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FEASIBILITY OF USING SCANNING ACOUSTIC MICROSCOPY TO PROVIDE LARGE AREA, HIGH SPATIAL AND THICKNESS RESOLUTION MEASUREMENTS OF FLEXIBLE POLYMERIC THIN FILMS

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Feasibility of Using Scanning Acoustic Microscopy to Provide Large Area, High Spatial and Thickness Resolution Measurements of Flexible Polymeric Thin Films

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ABSTRACT

This report details work carried out to determine the feasibility of high spatial and thickness resolution measurements of flexible polymeric thin films using immersion-based scanning acoustic microscopy (SAM). In principle, by measuring the ultrasonic time of flight (ToF) normal to the film surface and using the ultrasonic velocity of the material, the thickness of the film at any point can be determined. This work aims to verify the methodology and identify issues in the characterisation of film thickness or mapping which may contribute to uncertainty. A mid-range frequency/resolution scanning acoustic microscope system (75 MHz and 100 μm) was used in a variety of imaging modes, yielding signal intensity images, as well as cross-sectional slices, to maximise characterisation of the film surface topography and thickness distribution. The thickness maps generated using acoustic microscopy were verified and validated using appropriate calibration artefacts and estimates made of the measurement errors and their sources.

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Approved on behalf of NPLML by
M R L Gower, (Science Area Leader)

CONTENTS

1 BACKGROUND

Ultrasonic thickness gauging is a means of testing the local thickness of materials (usually applied to solids and frequently specifically metallics within an industrial setting) which are inaccessible to traditional mechanical thickness measurements e.g. closed vessels with one-sided access only, central region measurements on large area components, layer thicknesses in multilayer components or coatings/paint. Measurements must be calibrated for each material since the ultrasonic velocity in different materials, microstructures or at different temperatures change significantly and has an influence on the accuracy of thickness determination.

The method is based on the time taken for an ultrasound wave to travel through the material, also known as time of flight (ToF). These measurements are often performed in direct contact and from only one side. As a result, high frequency piezoceramic transducers (>5 MHz) with built-in delay lines are required to provide signal separation and optimal thickness resolution, couplants are also needed to join the probe to the measurement surface of interest which can be often rough. Alternatively an electromagnetic (EMAT) transducer can be used which avoids some of these issues but is restricted to use with metallic or magnetic materials.

Ultrasonic gauging works well for many applications e.g. internal oxide thickness in steam plant, steel pipe wall remnant thickness after internal corrosion losses, but also has some limitations. Contact techniques tend to be spot measurements, each averaged over relatively large areas (transducer diameters commonly 3-12 mm) and these are not easily suited to very thin, flexible, small radius curvatures or soft/compressible materials.

Immersion-based scanning acoustic microscopy (SAM) uses high frequency focused transducers (beamwidth <0.5 mm) and fast, accurate time sampling with large area scanning capability to provide detailed imaging of complex internal architectures. This work determines the feasibility of high spatial and thickness resolution measurements of flexible polymeric thin films using SAM. In principle, by measuring the ultrasonic time of flight (ToF) normal to the film surface and using the ultrasonic velocity of the material, the thickness of the film at any point can be determined. The thickness maps generated using acoustic microscopy are verified and validated using appropriate calibration artefacts and estimates made of the measurement errors and their sources.

2 EXPERIMENTAL PROCEDURE

2.1 SAMPLES

Three polyurethane sample films (suitable for biomedical application) were used to demonstrate the thickness measurement capability of scanning acoustic microscopy. These films were made from multiple layers of chemically modified polymer and were soft (compressible), flexible (deformable under gravity) and rubbery (stretchable). The film dimensions were approximately 100 mm square and < 200 μm thick. An example of a typical film is shown in Figure 1, also indicating the variability in thickness as observed from the upper film surface.

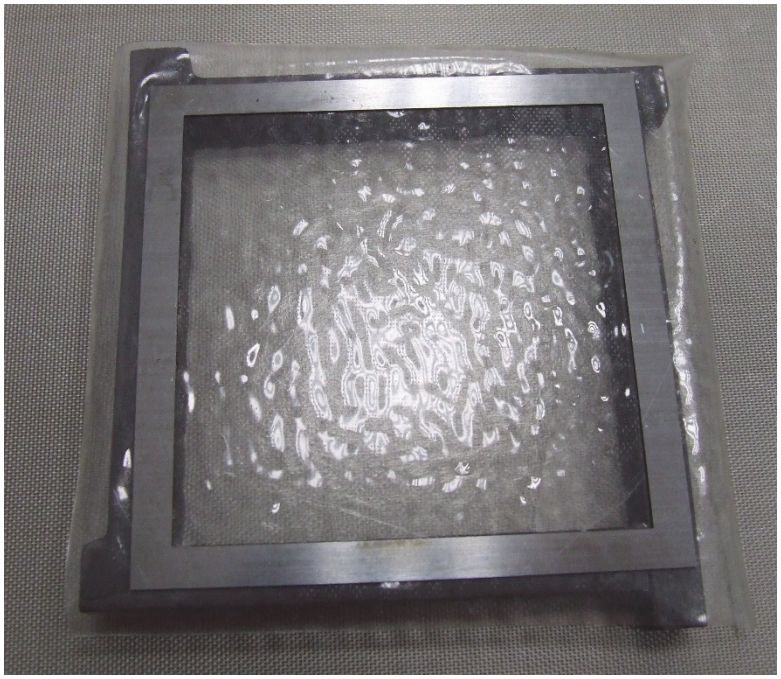


Figure 1 - Image of sample film, held tensioned in a clamping frame designed to enable SAM of the flexible polymer films.

2.2 CALIBRATION/VERIFICATION ARTEFACT

The suitability of employing scanning acoustic microscopy time of flight (SAM ToF), as well as the other thickness measurements used for reference and comparison in this study, was verified using a glass microscope slide with a single well. This is a highly reproducible artefact made from soda-lime silica glass, shown in Figure 2, having a uniform plate thickness and well-characterised acoustic properties which are readily available in the literature (e.g. an acoustic velocity for bulk longitudinal waves V_{glass} of 5900 m/s [1, 2]).

Figure 2 - Single well glass microscope slide used to calibrate SAM ToF thickness methodology.

2.3 SUPPORT RIG

An integral aspect of the measurement process was the optimal alignment of the films, flat and perpendicular to the focused ultrasound beam to prevent cosine orientation errors during thickness measurements. Simultaneously, the film needed to be fixed firmly in place to minimise vertical undulation or sideways movement during immersion scanning. This was achieved by forming a square window base support frame from 6.5 mm thick water resistant magnetic strip, internal window dimensions ~60 x 60 mm. This was bonded at the corners, using a room temperature fast cure two-part epoxy adhesive, to provide shape rigidity and stability. The frame was fixed magnetically inside the SAM immersion tank on a Resistalloy™ baseplate (corrosion resistant paramagnetic steel) and edged with water resistant grease (Rocol Sapphire Aqua2) to prevent fluid movement in or out of the space between the film and the base plate. A 5 mm wide window frame of 0.6 mm thick tinned steel with a matching internal aperture was manufactured and positioned over the magnetic support frame. The magnetic clamping between the support and the top window, fixed the sample film firmly but gently in place. This tensioning/clamping frame is shown in Figure 1 and given schematically in Figure 3.

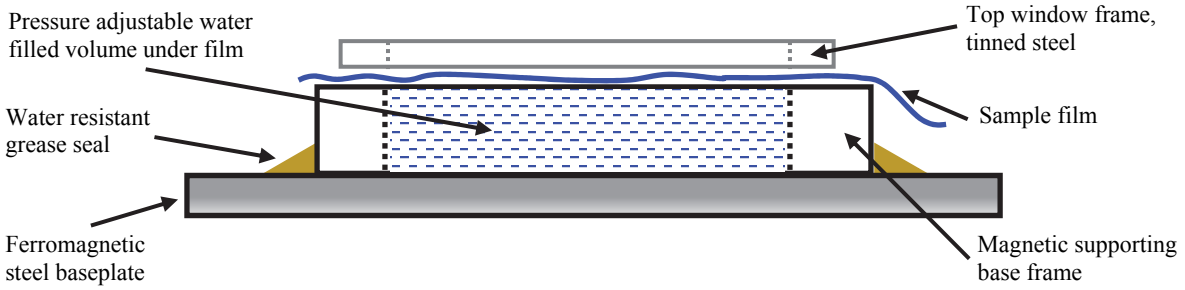


Figure 3 - Schematic of ultrasonic inspection support and clamping arrangement for flexible thin films (unsupported film area ~60 x 60 mm).

Each film was laid flat on the top window and gently pulled to tension it just enough to prevent global bulging or sagging. The aim was to minimise Poisson's contractions of the film thickness and hence provide a more realistic thickness determination whilst maintaining a smooth, nominally flat film surface. The sticky nature of the top film surface ensured this tension was maintained once the window was inverted onto the supporting frame. The thin window was sufficiently flexible to conform to the film and ensure a liquid-tight seal around the film. After positioning, both the upper and lower film surfaces were rinsed in weak detergent to minimise trapped air bubbles, and the assembly was immersed in the scanning tank.

The air contained in the enclosed volume beneath the film was released by lifting and tilting one corner of the top window frame, thus allowing the space to fill with water (shown water-filled in Figure 3). A flat film surface was obtained by adjusting the water volume (pressure) under the film. This was achieved by, again, slightly lifting one corner of the top window frame while simultaneously pressing the film surface to varying degrees, allowing water to flow in or out of the space as necessary. The film flatness was monitored using the ultrasonic time of flight between the transducer face and the film surface. This was measured at the four corners and centre of the freely suspended film area and flatness was confirmed when these values were equalised to within 0.1 μs.

On completion of all the scanning measurements, the film was marked at three corners to orientate and define the measurement region for later reference and subsequently removed from the support frame.

2.4 NON-DESTRUCTIVE TEST EQUIPMENT USED

A Sonix UHR 2001 scanning acoustic microscope, shown in Figure 4, was used to ultrasonically image the polymer film samples. The maximum scan area of the system is 250 x 150 mm and the system frequency bandwidth is 5 - 100 MHz. The inspected area is raster scanned, during which the excitation pulse is triggered and the signal amplitude is captured in synchronisation with the scan position to generate ultrasound images in pulse-echo mode. The integrated software and instrumentation controls all aspects of the transducer excitation, received signals, mechanical scanning motion and data capture/display. Samples are immersed in water to provide ultrasound coupling and scans are performed in pulse-echo mode at 5 - 100 μm lateral steps.

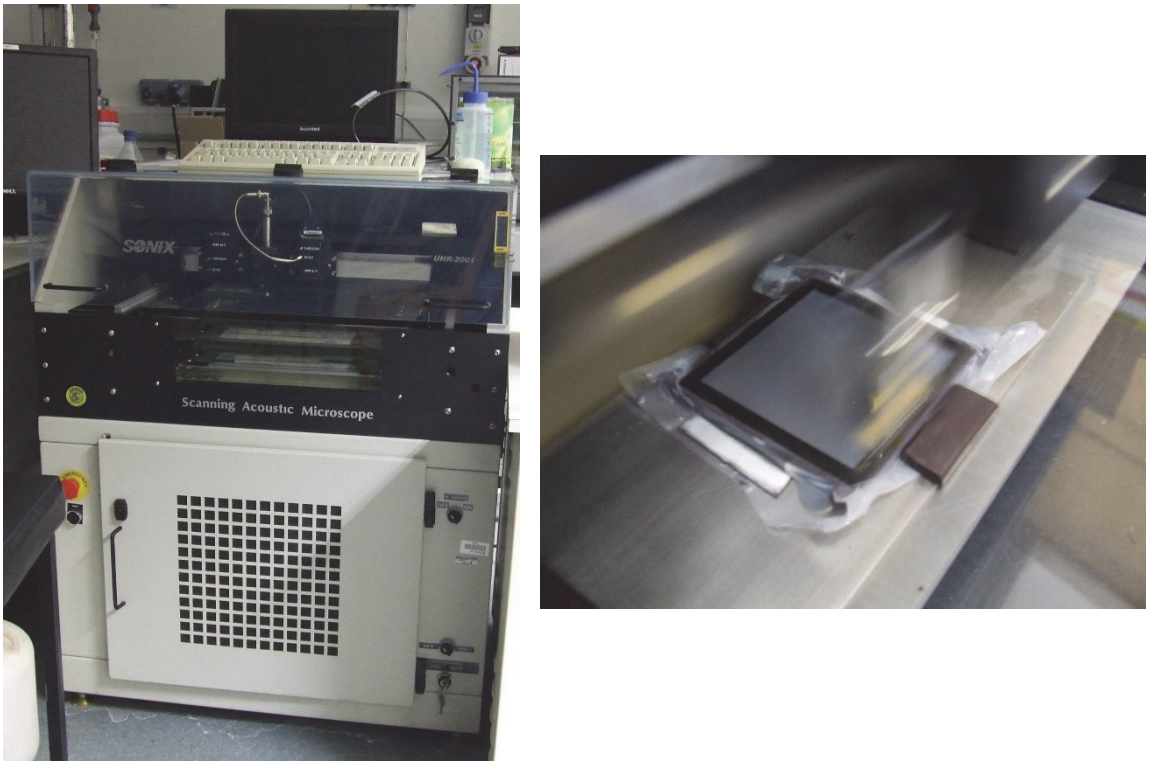


Figure 4 - Ultrasonic scanning microscope (left) and close-up of clamped immersed film during scanning (right).

