

Report

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Measurement of single and multi layer film coatings for medical implants

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Measurement of single and multi layer thin film coatings for medical implants

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ABSTRACT:

A novel instrument for the measurement of polymer film coatings is reported and evaluated. The device uses a measurement technique based upon conventional beam profile reflectometry but that has been adapted to work on highly curved surfaces such as those found on medical implants. The instrument was used to measure polymer film thickness on a range of test artefacts with varying film thickness and surface curvature. The results obtained were then compared with those made at the National Physical Laboratory using alternative measurement techniques to assess the accuracy of the measurement system.

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Table of Contents:

1. Background	7
2. Introduction	7
3. Principles of calibration/experimental techniques	8
3.1 Beam profile reflectometry	8
3.2 Artefacts	11
3.2.1 Different artefacts and the reason for them	11
3.2.2 Curvature	11
3.2.3 Material	12
3.2.4 Development of coating technique	12
4. Measurement of artefacts at NPL	14
4.1 Trench cutting	14
4.1.1 Focused ion beam (FIB)	14
4.1.2 Laser Machining	18
4.1.3 Masking	19
4.2 Systems used to measure the trenches at NPL	20
4.3 Suitability and problems of using each system	21
4.3.1 AFM	21
4.3.2 LEXT confocal microscope	23
4.3.3 SWLI	26
4.4 Measurement and results of NPL measurements	29
5. Measurement of artefacts with BPR	32
5.1 Initial measurements	32
5.2 Re-measurement of NPL standard samples	34
6. Analysis of measurements	35
7. Conclusions	36
8. Possible future work	37
9. Acknowledgements	38
10. References	38
11. Appendices	38
Appendix I: Development of the EOS BPR device and 4 day consultancy	39
Appendix II: Supplementary data and analysis from the re-measuring of the artefacts	45

Table of Figures:

Figure 1. Schematic diagram of BPR apparatus.....	9
Figure 2. SEM images of two different coating surfaces and their roughness.....	13
Figure 3. Current leakage to the stage during the FIB process.....	15
Figure 4a and b. SEM images of the 50 μm wire with the FIB trenches cut through.	16
Figure 5. Damaged edges make the coating surface level difficult to identify and measure.	17
Figure 6. Large deposits covering the trench base make any depth measurement unusable... ..	18
Figure 7a, b & c. Show typical examples of the Oxford laser trenches & measurements. a) shows the damage to the surrounding polymer, b) shows the unevenness of the trench base, while c) shows a measurement cross section , by the LEXT confocal microscope system, displaying problems with the measurement of a trench.	19
Figure 8. AFM image, slight movement with an AFM measurement of a wire can be seen.	22
Figure 9. AFM image of the centre of a trench and the edge of the sloped side.	22
Figure 10. LEXT measured image of one side of an artefacts trench.	23
Figure 11. Height map from a LEXT measurement, of a tube artefact.	23
Figure 12. Measured height profile of a plane along an artefacts trench	24
Figure 13. LEXT scan of a trench step.	25
Figure 14. LEXT scan of a trench step, showing, in red, the plane of measurement.	25
Figure 15. SWLI measurement of a sample trench.	26
Figure 16 a and b. 3dimentional measurement and profile of a sample and trench	27
Figure 17a. b. and c. SWLI measurement images of the gold plated sample.....	29
Figure 18a and b. A further example of a measurement made with the SWLI, showing the surface roughness and step measurement.	30
Figure 19. A SWLI image showing some of the noise experienced around the edge of the step, possibly due to optical effects similar to those mentioned in [3].....	31
Figure 20a and b. The difficulty in measuring wire samples with the SWLI.....	31
Figure 21. The software fit of a thick coating on a 1mm tube sample, giving a thickness of $14886 \text{ nm} \pm 600 \text{ nm}$ and $n = 1.65 \pm 0.10$	33
Figure 22. Shows the optical fringe image and software fit of a 2mm tube sample, giving a thickness of $4999 \text{ nm} \pm 300 \text{ nm}$ and $n = 1.73 \pm 0.12$	33
Figure 23. Shows measurements made using the BPR of a 2 mm diameter tube, (sample 2), clear and sharp fringe patterns are visible, giving a thickness of 7512 nm and $n = 1.717$	35
Figure 24. Shows measurements made using the BPR of a 1mm diameter tube, (sample 1, thick coating), giving a thickness of 23752 nm and $n = 1.790$	35
Figure 25. Picture of the BPR instrument	40
Figure 26. Optical set up of the device.....	41
Figure 27. Example of light intensity, to stage position, and variation between lenses.....	42
Figure 28. Sample 1, 2 mm tube.....	47
Figure 29. Sample 1, 1 mm tube.....	47
Figure 30. Sample 1, 50 μm wire.	47
Figure 31. Sample 2, 2 mm tube.....	48
Figure 32. Sample 2, 1 mm tube.....	48
Figure 33. Sample 2, 50 μm wire.	48
Figure 34. Sample 3, 2 mm tube.....	49
Figure 35. Sample 3, 1 mm tube.....	49
Figure 36. Sample 3, 50 μm wire.	49
Figure 37. Correlation of NPL to final N- EOS results.	50

1. Background

Stents are metallic-slotted tubes that are widely used in clinical practices as implantable devices in coronary and peripheral arteries in order to maintain blood flow through diseased vessels. However the major challenge in coronary stenting has been the high rate of re-narrowing of the blood vessel (restenosis), which is documented to be 20% to 30%. More recently drug-coated stents, which use polymer coating as a carrier to deliver potent therapeutic drugs to reduce restenosis, have substantially improved patient outcome. The drug is incorporated in the polymer and is then released in a controlled manner after insertion of the stent. The use of polymer-coated stents is growing rapidly and has become a very important therapy in interventional cardiology.

2. Introduction

This is a report that describes a joint project between three partners: Nightingale-EOS, Lombard Medical Technologies and NPL, aiming to develop reference, test artefacts to validate a unique ‘bench top’ measurement system capable of making measurements of the thickness and refractive index of thin film coatings deposited on medical implants such as stents and prostheses. The artefacts will enable the monitoring of film thickness and refractive index in a production context. The measurement system and test artefacts will offer significant improvements in measurement speed and cost-effectiveness over such other techniques as are currently available.

All medical implants are subject to varying degrees of rejection by their host. To reduce the rejection of implants, anti-rejection drugs are administered to the host along with anticoagulants. Coating of implants and stents with varying amounts of these drugs is now becoming the favoured method of administration, due to the fact that the area most vulnerable to rejection is the implant area. The total dose and dosage rate administered to the patient can be controlled by varying the concentration of the drug within the coating and the thickness of the coating upon the device; it is expected that both of these parameters will be measurable by the system under development as part of this project. Significant applications also exist for the measurement of inert biocompatible coatings such as amorphous carbon or various metal oxides, particularly on orthopaedic prostheses.

Nightingale-EOS is a start-up optical technology company that has been established to adapt proven semiconductor-industry measurement techniques for biomedical applications. Nightingale-EOS have developed the measurement system, a beam profile reflectometer (BPR), and were required to complete product development of the system, develop application software, refine calibration procedures, and make measurements of the artefacts for comparison with NPL.

Lombard Medical Technologies (LMT) is a specialist cardiovascular device and polymer coatings company which focuses on discovering, developing and commercialising innovative products that address the unmet needs of medical professionals across the world. LMT's polymer coatings division concentrates on drug delivery polymers with PEPTM (programmable elution profile) technology that allows several drugs to be released from a coating at different times and in different quantities. This technology is being used with partners from academia and the pharmaceutical industry to develop potential new coatings for drug eluting coronary stents. Their role within this project was to supply test artefacts for measurement, and develop the range of coatings and coating thicknesses required for the artefacts.

NPL's role was to review and validate the system design, develop prototype test artefacts suitable for use with the BPR measurement system and compatible with current medical implant devices, to perform measurements on the developed test artefacts, and to co-ordinate a comparison of these measurements with further measurements performed by Nightingale-EOS.

3. Principles of calibration/experimental techniques

3.1 Beam profile reflectometry

Beam profile reflectometry (BPR) was first developed in the 1990s by Therma-Wave Inc. of Fremont, California (now a subsidiary of KLA-Tencor Inc.), and is used by Nightingale-EOS Limited under licence. As originally applied to the measurement of dielectric films on flat silicon substrates, it is described in references [1] and [2]. Figure 1 shows a schematic diagram of the apparatus used to implement the method for the purposes of the work described in this paper. Laser light from a temperature-stabilised 640 nm diode source is relayed via a single-mode optical fibre and some collimating, beam-expanding and polarising

optics to a microscope objective lens with high numerical aperture of about 0.8. The lens focuses the collimated laser light onto a spot with a diffraction-limited size of approximately $0.9\text{ }\mu\text{m}$. Within this spot, light is incident at a continuum of angles ranging from normal incidence to the maximum allowed by the lens. Critically, there is a 1:1 correspondence between the angle of the light incident at the spot and the part of the lens through which the original collimated laser light passed. Assuming specular reflection in the focal plane, this correspondence is preserved when the reflected light passes back through the lens so that light, at a given spatial position within the reflected beam, may be unequivocally identified with a particular angle of reflectance. Hence, by placing a spatially-resolving detector (in this case a CCD camera) in the path of the reflected laser beam, light from multiple angles-of-incidence can be characterised simultaneously without any need for physically scanning the detector.

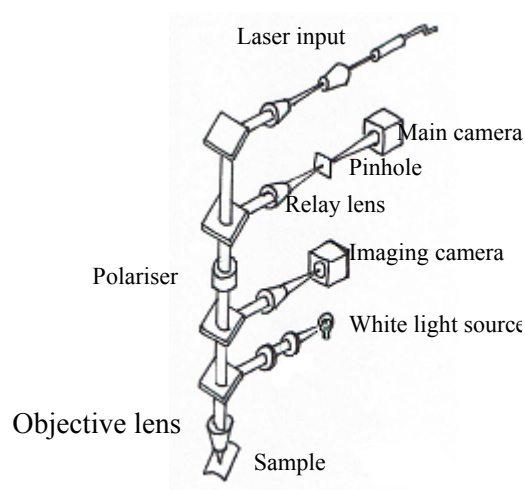


Figure 1. Schematic diagram of BPR apparatus

For samples with a thin-film coating, light reflected from film interfaces, not directly in the focal plane of the objective lens, is not fully re-collimated by the lens. Nevertheless, by elementary ray-tracing it can be shown that for a flat surface all such light rays, from all the interfaces in the system, are brought together in the rear exit pupil (or ‘Fourier plane’) of the lens. In this plane, therefore, an interference pattern is formed which the relay lens then focuses upon the detector, and it is this interference pattern which contains the information used subsequently to obtain the desired measurement parameters. Specifically, based on the polarisation of the incident laser light, two principal sections can be identified within the

Fourier plane that corresponds to s-polarised and p-polarised light. The patterns of reflectance-versus-angle within these sections can be modelled using the well-known Fresnel equations in order to obtain the thickness and refractive index of the film.

Specifically the relevant equations are, at each interface

$$r_s = \frac{\hat{n}_1 \cos \theta_1 - \hat{n}_2 \cos \theta_2}{\hat{n}_1 \cos \theta_1 + \hat{n}_2 \cos \theta_2} \quad r_p = \frac{\hat{n}_2 \cos \theta_1 - \hat{n}_1 \cos \theta_2}{\hat{n}_2 \cos \theta_1 + \hat{n}_1 \cos \theta_2} \quad (1)$$

and for each layer:

$$R_{s,p}^{(j)} = \frac{r_{s,p}^{(j)} + R_{s,p}^{(j-1)} e^{4\pi i \hat{n}_j \cos \theta_j t_j / \lambda}}{1 + r_{s,p}^{(j)} R_{s,p}^{(j-1)} e^{4\pi i \hat{n}_j \cos \theta_j t_j / \lambda}} \quad (2)$$

Where $\hat{n}_j$ are the complex refractive indices of each layer from the substrate to the medium, θ_j is the angles of incidence within each layer (as determined from Snell's Law), t_j is the layer thicknesses, $r_{s,p}^{(j)}$ is the amplitude reflectances at each individual interface and $R_{s,p}^{(j)}$ is the total amplitude reflectances summed to the j^{th} interface.

Where the sample surface is curved, the recombination of light from different interfaces in the Fourier plane of the lens does not work perfectly; rather, light reflected from one interface at a particular angle-of-incidence may interfere with light, which was reflected from another interface at a quite different angle. To account for this it is necessary to perform a full ray-tracing analysis of the light through the film. Nightingale-EOS has developed a highly efficient computational method for performing this analysis.

The optical pathway for the current work also includes a white-light source and some conventional imaging optics so that a real image of the sample, with the focused laser spot in the centre of the field-of-view, may be obtained to assist in locating the laser spot upon the sample. Additionally a five-axis sample handling stage (providing motion in X, Y, Z, tip and tilt) is provided so that the curved sample can be properly located beneath the lens.

3.2 Artefacts

3.2.1 Different artefacts and the reason for them

The amount of drug coating sprayed onto the stent of implants is currently measured by mass. There is no measurement of film thickness, which could in principle be used to derive the quantity of drug in the coating.

The test artefacts used to verify the BPR needed to be relevant to all medical applications, not just the range of stents currently used at Lombard Medical. It was agreed that, ideally, the range of coated test artefacts would be produced, with four morphologies, including flat artefacts and artefacts with a range of curvatures consistent with those found in typical stents, each with a range of coating thicknesses. For NPL to calibrate these artefacts a step or trench was required to be cut through the coating to the substrate. Reference to NPL Measurement Good Practice Guide No.37 [3] was made to help inform, where possible, the artefacts design. It was important that any trench cut did not damage the surrounding polymer coating or the substrate as this would alter the calibration depth at the trench, and would, therefore, not be representative of the thickness of the coating on the rest of the artefact.

3.2.2. Curvature

The different curvatures and thicknesses of the artefacts were selected to reflect the dimensions of stents currently in use and, therefore, which the BPR would be required to measure. Generally, stents have a radius of 1 mm to 2 mm, and the thicknesses of the polymer coatings normally applied to stents is in the range of 5 μm to 10 μm .

The BPR originated in the field of silicon wafer measurement, to this end its use and theory has been based on flat samples. Because of this a flat test artefact was included to help verify the system, completing the transition from the historical use of the device to the medical application implemented here.

Although Lombard Medical has expertise in coating stent samples of similar dimensions to the proposed artefacts, the structures are very different. Stents are a lattice, so solid tubes will disperse the polymer coating in a different way, as would a wire and the proposed flat samples. This led to LMT having to develop new methods of coating the test artefacts to ensure uniform coatings with a smooth and consistent finish.

3.2.3. Material

Stents, being medical devices for implantation into the body, are most commonly made from medical grade, Type 316S stainless steel. They also have a very high quality surface finish. The BPR requires the reflectivity of the substrate surface to be high to produce high visibility fringe patterns. It was important, therefore, to replicate, as closely as possible, the surface conditions of an actual stent on the chosen test artefacts. It proved difficult to obtain electro-polished 316S stainless steel with the desired artefact radii. Non-electro polished steel was initially purchased to assess its suitability, and eventually a syringe needle company was found capable of providing 316S stainless steel with the dimensions required for the test artefacts.

