

FIB Clean Up of ToF-SIMS Craters for Microstructural Characterization

V.S. Smentkowski and D. Ellis

General Electric Global Research Center, Niskayuna, NY 12309

Focused Ion Beam (FIB) techniques [1] are widely used in order to generate cross sections of samples which can then be analyzed using a variety of analytical instrumentation in order to obtain microstructural information. In order to rapidly generate FIB cross sections, small areas (typically 5 to 10 microns) are milled. Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) is a powerful surface analytical technique that is able to measure low concentrations of species [2]. ToF-SIMS depth profile measurements are collected by eroding a large area (50 to 1,000 microns in size) of a sample using an Ar⁺ or Cs⁺ ion beam for a certain amount of time, pausing the erosion, analyzing a smaller (central) region of the eroded area, and repeating the cycle until the desired depth has been reached. Ion images are often recorded at each depth and a full mass spectrum is saved at every volume element allowing for 3 D analysis [3]. Often times, both ToF-SIMS depth profile analysis and high resolution microstructural SEM characterization are required on the same sample. Ideally, these complimentary analyses would be performed in the same region of a sample. In this presentation, we will demonstrate that FIB techniques can be used to clean up the large area craters produced by ToF-SIMS depth profile analysis thereby enabling microstructural characterization over larger areas of samples.

FIG 1 shows a ToF-SIMS depth profile measurement that was collected on an Au/Al/Ti/Au/Si multilayer thin film stack; FIG 2 shows a SEM image of the ToF-SIMS crater. The crater side walls reveal significant topography (cone formation) [4] which prevents meaningful microstructural characterization of the crater wall. The top panel of FIG 3 shows a low magnification SEM image of a FIB cut that was generated at 90 deg to the ToF-SIMS crater wall; the FIB cut continues from the crater (left side) into an unperturbed region of the thin film stack (right side). The lower panel of FIG 3 shows higher magnification SEM images generated close to the ToF-SIMS crater (far left) as well as 15 and 60 microns in. We are in the process of using FIB techniques to perform serial cuts starting at the outer ToF-SIMS crater and moving inward until the SEM images no longer reveal damage associated with the erosion performed during the ToF-SIMS depth profile analysis – these data will be included in the presentation.

Advantages of this approach are 1) larger FIB cross sections can be made because a significant amount of material was removed during the erosion cycles in the ToF-SIMS analysis, 2) combining the larger area and statistically refined chemical data from the ToF-SIMS analysis with the microstructural characterization provided by FIB-SEM analysis, and 3) both the ToF-SIMS data and the FIB-SEM data were generated in the same region of the sample. The ToF-SIMS depth profile craters have a slope of about 45 deg (the ion column is positioned at an angle of 45 deg from the sample normal), an advantage of performing the FIB clean up at this angle is the enhancement in the layer thickness that will be available for microstructural (as well as chemical) analysis.

We would like to thank Lauraine Denault for providing high magnification SE images of the ToF-SIMS craters.

References

[1] C.A. Volkert and A.M. Minor. *MRS Bulletin* 32 (2007) 389.

[2] J.C. Vickerman, D. Briggs Eds. *ToF-SIMS Surface Analysis by Mass Spectrometry*; Surface Spectra/IM Publications: Manchester, U.K., 2001.

[3] V.S. Smentkowski, et al., *Anal. Chem.* 79 (2007) 7719.

[4] V.S. Smentkowski, *Prog. Surf. Sci.* 64 (2000) 1

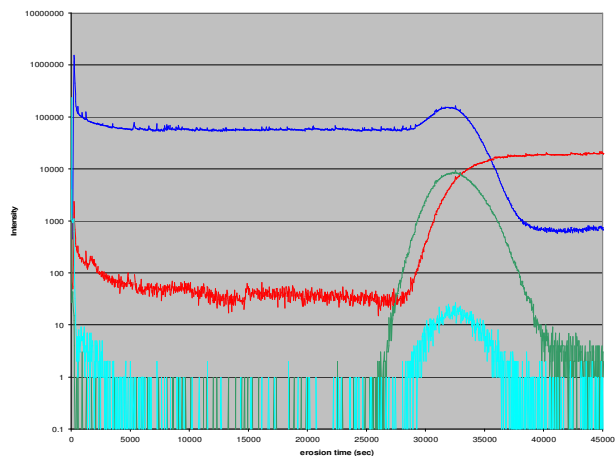


FIG1 ToF-SIMS depth profile traces. Time is proportional to depth. Dark blue = Al, light blue = Au, green = Ti, and red = Si.

