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ANALYSIS*

**NOMENCLATURE, SYMBOLS AND
UNITS RECOMMENDED FOR *IN SITU*
MICROANALYSIS**

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Comments on these recommendations are welcome and should be sent within 8 months from December 1983 to the Secretary of the Commission:

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Comments from the viewpoint of languages other than English are especially encouraged. These may have special significance regarding the publication in various countries of translations of the nomenclature eventually approved by IUPAC.

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NOMENCLATURE, SYMBOLS AND UNITS RECOMMENDED FOR IN SITU MICROANALYSIS

The extensive use of physical techniques for the characterization of microstructures of solid materials has been realized by the Commission and recommendations concerning in situ microanalysis are provided in order to unify and simplify communication. Standard technique names are proposed and general nomenclature is clarified. Acronyms for the individual techniques as well as symbols and units for reporting data are presented.

INTRODUCTION

In situ microanalysis as an important part of physical analysis contributes considerably to the development and expansion of modern analytical chemistry. As a consequence of rapid growth and diversification a variety of nonconsistent technique names exist. It is therefore the purpose of this project to clarify nomenclature by giving definitions and by proposing tentative technique names and acronyms.

This report is a continuation of the project of this Commission "Recommendations for Nomenclature, Standard Procedures and Reporting of Experimental Data for Surface Analysis Techniques" (ref. 1). Further definitions and recommendations concerning in situ microanalysis are given in the publication "Manual of Symbols and Terminology for Physicochemical Quantities and Units" (ref. 2) and the reports "Nomenclature, Symbols, Units and their Usage in Spectrochemical Analysis" (ref. 3).

1. GENERAL AND TECHNIQUE NOMENCLATURE

In Situ Microanalysis: Term used for direct analytical investigation of the microstructural domains of a solid by focused beams of particles and radiation. Analytical characterization includes obtaining information about type, quantity and distribution of the elements, their state of chemical bonding, morphology and crystalline (geometric) and electronic structure of the individual phases. The combination of this information serves as a basis for the property-relevant characterization of solids.

Electron Probe Microanalysis (EPMA): General term for methods using bombardment of a solid specimen by electrons which generate a variety of signals providing the basis for a number of different analytical techniques.

Electron Probe X-Ray Microanalysis (EPXMA): Any analytical technique which uses the excitation and evaluation of the characteristic X-ray spectrum of a solid specimen by a focused electron beam (typically of a diameter of less than 1 μm). Qualitative and quantitative X-ray analysis is the main feature of EPXMA. For obtaining supplementary information the secondary, backscattered and absorbed electrons are also frequently observed. Qualitative and quantitative X-ray analysis is, however, the main feature of EPXMA.

Scanning Electron Microscopy (SEM): Any analytical technique which involves the generation and evaluation of secondary electrons (and to a lesser extent back scattered electrons) by a finely focused electron beam (typically 10 nm or less) for high resolution and high depth of field imaging.

Transmission Electron Microscopy (TEM): Any technique in which an electron transparent sample is bombarded with an electron beam and the intensity of the transmitted electrons which is determined by scattering phenomena (electron absorption phenomena) in the interior of the sample is recorded. TEM essentially provides a high resolution image of the microstructure of a thin sample.

This technique is often just called electron microscopy. The term transmission electron

microscopy is however recommended for the sake of a clear distinction from other electron microscopic techniques.

Scanning Transmission Electron Microscopy (STEM): A special TEM-technique in which an electron transparent sample is bombarded with a finely focused electron beam (typically of a diameter of less than 10 nm) which can be scanned across the specimen or rocked across the optical axis and transmitted, secondary, back scattered and diffracted electrons as well as the characteristic X-ray spectrum can be observed. STEM essentially provides high resolution imaging of the inner microstructure and the surface of a thin sample (or small particles), as well as the possibility of chemical and structural characterization of micrometer and sub-micrometer domains through evaluation of the X-ray spectra and the electron diffraction pattern.

Although there is a large degree of overlap between STEM and TEM with the latter also providing the possibility of X-ray analysis in special instruments, it is recommended to make this distinction since the scanning feature in combination with the very small beam diameter provides largely extended analytical capabilities. The term "analytical electron microscopy" which is found in the literature is undesirable because it is neither precise nor meaningful.