The non-electro-polished substrates when viewed under the microscope were quite rough and underlined the need for electro-polished tubes and wires. Measurements with the BPR were still possible but location of the measurement had to be made carefully to ensure a good reflection, and the measurement uncertainty of the subsequent fringe analysis was higher.

3.2.4. Development of coating technique

The technique generally used by Lombard Medical for coating stents proved unsuitable for coating the test artefacts. Although the tube samples were of similar dimensions and could be mounted in the same way as a conventional stent, the solid surface caused problems with the flow rate of the polymer and, therefore, the uniformity and thickness of the coating produced.

The company spent a large amount of time trying to perfect the best coating finish for the artefacts. Initial tests produced quite a rough surface, but the process was modified and improved to produce smoother coating finishes. Figure 2. shows examples of this.

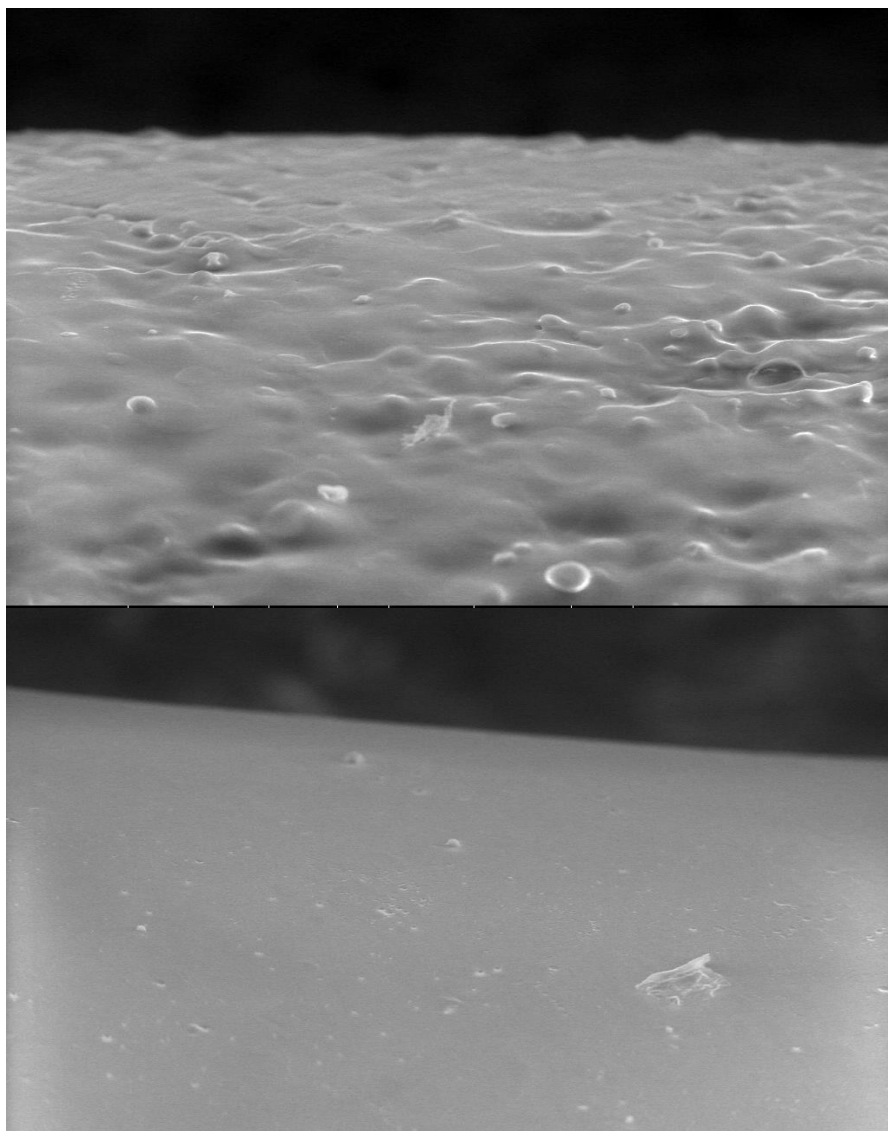


Figure 2. SEM images of two different coating surfaces and their roughness.

All artefacts were coated on a Sono-Tek Medicoat Benchtop Coater using a 0.5% solution of poly(vinylbutyral) in CHCl_3 . All coatings were performed using a flow rate of 0.09 ml/min with an ultrasonic power of 1.5 W. The artefacts were cleaned prior to coating by sonicating in a 7.5% solution of NaHCO_3 followed by 2-propanol and finally deionised water. The artefacts were then dried at 100 °C for 20 hours.

The exact process used for coating each type of artefact varied slightly as detailed below: The three flat artefacts had a problem with the dispersion of the polymer, with the following procedure producing the best result. The artefacts were coated receiving four, ten and twenty passes respectively on the ultrasonic spray coater, with the artefact moving at a horizontal

speed of 0.13 cm/s at a distance of 28 mm from the spray head. After coating, the artefacts were held in a CHCl_3 atmosphere for 10 seconds before being dried under vacuum at 40 °C for 20 hours.

The two diameters of tube artefacts were mounted on a mandrel for coating. Three of each were coated receiving eight, sixteen and twenty four passes respectively on the ultrasonic spray coater with the artefact moving at a horizontal speed of 0.13 cm/s and rotating at 40 rpm under the spray head at a distance of 24mm from the spray head. After coating, the artefacts were held in a CHCl_3 atmosphere for 10 seconds before being dried under vacuum at 40 °C for 20 hours.

The wire artefacts had to be mounted in a C-shaped piece of metal to keep the wire straight and under tension, which also allowed the coating to be sprayed around the whole wire, in a similar way to conventional stents. They were coated in the same way as the other artefacts except that they received ten, twenty and thirty passes for the three thicknesses of coating.

4. Measurement of artefacts at NPL

Several methods were investigated while attempting to produce suitable trenches in the polymer coatings on the test artefacts.

4.1. Trench cutting

4.1.1. Focused ion beam (FIB)

The University of Surrey have a focused ion beam system, and this was initially thought to be the best method to accurately cut through the polymer coatings on the test artefacts, while not damaging the substrate. It was initially thought it would not be a problem to micro-mill the trenches using the FIB, although it had been previously noted that the polymer type could sometimes affect the accuracy of the cutting process.

Three coated, 50 μm diameter wire artefacts were initially provided for testing. The milling method used created a step on one side and a series of steps on the other; a sharp edge was then cut by further fine milling to ensure a vertical surface. Some surfaces can curve at the edge, but this can be rectified with an ion beam mask to protect the surface during the milling.

The ability to mill through the polymer without subsequently cutting through the substrate was controlled by feedback from the FIB system, which monitors the current leaking to the FIB mounting stage during the milling progresses. Figure 3 shows an example of this process. When milling the polymer the leakage current is low as the polymer is insulating and we only see what is scattered in the FIB chamber.

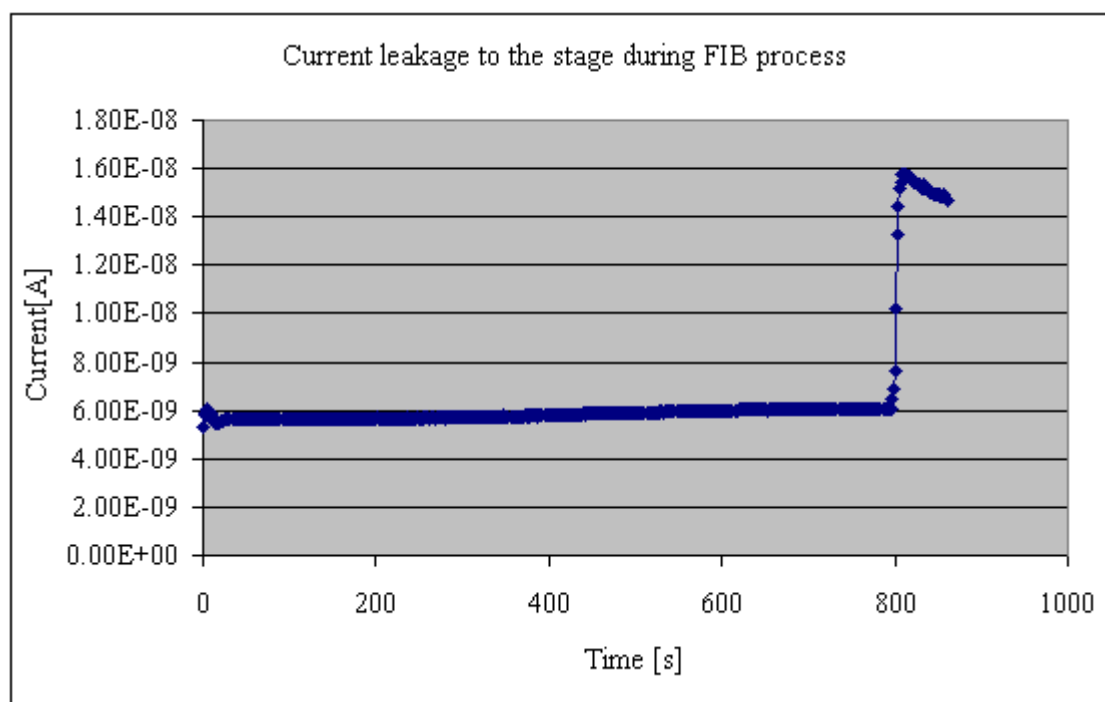


Figure 3. Current leakage to the stage during the FIB process.

The large spike corresponds to the point where the polymer is so thin that current can leak into the wire and through to the FIB stage. This means that there will be a very small error associated with some milling after all the polymer has been removed, but the amount of overcut would be very small, of the order of a few nanometres, since it only corresponds to a few passes of the milling beam. With each pass milling off a layer or two of atoms, it is

unlikely that even the substrate's native oxide will have been removed. Figure 4a and b show two SEM images of trenches cut by the FIB system in 50 μm wires.

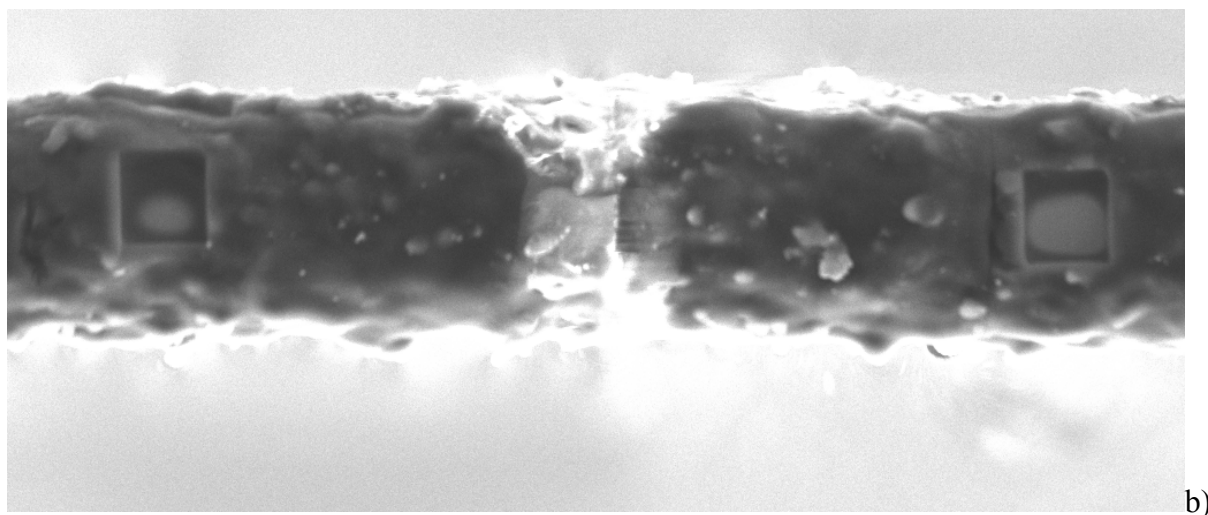
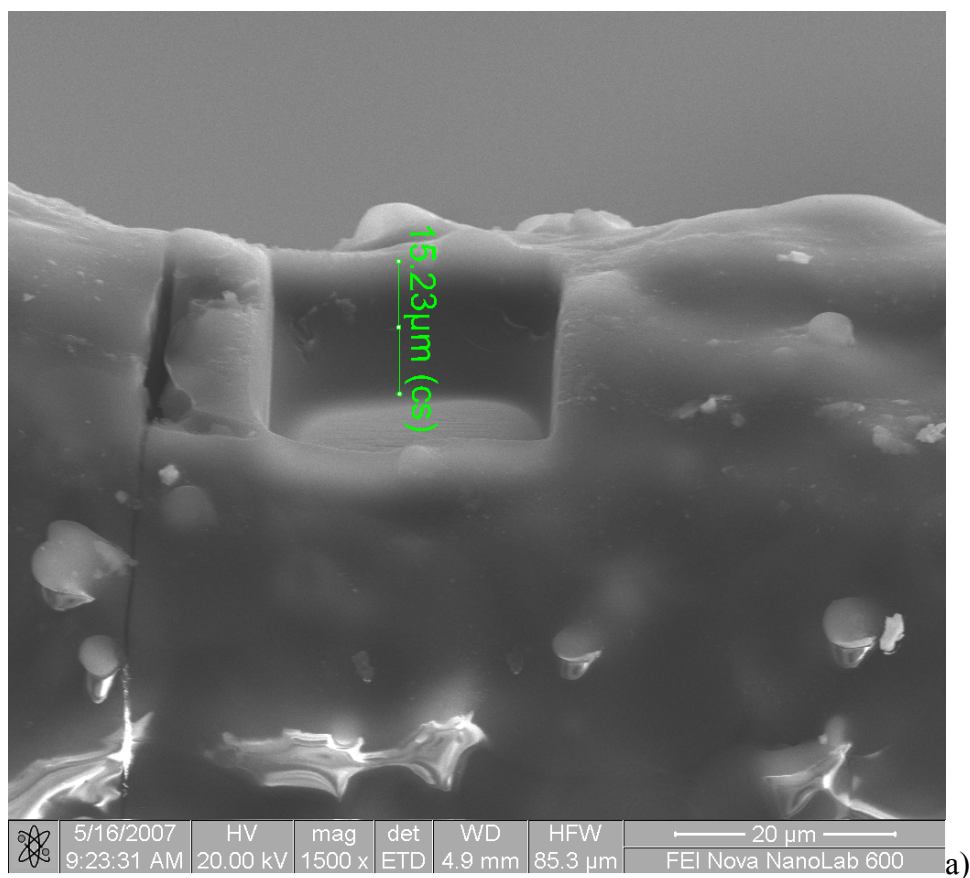


Figure 4a and b. SEM images of the 50 μm wire with the FIB trenches cut through.

Unfortunately problems arose when using this method with the tube artefacts. There were difficulties in milling the coatings on these artefacts as a differential milling rate caused

smearing of the trench leading to variation in the cut depth of up to a third of the coating depth. This was originally thought to be due to the surface roughness of the polymer coatings, but further investigations with smoother coatings suffered the same problem. It was subsequently believed that the problem was caused by a combination of sample charging, which caused drift, and also a significant difference in the milling rate once into the coating layer possibly due to a crystalline structure within the coating material. These problems were not experienced with the initial wire artefacts, but since the polymer used was the same for both it was difficult to know the exact reason for the problem. Grounding of the steel tube artefacts was tried but still did not resolve the problem. It was thought that application of a gold coating could have helped the milling process, but that would have rendered the artefact unusable for use with the BPR.

