

Ultrasonic Monitoring Of Reactive Polymer Processing

Summary

Reactive processing such as injection moulding of thermosetting resins can now be monitored non-intrusively using ultrasonic pulses. In this technique, transducers placed in the mould measure the time of flight of an ultrasound pulse, which in turn is related to the stiffness of the test material. As the curing reaction proceeds, material stiffness increases while the time of flight of the pulse decreases as the wave velocity reduces. The time of flight (and thus velocity) can be measured up to an accuracy of 1ns. A number of industrial trials of this technology have been conducted covering injection moulded and compression moulded products. Improvements in productivity and/or enhancement of quality assurance are the direct benefits of this technology. A new project, which aims to demonstrate the benefits of ultrasonic monitoring of materials processed for the construction industry is being launched.

CS Brown, C.B. Hobbs and P.H.J. Spitteler

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Ultrasonic Measurement

Ultrasound is a well established technology used for medical imaging, flaw detection of composite structures and measuring thickness. Recent advances in transducer technology make it possible to employ ultrasound to continuously monitor curing up to 200°C. Since ultrasound can travel through steel mould walls, the state of cure can be monitored *in situ* without disturbing the product during moulding [1].

The "stiffness" of a material increases with an increase in the degree of cure. The degree of curing can be followed by determining the velocity 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 is inversely proportional to damping, $\tan \delta$. The time of flight of the ultrasound pulse and power are also affected by the thickness, the density of the product and temperature [2].

The frequency of the ultrasound pulses and the type of transducers need to be matched with the materials and type of mould used for an optimal result.

The NPL Method

In NPL's cure monitoring method, the ultrasonic pulses are continuously sent (see [Figure 1](#)) through the curing material. The software package, **Ultralyser** has been developed ([Figure 3](#)) to automatically analyse and monitor the received ultrasonic pulses and display variations in the time of flight and the signal amplitude. The change in the pulse amplitude during cure of a reactive polymer is shown in [Figure 2](#). Curves obtained in this manner can be likened to the fingerprints of the test material during cure.

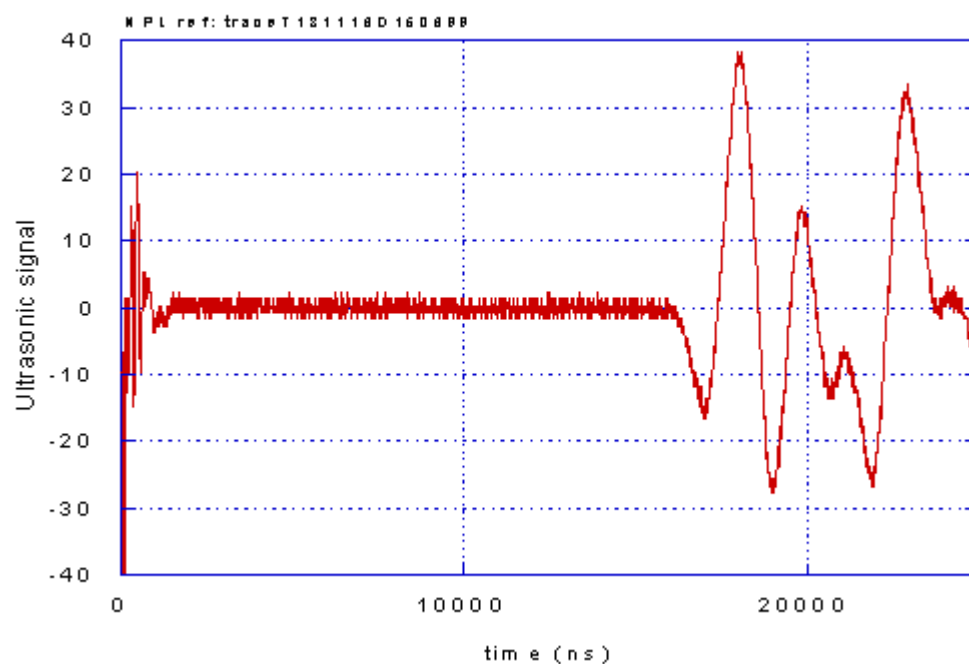


Figure 1 Ultrasonic pulse as received through a sample using a 0.5 MHz transducer capable of operating up to 200°C.

