**Holographic and electronic speckle pattern interferometry applied to the measurement of static and dynamic mechanical properties of elastomers**

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HOLOGRAPHIC AND ELECTRONIC SPECKLE PATTERN INTERFEROMETRY APPLIED TO THE MEASUREMENT OF STATIC AND DYNAMIC MECHANICAL PROPERTIES OF ELASTOMERS

BY

ADRIAN CHARLES ROWLAND

A MASTERS THESIS

SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS OF THE AWARD OF MASTER OF PHILOSOPHY OF THE LOUGHBOROUGH UNIVERSITY OF TECHNOLOGY

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ABSTRACT

Taking as its starting point a holographic interferometric technique, used to measure the static and dynamic bulk moduli of elastomers, this thesis describes the progression of research aimed at extending the range and nature of these measurements. The use of Electronic Speckle Pattern Interferometry (E.S.P.I) as an alternative optical technique is described. The use of amplitude and phase modulation within this interferometer is also described.

These modulation techniques are shown to be advantageous in manipulating fringe distributions making data extraction easier and more accurate. This allows measurement, over an extended frequency range, of the pressure and temperature dependence of bulk moduli and also Young's moduli for elastomer samples. The visco-elastic components of these moduli are also investigated by using E.S.P.I. to measure the loss component \( \tan \delta \) of the vibrational response of the elastomer.
ACKNOWLEDGEMENTS

The author wishes to thank Dr B P Holownia for his guidance and friendship throughout the course of this research and the late Dr John Butters for his role as director of research.

Many people deserve thanks for the assistance and support I received whilst working in the Department of Mechanical Engineering in particular Mr Brian Bergquist, Mr Mick Smeaton, Mr Ken Topley and the technicians of the Mechanical Workshop. Other colleagues and friends who I would like to thank for their invaluable support; Dr John Powell, Dr Paul Montgomery, Dr Cathy Wykes and Dr Fernando Mendoza. I would also mention other members of the Optics Group at Loughborough who made my time there entertaining; John Tyrer, Liz Raymond and Paul Henery. Thanks also to Dorethy Kelly for her unending patience in the preparation of this thesis.
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1. INTRODUCTION

Holographic and speckle pattern interferometry are finding many applications in engineering metrology. These are whole field techniques which allow a range of fine measurements over comparatively large areas. Holography in particular has been used in a mainly qualitative way because of the difficulties involved in extracting fringe data. Previous work (1) has shown that holography can be used to measure accurately, volume contractions capable of yielding values for the static and dynamic bulk modulus of elastomers.

The accurate measurement of material constants is becoming increasingly important with the prolific use of numerical methods of stress and strain analysis. Many techniques are already available to measure most mechanical properties with high accuracy. Elastomers, a generic term for rubbers, prove generally more difficult particularly the measurement of bulk modulus and Poisson's ratio. The Poisson's ratio of rubbers is generally between 0.49 and 0.4999 which has led to them being wrongly termed incompressible. This term would be reserved for a material with a theoretical Poisson's ratio of 0.5. It does, however, mean that large compressive forces are required to produce discernible volume reduction, hence the use here of sensitive optical techniques.

Further complications arise because the dynamic mechanical properties of elastomers have elastic and viscous components. It is the vectorial product of these two components which determines the strain, at a characteristic phase angle to the applied stress.

Published work on the measurement of dynamic bulk moduli of elastomers has concentrated on acoustic properties of
materials in the ultrasonic region. Little has been published on measurements in the region 0 - 1kHz where materials with glass transition temperatures near room temperature exhibit dramatic changes in properties.

The work detailed here describes the use of holographic and electronic speckle interferometric techniques capable of resolving very small surface displacements. Modulation of amplitude and phase of the laser light is used to manipulate standard interferometric sensitivities to reduce complexity and extract information from resultant fringe fields.

The developed technique is then used to make initial measurements of the frequency, pressure and temperature dependence of static and dynamic bulk moduli of elastomers in the region of 0 - 1kHz, 0 - 40 bar and over a limited temperature range. These measurements, together with measurements of Young's modulus, can be used to characterize the mechanical properties of the material through the Lamé constants for isotropic materials.

The results are presented in a way which is intended to highlight the rationale for various choices of technique. In this respect the results of early work are described critically before going on to concentrate on results obtained from the developed technique.

For the sake of brevity in the following chapters the reader is assumed to have a prior knowledge of the optical techniques used and of the principles of mechanical properties of elastomers.
2. BACKGROUND TO EXPERIMENTAL WORK

Holographic Interferometry as a means of measuring static and dynamic displacements of optically rough surfaces has only been possible since the development of the LASER in the early nineteen sixties. The off axis holographic technique of Leith and Upatnieks (2) was first extended to study the motion of vibrating objects by Powell and Stetson(3). Object motion during holographic exposures usually causes failure of the image recording. Powell and Stetson, however, showed that if the object is excited sinusoidally with very small amplitudes, a pattern of dark and light areas or fringes are observed on the reconstructed holographic image. They showed this modulation of the image amplitude to be given by

\[ M = J_0 \left( \frac{2\pi}{\lambda} \right) \left\{ \cos \theta_1 + \cos \theta_2 \right\} m(x,y) \]

where \( \theta_1 \) and \( \theta_2 \) are the angles of illumination and viewing relative to the object vibration \( m(x,y) \), \( J_0 \) is the zero order bessel function of the first kind. This approach allows a quantitative study of very small amplitude vibrations.

Burch and Ennos (4) noted that interference effects could be obtained between an object and its holographically recorded image. This was achieved if the two were superimposed by replacing the processed hologram plate in precisely the same position in which it had been recorded. This allowed real time study of the static displacement of the object.

From these beginnings, the field of holographic interferometry has developed to cover a vast cross section of possible measuring techniques. It would not be relevant to carry out a comparison of the advantages and
disadvantages of all of these techniques. Instead, this text will concentrate only on those areas of particular interest to the work carried out here.

Holownia (1) has outlined the use of holography to measure the radial contraction of a spherical elastomeric sample due to the application of hydrostatic pressure. From this measurement the bulk modulus of the material was calculated. The measurements were made on samples subjected to static pressure, using double exposure holography, and samples subjected to sinusoidally varying pressures, using time averaged holography. The technique involved viewing a spherical sample through a glass observation window in a steel pressure cell, see Fig.2.1. The sample was submerged in a pressurising fluid, either oil or glycerine, by means of which pressure changes were applied. The laser light scattered from the sample passes through the fluid, on approaching and returning from the sample surface. Thus the pressure-induced refractive index changes of the fluid causes phase changes in the scattered light. These phase changes together with those introduced by the sample surface movement, form the interference fringes observed in the reconstructed image.

The theory given by Holownia requires an exact knowledge of the bulk modulus of the submerging fluid in order to give an accurate measure of the fringe shift introduced by the refractive index changes. To this end a steel ball is studied. Having a much higher bulk modulus than the elastomer, the steel ball undergoes minimal radial contraction due to the applied pressures. The fringes produced are due to refractive index changes only and this information is used to calculate the bulk modulus of the fluid.
FIG 2.1  Pressure cell, Holownia[1]
To achieve this the Gladstone Dale empirical relationship was used, an experimentally determined equation relating changes in density to refractive index changes. This expression is normally used for gases (pp344, 13). However, a similar choice was made by Jones and Bergquist(5) in their dynamic calibration of pressure transducers.

Their optical system consisted of an oil filled pressure vessel with optically flat windows which was placed in one arm of a Michelson or Jamin Interferometer. The pressure induced refractive index changes were detected as fringe shifts by a photodiode arrangement. The phase change of the light, wavelength $\lambda$, passing through the oil cavity, length $L/2$ with fluid of refractive index $n_0$ and bulk modulus $K_0$, is shown to be given by:

$$\Delta \phi = \frac{2\pi}{\lambda} \left( \frac{n_0 - 1}{K_0} \right) L \Delta P$$

this is for a pressure change $\Delta P$

However, in the experimental analysis, the expression $\left( \frac{n_0 - 1}{K_0} \right)$ is replaced and the slope of experimental results used in the equation for $\Delta \phi$. This means that it is the linearity of the Gladstone Dale expression which is used not the value of the constant, given by:

$$\frac{n_0 - 1}{\rho_0} = \text{CGD}$$

where $\rho_0$ is the density of the fluid.

Poulter et al(38) in their measurements on the effect of pressure on the refractive index of parafin oil and glycerine show that the alternative Lorentz-Lorenz equation:

$$\frac{(n_0^2 - 1)}{(n_0^2 + 2)} \frac{1}{\rho_0} = C_{LL} = \text{Constant}$$
holds true for both liquids over the pressure range 1 - 10000 Atm., although it must be mentioned that the Gladstone Dale constant also remains stable for the glycerine results given. The difference in using either of these two expressions is shown in chapter 3. Results obtained by Holownia(1) for the bulk modulus of glycerine do, however, agree with published values.

Holownia by using spherical samples, made the application of triaxial loading, required to give pure bulk deformation, very much easier. The introduction of static or dynamic pressures was used to cause such pure strains; the fluctuating pressures themselves were generated by a hydraulic servo valve connected to the pressure chamber. Static pressures were applied via a close fitting weighted piston and dead weight.

The holographic recordings made for an isotropic ball sample resulted in a set of circular fringes concentric with the ball centre, see Fig.2.2. To extract fringe data from these recordings required taking a photograph of the holographic image in order to measure the fringe spacings and their positions on the ball diameter. This information was used to plot fringe order against cosine of the angle subtended at the centre of the ball by the fringe position. The actual fringe order on the ball is unknown as the zero order and subsequent higher order fringes disappear from the image. It is thus the slope of this graph which yields the value of $\Delta R$ the radial contraction of the ball or $K_0$ the bulk modulus of the pressurising fluid.

Two elements are missing from the analysis of these fringes:
FIG 2.2 Holographic recording, Holownia
1) The holographic technique with normal viewing and illumination is sensitive to movements in the direction of viewing. Thus the radial contraction is detected with increasing sensitivity as the angle $\theta$ increases, see Fig. 2.3.

![Fig. 2.3 - Change in fringe sensitivity with angle $\theta$](image)

2) The change in fringe distribution between double exposure and real time studies is determined by the functions describing the fringes of each. For double exposure the fringe distribution is governed by $\cos^2$ as compared with $J_0^2$ for time average fringes.

It can be seen that the optical technique used by Holownia was not fully developed. Throughout the following section of this chapter the measurement technique already outlined will be developed and new methods introduced which were considered to extend the measurement of dynamic bulk modulus $K^*$ by studying the viscous loss factor $\tan \delta$.

Time averaged holography, when used to study a vibrating object, is capable of yielding dynamic amplitude values over the object surface. The relative phase of vibration of any part of the object, with respect to other parts on the object or the forcing function, is lost due to the time integral effect of the recording medium. Several
techniques have been developed capable of retrieving this phase information, which is stored as a modulation term in the phase of the scattered wave from the object.

Archbold and Ennos\(^6\) used a rotating shutter system to produce two pulses of laser illumination, \(\frac{1}{20}\) of a period in duration. From this illumination pulse train they split and amplified a signal which was used to drive a piezoelectric transducer attached to an aluminium disk. The vibrating disk was then viewed through a real time hologram of the continuously illuminated stationary disk. They comment on the increased visibility of higher order vibration fringes and on being able to identify more resonant modes on a frequency scan than were observed without strobing. By using a rotating mirror and polarising filter they were able to alter the relative phase of the vibration and strobing. By advancing the phase of vibration for a fixed stationary resonant mode, they were able to identify the phase relation between areas on the plate. This, however, was only a qualitative method and no accurate phase measurements were attempted.

A study of vibration amplitudes using this real time approach produces a more complex result than that obtained from time averaged recordings. This is due to the static element introduced by the recording of the stationary object. If subsequent vibration modes are equidistant about the disks stationary position, half the vibration amplitude is observed, but if areas vibrate about a mean point, which is not the stationary position, fringes will tend to merge or blur.

Zaidel et al\(^7\) were looking at a very similar technique but using a rotating shutter system to modulate both the object and reference beam amplitude. The shuttering system allowed the periods between illumination to be
altered so that illumination pulses could be produced with very short gaps between them. Once again by strob ing a vibrating object and observing through a real time hologram of the unstrobed stationary object, they were able to increase the number of higher order vibration fringes visible. They also noticed that a time averaged recording of the strobed object, together with strobed reference, improved fringe visibility further. This was due to the general poor efficiency of their real time holographic recordings, the fringe visibility being a function of the hologram efficiency and transmitted object intensity. The technique described does not extend to varying the phase of the strobing relative to object vibration. They do, however, show the effect of reducing the period between strobing such that the illumination occurs at points separated by less than a half period of the vibration cycle. This reduces the object motion allowing large amplitude vibrations to be studied.

One problem they have not mentioned, is knowing when to effect the strobing when using strobed time averaged holography. The timing of illumination pulses determines the object displacement which is observed. So to observe maximum object displacements the strobing must be effected at moments of peak displacement. For this reason it is almost always necessary to use real time holographic recordings, with their inherent problems, in order to study the phase of object variation, using strobed holography.

One alternative, using amplitude modulation of the reference beam only, was outlined by Takai et al.(8). They show that if the amplitude of the reference beam is sinusoidally modulated at a frequency equal to that of the vibrating object and at a relative phase \( \phi \) the resulting image intensity distribution will be given by

\[
I = J_1^2(\alpha) \cos^2 \Delta
\]
where $J_1^2(a)$ is a characteristic result obtained by the single sideband suppressed-carrier theory of Aleksoff\(^{(11)}\) and $\Delta = \phi - \theta$ where $\theta$ is the phase of the object vibration. So it is shown that the bessel function fringe pattern is modulated by $\cos^2$ with a maximum value when $\phi = \theta$. The fringe distribution shows a bright fringe in areas where the phase of reference amplitude modulation equals that of the object vibration. The major drawback of this technique is that $\cos^2 \pi = \cos^2 0$ so areas $180^\circ$ out of phase cannot be separated.