Transmission High Energy Electron Diffraction (THEED): Any technique which is based on the diffraction of high energy electrons ($E_0 = 10 - 200$ keV) in crystalline materials and evaluation of the angular distribution of the transmitted electrons. The diffraction pattern represents an image of the reciprocal lattice and therefore contains information about crystal structure.

This technique is also often called Selected Area Electron Diffraction (SAED). For consistency with other electron diffraction techniques however the term THEED is recommended.

Reflection High Energy Electron Diffraction (RHEED): Any technique which measures the angular intensity distribution of electrons "reflected" from a crystalline surface under bombardment with high energy electrons near grazing incidence. The diffraction pattern provides a very surface sensitive information (information depth ~ 1 nm) on the atomic arrangement of the top layers of a solid.

Low Energy Electron Diffraction (LEED): Any technique which measures the angular intensity distribution of electrons reflected from a crystalline surface under bombardment with low energy electrons ($E_0 < 500$ eV) in larger angles of incidence. The diffraction pattern also provides very surface sensitive information on the atomic arrangement of the top layers of a solid.

In Situ Micro-X-Ray Diffraction (Kossel-technique): Any technique which utilizes the diffraction of X-ray generated in a microstructural domain of a solid under bombardment with a finely focused electron beam, thus providing an X-ray diffraction pattern of this microstructural domain. The pattern can be recorded with a film either on the reflection or transmission side of the specimen (in the latter case the crystalline sample has to be a thin film or a small particle).

(Transmission) Electron Energy Loss Spectroscopy (TEELS): Any technique in which an electron transparent specimen is bombarded with a finely focused electron beam and the energy distribution of the transmitted electrons is measured. This energy spectrum contains features corresponding to discrete losses of energy of the transmitted electrons due to excitation of electronic or plasmon states and provides information on the identity (and in some cases) chemical bonding of the elements in the sample.

Reflection Electron Energy Loss Spectroscopy (REELS) (also called High Resolution Energy Loss Spectroscopy (HRELS)) (ref. 4): Any technique in which a specimen is bombarded with a focused low energy ($E_0 < 10$ eV) electron beam and the energy distribution of the reflected electrons is measured. This energy distribution contains features corresponding to discrete losses of energy of the reflected electrons due to excitation of vibrational and plasmon states and provides information on the type and geometric structure of compounds at the surface of the specimen.

Auger Electron Spectroscopy: Any technique in which a specimen is bombarded with keV-energy electrons or X-rays, and the energy distribution of the electrons produced through radiationless de-excitation of the atoms in the sample (Auger electrons) is recorded. The derivative curve may also be recorded (ref. 1).

Ion Probe Microanalysis (IPMA): Any technique in which the specimen is bombarded by a focused beam of (primary) ions (diameter less than 10 μm) and the (secondary) ions ejected from the specimen are detected after passage through a mass spectrometer (ref. 1).

Ion Microscopy refers to the use of the SIMS technique to obtain micrographs of the elemental (or isotopic) distribution at the surface of a sample with a spatial resolution of 2 μm or better (ref. 1).

Laser Raman Microanalysis (LRMA): Any technique in which the specimen is bombarded with a finely focused laser beam (diameter less than 10 μm) in the UV or visible range and the intensity vs. wavelength function of the Raman radiation is recorded yielding information about vibrational states of the excited substance and therefore also about functional groups and chemical bonding.

Particle Induced X-Ray Emission Spectroscopy (PIXES): Any technique in which the specimen is bombarded with a focused beam of high energy particles (protons, α -particles or heavier ions) and the characteristic X-ray spectrum generated in the specimen is recorded.

Laser Micro Mass Spectrometry (LAMMS): Any technique in which a specimen is bombarded with a finely focused laser beam (diameter less than 10 μm) in the UV or visible range under conditions of vaporization and ionization of sample material and in which the ions generated are recorded with a time-of-flight mass spectrometer.

Laser Micro Emission Spectroscopy (LAMES): Any technique in which a specimen is bombarded with a finely focused laser beam (diameter less than 10 μm) in the UV or visible range under conditions of vaporization and thermal excitation of electronic states of sample material and in which the photon emission spectrum is observed.

2. RELATED TERMS

Inner Orbital X-Ray Emission Spectra: X-ray spectral lines generated by the transition of electrons between "inner orbitals". The term "inner orbital" serves to designate the sharp, not degenerate electronic levels of atoms in a solid and therefore is a general term for all orbitals except the valence band.

