11. Scanning electron microscopes and electron probes

Contents

11.1 System structure and working principle of scanning electron microscope

The imaging principle of the scanning electron microscope is completely different from that of the transmission electron microscope. Instead of using an electromagnetic lens to focus the image, it uses a finely focused electron beam to scan the surface of the sample, and uses a detector to receive various physical signals that are excited to modulate the image.

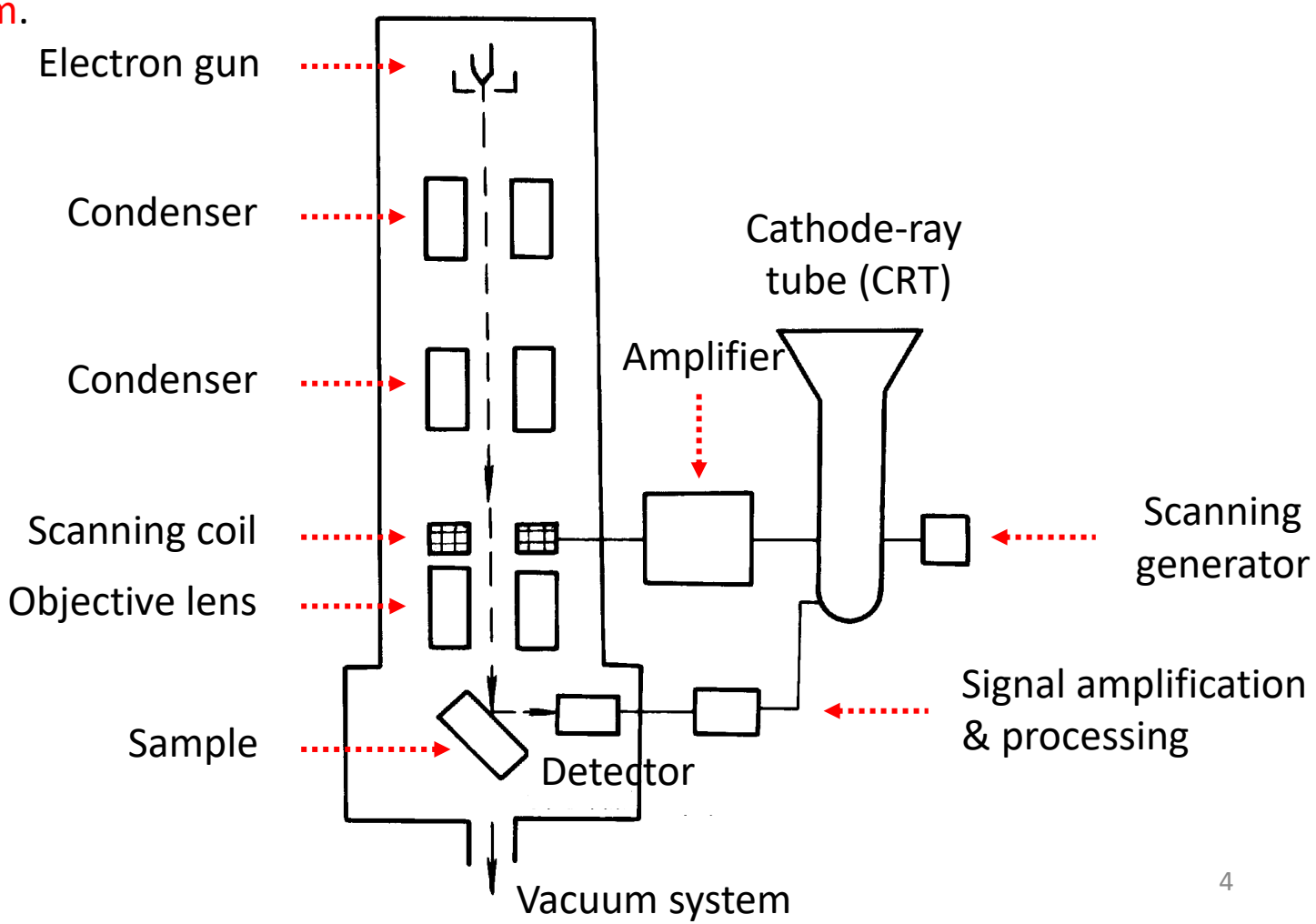
At present, the resolution of secondary electron images of scanning electron microscopes is better than 3 nm. The resolution of high-performance field emission gun scanning electron microscopes has reached about 1 nm, and the corresponding magnification can be as high as 300,000 times.

Compared with optical microscopes, scanning electron microscopes not only have high image resolution but also have a large depth of field, so they have obvious advantages in fracture analysis.

Scanning electron microscopes began to develop in the 1960s. As their performance continues to improve and their functions gradually improve, one scanning electron microscope can now simultaneously realize isotopic analysis of tissue morphology, micro-region composition and crystal structure. It has now become a major player in materials science. An indispensable analytical tool in other research fields.

11.1 System structure and working principle of scanning electron microscope

As shown in the figure, the scanning electron microscope consists of three basic parts: the electron optical system, the signal collection and image display and recording system, and the vacuum system.



11.1 System structure and working principle of scanning electron microscope

- Electronic optical system (lens tube)

1. Electron gun

The electron gun in the scanning electron microscope is the same as the transmission electron microscope. There are also two types: thermal emission and field emission, but the accelerating voltage is lower, generally up to 30 kV.

2. Electromagnetic lens

The electromagnetic lenses in the scanning electron microscope are not used for focusing imaging, but are condensers. Their function is to gradually focus and reduce the size of the electron beam spot, from the 50 μm beam spot of the electron gun to several nanometers.

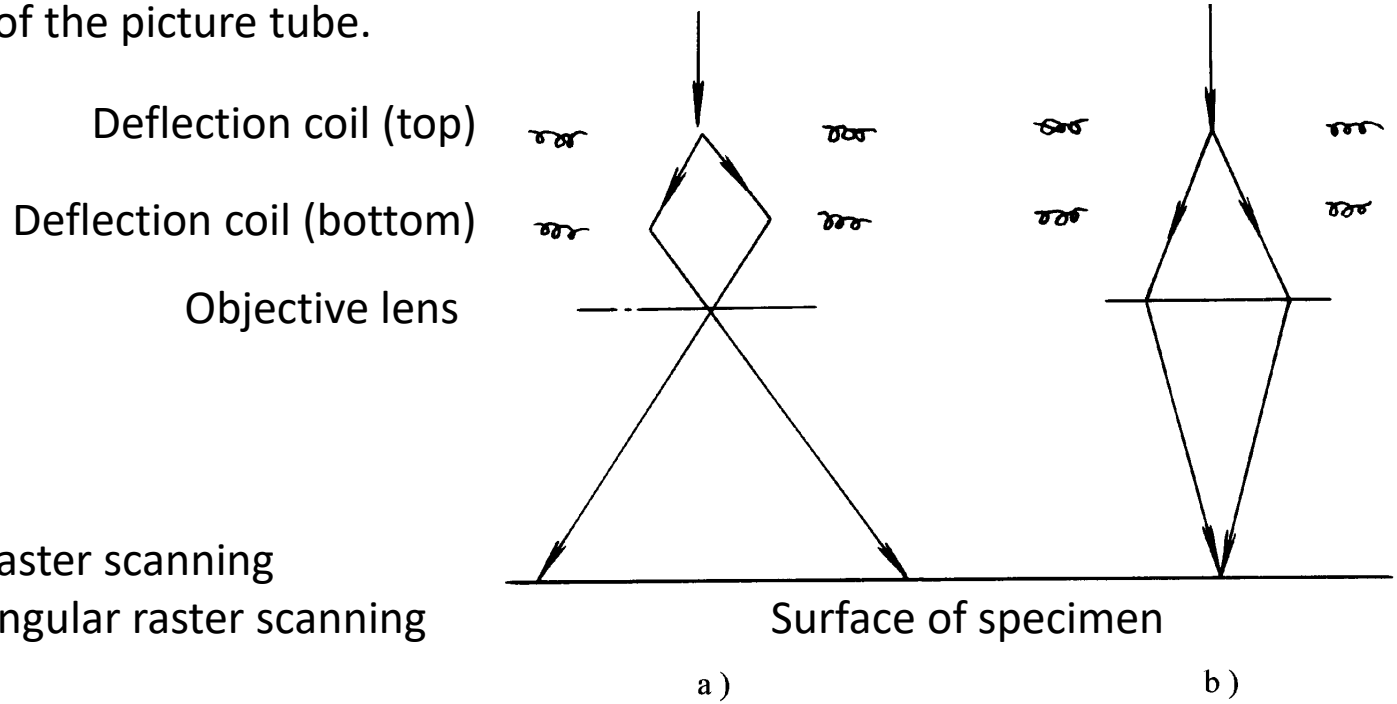
Scanning electron microscopes are generally equipped with three condensers. The first two condensers are strong magnetic lenses; the final lens is a weak magnetic lens with a longer focal length and is customarily called an objective lens. The beam spot size of the scanning electron microscope is about 3~5 nm, and the field emission scanning electron microscope can be as small as 1 nm.

11.1 System structure and working principle of scanning electron microscope

- Electronic optical system (lens tube)

3. Scanning coil

The function of the scanning coil is to deflect the electron beam and perform regular scanning on the sample surface. When analyzing the surface topography, the raster scanning method is used. When analyzing the electron channel pattern, the electron beam scans a square area on the sample surface. The angular raster scanning method is used to synchronously control the scanning of the electron beam on the sample surface and the scanning of the picture tube.



a) Raster scanning
b) Angular raster scanning

11.1 System structure and working principle of scanning electron microscope

- Electronic optical system (lens tube)

4. Specimen chamber

- The sample chamber is located at the bottom of the lens barrel. In addition to placing the sample, various signal detectors must be placed at appropriate locations.

- The sample stage is a complex and precise component that should be able to reliably carry or hold the sample and enable the sample to perform translation, tilt, and rotation movements to analyze each specific position or orientation on the sample.

- The sample chamber of the new scanning electron microscope is equivalent to a miniature laboratory equipped with various control functions, such as heating, cooling, stretching, bending, etc. The sample chamber is generally set to a high vacuum state. Some scanning electron microscopes can set the sample chamber to low vacuum or ambient vacuum according to analysis needs.

11.1 System structure and working principle of scanning electron microscope

- Signal collection and image display recording system

1. Signal collection

Signals such as secondary electrons and backscattered electrons are detected using a scintillation counter. After the electronic signal enters the scintillator, it causes ionization. The ions and free electrons recombine to produce visible light. The visible light signal enters the photomultiplier tube, and the optical signal is amplified and converted into a current signal output. The current signal is amplified by the amplifier and becomes a modulated signal.

2. Image display

The electron beam incident on the sample and the electrons in the Cathode-ray tube are scanned synchronously. The brightness of each image pixel on the fluorescent screen corresponds to the signal intensity at the corresponding position of the sample. Therefore, if the status of each point on the sample is different, the received signal intensity is also different, corresponding to the brightness of the image point on the fluorescent screen. Therefore, an image reflecting the surface status of the sample is displayed on the fluorescent screen.

11.1 System structure and working principle of scanning electron microscope

- Vacuum system

To ensure the normal operation of the scanning electron microscope, there are certain requirements for the vacuum degree in the lens tube: Under normal circumstances, if the vacuum degree of the lens tube reaches 1.33×10^{-2}~1.33×10^{-3} Pa, it can prevent the discharge between the electron gun electrodes and the sample contamination. The picture shows a scanning electron microscope photo of the actual object.

11.2 Working principle and application of environmental scanning electron microscope

- Basic components and characteristics of environmental scanning electron microscope

Environmental scanning electron microscopes generally have three imaging modes: high vacuum, low vacuum, and environmental scanning (ESEM) mode.

- The high vacuum mode is the traditional scanning electron microscope mode.

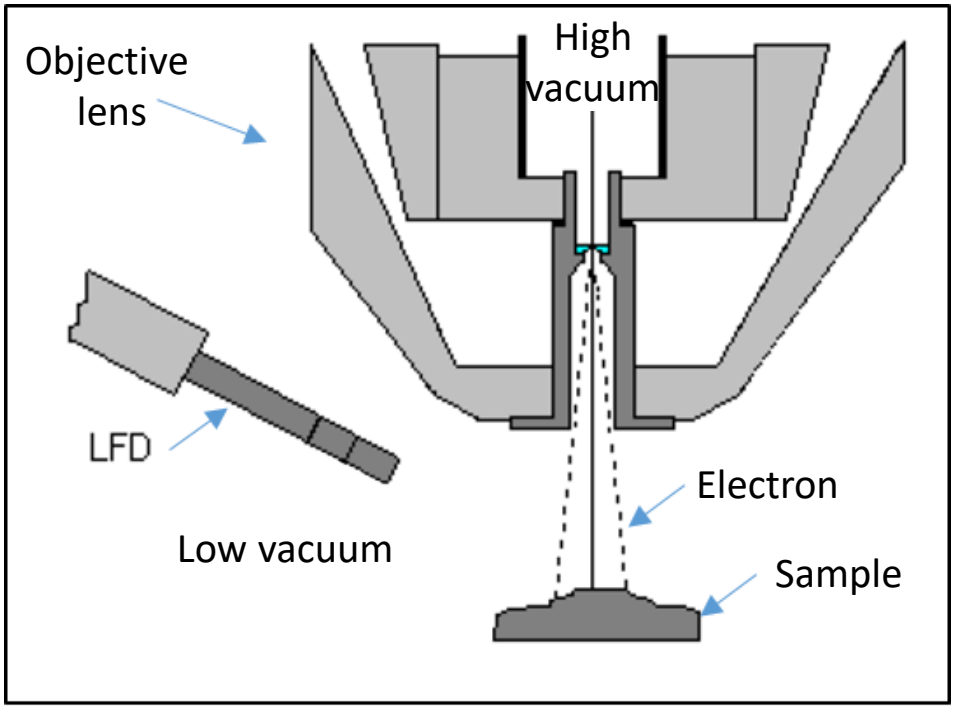
- The most significant difference between the environmental scanning electron microscope and the traditional scanning electron microscope is that the vacuum degree of the sample chamber of the former is much lower than that of the latter (about 2,600 Pa), which is closer to the ambient atmospheric pressure. As the name suggests, it is an environmental scanning electron microscope.

- The application of this technology has greatly broadened the observation range of sample types. Traditional scanning electron microscopy is only suitable for dry and conductive samples. In contrast, environmental scanning electron microscopy is based on traditional scanning electron microscopy and has added the ability to detect samples containing large amounts of water and non-conductivity.

11.2 Working principle and application of environmental scanning electron microscope

- Low vacuum mode with LFD electronic probe

The sample chamber of an environmental scanning electron microscope is in a low vacuum, while the electron gun and lens barrel are still in a high vacuum. A pressure difference diaphragm is added to the pole shoe. It isolates the lens barrel and the sample chamber and ensures that the electron beam is in a high vacuum state. In addition, the diaphragm protects components such as the electron gun and lens barrel from water vapor corrosion.

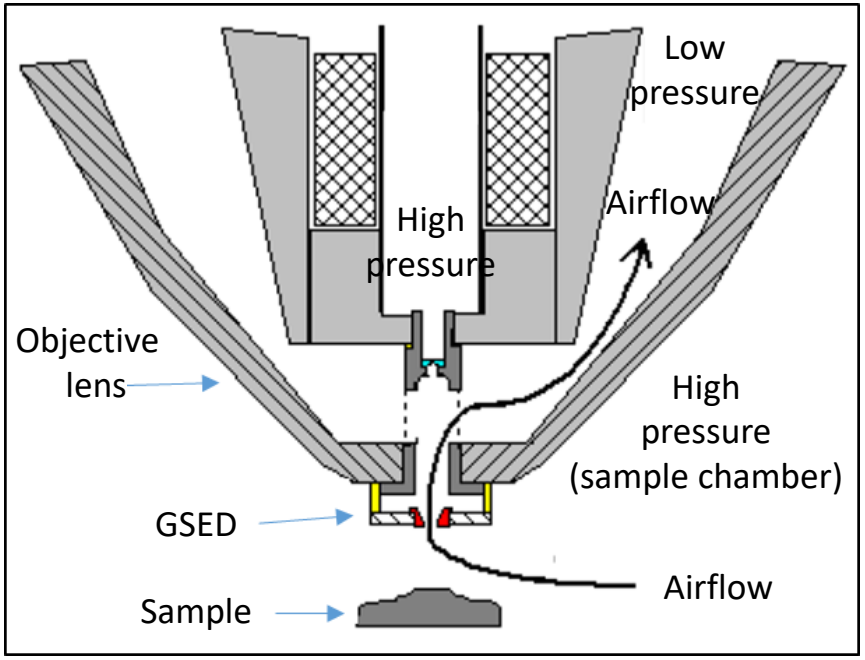


Mechanical pumps and molecular turbine pumps control the high vacuum of the electron gun and lens barrel. In contrast, the pressure of the sample chamber is controlled by adjusting the concentration of external water vapor and can be freely switched between 10 and 130 Pa. The electronic probe used in low vacuum mode is an LFD (large field detector).

11.2 Working principle and application of environmental scanning electron microscope

- Environmental Scanning Mode and GSED Electronic Probe

Compared with low vacuum, environmental vacuum adds a first-level pressure difference diaphragm to the latter. That is, a button-like diaphragm is installed under the objective lens. The whole is called GSED probe, as shown in the picture. The probe has two functions: one is to isolate the vacuum between the sample chamber and the lens barrel, and the other is to collect the electronic signal of the sample. When using this mode, the vacuum in the electron microscope can be divided into three sections.

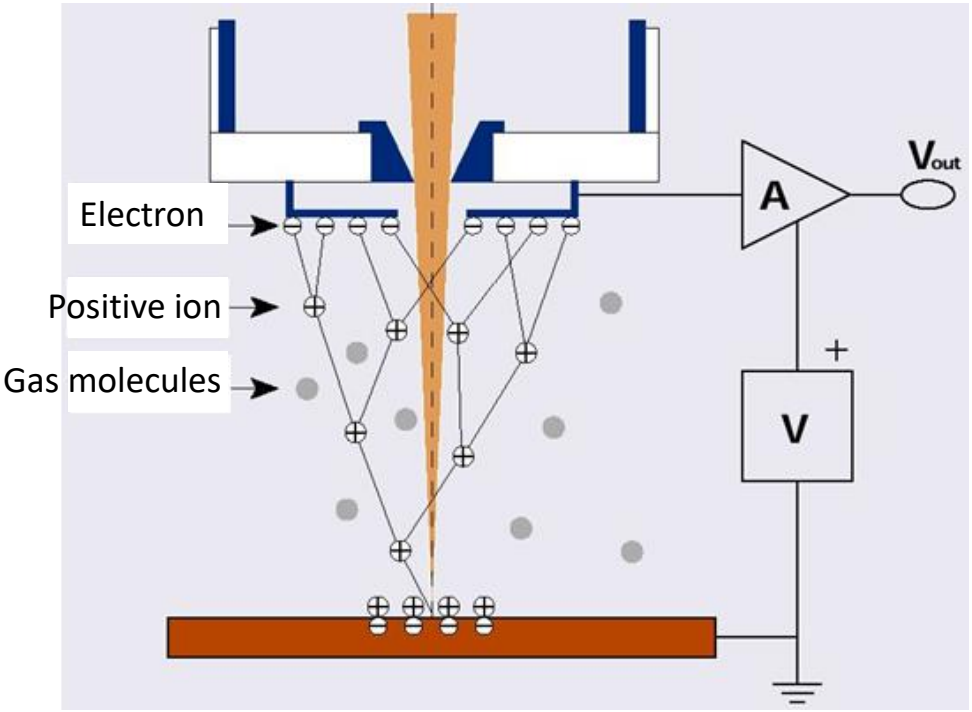


The electron gun and lens tube are in low vacuum between the high vacuum objective lens and the button-shaped diaphragm, and the sample chamber is in ambient vacuum. ESEM maintains a low vacuum in the sample chamber by continuously replenishing gas in the sample chamber. It also provides working gas for the secondary electron detector GSED. Water vapor is the most commonly used working gas.

11.2 Working principle and application of environmental scanning electron microscope

- Environmental Scanning Mode and GSED Electronic Probe

The secondary electrons generated by the interaction between the incident electron beam and the sample escape from the sample surface and accelerate upward under the positive voltage applied by the environmental secondary electron detector; these accelerated secondary electrons collide with gas molecules, ionizing them to produce positive ions and electrons (called environmental secondary electrons);



This process of electron acceleration and gas ionization is repeated, resulting in a proportional cascade amplification of the original secondary electron signal. The positive ions that are attracted by the charge on the sample surface and move downward can eliminate the charge.

