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A WIDE BANDWIDTH ULTRASONIC ABSORPTION SPECTROMETER FOR LIQUID MATERIALS

R.E. Challis and A.K. Holmes

University of Keele

Abstract

A novel spectrometer instrument has been developed for measuring ultrasonic near plane wave absorption in small volume samples of liquids over bandwidths up to 60 MHz. In principle the bandwidth could be increased to 100 MHz. The instrument is controlled by computer and on-line digital signal processing is used to correct for transducer insertion, radiation coupling, and electronic circuit responses. The data acquisition time is short and the instrument can be used to estimate wide bandwidth absorption spectra at intervals of 100 ms. The instrument can therefore be used to study the dynamics of systems undergoing chemical reaction. Experiments have been performed which show excellent agreement between absorption measurements made with the instrument and with traditional (slower) techniques.

Introduction

Measurements of the propagation velocity and absorption of ultrasonic waves as functions of frequency have long been used to elucidate the dynamics of chemical equilibria which can be driven by pressure gradients. A number of wave modes have been employed, the principal ones being plane waves and shear waves. The majority of existing methods are based on pulse echo, pulse transmission or cavity resonance methods and most are narrow band in nature, relying either on gated sinewave bursts as test signals, or on the fairly narrow band radiation from plate piezoelectric transducers. Cavity methods are inherently narrow band due to their fixed, or at best, slowly adjustable cavity dimensions. In order to cover an adequately wide range of frequencies to characterise an absorption process it is frequently required to change transducers and sometimes the test vessel as well. This suffers the joint disadvantages that i) transducer changes may lead to uncertainties of measurement that are difficult to control and ii) the time required to cover adequate frequency bandwidths is of the order of minutes, or even hours. In this note we present a new ultrasonic spectrometer instrument that overcomes these problems. It is based on the propagation of short (10 ns) wide bandwidth pulses between two circular transducers aligned coaxially on either side of a low volume (30 ml) fixed geometry test cell. The transducers are acoustically thick (1) and operate in a fast transient mode that offers, at low cost, bandwidths that are much greater than those available from conventional resonant devices. Signal transmission and data acquisition from the cell is controlled by computer and on line digital signal processing procedures are employed to partially correct for acoustic field diffraction, transducer insertion, and the dynamic responses of the transmitter and receiver electronic circuits. The bandwidth available for measurement is at present 60 MHz, although this could be increased by the use of a faster digitising system and a receiver amplifier of wider bandwidth. Where necessary the variance of absorption measurements can be reduced by improvements to signal to noise ratio in the raw transducer signals by coherent averaging. The data acquisition time for each absorption spectrum depends upon the number of averages required to control system noise and is typically 100 ms. This figure could be reduced if custom built computer hardware were used, although trade offs exist between the system repetition rate, signal to noise ratio, and available bandwidth. Rapid operation gives the advantage that the system can be used for rapidly repeated absorption measurements. It therefore has application to non stationary reacting systems as well as for more conventional absorption spectrometry.

The Test Cell

The cell is shown in fig 1. It is of 36 mm cubic shape with 10 mm dia x 5 mm thick piezoelectric ceramic transducers (PZT) fixed with adhesive into precisely machined bores in the end plates of the cell. One device acts as transmitter, the other as receiver. The bores are in accurate coaxial allignment and tight fitting locating dowels ensure correct allignment of the end plates during assembly. The inner active faces of the transducers are lapped down to form continuous surfaces with the cell end plates (+ -10 μm). The active faces of the two transducers are maintained parallel to within 0.05 degrees since a non parallelism error would cause a serious reduction in bandwidth. The transducer and cell dimensions have been set to values that cause the duration of the impulse response of the radiation coupling process between the two transducers in typical test media to be less than the difference in propagation times between the transducers by the most direct route (face to face), and other spurious routes such as propagation along the walls of the cell or by reflection off the side walls. The cell is housed in a temperature controlled enclosure within which provision is made for measuring temperature and pH in the test medium itself. Other sensors could be substituted for these where required.

The transmitter transducer is excited by a triggerable bipolar transistor avalanche generator that delivers short (10 ns) pulse transients of 100 v amplitude. The receiver transducer is terminated by a common base header amplifier followed by a voltage amplifier based on the SL 560c integrated circuit (Plessey Ltd). Amplified received signals are digitised using a proprietory radio frequency transient recorder (Le Croy TR 8828) which has a maximum sampling rate of 200 MHz and, in the current configuration, a maximum data length of 32k samples.

