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MEASUREMENT OF ULTRASONIC WAVE ABSORPTION AND PHASE VELOCITY IN THIN POLYMER SAMPLES: RECENT DEVELOPMENTS.

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ABSTRACT

This paper describes several procedures to measure the ultrasonic absorption and phase velocity in thin samples of polymer using a wide bandwidth, through transmission immersion technique. Measurements are described using traditional pulse windowing methods, where the Fast Fourier Transform (FFT) algorithm is utilised to obtain absorption and phase velocity spectra in thick samples. These methods are necessarily limited to acoustically thick samples so that individual pulses in the sample can be resolved. Thus, this paper also considers much thinner samples, and a novel technique is presented which permits characterisation of samples in situations where pulse overlapping occurs. Measurements on samples which can be as thin as 25 μm are presented, and these are compared to measurements made on thicker samples.

INTRODUCTION

The use of ultrasound for the study of engineering materials is well established and, in the case of polymer materials, is a useful tool for the characterisation of properties such as mechanical modulus, adhesive cure and other structural changes in the material. In many cases, the polymer material forms part of a composite structure which itself may need to be analyzed using ultrasound, for example in the case of adhesive bonds and carbon fibre composites. In order to understand and model the interaction of ultrasound with composite materials it is necessary to characterise the propagation characteristics of the polymer itself. Wave propagation in polymer materials can be represented by a complex wave number which incorporates the phase velocity and absorption coefficient of the sample. The measured absorption and phase velocity can then be related to mechanical properties of the polymer(1) or used as input to a model describing wave propagation in a composite material(2). Historically, a number of techniques have been used to ascertain the ultrasonic behaviour of polymers including spot frequency and gated tone burst methods. Whilst often very precise, these techniques only give information over a very limited bandwidth or at a few specific frequencies(3). It is often required to characterise the material over a much wider frequency range, for example in cases when the polymer forms part of a structure where wide bandwidth ultrasonic methods such as flaw detection are employed. For these reasons, pulsed ultrasound techniques have been developed to characterise the polymer, and the Fast Fourier Transform (FFT) is utilised to obtain the phase velocity and absorption coefficient as a function of frequency. A number of pulse windowing methods are described which can be used to characterise compression and shear wave propagation in a range of materials. The limitations of the pulsed based technique, when analysing thin samples, are described and a novel method for the characterisation of acoustically thin samples is presented. Results are given for samples of polystyrene (PS), polyethylene terephthalate (PET), polytetrafluoroethylene (PTFE) and a high strength adhesive.

THEORY AND MEASUREMENT METHODS

Figure 1a shows a slab sample of polymer material immersed in a water coupling medium and set at normal incidence between coaxially aligned ultrasonic transmitter and receiver transducers. The ideal unit impulse response of a typical sample in the time domain is given in figure 1b.

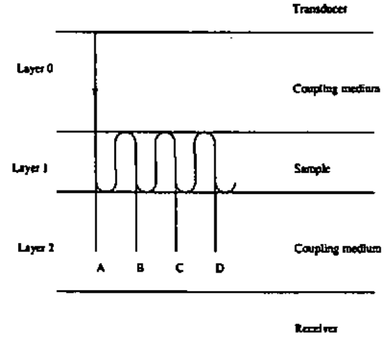

Figure 1a Slab sample of polymer arranged between transducers,

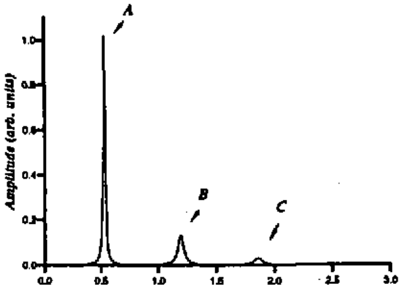

Figure 1b Ideal time domain response of polymer sample at normal incidence.

