High-accuracy (0.1 ± 0.2 nm) analysis of $Al_xGa_{1-x}As$/GaAs layers using beam-exit cross-sectional polishing and selective etching

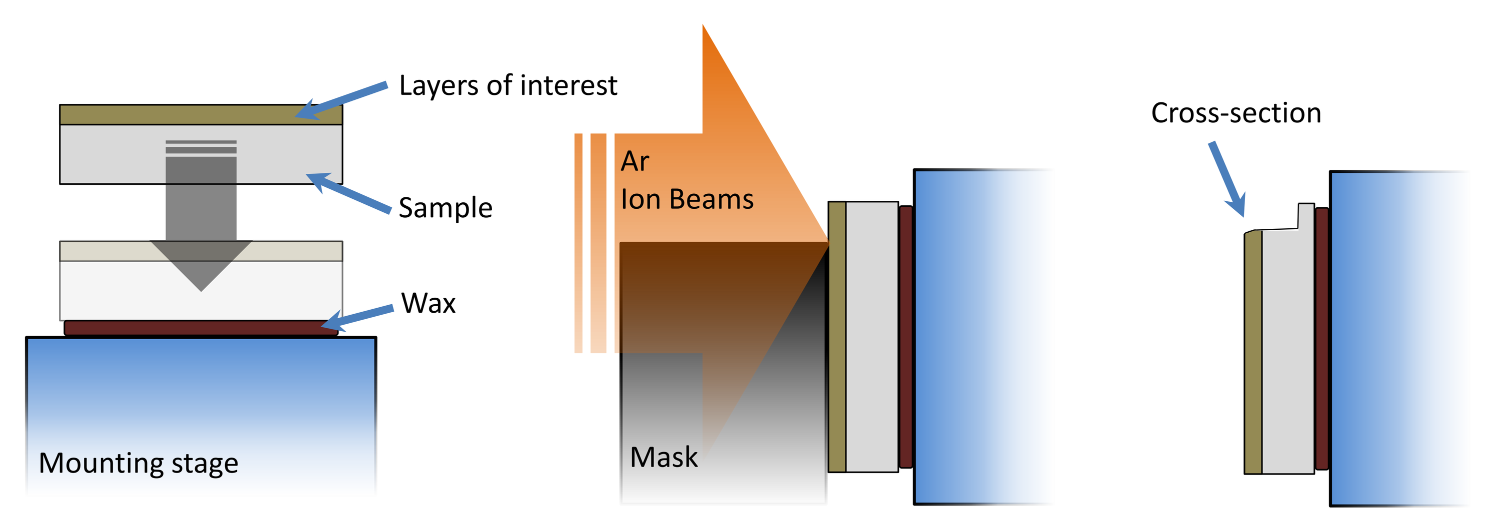
A. J. Robson,*[1] I. Grishin,[1] R. J. Young,[1] A. M. Sanchez,[2] O. V. Kolosov[1] and M. Hayne[1]
[1]Department of Physics, Lancaster University, Lancaster, LA1 4YB, UK
[2]Department of Physics, University of Warwick, Coventry, CV4 7AL, UK
***Email: a.robson@lancaster.ac.uk**

