TEM Sample Lift-out Using the Zyvex Nanoprober System

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Introduction

The Zyvex Nanoprober System, coupled with a focused ion beam (FIB) tool, is a complete solution for TEM sample preparation. The Zyvex System has the ability to perform the following tasks in a dual beam FIB (dbFIB) environment (see **Figure 1**).

- Probe bulk samples
- Take electrical measurements
- Manipulate samples
- Perform immediate *in-situ* transmission electron microscope (TEM) sample lift-out

The ability to prepare site-specific specimens (that can be removed from the bulk of a sample) provides more insight into defect analysis, process monitoring and control, and materials characterization of increasingly smaller areas. This is an extremely important application.

The Zyvex Nanoprober (**Figure 2**) is capable of performing the standard *in-situ* lift-out method using a tungsten probe along with FIB micro-machining and welding. Zyvex has developed a new approach to TEM sample lift-out by using NanoEffector® microgrippers to grasp the FIB-cut samples. This unique approach allows samples to be removed and placed onto a TEM grid inside either a scanning electron microscope (SEM) or an FIB system using, *in-situ*, the Zyvex Nanoprober.

The new Zyvex NanoEffector microgripper-based method is favorable over traditional methods since samples can be removed and manipulated without being contaminated by FIB deposition.

Background

Zyvex has developed an automated assembly technology for microsystems which is scaleable to nanosystems manipulation and assembly. Much of this research and development has been done using electrothermal micro-electromechanicial systems (MEMS)-based microgrippers to assemble many three-dimensional structured microsystems

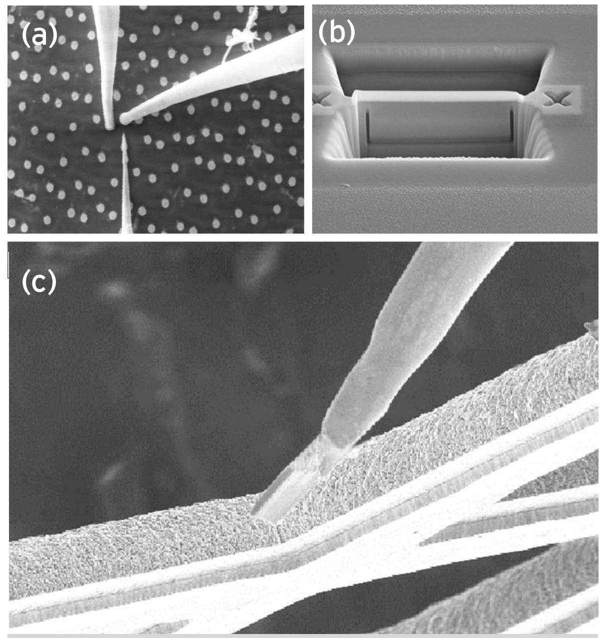


Figure 1
(a) Zyvex Nanoprober probing
(b) Zyvex Nanoprober cutting samples
(c) Zyvex Nanoprober lift-out

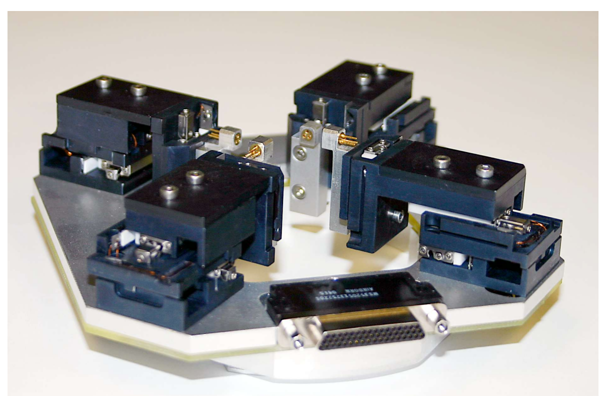


Figure 2 Zyvex Nanoprober System

(see **Figure 3**). MEMS grippers allow small, delicate objects to be maneuvered with nanometer-scale precision and accuracy. These grippers have other assembly applications, including TEM sample preparation.

