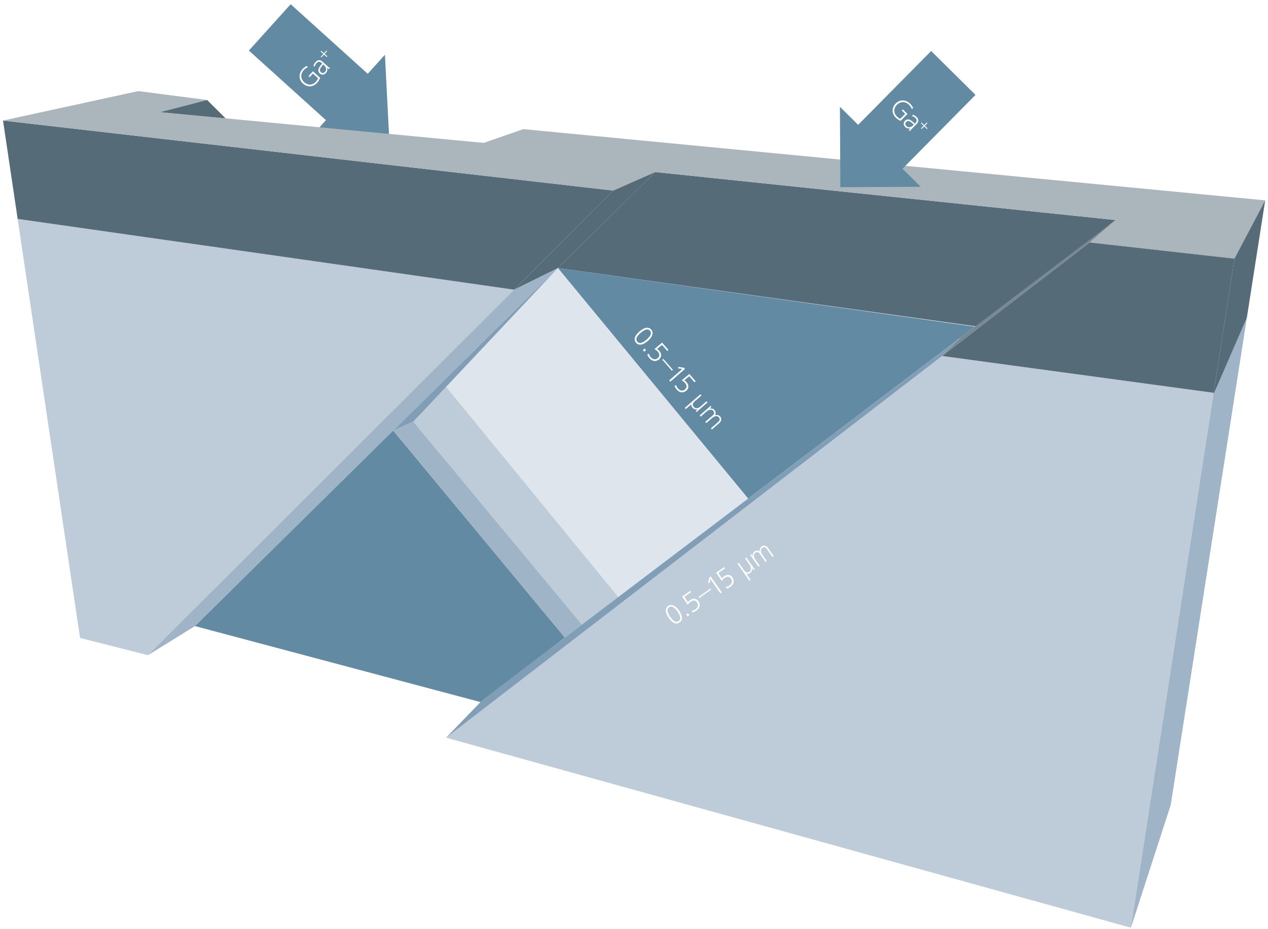
Application Note



X² STEM Lamella Preparation from Multi-composite Organic Electronic Devices with ZEISS FIB-SEMs

We make it visible.

