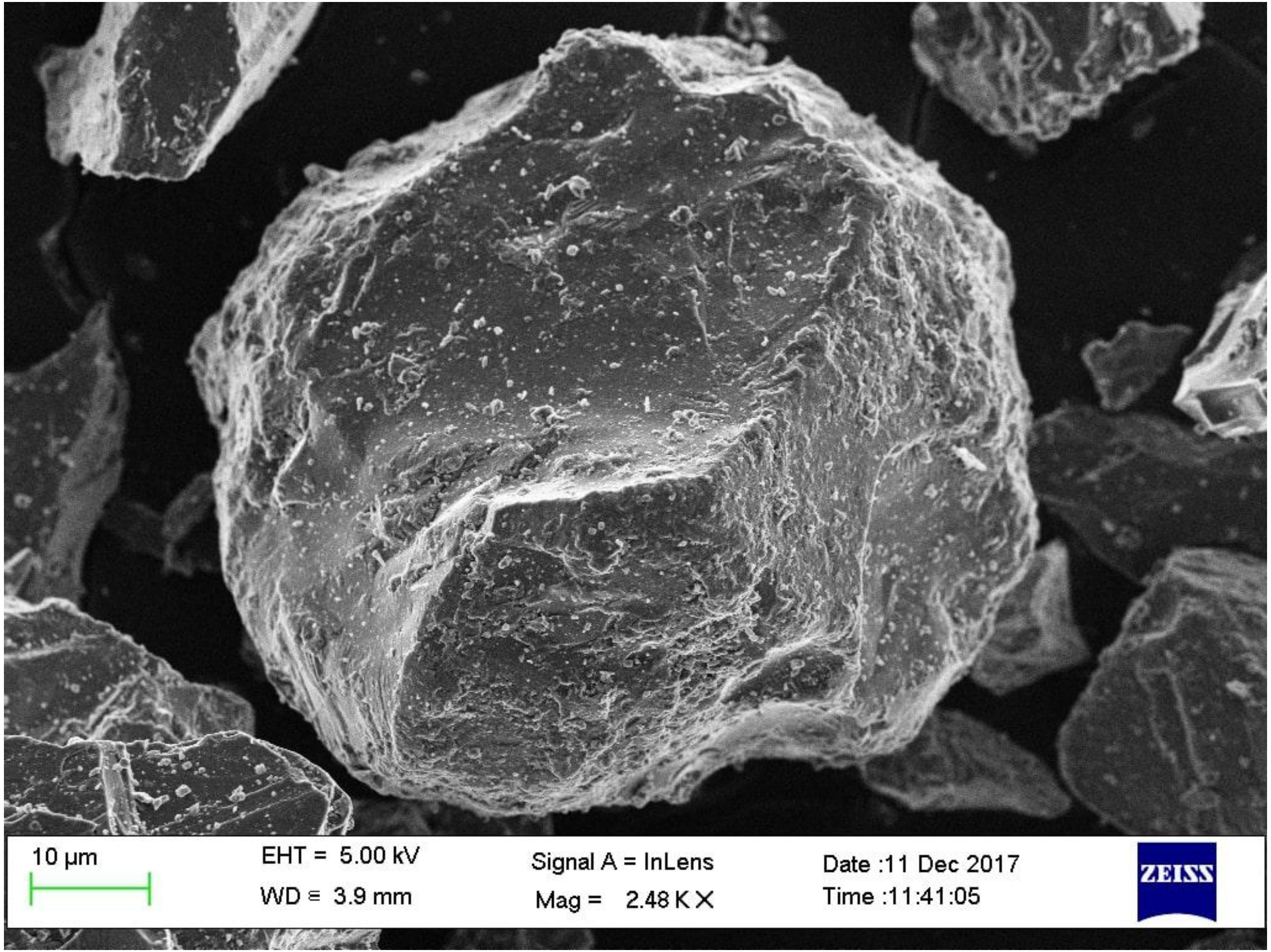
SEM and FESEM

<u>Platinum or carbon coating</u>. <u>Nonconductive specimens</u> collect charge when scanned by the electron beam. This causes <u>scanning faults</u> and other image <u>artifacts</u>. **Specimens must be electrically conductive**, at least at the surface, and electrically grounded to prevent the accumulation of electrostatic charge.

