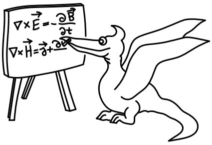

Serial: Window into the Nanoverse

Introduction

As one of the pillars of new knowledge, Richard Feynman established the possibility of exploring phenomena at the atomic level in his legendary article “There’s Plenty of Room at the Bottom.”[1] The fulfillment of this grand goal did not take long; only 22 years after the publication of this article, the first Scanning Tunneling Microscope (STM) was constructed, enabling the imaging of conductive materials with atomic resolution. This imaging method is extremely useful in countless applications, but it only allows measurements on conductors or semiconductors. It utilizes the quantum phenomenon of tunneling, which unfortunately cannot be explained in a few pages as it requires knowledge of quantum physics. We provide the reader with a literature reference at the very least [2].

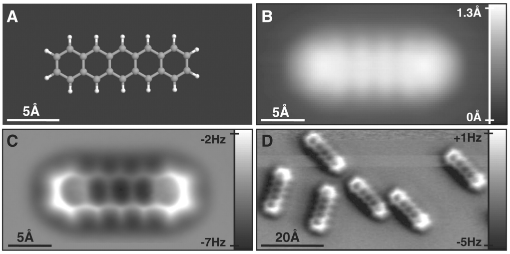

Figure 1: Image of naphthalene adsorbed on a surface captured using both AFM and STM.[3] Image A is a model of the molecule, image B was captured using an STM tip, and C and D are images captured using AFM.

From a research perspective, the ability to capture images with atomic resolution was a massive step forward. However, STM operating conditions entirely exclude the study of the atomic structure of membranes and organic molecules, which are typically non-conductive and exist only in aqueous environments. Atomic Force Microscopy (AFM) was developed for these and other purposes. As we will show, AFM operates on a principle much more intuitive to the reader and complements STM, together forming an exceptionally useful toolkit for surface studies. Nevertheless, the path to practical realization was somewhat more difficult. Interestingly, both STM and AFM originated at the Swiss branch of IBM, with the invention of STM earning Gerd Binnig and Heinrich Rohrer the Nobel Prize in 1986. The smallest stop-motion film was also created using an AFM tip.[4]

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[1] `https://web.pa.msu.edu/people/yang/RFeynman_plentySpace.pdf`

[2] `https://link.springer.com/book/10.1007/978-3-030-16649-6`

[3] `https://www.science.org/doi/10.1126/science.1176210`

[4] `https://youtu.be/oSCX78-8-q0`

Operating Principle

Phenomenological Approach

Without considering any physical phenomena, we can imagine an AFM microscope as a person given a specific task: to map a surface that is perfectly flat but has regions of varying temperatures. One would proceed by moving a finger above the surface and tactually identifying warmer and cooler areas. The closer the finger moves to the surface, the more certain the judgment, yet one could never get too close to avoid getting burned. By "feeling around" in this way, we could undoubtedly obtain a surface map that might be useful for someone.

In principle, an AFM microscope works very similarly, with the difference that instead of a finger, it has a perfectly sharp tip—down to a single atom at its apex—and it maps the surface morphology rather than temperature. Since repulsive forces acting between two atoms depend on their distance, maintaining the tip at a constant height above the surface allows the force acting on the tip to provide a "map" of the surface. Just like with a finger, the closer the AFM tip approaches the surface, the more easily damage occurs. The tip can, for example, pick up contaminants from the surface or, in the worst case, break against it.

As shown below, the actual operation of AFM is somewhat more complex, but the description above should be correct for a basic conceptual understanding.

AFM Design

Every AFM microscope consists of four basic components. The first and most important part is the cantilever, which facilitates contact/interaction with the studied surface. The cantilever can be imagined as a flexible rod with a very sharp tip at its end—so sharp that its apex consists of exactly one atom. There are several ways to achieve the required sharpness, such as electrolytic etching of the tip material, lithography, or focused ion beam milling. Different tip materials are suitable for studying various phenomena and surfaces; thus, no single specific tip is used. However, there is a list of common materials used, including silicon, copper, tungsten, silver, or gold.

