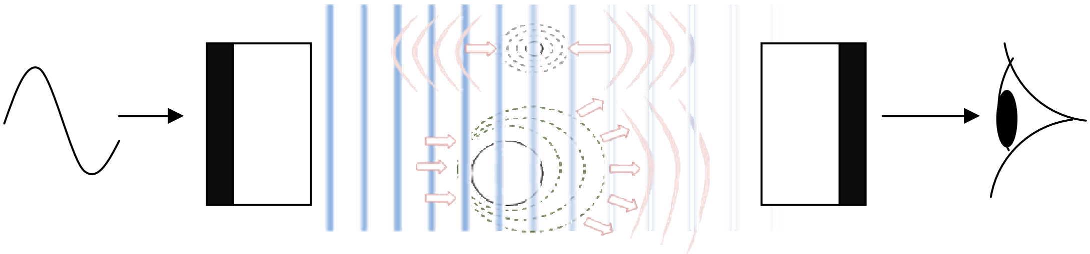
Process Ultrasound Spectroscopy for In-situ Characterisation

Introduction

Ultrasonic Spectroscopy (USS) is the characterisation of the ultrasonic response of a material to the propagation of a low energy ultrasonic wave. Ultrasonic Spectroscopy can measure the absolute frequency dependant velocity and attenuation of liquids and soft solids. Ultrasonic waves propagate through a medium as a mechanical wave Due to the nature of the Ultrasound wave the method has a number of inherent and useful features:

- Non-destructive, Non-Intrusive and in-situ
- Can be used on opaque samples allowing use at high concentration and strong colours
- Can be used on both liquids and soft solids
- Simple sample handling with no requirement for manual intervention
- Able to operate over a wide range of length scales from nanometers to millimeters

The interaction of the wave with the material is a function of the material structure including hydrodynamic and the thermo-elastic properties. Therefore Ultrasonic Spectroscopy data can be used to characterise any process which modifies the these parameters including the process kinetics, solids concentration, rheological behaviour and changes in particle size or material structure.

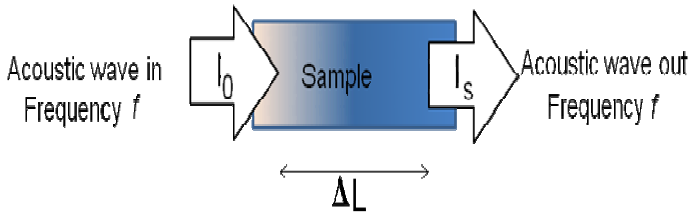


Ultrasound interacts with microstructure and mechanical processes modify wave amplitude and phase

Unlike optical methods, Ultrasound is not limited by the opacity of the sample and is hence able to work at high concentration or in coloured liquids such as pigments. The technique is sensitive to changes in particle size over a wide range of mean particle sizes approximately 10nm to 1mm

USS Method

The method measures the change in the amplitude and phase of the ultrasound signal over a known path length and provide volume averaged information on the material.



$$\alpha = \frac{1}{\Delta L} \cdot Log\left(\frac{I_o}{I_s}\right)$$

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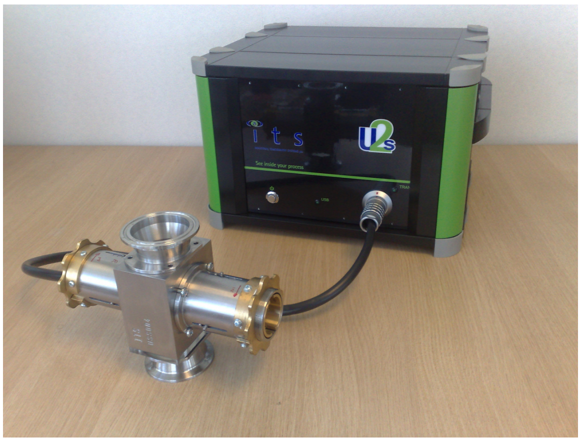
The Industrial Tomography Process Ultrasound Spectroscopy System U2s

The ITS U2s is a unique product, a single multipurpose ultrasound spectroscopy platform which can be applied to a wide range of materials from pure liquids to soft solids over a wide range of operating conditions.

