Applications of Multibeam SEM/FIB Instrumentation in the Integrated Sciences

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The Hague, Netherlands, 1690. Christiaan Huygens and Sir Isaac Newton, at odds with their competing wave/particle theories of light, today reached a compromise with their unveiling of a new multibeam SEM/FIB instrument featuring an SEM column designed by Huygens that uses electromagnetic lenses to focus the beam of electrons and a FIB column employing electrostatic lenses to focus the beam of ions devised by Newton. Onlookers were dazzled by its extensive capabilities and range of applications.

1. Introduction

Obviously this headline is purely fictitious, but dual beam SEM/FIB instruments are becoming increasingly popular in all facets of research in the physical and life sciences [1-2]. An example of one of the more recent additions to the market is the JEOL JIB-4500. This instrument consists of a vertically mounted SEM column, featuring a LaB_6 electron source (a field emission source instrument is also available), and a focused ion beam column mounted at 52˚ to the SEM with a Ga^+ liquid metal ion source (see Figure 1). As with all modern SEMs, a host of ports are available for complementary analysis techniques such as EDS, CL and EBSD. Additional features of the instrument are the gas injection systems. These allow deposits of C, W and Pt to be formed on the sample surface by the interaction of either the ion or electron beam with a suitable precursor gas delivered by the gas injection nozzles adjacent to the sample surface. Combined with the sputtering or milling action of the focused ion beam, this produces an all-in-one instrument capable of imaging, analyzing, removing, and adding nanometer-sized features to samples from the physical and life sciences.

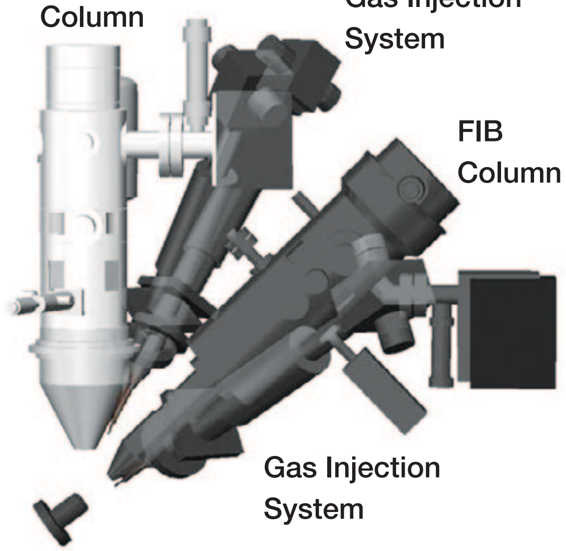


Figure 1: Schematic diagram of the JEOL JIB-4500 multibeam instrument.

