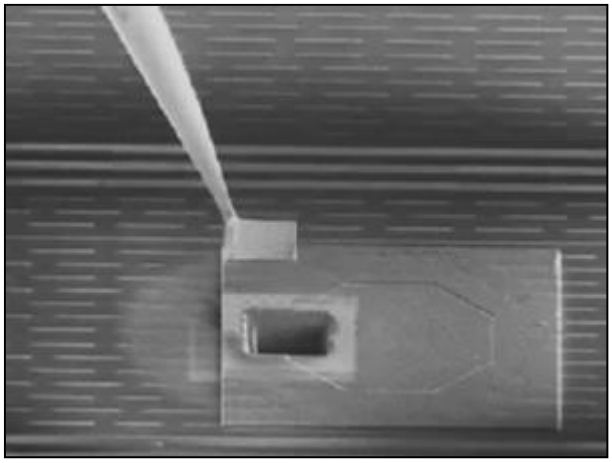
NanoScope FIB technology article

In-situ or Ex-situ? Which TEM section lift-out method is best?

In this article I will be discussing the relative merits of both methods of FIBxTEM section extraction and the factors to consider when choosing which is better for your sample types.



You may think this is a foregone conclusion, with *in-situ* foil extraction being clearly more advanced and more effective as an extraction method than *ex-situ*, but hold on to that thought, as there may be some less obvious issues that are worth your consideration.

Firstly let's define terms –

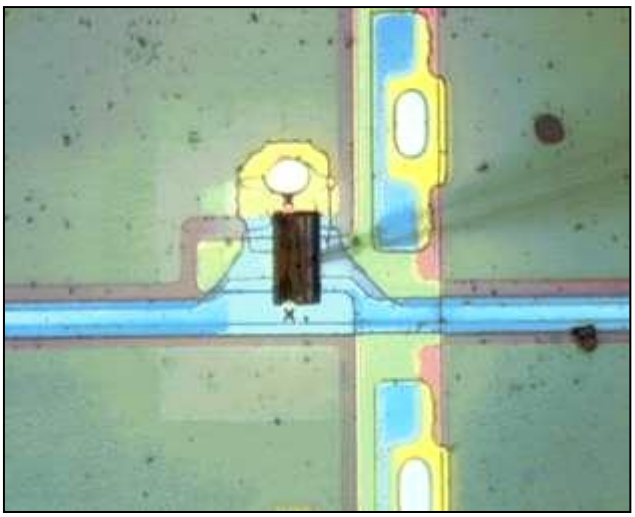
FIBxTEM section or foil = The site specific FIB milled sample biopsy that is FIB polished to become electron transparent and extracted to a TEM grid before being transferred to a TEM microscope for more detailed studies.

'*in-situ*' foil extraction – where a 'thick' biopsied section is transferred to a TEM grid inside the FIB-SEM instrument using a nano-manipulator and attached there before being FIB polished to the required 'thinness' and then transferred to a TEM.

'*ex-situ*' foil extraction – where a site specific feature is thinned to electron transparency BEFORE extraction, then cut free and unloaded from the FIB-SEM and where the section transfer to TEM grid occurs under an optical microscope on the lab bench, before being loaded into the TEM.

In-situ foil extraction (or lift-out) is the recommended approach promoted by both microscope vendors and extraction system suppliers alike. They claim that it is faster, safer, easier, requires less operator training, and produces a sample that is both of higher quality and also able to be 'reworked', if not perfect in all respects after the first iteration.

Ex-situ foil extraction however continues to have it's advocates. They claim that it is significantly cheaper, significantly faster, offers very high yields and is more versatile in the range of materials it may be applied to. It also removes any additional risks to the main instrument, reduces the time needed to prepare samples, and removes additional support costs for the extra hardware.



Advocates of *in-situ* claim that *ex-situ* extraction is difficult and suffers from low yield, whereas *ex-situ* advocates point to the materials limitations of *in-situ* and scoff at the excessive cost. We have to look at the merits but also the disadvantages of each technique if we are to form a balanced picture.

So lets compare…

1st Criteria - Speed.

Here the *ex-situ* approach has many advantages

During the FIB thinning process there is no break in the middle of the milling steps for transferring the sample to a grid before continuing to polish it to the desired thickness. You just pick your spot and thin to the target thickness – then release and unload. Transferring the section to a TEM grid is also very fast – the lift and transfer can take as little as 20 seconds – and the whole process including all alignments and handling, typically less than 5 minutes. **(see NanoScope Youtube video here)**

What is even more important is that these 5 minutes take place **outside the FIB tool**, so the main instrument can already be preparing the next sample and saving (typically) 20% of the total processing time.