11.3 Main features of scanning electron microscopes

- Resolution

The resolution of the scanning electron microscope is related to the type of detected signal, because different signals are generated in different depth ranges of the sample, see table,

Various signals originate from the depth range (nm) of the sample surface

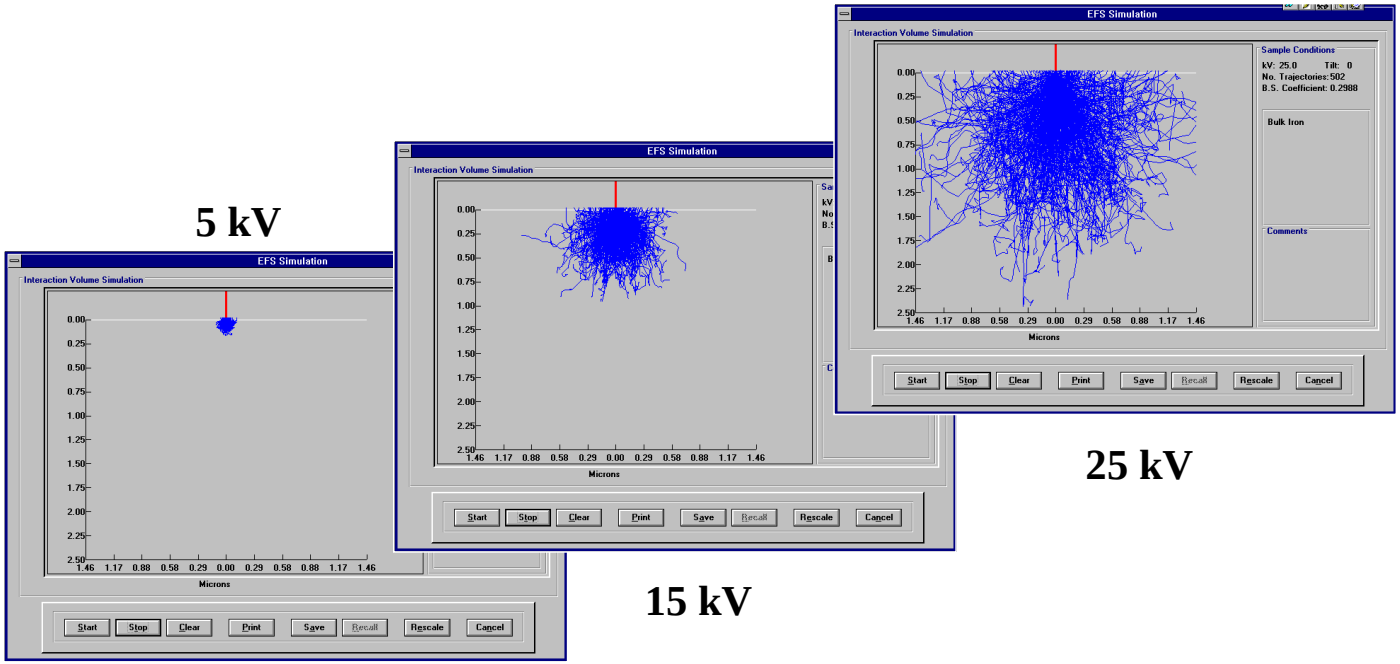
Signal	Secondary electron	Backscatter electron	Absorbed electron	X-ray	Auger electron
Depth	5~10	50~200	100~1000	100~1000	0.5~2

It can be seen that the depth of the sample that generates Auger electrons is the smallest, followed by secondary electrons, and the depth range of the sample generated by absorbed electrons and characteristic X-rays is the largest.

11.3 Main features of scanning electron microscopes

- Resolution

The electron beam generally expands into a drop-shaped area in the sample. The depth and shape of the expanded area are affected by the accelerating voltage and the atomic number of the sample. The expanded area increases as the accelerating voltage increases and decreases as the atomic number of the sample increases.



Computer simulation results of the electron beam expansion area in the sample under different acceleration voltages.

11.3 Main features of scanning electron microscopes

- Resolution

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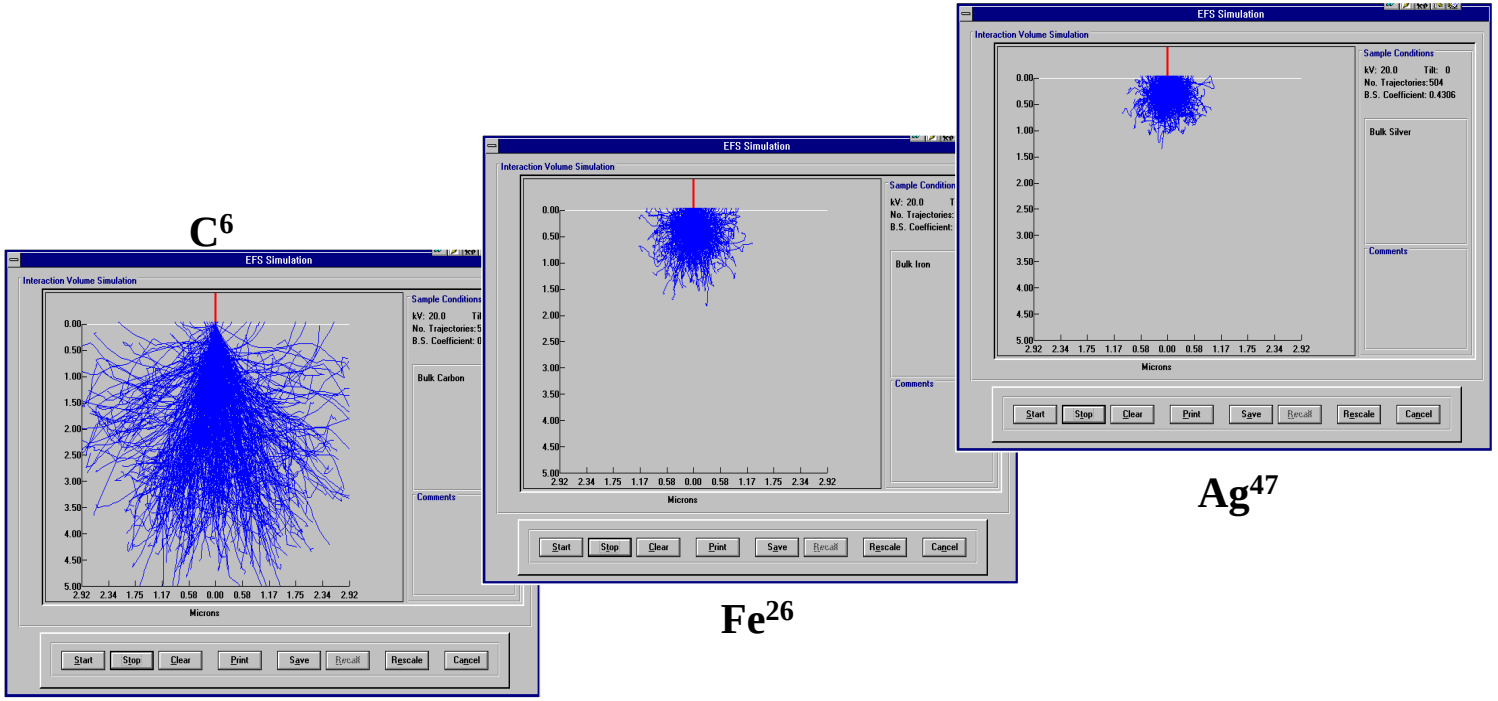
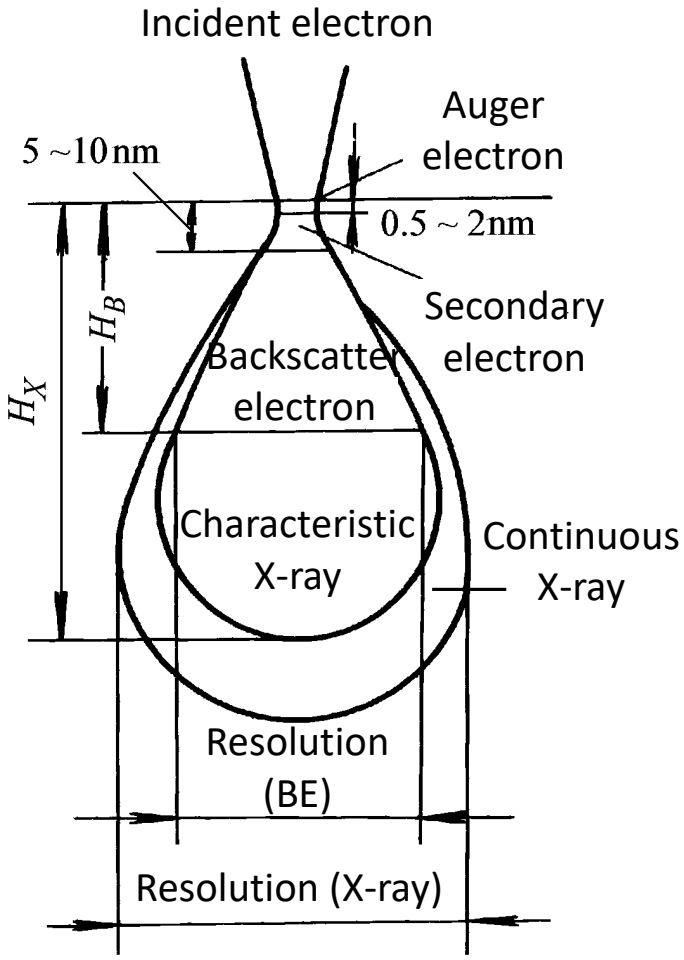


Computer simulation results of the electron beam expansion area in different samples when the accelerating voltage is constant.

11.3 Main features of scanning electron microscopes

- Resolution



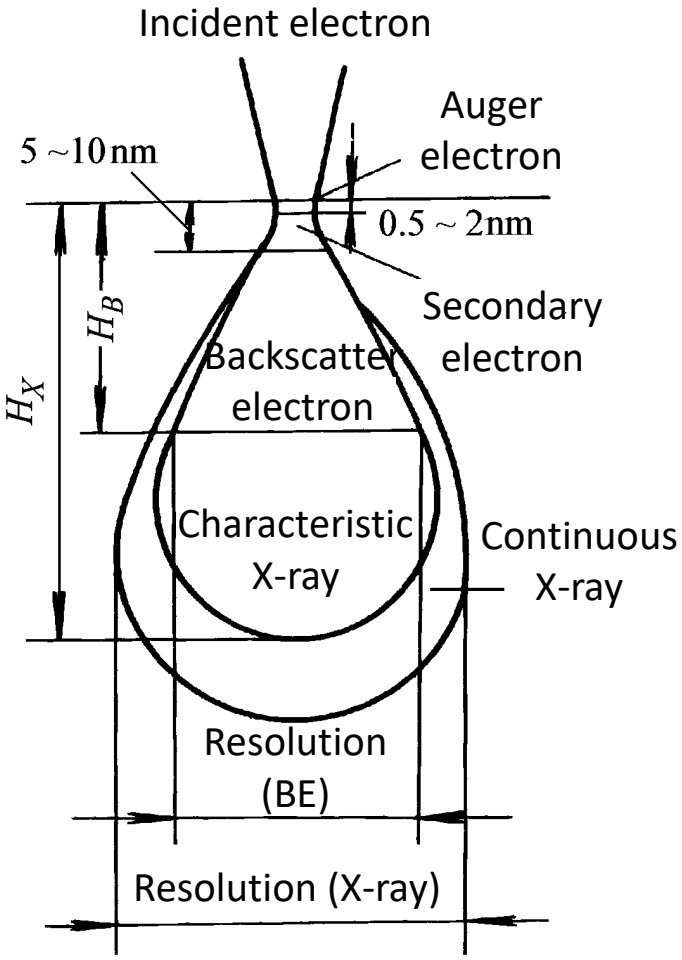
Signal imaging resolutions will decrease as the depth range over which the signal is generated increases. Because as the depth distance increases, the lateral expansion range of the electron beam also increases.

The energy of secondary electrons is very low, and the secondary electrons generated in a deeper range cannot escape from the surface; the Auger electrons generated in a deeper range lose their characteristic energy due to inelastic scattering by the sample.

The secondary electron image resolution is high since the sample area where secondary electrons are generated is small.

11.3 Main features of scanning electron microscopes

- Resolution

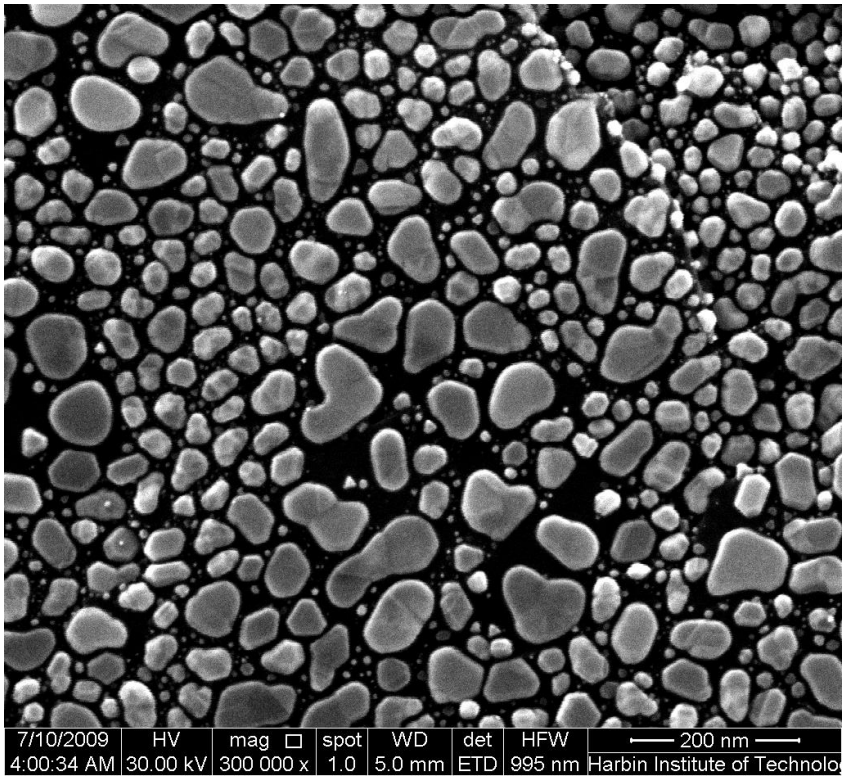


Because the depth range where backscattered electrons are generated is larger, the lateral expansion range of the electron beam at this depth is also larger, so the resolution of the backscattered electron image is lower than that of the secondary electron image; while the depth range where absorbed electrons are generated is larger, so the image resolution is lower. Because the secondary electron image has the highest resolution, it is customary to use the secondary electron image resolution for scanning.

Electron microscope resolution index characteristics X-rays and Auger electrons are used for composition analysis. The sample area that generates these signals is usually called the spatial resolution of micro-area composition analysis.

11.3 Main features of scanning electron microscopes

- Resolution



Determination of secondary electron image resolution.

Vacuum-evaporated gold film particle samples are usually used to determine the resolution of the scanning electron microscope. The minimum distance between particles measured in the photo is the image resolution of the scanning electron microscope at the magnification.

For example, if the minimum distance between particles is measured in a photo to be 0.30 mm, and the magnification of the photo is 300,000 times, the resolution is 1 nm; the secondary electron image resolution will decrease as the accelerating voltage decreases.

11.3 Main features of scanning electron microscopes

- Magnification

The amplitude of the incident electron beam scanning on the sample surface is A_s, and correspondingly, the amplitude of the cathode ray synchronous scanning on the fluorescent screen is A_c. The ratio of A_c and A_s is the scanning electron microscope magnification.

$$M = \frac{A_c}{A_s}$$

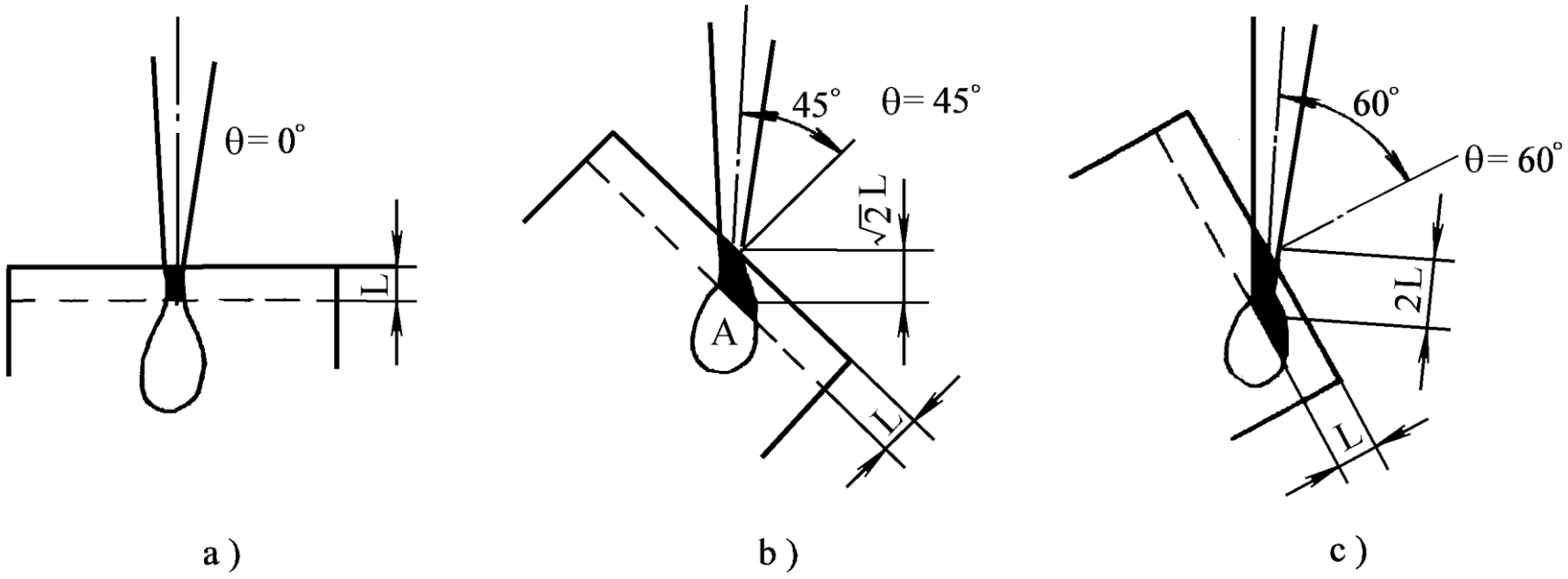
Since the size of the SEM screen is fixed, the magnification can be changed simply by changing the size of the scanning area of the electron beam on the sample. For example, if the width of the fluorescent screen is A_c = 100 mm and the scanning amplitude of the electron beam on the sample is A_s = 0.05 mm, the magnification is 2000 times.

The principle of magnification selection is that on the premise of being able to resolve the smallest structural details on the sample, a lower magnification should be used as much as possible to observe a larger sample area.

11.4 Surface topography contrast principle and its application

- Secondary electron imaging principle

The brightness of the image point in the secondary electron image depends on the secondary electron output corresponding to the sample position, and the output of the secondary electron is very sensitive to the orientation of the sample micro-area surface. It depends on the sample volume from which the secondary electrons are generated.



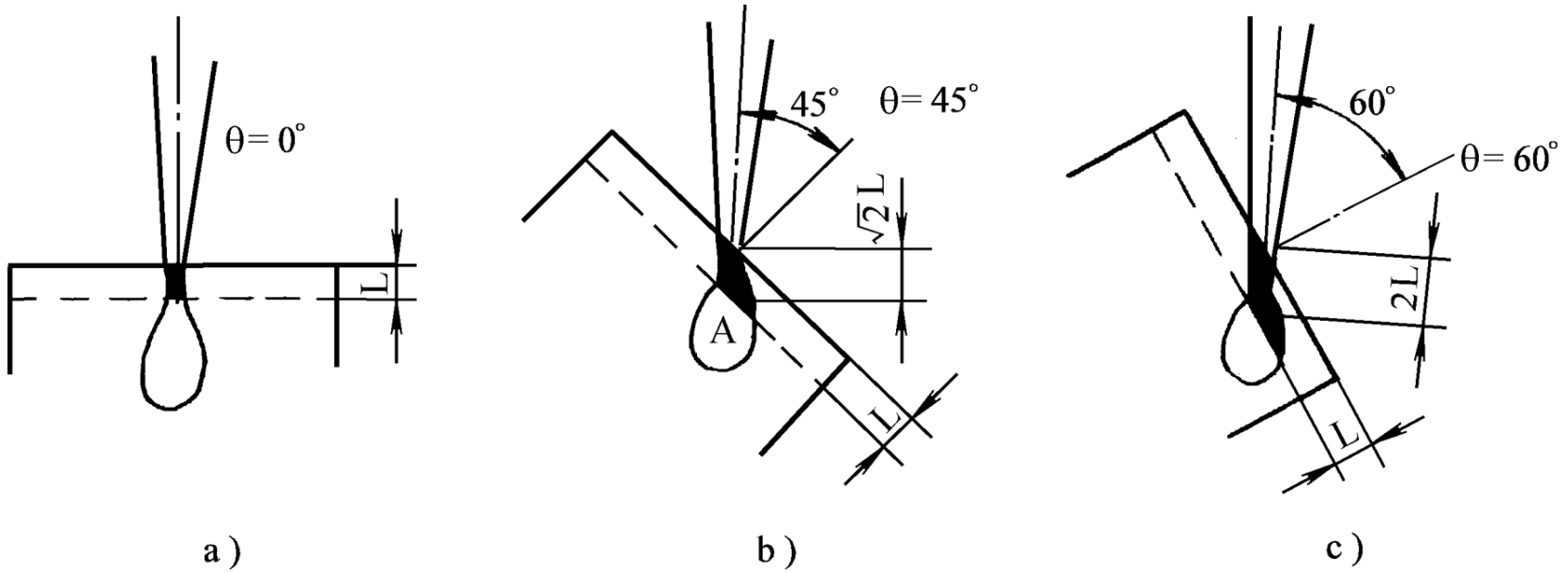
The secondary electron imaging principle

11.4 Surface topography contrast principle and its application

- Secondary electron imaging principle

As the angle θ between the normal line of the sample surface and the direction of the electron beam increases, the effective depth of excited secondary electrons increases, and the output of secondary electrons increases accordingly.

