Quick Sample Preparation and EFTEM Elemental Characterization of FAB Based Defects

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A purpose of microscopy is to magnify and enhance contrast between different regions of a sample, whether those regions may be different structures, different orientations of the same structure, regions of different atomic weight, or different chemistries. In the present case, elemental mapping in the energy filtered transmission electron microscope (EFTEM) is used to enhance contrast between elements in an apparent bundle of fibers previously seen through scanning electron microscopy (SEM) and a particle that appears to be a catalytic source for the fibers.

Automated in-line SEM imaging is a typical defect inspection technique in a semiconductor fabrication facility (FAB). Figure 1 shows three examples of nanofiber bundles that were detected through in-line SEM defect imaging and one off-line "manned" SEM image. From the "top-down" viewing orientation shown here, one may conclude that the fiber bundles are localized, perhaps having a single origination point. However, these images give little additional information to FAB engineers to pinpoint the true source of the problem leading to these defects. Defects of this size can easily have a huge impact on future steps in the process flow. Since they are considered "killer defects," finding their cause is of very high importance for product yield for a FAB.

To assist FAB engineers in discovering the nature of these defects, TEM based techniques were applied to defects indicated by the automated in-line SEM imaging systems. Through computerized full wafer alignment and navigation stage control, the defects of interest can be located for preparation of TEM cross-section samples.

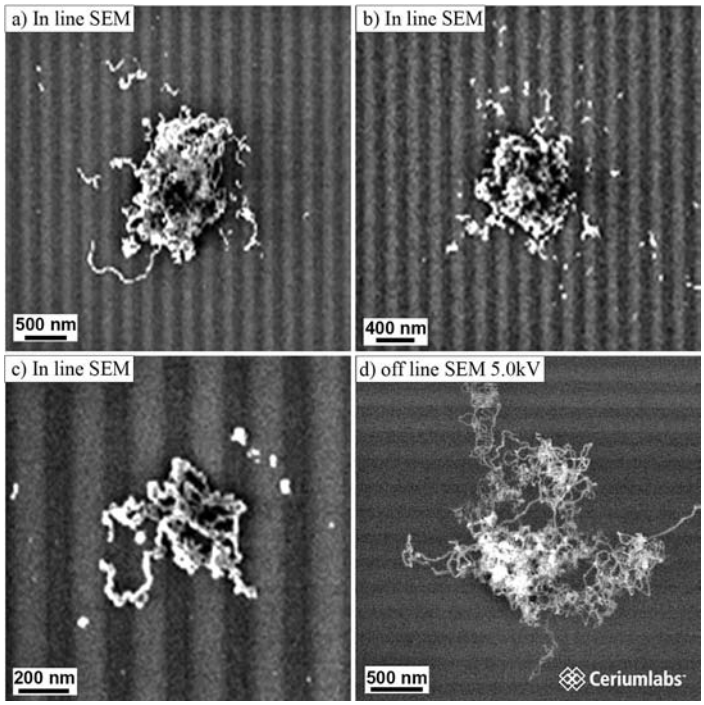

Figure 1. SEM images of typical nanofiber bundle as seen in "in line" FAB SEM and an "off line" SEM.

TEM sample preparation in the present work utilized an FEI XL835 dual-beam focused ion beam (FIB) system equipped with an Omni-Probe™ in-situ lift out extraction apparatus [1]. Using this sample preparation system, one can mill a "slice" with the ion beam and then observe the sample in a damage free manner with the electron beam to determine if a core defect is present in the newly exposed face of the TEM cross-section lamella. The lamella can then be further thinned from the opposite side to electron-transparency. In this way, the core of the defect is ensured to be within the sample prior to TEM imaging. The sample illustrated in this paper was prepared by first applying protective layers of Pt using an electron beam deposited Pt layer at 5kV followed by an ion beam deposited Pt layer using a 30kV Ga ion beam. This two-step process ensures that the Ga ions used in the ion-beam deposited Pt deposition are stopped in the electron beam deposited Pt layer and do not damage the region of interest. Bulk milling is performed using a Ga ion beam at 30kV followed by a 5kV "low-kV" clean up step to remove most of the amorphized material remaining on the surface of the lamella from the high energy mill [2]. Final sample thicknesses in the range of 50nm-100nm are typically achieved through this technique.