The transmitter is pulsed and a series of compression wave reverberations are excited in the sample. Referring to figure 1b, the effects of absorption in the sample on the received pulses are quite clear. Each of the ultrasonic pulses received from the sample can be described in terms of the transmission and reflection coefficients at the water-sample-water interfaces, the time delays through the sample, and the absorption in the polymer. Hence, referring to figures 1a and 1b the sample response may be written,

$$\begin{aligned} S(\omega) &= A + B + C + \dots \\ S(\omega) &= t_{01}t_{12}e^{-j\omega\frac{d}{c(\omega)}}e^{-\alpha(\omega)d} + t_{01}r_{12}r_{10}t_{12}e^{-j\omega\frac{3d}{c(\omega)}}e^{-\alpha(\omega)3d} + t_{12}r_{12}^{2}r_{10}^{2}t_{12}e^{-j\omega\frac{5d}{c(\omega)}}e^{-\alpha(\omega)5d} + \dots \end{aligned} \tag{1}$$

where r_{xy}, t_{xy} are the reflection and transmission coefficients (which can be assumed to be frequency independent), $c(\omega)$ is the phase velocity, d is the sample thickness, and $\alpha(\omega)$ is the absorption coefficient. In a real measurement system the ideal pulses shown in figure 1b will be convolved with the system response of the electronics and transducers, including effects such as the bandpass characteristics of the transducers and diffraction phenomena.

Thick samples: - pulse windowing techniques

Pulse windowing methods rely on windowing individual pulses excited in a sample in the time domain and comparing their magnitude and phase in the frequency domain to obtain the absorption coefficient and phase velocity. The techniques of Challis et al(4) require windowing the first two compression wave pulses excited in the sample, (*A* and *B* in figures 1a and 1b) and calculating the FFT for each pulse. This will give the first two terms of equation 1 which are also convolved with the response of the measurement system. The absorption relationship is obtained by taking the modulus of the first two terms, correcting for the reflection coefficients, and then dividing one by the other thus deconvolving any system effects as shown in equation 2.

$$\alpha(\omega) = \frac{1}{2d} \ln \left(\frac{|A(\omega)|}{|B(\omega)|} r_{10} r_{12} \right) \tag{2}$$

The phase velocity is obtained by calculating the phase response of the first two terms and then subtracting the phase of one from the other, as shown in equation 3,

$$c(\omega) = \frac{2d}{\left(\frac{\Phi(B(\omega)) - \Phi(A(\omega))}{\omega} \right) + (t_B - t_A)} \tag{3}$$

where t_A and t_B are time shifts that are applied to the pulses when calculating the phase in order to minimise phase wrapping in the FFT calculation.

In some situations it may not be possible to produce an adequate compression wave reverberation sequence in the sample (due to high losses or for very thick samples). In these situations the first pulse through the sample is compared to a reference pulse propagated in the water filled test cell without the sample in place, and slightly modified versions of equations 2 and 3 are employed to calculate the absorption and phase velocity$^{(5)}$. This measurement technique is used in the analysis of the thick samples presented in this paper and for shear wave measurements where the sample is set past the critical angle for compression wave excitation in the sample (typically 50° to 70° incidence). In this situation, only pure shear modes are excited in the sample and the first shear wave pulse received from the sample is compared to the reference pulse.

Thin samples: - parameter fitting technique

In recent work the authors have considered situations where the sample thickness approaches the wavelength of the highest frequency propagated in the sample. In this case, reverberations in the thin sample cause pulse overlapping in the time domain, and the calculations described above cannot be employed. Thus it is necessary to consider the complete sample response, rather than individual reverberations. Equation 1 may be modified to give the complete sample response in terms of a system transfer function as shown in equation 4.

$$S(\omega) = \frac{t_{01} t_{12} e^{-j\omega \frac{d}{c(\omega)}} e^{-\alpha(\omega) d}}{1 - r_{12} r_{10} e^{-j\omega \frac{2d}{c(\omega)}} e^{-\alpha(\omega) 2d}} \tag{4}$$

