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High-efficiency detector of secondary and backscattered electrons for low-dose imaging in the ESEM



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ABSTRACT

A new Combined System for high-efficiency detection of Secondary and Backscattered Electrons (CSSBE) in the ESEM consists of three detectors: an ionisation SE detector, an improved scintillation BSE detector, and a new Ionisation Secondary Electron Detector with an electrostatic Separator (ISEDS). The ISEDS optimizes conditions for electron-gas ionisation phenomena in the ESEM to achieve a strongly amplified signal from the secondary electrons with a minimal contribution from backscattered and beam electrons. For this purpose, it is originally equipped with an electrostatic separator, which focuses signal electrons towards a detection electrode and controls the concentration of positive ions above the sample. The working principle of the ISEDS is explained by simulations of signal electrons in a selected range of energies is described with Geant4 Monte Carlo simulations of electron-solid interactions and proven by experimental results. High-efficiency detection of the ISEDS is demonstrated by imaging a low atomic number sample under a reduced beam energy of 5 keV, very low beam currents of up to 0.2 pA, and gas pressure of hundreds of Pa.

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1. Introduction

The ability to perform research on most natural and synthetic materials in their native or minimally treated state, in different phases of matter, or during phase transitions emphasises the indispensability of an Environmental Scanning Electron Microscope (ESEM) in science and industry. The ESEM [1] allows direct observation of natural, electrically non-conductive and semi-conductive samples free of charging artefacts [2,3], fully hydrated or wet samples [4,5], plants [6,7], small live animals [8], or microgel particles

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http://dx.doi.org/10.1016/j.ultramic.2017.08.003 0304-3991/© 2017 Elsevier B.V. All rights reserved. [9] in conditions of thermodynamic equilibrium [10], as well as *in-situ* investigation of chemical reactions and samples under dynamically changing conditions (mostly in water vapour pressures of ones to thousands of Pa, sample temperature commonly from $-20 \,^{\circ}$ C to 1500 $^{\circ}$ C, and relative humidity up to 100%) [11,12].

Inevitable beam diffusion in gas causes unscattered beam electron fraction to decrease as well as probe current in a focused spot. It can be mitigated by lowering the distance in the gaseous environment which beam electrons (BE) pass through, increasing the BE energy, probe current and dwell time, or choosing a suitable gas type and its pressure [13,14]. Nevertheless, a useful signal from a beam spot region is supplemented with an unwanted background signal generated by inelastic beam-gas interactions and elastically scattered BE, generally governed by the Poisson distribution probability in the skirt region [15]. It can cause worsening of signal-to-noise ratio (SNR), image resolution, as well as the X-ray microanalysis spatial resolution in the ESEM. Investigation of samples with a low atomic number (low Z samples) or wet samples in their native state susceptible to radiation damage increases demand on new high-efficiency and energy-sensitive detection systems. Given the above, research and development of new detection systems for the characterisation of samples in a wide range of working conditions is one of the most important topics for future progress in the ESEM.



Abbreviations: BE, beam electrons; BSE, backscattered electrons; CSSBE, a Combined System for high-efficiency detection of Secondary and Backscattered Electrons; ESD, Environmental Secondary Detector; ESEM, Environmental Scanning Electron Microscope; GSD, Gaseous Scintillation Detector; GSED, Gaseous Secondary Electron Detector; ISEDS, Ionisation Secondary Electron Detector with an electrostatic Separator; ISI of the CAS, Institute of Scientific Instruments of the Czech Academy of Sciences; ITO, indium tin oxide; LVSTD, Low Vacuum Secondary Tescan Detector; PLA, pressure limiting aperture; PLA2, second pressure limiting aperture; PV, sample to PLA2 distance; PV_d, sample to detection electrode distance; PV_p, sample to separation electrode voltage; U_{DEF}, deflection electrode voltage; U_{DR}, drain electrode voltage; Us, separation electrode voltage; VPSE, Variable Pressure Secondary Electron detector; YAG:Ce³⁺, yttrium aluminium garnet doped by Ce; YAP:Ce³⁺, yttrium aluminium perovskite doped by Ce.

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So far, the most efficient detectors of secondary electrons (SE) in the ESEM use a principle of gas ionisation that proceeds as a cascade or avalanche between a grounded sample holder and a detection electrode supplied with a positive voltage, placed under a pole piece of an objective lens [16–18]. The signal electrons are accelerated by an electrostatic field. They then ionize gas molecules, generate ions and new electrons and thus amplify the detected signal [19]. The amplification depends namely on three factors: the intensity of the electrostatic field between the detection electrode and the grounded sample, the path length of the signal electrons through the gas, and the pressure and type of ionisation gas [20,21]. This principle was used in the Environmental Secondary Detector (ESD) [22]. Unfortunately, the ESD is unable to detect SE and backscattered electrons (BSE) separately [23]. For this reason, the ESD was developed into the Gaseous Secondary Electron Detector (GSED) [24]. The GSED provides a cleaner SE signal, but it still detects some BSE signal [23]. A multi-electrode configuration of the gaseous detector device was proposed by Danilatos to separate various signals emitted from the sample surface [25]. The physical principle of the multi-electrode configuration was implemented in the technical design by Armstrong et al. [24], and it is used commercially in the ESEMs of FEI Inc. Nevertheless, the BSE are not yet separated completely from the SE image. The contribution of BE, SE, and BSE to the total amplification of the signal by the ionisation of the gas was simulated by Meredith et al. [26]. Toth et al. [27] introduced a system for the detection of SE at gas pressures exceeding 1 kPa. This system uses a stainless steel needle-shaped anode with a radius of curvature of approx. 50 µm, which allows an optimal gradient of the electrostatic field for electron impact ionisation of gas molecules and detection of the amplified signal to be made free of dielectric breakdown. A similar detection system based on a biased nano-wire situated in close proximity to the sample and used as a detection electrode was introduced by LEO (Zeiss Company) [28]. An SE detector working on the same principle as the two detectors mentioned above, but with the detection electrode situated on the sample stage at a fixed distance from the sample, was designed by Fitzek et al. [29] The best of the commercially available detection systems of SE in the ESEM is the Helix (FEI Inc). This system uses the magnetic field of an ultra-high resolution magnetic immersion objective lens and the electrostatic field of an annular detection electrode placed at the bottom part of the objective [30]. The Helix detector allows a high resolution image (1.8 nm at the energy of BE 3 keV) at a short working distance (up to 3 mm) and low gas pressure (up to 200 Pa) to be recorded. A recent study in the field of generation and detection of a signals in high pressure conditions implies the significant role of positive ions [31]. For this reason, the Helix detector is equipped with an "ion trap electrode", which allows a number of ions in the vicinity of the sample and detection electrode to be controlled. The utilization of the magnetic field to the signal selection and amplification of the SE signal are very promising for the future detection of the signal electrons in the ESEM.

