Using Quantitative Electron Microscopy for Process Mineralogy Applications

P. Gottlieb, G. Wilkie, D. Sutherland, E. Ho-Tun, S. Suthers, K. Perera, B. Jenkins, S. Spencer, A. Butcher, and J. Rayner

Quantitative microscopy using scanning electron microscopy-based automatic measurement methods and data-processing techniques that provide broad ranges of applications is very popular in the mining industry. These systems determine the quantities and microtextures of the ore samples and metallurgical products to guide process development and troubleshoot processing problems.

INTRODUCTION

Microtextures and quantitative mineralogy are essential for guiding ore process design and optimization. Traditionally, mineralogists have performed much of the work manually using optical microscopy. Ore bodies, however, are often highly complex and cannot always be adequately quantified by manual quantitative microscopy. With current available technology, automatic image analyses are now possible. One of the earliest automatic systems was QEM*SEM, a quantitative electron microscopy-scanning electron microscopy system that the Commonwealth Scientific and Industrial Research Organization (CSIRO) has since developed into a general-purpose instrument for characterizing ores and investigating plant performance.

SYSTEM DESIGN

In most automatic image analysis systems, the sampling, specimen mounting, measurement mode, and data utilization schemes are optimized for specific mineral-industry applications. The QEM*SEM system design is very flexible and consists of a hardware module and a software module that control a scanning electron microscope to collect raw data, construct digital images, and process the data. The system acquires energy-dispersive x-ray (EDX) spectra and backscattered-electron image (BEI) information from each measurement point and identifies the underlying minerals using the chemical composition from x-ray information preferentially overbackscatter-electron brightness. The identified species represent individual minerals or groups of minerals with similar chemical compositions.

A database, using a priori knowledge of the mineral system, identifies minerals from low-count EDX spectra and constrains the possible identifications when there is a lack of uniqueness. Real-time processing of the signals generates digital images that classify pixels as mineral species. Characteristic peaks in the x-ray spectrum indicate the presence of a chemical element. The EDX spectrum is analyzed by windowing, background subtraction, overlap correction, thresholding, and the calculation of peak ratios to resolve individual element spectral lines.

The data obtained for each point analysis are compared with a database of mineral species. The database lists elements that must be present, and elements that may be present. Species with similar x-ray spectra and BEL such as chalcopyrite and cubanite, are differentiated by element ratios. Species with similar spectra but distinct BEL such as magnetite and hematite, are differentiated by BEL Some simple ore types can be analyzed by BEL alone using a subset of the system capabilities. Finally, off-line processing extracts mineral and particle statistics and condenses the identified species into mineral groupings suitable for interpretation.



Individual rock sections as well as particulate samples can be measured with the QEM*SEM system. Particulate samples are mixed with resin to form grain mounts that are sectioned and polished; the procedures ensure both random orientation and separation of the individual particles.

Several modes of operation are possible, with each analysis mode designed to work on a particular mineralogical or metallurgical problem. Routine analyses measure from 500 to 100,000 particle sections, depending on size fraction and modal distribution, and as much as a million or more particle sections for trace-metal searches. The following operations are generally available.

Bulk Mineralogical Analysis

The bulk mineralogical analysis (BMA) mode uses linear scans and can be used on drill core, rock, and particulate samples. To collect 40,000–100,000 points, an analysis runs for 30 to 90 minustes. The system records the number of intercepts of all mineral species; the length of each intercept; and the number and type of transitions between phases, including background (epoxy in particulate samples). From these data, modal

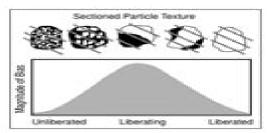


Figure 1. A schematic of the basis of stereological error, with sections (graylines) through liberated and composite particles of varying texture and the corresponding magnitude of bias.

