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MICROSCOPY AND MICROANALYSIS LAB

ELECTRON PROBE MICROANALYSIS (EPMA)

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Electron Probe Microanalysis (EPMA) is an analytical technique used to determine the elemental composition of a solid sample at the micrometer scale. EPMA is based on the principles of X-ray emission spectroscopy, and it combines the imaging capabilities of an electron microscope with the quantitative elemental analysis capabilities of an X-ray spectrometer. The technique is widely employed in various fields, such as materials science, geology, metallurgy, and semiconductor research, for studying both the composition and microstructure of materials.

The underlying theory of EPMA involves the interaction between a focused electron beam and a solid sample, which generates characteristic X-rays. When a high-energy electron beam is directed onto the sample, it can cause the ejection of an inner-shell electron from the atoms within the material, creating an excited state. To return to the ground state, an outer-shell electron transitions to fill the vacancy left by the ejected electron, and in the process, an X-ray photon is emitted. The energy of the emitted X-ray photon is characteristic of the specific element and the electron shell transition involved. By measuring the energy and intensity of the emitted X-rays, the elemental composition of the sample can be determined.

EPMA instruments typically consist of an electron microscope equipped with one or more wavelength-dispersive X-ray (WDS) spectrometers or energy-dispersive X-ray (EDS) spectrometers, which are used to analyze the generated X-rays. WDS spectrometers use a crystal to diffract the X-rays, providing high spectral resolution and allowing for the separation of X-ray lines from different elements. EDS spectrometers, on the other hand, utilize a semiconductor detector to measure the X-ray energy directly, providing faster analysis and simultaneous detection of multiple elements but with lower spectral resolution compared to WDS spectrometers.

One of the main advantages of EPMA is its ability to provide quantitative elemental analysis with high spatial resolution, typically in the range of 1-2 micrometers, and high sensitivity, down to trace levels of 0.01% or lower. Additionally, EPMA can be employed for non-destructive analysis of a wide range of materials, including metals, ceramics, glasses, polymers, and geological samples. However, EPMA also has some limitations, such as a limited ability to analyze elements with low atomic numbers (typically below boron), the need for sample preparation to obtain a flat, polished surface, and the potential for sample damage due to the high-energy electron beam. Despite these challenges, EPMA remains a powerful and widely used technique for the microanalysis of solid materials.

APPLICATIONS

MATERIALS SCIENCE AND ENGINEERING:

- Phase identification and distribution in alloys
- Analysis of coatings, thin films, and multilayer structures
- Study of interfacial phenomena, such as diffusion and segregation
- Characterization of ceramics, glasses, and refractory materials
- Evaluation of corrosion and oxidation processes

GEOLOGY AND EARTH SCIENCES:

- Mineral analysis and identification
- Study of rock and mineral inclusions
- Quantitative analysis of trace elements in minerals
- Characterization of meteorite compositions
- Assessment of element distribution in ore deposits

METALLURGY:

- Quantification of alloying elements in metals
- Evaluation of precipitates and microstructures
- Analysis of inclusions in steel and other metallic materials
- Assessment of welds and heat-affected zones

SEMICONDUCTOR AND MICROELECTRONICS:

- Elemental analysis of thin films and multilayer structures
- Assessment of dopant concentration and distribution
- Characterization of interconnects, contacts, and diffusion barriers
- Evaluation of defects and contamination in semiconductor materials

ENVIRONMENTAL SCIENCE:

- Analysis of airborne particles and pollution sources
- Characterization of soil and sediment compositions
- Study of biogeochemical processes, such as element cycling and uptake

CULTURAL HERITAGE AND ART CONSERVATION:

- Elemental analysis of pigments and paint layers
- Characterization of ancient artifacts and materials
- Assessment of degradation processes and conservation treatments

EQUIPMENT AND TECHNICAL SPECIFICATIONS

- ## JXA-8530F FIELD EMISSION ELECTRON MICROSCOPE

The JXA-8530F is a field emission electron microscope that allows qualitative and quantitative elemental analysis at the highest magnifications. The system is equipped with three WDS channels and a JEOL EDS system. One of the most important advantages of WDS is the low minimum detection limits (MDL) of elements. This benefit proves to be very useful in detecting trace heavy metals with minimum or no sample preparation required by other trace elemental analysis techniques, such as ICP.

At low kV (5-7kV) with WDS (wavelength dispersive X-ray spectrometer), integrated EDS, high probe current, and small probe diameter, the JXA-8530F is capable of extreme elemental analysis of sub-micron areas (approaching 100nm). This system offers high spatial resolution in X-ray mapping and customized, detailed analysis.

The FE electron gun produces an extremely small spot size at low accelerating voltage, even with high probe currents (40nm at 10nA and 10kV, 100nm at 100nA and 10kV), allowing for WDS analyses with high X-ray spatial resolution. The microprobe size is 1/5 to 1/10 the size of that produced in a thermionic-emission electron gun in conventional EPMA with tungsten filament or LaB6 tip.

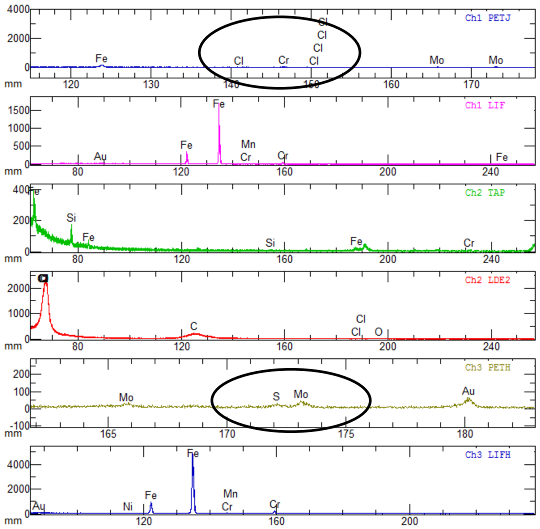
JXA-8530F users can acquire qualitative WDS spectra and semi-quantitative analyses by merely clicking a point on a secondary electron or backscattered electron image. Detailed analytical procedures can be tailored to research objectives through preset recipes. A combined WDS and EDS system provides a powerful tool for efficient data acquisition of quantitative analyses, high magnification beam scan mapping, and large area stage scan mapping.

TECHNICAL SPECIFICATIONS:

- Signals Detected: Secondary & backscattered electrons and several X-ray detectors.
- High Resolution of 3.0 nm at High vacuum
- New large crystal spectrometers for higher detection sensitivity for trace elements and increased count rate without sacrificing energy resolution and P/B ratio
- Higher sensitivity for trace analysis from Be to U.
- Detectable wavelength ranging from 0.087 to 9.3 nm.

EXAMPLES OF WORK DONE

EPMA analysis for corrosion sample showing distinctive S and Mo peaks, which overlaps in case of EDS analysis. Moreover, EPMA analysis can show trace elements when EDS fail to do so such in this case the Cr and Cl elements



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PICTURES OF EQUIPMENT

JXA-8530F Field Emission Electron Microscope