It has been specifically developed to provide a solution to in-situ process characterisation needs and can provide real-time information of on the microstructure of industrial materials.

- Use of fast measurement methods
- Specifically developed for in-line/in-situ applications.
- Modular design for customization to process needs
- Able to measure both the forward (Transmission) and reverse (reflected) signals

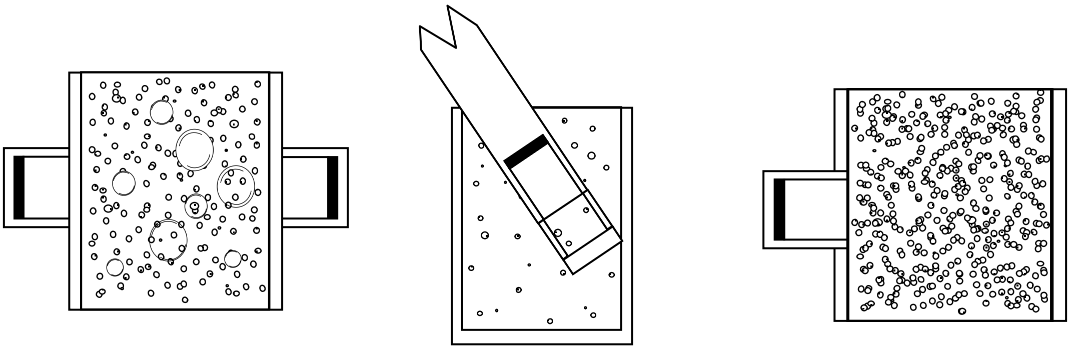


The ITS U2s with Transmission cell

The use of fast transient methods allows data collection in real time allowing the use of the system on dynamic and rapidly evolving applications, such as crystallization. The system can operate in pipes for diameters of 8mm to 100mm or vessels from 500ml up in applications with operating temperatures of 10-120C.The sensor itself is custom designed to match the application, and avoids the need to move the transducers s during measurements allowing a more robust and process compatible sensor. The sensor can either be a flow through cell for pipes or a dip in probe for reactors and large vessels.

The U2s can be configured to operate in a number of different methods to best match the application

1. The probe or pipe method: Low concentration liquids and colloids very fast measurements
2. The Coded transmission method: low concentration and High concentration liquids and colloids, moderate update rate.
3. The reflectance method, extremely highly attenuating materials almost solid.



Sensor Configurations

Ultrasound Spectroscopy Characterisation Capabilities

The Ultrasonic Spectroscopy data can be used to characterise a range of material or process properties and behaviours including:

- Measurement of density and or concentration of discreet phases mixed liquids or slurries
- Rheological characterisation of liquids and soft solids through phase velocity
- Indicative changes in attenuation spectra due to changes in material structure including particle Size of colloids in both slurries and emulsions
- Relaxation phenomena in biomaterials and soft solids
- The characterisation of liquids containing bubbles

Rheology

The rheology of a material can be calculated directly from the ultrasonic wave number. In the case of liquids the measured ultrasonic response is due to the longitudinal compressional propagation mode and hence the rheological parameter calculated is due to the compressional modulus.

The relationship between attenuation and phase spectra and storage and loss modulus is through the modification of the Wood Equation to include the complex wave number and complex modulus. Separating the complex Modulus into real and imaginary parts give approximations for the Storage and Loss Modulus.

$$\frac{\omega}{k^*} = \sqrt{\frac{M^*}{\rho}}$$

Storage Modulus

$$M' = \rho c^2$$

$$M'' = \frac{\quad}{\omega}$$

Loss Modulus

$$\eta = \frac{M''}{\omega}$$

Viscosity

where:

Complex Bulk Modulus: $M^* = M' + iM''$

Loss Modulus (Pascals): M''

Storage Modulus (Pascals): M'

Complex wave number: $k^* = \frac{\omega}{c} + i\alpha$

Frequency (radians): w

Density (kg/m^3): r

Velocity (m/s): c

Attenuation (Np/m): a

The Advantages of using USS for rheology are:

- Unlike Viscometers USS does not assume Newtonian behaviour and hence can provide greater information on complex rheology.
- USS length scales ensure that visco elasticity is linear and simplifies analysis
- USS is more sensitive at detecting start of processes, does not require bulk of material to be modified before detection.