In this paper the theoretical sample response given by equation 4 is fitted in a least squared error sense to the measured sample response as a function of frequency. The measured sample response is obtained by taking the FFT of the time domain sample response and dividing this by the FFT of the reference response without the sample in place. This has the effect of deconvolving the system response thus leaving only that of the sample. Given the acoustic impedance of the immersion medium and the sample thickness and density, it is possible to calculate the mean phase velocity (and in some cases the frequency dependent phase velocity) and an approximation of the frequency dependent absorption coefficient in the sample. It is necessary to approximate the absorption in the polymer sample to limit the number of terms required in the fitting process. In this work, two approximations have been used: a simple straight line approximation assuming a linearly increasing absorption with frequency, and a more detailed approximation based on an anelastic model of wave propagation(6). For many polymer materials these approximations have been shown to adequately describe the observed absorption and velocity dispersion in a number of materials(7). Figure 2 shows the error surface of the fitting process for a typical polymer sample which is 50 μm thick, assuming a mean phase velocity and a linear frequency dependence for the absorption coefficient.

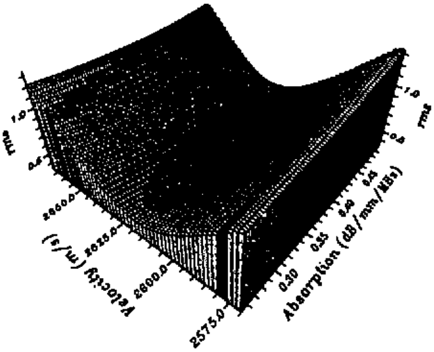

Figure 2 Error surface for parameter fitting technique.

EXPERIMENTAL APPARATUS

Figure 3 shows the experimental apparatus consisting of an immersion test cell, coaxially aligned transducers, sample mounting with a micrometer vertical axis adjustment, and a computer controlled, high precision goniometer stage. Horizontal and vertical axis alignment is provided to a resolution of 0.01° and 0.05° respectively with corresponding accuracies of 0.04° and 0.10° thus allowing the sample to precisely aligned to any angle of incidence required. The test cell can be fitted with a wide range of transducers including wide bandwidth acoustically thick transducers and conventional resonant transducers. Data is digitised at a 400 MHz sampling rate using a 9450 LeCroy digital oscilloscope which is connected via a GPIB data bus to a PC, and on to a local area network where the data is analyzed. When required, coherent averaging can be employed to give improvements of up to 30 dB in signal to noise ratio. A typical waveform propagated across the test cell using the wide bandwidth transducers is shown in figure 4.

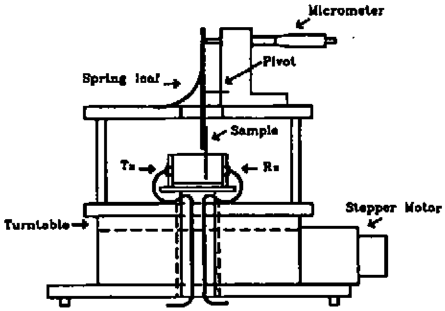

Figure 3 Experimental apparatus: test cell, sample mount and goniometer stage.

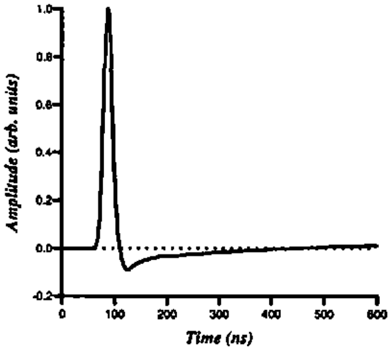

Figure 4 Typical wide bandwidth waveform propagated across test cell.

EXPERIMENTS AND RESULTS

Thick samples

Samples in the range 0.5 mm to 1.5 mm thickness were considered and both compression and shear wave measurements were made over a frequency range of 3 MHz to 40 MHz. In each case only the first pulse through the sample was considered. Figures 5a and 5b show the measured time domain compression and shear wave response for a 1.0 mm sample of polystyrene (PS) and polyethylene terephthalate (PET).

