

## Equipment for a classic SEM enabling environmental techniques

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Classic Scanning Electron Microscopes (SEM) are vacuum instruments, which disqualify insulator specimens or those with high vapour constituents, as for instance biological ones. These limitations can be overcome in Variable Pressure or Environmental SEM (VP/E SEM) in which the sample chamber operates at elevated pressure while the electron optical column is maintained at high vacuum [1]. This implies differential pumping of the two regions which have to be separated by an intermediate chamber with vacuum of order of  $10^{-2}$  mbar, limited by two pinhole throttling apertures (also called pressure limiting apertures – PLA) preventing intensive gas flow from the sample chamber to the high vacuum side. A second peculiarity of the VP/E SEM is an electron detection system which must work at the elevated gas pressure.

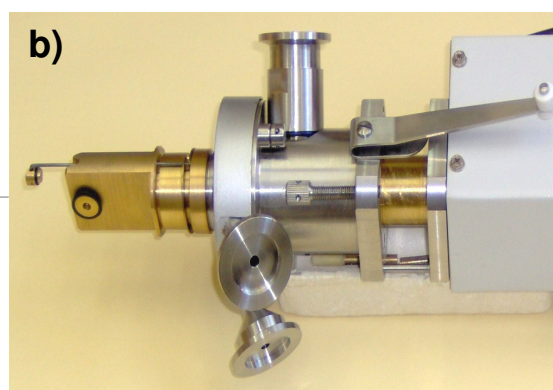
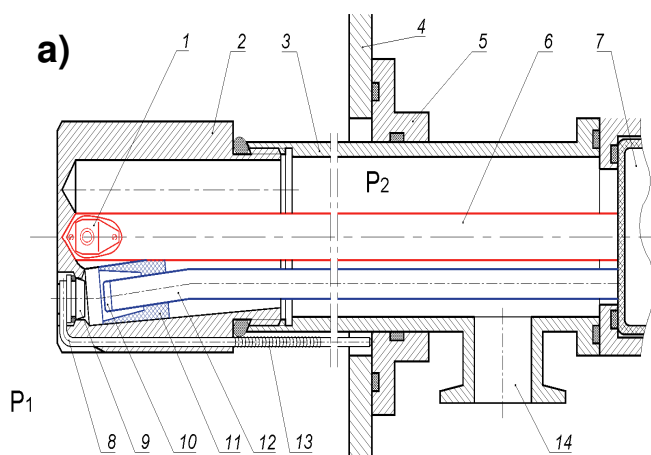
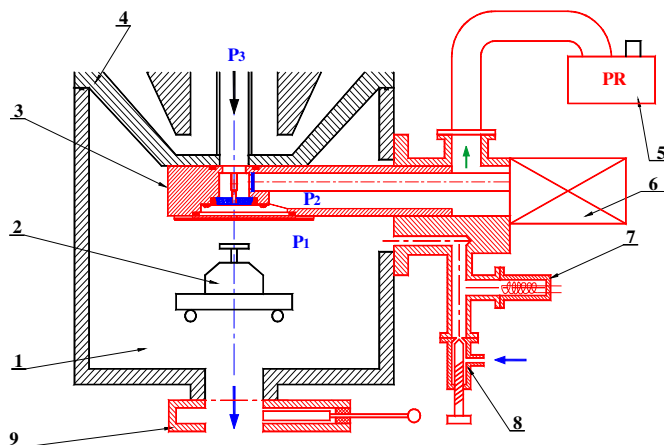
To extend capabilities of the classic instrument toward the VP/E technique, authors supplemented it with the two mentioned units [2], designed in the form of a simple attachment. The system (indicated with red in Fig. 1) can be mounted in a classic SEM, without changes in its original structure. The main part of the system is the vacuum and detection head, combining the intermediate chamber and electron detectors of chosen kinds. For instance, the vacuum and detection head shown in Fig. 2 contains two types of electron detectors. At the end of the main light pipe (6) on the head axis, a low vacuum secondary electron (SE) detector (1) is arranged. The detector extracts secondary electrons through the lower throttling aperture and is designed in the form of a unipotential electrostatic lens with the anode covered with scintillator (described in the adjacent paper). It can work both at pressures exceeding 10 mbar and in high vacuum, so it could be the sole SE detector for the whole range of the SEM applications. Additionally, this detector may also be combined with a backscattered electron detector. However, a body of the intermediate chamber (2) restricts the stage tilt angle and a small opening of the throttling aperture limits the maximum view field, so the head has been made movable to be shifted aside with one move of a lever when vacuum is good. Simultaneously, a pusher (8) opens an inlet valve (9) to an auxiliary SE detector of the Everhard-Thornley type, connected to the same photomultiplier (7) as the low vacuum one. Besides, the head is equipped with a ball-socket joint enabling its angular movements up or down and to both sides.