When $\theta = 0°$, the secondary electron output is the smallest; when $\theta = 45°$, the output increases; when $\theta = 60°$, the secondary electron output is greater.

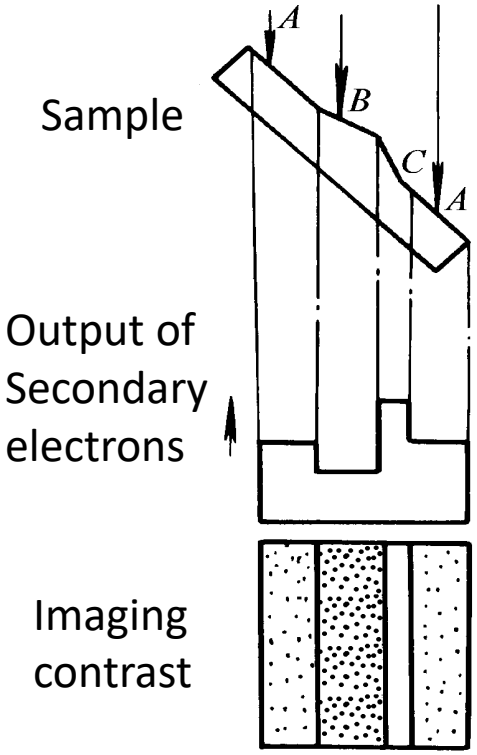


The secondary electron imaging principle

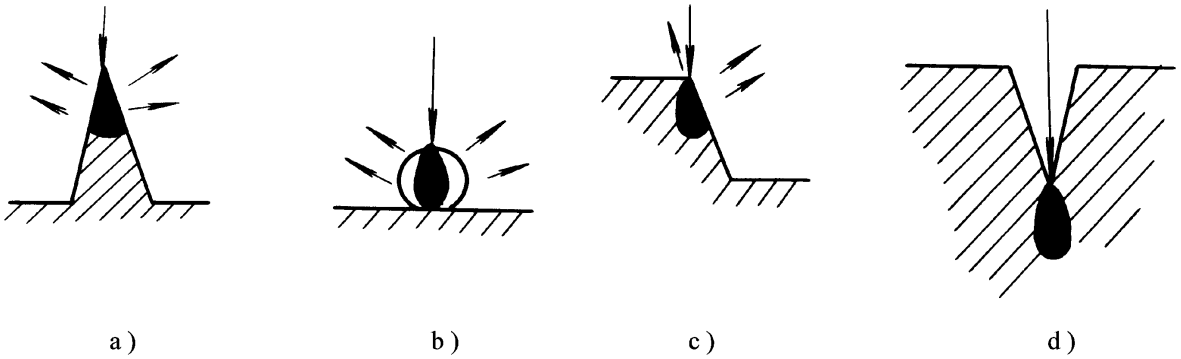
11.4 Surface topography contrast principle and its application

- Secondary electron imaging principle

According to the above principle, the secondary electron imaging contrast is shown in the figure. In the figure, the B plane has the smallest tilt angle, the smallest secondary electron output, and the lowest image brightness; the C plane has the largest tilt angle, and the largest image brightness.



The brightness of the image point in the image ultimately depends on the number of secondary electrons detected. The images of sharp corners, particles, etc. protruding from the surface are brighter; the images of grooves are darker, because although the secondary electron output is larger here, it is not easily received.



Schematic of secondary electron excitation in actual samples

11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

The surface topography contrast provided by the scanning electron microscope secondary electron image is extremely widely used, mainly including the following aspects:

- Fracture analysis: Determine fracture properties and fracture microscopic mechanisms
- Metallographic analysis: observing the morphology, size, and distribution of phases
- Powder morphology analysis: observe powder spatial morphology and size distribution
- Surface epitaxial film crystallization film analysis: analysis of crystalline film particle morphology and size
- Corrosion Analysis: Investigate Wear and Corrosion
- Mechanisms Failure Analysis: Analyze the causes of aging and analysis related to surface morphology, etc.

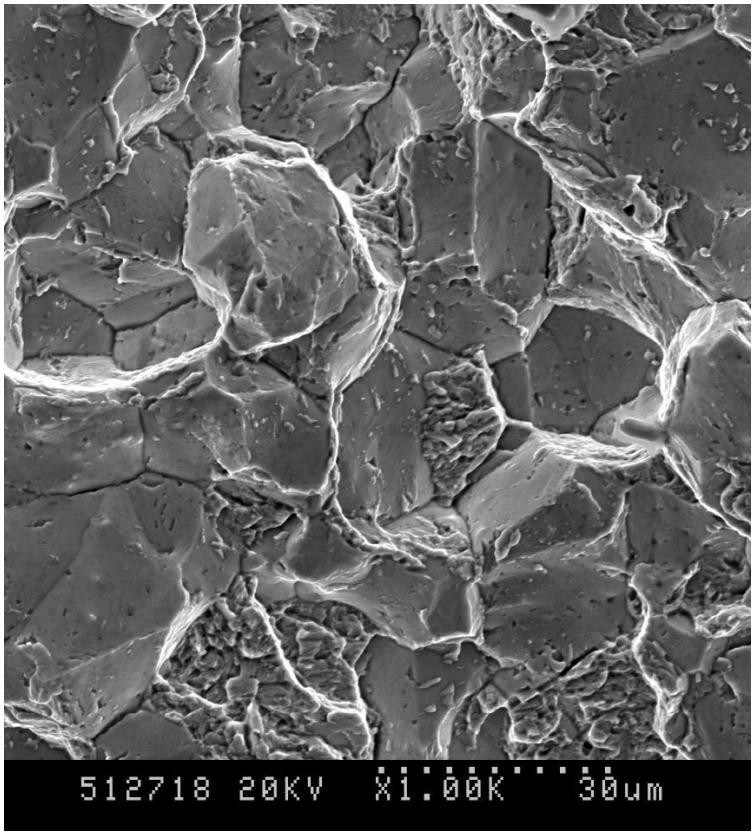
11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Fracture analysis

1. Intergranular fracture

The characteristic along the crystalline fracture surface is like a rock candy block, as shown in the figure. The fracture occurs on the surface of the grain and is brittle, with no signs of plastic deformation on the fracture surface.



Morphological Characteristics of Intergranular Fracture

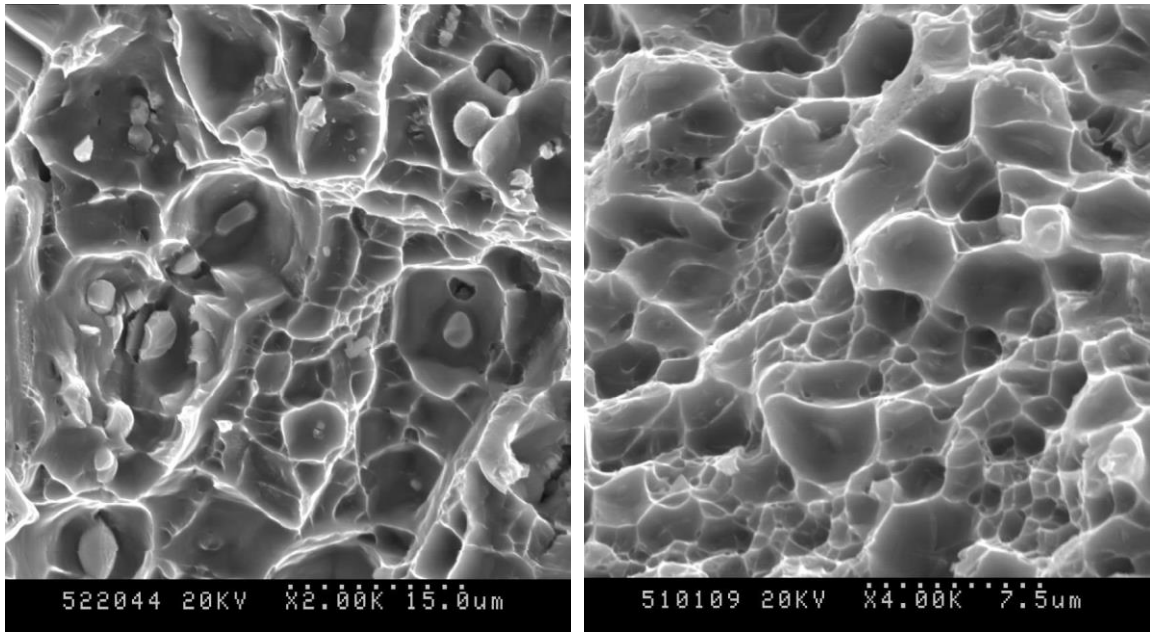
11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Fracture analysis

2. Dimple fracture

The dimple fracture is a transgranular ductile fracture, and its morphological characteristics are shown in the figure. The fracture surface is composed of dimples and tearing ripples. Second-phase particles can sometimes be seen at the bottom of the dimples and the fracture surface exhibits ductile fracture characteristics.



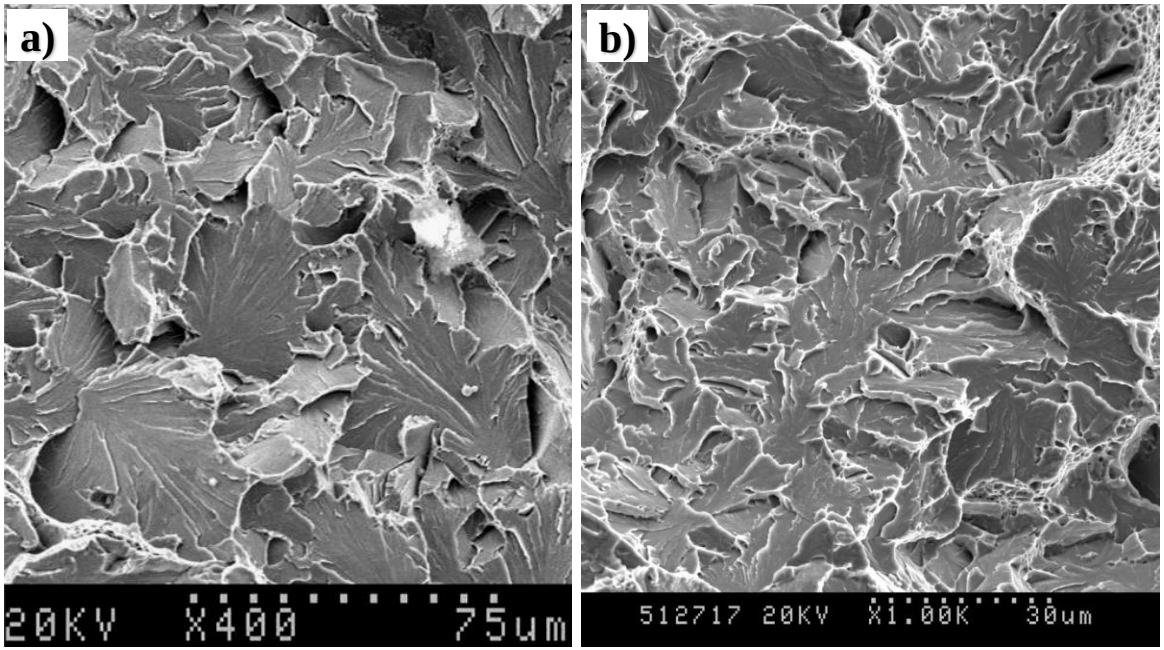
Morphological characteristics of the dimple fracture surface.

11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Fracture analysis

3. Cleavage (quasi-cleavage) fracture surface



A Cleavage (quasi-cleavage) fracture is a brittle, transgranular fracture along the cleavage plane. The fracture characteristics are shown in the figure. Multiple steps in the fracture merge with each other during the crack propagation process to form a river pattern.

Morphological characteristics of **a)** cleavage fracture and **b)** quasi-cleavage fracture

11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Fracture analysis

4. Fiber reinforced composite material fracture

The figure shows the secondary electron image of the fracture surface of carbon fiber-reinforced ceramic composite material. A large number of fibers are pulled out and exposed on the fracture surface, and holes are left after the fibers are pulled out.

Carbon fiber reinforced ceramic composite fracture surface

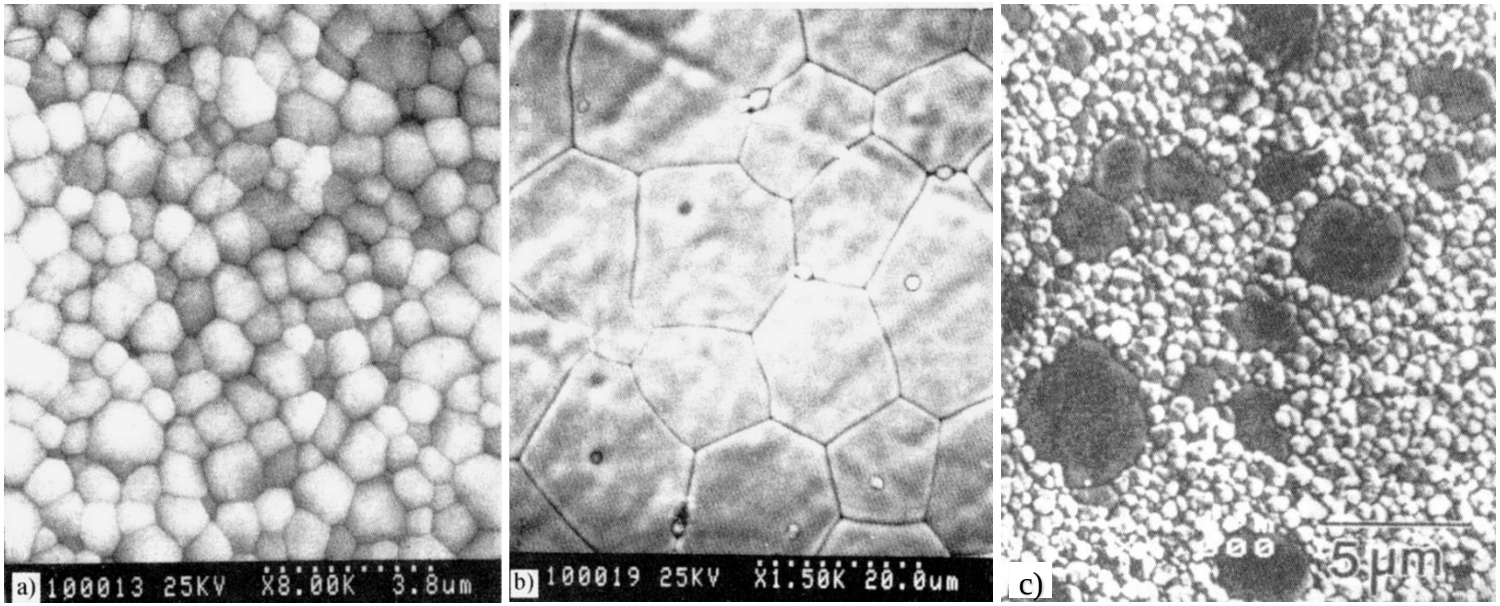
11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Sample surface morphology observation

1. Sintered body natural surface

The picture shows the secondary electron image of the surface of ZrO_2-Y_2O_3 ceramic sintering.



ZrO_2-Y_2O_3 ceramic sintered surface

a) t-ZrO_2 b) c-ZrO_2 c) (c+t)ZrO_2

11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Sample surface morphology observation

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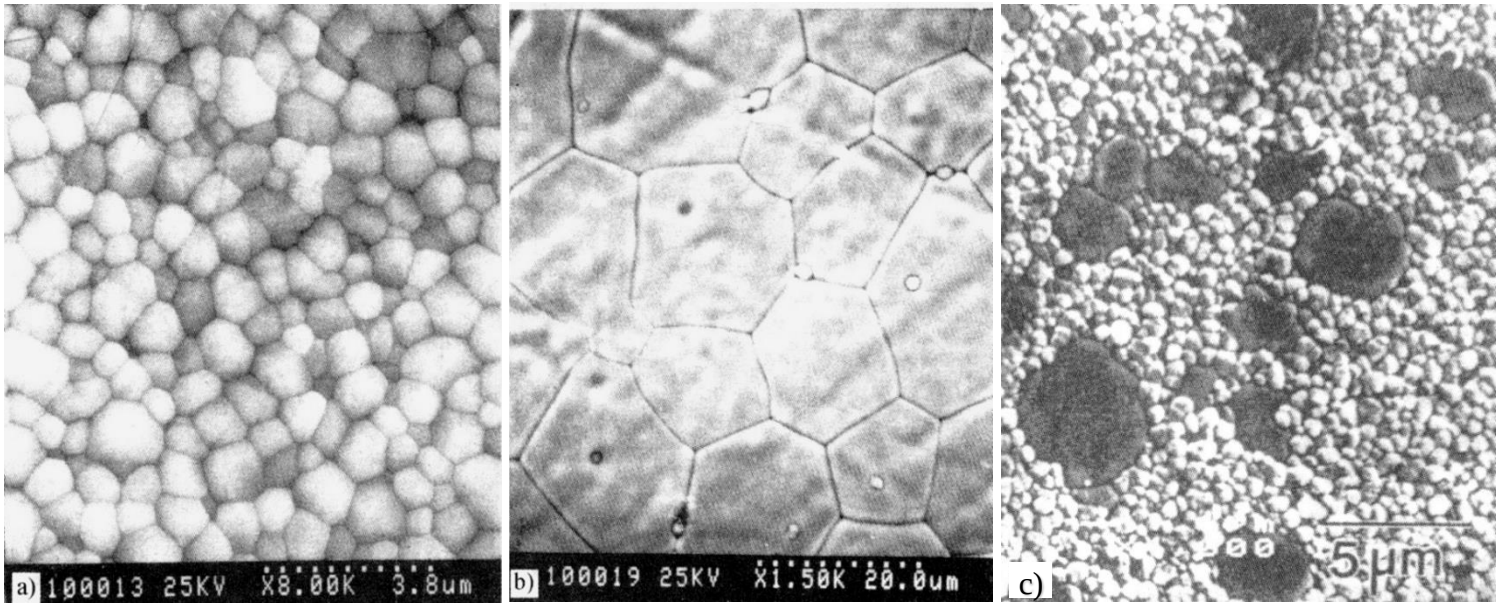


ZrO_2-Y_2O_3 ceramic sintered surface

a) t-ZrO_2 b) c-ZrO_2 c) (c+t)ZrO_2

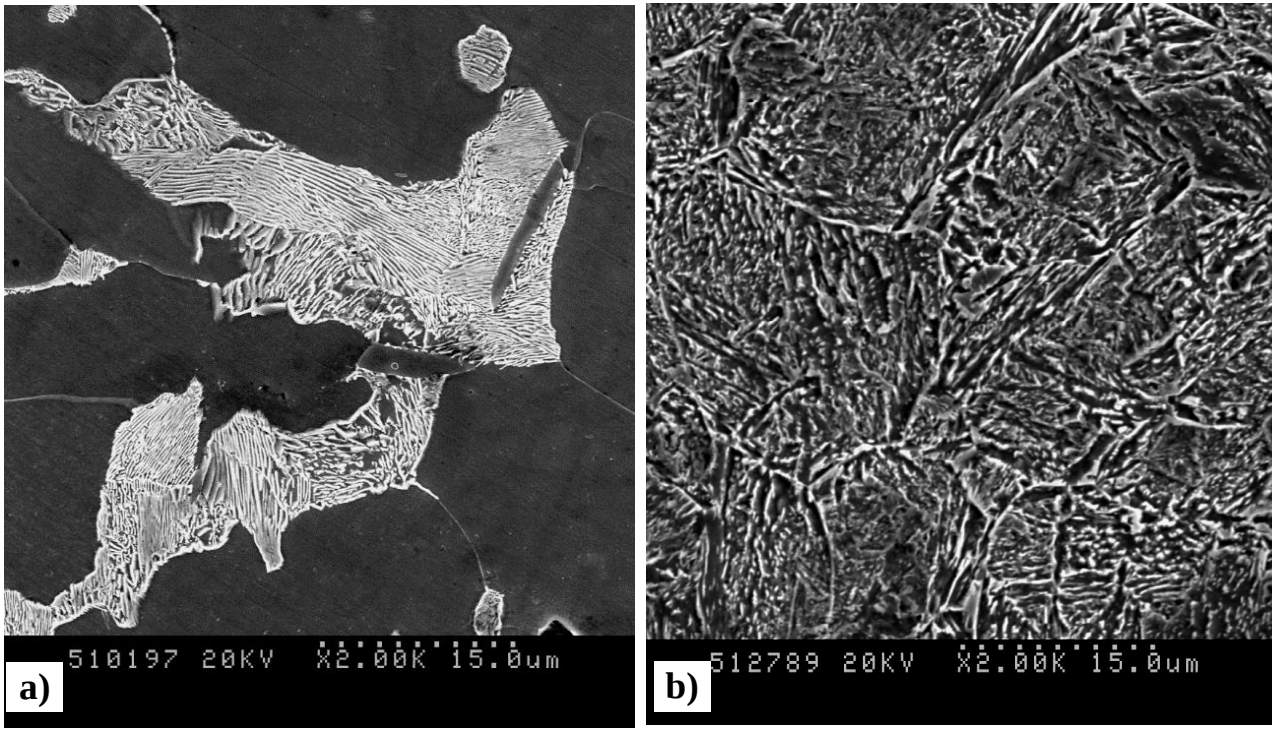
11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Sample surface morphology observation

2. Metallographic analysis

The picture shows the secondary electron image of the metallographic structure of steel. The sample can be observed on the scanning electron microscope after polishing and corrosion, but the corrosion degree of the sample is slightly greater than that of the light microscope sample.



