Electron Microscopy Laboratory (EML)



Scanning electron microscopy (SEM) became an indispensable tool for high-resolution imaging of surfaces with its ability to combine imaging with elemental microanalysis. SEM analysis of different materials revealing information about their morphology, chemical composition (by Energy Dispersive X-Ray Spectroscopy-EDS) and crystallographic characterization (by EBSD) is possible by using the currently available FE-SEM equipment at our laboratory.

BASIC PRINCIPLES

SEM is a method for high-resolution imaging of surfaces. It generates a beam of high-energy electrons in high vacuum environment, after which the electrons are focused into a small beam and then this beam is directed onto the sample. An image can be displayed by scanning the sample and collecting the appropriate electrons produced upon the electron-surface interaction with a special detector.



The generated signals upon specimen-beam interaction are schematically shown below. The primary specimen-beam interaction used for SEM analysis is known to be secondary electrons (SE). Since SE's are coming from a specimen depth of up to ~10 nm, it is possible to gather information on the surface morphology of the specimen with high resolution.

Our equipment allows obtaining high-resolution images featuring 1.2 nm resolution using the Field Emission electron gun. Furthermore, it is possible to obtain images from coated (high-vacuum mode) and un-coated (low-vacuum mode) samples.



ANALYSIS MODES:

High vacuum: conductive samples, powder samples, thin films, coated insulators. .

Low vacuum: Un-coated insulator samples, polymers, glass samples.

ESEM: Biological samples, hydrated samples.





RISING MOCH by Manual Value Tin Ball on KOH etched Si surface seems like a moon rising abov Si pyramids. This image is gathered from Quanta 400FEG SEM at 16100X and colored by using Adobe Photoshop Software.

Electron Probe Micro Analyser (EPMA)

Electron probe micro analysis is a technique for chemically analysing small selected areas of solid samples ($\geq 1 \mu m$), in which X-rays are excited by a focussed electron beam. By comparing intensities of characteristic X-rays coming from elements of specimens and standard materials, it is possible to obtain quantitative concentrations of ±1% accuracy with this technique. Photon energies or wavelengths of elements can also be obtained qualitatively by the help of characteristic X-rays. The spatial distributions of specific elements can be recorded in the form of line profiles or two-dimensional maps. Elemental concentrations can be obtained with two-dimensional maps.

FUNDAMENTAL PRINCIPLES

Electron Probe Micro Analyser (EPMA) has an energy dispersive spectrometer (EDS) and three wavelength dispersive spectrometers (WDS) having four different crystals (LDE2-TAP-PET-LIF). EPMA provides electron beam on specimens by the help of a probe which controls steady flow and beam size. Also, it measures number of characteristic X-rays of all elements by the help of WDS separating according to wavelenghts. Measurements are performed as both qualitatively and quantitatively by using WDS. WDS enables more high precision measurements than EDS.

SPECIMEN PROPERTIES

Thin sections having smooth surfaces and epoxy discs can be analysed at EPMA (Figure 1). For both specimen types, their surface should be well-polished. Properties for polished thin sections are 45-47mm length, 25mm width and 1mm thickness. Specimens which will be analysed put into process after carbon coating. Optimum thickness for carbon coating should be 20 nm. In order to achieve efficiency from carbon coating, the surface of the specimens should be smooth. Moreover, in our laboratory, calibration of standard specimen sets which used for quantitative analysis are performed periodically.



Figure 1. Specimens used in EPMA, a) thin section, b) epoxy discs

CASE STUDIES

A major part of our experiments are from geological materials (Figure 2a) especially rock forming minerals. In our laboratory, JEOL JXA-8230 model of EPMA is used and quantitative analysis giving major oxide values of the specimens, qualitative analysis, line profiles and 2D color maps (Figure 2b) are achieved by using EPMA.



Figure 2. a) Compositional view, b) line profile and elemental map of Fe and Mg from a pyroxene mineral (Atak Küçük & Toksoy Köksal, 2015. 68th Geological Congress of Turkey).

ILETİŞİM BİLGİLERİ

Experiment Principals: Aynur Atak Küçük, Zafer Artvin, Serhat Köksal (210 6481- 210 7430) e-mail: <u>mlabeml@metu.edu.tr</u>