The phase information of a vibrating object may also be determined holographically with the use of phase modulation of the optical beams used in the recording. This principle was first shown by Aleksoff\(^{(9)}\) in his study of a phase object, that is an object which only affects the phase of light passing through it. The object, an ADP (Amonium Dihydrogen Phosphate) crystal exhibiting the Pockel effect, was studied using normal holography and also with cross polarised filters. A hologram was then made with a reference beam which had been passed through an area of the crystal. The results showed that areas which had been bright in the original hologram i.e. zero order fringes, were now dark and that areas causing the same phase and depth of modulation as that of the reference path were now bright. Also areas $180^\circ$ antiphase with these areas were dark. Thus it was shown that the zero order bright fringe of a time averaged recording could be made to occur at areas not stationary and by altering the phase angle and depth of modulation, could be made to reconstruct at any area on the phase object.

This concept was developed by Neumann et al\(^{(10)}\) to cover time averaged and real time studies of vibrating objects, describing a method of complete phase mapping of a complex object.
vibration mode. The phase modulation was introduced to the object or reference beam by means of a piezoelectric mounted mirror. They showed light from a point on the object to be phase modulated according to the vibration at that point:

\[ \theta_s(t) = \theta_{so} + \frac{2\pi}{\lambda} a_s \cos (\omega t + \phi_s) \]

where \( \theta_{so} \) is the phase of the unmodulated object beam, \( a_s \) the amplitude of vibration, \( \phi_s \) the relative phase of object vibration and \( t \) the time varying phase of the vibration. Together with the reference wave, modulated by the mirror vibration, (vibrating at the same frequency as the object)

\[ \theta_r(t) = \theta_{ro} + \frac{2\pi}{\lambda} a_r \cos (\omega t + \phi_r) \]

Then for a time averaged recording of the object an expression similar to that obtained under normal illumination conditions results for the exposure variation

\[ \xi = A_r^2 + A_s^2 + 2A_rA_s J_0(2\pi at/\lambda)\cos \phi \]

where \( A_r \) and \( A_s \) are the amplitudes of reference and object wave except now \( a_t \) is given by

\[ a_t = \sqrt{\left[ a_r^2 + a_s^2 - 2a_r a_s \cos (\phi - \phi_r) \right]}^{1/2} \]

Thus \( a_t \) was shown to be the magnitude of a resulting phasor of the object and reference phases. The minimum value of this phasor, which represents maximum image brightness, was shown to be given when \( A_r = A_s \) and \( \phi = \phi_r \). The authors go on to describe how the same treatment can be used to derive an expression for a live fringe (real time) study, phase modulation being applied to the
reference beam replaying the real time hologram. This allowed them to study the phase of vibration of different areas of a square plate, excited by a phase referenced source, under several modes of vibration.

A comprehensive study of the theoretical and experimental implications of many types of temporally modulated holography, both amplitude and phase, is given by Aleksoff(11). He describes the use of phase modulation of the object wave to sensitise the holographic process producing a heterodyne system, altering the order of the Bessel function describing the resultant fringe field, allowing detection of very small amplitude vibrations. Other generalised considerations of such modulation techniques can be found in references (12) and (13).

The major problem of holographic studies used to measure the phase of object vibrations comes from the lengthy procedure of making repeated time averaged holograms with different modulation characteristics, or the difficulties of producing good real time recordings. The first is laborious and does not guarantee identification of important phase angles. The second involves using an in situ wet tank to remove the problem of accurate relocation. The more recent holographic cameras such as the thermoplastic camera of Newport Research Corporation or Baling Electro Optics wet film process, make this procedure easier but costly. An alternative interferometric technique which allows a much more flexible approach to the problem is that of electronic speckle pattern interferometry (E.S.P.I).

The fundamentals of the speckle pattern correlation interferometer were outlined in a paper by Leendertz(14). The speckle effect noticed when a laser illuminated surface is observed by an imaging system of finite
aperture size, is utilised to characterise the surface. Leendertz showed how the combination of two such speckle fields results in a third speckle pattern. This resultant pattern is sensitive to changes in either of the two original fields. If one of the surfaces producing these fields moves, introducing a phase change $\phi$ into its speckle field, the resultant pattern will undergo random changes in amplitude until $\phi = 2\pi$. The pattern will then return to its original form.

Leendertz also showed that a photographic negative of the focussed image of the resultant speckle pattern, when relocated accurately to produce minimum transmission of the speckle field (correlated) will detect movements of either surface by an increase in the transmitted light.

Experimentally the correlation is achieved by taking a photographic positive of the speckle pattern produced when one of the surfaces is deformed and locating this accurately with the first negative. High pass Fourier filtering of the transmitted light from the two transparancies enhances areas of high and low spatial frequency content. Areas where the object has not moved or where $\phi = 2\pi$ are correlated and have low spatial frequency content and are shown as dark areas. Intermediate areas have high spatial frequency content and are shown as light areas.

The replacement of the photographic recording medium with a video camera and the use of a smooth reference wavefront rather than speckled was outlined by Butters and Leendertz(15) in 1971. The advantages of an effective real time recording medium were seen as a major advantage over the previous speckle technique of Leendertz(14), the technique was seen to have great potential. The two stage transparancy was replaced by two video recordings of the deformed and undeformed
speckle patterns. Electronic inversion, then addition of one of these video frames to the other, together with electronic processing to simulate Fourier filtering yielded a video picture similar to that obtained by the photographic treatment. Also outlined in this paper is the time average use of this interferometer. The persistence of the camera tube acted as an integrating function and video processing was used to make the display equivalent to a time averaged hologram.

The technique developed quickly from these beginnings. The improvement of video processing techniques formed an area of major advancement. Developments in optical techniques have followed those of holographic interferometry.

A paper by Pedersen Lokberg and Forre (16) shows the improvement of E.S.P.I. results in time average study and also introduces the use of strobed E.S.P.I. to improve fringe contrast. The optical system was the same as that described by Butters and Leendertz (15) except for an electro-optic modulator which was used to amplitude modulate both the reference and object beams. Electronic processing was achieved by high pass filtering and full wave rectification. The authors demonstrate that the increased sensitivity of the standard vidicon tube used allowed stroboscopic operation of the E.S.P.I. system, at frequencies above the persistence time of the tube. However, for time averaged results, a 5 mW HeNe laser was used and for strobed results a 20mW HeNe laser was used. Results show that, as in holography, the cosine fringe distribution of stroboscopic illumination enables higher order fringe systems to be studied. They do not describe the use of strobed operation to study vibration phase measurement, they do mention the possibility of doing so.
Processing of the strobed signals was identical to the time average procedure indicating the comparability of the video processing to the square law detection system of photographic recordings. Pedersen et al point out that for time average study, the result of imperfect full wave rectification will lead to a resulting fringe function between $J_0(x)$ and $J_0^2(x)$ and presumably between $\cos(x)$ and $\cos^2(x)$ for stroboscopic fringes.

Lokberg\(^{(17)}\) has gone on to describe the use of the inherent high sensitivity of the vidicon tube in the study of chopped or short exposure time E.S.P.I. to study unstable objects such as the human ear drum. Also, in a paper written by Lokberg and Hogmoen\(^{(18)}\), the work of Aleksoff\(^{(9)}\) is extended to introduce phase modulation techniques into E.S.P.I. The expression developed for the intensity of an image point of the object $I(x,y)$ is the same as that of Neuman \(^{(10)}\) et al, i.e.

$$I(x,y) = I_0(x,y) J_0^2 \left[ \frac{4\pi}{\lambda} a_s^2(x,y) + a_r^2 + 2a_s(x,y)a_r \cos(\phi_s(x,y) - \phi_r) \right]$$

as $a_s$, $a_r$ and $\phi_r$ are varied, the minimum value of the bessel function argument and thus the maximum image intensity occurs for areas of constant $a_s(x,y)$ and $\phi_s(x,y)$ such that

$$a_r = a_s(x,y) \quad \text{and} \quad \phi_r = \phi_s(x,y)$$

Using this condition the authors demonstrate the advantage of the real time nature of E.S.P.I. operation to produce a much quicker method of amplitude and phase mapping of objects vibrating with a complex vibration mode. However, the visibility of higher order bessel function fringes is generally very poor with conventional time average E.S.P.I. and the reference phase wave modulation tends to
deteriorate these still further. A complete study of the vibrating surface may thus take some time, especially to identify the amplitude over a large area. In the case of a surface vibrating in uniphase, as is encountered in the study to be developed in this thesis, much more information is given and in clearer detail if amplitude strobing is used to measure phase.

Many other uses of E.S.P.I. have also been developed to rival and improve upon equivalent holographic interferometry. The real time advantage of E.S.P.I. allows a more flexible approach to the measurement of fine movements. For further reading, the following text is recommended, Jones & Wykes (19). This is one of the most recent comprehensive texts on the subject of holographic and speckle pattern interferometry.

As the aim of the work reported here was to develop an optical technique capable of yielding values of the dynamic mechanical properties, in particular bulk moduli, of elastomers, it would be useful to gain an appreciation of the difficulties involved. This may be best achieved by looking at previous techniques which have been published and at the specific nature of each.

The measurement of bulk moduli of elastomers falls into two categories. Firstly, isothermal or static - measurements in which the rate of applied stress is slow and the material is always in thermal equilibrium i.e. heat generation inside the material is minimal. Secondly, adiabatic or dynamic - measurements in which the stress cycling is high enough to prevent appreciable heat dissipation over the period of the cycle. Marvin and McKinney in their comprehensive discussion of volume relaxations in amorphous polymers (20), give the critical frequency, \( w \), above which measurements are largely
adiabatic and below which they are largely isothermal as

\[ \omega = \frac{K}{C_p \rho x} \]

where \( K \) is the thermal conductivity, \( C_p \) the specific heat at constant pressure, \( \rho \) the density and \( x \) the sample thickness. \( \omega \) is generally below 1 Hz for most polymers. They also give for the difference between \( B_s \), the static bulk compressibility, and \( B_d \), the dynamic bulk compressibility.

\[ B_s = B_d + \frac{T\alpha}{\rho C_p} \]

where \( B = \frac{1}{k} \), \( T \) is the absolute temperature and \( \alpha \) the volume thermal expansitivity.

The measurement of static bulk modulus is quite readily achieved by the application of large pressures to confined samples. Wood and Martin\( ^{(21)} \) have used a mercury filled dilatometer immersed in a temperature bath to measure the effect of pressure on the bulk modulus of natural rubber. Contractions of the sample due to applied static pressures are measured by the amount of mercury movement in a capillary. It is worth noting that in order to measure accurately the volume contraction of an elastomer requires the application of high pressures, depending on the sample volume. On this point Wood and Martin\( ^{(21)} \) and other workers such as Heydemann and Houch\( ^{(22)} \) have found that an increase in pressure has the effect of increasing the glass transition temperature of rubbers and polymers, that is, the temperature at which crystallinity begins to affect the mechanical characteristics of the material. This has the effect of changing the value of the property being measured. Jones, Parry and Tabor\( ^{(23)} \) in their review of work carried out on this aspect, provide numerous values of \( \frac{dT}{dP} \), that is the effective shift in temperature with pressure for static measurements of bulk modulus - this
of course varies with different materials but for rubbers that have been tested, the values do not exceed 0.03°C/atm., that is 30°C per 1000 atm.

A second technique developed by Matsuoka and Maxwell (24) was used to measure bulk modulus at constant rates of strain. The technique employed a precision bore and piston into which a tight fitting specimen was placed. The bore was surrounded by a heating element in order to measure temperature effects. The apparatus could be used with any suitable mechanical test machine on which forces were applied to the piston, and thus the sample pressures were calculated from piston area. The deflection of the sample undergoing pure bulk compression is measured from the travel of the piston with the aid of a dial gauge.

This type of arrangement has also been used by Warfield et al (25) in a method to measure static bulk and Young's moduli from the same sample. By making the sample a loose fit in the bore, the sample, when first compressed by the piston, is in axial compression dependent on Young's modulus E. As the sample deforms and fills the bore, it will start to undergo bulk compression dependent on the bulk modulus K. Holownia (26) has also used this method to measure the effect of carbon black as a filler on the elastic constants of elastomers.

The measurement of adiabatic (dynamic) bulk modulus K* or compliance B* = 1/k* proves to be more problematic than static K measurements. Primarily the problems arise from the large pressures required to produce measurable volume contractions in bulk compression. Techniques which use small loads relying on more sensitive measuring techniques are generally the rule.
The complex nature of a dynamic viscoelastic response requires that an additional value, the loss tangent \( \tan \delta \), also be measured. The accuracy to which this loss tangent can be measured is important in identifying the existence of bulk viscosity \( K^* \) in the response of a material.

Preliminary attempts in the measurement of \( K^* \) were fraught with problems. Philippoff and Brodnyan(27) developed a dynamic version of the mercury dilatometer for use on a dynamic mechanical test machine. The sample was immersed in mercury, enclosed in a pressure vessel and oscillating pressures were produced by the action of the test machine on a small well tolerated plunger. Calibration of the instrument was achieved by compressing the system when filled with mercury only. The displacement of the piston, measured by a strain gauge, gave a value for the compressibility of mercury. Comparison of this value with a known value gave the finite compressibility of the pressure chamber. Actual measurements were limited to frequencies between 0.0003 and 5 Hz at -25° to 95°C because of the limited dynamic range of the mechanical test machine used.

Results do not appear conclusive apart from determining that \( K'' \ll K^* \) and that an increase in temperature causes a drop in \( |K^*| \). The results given are for plastic materials but this is one of the few methods which use relatively large strains to make measurements.

An alternative technique was proposed by Marvin et al(28) involving the measurement of the propagation constants of ultrasonic longitudinal waves through a block of the test material. The longitudinal wave propagation is determined by longitudinal bulk modulus \( M^* \). The apparatus consisted of a glycerine filled tank with two pizoelectric
transducers one at either end. The test sample was placed in the glycerine between the two transducers. One transducer was then used to introduce an ultrasonic wave into the fluid and the second used to receive the signal passing through the test material. Measurement of attenuation ($\alpha$) and velocity ($C$) of the wave through the sample could be made from the comparison of the transmitted and received signal. These values were used in the following expressions:

$$M^* = M' + M'' = k^* + \frac{4}{3} G^*$$

but $M' = k' + \frac{4}{3} G' = \rho C^2 \left( \frac{1-a^2 C^2}{1+a^2 C^2} \right) \frac{1}{\omega^2}$

and $M'' = k'' + \frac{4}{3} G'' = 2 \rho C^2 \frac{(\alpha C / \omega)}{\left( 1+a^2 C^2 / \omega^2 \right)^2}$

By using values of $G^*$ obtained by previous workers they calculated $K^*$ over the frequency range $0.9 - 7 MHz$ and temperatures from $5-50^\circ$. However, using the method of reduced variables this frequency range was extended to cover $10^5 - 10^8 Hz$. The use of reduced variables to extend dynamic results is mentioned briefly later.

