

### Project Aims

- To inspect some of the effects of sputtering† on highly flat layers of Organic Semiconductor materials and soft materials
- To prepare highly flat layers of sample materials
- Measurement of step heights of the sputtered etches using Scanning Electron Microscope (SEM), White light Interferometry and Alicona Optical Profiler

### Introduction

Inspecting the effects of sputtering on Organic Semi-conductor materials and soft materials required the preparation of flat layers of such materials with very low surface roughness, appropriate for producing well defined etches using Argon gas ion beam sputtering. Measuring around the micron scale required the use of different instruments in conjunction, each with its own functions and limitations. While instruments like the White light interferometer are used for measuring step heights ranging from tens of nanometers, it requires a certain amount of sample preparation and suitable sample conditions like low roughness. The Alicona optical profiler is an optical system which could not be expected to provide highly accurate measurements at such scales consistently. The '3D Image Viewer' software on SEM images was tested for its accuracy, operation of software and calibrations before applicable measurements were achieved using it.

### Method

#### Step height measurement accuracy on soft materials using silicone calibration on '3D Image Viewer' software on SEM

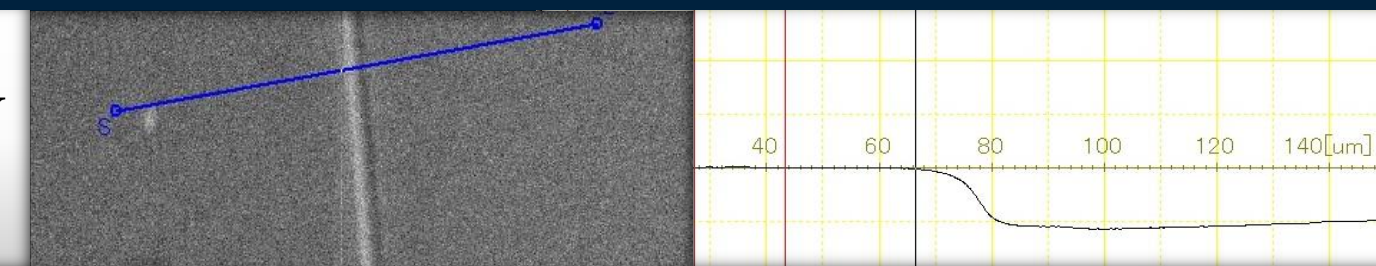
3D Image Viewer software allows the generation of 3-D models of samples using 4 directional images captured by 4 backscattered electron detectors of the SEM. These models can be used to measure surface roughness and step heights. To test the accuracy of measurements from the software, profiles of samples were measured with different iterations

### Testing flat layer formation using Mica, Field's metal and Polycaprolactone (PCL) melts and Glass slide arrangements.

Droplets of Field's metal were flushed with the Mica surface and allowed to solidify, therefore obtaining mica like surface on Field's metal. AlQ3 solution in Dichloromethane (DCM) was tested for layer formation on flat Field's metal using micropipette.

Layer formation was tested using PTFE tape mounted on microscope slides as well as Silicone release agent spread on microscope slides, both arranged in a wedge shape using wires of 100-300 micron diameter. Flat layers of organic electronic materials were obtained using Mica layer mounted on glass slides.

SEM Step height at x500 magnification using 15kV accelerating voltage.



