Nuclear Instruments and Methods in Physics Research A 645 (2011) 79–83

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research A

journal homepage: www.elsevier.com/locate/nima

Comparison of calculated, simulated and measured signal amplification in a variable pressure SEM

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ARTICLE INFO

Available online 29 December 2010

Keywords:
Electron–gas interactions
Monte Carlo simulations
Signal amplification
Analytical models

ABSTRACT

This paper presents computed dependencies of signal amplification of detected electrons on the water vapor pressure in a variable pressure SEM using the EOD software equipped with a Monte Carlo plug-in. We analyze the amplification at selected energies of signal electrons going through a high-pressure water vapor and total signal amplification by including a realistic simulation of the secondary emission from sample. The results are compared with experimental measurements and the dependencies of published analytical models.

1. Introduction

Relatively high pressure (up to 3000 Pa) of gases (mostly water vapor) in the specimen chamber of variable pressure scanning electron microscopes (VP-SEM), environmental scanning electron microscopes (ESEM), etc., enables examination of non-conductive, partially or fully wet samples as well as the study of dynamically changing physical or chemical conditions [1]. Besides these advantages scattering of primary electrons (PE) in a gaseous environment [2] causes worsening of the signal to noise ratio and it negatively influences the image resolution. This problem can be solved by decreasing the length in which the PE passes through the gas environment, increasing the PE accelerating voltage and probe current, using lower scanning speed and by choosing a suitable gas type and pressure [3,4]. Nevertheless, decrease in the probe current in the focused spot due to the primary electron–gas interactions lowers the number of the unscattered beam electrons and decreases the useful signal from the specimen. In such cases there are higher demands on the efficiency of the detection system.

So far the most efficient detectors for the detection of the secondary electrons (SE) in environmental conditions use the principle of gas ionization that proceeds as a cascade between a grounded specimen holder and the signal electrode supplied with a positive voltage [5]. Signal electrons are accelerated by the electrostatic field and ionize gas molecules and generate positive ions; the secondary electrons strongly contribute to the amplification of signal detected by the detector. The amplification of the detected signal depends on the intensity of the electrostatic field between the detection electrode and the grounded sample, on the gas path length of the signal electrons through the gas as well as on the pressure and type of the ionization gas [6].

High-pressure gases in the specimen chamber of electron microscopes create completely different and more complicated conditions for the detection of SEs than in a conventional SEM. Signal amplification arises due to ionization of gas molecules not only by various types of secondary and backscattered electrons, but also by the primary and the diffused electrons. Moreover, experimental results cannot provide quantitative information about separate types of signal electrons from the detected signal and any improvement of detection systems with the aim to discriminate unwanted signal components cannot be simply made. It requires computer simulations using properly defined and correct mathematical algorithms.

2. Simulation method

In order to understand the signal generation in the gaseous environment, the program EOD [7], used for the design of imaging, scanning and detection systems in electron microscopes, has been extended with a MC module to include the collision phenomena of electrons with the gases in the specimen chamber of the microscope. For this we use the data of cross-sections from the NIST database for ionization. Dissociation, vibration, rotation and momentum-transfer cross-sections for water molecules are included in the simulation algorithm.

The simulation uses integration of the equation of motion of electrons with variable and sufficiently small step, much smaller than the steps necessary to trace electrons in a non-gaseous environment. The step size is determined by the probability in which the interaction of electron with a gas molecule may occur, typically around 10%, and the interaction cross-section for given

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0168-9002/$ - see front matter
doi:10.1016/j.nima.2010.12.200

electron energy. According to the particle energy the MC routine selects if and which type of collision happens. After elastic collisions the electron energy is not changed, only its direction. If an ionization collision happens, a new electron is generated with a random angle and a small energy gain relevant for the ionization type. The energy of the first electron is decreased by the ionization energy and the energy transferred to the second electron; its direction is changed, depending on ionization level involved and electron energy. A correct simulation of these effects is vital for obtaining correct results for the signal amplification. A detailed description of the effects involved is given by Thiel et al. [8]. In our computations we do not include any effects related to generated ions (space charge, generation of secondary electrons as an impact on the sample, recombination of ions and electrons) and the secondaries arising from the impacts of BSEs on the microscope parts.

The computation starts with a set of electrons with given initial position, energy and angle. New "environmental" electrons are then added to the set. For each selected energy 2000 particles are started and then the amplification is determined from the total number of detected electrons. The results were calculated for SE energies of 3, 10, 20 and 50 eV and BSE energies of 100, 250, 500, 750, 1000, 3000, 10 000 and 18 000 eV; see Fig. 3.

3. Analytical models

A recent analytical model used to calculate the total amplified ion current detected with the environmental secondary electron detector was published by Meredith et al. [6]. An improved version of this model taking into account the nonlinearity of the Townsend's first ionization coefficient as well as newly calculated specific gas type coefficients was published by Thiel et al. [8]; see Eq. (1). Small modification of Thiel's model, reducing its total signal amplification by inclusion of the SE and BSE emission coefficients, was published by Morgan [9]; see Eq. (2).