X^2 STEM Lamella Preparation from Multi-composite Organic Electronic Devices with ZEISS FIB-SEMs

Author: Heiko Stegmann
Carl Zeiss Microscopy GmbH, Germany

Sylvia Mucke, Sven Quix
Plastic Logic GmbH, Dresden, Germany

Date: April 2015

Preparation of ultrathin lamellas from polymer samples using conventional focused ion beam (FIB) preparation protocols often results in strong bending, distortion and nonuniform thickness of the lamella. The X^2 method significantly improves stability of such lamellas. Using a dedicated ZEISS sample holder, ultrathin lamella can be produced from sensitive polymer samples with low distortion and uniform thickness. In this paper, we show the application of this technique to a multi-composite polymer / metal layer system of an organic display device using a ZEISS FIB-SEM (focused ion beam scanning electron microscope).

Polymer display specimen preparation and imaging

Polymer transistor technology allows the manufacturing of microelectronic circuits on flexible plastic sheets. The most advanced of such technologies uses a large area active matrix backplane to drive electrophoretic display or sensor media. Flexible displays with a thickness of a sheet of paper and pixel density of up to 150ppi can be produced.
The next generations of such electronic devices will integrate several different functions on a single plastic film, such as display, sensors and logic (‘system on a film’).

Process development and manufacturing of these devices require structural control at the nanometer scale. Ultrathin lamellas need to be prepared from specific device structures for electron microscopic imaging in transmission mode. Often, tiny defects in single pixels of the display need to be located and prepared. In order to avoid misinterpretation of interface morphology, cystallinity and thickness of the multiple polymer and metal layers, the lamellas must be thinner than 50 nm and free of preparation artefacts. This is very difficult to achieve by ultramicrotomy. For routine lamella preparation on such samples, focused ion beam (FIB) cutting followed by in-situ liftout of the lamella with a micromanipulator has turned out to be a much better solution. At Plastic Logic GmbH, such FIB lamellas are routinely prepared from production and development samples using a ZEISS FIB-SEM.

The materials used are also sensitive to electron beam damage. To reduce specimen damage by SEM imaging during FIB preparation, low acceleration voltages must be used. When imaging the finished lamellas in transmission, sub-nanometer resolution is not required to examine the device structures. As a result, the lamellas produced in the ZEISS FIB-SEM at Plastic Logic do not require imaging in a dedicated TEM or STEM, but can be imaged in the same tool at moderate voltage using a retractable STEM detector.

Polymers tend to bend, roll and melt under FIB induced thermal stress [1]. Even delamination of the stacked metal and polymer layers is possible. These problems can be minimized with the X^2 method. It increases the lamella stability and controllability of the FIB thinning process.

Conventional and X^2 FIB lamella preparation

The conventional method of preparing a thin, electron-transparent window with the FIB consists of symmetrical milling from the upper sample edge down (Figure 1, left). This is done step-wise with decreasing beam currents.

When the window is thinned below a certain threshold, warping is caused because of intrinsic or milling induced strain. Despite using adaptive milling techniques such as counter-tilting the lamella towards the beam, and special