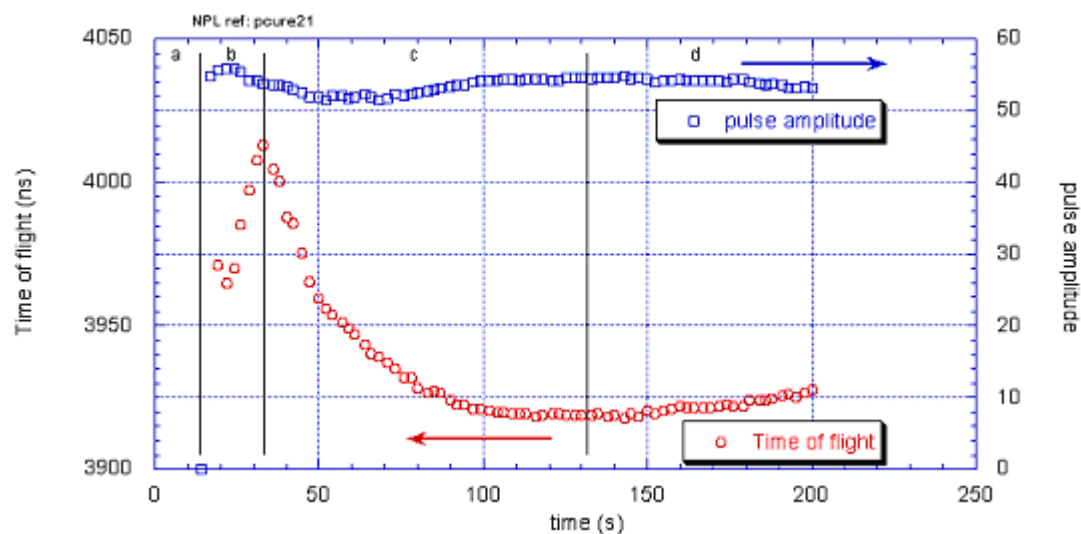


Figure 2. The change in the TOF and amplitude of an ultrasound pulse during a curing reaction. In zone a, the powdered material was compressed in the mould. In zone b, the TOF and pulse amplitude increase with temperature. Zone c shows the influence of cure. When the sample is kept in too long, regression can be observed (zone d).