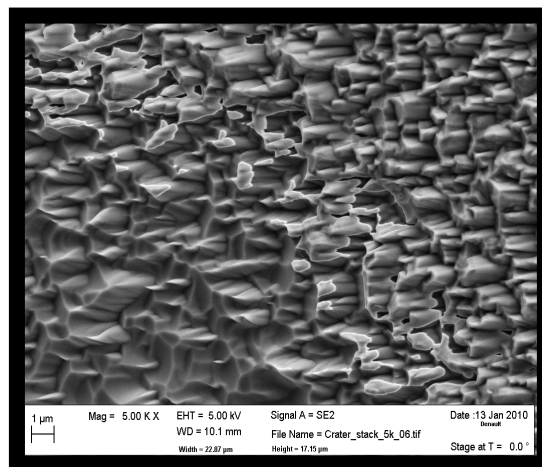


FIG 2 SEM image of side wall of the crater (Ar ions impinged the sample from the left side). Note cone formation [4].

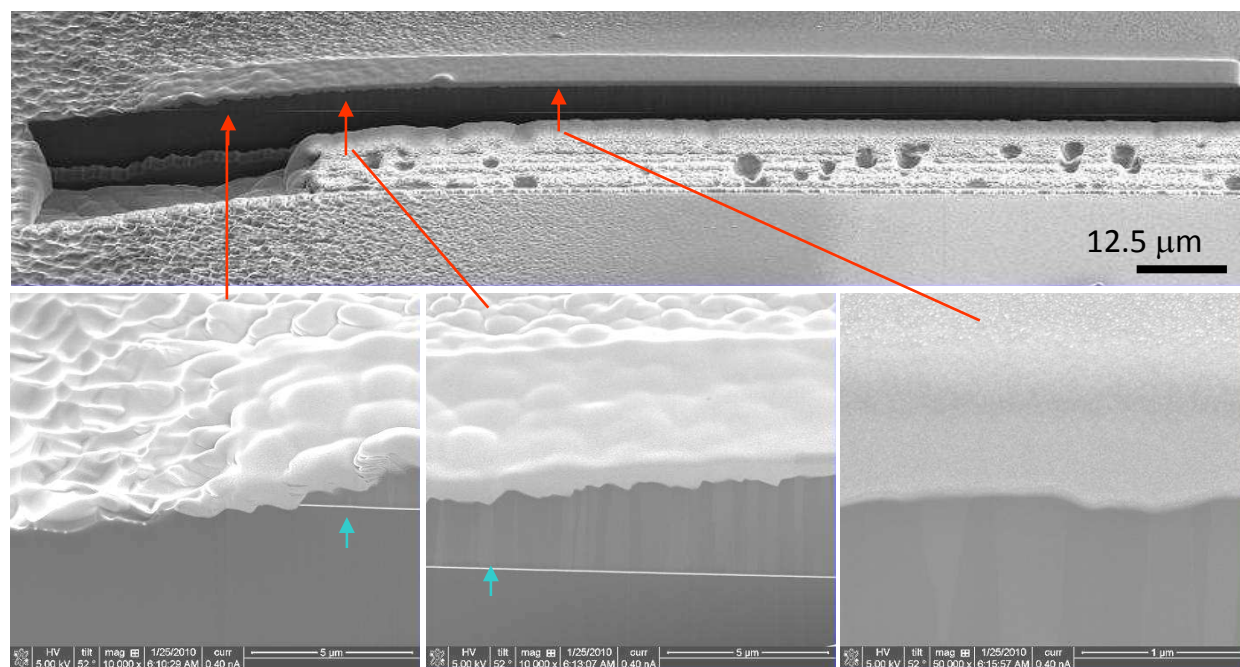


FIG 3. SEM image of a FIB cut that was made going from the crater (left side) to a region of the sample that was not perturbed by the ToF-SIMS erosion (top). The bottom images were taken at various distances in from the side wall of the ToF-SIMS crater (bottom). The bright line (indicated by the light blue arrows in the two lower panels) represents the Au layer (see FIG 1).