Results for Polyisobutylene show $K'$ to be $10$ times greater than $K''$, when $K''$ reaches its peak value. Values of $K''$ using this method are, however, open to quite large errors due to the high losses produced in shear wave propagation, the method used to measure $G''$. This is combined with errors introduced by the subtraction of $\frac{3}{4} G''$ from $M''$, both of nearly equal value. However, this technique, which has become known as the difference technique, has been successfully used by several investigators to study the frequency dependence of bulk moduli and to investigate the physical similarity, if any, between shear and bulk viscosity.
A development of this difference technique was forwarded by Kono(29). Using a rotatable slab of the test material he was able to determine both \( M^* \) and \( G^* \) by making the following measurements. At normal incidence of a longitudinal wave on the slab, generated as before by a piezo crystal in a fluid bath, no shear waves are generated in the solid. The propagation constants of the wave are therefore determined by \( M^* \) and were measured using the same technique as (28). When the plate is rotated through an angle to the source, a shear wave is also propagated through the material emerging at a different time and angle from the longitudinal wave. At a critical angle, \( \phi_c \), the longitudinal wave is totally reflected at the front face of the slab and only the shear wave is propagated. The propagation constants of the shear wave can then be measured.

A method which overcomes the problems of the difference method was described by McKinney et al(30). Using direct stress on a confined sample and measuring the volume strain produced, they were able to calculate \( K^* \) directly. The apparatus consisted of a small cylindrical pressure cell, 15cm diameter by 15cm deep, capable of holding pressures of 1000 atm. Dynamic pressures were produced by a piezoelectric ceramic transducer inside the cell filled with a light fluid in which a small sample of test material was held (400mm\(^3\)). The phase and amplitude of the resulting strains were measured by a second piezoelectric transducer also inside the cell. By ascertaining the system response to materials of known compressibility, which were believed to be constant over the temperature and pressure range used, they were able to calibrate the voltages of the second transducer. The dynamic pressures induced in the sample are of the order of \( 10^{-1} \)Pa. For the frequency range used the wavelengths of the sound waves produced were much larger than the cell.
dimensions so that no standing waves were produced inside the cell.

Subsequent work by McKinney and co workers (31)(32) using this apparatus has produced very comprehensive results of the dynamic compressibility of a natural rubber vulcanizate and Poly (vinyl acetate). Temperature variations, achieved by submerging the apparatus in a temperature controlled oil bath, were from -30° to 70°C and applied static pressures between 0 - 1000 bar were used. The range of their experimental results was increased by the use of the 'Universal' WLF equation reduced variable treatment extending the effective frequency range to cover from $10^{-2}$ to $10^{14}$ Hz and pressure range increased to -2000 to +2500 bar. These results are probably the most comprehensive available for the materials which they tested.

A final technique which has less practical use but does allow basic studies is the suspension method outlined in use by Wada and co workers (33). The method relies on the principle that particles in suspension in a fluid which are smaller than the wavelength of sound transmitted through the fluid, will undergo bulk compression as a sound wave passes through the medium. This assumes that no shear deformation occurs in the particles. This relies on a lack of shear viscosity in the fluid. Certain other conditions must be satisfied such as - the particles must be nearly spherical and must show a good affinity for the fluid used i.e. there should be no airbubbles at the material fluid interface and no swelling or dissolution of the material. For a particle size of 40µm in 1 litre of fluid, a frequency limit of 50 MHz exists, determined by the wavelength of the sound. There is also a lower limit of 100KHz. Their apparatus consisted of two quartz transducers - one used as transmitter the other as
receiver. Once again measurements are made of the velocity and attenuation of propagation. These lead directly to values of the storage and loss components of \( K^* \).

The use of reduced variables has been used by several of the authors whose work has been mentioned (27) (28) (31) (32) (33). The basis of this treatment of time dependent results from amorphous polymers, that is above their glass transition temperature, was outlined by Williams, Landel and Ferry (34). A detailed study of the theory on which this treatment stands will not be discussed here as it strays from the basic work. If the reader wishes to investigate this aspect further he is recommended to reference (35) and (20) where a detailed account of the principles involved will be found. By way of a brief explanation the basic concept of the theory relies on free volume, a theoretical concept representing the difference in material volume above and below the glass transition temperature. The dependence of relaxation (time dependent) processes of mechanical properties are presumed to be dependent on the temperature and pressure behaviour of this free volume. The effect of temperature on the measured properties is given by the Williams, Landel & Ferry (W.L.F) equation.

\[
\log \alpha_t = -C_1 (T-T_s)/(C_2 + T-T_s)
\]

where \( \alpha_t \) is the ratio between the values of a relaxation process at temp \( T \) and those at the reduced temperature \( T_s \). It was found (34) that the constants \( C_1 \) and \( C_2 \) have values of 8.86 and 101.6 for reference temperatures chosen to fit the above equation. The effects of pressure can be obtained by ascertaining a value for \( \delta T/\delta P \) as made reference to earlier (23), that is the effective shift of the glass transition temperature with pressure.
The measurement of Young's modulus $E^*$ for viscoelastic materials is on the whole much easier and much better documented. The adoption of techniques to be described in this thesis will lead to an attempt at measurement of $E^*$ by what is by comparison a rather complicated method. It is, however, the intention to develop a technique which will yield values of $K^*$ and $E^*$ from the same apparatus and test specimen much in the way that $K$ and $E$ are measured in\(^{18}\). For these reasons the reader is recommended to better texts which cover most of the many available measuring techniques and terminology of dynamic mechanical testing of elastomers.

Ferry\(^{35}\) covers comprehensively the measurement techniques as does Warner\(^{36}\). The international standard \(^{37}\) covers much of the notation and form of dynamic measurements of $E^*$ for elastomers.

The majority of the published techniques for the measurement of adiabatic bulk moduli have used frequencies in the $GHz$ region. For elastomers with glass transition temperatures near room temperature, the frequency range of interest falls between $0 - 10KHz$. McKinney et al have however used frequencies in this range. They have used small samples thus making it difficult to extrapolate to large bulk sample properties. There appears to be a need for a technique which will give results from large samples which will take account of microscopic inhomogeneities present in foam rubbers and composite formulations.
2.1 Experimental Development

As previously mentioned the holographic technique of Holownia\(^1\) was the starting point of the research reported here.

It will be useful to the reader if the developments made by research continuing directly from that of Holownia, are outlined separately from the experimental procedure used to obtain results. This will enable a better understanding of the particular problems encountered and techniques used to overcome them.

The experiment can be split into two broad areas which will incorporate the whole system, interferometric technique and sample geometry and loading.

2.1.1 INTERFEROMETRIC TECHNIQUE

Holographic recordings are sensitive to movements of the object being studied. They are also sensitive to movements of any of the optical components or holographic plate holder. Vibrational stability problems were therefore most common, especially when using large fluctuating pressures inside the pressure chamber. If the amplitude of these spurious vibrations exceeds \(\pi/4\) they will cause failure of the holographic recording. To anticipate such failure a second interferometer, a Michelson interferometer was employed to identify frequencies at which vibrations of the system were excessive. Using a mirror mounted on the side of the pressure chamber and a second mirror on the holographic plate holder, the Michelson interferometer was made
FIG 2.1.1

Holographic set-up, Holowina

Static Pressure Pot

Pressure Cell

Amplifier

Oscilloscope

Supply Pressure

Servo

Amplifier

Oscillator

Holographic Plate

Michelson Interferometer (To Monitor Vibrations)

Beam Splitter

Collimator

Electronic Shutter

LASER

Spacial Filter
sensitive not only to optical component vibration but to the mechanical stability of the subject support and plate holder.

Whilst using holographic interferometry, both reference beam phase modulation\(^{(9)}\) and amplitude strobing\(^{(6)}\) were investigated. Reference beam phase modulation proved difficult to use. The major problem was in producing a real time hologram of the object. A kinematic mount was used but emulsion shrinkage caused excessive re-location fringes. Alternative chemicals or insitu development were not tried.

Amplitude strobing, on the other hand, produced great improvement in vibrational stability of recordings. Strobing was achieved with the aid of a Pockels cell, an electro-optic device which relies on the linear change in birefringence induced by an applied electric field in a crystal exhibiting the Pockels effect. If linearly polarized light, such as that from a He Ne laser, is passed through the Pockels cell crystal at \(45^\circ\) to its optical axis, the polarization of the beam is rotated proportionally to the voltage. A polarizing filter on the output window of the Pockels cell will alter the amplitude of the emerging beam in accordance with this voltage.

Some of the interferograms produced using stroboscopic holography are shown in figures 2.1.2 and 2.1.3. These are a steel ball and a nitrile ball both pressurised in glycerine. The optical set up used to obtain these results is shown in figure 2.1.4.

A major problem encountered with strobed holography was the measurement of the phase of illumination pulses relative to the object vibration.
FIG 2.1.2  Strobbed hologram of steel ball submerged in glycerine, subjected to 6 bar pressure fluctuations at 50 Hz.
FIG 2.1.3 Strobed hologram of a NITRILE ball submerged in glycerine, subjected to 6 bar pressure fluctuations at 50 Hz.
timed to coincide with maximum and minimum pressure amplitudes. This relies on the object vibration being in phase with the pressure cycle. However, it was believed that for some of the rubbers being tested, this was not the case. A real time holographic recording can be used to identify the phase of a vibrating object relative to the forcing function (1), but this method was found difficult to reproduce as mentioned previously.

Some of the rubber ball specimens tested did not show any fringes when tested at normal loads. In order to investigate this further an electronic speckle pattern interferometer was incorporated into the previous arrangement, as shown in figure 2.1.5. With the E.S.P.I. system, it was shown that these ball samples were very sensitive to pressure and could not be tested with the hydraulic pressures which were normally used. It was also noted that using E.S.P.I., a greater range of excitation frequencies could be studied without problems arising from vibration of the optical and other components. This was partly due to the over critical response of the Michelson Interferometer which had been used to identify flat spots in the system. Also, because of the fast repetition rates at which interferograms are displayed with the E.S.P.I. system, one every 1/25 secs., the effect of instabilities, which cause failures in longer duration holographic recordings was reduced. It was also noted that by scanning the phase of the illumination pulses across the pressure cycle, the point of maximum object deformation could be identified. This led to greater development of E.S.P.I. for this application.

Using E.S.P.I. does, however, mean that frequencies below 10Hz cannot easily be studied due to the video scan rate (40 msec). This was not critical as changes in bulk
modulus with frequency have been shown to be logarithmic\(^{(34)}\) and the frequencies of interest at this early stage were towards 1kHz.

The effects of pressure on refractive index of the submerging fluid have been mentioned in the previous chapter. As a result of these effects, a new technique to E.S.P.I., but not to holography\(^{(37)}\), was investigated. The technique involves the use of an object localised reference mirror which enables the phase changes due to refractive index variations to be annulled. This technique will be described in more detail later and a theoretical consideration given in Chapter 3.

2.1.2 SAMPLE GEOMETRY AND LOADING TECHNIQUE

Sample geometry determines the sensitivity to loading and sample support determines the type of stressing which will result from loading.

(i) Sample Geometry

The relative incompressibility of rubber samples meant that the size of spherical sample chosen by Holownia required large pressures to produce measurable radial strain. Using larger spherical samples, which would require less pressure to compress, was not feasible because of the difficulties found in the production of homogeneous samples of the smaller size used. Figure 2.1.6. shows the effect of air bubbles beneath the surface of a ball and an example of an unsymmetrical fringe pattern produced by larger inhomogeneities. Both of the measuring techniques employed, holography and E.S.P.I., measure surface movement in the direction of observation. In order to sensitise the volume deformation to pressure, volumetric strain can be limited
FIG 2.1.6 Holograms showing inhomogeneities in rubber samples, due to moulding faults.
to localised surface contraction, thus introducing a greater surface movement for the same volume strain.

The sample geometry and support chosen with this property was a cylinder restrained from movement on all surfaces except one of its circular ends. The central deflection of such a cylinder face is approximately eight times the radial contraction of a sphere of comparable size subjected to the same pressure. A mathematical analysis, using finite difference techniques, showed that the main stress induced in such a cylinder when its free end was exposed to pressure was triaxial, apart from minimal shear stresses induced in the bound edges of the free surface.

The physical representation of this geometry, a rubber cylinder glued or moulded into a metal pot, provided much greater rigidity than was found with the spherical samples. They required much smaller pressures enabling higher frequencies to be studied and were easier to mould without inhomogeneities.

This sample geometry was also modified to allow bulk modulus specimens to be tested with uniaxial stressing allowing measurements of dynamic Young's modulus $E^*$ to be made. Removing the support from the other circular end of the cylinder and applying differential pressures across these exposed ends forced the sample to act as a thick diaphragm, the deflection of which is dependent upon $E$.

(ii) Loading Technique

The main criterion for bulk compression is triaxial loading. This was achieved for spherical samples by immersion in a pressurising fluid. Several fluids were
chosen - glycerine, transformer and mineral oil because of their transparent properties and relatively high bulk moduli. A pressure cell was required to confine the fluid and the cell needed an observation window because of the optical measuring techniques employed.

The design of the cell was changed several times over the period of the research. The original cell had a large internal volume to accommodate a change in sample size. It also had a small observation window to prevent flexure. Subsequent work showed that the large internal volume caused problems with fluid flow, due to fluid compressibility. The observation window has been enlarged to afford better viewing angles, window flexure being minimised by using thicker glass.

Static pressures were applied via a dead-weight piston. Dynamic pressures were generated by using a hydraulic power pack together with a servovalve, to produce sinusoidal fluctuations of pressure. Isolation of this equipment from the optical table produced major stability improvements. A servovalve with higher frequency response improved control of the pressure waveform.

The sample mount clamping arrangement was also changed. The previous cells relied on the clamping of the observation window to provide a restraint for the sample mount. This was sometimes inadequate and could also cause problems with window breakage. A metal back plate now clamps the sample mount against a metal butt giving more rigid support while allowing the window to remain fixed.