The *in-situ* approach **(see Youtube video here)** requires the TEM grids to also be loaded/unloaded into the FIB(FIB-SEM) in addition to the bulk sample. It also requires additional attachment/detachment/re-attachment and sample transfer steps using metal deposition and cutting steps, and there are two distinct sets of milling processes required on two different sample holders, both of which require alignment and optimisation. The sample release cuts also take place while the sample is thicker requiring longer.

On the topic of speed at least, there seems to be a clear winner.

2nd Criteria – Yield

When comparing the success rates of the two techniques we trigger a strong 'marmite' style user response. Advocates of both techniques claim > 95% success rates for experienced users. Most *in-situ* users believe that *ex-situ* extraction is more difficult and lower yielding, but this perception is not supported amongst advocates of *ex-situ* extraction. There are however many users who have tried *ex-situ* extraction and after a few failed attempts have reached the conclusion that it is not a reliable method. This perception is not routinely challenged and is also supported by the promotional messages of the major equipment vendors (wishing to sell more tools and more options.. of course).

Furthermore, as more people use *in-situ* extraction there is a wealth of data showing the success rates are more available, and due to the higher investment required, a higher likelihood that a specific user would persevere until the process is yielding well.

Technically there are also differences in the training process. When sections are damaged by the *in-situ* extraction process they do not tend to be 'lost' as they can be with the *ex-situ* approach, and so with additional time and effort 'some' type of usable sample may be able to be salvaged more of the time.

NanoScope Services Ltd
Office 30, Station Road Workshops, Station Road
Kingswood, Bristol BS15 4PJ, UK
Tel : +44 (0)1179576225
Contact@NanoScopeServices.com
www.NanoScopeServices.com

In my experience (and I routinely use both techniques, and in fact helped develop and supply them over a period of some years) the yields are about the same – at NanoScope we routinely achieve >95% yield using both methods, and the few failure modes we have seen are normally not related to the extraction process itself, but to some unusual aspect of the materials system being processed or the way the samples are presented.

With *in-situ* it's all about having your hardware aligned and stage positions saved, and with *ex-situ* it's all about knowing the loss mechanisms and how to avoid them, or recover from them if they occur. At NanoScope we call these the '**10 golden tricks and tips of high yield liftout'** and for those readers looking to improve their extraction skillset, we will soon be posting them on Youtube. If you would like a preview we'd be happy to chat with you, just click here.

3rd Criteria – Quality

This factor is more difficult to measure. One must first have a good idea of what type of TEM analysis one would like to perform, and measure your success against these criteria.

While all samples must be electron transparent, they can be customised to support specific types of study. For example, if you want good EDS data – a slightly thicker sample may be appropriate. If you wish to do high resolution studies in TEM mode, or EELS studies, then a very flat, very thin, low damage foil with parallel sides is better. For studying fine horizontal layers in STEM mode on a budget and to a time limit – a wedge shaped section may be preferred.

In the perfect world a sample where you can start thick, and then re-polish the section to a new format to give further information under different imaging conditions is the ideal approach– and for this an *in-situ* mounting technique offers real versatility. *Ex-situ* foils can be re-thinned with lowkV – but generally speaking it is more difficult to get a uniform result from one side only (although subsections may be re-thinned from both). For the best HR studies however (and the headline imaging results used to sell the big ticket FIB-SEM and TEM microscopes) it is widely accepted that the ability to re-thin *in-situ* extracted foils at very low kV's is now the method of choice.

So *ex-situ* extraction comes in second for ultimate sample quality management (mostly).

4th Criteria – Cost

The most commonly recommended *in-situ* nano-manipulator available for this application comes in its basic form with a hefty price tag of >120K$, and if you add some helpful control systems, can be even more.

