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Scanning Electron Microscopy for Characterization of Polymers

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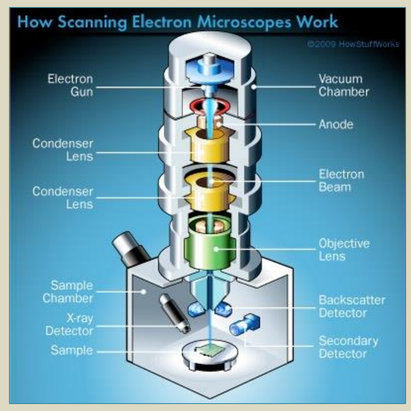
Introduction

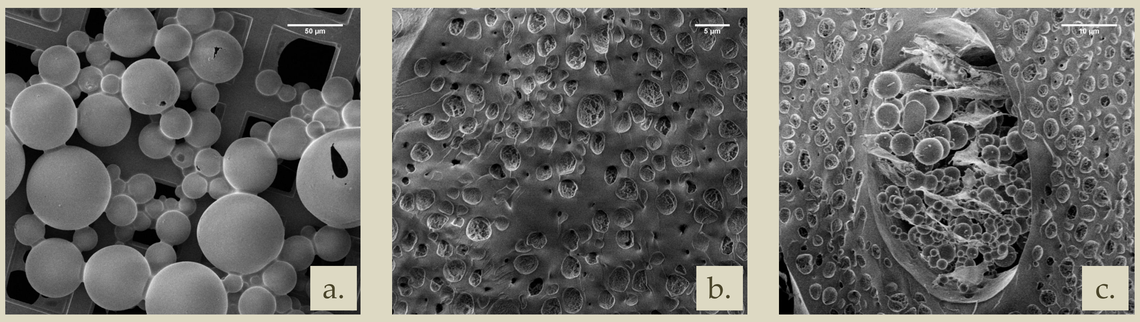
The scanning electron microscope (SEM) is a multifold tool that can be used for advanced characterization of polymers. One can easily get a *picture worth thousand words* with the SEM if the sample is prepared carefully, the right imaging technique and the right signal for detection is pursued. Besides imaging, elemental microanalysis and *in-situ* experiments, such as wetting or drying can be performed in an SEM.
Most polymers present vacuum incompatibility issues, are poor electrical conductors, sensitive to the electron beam and generate little signal, which makes the electron microscopy investigation tedious. We present here a few advanced scanning electron microscopy techniques, which allow for imaging polymers with minimal sample preparation, no chemical modification and no sample coating.



Low voltage SEM (0.5-5 keV)

Room Temperature, High Vacuum

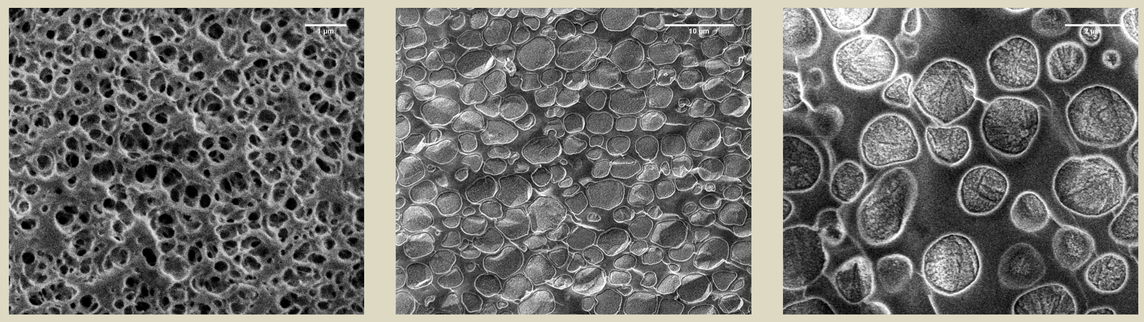
Charge compensation of the sample can be achieved by choosing the right accelerating voltage. At high acceleration voltage the number of electrons (secondary and backscattered) escaping the sample is lower than the number of primary electrons hitting the sample. As the acceleration voltage is decreased the number of escaping secondary electrons increases due to the smaller escape distance. At a certain acceleration voltage an equilibrium between primary electrons hitting the sample and electrons escaping is achieved. This equilibrium accelerating voltage can be found experimentally and used when imaging the sample without coating.



Low voltage SEM micrographs showing the topology of the polymer investigated. The samples are a) PMMA microcapsules dispersed on a Cu grid, b) and c) freeze dried hydrogels (HEMA with various concentrations of PEG DMA).