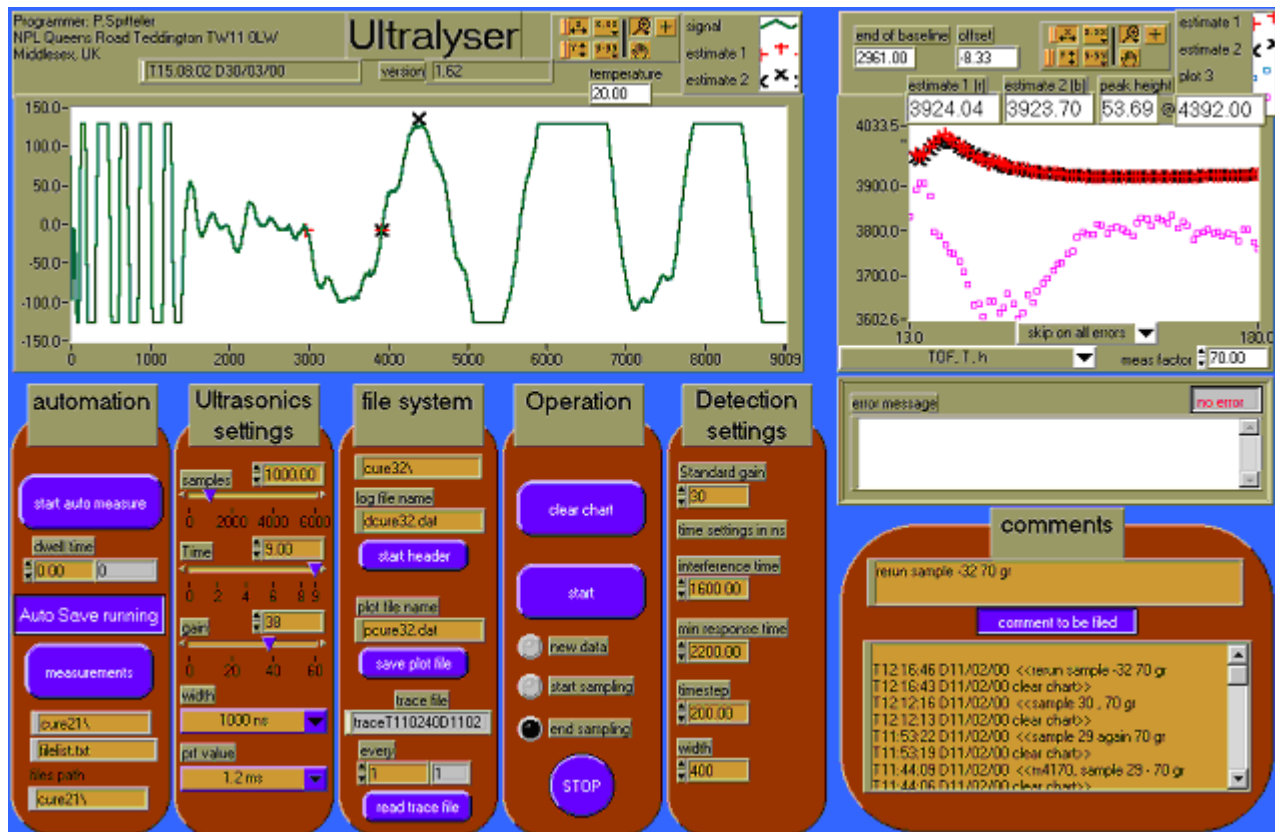


Figure 3. The user interface of the Ultralyser software. The signal as received is shown on the left and the resulting time of flight and signal power as it changes with time on the top right.

Industrial Trials

Industrial trials have been carried out with seven companies in the UK. Figure 2 shows a cure curve displaying four characteristic zones; **a**, **b**, **c** and **d**. In this case, a powder material was loaded and compressed. Ultrasound pulses of the applied frequency do not pass through air and are effectively conducted only when the powder has melted (zone **a**). The time of flight then increases with increasing temperature as can be seen in zone **b**. The cure reaction subsequently causes a drop in time of flight of the material in zone **c**. When the sample is left in the mould too long the time of flight increases again and the signal amplitude reduces, zone **d**. This correlates with a deterioration of product properties when the material is over-cured. This example demonstrates how the ultrasonic monitoring technology can determine optimum cure times.

The technology has potential not only for product development but also for quality control and perhaps most importantly for cycle time reduction leading to increased efficiency. A wide range of reactive materials has been studied using this technique including polyesters, amino moulding powders and highly filled materials such as the slate roof tile illustrated in figure 4.

