

Monitoring Processing of Reactive Polymer Systems using Ultrasonics

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ABSTRACT

Ultrasonic equipment to monitor the processing of reactive polymer systems has been upgraded to operate in a fully automatic mode and to monitor changes in peak height as well as the time of flight of ultrasonic pulses. Changes in time of flight can be measured to within one nanosecond. The system can therefore measure ultrasonic velocity very accurately which is directly related to the modulus of the material. As the modulus of the material changes during cure, the ultrasonic method can be used to monitor cure. Transducers have been mounted in industrial processing equipment (e.g. injection and compression moulds) and the performance of the equipment assessed. In one case it was shown that the ultrasonic monitoring equipment was able to identify optimum processing conditions. This has commercial implications for reducing cycle times to improve efficiency and in enhanced quality assurance. In three other cases promising results were obtained which indicated the potential of the equipment to lead to operating cost savings to the companies concerned.

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1. INTRODUCTION

The purpose of this project was to access the ability of ultrasonics to monitor the processing of reactive polymer systems. This document summarises the ultrasonic measurement method and briefly reports five industrial trials using the equipment. This document is deliverable 42.4 of project "Measurement of Reactive Polymer Systems", (6mpm9092) a "studio" project supported 50% by the DTI and 50% by the participating companies.

2. THE ULTRASONIC MEASUREMENT METHOD

2.1 INTRODUCTION TO ULTRASONIC MEASUREMENTS

In order to prepare for monitoring reactive processing during commercial scale production some laboratory measurements were carried out. In a typical experiment a sample is clamped in between two transducers (see Figure 1). At the start of one experiment one of the transducers emanates an ultrasonic compression pulse with a frequency in the range of 0.5 - 6 MHz. The second transducer receives the pulse after a short delay (time of flight). The pulse is then amplified and processed.

The timing of the pulse with respect to the time of emission is critical and puts high demands on the acquisition hardware. NPL therefore developed a specialised unit (named "EDS") to carry out this task. In this unit the time of flight is determined to better than 1 ns precision. A schematic diagram of the EDS unit is given in Figure 2.

The time of flight (tof) and the attenuation of the signal can be related to the elongational modulus. The elongational modulus L^* relates to the bulk compression modulus (K) and the shear modulus (G) as follows:

Equation 1
$$K^* = L^* - \frac{3}{4} G^*$$

The moduli are complex quantities and relate to the elongational moduli L' and L'' as follows

Equation 2
$$L^* = L' + iL'' \quad \text{with} \quad L' = \rho V_{\text{long}}^2 \left[\frac{\omega^2(\omega^2 - \alpha_{\text{long}}^2 V_{\text{long}}^2)}{(\omega^2 + \alpha_{\text{long}}^2 V_{\text{long}}^2)^2} \right]$$

$$L'' = \rho V_{\text{long}}^2 \frac{2\alpha_{\text{long}} V_{\text{long}}}{\omega} \left[\frac{\omega^4}{(\omega^2 + \alpha_{\text{long}}^2 V_{\text{long}}^2)^2} \right]$$

and therefore

$$\tan \delta = \frac{L''}{L'} = \frac{2\alpha_{\text{long}} V_{\text{long}} \omega}{\omega^2 - \alpha_{\text{long}}^2 V_{\text{long}}^2}$$

with ρ the density, V_{long} the ultrasound velocity, ω the ultrasound angular frequency and α_{long} the absorption coefficient.

This equation can be greatly simplified (ref 1) as long as $\tan \delta$ is smaller than 0.2. The $\tan \delta$

can then be written as $\tan \delta = \frac{2\alpha_{\text{long}} V_{\text{long}}}{\omega}$ with an error of less than 1%. This is generally the case with materials that have much more of an elastic than a viscous character ($L'' \ll L'$) at that frequency.

The new simplified relationships read:

$$\text{Equation 3} \quad L^* = L' + iL'' \quad \text{with} \quad L' = \rho V_{\text{long}}^2$$

$$L'' = \rho V_{\text{long}}^2 \frac{2\alpha_{\text{long}} V_{\text{long}}}{\omega}$$

The moduli of a material increase with an increase in the degree of cure. The degree of curing can be followed by determining the velocity V_{long} of an ultrasound pulse through accurately measuring its *time of flight (TOF)*. The *pulse amplitude* of the ultrasound signal also changes and is related to the remaining power of the outgoing pulse depending on dissipative properties of the material. More precisely, the time of flight is inversely proportional to the elastic part of the (elongational) modulus and the pulse amplitude inversely proportional to damping, $\tan \delta$. The effect of curing is schematically shown in figure 1. After curing the increased moduli lead to a faster pulse propagation through the material and less damping. The time of flight of the ultrasound pulse and the power are also affected by the thickness, the density (ρ) and the temperature.

Holes are drilled in a mould to accommodate the ultrasonic transducers, as shown in Figure 3 for example, with metal left so that the transducers are not in direct contact with the sample. The mould is then fitted in a press of which an example is shown in Figure 4.

3. REPORTS ON TRIALS OF ULTRASONIC MONITORING

In this section brief accounts are presented of industrial trials of the ultrasonic equipment carried out in conjunction with five companies.

3.1 BIP

3.1.1 Purpose of the BIP trial

To assess the feasibility of following the cure of polyester dough moulding, urea and melamine compounds in a 30 ton hydraulic press.

3.1.2 Equipment

Hydraulic press (machine 2400) set to 160 °C. Top platen operating at 160 °C lower platen at 175°C.

2.0 mm cavity mould.

Nominal charge 60g.

3.1.3 Materials

Dough moulding compounds BIP100200\cure1, 5580 DIOP and BIP100200\overcure1.
 Urea formaldehyde - C Standard, BIP110200\cure17, BIP110200\cure19, BIP110200\cure20,
 BIP110200\cure24, BIP110200\cure25, BIP110200\cure32,
 Melamine formaldehyde BIP110200\cure30
 Injection grade BIP110200\cure28.

3.1.4 Trial Set Up

The transducers were installed in the mould with a commercial high temperature couplant, Sono 600, applied to the active side of the transducers. The metal in the mould between the transducer face and the interior of the mould was 2.0 mm. The mould had been set up several weeks before the trial and kept for a number of days at the operational temperature. When the trial started no significant signal could be detected through the mould with a dough moulding compound, Figure 5. The top and bottom moulds were disassembled to find that the clamping force on the transducer was poor and the couplant had charred and therefore was not effective. The transducers were replaced, a ring washer added to apply a force to the transducer and the couplant replaced with silicone grease. A pulse signal was now obtained (Figure 6) on a 2 mm plaque placed in the mould.

3.1.5 Trial on a range of samples

The mould was fitted in the press and between 60 and 70g of material was quickly placed in the mould at operational temperature, the ultralyser programme was started and the platens closed. Samples of dough moulding compound BIP100200\cure1 and BIP100200\overcure1 were tested several times (Figures 7 and 8) but no sample time of flight was found to be measurable. The pattern that is visible in these figures is thought to be due to ringing of the press. The absence of a detectable pulse signal through the sample could be due to the scattering of the ultrasonic waves by the glass fibre filler when the resin is fluid at elevated temperature. The attenuation α_{long} increases with increasing temperature resulting in a weaker pulse at elevated temperature. The plaque at room temperature transmitted ultrasound but the signal waveform was unusual.

However, the cure behaviour of moulding powder BIP110200\cure21 could be successfully monitored and it was noted that removing the sample after different times in the press, i.e. when the time of flight was falling, beginning to rise and increasing rapidly, had a profound effect on the surface finish. At short times, (i.e. undercure) the plaque was slightly off white with a smooth finish, for the situation when the time of flight was beginning to rise (i.e. optimum cure) the sample was white with a good finish while at long times (i.e. overcure) an orange peel surface texture was evident. Thus there was a good correlation between ultrasonic time of flight measurements and surface finish.

Cure monitoring of a range of urea formaldehyde and melamine powders was undertaken with a range of charge weights and cure times. Figure 9 shows a typical ultrasonic signal. The cure traces of all these powders are shown in Figures 10-18. In these figures the measured time of flight for the ultrasound pulse is plotted against the time in the mould. The software provides two estimates of the time of flight (estimate 1 and 2) which agree well in all these figures. The measured peak height is also shown. The time of flight increases sharply at early times as the temperature of the resin increases (this is expected as the temperature rise will reduce the stiffness of the material and thus reduce the velocity of ultrasound.) A maximum is reached in the time of flight curves when the effect of cure starts to dominate (e.g. at 40 seconds in figure 10). The time of flight then reduces to a minimum or plateaux. In some materials regression occurs, i.e. the stiffness of the materials starts to reduce with time; the material is being over cured. Evidence for this can be seen in figures 11, 12, 15, and 18. The peak height behaviour is the mirror image of the time of flight behaviour; with reference to figure 11, the peak height falls to a minimum at about 40 seconds, just when the time of the flight reaches a maximum. The peak height then rises to a maximum at about 100 seconds, just when the time of flight reaches a minimum. This point is the optimum processing time. The peak height then reduces right out to 300 seconds. The ultrasonic measurements' optimum processing times appeared to agree well with optimum cure times that had been established previously by BIP.

3.1.6 Conclusions from this trial

A rise in the time of flight at long cure times correlated with a poor surface finish and indicated deterioration in material properties, suggesting a preferred time of cure, determined by ultrasound. The ability of the ultrasonic monitoring technology to determine optimum cure times has commercial implications for maximising productivity and quality assurance.

2. Urea formaldehyde and melamine formaldehyde moulding powders transmitted ultrasound satisfactorily and the cure reaction was successfully monitored by the variation in the time of flight
3. Dough moulding compounds (polyester resin based) gave distorted waveforms not amenable to measuring the time of flight under the conditions investigated.
4. It is important to apply a positive pressure to the transducers and to use a thermally stable high temperature couplant (silicone grease rather than Sono 600).
5. A variation in the peak height was also recorded which could be used as an alternative cure state indicator.

3.2 RAYCHEM

3.2.1 Purpose of the trial

To assess the feasibility of following the cure of cross linkable polyolefin compounds during injection moulding.

3.2.2 Equipment

A 25 ton Arburg 221-55-250 injection moulding machine (140 bar) fitted with a 2 mm cavity mould (70 mm square) side inlet was used in this trial.

3.2.3 Moulding conditions

Barrel temperature 70-80 °C.

Mould temperature for production of plaques 190°C.

Transducers fixed 3 mm from the moulding surface.

3.2.4 Materials

Three batches of polyolefin of varying degrees of stiffness.

a) rc1.

b) rc2.

c) rc3.

3.2.5 Trial

The lower transducer was inserted after silicone grease couplant had been applied to allow the ultrasound to be transmitted through the mould wall into the sample. The upper transducer was also inserted and spacers added to impart a positive pressure onto the mould.

The machine was run up to temperature and plaques produced from the rc1 compound. However, surprisingly, the sample signal, though observable, was too noisy to measure consistently by the software (Figure 19). Turning the heat off and cooling the mould with a plaque in place improved the signal but the noise level was still too high to give a repeatable time of flight measurement.

Removing the upper transducer and placing it directly on the top of the plaque gave a good measurable signal at room temperature and it was further observed that the ultrasonic beam was narrow as moving the transducer only a small distance (1-2 mm) out of alignment with the lower transducer reduced the signal strength received considerably.

To observe the effect of the thickness of mould metal under the transducers, the upper transducer was directly placed on a 3 mm sheet of steel resting on two plaques, (well greased with silicone couplant to ensure good acoustic contact). The signal response was similar to the transducer sited in the mould (Figure 20). The 3 mm metal sheet was replaced with a 1 mm thick sheet in the same set up as above and gave a significantly improved result (Figure 21). It was also noticed that moving the transducer out of alignment (3-4 mm) did not reduce the signal. The moulding metal interface apparently caused the ultrasonic beam to diverge strongly. Making the metal thicker then appeared to reduce the signal received by the transducer. Heating the lower mould to 190 °C reduced the sample signal considerably (Figure 22) giving a signal similar to the situation during injection moulding.

3.2.6 Conclusions from this trial

The steel-moulding interface appeared to diverge the ultrasonic beam. Small increases of the distance of the transducer to the interface then appeared to weaken the received sample signal to such an extent that the time of flight could not be measured. An impedance mismatch for the polyolefin in the melt state with the metal was believed to be responsible for this. This is in contrast to urea and melamine compounds investigated with BIP that have much higher moduli and gave much stronger signals.

Transmitting ultrasound through 1 mm of metal gave a satisfactory signal for time of flight measurement (at room temperature only).

