Advanced Particle Beam Technologies for Nano Characterization and Fabrication

J.J.L. Mulders*, J. Greiser**

*FEI Electron Optics, Building AAE, Achtseweg Noord 5
PO Box 80066, 5600 KA Eindhoven, The Netherlands, jjm@nl.feico.com
**FEI Electron Optics, Building AAE, Achtseweg Noord 5
PO Box 80066, 5600 KA Eindhoven, The Netherlands, jgreiser@nl.feico.com

ABSTRACT

Nanotechnology developments focus on materials and life science functionality at the very small scale, created by the local structure and interactions of the materials involved. Essential for understanding this local functionality is the visualization and the material-analysis of the nano-structures. In particular the 3-dimensional information at the nano and micro scale is an important way to characterize the material. This can be achieved by using high-resolution TEM tomography and by 3D slice and view applications of a DualBeam system, where its FIB column is used for milling and its SEM column for viewing and analysis. The DualBeam tool can also be used for the actual fabrication of nano scale structures by patterned ion beam milling, electron and ion beam induced depositions and electron beam lithography. The deposition technique offers direct deposition by control of a materials growth process from a gas phase and offers both high resolution and control of the growth in the third dimension.

Keywords: FIB, SEM, TEM, nano-charcterization, nano-scale production

1 INTRODUCTION

Since many years Transmission Electron Microscopes (TEM) and Scanning Electron Microscopes (SEM) have been used for high-resolution imaging and analysis of prepared, thin samples and of unprepared bulk samples respectively. Resolution improvements, low-dose techniques, adaptations to the sample's environment and addition of analytical capabilities have been recent changes for those instruments. As a result sample preparation for SEM has been minimized, the imaging capability for TEM has recently been extended down to the sub 1 Ångström level, and the availability of FIB capability has resulted in an new instrument where both the SEM and the FIB beam can be used in a combined way, allowing the full split between milling for sample modification on the one hand and non-destructive imaging and analysis on the other hand. Although many of the changes have been made due to the needs in the semi-conductor industry, they are generally applicable and fit very well into today's and near-future demands of research for nano technology. An interesting aspect of nano technology is the need for a multi-discipline approach having aspects of chemistry and physics and biology in addition to electronics that allows implementation of system control and measurement at the very small scale. An example can be found in the recent development of sensors with the direct aim to combine a minimum amount of material, to be analyzed with a maximum amount of accuracy or - in short - a higher specificity combined with a higher sensitivity. These objectives can only be achieved by manipulating material and building measurement systems at the nano scale.

2 LIFE-SCIENCE

2.1 Structural information

Over many years the structures in life-science have been studied with high resolution TEM, for a long time being the only technique allowing to visualize the high details of cell-based structures and mechanisms. As life-science materials are aqueous-based structures and electron microscopy is a high vacuum technique, sample preparation has been a key, for the application of the TEM on life-science materials. In addition, at very high magnification the electron dose applied to the sample is so large, that the structures can be destroyed by the imaging and analysis, and therefore special precautions have to be taken.

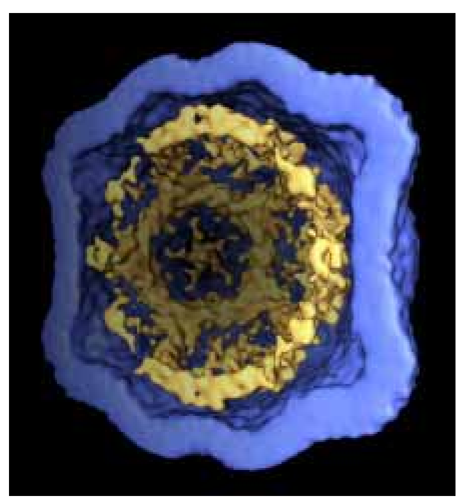

Figure 1: Cowpea mosaic virus: image obtained at low dose and at liquid He temperature

In practice this has been realized by keeping the sample at very low temperature (liquid Helium) with the electron density generally below 10 electrons / Å^2. An example of an image made under these conditions where the structure remains in-tact is given in figure 1.