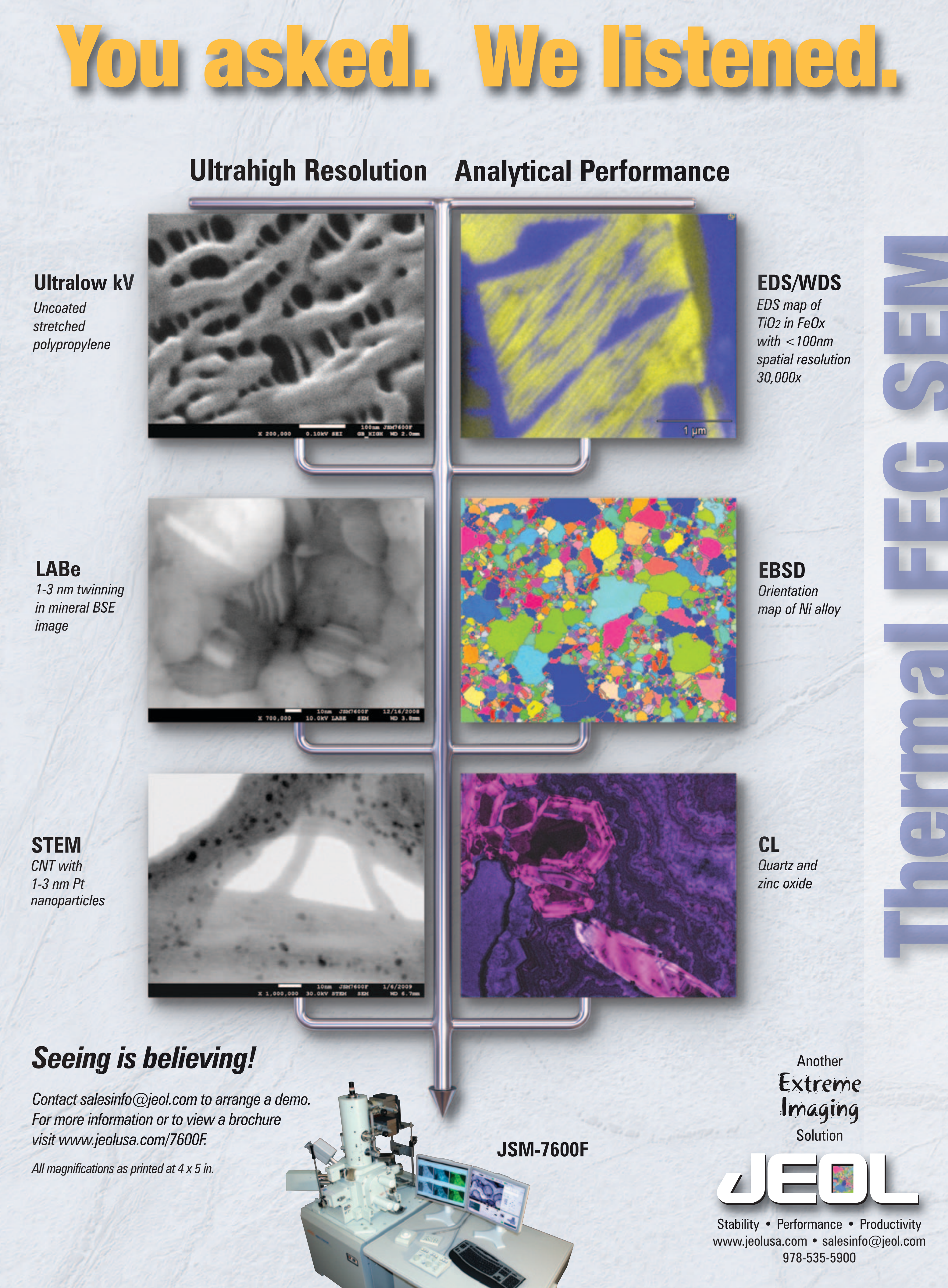
2. Applications

2.0 Cross-sectional Imaging with the SEM

Perhaps one of the most common applications of the multibeam instrument is the cross-sectional imaging of samples. The sample is tilted towards the FIB column such that the ion beam is normally incident on the sample surface. An area is milled out, typically 20-30 µm in width and 10-15 µm in length. A fairly high Ga^+ ion beam current (typically greater than 1 nA) is initially used to rapidly mill the area to a depth of 5-10 µm, and then a finer beam with a much smaller beam current (500 pA or less) is used to polish one face of the cross-section. The polished face can then be imaged directly with the SEM, or the sample can be tilted back to normal incidence with the SEM column and imaged with the FIB (section 2.2). If the very top surface layer is of particular interest, then the gas injection systems can be used to deposit a "thick" layer (~0.5 – 1 µm) of C or W to act as a sacrificial surface to protect the very top surface of the sample from ion beam damage. Figure 2 shows SEM images of a typical FIB cross-section through a portion of a novel solar cell architecture based on a dense array of nanostructures with an FIB-deposited C protective layer. Such images can be rapidly obtained in less than an hour and used to determine layer conformity and thicknesses at the top, bottom and sides of the vertical nanostuctures.