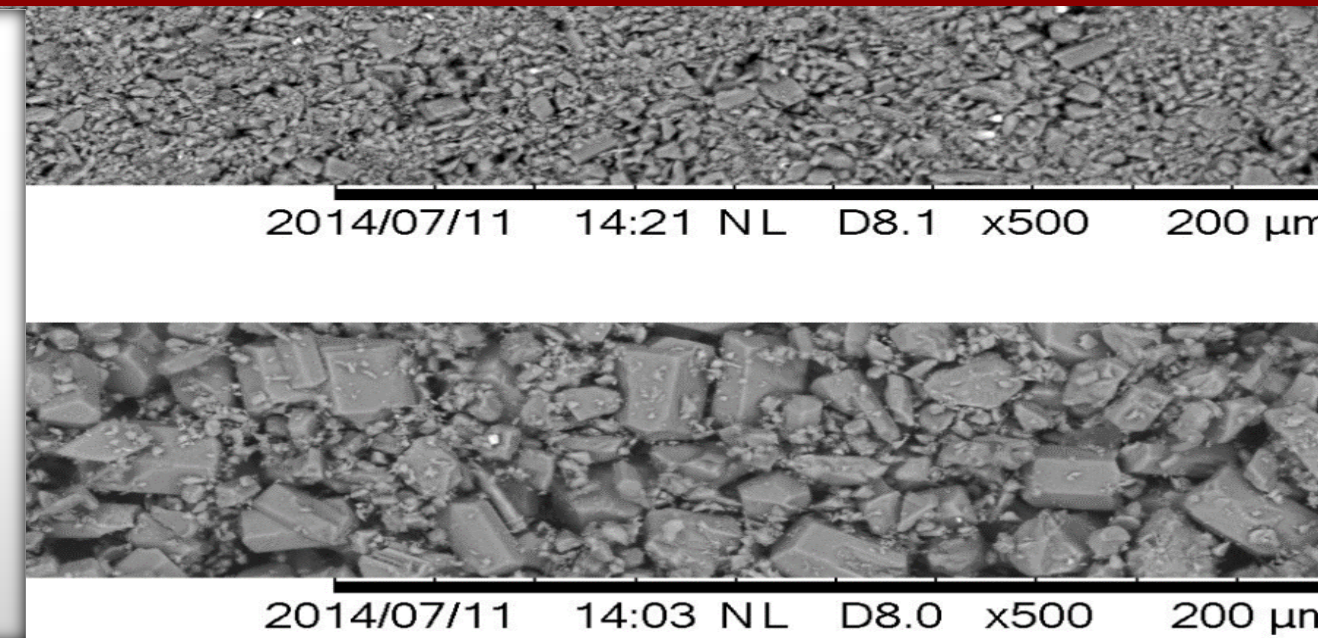
### Measurement of step heights

Most layers used for sputtering were obtained by slow cooling meniscus flushed with mica surface placed in oven at up to 85°C. The exposure conditions for sputtering included a cluster size of 1000 atoms with cluster energy 4000kV and exposure time of 7000s.

Layers undergoing successful sputtering were then analysed and step heights measured using the three instruments. For measurements using the Interferometer, Gold vapour deposition was carried out on relevant samples.