In order to improve the signal to noise ratio tests the following could be considered:

- The transducer hole in both top and bottom moulds could be bored out to a 1 mm thickness of metal,
- Higher power transducers could be used,
- Transducers with a less divergent beam could be investigated.

3.3 ZOTEFOAM

3.3.1 Purpose of trial

The work reported in this section demonstrates that valuable information on Zotefoam's packaging products can be obtained from ultrasonic monitoring. A good quality signal was obtained and the technique successfully followed the cooling of an extruded sheet after production. The technique is believed to have the potential to help Zotefoam achieve improvements in productivity.

Zotefoam exploit a unique process to produce an impurity free, closed cell, expanded polymeric (mainly polyolefin) foam used where no contamination can be tolerated from foam packaging or its presence in other uses. (Figure 23). The process involves an extrusion stage where the polymer is mixed with pigment and a peroxide and then passed through heated ovens to form a cross linked (cured) continuous sheet. This precursor material is then impregnated with nitrogen in a two stage process to produce a low density, much expanded foam.

To follow the curing process ultrasonics could be a good candidate and assist in increasing the throughput in the ovens (i.e. raising belt speed) amongst other potential benefits. Therefore some trials were performed to assess the attenuation of an ultrasonic signal and its temperature sensitivity produced from the NPL ultrasonics equipment on typical materials at this point in the process. Conventional means of measuring cure of thick sections had proved difficult and involved costly time consuming testing of only a small part of the continuous process by high temperature modulus testing in a tensile machine. Ultrasonic online monitoring opens up the possibility of continuous quality control (like a sentinel) as well as optimising or indeed controlling the line speed.

3.3.2 Trial

A first attempt to send an ultrasonic signal through a solid slab of LD 24 resulted in a measurable signal. However, as expected, a *foamed* material LDPE 45, did not give any response due to the air pockets in the sample. This same material with nitrogen dissolved (700Kg/m^2), *but not foamed*, gave a strong signal. A similar signal was also obtained from ZF45 removed straight from the cross-linking extrusion line which was still warm and displayed a rise in ultrasound velocity as the sample cooled (see Figure 24). Figure 25 shows the increase in ultrasound velocity for a sheet of EVA (designated ZF88) again while cooling immediately after removal from the extrusion\curing line. This effectively gives a measure of the average temperature of the sheet. All signals recorded were well within the measurable region.

Conclusions from this trial

- Ultrasonic measurements in transmission can be performed on the solid sheets produced by Zotefoam and were well within the operational limits of the NPL system.
- Temperature changes of the sheets have been measured by the cure monitoring system in terms of the time of flight or ultrasound velocity.

3.3.4 Recommendation

The ultrasonic cure monitoring system seems to be a good candidate to provide valuable information on Zotefoam's products during cure and temperature during processing of the precursor materials both off line and online. There is believed to be potential for the ultrasonic cure monitoring system to increase productivity and incur all round cost savings. A further trial of the NPL ultrasonic system at Zotefoam is therefore recommended.

ETERNIT

3.4.1 Purpose of the trial

A new type of lightweight slate is produced by Eternit that closely resembles the natural slate in composition and appearance, Figure 26, but is easier and simpler to install. The slates are cured in a mould. To follow this curing process ultrasonics could be a good candidate to assist in reducing cycle times further thereby increasing productivity and reducing costs. Some trials were therefore performed to assess the attenuation of typical signals through the Eternit slate roof tiles using the NPL ultrasonics equipment and also to investigate the temperature sensitivity.

3.4.2 Trial

The ultrasonic system consists of two ultrasonic transducers. One ultrasonic transducer emits an ultrasonic pulse that travels through the slate and is received at the other end by another ultrasonic transducer. The pulse takes a short time to travel. This “time of flight” is inversely proportional to the stiffness (more precisely the elastic part of the elongational modulus), and therefore an indication of cure. The temperature and density of the slate influence the time of flight as well.

A first attempt to send an ultrasonic signal through an old slate was made on 25/11/1999. A strong signal, as shown in the top left corner of Figure 27, was obtained indicating a high transmission. Figures 27 and 28 show the user interface of the program “Ultralyser” that is used to monitor the signal changes and to provide quality control. The slates produced on that day showed a markedly higher attenuation which is a direct result of a modification in the composition. The signal power received however, was still well within the measurable region.

To make an assessment of the temperature sensitivity a fresh slate from the mould was inserted in between the two ultrasonics transducers. A very short time after the slate was taken out of the mould a signal as shown in Figure 28 was received. At higher temperature the moduli of the slate are different and as the slate cools down the time of flight goes down. The decrease of the time of flight as the slate was cooling down is shown in Figure 29. This effectively gives a measure of the average temperature of the slate.

3.4.3 Conclusions

- Ultrasonic measurements in transmission can be performed on the slates well within the operational limits of the NPL system.
- Temperature changes of the slate have been measured by the cure monitoring system.

3.4.4 Recommendation

The ultrasonic cure monitoring system seems to be a good candidate to provide valuable information to Eternit on cure and temperature during moulding. There is potential for the ultrasonic cure monitoring system to suggest further reductions in production cycle time.

3.4.5 Future Work

The main target for future work would be to assist Eternit to reduce cycle times still further. From the above, this system appears to be capable of providing detailed information on the average temperature within the slate during the early part of the production cycle and also on the degree of cure during the final part of the cycle. The system is expected to be able to help optimise production if used in conjunction with well controlled heaters within the mould and possibly also preheating with microwaves.

It is proposed that Eternit's current development is modified to allow ultrasonic transducers to monitor the production of slates. It is proposed that a further trial is carried out over a 5 day period to assist Eternit in the optimisation of the production process to attempt to reach further significant reductions in cycle time.

3.5 REXAM

3.5.1 Purpose

To ascertain whether the state of cure of an epoxy coating on a copper foil ("B" staged) could be measured by ultrasonics. This is essential to know as further processing is carried out dependent on the first stage ("B" Stage) which affects the final properties.

3.5.2 Materials

Two samples were provided of different states of cure ("B" staged).

Copper foil thickness 0.018 mm.

Sample 1 (NPL code HPY-000A) partially cured coating thickness = 0.029 mm

Sample 2 (NPL code HPY-000B) thought to be of longer cure time were, coating thickness = 0.074 mm.

3.5.3 Ultrasound Testing

The time of flight by transmission through each sample, including the copper foil, was determined using the NPL "EDS" system and a set of 0.5MHz transducers assembled in the ultrasonics test rig at room temperature. Measurements were taken every 6 seconds for 3 minutes and averaged. The entire thickness of each sample was measured with a calibrated micrometer.

3.5.4 Results

By subtracting the copper foil thickness from the overall thickness for each sample the apparent velocity in m/s was calculated.

Sample 1 Velocity = 355.9 m/s.

Sample 2 Velocity = 494.5 m/s.

3.5.5 Conclusion

It can be seen that sample 2 has a velocity nearly 40% faster than sample 1.

The ultrasonic technique can distinguish the state of cure between two epoxy coated copper foils and thus shows potential for in-line monitoring and process control of commercial production of coated copper foils.

4. CONCLUSIONS

- NPL's ultrasonic monitoring system has been used to assess five companies industrial processes.
- Optimum processing conditions were identified for materials produced by BIP.
- The optimum processing conditions, determined by online ultrasonic monitoring, at BIP were shown to agree well with product properties and established conditions at BIP.
- It was not found possible to monitor cross-linking of Raychem's polyolefins nor BIP's polyester resins under the conditions investigated.
- Promising results were obtained with Zotefoam's extruded sheets, Eternit's slate roof tiles and Rexam's epoxy coated copper sheets.
- During the course of the project the Ultrasonic equipment has been upgraded so that it now operates in a fully automatic mode and it is able to follow changes in ultrasonic peak height as well as time of flight.

5. ACKNOWLEDGEMENTS

This report was prepared as part of a project on the measurement of materials properties related to processing carried out in a programme of research financed by the Engineering Industries Directorate of the UK Department of Trade and Industry.

NPL are pleased to acknowledge the substantial contributions made in carrying out this work by BIP, Raychem, Zotefoam, Ethernet, Rexam and HW Wallace.

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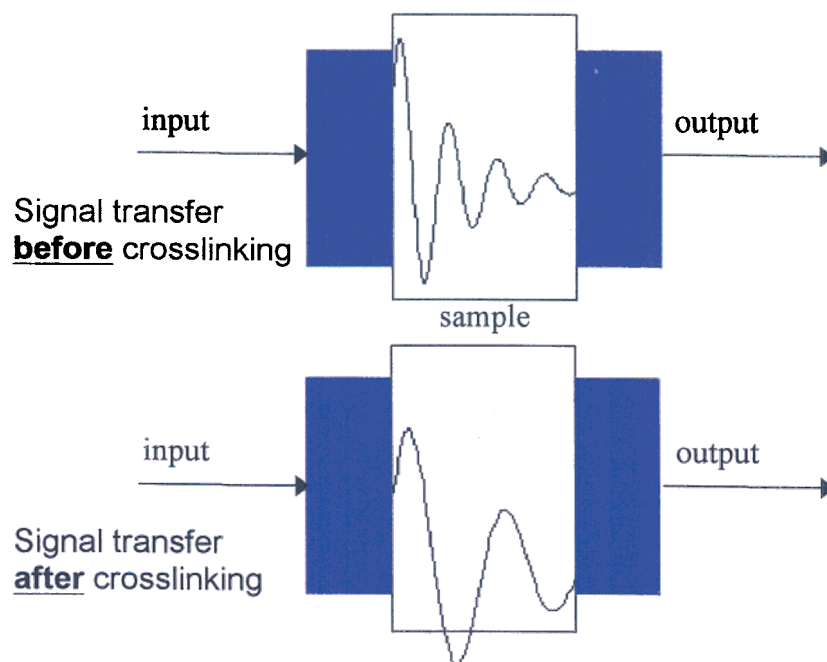
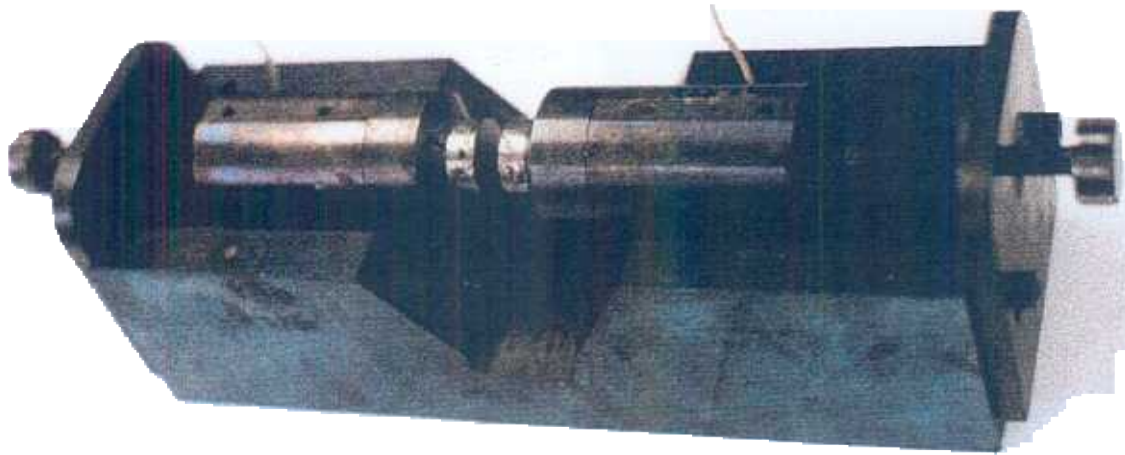


Figure 1. The laboratory rig used to clamp samples in between two transducers at room temperature (top). Schematic of an ultrasonic pulse measurement. A sample is clamped between two ultrasonic transducers (bottom).

