Scanning Electron Microscopy Unit

Introduction

Electron microscopy is a straightforward technique to determine the size and shape of particles. It can also reveal information on particle composition, for example by detecting X-rays, which are produced by the interaction of the electrons with matter, or by analyzing the way in which the electrons are diffracted. Electrons have characteristic wavelengths in the range of 0.1-1 nm (visible light: 400-700 nm) and some close to seeing atomic detail. The interactions of the primary electron beam (100-400keV) with the sample are summarized in Fig. 1:

- Depending on sample thickness, a fraction of the electrons passes through the sample without suffering any energy loss. These electrons are used in transmission electron microscopy (TEM).
- Electrons are diffracted by particles that have a favorable orientation towards the beam, enabling one to obtain crystallographic information.
- Electrons can collide with atoms in the sample and be scattered back; back scattering is more effective when the mass of the atom is increased.
- X-rays and Auger electrons are formed in the relaxation of core-ionized atoms, as discussed in the sections on AES and XPS.
- Electrons excite characteristic vibrations in the sample, which can be studied by analyzing the energy loss suffered by the primary electrons.
- Many electrons loose energy in a cascade of consecutive inelastic collisions. These are called secondary electrons; most of these had their last loss process in the surface region just before leaving the sample.
- The emission of a range of photons from UV to infrared is called cathodoluminescence and is caused by the recombination of electron-hole pairs. Thus, the interaction of the primary beam with the sample provides a wealth of information on morphology, crystallography and chemical composition.

Scanning electron microscopy (SEM) is carried out by rastering a narrow electron beam over the surface and detecting the yield of either secondary or back-scattered electrons as a function of the position of the primary beam. Contrast is caused by the orientation: parts of the surface facing the detector appear brighter than parts of the surface pointing away from the detector. The secondary electrons have low energies (10-50 eV) and originate from the surface of the sample. Back-scattered electrons come from deeper and carry information on the composition of the sample, because heavy elements are more efficient scatterers and appear brighter in the image. Dedicated SEM instruments have resolutions of about 5 nm. Simple versions of SEM with micron resolution are often available on Auger electron spectrometers, for the purpose of sample positioning. The main difference between SEM and TEM is that SEM sees contrast due to the topology of a surface, whereas TEM projects all information in a two-dimensional image, which is of sub nanometer resolution. A TEM instrument combines the two modes of electron microscopy.

As illustrated in Fig. 1, an electron microscope offers additional possibilities for analyzing the sample. Diffraction patterns (spots from a single-crystal particle and rings from a collection of randomly oriented particles) enable one to identify crystallographic phases as in XRD. Emitted X-rays are characteristic for an element and allow for a determination of the chemical composition of a selected part of the sample (typical dimension 10 nm). This technique is called electron microprobe analysis (EMA, EPMA) or, referring to the usual mode of detection, energy dispersive analysis of X-rays (EDAX or EDX).

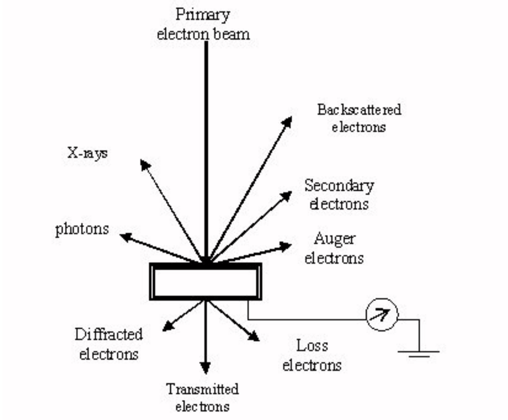

Figure 1: The interaction of the primary electron beam with a sample in electron microscopy produces a wealth of detectable signals.

Facilities & Infrastructure

The SEM equipment consists of a JEOL JSM-563 unit connected to an OXFORD LINK ISIS L300 for EDX analyses. It is located in the Department of Chemistry (Figure 2).

The development of the SEM unit in the future will necessitate the acquiring of a Scanning Tunneling Microscope (STM) as well as an Atomic Force Microscope (AFM) which represent the latest development in the field of microscopy for observation of matter at atomic level.

Figure 2: General view of the S.E.M. (Scanning Electron Microscopy) Unit

Services

The SEM unit provides services to Research groups of the University of Ioannina, other Universities and Research Institutes. Specifically, the following kind of support can be provided:

Research activities
Support of the research activities of various groups in the Departments of Chemistry, Physics, Medicine, Archeology, Materials Science and Technology. Relevant topics include study of texture of surfaces of materials, definition of the micro-structure of solids and studies of textural composition of archaeological objects.

Industrial activities
Control, identification and characterization of products from metallurgical units, silver smith small medium enterprises, food industry, pharmaceutical industries etc.

Environment
Interaction of pollutants with soil samples, identification of pollution aerosols etc.