The cantilever is further connected to a piezocrystal. Piezocrystals are materials that deform when exposed to an external electric field and, conversely, induce an electric field when deformed. Common piezoelectric materials include quartz or various types of ceramics. An alternating high-frequency voltage can then excite cantilever oscillations, which is advantageous for measuring surface interactions and, ultimately, surface morphology.

The deflection of the cantilever due to the interaction between the tip and the sample surface is measured by a laser that hits a mirror attached to the cantilever and reflects onto a photodiode detector, which converts the signal for a computer. When moving over the surface, the cantilever bends in various ways, tilting the mirror and changing the laser beam's position on the photodiode.

The final component of the apparatus is the feedback loop, which ensures a response to changes in the laser beam position and maintains the resonance frequency. Naturally, a device for ensuring precise movement across the sample is also included.

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[5] `https://en.wikipedia.org/wiki/Atomic_force_microscopy#/media/File:Atomic_force_microscope_block_diagram.svg`

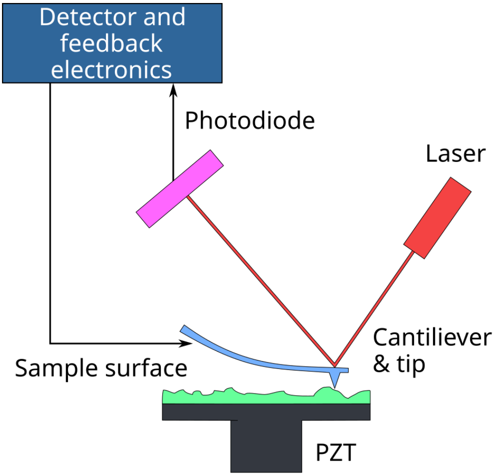

Figure 2: Schematic of an atomic force microscope.[5]

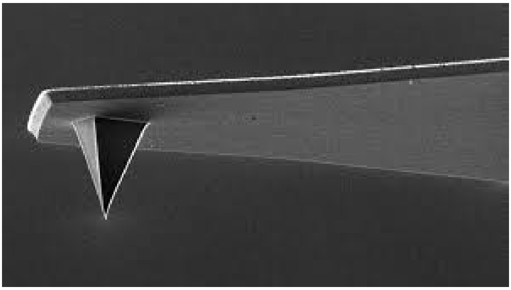

Figure 3: Image of an AFM tip.

Harmonic Oscillator

To understand the physical operation of AFM, we must first introduce the model of a harmonic oscillator. In classical physics, we consider this model for systems displaced from an equilibrium position by an external force that then oscillate around it. Such a system could be, for example, a mass m suspended on a spring of stiffness k moving only in the vertical direction. Newton's second law for our system takes the form

$$m\frac{\mathrm{d}^2 z}{\mathrm{d}t^2} = -kz\,,$$

where by moving kz to the left side, we obtain a differential equation in the well-known form

$$m\frac{\mathrm{d}^2 z}{\mathrm{d}t^2} + kz = 0\,.$$

This equation can be solved in several ways, which we leave to the reader. We include only a link to a summary of useful methods[6] and continue directly with the result

$$z(t) = A\cos\left(\sqrt{\frac{k}{m}}t + \varphi\right) = A\cos\left(\omega t + \varphi\right)\,,$$

where A is the amplitude of oscillation, ω is the angular frequency at which the mass oscillates, and φ is the initial phase. The relation $\omega = \sqrt{k/m}$ indeed holds, as confirmed by dimensional analysis.

Although it might seem at first glance that such a situation is never achieved in practice, the mass-on-a-spring model corresponds well to the free oscillation of a cantilever in an ultra-high vacuum. The result obtained from the harmonic oscillator model also allows us to measure the distance from the surface.

Static versus Dynamic AFM

In the early days of AFM, the main obstacle was the limited possibilities of the static mode—where the tip moves over the surface without oscillating, and the distance from the surface is determined from the known stiffness of the cantilever (imagined simply as a spring) k and by measuring the force F. In principle, such a machine would be beautifully simple, an ordinary force gauge scaled down to atomic size, but if research were that easy, only common sense would have been taught in schools long ago.

The dynamic mode differs from the static mode in that the tip oscillates along with the cantilever. Such a device can again be simplified as a system of coupled springs, see Figure 4. This approximation can be further refined by accounting for friction and forced oscillations of the cantilever, which you will address in the first problem of the series.

