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H. Seiler
Universität Hohenheim

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INVESTIGATION OF SURFACES AND INTERFACES BY ELECTRON OPTICAL METHODS

H. Seiler
Institut für Physik, Universität Hohenheim
D-7000 Stuttgart 70, West/Germany

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Abstract

The combination of methods for surface analysis with electron microscopes (EM) gives the possibility for surface and interface microanalysis. The paper deals with different methods a) for imaging of surfaces with high lateral resolution: Emission EM (EEM), Scanning EM (SEM), Reflection EM (REM) Transmission EM (TEM) using special preparation methods as replica techniques or cross section specimens b) for crystal structure investigation: Low and High Energy Electron Diffraction and Electron Chanelling Patterns and c) for material analysis using electron spectroscopy either of the emitted or scattered electrons.

The combination of all analytical methods in one instrument for surface microanalysis (SMA) however is difficult. The possibilities and limitations of different SMA instruments are discussed.

KEY WORDS: Surface microanalysis. Scanning-, emission-, reflection-, mirror-, low energy-, transmission electron microscopy. Electron diffraction and channeling. Electron spectroscopy.

Address for correspondence:
Hellmut Seiler
Institut für Physik, Universität Hohenheim
Gartenstr. 30, D-7000 Stuttgart-70, Germany
Phone No.: Germany/0711/459/2150

Introduction

Modern material science, semiconductor technology, environmental research, surface physics, biology, and medicine require more information on the surfaces or on the interfaces of their objects. Object details with the extension of some nm should be visualized, monolayers or even submonolayers on surfaces should be detected and analyzed, steps of atomic height should be measured and the crystal structure should be investigated. Surface microanalysis (SMA) - Fig. 1 - deals with the investigation of a) the TOPOGRAPHY b) the CRYSTAL STRUCTURE and c) the MATERIAL of the objects in small spots. For the investigation of the topography different types of electron microscopes (EM) are available: Transmission EM (TEM), Scanning EM (SEM), Reflection EM (REM), Emission EM (EEM), Mirror EM (MEM). Crystal structure investigation is possible by Low energy electron diffraction (LEED) or High energy electron diffraction (HEED) or electron chanelling pattern (ECP). For material analysis of surfaces often electron spectroscopic methods are used as Auger Electron Spectroscopy (AES), Secondary Electron Spectroscopy (SES), Plasma Loss Spectroscopy (PLS). Ionization Loss Spectroscopy (ILS), Electron Energy Loss Spectroscopy (EELS) either in transmission with high electron energies or in reflection with low energy electrons.

For many years the development of high resolution electron microscopes (EM) for imaging of thin foils (TEM) and of EM for the imaging of surfaces as REM, EEM, SEM and MEM on the one hand and the development of techniques for surface analysis as LEED and AES on the other hand were quite separate. Only in recent years, since ultra-high vacuum (UHV) EM are available and since the Auger spectrometers are equipped with focussing electron lenses surface microanalysis (SMA) is possible with high lateral resolution and high surface sensitivity. For SMA the EM should be combined with

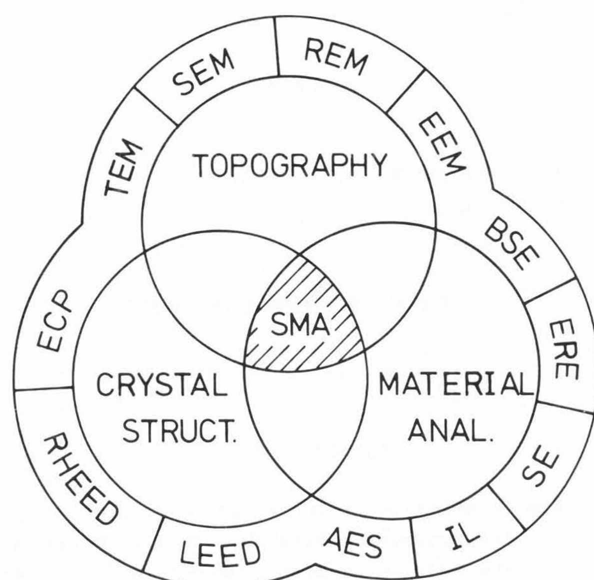

Fig. 1. Combination of different methods for surface microanalysis: SMA

as many methods for surface analysis as possible. Wittry (1980), Venables (1981, 1982), Hofmann (1986), Hauser and Seiler (1987), Seiler (1988). This is however difficult without moving the object, due to the limited available space in front of the object.

A survey is given on the most frequently used methods for i) imaging of surfaces and measuring of surface details with electron optical methods. ii) for the investigation of the crystal structure on surfaces. iii) for material analysis of surfaces by electron spectroscopy. For the combination of different analytical methods with EMs several instruments were built. The possibilities and limitations of different instruments for SMA are discussed. Newbury (1979), Wittry (1980). In the proceedings of the fifth Pfefferkorn Conference "Physical aspects of microscopic characterization of material", some of the instruments are described in detail. Kirschner et al. (1987). The paper deals not with the well known method for elemental analysis by detecting characteristic X-rays in an electron microprobe (EMP-EDX or EMP-WDX) Hren et al. (1979), Heinrich (1981), Williams (1984), Reimer (1985) and also not with X-ray photoelectron spectroscopy (XPS or electron spectroscopy for chemical analysis, ESCA). A survey on XPS with high lateral resolution was given recently by Chaney (1987).

A serious problem in SMA is the radiation damage due to the high primary electron current density necessary for a sufficient S/N-ratio. Fontaine et al.(1979), Bauer and Seiler (1980), Le Gressus et al. (1981), Pantano and Madey (1981), Casaux (1985). Interfaces can be studied either in transmission of cross section specimens with high spatial resolution or by depth profiling by ion sputtering combined with surface analytical methods.

Survey on electron optical methods for imaging of surfaces (Fig. 2)

Emission Electron Microscope: (EEM). Möllenstedt and Lenz (1963), Seiler (1968), Schwarzer (1981), Kampik et al. (1983)

An extended area of the surface is irradiated with UV-light, primary electrons or ions and the released photoelectrons, secondary electrons, elastically reflected electrons or ion induced electrons are accelerated and focussed by a cathode lens. Imaging of the surface is also possible by thermionic emitted electrons. Using high vacuum the images were influenced by adsorption or contamination layers which can destroy the material contrast. The EEM with ion released electrons allows the imaging of material or crystallographic contrast even in poor vacuum if the sputtering rate is greater than the contamination rate. Fig. 3 shows a perlite steel surface imaged in an EEM with ion released electrons in high vacuum showing a good material contrast. The change in surface topography during sputtering can be observed.