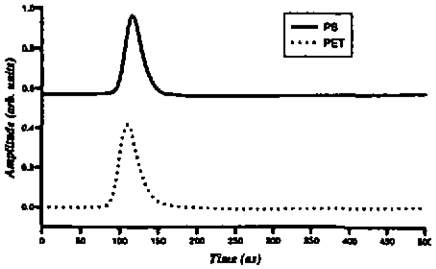

Figure 5a Measured time domain compression wave response for polystyrene (PS) and polyethylene terephthalate (PET).

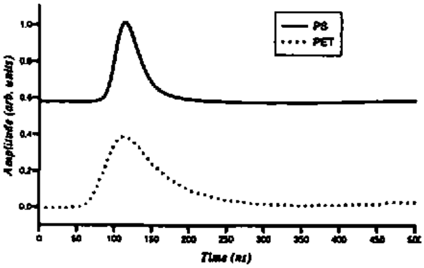

Figure 5b Measured time domain shear wave response for polystyrene (PS) and polyethylene terephthalate (PET).

Figures 5c and 5d show the calculated compression and shear absorption coefficient for the samples over a bandwidth of 3 MHz to 40 MHz. The results for the PET compare well to those made by Hartmann and Jarzynski[(3)] for samples of polyethylene.

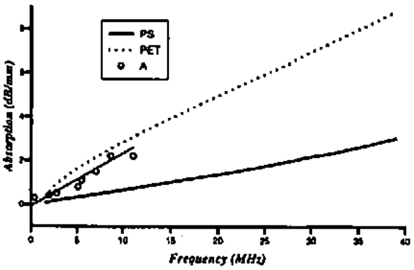

Figure 5c Measured compression wave absorption in polystyrene (PS) and polyethylene terephthalate (PET), and experimental data (A) measured by Hartmann and Jarzynski[(3)].

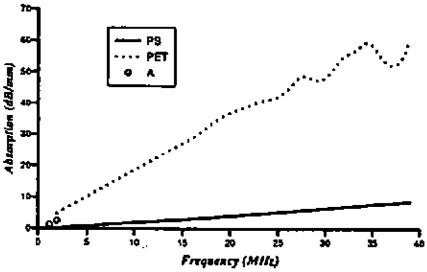

Figure 5d Measured shear absorption in polystyrene (PS) and polyethylene terephthalate (PET), and experimental data (A) measured by Hartmann and Jarzynski[(3)].

Figure 6a shows the measured time domain compression wave response for a 1.5 mm sample of Ciba Geigy AV119 adhesive.

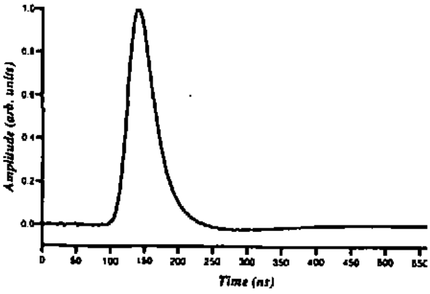

Figure 6a Measured time domain response for 1.5 mm sample of Ciba Geigy AV119 adhesive.

Figures 6b and 6c show the calculated compression wave absorption coefficient and phase velocity for the adhesive. It can be seen that the absorption and velocity dispersion is considerable as the frequency increases.

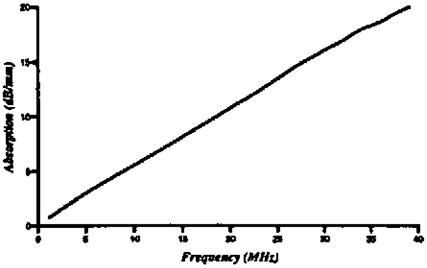

Figure 6b Compression wave absorption coefficient for AV119 adhesive.

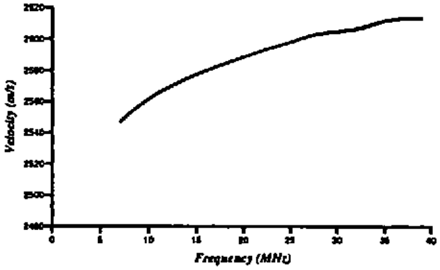

Figure 6c Measured compression wave phase velocity for AV119 adhesive.

