Depth Profiling of Thin Films Using the fibTOF for FIB-SIMS Measurements

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Thin films with desired mechanical, electrical, and optical properties are ubiquitous in modern life, with applications ranging from simple protection or antireflection coatings to hard layers for tools and complex multi-layer structures on semiconductor wafers. Thin films on complex substrates are also increasingly important in clean energy storage applications such as lithium-ion batteries. The deposition of such advanced thin films, especially on small parts with complex shapes, is a challenging process requiring complex development and verification activities. The use of a fibTOF secondary ion mass spectrometer can accelerate this development by providing information about the elemental composition of the layer structure. This information concerns the chemical homogeneity both at the surface and in buried layers (such as an adhesion layer), the detection of contaminants (often present at the interface between two layers) and the sharpness of interfaces between layers. Although secondary ion mass spectrometry has long been used to analyze thin films [1], the fibTOF advances the method by providing high resolution chemical images and does not require choosing the elements of interest before analysis. In this application note, we present two examples of inorganic thin film measurements using a fibTOF.

Example 1: A Vertical Cavity Surface Emitting Laser

Figure 1 shows a depth profile through part of a vertical cavity surface emitting laser (VCSEL) that was constructed from five gain regions using epitaxially bonded AlGaInAs-InP and a distributed Bragg reflector of many alternating GaAs-AlAs layers. For a description of the structure and operation of such devices, see Iga [2]. Figure 2 shows an enlargement of the aluminum depth profile, superimposed

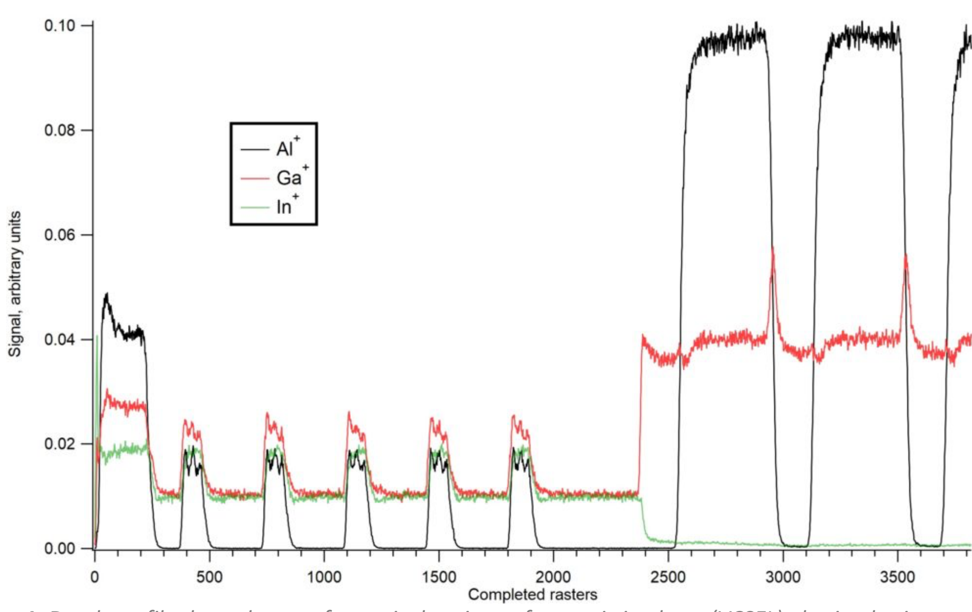

__Figure 1.__ Depth profile through part of a vertical cavity surface emitting laser (VCSEL) obtained using Tofwerk's fibTOF with a gallium ion focused ion beam microscope. For optimal depth resolution, the focused ion beam was used with a low energy of 3 keV. The various layers in the semiconductor stack can be clearly seen although the peak of the Ga+ signal between each Bragg reflector pair to the right of the image is an artefact of the sputtering process. The x-axis is given in completed rasters of the FIB microscope over the selected area; note that this is strictly proportional to depth if and only if the sputtering rate is constant (unlikely in a depth profile through different materials) and the current of the FIB beam is constant.