An alternative FIB system was also tried at the University of Cambridge but the milling times were extremely long with their system, in the region of 10 hours per trench. The results were not as clean as those produced by Surrey, and did not meet the requirements for the reference artefacts, as it was not possible to accurately mill to the substrate surface, leaving deposits in the base of the trench, which could not be quantified. Figures 5 and 6 show SEM images of the Cambridge FIB trenches.

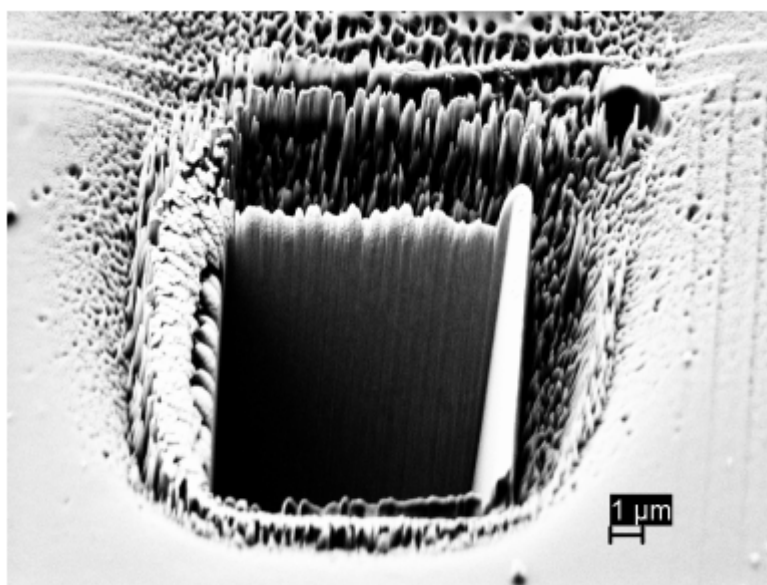


Figure 5. Damaged edges make the coating surface level difficult to identify and measure.

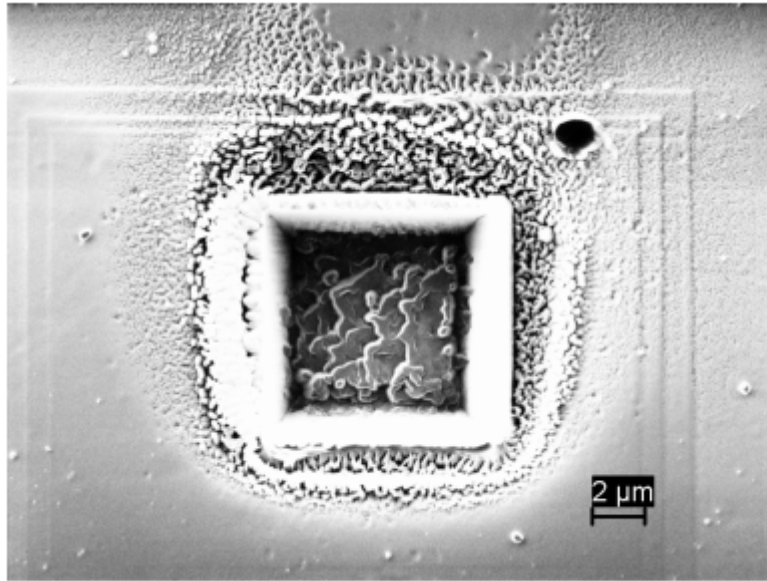


Figure 6. Large deposits covering the trench base make any depth measurement unusable.

Because of the problems encountered, the FIB system was considered unsuitable for creating the required trenches in the test artefact coatings.

4.1.2. Laser Machining

Oxford Lasers were approached to ascertain if any of their laser systems would be capable of cutting the required trenches through the polymer. Oxford Lasers were given four different coated artefacts and contracted to attempt laser cutting of them. A series of test holes were drilled at one end of each tube, to establish the best cutting parameters for the coating. A further series of twenty, (best cutting condition) holes were then drilled at the other end of the coating. This was ultimately unsuccessful, Figure 7 shows an SEM image of two typical test holes. The area surrounding the holes has been badly damaged by the process, and the base of the hole is uneven, indicating unremoved polymer residue on the substrate surface making usable measurements impossible.

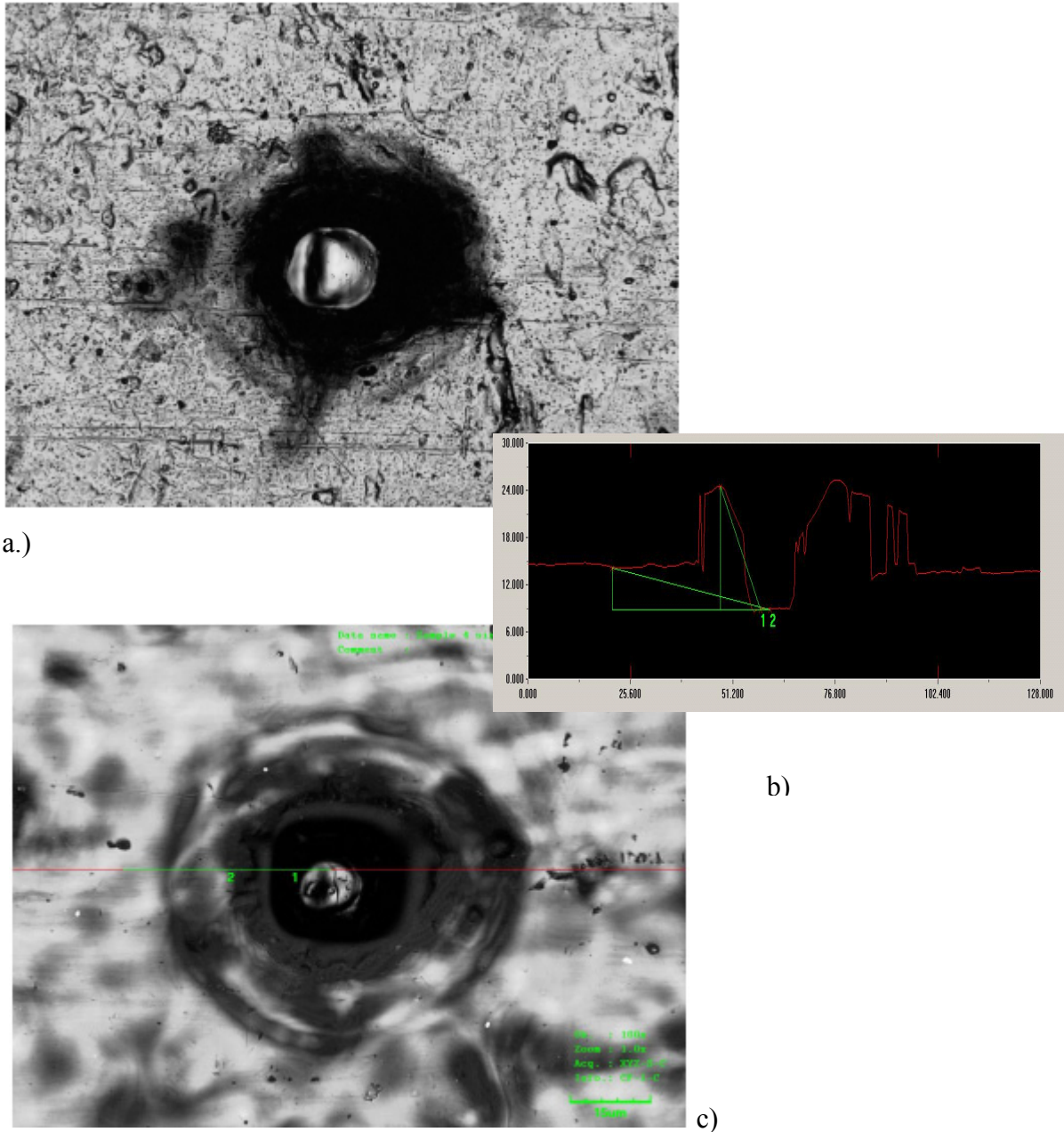


Figure 7a, b & c. Show typical examples of the Oxford laser trenches & measurements. a) shows the damage to the surrounding polymer, b) shows the unevenness of the trench base, while c) shows a measurement cross section, by the LEXT confocal microscope system, displaying problems with the measurement of a trench.

4.1.3. Masking

A final alternative to create the trenches in the polymer coating was to mask off an area of the substrate prior to the coating being applied. This technique, however, has several disadvantages. Firstly it is difficult to obtain trenches that are very narrow, making

measurement of the trench more difficult and time consuming. Also the definition of the trench produced is not as pronounced as with other techniques.

The photo fabrication company Photofab were consulted about the possibility of creating a photolithographic mask to produce small and well-defined trenches but it was decided that this would not be possible due to the difficulty in effective adhesion of the mask to the substrate.

Despite these limitations masking proved the most effective method for this work and was used to produce the test artefacts. A form of tape was used on the final samples as this proved to produce the most suitable trenches, and also reduced the artefact production time since they were created during the coating process as opposed to a separated stage in production. Essentially each artefact when coated had a central area masked which, once removed, left a trench through to the substrate surface. Due to the limitation of the tape positioning this trench was generally about a millimetre or two wide. The removal of the mask affected the definition of the step edge, causing there to be a slight slope from the coating surface to the substrate, which consequently made AFM measurements more time consuming.

4.2. Systems used to measure the trenches at NPL

Initially it was thought that several contact systems would be suitable for the measurement of the artefacts, but the size of the artefacts and trenches meant that this was not possible. It was decided to use three non-contact measurement systems owned by NPL to measure the artefacts: an Olympus (LEXT) confocal optical microscope, a Park Scientific atomic force microscope (AFM), and a Taylor Hobson Talysurf CCI scanning white light interferometer (SWLI). Unlike the AFM and SWLI, the LEXT system was capable of scanning the full width of the trench, with the system software used to stitch the scanned images together. Measurements made with the AFM were taken as separate scans, translating the system X-Y stage between scans, and allowing for significant overlap between the traces to ensure good scan alignment. In this way it was possible to build up a picture of the whole trench. Single images of each side of the trench were taken using a 10x magnification lens to give the broadest possible view of the step. The three systems all proved to have different limitations when measuring these types of artefact and the best obtained data for each (primarily obtained from the SWLI) was used when comparing results with the BPR.

The AFM had a maximum manufacturer specified z-range limit of 12 μm , but in practice it became apparent that this limit was closer to 8 μm . Due to this the AFM was only suitable for measurement of eight of the twelve artefacts.

4.3. Suitability and problems of using each system

4.3.1. AFM

The performance of the AFM was limited by the thickness of the coatings and was only capable of measuring steps of less than 8 μm . For measurement in the AFM, securing the samples was also a problem. This was most apparent with the 50 μm wire samples, as during measurement the AFM tip would move the sample distorting the measurement. Several methods were used to secure all the artefacts during measurement. Initially gauge blocks were spark eroded with a shallow trench to secure the tubes and wire to. This was achieved by placing the wire in tension across the gauge block and securing the ends. Unfortunately this still allowed for some movement during measurement (mostly with the wire samples), as a result adhesive carbon paper was tried, and this approach proved to be more effective, although there was still a small amount of movement with the wire sample during measurement. This can be seen in the AFM images, where some planes of AFM tip movement show shifts in the image (Figure 8). The X and Y movement of the AFM was also a limiting factor as it could not measure the whole trench in one scan, and also more importantly it was not possible to measure the complete step in one scan, due to the slightly sloping edge. The AFM stage had to be moved, and a series of progressive scans taken to build up the final image of the step. This made a single measurement of one side of a trench extremely time consuming as each scan could take ten minutes or more and at least three or four were needed for a measurement. There was no stitching software with the AFM so this had to be completed by hand.

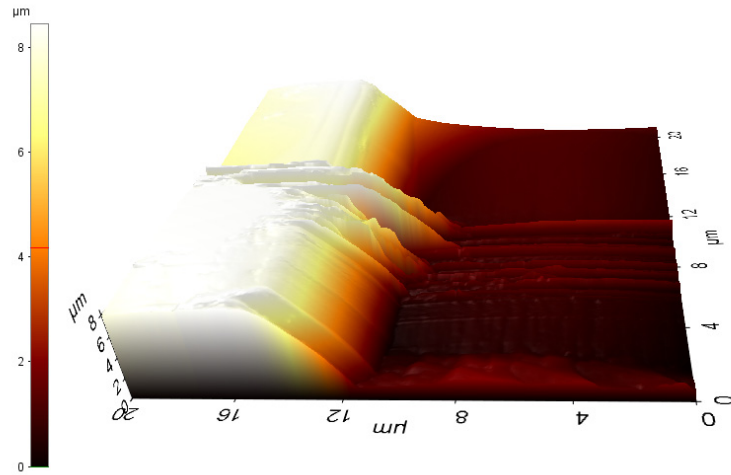


Figure 8. AFM image, slight movement with an AFM measurement of a wire can be seen.

Figure 9 shows one of the images from the AFM measurements. The surface topography is clearly visible but due to the limited resolution only the start of the slope/step can be seen so many images were needed to build up a profile of the full step. Stitching of these images can introduce errors in the determination of the dimensions of the trench.

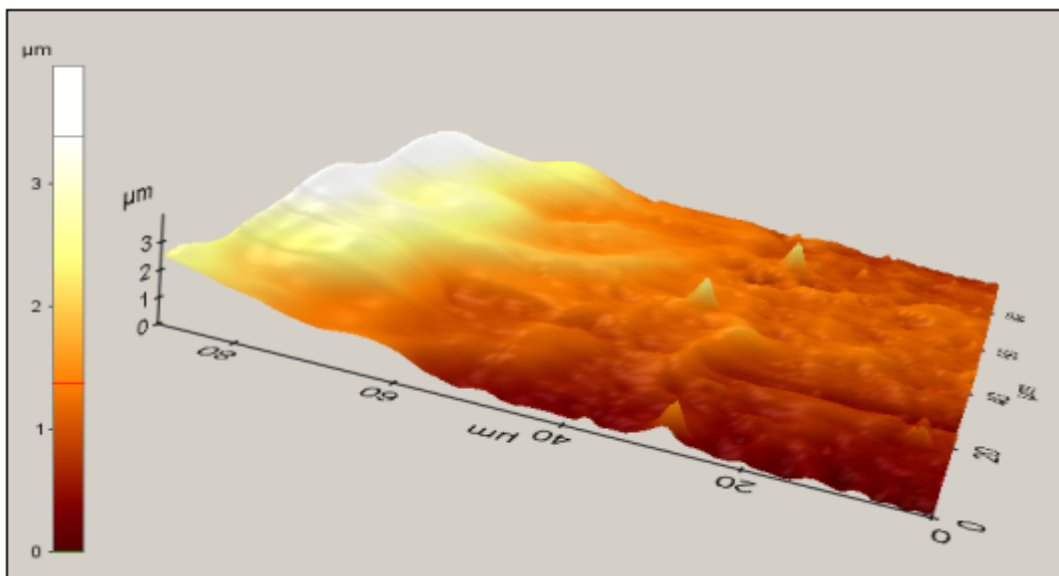


Figure 9. AFM image of the centre of a trench and the edge of the sloped side.