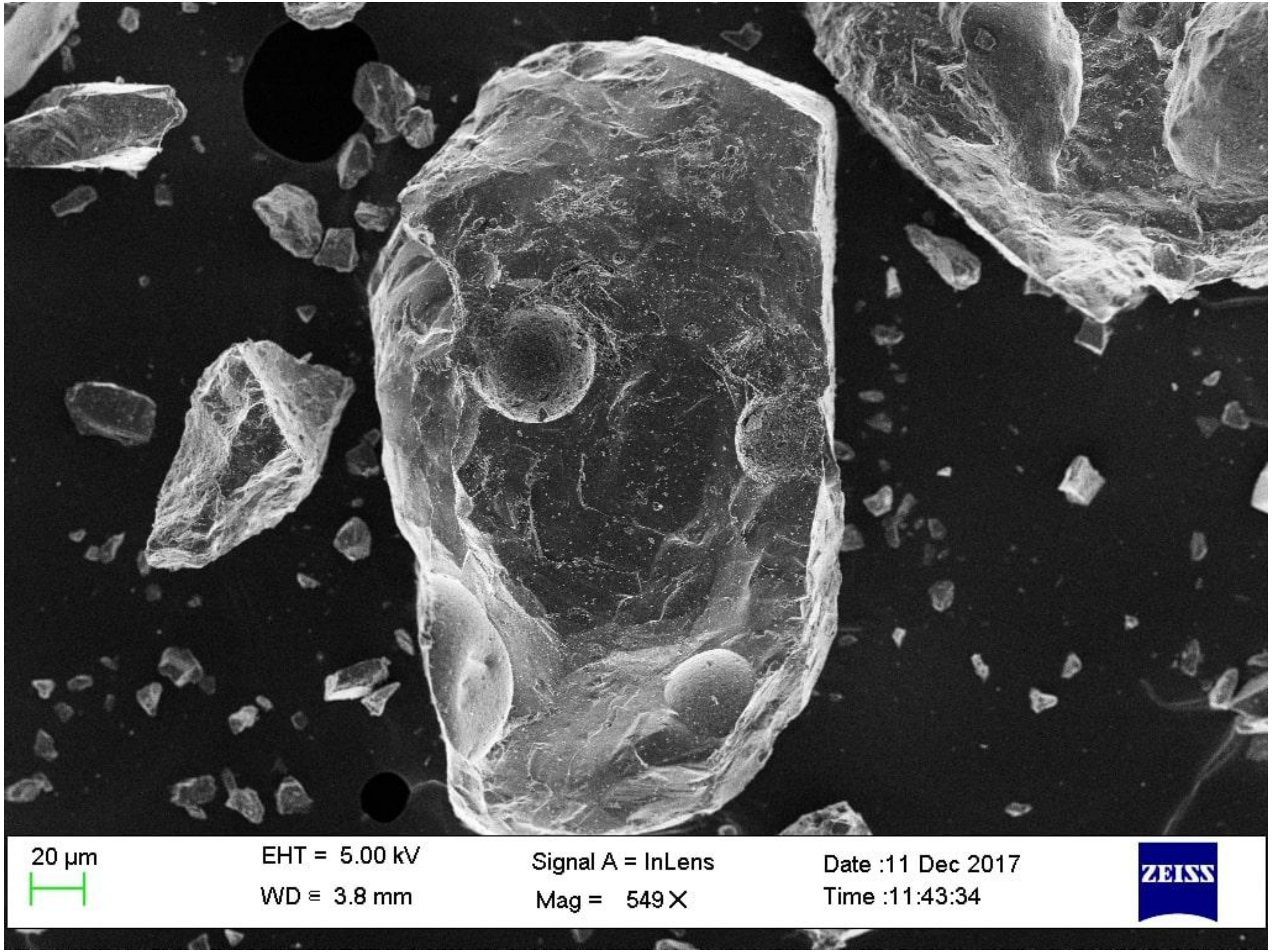
SEM and FESEM



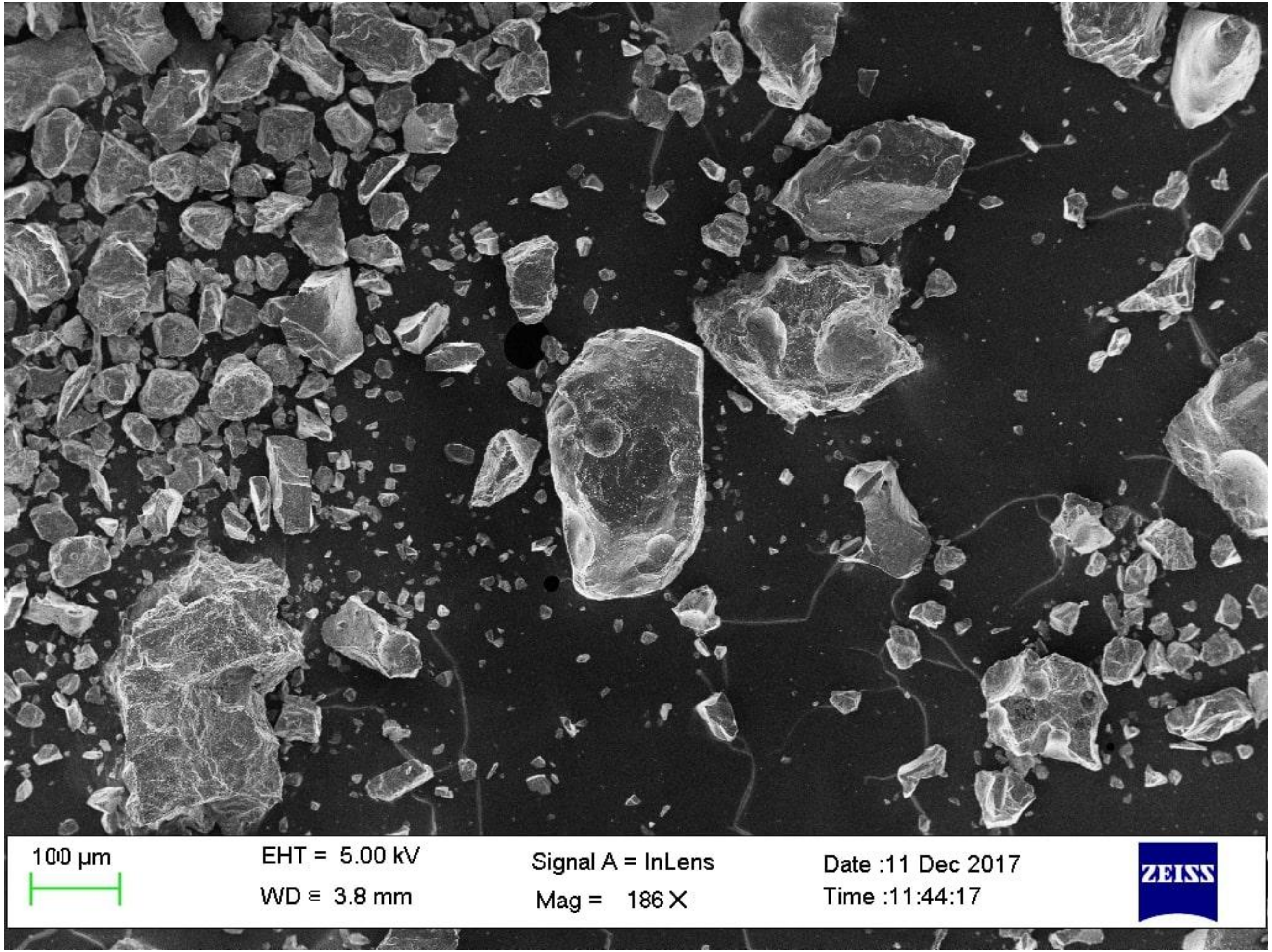