Staff & Contact Information

A Scientific Committee, consisting of staff members of the University of Ioannina is responsible for the SEM Unit. Information can be acquired from Prof. P. Pomonis.

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Representative Publications

1. "Pore anisotropy and microporosity in nanostructured mesoporous solids'' J. Knowles, G. S. Armatas, M.J. Hudson, P.J. Pomonis, Langmuir, 22 (2006) 410.
2. "Architecture at the Mesoscale: Morphogenesis of Novel Patterned Alkaline Earth Containing Mesoporous Silica" C.C.Pantazis, A.P.Katsoulidis, P.J.Pomonis, Chemistry of Materials, 18(2006)149.
3. "Comparative study of Morphometric Properties Characterizing the Complexity of Silicate Networks Probed by Adsorption of Nitrogen and Methanol" R. Denoyel, J.M.Meneses, G.S.Armatas, J. Rouquerol, K.K.Unger, P.J.Pomonis, Langmuir, 22 (2006) 5350.
4. "Microporosity, Pore Anisotropy and surface properties of organized mesoporous silicates (OMSi) cotaining cobalt and cerium" A.P.Katsoulidis, D.E.Petrakis, G.S.Armatas, P.J.Pomonis, J. of Mater. Chem., 17 (2007) 1518.
5. "Self-organized meso- and hybridic phases of Poly-Aspartic and Poly-Glutamic Aminoacids with Cationic Surfactants C_nTAB (n=14, 16) and TEOS" E. K. Kodona, Ch. Alexopoulos, E. Panou, P. J. Pomonis, Chemistry of Materials, 19 (2007) 1853.
6. "Structure and photocatalytic performance of TiO_2/clay nanocomposites for the degradation of dimethachlor", V. Belessi, D. Lambropoulou, I. Konstantinou, A. Katsoulidis, P. Pomonis, D. Petridis, T. Albanis, Applied Catalysis B: Environmental, 73 (2007) 292.
7. "A Kinetic Study of Methane and Carbon Dioxide Interconversion over 0.5%Pt/$SrTiO_3$ Catalysts", A. Topalidis, D.E.Petrakis, A. Ladavos, L. Loukatzikou , P.J. Pomonis , Catal. Today, 127 (2007) 238.
8. "A comparative study of substituted perovskite-type solids of oxidic $La_{1-x}Sr_xFeO_{3\pm\delta}$ and chlorinated oxidic $La_{1-x}Sr_xFeO_{3\pm\delta}Cl_\sigma$ form: Catalytic performance for CH4 oxidation by O_2 or N_2O" , A.A.Leontiou, A.K.Ladavos, A.E.Giannakas, T.V.Bakas, P.J.Pomonis, Journal of Catalysis, 251 (2007) 112.
9. "Chirality and helix stability of polyglutamic acid enantiomers" E.K.Kodona, Ch.Alexopoulos, E.Panou-Pomonis, P.J.Pomonis, J. Colloids and Interface Science, 319 (2008) 72.
10. "Transesterification of soybean frying oil to biodiesel using heterogeneous catalysts",K.G. Georgogianni, A.P. Katsoulidis, P.J. Pomonis, M.G. Kontominas Fuel Processing Technology, 90(2009) 671.
11. "Organized Mesoporous Silico-Nickelates (OMSiNi) and Silico-Lanthano-Nickelates

(OMSiLaNi): Crystallogenesis vs. Morphogenesis and Microporosity vs. Pore Anisotropy", A.P.Katsoulidis, E.T.Tsaousi, G.S.Armatas, D.E.Petrakis, P.J.Pomonis, Microporous and Mesoporous Materials, 122 (2009) 175.

12. "Mesoporous titania nanocrystals prepared using hexadecylamine surfactant template: Crystallization progress monitoring, morphological characterization and application in dye-sensitized solar cells", N. Alexaki , T. Stergiopoulos , A.G. Kontos, D.S. Tsoukleris , A.P. Katsoulidis , P.J. Pomonis , D.J. LeClere , P. Skeldon , G.E. Thompson , P. Falaras , Microporous and Mesoporous Materials, 124 (2009) 52.
13. "Anomalous diffusion during isopropanol decomposition on $(ZrO_2)_{1-x}$ $(CeO_2)_x$ Catalysts", A. Iosifidis, D.E. Petrakis, P.J. Pomonis, Applied Catalysis A: General 364 (2009) 199.
14. V.N. Stathopoulos, V.C. Belessi, T.V. Bakas, S.G.Neophytides, C.N. Costa, P.J. Pomonis, A.M. Efstathiou, Comparative Study of La-Sr-Fe-O Perovskite-type Oxides Prepared by Ceramic and Surfactant Methods over the CH_4 and H_2 Lean-deNO*x*, Applied Catalysis B, Environmental, 93 (2009) 1.