Correlation between TEM imaging and microanalysis for atom probe reconstruction verification

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The atom probe instrument field evaporates atoms from a specimen of interest and these atoms (now ions) are collected on a position-sensitive, mass-spectrum detector. By reconstructing the trajectory path and impact position of each ion from the field evaporation event, a volumetric reconstructed rendering of the material is generated with near atomic precision for each individual atom. The reconstruction method of an atom probe volume is dependent upon a constant evaporation field [1]. When the evaporation process proceeds through an interface of two different phases, the field can change resulting in aberrations in the atom probe reconstruction. These aberrations typically appear as density variations across interfaces and/or incorrect morphologies of precipitates within the matrix [2]. To help validate the atom probe reconstructions, TEM imaging and microanalysis can be employed. This proceeding addresses specific examples where the coupling of TEM can assist in the validation of the atom probe reconstruction. In addition, the proceeding addresses some experimental difficulties in bridging the two microscopy techniques.

Example #1: As a thin film is reduced in layer thickness, the film's structure can undergo a crystallographic change. For a multilayered stack of two different phases, this pseudomorphic phase transformation in each of the individual layers has been modeled by the competition between thermodynamic volumetric energy increases and interfacial energy reductions [3]. Using this model, Thompson *et al.* [3] predicted and confirmed the α -hcp to β -bcc transitions for Ti in a Ti/Nb multilayers. Atom probe reconstructions [3] of the bcc transformed Ti layers revealed near 20 at.% Nb interdiffusion into the Ti layers, as seen in Figure 1 (a). The Nb interdiffusion is a β -stabilizer and reduces the volumetric free energy change.

An FEI Titan F30 S/TEM equipped with electron energy loss spectroscopy (EELS) and an aberration corrector on the probe-forming lens was employed to investigate the compositional distribution in the Ti/Nb multilayers. To determine the composition in each layer, as measured by the EELS signal, the following equation was used:

$$\frac{N_A}{N_B} = \frac{I_A(\beta\Delta)\sigma_B(\beta\Delta)}{I_B(\beta\Delta)\sigma_A(\beta\Delta)}$$

where N_i is the numbers of atoms of type i, β is the semiangle spectrometer collection angle and Δ is the energy window range. The ratio of σ_B/σ_A is the sensitivity ratio, similar to the Cliff-Lorimer factors used in x-ray energy dispersive spectroscopy [4]. The STEM-EELS line profile, shown in figure 1(b), indicates reasonably good agreement with the atom probe data set. The slight difference in the individual layer thicknesses will be addressed.

Example #2: With continuing decreases in transistor sizes, new architectures are being developed. One of the major issues is current leakage with sub-100 nm nodes. A FinFET device, also referred as

a tri-gate structure, is a nonplanar, triple gate transistor that has a conducting channel wrapped around a thin Si 'fin.' The fin determines the effective channel length and can provide increased surface area for the electrons to travel. One of the major challenges is to determine the 3D dopant distribution in these devices. A series of As doped Si FinFET devices have been fabricated into atom probe tips using a site-specific FIB based technique [5]. The generic location where the site-specific atom probe tip was extracted is shown in Figure 2(a). The isosurface of 0.52 As atoms per nm³ 3D atom map reveals the As distribution, Figure 2(b). This image was then compared to a STEM-High Angle Annular Dark Field (HAADF) micrograph of a similar tip shown in Figure 2(c). A high-angle tilt series of STEM-HAADF images have been collected and compared to the isosurface reconstruction of similar regions in the atom probe data sets.

References

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Figure 1: Ti/Nb multilayer (a) atom probe compositional profile (b) EELS compositional profile.



Figure 2: (a) General location of the atom probe extracted tip (b) Reconstructed atom map (c) Representative STEM-HAADF image revealing a higher-Z contrast in an extracted atom probe tip.