

CHAPTER 1

INTRODUCTION TO IN-SITU ELECTRON MICROSCOPY

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This chapter gives an introduction to *in-situ* electron microscopy. The historical background, the achievements, and modern techniques of *in-situ* electron microscopy are briefly reviewed, and the limitations of the technique as well as the prospects for future developments are discussed.

1. Definition and History of *In-Situ* Electron Microscopy

The interest in structures with sizes on the micron, nanometer, and eventually atom cluster or molecule scale has resulted in the development of sophisticated tools of microscopy during the past decades. Today, materials or biological science cannot be imagined without the characterization techniques of modern transmission electron microscopy.^{1,2} Transmission electron microscopes permit a view into the interior of small objects and are complementary to scanning tip microscopies that provide images from specimen surfaces. Due to continuous efforts in electron optics, the lateral resolution of electron microscopes has now dropped below 0.1 nanometers, and this scale is already smaller than the distance between atoms in densely packed crystals or in molecules. Nowadays, images with the resolution of crystal lattice spacings are recorded as a matter of routine and even individual atoms have been observed, though in special systems only. With the ability of forming strongly focused electron probes, an electron microscope is also able to provide the platform for analytical techniques with high lateral resolution such as energy-dispersive X-ray or electron energy loss spectroscopy.

By looking through textbooks of electron microscopy, it appears that microscopy is just able to provide information from the space of the

objects. However, time is another parameter and, indeed, it has been demonstrated since decades that experiments can be conducted in real time inside the specimen chambers of electron microscopes. This field of experimentation became known as *in-situ* electron microscopy. The monitoring of the image in real time allows us to study structural changes in the specimens and, thus, to use the electron microscope as a ‘nanolaboratory’ for carrying out experiments on a small spatial scale.

In-situ electron microscopy dates back to the 1960s when serious problems in materials science, for example, the fatigue of metals for applications in aviation, had to be solved. The need to design spacious experimental setups in the specimen chamber of the electron microscope resulted in the development of high-voltage instruments operating at or above 1 MV and with large gaps between the objective pole pieces. The lateral resolution of these machines was hardly below 1 nm but it was possible to introduce specimen stages with dimensions of several centimetres. Electron-transparent metal sheets have been strained and monitored at the same time so that the movement of defects, e.g. dislocations, was accessible to direct observation.³ Dedicated stages were designed that allowed to heat specimens up to high temperatures in the microscope. In such a way, phase transformations were observable, though not on a very small scale. Imaging was in most cases carried out in diffraction contrast (bright or dark field imaging of specimens under Bragg conditions) whereas electron diffraction gave information about the crystallography during transformations of the material.

Some *in-situ* experiments were based on the accidental observation of dynamic processes in the specimen during normal inspection in standard stages. An important field has been electron irradiation of specimens which is generally unavoidable during electron microscopy. The energetic electron beams in high-voltage electron microscopes were used to generate and study radiation damage and to simulate the behavior of materials for applications in nuclear reactors or in space.

2. Modern *In-Situ* Electron Microscopy

Numerous advancements have been achieved in electron microscopy in the past decades. Not only has the spatial resolution been improved,

image recording techniques have been revolutionized by the application of CCD cameras, dedicated specimen stages have been made with micromechanical tools, and analytical spectroscopies with almost atomic resolution have been integrated into standard electron microscopes. All these improvements had their impact in the development of modern *in-situ* electron microscopy. Systems with dimensions on the nanometer scale are in the focus of interest at this time and particularly well suited to experiments in the electron microscope. These experiments have also led to the discovery of new phenomena on the scale of nanoparticles or atom clusters.

