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***In Situ* characterization methods**

by Aurélien MASSEBOEUF¹

in Transmission Electron Microscopy

Introduction

In situ Transmission Electron microscopy is a widely used term which enhances easily an experimental description. As an example, the use of a high energy electron beam to irradiate a sample is often referred to as an *in situ* TEM technique (M. Klimenkov, W. Matz, et al., 2000; J.-W. Liu, J. Xu, et al., 2012) while the claimed stimuli is in fact the one used to obtain an image of the sample. A reason could be that, *in situ* (from Latin “in place” or more commonly “at the right place”) is also an international word, easy to understand and exactly pronounced alike by quite every TEM user in the world (unlike TEM which has at least two known various forms in Europe : TEM and MET). *In situ TEM* is thus a continuously growing topic. From 3 occurrences per year in the 80's (~ 1% of TEM papers) it reached more than 300 occurrences (~ 7% of TEM papers) per year since 2010 (“Citation analysis realized via ISI web of Knowledge (c), last time consulted 1st of sept. 2012,” n.d.).

The aim of this chapter is to give some explanations around *in situ* TEM mostly dedicated to devices study. It will try to be as exhaustive as possible. However, as a photography of a (quickly) growing topic, it will perhaps be obsolete in the next ten years. This reminder is here to inform the reader that such a chapter was written in the middle of 2012 with results and instrumental breakthrough of that year and that it could be perhaps out of date when it will be read.

The first aim of *in situ* is to bring a new dimension to a TEM result. *In situ* techniques are then directly associated to a quantitative measure linked to a TEM image. The new dimension being brought by the experimentalist, it is supposed to be perfectly quantified. The analysis which is therefore made is a quantification along the new dimension of conventional TEM images. Quantitative imaging often claimed and required for TEM results, does not require, for the *in situ* case, complex image formation mechanism knowledge nor high level contrast simulation to bring quantification. However, a perfect knowledge of the physic at play is absolutely needed to link macroscopic observables to what happens at the microscopic scale.

The various forms of *in situ* techniques will be listed regarding their physical meaning conjugated to their experimental requirements. **Temperature** can be modified by heating or cooling the sample. For that specific case numerous commercial solutions are available. **Electro-magnetic field** applications are quite easy to produce inside a TEM. First of all because there are non local constraints, but also because TEMs are fitted with electromagnetic lenses that can be used for their generation. **Mechanical** traction or compression is an old topic which will be briefly reviewed. Focus will preferentially be made on the emergence of Micro-Electro-Mechanical Systems (MEMS) integration to produce a truly local and small displacement. **Chemistry** and **light interaction** will be lightly introduced as they mostly rely on a microscope modification. Finally we will add the **current** injection constraint which is by far an integration of quite all the previous topics. This last experimental group is on one hand a stand-alone topic as current can be used as well for resistance measurement as for magnetic domain walls motion. But on the other hand injected current is also the driving parameter for a temperature increase or a MEMS actuation.

The next section will thus be dedicated to a presentation of these various forms of *in situ* TEM

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techniques. The current and future available solutions for injecting currents into devices will be given in the following section. This section will also give a review of commercial and home-made sample holder designs. As samples sometimes need to be adapted for *in situ* experiments a last section has been dedicated to that specific part.

***In situ* in a TEM :**

So to speak, *in situ* TEM has the same approach as tomography or dynamical TEM where a third length dimension or a time scale are respectively added to a conventional bi-dimensional image. Here, the added dimension is not metrologic but analytic. Nevertheless such new dimensions can *a priori* be added using more complex mode of TEM such as spectroscopy or holography. Last but not least, such new dimensions added with *in situ* techniques are additive. For example one could imagine an experiment by simply bringing a third length dimension along with a temperature and a magnetic field scale. Therefore two more dimensions would be added to the tomogram giving rise to an extensive quantitative tomography experiment with five quantified dimensions. We will review in that section the different classes of *in situ* experiments that can be driven inside a TEM.