As structures represent 3 dimensional information, the traditional 2D projection image form a TEM has been expanded to muti-angle recordings of digital images (typically 140), that are processed by Software to obtain a real, 3 Dimensional reconstruction of a volume of interest. Automation of this procedure on the TEM has allowed this technique to be applied routinely. In this way structures can be positioned in space and this helps for a better understanding of their actual function. An example of such a 3D reconstruction is given in Figure2.

Figure 2 [1]: Visualization of small magnetite crystals in magnetotactic bacteria. This image is a part of a 3D dataset with the reconstructed volume, allowing any viewing angle.

2.2 Imaging in the natural state

The SEM technique has expanded to view wet samples in their natural state by adapting the vacuum conditions, to allow saturated water vapor in the chamber creating an equilibrium between the sample and its environment and hence preventing the sample from drying out.

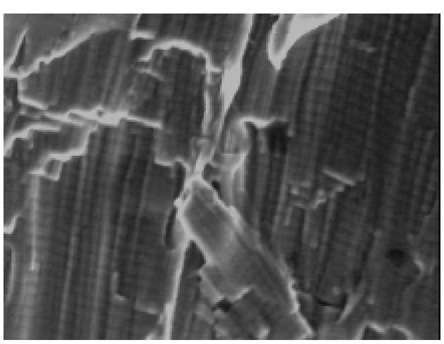

Figure 3: Muscle tissue in fully hydrated, natural state

Corresponding imaging techniques using dedicated detectors still allow the imaging of the sample surface under these conditions, using secondary electrons, hence showing topography. This technique is referred to as ESEM and has been implemented in most of today's FEI SEM instrumentation. An example of imaging under these sample environment conditions is given in figure 3.

2.3 Opening the 3D dimension of a bulk sample such as tissue or single particle

In addition to natural direct imaging using the ESEM technique, another way of preserving water containing samples is by quickly freezing them and, while frozen at a very low temperature of < 130 ^{o}C, create a fracture to open up the sample and view the inside of it. This is technique is known as cryo-SEM and it is applied to many life-science samples, but also to related materials, such as food and cosmetic products. An example of this is given in figure 4.

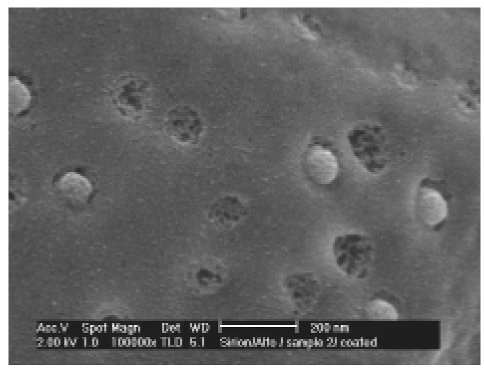

Figure 4: Cell membrane structure, generated by cryo fracture of the plunge frozen sample

By applying a FIB column from a DualBeam instrument, for the creation of a cross-section, it is even possible to cut through a very specific region of interest and hence reveal its internal structure, precisely at the area of interest. An example of a cut through a single Lacto Bacillus bacterium is given in Figure 5.

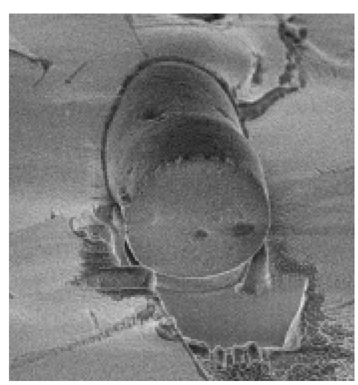

Figure 5: FIB cross-section through a selected site of a single, flash-frozen bacterium

3 MATERIAL SCIENCE

The electron and ion beam based techniques offer suitable analytical capabilities for materials at the micro and nano scale. Not only topographical information but also elemental distributions, crystallographic information and even magnetic information can be obtained with these instruments. In addition to 2 dimensional distributions, are replaced by three-dimensional information for a more fundamental description of the material.

3.1 Structural information

The latest developments in TEM have resulted in a break-through in resolution, where the 1 Ångström information limit has been exceeded. Modern electron optic lens design, correcting for higher order aberrations in combination with low beam energy spread, have allowed to pass this barrier and as a consequence atomic stacking in crystals can be shown with excellent clarity. Grain borders and dislocations can now be studied in more detail and with low uncertainty. An example of this achievement is given in figure 6, where the technique is applied to show the grain boundary of a gold crystal.