In recent years by Bethge et al. (1985), Nadakavukaren and Griffith (1985) and Bauer (1985) UHV-EEM were built with a resolution of 10-60 nm. Monoatomic layers and submonolayers may alter the work function, change the electron emission and can so be visualized. A serious limitation of the EEM is the fact that only flat surfaces can be imaged with high resolution.

Scanning electron microscope: (SEM). Reimer (1985)

In the SEM the object is scanned by a focussed electron beam and the signal of each object point modulates the intensity of the image point. Normally the energy of the primary electron beams is 10 - 30 keV. In order to minimize charging of insulator or semiconductor surfaces as well as to enhance the topographic contrast and the material contrast nowadays Low Voltage SEMs (LVSEM) are used. Pawley (1984), Hefter (1987). A certain problem in LVSEM is the small brightness of the electron gun, which may be increased by using LaB_6 or field emission guns. Another problem is the influence of the electric field of the SE-detector on the low energy primary beam. In order to minimize this influence, Zach and Rose (1986) developed a new detector of electric and magnetic quadrupoles, Schmid and Brunner (1986). With field

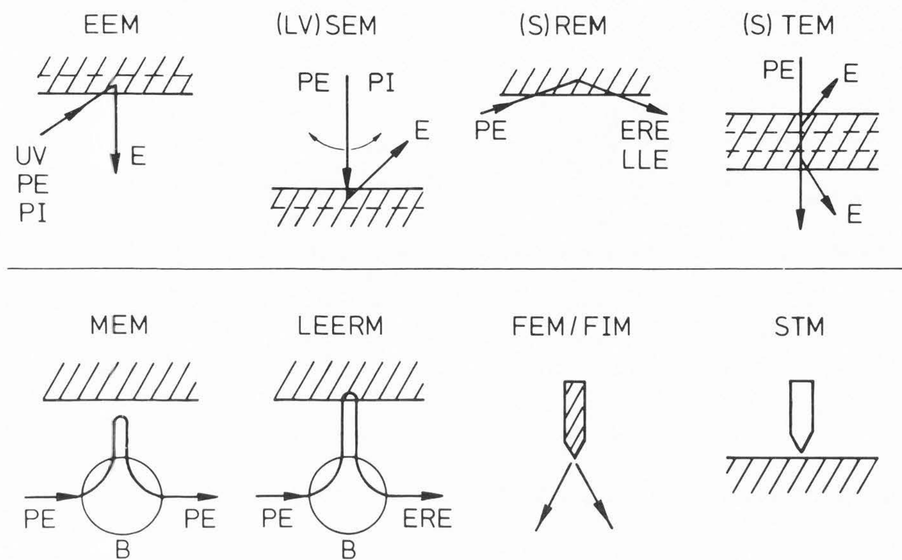

Fig. 2. Schematic illustrations of different electron microscopes for the investigation of surfaces.

emission guns a resolution at 30 keV of 2 nm and at 200 eV of 100 nm is possible. Ichinokawa (1986), Kirschner et al. (1986). In FESEM, a SEM with a field emission electron gun, and if the samples are inside of the pole piece of a highly excited objective lens a resolution of better than 1 nm seems possible, Nagatani et al. (1987).

Contrary to the EEM rough surfaces also can be investigated with the SEM. The main advantage of the SEM is the possibility to measure the signal of each object point and to modulate the image brightness by a combination of different signals, i.e. signals of different detectors.

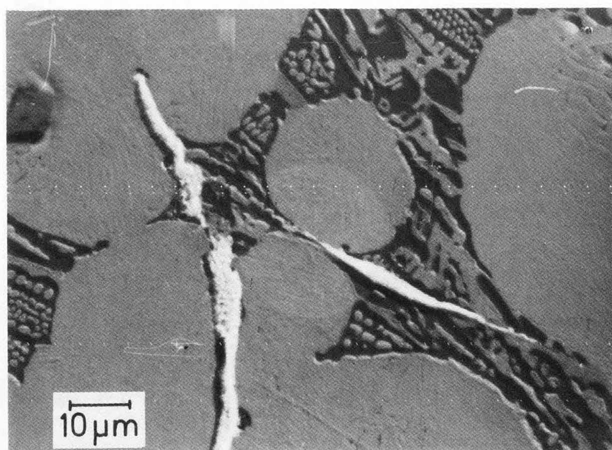

Fig. 3. Perlite steel surface imaged in an EEM with ion released electrons showing material contrast.

<u>(Scanning) Reflection electron microscope: (S)(REM)</u>

Since the beginning of EM it was tried to image the surface of bulk material under grazing incidence angle with reflected electrons. The first experiments with a REM were done by Ruska and Müller (1940). The REM (100 keV) has some limitations due to the severe foreshortening of the image (factor 20 - 50), the high sensitivity to surface roughness and the difficulty of correlating the images with microanalytical signals from small specimen regions. For the last problem a combination of REM with SEM is useful. Using primary electrons with energies of 2 - 20 keV the diffraction angles become greater than 5° - 16° with less foreshortening and less sensitivity to surface roughness . Nowadays a resolution of better than 100 nm has been shown for lower electron energies, for higher electron energies the resolution may be 1nm - and it is possible to recognize steps of atomic height - Yagi (1982), Hsu (1983), Cowley and Peng (1985), Cowley (1986).

In the SREM not only the elastically reflected electrons can be used for imaging of surfaces but also the electrons which have lost some 100 eV: Low loss electron (LLE). Treacy and Bellessa (1983). Wells (1986) showed pictures with high re-

solution of uncoated photoresist imaged with LLE under a glancing angle of incidence of 30° with an energy loss of about 300 eV. Broers et al. (1975) obtained a resolution of 2 nm.

(Scanning) Transmission electron microscope: (S)(TEM)

Normal TEM allows one to investigate thin specimens with a thickness less than 1 μm. With very thin objects it is possible to get atomic resolution. Takayanagi (1986). Very interesting is the profile imaging method. Smith (1986), Smith et al. (1987). Using diffraction contrast monoatomic layers on thin crystalline objects can be recognized. Lehmpfuhl and Warble (1986), Klaua and Bethge (1985), Yagi (1986). With a TEM however we can also get surface information of bulk material by backthinning. Goodhew (1972). By replica techniques sometimes combined with a gold decoration method it is possible to recognize steps of atomic height. Bethge and Keller (1965). Of course it is not possible by replica techniques to get information on the material of the surface.