Measurements of the adhesive properties have been used extensively in a multilayered wave propagation model for the study of adhesive bonds allowing very accurate simulation of the response of the joint to be made$^{(8)}$. This work has been important in the development of advanced signal processing techniques for the NDT of adhered automotive structures.

Thin samples

Measurements were made on thin polymer samples with thickness ranges of 25 µm to 250 µm. Using the parameter fitting technique and assuming a linear relationship both the compression absorption and phase velocity were calculated for thin samples of PTFE. Figure 7a and 7b show the fitted absorption and phase velocity spectra, also shown are the measured compression absorption and phase velocity for a 500 µm acoustically thick sample measured using the pulse windowing method described in this paper. It can be seen that the approximate absorption coefficient and phase velocity calculated for the thin samples compares well with that measured in the thick samples.

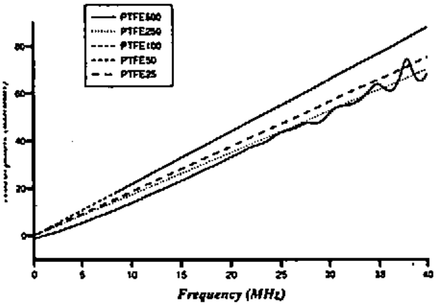

Figure 7a Fitted absorption coefficient for 25, 50, 100, and 250 μm samples. Solid line shows actual absorption in a 500 μm thick sample.

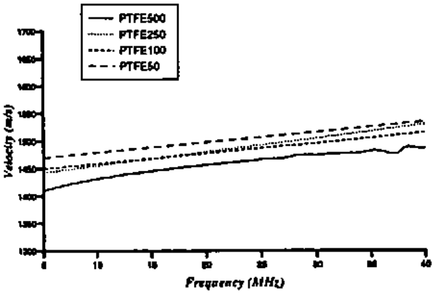

Figure 7b Fitted phase velocity for 50,100, and 250 μm samples. Solid line shows actual phase velocity in a 500 μm sample.

To illustrate the good degree of fit observed in the figures, equation 4 was applied in inverse form to the measured data to remove the sample response. Figure 8a shows the measured sample response for the 50 μm sample of PTFE and the effects of pulse overlap can clearly be seen. Figure 8b shows the data after correction for the sample response showing recovery of the original system response, comparing well with the actual response of figure 4.

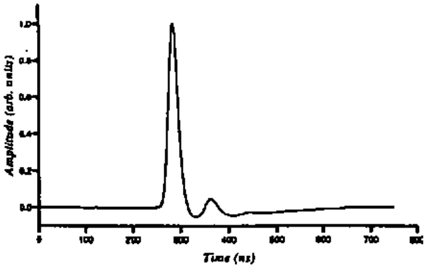

Figure 8a Measured time domain response for a 50 μm sample of PTFE.

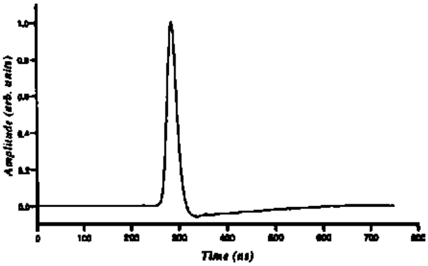

Figure 8b Data of figure 8a corrected for sample response.

CONCLUSIONS

Techniques for the measurement of compression and shear wave absorption and phase velocity have been presented which permit the characterisation of polymers over a wide bandwidth. The importance of such measurement techniques for NDE and NDT of polymers and polymer composite materials has been discussed. In a new development a technique has been presented which permits the characterisation of polymer samples which can be very thin, such that the sample thickness is often less than the wavelength of the interrogating ultrasound. This work has some considerable importance for the characterisation of thin polymer coatings, paint layers and thin adhesive bondlines.

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