PHYSICAL ELECTRONICS

3D FIB-TOF IMAGING

the Microstructure of an Alloy

OVERVIEW

3D characterization of materials with time-of-flight secondary ion mass spectrometry (TOF-SIMS) has traditionally been accomplished via depth profiling where a sputter ion beam erodes material from the probed specimen between TOF-SIMS image acquisition cycles. After many sputter and acquisition cycles, a 3D image of the probed volume may be reconstructed from the saved RAW data stream file. However, there are a number of difficulties with which to contend depending on the nature of the sputter ion beam and the matrix composition of the probed specimen. Differential sputtering, which arises when one component of the sample matrix is removed more readily than other components, may distort the true 3D chemical distribution in both inorganic and organic matrices. Accumulated beam damage from either the sputter or the acquisition ion beams, which alters the matrix chemistry as the depth profile proceeds, at best complicates interpretation of the 3D chemical distribution or, in the worst case, inhibits the collection of relevant chemical information. Thus, TOF-SIMS depth profiling is limited to probing less than 5 μm in "favorable" circumstances, and to less than 300 nm in

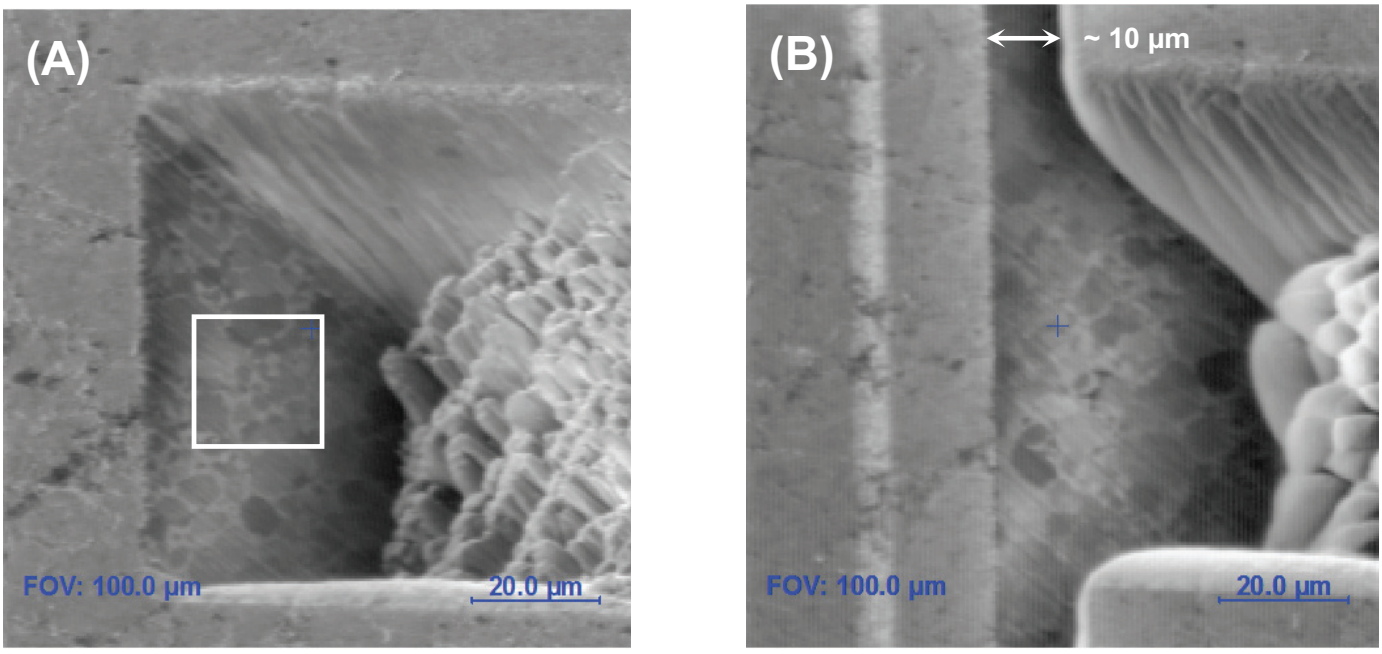

Figure 1: Primary ion-induced secondary electron (SE) images of the Cu-W alloy sample showing (A) the initial FIB-milled crater and (B) after 20 successive FIB sections. The approximate location of the 20 x 20 μm² area imaged by TOF-SIMS is indicated in panel (A) and the approximate "depth" of the imaged volume is indicated in panel (B).