Temperature control and irradiation :

One of the first effect of an electron beam interacting with a sample is a local heating due to inelastic scattering (even if such heating is in the order of a few degrees (Egerton et al., 2004)). The temperature has thus been quickly a parameter that has to be driven during TEM experiments. It is thus now possible to adjust a sample temperature towards low temperatures as well as the highest ones : from around the helium boiling temperature (~ 10 K) (Sutter et al., 2011) to the melting point of silicon (1700 K) (Nishizawa et al., 2002)

The major concern in such case is the thermal conduction between the sample and the cooling or heating point. Cooling capacities rely on a fluid circulation within the sample holder (sometimes with specifications added to the microscope itself). Time is the key to let the whole system adapt to the desired temperature. The important measure is here the temperature as near as possible to the object. It is traditionally realized using an exchangeable thermocouple which is sensitive to the selected temperature range, but is far from the field of view. For high temperatures, even if commercial designs enable to use standard TEM specimens, the temperature is limited to few hundred degrees. Higher temperatures are only possible by directly putting samples onto a filament that will be heated (Nishizawa et al., 2002; Yonezawa et al., 2012) (see Figure 1.d). That configuration only ensures a good heat transfer between the heating source and the sample. Such a design also limits the kind of sample that can be used. Nevertheless in such cases electron irradiation can be used to locally tune the temperature by using a small STEM probe. At this time, electron irradiation is mostly used to induce structure change in fragile samples as carbon based objects (J.-W. Liu, J. Xu, et al., 2012) or local formation of precipitates within thin foils (M. Klimenkov, W. Matz, et al., 2000). More complex systems also add an on-chip designed thermometer to perfectly measure the temperature closed to the observed part of the sample (Harris et al., 2011).

Electro-magnetic field :

As mentioned before, electrons are focused within a TEM using electromagnetic field. It is therefore easy to use the imaging magnetic field as an applied constraint. Lorentz microscopy (Chapman, 1984) is thus often used in conjunction with magnetic field application. One of the major advantage of electro-magnetic fields is their relative homogeneity regarding TEM sample size. The magnetic (or electric) field for example is considered as constant within the sample region

of the pole piece, or at least on the observation area when designed on the whole TEM sample (Kling et al., 2010). Nevertheless lots of developments are still being made on TEM holders design or samples themselves (Brintlinger et al., 2010) to provide electromagnetic fields in a local approach (Takeguchi et al., 2006) and in specific orientations (Uhlig et al., 2003; Cumings et al., 2008; Kling et al., 2010) or in a pulsed form (Yi et al., 2004).

Mechanical :

Along with electron irradiation, mechanical stress was perhaps the first *in situ* field of TEM. The first approach is to use a holder applying the strength uniformly at the macroscopic scale (Kubin and Louchet, 1979) (see Figure 1.c). In such a case the sample has to be previously designed to enable a thin area for TEM observation as well as an overall design to afford such a mechanical constraint. These *in situ* observations are generally carried out using diffraction, conventional, or even high resolution imaging (Oh et al., 2009). Following the general evolution of TEM sample holder design, new forms of strain application appeared in the last decade. Among others are the use of moving probe (Stach et al., 2001) for local indentation or MEMS devices to carefully apply controlled forces on reduced dimension objects such as material covered tips (Ishida et al., 2010), nanowires (Pant et al., 2011) or nanotubes (Muoth et al., 2009). Such an integration is thus becoming one of the major concerns for *in situ* strain experimentalists as it is the only known method to associate *in situ* strain application with the emerging methods of TEM as electron holography or tomography (Midgley and Dunin-Borkowski, 2009). The moving probe will be detailed in the next part of this chapter (see also Figure 1.a).

Chemistry:

One of the most known dreams of chemists would be the observation of chemical reactions at the atomic scale. Environmental microscopy is a continuously growing topic which brings some important instrumental development to the TEM community. The two main classes of ETEM are the observation of specimens under a gaseous (Yokosawa et al., 2012) or liquid (Jonge and Ross, 2011) atmosphere. The main tools used for this TEM variations are called TEM windows (Creemer et al., 2008). They are based on nitride or oxide silicon membranes. A detailed presentation of such objects will be given in the last part of that chapter. The principle in ETEM is to confine the desired