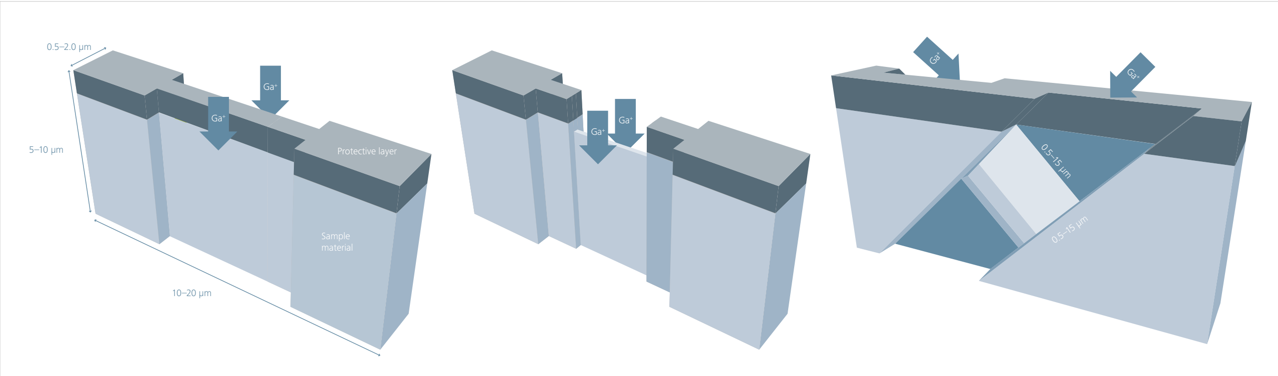


Figure 1 *Principle of conventional (left, center) and X^2 method (right) lamella thinning.*

mounting techniques such as milling from the bottom side up, large mechanically stable and evenly thin areas cannot be obtained from many materials, in particular from polymers.

Unintentional removal of the protection layer deposited on the sample surface before the lamella preparation is a further problem during thinning to the final thickness (Figure 1, center). When reducing the window thickness by only a few ten nanometers, the beam tails of the FIB beam can eat into the upper edge of the window by several micrometers. This can be mitigated by depositing very thick sacrificial protective layers, but this is a time consuming and expensive process.

The approach of the X^2 method to avoid these problems is to support the ultra-thin window at the region of interest by a closed frame. The frame is formed by the thicker material surrounding the window. This is achieved by an angular displacement between the front side and the back side FIB cut (Figure 1, right).

Because of this window geometry, a large region of interest can be milled to very small thickness without the bending or reduction of the lamella area [2]. The frame stabilizes the lamella and serves as sacrificial material to protect the thin window from damage by the beam tails. This is especially advantageous when applying low kV polishing at comparatively large beam diameters to achieve extremely small thickness while simultaneously reducing the Ga induced amorphisation layer.

ZEISS has designed a special specimen holder for the X^2 method [3]. The necessary transition between the two milling directions is triggered by a simple 180 degree rotation of the microscope stage (Figure 2). Simultaneously, the side of the lamella to be milled is turned towards the SEM lens for visual real-time control of the thinning process.
The milling angles are adjustable by movable stoppers. In the following paragraph, we show the application of this setup for the preparation of ultrathin lamellas from polymer display samples, and compare the results to those of conventional FIB lamella preparation.

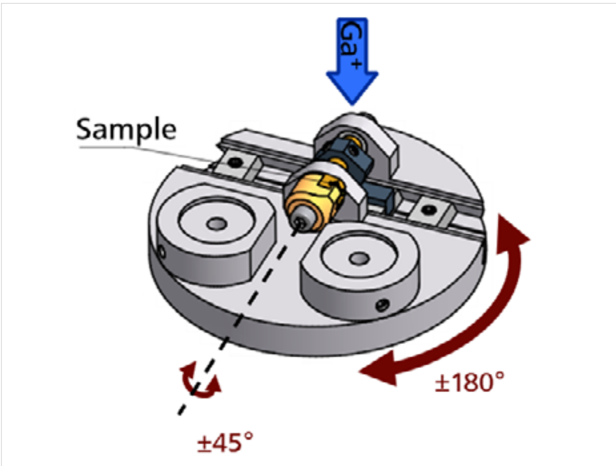


Figure 2 *X^2 sample holder. Rotation of the holder while the stage is tilted flips the TEM grid from the front side to the back side milling position.*

Experimental setup

Three lift-out lamellas containing a polymer-metal stack with multiple interfaces were prepared from a Plastic Logic polymer device in a ZEISS FIB-SEM. One lamella was attached at the side of a lift-out grid post ('side mount', Figure 3 top left), another one was attached to the top of a post ('base mount', Figure 4 top left). In both cases the grids were mounted in a standard lift-out grid holder. The third lamella was also attached in base mount, but with the grid mounted in the X^2 holder (Figure 5, top left).