We can observe that, assuming small displacements[7] and a constant force gradient over time, this model behaves as a harmonic oscillator, but shifted by some constant angular frequency $\Delta\omega$, because

$$k_{\text{tot}} = k\ + k_{\text{ts}} \quad \Rightarrow \quad m\frac{\mathrm{d}^2 z}{\mathrm{d}t^2} + z(k + k_{\text{ts}}) = 0\,,$$
$$\omega^2 = (\omega_0 + \Delta\omega)^2 = \frac{k_{\text{tot}}}{m} = \frac{(k\ + \frac{\partial F}{\partial z})}{m}\,. \tag{1}$$

where k_{ts} is replaced by the gradient of the force acting on the tip from the surface, since the spring force is defined as $F = -kz$, and thus k_{ts} is obtained by the derivative of F with respect to z. We also assume that the force changes slowly enough that its change between two consecutive oscillation periods is negligible.

Using the approximation $\Delta\omega \ll \omega_0$, Equation (1) can be modified into the form

$$(\omega_0 + \Delta\omega)^2 = \frac{k\ + \frac{\partial F_{\text{ts}}}{\partial z}}{m}\,,$$
$$\omega_0^2 + 2\omega_0\Delta\omega + \Delta\omega^2 = \frac{k}{m} + \frac{\frac{\partial F_{\text{ts}}}{\partial z}}{m}\,,$$
$$2\omega_0\Delta\omega + \omega_0^2 = \omega_0^2 + \frac{omega_0^2}{k}\frac{\partial F_{\text{ts}}}{\partial z}\,,$$

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[6] `https://www.math.toronto.edu/selick/B44.pdf/`

[7] This clearly holds for van der Waals forces, as they typically act over distances of 10 nm and the displacement will be $\ll 10\,\mathrm{nm}$.

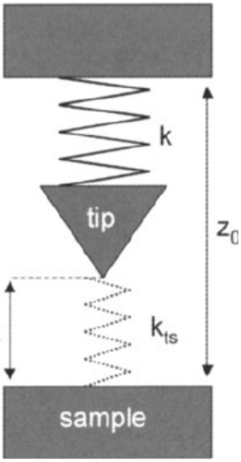

Figure 4: Simplified physical model of dynamic AFM. It consists of an oscillating tip on a spring of stiffness k, which is connected to the surface by an imaginary spring of stiffness k_{ts} modeling the interaction.

because $\omega_0 = \sqrt{k/m}$ and $\Delta\omega^2 \approx 0$. From this equation, we already obtain a formula allowing us to convert force (proportional to the distance from the surface) into a well-measurable quantity: the relative change in oscillation frequency[8]:

$$\frac{\Delta\omega}{\omega_0} = \frac{\Delta f}{f} \approx +\frac{1}{2k}\frac{\partial F_{\mathrm{ts}}}{\partial z}\,.$$

At first glance, it might seem we have two equivalent paths to measure interatomic forces (and surface morphology), with the static path appearing even easier on paper. The difficulty lies in the practical implementation, where one is significantly simpler and cheaper. Not that AFM is a cheap instrument regardless.

Consider the standard Lennard-Jones potential, which models the interaction between two neutral particles, see Figure 5. To outline the first drawback of static AFM, consider a spring representing a cantilever with a tip of stiffness k. We are able to measure the force if and only if the change in force is less than the imaginary spring stiffness k. Otherwise, the cantilever is necessarily pulled to a height where the restoring force of the spring is in equilibrium with the force from the surface.

In Figure 5, a sample static AFM trajectory is marked with a dashed line. Parts of the interaction between BC and DA are simply not measurable. The reason is that the spring force depends linearly on displacement. If we exceed point B, this slope is no longer sufficient to compensate for the repulsive forces, and equilibrium is reached only at point C. Analogously,

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[8] In AFM literature, you will typically encounter a negative sign, as the gradient of an attractive force is negative

a “snap” occurs between D and A. Not only is a whole portion of the force interaction range lost, but these observable snaps could easily result in tip damage. A possible solution to the snapping problem is simply creating a sufficiently “stiff” spring for which $\forall z : (\partial F_{\mathrm{ts}}/\partial z)|_z < k$ would hold everywhere.