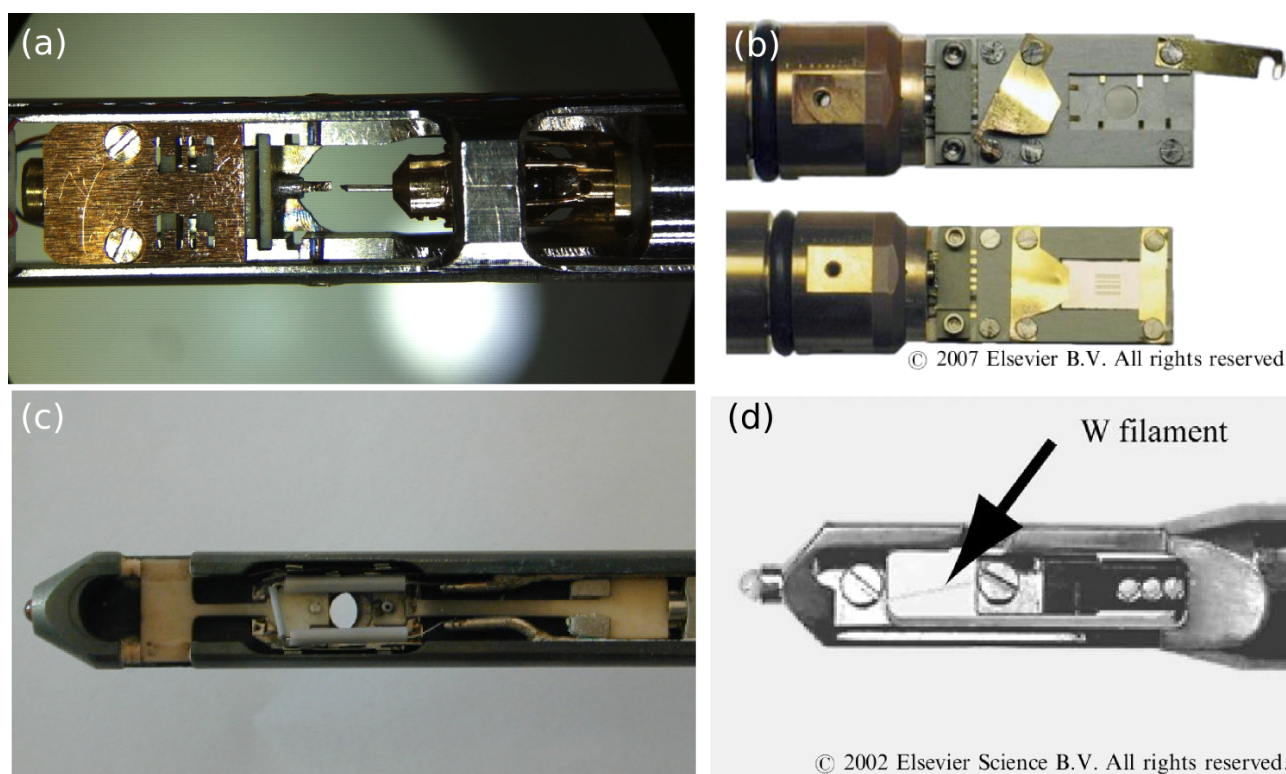


Figure 1: Sample holders for *in situ* a) Holder with a scanning probe for indentation, biasing or field emission measurement. b) Custom made sample holder for multi-contacted sample (8 contact pads – courtesy of (Kim, Kim, et al., 2008)). c) Sample holder for strain (traction) experiments. d) Sample holder for high temperature observations (courtesy of (Nishizawa et al., 2002)).

atmosphere within a restricted volume of the TEM chamber included between two of these membranes. Nevertheless such a designed volume is for many reactions not sufficient or too much fragile. Dedicated microscopes have thus been developed (Gai, 2002) to offer a full reaction chamber at the objective stage. Here the chamber is isolated from the rest of the column by using differential apertures.

Light :

Light interaction is becoming a timely topic since new forms of microscopy have been discovered such as orbital moments electron beams (J. Verbeeck et al., 2010) or plasmons mapping (Nelayah et al., 2007). Moreover TEMs designed for ultra-fast (Kim, LaGrange, et al., 2008) or dynamical (Zewail, 2010) microscopy showed the huge interest in bringing light towards a sample. If the column modification is sometimes used for dedicated experiments (as for dynamical processes) the light insertion (or detection) is rather simple using optical fibers (Tanabe et al., 2002). A challenge remains in conserving pre-formed light shapes within the TEM column. Alike Scanning Tunneling now commercially available with numerous manufacturers (see also next part), it is possible to add a Scanning optical probe (SNOM) within a sample holder (Xiang et al., 2012). Sample holders bringing at the same time a probe for electrical biasing or mechanical indentation have also been fabricated (Shindo et al., 2009).