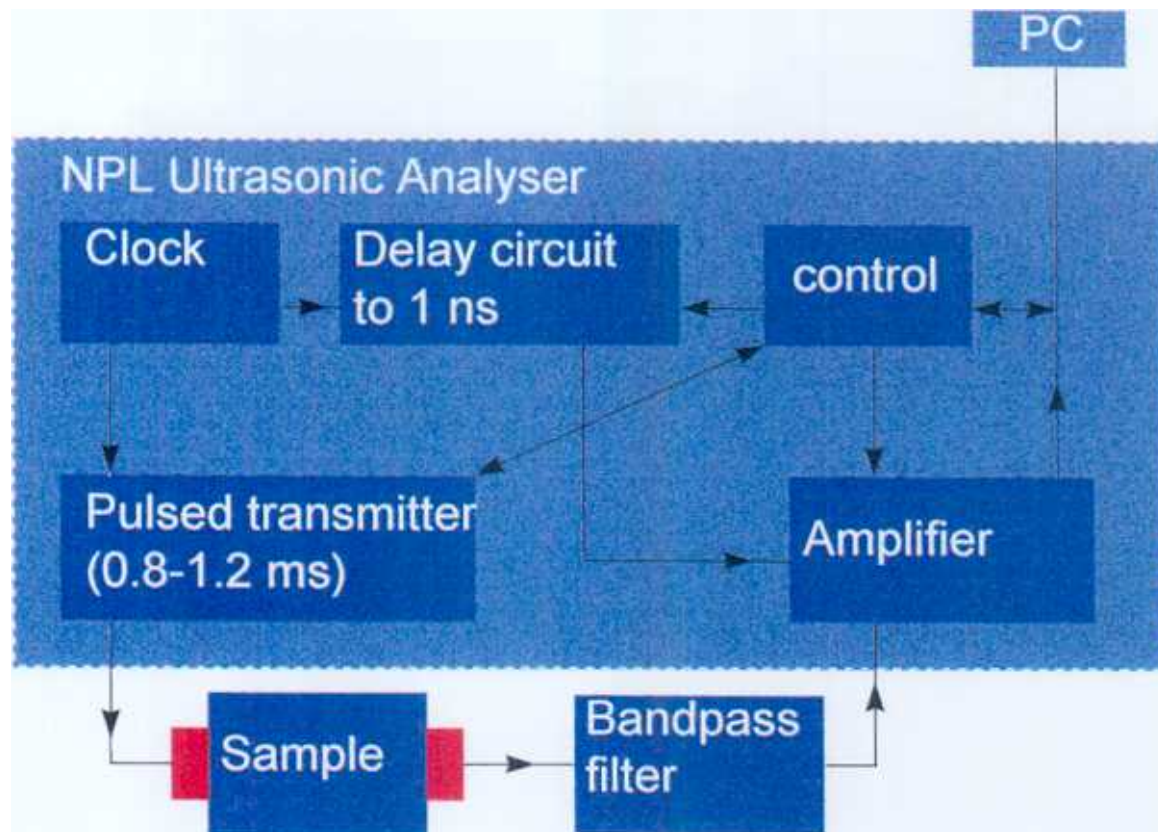


Figure 2. Schematic functional diagram of the EDS ultrasonics unit.

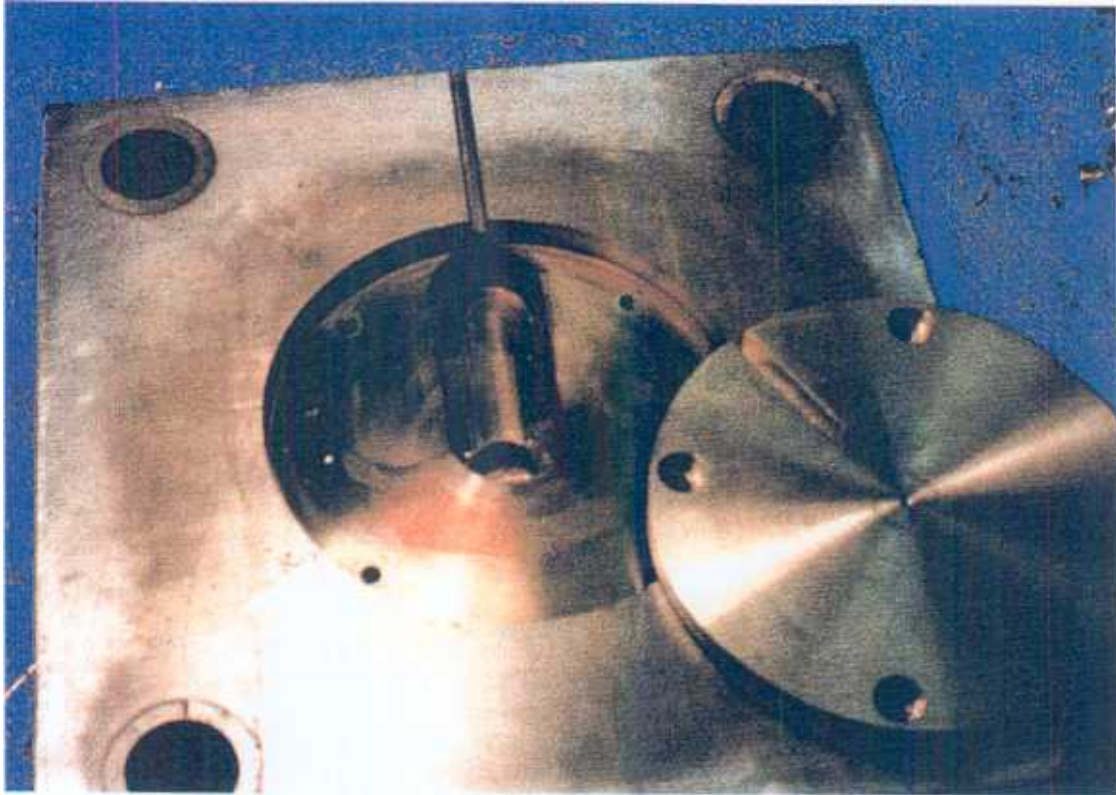


Figure 3. The backside of half the mould modified to fit transducers. The transducer fits in the middle. In this case the transducer has a side connection, the connecting wire is led out through a groove. The plate is fitted to close it up.

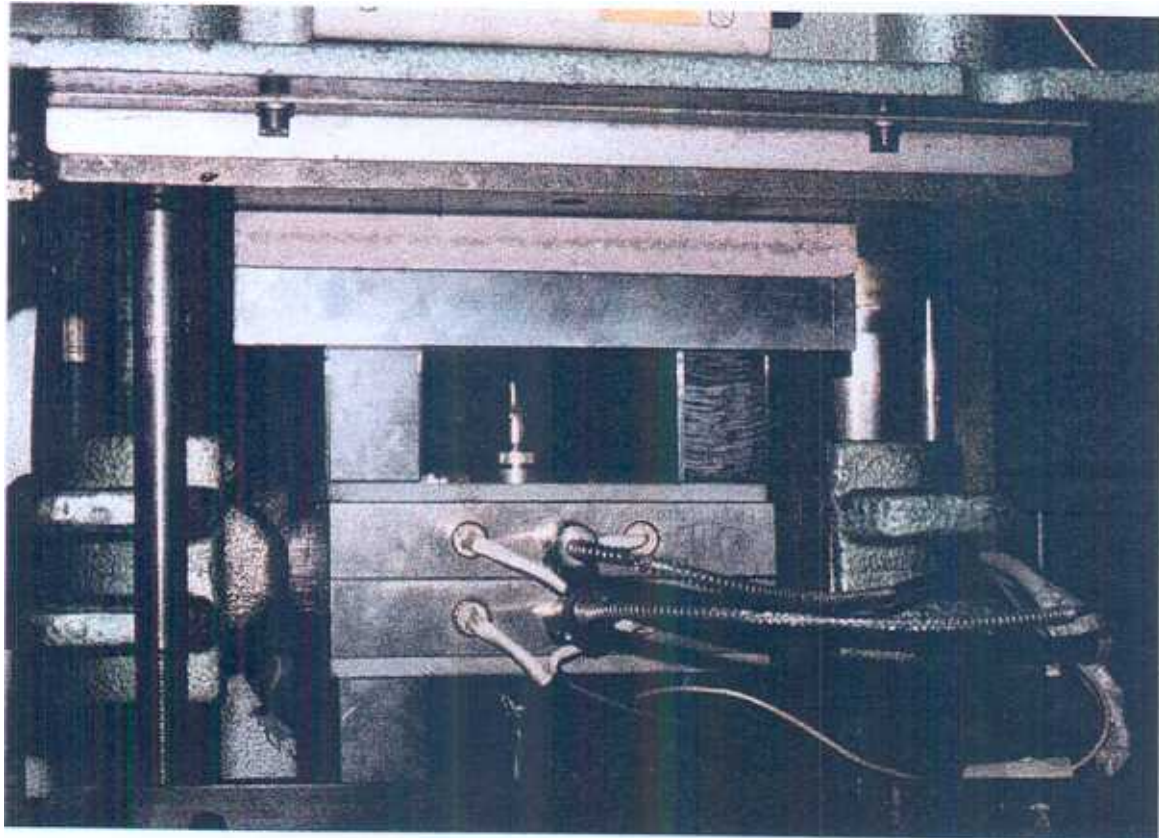


Figure 4. Mould fitted in a press. In this case the transducers have the wiring at the top. The transducers can be seen sticking out at the top and the bottom.

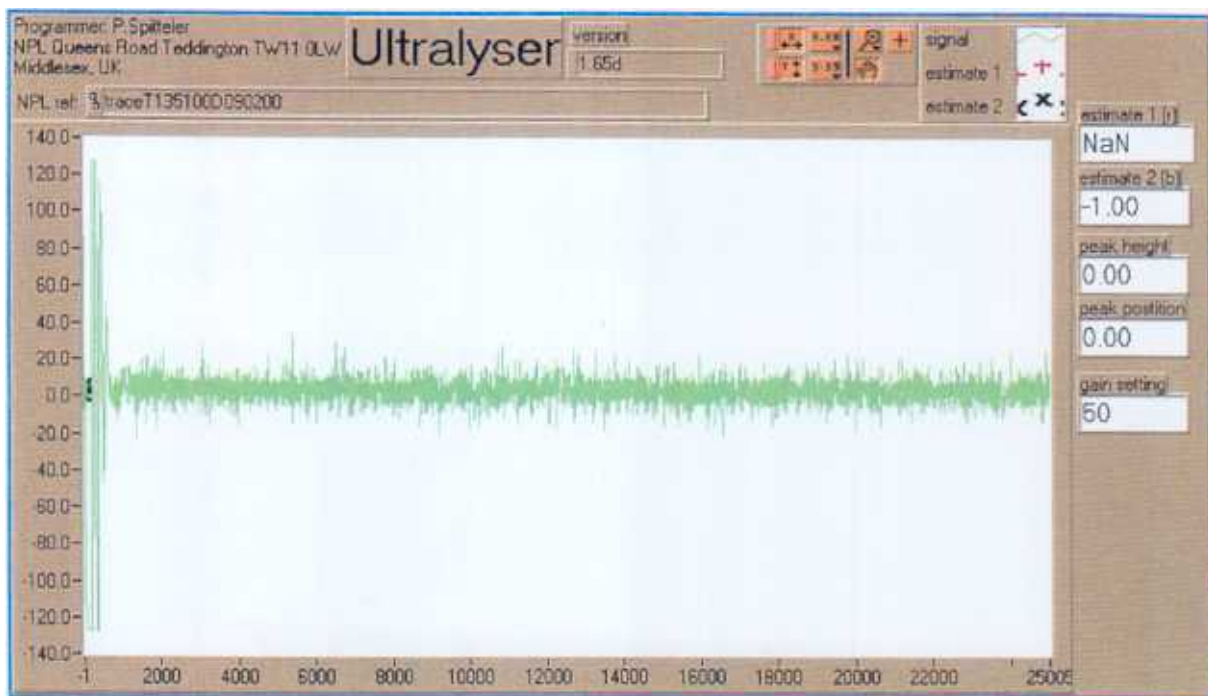


Figure 5. Ultrasonic trace through a polyester resin at BIP in the mould, showing no detectable peak. This was thought to be due to degraded coupling fluid.

