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Characterization of sub-micron features with the FE-EPMA

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The development of a thermal field emission cathode for an electron microprobe extends analytical capabilities into the sub-micron range. The simultaneous realization of small electron beam diameters at low accelerating voltages (5–10 keV) and moderate beam currents ($<$ 100 nA) allows high spatial resolution spot analyses as well as detailed X-ray mapping of composition contrast in the range of 100 nm. Compared to conventional electron sources, the FE-EMP has both improved SEM- and BSE–imaging capabilities and, in combination with the high spatial X-ray resolution, the concept of sample homogeneity takes on a whole new meaning.

It is important to realize that this new approach is influenced by a suite of new issues: reduced X-ray intensities, peak overlap, secondary fluorescence, potentially damaging current density, contamination problems, sample preparation and coating media. Overcoming these problems requires new, sometimes unconventional measurement strategies.

By means of three examples the advantages of the FE-EPMA will be illustrated:

1. The reaction rims in natural samples were characterized in order to study the transport mechanism for the rutile-titanite transformation. Small enrichments of Al and Fe at the nm scale along grain boundaries constrain which transport mechanisms were responsible for rim formation.

2. Micron-scale inclusions within minerals were analyzed where our $\sim$100 fg sampling mass was essential for avoiding a signal contribution from the surrounding crys-

tal. For such analyses is a detailed knowledge about the scattering volume and the beam diameter a key prerequisite for achieving accurate results.

3. The characterization of complex zoning patterns within small domains of accessory minerals (zircon, monazite, etc.) is on the one hand important for the understanding of growth and alteration processes and on the other hand compositional X-ray mapping is an essential first step for achieving many modern-day research objectives, e.g. dating.