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Miroslav Kolíbal

**SCIENCE, TECHNOLOGY
AND IN-SITU
ELECTRON MICROSCOPY**

VYSOKÉ UČENÍ TECHNICKÉ V BRNĚ
FAKULTA STROJNÍHO INŽENÝRSTVÍ
Ústav fyzikálního inženýrství

doc. Ing. Miroslav Kolíbal, Ph.D.

**SCIENCE, TECHNOLOGY
AND IN-SITU ELECTRON MICROSCOPY**

VĚDA, TECHNOLOGIE A IN-SITU ELEKTRONOVÁ MIKROSKOPIE

THESIS OF THE INAUGURAL LECTURE
IN APPLIED PHYSICS



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KEYWORDS

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KLÍČOVÁ SLOVA

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ABOUT AUTHOR



I carried out my Ph.D. studies on applied physics at Brno University of Technology (Czech Republic) under supervision of Prof. Jiří Spousta developing a Low Energy Ion Scattering (LEIS) instrument and later exploring the possibilities of morphological analysis of thin surface layers. During this period, I actively collaborated with the group of Prof. Peter Bauer at JKU Linz. After finishing my Ph.D. I continued the work at Brno University of Technology as a research fellow and since 2007 as a research assistant in the group of Prof. Tomáš Šíkola. I have been involved in various research projects dealing with hybrid top-down/bottom-up lithographic approaches for preparation of nanostructures and, more importantly, I have been given freedom to pursue new research directions. To gain a more interdisciplinary background and broaden my knowledge of electron microscopy, I have started a collaboration with FEI Company (currently Thermo Fisher Scientific), developing techniques for real-time observation of different processes inside an electron microscope. As a part of this activity, in 2009 I joined the group of Dr. Milos Toth at FEI R&D in Hillsboro, Oregon (USA). After returning to Brno, I have started my independent research in the field of nanowire growth and developed unique instrumentation and methodology for observation of nanowire growth in real time in a scanning electron microscope.

Being interested in one-dimensional nanostructures, I took the opportunity to join the group of Dr. Heike Riel at IBM Zurich Research Laboratory (Switzerland) in 2012, developing high-k dielectric templates for directed III-V nanowire growth. At the same time, I have joined a newly established centre of excellence CEITEC BUT in Brno, Czech Republic, where I carry research on semiconducting low-dimensional nanostructures. I was involved in the definition of equipment for the CEITEC Nano core facility.

Although electron microscopy of nanoscale materials is still my core research area, the interests of my group get broader as new people join the team and new projects are initiated. Currently, we get more involved in correlative microscopy, attempting to collect more signals than just the secondary electron contrast (e.g., using scanning probe microscopy, mass spectroscopy, etc. at the same location). The newly developed methodologies allow us to study appealing material systems inside a microscope: battery stacks, gas-phase catalytic reactions, etc. All this is possible via collaboration with leading companies (Thermo Fisher Scientific, NenoVision, onsemi) on hardware and via joint projects.

As my research is currently being done at both CEITEC and Faculty of Mechanical Engineering, I keep a strong link to the faculty students by teaching several undergraduate courses for both bachelor and master students, including Modern physics (tutorials), Surfaces and thin films (tutorials), Experiments in physics (for both basic as well as advanced programme), Preparation of low-dimensional structures, and Diagnostics of nanostructures (lectures). In addition to physics, I am also involved in teaching ‘soft skills’ (for example presentation skills) to master students (Diploma seminar for undergraduates) and Ph.D. students (Friday seminars for CEITEC Ph.D. students). My greatest pleasure is to promote students’ interest in experimental physics and science in general by supervising bachelor and master theses. I supervised 19 bachelor, 18 master, and 2 PhD students who successfully defended their final theses.

1. CONTEXT AND PURPOSE OF THE THESIS

The equations describing electromagnetic fields are James Clerk Maxwell's most famous scientific achievement. The equations were originally published in 1861 and 1862 and, in a different form, they were recast in 1884 by Oliver Heaviside. In 1925, Samuel Goudsmit and George Uhlenbeck made a bold proposal that fixed almost every issue in atomic physics at that time – they added a fourth quantum number. This number was recognised later as electron spin. Those are two examples of fundamental research, or perhaps better called curiosity-driven research, which searches for discoveries and explanations of *phenomena*. What is light? How are the electric and magnetic forces related? What is the mechanism behind the anomalous Zeeman effect? Curiosity-driven research is initiated by similar questions. Therefore, there is no rationale other than curiosity and a passion for learning about the world around us. Soon after, additional questions arise: can we generate electromagnetic radiation of different frequencies? Can we flip the spin? Scientists perform application-driven research with a clear purpose in their minds – they look for *principles*, i.e., how the phenomena can be utilised practically. Finally, this endeavor can potentially lead to new technology. Maxwell's equations stand as the basis of modern technology for the generation, propagation, and reception of electromagnetic radiation. Spin is the key phenomenon used in magnetic resonance imaging and drive-head technology in hard drives, among other things.

In physics, in particular, curiosity- and application-driven research are necessary prerequisites to the success story of the twentieth century. Indeed, a lot of effort is still needed to transition fundamental findings (phenomena and principles) into novel technologies. This effort also includes incremental advancements, leading to further technological development. The balance between the importance of incremental advancements and fundamental discoveries differs between the fields. However, as outlined above, a common mindset is that science is the driving force for technology development. However, not being commonly stressed is that novel technologies are required to achieve scientific breakthroughs. Otto Stern and Walter Gerlach conducted a famous experiment in 1922. In order to prove (or disprove) Niels Bohr's hypothesis of spatial quantisation of angular momentum, they built a unique device. They combined the emerging technology of atomic beams with a magnet to create a large anisotropic magnetic field. Gerlach had to construct one himself, as such magnets were not commonly available at that time. Combining available technologies with state-of-the-art know-how, they finally showed that the silver atomic beam splits into two after passing through the magnet. The experiment was correctly explained only after Uhlenbeck and Goudsmit proposed the existence of spin angular momentum. Although being praised as a foundational work in atomic physics, the experiment also had implications for further technological development. The technology of atomic beams subsequently evolved into molecular beam epitaxy, which enabled Nobel prize-award-winning research on the fractional quantum Hall effect (1998) and semiconductor heterostructures (2000). While the former is a subject of advanced solid state physics research, the latter is a cornerstone of modern semiconductor devices, e.g. LED lighting, solid-state lasers, etc. The story could continue further on, e.g., by describing the usage of lasers in curiosity-driven research. Similar "trains of discoveries" are the fundamental building blocks of modern technological society. I believe that it is of crucial importance that university students understand the complex mutual relationship between science and technology.

I aim to demonstrate the science-technology relationship using electron microscopy by describing its invention and use in science and, finally, in education. This thesis focuses on the unconventional use of an electron microscope. Instead of passive capturing of stationary images, I present famous experiments that use the microscope in a rather unusual way. The thesis briefly describes

demonstrations of puzzling predictions of quantum physics, namely a double-slit experiment with electrons and the even more intriguing Aharonov-Bohm effect. Through these demonstrations, the electron microscope has manifested its capabilities beyond simple imaging. In recent years, one of the efforts defining the field of materials science has been directed toward performing dynamic experiments inside the microscope, turning it into a complex materials science laboratory. In the second part of this thesis, I will present our small contribution to this effort. In our work, we focus on modifications of an electron microscope to allow for observation of processes happening at extreme conditions, e.g. high pressure and temperature. The development of unique instrumentation allows us to perform unique experimental work and, as such, corroborates the ideas outlined in the previous paragraphs.

2. DEVELOPMENT OF AN ELECTRON MICROSCOPE

“Do you know, I always thought unicorns were fabulous monsters, too? I never saw one alive before!” Well, now that we have seen each other,” said the unicorn, “if you’ll believe in me, I’ll believe in you.” Lewis Carrol

It took many fundamental discoveries and incremental designs to finally set up an electron microscope, as previously outlined. Due to the limited space of this thesis, I have chosen to introduce just a few (see Figure 1). I have already mentioned Maxwell’s equations describing the electromagnetic field. In 1897, J. J. Thomson discovered the electron, the first subatomic particle. The reader of a typical physics textbook might think that Thomson did a simple experiment: he applied simple physics to cross electric and magnetic fields, let electrons deflect under the Lorentz force, and then obtained an electron mass which is much smaller than an atomic mass. There is always more to the truth than meets the eye in scientific discoveries. The electron beam created in Crookes tubes was called “cathode rays” back then, and many scientists were studying this phenomenon. J. Perrin found that cathode rays possess a negative charge. Both H. Hertz and P. Lennard investigated the transmission of cathode rays through various foils and substances (including air). E. Rutherford, who had just joined the Cavendish laboratory as a PhD candidate, contributed significantly to the development of a technique for measuring single charges. Indeed, the pathway to the identification of electrons was not straightforward. For example, H. Hertz found that cathode rays can be deflected by a magnetic field but not by an electrostatic field (!?). The history of each key scientific discovery is very rich. It teaches us that breakthrough discoveries do not come from anywhere, but result from many advances made by several scientists.¹ But in the end, J.J. Thomson put all the pieces together and concluded that it is a low mass m instead of a multiple charge q , causing such an unexpectedly large q/m value. Important to note is that electrons could not have been discovered without vacuum technology, which had advanced enough so that particles could pass through the Crookes tube without many collisions with residual atoms (vacuum quality varied from lab to lab, and it was a particularly poor vacuum which was behind puzzling observations of H. Hertz).

The properties of electrons were immediately studied, including the generation of electron beams. In Crookes tubes, electrons were generated by the ionisation of gases close to the cathode (hence the name ‘cathode rays’). Soon it was found that electrons can be generated by heating a metallic wire inside a vacuum or by applying strong (electric) fields to a sharp tip. The latter process is known as field emission. Understanding this process was possible once the quantum theory had been built. R. H. Fowler and L. H. Nordheim realised that its mechanism relies on quantum tunneling (see also later, Fig. 2b).

