A practical approach to test the scope of FIB-SEM 3D reconstruction

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Abstract. State-of-the-art focused ion beam (FIB) instruments have an ion column for sample modification and an electron column for scanning electron microscopy (SEM). 3D reconstruction of a sample volume can be achieved by serial sectioning using the FIB in combination with high-resolution SEM imaging of each cross-section. Usually, the resolution in the direction in which the sections are milled (z-direction) is much lower than in the plane of the cross-section (xy-direction) itself. Increased sampling in the z-direction can only be achieved by decreasing the distance between single sections. For a constant volume this is equivalent to increasing the number of sections, i.e. time and effort. To perform efficient 3D reconstructions the effect of the reduced sampling in the z-direction to the overall accuracy of the 3D reconstruction has to be known. We tested this approach with FIB conical test structures that were slice-and-view processed and subsequently reconstructed. Using a reference data set with a slice thickness (z-resolution) of 22 nm, data with z-resolutions ranging from 44 nm to 440 nm were created and reconstructed with commercial software. The calculated volumes for the simulated z-resolutions and their deviations from the reference volume are shown. Deviations of up to 35% occur and reach about 10% once the z-resolution was one fifth of the upper diameter of the conical structures.

1. Introduction
3D reconstructions by serial sectioning with focused ion beam (FIB) instruments, introduced some years ago [1-3], is now a standard feature or option of most available FIB-SEM instruments. Using the SEM for imaging, instead of the ion beam, allows non-destructive quantitative analysis of features smaller than 100 nm [3]. However, the process of serial sectioning with FIB is used not only for volumetric reconstruction of the microstructure, but is also the basis for quantitative 3D analytical methods, such as 3D electron backscatter diffraction (EBSD) [4] and 3D energy dispersive X-ray spectroscopy (EDS) [5] and a combination thereof [6]. In this paper we test the ability of serial FIB sectioning, using standard features of the FIB-SEM instrument, to enable efficient 3D reconstructions using SEM image data. The resolution in the direction in which the sections are milled (z-direction) is usually much lower than that in the plane of the cross-section (xy-direction) and so it is helpful to have an estimate of the effect of z-resolution on the overall accuracy of the 3D reconstruction, e.g. for reducing FIB time and effort. If the size of a structure of interest is known approximately, this information could be used to estimate the FIB parameters of an experimental setup for 3D reconstruction.

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2. Methods
A Helios NanoLab 600 (FEI Company, Hillsboro, USA) FIB-SEM was used to fabricate and section the microstructures for the 3D reconstruction test. 3D reconstruction was performed with ResolveRT 4.0.1 (Visage Imaging GmbH, Berlin, Germany).

2.1. Test structure fabrication
Conical structures with diameters of 1000 nm and 500 nm were generated on a silicon wafer sample by focused ion beam milling an area of approx. 7 µm x 6 µm (figure 1). The nominal milling depth was 1000 nm but the actual depth measured was 2700 nm (figure 2). Subsequently, the cavities were exposed to gas-assisted platinum deposition and manually observed until completely covered. We chose platinum, because it provides high contrast with respect to the silicon in the SEM secondary electron (SE) images, which makes it easier to threshold the data and perform a semi-automatic segmentation of the test features.

![Figure 1. SEM SE image of the test structure: five large circular cavities with a diameter of 1000 nm and nine small cavities with a diameter of 500 nm.](image1)

![Figure 2. SEM SE image of FIB cross-section number 37 out of a total of 316 sections.](image2)

2.2. Serial FIB tomography
In total, 316 serial FIB cross-sections were made and imaged in SEM secondary electron (SE) mode at a sample tilt of 52 degrees. SEM images were recorded at 10 keV beam energy, a probe current of 0.17 nA and with a pixel size of 10.4 nm. After tilt correction, the effective pixel size in the y-direction (slow scan direction) was calculated to be 13.2 nm. Each slice that had been milled to create the serial sections had a thickness of 22 nm, determined by the spot size of the ion beam, the relative beam overlap at 30kV ion energy and the beam aperture chosen to give 0.92 nA ion current. Here, the thickness of a single slice is defined as the z-resolution.

2.3. 3D reconstruction with different virtual slice thicknesses
With the SEM image data imported into ResolveRT, the images were aligned manually so that in the reconstructed coordinate system the slicing direction became normal to the image plane. To simulate different z-resolutions, further 3D reconstructions were made with down-sampled data sets using only every second, third, fourth (and so on) SEM image (see table 1). Segmentation was performed with the wand tool and a lower threshold of 78 (black) and an upper threshold of 248 (white).
3. Results

3.1. Volume reconstruction

Table 1 shows the calculated average volumes and standard deviations of the five large and nine small cavities at various z-resolutions, ranging from 22 nm to 440 nm. At the highest z-resolution the average volume of the larger structures is 1.253 µm$^3$ and 0.276 µm$^3$ for the smaller structures. A visualization of two of the 3D reconstructions can be seen in figures 3 and 4, using z-resolutions of 22 nm and 330 nm, respectively.

![Figure 3](image1)

Figure 3. Reconstructed cavity volumes of the test structures (316 serial FIB cross-sections, i.e. 22 nm per slice).

![Figure 4](image2)

Figure 4. Reconstructed cavity volumes of the test structures (21 serial FIB cross-sections, i.e. 330 nm per slice).

Geometric distortions of the 3D reconstruction of the large cavities are already evident at a virtual z-resolution of 110 nm, and very strong geometrical errors are present at a z-resolution of 176 nm and lower. These values correspond to a z-resolution to diameter ratio of 0.11 and 0.18, respectively. At the small cavities, distortions start appearing at a z-resolution of 66 nm, and strong errors occur at 110 nm or lower. These values correspond to a z-resolution to diameter ratio of 0.13 and 0.22, respectively.