4.3.2. LEXT confocal microscope

It was possible to make measurements with the LEXT system, although there were several issues with the results. The result of stitching several images to form the overall scan image can be seen in Figure 10 and 11, but this process added a certain uncertainty to the measurements. It was also difficult to take into account the levelling of the sample, (Figure 12). Because there was no stage levelling adjustment the measurement data had to be post-processed to remove nominal tilt.

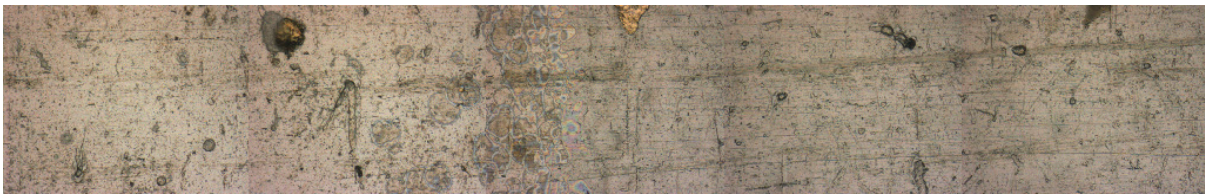


Figure 10. LEXT measured image of one side of an artefacts trench.

Figure 10 shows the composite image of a trench step produced by the LEXT system, the individual stitched images can be seen and the step can just about be made out centrally. Measurement with the LEXT system could be made from the stitched results by removing the nominal tilt plane from the measured image, shown in Figures 11, 12 and 14, allowing the step height to be evaluated more accurately.

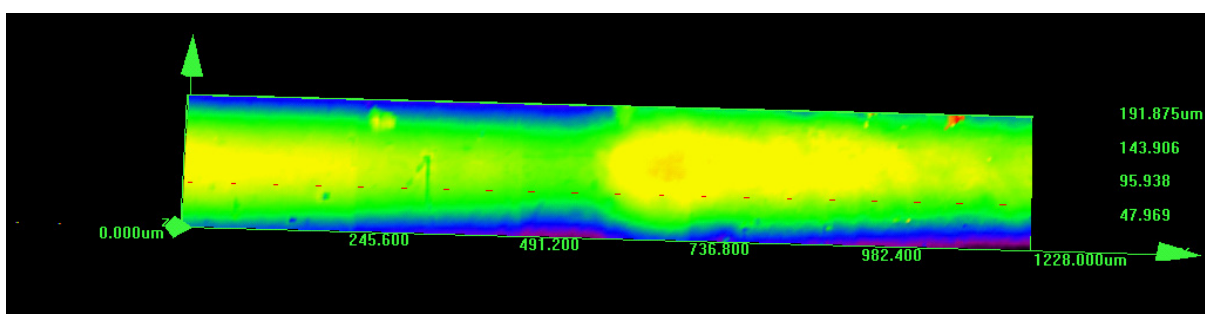


Figure 11. Height map from a LEXT measurement, of a tube artefact.

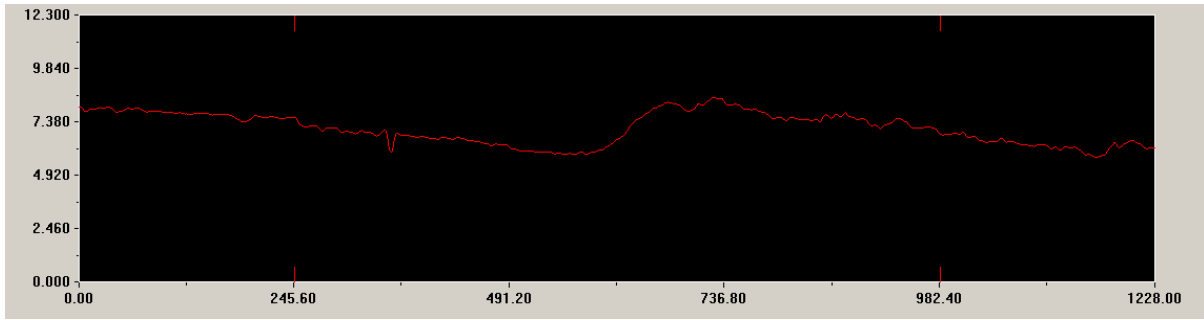


Figure 12. Measured height profile of a plane along an artefacts trench

What proved to be a major issue, however, was an effect due to the transparency of the polymer coating. The LEXT system, although still able to image the surface, appeared to have its measurements distorted by reflections from the substrate through the polymer. This meant that the imaged surface was a combination of two different surfaces, and could not be completely relied upon. Due to this the system did not accurately pick out the coating surface and drastically reduced the measured trench depth; this effect was highlighted when measuring a gold plated sample, which eliminated this effect and showed the depth to be very much greater. Partly due to this transparency, but also due to the reflective nature of the substrate, multiple peaks were formed in the results, especially around the trench edges. This “noise” was filtered out using software filtering but their effect on the results was not quantifiable, and added an uncertainty to the measurements (Figure 13 and 14 show scans of two trench edges, a lack of depth from the measurements can be seen. Figure 14 also shows, as a red line, a plane of measurement being taken across the sample).

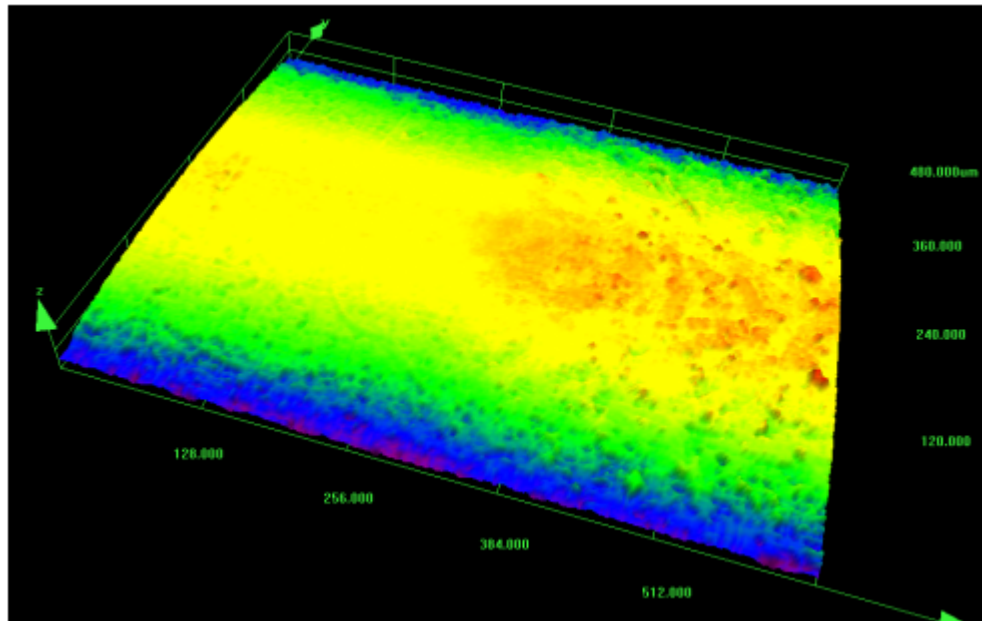


Figure 13. LEXT scan of a trench step.

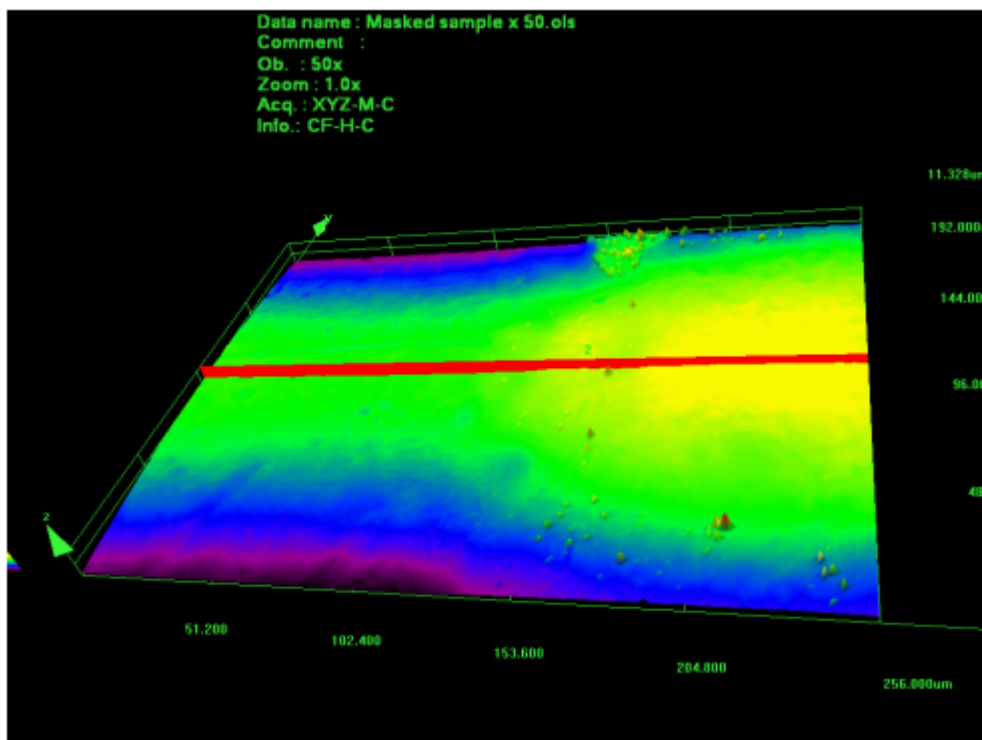


Figure 14. LEXT scan of a trench step, showing, in red, the plane of measurement.

The LEXT system also struggled to measure the wire samples due to their size and unevenness, with the results obtained deemed unusable.

4.3.3. SWLI

The SWLI was capable of scanning one complete side of the trench, but it was not possible to track across to the other so separate scans were made of each side. The system software had levelling capabilities, which was used to adjust for any tilting of the sample during data analysis.

Figure 15 shows a measurement image of a 2 mm radius artefact taken with the SWLI. The substrate surface has been measured well but the coating surface displays some noise, appearing as a series of peaks. This was common with all the measurements of the samples and was partly due to the unevenness of the coating surface, which the SWLI finds difficult to identify fringes on. It can also be seen that the peaks saturate at a certain height making the measurement quite difficult.

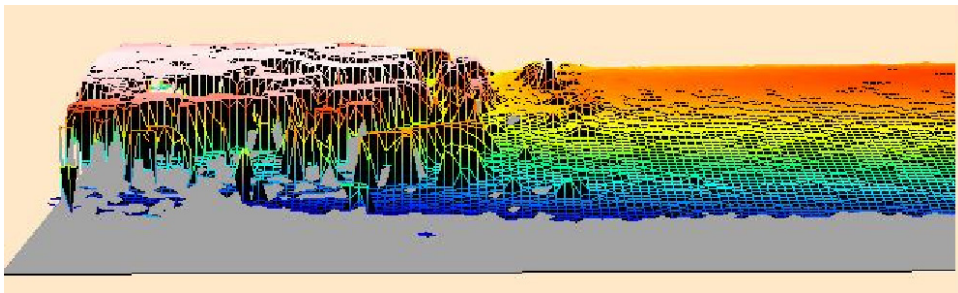
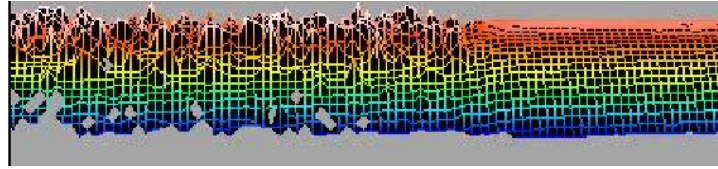
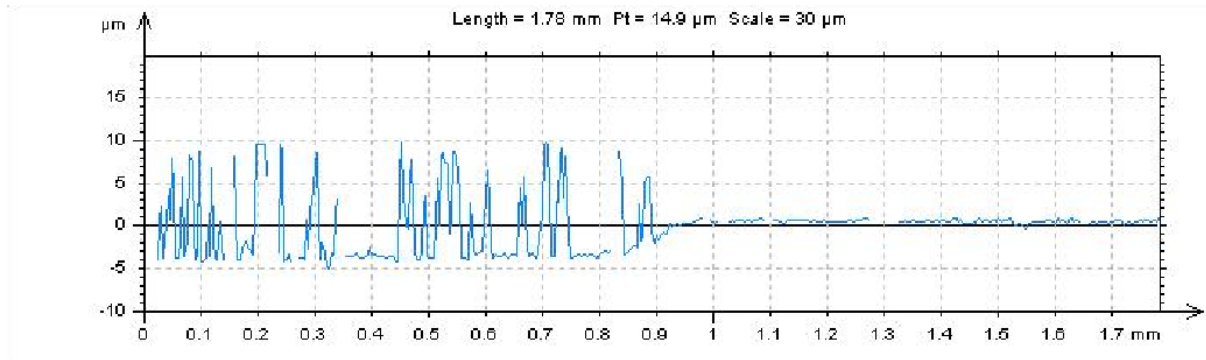


Figure 15. SWLI measurement of a sample trench.

A major issue with the measurements from the SWLI can be seen in Figure 16a and b. There are three levels measured for the step from which it is difficult to identify the actual substrate and coated surface from the profile. From knowledge of the sample the smoother surface on the right is the substrate whereas the noise would be the coated surface, although there is apparently a further surface below the level of the substrate.



a)



b)

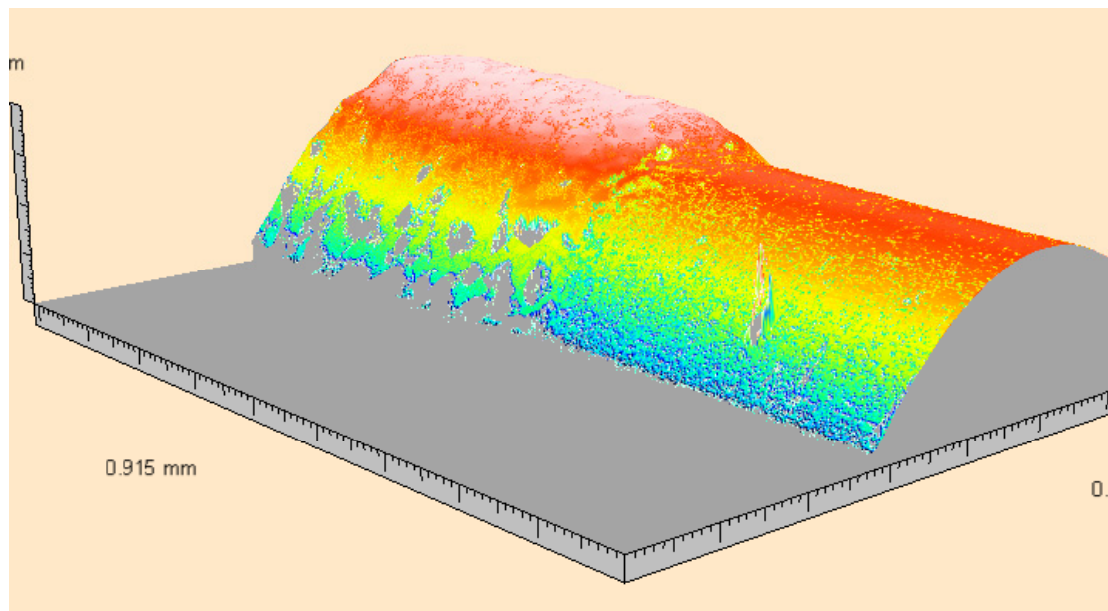
Figure 16 a and b. 3dimensional measurement and profile of a sample and trench

It was thought that secondary fringe patterns were being observed through the polymer coating from the substrate causing this additional level. Measurements on several samples showed that this effect decreased with the thicker coatings as less light, and hence secondary fringe patterns, are reflected through the coating.

