EPMA Present and Future

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Abstract. In electron probe microanalysis the objective has always been to gain the maximum information from a sample. To this end efforts have been directed at increasing detection efficiency, reducing the size of the excitation volume, analysing more difficult specimens and improving the accuracy of quantitative measurements whilst, at the same time, attempting to minimise the complexity of the analysis. The present paper comments on the progress that has been made in these areas, focuses on the latest hardware and software developments taking place and looks forward to how these may impact on the technique of EPMA.

Key words: X-ray spectrometers; X-ray detectors; surfaces and coatings; standardless analysis; expert systems.

Predicting the future for a technique is never easy as advances tend to occur in small incremental steps and then an unexpected development takes place which totally changes the course of events. An example was the advent of the lithium drifted silicon detector, with its high collection efficiency and ‘parallel’ mode of X-ray acquisition. This device rapidly revolutionised the way analyses were performed and also broadened the scope of electron probe microanalysis (EPMA) so that it has now become a key analytical tool in both scanning and transmission electron microscopy. Although the effects have been more gradual, increasing computing power over the past quarter of a century has had a major impact on EPMA too, making data easier to acquire and to quantify as well as providing more effective forms of data presentation. Nowadays spectra may be recorded and the elements identified and quantified at the press of a button, an indication of how far the technique has progressed.

Whilst developments in EPMA will continue to be difficult to predict, it is certainly feasible to study existing instrumentation, software and procedures in order to identify areas where improvements are desirable and then to consider whether there is any likelihood of achieving these. This is the approach adopted in the present paper.

Wavelength Dispersive Spectrometry

In wavelength dispersive spectrometry (WDS) it is notable that few substantial changes appear to have taken place since the early days of microanalysis. Crystals such as lithium fluoride remain in use and the gas proportional counter is still the standard method of X-ray detection. There has been progress though: most spectrometers are now fully focusing, automated and, in some cases, have their operation integrated with that of an energy dispersive spectrometer (EDS). Also the development of Langmuir-Blodgett type pseudo crystals for EPMA [1, 2] and, more recently, the layered synthetic microstructures [3, 4] have enabled major advances to be made in both qualitative and quantitative analysis of light elements.

The principal merit of WDS is its excellent spectral resolution which makes it superior to EDS for separating closely spaced X-ray lines and detecting trace elements. However, the serial method of X-ray collection renders it relatively slow at acquiring data even with automated spectrometers. If diffraction efficiency and/or spectrometer collection efficiency could be improved then this would permit

- faster analyses,
- measurement of lower elemental concentrations,
Wittry and coworkers [5, 6] have studied the theoretical efficiency of several doubly curved diffractor geometries by considering the effective area of each contributing to diffraction. One of the most promising has the diffracting planes parallel to the surface which minimises any plastic deformation during fabrication because no grinding of the crystal surface is necessary. This diffractor is in the form of a logarithmic spiral with the X-ray source at the spiral’s origin (Fig. 2). The angle of incidence is then constant for all points on its surface and the diffracted X-rays are focused into a caustic at which position the detector is placed. By rotating the spiral about an axis joining the source and caustic position a doubly curved form is obtained. A prototype logarithmic diffractor was developed for use in the TEM specifically to measure calcium in the presence of potassium. Initial data were promising although intensities were substantially lower than expected, a result attributed to an incorrect surface profile and/or poor alignment [7, 8]. However, the usefulness of doubly curved spiral diffractors is significantly impaired because they are only fully efficient for a single X-ray wavelength, the efficiency falling off quite rapidly on either side of this value.

Another means of improving WDS performance is by incorporating the relatively new technique of capillary optics in a radically different design of spectrometer as adopted commercially by Thermo Noran [9]. This has the benefit of improving both collection and diffraction efficiency by adopting parallel beam X-ray optics. Here the divergent X-ray beam from the specimen is focused prior to its interaction with the analysing crystal and is made parallel using two different methods. The central part of the beam (with a small divergence angle) is focused by means of a polycapillary optic whilst the extremities undergo specular reflection from the surface of a parabolic mirror, Fig. 3. By arranging the X-ray source to be placed at the mirror’s focus the