Zyvex NanoEffector Microgrippers (see **Figure 4**) are used for TEM sample lift-out. The grippers use electrothermal actuation to achieve a range of motion from zero to five microns of opening at the tips. An electrical potential is applied across the two anchor pads, passing current through the bent-beam structures. Joule heating within the beams results in localized thermal expansion. This thermal expansion drives flexures which amplify and create horizontal motion at the gripper tips.

In the MEMS fabrication process, there is a minimum spacing between the tips. Zyvex packages the NanoEffector Microgrippers onto an aluminum nitride sub-mount.

The sub-mount, with microgrippers attached, is then mounted on the end of the Zyvex Nanoprober plug. This gives the microgrippers the ability to move in x, y, z, and θ to grasp the sample and lift it out. The range of motion of the Nanoprober is excellent for this task.

Probe in-situ Lift-out Method

The Zyvex System is capable of performing the standard method of *in-situ* TEM lift-out using Zyvex NanoEffector tungsten probes. After locating the area of interest, a sample is micromachined in the dbFIB system. One of the four probes on the Nanoprober is then inserted into the area of interest. The probe is placed on top of the sample and then welded to the sample using FIB deposition. The tethers are cut to ensure the sample is entirely free. To lift out the sample and find the TEM grid, the Zyvex Nanoprober then moves up in z and over in x and y. The sample is then welded to the TEM grid for further thinning (if necessary). The steps to this process are shown in **Figure 5**.

Normal welding techniques are not always reliable because the probe does not weld very securely to the sample using the platinum deposition. It is easy to break the platinum deposition if the sample is not completely free at all times while trying to move the probe. Even vibrations from the gas injector can cause the platinum to crack.

It should be noted that this method requires some maintenance to the probe to keep it sharp. The tip must be

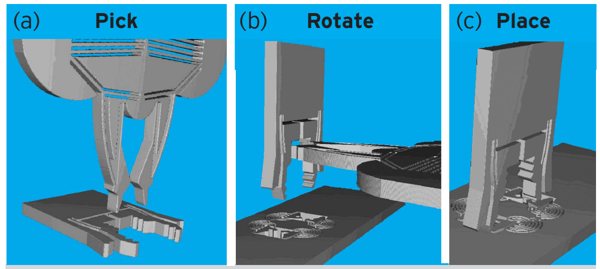


Figure 3 (a-c) Zyvex technology using microgrippers

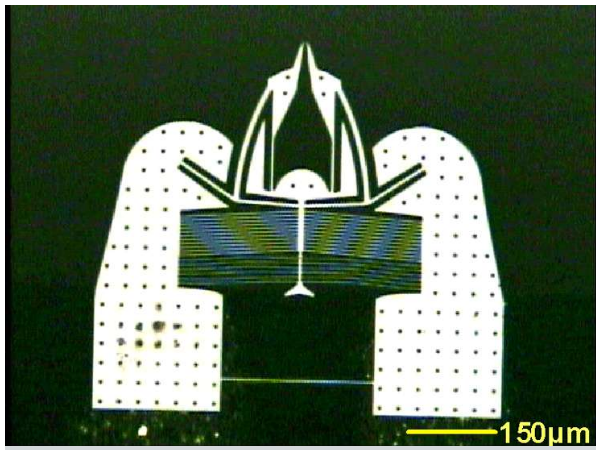
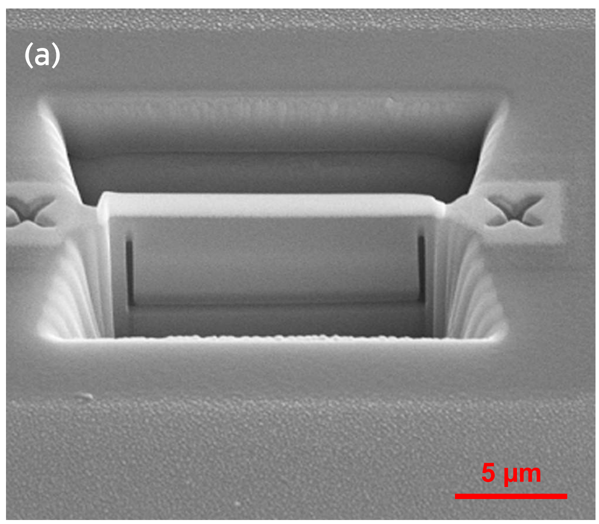


