

Ultrasonic Cure Monitoring of Two Industrial Processes

Summary

The use of ultrasound to monitor cure has now reached an advanced stage so that a system has been developed that can operate within industrial process conditions. This has been made possible with the availability of high temperature ultrasonic transducers, a unique NPL designed ultrasound operating system and more powerful computers. Thus an ultrasound signal can be transmitted through thick sections of highly attenuative material at process temperatures unobtrusively (without showing a witness mark on the product). To demonstrate the effectiveness of this cure monitoring and quality control technique two field trials have been carried out and are summarised in this measurement note. Good correlations are shown to relate the stiffness, as measured by the time of flight of the ultrasound signal, to the salient material properties. Benefits arising from the application of ultrasound monitoring would be a shortening of cycle times, an improvement in quality and a reduction of scrap. The more rapid achievement of acceptable and serviceable products on start up or changing grades would be an additional bonus leading to greater confidence in the subsequent enhanced specification.

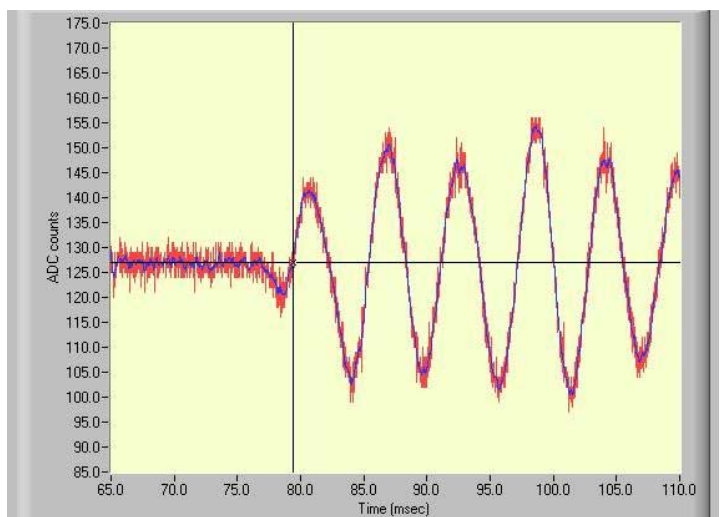


Figure 1. The ultrasonic pulse as received through a billet during bearing manufacture using a 0.5 MHz transducer at elevated temperature.

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January 2003

Ultrasound has been well used by industry in non-destructive testing for many years. The need for process monitoring has been well appreciated by converters for sometime so the marrying together of ultrasound technology to processing with production improvements is very attractive. The benefits for process monitoring would be an increase in productivity, efficiency and a reduction in costs. Other benefits arising would be an improvement in product quality and a reduction in scrap. The specific aim would be the shortening of cycle times and the greater confidence in an acceptable and serviceable product to a higher specification.

Many processes require temperatures up to 200 °C for perhaps hours for full conversion to a satisfactory product. Recent advances in ultrasound technology have enabled robust transducers to be manufactured that can run continuously at high temperature. Thus it is now possible to transmit ultrasound through steel moulds without disturbing or affecting the product in situ so that the degree of conversion can be monitored with suitable equipment [1].

The task

The task then is to apply the power of modern computers (and software such as Lab View), ultrasound signal generators and controllers operating high specification transducers with material science expertise to achieve a demonstrable and effective process monitoring system.

The ultrasonic measurement

During the conversion process, for instance in the curing or cross-linking of a reactive system, the

“stiffness” of the material increases with the degree of cure. This can be followed by recording the ultrasound pulse and accurately measuring the time of flight of the ultrasound signal through the product. The change in time of flight reflects the state of cure of the material under constant temperature where the thickness and density variations are negligible [2]. The time of flight can also be used to calculate the ultrasound velocity (if the thickness is accurately known) in the sample.

The amplitude of the ultrasound pulse detected depends on the dissipative (damping) properties of the material (the type and loading of filler will also affect the peak height by scattering the ultrasound). It is also dependent on the power and operating frequency of the transducers and again on the thickness of the product.