All in all, the prerequisites to build an electron microscope were known already around the year 1900. But how would this microscope work? What would be the resolution? The answer to the latter question became known in 1924, when L. De Broglie submitted his dissertation thesis. He successfully defended his idea that wavelength is also associated with matter. At that time, the wavelength-resolution relationship in optical microscopy was already known. The idea to build a microscope using energetic particles emerged almost immediately. Envisioning the manipulation of electrons by electromagnetic fields utilising Maxwell’s equations, H. Busch published a paper proposing to use the magnetic field of a coil to manipulate and focus electrons in 1926. The concept was similar to the use of photons in an optical microscope. Soon after, S. Davisson and L. Germer performed their famous experiment demonstrating the diffraction of an electron beam on a crystal

and fuelled the expectations of the new technology to come. Finally, in 1932, E. Ruska was the first to build an electron microscope that later enabled observing matter in real and reciprocal space with unprecedented resolution. Initially, the microscope had a lower resolution than an optical one. Thanks to an enormous effort driven by applications and economic interests of different companies, this soon changed. Numerous advancements were achieved as a result of the development of better vacuum pumps and brighter sources, especially LaB₆ and dedicated cold field emission cathodes. A milestone in terms of accessibility was the development of the scanning electron microscope (SEM) by M. von Ardenne (1938) and V. K. Zworykin, J. Hillier and R. Snyder (1942). This microscope soon became a workhorse in many laboratories, including rapidly growing semiconductor companies. The versatility of scanning electron microscopes was increasing with the development of additional detection techniques, e.g. environmental SEMs, energy-dispersive X-ray spectroscopy, diffraction techniques, etc. Indeed, the history of electron microscopy is tightly bound to a steadily increasing resolution. This is due to progress in the voltage sources' stability, the microscope's mechanical build, and electron optics. Another milestone in the development of the transmission electron microscope (TEM) was the development of aberration correctors, which allowed the achievement of remarkable resolution values in the subnanometer range. As such, electron microscopy is one of the most powerful techniques for probing matter and its properties.

The development and construction of an electron microscope (especially TEM) is a very challenging task. Hence, the production of electron microscopes is limited to several companies in locations related to the history of electron microscopy (Germany - Zeiss, United States – Thermo Fisher Scientific, Japan – JEOL and Hitachi). A significant part of the world's production is also located in Brno (Thermo Fisher Scientific, Tescan, Delong Instruments). The history of electron microscopy in Czechoslovakia is associated with prof. Aleš Bláha, who initiated electron microscopy-related research first at Škoda company and since 1946 at the Brno University of Technology, where he became a professor. He established dedicated laboratories and industry collaborations and engaged three of his students (Armin Delong, Vladimír Drahoš and Ladislav Zobač) to build an electron microscope. What would be celebrated today (as an outreach beyond university towards industry) became fatal for Prof. Bláha carrier as he was expelled from his position at BUT. However, his students were allowed to continue their development and, based on their work, the first commercial microscope manufactured in Czechoslovakia (Tesla BS 241) was introduced. Tesla was dismantled after 1989, but, thanks to its employees, smaller companies were established (Tescan, Tesla Elmi/Delmi, and Delong Instruments), which currently supply one-third of the electron microscopes in the world today.

The next chapter discusses two experiments that demonstrate the versatility of electron microscopy beyond standard imaging.

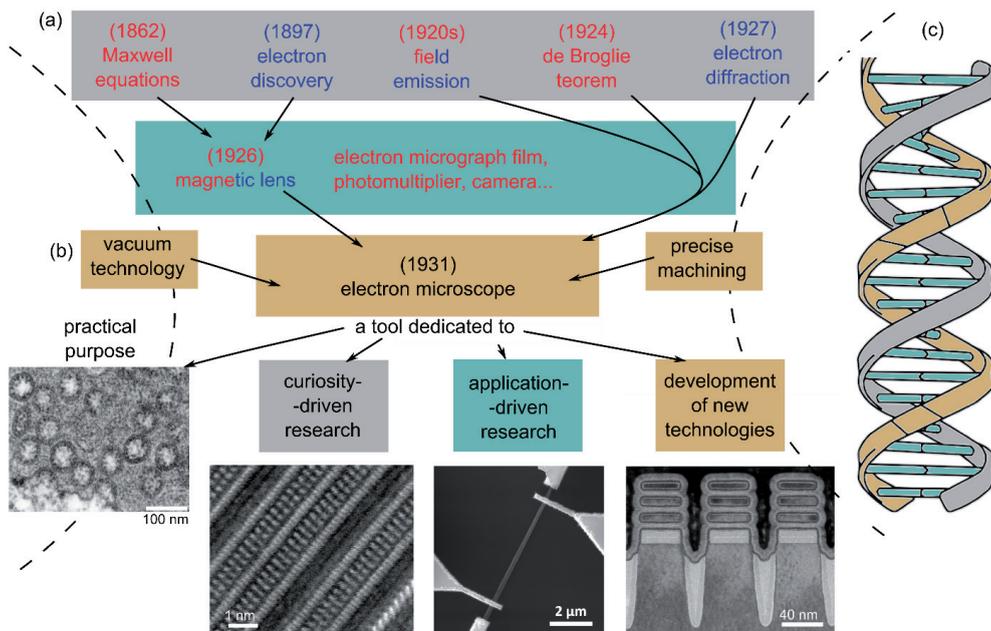


Fig. 1: Relationship between science and technology. (a) The pipeline model, most frequently presented. Curiosity-driven research (grey box) searches for new phenomena, while application-focused research uses these phenomena in search of principles (green box).² Finally, new technologies emerge from the cumulation of principles and related phenomena (sandy box),¹ as illustrated by the development of an electron microscope. Both theoretical (red) and experimental (blue) work was necessary to develop the building blocks of an electron microscope, an instrument assembled by Ernst Ruska under the supervision of Max Knoll in 1932. (b) New technologies serve humanity in various ways and promote disruptive changes in society³ (the image on the left shows the SARS coronavirus under an electron microscope; reprinted with permission from Centers for Disease Control and Prevention).⁴ But, not to forget, technologies are crucial for further development in all science and engineering fields. They enable the quest to understand nature, its laws and their utilisation for further advancements in science and technology. Bottom line, from left: TEM image of cross-sectioned SnS-TaS₂ misfit nanotube;⁵ SEM image of a WS₂ nanotube electrically contacted by four electrodes; stacked nanosheet transistor design by Intel's "3 nm" technology, imaged by TEM [source: Intel]. (c) A more realistic view of the science and technology relationship utilising a double helix⁶ – the full potential of both is unleashed only when they are mutually bound together.

3. QUANTUM THEORY AND ELECTRON MICROSCOPY

“When you can measure what you are speaking about, and express it in numbers, you know something about it, when you cannot express it in numbers, your knowledge is of a meager and unsatisfactory kind; it may be the beginning of knowledge, but you have scarcely, in your thoughts advanced to the stage of science.” Lord Kelvin

Quantum theory paved the way for many technological advancements, including the development of the electron microscope, as particularly documented in Fig. 1. However, this theory differs from other models of nature in that it sometimes contradicts our daily experience. Despite quantum mechanics being accepted as a valid theory in the scientific community a long time ago, the endeavour to prove its conclusions continues. In this chapter, the contribution of electron microscopy to this quest is highlighted.

Quantum theory is closely related to the theory of light. It was developed thanks to the “few discrepancies” found in physics at the end of the nineteenth century associated with light (photoelectric effect, Michelson-Morley experiment, and blackbody radiation). It was primarily a question of the nature of light and its propagation. Isaac Newton pioneered the corpuscular theory of light, which was soon replaced by the wave theory, which explained many light-related phenomena, e.g. interference, diffraction, refraction, etc. The most famous experiments demonstrating the wave nature of light are the double-slit experiment by Thomas Young (see Fig. 2a) and the two-mirror experiment by Augustin-Jean Fresnel. According to our everyday experience, experimenting with macroscopic particles (e.g., bullets) does not result in interference patterns (Fig. 2b). However, quantum matter behaves differently (Fig. 2c), and both results (interference or classic pattern) can be obtained depending on the conditions of the experiment. In search of the limits of the quantum world and theory itself, many experiments have been conducted on this puzzling observation.⁷ In addition to its fundamental importance, the double-slit experiment has also several applications - the most interesting one being the superconducting quantum interference device (SQUID), so far the most precise concept for measuring magnetic fields (Fig. 2d). The development of SQUID was only possible due to fundamental research in quantum physics, especially Brian Josephson’s theory of superconducting junctions. Even today, this classic textbook experiment is an inspiration for the development of, e.g., novel approaches to probe matter.⁸

As quantum particles, electrons should manifest all the strangeness of a double-slit experiment. The electron microscope offers both an electron source and an imaging screen, making it an ideal playground for these experiments (Fig. 3a). The electron source itself is, in fact, an excellent example of the quantum effect in use (Fig. 3b). The electric field E between a metallic tip and a nearby electrode results in an effective lowering of the work function Θ due to the combined effect of an electric intensity at the tip and the effect of an image force (which arises due to a single charge removed from the metal at a distance x from the surface). The resulting potential V can be calculated as

$$V = -eEx - \frac{e^2}{4\pi\epsilon_0 x}; \quad (\text{eq. 1})$$

where e is the elemental charge (negative for electrons) and x is the distance from the tip surface.