SEM and FESEM



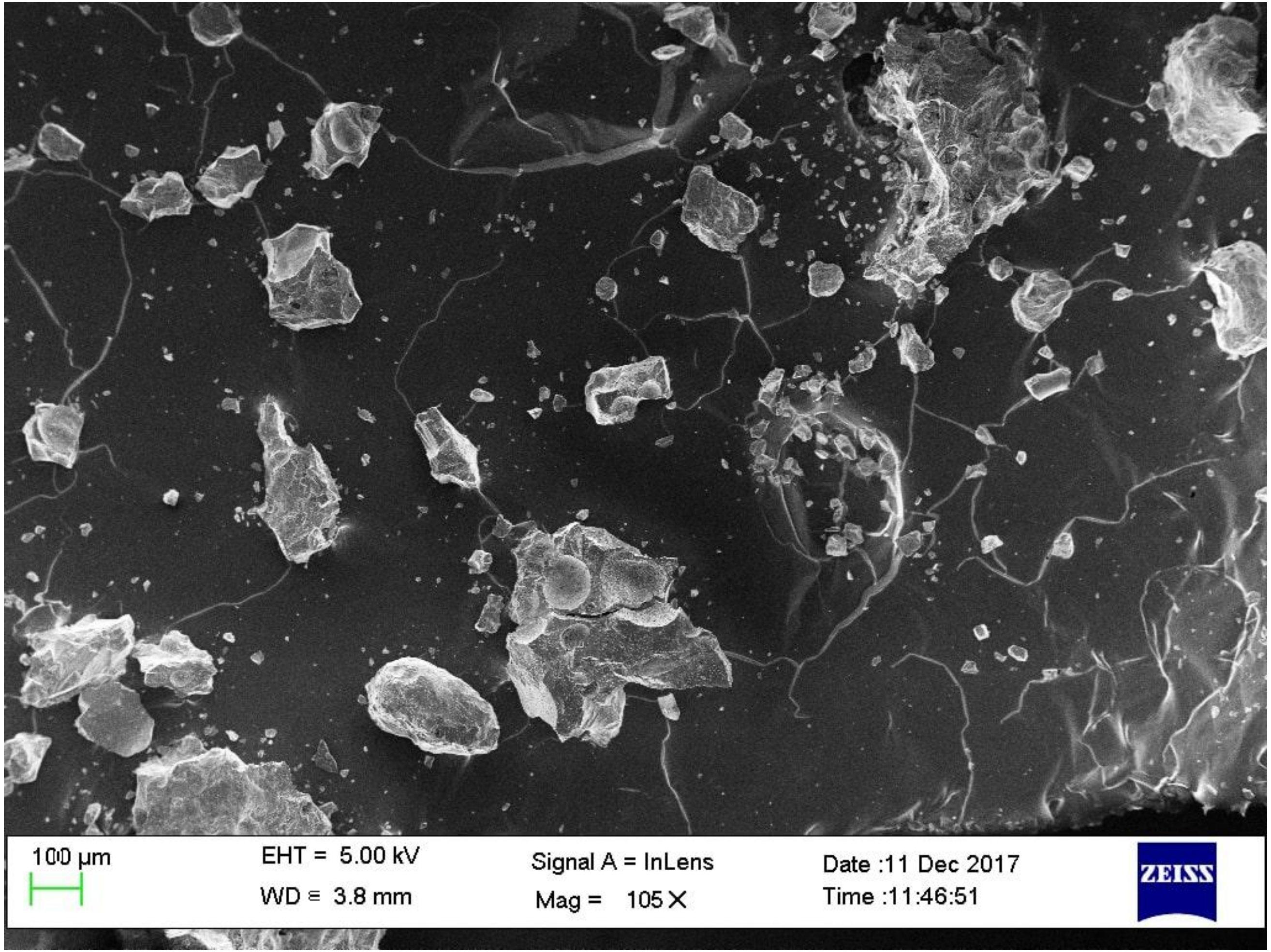
SEM and FESEM