"unfavorable" circumstances.

For some time, 3D characterization of materials has been accomplished with dual-beam FIB where a FIB gun is used to section the sample between SEM image acquisition cycles. [1] Most commonly, SEM is used in conjunction with energy dispersive x-ray spectroscopy (SEM-EDS) or with wavelength dispersive x-ray spectroscopy (SEM-WDS) to obtain elemental information. However, there are several limitations to this analytical methodology which include low sensitivity, especially for light elements. Other limitations include poor spectroscopic resolution, limited chemical information, and no information concerning molecular or structural chemistry.

In this Note we demonstrate the use of the FIB option (30 kV Magnum UHV FIB column from FEI Co.) on the PHI *nanoTOF* to extract 3D image information associated with a Cu-W alloy. The large solid angle and depth-of-field intrinsic to the TRIFT mass spectrometer are utilized to image the vertical wall of the FIB-milled and subsequently sectioned alloy matrix. Successive *in situ* FIB milling and TOF-SIMS image acquisition cycles are employed to image a 10 μm deep volume. After FIB milling the the initial crater, 3D imaging the 10 μm deep volume is accomplished in less time than is required to complete a TOF-SIMS depth profile using a low voltage sputter ion beam but without the limitations of differential sputtering. The imaged surface of the FIB-milled crater, indicated in Figure 1A, is defined as the surface. The depth of the 3D-imaged volume, indicated in Figure 1B, is defined by the cumulative length of the FIB line cuts.

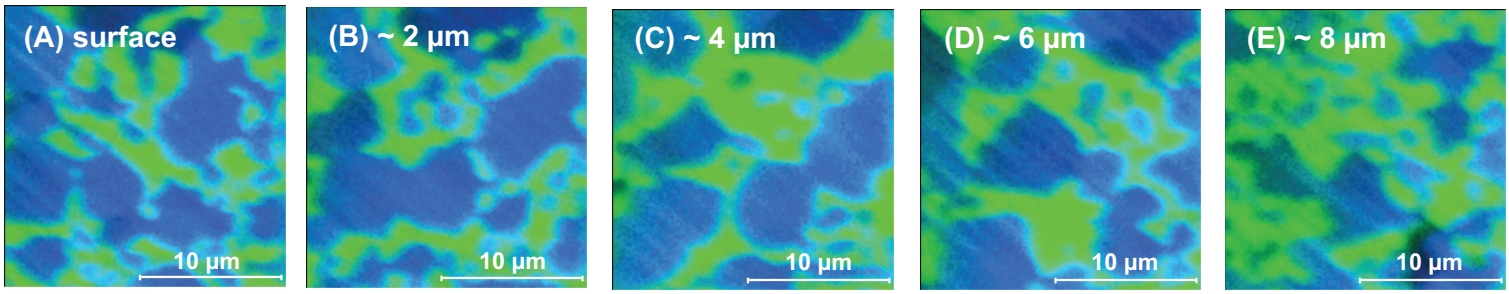

Figure 2: A set of 2D images-at-depth showing the Cu and W phases of the sample as color overlays of the Cu^+ (green, 63 m/z) and WO^+ (blue, 200 m/z) images. The field-of-view (FOV) in each image is 20 μm x 20 μm.

