Published paper
Dwyer-Joyce, R.S. and Hankinson, N. An ultrasonic technique for the measurement of elastic properties of soft surface coatings. *Tribology International*, 2006, **39**(4), 326-331.

http://dx.doi.org/10.1016/j.triboint.2005.02.003

An Ultrasonic Technique for the Measurement of Elastic Properties of Soft Surface Coatings

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Abstract

The properties of thin layers of materials can be different from those in the bulk form. The response of a coating to any given load and its ability to remain bonded to the substrate will depend on its elastic modulus and Poisson's ratio. In this study a measurement method based on ultrasonic bulk wave reflection was evaluated. As a model system, a thin layer of polyethylene was pressed between two solid steel bodies. The reflection spectra of longitudinal and shear ultrasonic waves were recorded from the coating. The frequencies at which the layer resonates were measured and from this the wave speeds deduced. The Poisson's ratio can be determined from these two wave speeds and if the layer thickness is known the modulus is also available. The tests yielded reasonable values for both. This approach is only suitable if the layer can be made to resonate by the available ultrasonic frequencies; typically this will be the case for thicker coatings (tens of microns). Further, good coupling between the layer material and the steel bodies is necessary so that the interfaces do not themselves act to reflect ultrasound. This is better achieved with a smooth soft coating.

Introduction

Soft surface coatings are applied to a wide range of engineering components. Physical Vapour Deposition is frequently used to apply lubricating surface coatings based on PTFE or Molybdenum Disulphide. Alternatively polymeric coatings are used for corrosion protection, heat or electrical insulation, decorative purposes, or to enhance structural damping. In recent years polymer coatings have been widely applied to protect the surfaces of magnetic media such as CD-ROMS or hard disks.

The durability of the surface layer depends on the properties of the surface coating and how well it is bonded to the substrate. The elastic properties are particularly important in the prediction of how the coating-substrate interface will respond when the component is subjected to distortion. The properties of a thin layer of a material are different to those of the bulk solid. So any test method must be based around the testing of small specimens. Depth sensing nano-indentation methods have proved useful in this respect. Monitoring the unloading of an indentor can be used to determine the reduced modulus of the surface material. The reduced modulus is a combination of the Young's modulus and Poisson's ratio for the material. Measurement of the velocity of an elastic surface wave will also give the reduced modulus [Rokhlin and Lavrentyev 1998]. However, in this case the modulus is determined in an orientation parallel to the contact surface. Many coatings have a variation in properties through the thickness and also spatially across the surface. It would be preferable therefore to measure the properties in the though thickness direction.

In this paper, an alternative method for measuring elastic surface properties is presented. The response of the surface layer to an elastic ultrasonic bulk wave is determined. The speed of

propagation of the wave and the resonant frequency of the layer are both functions of the modulus and Poisson's ratio. By determining these values for both transverse and longitudinal waves the elastic constants can be deduced.

Background

Two forms of ultrasonic bulk wave are frequently used in non-destructive testing; longitudinal (or normal) and transverse (or shear) waves. The velocities, c_τ and c_σ, respectively, at which these travel through a medium are given by (Krautkramer & Krautkramer 1990):

$$c_\sigma = \sqrt{\frac{E}{\rho}\frac{1-\upsilon}{(1+\upsilon)(1-2\upsilon)}} \tag{1}$$

$$c_\tau = \sqrt{\frac{E}{\rho}\frac{1}{2(1+\upsilon)}} \tag{2}$$

where, E is the Young's modulus, υ the Poisson's ratio and ρ, the density of the material through which the wave is travelling. When the ultrasonic wave strikes an interface between two materials a proportion of the wave amplitude reflected. This proportion (known as the reflection coefficient, R) depends on the difference in acoustic impedance of the two materials. When a wave strikes a layer imbedded between two other materials some proportion will be reflected at the front face and a further proportion at the back face. If the layer on a surface is sufficiently thick then it would be feasible to resolve the reflections from the coating interface and surface spatially and determine the acoustic velocity in the layer (if the thickness were known). This could be carried out for both normal and shear waves and equations (1) and (2) used to determine the elastic constants. This is a standard technique for determining elastic properties in bulk materials. However, most applied surface films are so thin that with conventional ultrasonic frequencies it is not possible to spatially separate the reflected signals.