Many *in-situ* experiments in the last years have been carried out with a spatial resolution of better than 0.3 nanometers. On this scale, the atom columns in well aligned crystals become visible. In some studies, the monitoring of even single heavy atoms within light materials has been achieved, though with considerable image noise in the recordings.⁴⁻⁶ But it still remains a fascinating goal of *in-situ* studies to ‘see the atoms moving’. An advantage of transmission electron microscopy is the parallel recording of the whole image. Scanning tip microscopies, on the other hand, need a certain time to scan the image and can ‘see the atoms’, but only one at a time so that dynamic processes are difficult to monitor. Dynamic processes where many atoms in a crystal lattice are involved and lattice planes change their position are ideally suited for high-resolution *in-situ* electron microscopy as will be demonstrated in the following chapters of this book.

However, some difficulties remained a challenge to the experimentalist. Lattice resolution of a crystal is only achieved when a low-indexed zone axis of a crystal is precisely aligned parallel to the electron beam. This condition is often difficult to fulfill in nanosystems that are subjected to mechanical, thermal, or electrical influence during the experiments. Another difficulty is the signal-noise ratio which is often quite high in high-resolution images that have to be taken with short exposure times. The image formation in high-resolution electron microscopy is based on phase contrast which has to be converted to amplitude contrast by the optical system to make the object details visible. The contrast in high-resolution images is generally much lower than in conventional mass-, thickness- or diffraction contrast images. Time-resolved *in-situ* electron microscopy

does not allow long exposure times of single image frames, therefore the problem of image noise cannot be overcome.

The output of *in-situ* electron microscopy is generally a series of images that is either taken manually frame by frame or recorded as a real time video. Image intensifiers with attached TV cameras have been used since decades although they provide noisy images when used at high magnification of the microscope. Nowadays, CCD cameras with high sensitivity and low noise are replacing photographic films or TV cameras. Multi-scan cameras enable us to record single frames or video sequences in the electron microscope, just like in digital consumer cameras or camcorders. With the steadily increasing computing power, online image or video processing is now possible. Offline processing allows the selection of suitable frames from the videos and to extract the whole information from the recordings.

In-situ experimentation needs specially designed specimen stages that fit into the objective lens and contain the whole setup around the specimen. Due to advancements in miniaturization techniques, specimen stages for many experiments can now be made small enough to fit even into the narrow gap of objective pole pieces for high resolution microscopy. Nowadays, high-voltage microscopes with large specimen chambers are only needed for very special setups or for irradiation experiments. Specimen stages for several applications are now available commercially, for example for heating, cooling, electrical probing, straining, or indentation of the specimen. Even scanning tunneling or atomic force microscopy tips have been integrated into these specimen stages so that mechanical manipulations of the specimen can be carried out by piezo drivers with highest precision and the simultaneous imaging of the specimen by TEM and AFM resp. STM became feasible.^{7,8} These stages allow *in-situ* experiments in almost every standard electron microscope. On the other hand, the columns of some microscopes have been modified for special experiments, for example, microscopy in a gas atmosphere,^{9,10} crystal growth in ultra-high vacuum,¹¹ ion irradiation of the specimen,¹² or the application of pulsed laser beams for nanosecond microscopy.¹³ Complicated setups have been attached to the columns of the microscopes that sometimes needed more space in the laboratory than the microscope itself.

The demands on the stability of the setup are particularly high in *in-situ* electron microscopy at high resolution because the specimen is not allowed to drift or vibrate by more than the desired image resolution. Moving parts of the specimen stages may cause mechanical vibrations, and local magnetic or electric fields may deteriorate the image formation in the objective lens. Another difficulty is the thermal expansion of the specimen stages which is unavoidable in heating experiments. Specially designed electronic image drift compensation systems have helped to overcome some of these problems. Furthermore, specimen preparation techniques had to be developed and adapted to the specific requirements of *in-situ* experimentation. Standard preparation techniques that are applied as a matter of routine for inspection in the electron microscope are often not suitable for *in-situ* experiments.