2.1 FIB Tomography

The images shown in Figure 2, however, only yield a small snapshot of the total structure. To ensure that such images are typical of the entire solar cell array, the serial sectioning and imaging capability of the dual beam FIB can be used to survey in an automated fashion a much larger region of the device. In this process, the ion beam progressively slices into the cross-sectional face at 40-60 nm intervals, and SEM images of the freshly exposed cross-section are imaged automatically with


You asked. We listened.
Ultrahigh Resolution
Analytical Performance
Ultralow kV
Uncoated stretched polypropylene
EDS/WDS
EDS map of TiO_2 in FeOx with <100nm spatial resolution 30,000x
1 µm
LABe
1-3 nm twinning in mineral BSE image
EBSD
Orientation map of Ni alloy
STEM
CNT with 1-3 nm Pt nanoparticles
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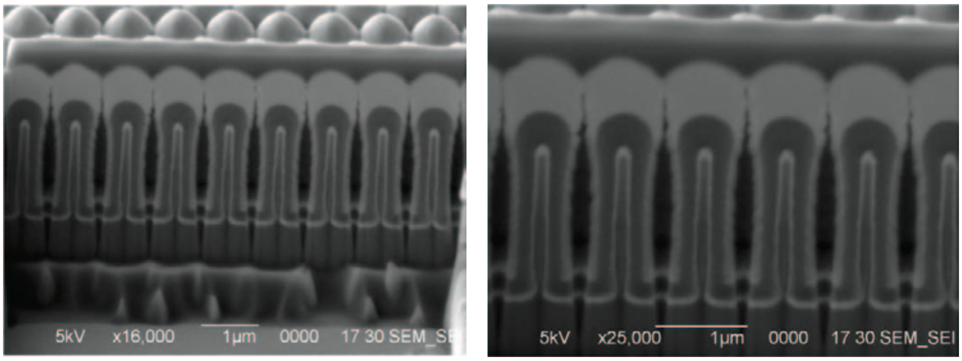

Figure 2: SEM images of FIB cross-section through a nanostructured photovoltaic architecture.

the SEM. The operator can dictate the thickness of each slice by varying the number of sections through a given thickness. The result in this case is a stack of 200-400 images through 10-30 rows of the vertical nanostructures which can be converted to a video sequence or used to create a volume rendering of the analyzed region as shown in Figure 3.

2.2 Cross-sectional Imaging with the FIB

Secondary electron images may also be produced using the focused ion beam as the imaging probe. In fact, the secondary electron yield is higher using the FIB, and thus images generally show a much better signal-to-noise ratio. However, any time the ion beam is used to image a sample, one must consider that the sample is being constantly sputtered and implanted with Ga ions. An advantage, however, is that imaging with the FIB gives very strong ion channeling contrast in the secondary electron images, as demonstrated by grain orientation contrast in the section through microelectronic wire bond shown in Figure 4.

2.3 TEM Sample Preparation

The idea of milling out an area of a sample and polishing the cross-section can be extended to include milling out the opposite side and thinning from both directions to produce a TEM sample. Here, FIB instruments have a huge advantage over conventional TEM sample preparation methodologies because site-specific TEM samples can be produced. Furthermore, they can be produced from almost any type of material, so problems associated with differential Ar^+ ion beam thinning rates are avoided. TEM sample preparation is facilitated even further in the Multibeam instrument by allowing one to observe with the SEM the thinning process being performed by the FIB as it progresses. This helps prevent accidental milling through the TEM sample as it approaches electron transparency (~100 nm in thickness) and minimizes damage to the sample from the energetic Ga^+ ions. Two commonly used methodologies exist for extracting TEM samples from the dual beam instrument. The first is to use a micromanipulator to lift the cut-out TEM sample from the mill trench and transport it to a formvar-coated TEM grid using simple electrostatic forces between the tip and the sample (Figure 5). This is the quickest way, but it is somewhat wrought with peril as occasionally static discharge between the sample and probe tip results in loss of sample. A disadvantage of this methodology is that if the sample is found in the TEM to be too thick, there is nothing that can be done, and a new sample must be made from the beginning. The second method for extracting TEM samples from the dual beam instrument is to weld the sample with either C or W using the gas injection systems to the micromanipulator tip while the sample is still fairly thick, free it from the bulk, and then attach it (again using the gas injection capability) to a slot grid, from which the top third is removed (see inset, Figure 6). Alternatively, special grids can be purchased for this purpose. After it has been attached, the sample can be thinned to electron transparency (Figure 6). Although there are more steps involved