Multiple and movable currents :

As a summary of the *in situ* variations presented above this last point present the current injection in a general way. Currents are needed in numerous of the techniques of *in situ* TEM. Magnetic coils, MEMS power supply or thermocouple activation are both tuned using currents. Variations inside the panel of currents injection are the among of contacts brought near the sample and their versatility (i.e. fixed or movables). Bringing more and more contacts towards the area of interest of the sample theoretically enables a mixing of various form of *in situ* TEM. In a same way, the possibility of selecting *in situ* where a current can be applied is an unfordable gain regarding the complexity of devices now observed in a TEM. The next sections of this chapter are thus dedicated to an exploration of the various form of contacting a sample within a TEM. First the two main families of sample holder will be presented, then the creation of the TEM sample to accept such contact will be explored.

Biasing in a conventional TEM :

As stated above, two approaches may be distinguished in injecting a current within a TEM sample. The historical one is the opportunity of bringing as much contacts as possible from the outside to the inside of the TEM via the sample holder. Current vacuum feedthrough is well known and widely available, but the connection between the TEM sample and the wire within a sample holder is not unique. We will try to exhaustively describe the available options. A new method was proposed in the late 20th century with the apparition of STM (scanning tunneling microscope) probes within sample holders. Last but not least, combinations of these two methods are now emerging. We will give a brief overview of the state of the art of such sample holders.

Multiple contacts :

At first an *in situ* TEM experiment aims at bringing wires on the sample under observation via the sample holder. Nowadays, two main characteristics differentiate multi-contact holders : the number and the versatility of the contacts. The more contact there are, the more versatile the sample holder is but less versatile each contact is. The main goal of this part is to give an overview and some examples of various contacting forms that exist in the literature. Nevertheless an important point in such sample holders is the design of the sample itself and a section is devoted to this subject at the end of the chapter.

The key point of multi-contact holders (see Figure 1.b) is to offer various constraints at the same time on a sample. An example is the use of currents to heat the sample, to measure the temperature and the resistivity of the sample as well (Verheijen et al., 2004; Harris et al., 2011). Such a simple analysis requires at least six independent contacts. When using MEMS inside the TEM to apply local strain, multiple contacts are also needed for the various parts of the MEMS to be actuated and controlled (Ishida et al., 2010). More generally speaking the amount of contacts will determine the number of actuation/measurement couples within the experiment (actuation being multidimensional – indentation is 1D, friction is 2D, etc.. - it can rely on multiple contacts as well).

Two methods can be employed to connect the sample at the sample holder : clamping or bonding. In the case of clamping, the clamp ensures the mechanical stability and the electrical contact as well (Zhang et al., 2005; Zhu and Espinosa, 2005). Such a design ensures a perfect reproducibility between sample insertions but it is less versatile because all the samples have to be adapted to the clamp. Moreover, the clamping has to be carefully designed to afford a high rate of closing/opening cycles without damaging the sample. The bonding method is more versatile but has to be handled more carefully. Bonding can be made by using wire bonding methods (gold, aluminum and more recently copper) (Breach and Wulff, 2010) or simple silver paste. The former is more reliable and conductive but also more traumatizing for the samples which may broke under the pressure used for bonding and the latter can be sometimes a poor conductor which can reach up to a few ohms per millimeter.

Finally the sample has to be integrally designed to accept the contacts. The use of such a sample holder can stand two different types of samples. The most used one is the free standing object. All the direct environment of the object is first designed and the object is then put at the right place for the constraint to be applied. The sample can be either a nanostructure (Muoth et al., 2009) or a material deposit on a former structure (Jalabert et al., 2012). In the first case the sample has to be clamped onto the structure by appropriate welding (Pant et al., 2011). A more complex design is the creation of all the environment around the existing object. In that case the object is first deposited onto a “TEM window” which will be lithographed later on (Tang et al., 2010; Lu et al., 2011)(see last section). The high advantage of using such a design is not necessarily to multiply the experimental constraints on an object but to multiply the number of objects under constraint on the same TEM sample (ten contacts for example enable the connexion of five different objects for simple resistivity measurements). First working transistors based on carbon nanotubes have been successfully observed using such an approach (Kim, Kim, et al., 2008) (see Figure 2.a).

Some other cases were also proposed, such as coupling a clamping method and a specific sample design but such methods are somehow limited to a few contacts (A. C. Twitchett, R. E. Dunin-Borkowski, et al., 2002). These experiments were the premises of a new form of holders bringing new forms of contacts.