Interfaces can be investigated in the TEM by cross-section specimens thinned by chemical etching and ion milling. Cross-sectional TEM allows the visualization of topography, microstructure and lattice defects with high contrast and a lateral resolution of better than 1 nm. This is especially important for the investigation of thin films in semiconductor device fabrication. Rehme and Oppolzer (1985), v. Criegern et al. (1985), Chew and Cullis (1987).

One of the advantages of a STEM is its ability to record simultaneously different signals: The signal of the elastically scattered electrons - sometimes registrated with an annular detector giving high resolution images such as atom visualization, the signal of the inelastically scattered electrons, giving a signal on the elemental composition by EELS, the signal of BSE giving information on the material and the signal of SE giving information on the topography of the surface of the object. Allen (1985), Reichelt and Engel (1986).

Mirror electron microscope: (MEM) Mayer (1957), Schwartze (1967).

In the MEM the object is of the same potential or slightly negative with respect to the potential of the electron gun. The primary electrons are deflected by a magnetic field B towards the object, they are reflected by the equipotential surface in front of the specimen and again deflected by the magnetic field B. It is possible to image the topography of the equipotential surfaces. In the MEM the object is not influenced by the electron beam. The measurement of height profiles as well as the measurement of potential differ-

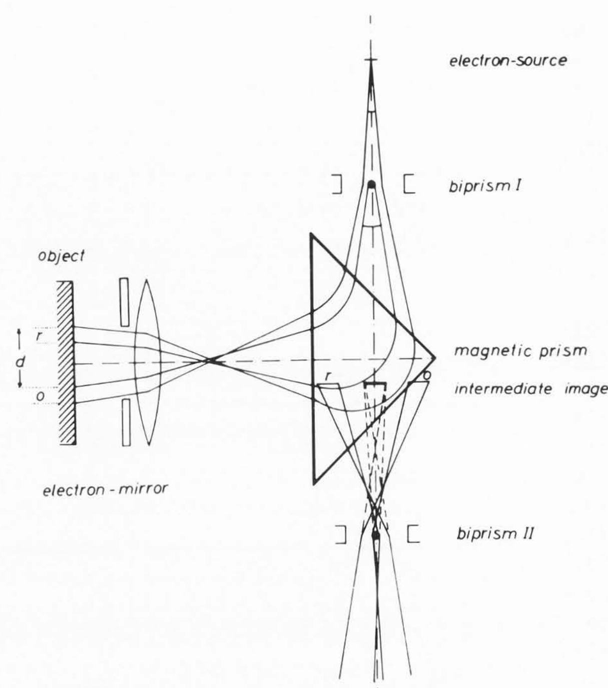

Fig. 4. Setup of the mirror interference electron microscope according to Lichte. (1983).

ences on surfaces is possible with the mirror electron interference microscope (MEJM) built by Lichte (1980, 1983) - Fig. 4 - . A plane electron wave is reflected at the object surface which is nearly at the same electrostatic potential as the electron gun and suffers local phase modulation due to local height variations of the surface. If two such modulated wave fronts reflected at different parts of the surface are superimposed by means of an electron biprism to form interference fringes, the local fringe shift is given by the local height variations which cause phase modulations. Object structures larger than 1 μm with a height of about 0.1 nm can be seen.

Low energy electron reflection microscope: (LEERM or LEEM)

The LEERM developed by Bauer and Telieps (1987) is in its principal setup similar to the MEM. The primary electrons can reach the object with low energy. Some of them are reflected elastically and a cathode lens forms an image of the surface. This instrument will be discussed later in detail.

Field electron microscope: (FEM) and Field ion microscope: (FIM)

In the FEM a small metal tip with a radius r 1 μm is at a negative potential U of several keV. By an electric field strength of $E \geq 10^9$ V/m

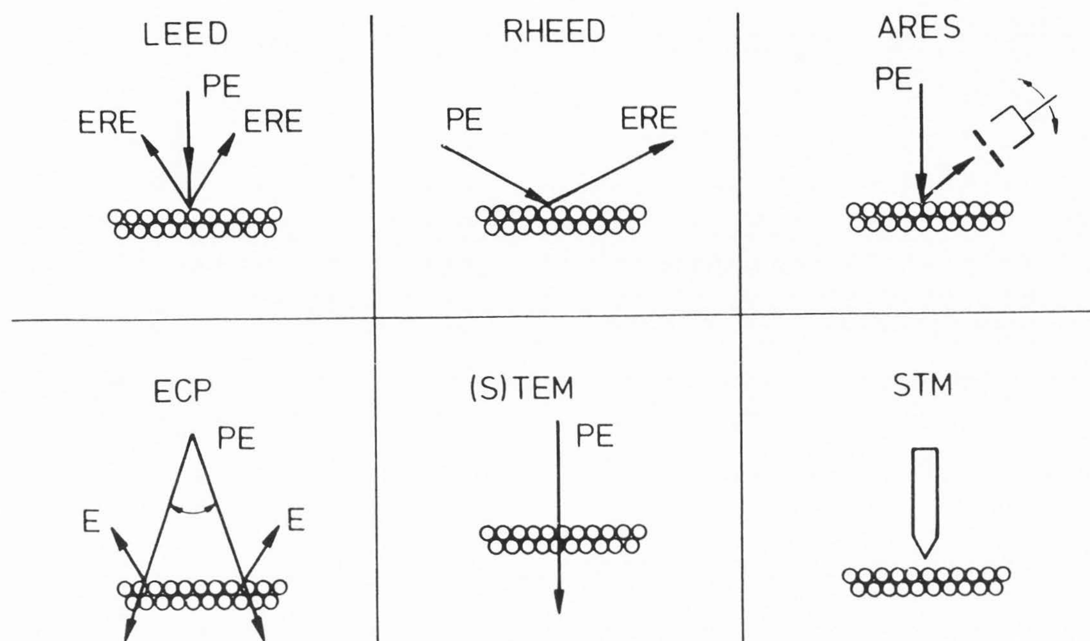

Fig. 5. Schematic illustrations of the different methods for the investigation of the crystal structure by electrons.

field emission of electrons occurs and the surface of the tip is imaged on a screen. Differences in work function between different crystallographic planes of the single crystal tip can be seen. Good and Müller (1956). In the FIM a small cooled single crystal tip is in an environment of He gas. The He atoms are polarized, attracted to the tip and ionized near the surface of the tip and accelerated in the electric field towards a screen. This FIM allows the imaging of single atoms and of the crystal structure of the tip. With a special technique "atomprobe" it is possible to release atoms from the surface and to analyze them in a time of flight mass spectrometer. Müller (1960), Kellogg (1987).