Modifications to this cell and sample holder were also made to allow the sample to act as a very thick diaphragm. A differential pressure across this diaphragm produces small deflections from which a value of Young's modulus can be calculated.
3. THEORY

Outlined in this chapter is the theory used to determine surface deflection from E.S.P.I fringe data. Particular emphasis will be placed on the simplification of data extraction by the use of modulation techniques in E.S.P.I. Interpretation of the deflection shape, by computer analysis of the fringe data, is shown to yield values of volume change in the sample.

The theory applies to a study of a submerged viscoelastic body undergoing static and sinusoidal volume contractions due to applied pressures in the submerging fluid. The measuring technique used is E.S.P.I. The theory derived for study using holographic interferometry has been given previously(1). Elements in this chapter are derivations from this original theory.

3.1 Light scattered from a submerged sample undergoing pressure induced volume contractions.

![Figure 3.1.1](image-url)
The free surface of the rubber cylinder AA, in Fig 3.2.1, characterised in cylindrical polar co-ordinates by $Z = 0$, $r = 0 - R$, $\theta = 0 - 2\pi$, is assumed when subjected to pressure to form the surface A'A' being characterised by $Z = Z(r \theta)$. For an isotropic material the deflection is symmetrical in $\theta$ and thus $Z$ may be expressed as $Z = Z(r)$.

Assuming normal illumination and viewing, the phase advance of laser light wavelength $\lambda$, travelling the distance $H$ to the surface and back through a medium of refractive index $n$ will be given by:

$$\phi = 4\pi nH/\lambda$$

The distance $H$ being given by:

$$H(r) = L + Z(r) \quad (2)$$

Comparing two states, the first unpressurised having conditions of pressure $P = P_0$ and refractive index $n=n_0$ with the second pressurised state $P = P_0 + \Delta P$ and $n=n_0 + \Delta n$.

Assuming differential changes in state we have

$$d\phi = (\delta \phi) \; dn + (\delta \phi) \; dz$$

$$\begin{align*}
(\delta n) & \quad (\delta z) \\
= 4\pi \; (Hdn + ndz) \\
\lambda
\end{align*}$$

$$d\phi = 4\pi \; (H \; dn + n \; dz)$$

$$dP \quad \lambda \quad dP \quad dP$$
3.1.1 EFFECTS OF PRESSURE ON REFRACTIVE INDEX

Consider the term $dn/dP$ (in equation 3) expressing the pressure dependence of refractive index. As mentioned, Holownia (1) makes use of the Gladstone Dale relationship to express this term in the following way:

$$\frac{n-1}{\rho} = \frac{n_0-1}{\rho_0} = C_{GD} = \text{Constant}$$

so

$$\frac{dn}{dP} = \frac{(dn)}{(\partial \rho)} \frac{(dn)}{(\partial \rho)} = \frac{(n-1)}{(\rho_0 \partial P \rho_0 \partial V)}$$

thus

$$\frac{dn}{dP} = \frac{(n_0-1)}{K_0}$$

Where $V_0$ is the initial volume of fluid and $\partial V$ the differential change due to pressure change. $K_0$ is the bulk modulus of the fluid.

The alternative Lorenz-Lorentz equation can be used to arrive at the following expression:

$$\frac{(n^2-1)}{(n^2+2)} \frac{1}{\rho} = \frac{(n_0^2-1)}{(n_0^2+2)} \frac{1}{\rho_0} = C_{LL} = \text{constant}$$

again

$$\frac{dn}{dP} = \frac{(dn)}{(\partial \rho)} \frac{(dn)}{(\partial \rho)}$$

here

$$\frac{dn}{dP} = \frac{(n_0^2+2)(n_0^2-1)}{6n_0} \frac{1}{K_0}$$

In this form both expressions (4) and (5) are linear as $n_0$ is constant. However, the gradient of equation (5) is 14% higher than that of equation (4) for a refractive index of 1.47 (that of glycerine).
In the following consideration the Gladstone Dale derived expression, equation (4), will be used as it has provided accurate results for the static bulk modulus of glycerine \(^{(1)}\). It is believed, however, that a compromise between the two expressions would provide a better approximation.

So substituting (4) in equation (3) and multiplying through by \(dP\) we have:

\[
\frac{d\phi}{\lambda} = 4\pi \left[ H(n_o-1) \frac{dP}{\lambda} + n_0 dz \right] K_o
\]

integrating this expression we obtain:

\[
\Delta\phi = 4\pi \left( L (n_o-1) \frac{dP}{\lambda} + n_0 Z 0 \right) - 6 K_o
\]

Here, \(Z(r)\) has been ignored in the expression for \(H(r)\) equation (2) due to the relative sizes of \(L \) and \(Z\).

\[L = 3 \text{ mm} \quad Z=6 \mu m\]

It is worth noting that equation (5) indicates that both the refractive index term and the surface displacement term are positive. This means that an increase in pressure will result in an increase in path length due to the surface moving away from the window and due to the refractive index change.
3.2 Static Pressure Changes

The application of a static pressure change $\Delta P$ to the submerging fluid, observing the surface using subtraction ESPI.

If we consider the initial intensity on the camera face plate to be given by:

$$I_1 = I_s + I_r + 2\sqrt{I_s I_r} \cos \psi$$

which is stored on the E.S.P.I. frame store, the intensity on the camera face plate after the application of an applied pressure $\Delta P$ will be given by:

$$I_2 = I_s + I_r + 2\sqrt{I_s I_r} \cos (\pi + \Delta \phi)$$

where $\Delta \phi$ is given by equation (6).

It has been shown by Jones & Wykes (19) that the resultant brightness on the TV monitor will be given by

$$B = C \left[ I_r I_s \sin^2 (\psi + \frac{1}{2} \Delta \phi) \sin^2 \frac{1}{2} \Delta \phi \right]^{1/2}$$

where $C$ is a constant defining the system electronic response.

This gives rise to dark and light fringes across the screen where a dark fringe is characterised by a minimum value of $B$ for which

$$\Delta \phi = 2N\pi \quad N \in {0, 1, 2, 3}$$
hence from equation (6) we have for a fringe order $N$ observed on the TV monitor.

\[ N = \frac{2 \pi L (n_0 - 1) \Delta P + n_0 Z(r)}{\lambda K_0} \]

The locus of constant $N$ for an isotropic material will be concentric circular fringes.

### 3.3 Dynamic Pressure Changes

If the sample is subjected to sinusoidal pressure variations of frequency $f$, the surface of the sample will undergo approximate S.H.M. where:

\[
\Delta P = P_0 \sin \omega t
\]

\[
Z(r) = Z_0 \sin (\omega t - \delta) \quad \omega = 2\pi f
\]

Here $\delta$ represents the phase delay or loss angle of the elastomer response.

Thus equation (6) becomes

\[
\Delta \phi = \frac{\pi L (n_0 - 1) P_0 \sin \omega t + n_0 Z_0 \sin (\omega t - \delta)}{\lambda K_0} - \delta
\]
3.3.1 TIME AVERAGED E.S.P.I.

Jones and Wykes (16) show that the resultant intensity of the time averaged speckle pattern on the camera face plate can be expressed as

$$I = I_g + I_r + 2\sqrt{I_g I_r} \left(J_0 \left(\frac{4\pi A}{\lambda}\right)\right) \cos \psi$$

$J_0$ being a zero order Bessel function and $I_r$ and $I_g$ the separate intensity of the reference and object wavefronts.

When processed by the ESPI electronics and displayed on a monitor this resultant intensity will correspond to a brightness given by

$$B = C \left[ \sigma_g^2 + \sigma_r^2 + 2\langle I_g \rangle \langle I_r \rangle J_0 \left(\frac{4\pi A}{\lambda}\right) \right]^{1/2}$$

where $\sigma$ is the standard deviation of the object and reference intensity and $\langle I \rangle$ the mean value of the intensity.

In this case $A$ is given by the Cosine rule and is the resultant of the two phase terms in equation (8).

i.e. $A = a^2 + b^2 - 2ab \cos \delta$

where

$$a = L \left(\frac{n_o - 1}{K_o}\right) P_o$$

$$b = n_o Z_o(r)$$

From a single fringe pattern it is impossible to identify the two unknowns $Z_o$ and $\delta$ so the fringe field is insoluble.
3.3.2 STROBOSCOPIC E.S.P.I.

Now consider the case where the laser beam is strobed to produce a train of short pulses at twice the frequency of the pressure signal, with a relative phase altered to coincide with peak and minimum surface displacements. Assuming the frequency of the pressure signal to be higher than 10Hz (defined by the persistence time of the camera tube, 0.1 sec), the resultant fringe brightness becomes that for addition ESPI.

$$B = C \left[ \sigma_g^2 + \sigma_r^2 + 2 \langle I_g \rangle \langle I_r \rangle \cos^2 \frac{1}{2} \Delta \phi \right]^{1/2}$$

The fringe brightness is not zero for dark fringes but contrast does not fall for higher fringe orders as with time averaged ESPI. So we have from equation (9)

$$B_{\text{MAX}} = C \left[ \sigma_g^2 + \sigma_r^2 + 2 \langle I_g \rangle \langle I_r \rangle \right]^{1/2}$$

when $$\Delta \phi = N\pi$$ where $$N = 0,1,2,3$$

$$B_{\text{MIN}} = C \left[ \sigma_g^2 + \sigma_r^2 \right]^{1/2}$$

when $$\Delta \phi = (2N + 1) \frac{\pi}{2}$$ where $$N = 0,1,2,3$$

therefore from equation (4) we have for a bright fringe

$$N_r = 2 \left[ L(n_0 - 1) \Delta P + n_0 Z(r) \right] \frac{k}{\lambda \eta} \quad \text{N} = 0,1,2,3$$

i.e. $$Z(r) = \frac{1}{n_0 \frac{1}{2}} \left[ N_r \frac{\lambda}{k} - L (n_0 - 1) \Delta P \right] - \frac{10}{n_0 \frac{1}{2}} k_o$$

$$N_r$$ being the fringe order at $$Z(r)$$
Z(0), is the deflection at the centre of the sample and is always the maximum. As Z(0) increases so the fringe radii increase as they move away from the sample centre. If the phase of the strobing is altered, the fringe diameters will increase or decrease depending on the positions being sampled by the subsequent illumination pulses. Fringe diameters will be maximum when illumination pulses occur at minimum and maximum deflection points. Thus, the phase delay between pressure signal and illumination pulses can be used to measure the loss angle.

Thus equation (10) can be used to define the shape of the deformed surface. This is assuming however that Ko is known at the particular frequency and static pressure and does not exhibit viscoelastic behaviour. Results have shown that both hydraulic oil and glycerine, used as submerging fluids, exhibit frequency dependent bulk moduli changes and both have a viscous component to their response. This behaviour introduced errors into the calculation of Z(r), and into the values of d in particular, due to a second phase term introduced in Ko. These errors are increased by the use of an assumed expression for dn/dP in equation (3).
Now consider the case of the object localised reference mirror shown in fig. 4.2.9. Here the reference beam and object illumination travel very similar paths through the submerging fluid. The reference wave must therefore contain a term identical to that of the refractive index term in equation (8)

\[ U_r = U \cos (\varphi_r + L (n_o - 1) \frac{P_o \sin \omega t}{K_o}) \]

The combination of object and reference waves will result in an intensity.

\[ I_t = I_s + I_r + 2\sqrt{I_s I_r} \cos (\varphi_s + \Delta \phi - \varphi_r - L (n_o - 1) \frac{P_o \sin \omega t}{K_o}) \]

or

\[ I_t = I_s + I_r + 2\sqrt{I_s I_r} \cos (\varphi + n_o Z_o(r) \sin (\omega t - \delta)) \]

where \( \varphi = \varphi_s - \varphi_r \)

If the object and reference beam are once again strobed over a short period of the cycle at \( 2\omega \) and at a phase delay, the resultant monitor brightness will be given by

\[ B = C \left[ \sigma_s^2 + \sigma_r^2 + \langle I_s \rangle \langle I_r \rangle \cos^2 \left( \frac{2\pi n_o Z_o(r)}{\lambda} \right) \right] \]

\[ \lambda \]

for a bright fringe order \( N_r \)

\[ N_r = \frac{2 n_o Z_o(r)}{\lambda} \]

\[ Z_o(r) = N_r \frac{\lambda}{2n_o} \]
This expression is used together with values of $\delta$ measured as described later from the oscilloscope traces, to describe the elastomer bulk response $K^*$. To achieve this the fringe data is taken directly from the screen and used in a computer analysis of this and other data.
3.4 Assumption of Spherical Deflection

Assuming that the surface A'A' can be considered as forming part of a spherical surface radius \( R \), when deformed with a central deflection \( h \). Consider an elemental volume

\[ \delta V = \pi y^2 \delta x \]

where

\[ y^2 = R^2 - x^2 \]

Integrating over the deflection from \( X = R - h \) to \( X = R \) to give the volume change

\[ \Delta V = \pi \left[ \int_{R-h}^{R} (R^2 - x^2) \, dx \right] \]
\[ V = \pi \left[R^2x - x^3\right] R \]
\[ l \quad 3 \quad R - h \]

i.e.

\[ \Delta V = \pi Rh^2 + h^3 \]
\[ 3 \]

h is very small in comparison to R and hence the \( h^3 \) term may be ignored.

\[ \Delta V = \pi Rh^2 \]

but \[ R = h + \frac{r^2}{2} \quad 2 \quad 2h \]

\[ \Delta V = \pi h^3 + \pi r^2h \]
\[ 2 \quad 2 \]

once again ignoring \( h^3 \) terms in relation to \( r^2 \) we have

\[ \Delta V = \pi r^2h \]
\[ 2 \]

the original volume of the cylindrical sample is

\[ V = 2\pi r^2l \]

therefore, the volumetric strain

\[ \frac{\Delta V}{V} = h \]
\[ \frac{2l}{V} \]
3.5 Computer Analysis of Fringe Data

The calculation of volume changes in the elastomer depends heavily on the exact shape of the surface deformation. Information about this shape is given by the fringes which contour the surface due to the equally spaced fringe field; the fringe separation being given by either equation 7,10 or 11 depending on technique.

The fringe field observed on the ESPI monitor is mapped from the screen to give information to sample image diameter D and fringe order diameters dN. The normalised fringe diameters, dN/D, are used to perform the volume integral.

\[ \Delta V = \pi \int_0^Z r^2 \, dz \]

by making the following numerical summation

\[ \Delta V = \pi R^2 \sum_{N=0}^{N_c} (d_N/D)^2 \, \delta N \]

where \( N_c \) is the central fringe order and \( R \) the actual sample radius.