Movable contacts :

A large range of applications appeared with the development of scanning probe microscopies and their piezo-driven mechanics. One of them was the integration of a moving probe inside a TEM holder (Iwatsuki et al., 1991). Nevertheless the low distances applicable with a piezo system only made it difficult to use. And it is only at the beginning of the 21st century that the well known inertial sliding or “slip-stick” mechanism (Pohl, 1987) was practically promoted in a STM

probe TEM holder (Svensson et al., 2003) (.).

The principle was not much to obtain a coupled STM/TEM image but to use the fine positioning system of nanometric probe in front of flat surfaces. In such a configuration the *in situ* experiment was extended to the positioning of the contact itself. The sample is traditionally grounded as the tip brings the bias. Biasing can thus be used to produce a field emission from a tip as small as a nanotube (Cumings et al., 2002). But the main interest in using such a tip is to inject a current in a specific local area of a device under study. P-n junctions can thus be precisely studied in terms of structure and electrical properties (Han et al., 2008; Park et al., 2010) as well as localized transport measurements (Chiaramonti et al., 2008), charged memories study (Choi et al., 2011; Q. Liu, J. Sun, et al., 2012) or various carbon-based material properties (Wang et al., 2006; Huang et al., 2010; Golberg et al., 2012). The ideal observation was the study of a working device using all the sensitivity of TEM techniques. The first experiments on MOSFET devices give a perfect knowledge of electrostatic potential within the conduction channel (Ikarashi et al., 2012) (see Figure 2.b).

The sample design is rather simple as it only needs a free half space to let the probe come to the surface of interest. FIB processes (described in another chapter of this book) are thus perfectly suited to such analysis. The only precaution needed being that the sample is effectively grounded.

The TEM holders are still developed to offer more and more interacting forms within the sample regarding *in situ* probing capacities. We can find two moving probes on the same sample holder (Murakami et al., 2006; Kawamoto et al., 2011) or a combination of a probe with a laser injection system (Shindo et al., 2009). Manufacturers now propose the combination of a moving probe and multi-contacts within the same sample holders. The 4 point measurement method traditionally used in the micro-electronic industry within a TEM sample holder might be accessible in a next future.

Comparison :

The TEM experimentalist has always to keep in mind the first *in situ* mechanism presented is that chapter : electron irradiation. It is worth noting that the few experiments on working devices have pointed out the huge effect of the imaging electron beam on the functions of the device (Kim et al., 2005; Ikarashi et al., 2012) (see also Figure 2).

The two presented methods are constantly developed and no one can say today if one will be the standard in a few years. One major consideration could be that the multi-contact holder is less complex to produce as it does not need any complex materials such as piezoelectric or probe holders. Moreover their design is more dramatically important with respect to the available samples. Lots of teams have thus fabricated their own sample holders, often starting from an originally manufactured simple holder. Some teams are still developing some high level moving probes systems (Siria et al., 2012). For specific analysis or for money saving it is sometimes better to produce a home-made sample holder as it will offer, without any doubt, the versatility expected by the TEM user. Nevertheless, lots of TEM experimentalists might be put off by TEM complexity, from grounding/charging precautions towards ultra-high vacuum problems. The fact has to be added that it is by far faster and easier to get commercially holders for conventional *in situ* operations as a manufactured system comes with all the electronic and software assistance.

The last point that will be discussed here is therefore a manufacturing limitation. Far from wishing to promote any of the TEM manufacturers, it is important to note that only Jeol Ltd. is using side entry sample holders of more that 10 mm in diameter. Such a size gives rise to a lot of space to design complex structures. In fact, quite all the high level developments in sample holders presented in that part have been made with that manufacturer equipment.