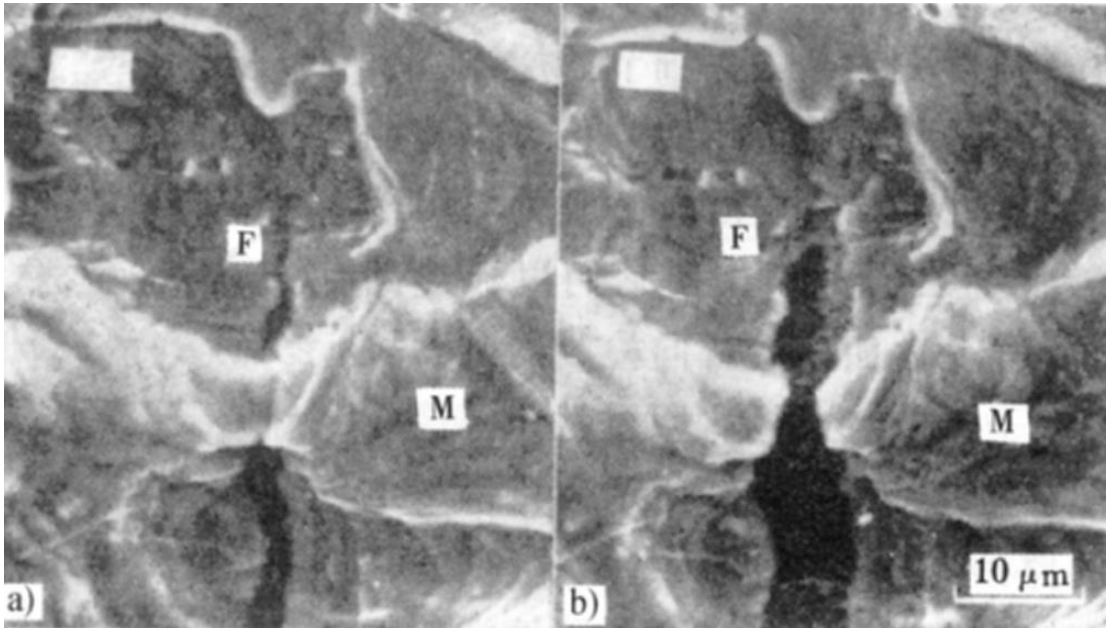
Secondary electron image of the metallographic structure of steel **a)** Ferrite plus pearlite **b)** Tempered martensite

11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

In-situ observation of dynamic processes of material deformation and fracture

As shown in the figure, the dynamic process of plastic deformation, crack initiation, crack propagation and unstable fracture of the material can be observed in situ using a dynamic stretching table.



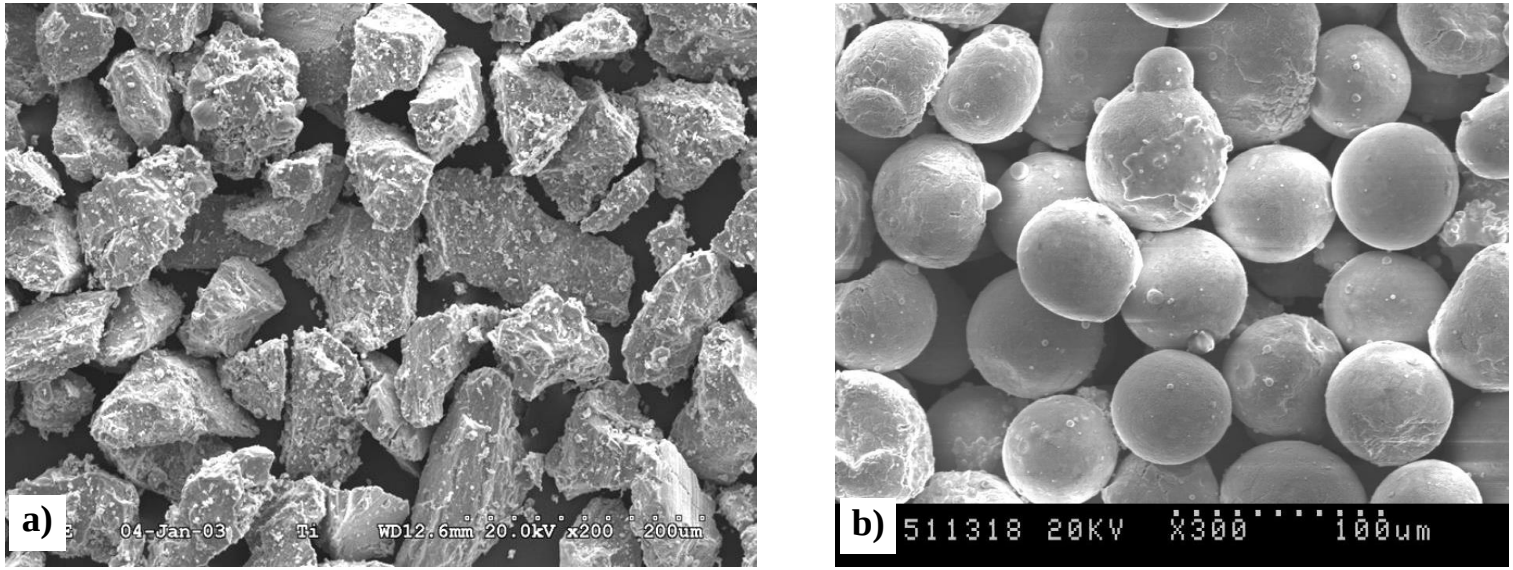
Dynamic in-situ observation of the tensile fracture process of ferrite (F) + martensite (M) dual-phase steel **a)** Crack initiation **b)** Crack propagation

11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Other applications

Powder analysis: The picture shows the secondary electron image of the powder sample. The three-dimensional image is very strong, and the spatial shape of the powder can be clearly observed. The sample does not need special treatment, it only needs to be evenly dispersed on the stage.

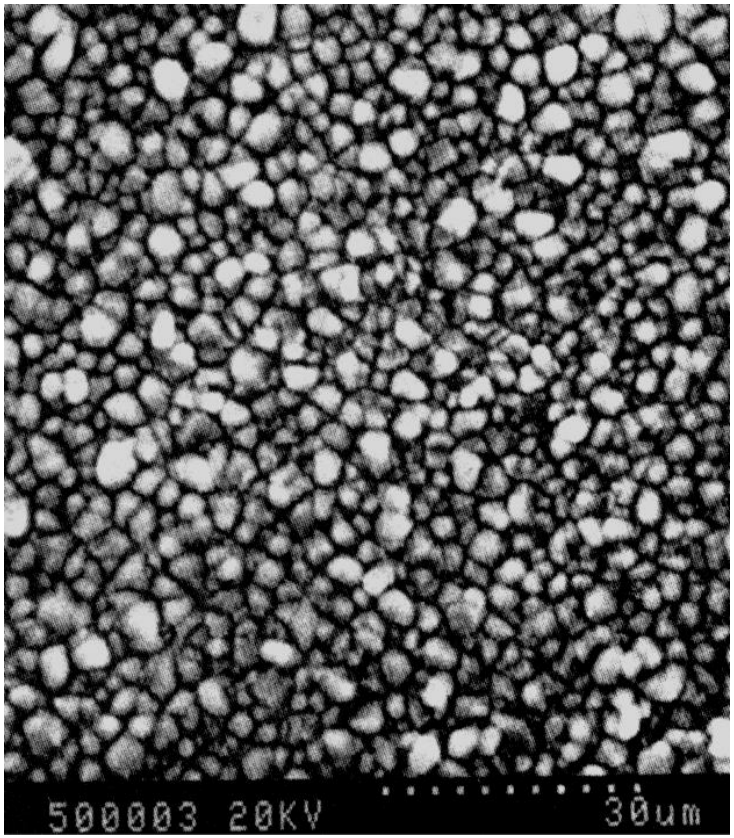


Secondary electron image of powder sample **a)** irregular shape **b)** spherical shape

11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Other applications



Morphology of phosphating film on the surface of low-carbon steel plate

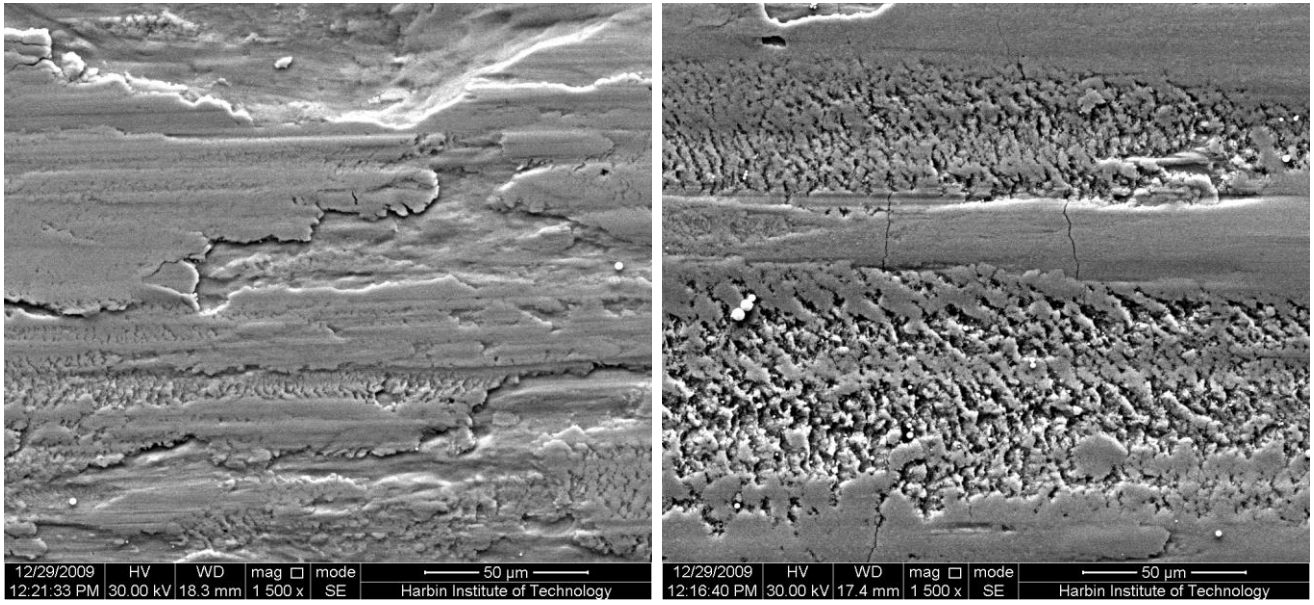
The picture shows the secondary electron image of the morphology of the phosphating film on the surface of a low-carbon steel plate. It can be seen that the phosphate film is in the form of spherical crystals with uniform size. The sample does not need to be processed and can be directly observed.

11.4 Surface topography contrast principle and its application

- Application of secondary electron image topography contrast

Other applications

Wear analysis: The picture shows the secondary electron image of the wear morphology of the alloy steel surface. Analyze the wear mechanism based on wear morphology characteristics and working conditions

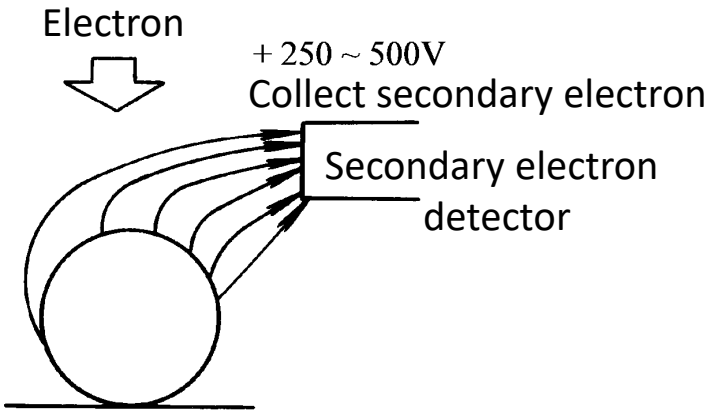


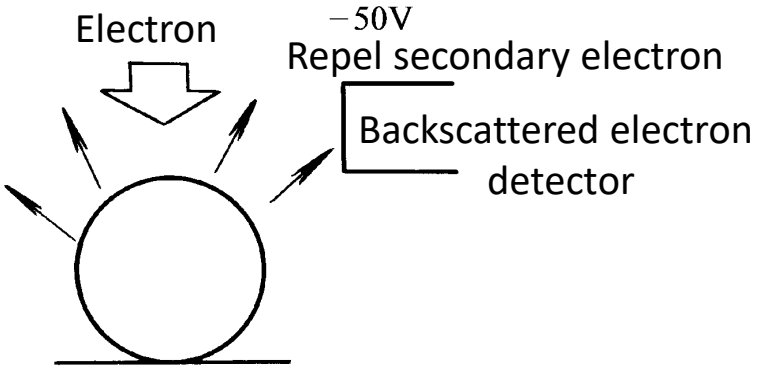
Secondary electron image of surface wear morphology of alloy steel

11.5 Topography contrast characteristics of backscattered electron images

- The principle and application of backscattered electron image contrast

Topography contrast characteristics of backscattered electron images





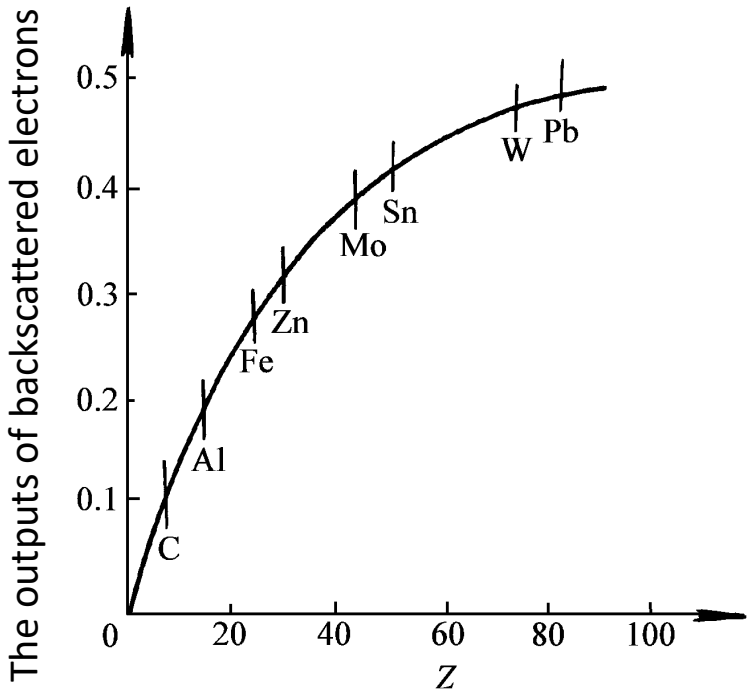
Backscattered electron image topography contrast features: Like secondary electron images, backscattered electron images can also provide surface topography contrast. However, compared with the secondary electron image, the morphology contrast characteristics of the backscattered electron image are:

1. The sample area where backscattered electrons are generated is large, so the resolution of the backscattered electron image is low.
2. The energy of secondary electrons is low, and the secondary electrons facing away from the detector can be detected under the attraction of the gate; while the energy of backscattered electrons is high, and the signal facing away from the detector is difficult to detect, see Figure 1, so the image has large shadows.

11.5 Topography contrast characteristics of backscattered electron images

- The principle and application of backscattered electron image contrast

Backscattered electron image atomic number contrast principle

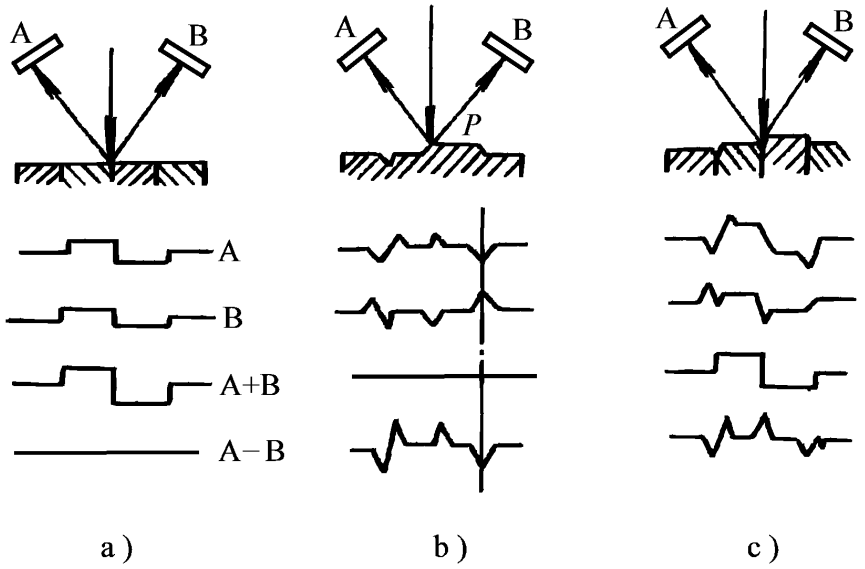


The figure shows the effect of atomic number on the output of backscattered electrons. The output of backscattered electrons increases with the increase of atomic number. In the range of atomic number Z less than 40, the relationship is approximately proportional. If the backscattered electron signal is used for imaging, the image corresponding to the area with a large average atomic number in the sample will be brighter, and the image corresponding to the area with a small average atomic number in the sample will be dark. Different phases have different element compositions, and their average atomic numbers differ. When backscattered electron imaging is used, different phases show different brightness.

11.5 Topography contrast characteristics of backscattered electron images

- The principle and application of backscattered electron image contrast

Backscattered electron image atomic number contrast principle



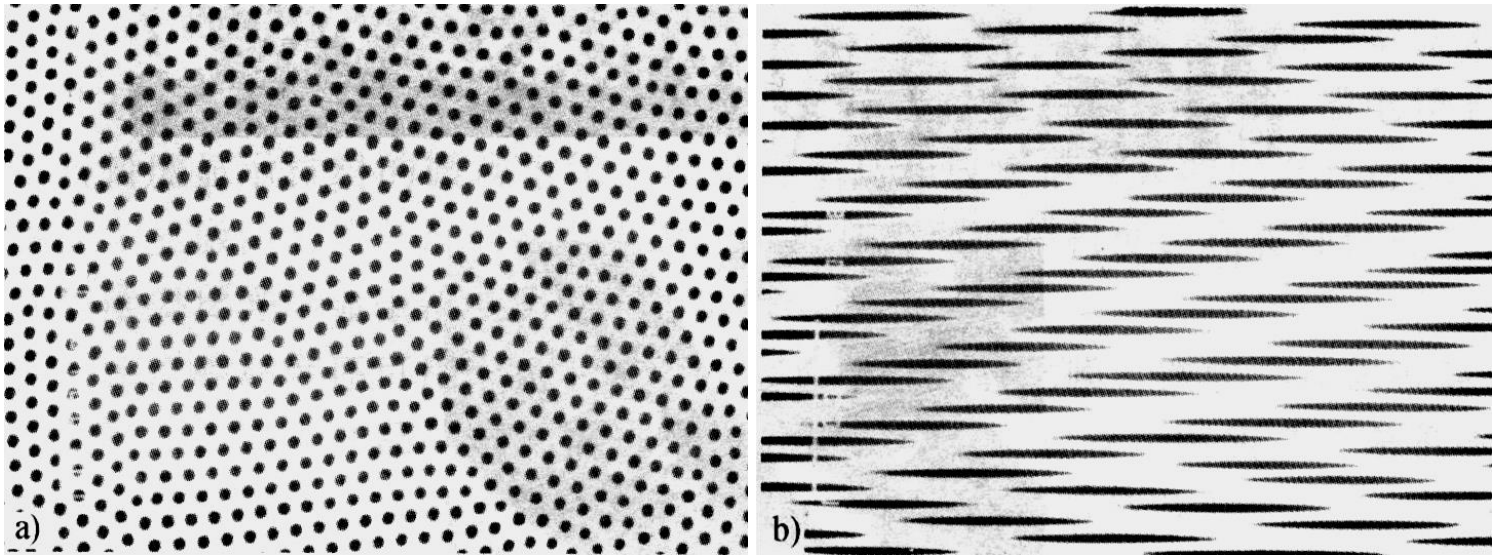
The working mechanism of detectors
a) Smooth surface **b)** Uniform composition
c) Differences in morphology and composition

To eliminate the interference of surface topography contrast on atomic number contrast, surface polishing can be used without corroding the sample, or a pair of (A, B) detectors can be used to detect signals. Adding the A and B signals can obtain the atomic number contrast image; subtracting the A and B signals can obtain the surface topography contrast image. The principle is shown in the figure.