When the thickness of the layer is small compared with the ultrasonic wavelength, the system may be treated as an array of springs in series. The reflection of the wave is a function of the stiffness of the imbedded layer according to a spring model (Tattersall 1973):

$$R = \frac{z_1 - z_2 + i\omega(z_1 z_2 / K)}{z_1 + z_2 + i\omega(z_1 z_2 / K)} \tag{3}$$

where z_1 and z_2 refer to is the acoustic impedance of the materials either side of the lubricant layer. The acoustic impedance is a product of the wave speed, c and the density, ρ. The stiffness per unit area, K, of a thin layer of thickness, h can be obtained from (Hosten 1991):

$$K = \frac{\rho c^2}{h} \tag{4}$$

Dwyer-Joyce et al. [2003] have used this approach determine the thickness of a liquid layer (lubricant films) between solid surfaces. If the acoustic impedance mismatch of the two materials either side of the layer is large (e.g. for a substrate-coating-air system) then R in equation (3) tends to unity and the stiffness (and therefore h or c) becomes hard to measure. The method works best when the materials either side of the layer have a similar acoustic impedance. For this reason it is necessary to create an imbedded layer by pressing a *contactor* onto the surface coating.

Equations (3) and (4) are suitable for thin imbedded layers. A more general treatment, based

on numerical solutions to the continuum wave equation as it passes across various interfaces, is given by Pialucha et al (1994). Figure 1 shows predictions of reflection coefficient against frequency for several thickness of imbedded layers (in this case polyethylene terephthalate polyester, PETP between steel half spaces).

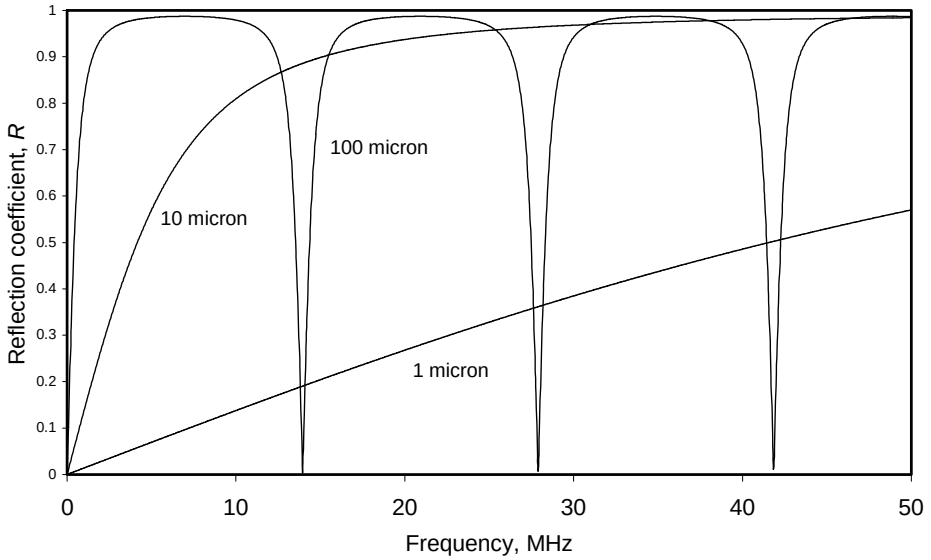


Figure 1. Continuum model predictions of the reflection coefficient spectrum from three thickness layers of PETP between two steel elements.

The reflection coefficient spectra shows minima where the layer is resonating and that particular frequency is completely transmitted through the layer. The location of these resonant frequencies is given by:

$$f_{res} = \frac{mc}{2h} \tag{5}$$

where *m* is the wave number. There are thus two methods available to determine the wave speed for an imbedded layer. Firstly, the reflection coefficient can be used to determine the layer stiffness (equation 3) and hence the speed (equation 4). Or, secondly, the resonant frequency can be measured and used to determine the speed directly (equation 5). The former approach is suitable for thin films, the latter for all films where a resonance can be obtained within the available frequency spectrum. In this work, it is the latter method that has been adopted.

Experimental Procedure

Layer Loading Apparatus

In these early studies thin polymer sheets were used to simulate a surface applied coating. The sheet was sandwiched between two flat polished steel specimens and compressed in a simple hydraulic loading apparatus. Figure 2 shows a photograph of the test apparatus.

The technique required the measurement of both longitudinal and transverse waves reflected from the surface layer. It was found to be necessary to take these measurements simultaneously. This was because removing and replacing transducers resulted in inconsistent coupling between the transducer specimen back face between test cases. The transducers were arranged above and below the layer in a load bearing housing as shown in Figure 3.