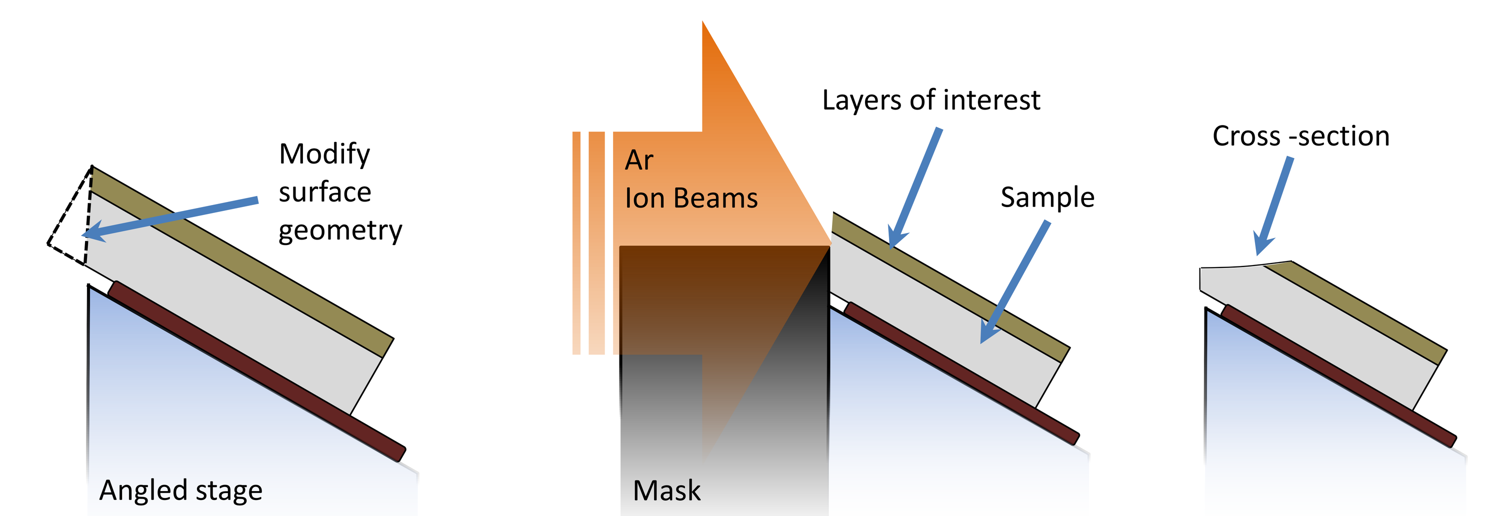
LANCASTER UNIVERSITY

Microscopy of semiconductor heterostructures on the sub-nm scale is presently only possible with techniques such as transmission electron microscopy (TEM), cross-sectional scanning tunneling microscopy and atom probe tomography, which are expensive, of limited availability and require considerable expertise. Here we report the use of a novel sample preparation technique, **beam-exit Ar-ion cross-sectional polishing (BEXP)** [1,2], which, when combined with scanning probe microscopy (SPM), has the potential to provide easy access to high-resolution microscopy of semiconductor nanostructures [3].

Conventional Ar-Ion Beam Cutting

The ion beam enters through the sample surface. However, this results in an area of a few micrometres at the beam entry which is unsuitable for scanning. As semiconductor nanostructures are often buried in the top hundreds to thousands of nanometres, the cut quality in this region is of utmost importance.





Beam-Exit Ar-Ion Cross-Sectional Polishing

The sample position is rotated, so that rather than entering through the top, the ion beam impinges upon the side of the sample at a shallow angle and exits the surface far from the masking plate.

This beam-exit point exhibits a much lower true nanometre scale surface roughness than the area close to the mask, making it suitable for SPM.

As BEXP cuts at an angle, the layers within the sample are "stretched out" over a larger area, allowing easier determination of small structures.

Methodology

Test samples with a variety of $Al_xGa_{1-x}As$/GaAs layers were grown using solid source molecular beam epitaxy (MBE). $Al_xGa_{1-x}As$ composition x varied between 0.2 and 1.0.

BEXP was performed in a Leica EM TIC020 ion beam cutter. After BEXP, the samples were allowed to oxidise before imaging.

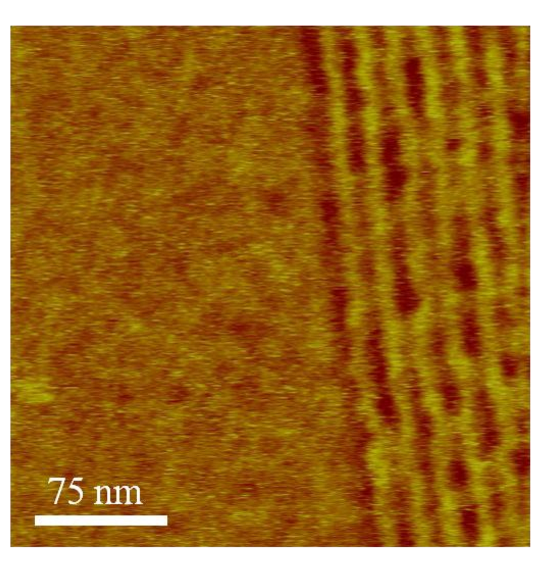
Samples were analysed using a Digital Instruments Multimode SPM in both tapping mode AFM and ultrasonic force microscopy (UFM).

