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Electron Probe Microanalysis: A Review of the Past, Present, and Future

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Abstract: The 50th anniversary of the application of electron probe microanalysis (EPMA) to the Earth Sciences provides an opportunity for an assessment of the state-of-the-art of the technique. Stemming from the introduction of the first automated instruments, the latest developments of EPMA and some typical applications are reviewed with an eye to the future. The most noticeable recent technical achievements such as the field-emission electron gun, the latest generation of energy and wavelength dispersive spectrometers, and the development of analytical methods based on new sets of first-principle data obtained by the use of sophisticated computer codes, allow for the extension of the method to the analysis of trace elements, ultra light-elements (down to Li), small particles, and thin films, with a high degree of accuracy and precision and within a considerably reduced volume of interaction. A number of working examples and a thorough list of references provide the reader with a working knowledge of the capabilities and limitations of EPMA today.

Key words: electron probe microanalysis, X-ray spectrometry, latest developments, capabilities and limitations, future developments

A Brief History from a Personal Perspective

The Early Days

In 2001, on the 50th anniversary of electron probe microanalysis (EPMA), Peter Duncumb published in this Journal a fascinating account of the early days dealing with the first 10 years of the technique when "there were still fewer than 20 instruments in existence or under construction around the world" (Duncumb, 2001). His account narrated the design requirements that inspired the various first-generation instruments and showed how the later commercialization had to converge over a few variants to reconcile the many conflicting requirements.

Fourteen years after Duncumb's paper, we can consider this year the 50th anniversary of electron microprobe applications in the Earth Sciences and in particular its widespread use in determinative mineralogy. The main manufacturers of dedicated EPMA instruments are now only two, but the practice of EPMA has expanded to all electron beam instruments, mostly thanks to the development of energy dispersive spectrometry (EDS), microelectronics, and computer capabilities.

In the Fall of 1970, I (R.R.) had my first encounter with an electron probe microanalyzer. The "Probe" was in a spacious lab on the third floor of the new Geophysical Sciences building of the University of Chicago. The austerity of the outside design of the building, inspired by the Tuscan town of San Gimignano with its 100 medieval towers, was in contrast with the modern and rational use of the interior. However, the medieval austerity was preserved in the very narrow, slit-type vertical windows, providing no distraction from the outside world, thus making the scientists concentrate on their indoor activities. My supervisor, Professor Joseph V. Smith, was in charge of the lab, and he personally took me through all the steps involved in obtaining the elemental analysis of silicate minerals from the X-rays generated by a focused electron beam capable of exciting the characteristic lines of each one of the elements present in the sample. The beam could also be made to scan over the sample surface to obtain a secondary electron image similar to that obtained in a scanning electron microscope (SEM) or, alternatively, one could obtain elemental maps by modulating the cathode ray tube (CRT) brightness with the spectrometers' output. In both cases, a useful image could only be obtained by taking a photographic exposure of the CRT in the slow scan mode. Figure 1 shows the layout of that instrument. Note the absence of any video display.

Besides having worked on the theory and practice of X-ray emission in minerals (e.g., Smith, 1965; Smith & Ribbe, 1966; Ruckledge et al., 1971), Joe Smith's ability with the actual instrument was exceptional. He could keep in mind all the readings (peaks and backgrounds) of the three spectrometers involved in the analysis of the nine or so elements, and he could drive the spectrometers, two at a time, a hand-crank in each hand (turning both clockwise or anticlockwise, but not always in unison), at such speed as to complete the data collection of each analysis spot in about 10 min! When considering read-out times of 20 s on both, peak and background, it meant that he could drive the machine (literally by hand) at the same speed at which it could collect the data! Beam

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