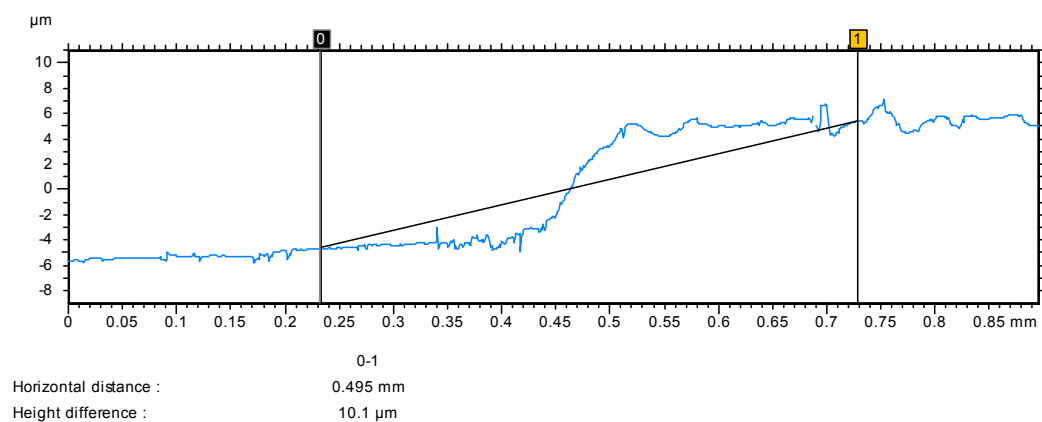
The height of the level is likely to be due to the refractive index of the polymer and would be expected to be a function of this and the thickness of the polymer coating. It could, therefore, be possible with knowledge of the refractive index of the polymer, to even calculate the thickness of the coating just from this.

To confirm that the imaged surfaces actually represent the true surface of the sample, a key sample was gold coated with a thin and even layer across its surface. This coating allowed the sample to be measured more accurately with the SWLI, eliminating the noise and also the effect caused by the fringes from beneath the coating. Measurement of this confirmed the assumption that the smooth side was the substrate surface and the levels of the peaks were the true representation of the artefact at its surface.

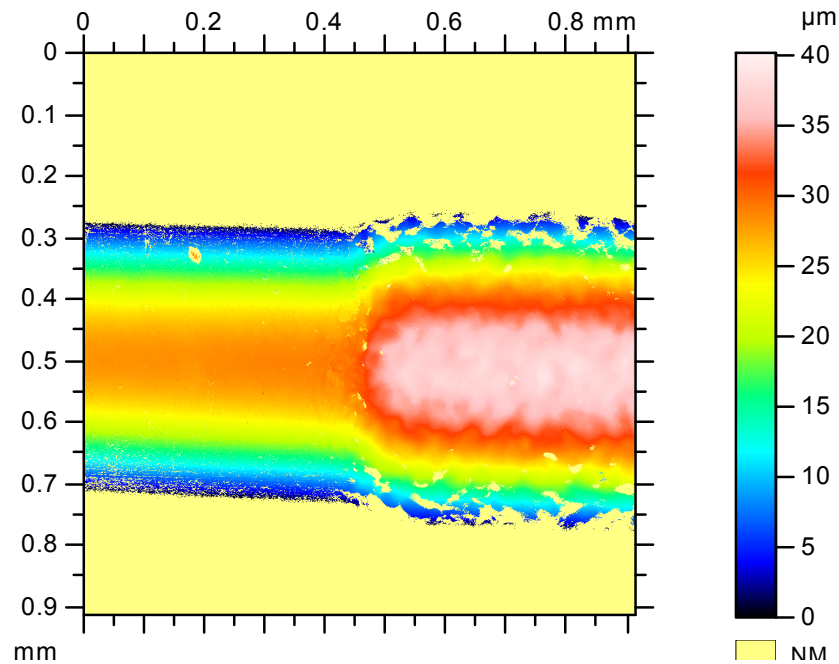
Figure 17a, b, and c show the gold-coated sample. The measured height of this was the same as the value evaluated prior to the gold coating being, within the uncertainty of the surface roughness.



a)



b)



c)

Figure 17a. b. and c. SWLI measurement images of the gold plated sample.

Although the SWLI had some difficulties on the thinner coatings due to the presence of secondary fringe patterns described above it still proved to be the most effective and accurate method for measuring these samples. Several measurements were still needed on both sides of the sample trenches to get good readings, but the final measurements made for comparison with the BPR were made using this system.

4.4. Measurement and results of NPL measurements

The SWLI was found to be the most suitable instrument for measuring the artefacts. It was possible to achieve clear and measurable steps from the scanned images, as shown in Figure 18a and b. The system still experienced problems with some of the measurements, for example Figure 19 demonstrates noise in the data around the edge of the step and which can be explained by standard optical effects similar to those mentioned in [4].

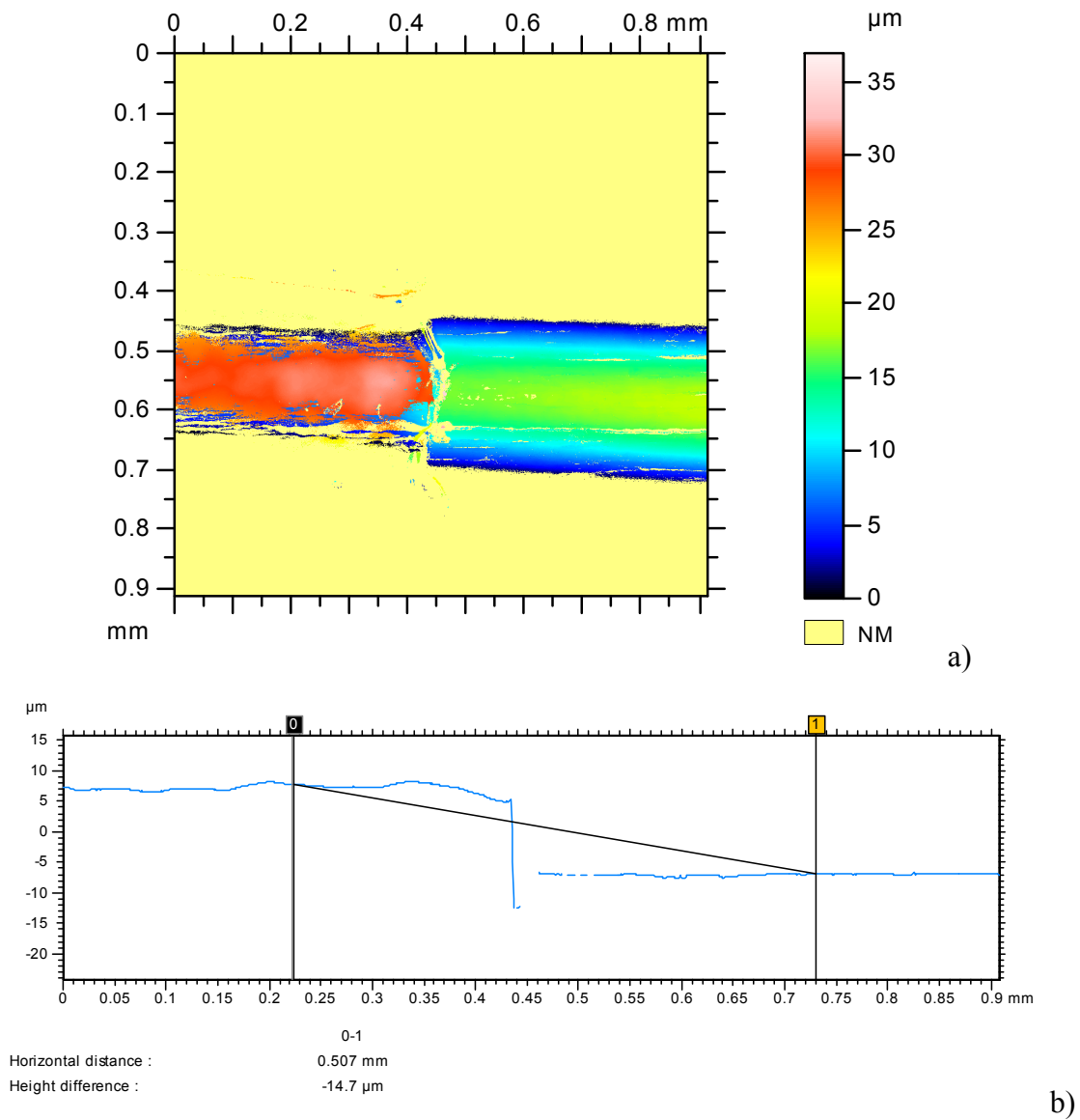


Figure 18a and b. A further example of a measurement made with the SWLI, showing the surface roughness and step measurement.

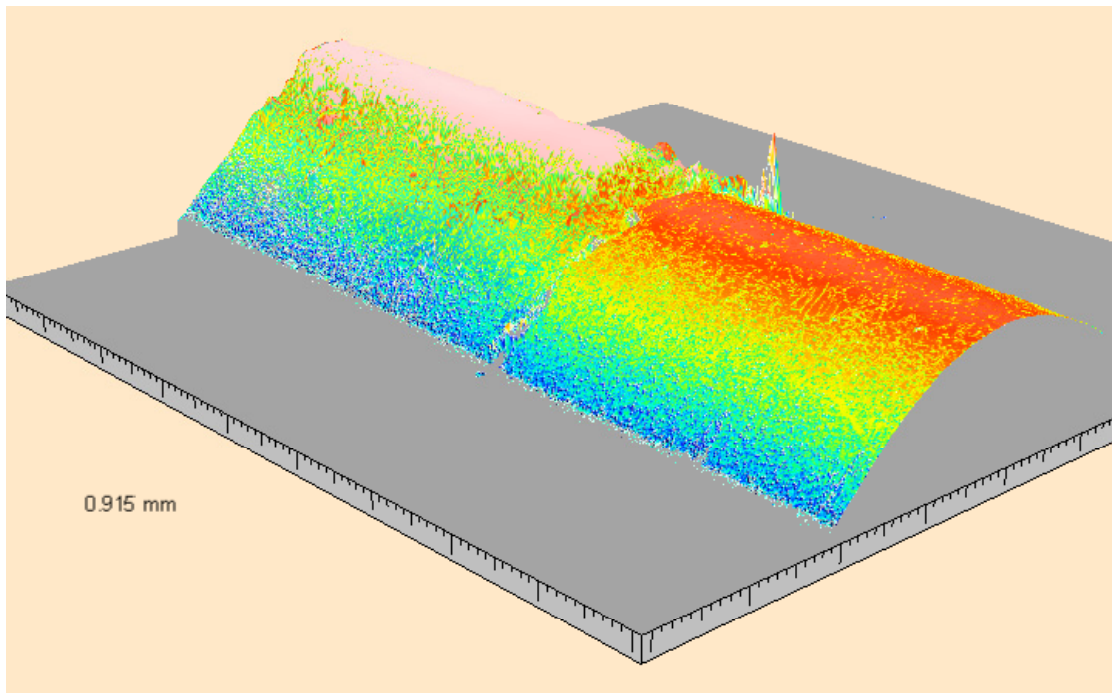
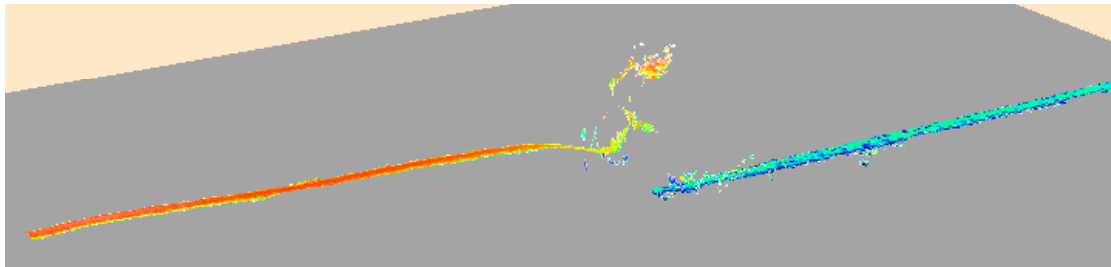
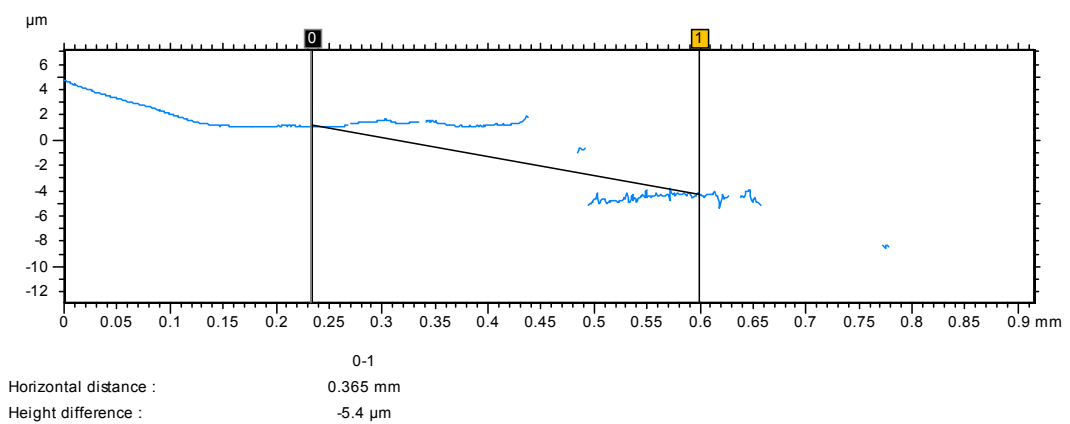


Figure 19. A SWLI image showing some of the noise experienced around the edge of the step, possibly due to optical effects similar to those mentioned in [3].



a)



b)

Figure 20a and b. The difficulty in measuring wire samples with the SWLI.

It also proved difficult to measure the wire samples (Figure 20a and b). This was largely due to the unevenness of the coating and damaged trench edges caused by the removal of the mask and which had a greater effect on the wire artefacts than the tubes. However, the majority of the measurements were found to be usable, and noise and errors in the scan could be filtered out, or were within the uncertainties caused by the unevenness of the coating surface. This was the data, which was used to compare with the results obtained by the BPR.

