Environmental and In Situ SEM/TEM

IM.3.P065
Multi-detector system for 3D imaging in the variable pressure and environmental conditions

W. Slówko[1], M. Krysztof[1]

[1]Wroclaw University of Technology , Faculty of Microsystem Electronics and Photonics, Wroclaw, Poland

witold.slowko@pwr.wroc.pl
Keywords: Variable Pressure/Environmental SEM, 3D imaging, quadrant ionisation detector

The authors developed a system enabling environmental techniques and three dimensional imaging (3D) even in a classic high vacuum scanning electron microscope (SEM) as it may be combined with a differential pumping unit capable to keep water vapour or another gas at pressure exceeding 10 mbar in the sample chamber. The system schematically shown in Fig. 1, is designed as a detachable equipment which does not implies changes in an original structure of the instrument. The 3D reconstruction of the sample surface is performed with use of the multi-detector method [1] which can be applied for smooth and weakly modulated surfaces in contrast to popular stereoscopic methods. This advantage may be very attractive particularly for biomedical applications.

A version of the 3D imaging method [2] developed by the authors assumes the Lambert's distribution of backscattered electrons (BSE) generated on the specimen surface and sampling this distribution in strictly defined azimuthal (Θ) and vertical angles (α or β) with a four folded BSE detector system. The detector system is combined with the intermediate chamber of a differential vacuum unit necessary to separate the sample chamber filled with gas from the electron optical column where high vacuum is maintained. In Fig. 2 these components are called the detector-vacuum head (3) and the whole 3D equipment contains digital signal acquisition unit (1) and five-channel signal amplifier (2) additionally. Signals from four BSE detectors amplified and converted into a digital form are finally processed by a computer to obtain the 3D surface visualisation.

The combined directional electron detector system [3] which fulfils mentioned demands is shown in Fig. 3. In general, it comprises two quadruple BSE detectors (a PIN diode for high take off angle BSE_1 accompanied by the ionisation type for low take off BSE_2) and a secondary electron (SE) one. The combined detector has an intermediate chamber (16) situated on the path of the electron beam (EB) to the sample stage (2). It is filled with gas with an intermediate pressure (P_2) lower than the working gas pressure (P_1) in the sample chamber where the place of emission of the electrons subject to detection is located. The detector is equipped with a lower aperture (1) throttling the flow of the gas to the intermediate chamber and in the space between the intermediate chamber and the sample stage it has four anodes (3) in the form of four electrodes identical in shape, arranged in pairs symmetrically around the electron beam axis, equipped with separate electrical leads and positively biased relative to the wall of the intermediate chamber. In the space between the anodes and the sample stage there is an external screening cathode (6) with a hole, delimiting from the sample stage side the maximal backscattered electron BSE_2 detection angle (β_2) at which the electrons enter the electric discharge area around the anodes. This arrangement creates an ionisation detector of low take off angle BSE_2 which can be multiplied thanks to secondary emission from the emission layer (17) covering cathode walls and the ionising avalanche in gas filing the sample chamber.

Inside the intermediate chamber there is a quadrant semiconductor BSE_1 detector (13) with four identical sectors active for electrons, arranged symmetrically relative to the electron beam axis, with their active surfaces facing the lower throttling aperture. This part of the combined detector can capture high take off angle BSE_1 coming into the intermediate chamber through the lower throttling aperture. To prevent limiting a maximal vertical BSE_1 detection angle (α_2) by the aperture its distance to the sample must be lower than the aperture diameter (lower than 1 mm). This limitation prevents investigations of semiliquid samples with semiconductor detector because it could suck such sample into the intermediate chamber. The ionisation BSE_2 detector can work at much higher distances (a few mm) and do not create such limitations for the sample nature (Fig. 4)

1. L. Reimer, R. Bongeler, V. Desai, Scanning Microscopy **1(3)**, 1987, p. 963.
2. J. Paluszyński, W. Słówko, J. Microsc. **233/1**, 2009, p. 10.
3. W. Slówko, Patent application nr PL399438, 06.06.2012.
4. This work was supported with the budged resources for science in 2010/13 as a research project.

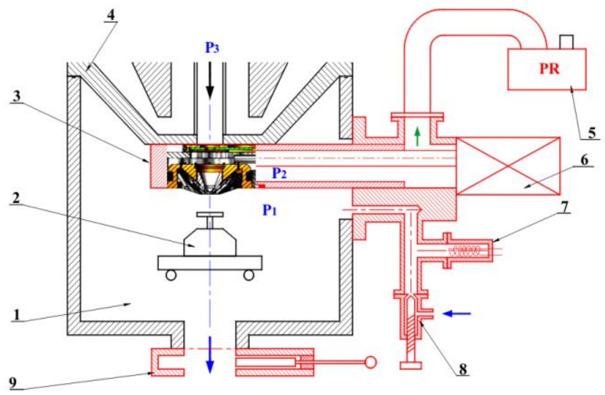

Figure 1. Scheme of the vacuum and detection system for 3D environmental techniques in a standard SEM:
1 - sample chamber; 2 - sample holder; 3 - intermediate vacuum and detection head; 4 - objective lens; 5 - rotary pump; 6 - five-channel amplifier, 7 - vacuum meter gauge; 8 - dosing valve; 9 - gate valve; P_1, P_2, P_3 gas pressures in particular zones.