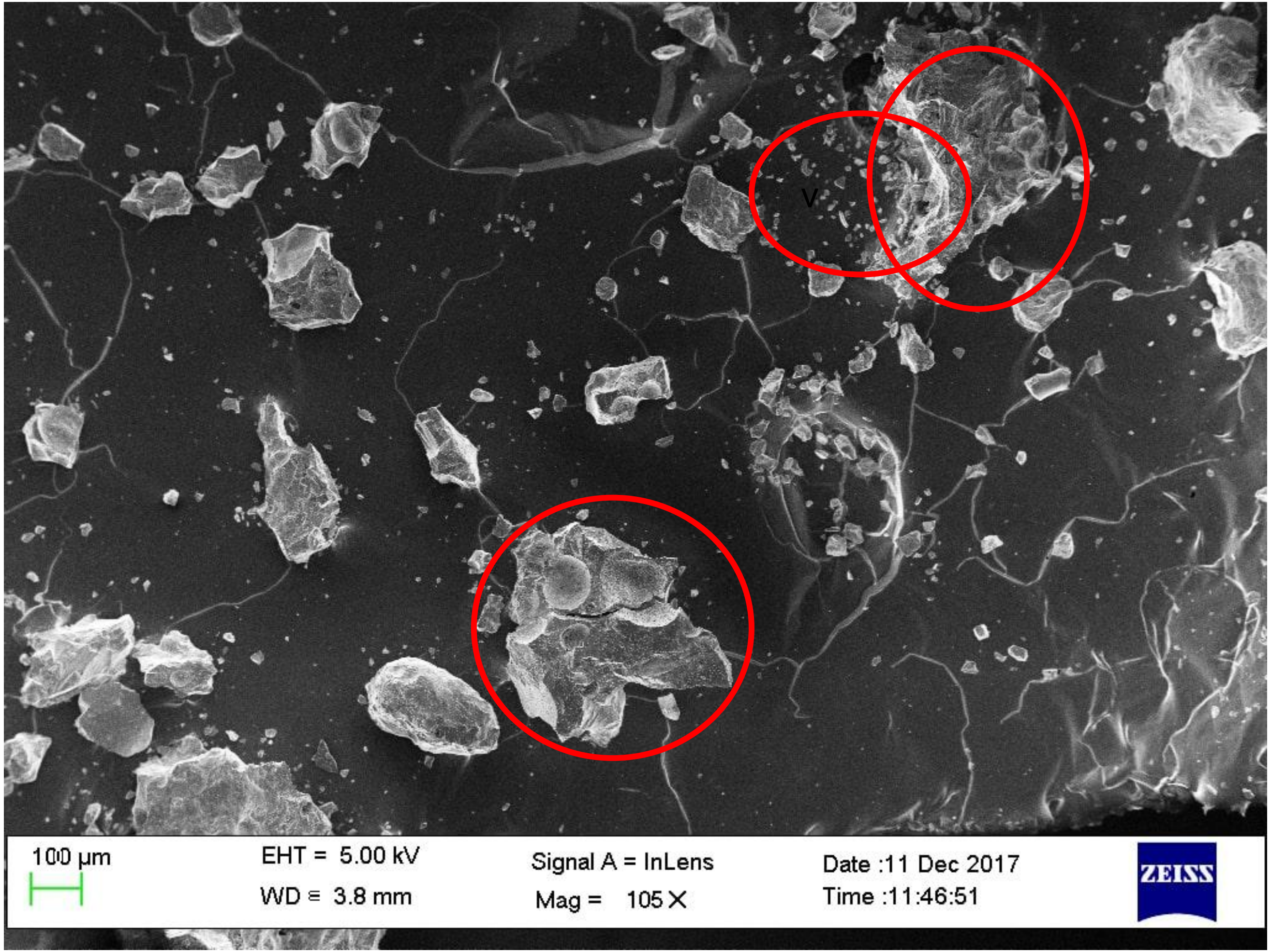
SEM and FESEM



SEM and FESEM



SEM and FESEM