5. Measurement of artefacts with BPR

5.1. Initial measurements

Initial use of the BPR showed an issue in the automation of the system. It was quite time consuming to align and take measurements. To help with this a four-day consultancy was setup, with Nightingale EOS to see what could be done to help in this area. Work on this consultancy is detailed in Appendix I.

The first results from the BPR showed some discrepancies from the NPL results, further fine tuning of the BPR highlighted some calibration issues, which improved the comparative results. The thickest coatings on each type of artefact were too thick for the current BPR optics to cope with. It was not possible to see clean fringes for any of them and it would appear that the number of fringes is too great for the pixel resolution of the camera. Figure 21 shows an example of this.

An initial calibration of the system was performed using a silicon “wedge” wafer with a pure SiO₂ layer of known refractive index but with thickness varying continuously with location. Using data from a given location on the wafer, it was possible to obtain a plausible calibration if any one of a set of discrete thickness values at that location is assumed. In this case, the calibration was performed using an assumed thickness that satisfied the following two criteria:

- Reasonable accuracy (within ~10 nm) when measuring a certified VLSI standard SiO₂ layer (unfortunately the thickness of the layer on the VLSI wafer was too thin to use for performing the calibration in the first instance).

- Reasonable correspondence to the expected order-of-magnitude thicknesses of the NPL artefacts.

For the measurements, the substrate properties in each case were assumed to be those of standard 316 stainless steel ($n = 1.71$, $k = 3.47$ at 640 nm). Only the s-polarisation plots were used, since alignment along that axis was more straightforward and these profiles are immune from birefringence effects, which are not yet fully modelled in the software. An example image from a measurement and software fit of the fringe pattern is shown in Figure 22.

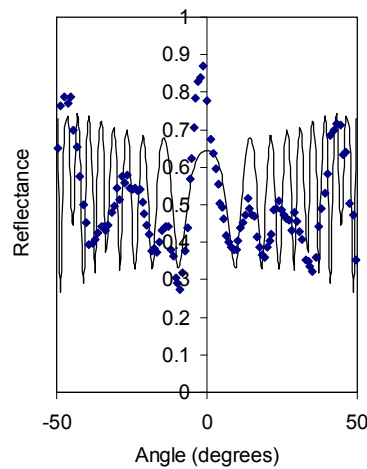


Figure 21. The software fit of a thick coating on a 1mm tube sample, giving a thickness of $14886 \text{ nm} \pm 600 \text{ nm}$ and $n = 1.65 \pm 0.10$.

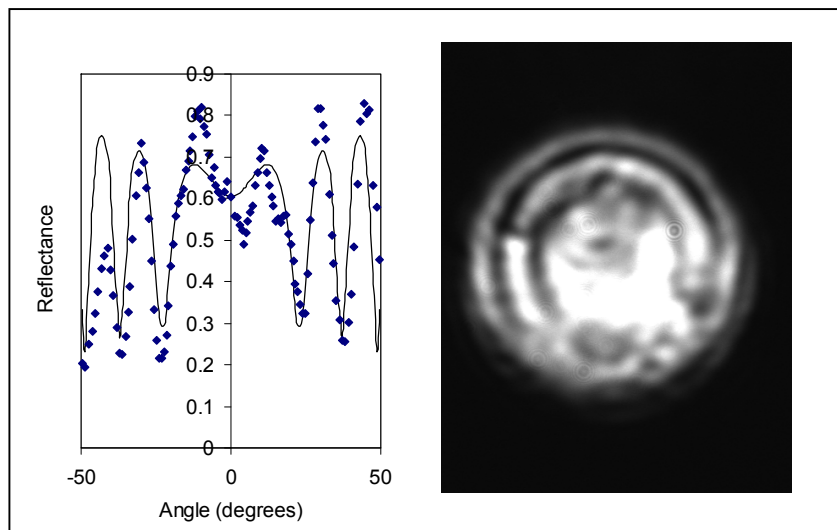


Figure 22. Shows the optical fringe image and software fit of a 2mm tube sample, giving a thickness of $4999 \text{ nm} \pm 300 \text{ nm}$ and $n = 1.73 \pm 0.12$.

Initial results and comparisons from this work were published elsewhere [5], a further final set of results were taken with the BPR essentially the thickness results are in line with what we had before, so for the thinner samples there is still good agreement between NPL measurements and the BPR. For the thicker samples, however, there are still far more fringes than would be consistent with a homogeneous layer of the thicknesses found. Nevertheless, it was possible to verify the thickness measurements approximately by a crude approach of focusing the BPR laser beam on the top and bottom interfaces of the film and measuring the z-displacement (and multiplying the displacement by an estimate of the refractive index).

It seems reasonably certain that there is evidence of inhomogeneity within the layers. For example ‘Flat sample 1’ data below, shows that although the fringe patterns are clean and symmetrical, they are not uniform – for a homogeneous film you would expect all the fringe heights to be the same, but clearly this is not the case here. This does not appear to be an artefact of the optics as this was checked several times, so is likely to be a real feature of the data. Modelling the film as two or three layers instead of one, it was possible to get the right number of fringes with a total thickness much closer to the NPL values. This is not straightforward, however, and to do it properly another bit of software development is required.

5.2. Re-measurement of NPL standard samples

Since the gathering of data for [5] and subsequent results, the Nightingale EOS BPR apparatus was modified in order to eliminate some of the optical effects, which affected the data from some of the samples. The fringe patterns subsequently obtained were clearer and sharper, with fewer aberrations and better overall resolution. Figure 23. shows a good example of this. In particular, it was possible to obtain clearly resolved fringes from the thicker samples where previously this had not been possible, Figure 24, and to get good-quality “p-polarised” data where previously only the s-polarised data was usable. A complete re-measurement of all the samples was therefore carried out at NPL using the SWLI. For each sample, two measurements were made, one from each side of the ‘trench’. The results of these measurements are shown in Table 2.

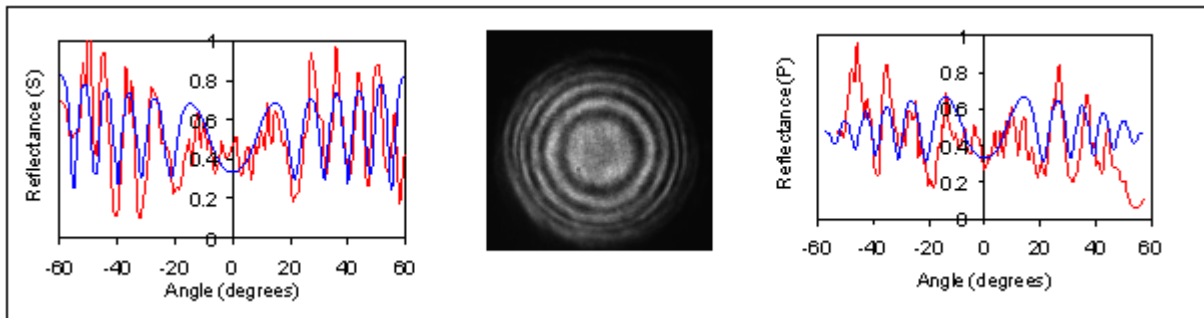


Figure 23. Shows measurements made using the BPR of a 2 mm diameter tube, (sample 2), clear and sharp fringe patterns are visible, giving a thickness of 7512 nm and $n = 1.717$

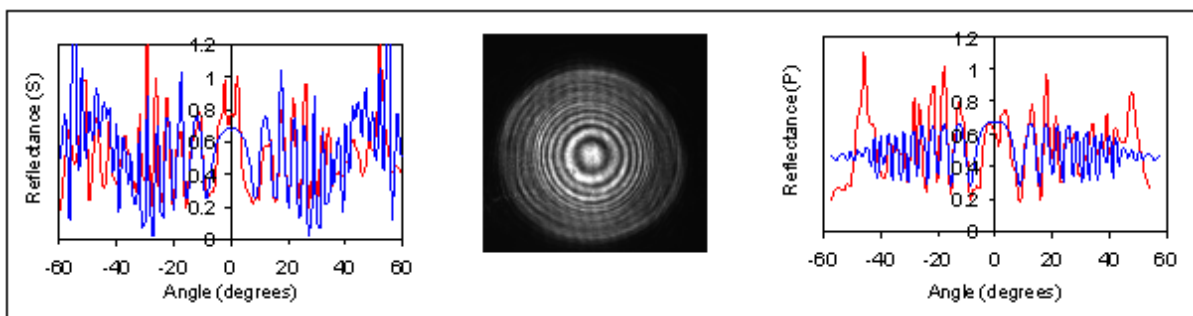


Figure 24. Shows measurements made using the BPR of a 1 mm diameter tube, (sample 1, thick coating), giving a thickness of 23752 nm and $n = 1.790$.

6. Analysis of measurements

Table 1 shows the NPL coating thickness measurements and the first set of BPR measurements of the test samples. The differences between NPL and the BPR range from 0.05 μm to 5.31 μm although all except the 50 μm wire sample are within 1 μm , which shows very good correlation. This is impressive considering a large discrepancy could have come from the coating thickness variation on the test samples. An example of this is the third wire artefact that gave BPR measurements ranging from 6 μm to 11 μm , similar in magnitude to the largest difference between NPL's and the BPR measurements.

Similar optical errors and effect as described in [4] were apparent in all the optical systems used by NPL in measuring the trenches, which affected the results. The BPR system is theoretically better suited than the three NPL techniques for measuring these coatings but it experienced problems with the thicker coatings, and in practice was unable to take accurate measurements due to a saturation of fringes of these thicker coated test samples.

Table 1: Table of coating thickness measurements, all values are in micrometres.

Artefact radius	NPL			BPR		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
2 mm	8.14	7.12	4.24		7.07	4.99
1 mm	15.65	8.51	3.30		9.42	3.66
50 μm	12.47	14.6	5.59			10.90

Table 2 shows the measurement results from the BPR with the clearer and sharper fringe patterns, after the first BPR apparatus modification. With the clearer images it had been possible to obtain measurements from the thickest sample coatings, but not the wire test samples.

The best results from the re-measure of the test samples give clearer measurements but still produced similar values to before, although the thicker sample measurements diverged considerably from the NPL results.

Apart from the thickest samples the difference to the NPL results for three test samples were less than 0.39 μm and the fourth was still less than 2 μm . For some samples the surface coating varied by up to 2 μm as well, which shows these comparative measurements to be very good and the clearer imaging of the BPR also gave better confidence in the validity of those measurements.

Table 2: Table of re-measured coating thickness measurements, all values are in micrometres.

Artefact radius	NPL			BPR		
	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
2 mm	8.14	7.12	4.24	13.37	7.51	4.27
1 mm	15.65	8.51	3.30	23.75	10.35	3.44
50 μm	12.47	14.6	5.59			

7. Conclusions

Despite an array of problems encountered in the production and development of suitable test samples, for measurements and comparison, final results have shown very good comparison between the NPL measurements and those made by Nightingale EOS with the BPR.

Considering the variation in the coating thickness across the surface, the errors around the step edge and measurement errors, the two sets of results are within the uncertainties of the artefacts.

Due to the nature of the coating and its effect on the measurement systems used, a large amount of noise and system errors were evident in all the measurements. The optical systems were affected the most, especially by the transparency and reflectivity of the materials used in the artefacts, and struggled to image the polymer surface accurately on the thin coatings. Verification of the BPR measurements with these artefacts showed good comparative results but highlighted a slight difference with some samples from the NPL measurements. The surface roughness and the coating thickness variation was a large part of this measurement error but it was also shown that there is a measurement limit for the BPR where the coating thickness is too great for the current system to measure, comparably to NPL. Nightingale EOS investigated this discrepancy and were able to identify the cause to be an error in the predicted Fourier plane of the lenses of the BPR. After contacting Nikon they accurately located the Fourier plane, and using several thick polymer-on-silicon samples, fine-tuned the alignment. After resolving this issue and re-measuring the artefacts again, the problem with the thicker samples, and on all three wires was gone. The measurements on these samples are at least as good as on the thinner, flatter samples. Overall thickness correlation across the whole range is now excellent. Appendix III shows the comparison of these results and measurement data of the re-measuring of all the artefacts.

The results between the AFM, LEXT, and SWLI techniques highlighted many key characteristics of the different systems as well as their limitations when applied to applications that have a different nature to the standard step artefacts they are generally used to calibrate and measure.

8. Possible future work

Although the work carried out confirmed the operation of the BPR there are several additional steps which could be undertaken to further verify performance.

It might be advantageous to gold plate all the samples to more accurately measure them as well as confirm the previous measurement, completing their analysis.

Further calibration of the optical measurement systems used could be performed by verifying them directly at the nominal step heights being measured. This could be achieved by measuring reference step gauges of these heights. Unfortunately this procedure had not been possible previously due to none being available at NPL, with the correct dimensions.

There were still remaining issues with the BPR due to speckle noise from the laser; this causes the various ‘spikes’ seen in the data, and without doubt effects the refractive index measurement to an extent (since the latter depends on being able to measure the fringe amplitudes accurately). Stephen Morris of Nightingale EOS believes he knows how to solve this issue and will be following this up in the future to improve the system further.

9. Acknowledgements

The project was funded by the DIUS, Measurement for Innovators Programme under a Joint Industry Project. Thanks also to David Cox, Claudiu Giusca, Alan Wilson, Robert Boyd, Louise Brown (NPL), and Bill O’Neil (University of Cambridge).

10. References

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- [2] J.T. Fanton, J. Opsal, D.L. Willenborg, S.M. Kelso and A. Rosencwaig, J. Appl. Phys. 73 7035 (1993)
- [3] R. K. Leach. NPL Measurement Good Practice Guide No. 37 – The measurement of surface texture using stylus instruments (2001)
- [4] F. Gao, R. K. Leach, J. Petzing and J. M. Coupland. Meas. Sci. Technol 19 (2008) 015303. Surface measurement errors using commercial scanning white light interferometers
- [5] L. Brice, S. Morris, J. Motely, R. K. Leach – Euspen International Conference – (May 2008)- Measurement of single and multi layer thin film coatings for medical implants

11. Appendices

Appendix I:

Development of the EOS BPR device and 4 day consultancy

Development of the EOS device and 4 day consultancy work:

The BPR device produced by EOS is as shown in Figure 25, with the optical measurement system set up on top of the microscope. The software used to control the instrument is written in Matlab.