3. The Techniques of *In-Situ* Electron Microscopy

A great variety of *in-situ* experiments has been carried out in the past decades. Many special setups have been designed and built in the electron microscopy labs. Only the most common types of experiments, or those where specimen stages or special setups are available commercially, will be summarized in the following. Figure 1 shows the principle of *in-situ* experiments in a schematic drawing.

The response of materials to mechanical stress was one of the first applications of *in-situ* electron microscopy.³ The straining of specimens in specially designed stages has been carried out at ambient or high specimen temperature.¹⁴ The nucleation, glide, or pileup of dislocations or the operation of dislocation sources has been made visible in impressive video sequences. In more recent experiments, nanoindentation of materials by tiny diamond tips have been applied to study deformation mechanisms on a small scale.¹⁵ The integration of an AFM into the TEM specimen holder⁷ allows one to measure small forces resulting from the elastic response of the specimen.

The variation of the specimen temperature by resistive heating of the holder has also been carried out since a long time. Modern heating stages allow imaging with lattice resolution at specimen temperatures up to more than 1000°C. Phase transformations such as solid-solid or solid-liquid

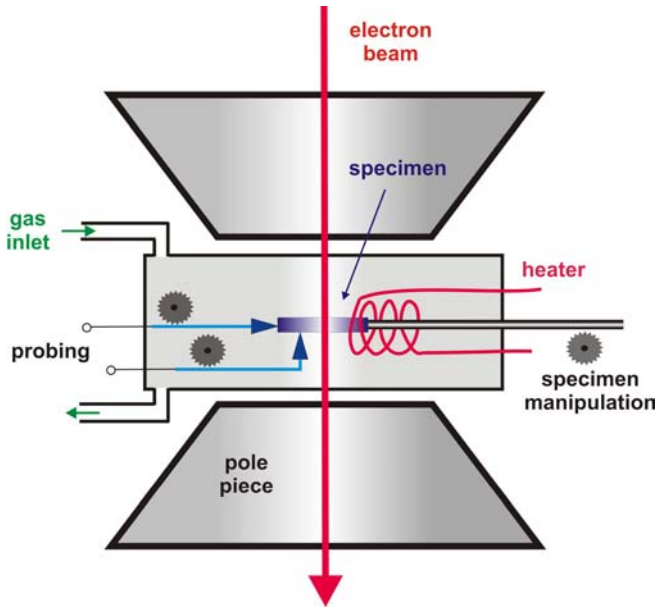


Fig. 1. Principle of *in-situ* experimentation in the transmission electron microscope. The specimen can be manipulated by heating, straining, electrical probing, or reaction in a gas atmosphere during inspection at high magnification. (In a real experiment, not all manipulations as shown here will be carried out in a single setup.) The space between the pole pieces of the objective lens limits the dimensions of the “lab inside the microscope”.

transitions or chemical reactions can be studied *in-situ* by varying the specimen temperature. *In-situ* microscopy of thermal effects is particularly interesting in nanomaterials because macroscopic characterization or analytical tools do not provide the information needed for understanding transformations of clusters or particles that, themselves, have dimensions which are only accessible by techniques of high-resolution microscopy. Other applications of high-temperature microscopy are, for example, crystal growth or epitaxy in specially designed ultra-high vacuum electron microscopes¹¹ or irradiation studies of materials.

The observation of chemical reactions of solids is a particular challenge of *in-situ* electron microscopy. Moving reaction fronts or the transformation of nanoparticles can be observed with lattice resolution.¹⁶ There are several examples of solid state reactions that were initiated by heating

the specimens and observed in real time. Not much detailed information exists about the course of reactions in nanoparticles, therefore *in-situ* observation of reactions promises very valuable information, for example in the technically most important field of nanoparticles in catalysis.

The study of reactions between solids and gases by applying environmental cells in the electron microscopes has been developed in the past years and resulted in a new field of electron microscopy^{9,10} (see Chapters 2 and 3). Specially designed gas flow and differential pumping systems have been attached to high-resolution electron microscopes and allow the observation of chemical reactions in a gas atmosphere at low pressure.

