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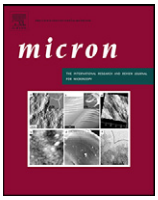

Skirting effects in the variable pressure scanning electron microscope: Limitations and improvements

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ABSTRACT

A new approach has been initiated to improve the spatial lateral resolution of the X-ray microanalysis and the backscattered electrons modes in variable pressure or environmental scanning electron microscope (VP-ESEM). This approach is based on correlation between two concepts: the electron beam skirt radius in the gas (R_S) and the generation volume radius (R_X) of X-ray signals and the generation volume radius (R_{BSE}) of backscattered electrons in the material. In order to follow the relationship between R_S, R_X and R_{BSE}, PMMA polymer, silicon oxide and aluminium are used. The results of the simulation show the existence of the best lateral resolution conditions named R (P, E) depending on the pressure and the energy for each material. This approach will enable us to propose some optimal experimental conditions to characterize different materials.

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1. Introduction

In Conventional Scanning Electron Microscope (CSEM) the penetration of the primary electrons in the material is accompanied by a broadening of the incident beam with a gradual loss of electrons energy. This is a direct consequence of elastic and inelastic scattering between electrons and solid atoms/molecules. The size of the envelope containing these interactions is commonly known as "the interaction volume" which is more elongated than the atomic number of material is lower (Goldstein and Yakowitz, 1975). During electron-material interaction, signals such as characteristic X-ray and backscattered electrons (BSE) can emerge from this interaction volume. In fact, the practical lateral spatial resolution is considered to be limited by many factors (Merli et al., 1995): the lateral spatial distribution of emitted signals "or the emission volume size within material", the primary electron beam size and the ratio between the signal and its random variation "noise". The approach used here to determine the lateral spatial resolution of the X-ray microanalysis and the backscattered electrons modes is based on the broadening of electrons within material. In high vacuum SEM, the higher lateral spatial resolution is the lower emission volume and consequently the lower interaction volume. Currently, there are several semi-empirical equations were proposed to calculate the depth electron penetration (Everhart and Hoff, 1971; Gruen, 1957; Kanaya and Okayama, 1972). On the other hand, a few studies were dedicated to calculate the lateral electron broadening (Lukiyanov et al., 2009). The Monte Carlo simulation is considered as the best way for estimating the lateral spatial resolution in the case of X-ray microanalysis and BSE electron imaging (Norman, 2001).

In variable pressure scanning electron microscope (VP-SEM), the collision of some electrons with atoms/molecules of gas is unavoidable, when the primary electron beam exit the final pressure limiting aperture PLA and enter in the specimen chamber. The main result of this collision is the scattering of electrons. However, the neutral gas itself can undergo some modifications due mainly to the ionization phenomena. In this case, the production of signals such as secondary electrons, backscattered electrons and X-rays, is not neglected. The presence of a gaseous environment in the VP-SEM modifies the primary electron beam profile then the electron beam can be generally divided into two fractions: (1) un-scattered beam, which retain the same distribution profile and also the same diameter as the original electron probe; (2) scattered beam, which affects the trajectory of the primary electron beam and distributed around it to forms what is known as a "beam skirting", usually spreads over the size of the electron probe diameter (Danilatos, 1988; Wight, 2001; Wight and Zeissler, 2000). It is of a paramount importance to know the magnitude and the extent of the electron beam skirt and how closely the scattered fraction can affect the spatial resolution of X-ray microanalyses and the BSE imaging in the VP-SEM. The fraction of beam scattered depends on several operating parameters: gas type, pressure, beam energy and the working distance. The scattered primary electron beam introduce "directly" a background noise by: firstly, signals generated from sample at large distance away from the focused probe, thus reducing signal

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to noise ratio of the analyses. Secondly, generating signals from gas adding a constant level of noise to the corresponding useful signals from the specimen.

It is of the utmost importance to be able to estimate the quantity of electrons scattered out from the electron probe. Although, the process of scattering is considered as a discrete process, this means that each electron that undergoes a scattering event between the final aperture and the surface of sample has a defined number of collisions. The statistical distribution of the electron scattered in the gas is generally governed by the Poisson distribution probability. Therefore, the results of scattering based on the average number of collision of all electrons (m) allow three different scattering regimes to be identified (Danilatos, 1988): (1) the single scattering regime ($m < 0.355$): in this case, the average number of collision is less than 5% which represent the minimal scattering regime; (2) the oligo-scattering regime ($0.355 < m < 3$): present the partial scattering regime where the average number of collision is between 5% and 95%. The most of environmental scanning electron microscope operate at this regime and the scattering effects on the primary beam become significant; (3) the plural scattering regime ($m > 3$): more than 95% of electrons are scattered out from the primary beam for the complete scattering. Hence, the plural scattering regime limits the capability of the microscope for useful X-ray microanalysis and imaging.