This idea is correct, and creating such a spring is possible today; however, higher stiffness naturally leads to a loss of sensitivity. The problem lies in the disproportionate complexity of the design required to measure the acting force. While precise piezoelectric elements could be used, it would still be an order of magnitude more complex than measuring changes in the cantilever’s oscillation frequency. To provide a numerical illustration, the strength of chemical bonds at the atomic level ranges from 1–10 nN.

For a better understanding of the graph in Figure 5, we remind the reader that the y-axis represents force and the x-axis represents distance z.

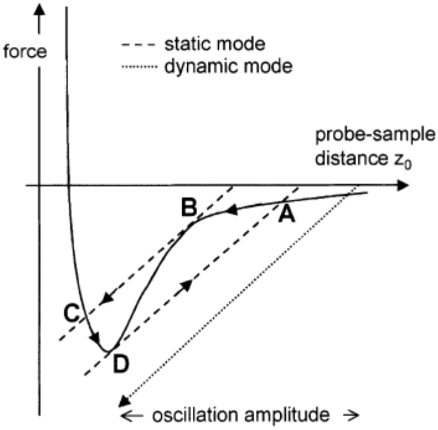

Figure 5: Tip trajectory during a typical interaction between two atoms.[9]

Transition from Optical to Electron Microscopy

The optical microscope works with visible light, and its resolution is limited by light diffraction at the aperture. In a simple form, this can be expressed by the Rayleigh criterion:

$$d \approx 0.61\,\frac{\lambda}{\mathrm{NA}}\,,$$

where d is the minimum distance between two points that can still be distinguished, λ is the wavelength of light, and NA is the numerical aperture of the objective. Even with short-wavelength visible light and high NA, the distance d is on the order of hundreds of nanometers. Magnification can be increased further, but without improved resolution, no new details appear.

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[9] B. Bhushan, H. Fuchs; Applied Scanning Probe Methods IV. Heidelberg: Springer Verlag, 2006. ISBN 978-3-540-26912-0.

Electron microscopy uses electrons instead of photons. Electrons have wave properties, and their de Broglie wavelength is very small at high energies. In practice, electrons are accelerated by high voltage, which reduces their wavelength and opens the possibility of imaging finer structures than in optics. Actual resolution is determined not only by wavelength but also by electromagnetic lens aberrations, instrument stability, and sample properties. Since electrons are significantly scattered in air and discharges can occur, electron microscopes operate in a vacuum.

Interaction of Electrons with Matter

The electron is a charged massive particle; therefore, when passing through matter, it interacts via Coulomb forces with both the nuclei and the electron shells of atoms. These interactions alter its trajectory and energy, trigger the emission of various secondary signals, and—due to the wave properties of electrons—lead to the interference of electron waves. These phenomena form the physical foundation of electron microscopy and determine the type of information that can be obtained about a sample.

In basic terms, electron microscopy is divided into Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM), based on the method of image formation and the beam-sample interaction. In SEM, an electron beam scans the sample surface sequentially, and the image is generated from signals produced during the interaction of the beam with the surface region of the material—primarily secondary electrons (SE), back-scattered electrons (BSE), and X-rays. Consequently, we primarily obtain information regarding surface morphology and chemical composition.

Conversely, in TEM, we observe electrons passing through an extremely thin sample. Image contrast thus arises from scattering, energy loss, and the interference of electron waves, providing information about the internal structure of the material. An overview of the potential products of electron beam-sample interaction is schematically illustrated in Figure 6. The specific signals used in SEM and the contrast mechanisms typical for TEM are described in detail in the following sections.

Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy is a method in which an image is created by sequentially scanning the sample surface with a focused electron beam. Unlike optical microscopy, where the image is formed simultaneously from transmitted or reflected light, SEM scans the sample point by point. At each point of impact, the interaction between electrons and the material generates signals whose intensity is then assigned to the brightness of the corresponding pixel in the image.

Electrons are produced in an electron source and, after acceleration (typically by voltages in the tens of kV), pass through a system of lenses and apertures that shape and focus the beam. Unlike optical microscopy, these are not glass lenses but electromagnetic lenses—coils creating a magnetic field that directs the movement of electrons. Since electrons are charged particles, their trajectory can be manipulated by a magnetic field to create a focused beam. The quality of this beam directly affects the resolution and quality of the resulting SEM image.

