

## AEM: FROM MICRONS TO ATOMS

Graham Cliff and Gordon W. Lorimer

Manchester Materials Science Centre, University of Manchester/UMIST,  
Grosvenor Street, Manchester, M1 7HS, ENGLAND

The "Manchester Connection" with analytical electron microscopy (AEM) goes back to 1913 and the work of Moseley which was carried out in the Physics Department of the University of Manchester<sup>1,2</sup>. It was Moseley who first pointed out that there is a simple relationship between  $Z$ , the atomic number of an element, and  $E_K$ , the energy of the characteristic K-shell X-ray. This relationship is enshrined in Moseley's Law,  $E_K = 10.3(Z-1)^2$ .

The origin of the modern bulk microprobe analyzer lies in the Ph.D. project of Castaing<sup>3</sup>. Under the supervision of Guinier, Castaing combined an electron microscope and an X-ray spectrometer and obtained a current of a few nA in an electron beam under a micron in diameter. Although enormous advances were made in instrumentation and quantification in the 1950's and 1960's, the spatial resolution for microprobe analysis remained at about  $1 \mu\text{m}^3$  or a mass of about  $10^{12}\text{g}$ , no matter how small the diameter of the incident electron beam. This limitation arises from the physics of the interaction of a high energy electron beam with a solid sample.

It was only possible to make a significant improvement in the spatial resolution for analysis by eliminating the diffusion-limited, activation-volume inherent in bulk microprobe analysis, i.e. by using a thin specimen. Duncumb<sup>4</sup> made the first attempt to combine the transmission electron microscope (TEM) and electron microprobe analyzer (EPMA) into EMMA-1, an "upside-down" Siemens electron microscope fitted with a crystal spectrometer. With this instrument Duncumb obtained chemical information from thin samples, but the electron optics were not designed to form a fine analytical probe. The most significant results were obtained from extraction replicas<sup>4</sup>.

The experience with EMMA-1 led, via two two prototype instruments EMMA-2 and EMMA-3, to the joint development by Cooke and Duncumb<sup>5</sup> (Tube Investments Research Laboratories) and Openshaw (AEI Scientific Apparatus)<sup>6</sup> of the first commercial analytical TEM, EMMA-4, which was based on an AEI EM802 TEM. The first instrument sold was installed in the Metallurgy Department of Manchester University in 1970. EMMA-4 was fitted with two crystal spectrometers (the original energy-dispersive analyzer was a gas-flow proportional-counter) and egress of X-rays was a major design problem. Cooke and Duncumb<sup>5</sup> and Openshaw<sup>6</sup> overcame this problem by the use of a conical "Lepoole minilens" to form the analysis probe which had a long working distance of 3.5 cm and, by today's standards, a large value of  $C_s$ , 4.2 cm. With EMMA-4 it was possible, for the first time, to form an analysis probe of minimum diameter  $0.13 \mu\text{m}$  which contained a current of 10 to 100 nA.

With EMMA-4 and a sample 100nm thick it was possible to obtain analytical information from a volume  $\sim 10^3 \mu\text{m}^3$ .

Since 1970 the spatial resolution for analysis in the analytical TEM has increased dramatically. The high X-ray collection efficiency afforded by an energy-dispersive spectrometer (0.05-0.2 sr as compared to 0.01-0.003 sr for a wavelength-dispersive spectrometer), high brightness sources, improved probe-forming lenses and the correct use of apertures to limit the effects of spherical aberration<sup>7</sup> have combined to produce a decrease in the analysis volume and the minimum detectable limit (MDL). The decrease in MDL with time from 1960 to the present is shown in Figure 1. It is interesting to note that the extrapolation to one atom occurs in the 1990's.

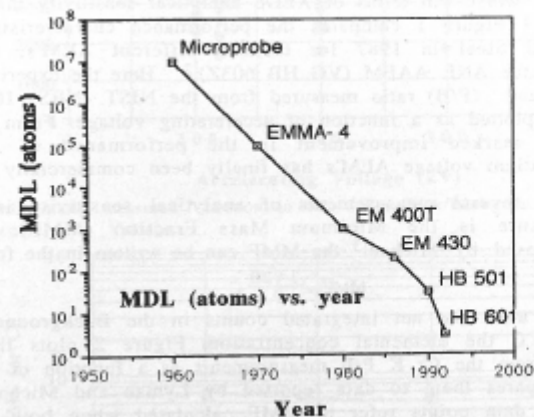


Figure 1. MDL as a function of year.

### References

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