11.5 Topography contrast characteristics of backscattered electron images

- Application of atomic number contrast in backscattered electron images

Using backscattered electron imaging, different phases show different brightness due to different average atomic numbers, which can be used to analyze the phases' composition, shape, size, and distribution. The figure below shows the backscattered electron image of the eutectic structure morphology of the Al-Li alloy.

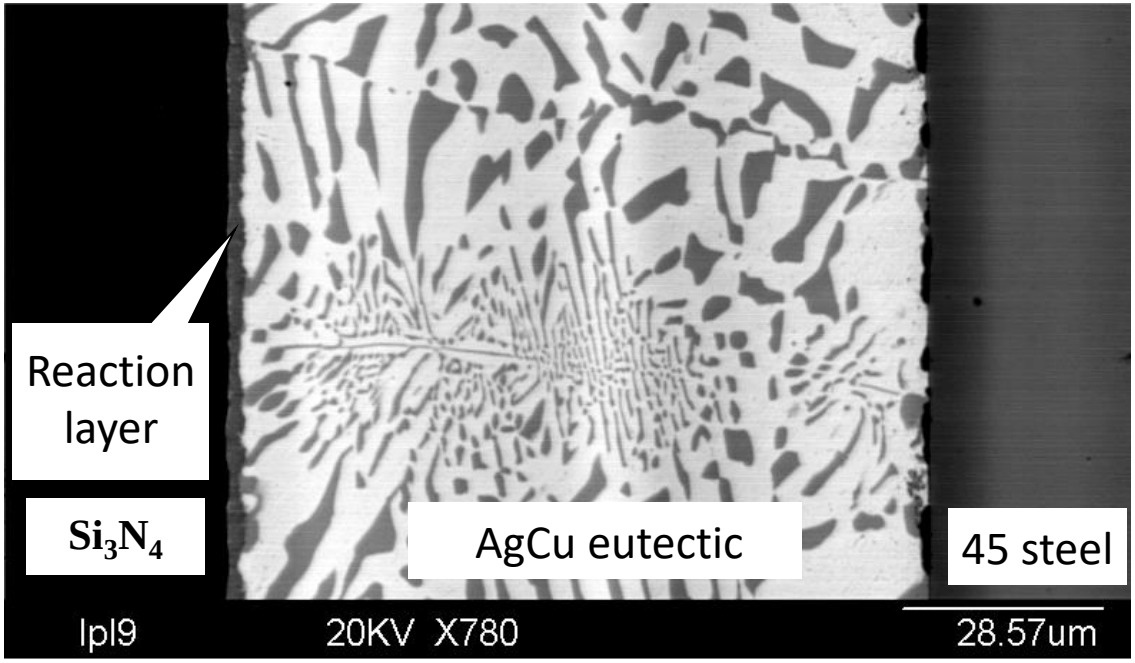


Backscattered electron image of cast AlLi alloy eutectic structure **a)** Cross section **b**) Longitudinal section

11.5 Topography contrast characteristics of backscattered electron images

- Application of atomic number contrast in backscattered electron images

The figure below shows the non-backscattered electron image of the structure of the Si_3N_4 ceramic and steel brazed joint. The solder is **AgCuTi**, and the joint structure is composed of the **TiN** reaction layer at the Si_3N_4 ceramic interface and the **AgCu** eutectic structure.

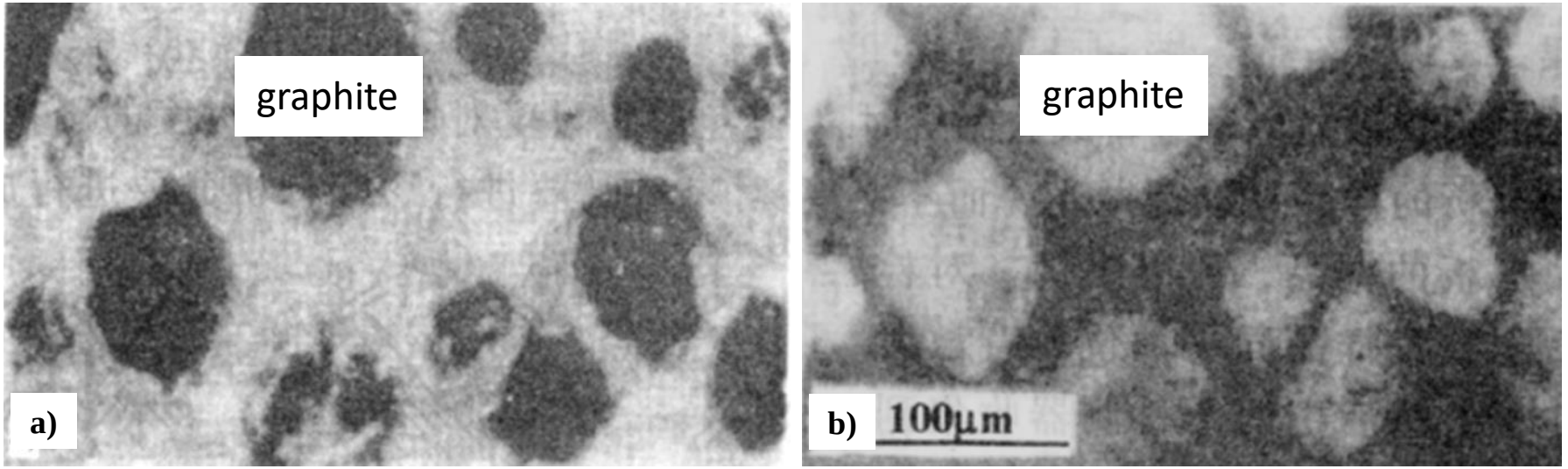


Backscattered electron image of the microstructure of the Si_3N_4 ceramic and steel brazed joint.

11.5 Topography contrast characteristics of backscattered electron images

- Absorbed electron image contrast principle and its application

The contrast of the absorbed electron image is the atomic number contrast. The yield of absorbed electrons is opposite to that of backscattered electrons, and decreases as the atomic number of the sample increases. Therefore, the contrast of the absorbed electron image is opposite to that of backscattered electrons, as shown in the figure. The resolution of absorbed electron images is lower than that of backscattered electrons.



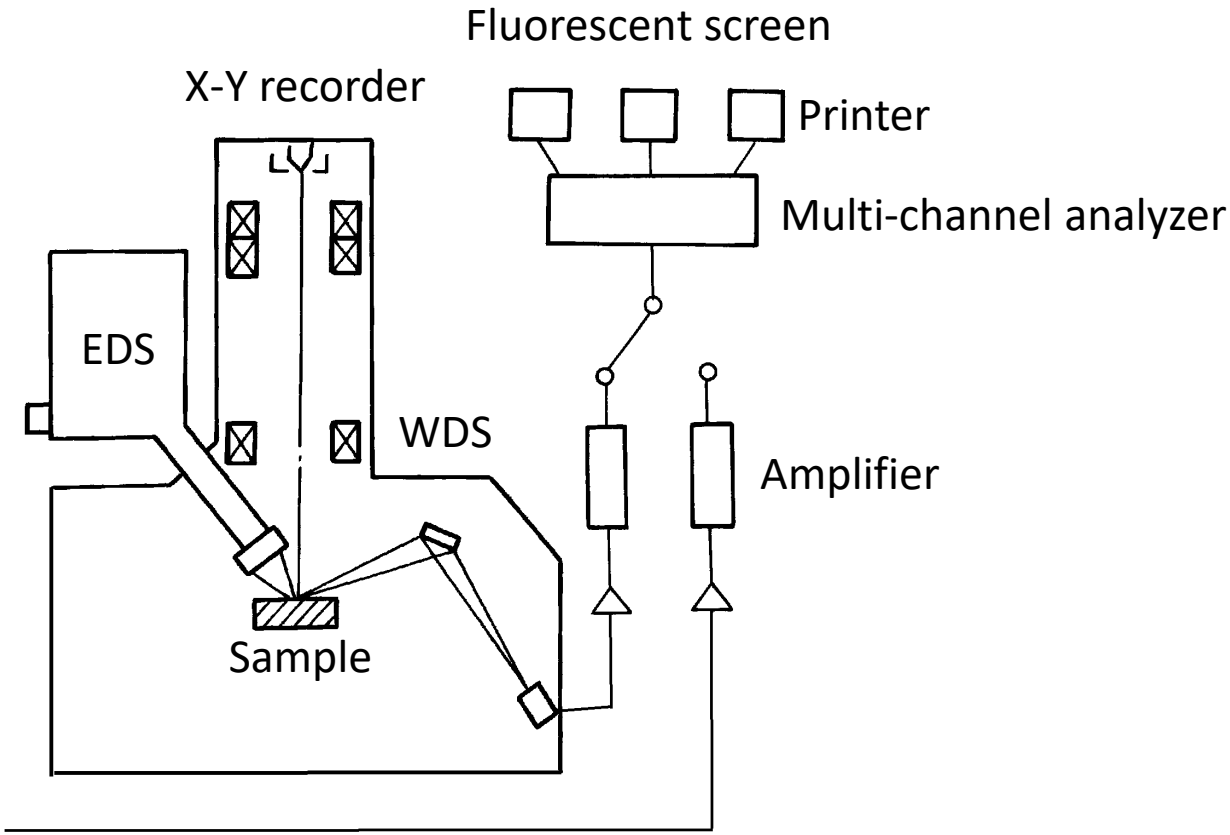
Comparison of absorption electron images and backscattered electron images of ductile iron **a)** Backscattered electron image **b)** Absorbed electron image

11.6 The structure and working principle of the electronic probe instrument

- The electron probe is a high-efficiency analytical instrument developed based on the principles of electron optics and X-ray spectroscopy.
- The electron probe uses the detector to receive the characteristic X-ray signal of the sample and perform qualitative and quantitative analysis of micro-area components.
- The overall structure of the electron probe is basically the same as that of the scanning electron microscope, but the accelerating voltage is higher (up to 50 kV), and the beam current is larger to obtain a sufficient overvoltage ratio and signal strength.
- The electron probe has both morphological observation and component analysis functions. Its main function is micro-area component analysis. The energy spectrometer, a detector that detects X-rays, can also be installed as an accessory on a transmission electron microscope, allowing the transmission electron microscope to perform in-situ analysis of microstructure, crystal structure, and chemical composition.

11.6 The structure and working principle of the electronic probe instrument

The schematic shows an electron probe. Its electron optical and vacuum systems are the same as those of a scanning electron microscope. The signal detection system of the electron probe is an X-ray spectrometer. The spectrometer that detects the wavelength of X-rays is called a wave spectrometer (WDS), and the spectrometer that detects the energy of X-rays is called an energy spectrometer (EDS).

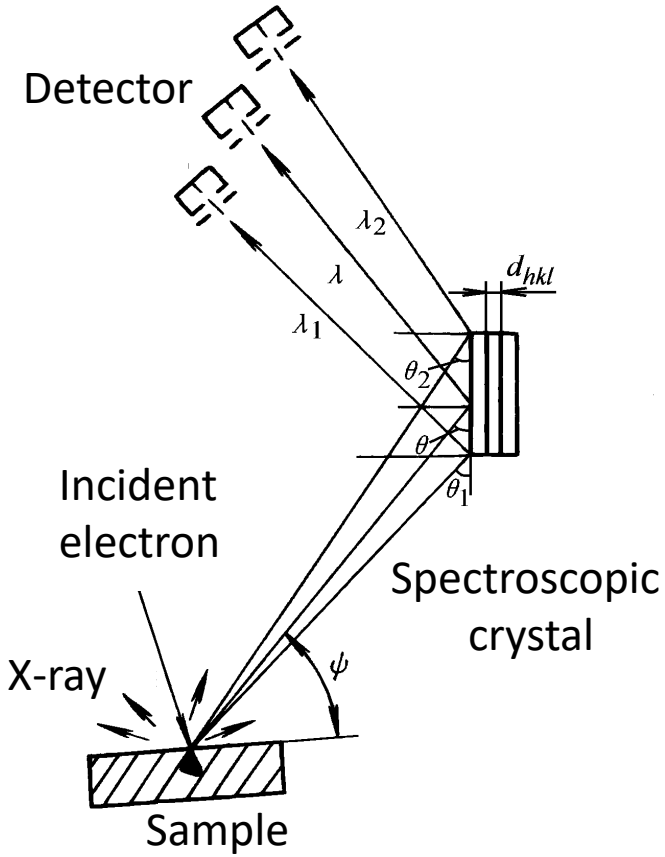


11.6 The structure and working principle of the electronic probe instrument

- Wavelength dispersion spectrometer

1. Operational principle

As shown in the figure, X-rays excited within the sample are emitted in various directions outside the sample surface, and each direction has X-rays of different wavelengths.



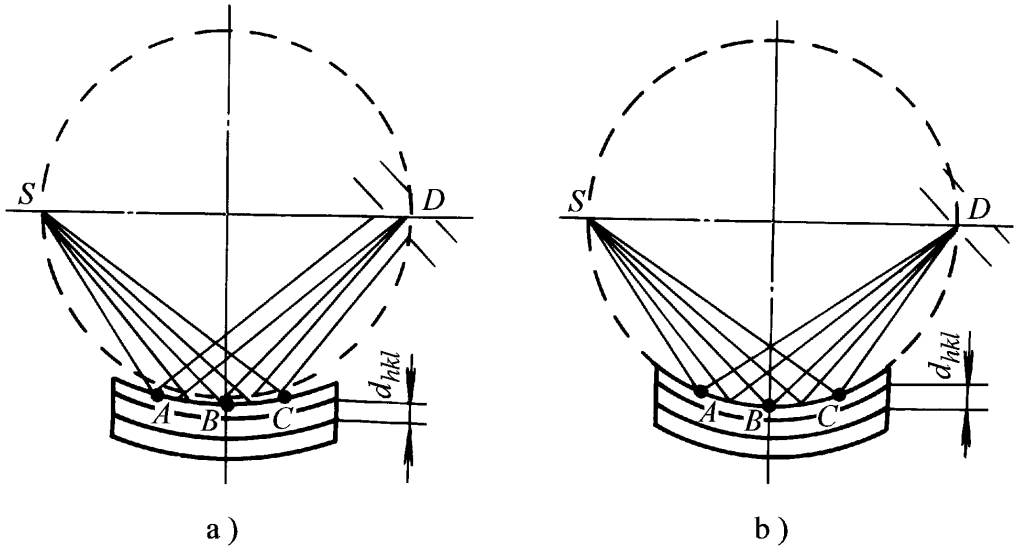
Place a crystal (with a crystal plane spacing ***d***) above the sample. According to Bragg's law $2d\sin\theta=\lambda$, it can be known that X-rays of different wavelengths λ can be detected in different 2θ directions, thereby achieving the dispersion and detection of X-rays. The detection principle is that Spectroscopic crystals with known interplanar spacing are used to detect X-rays of unknown wavelengths, but the detection efficiency of planar spectroscopic crystals is very low.

11.6 The structure and working principle of the electronic probe instrument

- Wavelength dispersion spectrometer

1. Operational principle

If the spectroscopic crystal is elastically bent and the ray source S, the spectroscopic crystal surface, and the detection window D are located on the same circle, the diffracted beam can be focused, and the detection efficiency can be improved.

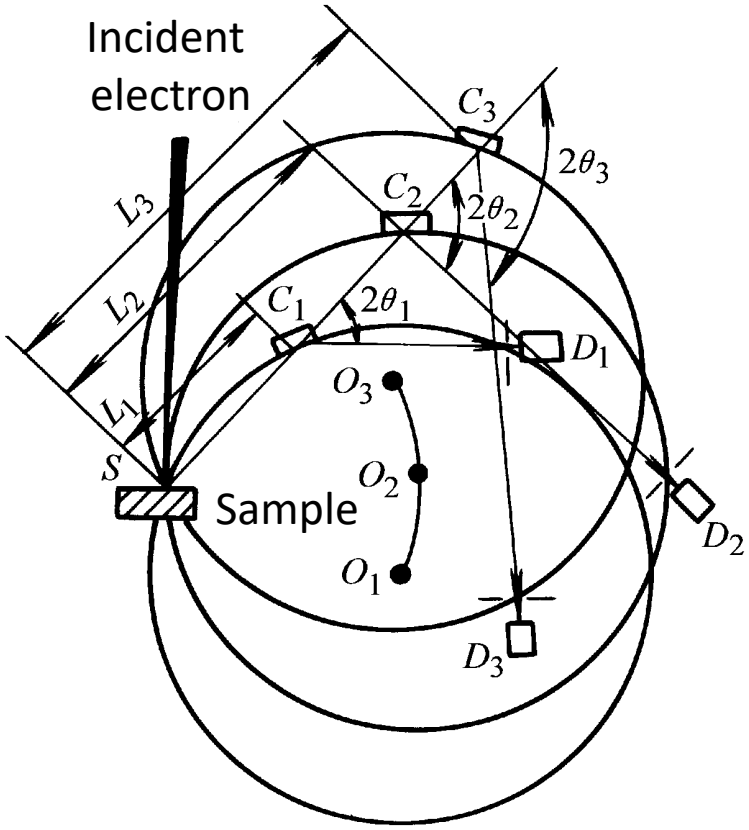


If the bending radius of the crystal is twice the radius of the focusing circle, it is called the John-type focusing method or the semi-focusing method; if the crystal bending radius is equal to the radius of the focusing circle, it is called the Johnson-type focusing method or the full-focusing method. At present, new spectrometers mostly use the full focusing method.

