

# FIB process takes materials analysis into new realms

The novel integration of ion and electron beams into one tool with precision sample handling abilities is providing a one-stop shop for many characterization needs, writes **Lloyd Peto**.

The examination and analysis of multi-layered films and complex structures is often challenging. Site-specific analysis, as required for single-site semiconductor device failures, is especially demanding. Problems are exacerbated when working with materials that are sensitive to the preparation techniques being used and/or environmental factors during preparation. Laser devices, for example, are almost impossible to remove from can-type packages without damage, so the detailed analysis of laser facets or failure sites on returns is normally only attempted when essential. Recent advances in equipment and analysis techniques are now removing many of the existing limitations that are imposed by complex structures and sensitive materials.

Focused ion beam (FIB) instruments are widely used in the semiconductor and data-storage industries for many applications, including 3D materials analysis, photolithographic mask repair, circuit edit and general nanoengineering. The ability to focus a beam of ions onto an area of a sample with precision and accuracy means that specific sites can be machined by the beam to modify the structure by sputtering away material. Buried structures such as HBTs can also be cross-sectioned and imaged using the ion beam. Imaging the area of interest is accomplished by scanning the ion beam across the sample and detecting the electrons emitted as a result of the interaction between the ion beam and the surface. Scanning ion microscopy has evolved to a point where it rivals high-resolution SEM as a microscopy technique in its own right.

## FIB sample preparation

Preparing thin samples by FIB machining for subsequent TEM analysis is a widely accepted technique in the silicon-based IC industry (see figure 1). The benefits of using FIB technology in this way are very clear; the process is very fast and labor saving when compared

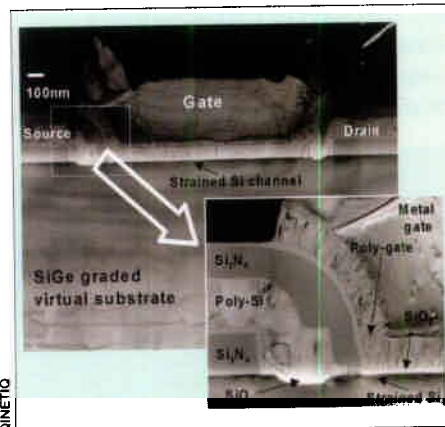


Fig. 1. TEM image of a foil machined by FIB from a strained silicon MOSFET device. The dark layer at the top is tungsten deposited as part of the FIB sample preparation.

**Recent developments have resulted in a unique integrated dual-beam system that combines a field emission electron beam with an ion beam.**

with sample preparation by mechanical grinding and polishing. It is very site specific (placement accuracies down to  $\pm 500 \text{ \AA}$ ), independent of different material hardnesses within a single foil and is the only fully instrumental technique that does not require any mechanical preparation of the bulk sample. These physical benefits are now routinely combined with multi-site automatic operation of the FIB instrument.

Recent developments at FEI Company, a manufacturer of FIB instruments and a provider of FIB analysis services, have resulted in a unique dual-beam system that combines a field emission (FE) electron beam with an

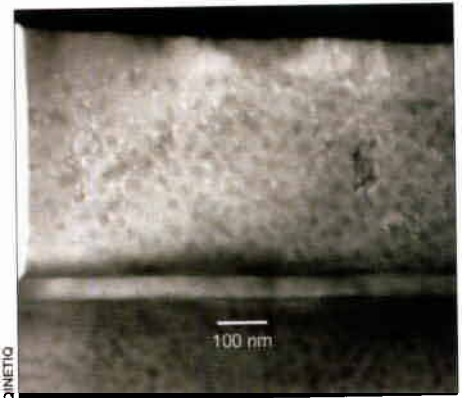


Fig. 2. High-magnification TEM image from part of a FIB foil taken using a single-beam instrument from an InGaP HBT. The speckled appearance is due to materials redeposited during the final ion-beam polish of the foil.

ion beam. This gives the ability to prepare a thin section of material and perform electron-beam analysis of that section within a single tool called a DualBeam instrument. The developments described within this article are specific to FEI's Strata DB235 DualBeam tool, and the TEM images were taken by the materials characterization group at QinetiQ in the UK.