The position of the beam impact point is controlled during measurement by a scanning coil, which moves it across a selected area of the sample line by line. Various signals are generated at individual points, differing in their production mechanism, energy, and the depth from which

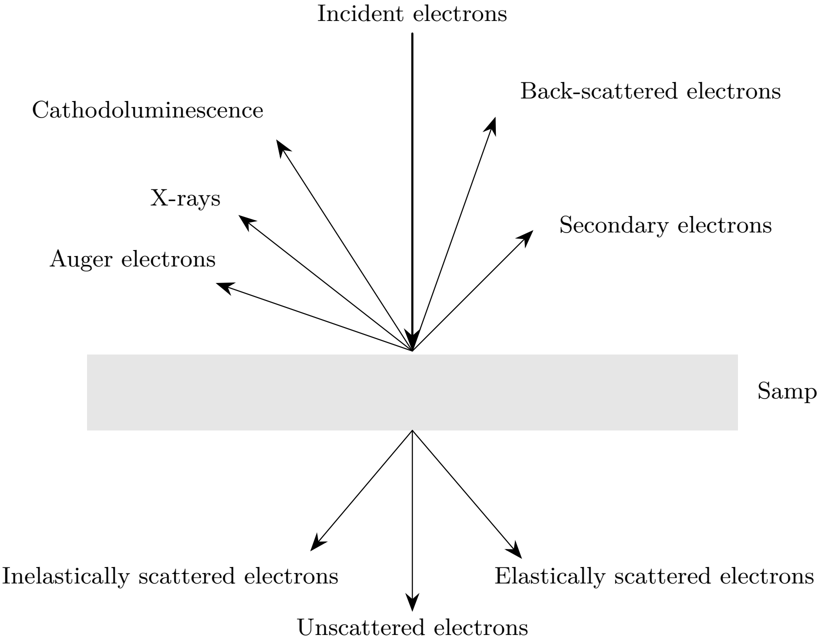

Figure 6: Schematic representation of electron interaction with a sample.

they escape the sample. The choice of the detected signal determines the type of information the image provides.

Secondary electrons (SE) are produced by inelastic collisions of primary electrons with the electrons of atoms in the sample. In such an interaction, a primary electron transfers part of its energy to an electron in the material, which may then be ejected from the atom as a secondary electron. SE typically have low energies (up to 50 eV), meaning they escape only from a very shallow depth—on the order of a few nanometers. Consequently, they are extremely sensitive to the state and shape of the surface. The SE signal primarily provides topographical information, emphasizing edges, pores, protrusions, or roughness, and enables high spatial resolution.

Back-scattered electrons (BSE) represent a different signal group. These are primary beam electrons whose trajectories are deflected within the sample by Coulomb interaction with atomic nuclei, subsequently leaving the sample toward the detector. Unlike SE, these are not low-energy electrons ejected from an atomic shell, but electrons that have retained a significant portion of their original energy; thus, the BSE signal has much higher intensity than the SE signal. Because they originate from a larger interaction volume, they carry information from greater depths, and their spatial resolution is generally lower than that of SE. Their major advantage, however, is sensitivity to chemical composition—specifically to the average atomic number of the elements. Atoms with higher atomic numbers scatter primary electrons more efficiently; therefore, more back-scattered electrons escape from such regions, making them appear brighter in a BSE image. BSE imaging is thus primarily used for compositional contrast to distinguish regions with different compositions or phases.

Another important product of electron-matter interaction is X-ray radiation. It is produced when a primary electron transfers sufficient energy to an electron in an inner shell of an atom (e.g., the K, L, or M shell[10]) and ejects it. This creates a vacancy in the inner level, which is then filled by an electron from a higher energy level. This transition releases energy in the form of an X-ray photon (RTG), proportional to the energy difference between the initial and final levels. Since electron energy levels are specific to each element, the energy of emitted X-ray photons is characteristic of that element, forming the basis of elemental analysis via Energy Dispersive X-ray Spectroscopy (EDX).

An EDX detector records the energies of individual photons and constructs a spectrum of intensity versus energy, where peak positions identify the elements present and peak intensities provide information on their relative abundance. This method is used for point analysis, line profiles, and elemental mapping. However, one must account for the fact that X-rays originate from a relatively larger interaction volume than secondary electrons, so the spatial resolution of chemical information from EDX is typically poorer than the topographical resolution of SE.

