X–RAY MICROANALYSIS IN ESEM

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Introduction

In comparison with the classical scanning electron microscopy (SEM) environmetal scanning electron microscopy (ESEM) has certain advantages connected with presence of a gas environment in surroundings space of the observed specimen. The X-ray microanalysis performed in ESEM requires take into account presence of this environment. Interactions of electrons with gas atoms are pictured on Fig.1. Inelastic scatterings at which characteristic and bremsstrahlung X-rays from gas atoms arise have great influence on X-ray microanalysis of the specimen. Moreover creation of the so called electron skirt, i.e. scattering of primary beam electrons into surrounding which arises due to elastic scattering, complicates performed analysis.

Danilatos [1] presents following relation for the skirt radius

$$r_S = \frac{364\ Z}{E} \left(\frac{p}{T} \right)^{\frac{1}{2}} L^{\frac{3}{2}} \qquad (1)$$

where: r_S skirt radius (m); p pressure (Pa);
Z gas proton number; T temperature (K);
E beam energy (eV); L beam path length in gas (m).

Skirt electrons significantly degrade spatial resolution of X-ray microanalysis and cause artifacts, which complicate elemental analysis.

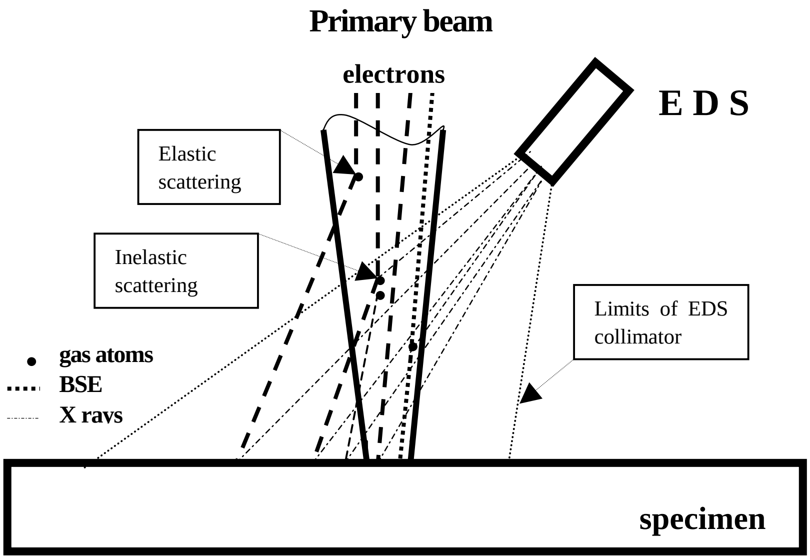

Fig. 1 *Electron scattering and X - rays collection*

Results and discussion

Artifacts related to presence of gas environment

Scattering of primary beam in given gas was calculated on the basis of equation (1) by Newbury [2]. The skirt radius is many orders higher than the radius of the primary beam (Fig.2).

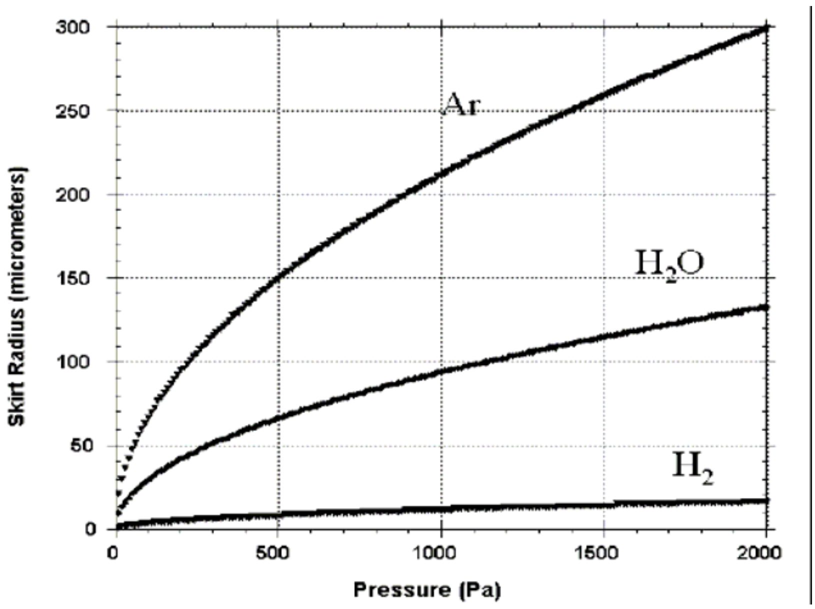

Fig. 2 *Beam broadening in ESEM (20 keV, 5 mm beam path length) [2]*

At classical signal detection in ESEM primary beam electrons which are transferred to the skirt due to gas scattering contribute only to signal noise. At X-ray analysis scattered skirt electrons interact also with atoms of specimen and produce X-rays and we do not know which X-rays are produced by primary beam and which are produced by skirt electrons. The skirt radius is proportional to $p^{1/2}$ and the skirt area is proportional to r^2, therefore area is proportional to pressure. Size of skirt can be reduced by increasing beam energy, by reducing gas environment proton number, by reducing pressure in the specimen chamber, by decreasing gas path length or by increasing temperature.