11.6 The structure and working principle of the electronic probe instrument

- Wavelength dispersion spectrometer

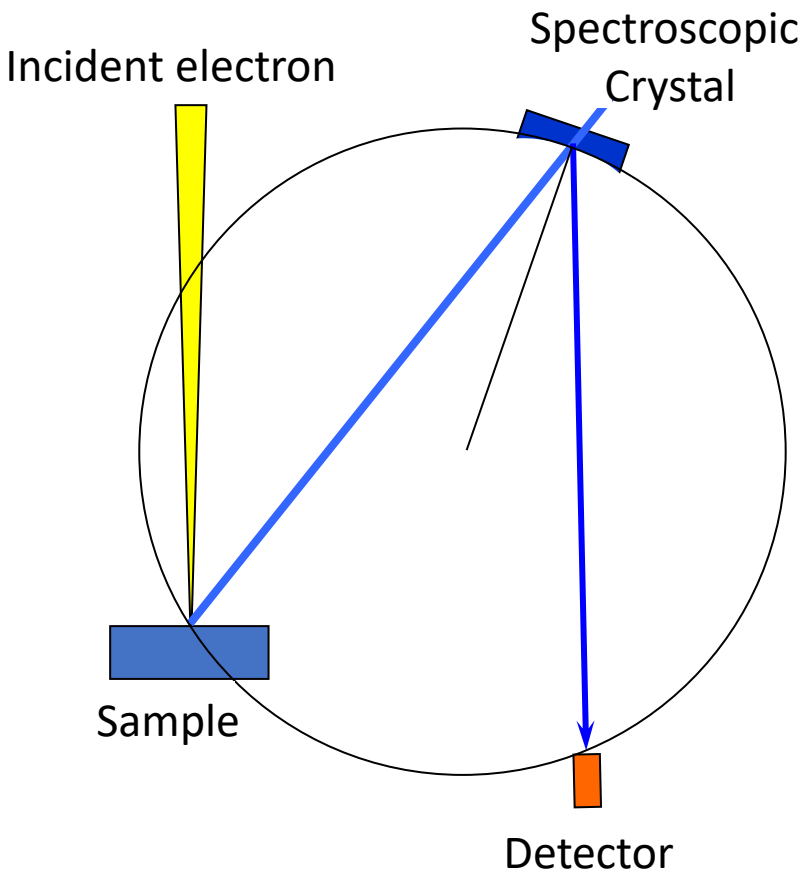
1. Operational principle



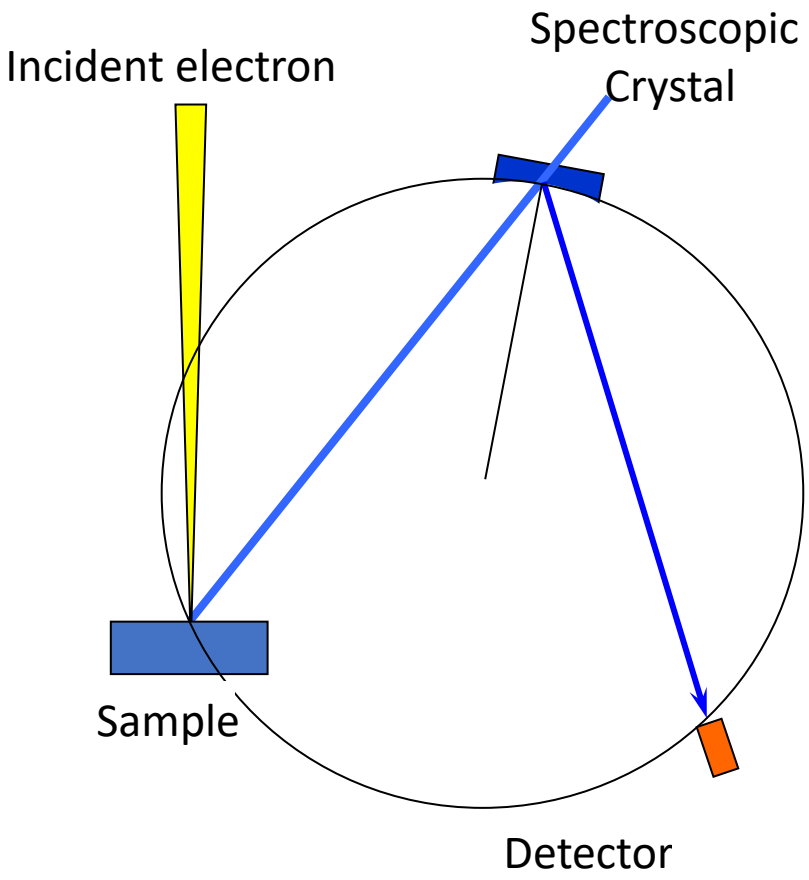
In a linear spectrometer, the spectroscopic crystal moves along a straight line. The working principle is shown in Figure—the crystal position ***L*** and the focusing circle radius ***R*** satisfy $L = 2R\sin\theta$. Find θ from the known ***L*** and ***R***, and then use the Bragg equation to calculate the characteristic X-ray wavelength λ .

The advantage of the linear spectrometer is that when detecting X-rays of different wavelengths, the exit angle ψ can be kept constant, which is beneficial to absorption correction during quantitative analysis. The disadvantage of the linear spectrometer is that its structure is complex and it occupies a large space.

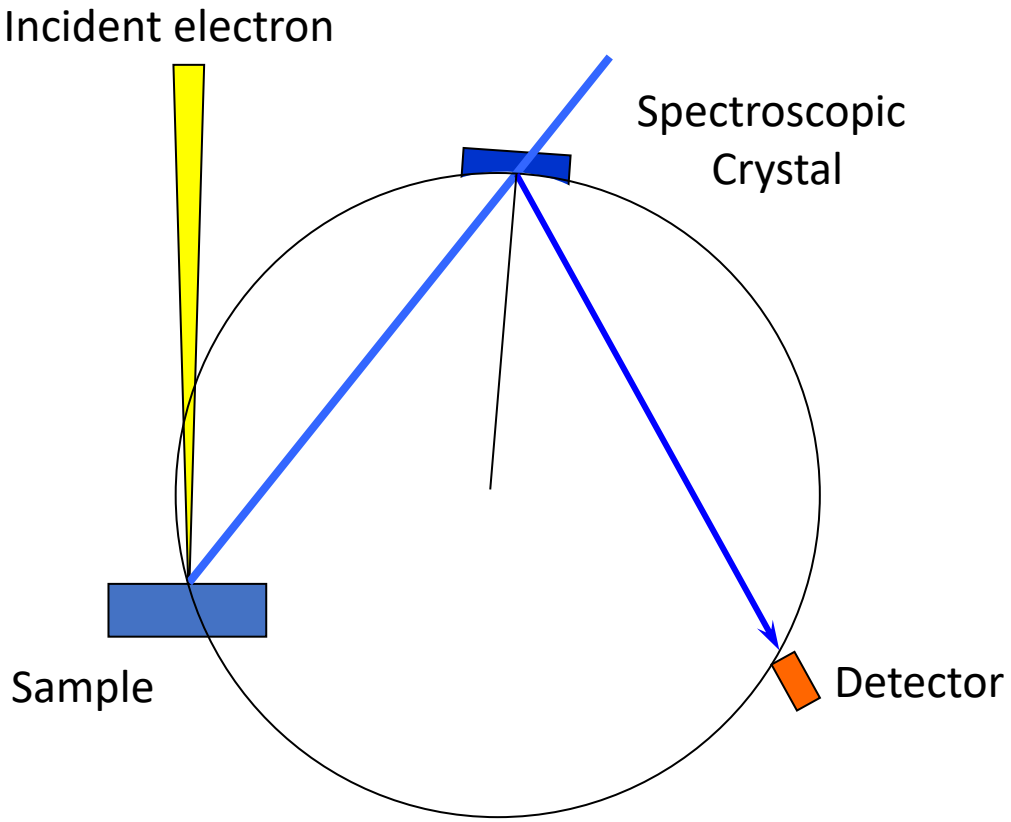
11.6 The structure and working principle of the electronic probe instrument



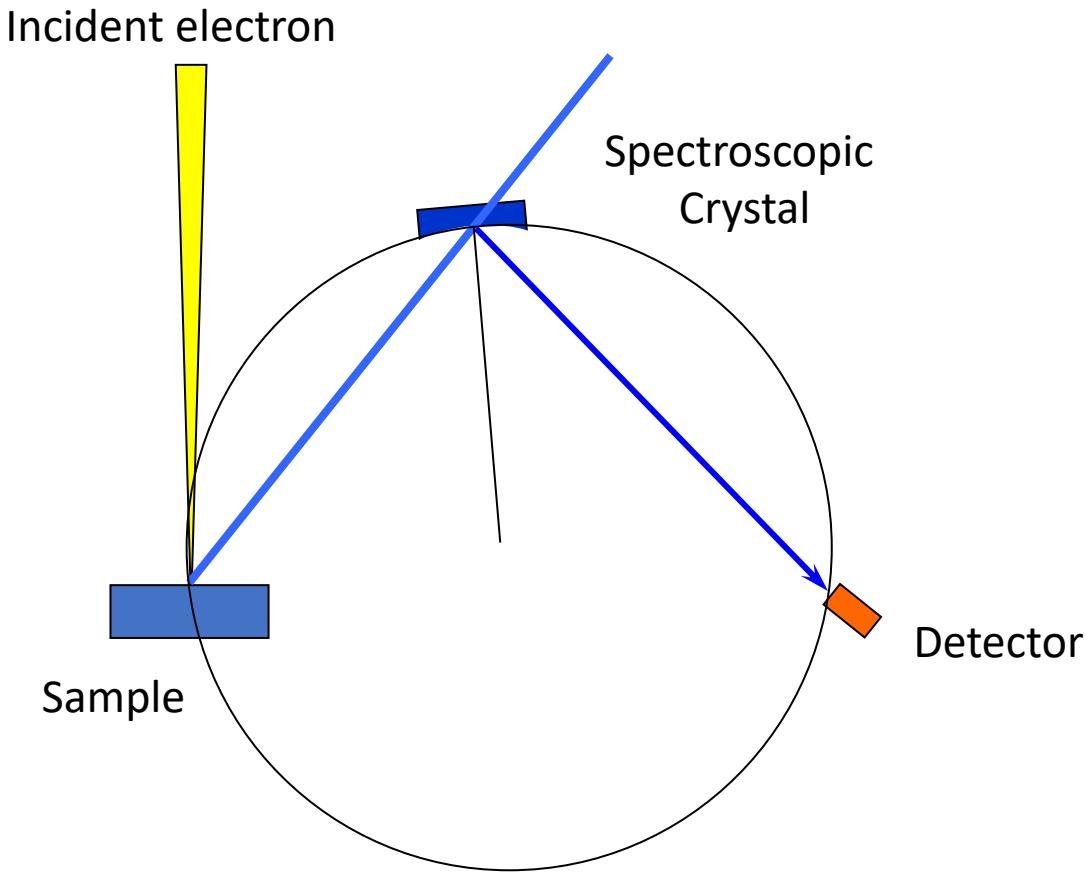
11.6 The structure and working principle of the electronic probe instrument



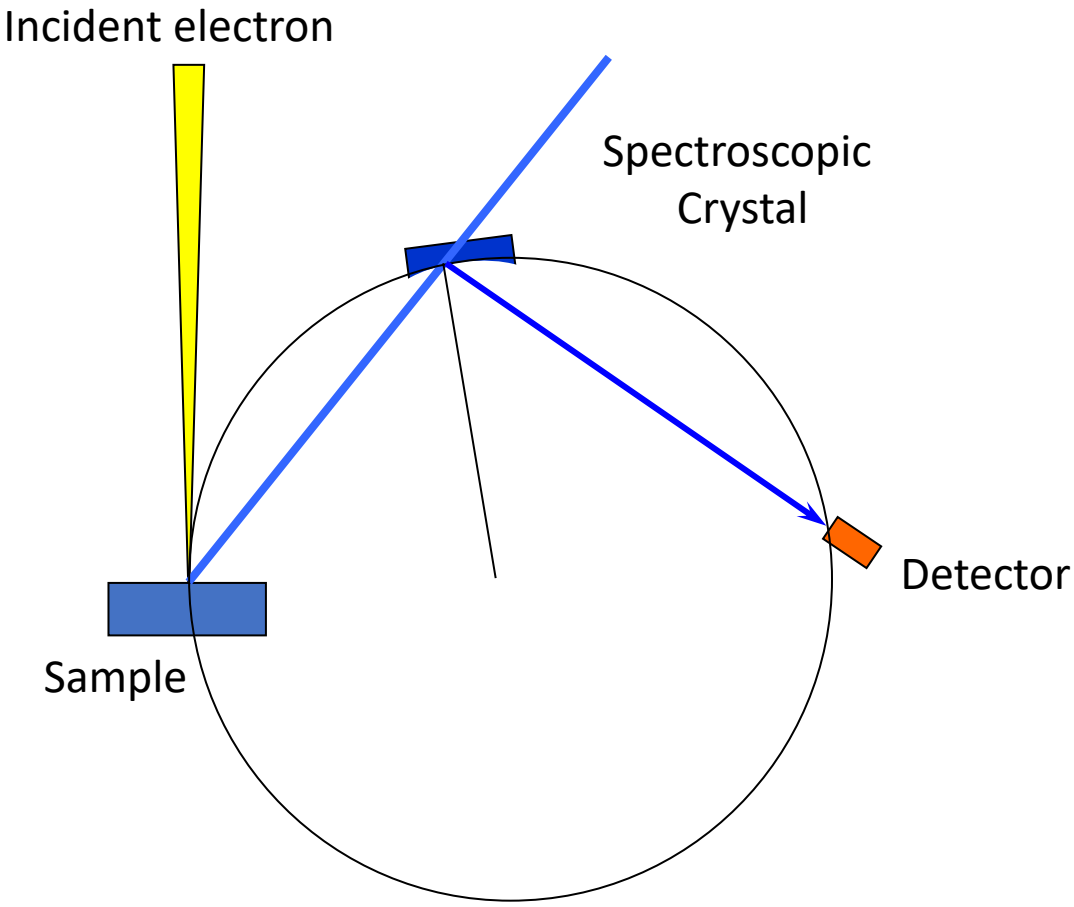
11.6 The structure and working principle of the electronic probe instrument



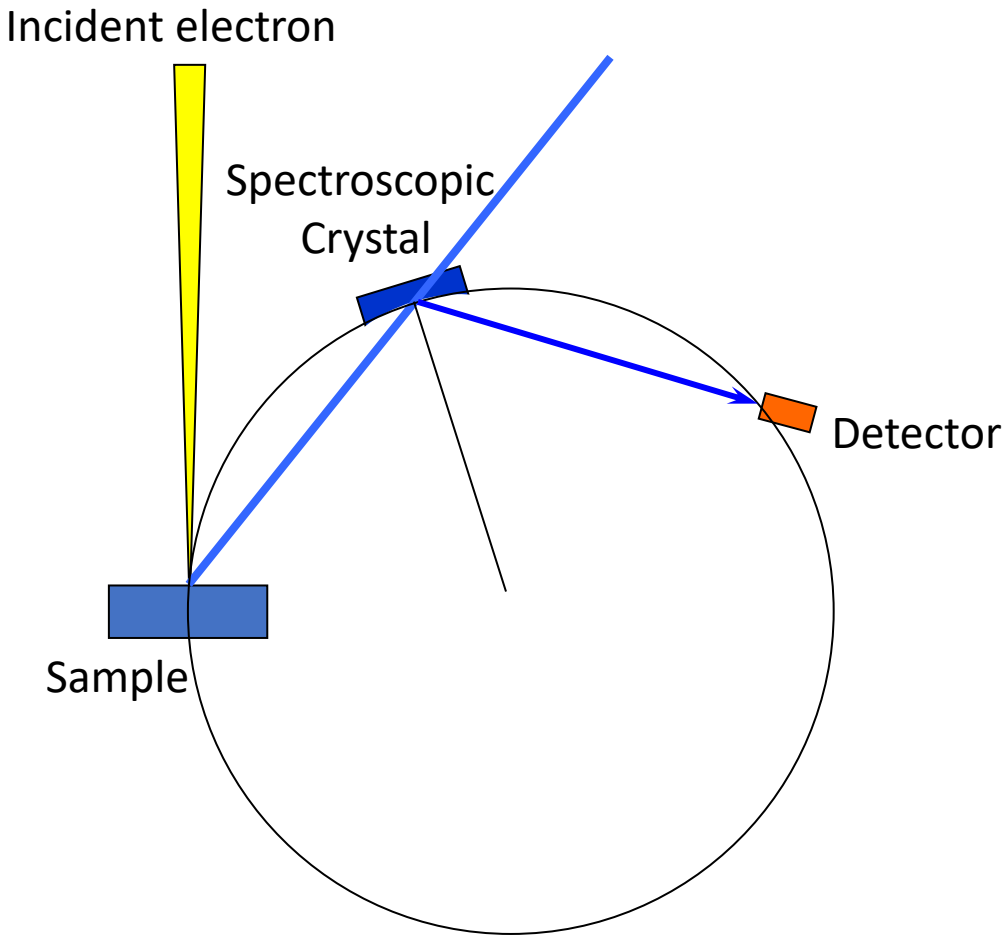
11.6 The structure and working principle of the electronic probe instrument



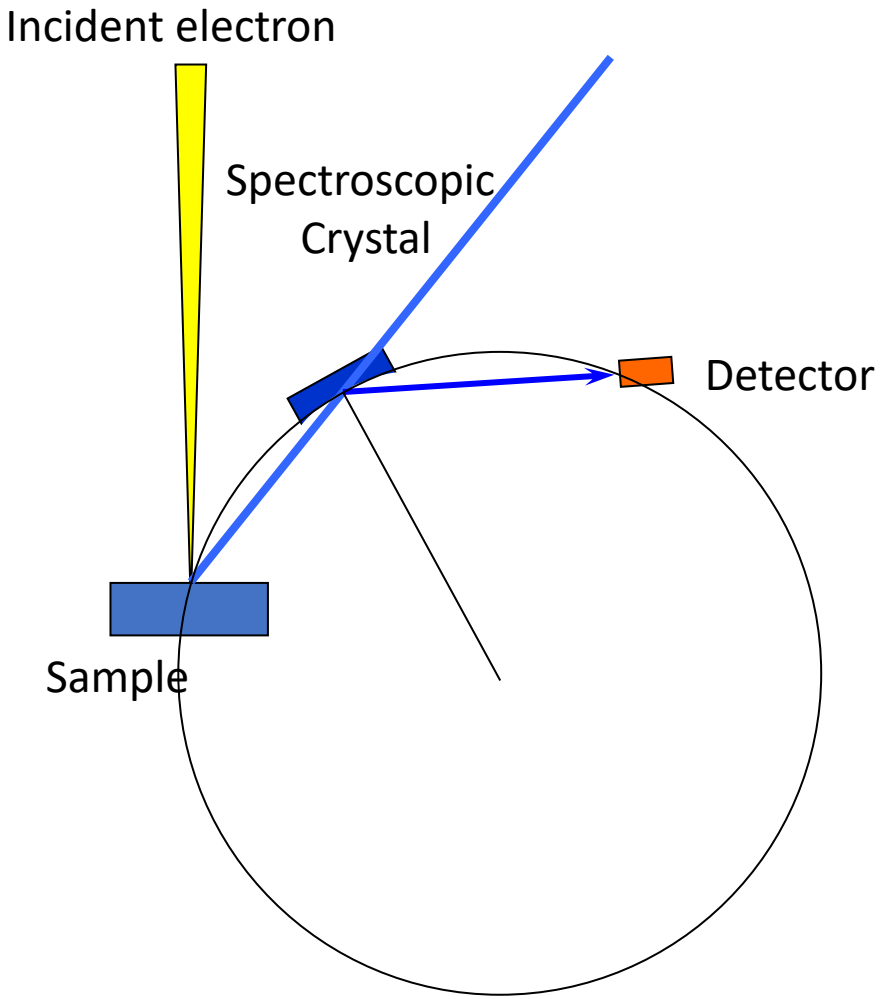
11.6 The structure and working principle of the electronic probe instrument



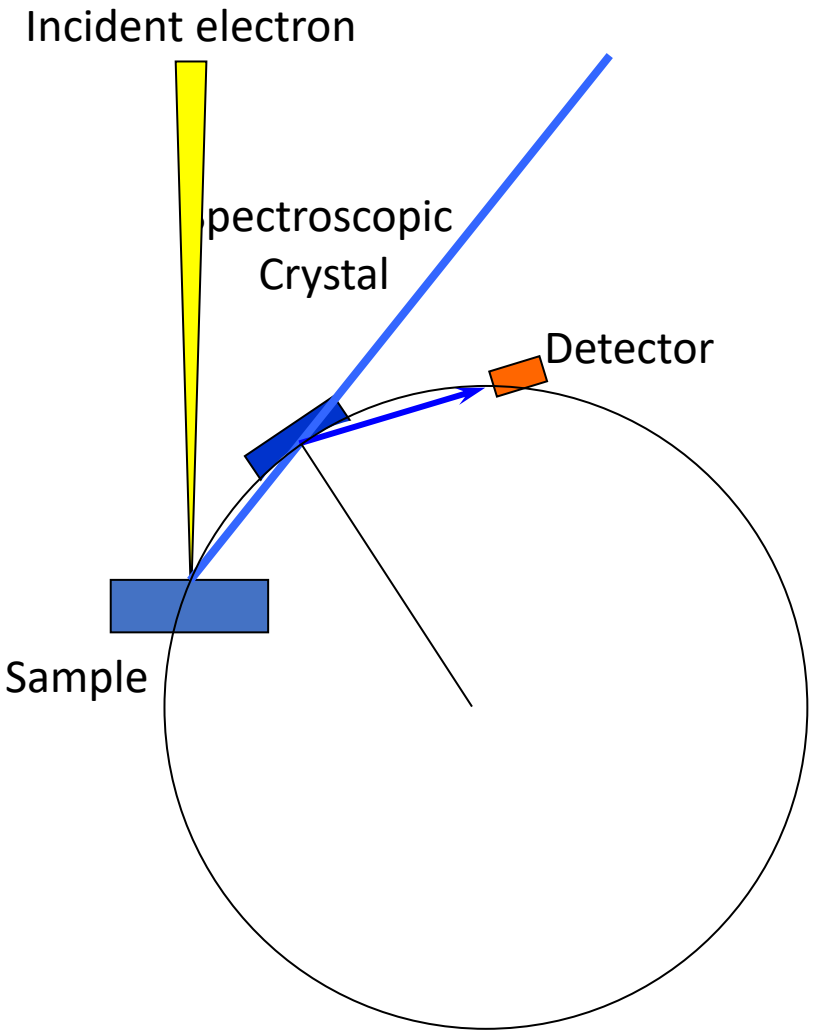
11.6 The structure and working principle of the electronic probe instrument