Bubbles and Aerated liquids

The effect of air bubbles in a material is to introduce resonant behaviour at low frequencies. The acoustic wave impinging on a bubble cause the thin walls to oscillate at the nature frequency of the bubble. This natural frequency is defined by the bubble size, the pressure in the bubble and the surrounding material properties. For simple cases of monodisperse bubble populations the size of the bubble can be determined by using the Minnaert Equation

$$f = \frac{1}{2\pi a} \cdot \sqrt{\frac{3\gamma P_a}{\rho}}$$

For air in water at standard pressure the relationship is

$$f.a = 3.26 m/s$$

This can be used to provide information on the characteristics of the bubbles in a sample. In the presence of Bubbles the attenuation and velocity characteristics of the response are dominated by the bubble response and it is not possible to determine other microstructure parameters

Applications

Liquids: The acoustic velocity of a liquid is dependent on the density and bulk modulus where density defines the mass per unit volume which must be acted upon by the mechanical force and bulk modulus is a measure of the resistance to that movement. Since density and bulk modulus are temperature dependant the velocity is also temperature dependant. The theoretical velocity can be determined from the Wood equation. If the liquid is a mixture of two liquids it is possible to determine the concentration of the two liquids using a modified version of the Wood equation where the average density and bulk modulus are calculated using mixing equations.

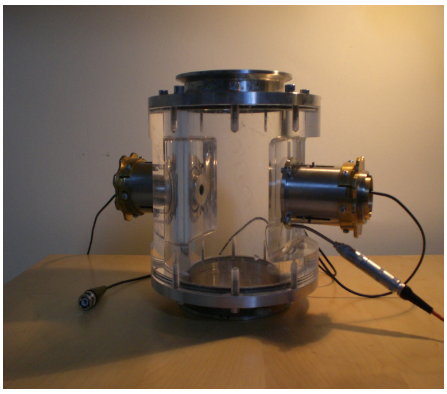
Emulsions: Ultrasound is scattered by emulsion droplets due to the differences in the thermodynamic properties between the continuous and disperse phases. At dimensions smaller than the wavelength the ultrasound wave compresses the volume of the material leading to small changes in the dimensional and thermal conditions of the droplets. This change results in changes in attenuation and phase of the wave. These changes are indicative of the droplet size , distribution and volume concentration.

Aerated Materials:. The structure of bubbles is maintained by surface tension cavity. When ultrasound encounters a bubble the structure vibrates with a frequency content proportional to bubble size. This response is resonant and has a high Q therefore signature of the bubbles will focus the energy and dominate the response. Therefore in an aerated sample the liquid and other disperse characteristics are obscured by the bubble response. Hence is possible to only measure bubble characteristics The method is also not able to determine the gas fraction as the magnitude of the response is very non-linear and hence cannot be determined analytically.

Suspensions: Particles absorb and diffract ultrasound energy and hence the change in ultrasound energy of a wave in a slurry or emulsion is a function of the particle size. Particle size determination from the ultrasonic attenuation spectra is based on a first principles model. Significant information is needed in order to accurately determine the particle size and distribution even in relatively simple applications. Complex or multimodal particle size distribution requires significant effort to development robust solutions. However whether a particle size model is used or not the change in ultrasonic spectra reflects change in particle size. In a process situation use of the spectra is sufficient to control the process.

Sensors

The U2s system is modular and be used with a wide range of sensors including bespoke designs for specialist applications.



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