The recorder communicates via an IEEE 488 bus to a host computer (LSI 11-73, Digital Equipment corp.). The machine level driver routines that control data acquisition can carry out coherent averaging to reduce incoherent noise in the signal. Further computations are carried out to reduce errors due to transient radiation coupling between the transducers and due to the insertion properties of the transducers themselves. These are discussed in the next section.

The Acoustic Signal Pathway

When the transmission transducer is pulsed electrically, near plane wave acoustic disturbances are generated at the front and back faces of the device (1); their time domain shape is that of the exciting voltage waveform with some distortion due to piezoelectric regeneration (2). The disturbances propagate away from the electrode regions of the transducers as four near plane bounded waves: wave (a), fig. 2, enters the transducer backing medium and is lost and wave (b), generated at the front face of the device, propagates into the test medium. Waves (c) and (d) enter the transducer and propagate to and fro in the device. At integer multiples of the propagation time across the transducer element, components (c) and (d) are partially transmitted into the backing and load media. The result in the load medium, the test fluid, is a series of short pulses separated by the transmitter device internal propagation time. The pulses become successively more distorted due to piezoelectric regeneration and eventually develop into the near sinusoidal half wave resonance of the transducer. After propagation through the test medium the pulses enter the receiver and reverberate within it in a similar way. The electrical signal at the receiver terminals consists of a series of pulses representing these reverberations, which eventually become more complicated due to reverberations in the test medium and in the structure of the cell itself. In this instrument we gate out for processing the segment of signal at the receiver which begins at the first arrival of the transmitter front face wave and lasts until the arrival of the next pulse at the receiver, that which was originally formed at the back face of the transmitter. This gated signal segment is formed by the insertion effects of both transducers up to one device propagation time, transient radiation coupling between the transducers, and the acoustic absorption in the test medium which we require to determine. Digital signal processing procedures are applied to this signal segment to correct for the following effects: i) transducer insertion, based on known models of transducer behaviour (2). ii) transient radiation coupling based on a plane wave - edgewave model (3). iii) the transmitted pulse shape. iv) the receiver amplifier transfer function. The corrected raw data then represents the frequency domain response of the test medium to near planewave acoustic pulses propagated through it. Further computations then yield the absorption coefficient as a function of frequency ($\alpha(f)$), absorption per wavelength versus frequency ($\alpha\lambda$), and absorption coefficient divided by frequency squared (α/f^2).

Preliminary results

To test the performance of the spectrometer wide bandwidth absorption measurements were made on 0.1 molar solutions of Hexadecylpyridinium Chloride (HDPC) containing 0.025, 0.05, 0.1, 0.15 and 0.2 molar pentan - 1 - ol. This alcohol-surfactant combination was chosen because its absorption behaviour is well understood and has been documented for multi-spot frequency measurements by Gormally et al (4). Fig 3 shows the function α/f^2 for the 3 to 50 MHz range measured with the spectrometer, with the results of Gormally et al plotted as crosses. It is clear that excellent agreement has been obtained.

Concluding Remarks

This paper has presented a novel instrument that enables ultrasonic absorption measurements to be made in liquids over wide bandwidths using a short pulse transmission technique incorporating digital signal processing. It has the advantage that data recording time is several orders of magnitude less than conventional multiple spot frequency techniques. The prototype device can capture data for a wide band absorption spectrum in about 100 ms. Applications of the instrument will therefore include measures of absorption in chemical systems which are reacting, and will provide a means to study reaction dynamics. A relatively simple extension to the signal processing arrangements will enable acoustic wave velocity dispersion versus frequency measurements to be included in the operation of the instrument.