The total amplified ion current I_{ion} or the total current of electrons emitted from the sample and amplified in partially ionized gases I_{ese} and detected with the detection electrode of the detector is stated by Thiel et al. [8] as

$$I_{ion} = I_{PE}\left(e^{\alpha_{ion}^{sw} d_{eff}} - 1\right)\left\{\frac{S_{PE}p}{\alpha_{ion}^{sw}} + \frac{\eta S_{BSE}p}{\alpha_{ion}^{sw}}\left(\frac{\overline{d}_{BSE}}{d}\right) + \delta\right\}k \quad (1)$$

The total amplification of the ionization detector A_{ESD} is defined by Morgan [9] as a ratio of the I_{ion} to the total amount of emitted electrons from the sample.

$$A_{ESD} = \frac{I_{ion}}{I_{PE}(\eta+\delta)} \quad (2)$$

Here I_{PE} is the primary beam current, p is the specimen chamber pressure in Pa, δ and η are the SE and BSE emission coefficients, d is the sample to detection electrode gap in mm, d_{BSE} is the average BSE path length in mm, S_{PE} and S_{BSE} are the field-independent ionization efficiencies of the primary electrons and BSEs in ion pairs/(mm Pa). For the calculation of S_{PE} an energy equal to 75% energy of primary electrons was used. The factor α_{ion}^{sw} is called Townsend's first ionization coefficient and gives an average value of ion pairs per unit of length, created by the SEs and their products in electron–gas interactions accelerated by the field of the detection electrode. The quantity k relates to the effect of positive ion impact at the sample. It is a gas-specific amplification factor related to the inelastic scattering cross-section. The pressure- and field-dependent effective gap distance d_{eff} represents a specific fraction of d when the value of α_{ion}^{sw} is approximately constant.

All the above mentioned analytical models use a simplified description of processes accompanying the impact ionization of gas molecules by signal electrons and its signal amplification in high-pressure conditions and they demonstrate the influence of microscope parameters on the shape of signal detected by an ionization detector. These models are based on simplified assumptions for the gas plate capacitor and use a physical theory for partially ionized gases published by von Engel [10]. The grounded sample, ideally straight and smooth, acts as a bottom electrode of the gas capacitor regardless of the existence of recombination sample processes, gas pressure distribution between the sample and the pressure-limiting aperture, nonlinear processes of signal amplification, etc.

4. Experiment

An experimental measurement investigating the dependence of the detected signal from a thin gold plate on the water vapor pressure as well as on other parameters was carried out using a special sample. Two holes were bored into a carbon cylinder with the diameter of 4 mm and the length of 8 mm. First, an eccentrically situated hole with the diameter of 0.5 mm and depth of 6 mm was used as a Faraday cage for measuring the absorbed primary electron current. The primary current was kept constant and measured precisely at the pressure of 0.01 Pa for each experiment using the pico-amperemeter KEITHLEY 485. The second hole, situated approximately at the center of the carbon cylinder was

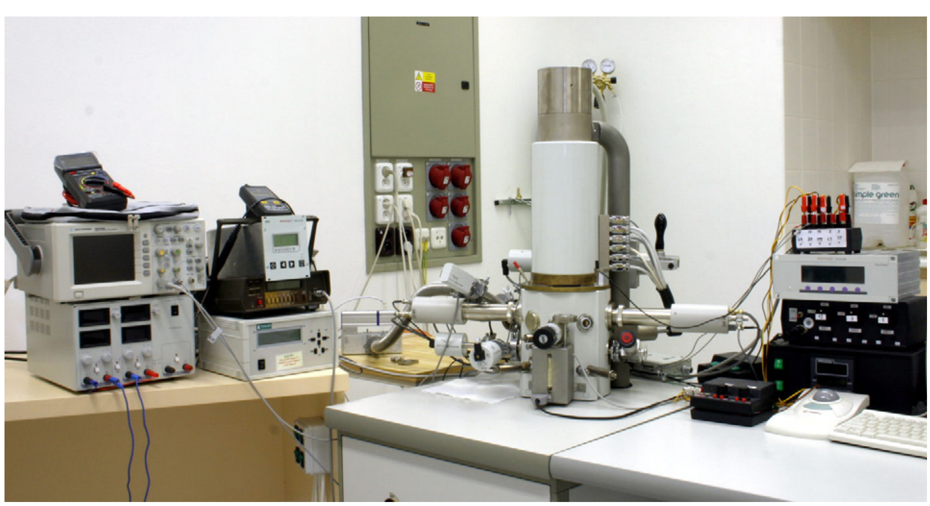

Fig. 1. Non-commercial experimental scanning electron microscope AQUASEM-II.