Figure 2. Test rig used for pressing a polymer layer between two steel specimens.

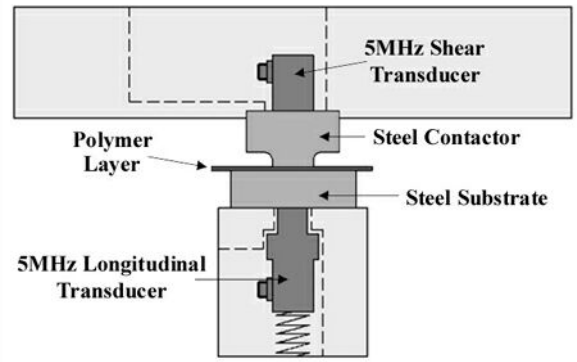


Figure 3. Arrangement of the ultrasonic probes for the measurement of longitudinal and transverse reflected waves.

Test Specimens

The substrate was simulated using a polished steel 10mm thick plate. On this was placed a layer of PETP film of thickness 175 μm. The layer was then pressed by a steel contact specimen, with a 15 mm diameter contact face, as shown.

Ultrasonic Transducers and Pulsing Apparatus

The transducers used were of the conventional Panametrics ceramic piezo-electric planar contact type with centre frequencies of 5 MHz. Both transducers were wide band with useful frequency ranges between 4 and 9 MHz for the longitudinal probe, and 2 and 8 MHz for the transverse probe. A pulser receiver unit was used to both excite and receive the ultrasonic signals. Each transducer was excited independently and used in pulse echo mode, rather than simultaneously to avoid signal interference through transmission. A digital oscilloscope was used to capture reflected waveforms that were subsequently passed to a computer for signal processing. Figure 4 shows a schematic layout of the apparatus.

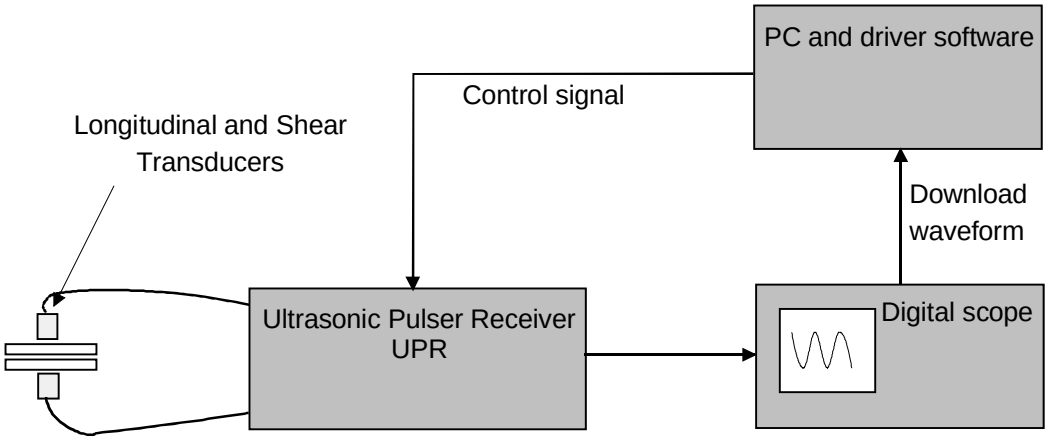


Figure 4. Schematic diagram of the ultrasonic pulsing and receiving system.

Collection of Reflection Signals

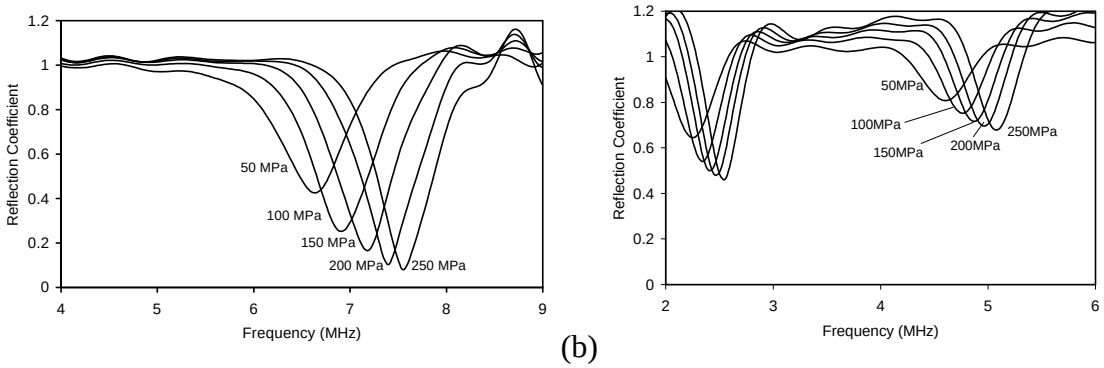
The first step in the procedure was to record reference signals for each transducer. When the specimens were unloaded (and the polymer layer was out of contact with either surface) then the waves reflect from a steel-air boundary. The waves reflect completely and the received signal was thus equal to the incident signal. This signal was then used as a reference; all subsequent reflected signals were divided by this reference to give the reflection coefficient.