Taking in particular the case of strobed E.S.P.I. using the O.L.R.M., \( \delta N \), the fractional fringe spacing is corrected using equation 11 to give the true depth \( z \). Thus, the numerical integration is corrected for system magnification effects and the fringe orders converted to actual deflections. The volume change calculated in this way corresponds to a pressure of \( 2P \cos \delta \). This is because the strobing is in phase with deflection and is lagging the pressure by \( \delta \). The bulk modulus is calculated using the simple expression
\[ K = \frac{2P \cos \delta}{\Delta V/V} \]

where \( V \) is the original volume. The assumption is made that \( V \) corresponds to an undeformed volume. This is not true if the sample is subjected to a static pressure of 20 bar. The volume difference, however, due to the high values of \( K \) encountered, is negligible.
4. EXPERIMENTAL EQUIPMENT AND PROCEDURES

Section 2.1, in which the development of experimentation was outlined, gave details of the advancement of original work. In this section the experimental procedures which constitute the derived methodology are described together with a more detailed description of the equipment design and usage.

4.1 Measurement of Bulk Modulus

Initially consideration will be given to the various methods of sample preparation and support used, followed by details of the pressure cell and associated instrumentation used to apply static and dynamic pressures to the sample. This pressure cell sample arrangement was incorporated into two main optical arrangements. These will be described with particular emphasis on the techniques used to simplify and manipulate the E.S.P.I. fringes produced in order to make displacement and phase measurements more accurate and repeatable. The electrical control and monitoring of the experimentation will be discussed. In particular the form of operation with regard to control paths and monitoring requirements, together with a description of the procedure used to obtain surface displacement fringes and displacement time delays for static and dynamic pressure loading.

4.2.1 PREPARATION OF SAMPLES

All of the specimens of rubber for which results are given in the results section, except for nitrile rubber used to obtain preliminary results, were supplied by the Ministry of Defence. They were
supplied either compression or transfer moulded into metal pots or in sheet form from which billets were cut and turned to size. The cut samples were glued using epoxy resin based adhesives into the same type of metal pots. The exact form of these sample support pots is shown in figure 4.1.1. This type of sample support was used for bulk testing only. Insufficient results were obtained to investigate the effect of the glue on the sample response. Cast samples did, however, tend to produce more symmetrical fringe patterns. Glued samples were also difficult to produce without including small air pockets between the rubber and the metal support. Several adhesives and techniques were tried using, epoxy based adhesives with a small hole drilled in the rear of the metal support to allow the escape of air, proved to be the most reliable.

In an attempt to derive a sample configuration which could be tested in both bulk compression and uniaxial tension, the sample support was changed to the configuration shown in fig. 4.1.2. The use of this type of sample support in uniaxial tension will be described in a later chapter.

Two options were available in order to use open-ended samples (fig.4.1.2) in bulk measurements. Firstly, the sample could be used with equal pressures applied to both free surfaces and thus subject the material to triaxial compression. To achieve a reliable result from this kind of loading, it would be necessary to rely on both surfaces taking on equal displacement and simply take twice the value measured on one surface. This would depend on equal conditions applying on both surfaces which, in practice, was very difficult to achieve.
FIG 4.1.1 Sample supports into which rubber samples were glued or moulded.
Section A-A

THREAD
Φ 33 PITCH?

4 CLEARANCE
M3 ALLEN SCRS.

2 HOLES Φ6
ON Φ28 IN
LINE WITH
5mm SLOTS

4 M3 HOLES
ON Φ33
X 6 DEEP

SAMPLE HOLDER AND BACKING PLATE
Material: MS | Scale: 2x F.S. | 9.12.83 | Drawn by. A. ROWLAND

FIG 4.1.2 Open ended sample support used in both uniaxial and bulk compression measurements (used with backing plate).
An alternative method was to alter the sample so as to obtain conditions similar to the original sample support. To arrive at the configuration previously used for bulk samples (fig. 4.1.1), a backing plate was secured to the rear of the sample as shown in Figures 4.1.2 and 4.1.3. This allowed the sample to be used as an ordinary bulk sample.

Great care was taken to ensure that the rear surface of the samples and the mating surface of the backing plate were flat. This was achieved by grinding both surfaces and then polishing them to a 1/2 mirror finish. Problems occasionally arose when some rubber samples were treated in this manner as they were preferentially removed by the grinding and polishing process. This resulted in a hollow surface which allowed air into the union. The problem of trapped air in these samples was seen to be a major problem and is discussed further in a later chapter.

All samples, whether tested in bulk compression or uniaxial tension, had their front faces, this is the surface which is viewed by the E.S.P.I. system, flat ground. This ensured that the unpressurised surface profile was known. The front faces were then sprayed with a thin layer of white cellulose based paint to increase the scattering effect of the surface.

Later samples were also prepared with a thin piece of front silvered mirror which was glued using cyonacrylate adhesive to the metal support surrounding the front face of the rubber (fig. 4.1.4). This mirror was used in the reference arm of the E.S.P.I. system in order to remove the effects introduced by refractive index changes in the submerging fluid. This operation will be discussed in more detail later.
FIG 4.1.3 Open ended sample supports showing conversion of uniaxial samples, left, to bulk samples, right, by addition of a backing plate.
FIG 4.1.4  Ground and painted front face of the sample with a small mirror attached to the sample support, bottom right.
Once prepared, the sample could be secured into the pressure cell for testing.

4.1.2 PRESSURE CELL AND HYDRAULICS

(i) Pressure Cell

The pressure cell used in the majority of experimentation consisted of three components. The cell itself, fig.4.1.5 (a), is a thick walled cylinder arrangement which helps minimise side wall movements.

The cell body has three access holes through which various functions can be performed. Two are arranged so as fluid may enter into the cell, either to the top of the bore or to the bottom, through which fluid may also be drained. A third hole enters the cell at the centre by means of an inclined bore through which pressures can be monitored. All channels are sculptured so as to allow the easy passage of air bubbles to the top of the cell where they can escape. The cell has four feet at its base so it could be securely bolted to the T sloted machine bed which served as an optical table. At the front of the central bore an 8mm lap was left against which the sample holder (fig.4.1.5(b)) was butted. This sample holder was designed to hold the sample support (fig. 4.1.1) rigidly inside the pressure cell bore. For this reason it was toleranced to be a good sliding fit inside the bore.

The depression in the outer diameter of the sample holder is designed to distribute fluid pressure evenly around the sample support. The
Front and back surfaces ground.

NOTE! All dimensions are given in mm.

FIG 4.1.5 a) Pressure cell.
varying sized holes radially spaced around the holder were designed to introduce pressures evenly into the inner area where the sample is secured. A small location pin insured that the sample mount was held in the correct orientation inside the cell bore. Once held by this location pin, the sample support could be screwed into the sample holder by means of a specially designed spanner using holes in the rear of the sample support (fig. 4.1.1 and 4.1.2). Once the sample was in place the whole assembly could be clamped by the backing plate (fig. 4.1.5(c)). The backing plate incorporates in its design a sculptured grove which runs across the pressure cell bore and allows pressures at the rear of the sample support to be monitored through the central access hole (see fig. 4.1.5(a)).

The length of the backing plate was such that when clamping the sample mount firmly, with a thin gasket between the two, the back plate formed a good seal against a rubber 'O' ring on the pressure cell body. This arrangement provided ample support for the sample and sealed the cell against leakage.

A 25mm thick glass observation window was also secured to the front face of the pressure vessel using a metal ring secured by the six bolts together with a rubber 'O' ring seal. Tests using E.S.P.I. to establish the flexure of this window due to normal test pressures showed that there was minimal movement with the clamping used. The window was not removed when replacing test samples as they were removed from the rear of the cell.
FIG 4.1.5 c) Pressure cell backing plate used in bulk compresion tests to clamp sample holders.

FIG 4.1.5 b) Sample holder used in bulk compression tests.
Once the sample was secured the cell was filled with glycerine from the static piston. A settling time of about two hours was required for the glycerine to become still and for air bubbles to rise to the top of the cell. This settling time was much shorter when transformer oil was used as the submerging fluid.

(ii) Hydraulics

As mentioned previously, the cell body has three access holes to the central bore. Two of these were used to introduce hydraulic pressures into the cell. The access hole at the bottom of the bore was connected to a dead weight piston arrangement as used by Holownia, to apply static pressures. The diameter of the piston could be changed from 10mm to 18mm to make the application of smaller pressures easier.

The top fluid input was connected to a hydraulic servo valve which was used to apply sinusoidal pressures inside the cell. The feed pressure for the valve was generated by an 11 tooth gear pump which was set to supply a constant 40 bar pressure. A pump ripple of typically 2% was inherent with this supply at a frequency of 55 Hz. By restricting flow through the cell, this ripple could be smoothed slightly but when applying light pressures the ripple was of similar magnitude to the desired pressure signal. This restricted the minimum pressures which could be applied.
The internal pressure which was produced by the presence of the hydraulic supply pressure inside the cell, was monitored via a dial gauge on the top input to the cell. This static pressure was generally 20 bar when the valve was balanced and working with a 40 bar feed pressure. The zero position of the servovalve spool could be controlled electrically to vary the static pressure inside the cell independently of the dynamic pressures. A fall off in valve frequency response was incurred in doing so. Lower frequencies were thus used to assess the changes in K due to applied static pressure. Dynamic pressures inside the cell were monitored by using a pizoelectric pressure transducer device.

The pressure cell, complete with sample hydraulics and electrical connections, is shown in fig.4.1.6. The cell is shown bolted to the optical table where it forms part of the optical system.

4.1.3 OPTICAL SYSTEM

The pressure cell was structured into the overall system as shown in fig. 5.7. The optical layout shown is that of a typical correlation speckle pattern interferometer using a hole in the mirror E.S.P.I. head. The laser used was a Spectra Physics 124 He Ne laser. Measured light outputs were between 19 and 21 mW making it a class 3b laser. The laser was aligned with a KD *P crystal Pockels cell and polarizing filter such that the light output could be modulated to produce square wave illumination pulses of variable mark space ratio. This was used to produce an amplitude strobing effect. The modulated beam was
Pressure cell and hydraulics. Pressures can be applied to the sample, centre, either statically by dead weight piston, right, or dynamically by servohydraulic valve, centre top. Pressures inside the cell were measured using the dial gauge, top right, for static pressures up to 40 bar and dynamically by piezo-electric pressure transducer and amplifier, left.
FIG 4.1.7 Overall system configuration showing the pressure cell and hydraulics incorporated into the optical and electrical control systems.
then split by a 1° wedge beam splitter, the first reflection being used as the E.S.P.I. reference beam, the second as a signal for a photodiode light meter, used to analyse the beam modulation. The continuing beam was spatially filtered and used to illuminate the rubber sample.

The E.S.P.I. optical head used was a hole in the mirror type. The hole refers to a 25μm hole in the mirror which is placed at 45° behind the imaging lens of the head. The reference beam is expanded just behind this mirror and is spatially filtered through the pinhole. This arrangement helps to insure axial alignment of the reference beam with light scattered from the object which has been gathered by the imaging lens. This alignment is necessary to satisfy the conjugacy requirements of E.S.P.I. (reference 16 Chap. 4).

A Link video camera fitted with a newvicon tube was positioned to produce a sharp picture of the rubber surface. The video signal of the phase referenced speckle pattern was processed in two different ways depending on whether E.S.P.I. subtraction or time averaged fringes were required. The basic electronic processing consists of high pass filtering to remove cross screen illumination variations. These are usually produced by the gaussian intensity distribution of the laser beam. The video signal is then differentiated and full wave rectified to simulate square law detection.

The hardware used is shown in fig. 4.1.8. The small pieces of tissue under certain optical components acts as a gasket between the magnetic bases of the components and the table. This improved the
FIG 4.1.8 Optical hardware including laser, Pockels cell, beam splitter, mirrors, spatial filters, neutral density filter and hole in the mirror E.S.P.I.
vibrational stability of the optics which was of prime importance in improving fringe contrast.

Because of the complex behaviour of the pressurising fluids used (glycerine and mineral oil), both exhibiting a viscoelastic response, errors are introduced into measurements of rubber bulk modulus $k^*$ and loss angle $\delta^*$. This is due to the inclusion of a fringe shift due to the refractive index changes of the submerging fluid. This can be removed by calculation but accuracy suffers if fluid properties change. A more accurate result can be obtained by using a technique, developed from Mottier (37) for use in E.S.P.I. called here object localised reference mirror.

(i) Object Localised Reference Mirror (O.L.R.M.)

E.S.P.I. works on a comparison of two beams of the same laser light. Normally one of these beams is modulated by movement of an object and the difference between it and an unmodulated reference are examined. In the case of the submerged sample, there are two distinct modulations which affect the object beam:

1. modulation due to surface movement of the elastomer sample;
2. modulation due to pressure-induced refractive index changes in the pressurising fluid and any window flexure.

It is only the sample movement which is of interest. The refractive index changes are merely a noise function which must be removed. This can be achieved
by calculating out the effect. If, however, the modulation due to refractive index change is introduced in to the reference beam also, then when the two beams are compared, only the modulation due to the sample movement will be identified. This was readily achieved by placing a mirror on the sample support (fig.4.1.4). The mirror is incorporated into the reference arm of the E.S.P.I. as shown in fig.4.1.9. Having passed through the pressurising fluid twice at the same angle as the object illumination, the reference beam will have become modulated by the refractive index changes in exactly the same way as the object beam. The resulting fringe patterns are then due only to movements of the sample surface. The loss angles measured correspond to these surface movements and do not include effects due to viscoelastic fluid properties.

Problems were encountered with this technique if localised changes in refractive index occurred near the sample mounted mirror. Glycerine is an inhomogeneous liquid and is also immiscible with mineral oil so, on occasions when both fluids were used in the cell, localised inhomogeneities were caused by areas of varying refractive index. The result of this was the introduction differential refraction of the reference beam causing loss of fringe contrast.