Valence Band X-Ray Emission Spectra: X-ray spectra generated by the transition of electrons involving the valence band. The term valence band designates the set of degenerate energy levels of a solid which originates through the interaction (splitting) of atomic electronic levels in bond formation.

Primary Electrons (PE): Electrons generated by a thermal or field emission source used to bombard the specimen for generation of the analytical signals.

Beam Current: The number of primary electrons reaching the surface of the specimen per unit time expressed as electrical current. Recommended symbol: i_B ; unit: A; typical range: nA. Recommended measurement technique: Faraday cage.

Absorbed Electrons: Term for the (excess) electrons present in a specimen under electron bombardment which are lead to ground and measured as specimen current. The number of absorbed electrons per unit time (or the specimen current), equals the number of primary electrons minus the number of backscattered, secondary and transmitted electrons per unit time. Therefore the fraction of electrons being absorbed depends on many parameters, including the composition and thickness of the specimen, the primary electron energy, the electron incidence angle and local electrostatic fields when present.

Recommended symbol for specimen current: i_s unit: A; typical range: nA. Absorbed Electron Coefficient (symbol a_e): Number of absorbed electrons per primary electron.

Secondary Electrons (SE): All electrons emitted from the surface of a solid except back scattered primary electrons.

In practice: Electrons emitted from the surface of a solid under particle bombardment which have a kinetic energy of less than 50 eV are called secondary electrons.

Secondary Electron Yield (symbol δ_e): The number of SE's generated per primary electron for a given specimen and experimental conditions: The secondary electron yield depends on the (mean) atomic number of the excited area of the sample, the angle between electron beam and sample surface, the primary electron energy, thickness of the sample and sample potentials.

Back Scattered Electrons (BSE): All primary electrons which are scattered out of the original direction and retransmitted through the surface of the solid. In practice: Electrons emitted from the surface of a solid under electron bombardment which have a kinetic energy in the range between 50 eV and excitation energy (E_0).

Back Scatter Coefficient (symbol η_e): Number of BSE's generated per primary electron for a given specimen and experimental condition. The back scatter coefficient depends on (mean)

atomic number of the excited area of the sample, angle between electron beam and sample surface, primary electron energy and thickness of the sample.

Excitation Energy (sometimes called acceleration energy or initial energy): Kinetic energy of bombarding particles at the surface of the solid. Recommended symbol: E_0 ; unit: keV or eV.

Critical Excitation Energy: Minimum energy to excite a specific analytical signal. The term critical excitation energy is most frequently used in connection with the generation of X-ray spectra, designating the ionization (binding) energy of the orbital on which the transition ends. Recommended symbol: E_q (q = term for levels); unit: keV or eV.

Beam Diameter: Diameter of the beam between which 68 % of the electrons fall. For Gaussian beam shapes this corresponds to the 2σ -value of the intensity distribution of the beam. Measurement can be carried out by scanning a beam across a sufficiently sharp edge and recording the transmitted electrons.

Lateral Resolution: For practical purposes in *in situ* microanalysis it is suitable to distinguish between lateral resolution for qualitative and for quantitative analysis.

For qualitative analysis the lateral resolution should be defined as the minimum distance of two points (areas) on the specimen corresponding to signal levels of 16 and 84 percent. For electron signals originating from top surface layers like secondary or Auger electrons and secondary ions the lateral resolution for qualitative purpose corresponds to the beam diameter. For signals originating in a greater depth of the sample (back scattered electrons, X-rays) the lateral resolution is worse than the corresponding value of the beam diameter due to the diffusion of the primary electrons. The lateral resolution may be determined exactly with sandwich specimen or sharp edge specimen, or approximately with specimen showing a regular microstructure of known dimension.

Recommended abbreviation: Lat. Res. (qualitative); unit: m; range: nm or μm .

For quantitative analysis the lateral resolution should be defined as the minimum distance of two points (areas) on the specimen corresponding to signal ratios of 10^4 . This condition assures that the value for the lateral resolution defines the diameter of the analytical area which yields the total analytical signal. Due to the effect of electron diffusion in a solid and "tail effects" in ion beam analysis the quantitative lateral resolution is significantly larger than the beam diameter.

Recommended abbreviation: Lat. Res. (quantitative); unit: m; range: nm or μm .

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