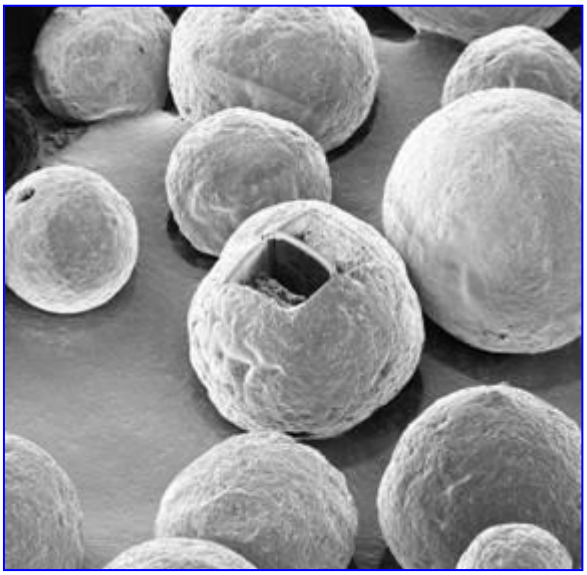
Rivals to this solution can be less expensive, but can also be more difficult to use. Any add-on in-chamber hardware that may not be well integrated into the main system architecture will carry some risks to the tool itself if not correctly operated.

Because of this, many manipulators are demounted and stored under normal multi-user conditions, and reserved for the use of the instrument supervisor. This adds time and complexity to the routine extraction of sections.

NanoScope Services Ltd
Office 30, Station Road Workshops, Station Road
Kingswood, Bristol BS15 4PJ, UK
Tel : +44 (0)1179576225
Contact@NanoScopeServices.com
www.NanoScopeServices.com

The costs of *ex-situ* solutions are also significant. The hardware consists of an optical microscope with long working distance lenses and a nano-manipulator suitable for the precision required. Buying a suitable optical microscope for use for this application alone could potentially be similar in cost to the more reasonably priced *in-situ* solutions. (a few 10's of 000's €). The advantage here of course is that all the major optical microscope vendors now offer reasonable long working distance (LWD) lenses to upgrade any suitable instrument you may already own, and these are far more reasonable to acquire. Extraction is quick so the system is used for only a few minutes a day and may be routinely used for other applications, thus spreading the costs further. There are some additional bits needed (for making probes) but these are all very reasonably priced.

One must also consider the value 'through-put' of the hardware concerned as a cost element. A FIB or FIB-SEM solution costs several 100's of 1000's of €'s and so every minute the instrument is being used has a value. *In-situ* extraction takes up instrument time and carries risk to the instrument and also the manipulator needle itself if incorrectly handled. If the time needed for foil extraction adds 20% to the overall section preparation time (not uncommon) and additional costs in consumables and maintenance and operational support – then this has to also be considered. Crudely put, if you own 4 FIB systems for making sections – you will need a fifth just to do the lift-out and no microscopy at all.



If we revisit the speed advantages of ex-situ extraction from Criteria 1 – we see that ex-situ comes out a clear winner for Criteria 4.

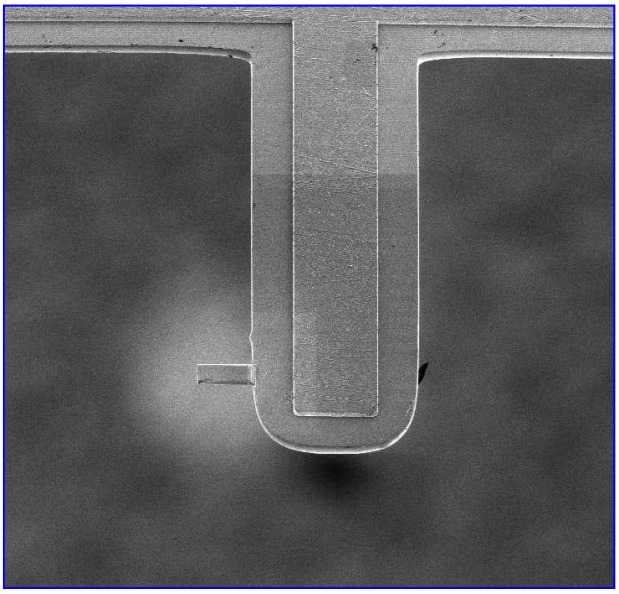
5th Criteria – Versatility

Which materials limitations do I need to be aware of? If you work exclusively with Semiconductors then either approach will support you well. If however you work with a wide variety of materials systems then there are other issues to consider.