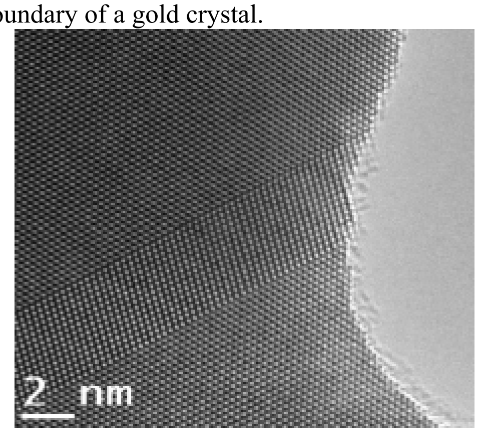

Figure 6: Image of a gold crystal showing the edge and the very clear separation on the twin grain boundary. Atomic structures shown at the boundary (some Moiree effect is introduced by the .pdf conversion).

3.2 Three dimensional image and analysis

The FIB capability to mill away material can be expanded to successive slicing in the material and subsequent imaging of the newly revealed surface. This automated process, referred to as slice an view will then allow the creation of a three dimensional volume of the internal structure of the material. This be done with images, but also with analysis data of the material and hence a 3D data set of a volume is created. For example, a materials crystallography can be determined using EBSD mapping on each, newly revealed slice, create by FIB. In this way a three dimensional crystallographic volume is visualized, and available for statistical analysis of the voxels.

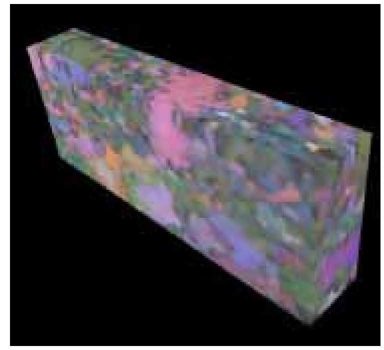

Figure 7: DualBeam 3D analysis of a 3 x 1 um volume of steel. Each color represents an orientation of a steel grain. Data suited for statistical crystallographic analysis.

4 CREATION OF STRUCTURES

Electron and ion beam equipment cannot only be applied for imaging and analysis at nano and micro-scale, but they can also be used to create structures at this level.

4.1 Pattern transfer with FIB

As the focused ion beam has enough energy and momentum transfer to remove material, by adding a patterning capability, it is possible to remove material in a very well controlled way and with a controllable depth. This process can be purified and / or made more efficient, but the local addition of etching gases close to the sample. Accuracy of this process and its material independency have allowed to apply this technique over a wide range of materials. Patterning can be applied to simple, repetitive structures or to more complex patterns such as controlled by a color-coded bit map, where intensities and pixels are translated into dwell times and actual beam positions. The technique can be applied for example in the creation process of a photonic array, where the regularity and the small size of holes milled in the material, are crucial for the final functionality.

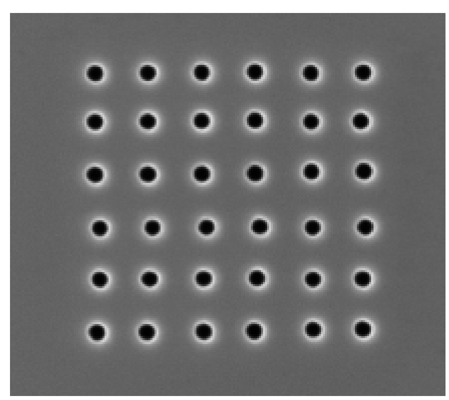

Figure 8: Matrix of 150 nm holes in InP, directly created by milling with the ion beam.

4.2 TEM sample preparation

As the ion beam can be so well controlled, it is also ideal for generating a very small, thin slice of material - thin enough to make it electron transparent and this at a location that is of interest to the researcher. In other words, the ion beam can be applied to create a site-specific TEM lamella that can be studied in a high resolution TEM. Because the ion beam milling process is quite independent of the material type, it is ideal to make TEM samples of interfaces between hard and soft material, such as for metal / polymer interfaces. The process is highly automated, accurate and fast compared to conventional TEM sample preparation techniques, that are not site specific and may suffer from a mix-up of the materials during the preparation and hence destruction of the actual interface of interest.