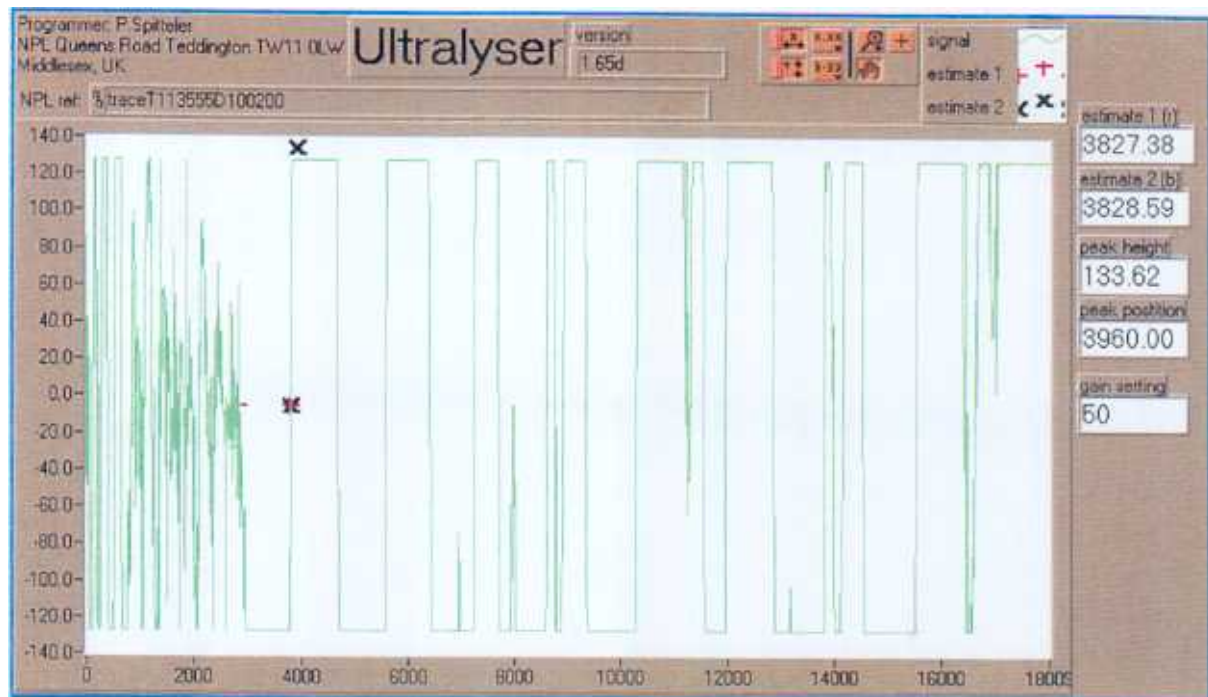


Figure 6. Ultrasonic trace (on the same scale as figure 5) showing a detectable signal after cleaning and applying silicone grease as compliant.

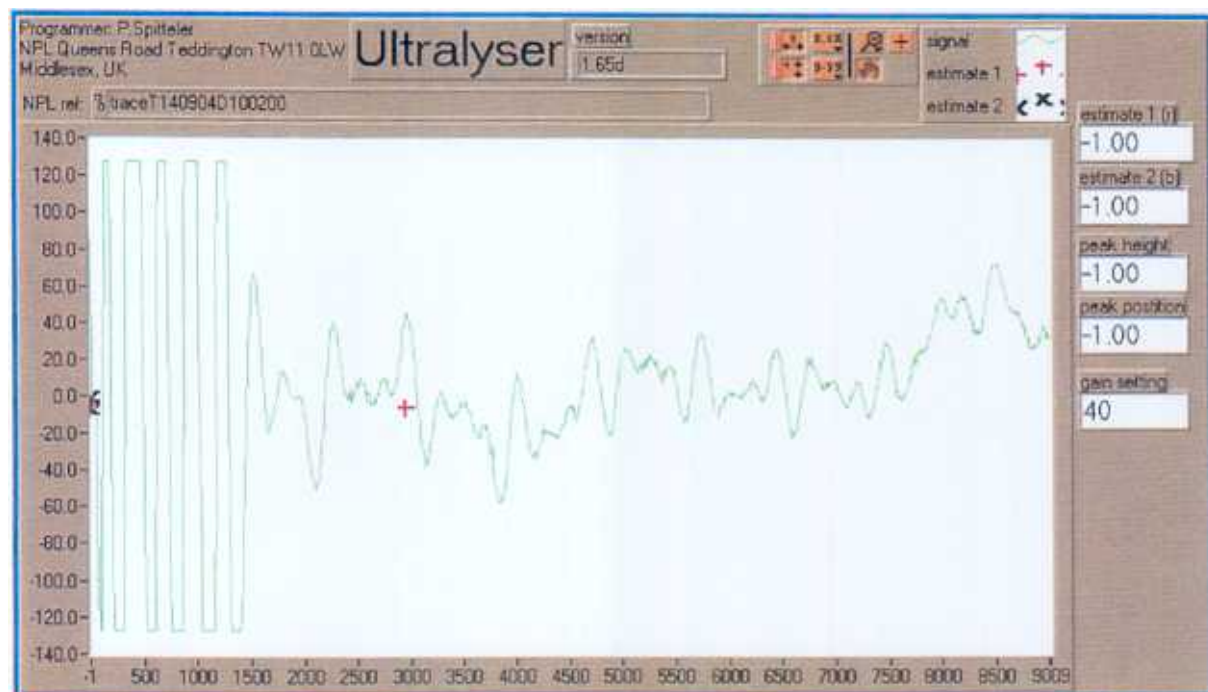


Figure 7. Ultrasonic trace of a polyester resin during compression moulding. The pattern in the signal is due to ringing of the press as a result of the Ultrasonics pulses and not a pulse through the moulding material. Scattering by the glass fibre filler could be the cause of the absence of a detectable signal.

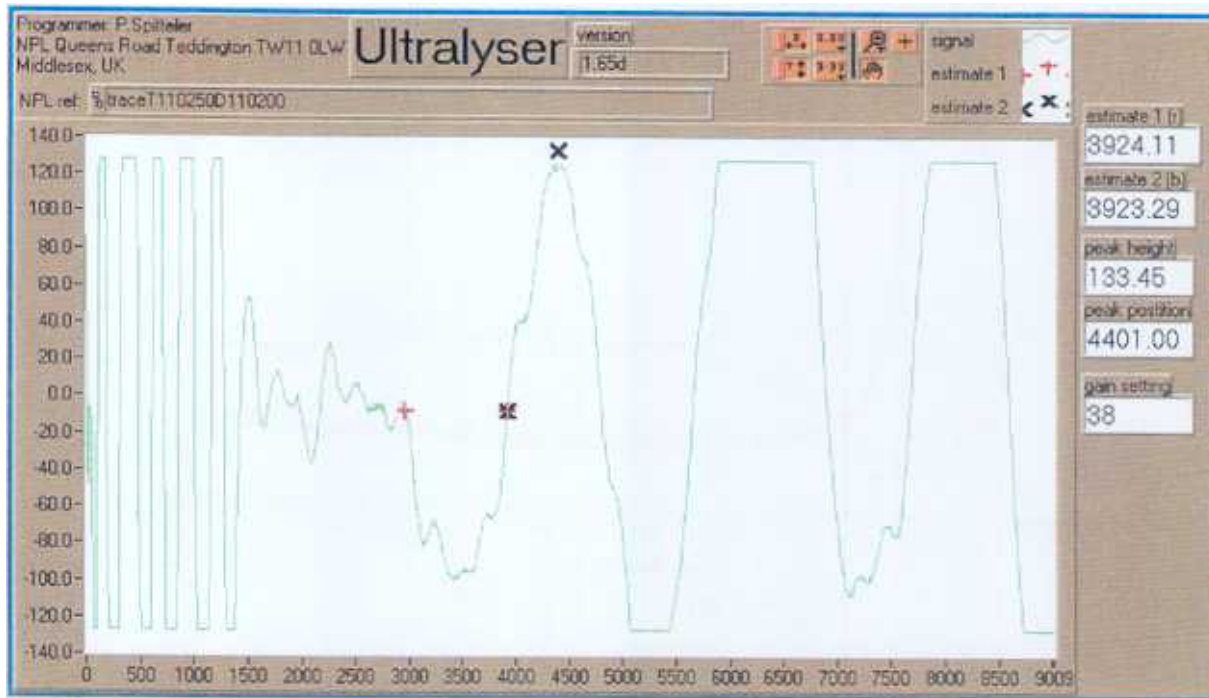


Figure 8. Ultrasonic trace for a second polyester resin. As with figure 7 no time of flight could be measured.

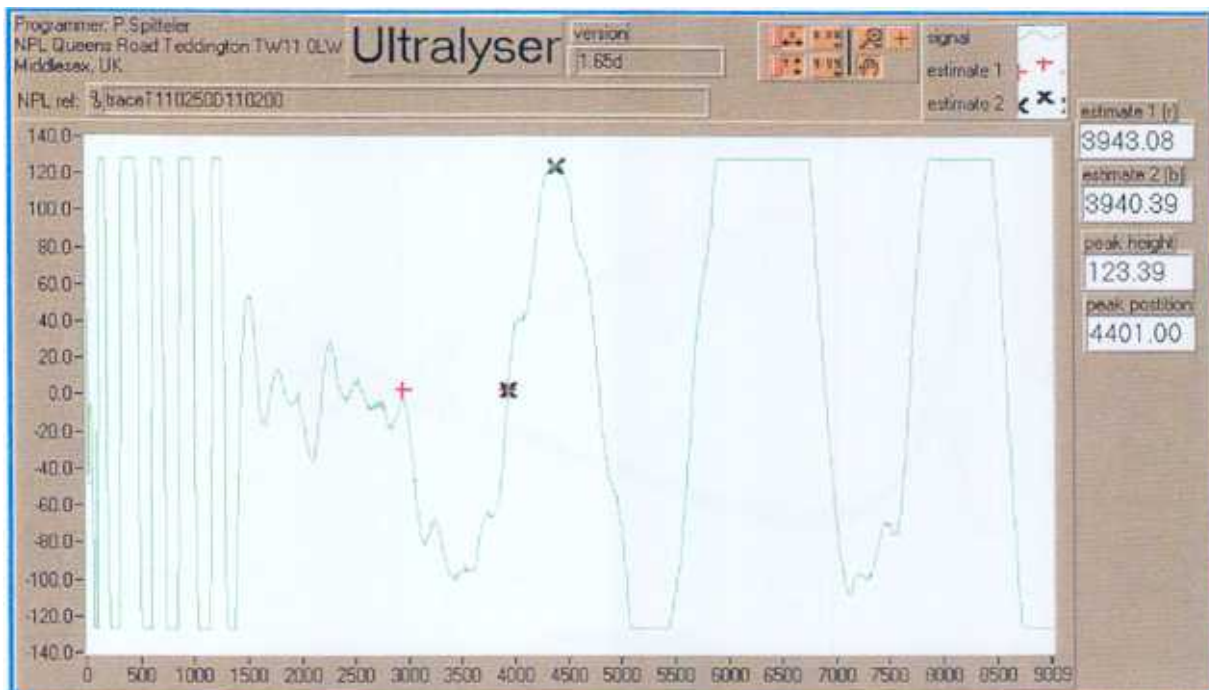


Figure 9. Ultrasonic trace of a urea formaldehyde resin during compression moulding. The time of flight and peak height were both detected from this trace.

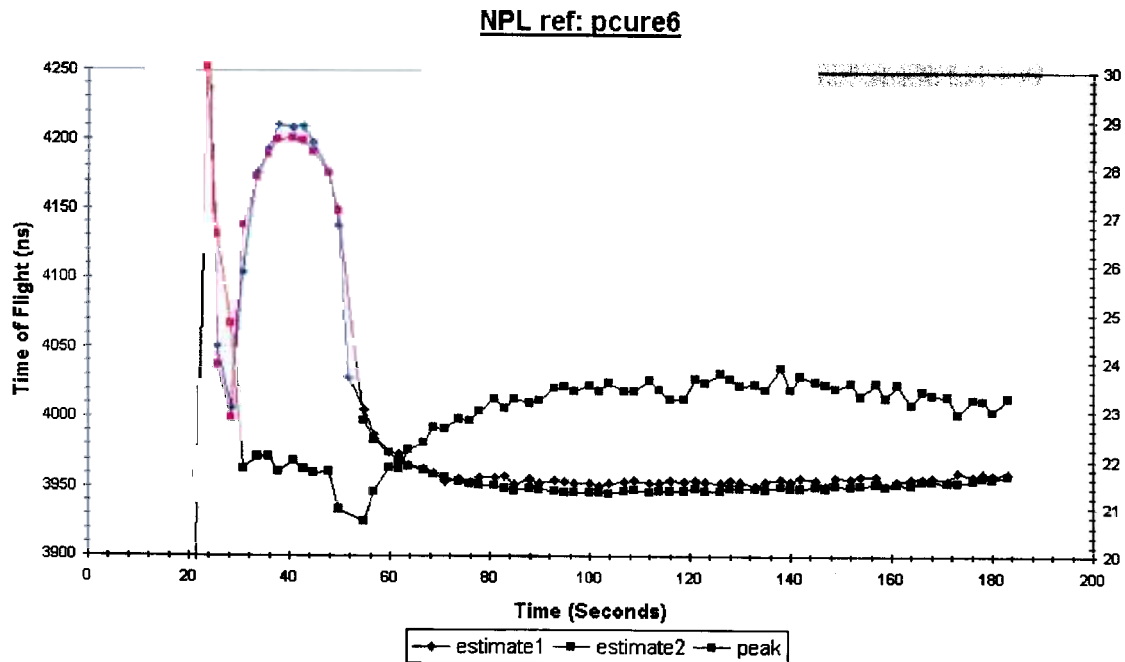


Figure 10. Plot of time of flight and peak height of an ultrasonic pulse propagating through a melamine resin. The two estimates of time of flight (estimate 1 and 2) both increase up to a maximum at about 40 seconds and then level out after about 70 seconds. Such data can be used to determine optimum cycle times.