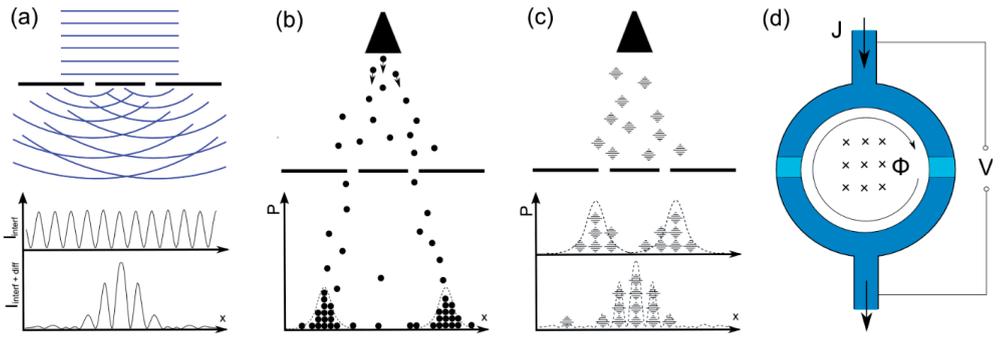


Fig. 2: Double-slit experiment. (a) A double-slit experiment in optics, utilising a plane wave (blue). The two slits in the plate act as coherent light sources; the waves interfere, and an interference pattern (top) is formed on the screen behind the plate. The pattern is modulated by diffraction on the slits (bottom). (b) The very same experiment with macroscopic particles (e.g. bullets) results in the formation of two maxima in detection probability right behind the slits. (c) If the experiment is performed with quantum particles, the result (pattern on the screen) depends on experimental conditions. If the observer can distinguish which slit the particle passed through, a classical pattern similar to (b) is observed (top). In another case, an interference pattern is detected just like in (a). Although this is a classic textbook example, this experiment has a significant practical use. The SQUID device, as pictured in (d), is reminiscent of the double-slit experiment. The two slits are replaced by Josephson junctions (light blue) carrying a current J , and interference (detected as alternating voltage V) is observed due to the phase difference caused by the (external) magnetic flux Φ enclosed within the loop (dark blue).

The work function changes as $\Delta\theta$, and can be obtained as a derivative of V . There are two possibilities of an electron escaping from the metal, either by thermionic emission (which is in the presence of a high electric field superseded by Schottky emission) or by tunneling through the potential barrier. The latter case is called field emission and is purely a quantum mechanical effect (see discussion in the first chapter).

In the case of electrons, a so-called biprism replaces the double slit in the microscope setup for the experiment. Biprism comprises two grounded electrodes and a metallic wire in between with an applied positive potential with respect to the electrodes. As the planar electron wave moves in the electric potential of the wire, it acquires additional components in the x -direction. Interestingly, these x -components have different signs for waves moving on opposite sides of the wire,

$$\phi_L(x, z) = e^{ik_z z} e^{ik_x x}; \quad \phi_R(x, z) = e^{ik_z z} e^{-ik_x x}. \quad (\text{Eq. 2})$$

The potential field bends the waves' propagation direction towards the microscope's optical axis, so they interfere on the screen. The intensity of the interference pattern can be calculated as

$$|\phi(x, z)|^2 = |\phi_L(x, z) + \phi_R(x, z)|^2 = |e^{ik_z z} e^{ik_x x} + e^{ik_z z} e^{-ik_x x}|^2 = 4\cos^2 k_x x. \quad (\text{Eq. 3})$$

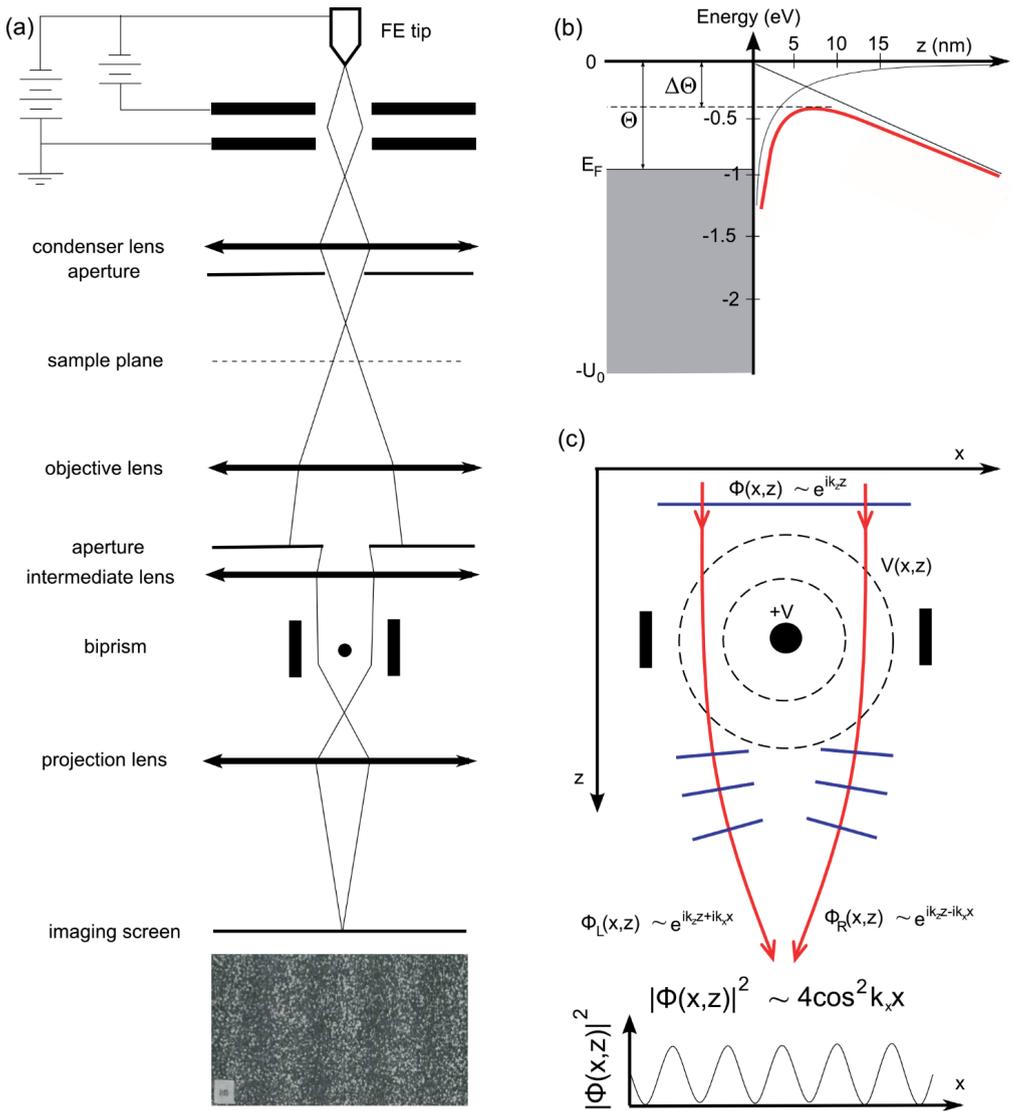


Fig. 3: Quantum phenomena in an electron microscope I. (a) A schematic of a transmission electron microscope, including a biprism acting as a double slit.⁹ (b) Energy diagram of electron emission from the metallic tip. Metal has filled all states up to the Fermi level (for simplicity, the Fermi-Dirac distribution of electron energies is neglected). The work function of the metal Θ is significantly reduced by $\Delta\Theta$ if an electric field is applied to the tip. The resulting potential is shown in red, and the sum of applied electric potential and field from a ‘mirror’ charge (both in black). The graph was calculated using $E = 40 \times 10^6$ V/m. (c) Working principle of a biprism, which gives rise to the interference pattern shown in a). The biprism consists of a wire with an applied electric potential $+V$ and two grounded electrodes. The electron plane wave (blue) acquires an additional opposite phase shift while passing on both sides of the wire. The phase shift difference gives rise to the interference pattern on the screen. The interference pattern was reprinted with permission from American Institute of Physics, ref. 9.

So far, we have treated the experiment as if electrons were waves. And indeed, they may behave as waves, as mentioned in Chapter 1. In such a case, the interpretation of the interference experiment with electrons in Fig. 3 is straightforward: the interference pattern is observed on the screen as predicted by Eq. 3 (see experimental results in Fig. 3a, bottom). However, the devil is in the details. The electrons possess an energy of 50 keV, which allows them to travel at a speed of $0.4c$ through the microscope column of approx. 1.5 m in length. Hence, the travel time from the source to the screen is approx. 10^{-8} s. The electron current was lowered to 10^3 electrons per second, meaning an extremely low probability that two electrons would travel within the microscope at any time. Most of the time, only a single electron is on its way to the screen. Hence, where does the interference pattern come from? The only plausible explanation is that the electron interferes with itself! One should notice how the interference pattern appears from the apparent noise as one electron after another hits the position-sensitive detector array.⁹ As a result, quantum mechanics reveals one of its tricky properties - electrons behave like particles when emitted or detected. However, it is impossible to understand what happens in between the two events.

It is difficult to accept this “explanation” of quantum-mechanical results. Quantum mechanics has surprised its creators even more, and the electron microscope has again been instrumental in demonstrating its strangeness. In 1948, W. Ehrenberg and R. E. Siday reported that the refractive index in electron optics, a unique quantity, depends on the vector potential \mathbf{A} of the electromagnetic field.¹⁰ There was not much attention given to the paper, which primarily targeted the electron microscopy community. Ten years later, in 1959, Aharonov and Bohm stressed the importance of vector potential \mathbf{A} in quantum mechanics.¹¹ That was a revolutionary hypothesis. At that time, the vector potential (as well as the scalar potential) were quantities without any detectable manifestation in nature. J. C. Maxwell created it to make calculations easier. Only the product of the rotation of the vector potential, the magnetic induction vector \mathbf{B} , is measurable:

$$\mathbf{B} = \text{rot } \mathbf{A} = \text{rot } (\mathbf{A} + \nabla f) \quad (\text{eq. 4})$$

Another issue is that the vector potential is not unique, as illustrated in eq. 4. If a gradient of any (continuously differentiable) scalar function f is added to \mathbf{A} , the product of rotation is the same since the $\text{rot grad } f = 0$. As a result, most physicists did not accept vector potentials as significant in quantum mechanics, and any experimental evidence was immediately questioned.