Bilde-Sorensen [3] described the beam stop method and pressure variation method for correction of influence of skirt electrons.

Presence of gas in the specimen chamber leads to generation of characteristic X-rays from gas atoms and leads also to the fall of counts in X-ray spectrum from analyzed specimen.

Charging

A great advantage of ESEM is the possibility to observe specimens of insulating character without necessity of their treatment. Newbury found out how the Duane-Hunt limit changes at influence of charging and without this influence. E.g. it is seen that at 53 Pa owing to charging it is difficult to detect peak of ZnKβ and $PbL\alpha_1$peak is significantly degraded. At 53 Pa the Duane Hunt limit dropped to 12 keV in comparison with approximately 15 keV at 266 Pa, see Fig.3.

Influence of the specimen

X-ray microanalysis in ESEM depends on a specimen configuration. There is a term "analytical blank" – it is the irreducible background level for signals. The spectrum obtained from the blank contains X-ray peaks arising from substrate, X-ray peaks from environmental gas, as well as X-ray peaks from the skirt. The operational analytical blank for ESEM considers the additional contributions (in contrast to classical SEM) that arise from the actual environment of the prepared sample, for example from particles surrounding the point of actual interest. The degree of success of microanalysis depends upon the configuration of specimen. Since the skirt electrons have high energy, it should be ideal to perform a large homogenous target. To reduce skirt spectrum contribution, we can use thin foil substrates (C, Al or Si) for small size specimen particles. If we need to analyze a specimen like fiber it is useful to put the fiber over a deep large diameter hole in a carbon block. So it is possible to eliminate the effect of skirt electrons.

The most difficult analytical situation is in the case of several phases heterogeneous microstructure analysis. The effective beam footprint due to gas scattering produces composite spectrum from all this phases.

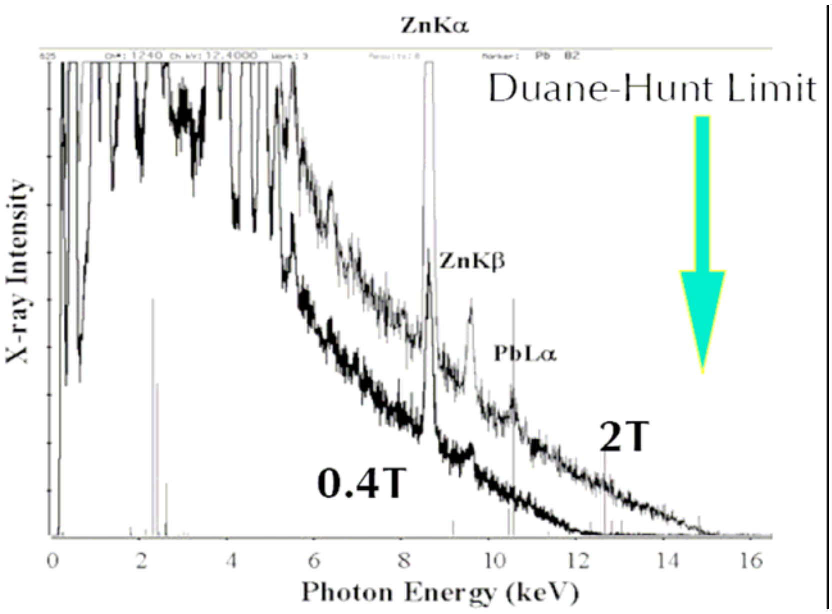

Fig. 3 *Duane-Hunt limit changes at charging (0,4 T = 53 Pa) and without influence of charging (2T = 266 Pa) [2].*

Conclusions

X-ray microanalysis in ESEM is a useful tool, but the analyst must recognize inevitable limitations that result from electron scattering in gas. The impact of electron scattering causes disadvantageous general increase of concentration limits of executed analysis by additional X-ray peaks contributed by the skirt.

Acknowledgements

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References

1. G.D. Danilatos, Foundations of environmental scanning electron microscopy, Advances in electronic and electron physics, vol. 71, Academic, p.109-249 .
2. D.E. Newbury, X-ray Microanalysis in the Variable Pressure (Environmental) Scanning Electron Microscope, Journal of Research NIST, 107, 2002, p. 567-603.
3. J.B. Bilde-Sorensen, C.C. Appel, X-ray spectrometry in ESEM and LV SEM: Corrections for Beam Skirt Effects, http://www.risoe.dk/afm/news1new_old.htm.