For this reason, together with the added ease of using mineral oil to fill the pressure cell, all tests using the localised mirror were carried out in mineral oil. This, however, limits the frequency response of the valve due to the greater flow produced by bulk contraction of the fluid.
FIG 4.1.9  Detail of the optical beam path when using the object localised reference mirror (O.L.R.M.)
4.1.4 ELECTRICAL CONTROL AND MONITORING
(see figs. 4.1.7/9/10)

In order to produce phase related strobos, it was necessary to provide a signal which was phase related to the signal used to drive the hydraulic servovalve. These signals were provided by a dual output variable phase generator (V.P.G). The strobing signal was first used to provide a reference frequency for a double pulse generator D.P.G. A signal was produced from the (D.P.G) such that the pulse frequency was twice that of the driving frequency and the mark space ratio was about 1:5. This signal was then fed to a high voltage amplifier used to drive the Pockels cell. The Pockels cell was aligned using fine x, y and z adjustments so as its optical axis was at 45° to the laser beam polarisation and the beam was passing through the centre of the crystal. Electrical control was thus established using the V.P.G., the fixed output driving the servo valve amplifier and the variable output driving a double pulse generator.

Various phase shifts occurred through the control paths before the drive signals were reproduced as either pressure or illumination fluctuations. The Pockels cell control path had a fixed phase relationship with respect to the input signal, introduced by the double pulse generator - approximately \( \pi/4 \) rads. The servo-valve, because of its inductive nature, had a variable phase response depending on the frequency of the input signal. It is, in fact, the increase of the inductive load at higher frequencies which ultimately limits the valve frequency response. This variable phase delay made it impractical to use the drive signals, as signals. Monitoring of both final signals was, therefore, necessary.
FIG 4.1.10 Electrical control and monitoring equipment. Consisting of variable phase and double pulse generators, twin dual beam oscilloscopes and Pockels cell amplifier, right. E.S.P.I. monitor and processing electronics, left.
The pressure inside the cell was detected by a piezoelectric pressure transducer, the signal from which was amplified. Both pieces of equipment were supplied by Kistler Instruments who quote the response time of the crystal and amplifier as around 6 µsec for full scale deflection of 250 bar (170 kHz maximum response). Each transducer is supplied with its own calibration number which, if dialled into the amplifier unit will give a linear calibrated response over the working range of the transducer. Sensitivity changes allow different values of Volts/bar to be given as an output signal.

The illumination modulation was monitored by a silicon photodiode and amplifier. The response time of which could only be judged by monitoring the phase delay between initial signal and measured response, knowing that both the double pulse generator and voltage amplifier were capable of high kilohertz if not megahertz response. From this it was found that the phase shift between input and measured signal remained fixed over the range of frequencies used in experiments.

Both signals were subsequently monitored on a dual beam oscilloscope, which was calibrated and was used to measure dynamic pressure levels directly as a voltage. The oscilloscope traces shown in fig.4.1.11a are typical of those observed during experimentation.

Illumination/pressure cycle phase variations could be observed by monitoring the two signals on the oscilloscope (see fig.4.1.11a and b). Where such variations occurred the two signals were displayed on a second oscilloscope, one signal displayed on the
x-axis the other on the y-axis of the screen, this produced a Lissajous figure of the two waveforms (fig.4.1.12). This provides a clearer indication of the extent of the phase difference between the two signals allowing a nulling of the phase difference for measurement purposes.
a) Pressure and strobing in phase at 500 Hz. Oscilloscope trace, time scale 1/125 sec per div at 1 Volt/bar.

b) Pressure and strobing 12 deg. out of phase scale as above).

FIG 4.1.11 Oscilloscope traces showing pressure signals, sinusoidal traces, from the pressure transducer and strobing signals, pulsed trace, from the photodiode.
FIG 4.1.12 Lissajous figures of the traces shown in fig 4.1.11 a) and b). Pressure signals are displayed on the time axis and illumination signals on the y axis.

a) Pressure in phase with strobing (see fig 4.1.11 a).

b) Pressure leading strobing by 12 deg. (see fig 4.1.11 b).
4.1.5 STATIC BULK MODULUS MEASUREMENTS

Static loadings were applied as described earlier, to either an 18mm or 9mm diameter plunger. Pressures were calculated from load over area.

Static pre-stressing was used to measure certain static values in order to eliminate fringe irregularities due to small flaws in the bond circumference or any air bubbles trapped during preparation. A full study of the effect of pre-stressing was also made. Measurements were taken from E.S.P.I. subtraction fringes observed on a TV monitor. The fringes were generated by surface movement caused by a known pressure.

Fringe positions, relative to the sample diameter, were marked on a paper strip directly from the monitor screen. Every half fringe was marked. The fringe positions and calculated pressure, together with sample depth, were used in the computer analysis to calculate a value of K.

In order to compare values obtained by this method the same samples were tested using the mechanical compression method outlined in (26). In order to utilise the potted cylindrical samples a special piston and barrel arrangement was used, fig.4.1.13. The samples were secured into the barrel by the clamping force of the loose plate. The piston was then forced using an Instron mechanical test machine. The resulting displacements were measured by an extensometer placed across the gap between extension bars. K values were calculated from piston displacement and applied pressure readings.

In order to obtain E.S.P.I. subtraction fringes a video frame store is required. This equipment was not available during long periods of the experimentation. This has
a) Barrel showing sample clamped in position.

b) Piston in position. For accurate measurements an extensometer could be placed across the 10 mm gap.

FIG 4.1.13 Static bulk compression tester for use with potted cylindrical samples.
meant that only limited results have been taken to show that the method is capable of yielding results for isothermal values of bulk modulus.

4.1.6 DYNAMIC BULK MODULUS MEASUREMENTS

Dynamic pressures were applied via the servovalve, as described previously, controlled by one output of the variable phase oscillator. The pressure signal from the cell was displayed on the oscilloscope; all dynamic loadings were superimposed on top of a $2 \times 10^6$ Pa static pressure unless otherwise stated.

Measurements were made with the aid of strobed, phase modulated E.S.P.I. fringes viewed on a video monitor. The timing of the laser strobing was altered until fringe diameters reached a maximum indicating that strobing was occurring at peak and minimum distortion. The fringe positions were then marked on a paper strip directly from the screen. Every half fringe was marked and their positions measured. Pressure values were read from the oscilloscope screen. This information, together with sample depth, was used in computer analysis to calculate values of $K^*$. Due to the loss component of bulk modulus of some of the rubbers tested, the strobing was not always in phase with the pressure cycle. The phase difference was measured using the second oscilloscope as described previously (see fig.4.1.11). This phase angle was measured using the dial indicator on a variable phase generator.

4.1.7 TEMPERATURE DEPENDENT MEASUREMENTS

A redesigned backing plate used for uniaxial measurement (fig.4.2.4) has incorporated in it a 15 Watt heater coil capable of raising the cell temperature by 20°C at a rate
of 2°C per hour. This is sufficiently slow to allow measurements to be made while the heater is running without introducing large energy inputs to the sample during these measurements. The sample temperature was measured indirectly by a thermocouple which, although immersed in the glycerine and not in contact with the sample, provides an adequate reading given the small thermal gradients present in the cell. The bulk deflections were measured as before, the temperature being measured by the thermocouple probe.

4.1.8 VOLUMETRIC STRAIN. CALCULATIONS

Calculation of the volume change represented by the fringe data was originally achieved by assuming a spherical deflection shape. Using only the central fringe order, volume changes were easily calculated.

Computer analysis has shown that the shape of the deflection is a complex function of $K$ and $v$ and varies with small changes in $v$. Figures 4.1.14 a,b and c show the dependence of shape on $K$ and $v$, keeping $E$ constant. Order of magnitude changes in central depth are due to changing values of $K$, due to the relationship

$$K = \frac{E}{3(1-2v)}$$

The calculation of the volume change depends heavily on the exact shape of deformation. Information about the deflection shape is given by the E.S.P.I. fringes which produce a contour pattern on the surface due to the equally spaced fringe field. So fringe spacings across the sample can be used to generate a shape curve by least squares polynomial curve fit. The generated curve is then used to carry out a numerical volume integration to calculate the volume change.
FIG 4.1.14 Effect of Poisson's ratio on deflection shape of exposed cylinder end, keeping E constant (results obtained by computer numerical analysis).
4.2 Experimental Determination of Young's Modulii

4.2.1 INITIAL CONSIDERATIONS

The measurement of Young's modulus has been achieved in many ways, some of which are outlined by Ferry (35). The form of measurement chosen here was determined by the following considerations:

i) the requirement that the samples used be similar in dimensions to those used in bulk modulus measurements. This was to maintain the scale of physical effects in the determination of both moduli;

ii) the strains involved should be of similar magnitude to those used in bulk modulus experiments: firstly because of the limited range of the optical measurement technique and secondly to maintain continuity between the two sets of experiments;

iii) to utilise essentially the same equipment in both sets of measurements in order to produce a more compact experimental unit.

This would not, of course, lead to the most effective method of measurement of E* but would validate the combination of values of E* and K* measured in this way.

4.2.2 THEORETICAL CONSIDERATIONS

Given the bulk sample configuration, fig.4.1.1, the loading needed to produce a deflection strongly dependent on E, was chosen to come from the front and rear of the sample. In order to achieve this the metal supporting the rear of the sample had to be
removed thus allowing free movement of the rubber along the loading axis,

FIGURE 4.2.1: Open-ended sample with metal backing plate removed, showing loading axis.

This arrangement can be considered as a thick plate, radius a and thickness t, rigidly supported around its circumference

$$W = wTa^2$$

FIGURE 4.2.2: Simplified analogy of thick plate rigidly supported around its circumference.

The maximum central deflection $y$, of such a plate due to a uniform load per unit area $w$ is governed by a simple relationship involving the material constants $E$ and $v$.
This relationship holds true for $t < a/2$ but in the case of the rubber sample $t > a/2$ and the expression defining the central deflection becomes a more complicated function of $E$ and $\nu$.

\[ y = \frac{3W (1-\nu^2)a^2}{16 E t^3} \]

\[ y = f(E, \nu, P) \]

FIGURE 4.2.3: $y$ the deflection of the front surface resulting from shear and longitudinal extension.

It was envisaged that the complications in the dependence of $y$ upon $E$ could be overcome by matching a measured deflection with one calculated by a finite difference method. Estimated values of $E^*$ and $\nu$, linked through equation (4.2) to $K^*$, would be used in an iterative process which will eventually lead to values of $E^*$ and which satisfy both the measured bulk modulus and central deflection.

\[ K = \frac{E}{3(1-2\nu)} \]
i) **Practical Solutions**

By applying a differential pressure across either end of the rubber sample and mount (see Fig.4.2.1) the required axial loading could be obtained. This was achieved using the existing pressure cell and hydraulics. The modifications required to make this possible have been outlined briefly already but will be covered here. The resulting central deflections were measured, using E.S.P.I., in the same way as bulk deflections. The phase angle allied to the delay between surface displacement and loading was also measured using techniques outlined for bulk experiments. Calculations based on equation .4.1 have been used to give an indication of the trends observed so far. Insufficient time was available to develop a full iterative technique based on finite difference methods.

ii) **Equipment Modifications**

Most of the equipment utilised for uniaxial measurements was used for bulk modulus experiments also. A major change, however, was the backing plate, see Fig.4.1.5(c), redesigned to provide a second input to the pressure cell, Fig.4.2.4. A nozzle arrangement distributes the incoming pressure pulse evenly without restricting flow. A recess in the backing plate, inside the bore, enabled a heating coil to be wound onto the backing plate. This can provide approximately 15 Watts of heating power, capable of raising the cell and sample temperature to around 40°C if required.

The sample holder in which the sample and mount are held was also redesigned, see Fig.4.2.5, to distribute
Alternative backing plate for pressure cell. Used in uniaxial tests to apply separate pressures to the rear of the sample.
Sample mount designed to allow different pressures around the sample support, in front, distributed from the circumference and behind, delivered from the cell backing plate of fig 4.2.4.
the top pressure input in the cell (Fig.4.1.5(a)) to the front of the sample only. Thus the front and rear portions of the cell can be separated enabling different pressures to be supplied to each side of the sample.

4.2.3 EXPERIMENTAL LIMITATIONS

i) Loading

To produce a forced sinusoidal response in the rubber, it was necessary to introduce pressure to both open faces of the sample. This was achieved by taking advantage of the dual output capacity of the servovalve already used. The pressures were brought into the cell as outlined by Fig.4.2.6

FIG.4.2.6
Cut away section through pressure cell showing pressure distribution.
However, problems were encountered in isolating the two areas of the pressure cell. Leakages around the sample holder and the fact that the sample acts as a diaphragm between the two pressurised areas made precise pressure control difficult. Also, the hydraulic power pack used generated a pump ripple on top of the pressure signal. This ripple was of the same order of magnitude as the dynamic pressures intentionally introduced. These factors made the proposed loading almost impossible to achieve.

A compromise was made which allowed results to be obtained and which gave a qualitative measure of the rubber response. Results were obtained by pressurising from the rear of the sample only. This, however, relies on the rubber rebounding under its own stored energy, creating a forced-free loading situation. This introduces more hysteresis than would normally be expected from push-pull loading.

ii) Sample Failure

The rubber is subjected to complex stressing when loaded axially. Under test conditions the shear stresses induced in the material were generally small. If, however, the bond between the sample and the metal mount was weak, these shear stresses caused failure of the bond rendering the sample unusable. From testing several sets of samples, it appears that samples which have not been moulded in the mount or samples which do not wet the surface of the metal sample supports during vulcanisation, are particularly prone to failure. This results in part, or all of the circumferential bond between sample and support failing, which makes the sample unusable for further testing. A glueing procedure was developed involving
the use of cyano-acrylate and epoxy resin adhesives which allowed these samples to be repotted.

4.2.4 RESULTS PROCEDURE

The test samples used were glued or moulded into the open-ended sample mount (Fig.4.1.2). The mount was secured into the pressure cell and hydraulic oil used to apply pressure to the front or rear of the sample only. The front surface was either exposed to air or submerged in oil.

It was generally found that better results were obtained if oil was used in front of the sample. The oil could then be used to raise the static pressure on both faces of the sample. Static pressures were applied using the deadweight piston and dynamic pressures via a hydraulic servovalve. The front surface of the sample was observed by the E.S.P.I. system. All deflections and pressures were measured in the same way as those observed in bulk modulus experiments. The loss angle of the deflection, that is the phase delay between the pressure cycle maximum and deflection cycle maximum, was measured in the same way as the phase angles observed in bulk modulus measurements.