As motioned above, the Poisson distribution may serve as an excellent mathematical model to illustrate the new electron distribution resulting from the collisions of electrons with gas in the VP-SEM. It is well know the probability distribution $P(X)$ that the electron scattered x times is given by this equation (Danilatos, 1990c):

$$P(X) = \frac{m^X e^{-m}}{X!} \tag{1}$$

With

$$m = \sigma_T \cdot n \cdot D \tag{2}$$

Here m is the average number of scattering event per electron, where σ_T is the total scattering cross section, n is the concentration of gas particles, and D is the distance travelled by the electron between PLA (pressure limiting aperture) and the specimen surface.

The primary electron beam usually spreads over the size of the electron probe diameter given a "beam skirting" process characterized by a skirt radius (Danilatos, 1988):

$$R_S = \frac{364 \cdot z}{E}\left(\frac{P}{T}\right)^{1/2} L^{3/2} \tag{3}$$

In addition, the accelerating voltage is directly involved in the R_S. When the accelerating voltage increases, the "skirt" will decrease. Thus, the fraction of scattered electrons will also decrease.

Several of measurements artifacts appear for both microanalysis and imaging when the atmosphere gas is already in analysis chamber. In fact, there is a loss of resolution, decrease of the signal to noise ratio, interactions between emitted X-ray signals and gas, contribution of gas to the spectrum. . .then the qualitative and ideally quantitative information are disturbed and the results from the analyses will difficult to interpret.

A number of thoughtful papers have been published studying the complication imposed by the beam skirting that can greatly alter the results achieved with X-ray-microanalysis in VP-SEM (Danilatos, 1994; Gilpin and Sigee, 1995; Khouchaf and Boinski, 2007; Khouchaf and Verstraete, 2002; Mansfield, 2000; Mathieu, 1998; Sigee and Gilpin, 1994). The phenomenon of electron beam scattering obviously means that there is a significant contribution to the EDS spectrum from: (1) the presence of the environmental gas, (2) the outside the focus of the primary probe. These spurious X-rays signals limited the performance of X-ray microanalysis and prevent the high resolution of the measured Si-EDS spectrum. Considering first the case of contribution from the gas signals on spectrum quality, both characteristic and continuum (bremsstrahlung) X-rays are produced when the primary electron beam (also the backscattered electron beam from the specimen) interact inelastically with gas atoms. The extraneous X-ray peaks due to the environmental can be easily detectable on EDS spectrum and their intensities increase with increasing pressure. On the other hand, a gradual lowering of the peak intensity of the interest material is viewed due to the elastic scattering into the skirt. Indeed, the elastic scattering lead substantially to the reduction of beam current within the focused probe at the interest area on specimen, significantly degrading the lateral spatial resolution of X-ray microanalysis. Secondly, the contributions from the non-focused beam by both elastically or inelastically scattering gives the characteristic and continuum X-ray appropriate to each electron scattered location on the specimen. As a consequence of this, the X-rays produced by the remotely scattered skirt electrons are indistinguishable from those produced by the focused probe which then affects the signal to noise ratio in EDS.

The effect of beam skirting especially on the X-ray microanalysis under helium and water vapor environment is well recognized (Arnoult et al., 2011; Khouchaf et al., 2011; Khouchaf and Verstraete, 2004). To overcome these limitations and to remove the artifact generated by beam spreading in VP-SEM, two major different ways have been proposed: (a) the beam-stop method (Bilde-Sorenson and Appel, 1996, 1997) which based upon comparing two recorded spectra. The first spectrum is acquired using a fine needle of a well-known element that is not present in the sample of interest. The second spectrum is acquired in the same condition as if recorded the first spectrum without using the needle, where this spectrum contains the peaks from the area of interest and from the skirt contribution. The corrected spectrum due to the unscattered beam alone can be obtained when the peaks of needle are stripped from the first spectrum, then that the modified spectrum is subtracted from the second spectrum to eliminate the effect of the skirt. However, there are some inconveniences encountered in this method because of the time consuming, also the micromanipulator to stop the unscattered electron beam needs very precise control and the subtraction is always not perfect (Gauvin, 1999; Mansfield, 2000); (b) the pressure variation technique which based upon on predicting the true spectrum that would be obtained at "zero-scattering regime". The exact procedure as described in detail by Bilde-Sorenson and Appel (1996, 1997), Doehne (1997), Mansfield (2000) and Newbury (2002) consists to recorded two spectra at different pressure under identical conditions. The two spectra are subtracted from each other leading to calculate the corrected intensity for zero pressure for a given working distance. Another pressure variation technique was proposed by Gauvin (1999). This method consists to plotting a linear relationship of the measured intensity I as a function of the unscattered beam intensity fraction f_P. The corrected intensity I_P is given when $f_P = 1$. Even the Gauvin method appears more precise than the Doehne method; the potential limitations of the two variable pressure methods are depending on choosing of the appropriate experimental conditions such as pressure, and accelerating voltage (Le Berre et al., 2007).

The objective of this paper is to introduce a new method for choosing the optimum experimental parameters needed for X-ray microanalysis and BSE imaging in VP-SEM. This method is based on minimizing the degradation of the resolution by comparing the extent of the skirt in the medium gas with the broadening of the incident electron beam within material. The best resolution at a given pressure and energy will be calculated.

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