EXPERIMENTAL

FIB crater: The initial crater was milled into the Cu-W alloy near the central region of the sample (away from the edges of the sample) using a 30 keV Ga^+ beam with a DC current of 7 nA. The initial crater was milled with dimensions of 60 μm x 120 μm using a 5-step cross-section pattern at a dose of 80 nC/μm^2 with a 90% beam overlap and 3 μsec dwell time. The resulting crater was approximately 50 μm deep at the vertical back wall. No gas injection system (GIS) was used and no polishing step was utilized; nevertheless, the curtaining is minimal and did not impede the TOF-SIMS image acquisition.

FIB sections: FIB cross-sections were cut using a 30 keV Ga^+ beam with a DC current of 7 nA. Each line cut pattern had dimensions of 0.5 μm x 125 μm, and a dose of 240 nC/μm^2 was used with a 90% beam overlap and 3 μsec dwell time. Each line cut required approximately 12 minutes to mill. Again, no polishing step was utilized.

TOF-SIMS imaging: An unbunched 30 keV Au^+ primary ion beam, operating at a DC current of 0.5 nA and a digital raster of 256 pixels x 256 pixels, was used to acquire images of the vertical FIB-milled crater wall (image area indicated in Figure 1A) in the positive secondary ion polarity. The measured Au^+ probe diameter is <200 nm. In all, twenty (20) RAW data stream files were collected at a 20 μm x 20 μm field-of-view and using 5 minutes to acquire each data file. The sample was not moved between successive FIB section and TOF-SIMS acquisition cycles. An oxygen leak ($1x10^{-4}$ Pa) was used to enhance the positive polarity secondary ion signals.

RESULTS AND DISCUSSION

Color overlays of the Cu^+ (green) and WO^+ (blue) TOF-SIMS images are presented in Figure 2. These 20 µm x 20 µm 2D images-at-depth were collected at discrete depths during the successive FIB milling and TOF-SIMS image acquisition cycles. Note that, in all, twenty FIB mill and TOF-SIMS image cycles were performed to a total depth of 10 µm (see Figure 1B). The small grains of copper and larger grains of tungsten are clearly visible, but much of the copper appears at the tungsten grain boundaries. There are visible striations in each image (i.e. curtaining) that arise from the high current FIB milling process. The curtaining effects may be reduced or eliminated by local ion beam-assisted deposition of a protective layer, or by FIB polishing steps consisting of successively smaller FIB beam currents to produce a smooth surface prior to the FIB sectioning steps.

The twenty individual RAW data stream files from the TOF-SIMS analyses may be compiled into a single RAW file for the purposes of extracting depth profiles and 3D images. The concatenated depth profiles of Cu^+ and WO^+ are rendered in Figure 3. For comparison, the total ion profile is also shown. Notice that, other than some minor undulation of the Cu^+ and WO^+ signals, the profiles are quite unremarkable. The 3D distributions of copper and tungsten are observed in Figure 4 where the Cu^+ (green) and the WO^+ (blue, opacity is 0.5) iso-surfaces are presented in an overlay. In these iso-surface overlays, the full 3D distribution of alloy components may be readily observed and fully explored. Thus, the entire 20 µm x 20 µm x 10 µm volume may be characterized. This level of 3D materials characterization would not be possible via conventional TOF-SIMS depth profiling with a low voltage sputter ion beam due to the effects of differential sputtering and to a prohibitively long acquisition time.

CONCLUSION

The PHI *nanoTOF*, in conjunction with *in situ* FIB milling, provides a platform for 3D chemical imaging of large volumes owing to the design of the TRIFT analyzer to have a large solid angle of collection.