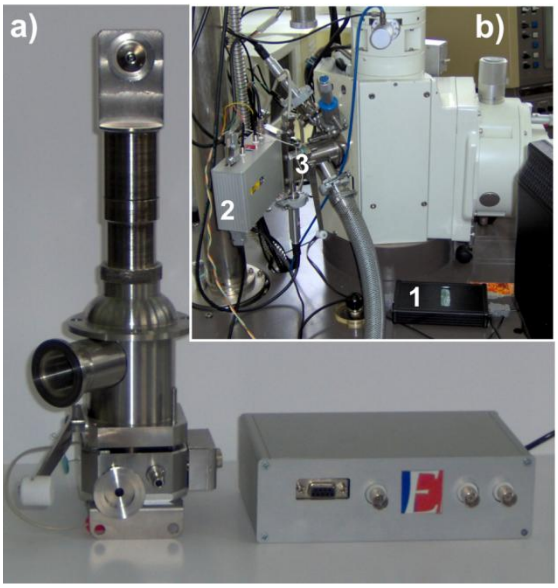

Figure 2. Detector-vacuum system for 3D imaging: a) detector-vacuum head and the five-channel signal amplifier, b) detector-vacuum equipment mounted to the JSM-840 microscope (1 - digital signal acquisition unit, 2 - five-channel signal amplifier, 3 - detector-vacuum head with the vacuum equipment, i.e. two vacuum meter gauges, dosing valve, vacuum line, etc.).

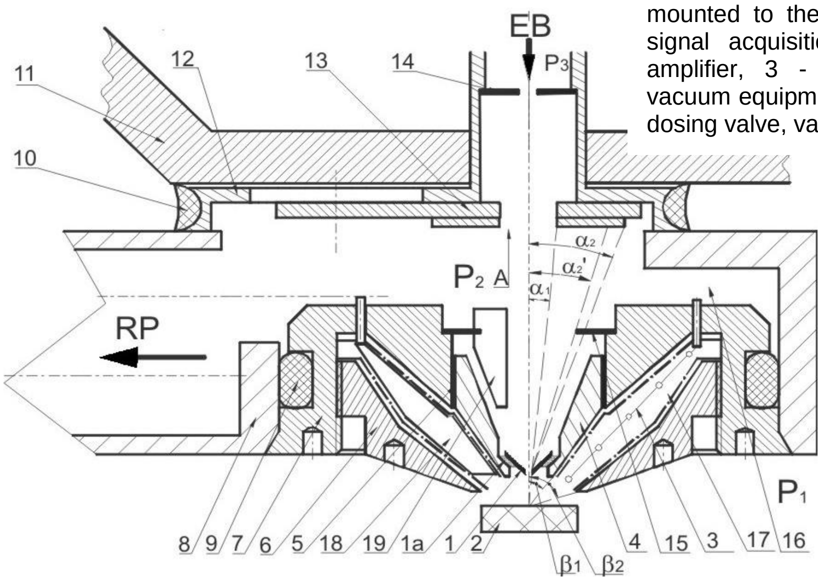

Figure 3. Combined directional electron detector: 1 – lower throttling aperture, 2 – sample stage, 3 – anode, 6 – external screening cathode, 11 – objective lens, 13 – quadrant semiconductor BSE_1 detector, 14 – upper throttling aperture, 16 – intermediate chamber,17 – emission layer, α_1, α_2 – minimal & maximal vertical BSE_1 detection angle, β_1, β_2 - minimal & maximal vertical BSE_2 detection angle, P_1, P_2, P_3 – gas pressures in particular zones, EB – electron beam, RP – vacuum system.

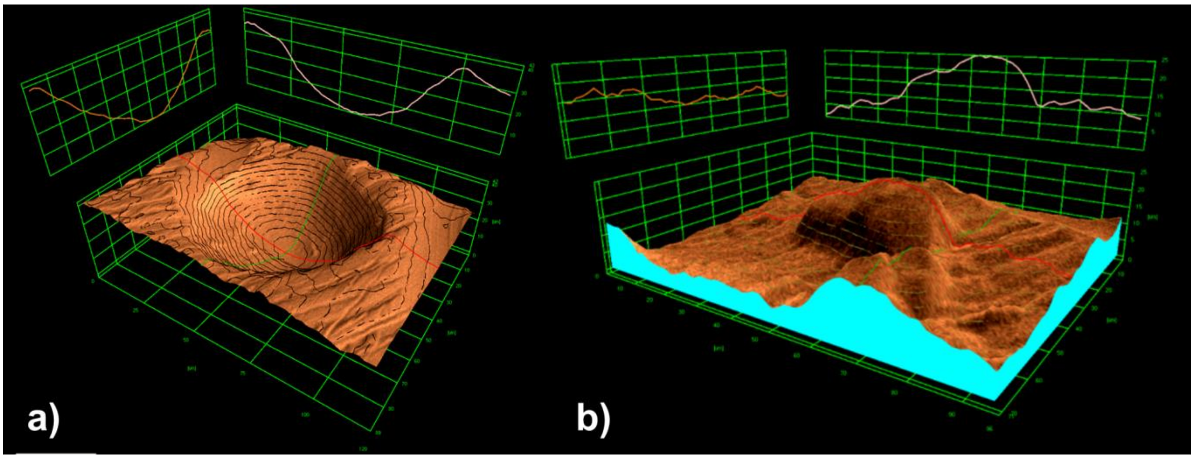

Figure 4. Images obtained from the ionisation BSE_2 detector: a) trace of a needle in polystyrene, b) impurities on the polystyrene surface (texture from a difference of BSE images, air 2 mbar, 20°C, horizontal view-fields: 120 and 95 μm respectively)