It is not difficult to guess why. Fig. 4a shows such an experimental proposal, sharing many similarities with a double-slit experiment. The difference is the source of a vector potential, located right behind the double slit between the slits so that no electron can pass through the area where $\mathbf{B} \neq 0$. The source of a vector potential is an infinite solenoid, whose characteristic is a constant \mathbf{B} inside the solenoid but zero \mathbf{B} outside. Vector potential \mathbf{A} , on the other hand, is non-zero outside. Vector potential \mathbf{A} affects the electrons passing through the double slit, and they acquire different, opposite phases. This phase difference causes the original interference pattern (without a solenoid) to shift (see Fig. 4a).

It is also due to Tomomura et al.’s ingenious demonstration that the physics community now accepts the Aharonov-Bohm effect.¹² In the initial attempts to demonstrate the effect, different substitutes for the infinite solenoid were used. Many arguments against these experiments were based on the fact that the vector potential sources used were not ideal infinite solenoids, and the electrons were affected by \mathbf{B} , which was nonzero in electron paths.

Tomomura et al. used a toroidal magnet placed in front of the biprism in such a way that one part of the electron wave passed through the toroidal core. The other served as a reference (Fig. 4b). The

two waves interfere behind the biprism. The resulting hologram is reconstructed to get the interference pattern of the electrons (Fig. 4d). By using superconductive cladding, this solution effectively eliminates any possibility of magnetic field \mathbf{B} emanating from the toroidal magnet (Fig. 4c). There is still the effect of vector potential \mathbf{A} on the electron. The effect causes a shift between interference patterns inside and outside the toroid, proving the existence of the A-B effect (Fig. 4d).

These experiments serve as direct real-world demonstrations of quantum mechanics. They demonstrate that quantum mechanics is the most accurate description of our world invented by physics so far. In the next chapter, I will demonstrate how advanced electron microscopy serves fundamental research in materials science.

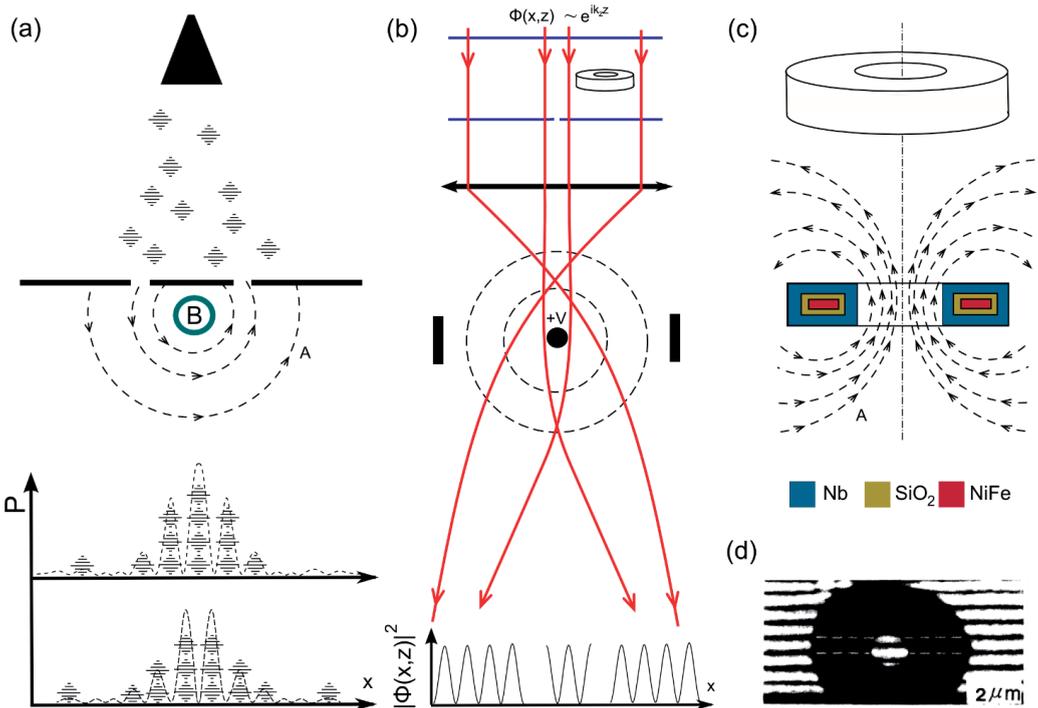


Fig. 4: Quantum phenomena in an electron microscope II: Aharonov-Bohm effect. (a) A modified double-slit experiment gives rise to one of the most debated quantum effects. The probability of detection in a classic double-slit experiment is shown on top. When a source of vector potential \mathbf{A} is placed behind the slits (e.g. infinitely long solenoid) such that the electron cannot pass the area where $\mathbf{B} \neq 0$, the detection probability shifts (bottom) due to the acquired phase shift difference. (b) Realisation of the experiment in an electron microscope. Instead of an infinitely long solenoid, a toroidal magnet (depicted in (c)) with superconductive niobium cladding is used. Here, biprism acts as a lens, allowing the creation of a hologram from the reference wave (left) and object wave (right, passing around and through the toroid core). If the hologram is reconstructed, depending on experimental conditions, the phase difference acquired is seen as a shift between the interference fringes inside and outside of the toroid (d) (reprinted with permission from American Physical Society, ref. 12).

4. BEYOND THE STANDARD ELECTRON MICROSCOPE: IMAGING THE FORMATION OF QUANTUM MATERIALS

“*Measure what can be measured, and make measurable what cannot be*”, Galileo Galilei

Even though electron microscopes are universal tools, they are unlikely to be used for the demonstration of quantum circuitry and electronics; rather, they are used for the study of solid-state materials. Recent years have shown an overwhelming interest in so-called quantum materials, re-ignited by the isolation of graphene in 2004. These materials can serve as tracks for electrons (and more interestingly, other quasiparticles), as in conventional electronics. In graphene, the electron dispersion is linear, similar to that of photons. As a result of this unusual behaviour, quantum effects can be observed within solid-state devices; researchers have already demonstrated different types of interferometers built from a single graphene layer.¹³ However, the requirements for material quality are stringent. As a result, most quantum devices are built using materials exfoliated from bulk crystals, which was the technique originally used in 2004. However, this process is not scalable. Hence, the scientific community’s attention turned to the established deposition techniques; physical and chemical vapour transport. Deposition techniques result in higher defect concentrations compared to exfoliation. Research on the deposition of quantum materials thus faces challenges similar to those of silicon manufacturing half a century ago. There is an urgent need for high-purity, scalable quantum materials. Here, electron microscopy enters our story again, but in a different way than expected. Instead of post-growth imaging of these materials after deposition, the material formation process itself can be observed in real time. Such an approach is termed *in situ microscopy*, and our group is a proud part of this community.¹⁴ In situ electron microscopy has proven to be a valuable tool in many areas of science, ranging from catalysis¹⁵ to materials research.¹⁶ These papers document the most attractive impact of in situ microscopy: observation of phenomena that remain hidden if the system is not monitored under reaction conditions.¹⁷

But there are also other impacts which could eventually outweigh the first one mentioned above. The rise of silicon-based electronics was based on a single key property of the semiconductor-oxide interface: its defect density. Huge investments in perfecting silicon crystal growth and thorough investigation of the properties of the silicon-oxide interface resulted in the manufacturing of the purest man-made material on our planet; for this reason, silicon is used not only in electronics but also in other areas, for example, in metrology.¹⁸ “Perfection” in materials science is often achieved via the concerted use of theoretical modelling and experimental work. The models need to be verified, and, if applicable, they require real experimental inputs like rate coefficients, activation energies, etc.¹⁹ In situ electron microscopy is an ideal tool to satisfy this need, providing valuable inputs for modelling-aided materials design. In further text, I will demonstrate these impacts on materials science utilizing our own, currently ongoing work. Firstly, I will demonstrate that even experiments performed in a high vacuum can provide valuable insights. Next, I will discuss how high-vacuum-based electron microscopes can be used to observe processes happening at high temperatures and pressures.