Scanning tunneling microscope:(STM) Celotta (1988)

The STM was developed by Binnig et al. (1983) the Nobel prize winners 1986. A fine tip is scanned over the surface of the specimen. The tunneling current which depends strongly on the distance is held constant by variation of the distance from the tip to the surface. This change in distance is registrated on a monitor. Single atoms of the surface can be recognized and it is possible to investigate not only metal and semiconductor surfaces but also the surfaces of biological objects.

Survey on methods for the investigation of crystal structure by electrons (Fig. 5)

Low energy electron diffraction: (LEED)

In principle this method was used already by Davisson and Germer (1927) and gave the first experimental proof for the wave nature of the electron.

The single crystal is bombarded with electrons of about 50 - 500 eV, corresponding to a wavelength of the electrons similar to the distance of the atoms. The diffraction pattern of the elastically reflected electrons ERE allows one to determine the crystal structure of the uppermost monolayer.

Reflection high energy electron diffraction: (RHEED) (also HEED or SHEED: Scanning high energy electron diffraction)

Under grazing incidence of the primary electron beam with energies > 10 keV it is possible to determine the crystal structure of the surface.

Angle resolved electron spectroscopy: (ARES)

By variation of the acceptance angle of the spectrometer it is possible to record the signal of the emitted electrons which in some cases gives information on crystal structure. Zimmer et al. (1984).

Electron channeling pattern: (ECP)

By variation of the direction of the incident PE relative to crystallographic directions the yield of emitted SE and BSE changes. So by regis-

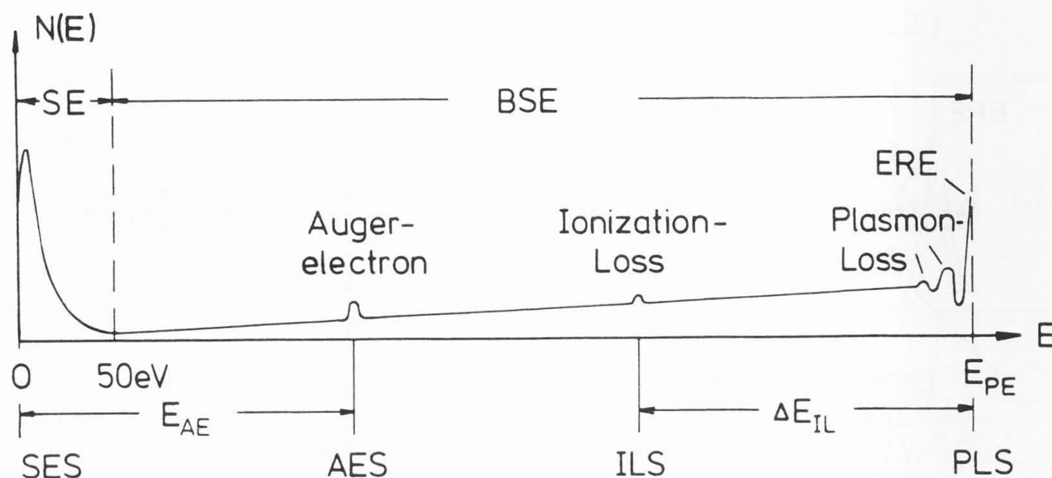

Fig. 6. Schematic energy distribution of electrons released at a surface by PE impact with an energy E_{PE} 2 keV.

tration of the ECP it is possible in the SEM to determine the crystal structure near the surface of the object. The contrast of the ECP, depends very sensitively on thin amorphous layers or contamination at the surface. The Bloch waves are strongly attenuated with increasing depth and so the ECP are formed in a layer with a thickness of some 10 nm at the surface. Seiler (1976), Kuhnle (1974). ECP can also be observed with PE energies less than 2 keV, however the Bragg angles become rather great. Seiler et al. (1975).

Atomic imaging

With atomic resolution with the STM, TEM or STEM it is possible to see the crystal structure directly. Smith (1986).

Survey on material analysis of surfaces by electron spectroscopy. Ibach (1977), Seiler (1985).

The measurement of the energy distribution of the scattered primary electrons or of the emitted electrons yields much information on the surface or the interface of the specimens. Fig. 6 shows schematically the energy distribution of electrons released by primary electrons (PE) with energies 100 eV < E_{PE} < 3000 eV.

Secondary electrons: (SE) Seiler (1983).

The SE-yield integrating over all energies of the emitted low energy electrons with energies E_{SE} < 50 eV is mostly used in the SEM for imaging of surfaces. The SE-yield however is not well suited for analytical purposes, the energy distribution of the SE contains more information on surface material.

Backscattered electrons: (BSE) Niedrig (1982).

The BSE-coefficient, integrated over all energies of the BSE with energies 50 eV < E_{BSE} < E_{PE} allows material analysis of plane surfaces in the SEM, however the BSE stem from depths up to half the range of the PE, so this signal is not very surface sensitive.

Auger electrons: (AE)

The AE are mostly used for material analysis of surfaces. By sputtering also interfaces can be investigated by AES. Using sensitivity factors a quantitative analysis of surface material is possible. Seah (1983).

Elastically reflected electrons: (ERE) Jablonski (1985).

The ERE are not only useful for LEED investigations but also for material analysis of the surface. It was shown by Gergely (1981) and by Schmid et al. (1983) that a monotone dependency exists between the number of elastically reflected electrons per primary electron and the atomic number of the target. So a material analysis of the surface is possible by measuring the ERE, similar as with BSE. In contrary to the BSE's however the ERE are reflected at the uppermost monolayer of the surface.

Ionization loss: (IL)

BSE which have ionized a surface atom in inner shells without other energy losses have defined energies with respect to E_{PE}. In reflection this is a rather small signal even compared with AES. In transmission the electron energy loss spectroscopy (EELS), investigating the absorption edges of inelastically scattered electrons is a very valuable method for material analyses of thin foils and interfaces. The EELS signal especially for low atomic number elements is some order of magnitude greater than the X-ray signal.

Plasmon loss: (PL)

BSE which have excited surface or volume

	SE	BSE	AE	ERE	-PL	-IL
Yield	0.3	0.3	10^{-4}	0.03	0.01	$< 10^{-4}$
Material analysis	no	(yes)	yes	(yes)	((yes))	yes
Escape Depth	nm	0.5R	nm	nm	nm	nm
Information Depth	0.5R	0.5R	<0.5R	nm	nm	nm
Surface sensitive	yes	no	yes	yes	yes	yes

Table 1 Survey on different signals in electron spectroscopy for SMA

plasmon oscillations without other energy losses have defined energies with respect to E_{PE}. Bohm and Pines, (1953). This PL spectroscopy (PLS) or elastic peak electron spectroscopy deals with the low energy vicinity of the ERE. Gergely (1986). The excitation of plasmon oscillations can also be observed in transmission of thin foils in EELS. Raether (1980). The excitation of surface plasmon losses strongly depend on thin layers on the surface.

