

Scanning Electron Microscopy / Electron Probe X-ray Microanalysis (SEM/EPMA)

- fast inspection
- topography
- material contrast
- semi-quantitative analysis
- element mapping

Scanning Electron Microscopy (SEM) is a well-known and very popular imaging technique, making use of the emission of electrons from a surface when irradiated by a scanning electron beam. The information in the images is based on either topography or composition. The X-rays that are emitted as a result of the electron irradiation provide quantitative information on the local chemical composition.

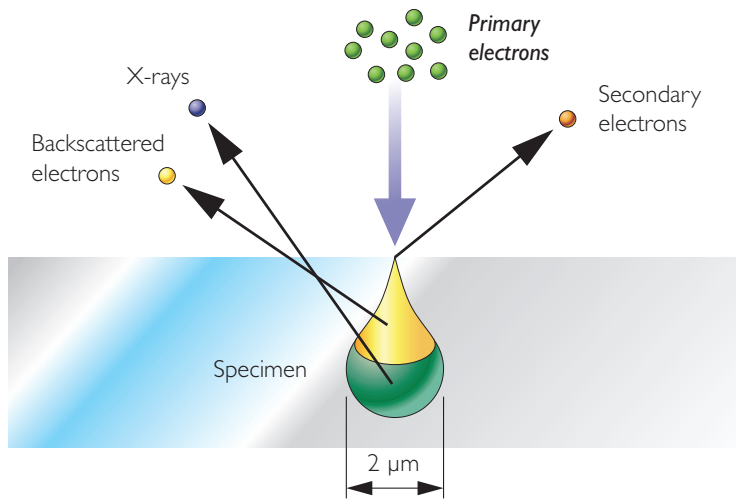


Fig. 1: Schematic of the various signals generated by the interaction of the electron beam with the sample.

Specimen beam interactions

Scanning Electron Microscopy (SEM) and Electron Probe X-ray Micro-Analysis (EPMA) can be applied as separate techniques for microscopy and analysis, respectively, or as a combination of both.

In both techniques a narrow beam of mono-energetic electrons is focused within the area of interest of the specimen surface. The accelerating voltage of this primary beam can be varied from 100 V to 30 kV. With SEM and EPMA different effects of these electrons on the material are used for analysis.

When the electron beam hits the sample, the interaction of the beam electrons with the sample atoms generates a variety of signals (see figure 1). These include secondary electrons (electrons from the sample itself), backscattered electrons (beam electrons that bounce off of atoms in the sample), and X-rays.

Scanning Electron Microscopy (SEM)

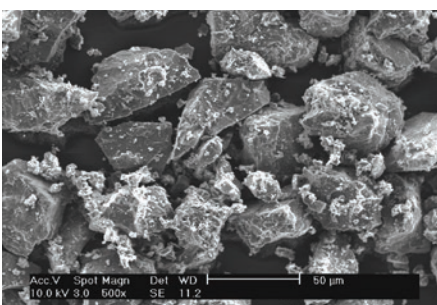
In SEM the focused electron beam is scanned over the area of interest. The resulting secondary (SE) and backscattered (BSE) electrons are used for making an image of the sample.

Secondary electrons are created by an incident electron that transfers energy to an electron of the sample. This excited electron then leaves the sample with a very small kinetic energy. Due to this low energy, only SE's that are created near the surface can exit the sample and can be detected. Any variation in topography of the surface will change the yield of SE's. This yield depends on the tilt angle of a surface, the enhanced emission at edges and small particles, and surface roughness (see figure 2a).

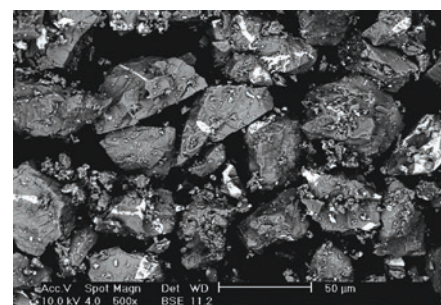
Backscattered electrons are beam electrons that bounce back from the sample after collision with atoms in the specimen. The most important contrast mechanism of backscattered electrons is the dependence of the backscattering coefficient on the mean atomic number: regions with higher atomic number elements appear brighter than lower atomic number elements in the BSE image. This allows phases with differences in atomic number to be recognized (see figure 2b). The yield of backscattered electrons is very sensitive to small crystal tilts. This allows differently orientated grains in polycrystalline material to be discriminated (see figure 3).

Fig. 2: A Pd containing powder imaged using three different techniques, providing complementary information: (a) SE image (b) BSE image (c) X-ray element mapping of Pd. The SE image provides a large depth of field, but does not display compositional variations. In the BSE image Pd-rich areas appear brighter. These bright regions correspond well to the Pd-rich regions in the X-ray map.

