

# Quantification of layer composition in compound semiconductors

## Keywords

GCIS depth profile, vertical cavity surface emitting laser (VCSEL), single photon LED (SPLED)

## Application Note MO442(A)

Further application/technical notes are available online at:  
[www.kratos.com](http://www.kratos.com)

## Introduction

Compound semiconductors are the key underpinning technology in optoelectronics, and also used in electronic applications with specialist requirements (e.g. power). The ability to engineer the electronic and optical properties of compound semiconductor alloys, for example in terms of their alloy composition, which may be binary, ternary, quaternary or quinary, and grow multiple layers of different semiconductor alloys on top of each other (heterostructures), is a key part of their success. Indeed, the global compound semiconductor market was worth \$66bn in 2016, and predicted to be worth \$143bn by 2023<sup>[1]</sup>. However, despite the clearly important role of composition in compound semiconductors, its accurate determination remains a challenge, especially in devices where there are many different layers.

Excellent and extreme examples of this are devices that contain distributed Bragg reflectors: alternating layers of high- and low-refractive-index material (typically GaAs/Al<sub>x</sub>Ga<sub>1-x</sub>As) to create a stop-band where a very particular set of wavelengths are almost fully reflected (ideally over 99.9%). For example, vertical cavity surface emitting lasers (VCSELs) are tiny (low-cost) semiconductor lasers that use a pair of DBRs to form the mirrors of the lasing cavity. VCSEL demand for their traditional application of communications continues to rise, while the non-communication market is expected to increase nearly 8-fold by 2022<sup>[2]</sup>. In VCSELs, the quality and consistency of the DBRs is important, as a VCSEL has a gain length on average 10<sup>5</sup> times smaller than an edge-emitting laser, and therefore needs ultra-high reflectivity mirrors to achieve a reasonable threshold current<sup>[3]</sup>. Examples of other, emerging, devices that use DBRs are single photon LEDs (SPLEDs); these are needed for quantum key distribution in quantum cryptography networks<sup>[4]</sup>.

This study focuses on these DBRs, and methods to accurately characterise their structure, including determining whether the semiconductor layer growth has proceeded as desired. X-ray photoelectron spectroscopy (XPS) depth profiles are taken to measure the chemical composition of the DBR layers to further characterise the growth. Even a small change in Al composition affects the refractive index, thus changing the optical path length of the layer, with consequences for everything from mirror characteristics to laser output wavelength. XPS yields quantitative information regarding Al content for the DBR structure, which directly relates to device performance.

## Experimental

The SPLED structure was grown by molecular beam epitaxy at Lancaster University, UK [5]. XPS analysis was performed using the Kratos AXIS Nova spectrometer. The Minibeam IV focussed ion gun was used for depth profiling in 4 keV Ar<sup>+</sup> mode. This ion source has a bend in the ion column to remove energetic neutrals which could degrade the interface resolution of this multilayer sample. Sample rotation using 90° increments at each etch cycle was used to minimise sample roughening during the etching. Compucentric rotation about a point during etching is easily implemented by a simple tick-box in the ESCAPE acquisition software.

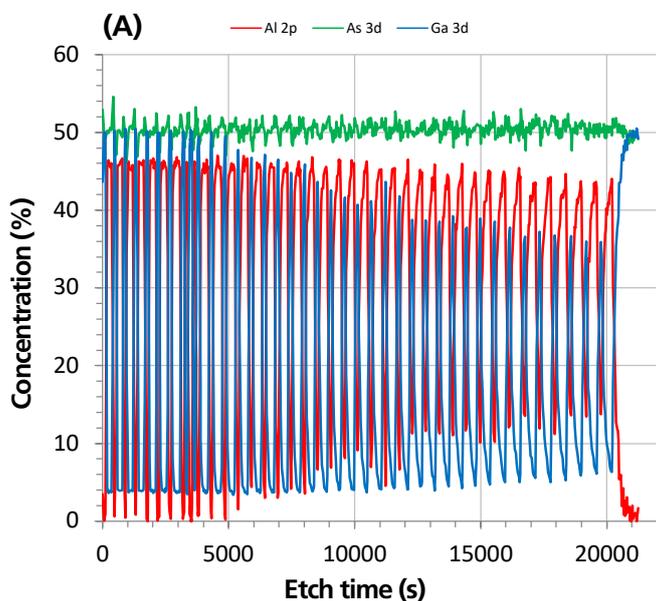


Figure 1A: Complete depth profile of SPLED structure.