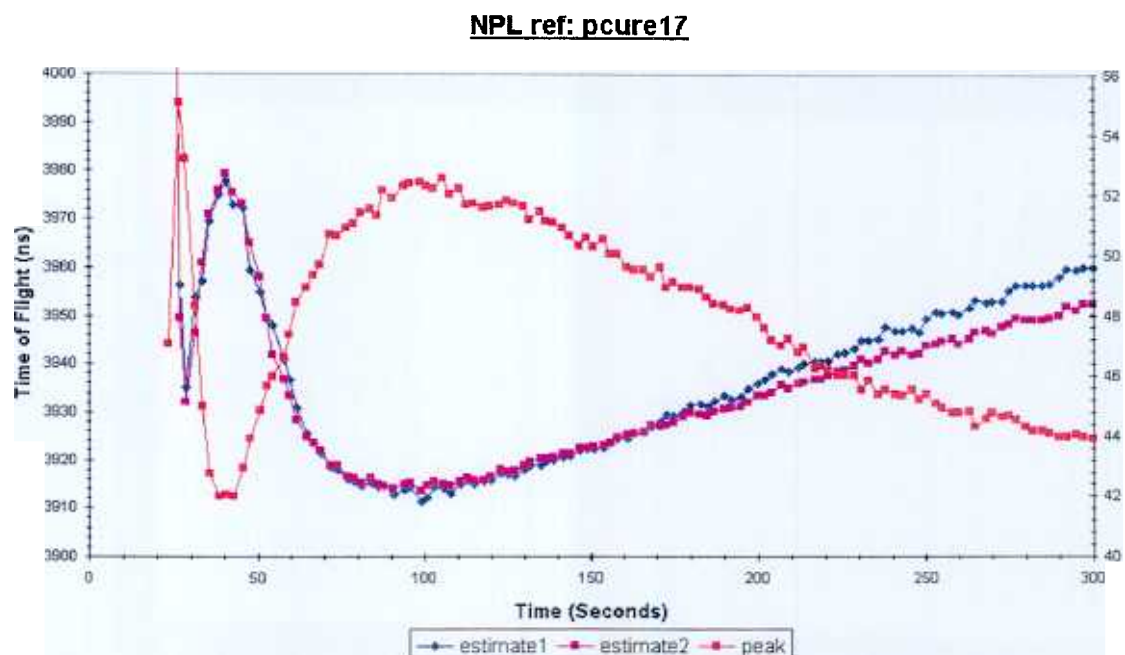


Figure 11. Plot of time of flight and peak height against time in mould (as figure 10) but for a urea formaldehyde that exhibits regression (i.e the time of flight can be seen to increase after about 100 seconds associated with a reduction in stiffness). The peak height behaviour is mirrored in the time of flight.

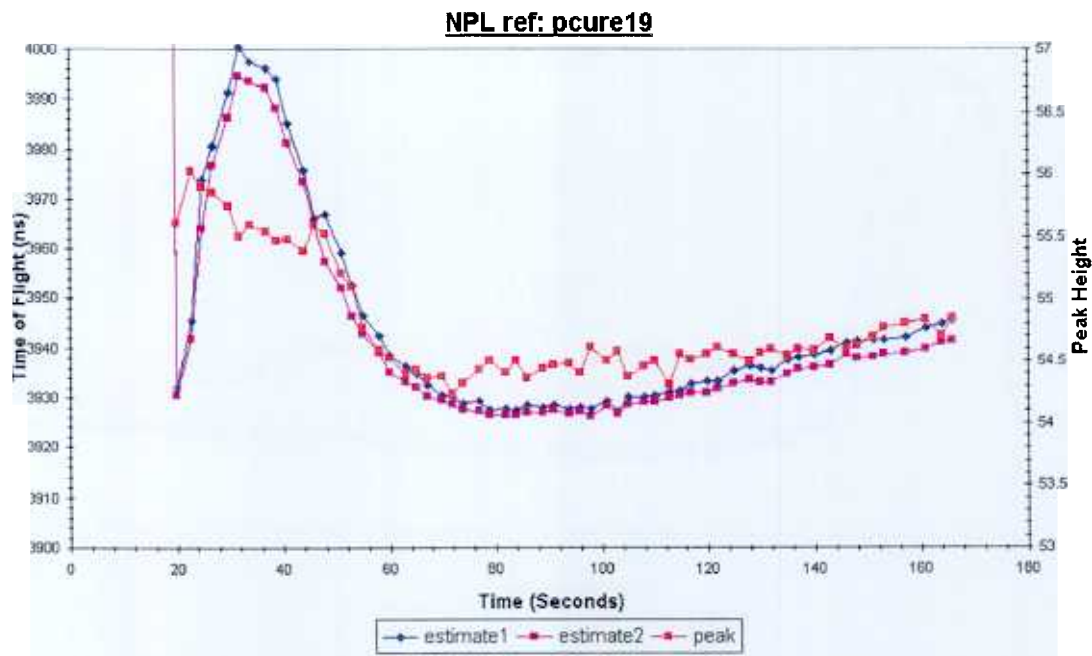


Figure 12. Time of flight and peak height plot for a different urea formaldehyde. (see figure 7).

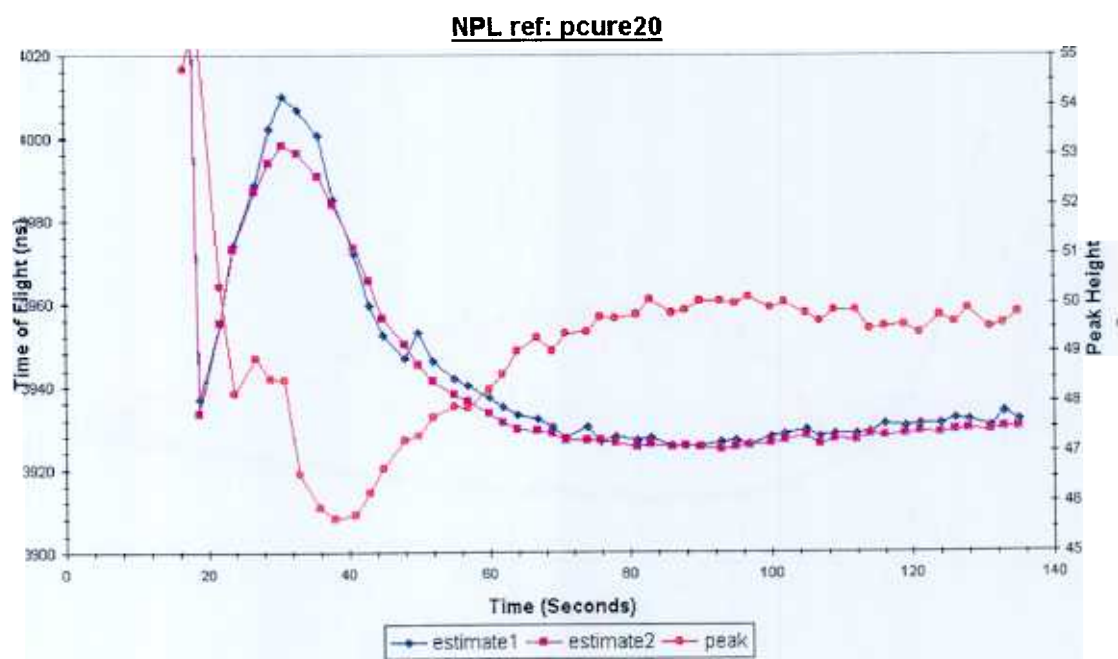


Figure 13. Time of flight and peak height plot of a urea formaldehyde during moulding.

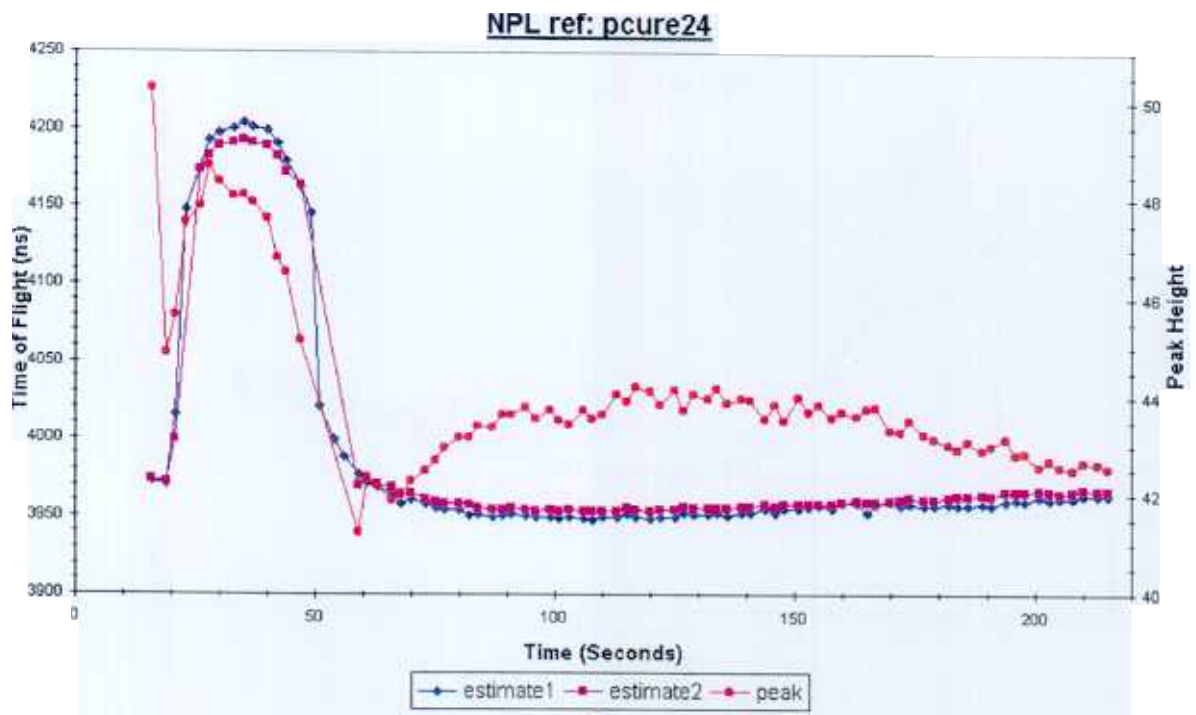


Figure 14. Time of flight and peak height plot of a melamine resin during cure.

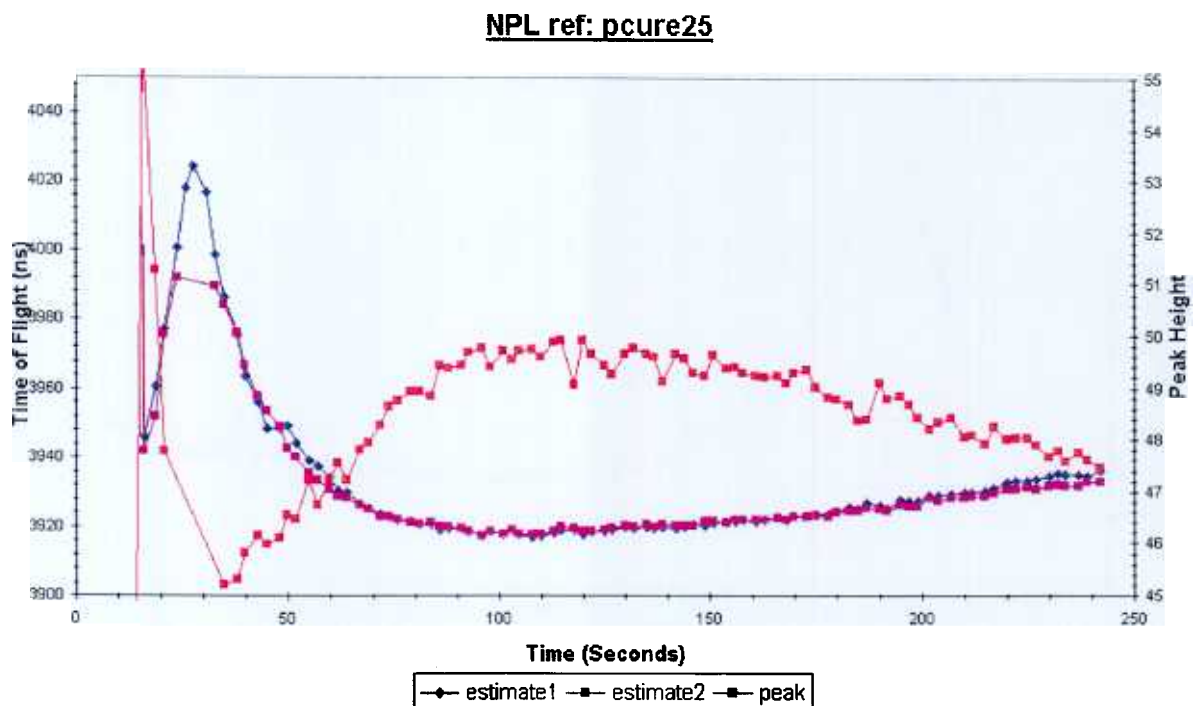


Figure 15. Time of flight and peak height plot of a urea formaldehyde during moulding. Some regression can be seen after about 120 seconds.