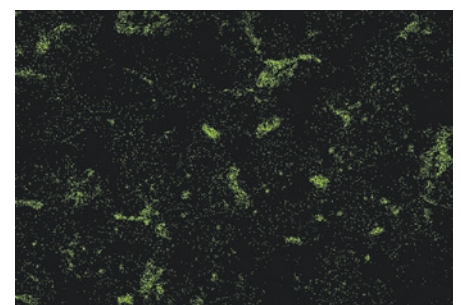
a



b



c



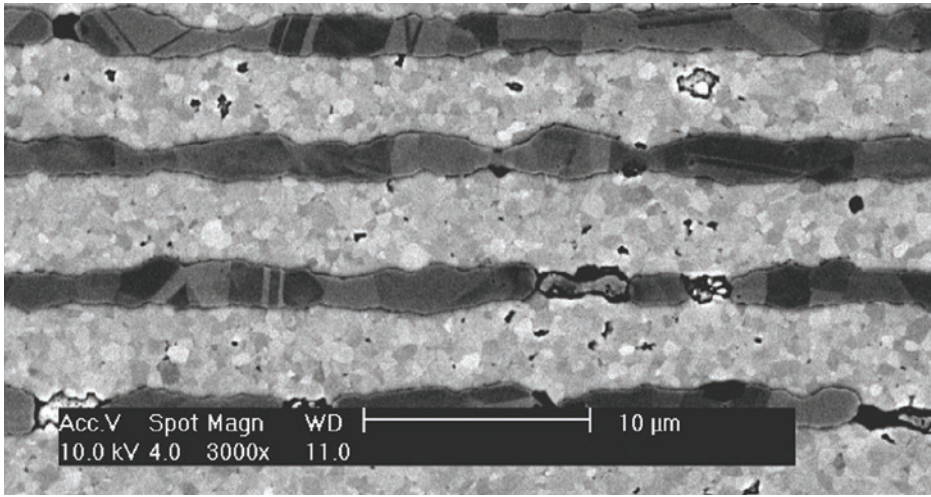


Fig. 3: BSE image of a cross-section of a ceramic multilayer capacitor: This multilayer consists of an alternating sequence of two materials. These different materials can easily be distinguished by element contrast. Within each layer, many crystals can be discerned due to the sensitivity of BSE's for crystal orientation.

Electron Probe X-ray Micro-Analysis (EPMA)

As a result of the interaction between the primary electrons and the local material, characteristic X-rays are emitted by the constituent chemical elements. From the energy or wavelength and intensity distribution of these X-rays the local chemical composition can be derived not only qualitatively, but also quantitatively. An essential feature of EPMA is the localized excitation of a small area of the sample surface with the finely focused electron beam. The information depth can vary from 100 nm to several μm , depending on the accelerating voltage of the primary electrons, the average atomic number of the material under investigation, and the energy of the analyzed X-rays. The electron beam can be scanned across the surface to give a 2D image of the material composition (see figure 2c). The lateral resolution is limited by the interaction volume in the sample. This volume is depicted by the yellow and green

pear-shape in figure 1.

The size of this volume is dependent on the composition of the sample and on the accelerating voltage of the primary beam. In practice, the lateral resolution is limited to a few micrometers. In EPMA the X-rays are separated in two ways: on the basis of energy (EDX) or on the basis of wavelength (WDX).

Energy Dispersive X-ray micro-analysis (EDX)

EDX is a powerful method for fast identification of materials, sometimes even at sub micron scale. Energy dispersive spectrometers have the advantage that all X-ray energies are recorded simultaneously and the spectrum (see figure 4) is displayed directly by means of a multi-channel analyzer. A disadvantage of EDX is the generally poor detection limit in comparison to WDX. This is due to a low peak-to-background ratio in the case of low concentration levels, and/or peak overlap problems related to the

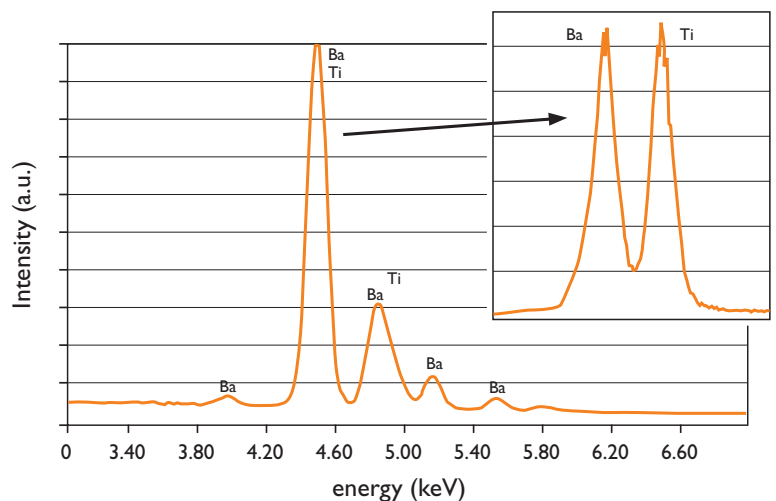
spectral resolution of only 130 eV.