Electrons which have undergone phonon losses provide information on adsorbed molecules. The energy losses in the range of some 100 meV cannot be resolved unless the primary beam is premonochromatized and the resolving power of the spectrometer is high enough: high resolution electron energy loss spectroscopy (HREELS) Ibach (1977), Thiry et al. (1987), Koel (1985). All peaks in the energy distribution of the emitted electrons can be used for characterization of the surface.

The energy distribution of the scattered or emitted electrons can be measured by different spectrometers. Kirschner (1983), Ibach (1977). In surface analysis mostly retarding field analyzer (RFA), cylindrical mirror analyzer (CMA) or hemispherical analyzer (HSA) are used. The RFA - also used in LEED-observations - allows crystal structure analysis. However the S/N-ratio of the RFA in material analysis with AES is rather low compared with CMA and HSA due to the high background of BSE.

Table 1 summarizes different signals which we get by electron spectroscopy. The yield of the SE and of the BSE is fairly great compared with the yield of the AE. So for imaging of surfaces mostly the SE or BSE are used. SE-yield and BSE-coefficient both depend on the material, the topography and the crystal structure of the surface. By imaging of a surface we get material-, topography and crystallographic contrast. With BSE on plane surfaces material discrimination is possible.

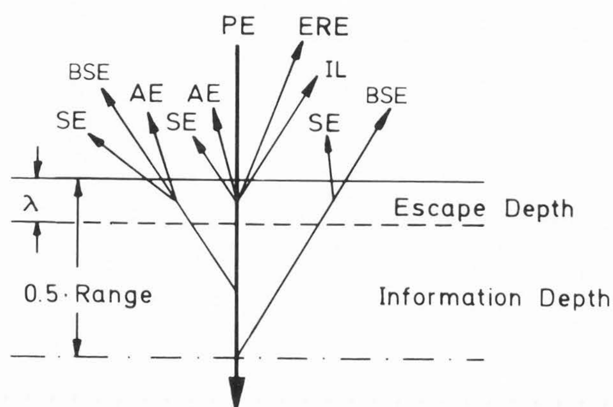

Fig. 7. Escape depth and information depth of different signals in electron spectroscopy and imaging.

For surface analysis the difference between escape depth and information depth is important (see Fig. 7) Seiler (1976). The escape depth of the SE and of the AE from metals is about 2 nm, the escape depth of the BSE is about half the range of the PE and so at E_{PE} 20 keV about 0.5 μm. The information depth is the distance normal to the surface contributing to the signal. The BSE also release SE and AE and so object details beneath a layer greater than the escape depth of the SE and AE influences the SE- and AE-yield. This effect can be eliminated by signal mixing if the BSE are measured with a separate detector. The escape depth of the ERE is smaller than that of AE of the same energy and contrary to SE and AE, the signal height is not influenced by object details far beyond the escape depth of SE and AE.

Surface microanalysis (SMA).

The combination of imaging with high resolution, crystal structure analysis and material analysis with high sensitivity for universal SMA is difficult: High resolution needs an objective lens with short focal length and so a short working distance. (The coefficient of spherical aberration is of the same order of magnitude as the focal length). So there is not enough space for a spectrometer with a large acceptance angle. The information contained in LEED or RHEED pattern is an average over rather large areas and so local variations in topography or adsorption layers with diameters ≤ 0.1 µm cannot be seen. Material analysis with high sensitivity needs a high primary beam current (in order to get a high S/N ratio) which is difficult to get in a probe with small diameter.

SMA using ERE

The difficulties in SMA can be explained by a simple setup - Fig. 8 - with a rather poor resolution which allows crystal structure analysis by LEED, material analysis by AES and imaging of the surface by ERE. Bauer et al. (1982). Normally in LEED instruments the impact point of the PE is unknown. In the case of homogeneous objects this is not a serious disadvantage, however for inhomogeneous objects the exact object point under investigation should be known. Moreover for AES mostly energies of the PE of several keV are used whereas for LEED investigation energies of several 100 eV are used. So the object area analyzed by AES may be different from the point analyzed by LEED because a change in PE energies may result in a deflection of the primary beam. Scanning of the primary beam in order to image the surface as in a SEM can normally be achieved by deflection plates which would however disturb the LEED pattern. So in this case the primary beam is deflected by means of two perpendicular pairs of Helmholtz coils. A very surface sensitive imaging of the surface is possible by using the ERE as the signal of each object point to modulate the intensity of the image point. So imaging, crystal structure and material analysis is possible however with a low resolution.

In order to image surfaces with high resolution and for crystal structure analysis combinations of an EEM with LEED was built by Bauer (1985), Telieps and Bauer (1985), Telieps (1987) Telieps et al. (1987) and Delong and Kolarik (1985). Fig. 9 shows schematically the LEERM developed by Bauer and Telieps (1987). As shown in Fig. 6 the ERE reflected on clean surfaces in UHV, have a very small energy distribution. So the use of electron lenses is possible. The primary beam from a field emission gun is separated from the reflected beam by a magnetic deflection field.

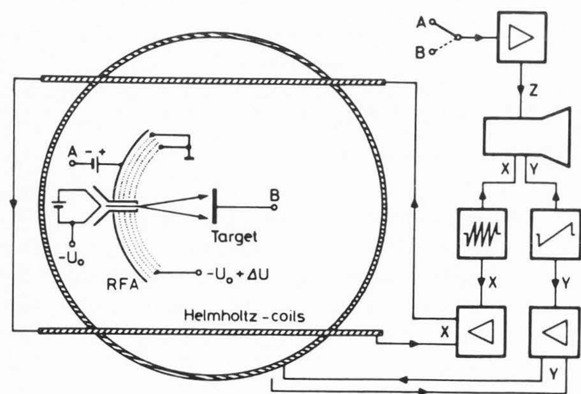

Fig. 8. Setup of an instrument for imaging of the surface by ERE, material analysis by AES and crystal structure analysis by LEED according to Bauer et al. (1982).