It has already been realized in the early days of electron microscopy that the energetic electron beam may alter the structure of the specimen. This has been applied in numerous studies of radiation damage of materials,¹⁷ for example, for applications in nuclear technology. More recently, interesting transformations of materials, in particular of nanoparticles, have been observed to occur under electron irradiation.¹⁸ The experiments are often straightforward because the same beam is used for both imaging and modifying the specimen.

A related topic is ion irradiation of specimens which has been realized by attaching the beam tube of an ion accelerator to the specimen chamber of an electron microscope.^{12,18} In such a setup, the ion beam is directed onto the specimen so that observation at high resolution is possible during ion irradiation. However, large setups outside the microscope are needed, and these experiments have only been carried out in a few specially designed microscopes. The modification of specimens by electron or ion beams has also been used for pre-defined structuring of the specimens. By using focused beams, different techniques of lithography have been developed and later applied in systems outside the microscope on a larger scale.

When an electrical current passes through a conducting specimen, the current-voltage characteristics can be measured. This has been carried out by electrically probing microscopic structures within specimens in special stages so that the relationship between structure and electrical properties could be investigated.^{19,20} Furthermore, the structure and properties of the specimen material may be modified by applying an electrical current. A specimen stage has recently been made available where a STM is integrated into the TEM stage so that the advantages of both techniques can

be combined. Electrical currents in the specimen can be measured with high precision through the STM tip.

The application of a magnetic field to ferromagnetic specimens causes a certain magnetisation and has been studied on a small scale by Lorentz electron microscopy or by phase contrast imaging with a biprism in the electron beam. The micromagnetic behavior of small magnetic structures, for example, hysteresis loops, magnetoresistive signals, or thermal effects have been observed while changing the local magnetic field.²¹

In-situ electron microscopy is normally limited to time scales above the minimum exposure time of an image. Experiments on much smaller time scales have been carried out by applying pulsed electron beams from photocathodes that are illuminated by pulsed laser beams. Triggering the image recording with the same pulses permits the study of dynamic processes on a time scale down to 10^{-13} seconds.^{13,22,23} However, these experiments need a specially designed electron microscope and an extensive external setup.

The principles of image formation that are applied for *in-situ* experimentation are basically the same as for usual static characterization of specimens. Most chapters of this book were written with focus on lattice resolution electron microscopy in the imaging mode because this technique is rather new and has shown many spectacular phenomena on the nanoscale. However, imaging in diffraction contrast remains important for many problems of *in-situ* electron microscopy as shown in Chapter 4.

4. Limitations of *in-situ* Electron Microscopy and Future Demands

In-situ electron microscopy observes dynamic processes on a small spatial scale. Of course, the time scale of the processes may span over many orders of magnitude. Thermally activated processes depend exponentially on the temperature; for example, the diffusivity of atoms may vary by six orders of magnitude when the temperature is varied by only 300 K. Hence, it is obvious that just a narrow time window is accessible to dynamic observation. The lower limit is given by the exposure time of one video frame which is approximately 0.05 seconds. An upper time limit is normally set by the regular start-up and shut-down procedures of the microscopes which is in most labs done every day. Of course, this time

could be expanded in some exceptional experiments, but a typical upper time limit of 50,000 seconds appears realistic. This gives us an accessible time scale over six orders of magnitude. However, as mentioned above, it has already been demonstrated that in a few special systems much shorter time scales can be explored by pulsed electron beams.

Another factor which limits the applicability of *in-situ* electron microscopy towards long observation times is radiation damage of the specimens. If irradiation is not the purpose of the study, it is important to minimize the electron dose on the specimen during an experiment and to work at low acceleration voltages to avoid ballistic atom displacements. Contamination of the specimen with organic molecules is another limiting factor when working close to room temperature.