The Two-Stage SE Detector and Intermediate SE Detector were introduced by Slówko [32]. These detectors contain a scintillation detector of the Everhart–Thornley type combined with a microsphere plate [33]. The same author also published a Combined Directional Detector [34] for the three-dimensional imaging of electrically non-conductive and semi-liquid samples in the ESEM, newly equipped with a coaxial ion micro-source [35]. This detector comprises two quadruple (4Q) semiconductor BSE detectors for the detection of high take off angle BSE1 and an ionisation type of detector for the detection of BSE2 and SE.

The Low Vacuum Secondary Tescan Detector (LVSTD) was developed by Jacka et al. [36]. This detector is based on the modified design of the Everhart-Thornley detector with a vacuum separated and differentially pumped detection chamber located behind a Microlens Differential Barrier and pumped by a small turbomolecular pump. A similar working principle is used in the scintillation SE detector developed by Jirák et al. [37] from the Institute of Scientific Instruments of the Czech Academy of Sciences (ISI of the CAS). In this detector, the biased scintillator (8–12 keV) is located in the differentially pumped chamber, separated from the specimen chamber by a system of two pressure limiting apertures (PLA). These apertures, with applied voltage, create an electrostatic lens which focuses the signal electrons through to the scintillator.

The study of specimens under the high pressure conditions in the ESEM need not be necessarily associated with "direct" signal electron detection. As was pointed out by Danilatos [38], photons can also be detected. Luminescence in gas ("gaseous scintillation"), a consequence of inelastic excitation collisions of the signal electrons with the gas molecules and their subsequent deexcitation (a process lasting approx. 10^{-8} s), is accompanied by the emission of light quanta in the range from ultraviolet to infrared radiation, as shown by Takahashi et al. [39] and Fraga et al. [40]. This principle is applied in the Variable Pressure Secondary Electron Detector (VPSE detector), patented by the Zeiss company [41], and the Gaseous Scintillation Detector (GSD), presented and described by Morgan et al. [42], which collect and transfer the detected photons via a light-guide to a photomultiplier. A comparison of the GSED and GSD in the operating conditions of the ESEM was published by Morgan et al. [42].

Autrata et al. introduced a very sensitive BSE detector using the original YAG: Ce^{3+} (yttrium aluminium garnet, $Y_3Al_5O_{12}$ doped with Ce^{3+}) [43] and YAP: Ce^{3+} (yttrium aluminium perovskite, YAlO₃ doped with Ce^{3+}) [44] scintillators for the study of the material contrast of samples. This allows the observation of wet biological samples at lower energies of BE [45,46].

Due to a demand for the simultaneous detection of SE and BSE, the YAG BSE detector was developed into the combined detector. BSE are detected by the scintillator and SE by the ionisation detector [47]. A hole in the scintillator of the combined detector creates the PLA for differential pumping of the ESEM [48]. The ionisation SE detector of the combined detector is created by a thin electrode from indium tin oxide (ITO), deposited on the bottom side of the scintillator. Many types of materials and shapes of detection electrodes were tested for the separation of BSE and SE signals [49], for the recording of spatial distribution of signal electrons [50], and for the study of biological samples [51,52] in our non-commercial ESEM AQUASEM II. Another version of the combined detector allows the signals from lateral sides of the scintillator to be detected separately using a halved scintillation single crystal. A comparison of YAG: Ce³⁺, YAP: Ce³⁺ and plastic scintillator NE102A [53], as well as a design of an optimum shape of light-guides [54], were published by Danilatos. These detectors are mostly situated below the ESEM objective lens. Some special "in lens" versions were also designed and situated inside the differentially pumped chamber for detection of SE going through the PLA2 up to the ESEM objective [55,56].

This paper introduces a new Combined System for highefficiency detection of Secondary and Backscattered Electrons (CSSBE) in the ESEM. The description of the detection system is focused on a new Ionisation Secondary Electron Detector with an electrostatic Separator (ISEDS); however, the other parts of the CSSBE (the combined ionisation detector and the improved scintillation BSE detector) will be briefly mentioned.

2. Materials and methods

2.1. The CSSBE detector

The CSSBE for simultaneous high-efficiency detection of SE and BSE in the ESEM contains three mutually integrated detectors (see Download English Version:

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