The attach/detach, post-thinning or 'sub region' thinning processes used for *in-situ* extraction - all rely on materials that have sufficient structural stability to support themselves only from one side. However there are many materials where this is not suitable. It does not apply to some delicate, brittle, or soft materials such as polymers, inter-metallics, voided materials, cracked features, amorphous materials, lowK materials (Semi), compressed or sintered powders, composites with low internal adhesion or delaminated structures. These materials require more delicate handling. The *ex-situ* extraction process relies on the electro-static attraction of the material to the nano-manipulator and then to the grid itself. After transfer the foil itself is then fully supported on a film in the TEM grid. At NanoScope we have successfully prepared TEM sections from all of these difficult materials systems using *ex-situ* extraction for transfer.

The different FIB milling processes also limit the scope of the materials systems that can be prepared.

NanoScope Services Ltd
Office 30, Station Road Workshops, Station Road
Kingswood, Bristol BS15 4PJ, UK
Tel : +44 (0)1179576225
Contact@NanoScopeServices.com
www.NanoScopeServices.com



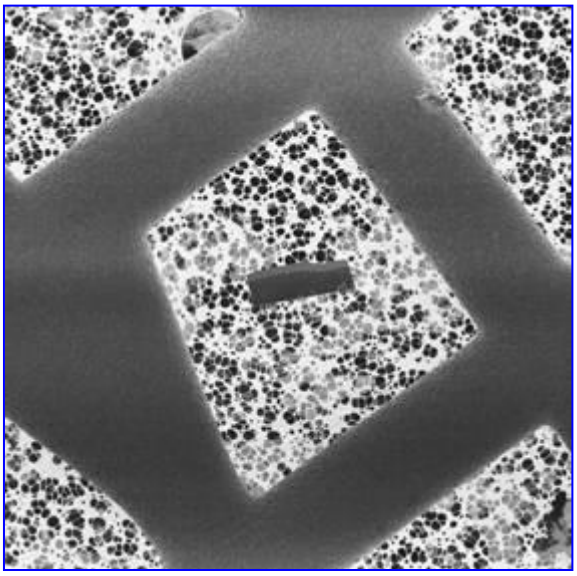
During *ex-situ* section polishing the foil stays connected to the bulk sample at 2 or more positions until the last moment. This can help ensure that the foil itself stays flat and that any deformations that occur in the foil during the most crucial stages of thinning, can still be controlled.
If a foil is held only at one edge – any deformation will cause significant movement in the position or aspect of the foil, and reduce the ability of the TEM user to align to and see, the target area. If a foil is made of a homogeneous material then these effects may not be severe – but when thinning sections from complex materials systems or with complex structures within them, there are some further issues to consider.
As a section gets thinner, these effects are more likely to appear as different elements within the section may react in different ways. Section distortions may include (but are not be limited to) the following.
Bowing (caused by uneven polishing or internal materials stress while clamped)
Curling (either symmetrically or asymmetrically)
Bending (can also happen during polishing, SEM inspection or FIB low kV treatment)
Rolling - even like a Roller blind – seen with some polymers
Flapping (when a potion of the section to one side of a thinned sub-region folds over)
Or my personal favourite '**flapping about a specific horizontal layer'** like a lowK dielectric (or 'cat flapping' as it has been called).

NanoScope have developed a method for preventing all of the above failure modes for an *ex-situ* preparation process – improving the tendency of the foil to stays optimally flat for fine polishing and lowkV cleaning, while at the same time allowing the foil to release any intrinsic materials or structural stresses during the thinning process. I hope to be able to discuss this topic in greater depth in a future blog. **For more information about this please contact NanoScope**

6th Criteria - Ease of Use
Both systems perform a non-trivial task, both require skill and understanding of process and material, but we must also look from the usability perspective and this is based on the principles of how each process is delivered.
Most *in-situ* nano-manipulator solutions are mounted at one point (normally the stage or the EDS port) and the tip is driven to the sample using rotational actuators and a length adjustment. This means that the tip of the needle moves in arcs (polar co-ordinates) or linear L, but always not-orthogonal to the instrument stage. As an operator this means if you extend a 'roof mounted, downward tilted' needle in L, it also moves in X and Z which can make life difficult.
The difficulties of operating a non-orthogonal system means operators start off by breaking needles and losing samples. A damaged needle costs money and many hours to replace and re-sharpen using the ion beam. Instead you find (if one visits a few users) that experienced operator with a 'high yield process' have found a quicker and more repeatable method. They tend to insert the carefully maintained (and carefully guarded) needle to a known central position at the centre of the field of view, and **move the sample to the needle.** The main instrument stage moves with clearly separated

NanoScope Services Ltd
Office 30, Station Road Workshops, Station Road
Kingswood, Bristol BS15 4PJ, UK
Tel : +44 (0)1179576225
Contact@NanoScopeServices.com
www.NanoScopeServices.com

axes in X, Y and Z making this far more controllable. The SEM in this case and indeed the needle movement system (other than insert-retract) are seldom used in order to save time and reduce errors.**
Help for the novice is also available in a FIB-SEM instrument by being able to view the approach of the needle position from the perspectives of 2 different imaging systems to confirm how the section is being moved.
For *ex-situ* lift-out the feedback is divided. Those who routinely use it (like me) are of the opinion that is it easy, reliable and fast. This is why service labs tend to like it – after all, time is money.