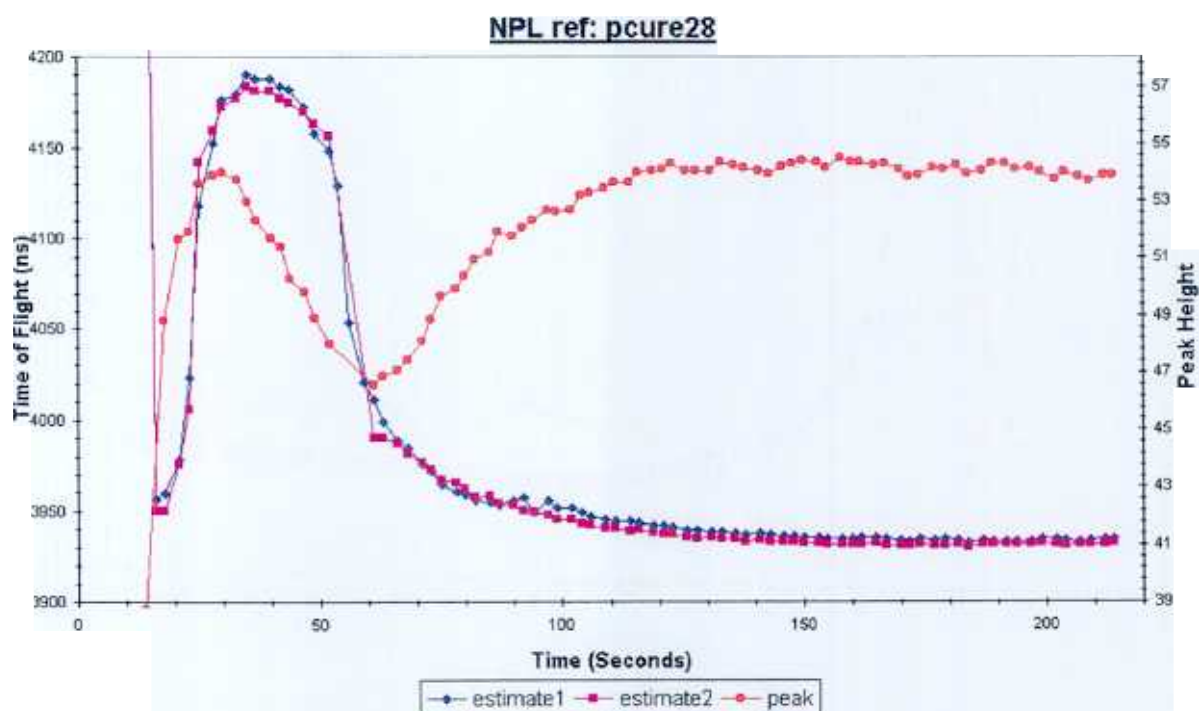


Figure 16. Time of flight and peak height plot for an injection grade Urea/melamine during moulding.

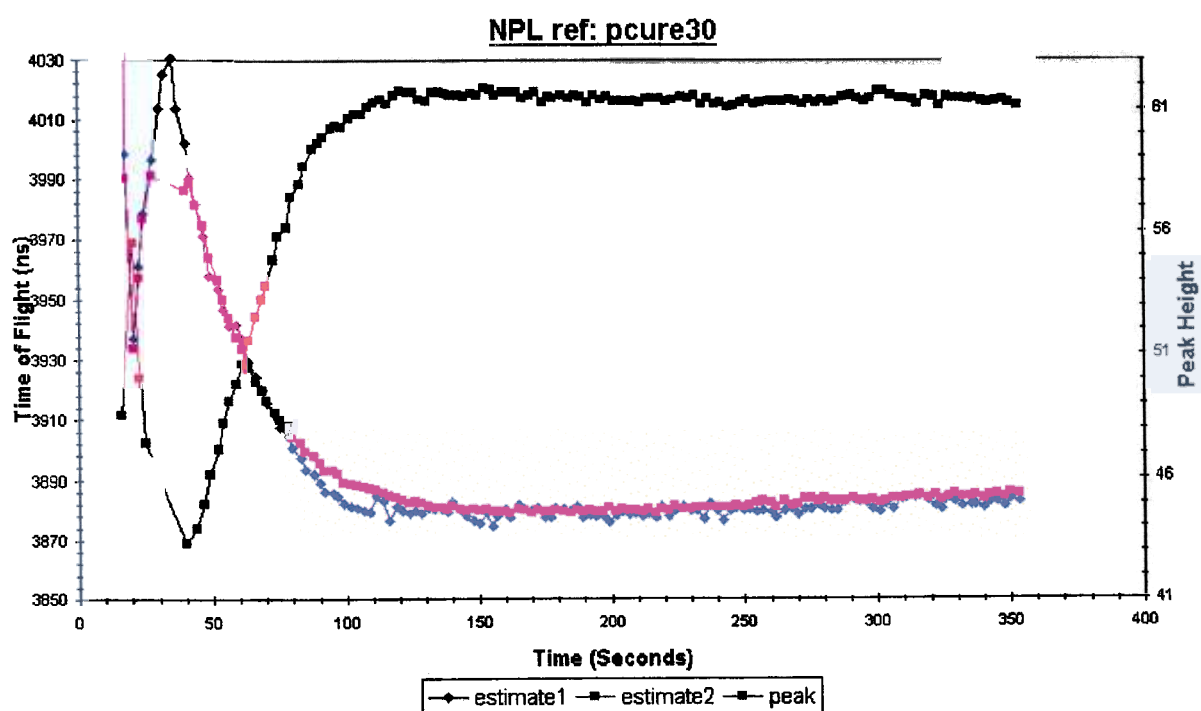


Figure 17. Time of flight and peak height plot for a melamine resin during cure (seen as a reduction in time of flight and an increase in peak height between about 40 seconds and about 120 seconds).

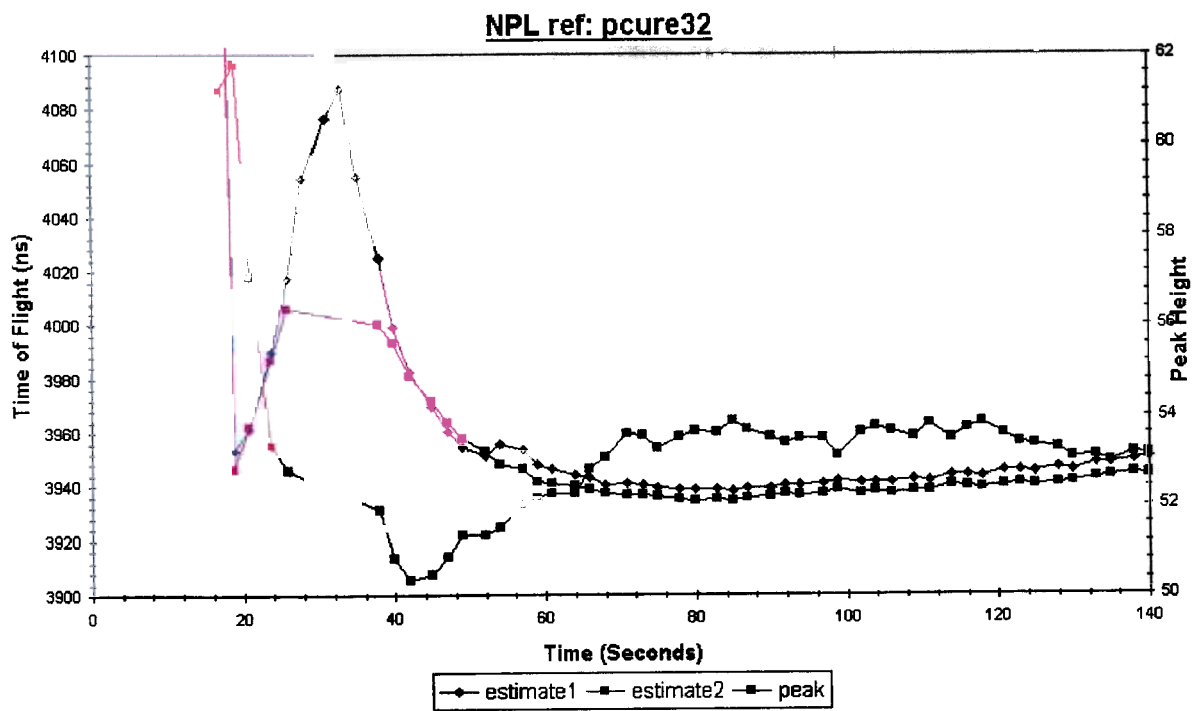


Figure 18. Time of flight and peak height plot for a further thermoset during moulding.

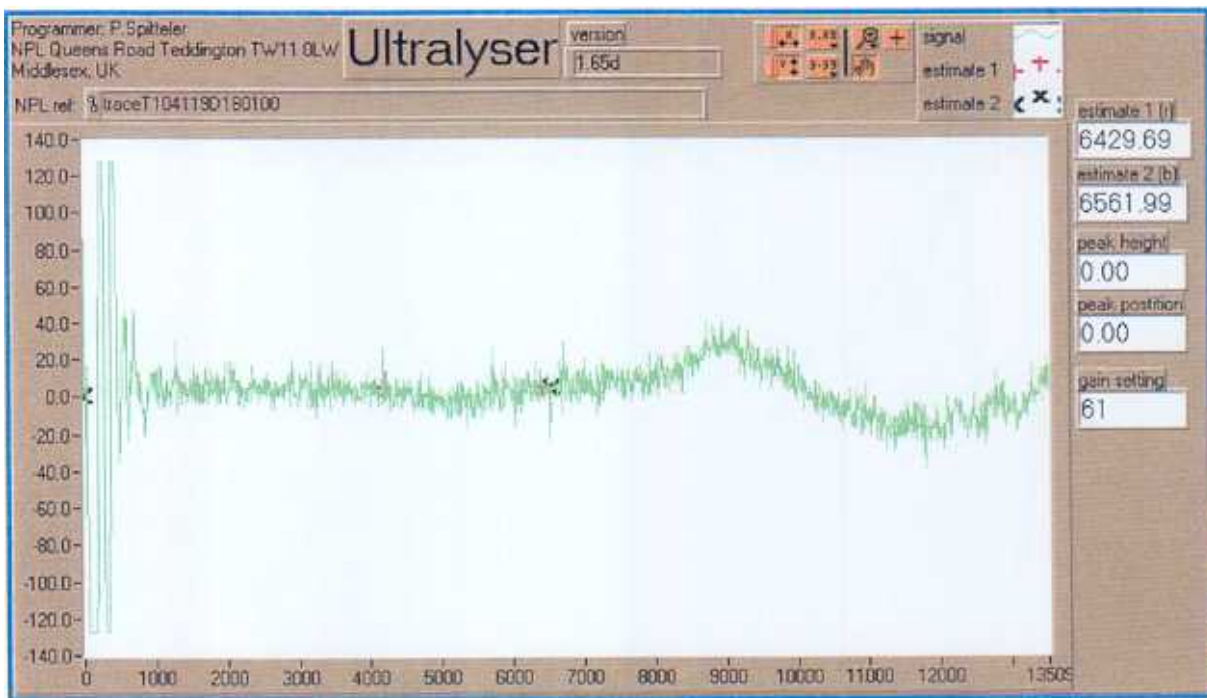


Figure 19. Initial ultrasonic signal from the Raychem trial. The signal to noise ratio was poor leading to unreliable time of flight measurements. .