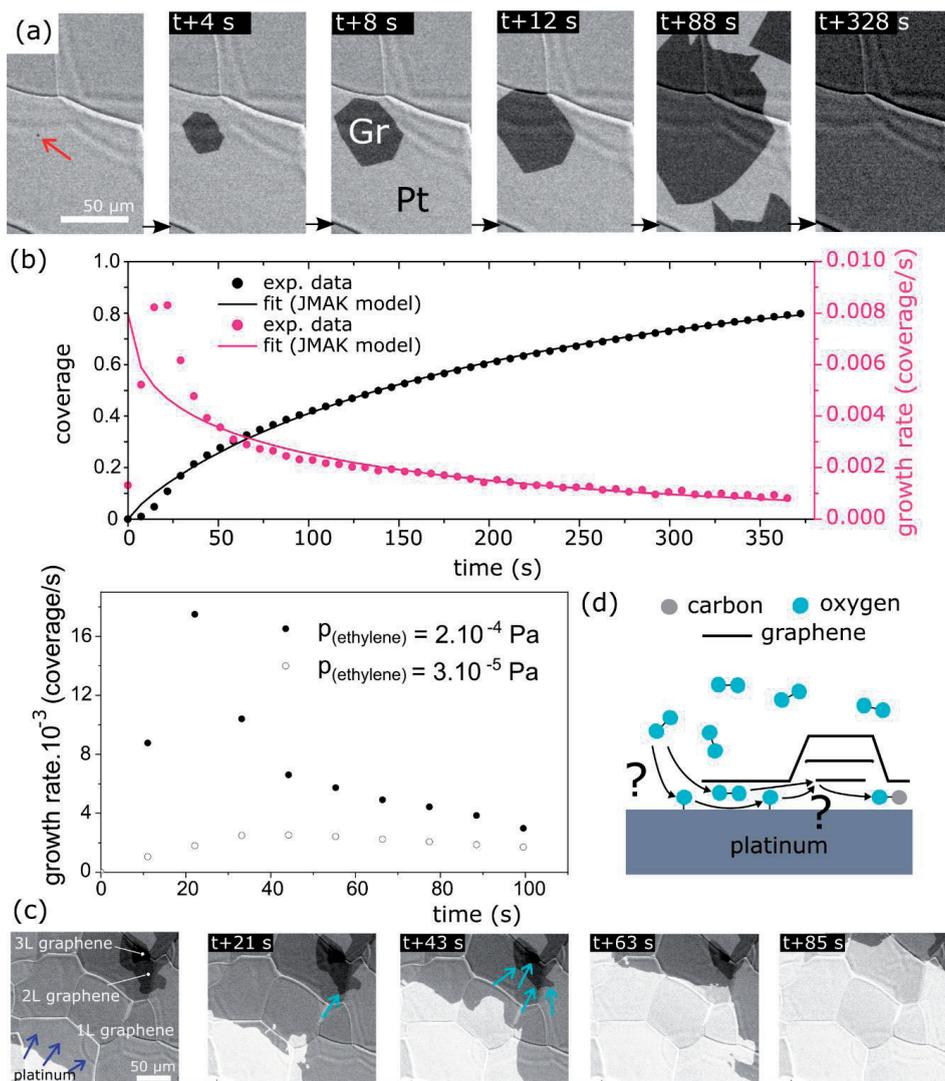


Fig. 5: Utilising a very high vacuum to study the CVD growth of graphene and its etching. (a) Image sequence depicting the growth of graphene on a Pt polycrystal. Carbon is supplied by dissociating ethylene molecules; the base pressure in the chamber is 6×10^{-7} Pa. Therefore, the hydrogen and oxygen fractions in the residual gas are very low. The red arrow marks the nucleation site. (b) Dependence of coverage and growth rate on time (taken on a different grain than in (a)). (c) Image sequence demonstrating etching of graphene within the van der Waals gap. Graphene is prepared in the “inverted wedding cake” geometry (see the schematic in (d)). The etching rate of the first layer of graphene by oxygen is constant, and the etching front (dark blue arrows) of the top graphene layer propagates from the left bottom corner up and to the right. Surprisingly, the graphene layers beneath the top one start to etch even before the etching front reaches its edges (cyan arrows). The schematic in (d) shows just a few possible etching scenarios: dissociative oxygen adsorption, diffusion of atomic or molecular oxygen, and one of the possible etching products, the CO molecule.

Chemical Vapour Deposition (CVD) of graphene does not require high pressures. Instead, a very high vacuum turns out to be advantageous because the effect of different molecules within the residual gas in the growth chamber can be recognised and suppressed. Fig. 5a shows the growth evolution of graphene flake on Pt polycrystal. Automated image recognition makes it easy to plot different growth characteristics (e.g., area/growth rate versus time, etc.) and to model them with conventional growth models.²⁰ Comparison with experimental data allows for the fitting of input model parameters and thus a deeper understanding of growth mechanisms. It is obvious that in situ experimental data are critically important. Specifically, while the classical growth model predicts a steadily decreasing growth rate as a result of the shrinking catalyst substrate surface, our data show a deviation from this behaviour in the early growth stage. Initially, the growth rate increases over time. This behaviour suggests that in the early stages, the growth is fed by the direct decomposition of the precursor and another source of carbon atoms. It is plausible that the carbon dissolved in the bulk platinum is considered as the additional carbon source. Such conclusions allow us to build more realistic growth models and lead to customised and optimised growth recipes.

In situ observation of chemical reactions is irreplaceable in identifying effects that are hidden from common ex situ analyses. For example, Fig. 5c shows the etching of multilayers of graphene (again on a platinum polycrystal) by oxygen. The graphene was prepared in an “inverted wedding cake” configuration, which is schematically shown in the figure as well. As a matter of fact, one would expect the outer graphene layer to etch first, followed by the layers below, until the first graphene layer on top of the platinum is etched away. The image sequence reveals a surprising fact: the bottom layers start to etch even before the etching front of the top graphene layer reaches them! This observation implies an exciting conclusion. The oxygen molecules (or atoms) diffuse *below* the outer graphene layer, within the so-called van der Waals gap, and etch layers below the cover of the outer graphene layer. On the basis of in situ imaging, the etch rates could be quantified and compared between the different layers. Theoretical modelling predicts an enhanced reaction rate within the gap,²¹ which would be a promising approach towards, e.g., rate enhancement of catalytic reactions. In situ real-time imaging is the most relevant and reliable way to prove or disprove this hypothesis.

Ultra-high-vacuum experiments are extremely valuable, but their relevance to realistic process conditions remains limited. One of the most striking examples is the question concerning the existence of platinum oxide,²² which has been bothering the catalysis community for decades. According to current understanding, platinum oxide forms on the surface at high oxygen pressures (above several millibars), whereas at lower pressures, oxygen is only dissociatively adsorbed. Thus, the reaction mechanisms deduced from many high-vacuum studies cannot be projected to real catalytic conditions. Our current knowledge of this essential industrial catalyst is still incomplete. More examples of surface structures that exist only under high pressure conditions could be found.

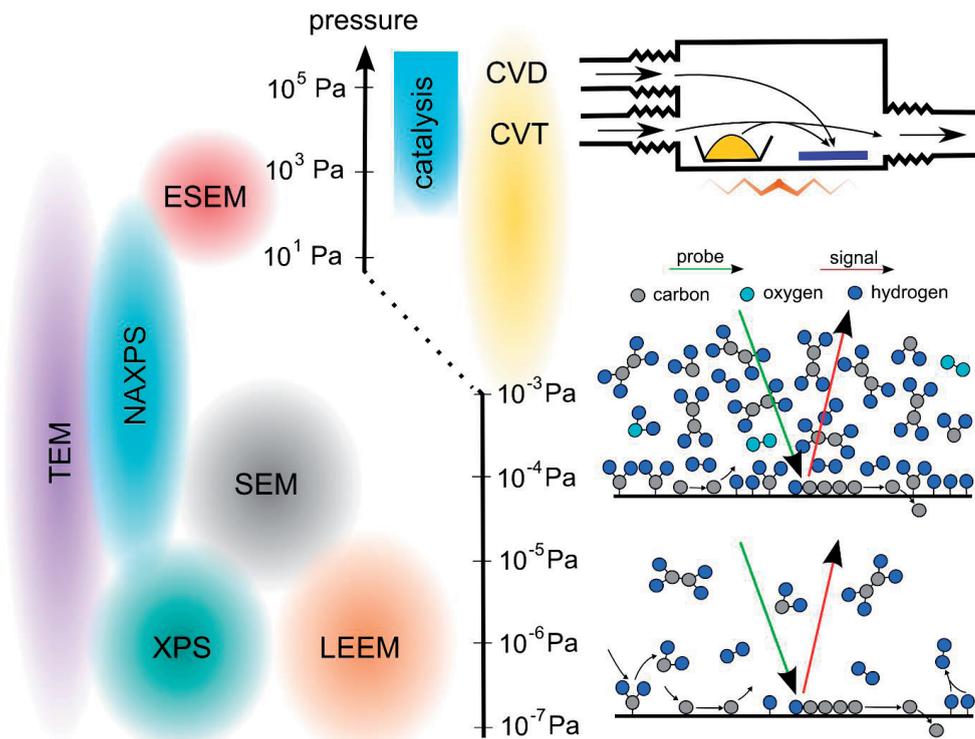


Fig. 6: Pressure gap(s). On the left, operational pressure ranges of standard scanning electron microscopy (SEM) and low-energy electron microscopy (LEEM), including X-ray photoelectron spectroscopy (XPS) as a representative of the surface sensitive spectroscopic technique, are shown. Environmental SEM (ESEM) and near-ambient XPS (NAXPS) are 'high pressure' versions of these techniques. Catalysis and chemical vapor deposition (CVD) and chemical vapor transport (CVT) are examples of processes working at higher pressures (on the right). Pressure gaps between analytical techniques and real processes are apparent. On the other hand, thanks to the recently employed MEMS-based approach (Fig. 7d), the TEMs operate over a wide pressure window. The sketches on the right illustrate two main issues emerging with increasing pressure. First, the probe or signal (or both) is significantly attenuated at higher pressures. Next, the signal is compromised by many 'parasitic' processes, which cannot be easily separated from each other, as in the case of a very high vacuum. However, the pressure gap arises primarily because of the inability of the techniques to work at elevated pressures. This is limited mainly by some parts of the analytical system that require high vacuum for operation.