Once the steel-air signal was recorded, the layer of PETP was introduced and the assembly loaded using the hydraulic cylinder. Signals were recorded from both transducers at contact pressure increments of 50MPa. The reflected spectra, for both longitudinal and transverse waveforms, relative to the steel-air reference (i.e. the reflection coefficient spectra) were then observed under varying layer contact pressure.

Results

Figure 5a shows the reflection coefficient observed with the longitudinal transducer for a series of applied contact pressures. In each spectrum a clear resonant minima was observed. As the load on the film was increased, the position of the minima occurred at higher frequencies.

Figure 5b shows a similar set of results but for the reflected transverse wave. In this case two minima were observed in each spectrum. This is because the resonant frequency for shear waves is lower than that for longitudinal waves (as determined from equations 1, 2, & 5). Thus, within the useful bandwidth of the transducer (2 - 8 MHz) there was enough energy to excite at the first and second resonances.



(a) (b)

Figure 5. Reflection coefficient spectra for a 175μm layer of PETP pressed between two steel

plates, at a range of contact pressures, using (a) longitudinal waves, and (b) shear waves.

In both cases, the resonant frequency was clearly dependent upon the pressure applied across the layer. As the load was increased, the thickness of the film reduced and its resonant frequency increased.

The ratio of the transverse to the longitudinal resonant frequency ($f_{res\tau}/f_{res\sigma}$) is independent of the layer thickness h and equal to the ratio of the respective wave speeds (c_τ/c_σ). This ratio is approximately constant (varying from 0.332 to 0.349) as shown in figure 6. Poisson's ratio can then be calculated readily by division of equations (1) and (2) giving a value of υ=0.43±2%.

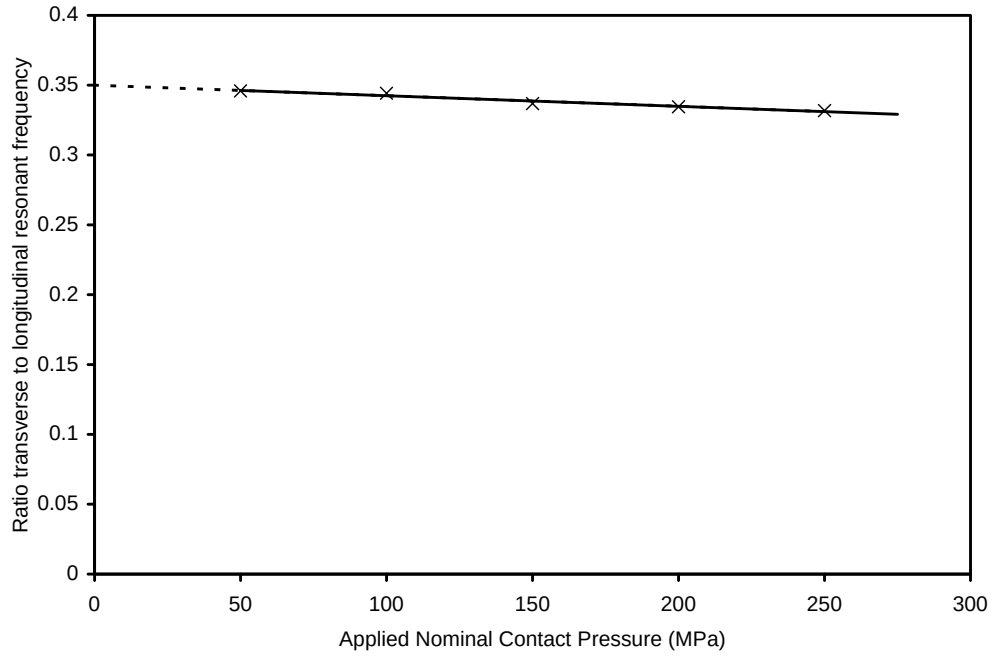


Figure 6: Ratio of transverse to longitudinal resonant frequencies as a function of pressure.