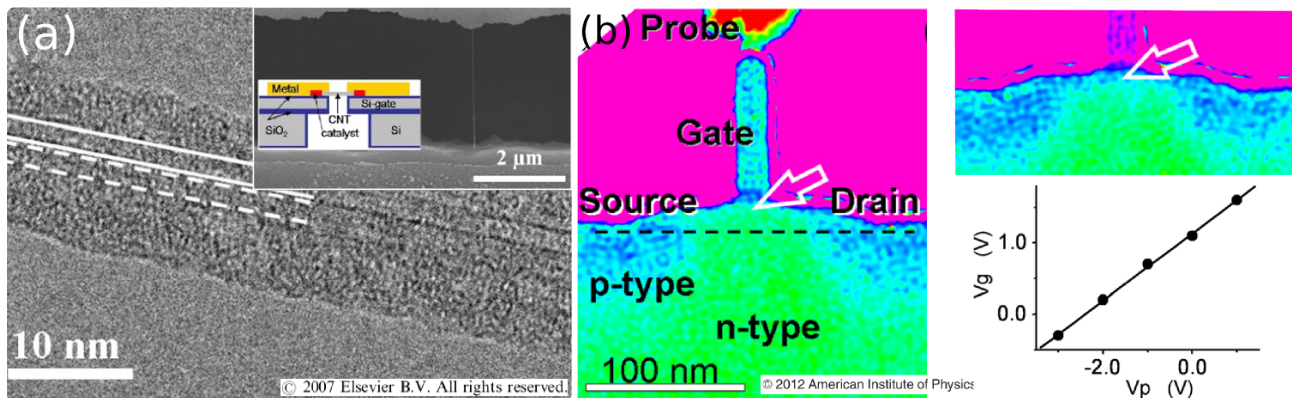


Figure 2: Working devices analyzed in-TEM. a) Field effect transistors (FET) based on CNT bundles mounted in TEM with a multi-contact holder. Observation of CNT during FET function finally showed a huge influence of the electron beam irradiation. Inset is showing a SEM image of the suspended CNT acting as an electron channel (courtesy of (Kim, Kim, et al., 2008)) b) MOSFET electric potential analysis using a moving probe sample holder. Electron holography is used to reveal the electric potential within the MOSFET channel at 0 (left image) and 2 V (right zoomed image). As electron irradiation is perturbing the work of the device (a positive gate tension (V_g) is found for a 0 V (V_p) probing), the authors have taken into account its influence to get quantitative measurement of the gate work-function. (courtesy of (Ikarashi et al., 2012))

Sample design :

Most samples studied in *in situ* experiment are nanostructures. Due to their low dimensionality and uniformity nanowires (nanotubes) and nanoparticles are well suited to pass a current or to be mounted on a probe. Nevertheless new TEM samples preparation techniques as well as micro-electronic traditional processes can now be merged to provide complex structures suitable for TEM *in situ* observation. We will see in the present section some of the most common tools that can be used to easily design a TEM sample that can be fitted within sample holders described above. Focused ion beam and nanolithography are now common techniques used to provide thin connected samples. One can start from a bulk specimen in which thin foils or devices are located or one can design a dedicated environment around an already TEM compliant sample.

It is worth noting here that common TEM samples can be used for *in situ* techniques such as temperature, strain or magnetic studies. Considering the moving probes presented above, they can be easily produced by the usual processes used for STM tips (Ibe et al., 1990).

Focused Ion Beam :

That section will not deal with traditional sample preparation techniques that can be used to prepare conventional TEM lamella that are now commonly used in the TEM field (and which are extensively examined in another chapter of that book). This part is more dedicated to an overview of unusual but simple methods that can lead to well adapted TEM samples for *in situ* TEM. The old fashioned H-bar method (Castany and Legros, 2011) was progressively abandoned in favor of the lift-out method that is by far more convenient for most applications. However its extremely robustness (the TEM sample belongs to the micrometric scale) is a perfect asset regarding the moving probe system presented above. It offers a flat surface, with a small thin area that can be easily reached by a moving probe within the TEM. Moreover, several H-bar samples can be etched within the same sample that can lead to a high density of TEM windows for destructive indentation experiments (Chiaromonti et al., 2008). Last but not least, such a geometry can also be used as a