Values of $K^*$ for the samples were measured using the technique outlined in section 4.1. An iterative technique was used to find the combination of $E$ and $\nu$ linked by equation (4.2) to $K$ which when substituted in equation (4.1), gave the central deflection which was measured experimentally.
5. DISCUSSION OF RESULTS

This chapter presents results obtained over the whole period of experimentation. Some preliminary results are used to show how the measurement of bulk modulus was improved upon. The changes in procedure made to bring about these improvements will be discussed.

The final procedure arrived at will be examined in terms of its effectiveness in reducing the complexity of measurements. Results obtained using this final procedure are presented which describe the frequency, pressure and temperature dependence of dynamic bulk modulus for various rubbers. These will be used to discuss the accuracy of the measurement technique. All results were obtained using E.S.P.I. unless otherwise stated.

Measurements of dynamic Youngs modulus are also discussed. Although limited in extent, these are used to describe the method of characterisation of elastomer properties.

5.1 Preliminary Results

i) **Measurement of sample surface deflection shape**

These are preliminary results used to highlight the rationale behind the final procedure used to obtain results in section 5.3.

During early work it was assumed that the free surface of the rubber samples, fig.4.1.4, took on a spherical profile when subjected to pressure loading. Volume strains were therefore calculated using the approach outlined in 3.4 using the central fringe order. Table 1 (results Appendix 1) shows a set of results for various rubbers obtained using this approach.
The accuracy of these results depends on the accuracy with which the central fringe order is read. This was of the order of 1/2 a fringe, giving an accuracy of about ±10%, depending on the total number of fringes. This total number was limited by poor fringe visibility.

The true deflection shape of the rubber surface is defined by a complex function of K & ν. A spherical assumption provides a poor approximation of this shape. The fringe spacings were therefore used to give a contour map of the deflected surface – see sections 3.4 & 3.5. This provided a much better evaluation of the volume change and hence the bulk modulus.

Figure 5.1.1 shows three curves representing the measured deflection shape for three different rubber mixes. These were generated by a polynomial least squares curve fit using measured radial fringe positions for fringe orders N=0, 0.5, 1.0 etc. These deflection shapes exhibit a similar trend to calculated values obtained by finite difference techniques, figure 4.1.14 a,b and c (values of ν for W203 A,C and E are around 0.4998). The diagrams show that initial curvature is high at E/R=1.0, and as E/R → 0 the profile becomes almost flat. (N.B. Fig. 5.1.1 'c' represents a different frequency to figures a and b).

Figure 5.1.2 shows curves of K* versus log of frequency for W203 A,C and E obtained using this contouring technique. Table II shows these results, along with comparison values of K*, calculated using central fringe order and assumed spherical deflection. Values of loss angle δ are also given. These were measured using the method described in Chapter 4.
FIG 5.1.1 Sample surface deflection shape under triaxial compression (experimental results).
FIG 5.1.2 Dynamic results for W203A, C, E (table II results)
ii) Effect of Superimposed Pressures

Discrepancies were noticed in values of static bulk modulus for nitrile rubber measured using the optical technique and mechanical testing (1).

The mechanical testing was carried out on the optical samples using the compression test rig shown in figure 4.1.13 together with an Instron tensile test machine. Similar test methods have proved reliable in the past (1) (26) (25).

The observed discrepancies were believed to be due to the amount of prestressing the samples were subjected to during measurements. In the case of mechanical testing this is the stress required to produce a measurable displacement and in the optical case is due to the static pressures unavoidably present during some measurements.

In order to investigate this comparative tests were made making note of the prestressed pressure in the sample.

Table III shows the results of this investigation. It can be seen here that mechanical and optical techniques both show large changes in measured values of $K$ for increasing static pressure. However they show good agreement with each other at the same pressure - the previous anomalies can thus be explained by the existence of discrepancies in static pressure at the time of measurement.

A set of results was then obtained for Norsorex rubber, keeping static pressure constant at 20 bar and varying frequency. These results are shown in fig.5.1.3. The procedure was then altered so that frequency was kept constant and static pressure varied over the range 10 to 30 bar. These results are shown in fig.5.1.4. This
FIG 5.1.3  

$K^-$ versus Log of frequency for two samples of NORSOREX rubber (static pressure kept constant at 20 bar).
FIG 5.1.4 $K^*$ versus superimposed static pressure for NORSOREX rubber at two frequencies
latter curve shows how large variations in $K^*$ can occur over a small range of static pressures. Failure to monitor and keep static pressure constant, would obscure any frequency dependent changes in $K^*$.

iii) **Effect of viscoelasticity of submerging fluid**

To compare the effect of different submerging fluids results are given in Table III for $K^*$ values measured with a sample of nitrile rubber submerged in transformer oil. The differences between these values and those obtained with the sample submerged in glycerine are within experimental errors observed in measurements at this time.

Observations were then made of the optical behaviour of the glycerine from static to dynamic conditions. A study carried out using a steel ball submerged in glycerine, as described by Holownia (1), produced the results shown in fig.5.1.5.

The static value of $K_o$ (see Table V) obtained from experiments agrees with published values (Kay & Labey). Values of $K_o^*$ show no particular trend. It would be reasonable therefore to assume a mean value of $5.15 \pm 10\%$. If, however, the values obtained at an unknown static pressure are ignored, a value of $K_o^* = 5.03 \pm 7\%$ results. This is at a static pressure of 20 bar. This figure is 17\% higher than that for the static value $K_o$. This corresponds to the difference between isothermal and adiabatic measurements Marvin & McKinney (20) give, typically in the region of 20\%.

Of importance in these results is the large variation in the viscous component of $K_o$, defined by the angle $\delta$. In fig.5.1.5. some of the $S$ values are plotted as negative, although the actual angles **must** be positive.
FIG 5.1.5 $K^*$ versus Log of frequency for glycerine, used as submerging fluid
The illumination pulses used to determine $\delta$ repeat at intervals of $\pi$ radians, with respect to the pressure cycle. The negative plotted values of $\delta$ could therefore represent, realistically, $(\delta+\pi)$ or $(\delta+2\pi)$.

Values of $\delta>\pi/4$ are highly unlikely in bulk response. Causes for such a response were considered. As mentioned the monitoring system, through which illumination pressure signals were detected, was not suspected of error. A remaining explanation could have been a time related difference between the pressure at the transducer and at the front surface of the steel sphere. This was dispelled by considering the wavelength of bulk waves at the highest frequency used. This can be calculated using the expression:

$$\lambda = \frac{1}{f} \sqrt{\frac{K_0}{\rho_0}}$$

For glycerine at 1KHz $\lambda = 1.5m$, this would preclude any standing waves being formed in the cell as the major cell parameters are very much smaller than 1.5m.

This anomaly, together with the errors introduced from an assumed expression for $dn/dp$ in equation 3, prompted investigation into ways of removing the effects of the submerging fluid from the fringe field.

Reference wave phase modulation provided a way of achieving this. Using an object localised reference mirror (O.L.R.M.) specific phase modulation could be introduced into the reference wave. This modulation technique together with amplitude modulation was used to obtain the remainder of the bulk moduli results which will be discussed in detail in section 5.3.
5.2 Optical Modulation Techniques

The effectiveness of the modulation techniques applied to the interferometer can be judged by observing fig. 5.2.1. This is a fringe pattern obtained using an O.L.R.M. and amplitude strobbing. The fringe contrast is high and the metal support surrounding the rubber sample free of fringes.

By observing the fringes as they form due to increasing pressure it is observed that no fringes move over the support. In terms of E.S.P.I. addition fringes, this means that no phase shift has occurred over this area, i.e. the phase modulation introduced by the O.L.R.M. cancels the phase shift which causes the dark outer ring in Figure 5.2.2. An excellent illustration of this action is shown in Fig.5.2.3 in which two almost imiscible fluids cover the rubber sample. The lower level through which the reference beam passes is glycerine and the upper layer hydraulic oil. The boundary between the two layers can easily be seen, defined by the change from light to dark of the outer metal support. The small dark area to the bottom right shows an inhomogeneity in the glycerine causing localised loss of fringe contrast.

The theory shows (Chapter 3) that for a submerged viscoelastic body, the phase term $\Delta \phi$ is complicated and for time averaged E.S.P.I. leads to an insolvable fringe field. Amplitude strobing improves fringe contrast because $\cos^2$ fringes suffer no reduction in contrast as do $J_0^2$ fringes. $\cos^2$ fringes also have equally spaced solutions.

Strobing allows measurement of the loss angle $\delta$ of the samples viscoelastic response. This on its own is not sufficient as results obtained using strobing only lead to
FIG 5.2.1 Strobed E.S.P.I. fringe pattern obtained using an O.L.R.M. and amplitude strobing. The fringe discontinuity, bottom left indicates non isotropic behaviour.
FIG 5.2.2  Strobed E.S.P.I. fringe pattern obtained without using an O.L.R.M.
FIG 5.2.3 Strobed E.S.P.I. fringe pattern obtained with two immiscible fluids covering a rubber sample. The O.L.R.M. is located on the left in the lower fluid.
unpredictable results. This is due to the included fringe shift due to the refractive index changes in the submerging fluid.

The bulk modulus of the fluid $K_0$ is a viscoelastic quantity having a viscous component which introduces a second phase term $\phi_0$ into the phase term $\Delta \phi$. Using an O.L.R.M. to introduce reference beam phase modulation, tailored to remove the refractive index shift, means that the pressure dependent term $\partial n/\partial P$ in eqt 3 disappears leaving a simplified term (Equation11). This makes the measurement of $\phi$ and $Z$ easier and more accurate.

5.3 Dynamic Bulk Modulus

In order to validate the experimental technique outlined for the measurement of dynamic volume viscoelasticity the following sections give some of the results obtained. This will also help to define the scope of the measurement technique.

The results given were obtained using strobed E.S.P.I. with reference wave phase modulation, using an O.L.R.M. A brief discussion of the trends exhibited by the results and of the possible errors in these values will be given.

5.3.1 FREQUENCY DEPENDENT MEASUREMENTS

The frequency range over which measurements were made was 50-1000 Hz. Values of $K^*$ were obtained with a superimposed static pressure of 20 bar. Dynamic pressures of less than 1 bar were used to make measurements. Results are shown for several rubber mixes which were supplied by the M.O.D. These rubbers will be designated by the following names:- ES700, EP25, NITRILE (KRYNAC 25-50), NEOPRENE/NITRILE, NORSOREX (as cast), NORSOREX
(cut from block material) and KRYNAC. Results are tabulated in Appendix 1. Values of $K^*$ have been plotted against log of frequency along with loss angle $\delta$ in figures 5.3.1, 2, 3, 4, 5 and 6 respectively.

Referring to the results of McKinney et al (24) (26) and (30), the characteristics of a glass transition due to increasing frequency are (i) a smooth transition between two almost constant levels, from a lower to higher value of $K^*$; (ii) a peak in the loss angle $\delta$ centred at a frequency in this transition region.

Results for EP25, Fig.5.3.1, seem to be leaving just such a transition region. Values of $K^*$ rise from around 3.0 GN/m$^2$ at 50Hz to level off at 3.8 GN/m$^2$ at 700Hz. Values of $\delta$ seem to peak at about 600Hz but this peak does not appear to be centred on the transition region of $K^*$.

Values of $K^*$ for Nitrile (KRYNAC 25-50), Fig.5.3.2, also seem to form part of a transition region. This time, however, at the beginning of a transition. $K^*$ is almost constant at 3.7 GN/m$^2$ from 50 to 100Hz then rises sharply and continues to do so until the highest measured frequency of 500Hz. The frequency was limited here because hydraulic oil was used as the submerging fluid. Large pressures could not be generated at higher frequencies due to the oil's low bulk modulus.

Values of $\delta$ show an uncharacteristic double peak, the second of which appears to coincide with the transition in $K^*$. This double peaked response in $\delta$ seems to be repeated by results for Neoprene/Nitrile, Fig.5.3.3. Values of $K^*$, meanwhile, rise slowly with no perceptible trend. There are no indications in the discussion of Marvin & McKinney (37) of such a response for elastomers but no comparable results are available for NITRILE rubbers.
**FIG 5.3.1**  $K^*$ versus frequency for ES700 and EP25 together with $\theta$ for EP25
FIG 5.3.2  $K^*$ and $\delta$ versus frequency for two samples of NITRILE(KRYNAC 25-50)
FIG 5.3.3 $K^*$ and $\delta$ versus frequency for two samples of NEOPRENE/NITRILE
FIG 5.3.4 $\kappa^*$ and $\delta$ versus frequency for NORSOREX (as cast).
FIG 5.3.5 \( K^* \) and \( \delta \) versus frequency for NORSOREX (cut from solid).
FIG 5.3.6  \( K^* \) versus frequency for KRYNAC
(\( \theta \) values all 0).
Results for Norsorex (as cast), Fig.5.3.4, do not show any trend in $K^*$ as values all lie within experimental error. $\delta$ on the other hand shows a peaked response similar to that of a transition.

$K^*$ values are also constant with a slight upward trend for Norsorex (cut from solid), Fig.5.3.5. The values are somewhat higher than for the 'as cast' sample, Fig.5.3.4. $\delta$ values, however, show a great deal of scatter. They have been represented as a curve but the trend appears to be random. Once again little exists in published results of other elastomers to indicate this as a true rubber response. Norsorex (as cast) and Norsorex (cut from solid) are essentially the same material except that the solid sample contains less air as voids in its microstructure. This does not help explain the variations in $\delta$ of Fig.5.3.5.

Finally, results for KRYNAC, Fig.5.3.6., show an upward trend in $K^*$ similar to that which might be expected at the start of a transition. $\delta$ values, however, are zero over the whole range which is unusual given the behaviour of for all the previous rubbers. This is the only rubber not to have a peak in $\delta$. More significantly, all other curves show a peak in $\delta$ between 400 to 800Hz regardless of whether this response would be expected from changes in $K^*$. The explanation for this must lie in the inclusion of an added response in the viscous response of the rubber. This would most reasonably be explained by air trapped in the sample mount.

5.3.2 PRESSURE DEPENDENT MEASUREMENTS

The effect of pressure on static values of $K$ has been discussed briefly in 5.1. Here the effect of superimposed static pressure on dynamic $K^*$ values will be
discussed in greater depth. A similar group of experiments to those in 5.3.1. were carried out for EP25 NEOPRENE/NITRILE and NORSOREX (as cast) this time observing the effect of static pressure over the range 5-40 bar, on K* values at various fixed frequencies. EP25, Fig.5.3.7., demonstrates a typical response of a rubber near its glass transition increasing pressure corresponding to an increase in temperature. At 50Hz the effect of an increase in pressure is to cause a sharp increase in K*. At higher frequencies, 300 and 500Hz, K* has already reached a steady value, see Fig.5.3.1., so increasing pressure produces little change in the value of K*.

