17.3 Other special techniques

Beside the various electron spectroscopic techniques, there are several other special techniques which were developed especially the *in situ* microanalysis and surface analysis of solids. Those techniques which are applied in surface analysis but also widely used in other fields are described in subsection 17.7.

Electron probe microanalysis (EPMA)

This is a 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. See 17.2.2.

Electron probe X-ray microanalysis (EPXMA)

This describes 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.

Field ion microscopy (FIM)

The field ion microscope (FIM) has the capability of imaging surfaces with high lateral resolution on an atomic scale. An imaging gas (usually a noble gas or hydrogen) is ionised near the hemispherical emitter surface and the ions are accelerated in the high electrostatic field towards a phosphorescent screen or image amplifier where the image is displayed. Contrast in the field ion image is due to local variations in ion yield caused by local differences in field strength, charge penetration into the solid and tunnelling probability (wave overlap). At cryogenic temperatures the position of individual lattice atoms can be located with a precision of better than 0.1 nm.

In situ micro X-ray diffraction (Kossel technique)

This describes any technique which utilizes the diffraction of X-rays 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).

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.

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.

Ion scattering spectrometry (ISS)

Any technique using low energy (<10 keV) ions in which the bombarding particles scattered by the sample are detected and recorded as a function of energy and/or angle. This technique is used mainly for determining the composition and structure of the first atomic layer of a sample.

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.

Laser microprobe mass analysis (LAMMA)

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 mass spectrometer.

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.

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 ($<$ 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.

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.

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 patter provides a very surface sensitive information (information depth $\approx$ 1 nm) on the atomic arrangement of the top layers of a solid.

Rutherford Backscattering (RBS)

Also referred to as *backscattering spectroscopy* (*BSS*). Any technique using high energy particles directed towards a sample, in which the bombarding particles are detected and recorded as function of energy and/or angle. The technique is mostly used for determining depth distributions of elements based on the energy of backscattering particle. In general, He^+ or H^+ particles are used at energies in the order of 100 keV to some MeV.

Scanning electron microscopy (SEM)

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

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, backscattered 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 characterisation of micrometer and submicrometer 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.

Secondary ion mass spectrometry (SIMS)

Any technique in which the sample is bombarded with a stream of (primary) ions and the (secondary) ions ejected from the sample are detected after passage through a mass spectrometer.

Thermal desorption spectroscopy; Temperature programmed desorption (TPD)

Thermal desorption consists of the removal of particles from the surface by thermal excitation induced by heating the substrate. The desorbing particles are detected by measuring the pressure increase or more specifically by mass spectrometric determination of desorbing particle fluxes. Desorption processes also have been measured by monitoring the time-dependent surface coverage, for instance for work function data. The thermal desorption ("flash-filament", "flash-desorption" or "temperature programmed desorption") method is used to study the surface composition and the binding states of adsorbates, the population of these states, as well as the kinetics of conversion and desorption.

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.

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.