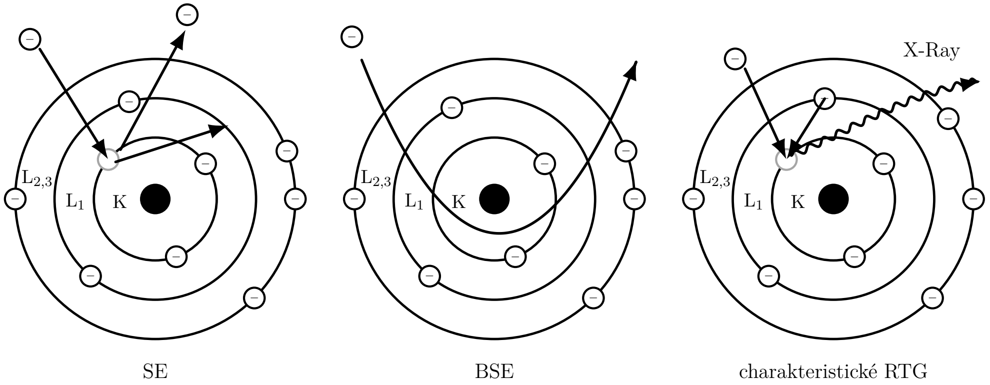

Figure 7: Generation of a secondary electron, a back-scattered electron, and characteristic X-rays.

Transmission Electron Microscopy (TEM)

Transmission Electron Microscopy is based on a principle similar to SEM, but the difference is that in TEM, we observe electrons that have passed through the sample. Images and other data are obtained from their scattering, energy loss, and interference. In TEM, electrons are also generated in a source, accelerated by an electric field, and shaped into a beam using electromagnetic lenses. However, in TEM, the beam is usually not focused to a point but is a parallel beam. Higher acceleration voltages are typically used—around 200 kV (often ranging from 100–300 kV)—compared to SEM. Higher energy increases electron penetration, allowing them to pass through thin samples with less loss and scattering. Furthermore, higher voltage

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[10] The letters K, L, M, etc., indicate the shell layer relative to the nucleus. Some levels may split, leading to designations such as L$_1$ or L$_2$ as seen in Figure 7.

[11] `https://www.jeol.com/words/semterms/20121024.073058.php#gsc.tab=0`

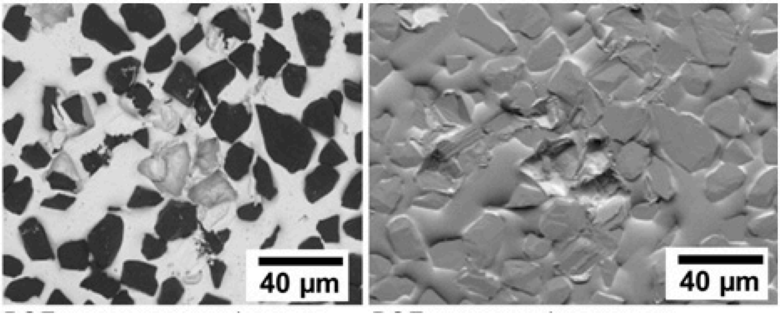

Figure 8: Comparison of BSE signal (left) and SE signal (right).[11]

decreases the de Broglie wavelength, which is beneficial for achieving high resolution. For a sufficient number of electrons to pass through, the sample must be very thin, typically under 100 nm.

During transmission, some electrons pass through without significant deflection, some scatter elastically, and others inelastically. The distribution of intensity among these components determines the TEM image contrast. A significant advantage of TEM over SEM is that, in addition to the image, one can observe a diffraction pattern (diffractogram), which carries information about the crystalline structure and orientation of the material.

When the electron beam hits a crystalline substance, electrons scatter off the periodically arranged atoms of the crystal lattice. Due to their wave properties, the scattered electron waves superimpose and interfere. In certain scattering directions, their phases align, resulting in constructive interference and an intense spot in the diffractogram. In other directions, destructive interference occurs, suppressing the intensity. The path difference of the scattered waves determines the type of interference. The geometry of scattering on atomic planes (Fig. 9) leads to Bragg's Law:

$$2d\sin\theta = n\lambda,$$

where d is the interplanar spacing, θ is the Bragg angle, λ is the electron wavelength, and n is an integer. When this condition is met, constructive interference occurs, and a diffraction maximum appears.