Figure 4 Power-open electrothermal grippers



re-etched every time it becomes dull from the welding and cutting contamination process. This method also risks contaminating the sample.

The Zyvex *in-situ* Lift-out Method

Current methods of probe *in-situ* lift-out are cumbersome, time consuming, and may contaminate the sample in the process. Zyvex believes there is a better solution to this task. By eliminating the steps of welding and milling the probe to the sample, all of the limitations above can be surmounted.

The Zyvex lift-out method uses NanoEffector Microgrippers powered by electrothermal actuators. These state-of-the-art microgrippers allow the sample to be easily grasped and manipulated freely — without risking contamination. When Zyvex's microgrippers are powered, they open. When the power is turned off the microgrippers close around the sample in order to retrieve it from the FIB-cut trench. The gripping motion is achieved by thermal expansion and motion amplification which has been optimized to achieve appropriate deflection with sufficient force at the tips.

Ideally, for efficient TEM lift-out, the samples are picked and placed in a serial automated fashion onto a carbon-backed TEM grid. This process would be useful for a very high efficiency design. If a higher quality of analysis is necessary, and efficiency is not as much of a concern, the samples could be welded onto the side of a TEM grid.

With the Zyvex microgripper lift-out process, it is possible to retrieve FIB-cut samples of varying sizes since the micro-grippers have a variable opening.

Another benefit of this new process is that the sample need not be completely cut free to lift it out. Our samples are undercut and are left with only one tether. The gripping force causes the tether to controllably break during lift-out.

As shown in **Figure 6**, the grippers are aligned over the sample using the Nanoprober. The power is turned on to open the grippers above the sample.

Then the grippers move down, inserting the gripper tips around the sample. The power is turned off and the sample is grasped as the tether is broken. The sample is securely grasped and is then removed from the trench. **Figure 7** shows the lift-out sequence.