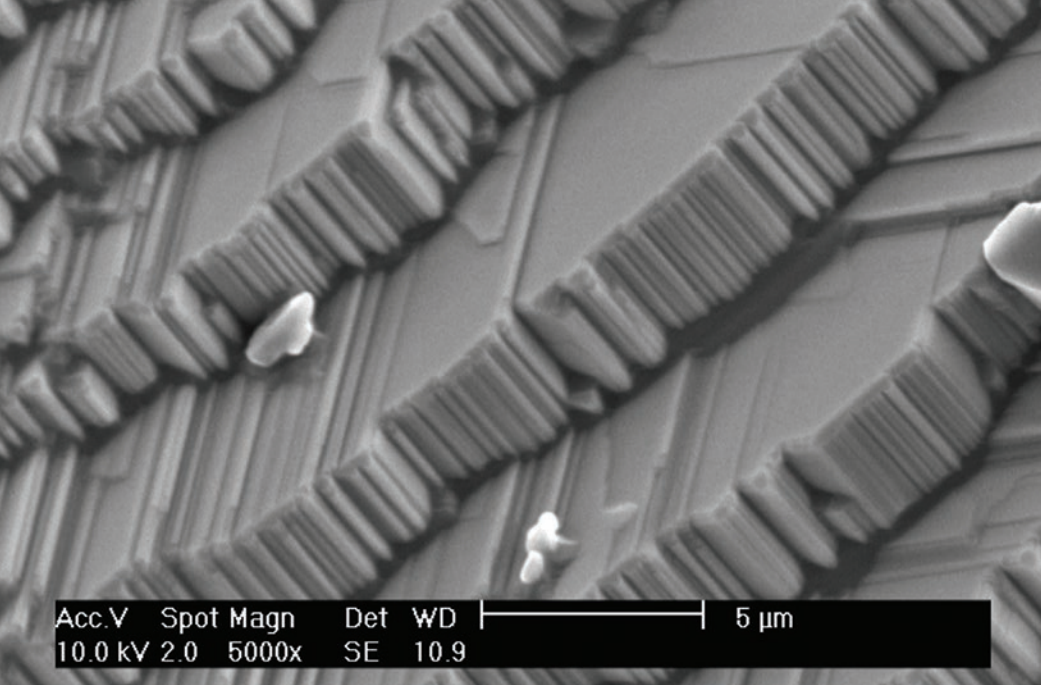
Computer programs facilitate semi-quantitative analysis for several types of specimens, such as metal alloys, glasses and ceramic materials. Furthermore, the layer thickness of thin layers can be calculated (from EDX spectra obtained at the surface of the sample).

Wavelength Dispersive X-ray micro-analysis (WDX)

WDX separates the X-rays by wavelength using a diffracting crystal spectrometer. In order to obtain a WDX spectrum the wavelength has to be scanned by tilting this detector unit. Because of this principle WDX is intrinsically more time-consuming than EDX, but WDX overcomes almost all limitations of EDX. Spectral resolution (≈ 5 eV), and peak-to-background ratio are much better (see figure 4). Detection limits below 0.01 wt.% are not exceptional. WDX can be used for quantitative analysis by using standards for all elements present in

Fig. 4: EDX spectrum of BaTiO_3 . The Ba and Ti peaks at 4.465 keV and 4.508 keV respectively can not be distinguished due to the limited energy resolution of EDX. Inset: part of a WDX spectrum showing these Ba and Ti peaks separately.





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the sample. After subtracting the background, a so-called k-ratio is calculated by dividing the net intensity of the sample by the net intensity of the standard. From the k-ratios of all elements present, and after correction for matrix effects, the composition can be determined. For thin layers on a substrate not only the composition but also the layer thickness can thus be calculated.

Lateral distributions

Elemental X-ray mapping is a very informative method of displaying the distribution of elements in the examined specimen (e.g. cross-sections of layered structures, diffusion couples and welded, brazed or soldered joints, see figure 2c). Line scans can also be recorded, giving a (semi-) quantitative impression of concentration profiles, e.g. in the case of diffusion experiments.

Applications

- morphology of solid materials
- elemental information of solid materials
- lateral distribution of elements
- composition and thickness of thin layers
- identification of contamination
- reverse engineering
- trouble shooting

Characteristics

SEM		
Sample type	Solid materials (metals, ceramic, glass, plastics): bulk, thin films, powders, surface, fracture surface, cross-section	
Sample size	150 x 150 x 30 mm max	
Magnification	10x - 200,000x	
Lateral resolution	1 nm	
Sample preparation	Depending on application: • none • coating with Pt or C in case of non-conducting samples • preparation of cross-section by fracture, mechanical polishing ("Schliff") or Focused Ion Beam (FIB)	
EPMA		
	EDX	WDX
Elemental range	Z ≥ Boron	Z ≥ Beryllium
Resolution	130 eV	5 eV
Information depth	> 100 nm	> 100 nm
Detection limit	0.1 wt.%	0.01 wt.%
Lateral resolution	0.5 – 4 µm	0.5 – 4 µm



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