11.6 The structure and working principle of the electronic probe instrument



11.6 The structure and working principle of the electronic probe instrument

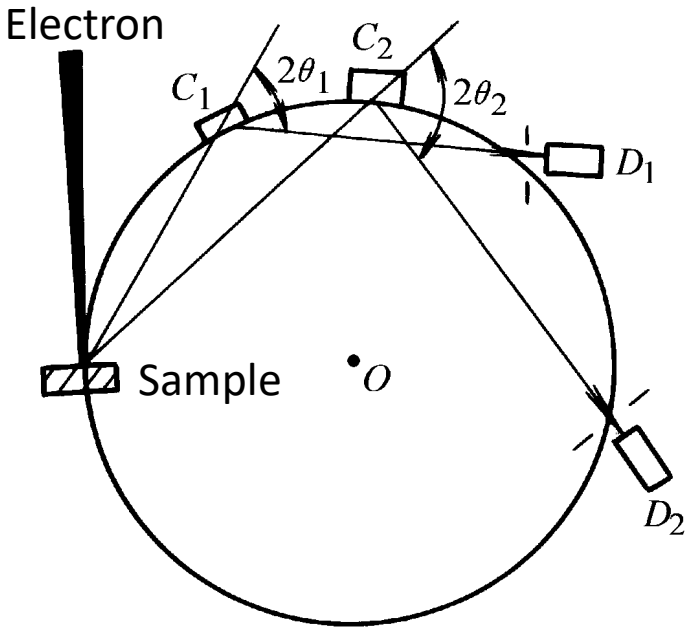


11.6 The structure and working principle of the electronic probe instrument

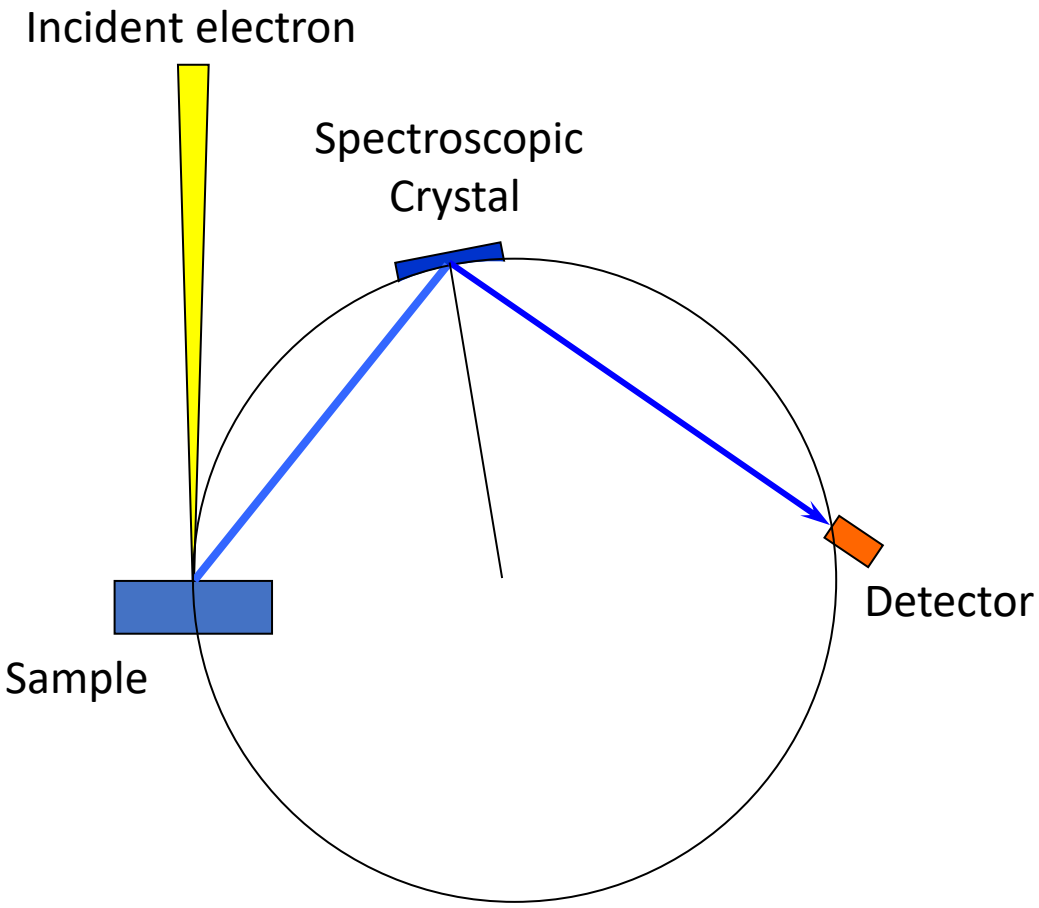
- Wavelength dispersion spectrometer

1. Operational principle

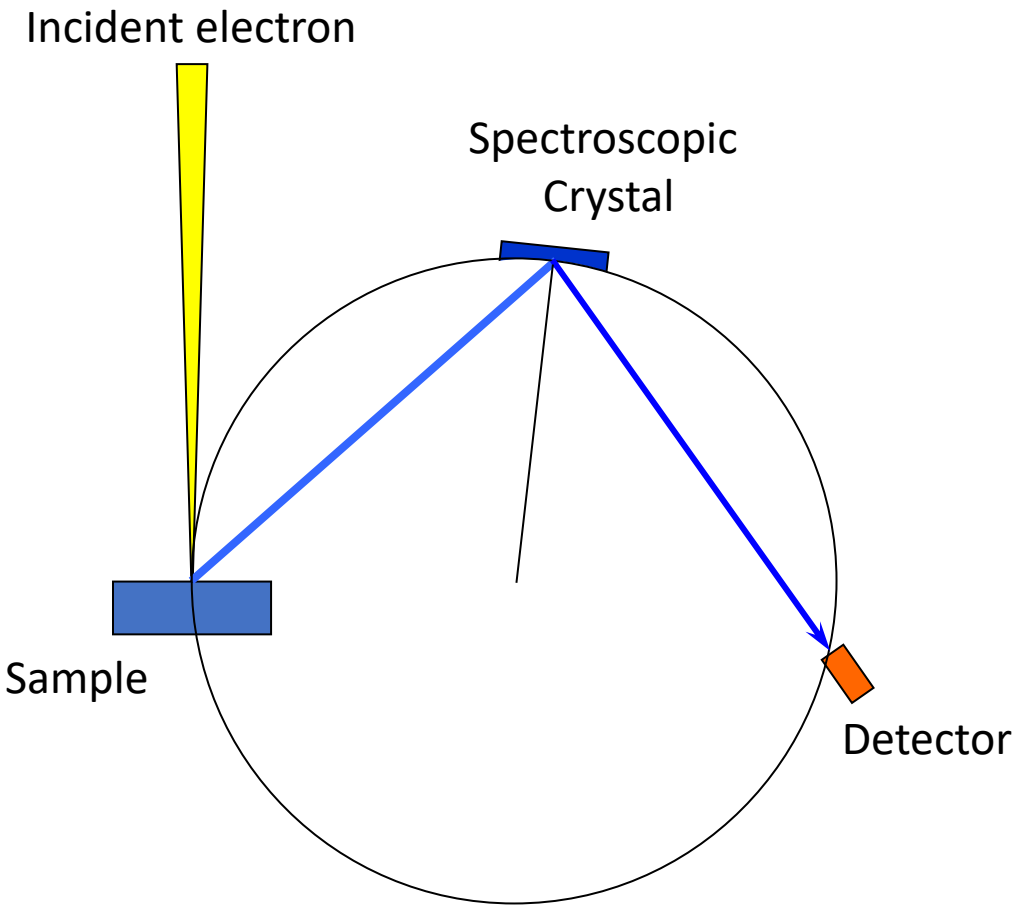
When the rotary spectrometer detects X-rays of different wavelengths, the spectroscopic crystal moves on the focusing circle and the detector moves on the same circle at a corresponding angular speed of 2 times. The advantage of the rotary spectrometer is that it has a simple structure, but the disadvantage is that when receiving X-rays of different wavelengths, the exit angle ψ will change, which is not conducive to absorption correction during quantitative analysis.



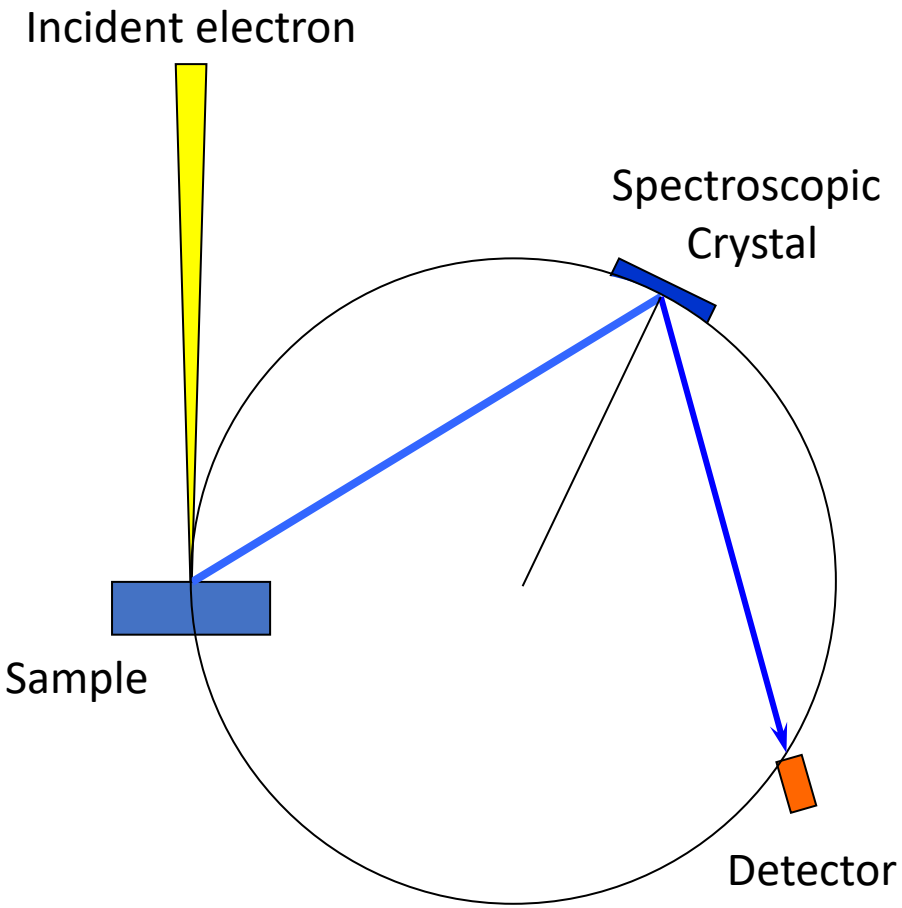
11.6 The structure and working principle of the electronic probe instrument



11.6 The structure and working principle of the electronic probe instrument



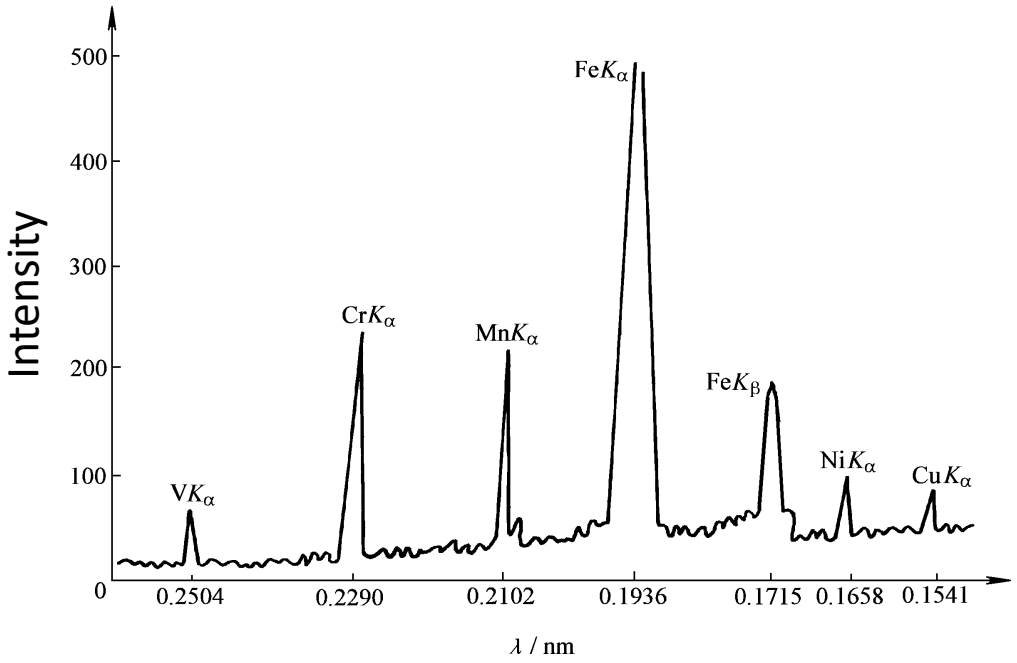
11.6 The structure and working principle of the electronic probe instrument



11.6 The structure and working principle of the electronic probe instrument

- Wavelength dispersion spectrometer

2. Analytical method



The spectroscopic crystal is continuously moved, and the spectrometer continuously detects and receives X-rays of different wavelengths.

The wavelength of the characteristic peak of the X-ray spectrum, as shown in the figure, corresponds to the atomic number of the element, and the peak intensity corresponds to the content of the element.

X-ray spectrum of alloy (**0.62Si,1.11Mn,0.96Cr,0.56Ni,0.26V,0.24Cu**) analysis

11.6 The structure and working principle of the electronic probe instrument

- Wavelength dispersion spectrometer

2. Analytical method

- Determine the sample analysis point. Use a special optical microscope equipped with an electron probe to accurately focus the analysis point on the circumference of the focusing circle. At this time, the analysis point is exactly at the center of the microscope eyepiece scale.

- The selection of spectroscopic crystals has a limited range of X-ray wavelengths that spectroscopic crystals can detect with a certain crystal plane spacing. Therefore, an electron probe is usually equipped with 3 to 5-channel spectrometers. Each spectrometer is equipped with 2 spectroscopic crystals, which can cover Be ~ U characteristic X-ray wavelength range of all elements. During analysis, the appropriate spectroscopic crystal is selected based on the wavelength of the characteristic X-ray of the element.

11.6 The structure and working principle of the electronic probe instrument

- Wavelength dispersion spectrometer

2. Analytical method

The table lists commonly used spectroscopic crystals and their applicable wavelength ranges.

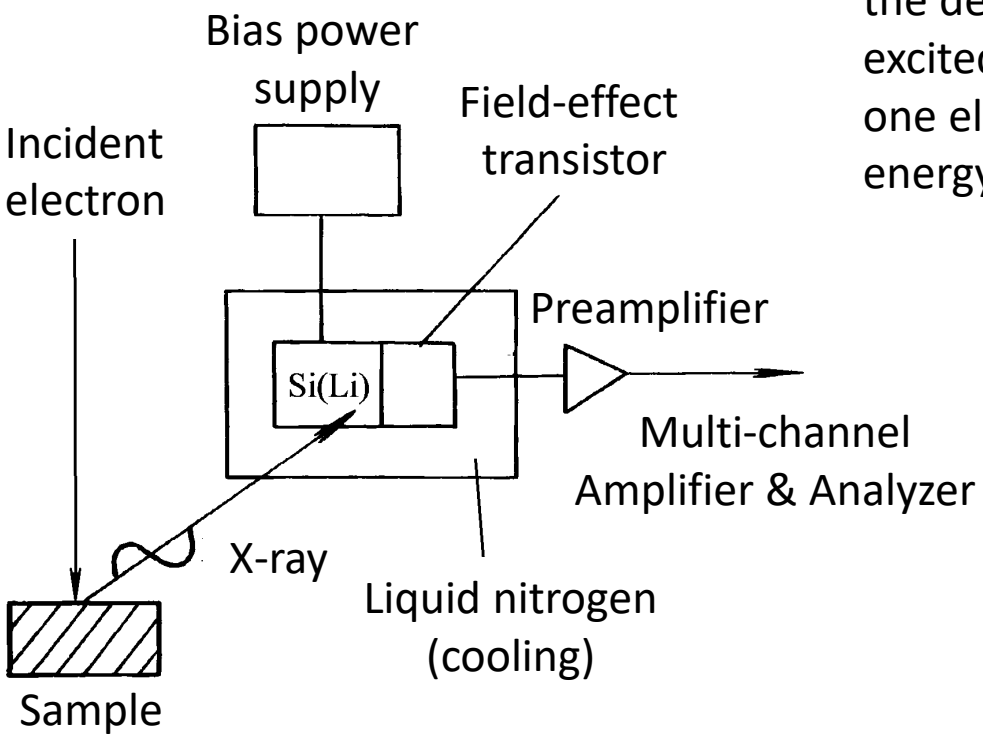
Common spectroscopic crystal data

Crystal	Plane	2*d*/nm	Wavelength λ/nm
LiF	(200)	0.40267	0.08~0.38
SiO_2	(10-11)	0.66862	0.11~0.63
PET	(002)	0.874	0.14~0.83
RAP	(001)	2.6121	0.20~1.83
KAP	(10-10)	2.6632	0.45~2.54
TAP	(10-10)	2.59	0.61~1.83

11.6 The structure and working principle of the electronic probe instrument

- Energy dispersion spectrometer

1. Operational principle



The energy spectrometer uses Si (Li) crystal as the detector. When X-rays with energy ΔE enter the detector, N electron-hole pairs will be excited, and the energy required to generate one electron-hole pair is ε. Then, the X-ray energy is:

$$\Delta E = N\varepsilon$$

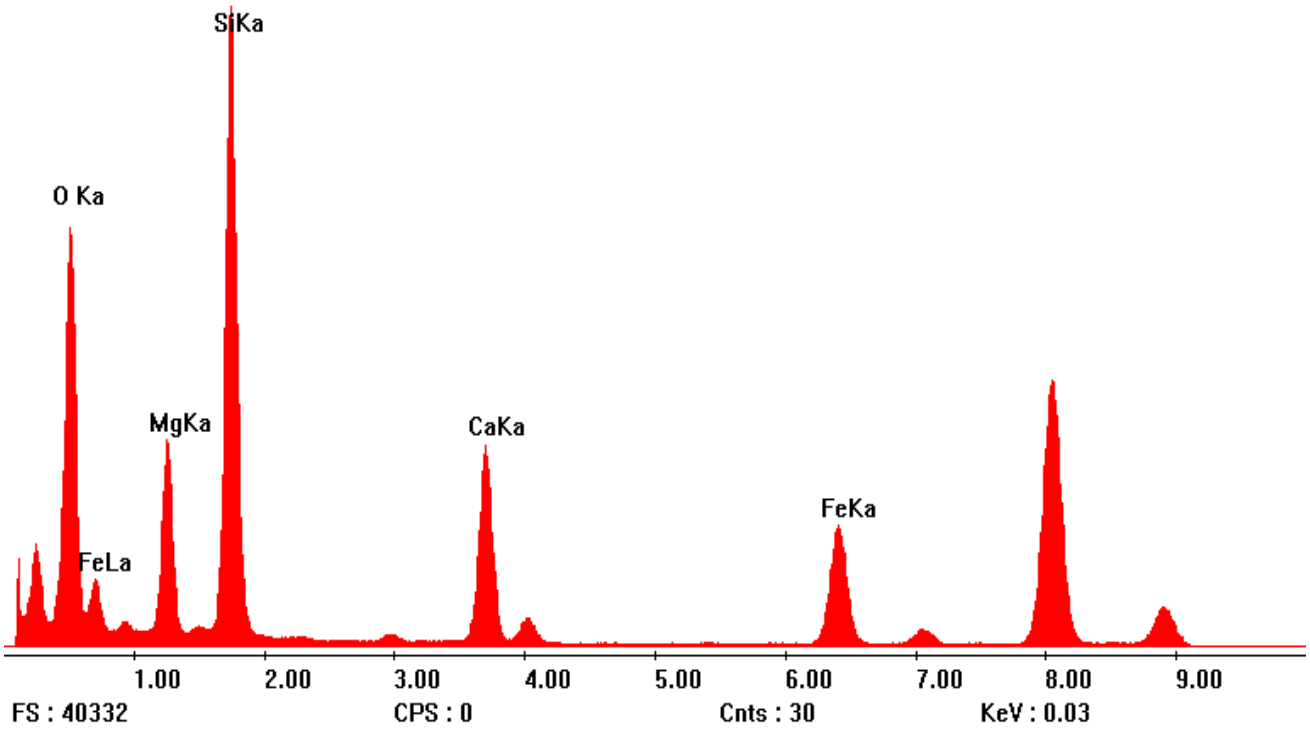
As long as the number of electron-hole pairs is detected, the energy of the X-photons can be calculated and then classified using a multi-channel pulse height analyzer to draw a spectrum based on energy dispersion.