Numerous experiments conducted by authors proved that a classic SEM equipped with the described attachment is capable to work not only in high vacuum conditions but also fulfils functions of VP/E SEM imaging perfectly almost all possible samples from insulators to liquid water (Fig. 3).

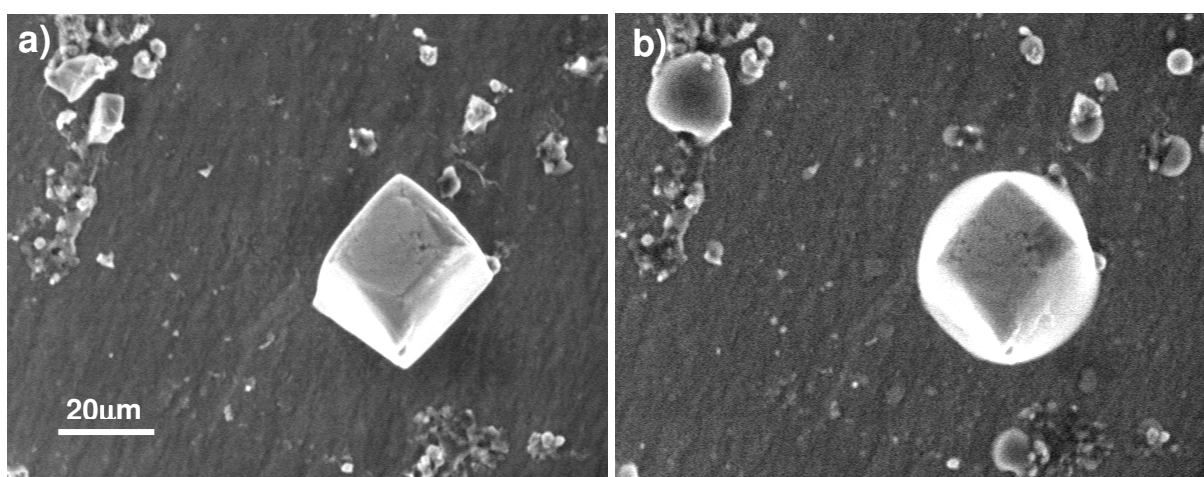
1. G.D. Danilatos, *Advances in Electronics & Electron Physics*, **71** (1988), A.P. London.
2. W. Słwko, *J. Microsc.* **224** (2006) p97.

**Figure 1.** Scheme of the intermediate vacuum and detection system:

1 – sample chamber; 2 – sample holder; 3 – intermediate vacuum and detection head; 4 – objective lens; 5 – rotary pump; 6 – photo-multiplier block; 7 – vacuum meter gauge; 8 – dosing valve; 9 – gate valve.



**Figure 2.** Vacuum & detection head without the vacuum equipment: a) schematic section (1 – low vacuum SE detector; 2 – intermediate chamber; 3 – connecting pipe; 4 – sample chamber wall; 5 – sealing block; 6 – main light pipe; 7 – photo-multiplier; 8 – pusher; 9 – inlet valve; 10 – scintillator; 11 – fixing insulator; 12 – auxiliary light pipe; 13 – spring; 14 – nipple), b) overview with opened inlet valve.



**Figure 3.** Phases of water condensation on a NaCl crystal and dust particles deposited on an Al foil (water,  $\sim 1^\circ\text{C}$ ,  $P_1 = 7$  mbar): a) condensation begins, b) NaCl crystal covered with a droplet of water.