Cryo (-130 to -140 °C), High Vacuum

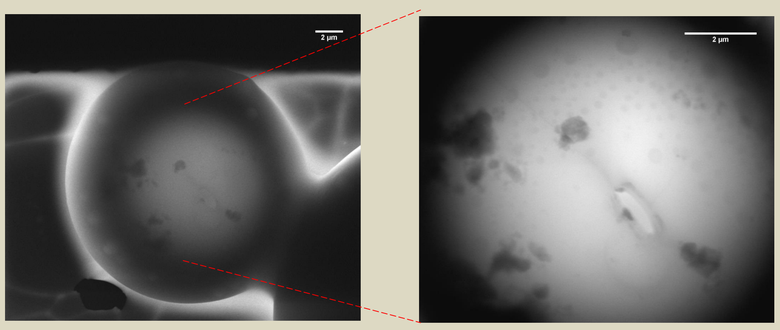
For the cryo SEM the sample is cooled rapidly (10^5 K/s) to liquid nitrogen temperature in order to transform the water content in to amorphous ice. After the cooling step, the sample is kept at temperatures below the glass transition temperature of water. In order to get a fresh surface the sample can be either fractured or sublimated at -90°C and then imaged at (-130 to -140°C) with an incident electron beam accelerated to the equilibrium voltage.



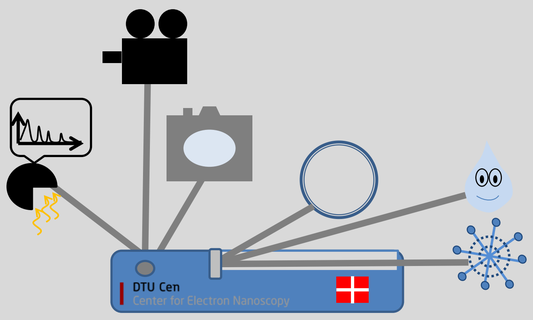
Low voltage cryo SEM micrographs showing the topology of various hydrogels (HEMA with various concentrations of PEG DMA). The samples are plunged freeze in liquid nitrogen and sublimated at -90°C.

Scanning Tranmission Electron Microscopy (STEM) in SEM

When the sample is smaller than the interaction volume most of the scattering will occur in the forward direction. Therefore a STEM detector, which detects the transmitted electrons, can be used.

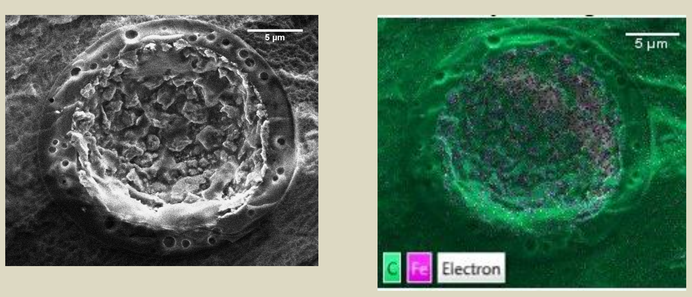


STEM micrographs showing PMMA microcapsules with encapsulated Fe_2O_3 nanoparticles.



Energy Dispersive X-rays (EDS)

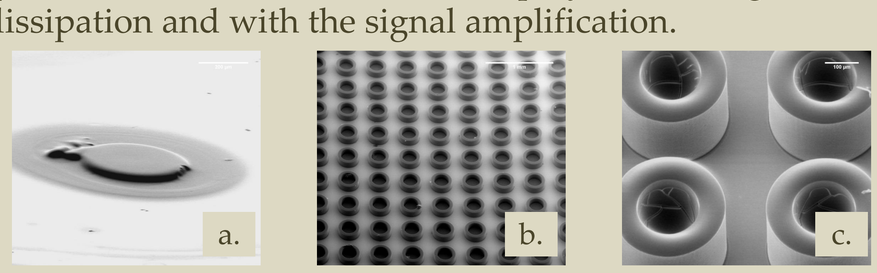
When the incident electron interacts with the sample, if the energy allows, a characteristic X-ray photon can be emitted by the sample. If the characteristic X-rays are detected the elements present in the sample can be identified.



Cryo SEM micrograph showing the cross section of a PMMA microcapsule with encapsulated Fe_2O_3 nanoparticles and the corresponding EDS mapping.

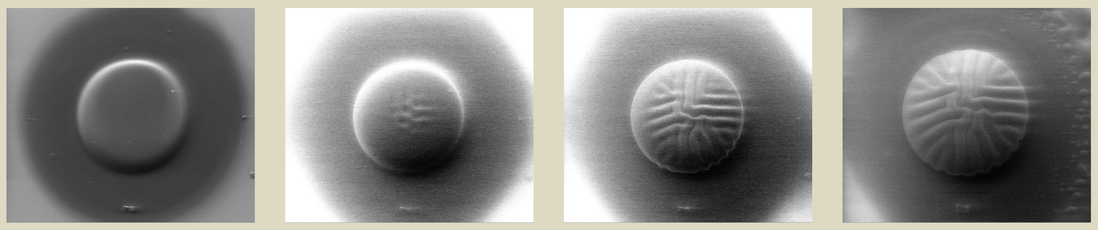
Variable Pressure (VP) SEM (Low and Environmental SEM)

In the VP SEM an auxiliary gas, such as water, is used in order to ad the SEM of poor electrical conductors such as polymers. The gas aids with the charge dissipation and with the signal amplification.



VP SEM micrograph showing the topography of crossed linked hydrogel (a), cross linked SU8 containers (b) and cross linked SU8 containers filled with hydrogel (c).

The VP SEM can be also used for *in-situ* experiments, such as drying, wetting, electron beam deposition and etching.



Increased water vapor pressure

In-situ VP SEM swelling of hydrogel.

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