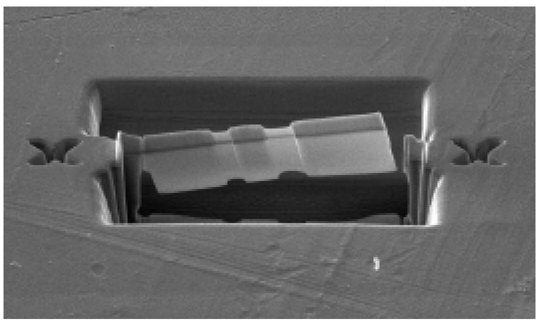

Figure 9: Ion beam created TEM sample in a coated polymer, ready for lift-out and transfer to the TEM

With this technique it now becomes possible to generate many TEM samples from a small area of the bulk sample, and hence ensure statistical validity of the analysis.

4.3 Patterning with the electron beam

Electron beam lithography using the electron beam from the SEM is a well-established technique to transfer a (CAD) pattern into resist and later into for example in a metal film. Also the electron column from a DualBeam is suited to do this and structures down to 20 nm can easily be created this way. Of course, resist processing is an essential part of this technique but there are many capabilities using high resolution or duo-layer resist that is thin enough to create very small structures. The stability of the FEG based equipment allows exposure over a longer period of time and using back-ground patterned (gold) structures, multi-field stitching and layer to layer alignment become possible as well.

4.4 Direct deposition techniques

With the ion beam or the electron beam it is possible to decompose a gaseous precursor that is supplied close to the beam impact area. This technique referred to as Electron Beam (or Ion Beam) Induced deposition allows the creation of very small structures by patterning and direct deposition of for example a metal, that is released from the gaseous organo-metallic vapor. Indeed small structures can be created with this as is shown in figure 10 and 11.

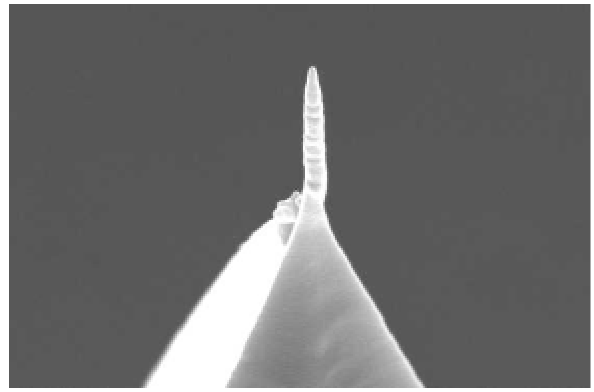

Figure 10: A 30 nm diameter Cobalt tip grown with electron beam induced deposition, on top of a diamond needle, to create a magnetic tip with very small apex radius for high resolution MFM measurements.

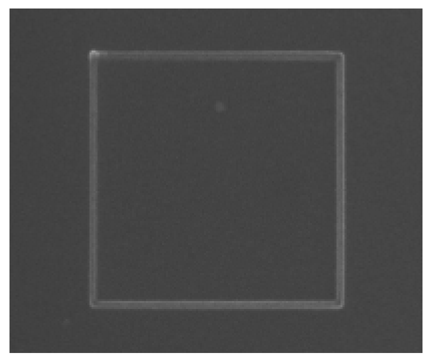

Figure 11: Rectangular frame of directly deposited gold. The frame is 500 nm wide. Line width and height 15 nm

5 ENABLING TECHNOLOGY

The capabilities of modern TEM, SEM, FIB and DualBeam instrumentation is still expanding and focus is given on both technological improvements, but clearly also on making this new technology accessible to the user by very extensive software control still condensed into an easy-to-use graphical user interface. Current limits will be pushed, new ideas implemented and working together with researchers in nano technology will help to implement the most relevant new functions in a user friendly way. In this way FEI's technology is now and will stay an enabling technology for the researchers in nano technology.

IMAGE COURTESEY

[1] M. Weyland, R. Dunin-Borkowsk, P. Midgley - Cambridge University and M. Otten, FEI Company