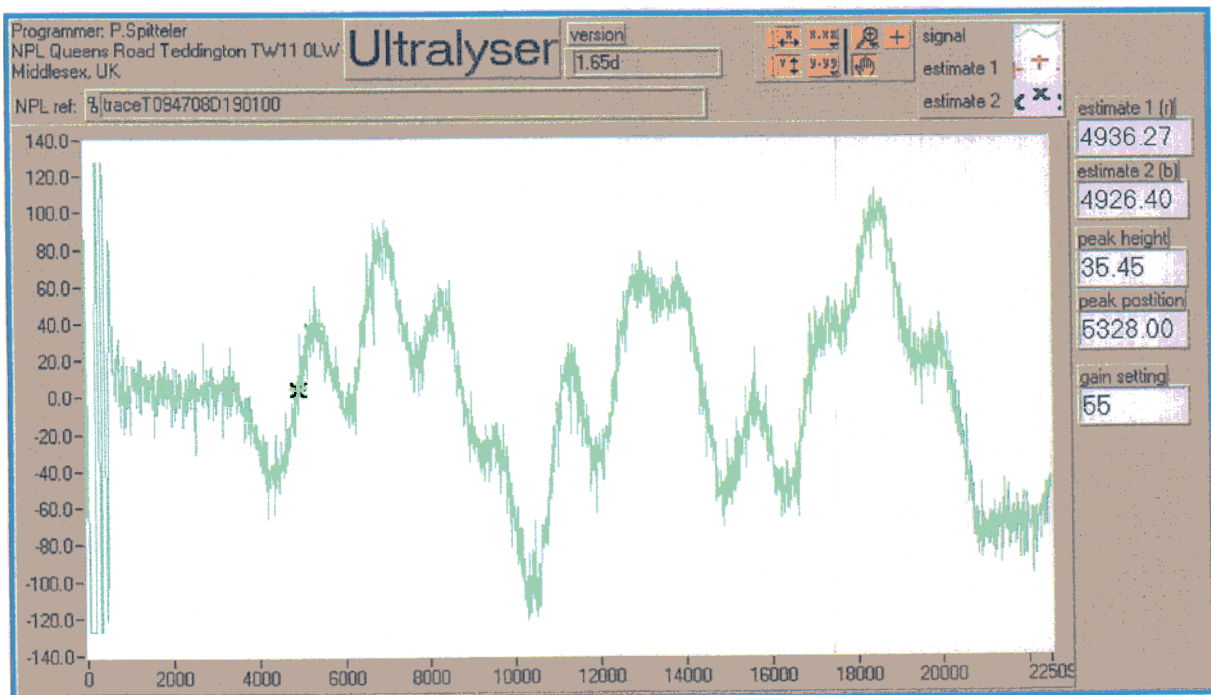


Figure 20. Ultrasonic signal through a 3mm metal plate and two plaques of resin mounted on the bottom plate of the mould.

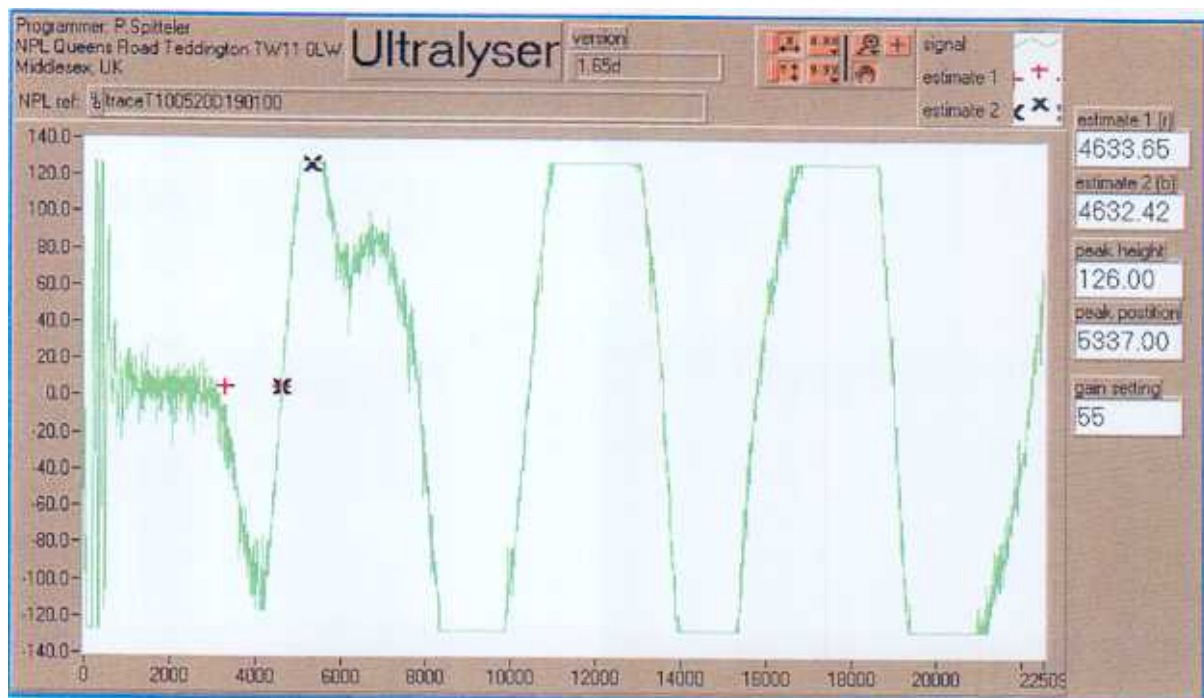


Figure 21. Ultrasonic signal with the 1mm metal plate and two plaques of resin mounted on the bottom plate of the mould. A much stronger signal is seen than in the case of a 3mm plate (figure 20).

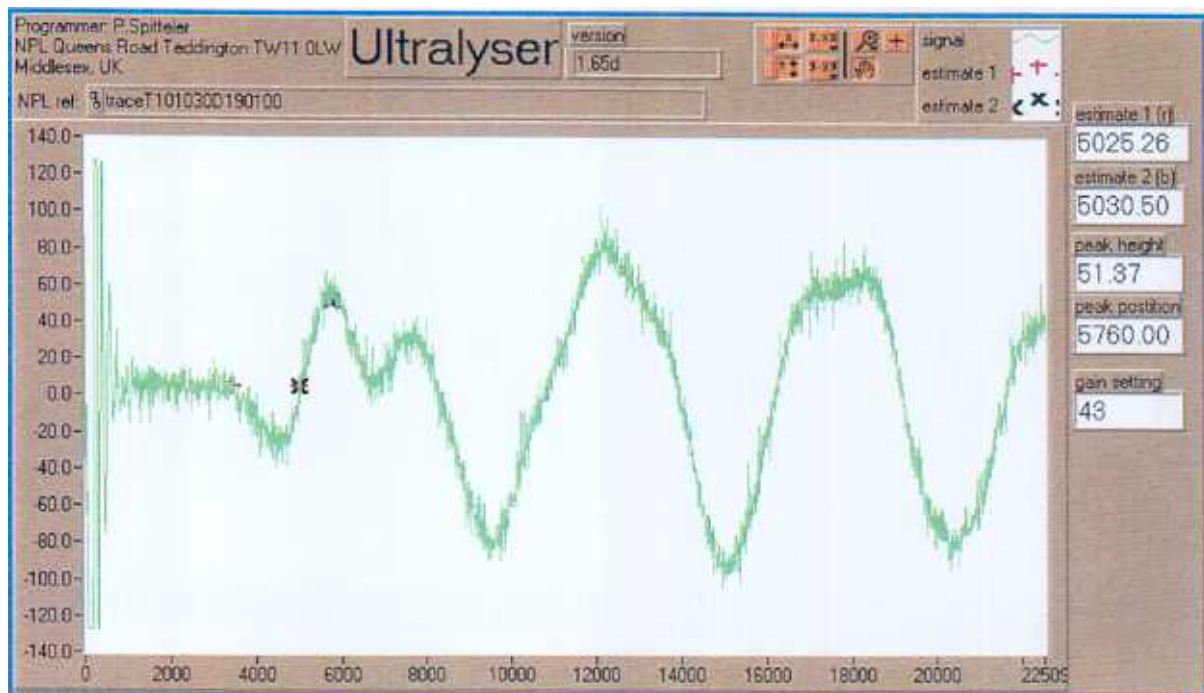


Figure 22. Ultrasonic signal on heating to 190°C; the signal strength reduced.

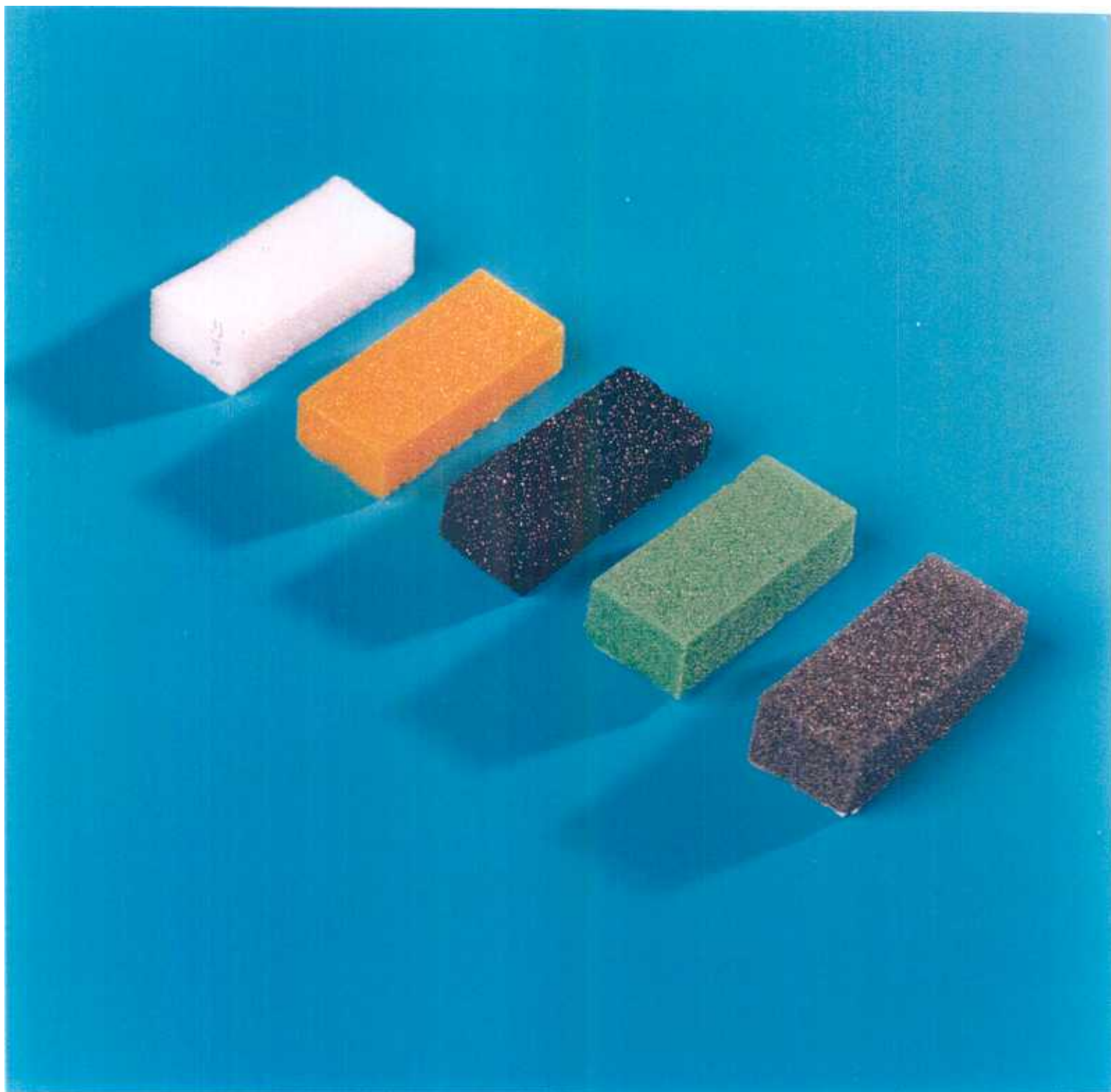


Figure 23. Examples of foam materials produced by Zotefoams. The length of each samples is 35mm.