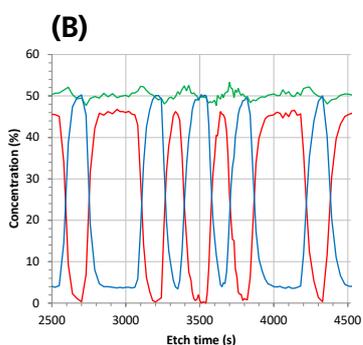


Figure 1B: zoomed-in emitter region.

## Results

The SPLED wafer was mounted onto the rotation platen and loaded into the instrument. Depth profiling was performed using high-energy monatomic 4 keV Ar<sup>+</sup> ions with the focussed beam

## Related documents available online:

  Smart phone screen - depth analysis of alkali and alkali earth metals Applications note MO427	  Applications note: Reduction in preferential sputtering of TiO <sub>2</sub> Applications note MO403
---	--

[Click to download](#) 

[Click to download](#) 

rastered across the surface to form a uniform etch crater. Fast (15s) XP spectra were acquired after each etch cycle to determine the composition of the newly exposed surface. Small-spot spectroscopy was used to limit the influence of crater edge effects on the interface resolution. Figure 1 shows the complete depth profile for the SPLED structure. The alternating GaAs/Al<sub>x</sub>Ga<sub>1-x</sub>As layers can be seen by the dramatic variation in Al content. In this structure the Al<sub>x</sub>Ga<sub>1-x</sub>As layers are designed to have an Al fractional content, *x*, of 0.9 (elemental content of 45%) - here the XPS quantification shows a consistent value of ~46% for the Al<sub>x</sub>Ga<sub>1-x</sub>As layers well within the accuracy of the technique. Importantly, the composition of the emitter layer (see Figure 1B) is seen to be consistent with the desired composition. After etching through the material for several further mirror layers the profile begins to lose form. This is most probably due to blending and roughening of layers due to the ion etching process. For this mode of etching this is a known issue. Unfortunately due to the total thickness of the layers it was not feasible to repeat the experiment with low energy ion (250-500eV) due to the significant increase that would cause in experiment time.

Post-acquisition XP images were acquired for the Al 2p and Ga 3d photoelectron lines to determine the etch crater shape and positioning (Figure 2). The images clearly show the layer structure of the device and the presence of the emitter layer 8 repeat units into the device.

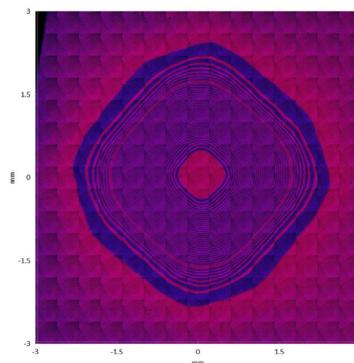


Figure 2: Stitched XPS image of etch crater; Ga (RED) Al (BLUE).

## Conclusion

XPS is an enabling technology which yields empirically quantitative information regarding the composition of compound semiconductor alloys. Here we analyse novel SPLED structures to characterise Al content. Using conventional depth profiling methods with Shard step-wise rotation we were able to depth profile a full device. The Al content was seen to agree with expected compositions for the different layers and will be useful for modelling the performance of cell structures.

## Acknowledgements

Many thanks go to Tom Wilson, Peter Hodgson, Alex Robson and Manus Hayne for sample preparation and fruitful discussions, who would like to acknowledge EPSRC (grant number EP/P034233/1) and to IQE plc for financial support, through a CASE Award for Tom Wilson.

## References

1. [alliedmarketresearch.com/compound-smiconductor-market](http://alliedmarketresearch.com/compound-smiconductor-market), accessed 2/2/2018.
2. [yole.fr/IR\\_LED\\_LASER\\_MARKET\\_TRENDS.aspx#.WnSRQkILFaQ](http://yole.fr/IR_LED_LASER_MARKET_TRENDS.aspx#.WnSRQkILFaQ), accessed 2/2/2018.
3. P. Moser, "Energy efficient oxide confined VCSELs for optical interconnects in data centers and supercomputers", PhD thesis, Technische Universitt Berlin, Fakultt II - Mathematik und Naturwissenschaften (2015).
4. [paneuropeannetworks.com/science-technology/quantum-rings-to-rule-them-all/](http://paneuropeannetworks.com/science-technology/quantum-rings-to-rule-them-all/), accessed 2/2/2018.
5. [lancaster.ac.uk/physics/research/experimental-condensed-matter/quantum-nanotechnology/](http://lancaster.ac.uk/physics/research/experimental-condensed-matter/quantum-nanotechnology/), accessed 2/2/2018.