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United States. National Bureau of Standards

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Electron Energy-Loss Spectroscopy in the Electron Microscope R.F. Egerton, 2011-07-29 Within the last 30 years electron energy loss spectroscopy EELS has become a standard analytical technique used in the transmission electron microscope to extract chemical and structural information down to the atomic level In two previous editions Electron Energy Loss Spectroscopy in the Electron Microscope has become the standard reference guide to the instrumentation physics and procedures involved and the kind of results obtainable Within the last few years the commercial availability of lens aberration correctors and electron beam monochromators has further increased the spatial and energy resolution of EELS This thoroughly updated and revised Third Edition incorporates these new developments as well as advances in electron scattering theory spectral and image processing and recent applications in fields such as nanotechnology. The appendices now contain a listing of inelastic mean free paths and a description of more than 20 MATLAB programs for calculating EELS **Quantitative Electron Microscopy** Gunter F. Bahr, Transmission Electron Microscopy Ludwig data Reimer, 2013-11-11 Transmission Electron Microscopy presents the theory of image and contrastformation and the analytical modes in transmission electron microscopy Theorinciples of particle and wave optics of electrons are described Electron specimen interactions are discussed for evaluating the theory of scattering and phase contrast Also analysed are the kinetical and dynamical theories of electron diffraction and their applications for crystal structure determination and imaging of lattices and their defects X ray microanalysis and electron energy loss spectroscopy are treated as analytical methods. The third edition includes a brief discussion of Schottky emission guns some clarification of minor details and references to the Transmission Electron Energy Loss Spectrometry in Materials Science and the EELS Atlas recent literature Channing C. Ahn, 2006-03-06 This book CD package provides a reference on electron energy loss spectrometry EELS with the transmission electron microscope an established technique for chemical and structural analysis of thin specimens in a transmission electron microscope Describing the issues of instrumentation data acquisition and data analysis the authors apply this technique to several classes of materials namely ceramics metals polymers minerals semiconductors and magnetic materials The accompanying CD ROM consists of a compendium of experimental spectra **Properties and** Microstructure R. K. MacCrone, 2013-10-22 Treatise on Materials Science and Technology Volume 11 Properties And Microstructure covers the parameters important to understanding microstructural effects The book discusses the direct observation and characterization of defects in materials the cause and effect of crystal defects in silicon integrated circuits as well as the microstructure of some noncrystalline ceramics. The text also describes microstructural defects in the important semiconductors silicon and germanium microstructural effects in glasses microstructural effects on the mechanical properties of ceramics and finally microstructures in ferrites Materials scientists materials engineers and graduate students taking related courses will find the book invaluable Principles of Electron Optics, Volume 3 Peter W. Hawkes, Erwin

Kasper, 2022-02-21 Principles of Electron Optic Volume Three Wave Optics discusses this essential topic in microscopy to help readers understand the propagation of electrons from the source to the specimen and through the latter and from it to the image plane of the instrument In addition it also explains interference phenomena notably holography and informal coherence theory This third volume accompanies volumes one and two that cover new content on holography and interference improved and new modes of image formation aberration corrected imaging simulation and measurement 3D reconstruction and more The study of such beams forms the subject of electron optics which divides naturally into geometrical optics where effects due to wavelength are neglected with wave optics considered Includes authoritative coverage of the fundamental theory behind electron beams Describes the interaction of electrons with solids and the information that can be obtained from electron beam techniques Addresses recent relevant research topics including new content on holography and interference new modes of image formation 3D reconstruction and aberration corrected imaging simulation and measurement Advances in Optical and Electron Microscopy T Mulvey, C. J. R. Sheppard, 2017-07-14 Advances in Optical and Electron Microscopy Volume 11 compiles papers on the important developments in optical and electron microscopy This book discusses the instrumentation and operation for high resolution electron microscopy diffraction pattern and camera length and electron microscopy of surface structure. The history of surface imaging by conventional transmission electron microscopy ion probe microscopy and secondary ion mass spectrometry with high lateral resolution are also elaborated This text likewise covers the acoustic microscopy quantitative methods biological applications and near surface imaging of solids and interior imaging This publication is a beneficial to students and individuals researching on optical and electron microscopy **Publications of the National Institute of Standards and Technology ... Catalog** National Institute of Standards and Technology (U.S.),1977 **Energy-Filtering Transmission** <u>Electron Microscopy</u> Ludwig Reimer, P.W. Hawkes, 2013-06-29 Energy Filtering Transmission Electron Microscopy EFTEM presents a summary of the electron optics the electron specimen interactions and the operation and contrast modes of this new field of analytical electron microscopy The electron optics of filter lenses and the progress in the correction of aberrations are discussed in detail An evaluation of our present knowledge of plasmon losses and inner shell ionisations is of increasing interest for a quantitative application of EFTEM in materials and life sciences This can be realized not only by filtering the elastically scattered electrons but mainly by imaging and analyzing with inelastically scattered electrons at different energy losses up to 2000 eV The strength of EFTEM is the combination of the modes EELS ESI ESD and REM Publications of the National Bureau of Standards ... Catalog United States. National Bureau of Standards, 1977