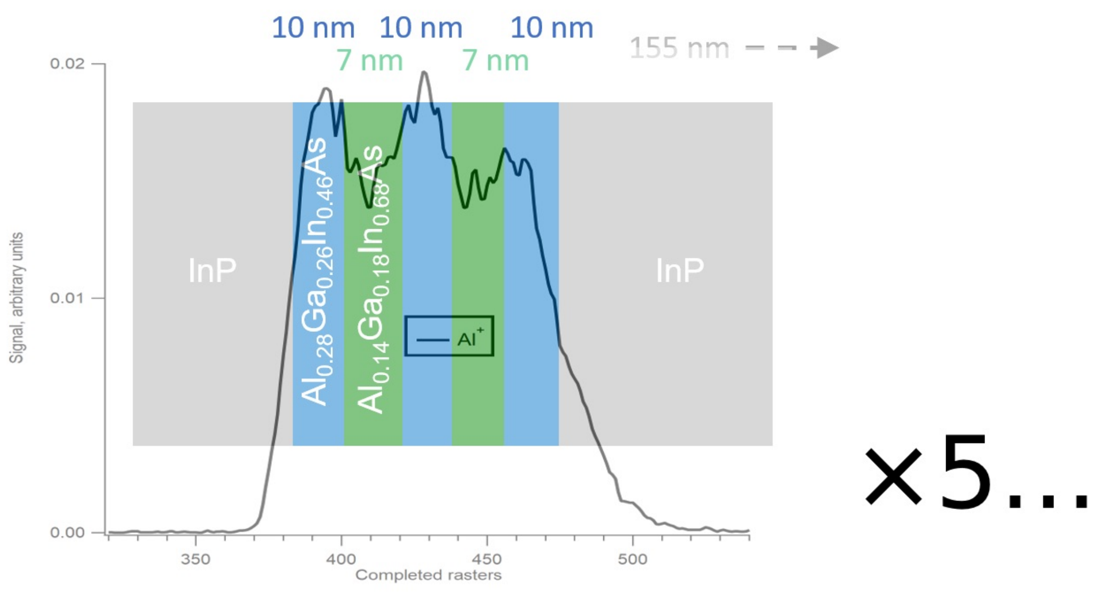

__Figure 2.__ A zoom of part of the Al+ trace from Figure 1 (black line) superimposed on a schematic indication of the thin film layers that define the quantum well gain region of the VCSEL device. There are five such regions (see Figure 1) each consisting of three more aluminum-rich layers (indicated in blue) are each 10 nm thick, separated by 7 nm of a layer with less aluminum (indicated in green). The trace from the fibTOF SIMS measurements shows three peaks in the aluminum concentration as expected.

on the structure of the device, showing structure within the gain regions.

The depth profile was acquired using a fibTOF instrument for the measurement of secondary generated by a focused ion beam microscope (in this case using Ga+ ions), using the same VCSEL sample reported by Whitby et al. [3]. Depth resolutions better than 8 nm have been demonstrated, and lateral resolutions better than 25 nm. It is important to recognize that even thinner layers of nanometer thicknesses, such as adhesion layers of chromium, titanium or titanium oxide, can be detected.

Example 2: Confirming the Presence of an Adhesion Layer in a Thin Film Stack

In this example, measurements from a fibTOF were used to answer the question as to whether or not an adhesion layer (thought to be titanium and/or platinum) was present at the interface between a gold coating and a lithium tantalate substrate.

The presence of a platinum adhesive layer between the gold coating and the substrate was confirmed. With this instrument, the signal from gold at m/q 197 could not be completely resolved from a signal due to TaO^+, and the other isotopes of TaO were in too low an abundance to be helpful. However, the *a priori* information about the sample composition made the interpretation unambiguous. Although analysis of mass spectral information should be approached with an open mind, it is also important to use any information one has about the sample (or typical contaminants in the measurement system). Another technique that can be seen in the figure is the use of the adduct ion signal from $GaPt^+$ which in this material was stronger and at less risk of isobaric interferences than the Pt^+ ion signal.