NPL has developed a software package (EDS logger) that automatically monitors and analysis the ultrasonic pulses and displays changes in time of flight and the peak height (a measure of the damping and is related to $\tan \delta$) with process time. The temperature (up to eight positions) within the system is also recorded alongside the instrument settings. A separate analysis package (manual EDS analysis) allows the files created to be read and an accurate determination of the ultrasonic parameters to be determined manually in addition to the file constructed while monitoring.

The Figure 2 below is an example of the ultrasound logger showing the instrument settings, the last ultrasound trace and the change in the measured time of flight and peak height since the programme was started.

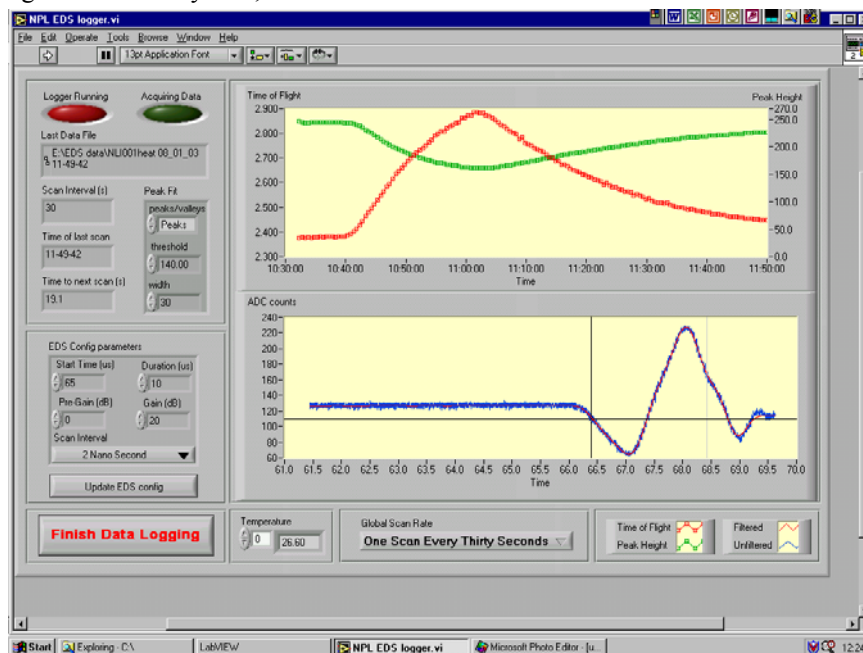


Figure 2. An example of a display screen for a sample that is heated and cooled.

The industrial trials

Two trials are reported here on a phenolic marine bearing material and a cross-linked polyolefin, which is subsequently foamed and sold as a sheet often used for packaging applications. A typical trace for an ultrasound pulse is shown in Figure 2 during the curing of a phenolic bearing in an air-heated oven at 140 °C.

Tests were also carried out to investigate the strength of the received signal (as well as the maximum measurable thickness of the billet wrapped around the mandrel) and to assess the effect of thickness on the time of flight. This showed that for a bearing material measured radially, a thickness up to 100 mm may be satisfactorily tested at room temperature.

The effect of temperature on the ultrasound signal is illustrated in Figure 3 on cooling a billet after curing.

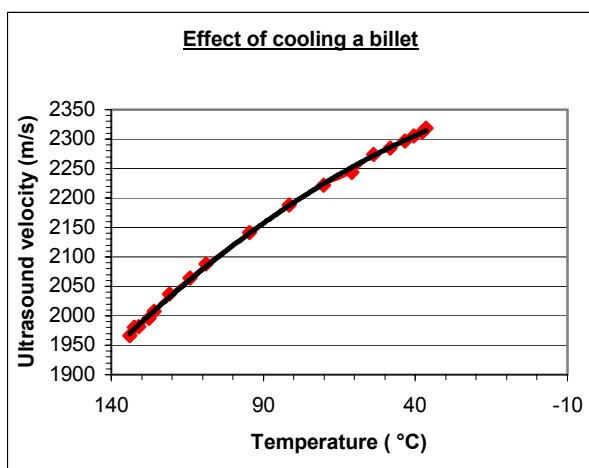


Figure 3. Change in the ultrasound velocity with temperature.