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Transmission

Electron Microscopy C. Barry Carter, David B. Williams, 2016-08-24 This text is a companion volume to Transmission

Electron Microscopy A Textbook for Materials Science by Williams and Carter The aim is to extend the discussion of certain

topics that are either rapidly changing at this time or that would benefit from more detailed discussion than space allowed in the primary text World renowned researchers have contributed chapters in their area of expertise and the editors have carefully prepared these chapters to provide a uniform tone and treatment for this exciting material. The book features an unparalleled collection of color figures showcasing the quality and variety of chemical data that can be obtained from today s instruments as well as key pitfalls to avoid As with the previous TEM text each chapter contains two sets of questions one for self assessment and a second more suitable for homework assignments Throughout the book the style follows that of Williams Carter even when the subject matter becomes challenging the aim is always to make the topic understandable by first year graduate students and others who are working in the field of Materials Science Topics covered include sources in situ experiments electron diffraction Digital Micrograph waves and holography focal series reconstruction and direct methods STEM and tomography energy filtered TEM EFTEM imaging and spectrum imaging The range and depth of material makes this companion volume essential reading for the budding microscopist and a key reference for practicing researchers using these and related techniques **Principles and Techniques of Electron Microscopy** M. A. Hayat, 1977 Specimen damage in the electron microscopy Freeze drying for electron microscopy Image reconstruction of electron micrographs by using equidensite integration analysis G banding of chromosomes Autoradiographic localization of DNA in nonmetabolic conditions Optical analysis and reconstruction of images Mirror electron microscopy Electron microscopy of banded mammalian chromosomes Equidensitometry some neurobiological applications Publications of the National Bureau of Standards, 1976 Catalog United States. National Bureau of Standards, 1977 Visualization of Nucleic Acids Gerard Morel, 1995-03-30 This book presents a review of the principle approaches for visualizing DNA and RNA Using scanning tunneling and atomic force microscopes the three dimensional image of the surface of nucleic acids can be seen at atomic scale resolutions Spreading methods provide useful details on structural features of isolated molecules but the major constituent of living matter is water and the cryomicroscope makes it possible to look at DNA in its aqueous environment Genes can be detected simultaneously in situ in chromosomes using fluorescent probes and also at the electron microscopic level In cells nucleic acids are localized and quantified by dyes electron microscopy is used with cytochemical immunocytological nuclease and in situ hybridization methods The main potential applications for pathological studies are shown with particular aspects such as viral nucleic acids and in situ PCR **Biomedical Applications of Microprobe** Analysis Peter Ingram, John D. Shelburne, Victor L. Roggli, Ann LeFurgey, 1999-10-29 Biomedical Applications of Microprobe Analysis is a combination reference laboratory manual for the use of microprobe analysis in both clinical diagnostic and research settings Also called microchemical microscopy microprobe analysis uses high energy bombardment of cells and tissue in combination with high resolution EM or confocal microscopy to provide a profile of the ion metal and mineral concentrations present in a sample This allows insight into the physiology and pathophysiology of a wide variety of cells and