Furthermore, it is not only the presence of such intermediate structures, but also their activity within the process studied and the overall reaction kinetics that profoundly affect the reaction output. Intensive exchange of species between the solid surface and the gas phase is limited in high-vacuum experiments. Thus, these studies offer just a glimpse of the complex mechanisms involved in most reactions. This gap between the working conditions of available analytical techniques (with particular emphasis on electron microscopy) and the reaction conditions of realistic processes is depicted in Fig. 6. Low-energy electron microscopy (LEEM) is extremely surface-sensitive due to

the low landing energy of primary electrons (0-40 eV). That is achieved via a cathode-lens effect - a high voltage applied to the sample. The risk of sparking between the sample and the objective lens increases at elevated pressure, possibly resulting in irreversible sample damage. As a result, this effect limits the maximum pressure in the chamber during measurement. Other electron microscopes can cover a higher pressure range. Equipped with sets of pressure-limiting apertures, the modified microscopes can operate up to relatively high pressures. These solutions, however, come at the cost of deteriorating resolution, since various other detection schemes are required. The situation is more complicated for SEMs, as compared to TEMs. TEM has the advantage of having a very small sample and related sample volume within the microscope. SEM is more versatile; however, the large chamber, potentially filled with a gas, represents a significant challenge, especially for safety reasons. Hence, with TEM offering atomic resolution and simpler adaptation to high pressures, why bother with SEM? The small sample size in TEM is its Achilles heel. Imaging technologically relevant processes often requires large fields of view. A complete picture of the process is not provided by atomic resolution. Imagine a pocket watch - what would the movement of a single cogwheel tell the engineer about the working principle of the watch? From this perspective, SEMs are an indispensable tool in material research, providing variable viewfields of bulk samples. Fig. 6 shows some technological processes relevant to in situ electron microscopy studies. Catalysis is a domain of surface science; however, the techniques with the best surface sensitivity require a very high vacuum. Therefore, the pressure gap in catalysis research is the subject of intensive and continuous instrumental development. Chemical Vapor Deposition and Chemical Vapor Transport (CVT) are conducted at high pressures as well. Performing these processes in SEM is often considered challenging, especially because of the possible contamination by process species. However, in the following paragraphs, I will show that these experiments are very rewarding once performed.

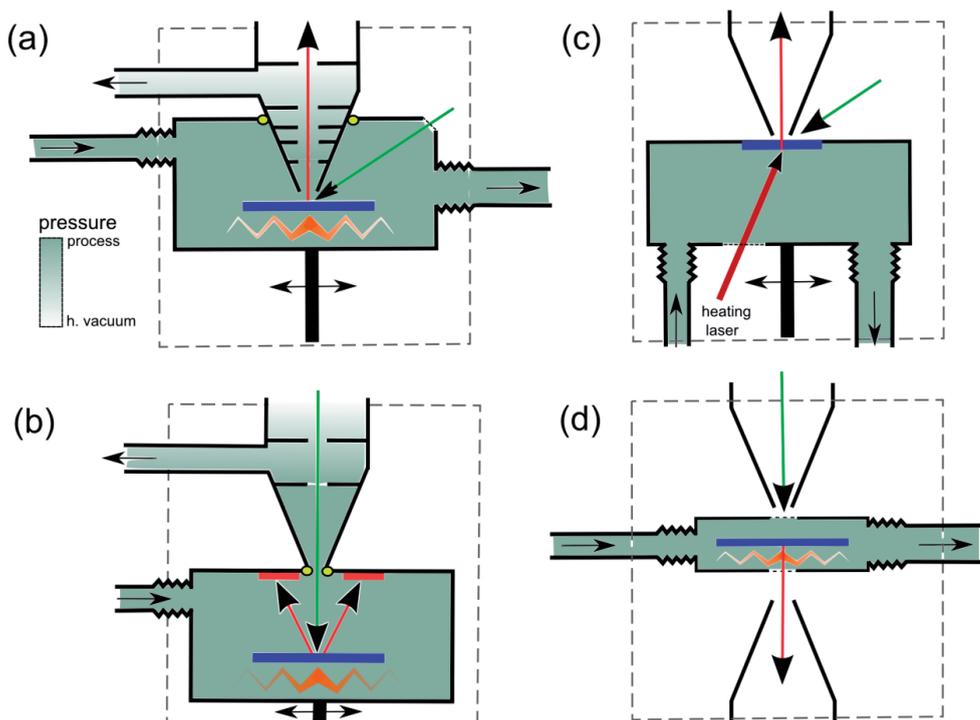


Fig. 7: How to bridge the pressure gap. (a) A differentially pumped analyser is used in NAXPS, similar to ESEM. (b) The concept of an inverted cell allows the use of a standard XPS analyser. (c) Reactor-in-SEM, where the detection system is placed inside the reactor (utilised by our group in).²³ (d) Miniaturization of the reactor to the extreme: a sandwich made of two atomically thin membranes with the reaction volume in between, including a MEMS sample heater. This universal approach is commonly used in TEM and can also be used in XPS and SEM. The pros and cons of these solutions are discussed in the text.

There are many lessons to be learned from the workhorse of surface science, X-ray photoelectron spectroscopy (XPS). The most pursued approach is to design a differentially pumped analyzer and a high-pressure sample-containing cell (Fig. 7a). Pressures can reach up to 130 mbar²⁴ and even higher these days. The application of similar principles to SEM is known as Environmental SEM (e.g., ESEM), where a series of pressure-limiting apertures lower the pressure gradient across the electron column. In connection with a dedicated pressure cell,²⁵ such a system can offer high-pressure imaging at a slightly deteriorated resolution. Compared to XPS, the detection system is placed inside the cell (Fig. 7b). We have demonstrated the successful use of such a setup for gallium oxidation reactions.²³ These experiments identified a number of significant pitfalls in this design. Reaction products easily and quickly contaminate the detector, resulting in its failure. Therefore, a distinct detector design or a conceptually different solution that places the detection system outside the pressure cell, preferably in the high-vacuum environment of the microscope chamber, must be sought. The latter concept has the advantage that the pressure cell (from now on called a reactor) can be integrated into an existing SEM without significant hardware changes. One of the approaches is to use an inverted pressure cell (Fig. 7c), where the rear side of the sample is exposed to the probe beam. The reaction proceeds on the front side. Indeed, the sample has to be relatively thin for the

ejecting signal to be detected above its rear side – usually, the graphene membrane support for reactive nanoparticles is used.²⁶ In transmission electron microscopy, similar approaches have been commercialised using encapsulated MEMS chips (Fig. 7d). Once again, the sample has to be very thin. Hence, even though almost realistic process conditions can be reached within these solutions, the sample is ill-defined, rarely reflecting real-world bulk samples and their surfaces. Unlike TEMs, SEMs permit using bulk samples even on MEMS chips enclosed within a small volume that can be pressurized (see Fig. 8a). This technology was developed in Thermo Fisher Scientific. The manufacturer and our group have been working closely on its applications. One of the experiments currently being conducted in our laboratory is the growth of 2D materials on liquid substrates.

The seamless stitching of domains is crucial to the formation of monocrystalline 2D materials on large scales. The emergence of electronically detrimental domain boundaries can be mitigated by tailoring the crystallographic orientation of the substrate to match the symmetry of the 2D material (see Figs. 8a-d). This is possible for graphene, which exhibits six-fold symmetry, but is increasingly difficult for other materials of interest. Therefore, a universal approach is being sought. Rheotaxy (growth on a liquid substrate) was proposed as a viable method to achieve domain ordering and self-assembly in 2012 for graphene.²⁷ However, it remains poorly explored due to a lack of in situ experimental techniques that confirm or disprove hypotheses about the formation and stitching mechanisms.²⁸ On a solid substrate, the most straightforward strategy is to suppress subsequent nucleation events after the initial one, which avoids any stitching at all. That is, however, very challenging to follow experimentally (see, e.g., Fig. 5a, where multiple nucleation events are observed), as there are too many knobs to turn. As stated at the beginning of this chapter, the growth modelling suffers due to the lack of reasonable quantitative inputs. Diffusion coefficients of atomic building blocks, attachment/detachment rates, and nucleation barriers need to be quantified experimentally to make the growth simulations quantitative. However, these data are unknown; their absence is the biggest obstacle to modelling real systems. Hence, a lack-of-data-based gap exists between growth experiments and growth modelling.²⁹ I have already shown in Fig. 5 that such data can be obtained from in situ microscopic experiments.