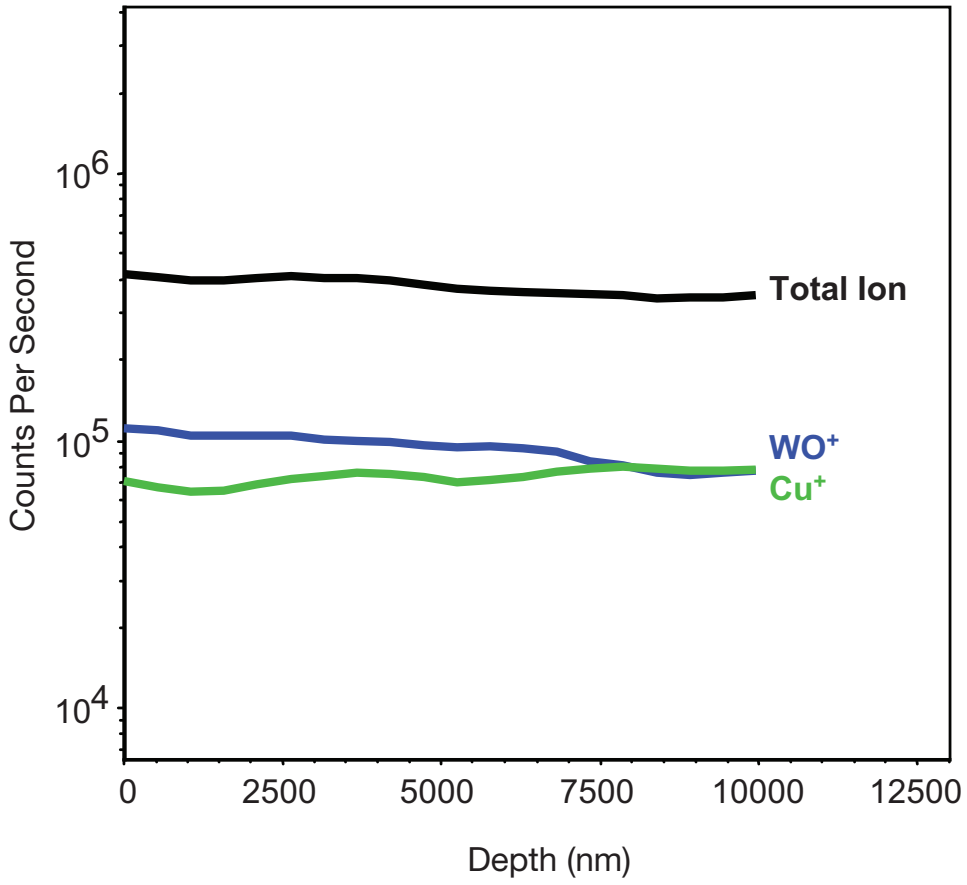

Figure 3: Concatenated depth profile, generated from the 20 individual RAW data stream files, showing the profiles of Cu^+ (green, 63 m/z), WO^+ (blue, 200 m/z), and the total ion signal. Note that the depth scale is defined by the cumulative length of the FIB line cuts in the plane perpendicular to the surface of the sample (see Figure 1B).