Table 1. The reconstructed average volumes and standard deviations of the five large and nine small cavities according to the chosen z-resolution.

| Slice thickness (nm) | Average volume large structures (µm$^3$) | Standard deviation volume large structures (µm$^3$) | Average volume small structures (µm$^3$) | Standard deviation volume small structures (µm$^3$) |
|---------------------|----------------------------------------|---------------------------------|---------------------------------------|---------------------------------|
| 22                  | 1.253                                  | 0.206                           | 0.276                                 | 0.039                           |
| 44                  | 1.282                                  | 0.214                           | 0.286                                 | 0.041                           |
| 66                  | 1.272                                  | 0.189                           | 0.284                                 | 0.041                           |
| 88                  | 1.259                                  | 0.169                           | 0.287                                 | 0.047                           |
| 110                 | 1.353                                  | 0.236                           | 0.310                                 | 0.046                           |
| 132                 | 1.310                                  | 0.207                           | 0.296                                 | 0.045                           |
| 176                 | 1.366                                  | 0.273                           | 0.294                                 | 0.044                           |
| 220                 | 1.405                                  | 0.276                           | 0.331                                 | 0.061                           |
| 264                 | 1.370                                  | 0.223                           | 0.310                                 | 0.050                           |
| 330                 | 1.481                                  | 0.392                           | 0.324                                 | 0.060                           |
| 440                 | 1.783                                  | 0.303                           | 0.284                                 | 0.015                           |

3.2. Deviation in reconstructed cavity volumes with respect to z-resolution

In general the results shown in table 1 indicate that the reconstructed average volumes for both types of structure in this test tend to become larger for lower z-resolutions. The deviations of the average
volume of the cavities reconstructed at the various z-resolutions with respect to the reconstructed reference volume at highest z-resolution oscillate strongly, both for the large and the small cavities (see table 2). Deviations from the reference volume larger than 10% occur once the z-resolution is lower than approximately one fifth of the diameter of the test structures, i.e. at a z-resolution of 220 nm for the larger and 110 nm for the smaller cavities. Whereas the maximum deviation from the reference volume is at the same magnitude for both kinds of structures, the minimum volume deviation remains below 10% at all z-resolutions, except for the case of the large structures at 440 nm. The low deviation for the small structures at that resolution cannot be explained at present.

Table 2. Deviation of the average, maximum and minimum volumes of the reconstructed test structures at various z-resolutions with respect to the reconstructed reference volume using all 316 serial FIB cross-sections.

| Slice thickness (nm) | Average volume deviation of large structures (%) | Max./min. volume deviation of large structures (%) | Average volume deviation of small structures (%) | Max./min. volume deviation of small structures (%) |
|---------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|-----------------------------------------------|
| 44                  | 2.25                                          | 3.18/1.69                                     | 3.50                                          | 5.68/1.62                                     |
| 66                  | 1.88                                          | 4.00/0.21                                     | 3.28                                          | 8.10/0.51                                     |
| 88                  | 5.44                                          | 6.92/2.56                                     | 8.11                                          | 13.68/4.33                                    |
| 110                 | 7.79                                          | 9.82/5.87                                     | 12.25                                         | 16.76/7.83                                    |
| 132                 | 4.64                                          | 5.78/3.28                                     | 7.06                                          | 12.11/0.47                                    |
| 176                 | 8.47                                          | 16.59/2.69                                    | 6.44                                          | 9.58/0.24                                     |
| 220                 | 11.61                                         | 15.63/6.42                                    | 19.99                                         | 28.30/3.47                                    |
| 264                 | 9.58                                          | 11.52/7.73                                    | 15.40                                         | 29.60/4.53                                    |
| 330                 | 16.86                                         | 33.90/2.09                                    | 17.43                                         | 27.90/5.58                                    |
| 440                 | 34.06                                         | 45.69/20.70                                   | 1.31                                          | 2.37/0.72                                     |

4. Conclusions

Deviations with respect to the reference volume larger than 10% were observed at a z-resolution of approximately one fifth of the diameter of the test structures. However, qualitative analysis of the 3D reconstruction shows visible geometric distortions already at a z-resolution of one tenth of the diameter of the test structures. The oscillations of the deviations itself could be due to the experimental setup of this test or due to the interpolation mechanism of the ResolveRT software. Therefore, the volume reconstruction seems only to be an indicator of the quality of the 3D reconstruction of the FIB cross-sections. Additionally, the geometric similarity of the reconstructions could be taken into account to quantify what can be seen as geometric distortions in the 3D reconstruction at lower z-resolution [7].

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References

[1] Sakamoto T, Cheng Z, Takahashi M, Owari M and Nihei Y 1998 Jpn. J. Appl. Phys. 37 2051
[2] Inkson B J, Mulvihill M and Móbus G 2001 Scripta Mat. 45 753
[3] Holzer L, Indutny F, Gasser P, Münch B and Wegmann M 2004 J. Microsc. 216 Pt1 84
[4] Groeber M A, Haley B K, Uchic M D, Dimiduk D M and Ghosh S 2006 Materials Characterization 57 259
[5] Kotula P G, Keenan M R and Michael J R 2006 Micros. Microanal. 12 36
[6] West G D and Thomson R C 2009 J. Microsc. 233 Pt3 442
[7] Maple C and Wang Y 2004 8th Int. Conf. on Information Visualisation (IV’04), 2004 363-369