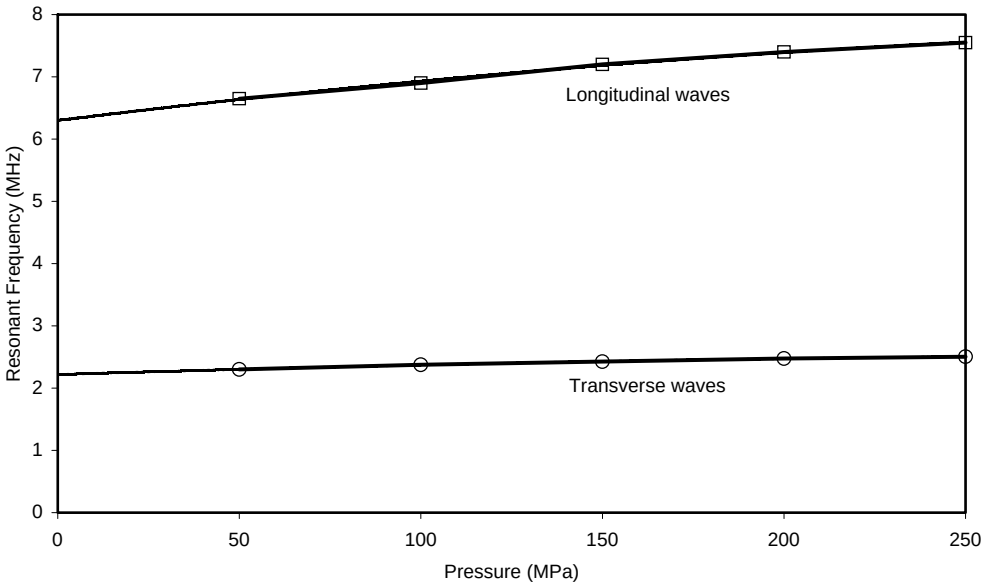


Figure 7. Variation of layer resonant frequency, for longitudinal and transverse waves, with applied nominal contact pressure.

In figure 7, the resonant longitudinal and transverse resonant frequencies are plotted against pressure. A best-fit curve is extrapolated back to the y-axis to give a value for the resonant frequency at a zero pressure (i.e. when the film is at its original thickness of 175 μm). This approach gives values for resonant frequency of 6.3MHz for longitudinal waves and 2.2MHz for transverse waves.

If the density and thickness of the coating are assumed correct (obtained from manufacturer's data as 1350 kg/m^3 and 175 μm respectively) then one or other of these resonant frequencies can be used to determine the Young's modulus of the layer using equation 5. This calculation gives a measured modulus of 2.3 GPa.

The modulus of the film alone as determined in a uniaxial tension test is between 2 - 3 GPa. And the reduced modulus, $E^*=E/(1-\upsilon^2)$ determined using a nano-indentation method (as described by Oliver and Pharr 1991) is between 3.6 - 5.1 GPa depending on indentation location. The reduced modulus calculated from the data, determined in this ultrasonic study, is 2.8 GPa. There is reasonable agreement between these methods; although the ultrasonic method does gives a result somewhat lower than that determined from nano-indentation.

Application to a Bonded Surface Coating

This approach has been evaluated on a bonded surface coating. The coating was epoxy based and used as a commercial damping coating applied to aerospace structures. The elastic properties are important to the designers because they are necessary for the prediction of damping behaviour. Figure 8 shows the measured resonance frequencies as the contact specimen is loaded against the coated surface. Again the resonance frequency increases slightly as the coating is compressed. The ratio of the two resonance frequencies give the velocity ratio as c_t/c_σ=0.44 and hence (from equations 1 and 2) a Poisson's ratio of υ=0.38.