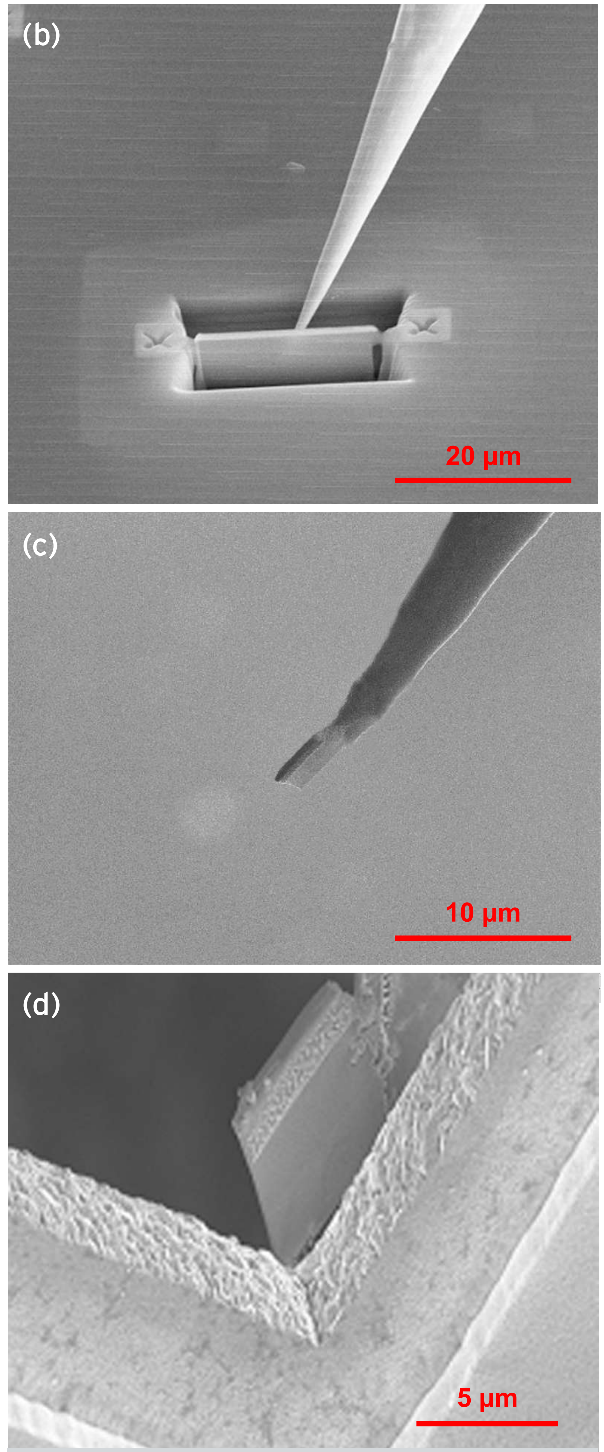


Figure 5 (a-d) *In-situ* probe method for TEM lift-out

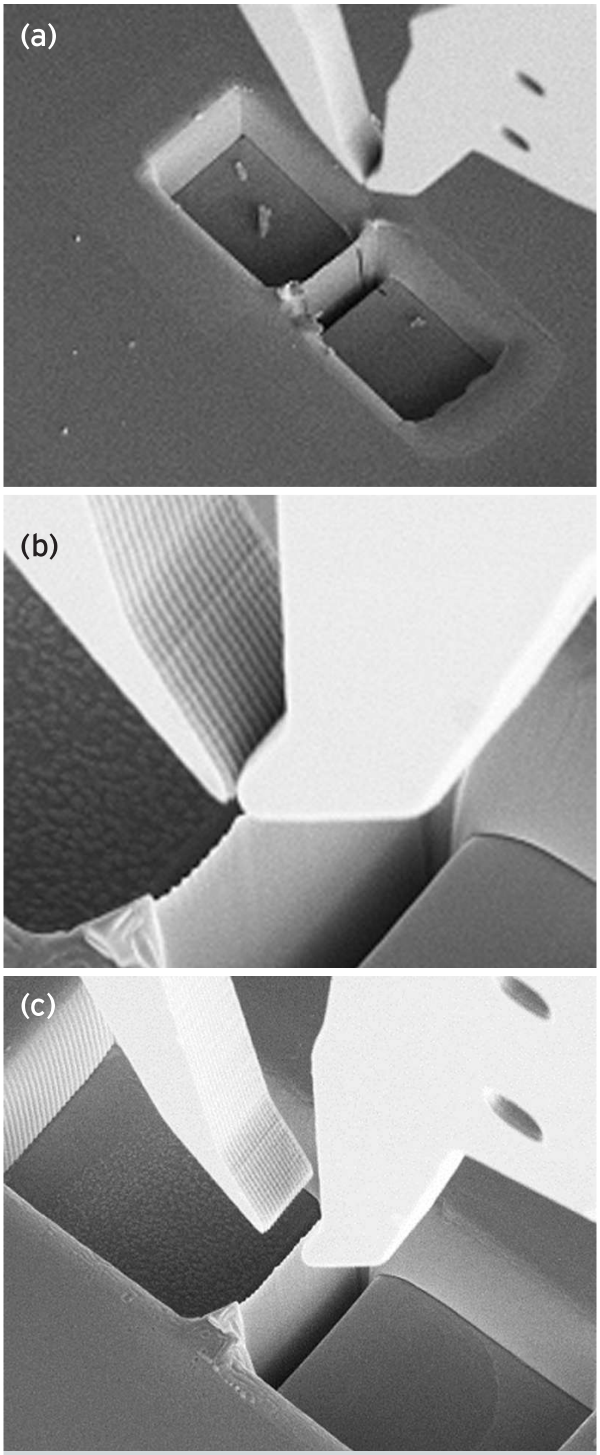


Figure 6 (a-c) Preparing to lift-out using MEMS grippers

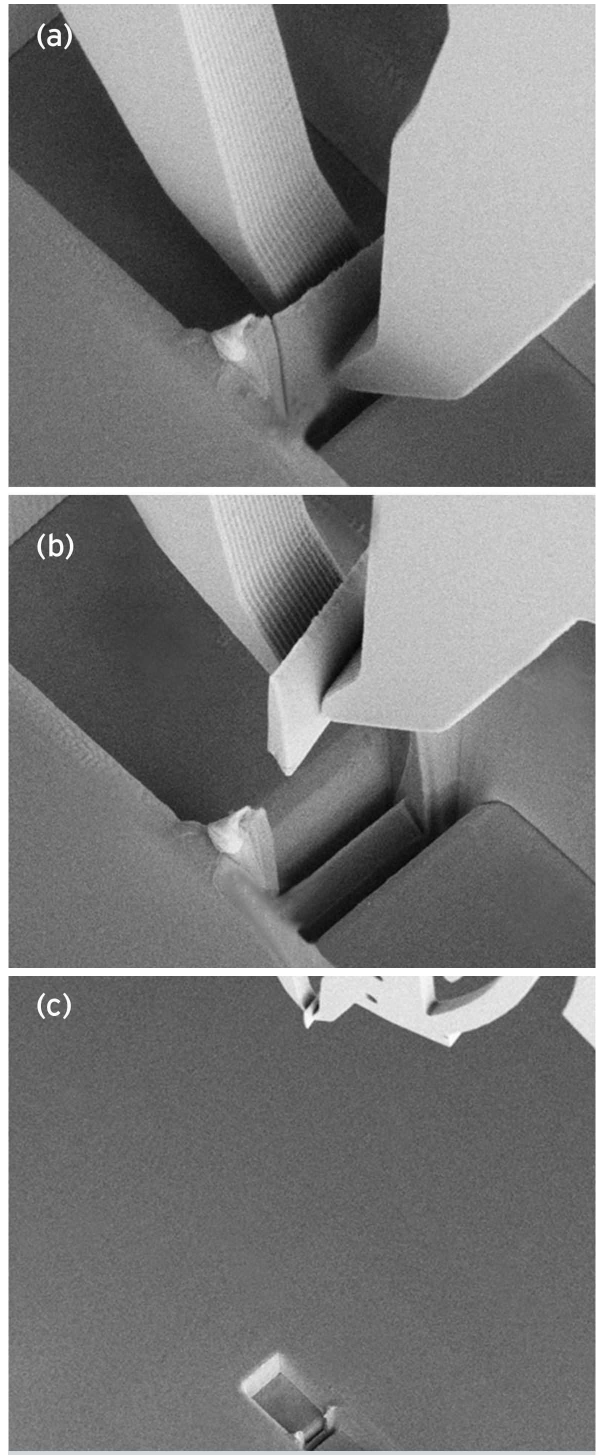


Figure 7 (a-c) Lifting out the sample with MEMS grippers

The final step is to locate the sample to the TEM grid by moving in x and y and rotating the stage. Then the sample is placed into the groove of the grid, and welded in place. It is also possible to set the sample down by opening the gripper to release it. You can also perform this process on a carbon backed grid. **Figure 8** shows the sample placed onto the side of the grid as a demonstration of the ability to release the samples without stiction. Welding to the side of the grid is necessary for re-thinning the sample.

