Electron tomography of nanostructures in the SEM

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This paper describes the implementation of electron tomography in the scanning electron microscope, and discusses the potential of this recently proposed 3-D imaging technique [1] [2].

Nowadays, the shape of the specimen is intuitively interpreted from a single image providing sufficient depth-of-field or from a combination of images acquired at different conditions, where the contrast features of the specimen details suggest their disposition in space. The introduction of the focused-ion beam allows one to visualize sub-surface structures and to perform a three-dimensional reconstruction through the slice-and-view method. Under the assumption that the slicing advances homogeneously and exposing a flat surface, the inner structure of the sample is revealed by contrast variations arising exclusively from the local variations in the specimen composition. These variations are either intrinsic to the specimen or have been introduced by the preliminary preparation, as is the usual case of biological samples. Sample preparation, progressive slicing, as well as the detection strategy used for image acquisition are the key elements in determining the resolution and the significance of the three-dimensional reconstruction.

Electron tomography is among the most promising and rapidly developing techniques for 3-D reconstruction as it combines a reliable reconstruction algorithm with the signal corresponding to incoherently scattered electrons in the Scanning-Transmission (STEM) imaging mode [3][4]. The STEM is already implemented not only in the Transmission Electron Microscope (TEM) but also in the Scanning Electron Microscope (SEM), where it takes advantage from some peculiar characteristics of the experimental set-up [5]. The STEM-in-SEM approach attains nanometric resolution and is free from aberrations caused by post-specimen imaging lenses; in addition it is possible to collect transmitted electrons over a wide angular range [6][7].

The optimization of detector design and performance, together with the formulation of a tailored detection strategy, make the contrast comply with local variations of composition or projected thickness. The bright-field component of the transmitted electrons can be effectively separated from the dark-field one, by varying the specimen-detector distance and the collection conditions of the STEM detector [4].

The capability of the STEM-in-SEM imaging mode to preserve the monotonic variation of the signal with specimen thickness meets the key requirement for tomographic reconstruction, and thus opens up the perspective for the 3-D reconstruction of nanosized samples, such as nanowires or carbon based nanostructures. In addition, the large value for the maximum detection angle ensures a complete detection of the scattered electrons, even in case of relatively large specimen thickness. In the case of tomography, these features are essential to maintain the proper image contrast when the specimen is rotated through the tilt series.

Fig. 1 (left) shows the secondary-electrons image of a ZnO crystalline nanostructure supported by a standard holey-C copper grid. This comb-like structure exhibits a number of parallel nanowires with uniform section (average width 176 ± 15 nm) and tapered termination. Two STEM images from the tilt series of the ZnO nanowires are also shown in Fig.1. The tilt series of 16-bit 1024x768 images was obtained by rotating the sample over a 110° range, with 1° step. ImageJ [8] with the TomoJ plug-in allowed obtaining the tomographic reconstruction [9].

The perspective view of the reconstructed volume is shown in Fig. 2. The main features of the samples are properly retrieved such as the disposition of the wires, their uniform section and the tapered termination, therefore demonstrating the potential of the method.

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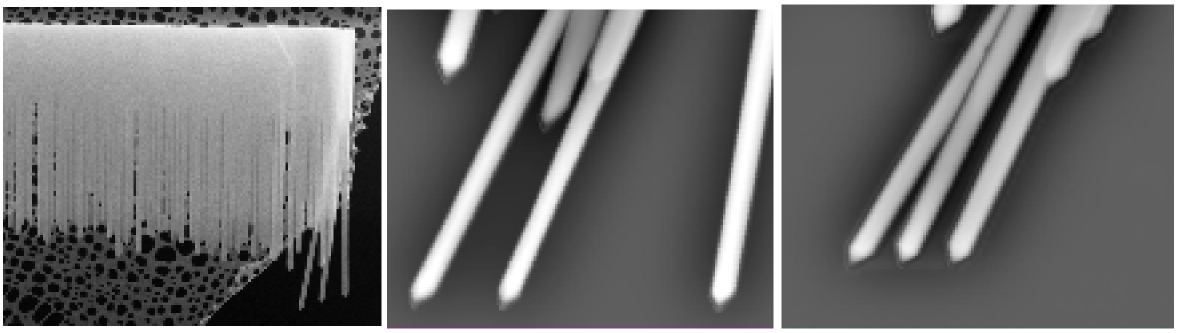

Figure 1. *(Left) Secondary-electrons SEM image of the ZnO comb-like nanostructure. The 3 nanowires in the lower right part of the comb have been considered for reconstruction. (Center) Transmitted-electrons SEM image of the nanowires. Such a STEM image fulfills the projection requirement and is suitable for tomography. The imaged area of interest measures 4.5×4.5 μm, corresponding to a primary magnification of 50.000×. (Right) Transmitted-electrons SEM image of the nanowires at 58 ° tilt relative to the image at center.*

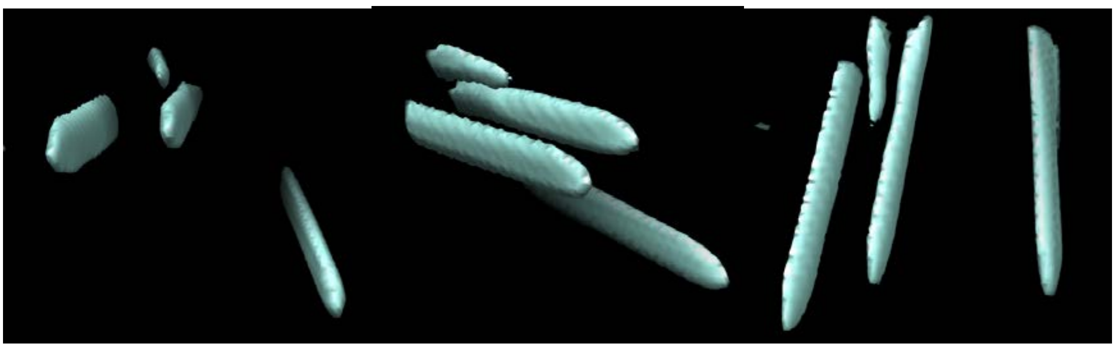

Figure 2. *Volume renderings of the nanowires. The disposition of the wires, their uniform section and the tapered termination are properly retrieved by the tomographic reconstruction.*