Trace Element Mapping Using the EPMA

Nick Bulloss, Ph.D., Boise State Center for Materials Characterization

The electron microprobe analyzer (EPMA) is particularly well suited for determining the distribution of small elemental concentrations with excellent spatial resolution. Trace elements in the 100ppm range require long count times to achieve sufficient counting statistics for a valid measurement. To overcome these long count times, we can use multiple wavelength-dispersive (WDS) spectrometers measuring the same element and sum the resultant maps.

In this example the concentration of Zr has been analyzed in a titanite (CaTiSiO₅) using laser ablation inductively-coupled mass spectroscopy (LA-ICPMS). The LA-ICPMS has much lower detection limits than the EPMA but it does not have the high spatial resolution.

LA-ICPMS analyses measured Zr concentrations ranging from 125-250ppm in this titanite grain. After the LA-ICPMS data was collected, the EPMA was used to determine the distribution of Zr in the titanite grain. All five spectrometers were peaked on the Zr Lα peak and a stage map was collected using a beam current of 2000nA and a beam dwell time of 50msec. The map set took approximately four hours to complete. Looking at a single Zr map (Fig. 1) we can see the black circles from the laser ablation analyses and we can see some variation in the Zr concentration in the titanite. However, we cannot see any fine detail to the distribution of Zr.

If we sum the Zr maps from all five spectrometers we get a much clearer picture of the Zr distribution detail (Fig. 2).

To achieve the fine detail seen in figure 2 using only a single wavelength-dispersive spectrometer we would have needed to run the maps for about 20 hours.

The high spatial resolution of the EPMA enables the determination of the distribution of elements on the sub-micron scale. The EPMA’s CalcImage software has the capability to perform image math (in this example, the addition of five individual image maps). The image math feature allows us to use multiple spectrometers to collect the same element data, thereby significantly reducing analysis times.