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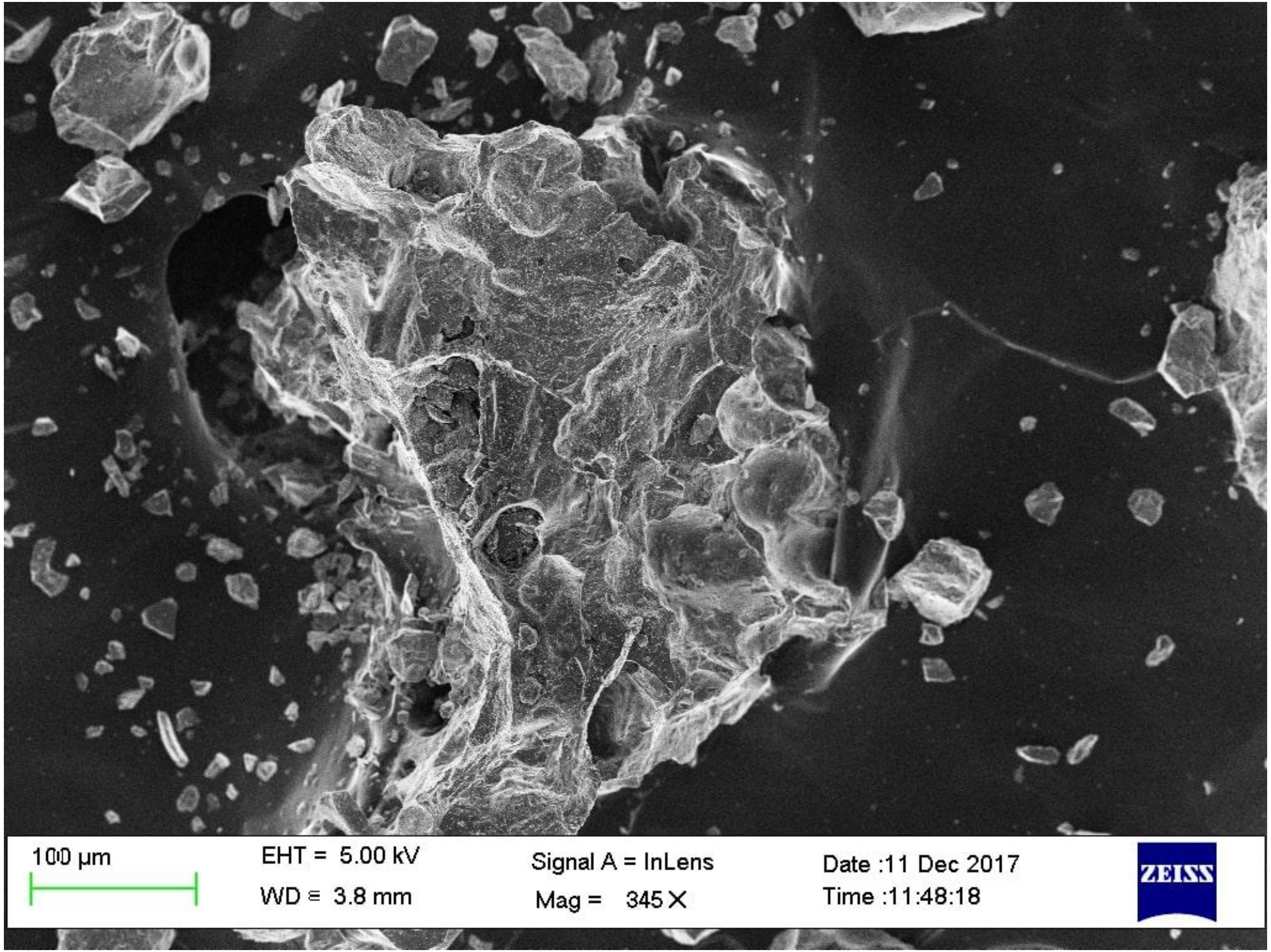