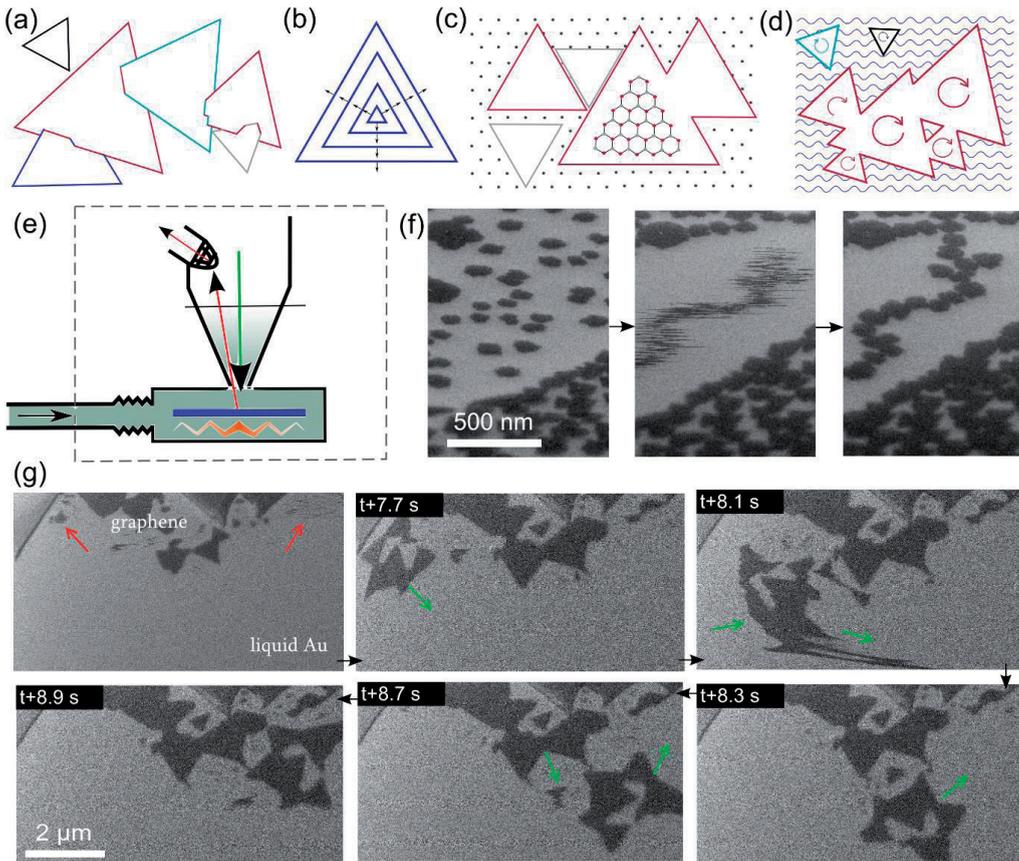


Fig. 8: Toward CVD rheotaxy in a scanning electron microscope. (a) Multiple random nucleation events result in the formation of a polycrystalline layer, where the grain boundaries separate different oriented domains as they meet during growth. This is the most significant issue for the scalability of the 2D-materials growth process. The solutions include (b) nucleation of a single nucleus while suppressing other nucleation events, and (c) growth of oriented domains, which requires, e.g. single-crystal substrate. It works for graphene, but for 2D materials with lower surface symmetry, this approach cannot prevent, e.g. twin GB formation (see panel (c), where red and grey edges meet). If a liquid substrate is used, the domains are hypothesized to rotate freely and align (d). A schematic of a MEMS-based μ Reactor is shown in (e). (f) Image sequence showing the transition of a gold substrate from solid (left) to liquid (middle) at 1050 °C. The phase change is fast (the scan time is 0.18 s) and the graphene grains float on the liquid surface until they are anchored (right). (g) Graphene can nucleate even on the liquid surface (red arrows) at 1085 °C, and the growing grains are again very mobile (green arrows in the images). The movement stops immediately if the domains are attached to a stable large-domain cluster. Interestingly, no assembly is observed, compared to other reports.²⁷

The MEMS chip enclosed in a microreactor (Fig. 8e) allows the melting of any piece of material with a melting point of up to 1100 °C. We have chosen gold ($T_m = 1064$ °C) as the growth substrate,

as it has a very low vapour pressure, ensuring its stability in the liquid phase during the experiment (Fig. 8f-g). The individual graphene domains grown on the surface of liquid gold partially exhibit expected behaviour; they are very mobile on the surface. Contrary to theoretical predictions, they are rarely seen to align and seamlessly coalesce (Fig. 8f). Instead, they behave as though they are stitched together without any evidence of regular assembly. Our further research into this phenomenon will focus on the effect of the surrounding atmosphere on the floating grains (specifically hydrogen, which can potentially terminate graphene edges), as well as nucleation probabilities on solid and liquid surfaces (although it is claimed difficult to see nucleation events on a liquid surface, Fig. 8g). Such data are of general interest, even beyond the community of 2D materials.

MEMS reactors offer close-to-real process conditions, but sample preparation is time-consuming and challenging. Hence, our latest activities aim to develop a macroscale reactor that permits the use of real bulk samples (e.g. pieces of silicon wafer), thus mimicking CVD and CVT tubes. Such an approach also offers other advantages over microscale reactors. Most importantly, the precursor for growth can be vaporised close to the sample, similar to conventional CVD and CVT tubes (see schematic in Fig. 6, top right). Complicated precursor delivery systems are thus avoided; however, it poses new challenges to reactor design.

Nevertheless, we have managed to build such a system (Fig. 9c) and demonstrate its use within the real-time in situ experiment of ZnSe nanowire growth (Fig. 9d,e). ZnSe powder is vaporised by the precursor heater. A carrier gas transports the vapour toward the sapphire substrate, which is held at a different temperature by a separate heater (usually at a lower temperature than the precursor). The sapphire substrate is covered with gold nanoparticles, which serve as collectors for both Se and Zn atoms, making their condensation site specific at the droplet location due to the vapour-liquid-solid process.³⁰ The droplets are pushed within the predefined trenches on the substrate by the growing ZnSe crystal behind, and thus in-plane nanowires are formed on the substrate. This technology has been envisioned to become one of the approaches towards nanowire-based electronics³¹ (Fig. 9a). However, nanowire-based architecture requires a thorough understanding of growth mechanisms, especially those that govern the growth direction of nanowires. Because of the in situ microscopic approach, we were able to watch the nanowires grow in real time and reveal the causes of their misalignment with the predefined trenches on the substrate. These include structural defects within the trench structure, surface contamination, and noncatalyzed deposition of ZnSe on the surface (Fig. 9d). Therefore, this study presents a clear picture of the requirements for substrate and process cleanliness in the (possible) future. In addition, we were able to track individual nanowires and quantify their growth rate dependence on nanowire radius (Fig. 9e). Such data are possible to fit with appropriate growth model³² and provide valuable insight into the growth process. In this particular case, the slope of the dependence in Fig. 9e gives information on the dimensionality of surface diffusion. Similar data are impossible to extract from the ex-situ growth experiments, because the nucleation delay significantly affects the resulting nanowire length. As a result, ex-situ analysis often leads to erroneous conclusions.

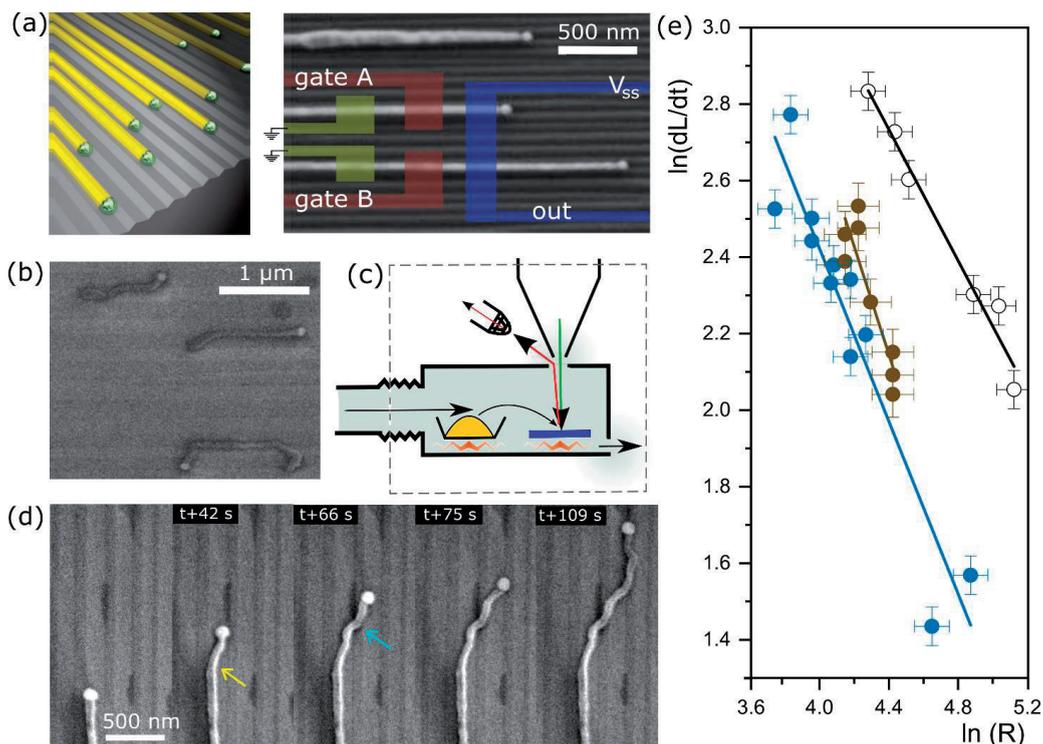


Fig. 9: Mimicking CVT reactions. (a) Electronics based on in-plane nanowires is envisioned as a promising technology, especially due to the integration of other materials on a silicon platform. The requirement is absolute control of the growth direction (reprinted with permission, © Ernesto Joselevich). Ideally, one can build simple logic gates based on these nanowires (the image on the right shows a PMOS NAND gate). (b) In reality, it is often not the case, as the in-plane growing wires are found outside the predefined trenches on the substrate surface. (c) A schematic of a macroscale reactor designed to mimic CVT growth of these nanowires in an SEM. The detection system is placed outside the reactor (contrary to the previous design shown in Fig. 7c), significantly improving the resistance to contamination. The color coding and symbols are identical to those used in Fig. 7. (d) An image sequence showing the growth of a ZnSe nanowire, guided by a gold droplet, on a surface of sapphire. Several events can divert the nanowire from its original straight growth trajectory – unseen contamination on the surface (yellow arrow), carbon contamination (cyan arrow), and possibly also corrugation of the surface trench geometry (not shown, see ref. 32). (e) Nanowire growth rate dependence on radius (both in logarithmic scale). Each point represents a single nanowire, different colors mark different experimental runs.

5. SUMMARY, IMPACT AND OUTLOOK

“Seeing is believing is a blind spot in man’s vision.” Buckminster Fuller

In this thesis to the inaugural talk, I first used electron microscopy to demonstrate the relationship between science and technology. This relationship is often simplified with the pipeline model. However, the reality is far more complex, as described in the first part of the thesis. I disproved the pipeline model in the second part by turning it upside down. I have shown that technology serves as a facilitator for fundamental research driven by curiosity. The examples given include the double-slit experiment and Aharonov-Bohm effect, ingeniously demonstrated using an electron microscope by Tomonaga et al. Indeed, there are even better examples of the inverted pipeline (e.g. discovery of cosmic microwave radiation) than the one presented here. Still, the choice was made concerning my own work and, of course, concerning the tradition of electron microscopy research and development in Brno. I believe that an understanding of the science-technology relationship should be explained to students during university courses because it is important to our society in general. Attention should be paid to all the different disciplines that contribute to the technological advancement of mankind.

