MICROANALYTICAL FACILITIES AT HP-HT LABORATORY AT ISTITUTO NAZIONALE DI GEOFISICA E VULCANOLOGIA, ROME (ITALY)

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FESEM is suitable for a number of applications including morphological, textural and microstructural analyses as well as semi-quantitative analyses.

The usefulness of quantifying elemental compositions and observation of microstructures is invaluable in the sciences of mineralogy, petrology, structural geology and materials research. Morphology, texture and microstructure of minerals and rocks can be observed. The composition of several mineral phases as well as the composition of a single mineral phase down to and even below the level of detection is possible. In this way it is possible to correlate microstructural to specific mineralogical and petrological information. Scanning electron microscopy (FESEM) or electron probe microanalysis (EPMA) are generally considered micro-analytical techniques which are able to image or analyze material at a resolution offered by visible techniques.

By image we mean photograph an object much smaller than we can see, even with the aid of an optical microscope. The FESEM generates much less electrostatically distorted images with spatial resolution lower than 1 nm, i.e., from 3 to 4 times higher resolution than conventional SEM. FESEM is equipped with detectors for x-ray (EDS) and the secondary and back scattered electrons and can increase probe current up to 200 nA improving signal to noise ratio in x-ray map. Moreover it is possible to collect chemical maps and images from a large area of sample through a special software (navigator).

Backscattered electrons (BSE) are high energy electrons emitted from the specimen as a result of the high energy electron probe's interaction within the specimen. BSE intensities in the result of elastic events between primary electrons and other electrons within the specimen which are relatively tightly bound. BSE intensities are very much a function of the specimen's atomic number i.e., the higher the atomic number, the brighter the image.

By analyze we mean identify the elements (e.g., silicon, iron, etc.) of which the specimen is composed. Elemental analysis can also be accomplished at a micro-scale; for example, EPMA can probe a specimen in small cuts (fractions of a millimeter (0.1 mm), and not only dorky) the element present that measure them with a high precision (less than than thousand part per million). However they do have their limits. For example, not all specimens can be exposed to the high vacuum within the specimen chamber. Also, elements lighter than atomic number 8 (oxygen) can not be measured without reservations, and EPMA is not sensitive to many elements below 100 ppm. Still, this instrumentation has proved invaluable, especially for mineralogy and petrology. A high precision can vary one megawatt or another to circumvent instrumental weaknesses. The EPMA is designed to measure qualitatively composition of a solid material and is particularly useful for the high precision (less than thousand part per million) and low detection limits (commonly a few tens to few hundreds ppm). Sample of interest can be as small as a few microns across.

Chemical mapping with EPMA of Albani Hills phonolithe sample used for decarbonation experiments (on the top). Distribution of colors allows understanding of the chemical distribution in the analyzed sample. Backscattered image analysis with FESEM of the same sample (on the bottom).

FESEM is particularly suitable for grain size and shape analysis.