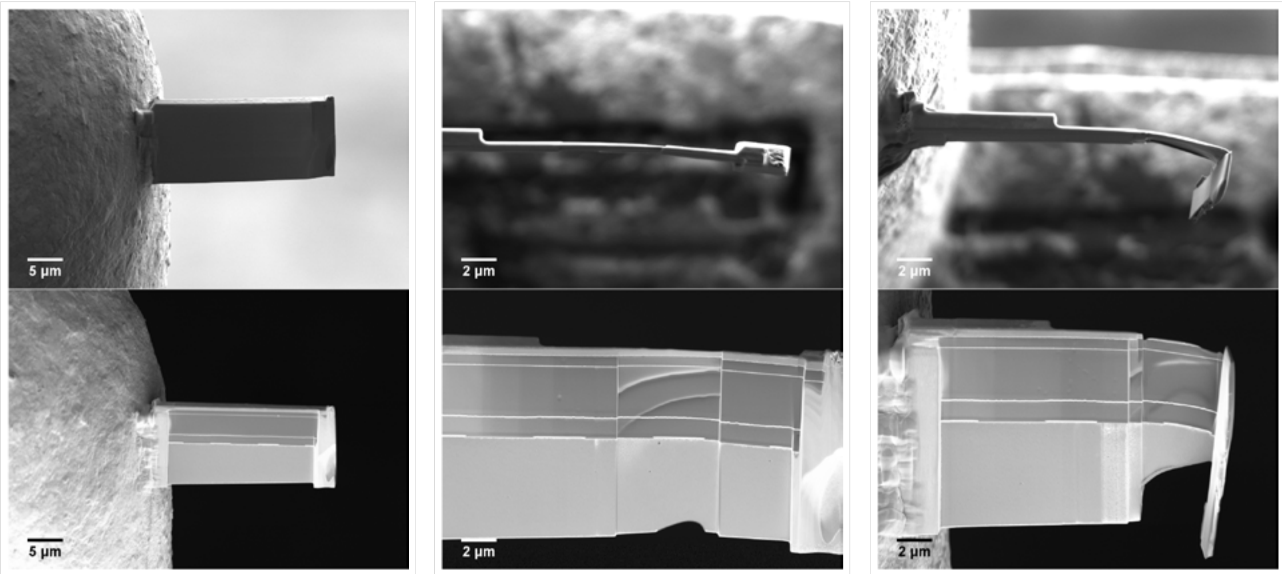


Figure 3 *Conventional FIB lamella preparation, side mount. SEM micrographs recorded with SE2 detector (top row) and Inlens SE detector (bottom row). The lamella is bending early during thinning. Further thinning is not possible.*

In a first thinning step, the thickness of the lamellas was reduced from an initial 1 µm to approximately 300 nm using 240 pA FIB current at 30 kV. At this step already, the side mount lamella, being supported at two sides only, showed considerable bending (Figure 3, center). No bending occurred with the bottom mount lamella on the standard and on the X^2 holder at this point (Figures 4, center, and 5, bottom left and top center), due to them being supported on three, respectively four, sides by the surrounding bulk material. In the following thinning step that used 20 pA current at 30 kV to produce an approximately 8 µm wide electron-transparent window, the side mount lamella developed very strong bending (Figure 3, right), so that further thinning was not possible. In comparison, the bottom mount lamella on standard holder showed reduced bending. However it eventually perforated over the complete window width due to the residual bending (Figure 4, right). Therefore, it was not possible to achieve a uniform thickness across the whole lamella. The X^2 lamella, however, stayed flat and could be thinned much more evenly until being perforated in one small spot (Figure 5, bottom center). The final X^2 lamella was imaged without further processing at 28 kV in transmission