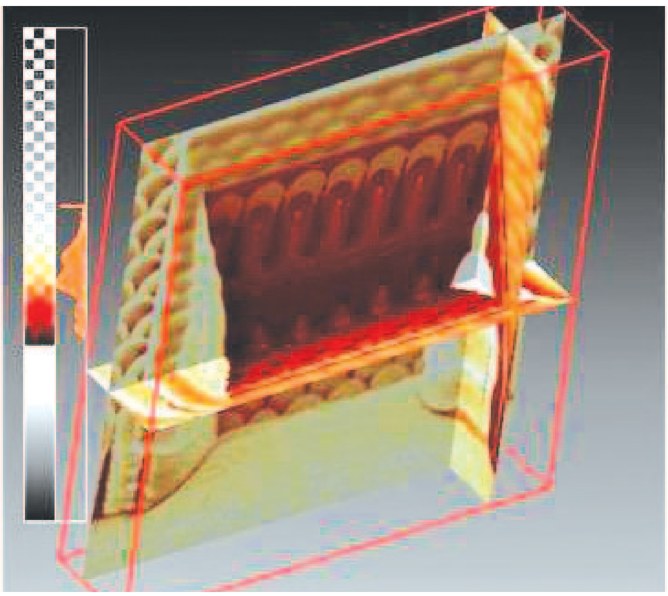

Figure 3: 3-D representation of photovoltaic vertical nanostructures obtained by serial sectioning and simultaneous imaging in the Multibeam instrument.

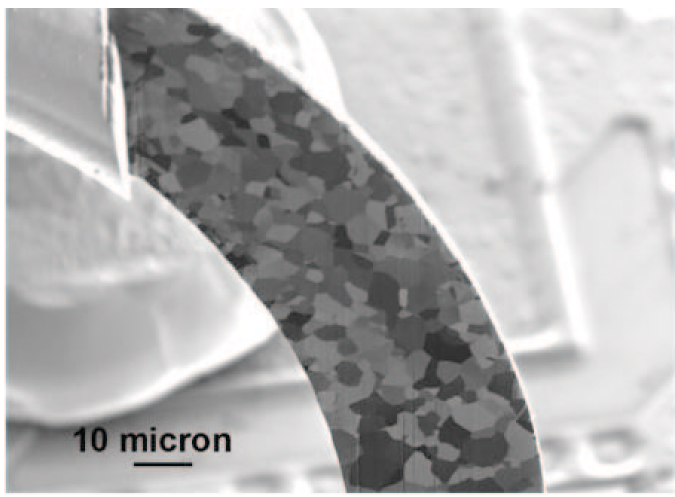

Figure 4: FIB secondary electron image from FIB cross-section through a section of microelectronic wire bond showing strong ion channeling (grain orientation) contrast.