tissues This book describes methods for obtaining detailed information about the identity and composition of particles too small to be seen with the naked eye and describes how this information can be useful in diagnostic and biomedical research Up to date review of electron microprobe analysis Detailed descriptions of sample preparation techniques Recent technologies including confocal microscopy infrared microspectroscopy and laser raman spectroscopy Over 100 illustrations with numerous specific applications Contributions by world renowned experts in the field Brief summary of highlights precedes each chapter Image Formation in Low-voltage Scanning Electron Microscopy Ludwig Reimer, 1993 While most textbooks about scanning electron microscopy SEM cover the high voltage range from 5 50 keV this volume considers the special problems in low voltage SEM and summarizes the differences between LVSEM and conventional SEM Chapters cover the influence of lens aberrations and design on electron probe formation the effect of elastic and inelastic scattering processes on electron diffusion and electron range charging and radiation damage effects the dependence of SE yield and the backscattering coefficient on electron energy surface tilt and material as well as the angular and energy distributions and types of image contrast and the differences between LVSEM and conventional SEM modes due to the influence of electron specimen interactions Publications United States. National Bureau of Standards, 1977 **Desorption Induced by Electronic Transitions DIET V** Alan R. Burns, Ellen B. Stechel, Dwight R. Jennison, 2013-03-07 This volume in the Springer Series on Surface Sciences presents a recent account of advances in the ever broadening field of electron and photon stimulated sur face processes As in previous volumes these advances are presented as the proceedings of the International Workshop on Desorption Induced by Electronic Transitions the fifth workshop DIET V was held in Taos New Mexico April 1 4 1992 It will be abundantly clear to the reader that DIET is not restricted to desorption but has for several years included photochemistry non thermal surface modification exciton self trapping and many other phenomena that are induced by electron or photon bombardment However most stimulated surface processes do share a common physics initial electronic excitation localization of the excitation and conversion of electronic energy into nuclear kinetic energy It is the rich variation of this theme which makes the field so interesting and fruitful We have divided the book into eleven parts in order to emphasize the wide range of materials that are examined and to highlight recent experimental and theoretical advances Naturally there is considerable overlap between sections and many papers would be appropriate in more than one part Part I focuses on perhaps the most active area in the field today electron attachment Here the detection and characterization of negative ions formed by attachment of elec trons supplied externally from the vacuum are discussed In addition the first observations of negative ions formed by substrate photoelectrons are presented **Electron Probe Microanalysis** Karl Zierold, Herbert K. Hagler, 2013-03-08 The aim of electron probe microanalysis of biological systems is to identify localize and quantify elements mass and water in cells and tissues The method is based on the idea that all electrons and photons emerging from an electron beam irradiated specimen contain information on its structure and composition In particular

energy spectroscopy of X rays and electrons after interaction of the electron beam with the specimen is used for this purpose However the application of this method in biology and medicine has to overcome three specific problems 1 The principle constituent of most cell samples is water Since liquid water is not compatible with vacuum conditions in the electron microscope specimens have to be prepared without disturbing the other components in parti cular diffusible ions elements 2 Electron probe microanaly sis provides physical data on either dry specimens or fully hydrated frozen specimens This data usually has to be con verted into quantitative data meaningful to the cell biologist or physiologist 3 Cells and tissues are not static but dynamic systems Thus for example microanalysis of physiological processes requires sampling techniques which are adapted to address specific biological or medical questions During recent years remarkable progress has been made to overcome these problems Cryopreparation image analysis and electron energy loss spectroscopy are key areas which have solved some problems and offer promise for future improvements

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