THE OKLAHOMA STATE UNIVERSITY ELECTRON MICROPROBE LABORATORY: APPLICATIONS IN NANOTECHNOLOGY

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The Oklahoma State University (OSU) Electron Microprobe is an analytical instrument that images and analyzes any material that is stable in a vacuum environment. The machine can both qualitatively and quantitatively identify elements present in material as small as ~1μm with a small degree of error. Electron Microprobe analysis is non-destructive and is used for detailed analytical study of important features in rocks, including compositional zoning of individual grains or reaction zones between grains. The machine is also commonly used by academic and industrial researchers interested in the compositions and shapes of their nanomaterials.

In Electron Microprobe analysis, high velocity electrons generated under vacuum conditions from a tungsten filament are focused through a series of electromagnetic lenses into a narrow (~1μm) beam. When the beam impacts the sample, elements in the sample emit various types of electrons and X-rays. These are used to take pictures of the sample and obtain chemical compositions. Secondary Electrons ejected from the sample surface show topographic features. Backscattered Electrons (BSE) are primary electrons emitted as a result of elastic collisions with specimen Electrons. BSE emission intensity is a function of the specimen's atomic number. For example, materials with ^{26}Fe will appear brighter than those containing ^{12}Mg.

When electrons from outer shells of an atom fall in to replace a discharged electron, they emit characteristic X-rays. These X-rays have energies specific to the elements from which they were emitted, and the amount of characteristic X-rays emitted by each element in the sample reflects its concentration. X-rays intensities are measured by either using wavelength-dispersive spectrometry for quantitative data or energy-dispersive spectrometry for qualitative mineral identification. The Electron Microprobe can also create X-ray element maps showing the relative concentrations of elements within a mineral. The electron beam is scanned across the sample, stopping at regular intervals to count the number of X-rays within a predefined energy window arriving at the detector. These maps show the concentration differences not detectable using Backscattered or Secondary Electron images.

Most of the periodic table can in principle be analyzed (boron through uranium) using the OSU Electron Microprobe, but analytical sensitivity ranges from a low of a few parts per million for optimum cases to several weight percent for some elements. Volume sampled is typically a few cubic microns. Samples must be clean and stable in a 10^{-5} torr vacuum environment. Analyses quality depends on sample preparation, character of the material, and availability of appropriate primary and secondary calibration standards for the desired elements. The OSU Electron Microprobe lab has 68 mineral and glass standards, and may be able to serve your needs.