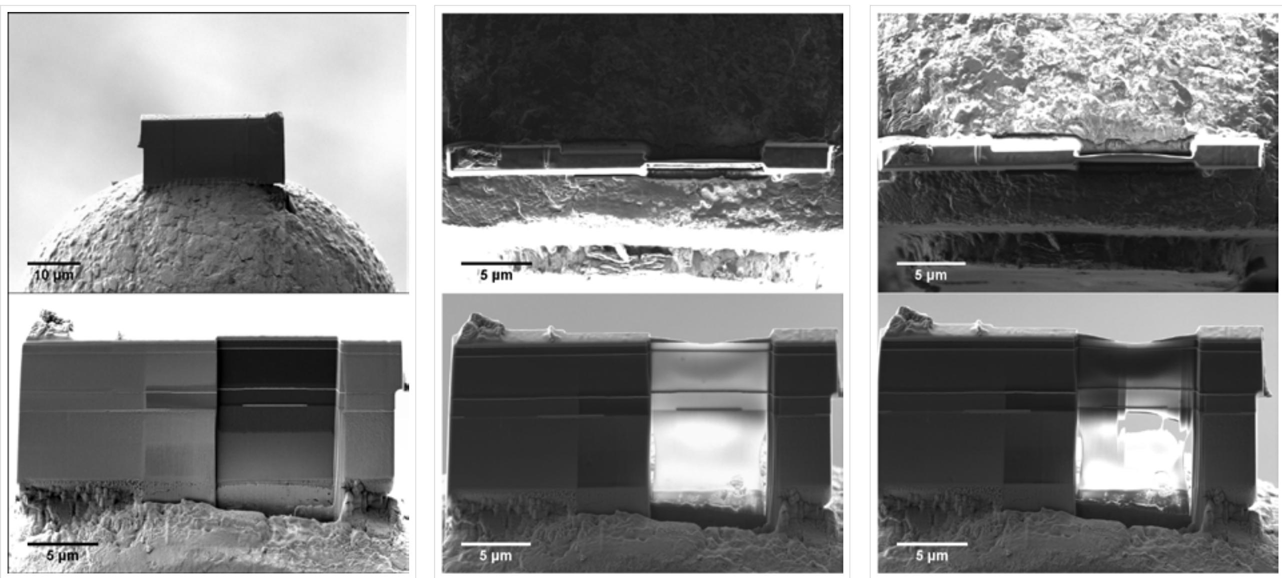


Figure 4 *Conventional FIB lamella preparation, bottom mount. SEM micrographs recorded with SE2 and Inlens SE detectors. Bending is reduced, but still present. When thinning further, this leads to uneven thickness and, eventually, to destruction of the lamella.*