In the third part, I focused on our work in the field of in situ microscopy. By taking advantage of the fertile soil of microscopy research in Brno, and collaborating with other industrial entities, we are able to visualise things not seen before. Applying the above principles allows us to perform curiosity-driven research due to the development of novel instrumentation. Within these studies, we collected valuable data on processes envisioned as future directions in the preparation of nanoscale materials. In this part of the thesis, I have also demonstrated that there are ways to mitigate the pressure gap between the operational conditions of an electron microscope and a real process environment. However, it is far from resolved. Bridging this gap will require unrelenting effort in the coming years, and it is a very promising field of research and development.

It is very tempting to draw conclusions based solely on unique microscopic observations. However, the microscopic experiments must be corroborated by other techniques and analyses. The choice of experiments presented here was made with respect to this fact; the obvious example of incomplete description is the etching of graphene within the van der Waals gap shown in Fig. 5c. To perceive the world around us, we also need other senses (e.g., the microscope, see Fig. 10). Consequently, implementing novel in situ techniques within an electron microscope is another promising way to increase the impact of these studies. We are currently conducting research in this area. Indeed, the development of working solutions is challenging; however, it is very rewarding when it is successful.

I have also shown that electron microscopy serves as an appealing demonstration tool of various quantum phenomena. As such, it can be easily implemented in multiple university courses. As a side effect, students become increasingly aware of the microscope operating principle and the technology advancement it represents. I utilise the experiments of Tomonaga mentioned in this talk in “Modern physics” courses. The results of our in situ experiments serve as input data for calculations in the “Surfaces and Interfaces” course. In a lecture in “Diagnostics of nanostructures”, I make use of many of our experimental results to demonstrate the capabilities of electron microscopy and spectroscopy.

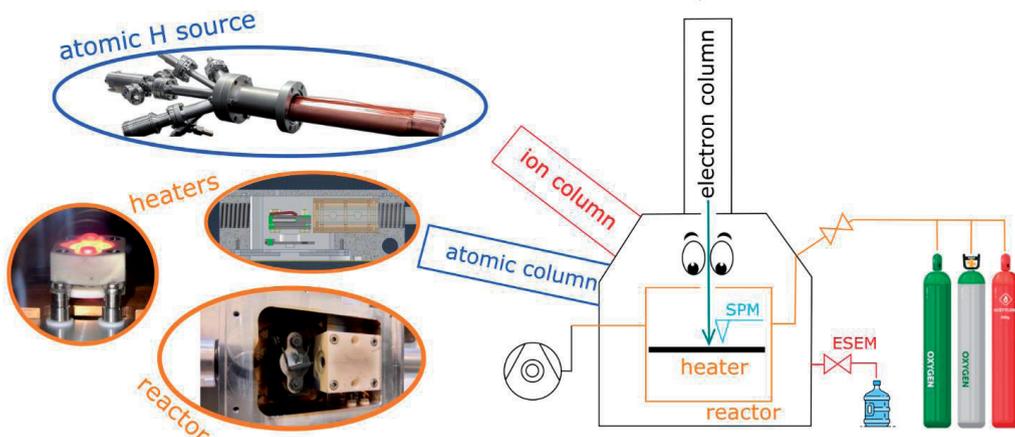


Fig. 10: Electron microscope as a laboratory for research on quantum materials. *A conventional scanning electron microscope (black) is a dedicated tool for observing bulk samples, utilising an electron beam as a probe and various detection schemes for detecting distinct signals (electrons, X-rays, and light). These two principal components are continuously improved over time. Disruptive events in microscope development were the introduction of the focused-ion beam (FIB) and ESEM (red). Our work focuses on utilising SEM for complex experiments dealing with (quantum) materials. Such an aim requires modifications of the microscope, including the development of unique instrumentation. This includes, e.g. a column for the generation of atomic beams (blue), reactor-in-SEM including a hot stage and a gas injection system for in-situ microscopy (orange), etc. These activities would not be possible without the support of an industrial partner (Thermo Fisher Scientific). Image formation is not necessarily limited to electron-beam-induced signal; in collaboration with NenoVision, we pursued the pathway towards correlative imaging utilising scanning probe microscopy. We develop methodologies for advanced modes of SPM (e.g. Kelvin probe force microscopy) in conjunction with the electron beam. The know-how on surface science and related processes (and characterisation techniques) we carry allows us to get a deep insight into the formation of quantum materials, their interaction with the electron beam, and behaviour under reactive atmosphere and under real process conditions.*

As introduced in Chapter 4, we focus on synthesis of (nano)materials, their polytypes or assemblies that are not naturally occurring in nature. Electron microscopy is only one of the tools we use. Our work can be considered multidisciplinary and, of course, cannot be performed by an individual. I have had the opportunity to gather a group of enthusiastic people with complementary interests (Fig. 11) who share the passion for physics, materials science and in situ microscopy. Where appropriate, we transfer the newly developed processes of material growth into an electron microscope and perform experiments like those presented in this thesis. The group welcomes bachelor and master students as well; new students within the group have a wide choice of what to focus on: experimental work, designing new instruments and tools, modelling, data evaluation and programming, etc. Hence, in addition to being an appealing research direction, in situ microscopy (and materials research in general) is an attractive platform for the education of undergraduate and PhD students.

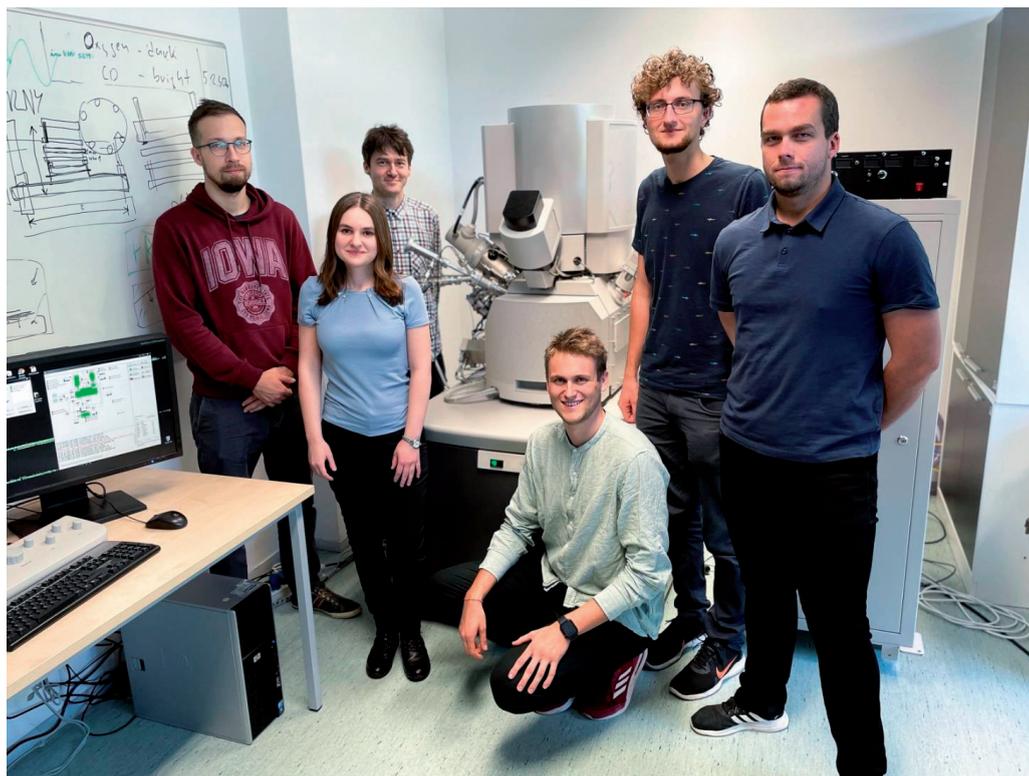


Fig. 11: The team in the lab, spring 2021. From left: Tomáš Musálek, Kristýna Bukvišová, Miroslav Kolíbal, Karel Vařeka, Michal Drozd, Daniel Citterberg. Moreover, other team members not present on the photograph have to be acknowledged, as they contributed to the results and group activities: Martin Kovařík, Jiří David and Matěj Nedvěď (all on Erasmus leave), Michal Dymáček, Marek Patočka and Hossein Mirdamadi.

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ABSTRACT

This thesis demonstrates my approach to research and education, two integral parts of my academic career so far. In the first part of this thesis, the complex relationship between science and technology is exemplified using electron microscopy as an example of technology built upon the foundations of quantum theory. Several examples illustrate the use of electron microscopy as a demonstration tool for quantum physics lectures. The largest part describes the in-situ experiments within electron microscope, revealing the latest achievements in mimicking the reaction conditions required for the growth of nanoscale materials. Examples include graphene growth and etching within a van der Waals gap on a platinum surface, graphene rheotaxy on liquid metal, and ZnSe in-plane nanowire growth on patterned sapphire. An outlook for the research field, particularly focusing on our own activities, is briefly discussed at the end.

ABSTRAKT

Tato práce si klade za cíl nastínit můj pohled na výzkum a vzdělávání, dvě nedělitelné součásti mé dosavadní akademické kariéry. V první části je popsán komplexní vztah vědy a technologií na příkladu elektronové mikroskopie, tedy technologie vybudované na základech kvantové teorie. Je uvedeno několik příkladů kvantových jevů, které lze demonstrovat pomocí elektronového mikroskopu. Největší prostor je věnován in-situ experimentům v elektronovém mikroskopu. Jsou popsány nejnovější výsledky dosažené ve snaze dosáhnout v mikroskopu takové experimentální podmínky, které panují v reaktorech pro přípravu nanomateriálů. Mezi popsané příklady patří příprava a leptání grafénu v prostoru van der Waalsovské mezery mezi grafénem a povrchem platiny, tvorba grafénu na tekutém kovu a tvorba ZnSe nanodrátů na tvarovaném povrchu safiru. V poslední části je krátce diskutován výhled a budoucnost tohoto výzkumného směru, s důrazem na naše aktivity.