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FIB Specimen Preparation for STEM and EFTEM Tomography

S.M. Schwarz[1,2] and L.A. Giannuzzi[1,3]

[1]University of Central Florida, Orlando, FL 32816, [2]NanoSpective Inc., Orlando, FL 32826,
[3]FEI Company, Hillsboro, OR, 97124

Electron tomography using transmission electron microscopy (TEM) and related techniques (e.g., scanning transmission electron microscopy (STEM) or energy filtered TEM (EFTEM)) allow for 3D microstructural and elemental mapping of specimens, and has been used successfully in the biological sciences where mass-thickness contrast dominates these mostly amorphous materials [1]. Recently, Z-contrast STEM via high angle annular dark field (HAADF) tomography has also been used successfully in the physical sciences. STEM and EFTEM tomography are more useful techniques for crystalline materials, since diffraction contrast in conventional TEM images can hinder image reconstruction. Typical tomography routines utilize conventional electron transparent foils, whereby the dimensions of the specimen perpendicular to the electron beam may be orders of magnitude greater than the specimen thickness parallel to the electron beam. Using this conventional specimen geometry, the effective specimen thickness increases as the specimen is tilted through +/- 70 degrees necessary for the tomographic acquisition. Thus, performing EFTEM at higher tilts can become impossible since multiple scattering events can hinder the acquisition of adequate or reliable maps. In addition, changes in effective specimen thickness due to specimen tilting can also vary the contrast in HAADF STEM images.

In order to overcome the specimen thickness limitations with tilting, we have prepared site-specific cylindrical electron transparent FIB milled specimens using the in-situ lift-out technique [2]. After a large piece of specimen (10 μm x 10 μm x 5 μm) is lifted out and secured onto a TEM grid, a cylindrical electron transparent specimen is FIB milled in a similar manner to that reported e.g., in atom probe applications [3]. The collection of an image tilt series may then be performed with the long axis of the specimen cylinder in the plane of tilting such that the specimen thickness is always the same regardless of specimen tilt angle. Specimen preparation and TEM analysis was performed on an FEI 200TEM FIB and an FEI TF30 operating at 300 keV, respectively.

FIG. 1a shows a zero loss EFTEM image of a pre-ceramic polymer (Si-C-O) coating on stainless steel specimen heat-treated at 850^{o}C for 30 minutes in air. Significant Fe interdiffusion from the substrate into the coating was observed. FIG. 1b shows an EFTEM oxygen map and FIG. 1c shows a HAADF STEM image of the same specimen as in FIG. 1a. Another example of this specimen preparation method is given for a PMOS gate structure in FIG. 2a-c, where FIG. 2a is a BFTEM image, FIG. 2b is an EFTEM oxygen map, and FIB. 2c is a HAADF STEM image. This cylindrically prepared specimen geometry is not limited to the examples shown herein, and can also be used to collect images for holography tomography, as well as other TEM tomography techniques [4].

References

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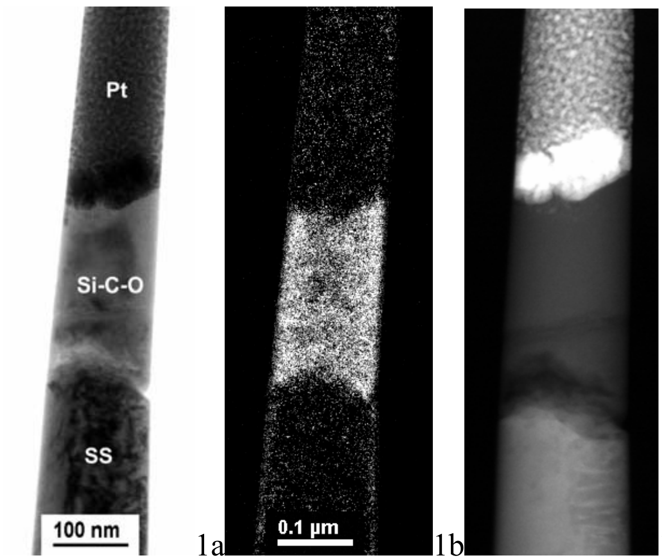
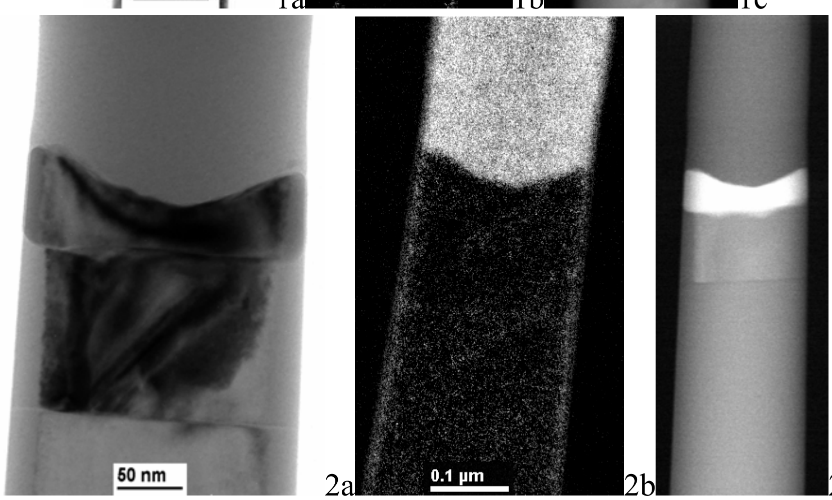


1a 1b 1c



2a 2b 2c

FIG. 1. A pre-ceramic polymer coating on a stainless steel substrate shown in an (a) Zero Loss EFTEM image showing, (b) an EFTEM oxygen map and (c) a HAADF STEM image.

FIG. 2. A gate structure from a PMOS device shown in (a) a BFTEM image, (b) an EFTEM oxygen map, and (c) a HAADF STEM image.