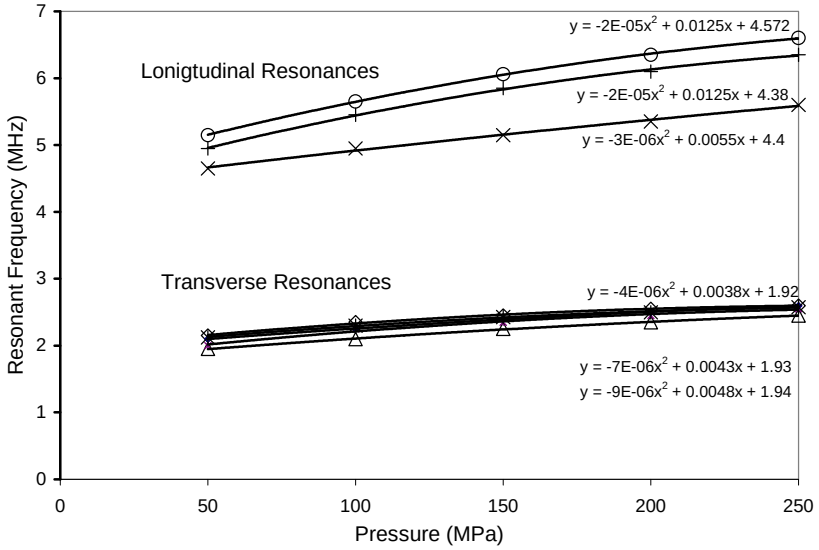


Figure 8. Resonant frequencies for longitudinal and transverse waves at an epoxy coated surface pressed against a steel specimen.

The different curves represents separate tests on new areas of surface. There is some scatter between measurements; whilst the two waves forms are recorded from the same contact case,

this still leads to some scatter in the ratio of resonance frequencies. There variation is probably caused by the variation loading conditions and whether a uniform loading is achieved over the contact. In this case, there is no value available for the coating thickness or density, so it has not been possible to determine the modulus.

Discussion

Limits of Applicability: Coating Thickness

The method adopted in this paper relies on the measurement of a layer resonant frequency. As such it can only be used when that frequency can be excited within the wave frequency used. Typically, it is possible to use ultrasonic frequencies up to around 50MHz; above this value equipment becomes expensive and the higher frequencies tend to be attenuated by a relatively small thickness of substrate. For a PETP layer this then gives a minimum measurable film thickness (from equation 5) of 22 μm. Layers thinner than this will not excite a resonance at longitudinal frequencies below 50 MHz. This method will be suitable therefore for thicker types of surface coating.

The Stiffness Approach

In principle, it is possible to determine the elastic constants from the layer stiffness obtained via reflection coefficient measurements (using equation 3 and 4). Some preliminary experiments were carried out to investigate this method. Experiments were performed on a 30 μm PETP film and the stiffness of the layer determined. However problems arose with the loading of the film between the two steel bodies. A rough surface contact also has an inherent stiffness (Drinkwater et al. 1996). The measured reflection coefficient is dependent not only on the stiffness of the PETP layer but also on the stiffness of the steel-PETP interface. Whilst the interface is relatively stiff compared with the layer, it proved difficult to get a repeatable coupling between the PETP and steel. The stiffness variations were significant when it came to determining elastic constants (but did not greatly affect the position of the resonant frequency).

Limits of Applicability: Coating Material

The use of a hard surface coating may also cause problems with the coupling between layer and contactor. The polymer layer permits relatively close contact (and therefore stiff interfaces); whilst a harder coating may result in a stiffer interface. The present method is currently most appropriate for thick soft layers. A liquid couplant (gel, water, or grease for example) could be used to improve this coupling. Unfortunately this coupling layer would also have an associated stiffness that would then need to be separated from the measured stiffness to extract the layer thickness. The stiffness of a couplant layer would be hard to quantify as it depends on the roughness of the surfaces being pressed together. However, if it is stiff compared to surface coating then it could be neglected. The relationship between the nature of the couplant layer and possibility of harder coating measurement remains to be investigated.

Conclusions

An ultrasonic wave can be used to cause a thin imbedded layer to resonate. Resonant frequencies will be transmitted through the layer whilst others will be completely or partially reflected. These resonant frequencies, for both longitudinal and transverse ultrasonic waves, depend on the normal and shear velocities respectively. Once these are known it is possible to determine Poisson's ratio directly, and if the layer thickness is known, Young’s modulus. The technique has been applied to layers of PETP film pressed between steel bodies and also an epoxy based damping coating.

This approach gives a simple method for determining the, through thickness, elastic properties of thin layers. It relies on, a known density and coating thickness, good coupling between layer and contactor, and that the coating is thick enough to resonate. The nature of the measurement approach is such that it is most suited to relatively thick soft polymer coatings.

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