## The DualBeam advantage

The advantage of the electron and ion beam integration system developed at FEI's laboratories is that all of the benefits offered by FIB and high-resolution FESEM instruments can be used without compromising either technique. The beams can also be used simultaneously in a single-point operation. This mix-and-match capability allows the user the freedom to select the excitation method, imaging technique used and the analysis preferred depending on what is available from the specimen. This affords a level of instantaneous investigation that was not previously possible. However, the effectiveness and precision of a dual-beam instrument is highly



Fig. 6. Image showing a FIB-prepared foil in a copper TEM sample grid.

and then cleaving up the sample and grinding it down to less than 50  $\mu\text{m}$  thick. This pre-thinned slice is then attached to a modified TEM sample grid and mounted in the FIB instrument prior to ion-beam thinning (see figure 4). After FIB processing, the grid was then removed from the FIB vacuum and transferred to the TEM for analysis. This procedure took 2.5–3 hours and required considerable operator skill.

*Ex situ* extraction is currently the most widely used technique. Any sample can be mounted and loaded into the FIB instrument with no mechanical preparation. Samples do not even need to be removed from packaging, as long as access to the top surface of the sample can be achieved with the ion beam. A thin foil can then be cut out by the ion beam, leaving it free standing in a FIB-milled trench on the surface of the sample (see figure 5). This sample is then removed from the instrument and with the use of an optical workstation, an electrostatic needle is used to extract the thinned foil and transfer it to a support film (normally holey carbon) on a copper TEM grid (see figure 6). This extraction technique requires a little experience before success rates of equal to or greater than 95% can be achieved routinely. However, it is highly effective and can be used on an enormously wide variety of materials, even ones with poor structural rigidity such as compressed powders and samples with voids or cracks. Unfortunately, exposing the sample to the atmosphere during transfer to the TEM can be problematic for materials such as those with a high aluminum content, which

can suffer from immediate degradation when exposed to atmosphere.

*In situ* extraction has been made possible by the addition of a micromanipulator to transfer the sample to a TEM grid and a further manipulator, known as a FlipStage, which flips over the sample onto the STEM detector. As a result, all of the steps from sample preparation through to STEM analysis can take place without ever removing the material from the vacuum in a DualBeam tool. This technique requires a lower level of operator training to complete, and has a success rate as high as *ex situ* extraction for an experienced operator. The procedure relies on the specimen being able to support itself (so powdered or brittle samples are less suitable), but it does permit re-thinning by the ion beam for specimens that require it.

### Elemental mapping

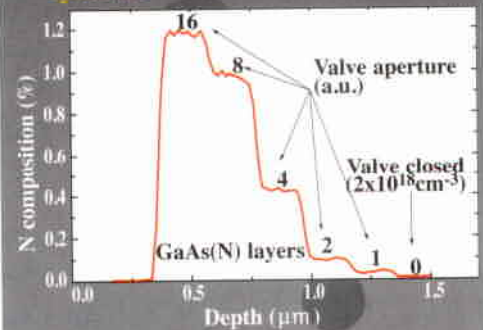
By allowing thinned samples to be examined *in situ* using the SEM function in a DualBeam instrument, it becomes possible to use other techniques that are available to SEM users. The energy dispersive X-ray (EDX) unit, which is mounted on most SEMs for elemental mapping, is an excellent example. The volumetric effect that reduces the mapping resolution to about 1  $\mu\text{m}$  for thick samples is removed when analyzing thin foils. EDX mapping of foils using this *in situ* method can achieve spatial resolutions of 35–50 nm. When these elemental results are then combined with the 0.6–1 nm high-contrast STEM imaging, we have some complementary analysis capabilities that are fast and very useful, and on a sample that can be made from bulk material without any mechanical preparation at all.

Tools such as the Strata DB235 bring together a total sample preparation and high-resolution imaging and analysis capability on a single-chamber system. Such an instrument complements existing sample-preparation techniques and also reconfirms TEM as a high-end analysis instrument. It enables the rapid production of many more thin samples and takes on a high proportion of the routine analysis work, providing efficiency improvements at all stages of the analysis process. ●

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dependent on the way the instrument is designed and operated. The design of an instrument with co-located ion and electron beams, each with its own requirement for electrostatic beam steering and focusing optics, is a non-trivial exercise. High-resolution imaging places stringent requirements on vibration isolation, and the ability to move thinned samples inside the instrument requires precision in mechanical placement and alignment, combined with high levels of repeatability.

FIB systems are used for an increasingly wide variety of materials including ceramics, polymers, superconductors, piezo-electrics and many different types of compound semiconductors. Some aspects of the FIB-SEM process offer unique advantages for compound semiconductors. The most commonly used foil size ( $20 \times 10 \times 0.1 \mu\text{m}$ ) makes this process ideal for looking at structures fabricated on the surface of any type of semiconductor substrate and for analyzing thin films or surface treatments. Compound semiconductors are generally more sensitive than silicon to crystalline damage at the surface when prepared using FIB techniques. During the final ion-beam polishing stages of making a TEM foil with a single-beam FIB instrument, it has become common practice to avoid imaging the face of the foil with the ion beam to minimize any unnecessary crystalline disturbance. The effect of this practice means that the foil surface cannot be quality checked by imaging after the final polish.