The lateral resolution of *in-situ* electron microscopy depends on the specimen stage but is nowadays close to the specified resolution limit of the microscope when modern miniaturized *in-situ* stages are used. However, the space that is needed for the experiment inside the objective lens is crucial. With increasing gap width in the pole piece, the resolution of the lens decreases due to increasing spherical aberration. It is to be expected that this problem can be partly solved when aberration correctors are applied.²⁴

The applicability of electron microscopy is often limited due to the concern whether the results on thin specimens are representative for bulk materials. If the behavior of macroscopic bulk materials is of interest, special care has to be taken that artefacts due to thinning or small-particle effects are avoided. However, nanoparticles which are in the focus of current interest are small systems and do not have to be thinned for electron microscopy experiments and observation.

Of course, every experimental setup has its own limits but there are some common problems that always appear. The mechanical and thermal stability of the setup has to be optimized so as to minimize vibrations or drift during observation. Improvements due to the development of new *in-situ* stages can be expected in the near future. The enormous efforts and achievements in the mechanics of tip microscopies (STM, AFM) can also be applied in TEM stages to move the specimen with almost atomic precision by piezo drivers. Everything is facilitated when such stages are available commercially because micromechanical engineering cannot be

done in most laboratories of electron microscopy. Some developments are already on the market and have been used in novel experimentation techniques (see Chapter 6). The affordability of such stages is, as usual, another important point. Improvements of other *in-situ* setups are to be expected, for example in heating stages with less thermal drift or applicability at higher temperature²⁵ or in gas reaction cells with higher flexibility for studying different types of chemical reactions.¹⁰

The spatial resolution of modern TEMs has already been extended below the 1 Å level by applying aberration correctors.²⁴ Another advantage of aberration correction is the easier interpretation of phase contrast images when the coefficient of spherical aberration is close to zero. As an example, delocalization artefacts in the images can be eliminated. As stated above, *in-situ* electron microscopy profits from high resolution imaging at lower voltage of the electron microscope and a larger gap in the objective pole piece. Both can be achieved with correction of the spherical aberration of the objective lens. The application of aberration-corrected condenser systems will enable us to focus electron beams onto spots in the 0.1 nm range and so manipulate specimens on the atomic scale. Nevertheless, high-voltage electron microscopy will continue to have its justification (Chapters 7 and 8). Many specimen materials or special setups for *in-situ* experimentation do not allow the preparation of specimens with thickness in the 10 nm range. Due to the lower inelastic scattering of energetic electrons, high-voltage microscopy remains the only useful technique for obtaining images from thicker specimens.

For many reasons, considerable further improvements in spatial resolution of electron microscopes is not expected in the near future, and the usefulness of resolutions towards the 10 pm range in materials science is not undisputed. But even if there is not too much ‘room at the bottom’, there is plenty of time at the bottom, and *in-situ* electron microscopy should be considerably extended towards time scales below 0.1 seconds. Many processes on the atomic scale happen within femtoseconds, so there remain 14 orders of magnitude almost unexplored. Modern electron detectors are quite sensitive, but exposure times below 0.01 seconds appear unrealistic with the present beam current densities. Due to radiation damage of the specimens, brighter continuous beams are not desirable, but pulsed electron beams promise to open new windows. A few

very exciting advances by the application of pulsed electron beams have already been reported,^{13,22,23} but an *in-situ* technique that is able to cover the whole remaining time scale is far from feasible today.

5. Concept of this Book

One of the motivations for this book was the fact that *in-situ* experimentation is hardly treated in textbooks about electron microscopy. In this book recent technical developments and advancements of *in-situ* electron microscopy are presented in a collection of articles. The main focus is on transmission electron microscopy with high resolution. Although the book was edited with the intention to give a concise overview, not all aspects of *in-situ* transmission electron microscopy were treated, for example, *in-situ* microscopy of magnetic materials²¹ or the application of pulsed electron beams.¹³

It was not the purpose of this introductory *Chapter 1* to provide an overview of the extensive literature about *in-situ* electron microscopy. Only reference to a few review or milestone papers is given here. Collections of papers about recent work in *in-situ* electron microscopy can be found in some special issues of journals or conference proceedings^{26–28} and in the following chapters of this book.