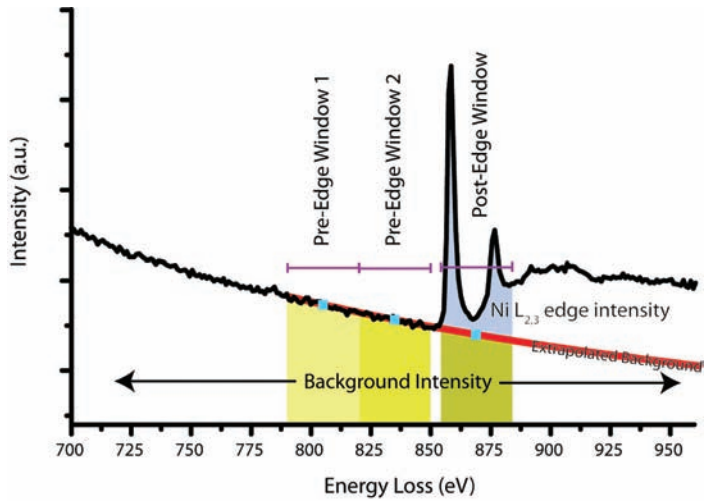

Figure 2. Schematic of the three-window EFTEM elemental mapping technique as applied to the Ni $L_{2,3}$ edge in an EEL spectrum. Two pre-edge windows are used to model and extrapolate the background intensity to subtract from the post-edge window on a pixel-by-pixel basis.

Conventional and energy filtered TEM (EFTEM) imaging was performed using a Philips/FEI CM300 TEM, equipped with a Model-850 Gatan Imaging Filter™ (GIF-2002). Conventional bright field images were acquired at an accelerating voltage of 300kV and the EFTEM images were recorded at 297kV. Elemental maps were calculated using the three window technique illustrated in figure 2. An electron energy loss spectrum (EELS) from 700eV to 960eV energy loss is shown in figure 2 as a black line, which includes the Ni $L_{2,3}$ edge. The EFTEM three-window elemental mapping technique makes use of the EELS spectrum to generate an elemental map using core electrons from defined energy windows, shown in yellow in the figure. As can be seen, the edge intensity sits on a decaying background, which should be subtracted in order to create a more accurate elemental map. The three window technique achieves this by using two pre-edge windows to calculate a background based on a two-parameter model, typically of the form $I=AE^{-r}$, where I is the intensity at energy loss E which are shown as light blue points, and A and r are the fitting parameters. The third image filtered by the energy selecting window is called the post-edge window, which includes intensity from the core-loss electrons and the background.

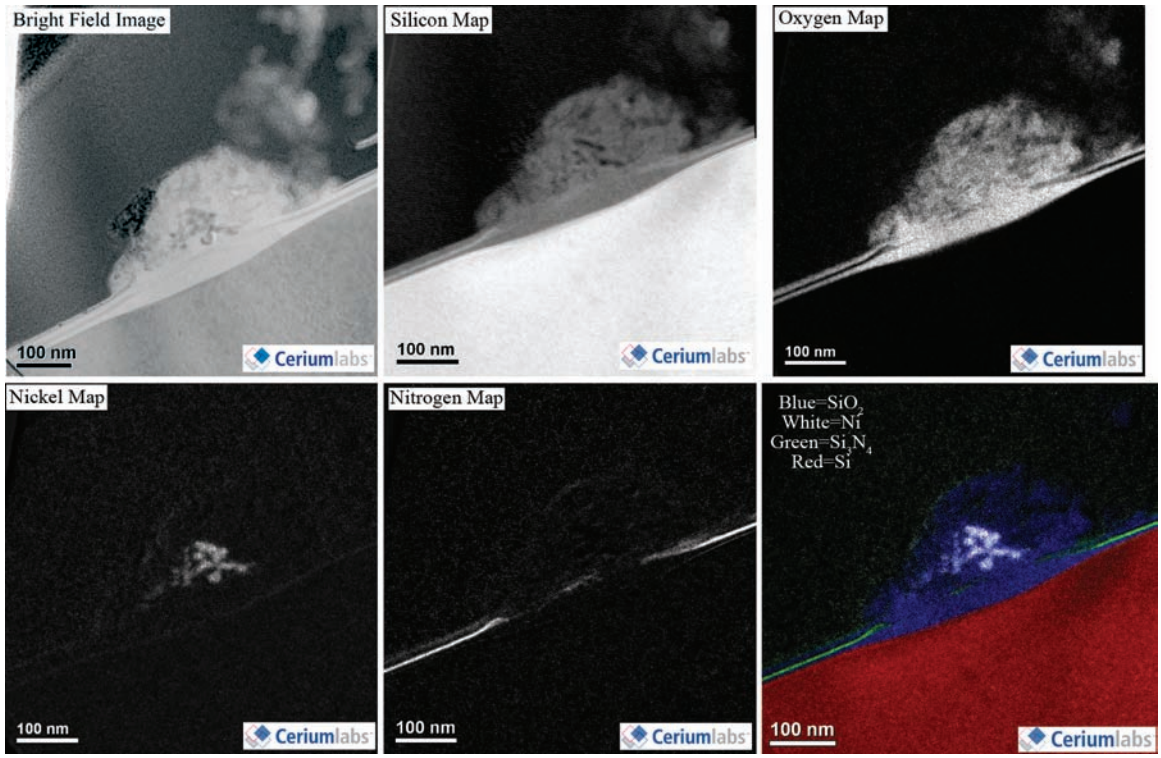

Figure 3. Bright field TEM image with corresponding EFTEM elemental maps and false color composite of the elemental maps. Together, the maps show that the nanofiber bundle is composed of Si-oxide fibers with a Ni particle at the center of the bundle which likely broke the underlying ONO layer creating a diffusion pathway to the substrate.