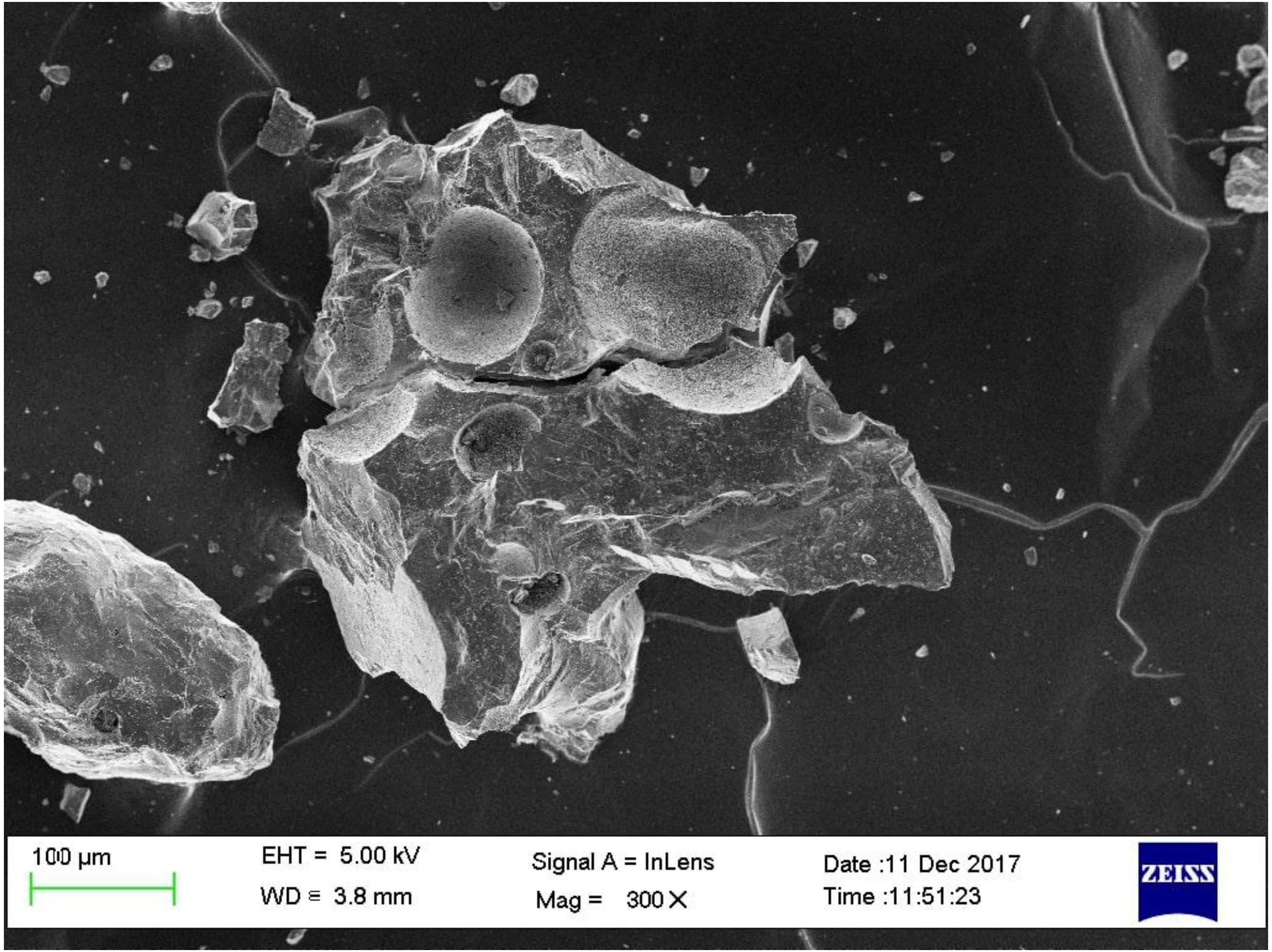
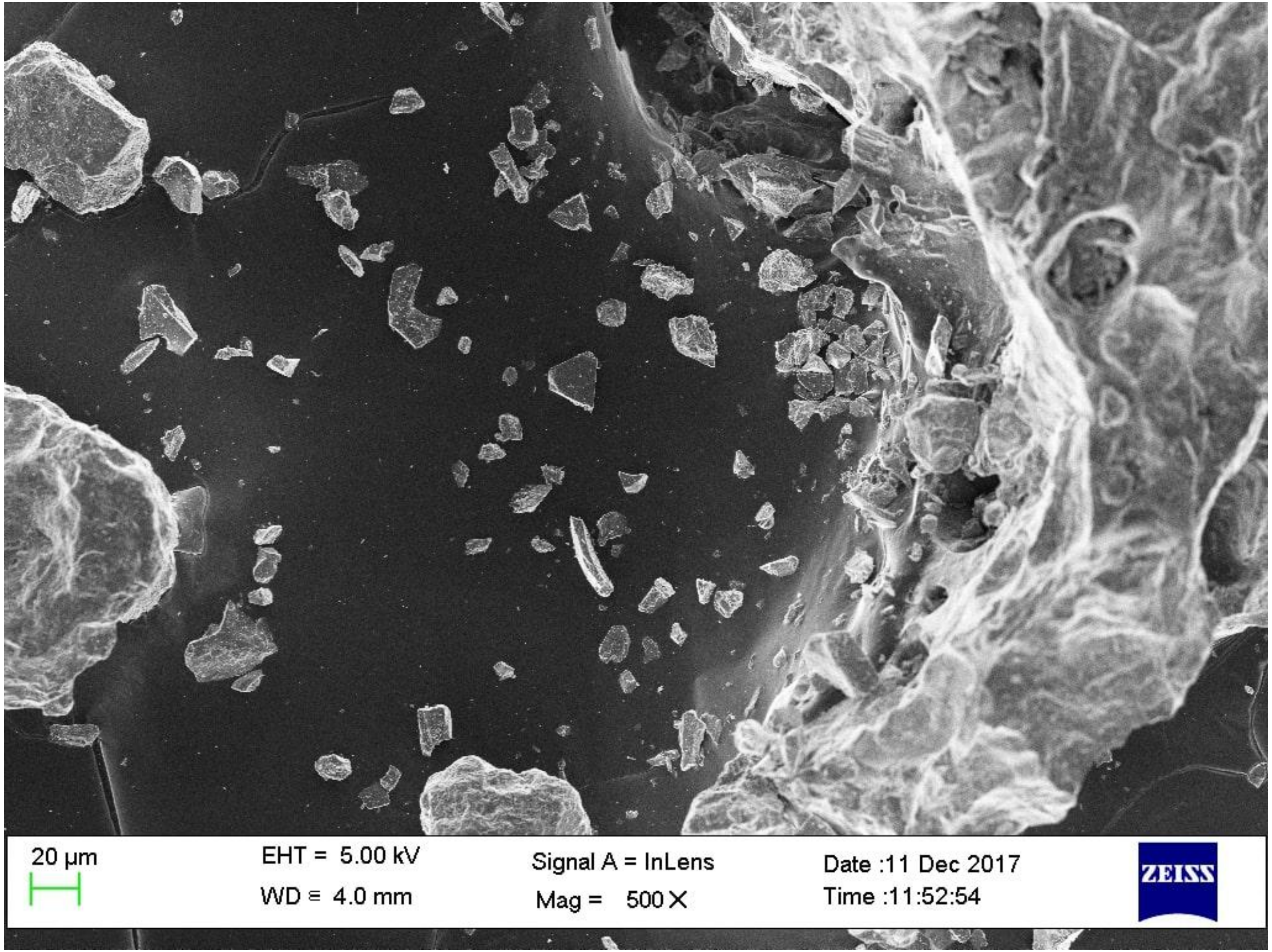