This is a lower frequency range, large area, high penetration SAM ultrasonic inspection method. It is particularly sensitive to air interfaces, even very thin delaminations, and is effective for penetrating through most materials including liquids, solid polymers, ceramics and metals, multi-material or layered architectures. The system comprises a flatbed xyz axis scanner. It lends itself to multiple data representation and visualisation schemes which highlight different aspects of the inspected part: warpage, thickness of layers, depth location of features and internal/surface defects by virtual ultrasound sectioning in any direction or over any volume.

2.5 INSPECTION PROCEDURE

Following initial trials with an example film, all SAM measurements were subsequently made at 75 MHz (beam width ~0.2 mm) in pulse-echo mode and with the transducer focused on the rear face of the flexible film (focal length ~12 mm). All sample scans were performed with the same basic system settings, listed below.

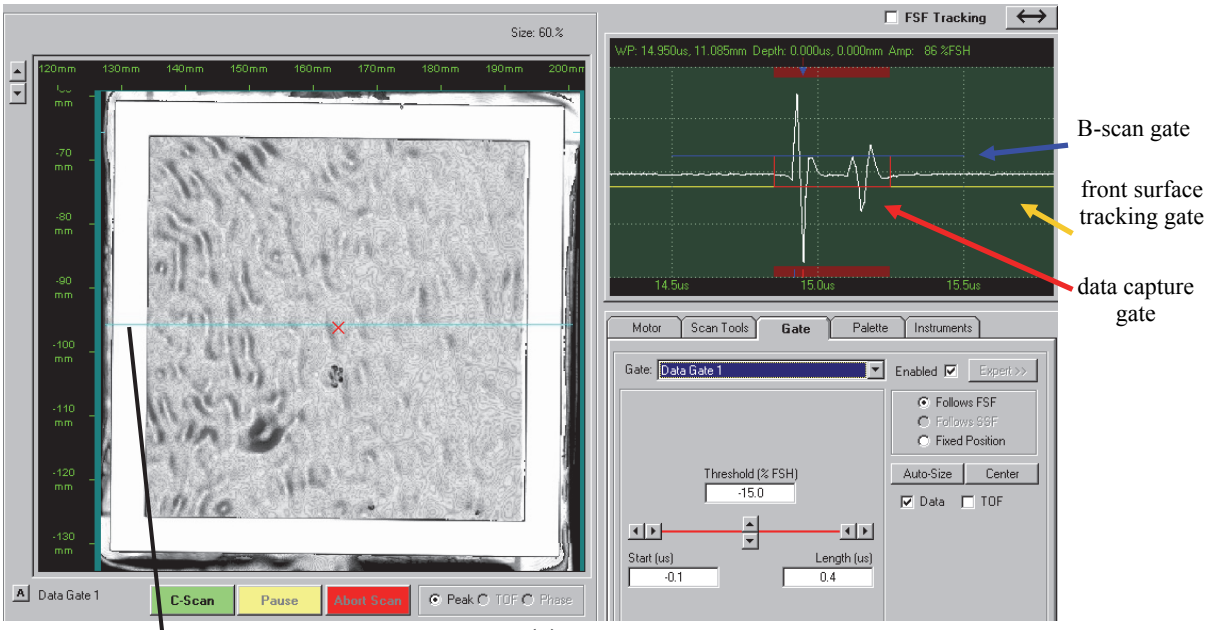
- 0.5 V receiver range,
- 330 V excitation spike with an extra ultrasonic pulse energy boost applied,
- scan area 75 mm x 75 mm,
- 100 μm spatial sampling interval,
- 75 mm/s scan speed (automatically reduces to 70 mm/s at highest sampling frequency to accommodate increased data capture rate) with acceleration/deceleration rates of 500 m/s^2,
- 32 dB receiver gain.

Pulse-echo SAM produces a reflected waveform at each sampled point, i.e. a series of return echoes with time, effectively probing the part depth. These return signal intensity-time series are called A-scans and are the basis for all acoustic imaging. Adjustable gating is used to define a threshold triggered time window within an A-scan, corresponding to a depth interval, from which amplitude or time of arrival information (usually from the largest signal within the window) can be acquired during uninterrupted mechanical scanning. Simple A-scan data can be captured, processed and represented in many ways. A typical A-scan and data capture setup from the sample films is shown in Figure 5.

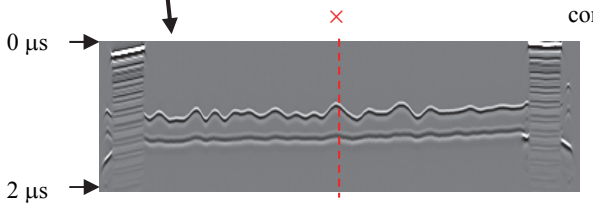
Several threshold triggered gates were used to capture data from the A-scans acquired at each scan point of the sample films, as indicated in Figure 5(a):

- A front surface following gate was used to identify the location of the top of the film as triggered by a minimum threshold amplitude;
- A data capture gate was located relative to the front surface trigger to capture the peak signal amplitudes above a minimum threshold level, and their arrival times, for post-processing as C-scans or thickness scans;
- A B-scan gate was located at a fixed time to capture all of the A-scan data without thresholding, for post-processing as B-scans.

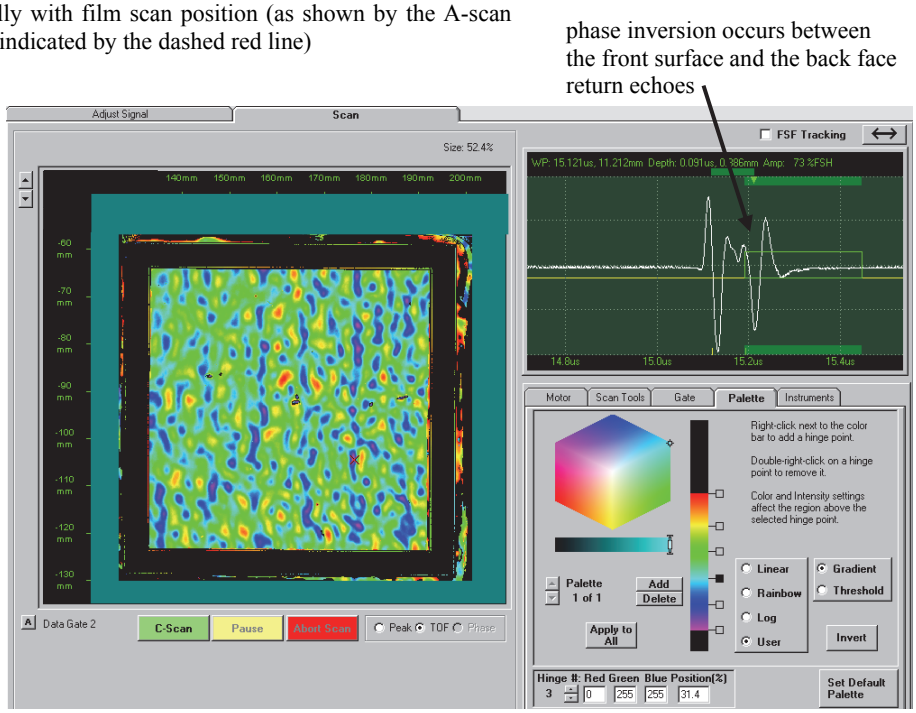
Examples of the data representations A-, B-, C-scans and thickness maps are shown in Figure 5(a) – (c).



(a) A typical A-scan return signal from the point marked 'x', capture gates and associated thresholds (right) and the C-scan image (left) corresponding to return amplitude with film scan position



(b) A typical B-scan cross-section from the scan line highlighted where the A-scan intensity data is presented in greyscale and plotted vertically with time and horizontally with film scan position (as shown by the A-scan data at position 'x' indicated by the dashed red line)



(c) A typical thickness (ToF) map where the colourscale corresponds to the time lapse between signal arrivals (shown in the A-scan) for every film scan position

Figure 5 - SAM data capture and representation.

The captured data were processed for subsequent display according to the following descriptions:

- C-scans were captured by monitoring the maximum signal amplitude returned within a fixed length time window, offset from the front surface echo by a defined delay. These are a representation of the amplitude response of a whole plane of the inspected part at a particular depth and correlate to an acoustic 'image' shown in Figure 5(a).
- B-scans were captured by monitoring the time variation, relative to the start of a gate which is fixed in time, of the whole A-scan. This represents the A-scan amplitude with time (converted to greyscale) for every point in a single scan line across the part showing reflection locations with depth and correlates to an acoustic slice cross-section as shown in Figure 5(b). As such, variations in the relative signal arrival times from the part can be analysed for distortion or thickness or locating defect interfaces. The separation between the front and back surface echoes at any point is directly related to the film thickness via an accurate and constant material acoustic velocity.
- Thickness scans were captured by measuring the absolute time delay between the front surface and back surface arrival times and plotting this over the whole scanned area as shown in Figure 5(c). Again, for an assumed constant material velocity, this is directly proportional to the material thickness.

Previous work carried out at NPL has shown some value in performing scans at different data sampling frequencies to highlight thickness or surface variations of an inspected part. Four separate scans were performed for each sample film to optimise different characterisation parameters, individual scan settings are described in detail in Table 1:

Scan (1). Front and back face C-scan and thickness maps – surface and back face signal intensity maps and a thickness map based on the time difference between arrival of front and back face signals;

Scan (2). Back surface C-scan and thickness maps – back face signal intensity maps and a mathematically convertible thickness map based on the time differences between front surface trigger and back face signals;

Scan (3). High sampling frequency back surface C-scan and thickness maps – back face signal intensity maps and a qualitative thickness map based on the time differences between front surface trigger and back face signals (banded every 32 ns of transit time) useful to help visualise the surface topography, displaying detailed thickness contours and highlighting underlying thickness trends or randomness;

Scan (4). Low sampling frequency front face C-scan and full B-scan – front face signal intensity maps providing contour indications and cross-section slices for each scan line to provide qualitative visual representations of the sample film orientation/alignment and thickness.

