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Charging : a limitation to perform X-ray microanalysis in the Variable Pressure Scanning Electron Microscope

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The variable pressure scanning electron microscope (VP-SEM) allows the imaging of insulators without coating. The electrons, resulting from the interaction between the incident beam and the specimen, interact with gas molecules. Some positive ions are created and they move towards the surface of the specimen in order to neutralise it. Charging (positive or negative) occurs when a material is not able to conduct effectively the beam energy imparted to it. Charging is a phenomenon which is not perfectly known, due to the difficulty to evaluate it. Nevertheless, several studies have been published and some techniques have been proposed to evaluate this phenomenon. A measure of the specimen charging can be performed by measuring the Duane-Hunt limit (DHL) [1].

Most of the time, a linear fit is used to attend this value. But the range used for this fit can be different between an experiment to an other [2,3]. Nevertheless this value could be difficult to obtain with this kind of method for specimen presenting a high charging as illustrated on Fig 1. In this case, the mean value of the charging range appears to be the best method as presented on Fig 2.

It is possible to prevent charging using appropriate experimental conditions but skirting keeps a limitation for the performance of X-ray microanalysis in the VP-SEM. Two types of methods were presented to take into account this phenomenon : the beam-stop method and the pressure-variation method. One of these pressure-variation methods is the one developed by Gauvin [4], which used the fraction of the unscattered beam, fp, in opposition with the direct pressure used by Doehne [5]. The best advantage of Gauvin's method is the absence of assumption for the variation of the X-ray intensity with pressure. A linear behaviour of the X-ray intensity with fp is predicted and the corrected intensity is obtained at fp=1. This linear behaviour seems to be only valid until the appearance of negative charging (Fig 3.). In fact, a change in the slope is observed at the same point that the DHL becomes lower than 15keV(accelerating voltage value).

This paper will present the influence of different conditions (accelerating voltage, pressure, livetime…) on the evolution of the charging of several insulators materials (ceramics, glass …). The linear part of I=f(fp) will be extrapolated until fp=1 to obtain the corrected X-rays intensities, that will allow to perform quantitative X-ray microanalysis.

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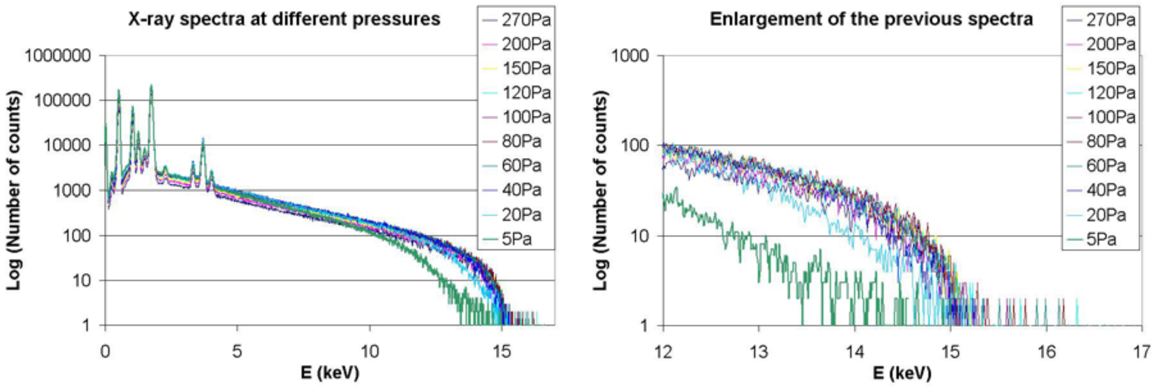

Fig 1. Energy-dispersive X-ray spectra of a glass obtained at 15keV and different pressures with an Oxford EDS system attached to the Hitachi S-3000N.

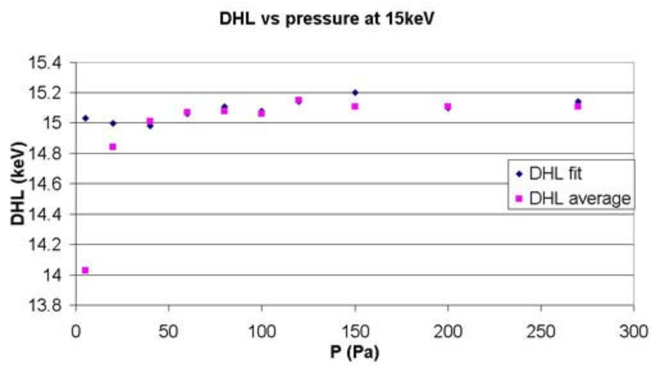

Fig 2. Dependence of the DHL obtained by two different methods with the pressure at 15keV.

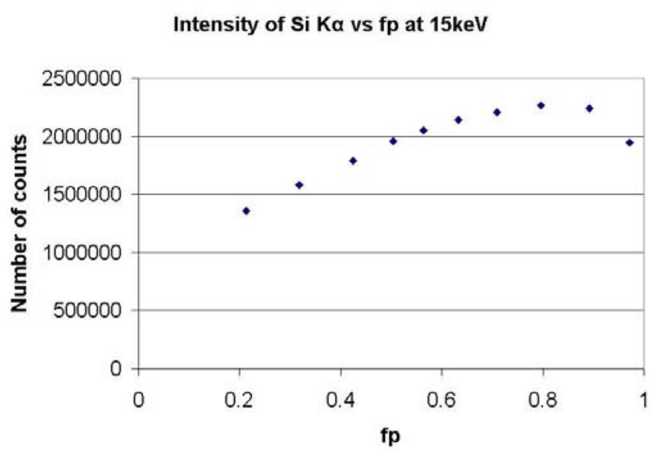

Fig 3. Evolution of the intensity of the Si Kα of a glass sample with fp at 15keV.

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