A diffractogram provides information on internal structure, particularly the degree of crystallinity, interplanar distances, and often phase composition. Using Bragg's Law, interplanar distances can be determined from the positions of diffraction maxima, while their intensity and width carry information about atomic arrangement, preferential orientation, crystallite size, and micro-strains. Factors such as thermal vibrations and defects also influence the intensity and width of individual spots.

The character of the diffractogram varies significantly by sample type. For a single crystal—a sample consisting of one continuous crystal with uniform orientation—diffraction conditions are met only for precisely defined crystal orientations; thus, the pattern appears as a set of discrete spot reflections. For a polycrystalline sample with random orientations of many small grains, some grains will satisfy Bragg's Law for every family of lattice planes, resulting in Debye-Scherrer rings on a 2D detector. Conversely, amorphous materials lack long-range periodic

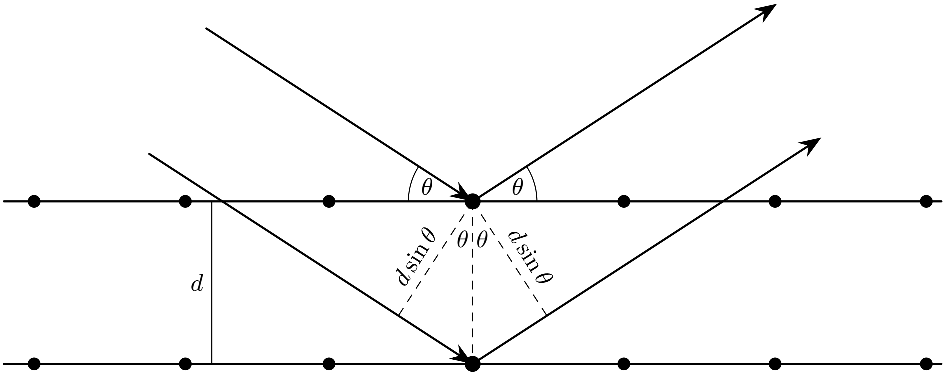

Figure 9: Electron diffraction on a crystal lattice.

order, so sharp Bragg reflections do not form, and the diffractogram contains only broad, diffuse halos. Examples are shown in Fig. 10.

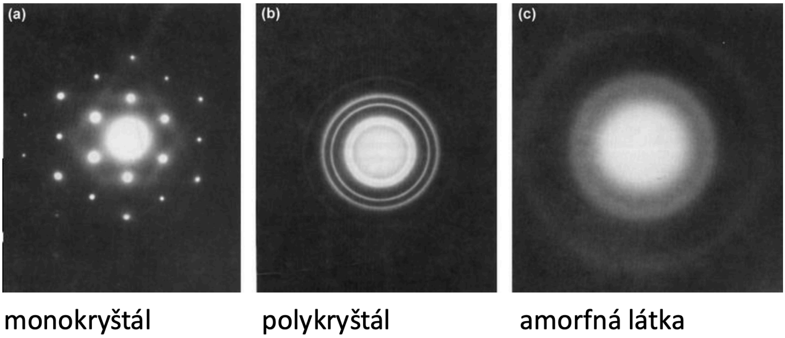

Figure 10: Diffractograms for single-crystal, polycrystalline, and amorphous samples.[12]

Contrast in TEM can have several origins. One is scattering contrast, which occurs because some electrons are deflected or lose energy during transmission and thus do not contribute equally to image formation. Thicker regions or areas with higher average atomic numbers scatter electrons more intensely and often appear darker. This contrast is vital for samples with non-homogeneous thickness or compositional differences.

Another major mechanism is diffraction contrast, arising from elastic scattering on the crystal lattice. The brightness of specific regions depends on whether they satisfy diffraction conditions at the given microscope settings. Diffraction contrast is sensitive to crystal orientation, lattice defects, grain boundaries, twinning, or strain, making it highly useful for studying microstructure.

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[12] M. Karlík, Transmisní elektronová mikroskopie: principy a aplikace. Praha: České vysoké učení technické v Praze, 2004. ISBN 80-01-02978-0.