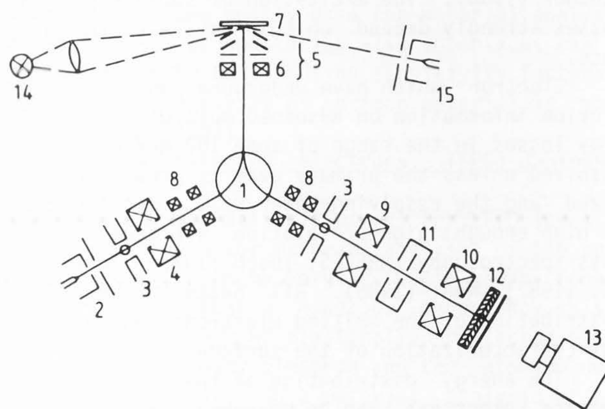

Fig. 9. Setup of the LEERM or LEEM according to Bauer and Telieps (1987).

1: magnetic field
2: field emission electron gun
3: quadrupole
4, 9, 10: electron lenses
5: cathode lens
6: stigmator
7: object
8: alignment coils
11: filter lens
12: channel plate
13: camera
14: UV-lamp
15: electron gun

Within the cathode lens the primary electrons are decelerated and reach as a parallel beam the object with an energy of some 100 eV. The elastically reflected electrons from a single crystal form the LEED pattern in the back focal plane of the cathode lens. The LEED pattern can be magnified by additional lenses.

For imaging the electrons of the (00) pattern are selected by a small aperture. This aperture minimizes the aberrations of the cathode lens. If necessary the inelastically BSE and SE can be rejected by an electrostatic filter lens. The LEERM has a spatial resolution of about 20 nm and monoatomic steps on single crystal faces can be seen and measured by phase contrast. The electron wave reflected from the one side of the step has a path difference of /2 relative to the wave from the other side. So Fresnel diffraction can be observed. By variation of contrast by changing the energy of the electrons the height of the steps can be measured. Another contrast arises by the difference in the coefficient of the ERE. This microscope also gives the possibility to image the surface by photo- or thermionic electron emission.

A combination of a LV-SEM with a field emission gun and a RFA on one side was built by Kirschner et al. (1986), Ichinokawa (1986). This instrument allows one to investigate crystal structures with a diameter of 60 nm at a primary energy of 250 eV by LEED and material analysis by AES and imaging with ERE.

Surface microanalysis using PLS

The energy loss peaks in the vicinity of the ERE can also be used for imaging of surfaces. So different grains on clean polycrystalline Al specimens can be seen Ichinokawa et al. (1981), Le Gressus (1982, 1984) and hydride phases can be detected Bevolo (1985).

Surface microanalysis using AES. Hofmann (1987)

For material analysis of surfaces mostly AE are used, either using a UHV-SEM with a CMA or an HSA attached on one side or the primary electron gun with focussing lenses and scanning devices is integrated within the CMA. With a field emission gun a resolution of better than 50 nm can be reached. However crystal structure analysis is difficult using a CMA or HSA. For LEED investigation there is no space for an RFA, for HEED a tilting of the object is necessary and for the registration of ECP rather large tilting angles of the primary beams of several degrees depending on the energy of the primaries is necessary. This is difficult to realize. Seiler et al. (1975). In the normal ECP-mode a rather extended area of the surface is necessary to get large tilting angles. ECP's can however also be observed by an angular scanned beam on a fine point on the sample surface, a method which has been used in normal SEM earlier. Nowadays this is also possible in some newly developed Auger microprobes. Sakai et al. (1988).

AES is not only possible on bulk material but also on thin films. Widmann and Seiler (1977). An interesting setup for AES in a TEM was proposed by Kruit (1986).

Surface microanalysis using SE

SE-yield. Seiler (1982, 1983) The SE-yield δ, integrated over all energies of the emitted slow electrons with energies $E \leq 50$ eV is mostly used in the SEM's to modulate the image brightness. δ depends on topography, crystal structure and material of the surface. So the topography of the surface can be measured and the crystal structure can be determined. Different materials at the surface often can be visualized, a material analysis however is not possible because there is no monotone relation between δ and the atomic number. The escape depth of the SE is about 2 nm in metals and 10 nm in insulators. So δ is influenced by very thin adsorption or contamination layers.

Futamoto et al. (1985) and Venables (1986) have shown, that visualization of submonolayers in a UHV-SEM is possible by applying a negative bias (-500 V) to the sample: biassed SE imaging. The sensitivity is in some cases better than 0.1 monolayer.

The SE are not only released by the incident PE but also by the BSE. The signal of a normal Everhart-Thornley detector in the SEM consists of different types of electrons: Seiler (1983), Reimer (1985, 1986).

SE I: SE produced only by PE.
SE II: SE produced by BSE at the surface of the specimen.
SE III: SE produced by BSE at the wall of the SEM.
BSE IV: BSE emitted in the direction of the detector.

The SE I signal is the desired signal both for high resolution and for high surface sensitivity.

The BSE are backscattered within the object and are not as surface sensitive as the S.E. Reducing the signal of the SE produced by BSE gives more information on the surface itself. So the signal of the SE minus a signal proportional to the number of the BSE gives more information of the surface. Volbert (1982). Another possibility to reduce the SE III and BSE IV is the through-the lens signal detection. Koike et al. (1971), Spiers (1987). This system attracts axial SE through the magnetic field of the objective lens to a detector above the lens. This technique collects SE I and SE II and eliminates unwanted SE III and BSE IV, providing true surface information.

<u>SE-Spectroscopy. (Seiler (1985)</u> SES is useful in the SEM for material characterization, work function measurement and for observation of voltage contrast.

The energy distribution (ED) of the SE of different materials is quite different and changes by adsorption of thin layers or especially by oxidation. So the FWHM of the ED of the SE increases from 5.5 eV for Al_2O_3 to 10.5 eV for Al; for Cu we get a FWHM of 18 eV and for Pt 27.5 eV.