Chapter 2 gives an overview of environmental electron microscopy as carried out in specially designed microscopes where the specimen is in a gas atmosphere under the electron beam. Chemical reactions are studied *in-situ* with lattice resolution and gas-solid or liquid-solid interactions become visible. Processes of highest technical importance, e.g. the CVD technique, can now be studied in a reaction cell inside an electron microscope. Environmental electron microscopy is meanwhile indispensable in the chemistry of nanomaterials.

In *Chapter 3* a technical alternative to the extensive setup of dedicated environmental electron microscopes is shown. Specially designed heating stages and gas nozzles for the exposure of specimens to gases allow the study of reactions in a standard electron microscope. Chemical reactions and transformations between different phases of nanomaterials, e.g. solid-solid, solid-liquid, or solid-gas reactions are investigated. Technically important reactions such as the solid-state formation of SiC from Si and C

are imaged at high temperature in real time and at lattice resolution. It is also shown how heating stages can be applied to study the melting behaviour of embedded nanoparticles and nucleation phenomena at solid-liquid interfaces.

In *Chapter 4* it is shown how mechanical deformation of specimens can be carried out *in-situ* during inspection in the electron microscope. Nanoindentation of the specimen under the beam allows the study of the dynamics of dislocations and grain boundaries or superplasticity in crystalline materials. Straining of specimens at high temperature gives further insight into the plastic behaviour of crystal grains and the evolution of substructures.

Chapter 5 shows the application of *in-situ* hot stage electron microscopy to the study of interphase boundaries. The collective motion of atoms at interfaces is made visible with lattice resolution. These observations are indispensable in the understanding of phase transformations at the atomic scale. Examples for order-disorder and precipitation phenomena in metallic alloys are given.

The electrical and mechanical manipulation of specimens is presented in *Chapter 6*. By using a dedicated *in-situ* specimen holder, electrical probing experiments, e.g. of carbon nanotubes filled with ferromagnets, are carried out. Current-voltage characteristics of nanoparticles are measured with such a device. Piezo drivers in the holder also allow the mechanical deformation which is shown here in the example of bending of nanoparticles. This chapter also shows the application of cooling and heating holders for the construction of nanodevices, e.g. a thermometer on the basis of a filled nanotube.

Chapter 7 is devoted on the one hand to ion irradiation of specimens which has been realized by connecting an ion beam line to the specimen chamber of a high-voltage electron microscope. With such a setup, ion implantation processes can be studied *in-situ* with high spatial resolution. This is shown here in the example of the implantation of Xe atoms into a metal matrix which enables us to monitor the growth and behaviour of Xe crystals with lattice resolution. As a second subject, this chapter treats the fabrication of nanostructured materials by electron beam-induced deposition of metals. This is realized by the decomposition of metal-organic gases on a substrate under the electron beam in the microscope. *In-situ*

observation allows the monitoring of the growth of pre-defined nanostructures with high resolution.

Chapter 8 treats the effects of electron irradiation on the specimens in the electron microscope. The displacement of atoms by knocks from the energetic electrons in the beam leads to a restructuring of the materials and, in certain systems, to new morphologies and phases of nanoparticles. The combination of heating and electron irradiation allows us to control the defect dynamics in the systems under the beam and to generate new structures by self-organization processes. Examples are shown for structures based on graphite such as carbon nanotubes.

Chapter 9 shows the detection limits of modern *in-situ* high-resolution electron microscopy. Individual point defects such as vacancies or interstitial atoms as self-interstitials or foreign atoms are observed in real time. Electron irradiation is also used here to create point defects in graphitic nanostructures.

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