Table 1 - Summary of inspection settings

<table>
<tr><th>Inspection mode</th><th>Sampling frequency (MS/s)</th><th>#B-scan gate (µs)</th><th>*Front surface C-scan gate DG1 (µs)</th><th>*Back surface C-scan gate DG2 (µs)</th></tr>
<tr><td>Front and back surface C-scan and ToF</td><td>1000</td><td rowspan="3">N/A</td><td>Start –0.05
Length 0.1
Threshold –15%FS</td><td rowspan="3">Start 0.065 or 0.070
Length 0.256
Threshold 15%FS</td></tr>
<tr><td rowspan="2">Back surface C-scan and ToF only</td><td>[a]1000</td><td rowspan="2">N/A</td></tr>
<tr><td>8000</td></tr>
<tr><td>Front face C-scan and full B-scan</td><td>100</td><td>Start 14.5
Length 1.0
No threshold</td><td>Start –0.1
Length 0.4
Threshold –15%FS</td><td>N/A</td></tr>
</table>

‘S/s’ = samples/second sampling frequency
‘FS’ = full scale
‘N/A’ indicates not applicable
‘#’ gate fixed in absolute time
‘*’ gate with fixed delay relative to front surface echo
‘[a]’ scan data used for thickness calculations

Front wall echo located at ~ 15 µs

Front surface follower gate in all cases set to start @12.5 µs with a length of 5 µs and –15%FS threshold

3 RESULTS AND DISCUSSION

Acoustic images generated using SAM are generally intuitive and readily interpreted. The contrast mechanism arises from the interaction of the acoustic elastic waves with the materials encountered during propagation. The waves may be absorbed, scattered and/or reflected at any boundary where the acoustic properties of the materials change along the propagation path, providing a high degree of sensitivity to variations in material properties (acoustic impedance), interfaces and surface topography. Any solid to air/vacuum interface results in the reflection of almost 100% of the incident energy due to the vast difference in acoustic impedances which makes this technique very sensitive to air-based defects, e.g. voids, porosity, delaminations etc.

Pulse-echo/reflection mode uses a single focused ultrasonic transducer that transmits the outgoing pulse and also receives return echoes. This mode can locate the depth or identify which interface has a defect and provides images with a high degree of spatial detail.

A selection of typical results from the SAM inspections for one sample film used in the study are given in Figures 6 – 10. Dimensions and measurement scales are indicated.

Some isolated regions of the thickness scan results show discontinuous data, as highlighted in Figure 6 and visible in B-scans in Figure 10, due to one of the following:

- Front surface signal loss (and corresponding back face signal loss) due to sudden localised changes in surface topography scattering the beam such that no threshold triggering is obtained for either front and/or back surface gates;
- Triggering within a gate on a different signal than expected such as:
  - ⇒ for low front surface threshold level settings, distortion of the front wall signal due to anomalies causes a small amplitude return before the main front face signal which can lead to premature triggering and a step increase in the transit time measured or a step drop in intensity;
  - ⇒ for back face signal gates, data are captured for time and intensity from the largest amplitude peak signal - consistently either the positive or negative peak - but surface anomalies can sometimes alter the balance of the relative peak amplitudes resulting in triggering on the alternative peak which is several nanoseconds displaced often with seemingly little change in signal amplitude;
- Film thicknesses corresponding to > 255 ns ToF resulting in data wraparound (then 256 ns = 0, 257 ns = 1 etc. in the data captured to file); this is easily recognised from the discontinuous nature of the data over a relatively large area where this occurs since usually the film surface data are smoothly continuous.

All signal amplitudes are recorded within B-scan gates, even sub-threshold, which can often aid in discriminating between the effects described above, e.g. surface anomalies, out of range thicknesses etc. Examples are given in the selected B-scans in Figure 10 where the full-scale vertical axis of each cross-section slice corresponds to 1 μs.

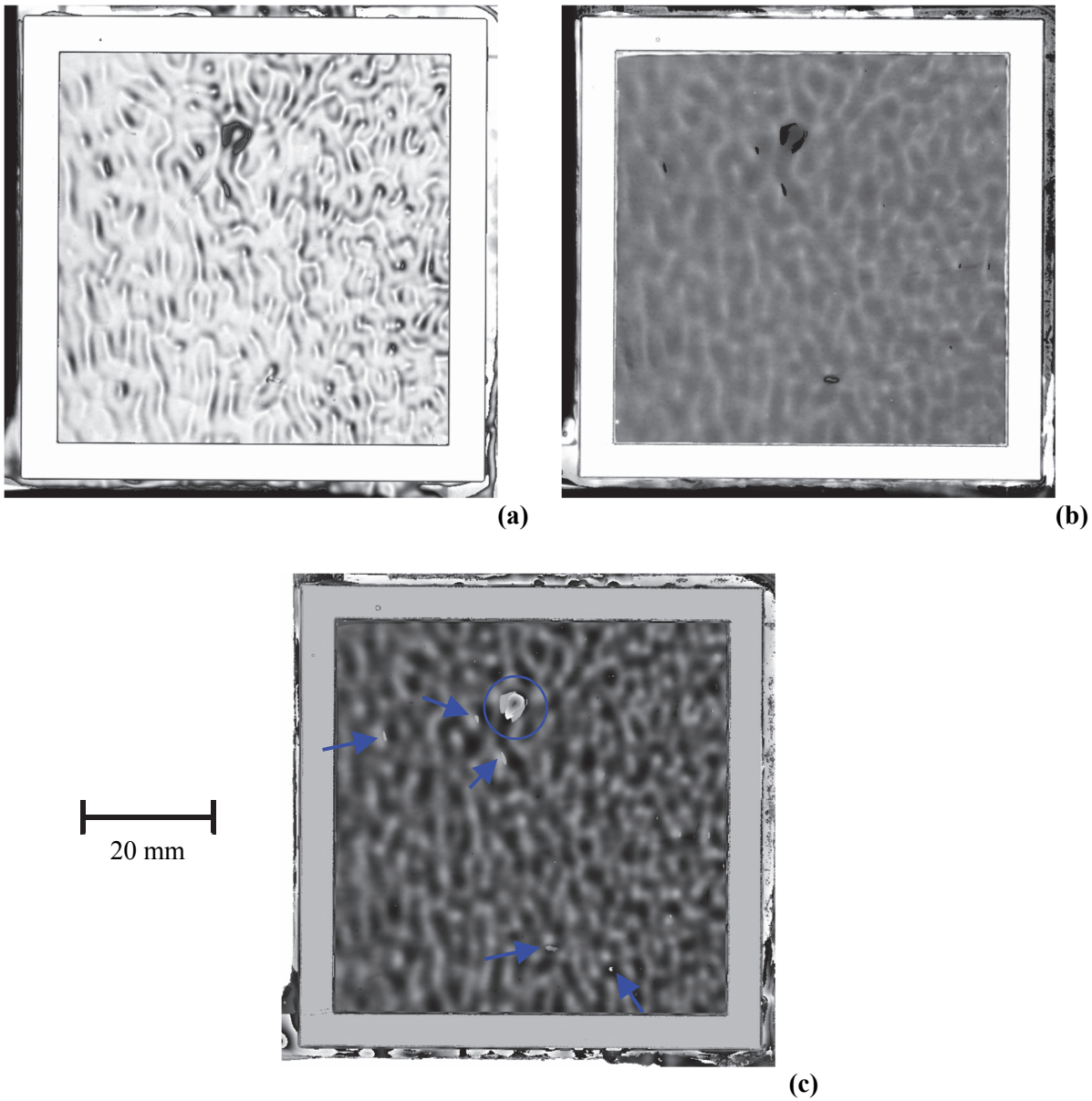


Figure 6 - Ultrasonic SAM images of a sample film produced by scan type 1 showing (a) top surface signal intensity map, (b) back face signal intensity map and (c) film thickness map. Discontinuities caused by large thicknesses are marked with circles, signal loss/wrong trigger effects are marked with arrows.

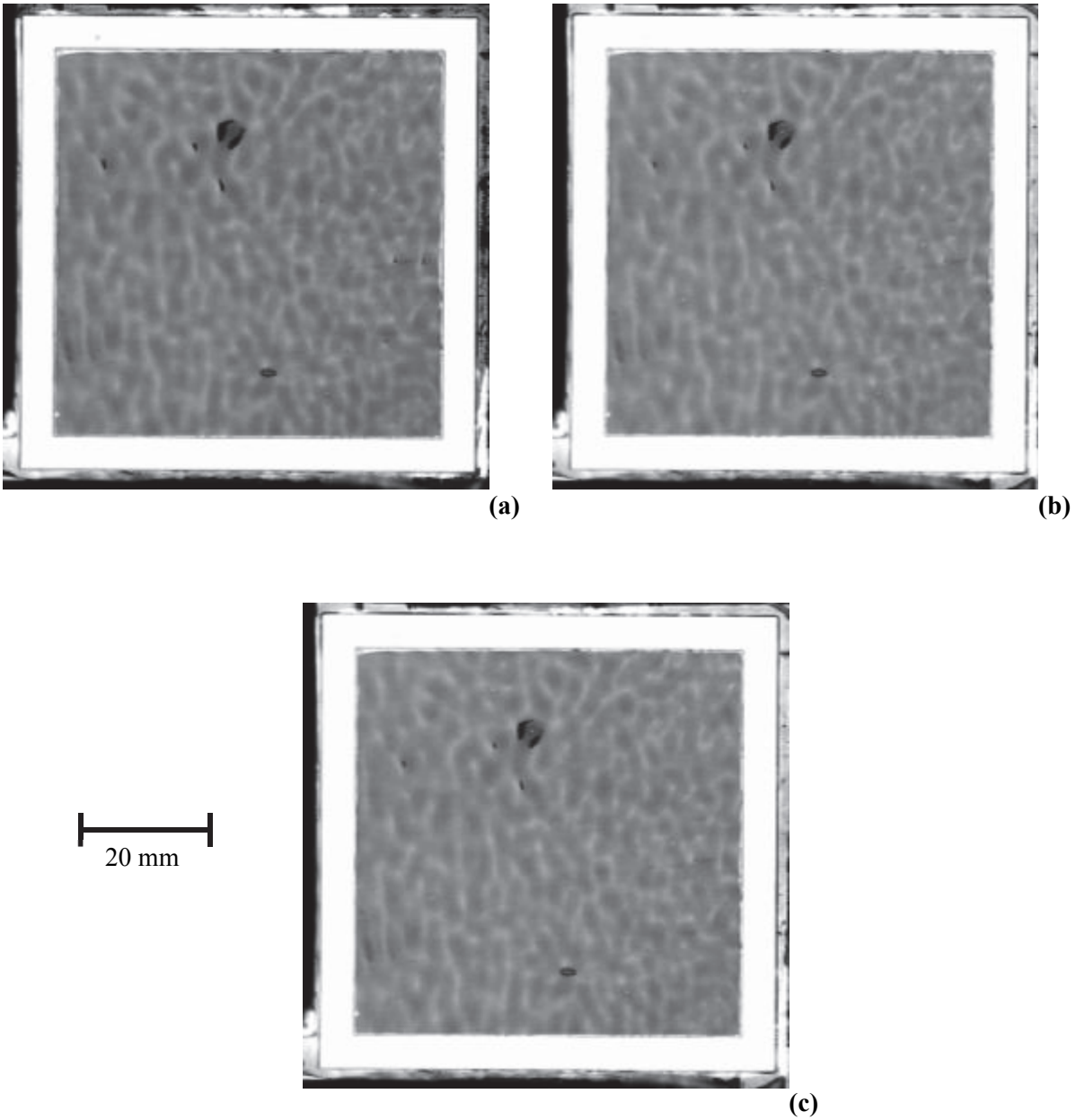


Figure 7 - Ultrasonic SAM images of a sample film showing repeatability of back face signal intensity maps produced by inspections of the same sample configuration with (a) scan type 1, (b) scan type 2 and (c) scan type 3.