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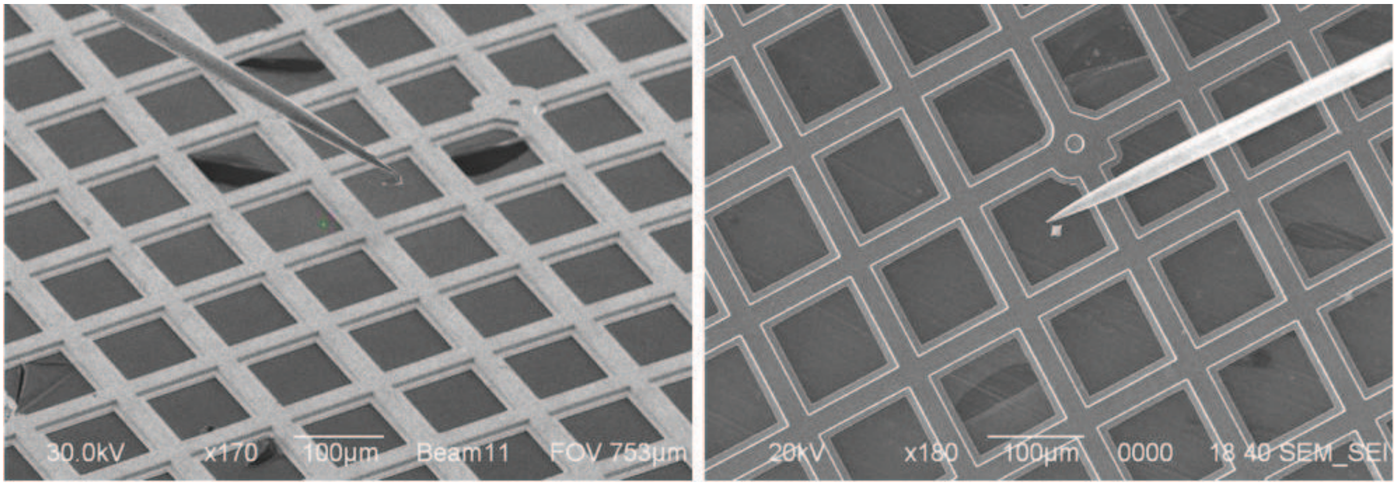

Figure 5: Lift-out and placement of TEM sample on formvar-coated Cu grid, using only electrostatic forces.

in this process, it does have the advantage that the sample will not be lost due to static discharge, and the sample can be re-thinned by FIB if it is found to be too thick in the TEM.

2.4 Nanofabrication with Gas Injection Systems

The deposition processes using the gas injections systems are much more useful than simply providing sacrificial surface layers for welding TEM samples to micromanipulator tips and TEM grids. They are also frequently used to build or modify nanostructures. Practically, an area is selected within the microscope field of view, defining the location of the deposition. A nozzle is brought to close proximity of the surface, and a valve is opened to begin the flow of precursor gas (phenanthrene for C deposits, tungsten hexacarbonyl for W deposits) surrounding the defined area. The beam is raster scanned within the defined area to dissociate the adsorbed precursor gas molecules, with the volatiles being pumped away and a deposit left behind. Either the electron beam or the ion beam can be used, but ion beam induced deposition is a much faster process, albeit the deposits will contain implanted Ga from the primary ion beam. Thus, the milling and deposition capabilities of the multibeam instrument can be used, for example, to build prototype biological sensors consisting of a FIB-deposited C nanorod placed on a FIB-milled AFM cantilever tip (Figure 7). Tungsten depositions are especially useful, owing to their electrical conductivity. Thus, they are commonly used in the microelectronic industry for semiconductor device modification. Manufacturers can modify new chip designs by severing and adding electrical connections to make prototypes without the necessity of the time-consuming and expensive process of mask design and fabrication. In addition, it has been found that the W deposits are superconducting at cryogenic temperatures [3-4] (Figure 8), which may eventually lead to a host of new applications.

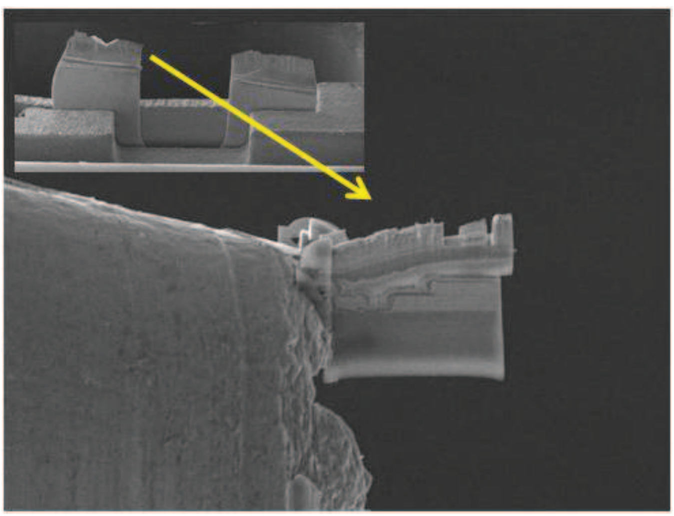

Figure 6: Thick TEM specimen attached to the side of modified slot grid (inset) ready for final thinning (and re-thinning if necessary).

