3D Imaging and Analysis

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STEM-EDX nano-tomography in a TEM

K Lepinay[1,2], F Lorut[2], R Pantel[2], T Epicier[1]

[1]MATEIS, umr CNRS 5510, Bât. Blaise Pascal, INSA de Lyon, F-69621 Villeurbanne Cedex
[2]STMicroelectronics, 850 rue Jean Monnet F-38926 Crolles Cedex, France

thierry.epicier@insa-lyon.fr
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Performing 3D experiments at a nanometric level in a Transmission Electron Microscope (TEM) has almost become a routine technique in Materials Science. Basically, tomography in the TEM is performed as it is in X-ray tomography: a series of tilted projections on the object of interest is acquired over a large angular domain; after in situ and post-mortem alignment, these images are used as an input for dedicated algorithms enabling the volume to be reconstructed [1, 2]. The development of tilting tomography in Materials Science has indeed been slowed down, compared to tomography in Biology, because conventional TEM (bright-field) imaging does not generally fulfil the 'projection requirement' in the case of crystalline materials; when tilted, any crystalline object experiences contrast variations due to Bragg diffraction effects, which makes that the intensity in projections is not proportional to the "mass-thickness". As a consequence, the reconstruction cannot properly render the actual 3D structure of the sample. Practical approaches were possible when acquisition of signals independent from elastic diffraction became available to allow realistic tilting sequences: this has been achieved thanks to STEM-HAADF (Scanning TEM, High Angle Dark Field imaging) [3] and EFTEM (Energy-Filtered TEM) [1, 4].

Very interestingly, these two techniques, which have been extensively used and developed for nano-tomography during the last decade, offer the additional advantage to provide chemical information. In HAADF, incoherent scattering yields to a contrast varying as roughly Z^2, which thus permits to discern phases with sufficiently different mass-thicknesses. In EFTEM, elemental maps can be acquired using ionisation edges of different elements. However, each of these techniques has drawbacks: for materials with similar atomic density and atomic numbers, HAADF cannot provide a sufficient contrast for evaluating the chemistry. Concerning EFTEM, several images need to be acquired at each tilting position (at least 3 per element), and the method is very time consuming. It was then tempting to think about tomography using EDX (Energy-Dispersive X-ray) elemental mapping. But this idea remained a dream for a long time because EDX mapping is usually by far been much longer than STEM-HAADF of even EFTEM imaging [4].

This situation has recently changed owing to major technical improvements, all available on the same commercial instrument, regarding (i) the new generation of EDX analysers (the so-called SDDs: Silicon Drift Detectors), (ii) high brightness field- emission electron sources, (iii) the parallel coupling of several (4) detectors around the sample, owing to an optimized geometry of the pole pieces [5]. The aim of this contribution is to give some recent illustrations of nano-tomography performed in a TEM using the STEM-EDX technique. Several experiments will be presented, which were all carried out on the FEI-Tecnai OSIRIS microscope installed at ST Microelectronics in Crolles, F; accordingly, most results will concern devices for applications in microelectronics.

The first example presented here concerns an advanced 28nm FDSOI transistor (Figure 1 a-b) [6]). STEM-EDX nano-tomography was performed on FIB-milled needle of 150 nm of diameter (Figure 1 c)). The reconstructed volume is shown Figure 1 e) to g): information could be easily extracted from the volume to re-project single slices or sub-stacks of slices from any directions as in classical tomography but with a direct chemical meaning. Figure 2 refers to multi-gate devices such as gate-all-around nanowires, a perspective beyond the end of the CMOS roadmap. An efficient failure control in conventional 2D-TEM would require being able to prepare thin foils in any direction of such (small) devices: not only this procedure will be time-consuming, but it is not known a priori which viewing orientation would better reveal the possible defects. 180° EDX-tomography on FIB-prepared needles allows the device to be examined without any restriction, nor shadowing effect according to the 3D reconstruction.

Results (i.e. benefits and issues) will be discussed in terms of irradiation effects and mechanical instabilities, which are obviously key issues in all experiments. Most acquisitions were carried out under the following typical conditions: i) beam energy 120 kV, probe size and current typically 0.5 nm and 1 - 2 nA respectively, ii) tilt series over 180° every 2 to 5°, iii) EDX maps about (500-800) pixels², dwell time between 500 and 1000 µs. All these parameters lead to acquisition times of a few minutes per map, and a total duration of 6 to 8 hours. We also aim at comparing STEM-EDX nano-tomography with other complementary techniques, such as HAADF and EFTEM as already evoked, but also Atom Probe Tomography [8], a very efficient technique for chemical analysis in 3D.