The onset of the ED of the SE depends on the work function difference between sample and analyzer. Jansen et al. (1980), Akhter and Venables (1981), Argile et al. (1984), Bauer and Seiler (1986). Fig. 10 shows the energy diagram for the determination of the energy of the SE. The samples

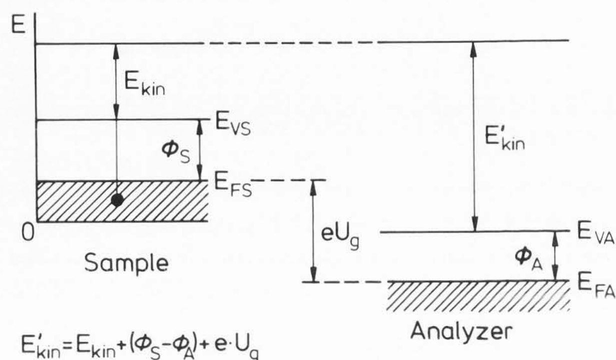

Fig. 10. Energy diagram for the determination of the energy of the SE, E_V: Vacuum level, E_F: Fermi level.

are biased to a negative potential U_g of about -10 V. This potential shifts the whole SE-spectrum in an energy range being more suitable for the CMA. A SE leaving the sample surface (work function Φ_S) with the energy E_{kin} is measured in the energy analyzer (work function Φ_A) with the energy $E'_{kin} = E_{kin} + (\Phi_S - \Phi_A) + eU_g$ and for the onset $E_{kin} = 0$ we get $E'_{kin} = \Phi_S - \Phi_A + eU_g$. A negative potential or a greater work function Φ_S of the sample shifts the onset of the SE-ED to higher energies. Fig. 11 shows the change in the shape of the ED and the shift of the onset of the ED during sputtering through a thin layer of Ag on Pt. Bauer and Seiler (1988).

In a SEM with an energy spectrometer the variation of the work function can be displayed directly.Combined with AES this allows one to correlate work function maps with elemental distribution on the surface. Bachmann et al.(1987,1988).

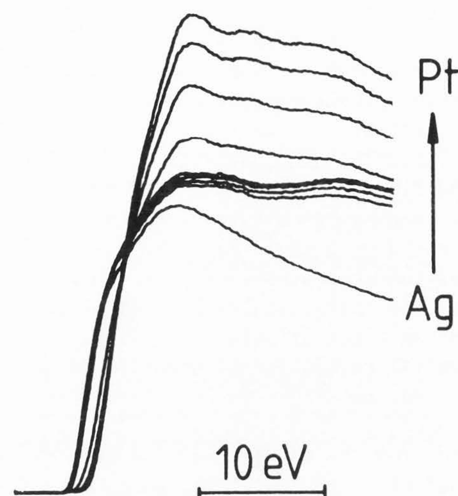

Fig. 11. Change of the energy distribution of the SE during sputtering through a thin layer of Ag on Pt according to Bauer and Seiler (1988).

<u>SE - with polarization analysis (SEMPA)</u> SE emitted from a ferromagnet are spin polarized. This spin polarization can be detected with several detectors. The LEED- and the Mott-detector are mostly used. In combination with a UHV-SEM this gives the possibility to measure the magnitude and the direction of magnetization with high resolution (Kirschner 1987, 1988), Koike et al. (1987), Hembree et al. (1987).

Microanalysis of interfaces

For the investigation of interfaces with high lateral and depth resolution different techniques are used:

a) Depth profiling by sputtering in combination with surface sensitive analytical methods as AES, SSIMS (Static secondary ion mass spectroscopy) ISS (Ion surface scattering), SNMS (Surface neutral mass spectroscopy). Hofmann (1985), Oechsner (1987).

b) Investigation of cross - sections in TEM. Oppolzer and Rehme (1985), Cerva et al. (1987). The specimens for cross sectional TEM were prepared using standard procedures: two pieces were glued together front to front and slices were cut from this structure by a dicing saw. Chemical polishing and low energy argon ion milling provide large transparent regions for TEM observation. Willer and Oppolzer (1987), Chew and Cullis (1987).

Fig. 12 shows an example from Criegern et al. (1985) of interfaces of Ta-Si-layers with a dis-

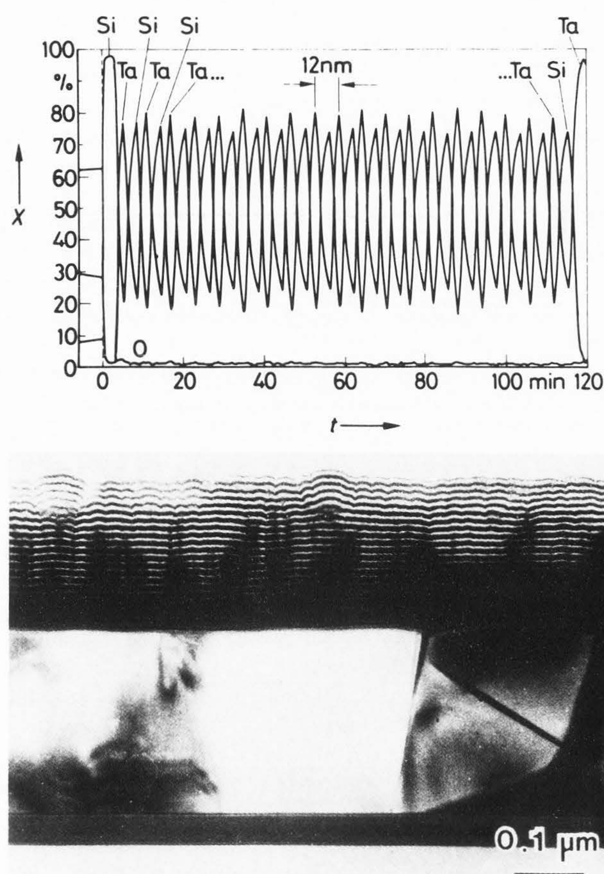

Fig. 12. Investigation of the interfaces of a Si-specimen with layers of Ta and Si on the surface. Top: by depth profiling, bottom: by cross sectional TEM, according to v. Criegern et al. (1985).

tance of the Ta-layers of 120 nm on a Si-sample by AES-sputter profiling and cross-sectional TEM. The cross-sections can be investigated in an analytical TEM. The crystal structure of selected areas can be seen by electron diffraction and the elemental composition can be determined by X-rays and EELS. Thiry et al. (1987).

Conclusions

A survey is given on different electron optical methods for imaging of surfaces. Commercial instruments are the SEM, LVSEM, FESEM, in lens FESEM, TEM, STEM, FIM, FIM/atomprobe and recently the STM. For the investigation of surfaces UHV versions are available. EEMs with thermionic, UV and ion released electrons commercially have been built for several years. (Ion-EMs with detection of the released secondary ions are available as well as a Scanning ion microscope using the released SE or secondary ions for imaging.) The REM, MEM, MEJM and LEEM are special instruments in several laboratories and to my knowledge not commercially available. Of these instruments only the LEEM works in UHV.

For crystal structure analysis HEED and ECP registration is possible in normal vacuum, LEED needs UHV. Nowadays not only HEED but also ECP investigations are also possible in commercial UHV apparatus. For material analysis using electron spectroscopy different electron spectrometers in UHV are available.

