Environmental and In Situ SEM/TEM

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Three dimensional imaging with combined electron detector in the Variable Pressure / Environmental SEM

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The three-dimensional imaging system has been developed in the form of an attachment to SEM, which consists of a combined directional electron detector, a frame-grabber and a PC-based processing unit. The detector head is integrated with the intermediate vacuum system to separate the sample chamber allowing gas pressure over 10 mbar and the electron optical column where high vacuum must be maintained. Quantitative information about the surface topography is obtained by digital processing of four input images acquired from four electron detectors [1]. This approach is often called a multi-detector method [2]. The multi-detector system developed by authors (Fig. 1) comprises two quadruple (4Q) backscattered electron (BSE) detectors (a PIN diode for high take off angle BSE_1 and the ionisation type for low take off BSE_2) and a secondary electron (SE) one. The multi-detector method is based on Lambert's angular distribution of backscattered (BSE) or secondary electrons (SE). A system of four detectors, placed symmetrically around the specimen (Fig. 2), allows to extract information about local topography and composition. The signal of a particular detector can be written as:

(1)

where: θ_p, φ_p - local surface topography in polar coordinates, δ_0 - secondary emission coefficient (depends on the material composition), d, c, θ_A - detector geometry coefficients, I_{PE} - electron beam current.

The local surface inclination may be obtained by dividing the difference by sum of the opposite detector signals. This provides a specimen topography profile if integrated along a scan line in the *x* direction. The second pair of detectors gives the initial profile in the *y* direction:

$$z(x, y_i) = a_x \int_{x_0}^{x_k} \left[\frac{I_A - I_B}{I_A + I_B} \right]_{y=y_i} dx + a_y \int_{y_{i=1}}^{y_i} \left[\frac{I_C - I_D}{I_C + I_D} \right]_{x=x_0} dy \qquad (2)$$

The 3D reconstruction procedure is as following. Four quadrants (4Q) of the chosen BSE detector produce four signals stored in the form of four input images as in Fig. 3b. They are processed to obtain a 3D pattern of the surface shape as a bit-map which can be visualized in any desired form. For instance, it may be a perspective view (Fig. 4a) with surface profiles shown on side grids as in Fig. 4b. This shape may be deprived of subtle details (mainly by the integration process occurring in the algorithm) usually present on every surface as a so called texture. The source of a surface texture overlaying the shape may be a combination of the input BSE images or advantageously an additional SE image shown in Fig. 3a. An impressive form of the surface shape presentation may be an anaglyph. Features of the semiconductor and ionisation 4Q BSE detectors are different. The latter can work at higher pressures and working distances of a few mm. Besides it gives a strong topographic contrast. As the result this detector can give a quantitative 3D image of the subtle meniscus of the semiliquid agar gel in Fig. 4b. Such features are particularly desired for biomedical applications. The semiconductor one captures electrons through the small throttling aperture so the sample distance must be much smaller than its diameter to prevent the detector from shadowing. This feature and its low topographic contrast (but high compositional one) make the detector destined for investigations of solid dielectric samples with strongly modulated surfaces as that in Fig. 3c. On the other hand, this detector can also work in high vacuum so the detector head is retractable to let it operate without the aperture limitations. The system has been mounted to a JSM840 microscope and additionally equipped with an environmental sample holder [3] which enables transfer of wet biological samples through the sample transfer chamber and facilitates keeping them in a natural state during investigations in the sample chamber.

1. J. Paluszyński, W. Słówko, J. Microsc. **233/1**, 2009, p. 10.
2. L. Reimer, R. Bongeler, V. Desai, Scanning Microscopy **1(3)**, 1987, p. 963.
3. W. Slówko, Patent application nr PL403014, 06.03.2013.
4. This work was supported with the budged resources for science in 2010/13 as a research project.

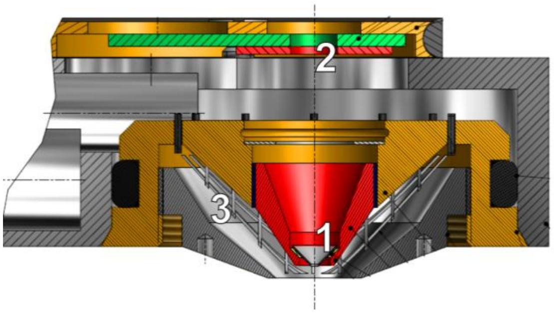


Figure 1. Cross-section of the combined electron detector:
1 - throttling aperture as the SE detector;
2 - semiconductor 4Q BSE detector:

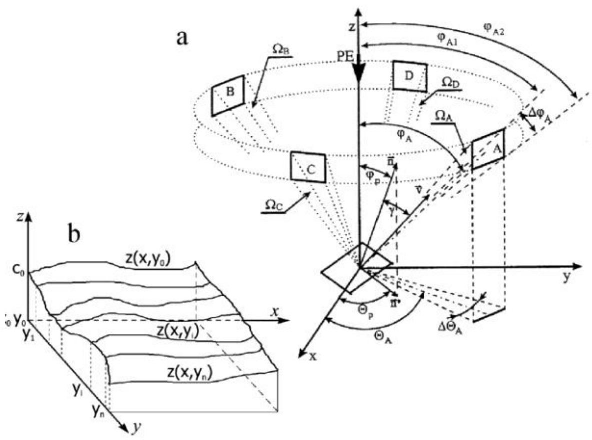
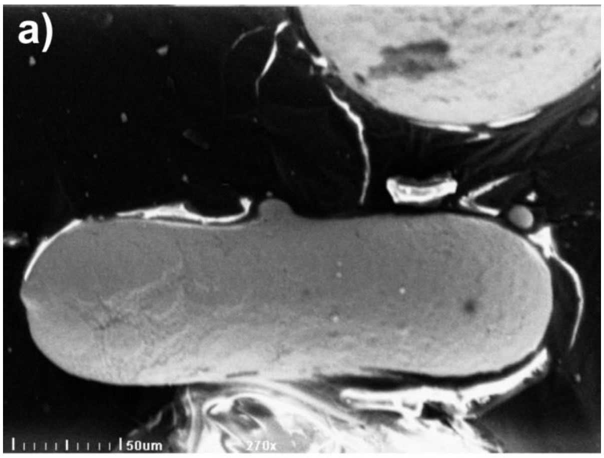
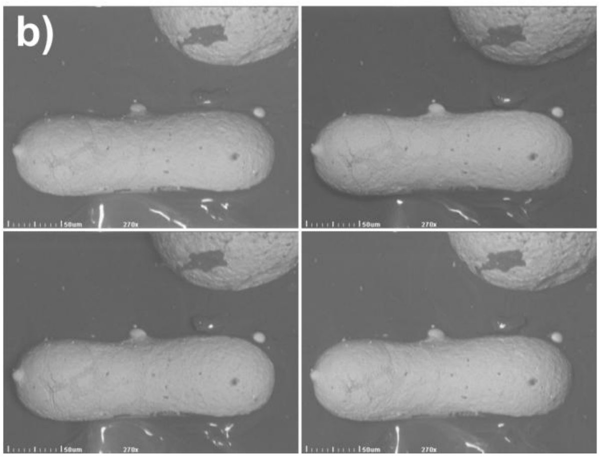


Figure 2. Illustrations to the 3D reconstruction method: a) four detector system in polar coordinates, b) surface shape reconstruction according to Eq. (2).





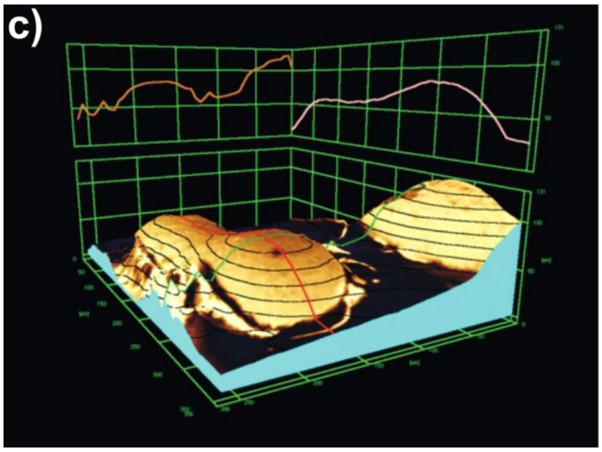
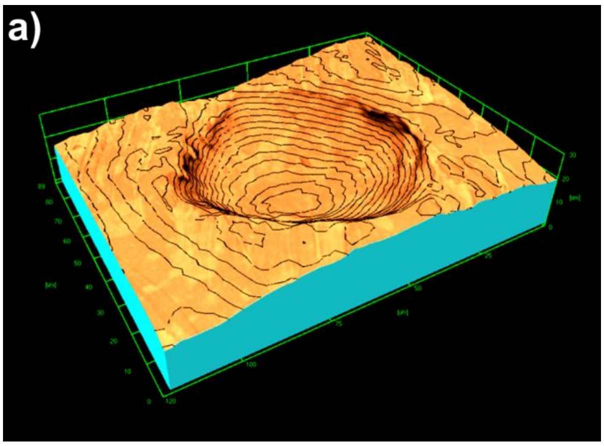


Figure 3.
Ceramic seeds on adhesive (air 2mbar, 20°C):
a) SE image used for the surface texture,
b) Four input BSE images taken from the semiconductor 4Q PIN detector for the 3D reconstruction,
c) axonometric view with level lines and surface texture taken from the SE image in a halftone color



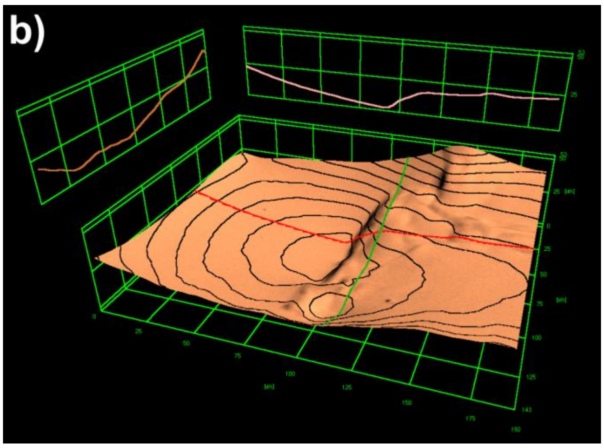


Figure 4. Images taken from the ionisation 4Q BSE detector: a) trace of needle in polystyrene (air 2 mbar, 20°C, a SE image as the texture), b) surface of agar gel with the wave caused by a submerged paper fiber (water 6 mbar, 1°C, texture from difference of BSE images).