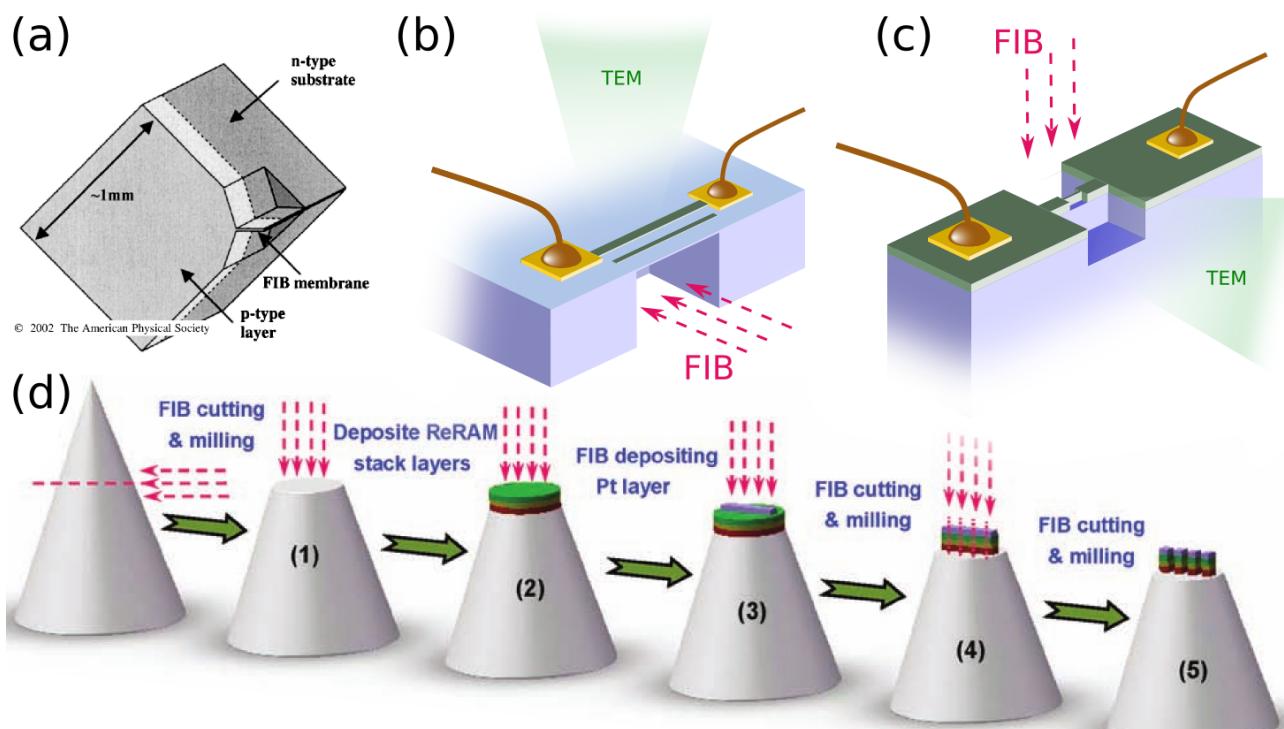


Figure 3: FIB use for *in situ* samples. a) TEM lamella made within bulk P-N junction sample using FIB (courtesy of (A. C. Twitchett, R. E. Dunin-Borkowski, et al., 2002)). b) Plane view sample produced by FIB. c) Cross-sectional sample used as a nanowire for passing current. d) FIB preparation of substrate and TEM sample preparation after material deposition (courtesy of (Q. Liu, J. Sun, et al., 2012)).

nanowire to pass current in the case of a thin foil (Figure 3.c). A less known technique is the plane view preparation using FIB (which could be referred to as the U-shape with respect to the H-bar denomination – see Figure 3.b). Such a technique can be easily used in combination with few lithographic processes (that can also be done using the FIB) to make a well suited geometry for strain experiments and/or current injections in nanostructures (Brintlinger et al., 2010).

One can also start from a bulk material (small enough to enter a TEM) and use its size to contact it easily within a TEM holder. The FIB is used there to produce a small visible area within that bulk sample in which the probed process occurs (A. C. Twitchett, R. E. Dunin-Borkowski, et al., 2002) (see also Fig 3.a). FIB can also be used to prepare a sharp surface that is used as substrate for material deposition afterwards, and then finally used to get one (or more) sample at a specific location that will be used for *in situ* measurements (Q. Liu, J. Sun, et al., 2012)(see also Fig 3.d).

TEM windows :

In addition to traditional carbon foils deposited on a copper grid, nano-objects are often dispersed on silicon nitride (or oxide) membranes (Grant et al., 2004). This solution, supported by a large commercial offer, is by far more convenient for a lot of *in situ* TEM techniques. First of all it proposes a flat surface supported by a quite thick and easy to handle substrate (few hundreds of μm) that can be used for material deposition using traditional growing processes (with a few precautions such as protecting the viewing hole from differential pressures). Moreover the insulating behavior of nitride ensures a perfect isolation of contacting pads which it supports.