The paper surveys the combination of different EMs with analytical techniques. Very useful is sometimes a combination of different EMs. For the investigation of the fundamentals of SEM - especially of the spatial distribution of the SE1 and SE2 - a combination of SEM and EEM was used by Hasselbach (1988). The emitter tip of a FEM has been investigated in FE-SEM. Kuroda et al. (1987). As to my knowledge up to now a combination of STM with its ultra high resolution and a high resolution EM in order to investigate the same surface with both imaging methods has not yet been built.

For a long time the ultimate resolution limit of the SEM using SE was theoretically considered to be about 1 nm due to the escape depth of the SE. Crewe (1985). Nowadays a resolution better than 1 nm was reported by several authors: Kuroda et al. (1987), Nagatani et al. (1987), Liu and Cowley (1987, 1988) and in lens-FESEMs with a resolution of better than 1 nm are now commercially available.

In a normal SEM the energy distribution and the angle distribution of the emitted SE is not very important, because the extraction field of the detector of about 10^4 V/m is strong enough to collect all emitted SE. However if small object details are investigated as in the testing of microelectronic circuits (Schönecker et al. (1986)) or in high resolution SEM, local field effects between adjacent object details cannot be neglected. Fig. 13 shows the electric field strength between object details at different potentials depending on their distance. The electric field of the Everhart-Thornley detector is about 10^4 V/m. If we have object details in a distance of 1 µm and a potential difference of 1 Volt the electric field strength is about 10^6 V/m, far greater than the field of the detector and so an influence on the detector signal is to be expected. Even small potential differences, caused by work function differences of adjacent object details may have an influence. This has to be considered in ultra high resolution SEMs, in the investigation of integrated circuits with small dimensions and perhaps this can explain the high contrast in biassed SE imaging too.

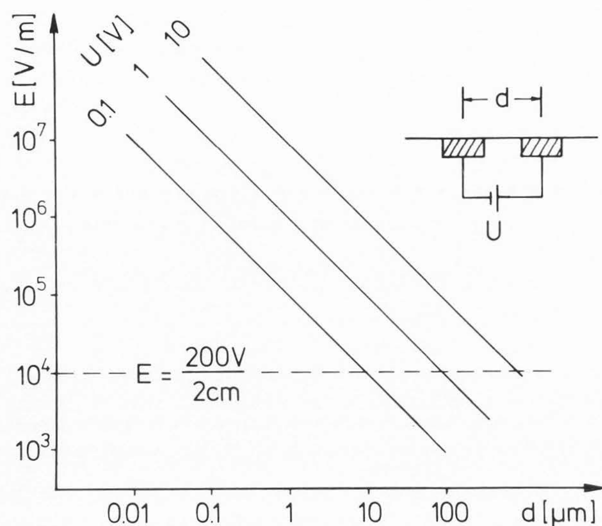

Fig. 13. Electric field strength between object details in a distance *d* with a potential difference *U* compared with the extraction field strength of the detector in a SEM of about 10^4 V/m.

Acknowledgements

The author thanks Dipl. Phys. H.E. Bauer for many helpful discussions, Prof. Dr. E. Bauer for the picture of the setup of the LEERM, Dr. H. Lichte for the picture of the setup of the MEJM and Dr. v. Criegern, Dr. Hillmer and Dr. Rehme for the picture of the investigation of the Si specimen with AES and TEM, Mrs. Neifer for the careful drawing of the figures and Mrs. Paukert for patient typing assistance.

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Discussion with Reviewers

J.M. Cowley: What is the depth resolution and lateral resolution that can be achieved in sputtering experiments? For example, what was the thickness of the Ag film for figure 11 and how much influence does the irregularity of the layers, seen in the electron micrograph of figure 12, have on the amplitude and profile of the oscillations in the depth-profiling record?

Author: The depth resolution in sputtering experiments is about some nm and depends on the investigated material and on the thickness of the sputtered layers. For Ta_2O_5/Ta we get about 2 nm for Ni/Cr 2-5 nm. The lateral resolution is mainly determined by the analytical method (AES, SIMS, ISS, SNMS, XPS). The thickness of the Ag film in Fig. 11 was 5 nm. The initial irregularities of the layers and the roughness of the surface influence the amplitude and the profile of the oscillations and also the depth resolution. A survey on depth profiling is given by Hofmann, S. (1980): Quantitative depth profiling in surface analysis. Surface and Interface analysis 2, 148-160. See also the references Hofmann S. (1985, 1986, 1987).

K. Kiss: Could you describe the backthinning technique?

Author: The backthinning technique first was used by Hirsch PB, Partrige PG, Segall RL. (1959): An EM study of stainless steel deformed in fatigue and simple tension. Philos. Mag. 4, 721-729. The specimen is first sliced off the sample. The slice is spark-cut to get disks with a diameter of 3 mm. The surface of interest is coated with a lacquer for protection and then the disc is thinned to electron transparency by ion milling from the back side. See also the references Goodhew (1972), Allen (1985), Rehme and Oppolzer (1985).

K. Kiss: Is the (S) REM a commercial instrument? What are its advantages over a high-resolution, commercially available SEM?

Author: In the normal SEM the specimen is behind the focusing lens, the SE detector is on one side of the specimen and the solid-state BSE detector on the lower polepiece of the focusing lens above the specimen. The image brightness is modulated by the SE and/or the back-scattered electrons. In the SREM the object is tilted, the electron beam hits the surface under an angle of 20 - 30° and the image brightness is modulated by the SE and/or the forward-scattered electrons. For the high resolution SEM or STEM the sample is in the high-field region of a condensor objective lens. The SE spiral up to the axis to the collector above the lens. In the high resolution SREM the specimen is tilted and the forward-scattered electrons are deflected by the lower half of the lens field onto a scintillator transmission detector.In an unmodified commercial SEM fitted with a condensor objective in the upper stage and a transmission detector it is possible to get images in the SEM-mode with SE or in the SREM-mode with the forward scattered electrons. Wells OC. (1988). Scanning reflection image from a solid specimen in the SEM with a condensor-objective lens. Scanning 10, 73-81. Wells showed, that the scanning reflection images can provide additional useful information in conjunction with the SE images. The main advantage of the SREM is the possibility to use the LLE or ERE to obtain more surface sensitive information. The necessary energy spectrometer or retarding field is however - to my knowledge normally not available in a commercial SEM.

K. Kiss: Could you comment on specimen preparation for the MEM?

Author: The specimen in the MEM is at the same potential as the electron gun. As in the EEM the investigated specimen should be polished in order to avoid electrical breakdown.