In High-Resolution TEM (HRTEM), phase contrast may also apply, resulting from the interference of electron waves with different phases after passing through the sample. Under specific conditions, HRTEM can resolve the periodic arrangement of the crystal as rows of atoms (specifically, rows of atomic columns along the projection), allowing for detailed structural study. However, interpreting HRTEM images is more complex and depends heavily on imaging conditions. An example is shown in Figure 11.

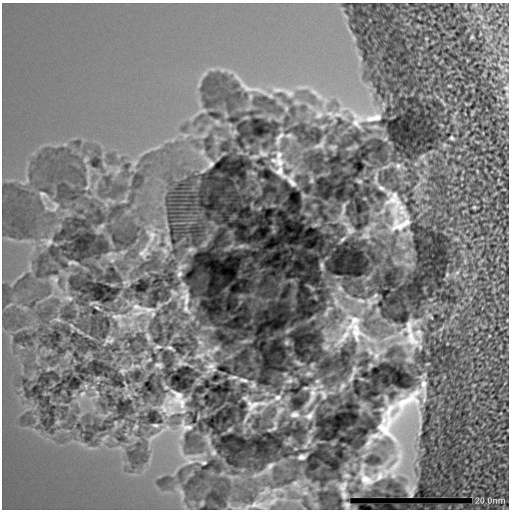

Figure 11: High-Resolution TEM (HRTEM) image.

Problem Images

Using the SEM image (BSE) and the model EDX spectrum, determine which chemical elements are present in the substance and which regions of the sample correspond to them. What else do you observe in the image, and how should a real EDX spectrum differ from the model? Provide detailed justifications for all your claims.

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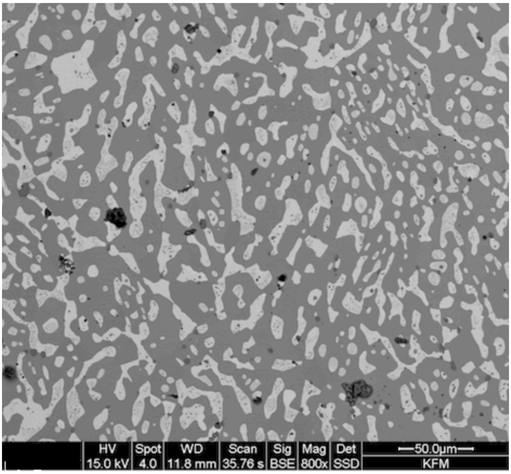

Figure 12: SEM image, BSE signal.

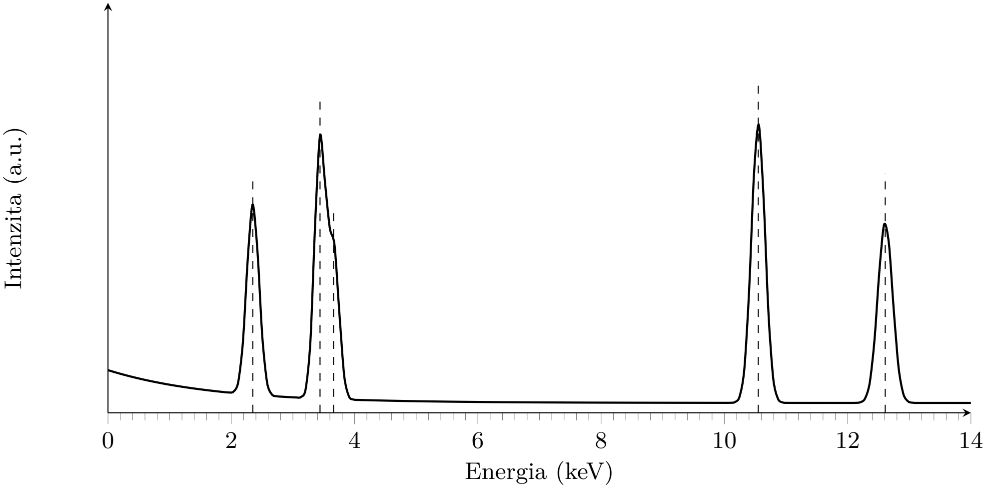

Figure 13: Model EDX spectrum.