Although high-quality foils are possible with both single- and dual-beam instruments, only the dual-beam systems can give direct feedback through the use of their electron-beam imaging capability during this final sensitive part of the foil production process. This extra degree of control permits thinner, higher quality foils to be made routinely, with a higher degree of confidence in the final results. Optimization of the final polishing methodologies in both FIB and dual-beam instruments means that high-quality foils with a thickness of 50–100 nm can be produced routinely, with no observable crystalline modification in the final specimen. The facility to take electron images in immersion (ultra-high resolution) mode while milling is possible because the electron and ion columns have been designed so that the beams are coincident at the sample stage's eucentric tilt axis, which is only 5 mm from the electron-beam lens.

Figure 2 shows a TEM image of part of a TEM foil sectioned from a GaAs/InGaP HBT structure in a single-beam instrument. The

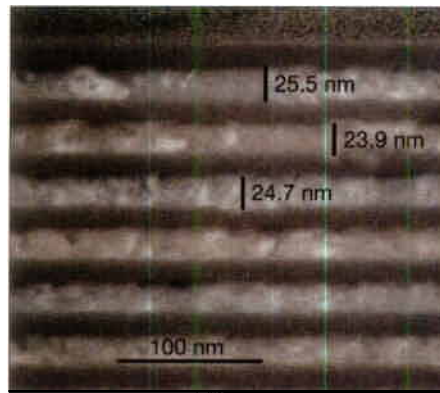


Fig. 3. A DualBeam STEM image of a GaAs/AlGaAs QW laser structure. The foil was prepared using a dual-beam system and the final surface is clear of redeposited material.



Fig. 4. The sample in this image was mechanically pre-thinned before being placed into a FIB system where the thin foil in the center was machined.

speckled appearance is the result of milled material that has been redistributed during the final polishing step. This might have been avoided or removed if the system operator had been aware of it. Figure 3 is a GaAs/AlGaAs QW laser structure prepared in a dual-beam instrument, which permits the final foil quality to be guaranteed and even examined (here by STEM) before transfer for TEM analysis.

#### Advances in detectors

Electron detectors are an area where technological advances are now yielding significant improvements in analysis capabilities. STEM imaging using conventional FESEM

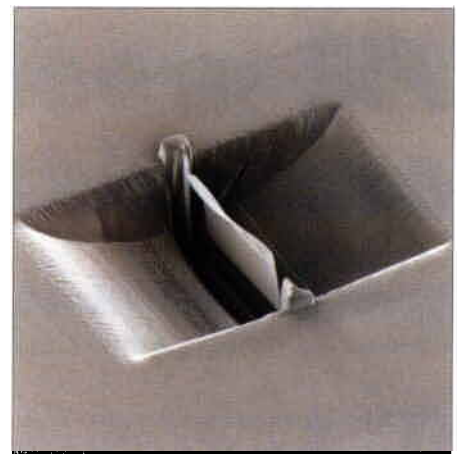


Fig. 5. Image showing a FIB-prepared TEM foil before *ex situ* extraction.

accelerating voltages can be added to new FESEM or DualBeam instruments. Thin samples can therefore be examined in transmission mode inside a DualBeam instrument. This has had several effects on the analysis procedures currently in routine use. Firstly, TEM can now be reserved for only the most challenging of investigations. Secondly, all samples for TEM can be pre-imaged in transmission mode to ensure that they are of the correct quality and thickness before any TEM time is used. Finally, many routine investigations can be solved without using valuable TEM instrument time, as the improved resolution (0.6–1.0 nm) offered by *in situ* STEM detection will be sufficient to provide the answers in many cases.

STEM detection does not permit diffraction information to be obtained, but it does permit good metrology information to be gathered because of the high levels of materials contrast seen with this technique. Low-contrast imaging can be a problem with transmission mode studies of thin specimens. This pre-examination and filtering of samples means that a significantly higher level of efficiency can be introduced to the analysis. Many more samples can be produced and successfully analyzed, and more information can be obtained in a shorter amount of time without a significant increase in costs or by having to change the existing laboratory equipment.

#### Advances in sample management

Several strategies are employed for extracting thin-foil TEM samples prepared by FIB. Conventional (old style) FIB-TEM sample preparation involved removing the material from whatever packaging it was enclosed in