A study was undertaken to assess the effect of post-curing the cured and machined bearing at various times and temperatures in an effort to understand the changes that take place and correlate the results with a compressive modulus test. Tests completed in the radial and axial directions are presented in Figure 4 for post-curing at 130 °C and 150 °C. The correlation found between the compressive modulus and the ultrasound velocity was good (the correlation coefficient = 0.98), so offering the possibility that an ultrasound test could replace the traditional method and reduce product unit costs.

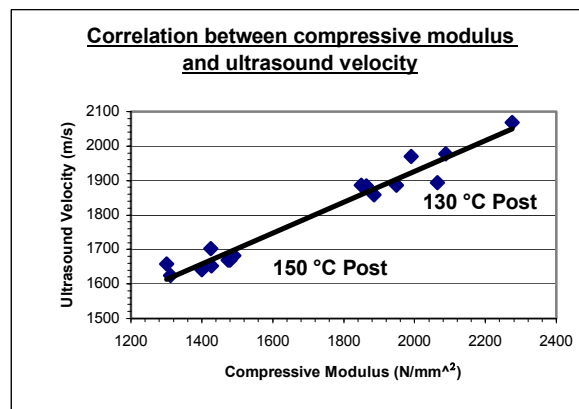


Figure 4. Shows a good correlation between the two stiffness tests.

The second trial on polyolefin resins

The polyolefin resin is extruded into large sheets, which are then chemically cross-linked with a peroxide or may be cross-linked off-line by treatment with high-energy radiation. Subsequent nitrogen impregnation at high temperature and pressure, followed at a later stage by rapid decompression and expansion at high temperature produces a low-density foamed sheet used, for example, in the packaging industry. The presence of cross-links prevents the collapse of the cell walls so the degree of cross-linking is crucial to the final product.

Ultrasound measurement of irradiated extruded slabs was used to assess the effectiveness of the irradiation treatment with a view to potentially replacing the existing quality control test. The results of the comparison between the two tests are shown in Figure 5 where the times of flight at room temperature and at 150 °C are correlated with the irradiation dose. A good correlation is found especially at room temperature though more data are needed before a firm recommendation can be made. If confirmed the ultrasound test method could then replace the traditional quality control test.

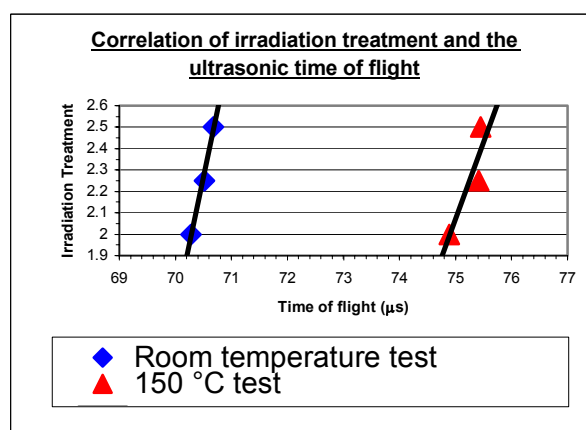


Figure 5. A graph showing the correlation of the measured time of flight and the radiation treatment.

Conclusions

Ultrasonic cure monitoring has been shown to be able to measure the change in state (stiffness) of a component in manufacture and can potentially replace or compliment the existing techniques used in quality control departments to reduce costs and ensure the quality of the final product.

For further information

More information on polymer processing and this ongoing topic can be found by joining the *Cure Monitoring Industrial Advisory Group* and/or by collaborating in research projects that address measurement issues in the polymer processing industry.

Acknowledgements

This measurement note was prepared as part of the “Measurements for the Processability of Materials Studio project: Monitoring the Processing of Construction Industry Materials” financed by the National Measurement System of the DTI and supported by companies in the Polymer Processing Measurement Club.

References

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

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