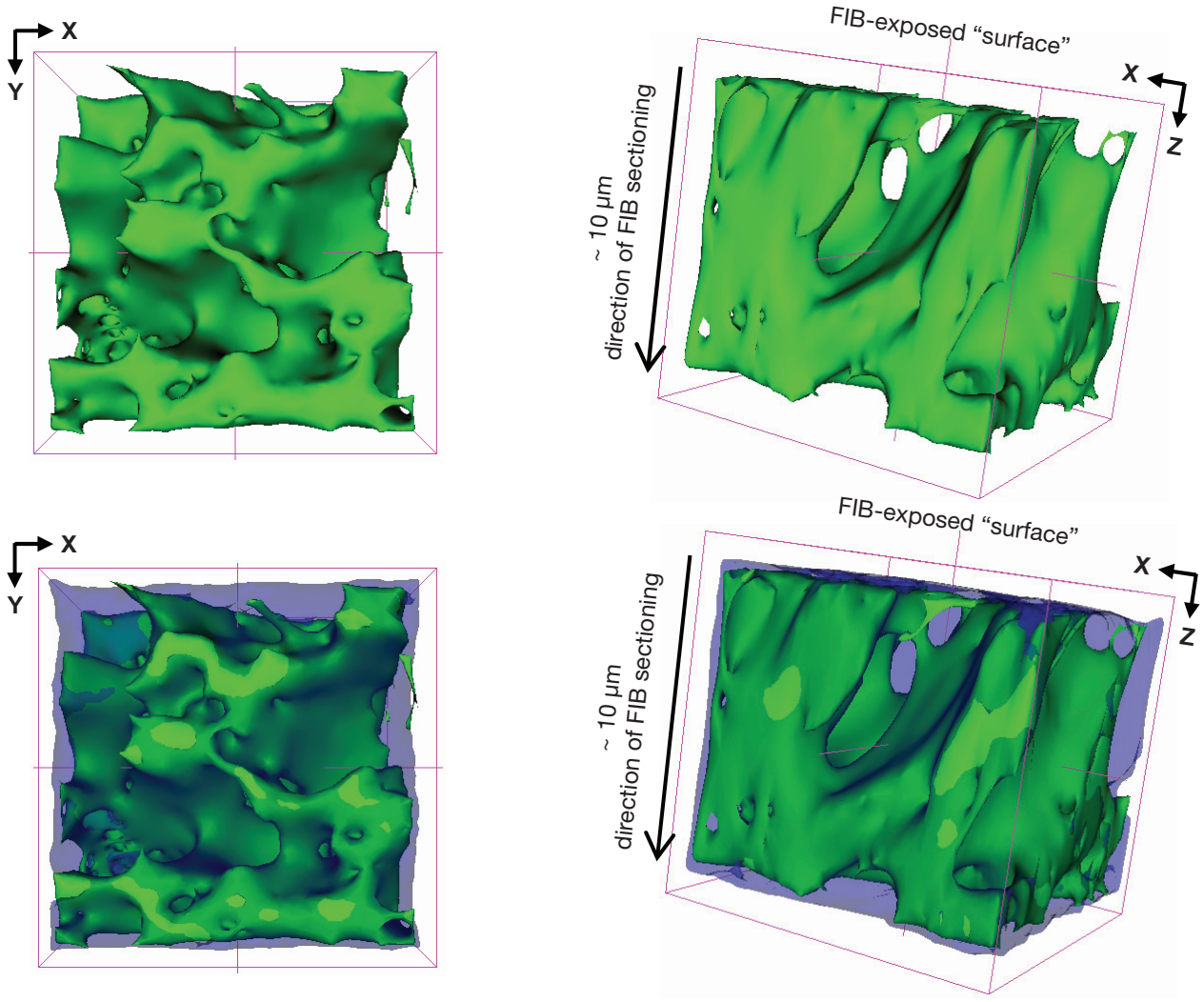

Figure 4: (Top row) 3D iso-surface images of Cu^+ (green, 63 m/z) and (bottom row) 3D iso-surface overlay images of Cu^+ (green, 63 m/z) and WO^+ (blue, 200 m/z). The distribution of copper and tungsten in the 20 µm x 20 µm x 10 µm volume is revealed without the artifact of differential sputtering. Note that the opacity of the WO^+ iso-surface has been reduced so that the interior distribution of the Cu^+ iso-surface image is visible. Note also that the depth scale is defined by the cumulative length of the FIB line cuts in the plane perpendicular to the surface of the sample (see Figure 1B).

The intrinsic characteristics of the TRIFT analyzer were demonstrated by imaging the vertically-oriented wall of a FIB-milled crater without e.g. tilting the sample. Thus, full 3D characterization of materials is possible with successive FIB milling and TOF-SIMS acquisition cycles, and 3D image reconstruction is straightforward because the sample is not moved between FIB milling and TOF-SIMS data acquisition.

ACKNOWLEDGEMENTS AND REFERENCES

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[1] Holzer, et al., *J. Micr.*, **216** (2004) 84-95.

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