Even with the large panel of commercial offers it is sometimes better to produce one's own membranes. The traditional TEM window presents small lateral dimensions that do not fit the lithography requirements (sample manipulations and resin deposit being the most important points). Moreover the instrumental need for such fabrications are really simple and can be found in most of cleanroom facilities. The main idea is to use a CVD grown nitride (resp. thermal oxide) grown on each surface of a thin (less than $300\ \mu\text{m}$ to fit common sample holders) wafer that will be lithographed on one side to create the opening holes. A simple wet (resp. dry) etching is then necessary to remove the silicon towards the opposite nitride (resp. oxide) side. Membranes

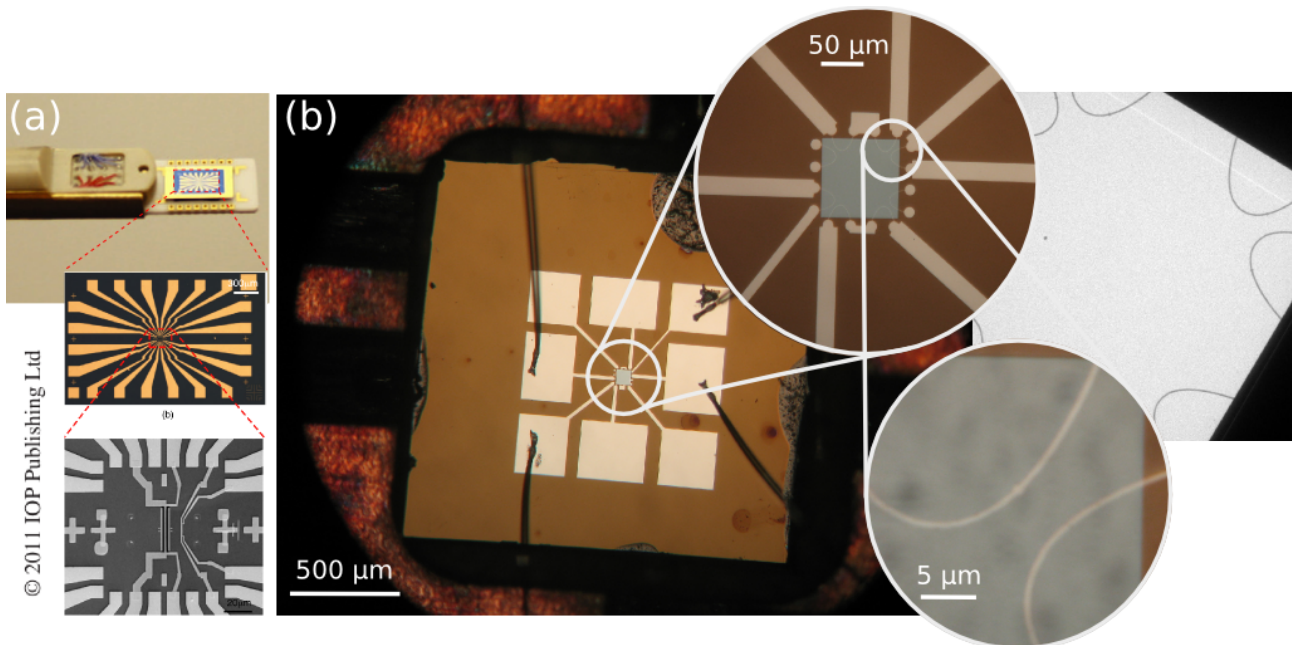


Figure 4: Silicon nitride windows. a) TEM thermal measurement platform used for nanowires under varying temperature analysis. Black areas in the middle of the bottom image are holes within the nitride membrane to let the electron passing through (courtesy of (Harris et al., 2011)). b) Permalloy stripes (400 nm width) grown on silicon membrane and connected to a sample holder with micro-bonding (4 pads connected). A TEM view of the sample (top right) is presented along with optical snapshots.

thickness can be decreased down to few tenths of nanometers. UV lithography can then be used to design contact pads and e-beam lithography to connect the sample to the pads. Complex designs (as complex as MEMS) can then be drawn with such technology to reach the concept of “in-TEM micro-laboratory” (Verheijen et al., 2004; Harris et al., 2011; Jalabert et al., 2012; Kallesøe et al., 2012). In such a concept, the sample is no longer the object under study alone but both the object and its direct environment. Figure 4 is presenting two designs of objects connected to a sample holder through the use of a silicon nitride membrane.

Conclusions :

In situ TEM is a really rich topic and can still be viewed as a non-traditional microscopy as it involves a lot of different processes. Mastering all the required processes may be long and tedious that is why it might sometimes be useful to proceed via the commercially available steps when they exist (sample holders, sample supports, contacting). The TEM experimentalist has to keep in mind that a home-made overall process will be more dedicated to the research than a commercial one. Still, *in situ* microscopy is now quickly growing mainly thanks to a lot of instrumental and processes developments. As stated above, such a short chapter might not be up to date for a long time.

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