The modeled background is extrapolated to predict the background contribution to the post-edge image, and is subtracted from it. This operation is carried out on a pixel-by-pixel basis to calculate the intensity at each pixel. When reconstructed, this calculation results in a spatial map of the elemental distribution [3]. This approach inherently does not consider the effects of plural scattering, and is susceptible to inaccuracies arising from features in the pre-edge region. However, with suitable sample thickness, edges that are well separated, and a slowly varying background, this technique is quite good.

A TEM cross-section sample was prepared from the fiber bundle shown in the off-line SEM image in figure 1. A montage of bright field and energy filtered TEM images of the corresponding TEM cross-section are shown in figure 3. One immediately notices the contrast between the fiber bundle, the substrate, and an extra layer that appears to have grown into the substrate. The surrounding, granular material is the electron-beam deposited Pt, and at the top left, one can see the interface with the ion-beam deposited Pt. Directly above the substrate, there is a triple layer of Si-oxide/Si-nitride/Si-oxide (ONO) which serves as the charge storage layer for the semiconductor device. At the center of the fiber bundle, there is a dark contrasting particle, but its chemistry can not be determined from the bright field image alone. Broad area energy dispersive X-ray (EDX) spectra were recorded to determine the elements present using a Noran X-Ray detector, and the Noran Vantage analysis system. Based on the EDX spectroscopy results, Si, O, N, C, Cu, Pt, and Ni were found. The Cu is likely from the Omniprobe support grid, the Pt and C are from the protective layer, leaving Si, O, N, and Ni as the elements of interest to map. These maps were calculated using the three window EFTEM technique and are shown in figure 3, along with a false color aligned composite image.

The silicon map in figure 3 shows a very strong signal from the substrate, a weaker signal from the fiber bundle, and sufficient contrast to clearly distinguish the ONO layers. In the oxygen map, one clearly sees that the growth into the substrate is oxygen containing, as are the fibers, and again, the ONO layers can be distinguished. Of particular interest is the Ni map that clearly shows that only the particle contains Ni and that the fibers do not. The nitrogen map shows that the nitride layer of the ONO films is broken, and when compared to the oxygen map, reveals that those breaks are likely filled with the oxide. One can easily visualize the spatial relationship between the elemental regions by combining these maps in a false color composite. Through this technique, one can paint a picture of what likely occurred. During processing, the Ni particle landed on the wafer and punctured the top oxide layer and the nitride layer providing a diffusion path for additional oxygen into the substrate/bottom oxide layer. During the subsequent rapid-thermal-anneal (RTA) step, there may have been a slight vacuum leak allowing water vapor or some other oxygen containing molecule into the RTA tool. At this point, the Ni particle served as a diffusion pathway and possibly a catalytic site for the O to react with Si from the substrate to grow the Si-oxide fibers, and as a pathway for the O to diffuse into the bottom oxide layer forming "birds-beaks" between the nitride layer and the substrate.

Through the use of dual-beam FIB with a full wafer stage and defect navigation control, along with defect locations determined through the in-line FAB monitoring system, the defects can be quickly found and prepared into TEM cross-sections. Using the OmniProbe™ *in-situ* extraction apparatus, these cross-sections can be mounted on the edge of a gridbar so that there is no support film underneath the sample to diminish the quality of the transmitted electron based results. Additional thinning can also be performed if desired after the lamella is mounted. Using a full analytical TEM with EDXS and energy filtered imaging capability, a defect can be brought into the lab, and in less than one shift, can be sufficiently characterized to enable FAB engineers to pinpoint the source of the problem and correct it to minimize downtime. ■

References:

[1] L.A. Giannuzzi et al., "FIB Lift-Out Specimen Preparation Techniques", in *Introduction to Focused Ion Beams: Instrumentation, Theory, Techniques, and Practice*, L.A. Giannuzzi, F.A. Stevie (eds), Springer-Verlag New York, 201 (2004).

[2] L.A. Giannuzzi et al., "Ion-Solid Interactions", in *Introduction to Focused Ion Beams: Instrumentation, Theory, Techniques, and Practice*, L.A. Giannuzzi, F.A. Stevie (eds), Springer-Verlag New York, 201 (2004).

[3] R.F Edgerton, Electron Energy-Loss Spectroscopy in the Electron Microscopy, 2nd ed., Plenum Press, New York, 1996

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