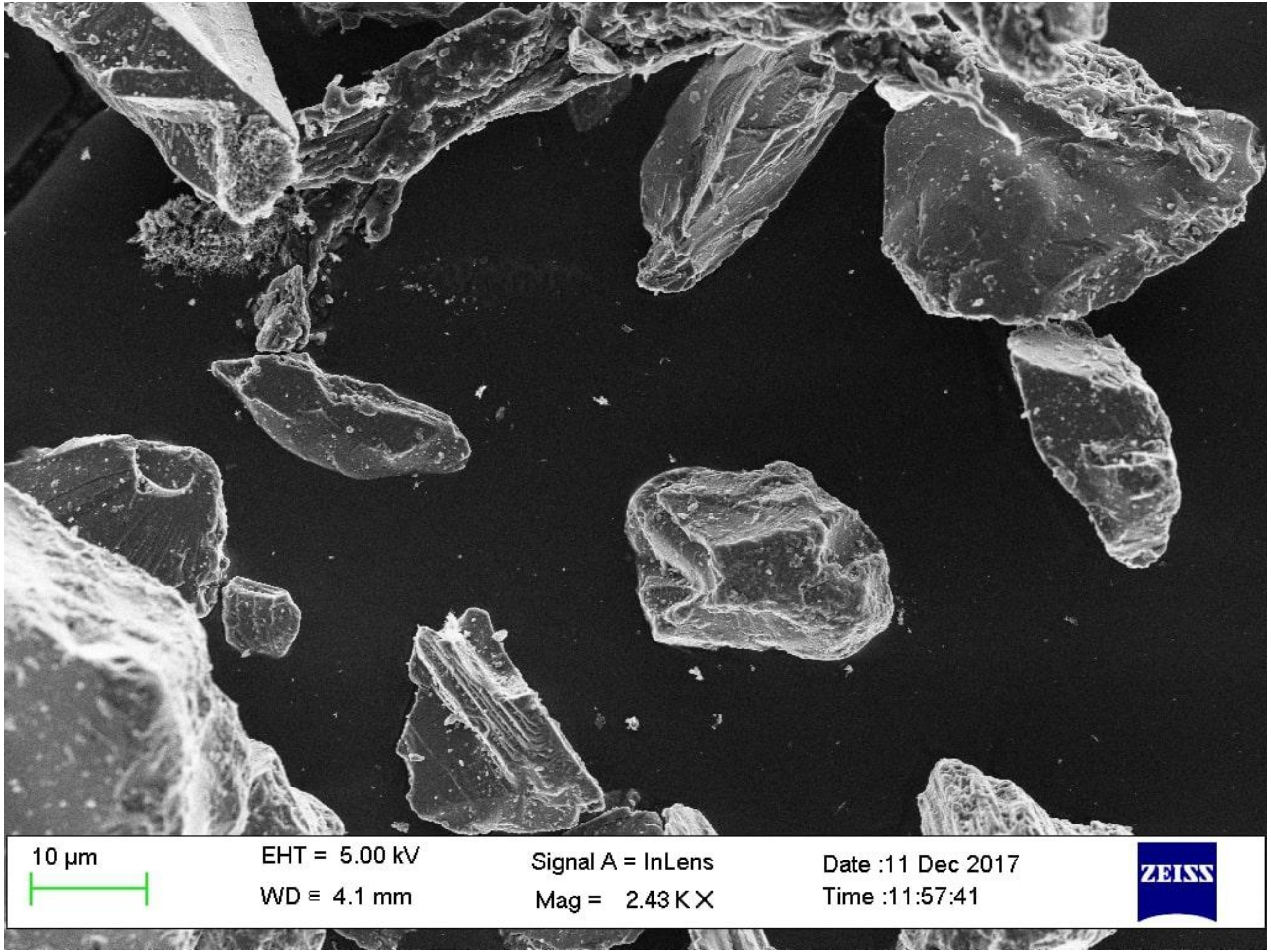
SEM and FESEM



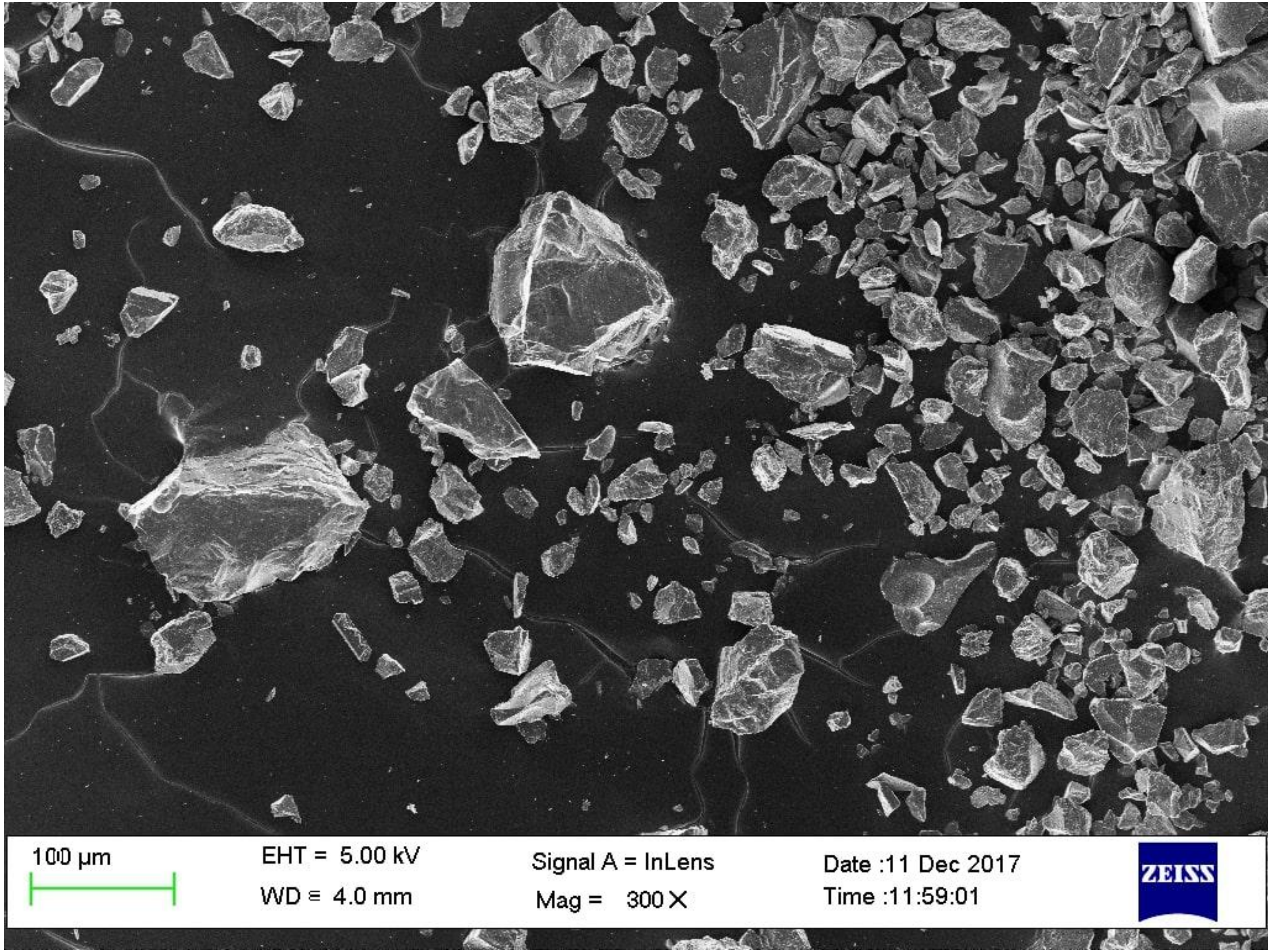
SEM and FESEM



SEM and FESEM



SEM and FESEM

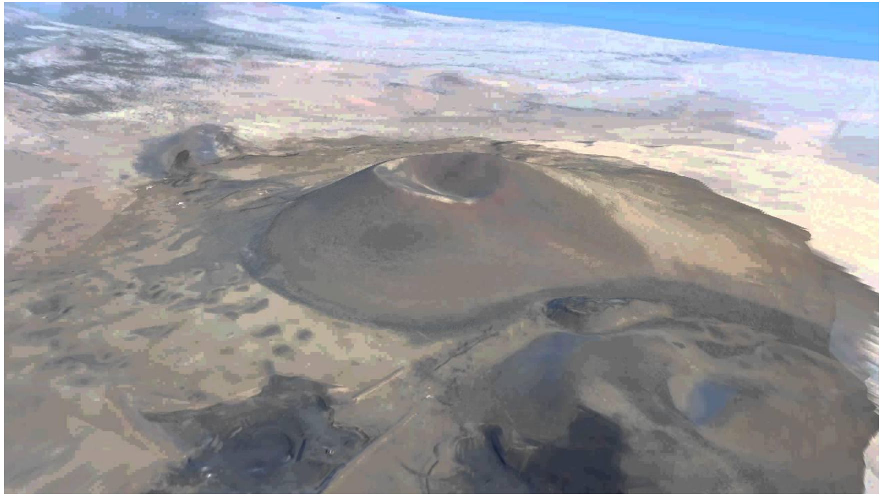


Cosmic dust analogs

Cosmic dust analogs: Geomorphology

Arizona,
USA

Cosmic dust analogs: Geochemistry


Merriam crater, Arizona

Constituent oxides	Apollo 14 sample 14163 (Papike et al. 1982)	JSC-1A (Ray et al. 2010)
SiO_2	47.3 %	45.7 %
Al_2O_3	17.8 %	16.2 %
CaO	11.4 %	10.0 %
FeO	10.5 %	-
Fe_2O_3	-	12.4 %
MgO	9.6 %	8.7 %
TiO_2	1.6 %	1.9 %
Na_2O	0.70 %	3.2 %
K_2O	0.55 %	0.8 %
MnO	0.135 %	0.2 %

Cosmic dust analogs: Geochemistry

Mauna Loa, Hawaii

Cosmic dust analogs: Exobiology

Río Tinto, Spain

High temperature and harsh chemical conditions.

Cosmic dust analogs: Exobiology

Concordia station, Antartica
-40 °C

Cosmic dust analogs: Exobiology

Atacama desert, Chile

Laboratory daily procedures

Laboratory daily procedures

For your own safety

Laboratory daily procedures

For the safety of your laboratory