But even here you will always hear the tag line 'but you must take care to avoid all the loss mechanisms and have a good understanding of recovery strategies'. Others who may be getting mixed results have a very different opinion. The learning curve is certainly steep and the yield 'very low' until all of these loss mechanisms are understood and controlled, which makes adoption of *ex-situ* lift-out a binary process. There is also not much readily available information on what to look for and how to handle it. For the sake of this discussion I will choose to advocate the former opinion, that if you want a fast, effective, high volume foil production process, then *ex-situ* offers some additional usability benefits.

**There are of course some clever control systems which can help with the issues created by polar movement, and these try to intelligently compensate for the non-orthogonal movement issues and give the user experience of a '1 axis at a time' Cartesian system. These systems indeed offer some relief from this issue – but again, come with an additional price tag.

7th Criteria - assorted

There are a few other technical factors worthy of mention, which may assist you in finally choosing one or the other technique.
Firstly in favour of *in-situ* extraction -

1) Some FIB-SEM solutions offer STEM detectors with a so called a 'FlipStage' which can help assess and accelerate the optimised polishing of very thin foils for the best TEM HR studies in a shorter time frame. If this represents the main reason for your instrument purchase then *ex-situ* is not the correct choice.
2) The *in-situ* technique also ensures that a materials system can be prepared and analysed within a single vacuum system – there are a few materials which are immediately compromised by exposure to atmosphere such as AlGaAs, where the Aluminium rich regions may corrode in short order when exposed to atmospheric water vapour.

And in favour of *ex-situ* extraction -

1) For the analysis of tiny features/defects. FIB is often used in this case when looking for very small features (perhaps <100nm's) as it enables a site specific and iterative analysis approach. But for maximum advantage ex-situ offers an additional benefit with these steps.
    - Step1 - perform conventional FIB sections with FIB or SEM imaging until you can see the beginning of the defect or feature.

NanoScope Services Ltd
Office 30, Station Road Workshops, Station Road
Kingswood, Bristol BS15 4PJ, UK
Tel : +44 (0)1179576225
Contact@NanoScopeServices.com
www.NanoScopeServices.com

- Step2 - learn what you can from it – using whatever imaging/analysis techniques you have available
- Step3 - polish it into a TEM section from behind – ensuring that 'most' of the feature is within the foil thickness
- Step4 - Transfer to TEM for further analysis

For *in-situ* extraction this presents a problem – because ion beam metal deposition is required to mount the section both to the needle and to the grid – thus coating the section in depo metal and meaning the feature must be re-polished before it can be imaged in the TEM. But if the defect is tiny – a few 10's of nm's only – then re-polishing is not an option as it will remove the majority of the remaining defect/feature from the face. Here the *ex-situ* approach ensures you can just stop polishing the side of the section where you have seen the defect or feature, and just polish it from the back until the section is ready for extraction.
For this 'tiny defect case' the ex-situ extraction process offers a clear advantage.
For an example of this approach please read the case study on the reverse side of our Spring Newsletter.

Conclusion

So with careful training and an unbiased approach, both these extraction techniques can be used to great effect. If you are not time or budget limited and wish to conduct an optimised analysis of a stable and solid sample then the *in-situ* extraction process offers some important advantages.
If however you are more interested in the fast routine analysis of the widest possible range of materials systems, and without breaking the bank, then *ex-situ* extraction offers advantages that cannot be overlooked.

In an ideal world of course – I would buy both, and **maybe a little training from NanoScope.**

We'd like to hear about your experiences with both types of foil extraction – write to us here.

NanoScope Services Ltd
Office 30, Station Road Workshops, Station Road
Kingswood, Bristol BS15 4PJ, UK
Tel : +44 (0)1179576225
Contact@NanoScopeServices.com
www.NanoScopeServices.com