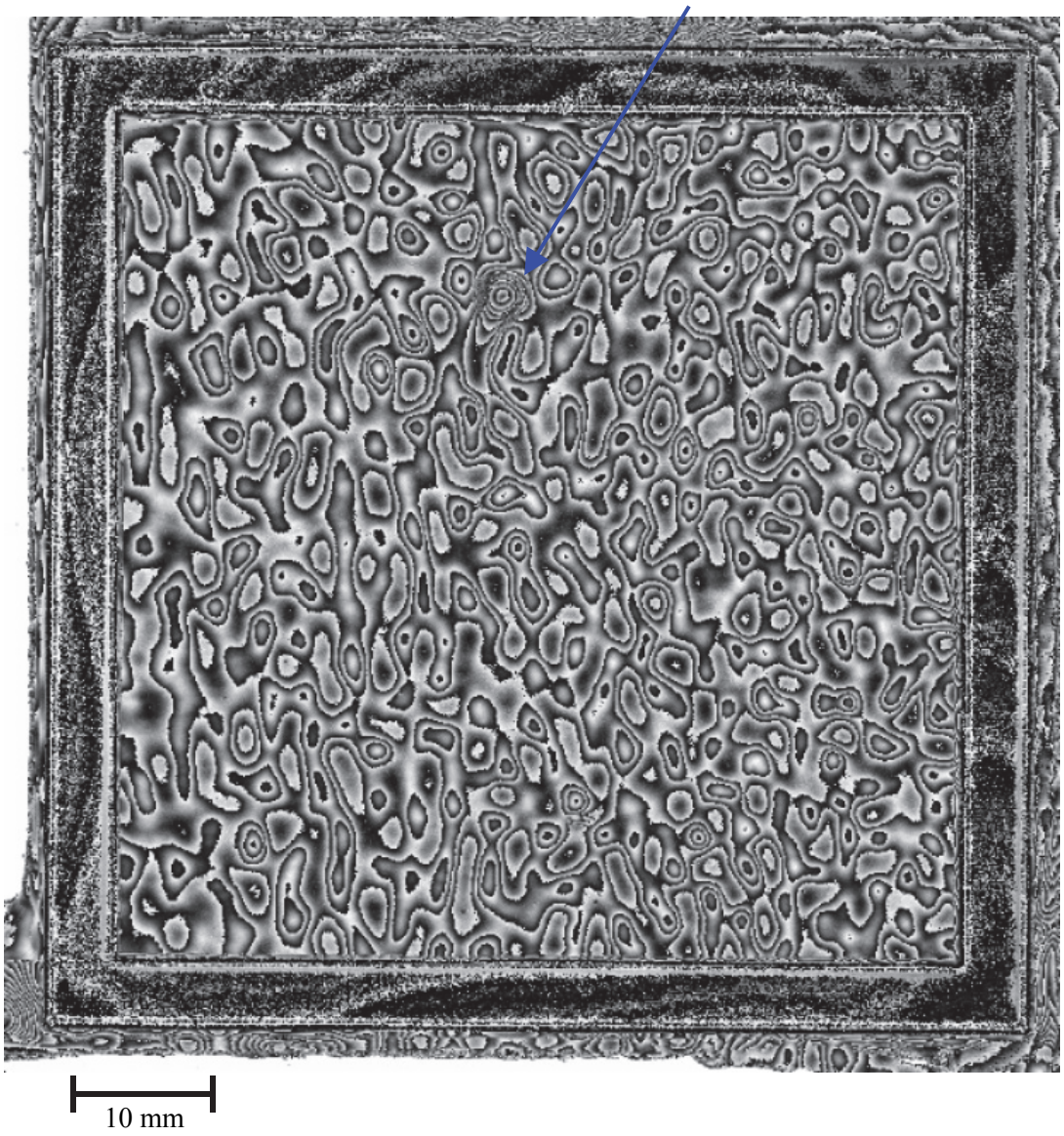


Figure 8 - Ultrasonic SAM images of a sample film produced by scan type 3 showing overwrapped thickness maps (pseudo thickness contours), the arrow points to closely spaced thickness bands indicating a steep-sided high point in the film thickness.

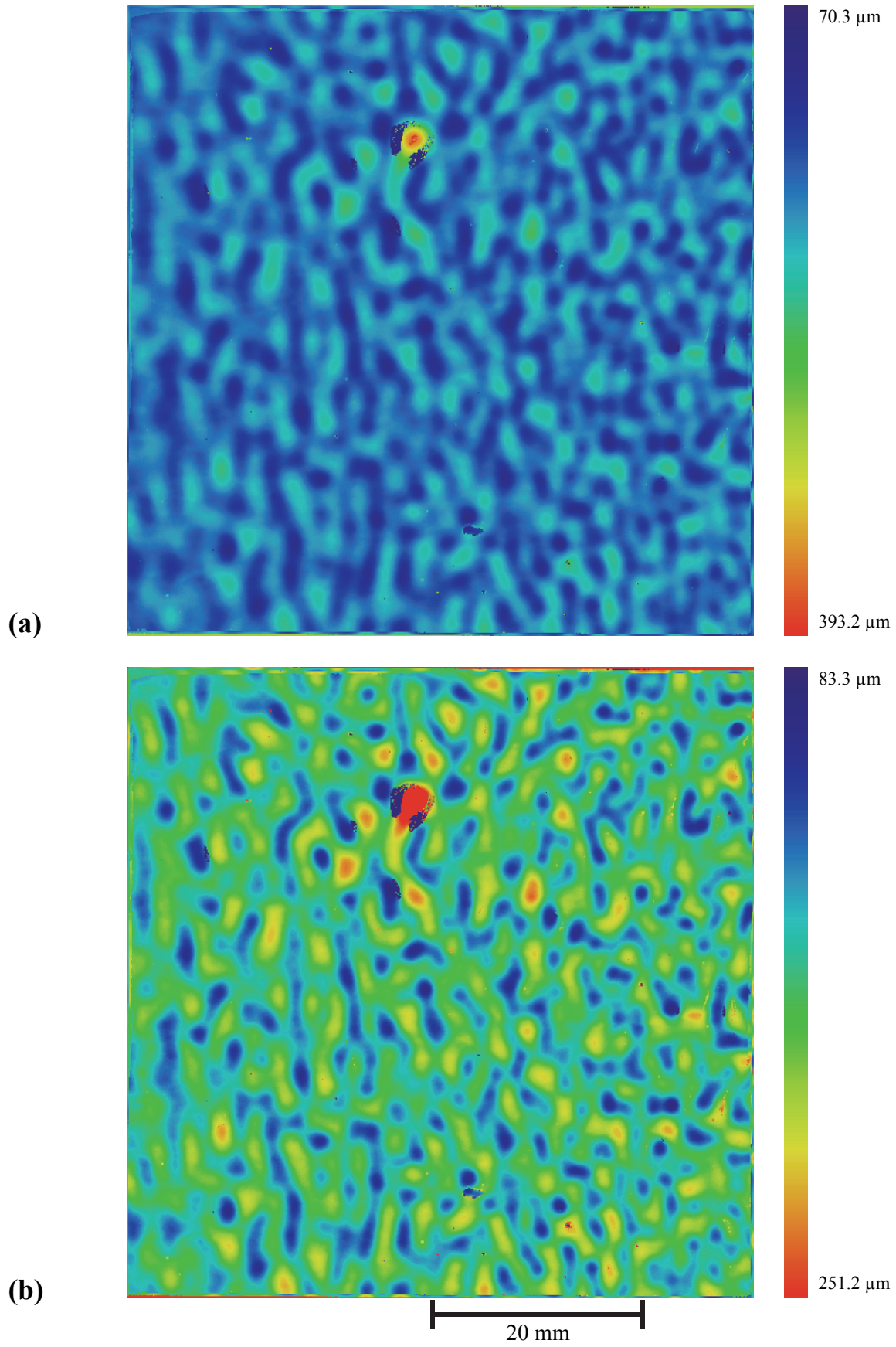


Figure 9 - Absolute thickness map of a sample film produced by scan type 2 at different thickness ranges of the same colour scale (E.M. spectrum).

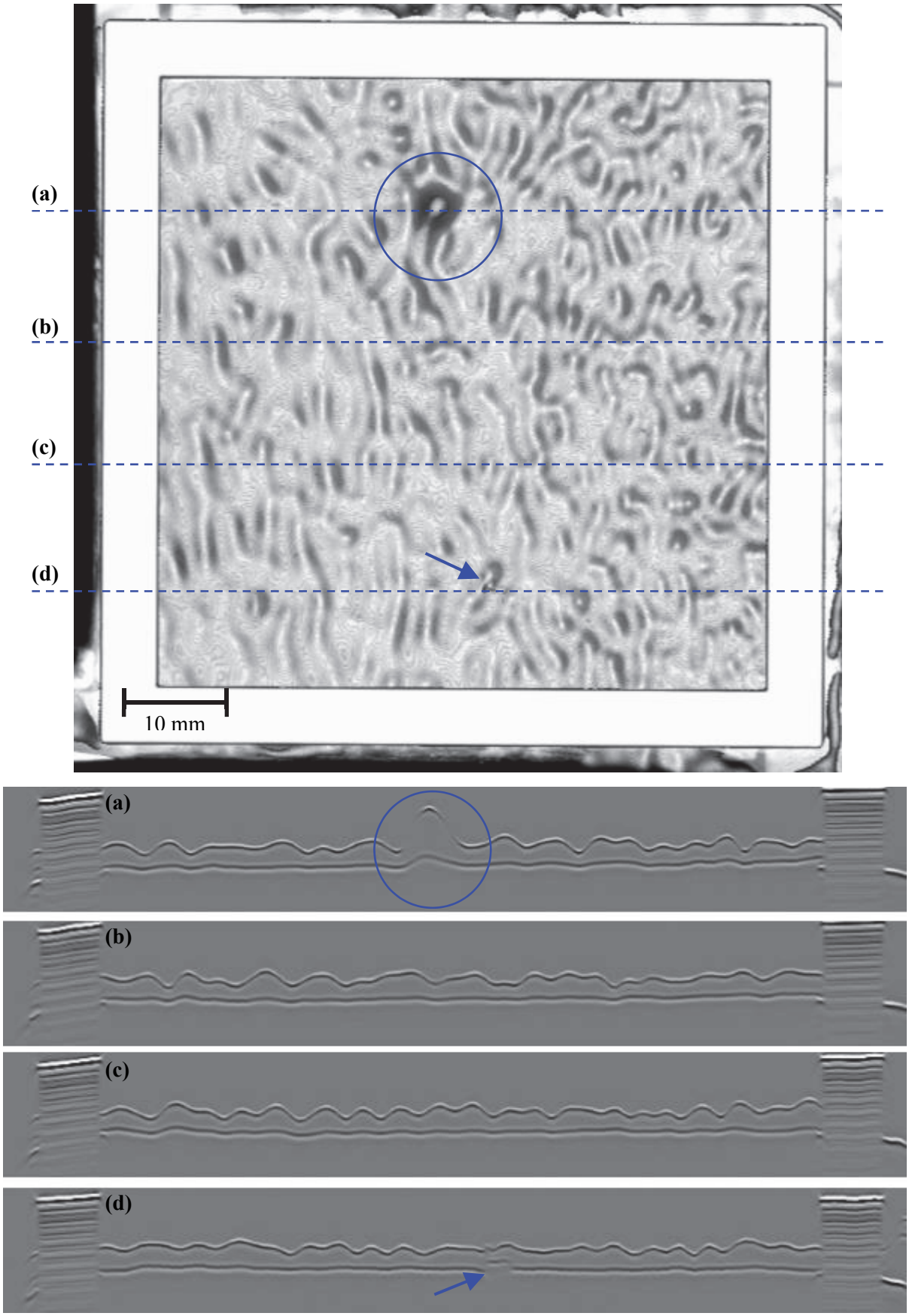


Figure 10 - B-scan cross-section thickness slices (lower) of a sample film produced by scan type 4 corresponding to the scan lines (a) - (d) in the intensity map of the top surface (upper). Circle marks peak thickness, arrow marks signal loss/wrong trigger.

3.1 INITIAL CONSISTENCY CHECKS

Checks were made to determine and verify the conversion from SAM 8bit ToF data (as recorded in thickness map datafiles) and the actual transit time in nanoseconds. In order to obtain this, the live oscilloscope timings were compared with the recorded data (adjusted for gate offsets) for certain fixed points (minimum thickness, maximum peak and second highest thickness) from all the sample films and the glass slide calibration artefact. The plot in Figure 11 shows this live versus converted recorded data. The recorded data very closely fit the straight line indicating 1:1 correspondence, indicating the reverse conversion process applied to the data to determine the absolute ToF is suitable. The largest deviations from parity occur at the longest measured transit times.