2.5 *In-situ* Electrical Characterization at the Nanometer Scale

The micromanipulator tips used to transfer TEM specimens to the grids are also themselves used to conduct electrical measurements at the nanometer scale. It is possible to place (at least) four of these inside the chamber of the JEOL JIB-4500 (Figure 9). Special low-noise triaxial connections are made to a source meter, which allows one to sweep, step, or pulse current and measure voltage or vice versa. Thus, the electrical properties of FIB deposits can be measured *in-situ*. These measurements can

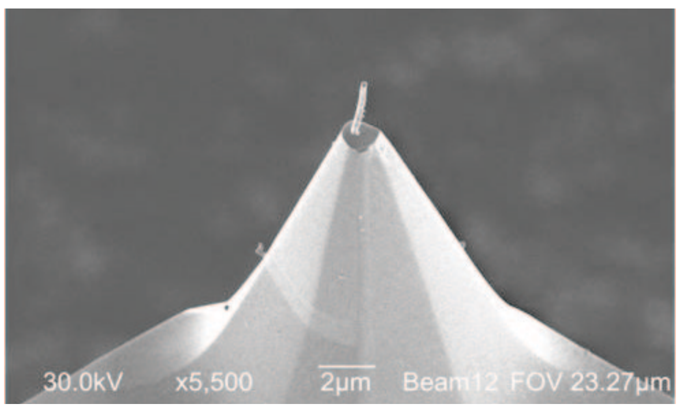

Figure 7: Prototype biological sensor consisting of a FIB-deposited C nanorod deposited on top of a FIB-milled AFM cantilever tip.

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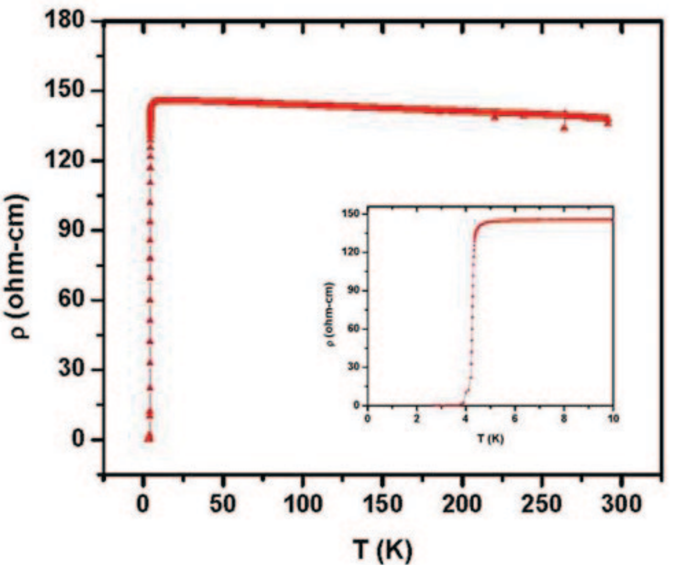

Figure 8: Graph of resistivity versus temperature of FIB-deposited W. The rapid decrease in resistivity shows the onset of superconductivity at approximately 5K (inset).

also be taken on nanostructures, including measurements from a single thin film coated vertical carbon nanotube (Figure 9).

A final, fun example of the capabilities of the JEOL JIB-4500 is the ability to load bitmap images into the operating software to mill or deposit more complicated structures. Although there are many scientific applications of this capability, the impact it has on generating enthusiasm towards nanotechnology within elementary, high school, and undergraduate students by using it for fun applications (Figure 10) cannot be underestimated.