Sample Contrast

Oxidised $Al_xGa_{1-x}As$ layers protrude above GaAs layers by an amount which varies with Al content x, allowing identification during imaging [4].

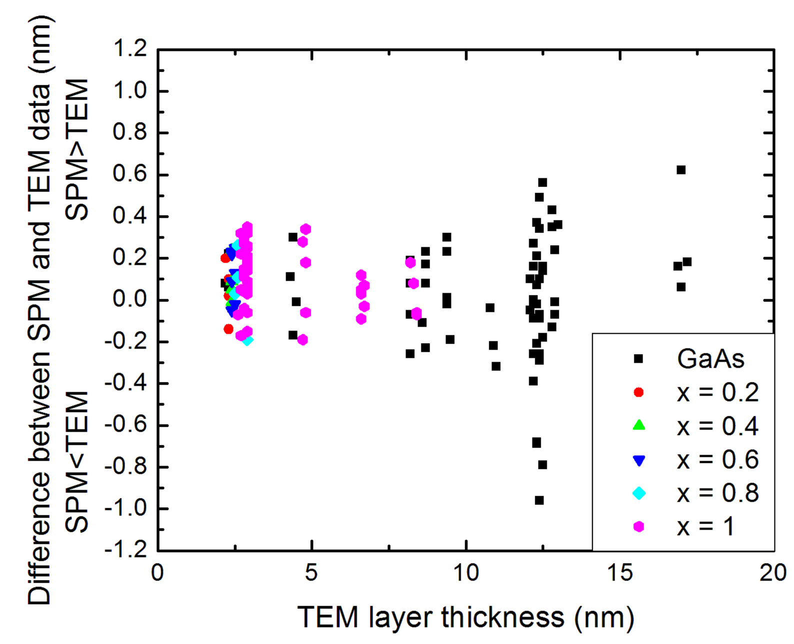
Contrast was enhanced via a 4:1 citric acid/hydrogen peroxide etch [5].

Figure shows a UFM image of a 2-nm AlAs/GaAs superlattice. Dark layers are AlAs, whilst brighter layers are GaAs.



Comparison with Transmission Electron Microscopy

Measurements for thin layers were made on 750 nm images produced at 512 samples/line. Thicker layers required larger scan sizes. Results were then compared with a TEM analysis of the same structure in a Jeol 2000FX TEM.