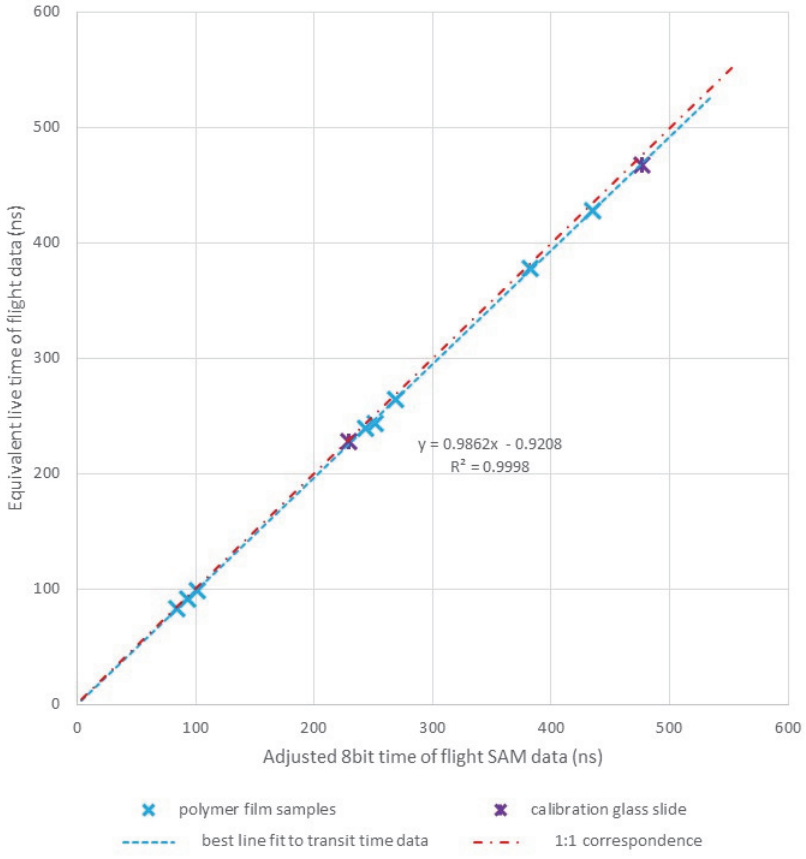


Figure 11 - Offset adjusted SAM ToF for selected results from all the sample films and single well glass slide calibration artefact.

3.2 TRANSIT TIME CORRECTIONS

Time of flight measurements are ideally captured from corresponding points of the front surface and back surface signals to determine an accurate transit time difference e.g. main zero crossover or first threshold crossing for each signal in turn, simultaneously accounting for the phase inversion which is observed in the back face waveform. The automated SAM system thickness determination employs the threshold crossing of the first signal as the timing point for the front surface, but uses the maximum peak within the second signal as the timing point for the back surface. This requires an appropriate correction to the transit time.

Figure 12 shows the waveform and timing points for an A-scan at one of the thinnest points in a sample film which corresponds to the minimum correction which could be applied across the whole area. As the frequency dependent attenuation increases through thicker film regions so the correction would also need to increase. In this case, the minimum correction determined for the waveforms of each calibration artefact / film sample are given in Table 2 and these are used to correct all measured ToF datapoints for each respective sample.

Future measurements would benefit from using an earlier timing feature close to the initial threshold trigger to reduce the correction applied and minimise the effects of dispersion on the waveform and transit times with increasing thickness.

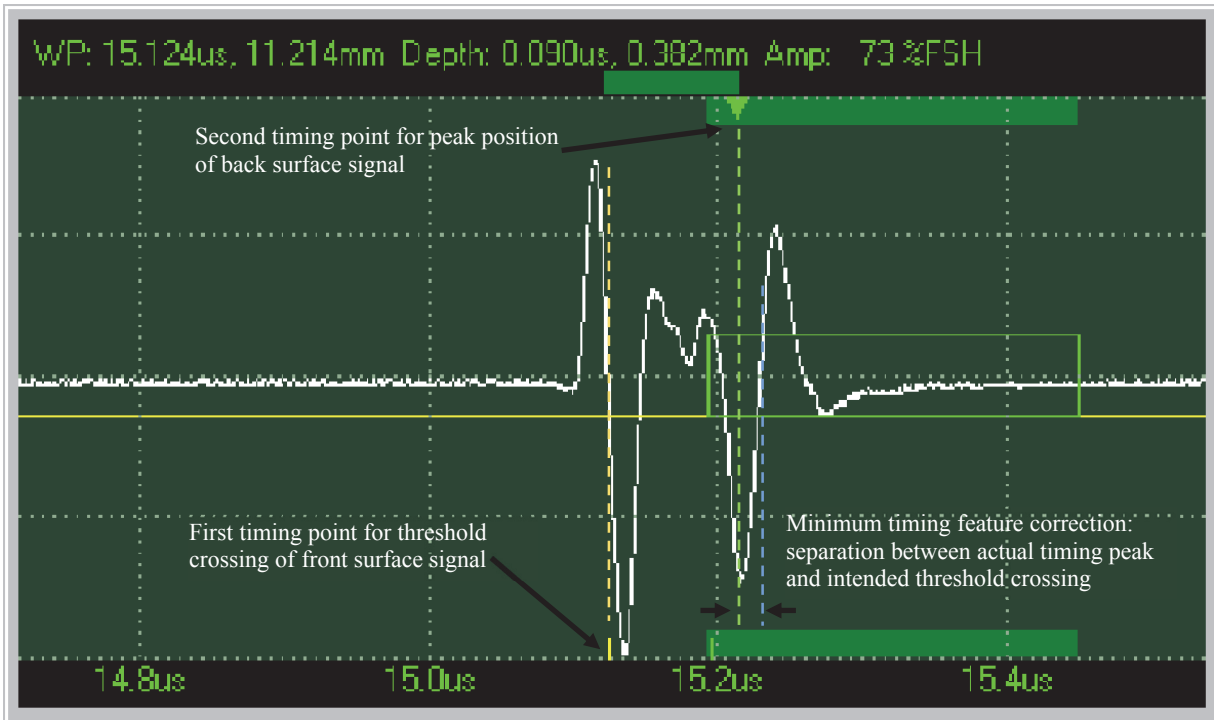


Figure 12 - Amplitude, triggering and timing features within an A-scan indicating the timing correction determined; due to phase inversion at the back surface the corresponding desired timing feature is now the positive threshold crossing. (Front surface tracking gate in yellow and data capture gate in green).

Table 2 - Corrections and offsets applied to raw SAM ToF 8bit file data

Sample	Minimum correction due to timing feature mismatch (ns)	Fixed gate start offset from front surface (ns)
PU reference film	– 3.9	42
Sample film#1	+ 15.2	65
Sample film#2	+ 16.7	65
Sample film#3	+ 14.8	70
Calibration glass slide	– 10.7	200

3.3 FILM VELOCITY CHARACTERISATION

A uniform thickness polyurethane (PU) film was used as a reference material to determine a characteristic velocity, subsequently assigned to the multilayer sample films. The reference film thickness was measured using a number of different methods to allow for independent verification and cross-checking. The results of these measurements are recorded in Table 3. The measurements performed were:

- Mechanical thickness measurement for reference using a flat anvil micrometer ~5 mm diameter,
- Optical thickness measurements by top surface 3-D optical profiling of an ~10 mm by 20 mm area of film laid smoothly (assuming perfect contact) onto a flat glass slide,
- SAM ToF measurements from front to back surfaces over ~60 mm square area of freely supported film over air,
- Optical thickness measurements through the film ~5 mm square area, each point returning either front or back surface depth, thickness then determined by correcting using literature values for refractive index [3].

Table 3 - PU reference film thickness characterisation

Measurement method	Thickness of measured section ± standard uncertainty (mm, µm or ns)	Comments
Mechanical **Mitutoyo Digital Micrometer**	0.041 mm ± 0.001 mm	Some mechanical compression expected
Acoustic **Sonix UHR 2001**	25.190 ns ± 0.366 ns	Provides a V_{PU} of 1722.0 ± 114.0 m/s calculated in conjunction with "optical surface" thickness measurement
Optical surface **Alicona IFM G5**	43.377 µm ± 2.802 µm	3.5 µm spatial resolution, 0.0787 µm depth resolution
Optical depth **Alicona IFM G5**	40.556 µm ± 0.711 µm	0.7 µm spatial resolution, 0.1380 µm depth resolution 26.165 ± 0.459 µm apparent thickness corrected by refractive index of 1.55*

'*' literature values for PU refractive index in the range 1.5-1.6 [3]

Except for the mechanical micrometer measurement, for which the standard uncertainty from the calibration certificate is quoted, all other methods generated population data over the measurement area from which the quoted standard deviations in Table 3 are calculated (from Gaussian distribution fitting of the data).

The thickness data determined by the independent methods are all comparable within the errors of the respective measurement methods. From these, the reference material velocity determined is 1722.0 ± 114.0 m/s.

The detailed data frequency plots for each of these methods, normal distribution fitting results and example outputs are provided in Figures 13 – 17.

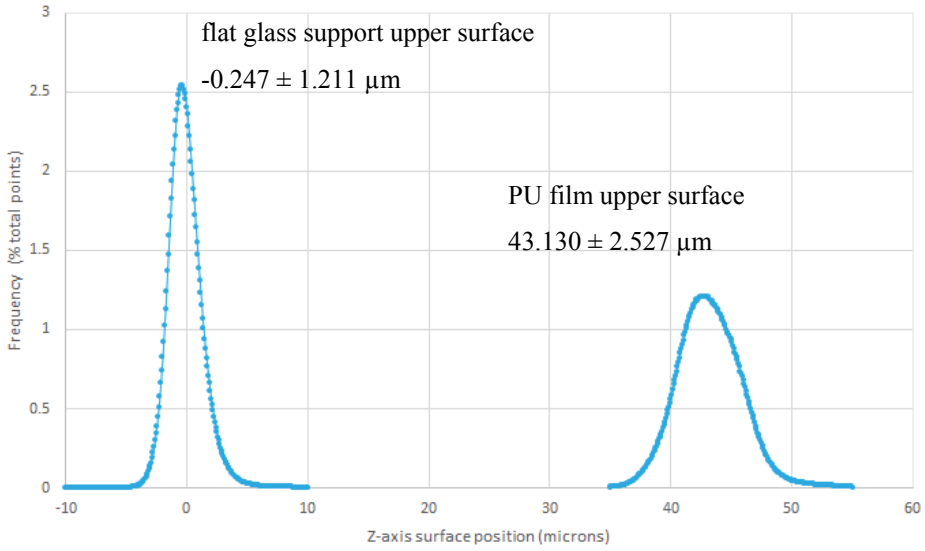


Figure 13 - Optical Alicona IFM G5 measurement populations of surface of PU reference film and peripheral glass slide support surface.

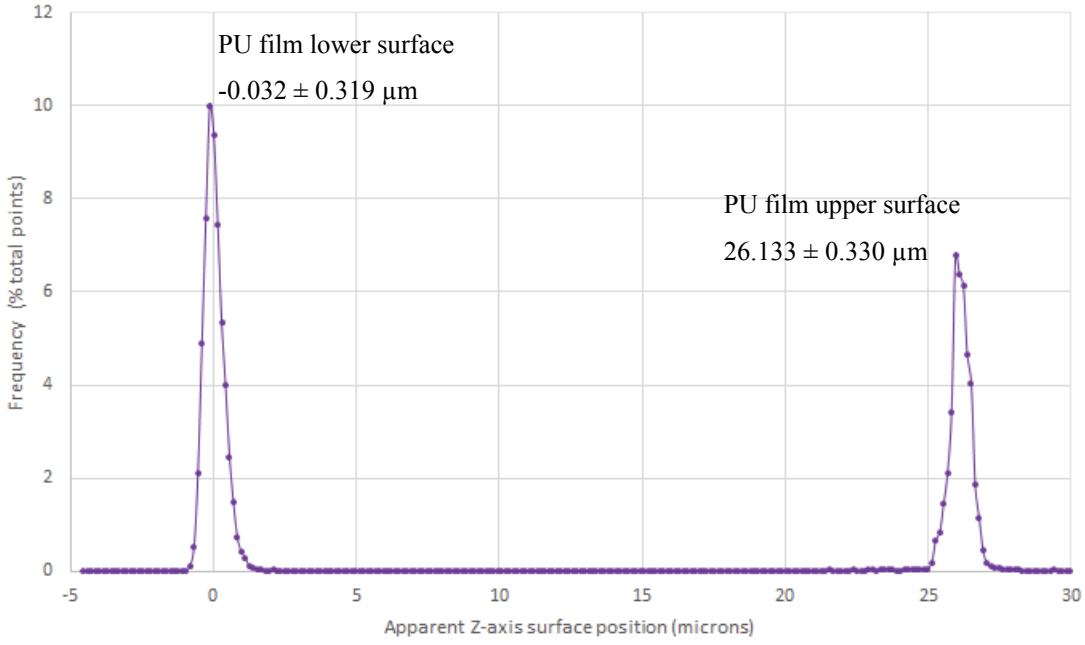


Figure 14 - Optical Alicona IFM G5 measurement populations of surface positions of reference PU film and 'through film' measurement of underlying support surface for refractive index/film thickness verification.