11.6 The structure and working principle of the electronic probe instrument

- Energy dispersion spectrometer

1. Operational principle

As shown in the figure below, the X-ray energy spectrum of oxide is shown. The abscissa is the energy and the ordinate is the intensity (or count).



11.6 The structure and working principle of the electronic probe instrument

- Energy dispersion spectrometer

2. Characteristics of component analysis with energy spectrometer

Compared with wave spectrometers, energy spectrometers have the following advantages:

- The detection efficiency of the energy spectrometer is high because the energy spectrometer probe can be placed near the sample analysis point, which can effectively increase the collection solid angle.
- Energy spectrometer analysis is fast because the energy spectrometer can detect and receive X-rays of different wavelengths simultaneously.
- The energy spectrometer has better stability and repeatability because it has no mechanical transmission device.
- The energy spectrometer has no special requirements for the sample surface. It can also be used for composition analysis of rough surfaces because the energy spectrometer does not require sample focusing.

11.6 The structure and working principle of the electronic probe instrument

- Energy dispersion spectrometer

2. Characteristics of component analysis with energy spectrometer

Compared with wave spectrometers, energy spectrometers have the following limitations:

- The energy resolution of the energy spectrometer is low. The Si(Li) detector is about 130 eV, while the energy resolution of the wave spectrometer is 5~15 eV. Low energy resolution broadens characteristic peaks, resulting in reduced peak heights and overlapping peaks with similar energies.

- The elemental analysis range of the energy spectrometer is small because the energy spectrometer detector window causes the absorption of low-energy X-rays. The element analysis range of the energy spectrometer was Na~U in the early stage, and is currently B and above elements, while the analysis range of the wave spectrometer is Be ~ U.

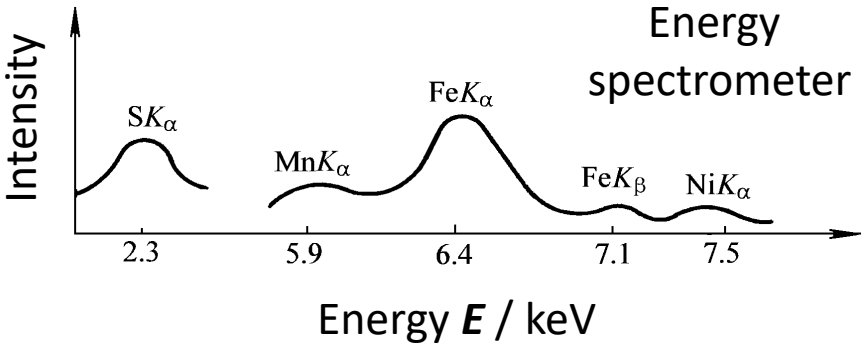
- The Si(Li) crystal of the energy spectrometer must be used at low temperatures, so it needs to be cooled with liquid nitrogen.

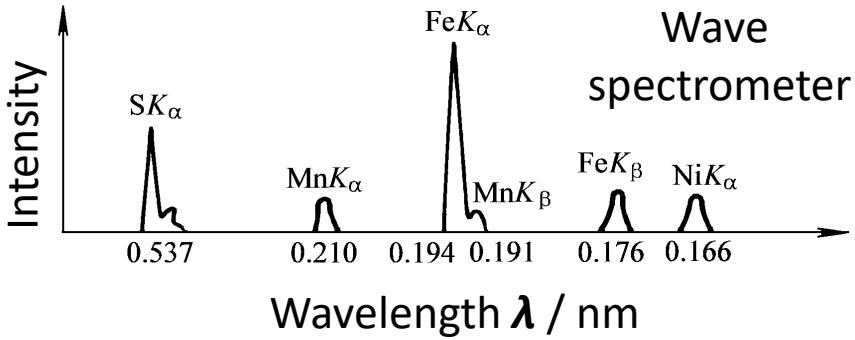
11.6 The structure and working principle of the electronic probe instrument

- Energy dispersion spectrometer

2. Characteristics of component analysis with energy spectrometer

The same sample was analyzed using an energy spectrometer and a wave spectrometer. The spectrum is shown in the figure.





11.7 Analysis methods and applications of electronic probe instruments

- Qualitative analysis

1. Point analysis

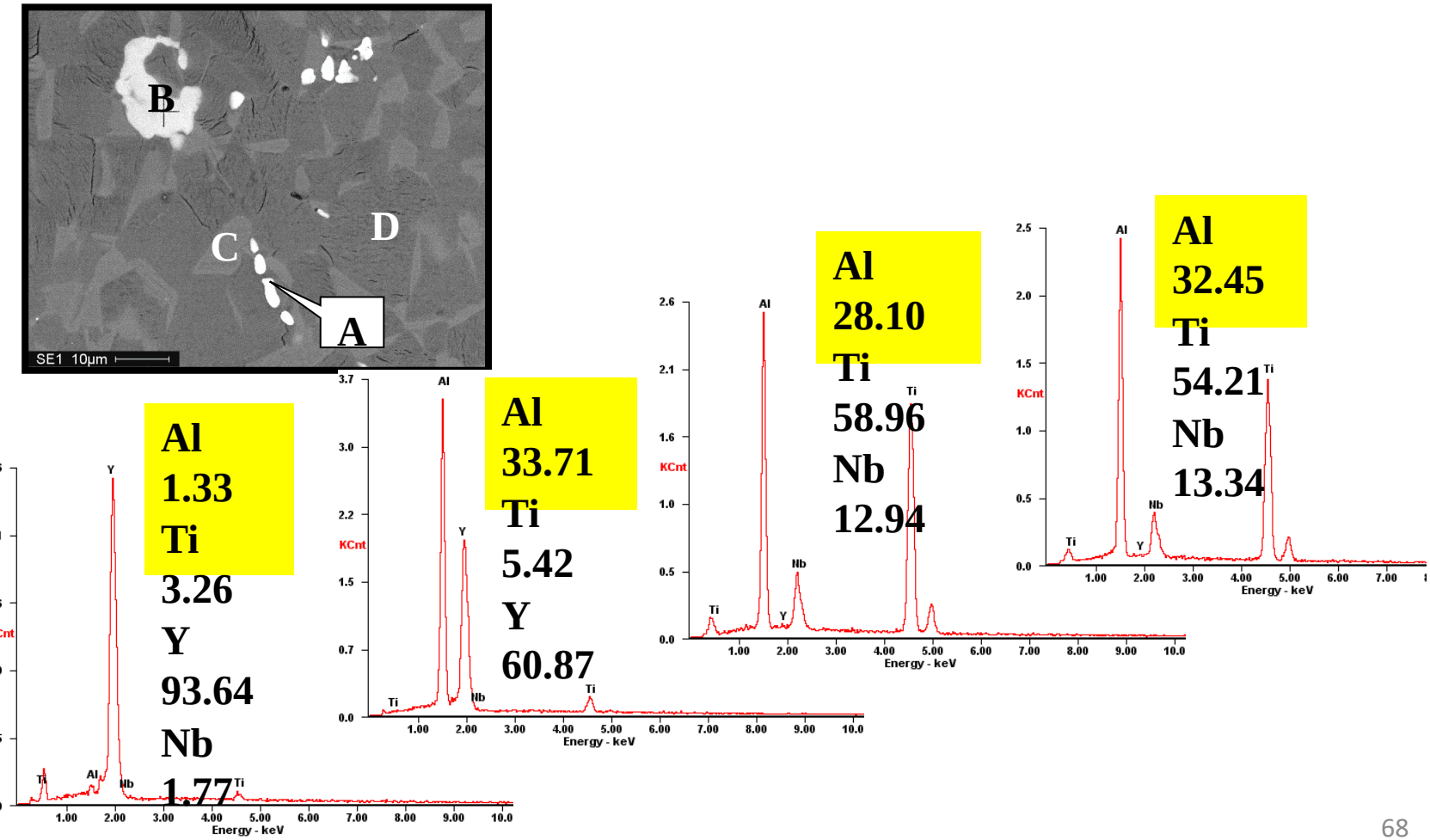
The electron beam is fixed on the selected sample analysis point, and the spectrometer continuously changes the position of the spectroscopic crystal and continuously receives X-rays of different wavelengths to obtain the full spectrum of X-rays at the analysis point or use an energy spectrometer to directly collect the X-rays at the analysis point.

According to the wavelength (or energy) of the characteristic X-ray in the spectrum, the type of element contained in the analysis point is determined. Point analysis is mainly used for elemental composition analysis of physical phases and, combined with quantitative analysis results, provides a basis for phase identification.

11.7 Analysis methods and applications of electronic probe instruments

- Qualitative analysis

1. Point analysis

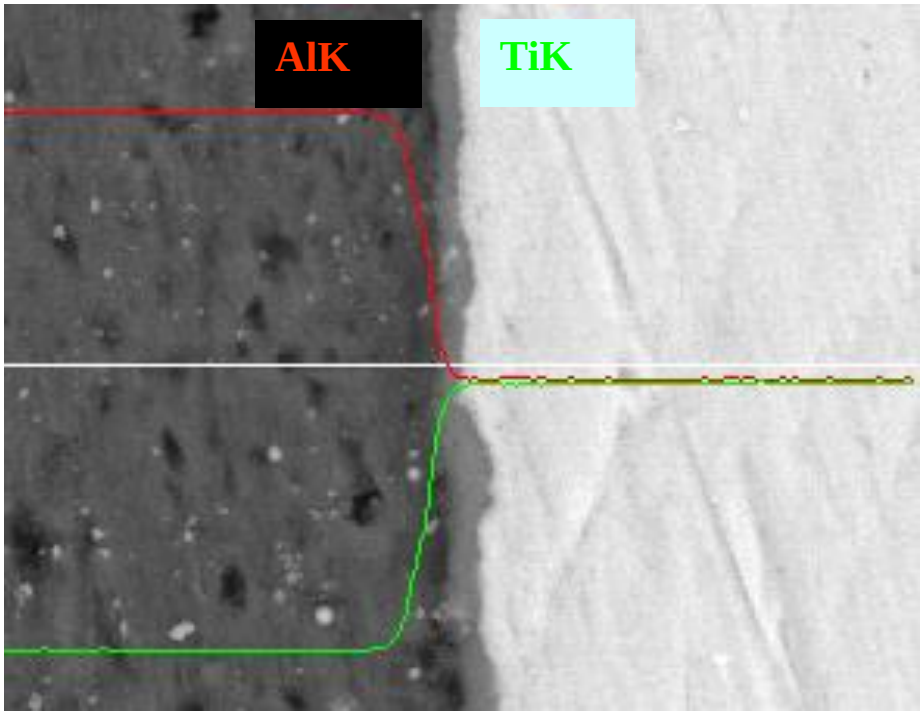


11.7 Analysis methods and applications of electronic probe instruments

- Qualitative analysis

2. Line analysis

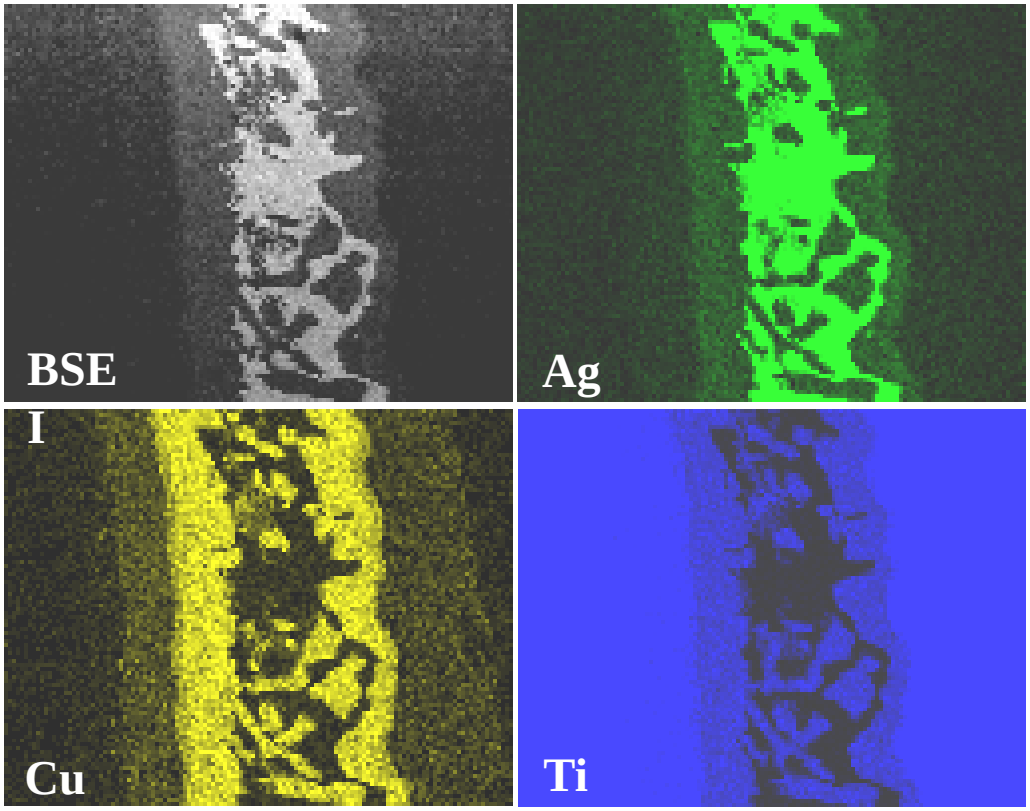
The electron beam is scanned along the selected straight line on the sample surface, and the spectrometer receives the characteristic X-ray of the element being measured. The concentration change curve of the element on the straight line of the sample can be obtained. It is mainly used to study elements at various interfaces.



11.7 Analysis methods and applications of electronic probe instruments

- Qualitative analysis

3. Area analysis



The electron beam is scanned in a selected area on the sample surface, and the spectrometer receives the characteristic X-rays of the selected element. The concentration distribution image of the element in this area can be obtained, which is mainly used to study the concentration distribution of elements in microstructure. It can also be used to display the morphology and distribution of group phases.

11.7 Analysis methods and applications of electronic probe instruments

- Introduction to Quantitative Analysis

The basis of quantitative analysis is the intensity of elemental characteristic X-rays, and its methods can be divided into the standard sample method and the standard-free method. The steps of the standard sample method are as follows:

1. Accurately measure the characteristic X-ray intensities I_0 and I of the detected elements of the standard sample and the sample under the same conditions. This intensity is the net intensity after background subtraction, overlapping peak stripping, and dead time correction. Calculate the intensity ratio K: $K = I / I_0$

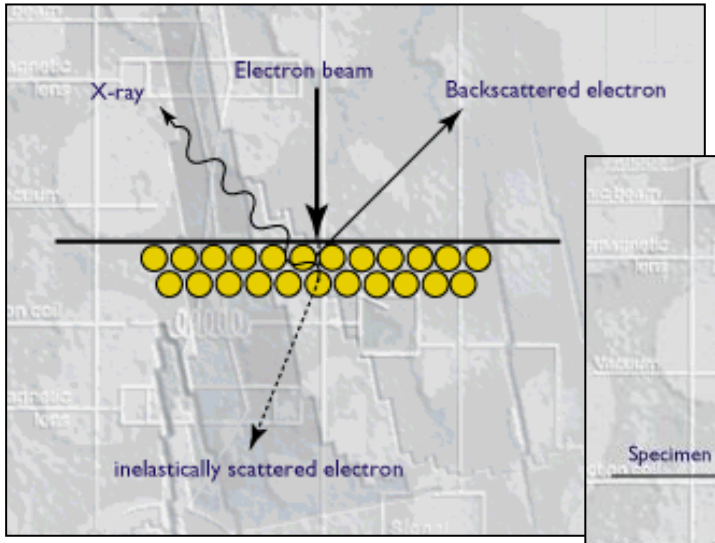
2. Convert the intensity ratio K into the mass fraction w of the element, and then correct the factors that affect the characteristic X-ray intensity. Under the conditions of pure standard sample: $w = ZAFK$

In the formula, Z is the atomic number correction; A is the absorption correction; F is the fluorescence correction.
For elements with $Z > 10$ and $w > 10\%$, the relative error is $\leq \pm 5\%$; its analysis area is less than $10\mu m^3$, if the material density is $10 g/cm^3$, the mass of the analysis area is only $10^{-10} g$.

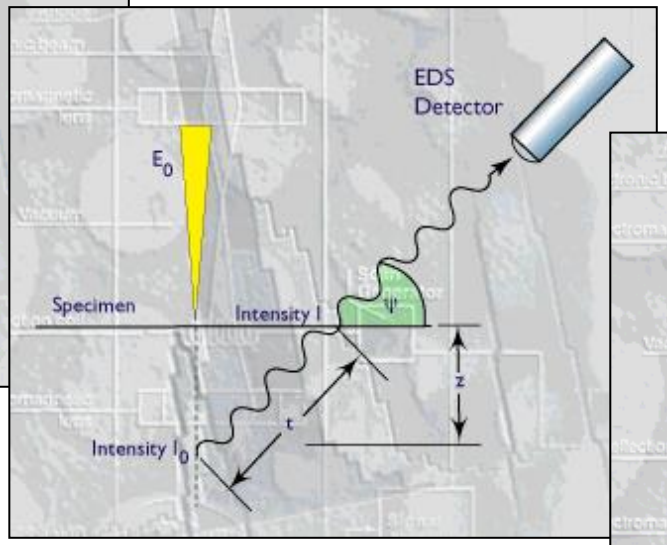
11.7 Analysis methods and applications of electronic probe instruments

- Introduction to Quantitative Analysis

The meaning of ZAF correction is shown in the figure below.

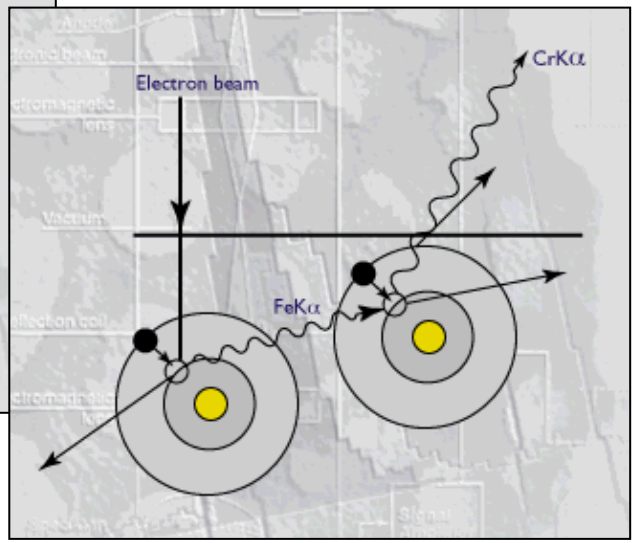


***Z* correction**



***A* correction**

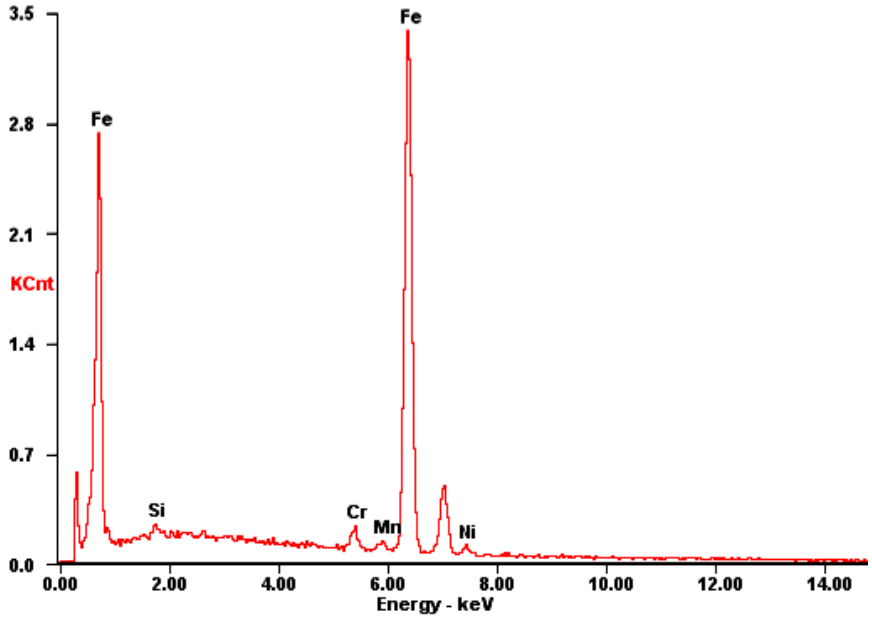
***F* correction**



11.7 Analysis methods and applications of electronic probe instruments

- Introduction to Quantitative Analysis

The figure below shows the analysis results of low alloy steel energy spectrometer.



Element	Wt%	At%
SiK	00.49	00.97
CrK	02.29	02.45
MnK	01.12	01.14
FeK	93.08	92.60
NiK	03.01	02.85
Matrix	Correction	ZAF

Example of quantitative analysis results of low alloy steel with energy spectroscopy