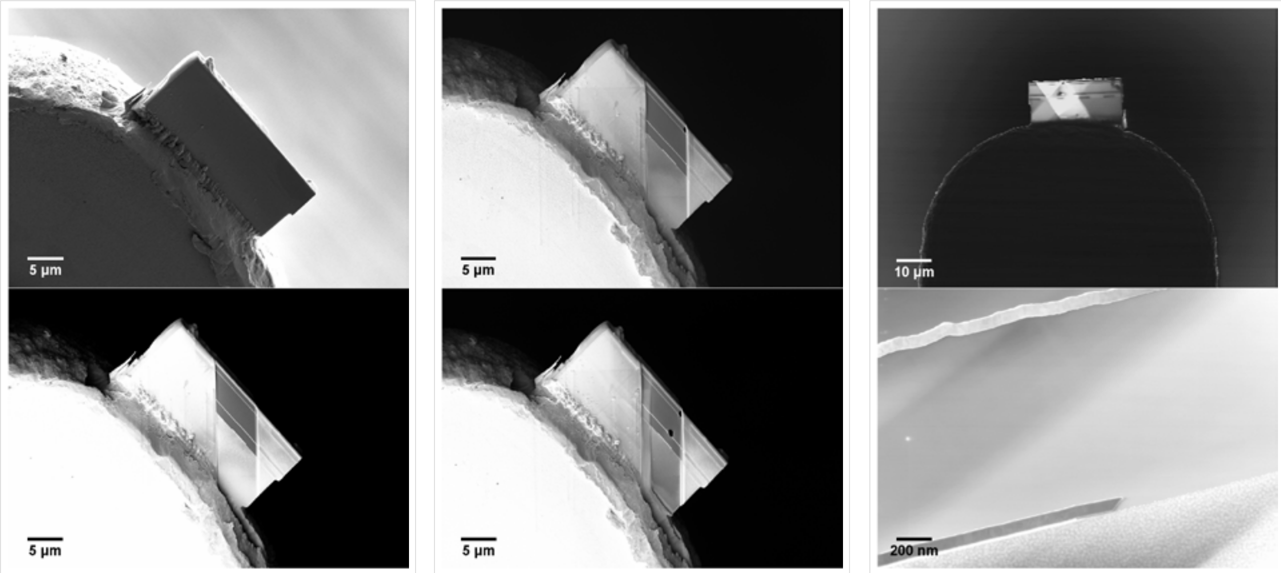


Figure 5 *X^2 lamella preparation. SEM micrographs recorded with SE2, Inlens SE and (right column) STEM detectors. The lamella is thinned until a small hole appears, but no bending occurs.*

using the retractable STEM detector of the ZEISS FIB-SEM (Figure 5, right). The lamella quality was sufficient to reveal nanometer sized polishing residue particles (Figure 6).

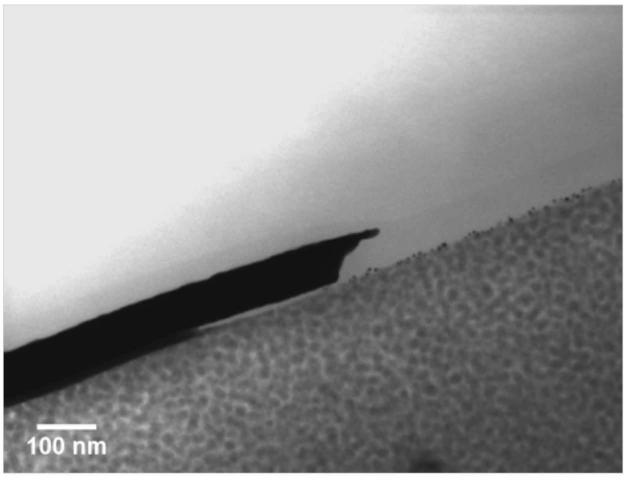


Figure 6 *28 kV STEM brightfield image recorded from the X^2 lamella in the ZEISS FIB-SEM.*

Summary

The above comparison of different FIB protocols for the preparation of electron-transparent lamellas from multi-composite polymer/metal layer system of an organic display device showed that conventional protocols produce bent and irregularly thick lamellas. The X² method, using a dedicated ZEISS sample holder, allows FIB preparation of ultrathin lamellas with low distortion and uniform thickness. They are thin enough and sufficiently free of preparation artefacts to be imaged by low voltage STEM in the same ZEISS FIB-SEM. Therefore, precise structural control of specific device structures at the nanometer scale is provided to process developers and manufacturing engineers of organic displays.

References:

[1] S. Kim et al., Ultramicroscopy, 111:191 – 199 (2011).

[2] L. Lechner et al., Microsc. Microanal. 17 (Suppl 2):628 – 629 (2011).

[3] R. Salzer et al., Microsc. Microanal. 18 (Suppl 2):654 – 655 (2012).

EN_42_013_159 | CZ 04-2015 | Design, scope of delivery and technical progress subject to change without notice. | © Carl Zeiss Microscopy GmbH

Carl Zeiss Microscopy GmbH
07745 Jena, Germany
microscopy@zeiss.com
www.zeiss.com/microscopy

We make it visible.