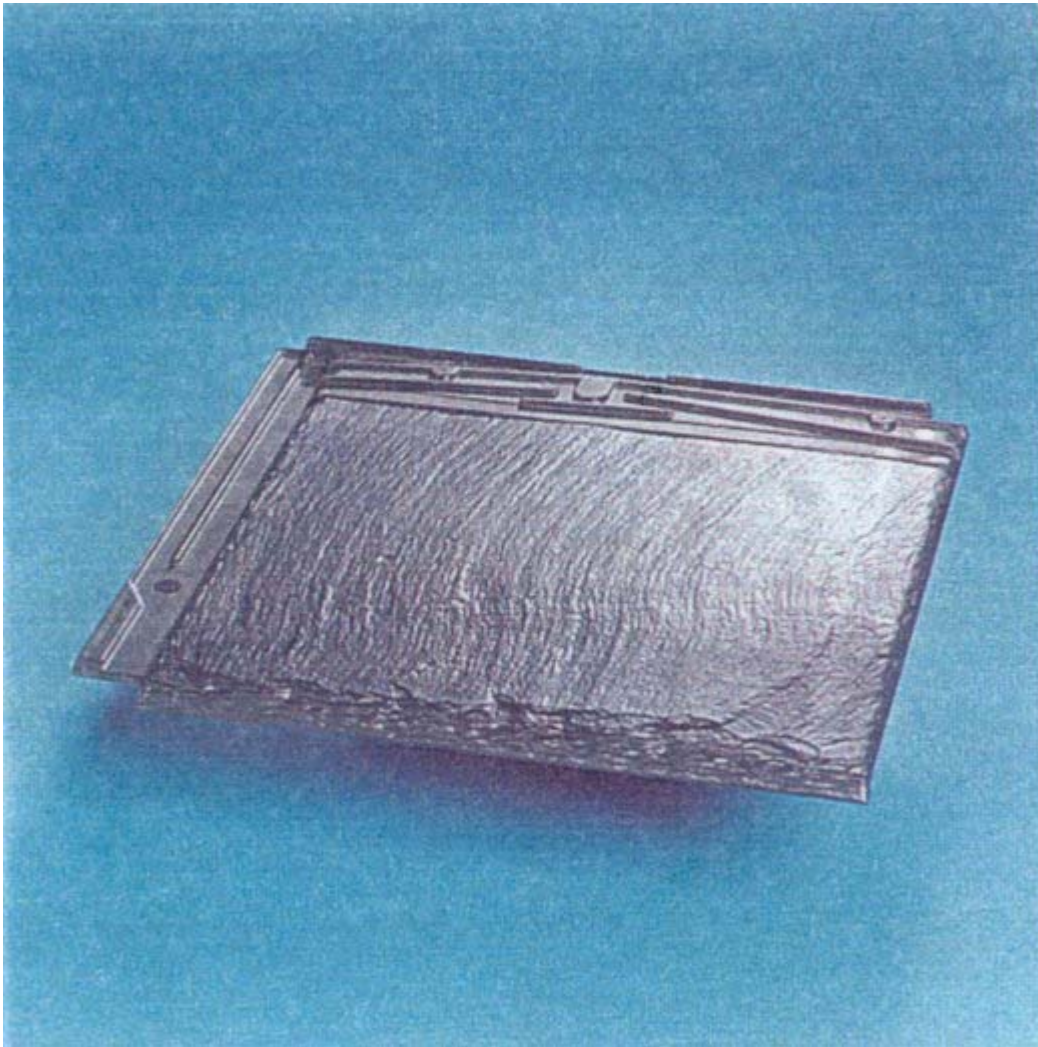


Figure 4. This moulded roof tile is an example of the type of product that has been investigated.

Conclusions

Ultrasonic monitoring technology can assist companies to increase efficiency by eliminating unnecessary, or even detrimental, residence time in the mould and by enhancing quality assurance.

A prototype of this instrumentation is now available for carrying out industrial trials as part of a new "studio" project.

Further Information

NPL plans to carry out a new project aimed at demonstrating the commercial benefits of monitoring the production of products or materials used in the construction industry. DTI funding is available through the "materials measurement studio project" so that financial support from participating companies of only 10% of the total cost is required.

Further information is also available from NPL on the *Polymer Processing Measurement Club*, and the following research projects which address measurement issues in the polymer processing industry:

- Heat Transfer Relating to Polymer Processing
- Viscoelastic Measurement Techniques for Polymer Melts
- Improved PVT Measurements for Polymer processing
- Automatic Optimisation of the Injection Moulding Process
- Industrial Assessment of Techniques to Measure Rheological Properties of Polymers During Processing.

Acknowledgements

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References

1. P A J Gibbs BPF Congress, NEC Birmingham, November 1994.
2. J Kirchhoff and D Mewes Paper 152, American Chemical Society Meeting, Orlando, Florida, September 21-24 1999.

For further information contact:

Mr Chris Brown
Centre for Materials Measurement and Technology
Tel: 020 8943 6769
Fax: 020 8943 6098
Email: Chris.Brown@npl.co.uk

Experts

[Link to Expert](#)

National Physical Laboratory
Queens Road
Teddington
Middlesex
United Kingdom
TW11 0LW

Tel: 020 8977 3222
Fax: 020 8943 6458
Email: materials@npl.co.uk

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