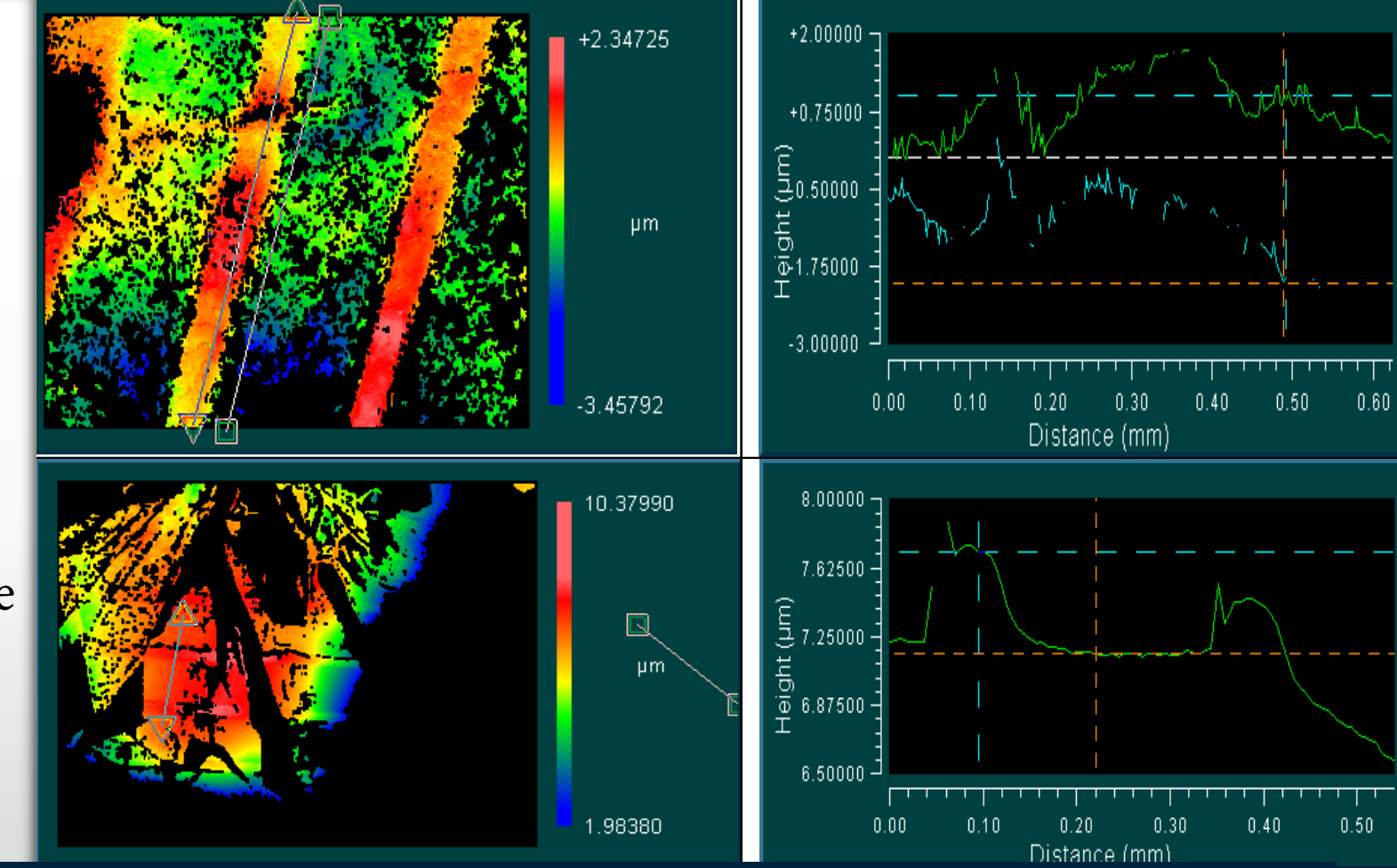
### Results

1. By placing the samples in a vacuum oven at <10mbar pressure, the drying up process is accelerated and therefore crystal growth is arrested. This causes the formation of layers with lower surface roughness



