

ATOMIC AEM - POISSONIAN PROBLEMS FROM GAUSSIAN PROBES!

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In a previous paper (1), the authors have described the engineering requirements needed to detect one atom in the analytical electron microscope (AEM) by using x-ray microanalysis. Whilst the requirements to achieve this goal cannot be specified at present for a particular instrument, the specification for machines being developed by Vacuum Generators have a calculated minimum detection limit (MDL) of fewer than 4 atoms (2). At these detection limits the usual Gaussian statistics which have applied in AEM give way to Poissonian statistics. This paper will look at some of the interesting consequences of AEM at the atomic level.

The energy dispersive x-ray spectrometers (EDS) used in AEM have percentage detection limits usually quoted as about 0.1 wt. %. For this to equal 1 atom as the MDL, the analysed volume, defined by the probe diameter and the specimen thickness, must contain about 1000 atoms. For a field emission gun (FRG) on an AEM, sufficient current (1nA) can be obtained in a small enough probe (1nm FWHM) (3) to allow analysis from a volume containing 1000 atoms (assuming adequate x-ray detection sensitivity) if the sample is about 20 atoms thick. A model of a suitable sample is shown in figure 1. It has a thickness of only 20 atoms (for Cu this would be about 5 nm). The volume of such a model sample, having a surface area of 2500 square nm (50 nm on each side), contains about one million atoms. In this model-volume 1000 solute atoms are randomly distributed. The solute concentration is therefore 0.1 %. Columns of this model specimen were analysed theoretically, with probe sizes varied to alter the volume of specimen sampled. We were thus able to monitor the results that would be obtained during the course of multiple point analysis in an AEM that had atomic detection sensitivity!

Figure 2 shows the calculated results of 25 randomly located point analyses in the model sample. It is assumed that the probe size and EDS detection efficiency allow an MDL of 2 atoms at the EDS detection limit of 0.1%. Note that for a sample of mean composition 0.1%, the data predicts that as many as 6 results from 25 analyses would not detect any solute. Note also the asymmetric nature of the Poissonian frequency distribution $P(r)$ of the data. This is a consequence of the small number of solute atoms in the sampled volumes. The frequency distribution of multiple point analyses will become more asymmetric as the probe sizes decrease and EDS collection efficiencies are increased. When the MDL becomes less than one atom this effect will be

most dramatic. Figure 3 shows 45 theoretical analyses from the 0.1 % model sample, analysed in an AEM for which the MDL has a calculated value of 0.3 atoms. It is noteworthy that 21 analyses would not detect any solute element because there are no solute atoms in the interaction volume!

References

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- 4) The authors acknowledge the assistance of Prof. G.W. Lorimer, Manchester Materials Science Centre and Dr. R. Barlow, High Energy Physics Group, Department of Physics, University of Manchester.

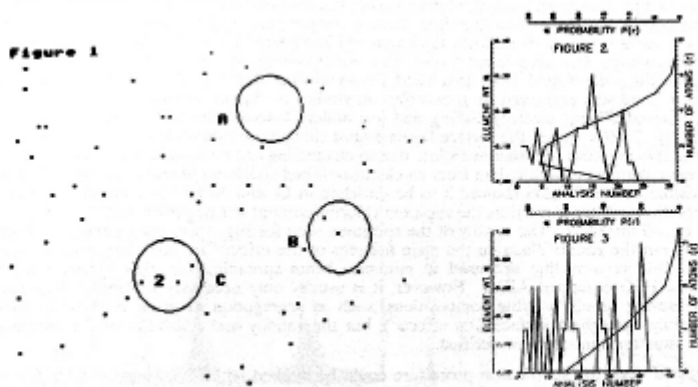


FIG 1.--Part of a 50x50nm area of a model sample 20 atoms thick. Solute atoms are randomly distributed through the model sample. Most atom columns have fewer than one solute atom. Columns marked 2 have 2 atoms. Only 11 atom columns had 2 atoms and none had more than 2 atoms. Circles represent analysed regions of sample. Area (A) contains no solute atoms, (B) contains 2, whilst (C) contains 5!

FIG 2.-- Calculated analysis results of 0.1wt.% solute in a model binary sample. The Poissonian frequency distribution $P(r)$ of the analysis results has a mean at 0.1% equivalent to a 2 atom MDL.

FIG 3.-- Calculated results from the same model specimen of figure 2. Here the distribution of analyses is calculated for an AEM with an MDL of 0.3 atoms.