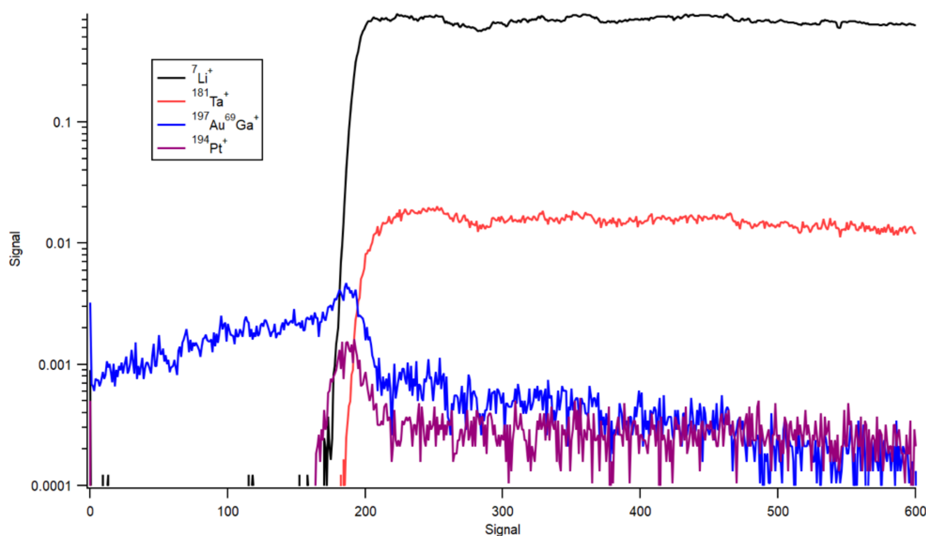

Figure 3. *Positive secondary ion depth profiles for gold coated lithium tantalate.*

Tips for the Best Depth Profiles

- For the best depth resolution, the focused ion beam should have the lowest energy possible without increasing the spot size so much that the shape becomes low quality.
- When producing a depth profile from the acquired data, it is important to adjust the lateral margins of the region of interest to that only data from the center of the pit is used, therefore avoiding milled pits with sloping walls.
- The floor of the crater should remain flat, often requiring some adjustments to the focused ion beam, pixel size, and scan rate or beam current.
- Some materials, under some conditions, can roughen or develop a wave-like surface when they are milled by a focused ion beam [4]. This will degrade the depth resolution but can be addressed by slightly altering the stage angle, changing the beam conditions, or by using an assist gas .
- If a larger sampling area is desired, measure in several places to ensure that the thin film is homogenous (e.g. at the center and edges of a wafer, or near and far from an ALD precursor inlet nozzle).

Complementary Techniques

Complementary methods to FIB-SIMS for investigating thin films composition and structure include X-ray reflectance, X-ray fluorescence, ellipsometry, or glow-discharge profiling — so long as the layers are homogenous on a scale of many millimeters. Making a cross-section of the thin films (either mechanically or with the FIB) can provide insight into the layering structure. For thicker films, techniques such as EDX or (near-field) Raman microscopy may be used to obtain information about composition and structure [5]. To obtain accurate information about the depth of features observed in the depth profile, an atomic force microscope can be used to measure the depth of FIB pits.

References

[1] Zinner, E. Depth profiling by secondary ion mass spectrometry. *Scanning 3*, 1980. DOI: 10.1002/sca.4950030202

[2] Iga, K. Surface-emitting laser — its birth and generation of new optoelectronics field. *IEEE J. Sel. Top. Quant.*, 2000. DOI: 10.1109/2944.902168

[3] Whitby, et al. High spatial resolution time-of-flight secondary ion mass spectrometry for the masses: a novel orthogonal ToF FIB-SIMS instrument with in situ AFM. *Adv. Mat. Sci. Eng*, 2012. DOI: 10.1155/2012/180437

[4] Shenoy, V. B. .; Chan W. L.; Chason, E. Compositionally Modulated Ripples Induced by Sputtering of Alloy Surfaces, *Phys. Rev. Lett.*, 2007.
DOI: 10.1103/PhysRevLett.98.256101

[5] Gucciardi, et al. Near-Field Raman Spectroscopy and Imaging*. Applied Scanning Probe Methods*, Springer, Berlin, Heidelberg, 2006.
DOI: 10.1007/978-3-540-37316-2_10

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