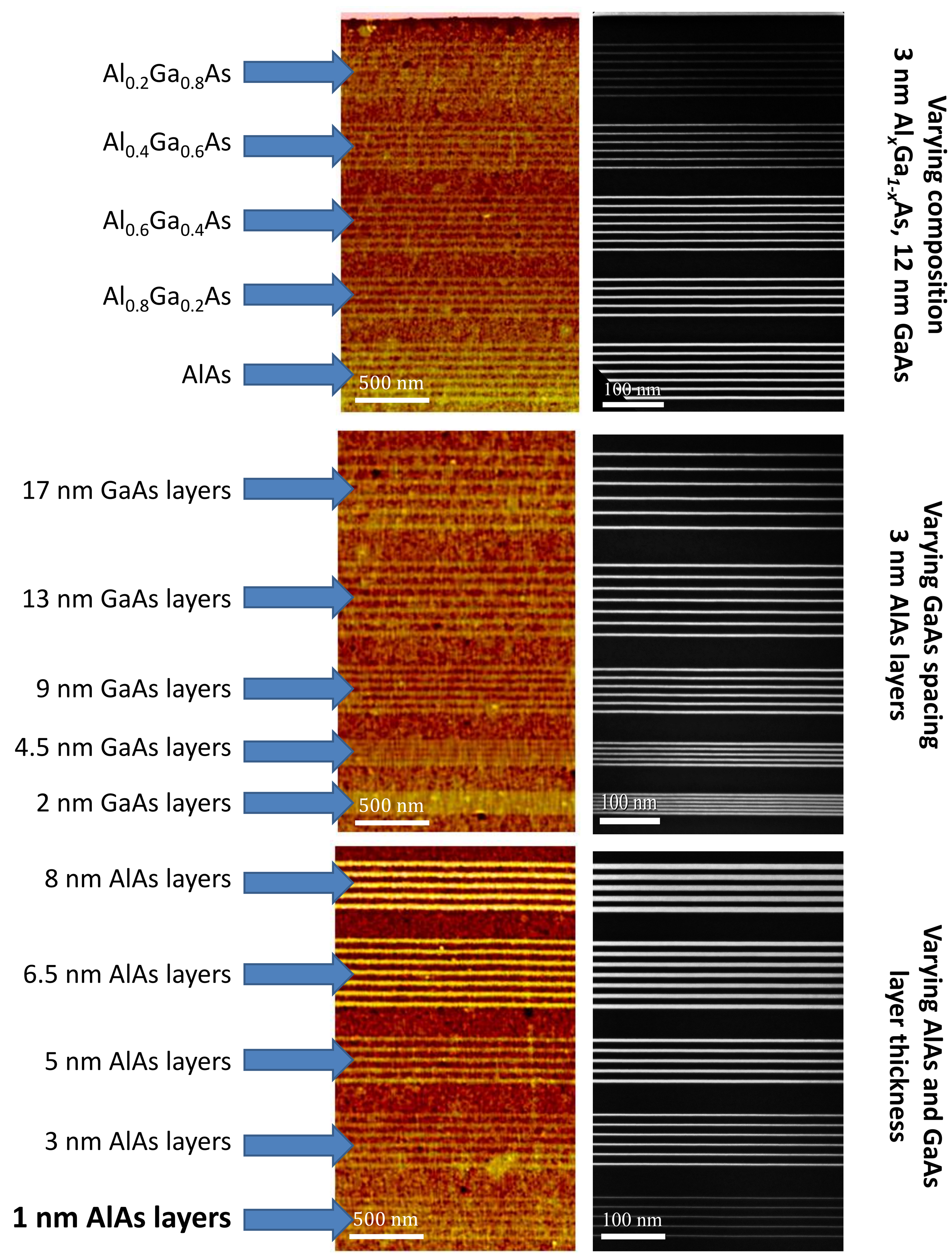
167 layers were analysed, the majority below 20 nm in thickness.

SPM measurements are very close to TEM values: for layers under 20 nm the difference is 0.1 ± 0.2 nm

Thicker layers display less absolute accuracy, though increased percentage accuracy.

Comparative SPM and TEM Images

Images have been scaled to produce a comparative view of BEXP-SPM and TEM.



Conclusions

Beam-exit Ar-ion cross-sectional polishing produces a cross-section through semiconductor samples with roughness on the atomic scale, making it suitable for qualitative and quantitative analysis of nanostructures with SPM.

We combined this technique with a light citric acid/ hydrogen peroxide etch, to image $Al_xGa_{1-x}As$/GaAs superlattice layers with a thicknesses as low as 1 nm. Over 167 thickness measurements were compared with TEM analysis of the sample. SPM results are within 0.1 ± 0.2 nm (1 ± 2 %).

BEXP-SPM thus shows great promise for the analysis of semiconductor heterostructures, especially devices with multiple layers such as vertical cavity surface emitting lasers and quantum cascade lasers.

References & Acknowledgements

[1] O Kolosov and I Grishin, Patent WO/2011/101613, (2011).
[2] O V Kolosov, I Grishin and R Jones, *Nanotechnology* **22** (2011), 185702.
[3] A J Robson et al., *ACS Appl. Mater. Interfaces* **5** (2013), 3241.
[4] F Reinhardt, B Dwir and E Kapon, *Appl. Phys. Lett.* **68** (1996), 3168.
[5] G C DeSalvo, W F Tseng and J Comas, *J. Electrochem. Soc.* **139** (1992), 831.
This work was supported by the Engineering and Physical Sciences Research Council

EPSRC Engineering and Physical Sciences Research Council