3. Conclusions

In conclusion, dual beam instruments, such as the JEOL JIB-4500, have a wealth of applications in the life and physical sciences. The instruments, with numerous ports available for accessories, are highly customizable and can be configured for each laboratory's specific need. The ability of the instrument to *in-situ* image, analyze, add, and remove material at the nanometer scale makes it one of the most powerful tools in the suite of microbeam instruments available to scientists today. Huygens and Newton would have been very proud. MT

Figure 10: Negative FIB carbon deposit of the Boston College logo.

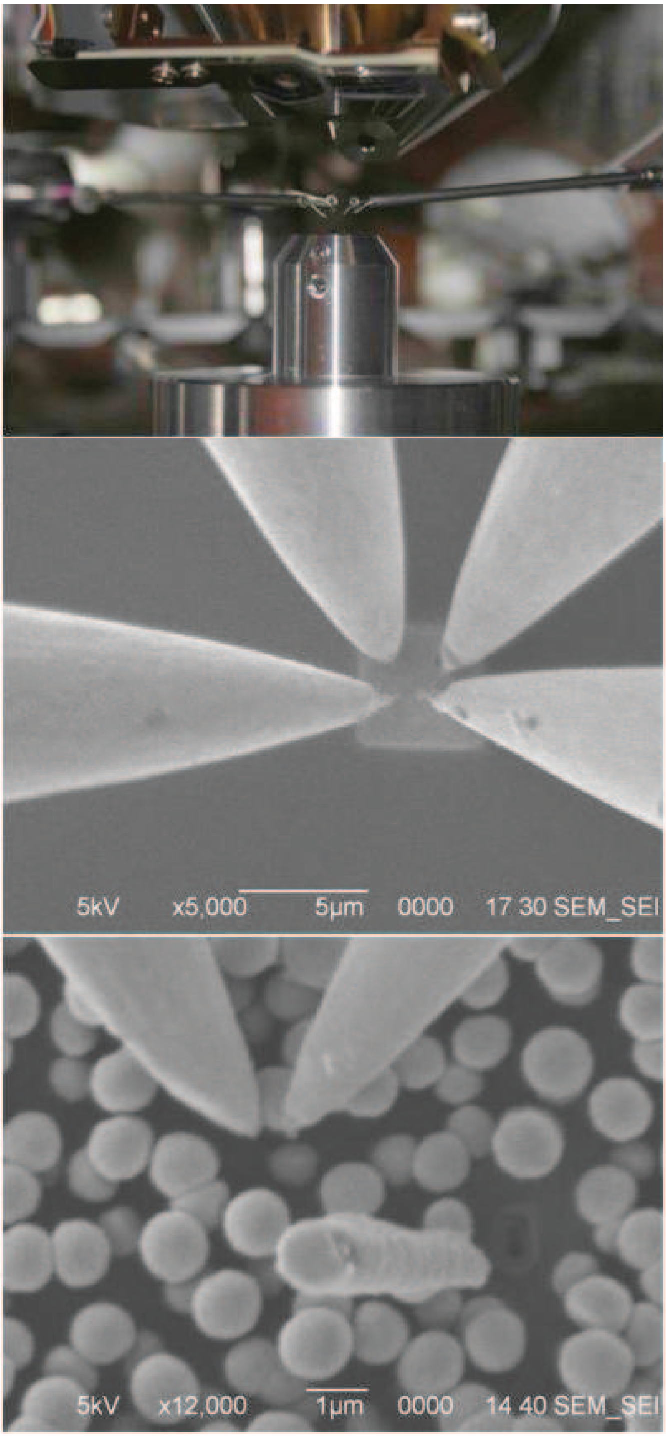

Figure 9: (Top) Four micromanipulator probes inside the JEOL JIB-4500 Multibeam instrument above the sample holder. (Middle) Four probes in place on a FIB tungsten deposit for a four-point probe measurement. (Bottom) Two probes contacting a single thin film coated carbon nanotube for electrical measurement.

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