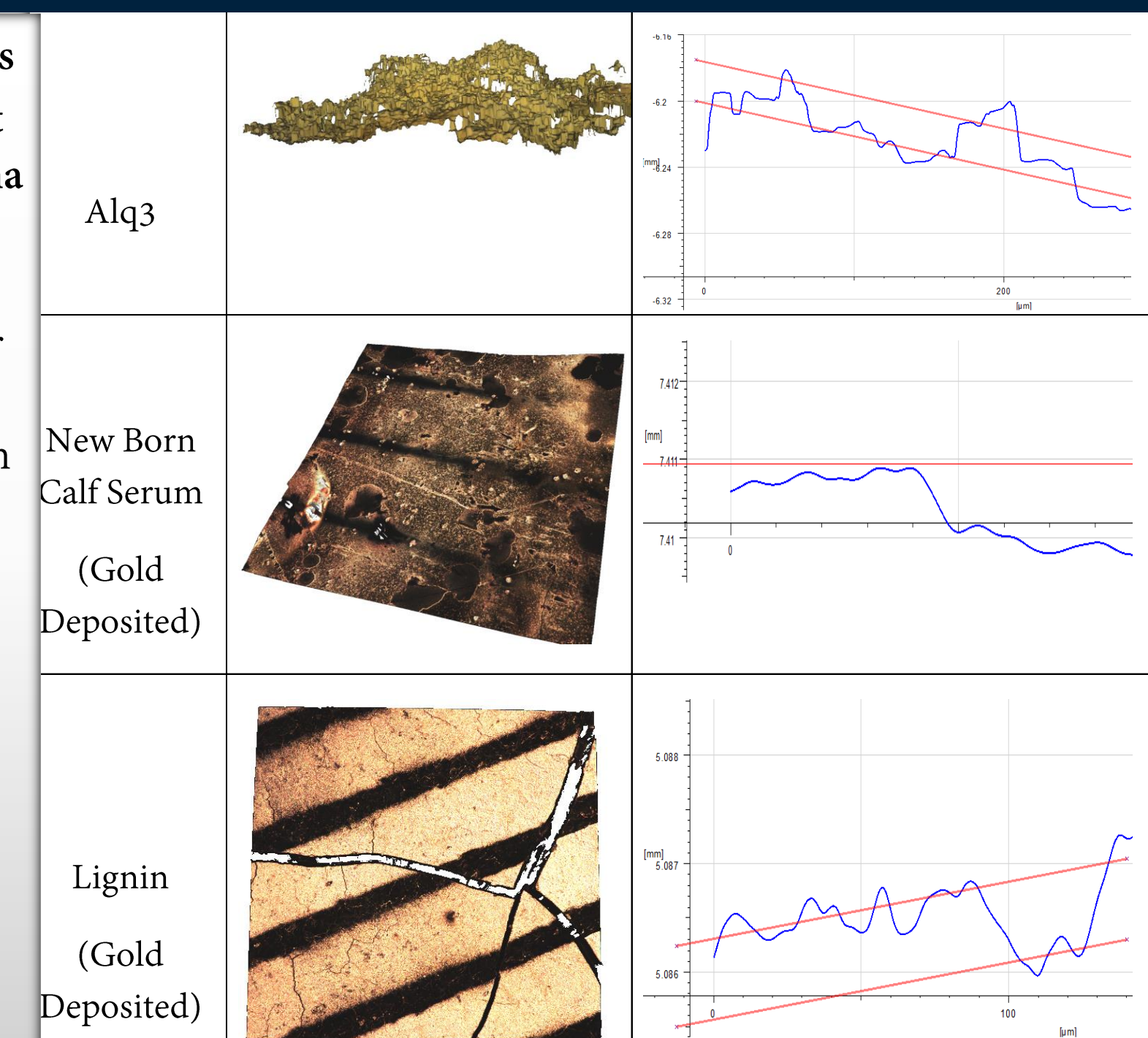
2. The depth accuracy measured on standard samples was ± 20% of the reference value. Measurement performance depends on calibration of the software for a given specimen & the observation conditions like magnification, accelerating voltage and working distance Step heights measured across up to ± 50° to the perpendicular to the step gave accurate results on '3D image Viewer' software

3. Interferometer Profiles and Measurements on Glucosamine (top) & Sucrose Helped in reaffirming the results obtained from the 3D image viewer software and the Alicona Profilometer



### 4. Sample profiles and measurement profiles on Alicona Profiler

Alicona optical profiler is used for measurement of form and finish on micro structured surfaces. As an optical system, it could be used for producing true images of the samples and 3D models of profiles as well as making measurements



### Conclusion

- This project helps provide data for analysis of polymers, soft materials and biomaterial surfaces without which it would be difficult to characterise new materials for e.g. organic photovoltaic cells or medical implants, among many other applications
- Progress was made in sample preparation and measurement techniques at micron scale through testing a series of suitable methods for investigating sputtering of organic semiconductors and other soft materials

† **What is Sputtering?** Sputtering is a process whereby atoms are ejected from a solid target material due to bombardment of the target by energetic particles. The sputtering process was carried out on selected layers using the ion gun of K-Alpha X-Ray Photoelectron Spectrometer, collecting data about the exposure conditions and the process.

**NEXUS** is the UK's National EPSRC XPS Users' Service, hosted by nanoLAB at Newcastle University. X-ray Photoelectron Spectroscopy, is one of the principal methods of probing the composition and electronic structure of surfaces, with research applications in electronics, semiconductor physics, novel materials and biomaterials, surface chemistry and functionalisation, sensor surfaces, adhesion, abrasion and tribology.