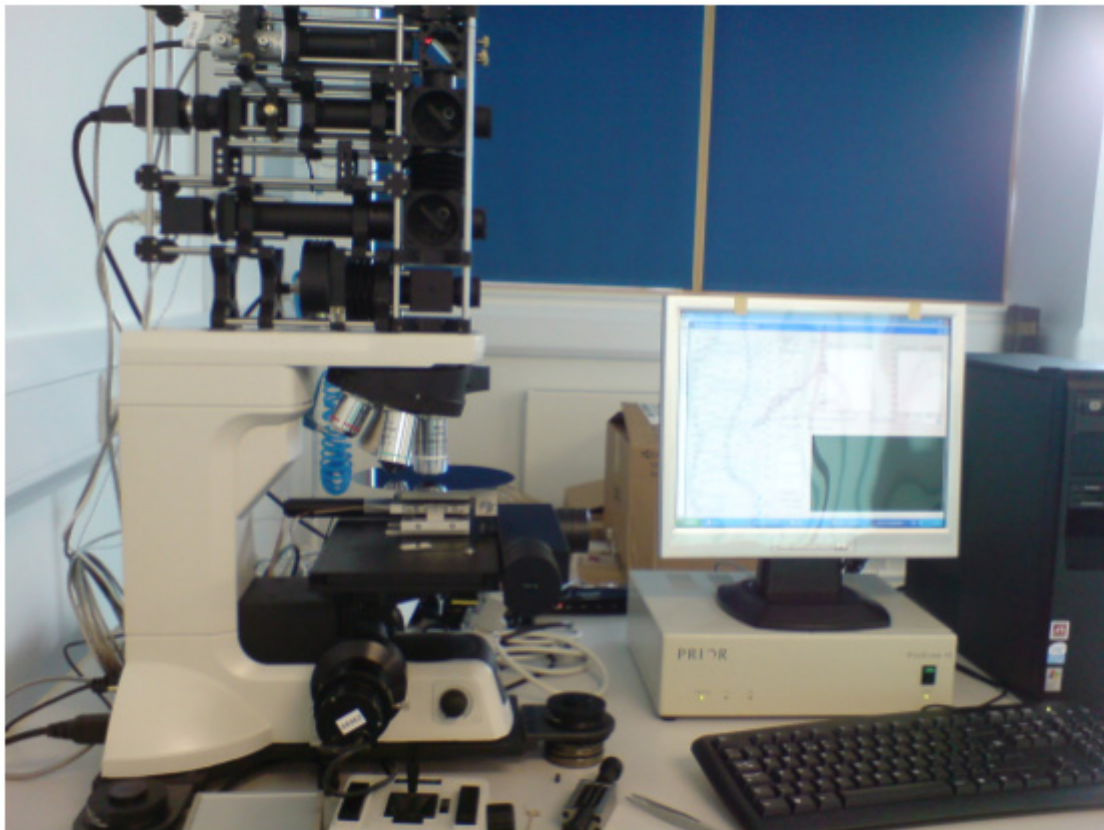


Figure 25. Picture of the BPR instrument .

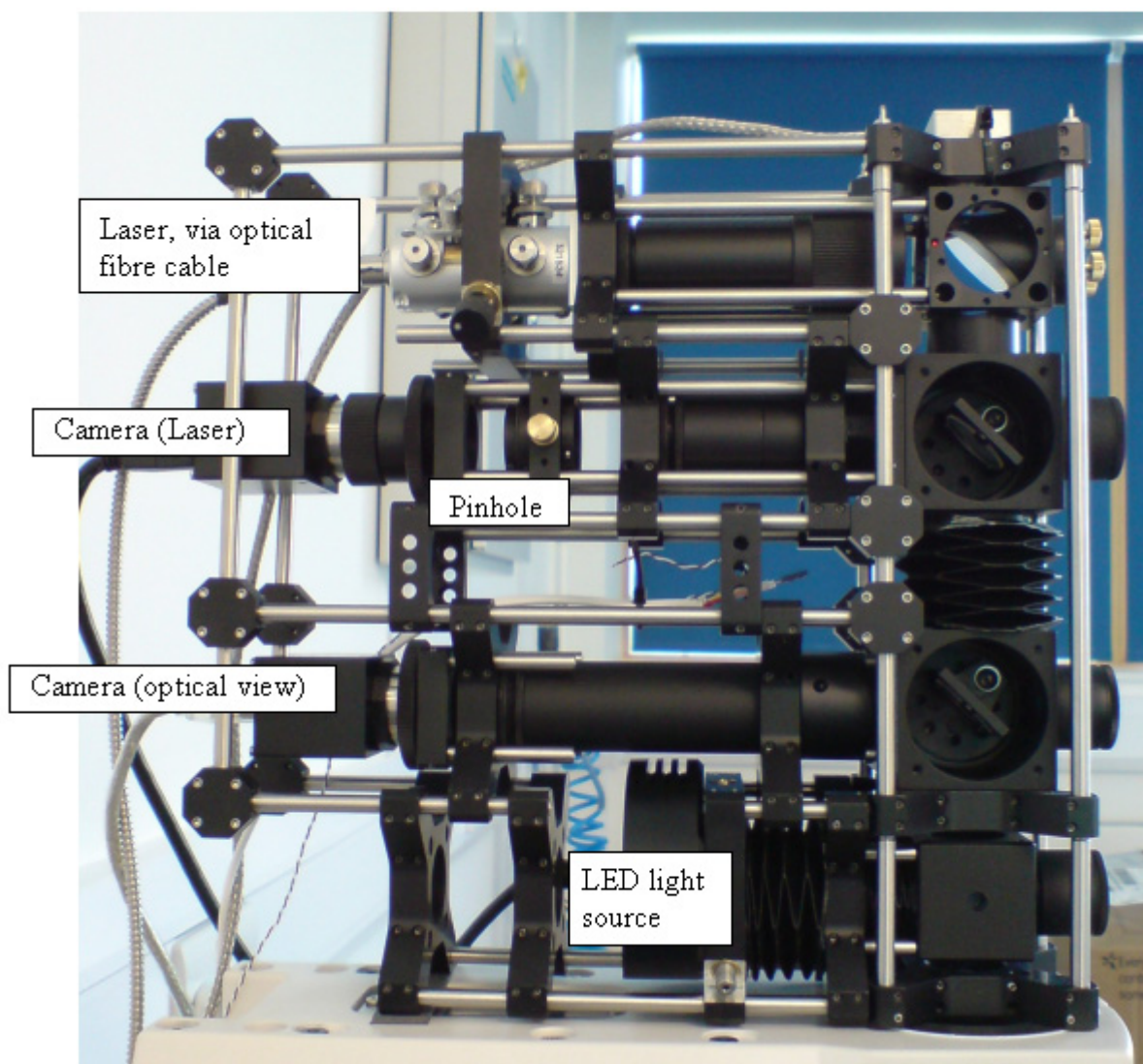


Figure 26. Optical set up of the device.

Figure 26. Shows the optical set up of the device. There are two separate cameras, one to display the optical image through the microscope and which is lit via an LED light source, and a second, which picks up the laser spot used for the actual measurements after it has passed through a 400 μm pinhole, (the diameter of the focused spot on the test surface is $\sim 13\mu\text{m}$.)

The main problem with the system was the time taken to align and take measurements. The focusing of the laser is the main area, which would be improved if automated; several methods trying to achieve this, were implemented, with varying success:

Basics: The main method for identifying the point of best focus is when the intensity of laser light, reflected back to the camera through the pinhole, is at a maximum. The higher the lens magnification the more distinctive the peak, there should be no false maximum peaks, so the maximum intensity will be at the point of the best focus. The intensity value is obtained from the image of the reflected laser spot and calculated for each grabbed frame from the relevant camera to give a value usually around 50 –100. Figure 27. shows a diagram demonstrating this relationship.

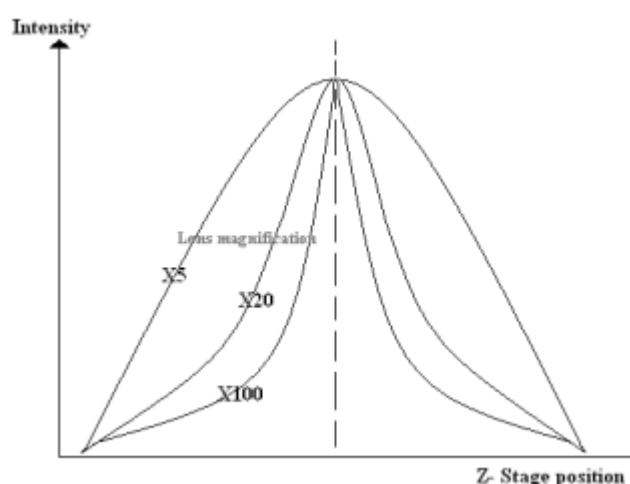


Figure 27. Example of light intensity, to stage position, and variation between lenses.

Of the different methods implemented to resolve the problems there have been issues with each:

Optimisation Routine: An off the shelf routine which takes samples across the range of the stage, and then goes to the maximum value. This would only take a sample of the data at set intervals and would occasionally miss the highest peak, even when adjusting the number of sample points.

Dynamic stepping: Running the stage through its range, while monitoring the intensity value seen, relating the maximum to the position after the sweep and automatically moving to that point. This did not seem to work due to delays in recording the relevant data. There would be a delay between the intensity value and when the position for that reading was taken to when a signal was sent to move a certain number of steps. There is also a delay and effect of the momentum of the stage when stopping, during this technique.

The camera displays 18 frames per second, which was thought to be a problem, as the stage might be travelling too fast to obtain enough intensity values to find the peak, and therefore sometimes miss the main peak.

An iterative routine is mainly used, starting with an initial fast sweep in both directions to home in on the maximum intensity, then a further sweep in 10um steps over the peak area, and finally, accurately, homing in on the peak by running at 1 um steps. This has a success rate of about 4 in 5, although sometimes falls down on the lower magnification lenses, due to noise. The time taken is also a factor of 10 slower than required, at about 10 seconds.

The intensity value used is from each image taken by the camera, the frame rate of this is important to the speed of the useful readings taken, and control of the stage in relation to the camera and data from it. Data is obtained via an Active X controller interface and a delay in receiving data from this control causes a discrepancy between the stage location and intensity value for that location

4-day consultancy

Based on the problems with the device a four-day consultancy was set up to resolve the synchronisation problems. This would improve the usability of the device, speeding up the measurement process to a level where it could be used commercially.

The cause of the problem was identified and by consultation with Nightingale EOS and the company who provided the control electronics (Prior), the best possible solution was identified.

Examining of the system, and control system manual, identified that the ProScan was provided without encoder inputs, and relied on the stepper motor pulse count to determine location, so there was no encoder to synchronise to. Fitting an encoder and building an interface would allow us to achieve synchronised, on-the-fly, image capture. Other alternative ways of synchronising using the TTL output were investigate through discussions with Prior.

An option based on their "trigger board" seemed suitable; it can be set up (using the TRIGSET command) to output a trigger pulse at specified distance intervals. This trigger pulse connects to the camera to initiate a frame capture. It is possible to set up to move continuously over a specified range and capture images at specified Z separations without the need to stop. This option was considerably cheaper than fitting an encoder. Both these options were put forward and formed the bases for future development of the BPR instrument.

Appendix II:

Supplementary data and analysis from the re-measuring of the artefacts

Re-measurement of NPL standard artefacts with new optic design

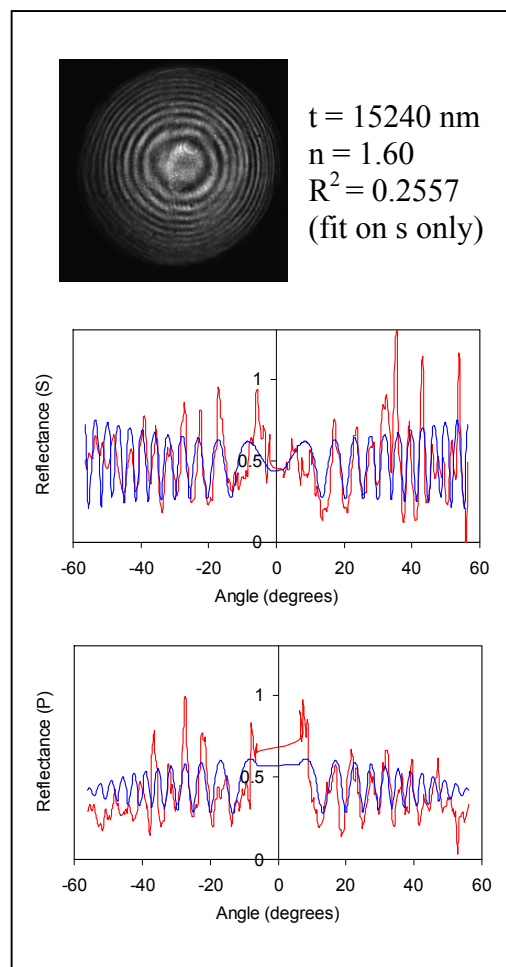
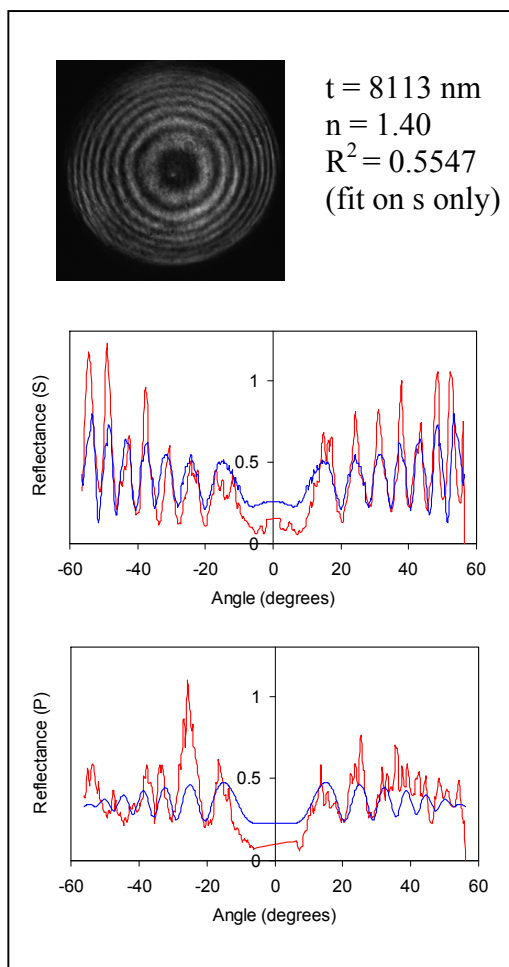
The results obtained after re-measuring the artefacts using the modified optical configuration are given below. Figures 28 to 36 show the data obtained by the BPR during this re-measure including the optical image of the fringe patterns and the fitted graphs. The coating thickness measurements for each are also shown including the refractive index obtained from the BPR measurement.

The issue of having too many fringes causing a difficulty in mapping the fringe pattern on the thicker (“sample 1”) films has been eliminated by correctly locating the image of the Fourier plane. Results are now, in general, more consistent with the NPL values presented in the EUSPEN paper [4].

Table 3 shows the original NPL measurements of the artefacts and these re-measurements by Nightingale EOS. As can be seen the values are very much more consistent, without the previous errors found with the thicker samples. The differences to the NPL measurements are all now very low; all but two (including the thicker coating samples) are less than 0.41 μm going down to 0.03 μm . The other two are still only in the region of 1 μm different from the NPL results. These are extremely good comparative results considering the variation in the coating thickness and surface roughness.

Figure 37 shows a comparison of this results looking at the R^2 comparative values. As can be seen overall thickness correlation across the whole range is now excellent ($R\text{-squared} > 0.98$) All the results here were obtained with essentially the same system settings, fitting on the s-polarised profiles and allowing the thickness, refractive index and scatter coefficients to be selected by the algorithm as apposed to fixing them before fitting the data. The range of values of refractive index obtained after data fitting is surprising, and is much wider than expected. Even so, allowing the index to take these values gives the best fit both to the raw data and to NPL’s thickness values, so this anomaly has been ignored for the time being.