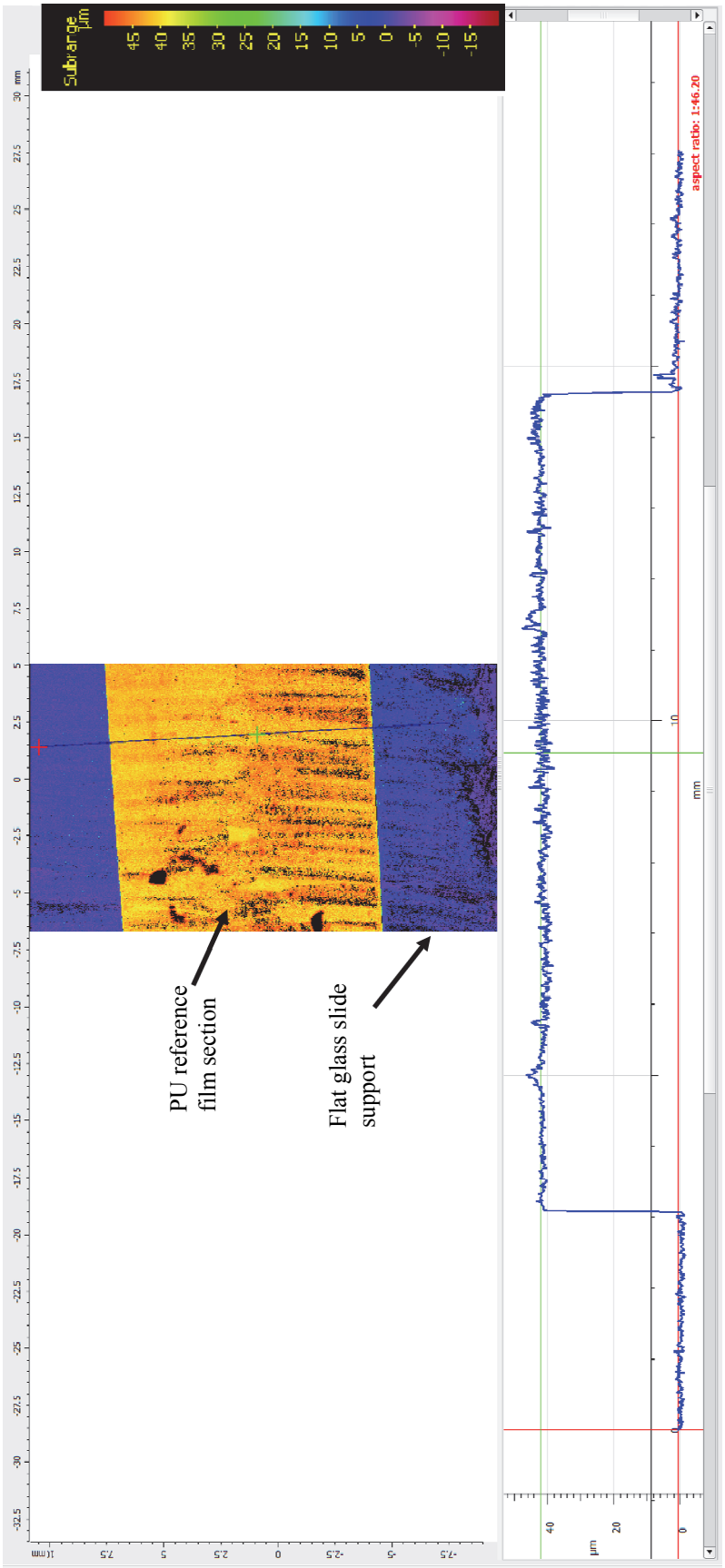


Figure 15 - Optical Alicona IFM G5 typical surface profile results for PU reference film supported on a flat glass slide (along the cross-section line shown in the image).

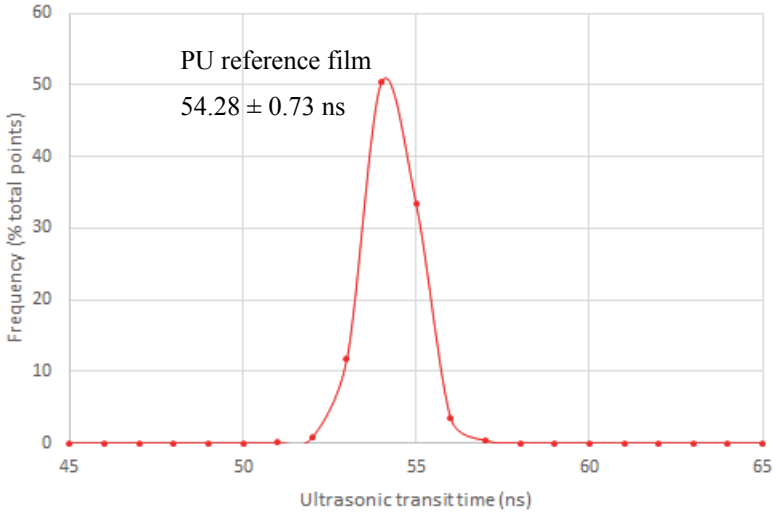


Figure 16 - Sonix UHR 2001 SAM measurement population of ToF through PU reference film.

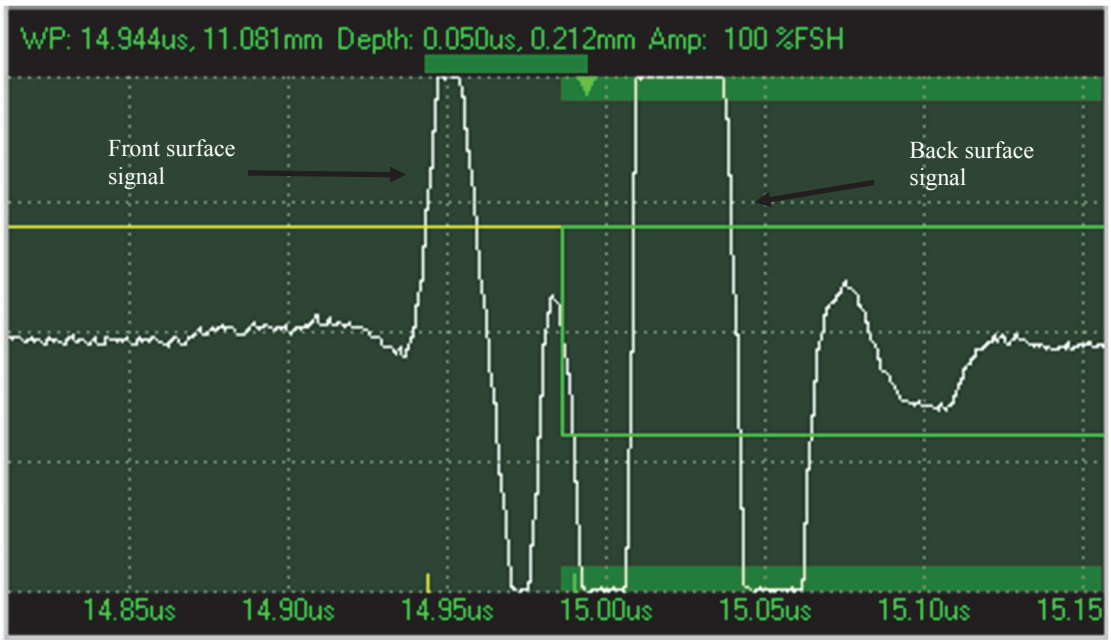


Figure 17 - Sonix UHR 2001 SAM typical A-scan showing front and back face returns from PU reference film (supported in clamping frame with air backing). Note: there is some unwanted overlap between front and back surface echoes.

Comparison between the acoustic velocity found from the literature [2, 4, 5] for the PU reference film and the value determined in this work is provided in Figure 18. This shows a wide range of quoted values are possible, highly dependent on the exact chemical formulation, but that the velocity determined in this work is reassuringly within the bounds of the upper and lower limits thus found.

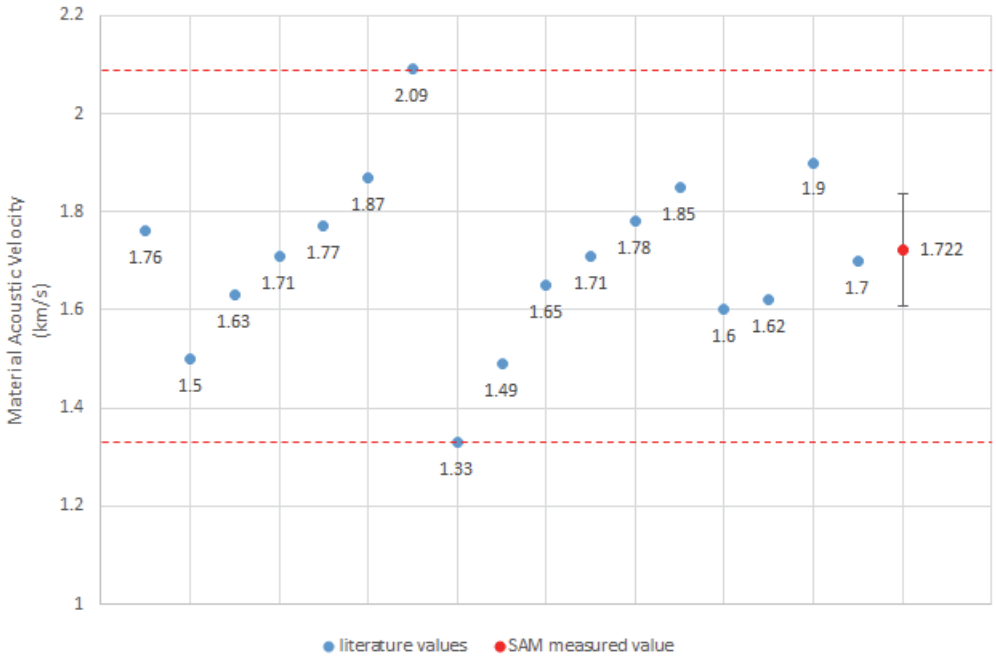


Figure 18 - Comparison between book values for acoustic velocity of polyurethane and value determined experimentally by optical thickness and SAM ToF measurements.

3.4 SAM TIME OF FLIGHT THICKNESS METHOD VALIDATION

The suitability of employing SAM ToF and optical thickness measurements to determine both the material velocity and the local variable film thickness of the samples was verified using the calibration artefact. Again, measurements were made using a number of independent techniques described below, the results of which are provided in Table 4:

- Mechanical thickness measurement for reference, using a 5 mm diameter ball-tipped micrometer,
- Optical thickness measurements by top surface 3-D optical profiling,
- SAM ToF measurements from front to back surfaces over ~20 mm square area centred on the single well.

Table 4 - Thickness estimates of single well glass slide calibration artefact

Method	Average thickness of flat region (mm)	Minimum thickness of dished region (mm)	Difference/well depth (mm)
Mechanical Mitutoyo Digital Micrometer	1.362 ± 0.001	0.631	0.731
SAM Sonix UHR 2001	1.376* ± 0.005	0.644*	0.732*
Optical Alicona IFM G5	1.375± 0.001	0.645	0.730

'*' calculated using V_{glass} of 5900 m/s for soda-lime silica glass (source [1, 2]), corresponding actual V_{glass} from mechanical thicknesses and SAM ToF data is 5924.6 m/s which is < 0.5% from accepted book value

The results show exceptionally good correlation between the different methods for the average flat surface and minimum thickness section measurements and, in particular, for the well depth. This provides some confidence in the methodology for an ideal scenario comprising a uniform thickness and homogeneous elastic material with consistent acoustic velocity.