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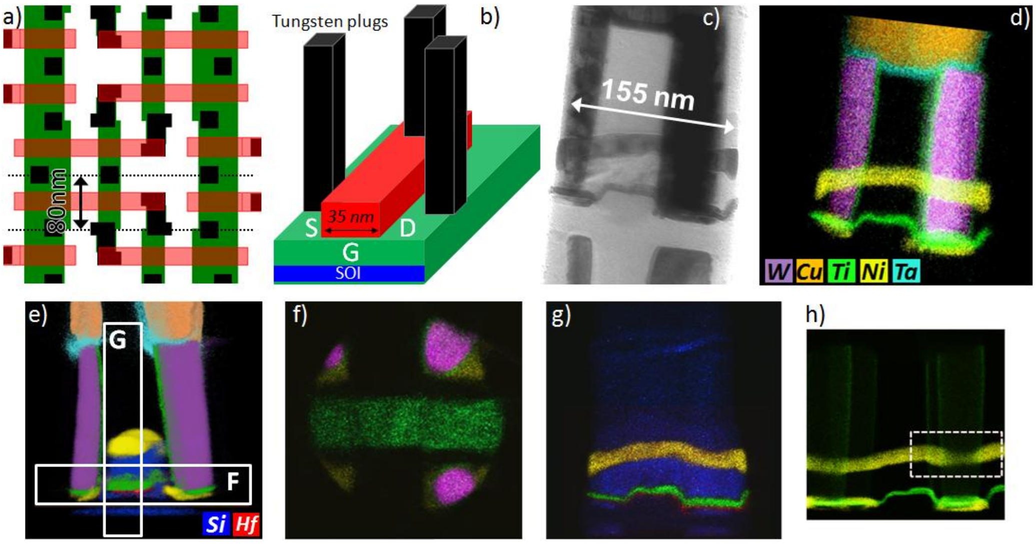

Figure 1. 3D STEM-EDX analysis of a 28nm FDSOI transistor. a): SDRAM layout (top view; active silicon is shown in green, the gate stack in red and the tungsten plugs in dark). b): schematic representation of a transistor. c): TEM image of the FIB tip (thickness about 150 nm). d): composite EDX map of the complete projection (acquisition at 120 keV, probe current 1.2 nA, tilt step 2° from -90 to 90°, 5 min. per map with size of 800x800 pixels²). e-g): 3D rendering of the chemical tomogram and volume extractions (45 nm - 150 slices -) along two perpendicular directions (frames F and G corresponding respectively to projections shown in f) and g)). H): typical usual 2D EDX map of Ti and Ni showing the shadowing effect produced by the W plug (to be compared to g)).

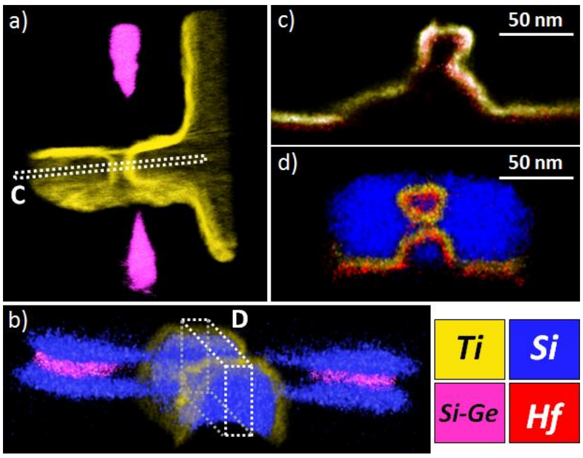

Figure 2. 3D chemical analysis of a complex device showing a failed system (a) and c)) and a well-processed one (b) and d)). Dotted frames in a) and b) show 'virtual' TEM lamellae extracted with any desired thickness and in any orientation to produce projections shown in c) and d). The visualization in d) demonstrates the correct and continuous high-K metal gate deposition all around the nanowire, contrarily to case c). The 3D resolution can be estimated around 3-4 nm (see details in [7]).