As previously mentioned some of the coatings have large variations in thickness, and it is possible to find areas with thicknesses very different from the ‘average’ thickness, again emphasising that the coating variation is the limiting factor.

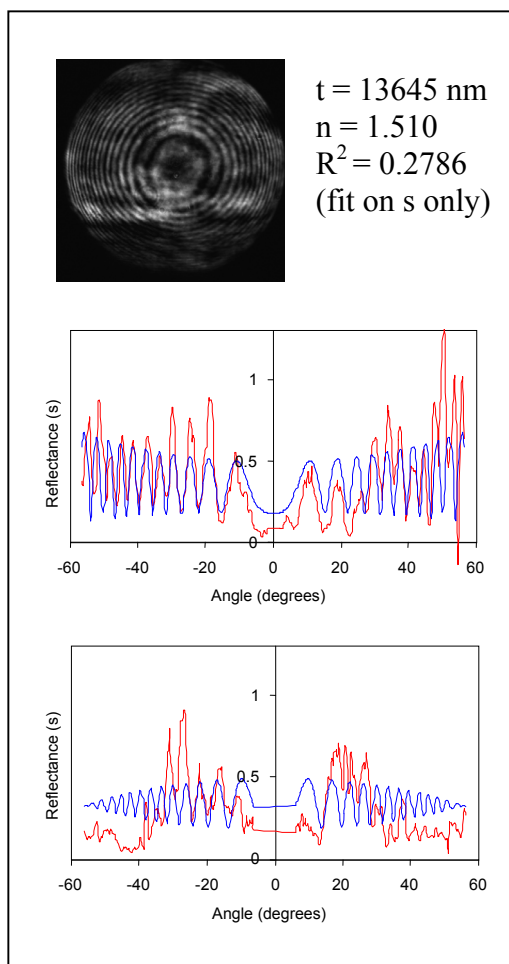


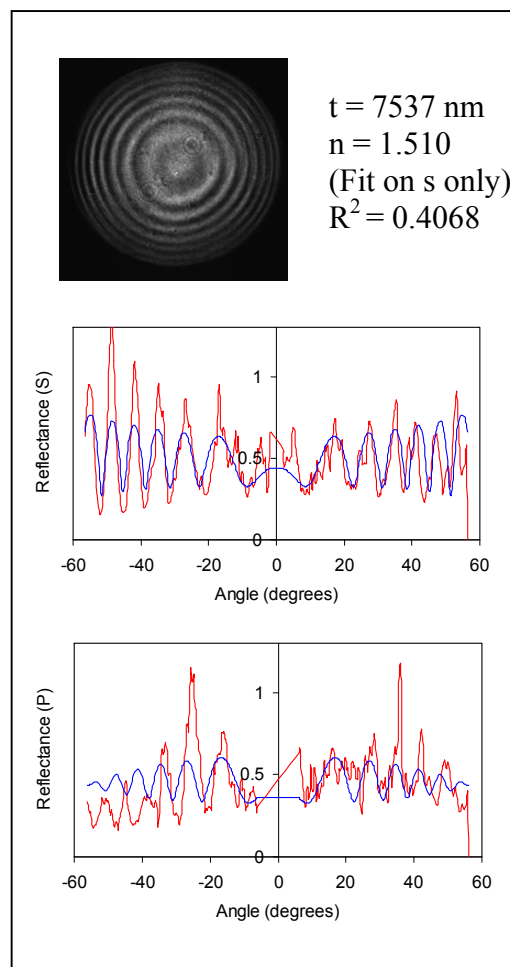
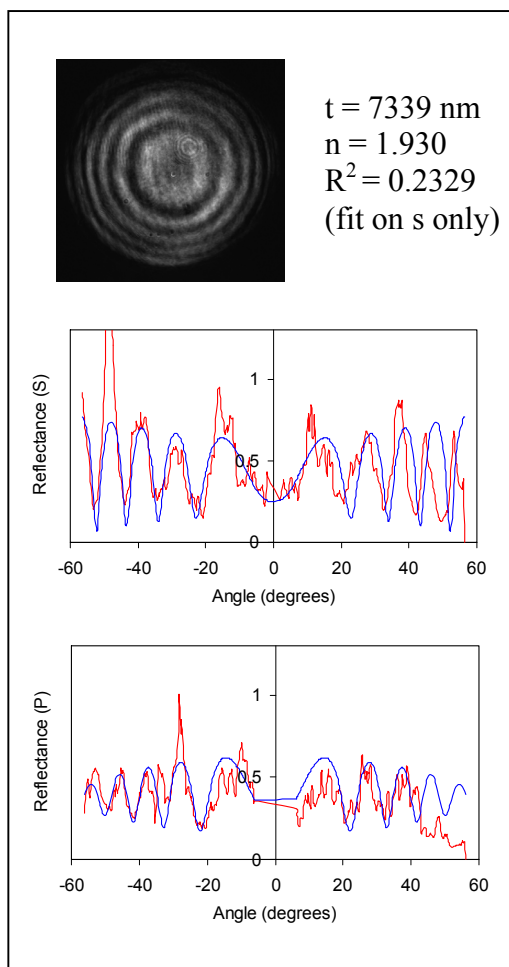
(Clockwise from top left)

Figure 28. Sample 1, 2 mm tube.

Figure 29. Sample 1, 1 mm tube.

Figure 30. Sample 1, 50 μm wire.



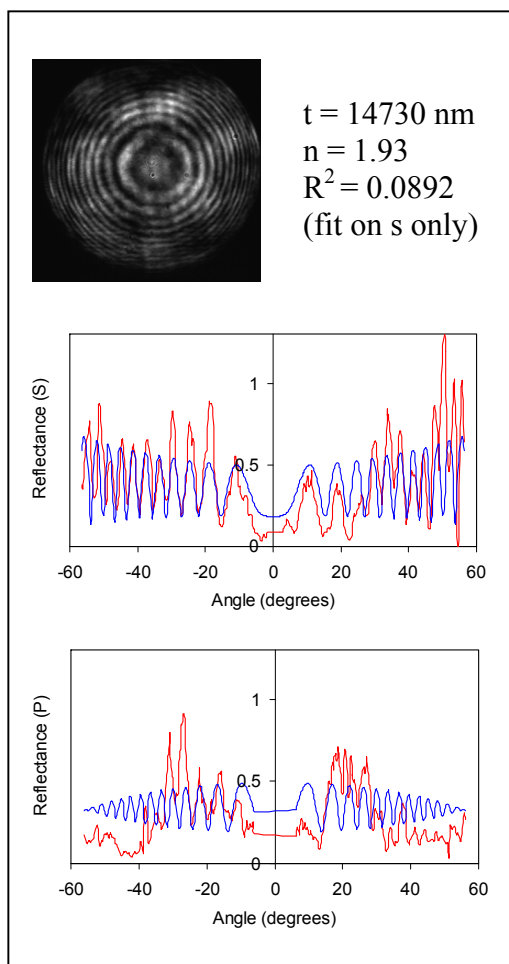


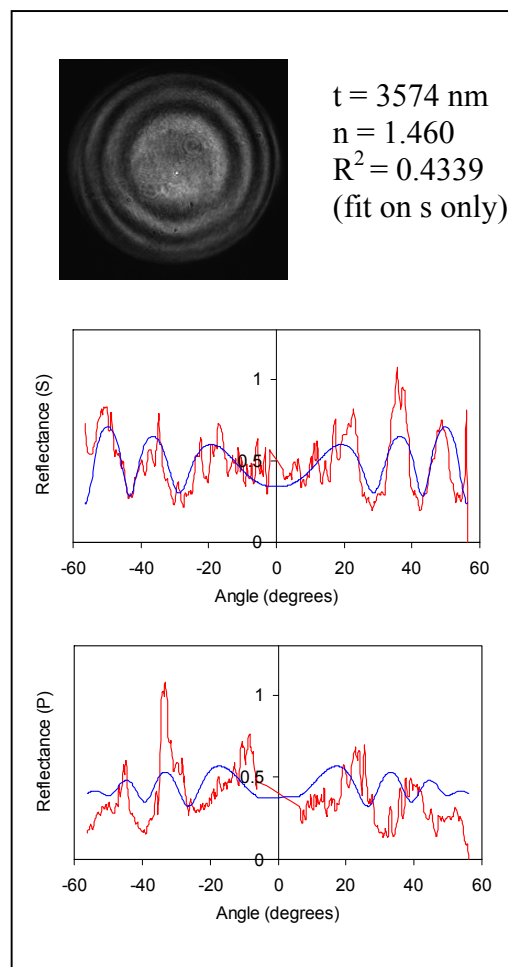
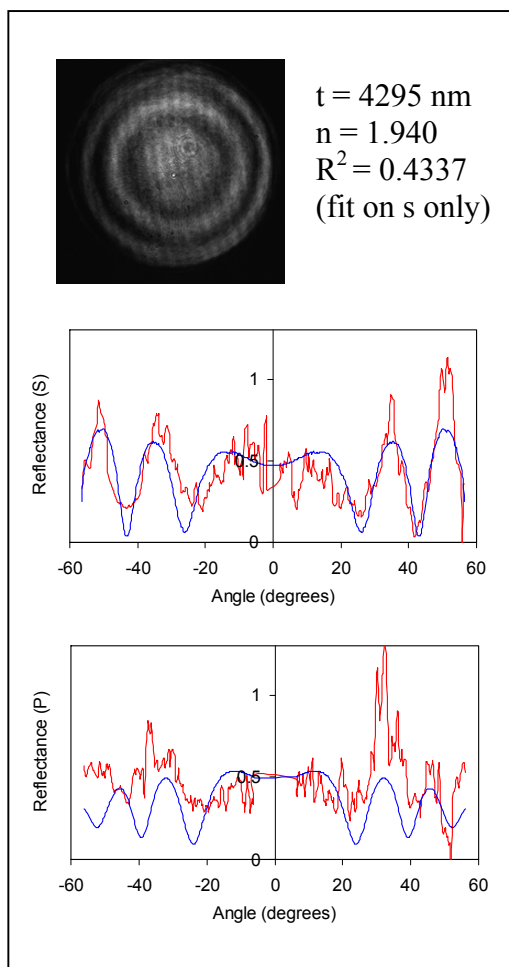
(Clockwise from top left)

Figure 31. Sample 2, 2 mm tube.

Figure 32. Sample 2, 1 mm tube.

Figure 33. Sample 2, 50 μm wire.





(Clockwise from top left)

Figure 34. Sample 3, 2 mm tube.

Figure 35. Sample 3, 1 mm tube.

Figure 36. Sample 3, 50 μm wire.

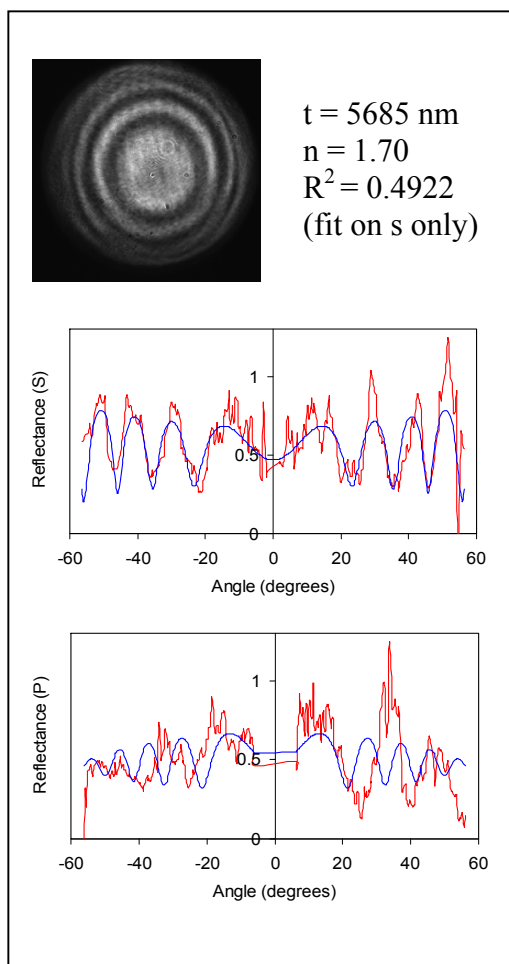
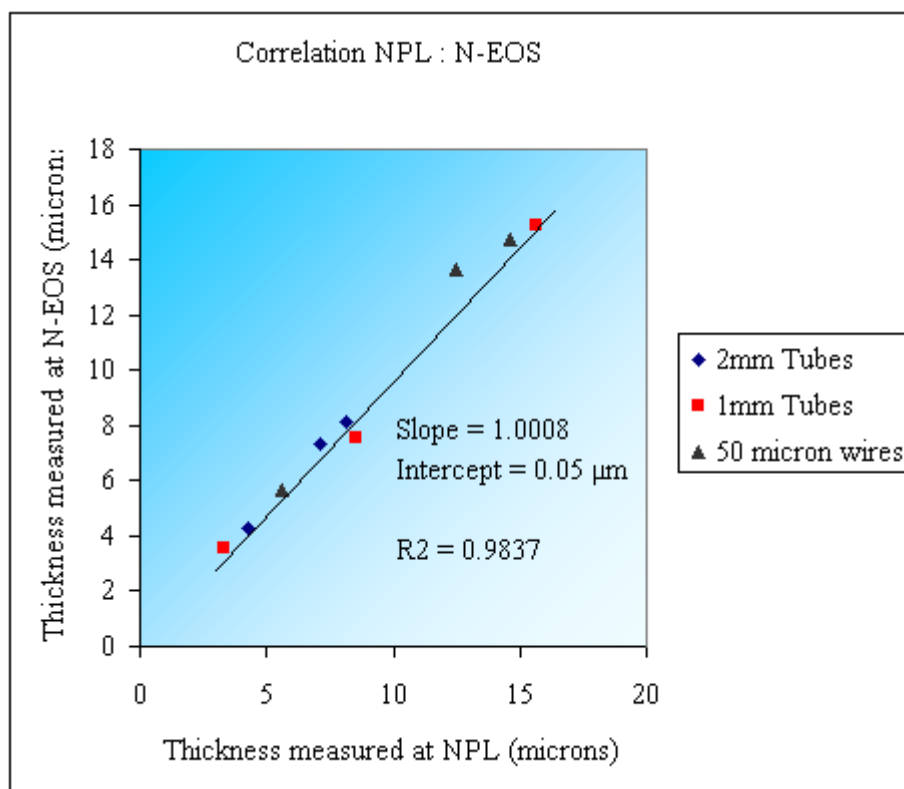


Table 3. Table of final results from NPL and Nightingale EOS

2 mm tubes			1 mm tubes			50 μ m wires	
NPL	N-EOS		NPL	N-EOS		NPL	N-EOS
8.14	8.113		15.65	15.24		12.47	13.65
7.12	7.339		8.51	7.537		14.6	14.73
4.24	4.295		3.3	3.574		5.59	5.685

**Figure 37.** Correlation of NPL to final N- EOS results.