The optical and SAM measurement data populations from which Gaussian distribution fitting parameters were used to provide glass slide calibration results are presented in Figures 19 – 27, including example raw data outputs.

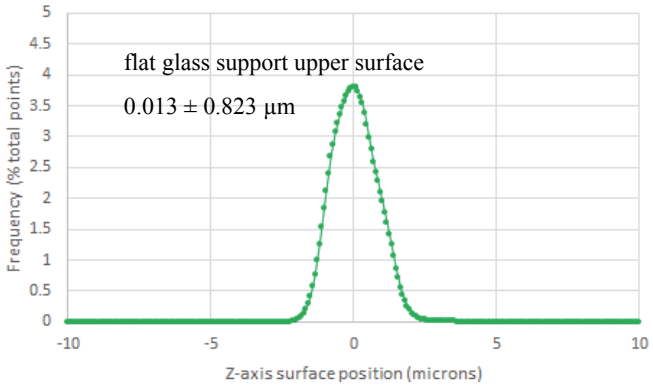


Figure 19 - Optical Alicona IFM G5 measurement population of upper surface of constant thickness glass slide region surrounding the single well.

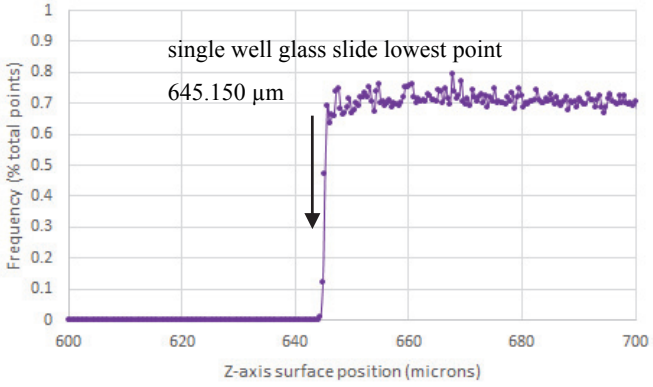


Figure 20 - Optical Alicona IFM G5 measurement population of upper surface of peripheral glass slide support surface.

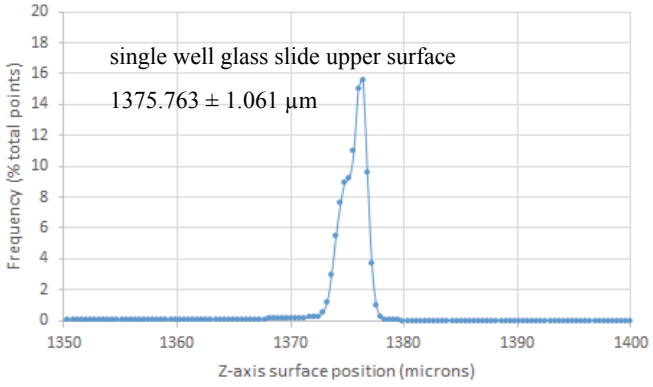


Figure 21 - Optical Alicona IFM G5 measurement population of upper surface of thinnest region of single well in the glass slide calibration artefact.

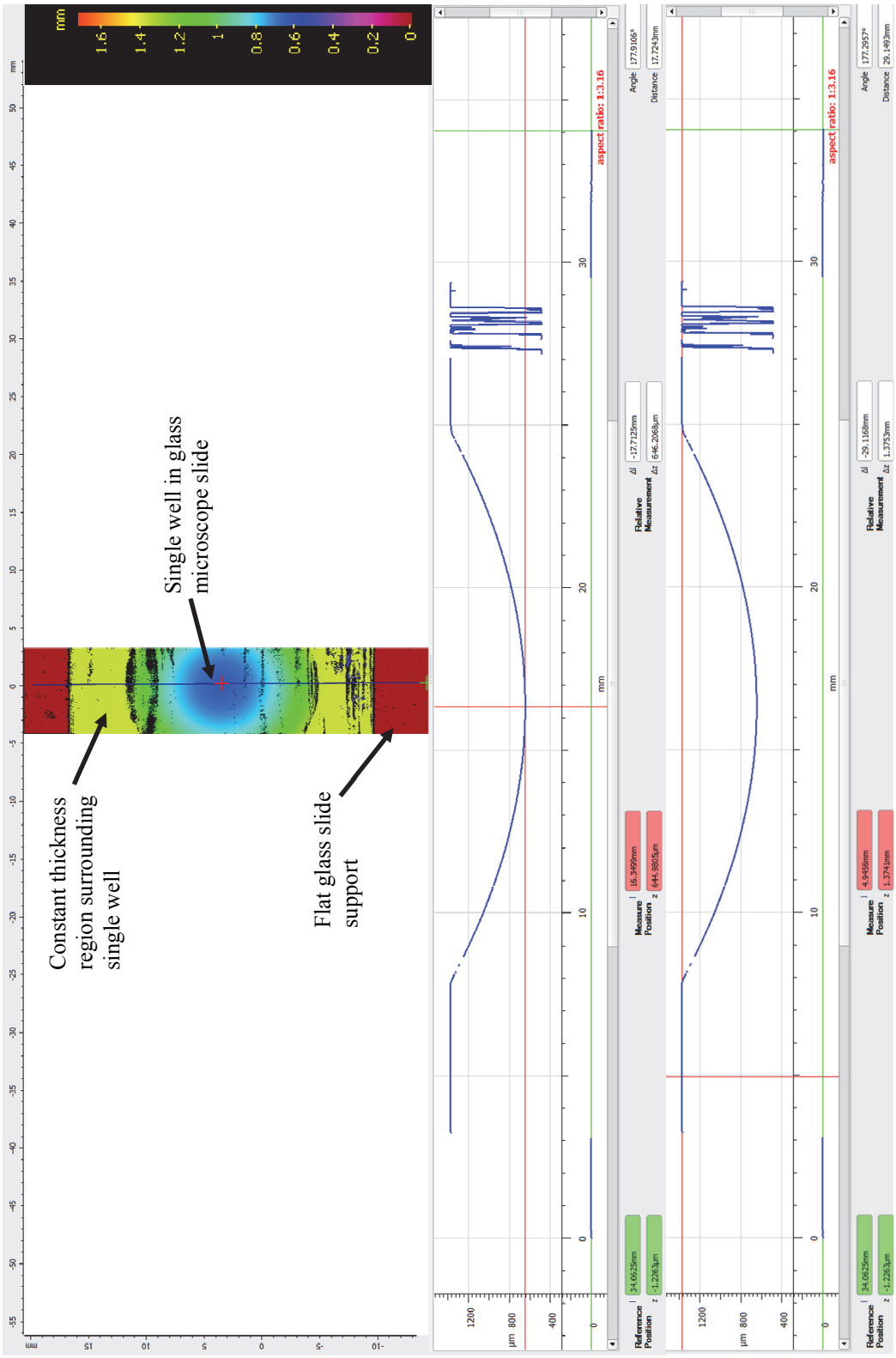


Figure 22 - Optical Alicona IFM G5 typical surface profile results for single well glass slide supported on a flat glass slide (along the thinnest cross-section line shown).

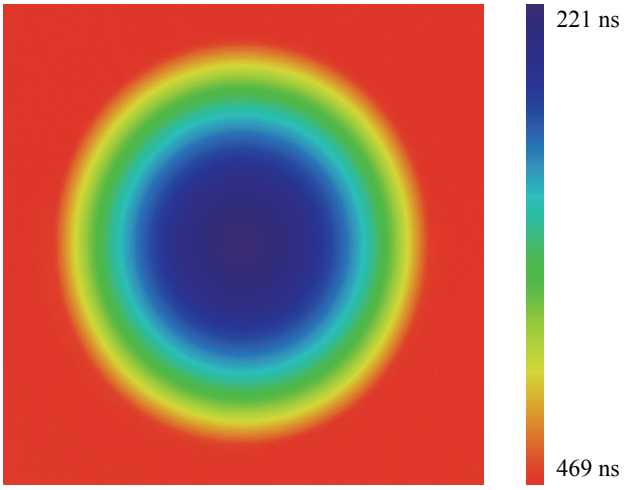


Figure 23 - Unwrapped single well glass slide SAM thickness map measured from front to back face. Scale showing nanosecond transit times across sum of forward and return journeys (twice the path length).

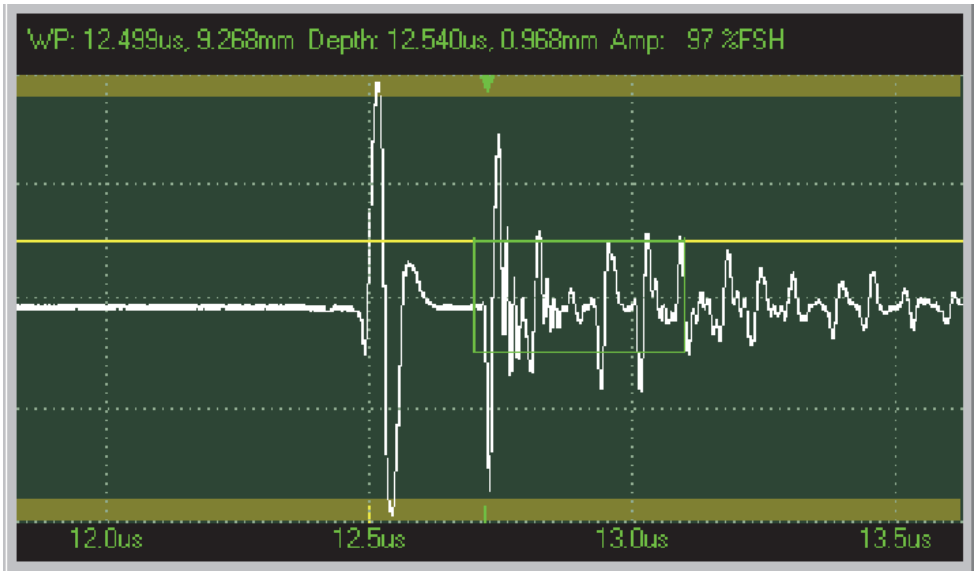


Figure 24 - Sonix UHR 2001 SAM typical A-scan showing front and back surface returns from thinnest point in single well glass slide. (Timing feature correction to the transit time data determined from this point).

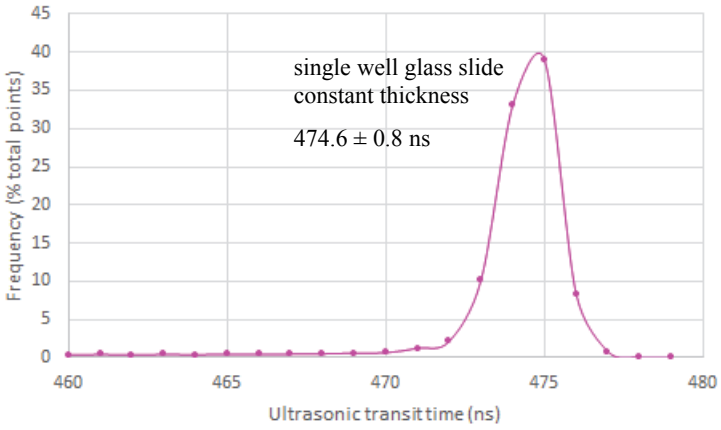


Figure 25 - Sonix UHR 2001 SAM measurement population of ToF through constant thickness glass slide region surrounding the single well.

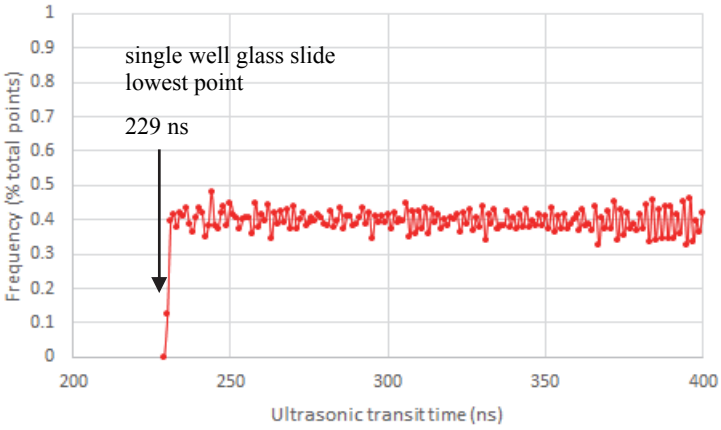


Figure 26 - Sonix UHR 2001 SAM measurement population of ToF through thinnest region of single well in the glass slide calibration artefact.