References

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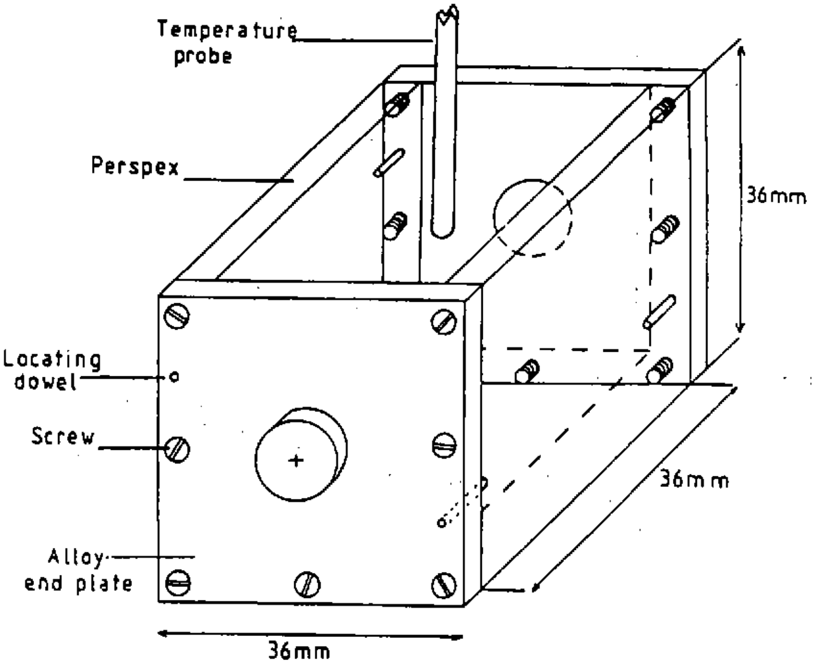

Fig 1 The test cell.

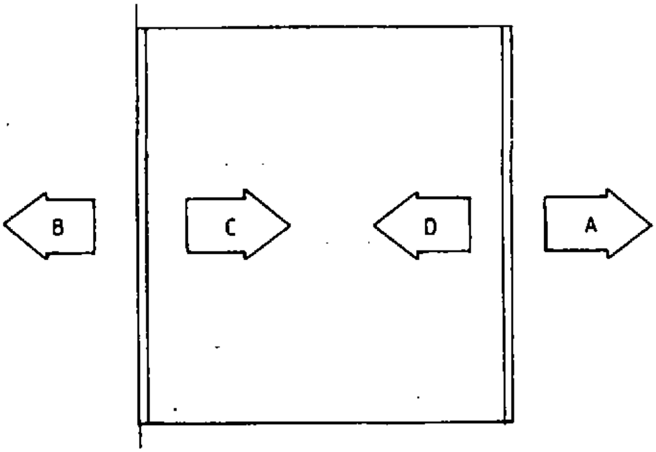

Fig 2 Sketch of piezoelectric transducer showing transient acoustic waves generated at its front and back faces. Wave (a) enters the backing medium and is lost, Wave (b) enters the test liquid at the instant of transducer excitation, waves (c) and (d) reverberate within the transducer and at each reverberation are partially transmitted into the test liquid. The response of the liquid to wave (b) only is used to determine absorption in the liquid.

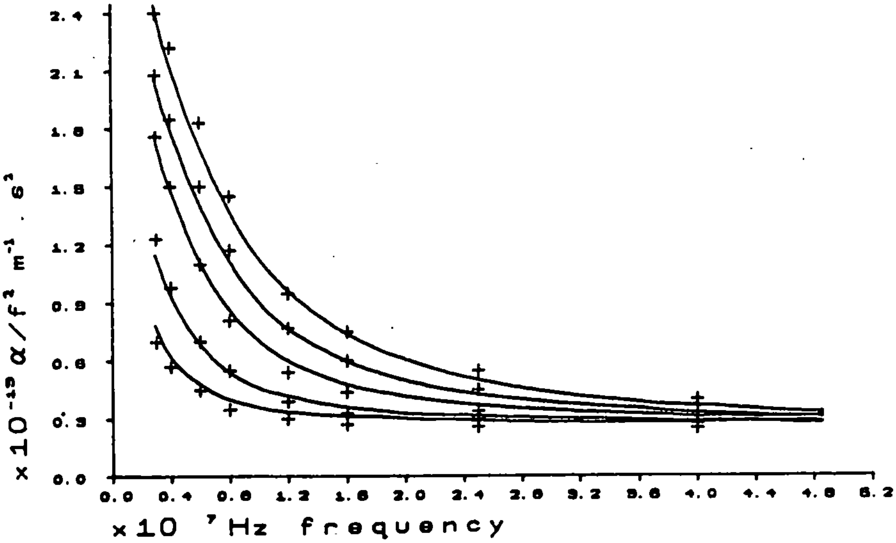

Fig 3 Preliminary results in 0.1 molar solutions of hexadecylpyridinium chloride (HDPC) containing 0.025, 0.05, 0.1, 0.15 and 0.2 molar pentan-1-ol. The curves show continuous α/f^2 data versus frequency, determined with the spectrometer, and the marked points are the spot frequency measurements obtained by Gormally et al (4).