Change in the Velocity of Ultrasound with Temperature on Cool Down Immediately after Extrusion NPL ref: ZF45

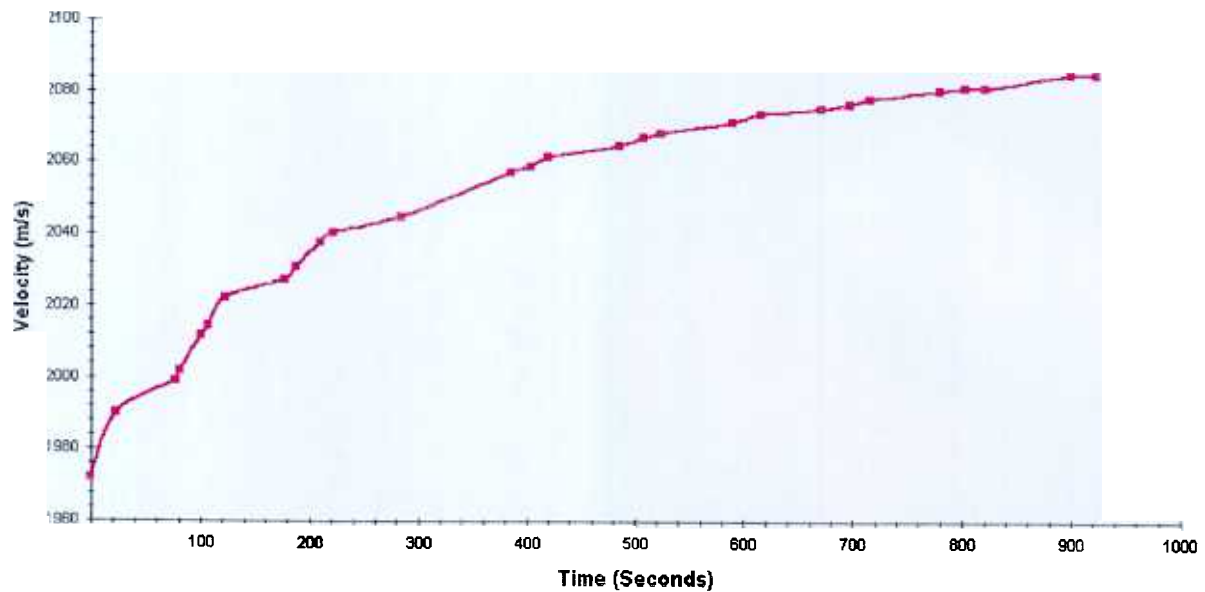


Figure 24. Ultrasonic velocity as a function of time during cooling of an extruded sheet of polyethylene, after cross-linking but prior to foaming.

Change in the Velocity of Ultrasound with Temperature on Cool Down Immediately after Extrusion NPL ref: ZF88

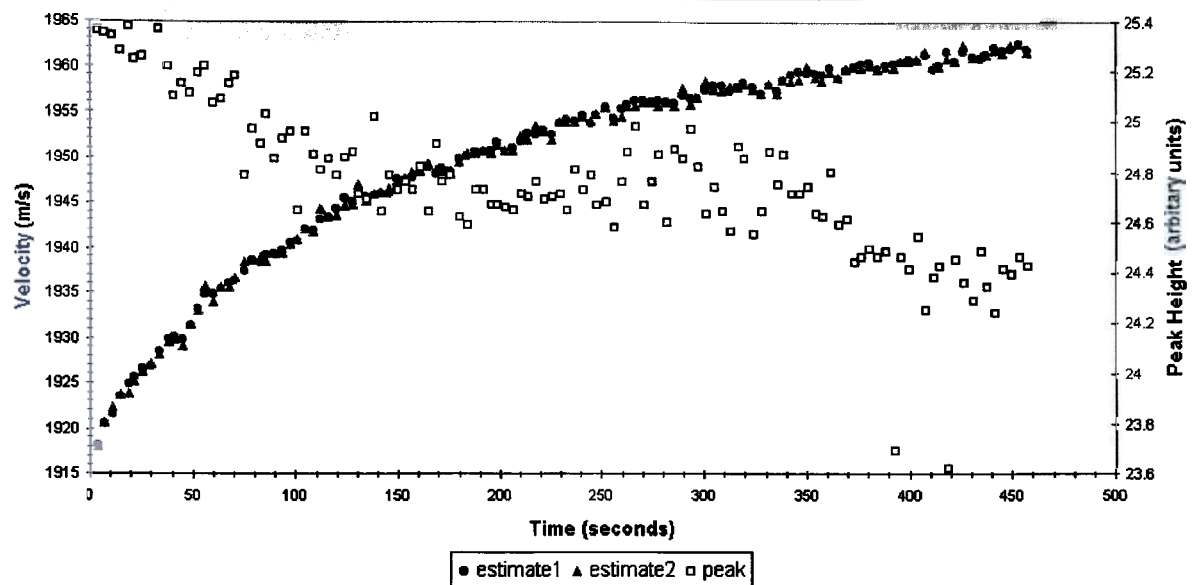


Figure 25. Ultrasonic velocity (solid circles and solid triangles) and peak height (open squares) against time for a EVA sheet after extrusion, during cooling.



Figure 26 Photograph moulded slate roof tile produced by Eternit.

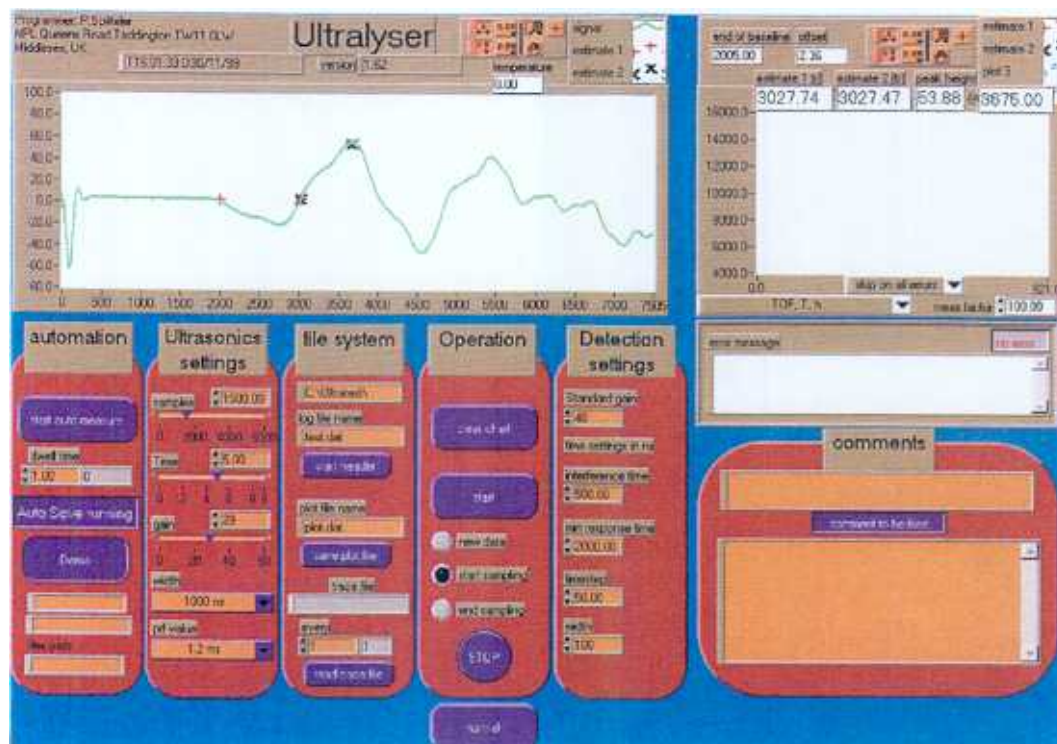


Figure 27. Operator user panel of Ultralyser software showing the result of an ultrasonics measurement of a moulded slate roof tile from Eternit.

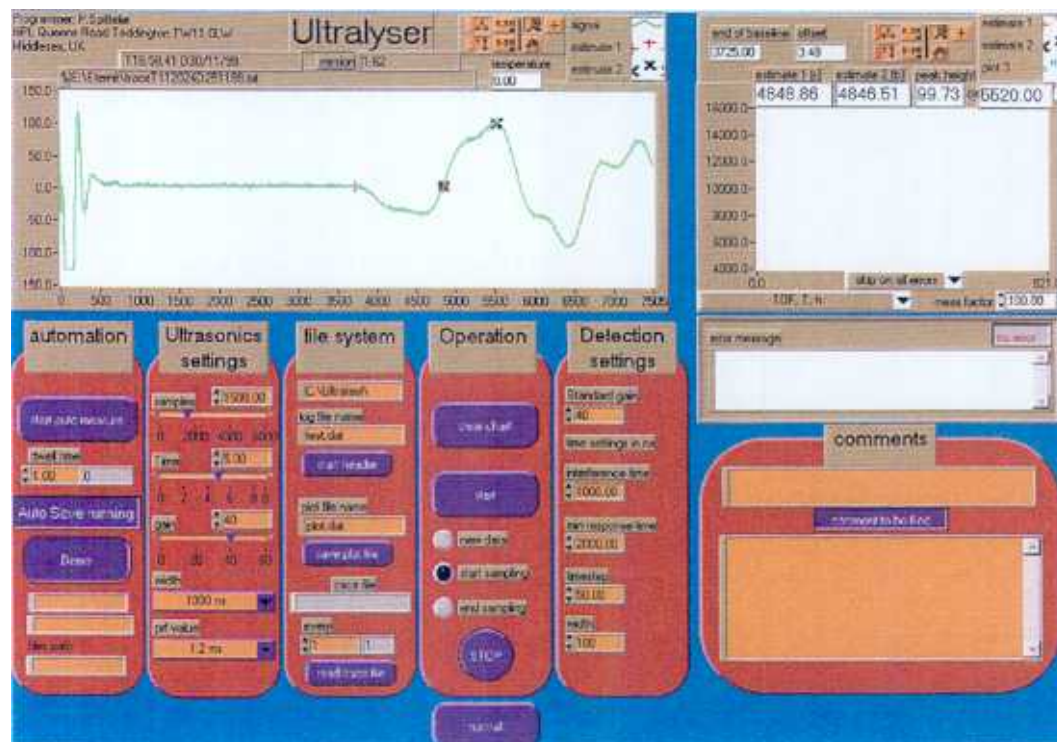


Figure 28. Operator user panel of ultralyser software showing an ultrasonics measurement of a slate roof tile straight after moulding.

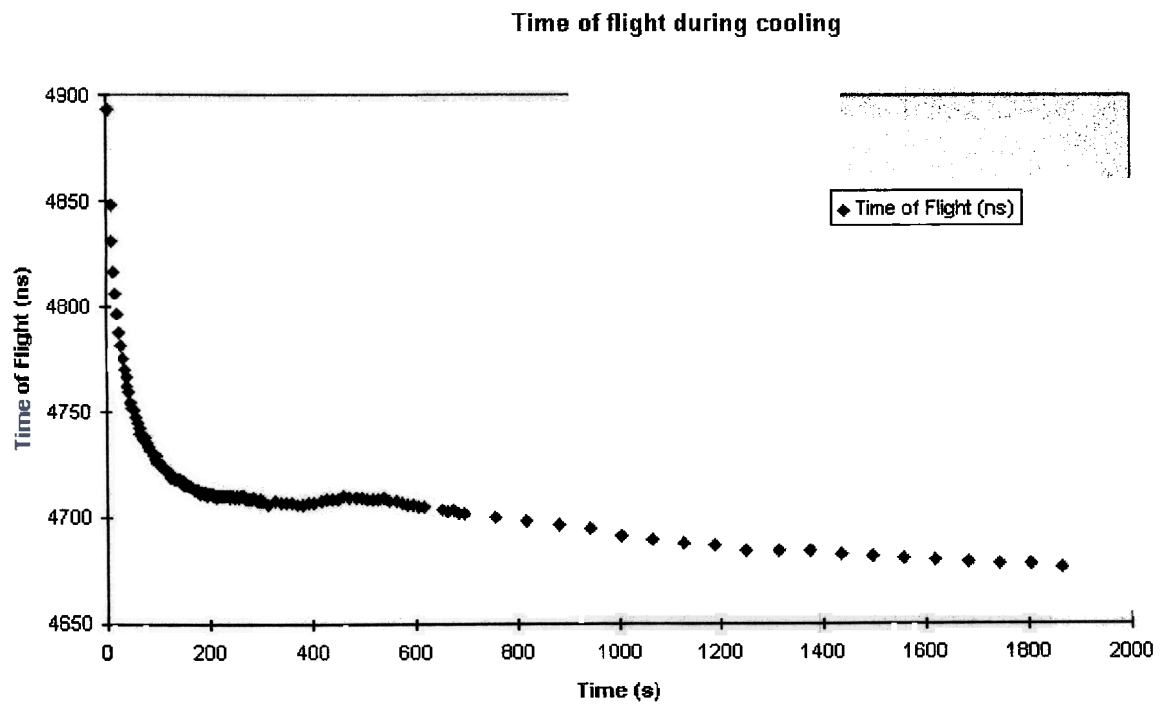


Figure 29. Plot of ultrasonic pulse time of flight against time after moulding. As the tile cools down the time of flight can be seen to reduce, as expected.