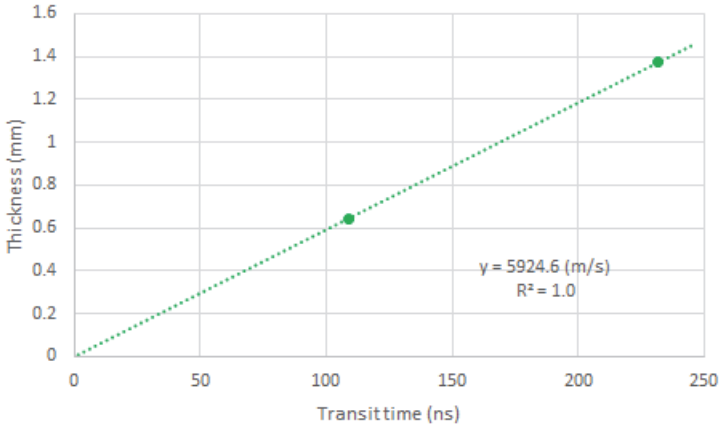


Figure 27 - SAM and optical measurement results for average maximum and single point minimum thickness against corrected ToF for single well glass slide artefact. Note: this provides a best fit line for soda-lime silica glass velocity <0.5% from book value.

3.5 ADDITIONAL CHECKS

The optical and SAM measurement data populations from which Gaussian distribution fitting parameters were used to provide sample film representative thicknesses for a small section of the sample films and associated uncertainties are provided in Figures 28 and 29.

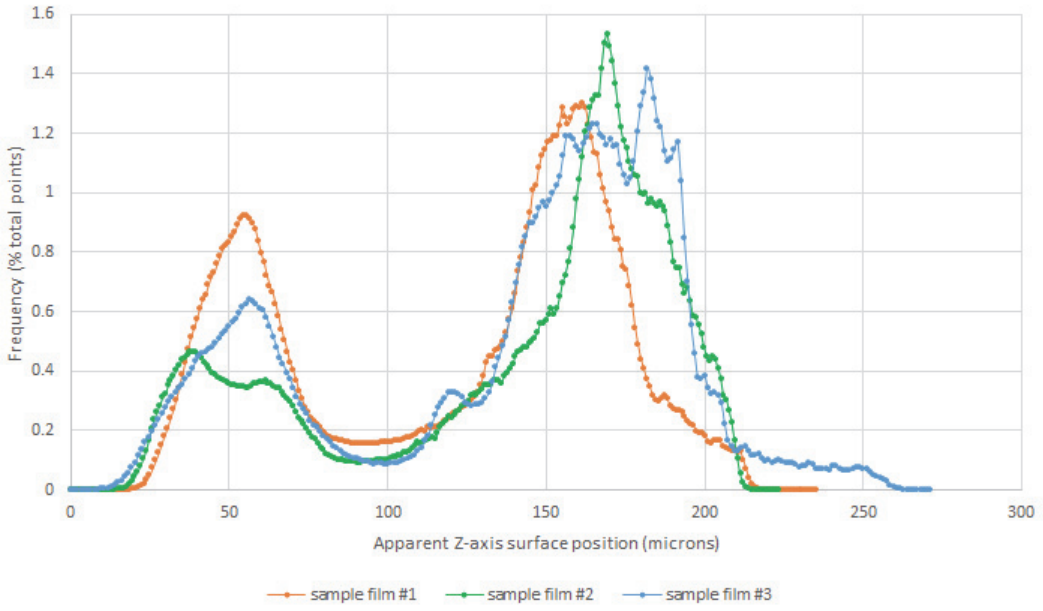


Figure 28 - Optical Alicona IFM G5 measurement populations of upper and lower surface positions as viewed "through film" for the ~15 mm square top left corner of each sample film. Note: the true thickness determined from this requires refractive index correction.

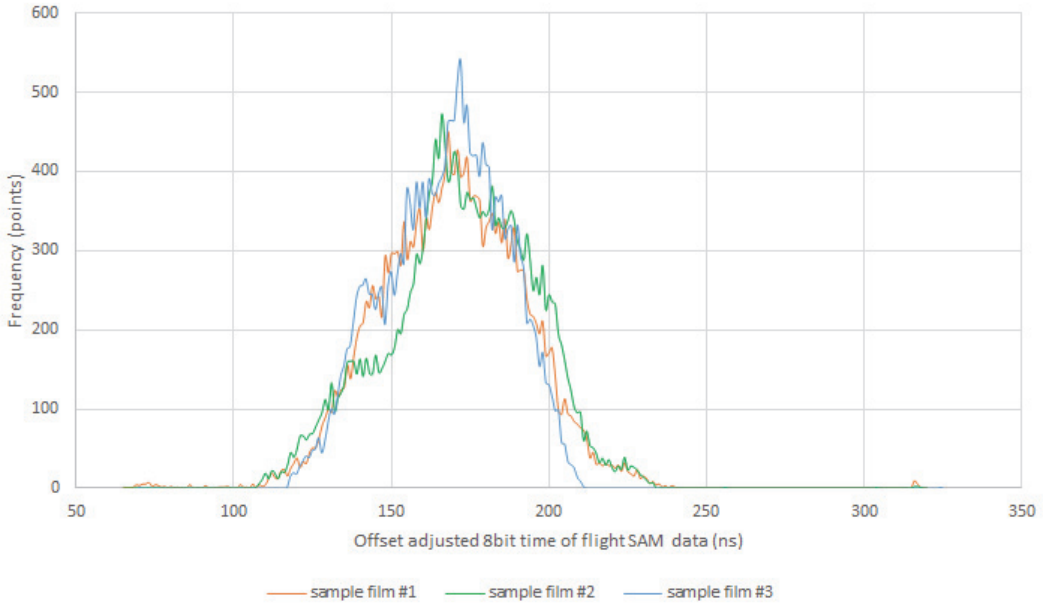


Figure 29 - Offset adjusted SAM ToF population results (not corrected for timing feature mismatch) for the 15 mm square top left corner of each sample film. Note: transit times correspond to twice the distance between front and back surfaces.

The comparison of optical and SAM population data and single reference mechanical (micrometer) measurements from the top left corner region of each sample film are given in Table 5. The disparity in results may be due to some or a combination of all of the following:

- Mechanical measurement is dominated by high points in the region but with mechanical compression likely due to compressibility of the material and localized contact (material may compress and spread under local high contact loads),
- Acoustic velocity of multilayer sample film is potentially significantly different to that measured from PU reference layer alone,
- Averaging of apparent thickness over the ultrasound beamwidth using SAM,
- Optical measurements are not made for both upper and lower surfaces for each measurement point rather only one or other is automatically detected and inferences made from these independent populations.

Table 5 - Comparison of thickness measurements for multilayer sample films

Sample area measurement	Sample #1	Sample #2	Sample #3
Mean optical lower surface position (μm)	53.5 ± 13.5	50.1 ± 23.7	53.4 ± 17.8
Mean optical upper surface position (μm)	157.5 ± 15.5	171.5 ± 17.4	168.4 ± 23.4
Calculated[1] mean optical thickness (μm)	161.1 ± 31.8	188.3 ± 45.6	178.2 ± 45.5
Mean SAM transit time (ns)#	169.4 ± 23.2	173.8 ± 23.2	168.8 ± 20.8
Calculated[2] mean SAM film thickness (μm)	158.9 ± 33.1	164.0 ± 34.3	158.1 ± 30.6
Mechanical (mm)	0.172	0.187	0.172

3.6 UNCERTAINTY OF MEASUREMENT

The uncertainties for the measurement equipment are listed in Table 6. The normal distribution standard deviation of each measurement population is given in Tables 3 and 4. An attempt to identify the error sources and an estimate of their magnitude and effect on the thickness or velocity measurements is given in Table 7.

Table 6 - Summary of equipment and calibration details

Equipment	UKAS-accredited calibration certificate N°.	Calibration date	Standard uncertainty
0-30 mm Mitutoyo digital micrometer ser.no.11010976	CN265457	02/18	± 0.001 mm
Sonix UHR 2001 scanning acoustic microscope	Verified using single well glass slide	n/a	n/a
Alicona IFM G5 3-D microscope ser.no.075621511514	CAL.17.8588	08/17	± 200 nm*

'*' claimed for 10x objective lens, not UKAS accredited

Table 7 - Error identification, estimation and the effect on measurement results

Error source	Estimated contribution	Effect of error on measurement
Alicona	(based on thickness map bin size) ~ 0.4 µm PU reference film ~ 1 µm multilayer sample film ~ 0.4 µm glass slide	± 0.56 µm for PU reference film thickness ± 2.2 µm for multilayer sample film thicknesses ± 0.4 µm for glass slide thickness
SAM	~ 2 ns	± 1.7 µm for PU reference and sample film thickness ± 5.9 µm for glass slide thickness ± 65 - 75 m/s on V_{PU} determination
Velocity	if combined polymer velocity 20% greater than PU reference (i.e. 2066.4 m/s)	± 2.1 µm for sample film thickness (based on SAM contribution)
	if combined polymer velocity 20% less than PU reference (i.e. 1377.6 m/s)	± 1.4 µm for sample film thickness (based on SAM contribution)
Timing feature offset	min 3 ns max 20 ns	~ 2.6 - 17.2 µm

Other potential sources of error, some of which are explored in Table 7:

- Poisson contraction in thickness due to tensioning of sample,
- Variability in frequency content (dispersion) for the viscoelastic polymers especially noticeable through thicker regions shifting timing features on waveform (stretching return signal), corrections were made based on minimum thickness level only for each sample film,
- Auto-selection of waveform timing features by system software,
- Further waveform stretching due to varying thickness over a minimum sampled region for each spot measurement using SAM (finite nature of interrogating ultrasound beam covers multiple point thicknesses),
- Additional error if velocity is significantly different for the chemically modified multilayers in combination compared to the PU reference film, and further errors with the variation in multilayer film thickness since the combined velocity could then be different at each point depending on the actual thickness of additional polymers.

4 CONCLUSIONS

SAM has been clearly proven a viable route to providing detailed, large area characterisation of thin polymer films which would be difficult to achieve by any other single technique. Even with several sources of error this method provides a good estimate of film thickness and its variability with excellent spatial resolution.

The methodology works well for a model elastic material system where the thickness is uniform, the material is perfectly homogeneous and the acoustic velocity is constant. In the case of the polymer films used here these basic characteristics are unlikely to be met satisfactorily; the material properties vary depending on the cure level and composition from point to point, the velocity may vary should the components of the multilayer differ significantly in acoustic velocity and the thickness over each sampled region is highly variable.

Some measurement issues still to be resolved:

- Improved upper window material (in clamping rig) to eliminate corrosion residue on film surface,
- Thicker PU reference material to improve accuracy of velocity determination (remove front and back face signal overlap),
- Constant thickness films of each individual material in the multilayer film to corroborate velocity and thickness validations,
- Independent waveform capture for each measurement point to enable precise determination of ToF without resorting to a timing feature mismatch correction.

5 REFERENCES

[1] *"A large ultrasonic bounded acoustic pulse transducer for acoustic transmission goniometry: modeling and calibration", Y. Bouzidi and D. R. Schmitt, J. Acoust. Soc. Am. 119 (1), January 2006, pp 54-64*

[2] *https://www.signal-processing.com/table.php*

[3] *https://warwick.ac.uk/fac/cross_fac/sciencecity/programmes/internal/themes/am2/booking/particle size/sample_dispersion__refractive_index_guide.pdf*

[4] *http://www.ondacorp.com/images/Rubbers.pdf*

[5] *http://www.classltd.com/sound_velocity_table.html*