The Zyvex microgripper lift-out process has many advantages over the standard probe lift-out method. It avoids many of the cutting and welding steps which shortens the process—saving time. Contamination is also less likely to occur since the tool is not welded to the sample. Also the microgrippers can be re-used for hundreds to thousands of lift-outs without requiring maintenance to maintain their performance. The standard probe lift-out method requires re-sharpening in the FIB after each sample lift-out. The Zyvex Nanoprober, with better than 5 nm resolution and 4 degrees-of-freedom, makes manipulation controlled and smooth.

Conclusions

NanoEffector Microgrippers used with the Zyvex Nanoprober for TEM sample lift-out is a new approach that overcomes some of the current limitations of probe *in-situ* lift-out. It is capable of performing lift-out with two processes — from four different orientations. The key advantages of the NanoEffector microgripper lift-out method are: faster lift-out times, less contamination, reliable process, reusable, no maintenance, and easy to use. Future developments for this application will implement automation to minimize user interaction and increase efficiency.

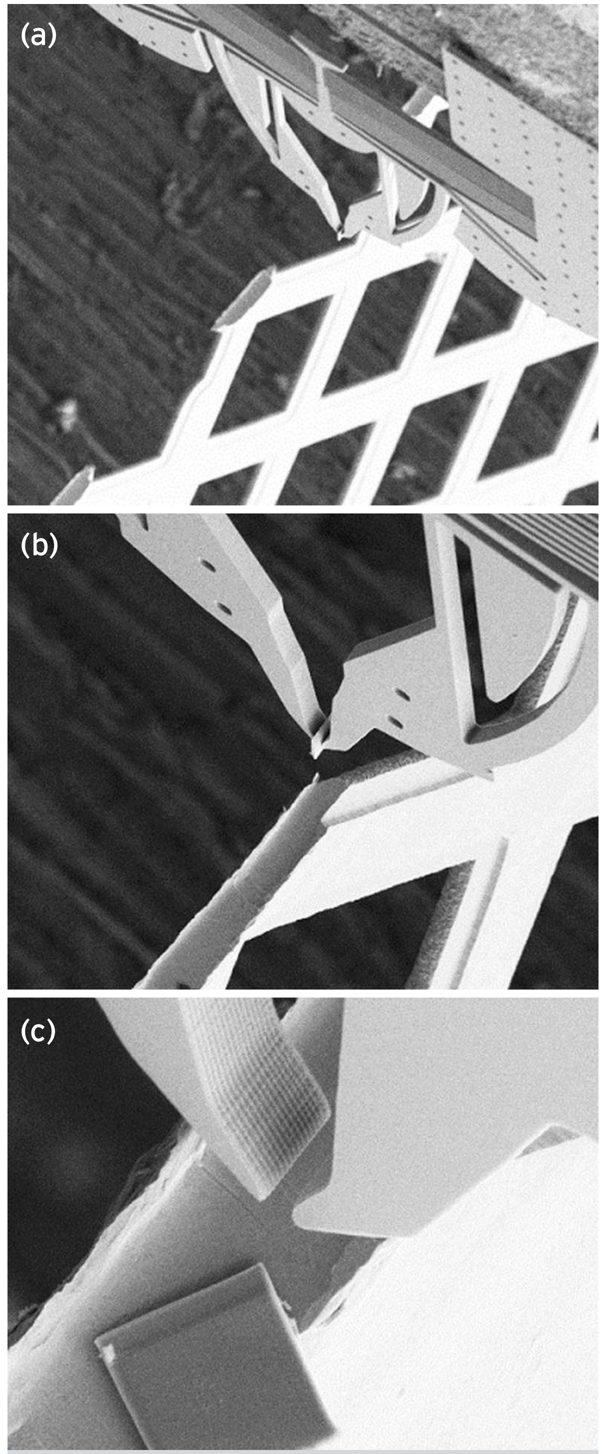


Figure 8 (a-c) Placement of sample onto grid

 Document: TEMS-ZZAN-001d

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