Values of θ also show a typical trend with values decreasing away from a peak value at the centre of a transition region. They do not appear, however, to be centred on the same frequency as for the results shown in Fig.5.3.1. This is particularly significant as has been discussed in the previous section and is believed to indicate the action of trapped air in the frequency results. Figs.5.3.8 and 5.3.9 show a somewhat different phenomenon being exhibited by NEOPRENE/NITRILE and NORSOREX respectively. Both show values of K* at two frequencies over a range of static pressures. The frequencies chosen are close together and K* values are similar at constant pressure, see fig.5.3.3 and 5.3.5. The curves show a similar trend to that of EP25, Fig.5.3.1, in that a transitional increase in K* occurs with increasing pressure. However, K* curves, Fig.5.3.8 and 5.3.9, are also very similar in trend. This would tend to suggest that the changes in K* with changing pressure are due mainly to air trapped in the sample mount. This behaviour is better demonstrated by results for NORSOREX (as cast) Fig.5.3.9, and NORSOREX (cut from solid), Table. As explained this is basically the same
FIG 5.3.7 \( K^* \) and \( \phi \) versus static pressure for EP25 at three frequencies.
FIG 5.3.8  $K^*$ and $\delta$ versus static pressure for NEOPRENE/NITRILE at two frequencies
FIG 5.3.9  $K^*$ and $\delta$ versus static pressure for NORSOREX (as cast) at two frequencies
material but the sample cut from solid contains less air as voids in its microstructure. For the cast sample $K^*$ varies greatly with static pressure as does $\delta$. For the solid sample $K^*$ hardly changes with increasing static pressure.

This explanation equally applies to results for KRYNAC, Table XII where $K^*$ values once again change very little for increasing static pressure. Both NORSOREX (cut from solid) and KRYNAC show little sign of air content. A discussion of the effect of air content on frequency and pressure dependent measurements is given in Section 5.5.

5.3.3 TEMPERATURE DEPENDENCE OF BULK MODULUS

It can be seen that the bulk modulus of NORSOREX alters little over the temperature range studied, Table XIII. The total temperature rise was 16°C starting from 22°C. Over this region it is not possible to identify a trend in the results. To extend the temperature range further it would be necessary to cool the cell first, using liquid nitrogen or solid CO$_2$. This should extend the range to cover at least 50°C.

5.3.4 EFFECT OF AIR INCLUSIONS ON BULK RESULTS

Air can be present in a test sample either due to inclusions or as voids. The inclusions within the bulk of the material may be formed during the moulding process due to low mould pressures or out gassing during curing. The voids will usually occur around the sample, between it and the sample mount.

The presence of this air in microscopic quantities is believed to alter significantly the measured value of $K^*$ and $\delta$. This behaviour can be seen in results obtained
for the pressure dependence of $K^*$ for the two samples Norsorex (as cast), fig. 5.3.9, and Norsorex (cut from solid), Table XI Appendix I. As cast the sample is likely to have a higher air content than the sample which was taken from a large slab of the solid material. This is due mainly to the greater experience of the M.O.D, who prepared both samples, in the production of the slab material. It has been mentioned that these results show marked differences with regard to the effect of changes in static pressure.

From other experimentation it was noted that to ensure minimal air content between the backing plate, fig. 3.2.2, and the sample, the two mating surfaces should be given a half mirror finish. This procedure was carried for Krynac.

The results obtained from this sample show high stability and little variation with changes in static pressure. The material was cut from a large slab and should have minimal air inclusions.

5.3.5 EXPERIMENTAL UNCERTAINTY

The errors introduced in the measurement of bulk modulus by the optical technique are (i) reading errors in the radial fringe positions; (ii) systematic and reading errors in the pressure signal; (iii) calculation errors introduced by assumptions in the theory.

(i) The diameter of every half fringe is measured from the camera monitor. Any magnification effect introduced by the E.S.P.I. imaging system and subsequent electronic signal processing is removed by using normalised radial positions. Normalisation is achieved by measuring the sample diameter from the monitor. Data is later rescaled by the true sample diameter.
Measurement of the fringe diameter is accurate to 2.5%. This figure becomes more important as the fringe diameter approaches that of the sample. Here deflection depths have a much greater effect on volume change. However, more fringes are present in this region due to the deflection shape. Here the polynomial curve fit is less dependent on the accuracy of individual fringe data points. However, in the worst case this positional inaccuracy will lead to a 5% error in the value of K.

(ii) Systematic errors due to inaccuracies in the pressure sensing device are small. The calibration point of the pressure transducer was set by the manufacturer and the linearity of the response is to within 1%. Reading errors are higher due to the method of reading peak to peak pressure signals from the oscilloscope screen. These errors were in the region of 5%. This leads to an uncertainty of around 6% in the value of K calculated from these pressures.

(iii) Calculation errors introduced by assumptions in the theory were minimised by using the O.L.R.M. technique previously described. This removed inaccuracies due to an assumed expression of for the submerging fluid. Other assumptions made are that the observation window does not deform and that the sample surface is initially flat.

The window flexure was tested using an opaque window with E.S.P.I. and was found to be minimal for the pressures used. The sample on the other hand does not deform from an initially flat surface. This is due to the static pre-load applied to the sample.
This, however, only introduces a possible error of around 0.1% into the value of K.

These errors combine to give a possible error in the value of K of 11%.

5.4 Dynamic Youngs Modulus

Results were obtained, using the method described in section 4.2, for the dynamic Youngs modulus of several rubbers over the frequency range 50 to 100 Hz. These results are tabulated along with the bulk modulus results for the same rubber. Also tabulated were values of \( v \) the poissons ratio derived from the equation

\[
K = \frac{E}{3(1-2v)}
\]

These results show some promise. Although insufficient study has been made to verify the dynamic results, static results are in agreement with other quoted values. Further work is required in order to remove errors introduced by unequal loading and to make improvements towards forced excitation rather than the forced-free excitation used on these results.

5.4.1 EXPERIMENTAL UNCERTAINTY

An analysis of the uncertainties introduced by reading errors indicates that the technique is stable and does not have a strong dependence on the accuracy of any one measurement.

There is a possible uncertainty in the estimation of central deflection of 1/4 of a fringe. This will result in a possible error in E of \( \pm 6\% \) when calculated in the above manner. Correspondingly there is an uncertainty in \( v \) the fifth decimal place of \( \pm 0.00005 \). 11% error
in the measured value of $K$ will result in minimal errors in $E$ but will cause $v$ vary in the fourth decimal place by $\pm 0.0002$. Errors in the measurement of $\sigma$, determined by repetition of readings, can be said to be within $\pm 4^\circ$ of the given value.
6. CONCLUSIONS

The aim of this work has been to develop an interferometric technique for the optical measurement of static and dynamic bulk and Young's moduli for elastomer samples.

The initial holographic technique has been shown to have grave limitations in the continuous study of dynamic events where amplitude and phase modulation techniques are to be employed. E.S.P.I. on the other hand shows itself to be well suited to this application, allowing more information to be gained in a shortened time span and providing a muchmore versatile measuring technique.

The use of amplitude modulation and phase modulation has proven very effective in the manipulation and extraction of fringe information. Amplitude modulation, because of the simple vibration mode studied, is capable of improving fringe contrast, at the same time making subsequent calculations less complex. Measurement of viscous losses were also made possible by using amplitude modulation.

Phase modulation, a powerful tool in vibration analysis, is shown to have major advantages in the reduction of fringe field complexity. By incorporating selective elements of the object induced phase variations into the reference arm of the interferometer, it is shown that the fringe function can be simplified, thus further reducing the complexity of the subsequent calculations.

Static and dynamic bulk and Young's moduli have been measured for various rubber mixes. These results are used to discuss the effects of frequency, temperature and pressure on the moduli of elastomers, especially those with glass transition temperatures near room temperature.
Results have also shown that both static and dynamic values of bulk modulus of elastomers are affected greatly by increases in static pressure. This is also shown to be the case in comparative mechanical tests. A similar trend was noted by McKinney et al.(31,32).

This behavior is thought in part to be due to the inclusion of air in the test samples. The presence of air in samples is shown to dramatically influence the measured values of static and dynamic bulk moduli. This finding is significant in as much as most manufactured elastomeric components are likely to contain air introduced in the forming process. It was found that less air was present in those samples which were cut from solid than in those cast into sample supports.

In final conclusion it is felt that this work offers insight into the use of temporal modulation techniques in interferometry. These modulations can be used to reduce fringe field complexities and reduce noise from unwanted object beam phase perturbations.
7. SUGGESTIONS FOR FURTHER WORK

An area where further work could be of benefit would be in the collection of a comprehensive set of frequency dependent results for comparison with results such as those of McKinney et al \(^{(31)}\). This would enable a comparison of the technique with a similar method.

It would also be of interest to investigate further the viscoelastic response of the submerging fluids. The aim of the work reported in this thesis has been to circumvent these behaviours by employing optical techniques. However the small amount of data collected seems to suggest an anomaly which should be addressed for the sake of certainty.

Concerning the definition of surface profile which has been shown to be critical in this measurement technique. An improvement could be achieved with the use of fringe stepping techniques recently described by Robinson (see footnote 1) for E.S.P.I. fringe analysis. Reference phase stepping can be used to improve the resolution of fringe fraction by allowing interpolation between fringe positions. Holographically and for Moire fringes this has been used to resolve 1/50 to 1/100 of a fringe. E.S.P.I. fringe patterns present more of a problem because of the less defined fringe centres. However with even 1/30 of a fringe resolution the surface contouring could be improved dramatically.

The technique used for the determination of dynamic \(E^*\) values also requires further work. This work should be aimed at producing a finite difference analysis of the rubber deformation given the uniaxial loading conditions defined in the experimental details. This treatment could then be used to predict surface deformations from various combinations of \(E^*\) and which are derived from the equation:
\[ K = \frac{E}{3(1-2\nu)} \]

\( K \) having been previously determined from experimentation. The actual deflection shape measured from the sample under uniaxial loading could then be matched to the computed shapes to provide the true combination of \( K \), \( E \) and \( \nu \).

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Tabulated Results Appendix I

The following results were obtained over the period September 1982 - September 1984. Unless otherwise stated, the results were obtained at room temperature taken to be 22°C. Pressures given are gauge pressures not true pressures.

Constants used.

\[ k_0 \text{ the static bulk modulus of glycerine} \]

\[ k_0 = 4.06 \times 10^9 \text{ N/m}^2 \text{ (Kay & Labey)} \]

\[ \text{refractive index of glycerine} \]

\[ \text{No} = 1.47 \text{ (Manufacturers' published figure)} \]
<table>
<thead>
<tr>
<th>SAMPLE REF</th>
<th>PREPARATION</th>
<th>FREQ. (Hz)</th>
<th>DYNAMIC LOADING (N/m²)</th>
<th>No. AT</th>
<th>K x 10^9</th>
</tr>
</thead>
<tbody>
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<td>ES700</td>
<td>Sample Cut</td>
<td>0.0</td>
<td>1.3 \times 10^5</td>
<td>7.5</td>
<td>3.28</td>
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<td></td>
<td>from cast</td>
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<td>7.0</td>
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<td></td>
<td>bar and</td>
<td>50.0</td>
<td>&quot;</td>
<td>7.5</td>
<td>3.28</td>
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<td></td>
<td>glued using</td>
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<td>7.0</td>
<td>3.5</td>
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<td>epoxy resin</td>
<td>500.0</td>
<td>&quot;</td>
<td>6.5</td>
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<td></td>
<td>adhesive</td>
<td>900.0</td>
<td>&quot;</td>
<td>6.0</td>
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<td>As cast</td>
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<td>1.3 \times 10^5</td>
<td>7.5</td>
<td>3.28</td>
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<td></td>
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<td></td>
<td>6.5</td>
<td>3.77</td>
<td></td>
</tr>
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<td>6.0</td>
<td>4.08</td>
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<tr>
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<td>6.0</td>
<td>4.08</td>
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<td>1.41 \times 10^5</td>
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<td>&quot;</td>
<td>5.0</td>
<td>4.2</td>
</tr>
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<td>&quot;</td>
<td>5.0</td>
<td>4.2</td>
</tr>
<tr>
<td>SAMPLE</td>
<td>FREQ Hz</td>
<td>FREQ.</td>
<td>LOAD</td>
<td>CENTRE</td>
<td>K x 10^3</td>
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<tr>
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<td>---------</td>
<td>-------</td>
<td>-------</td>
<td>---------</td>
<td>----------</td>
</tr>
<tr>
<td>W203A</td>
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<td>0</td>
<td>0.19</td>
<td>3.0</td>
<td>0.60</td>
</tr>
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<td></td>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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<td>1.87</td>
<td>6.5</td>
<td>3.12</td>
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<td>2.0</td>
<td>1.55</td>
<td>5.7</td>
<td>3.40</td>
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<td>1.55</td>
<td>5.5</td>
<td>3.38</td>
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<td>1.87</td>
<td>5.7</td>
<td>3.56</td>
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<td>1.55</td>
<td>4.6</td>
<td>3.90</td>
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<td>1000</td>
<td>3.0</td>
<td>1.55</td>
<td>3.8</td>
<td>4.46</td>
</tr>
<tr>
<td>W203C</td>
<td>0</td>
<td>0</td>
<td>1.25</td>
<td>3.8</td>
<td>3.58</td>
</tr>
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<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>1.7</td>
<td>1.55</td>
<td>4.5</td>
<td>3.9</td>
</tr>
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<td>1.55</td>
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<td>3.79</td>
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<td>4.5</td>
<td>3.88</td>
</tr>
<tr>
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<td>650</td>
<td>2.81</td>
<td>1.87</td>
<td>4.5</td>
<td>4.3</td>
</tr>
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<td>700</td>
<td>2.85</td>
<td>1.87</td>
<td>4.8</td>
<td>4.5</td>
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<td>1.87</td>
<td>4.6</td>
<td>4.7</td>
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<td>1.87</td>
<td>3.7</td>
<td>4.89</td>
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<td>W203E</td>
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<td>25</td>
<td>1.4</td>
<td>1.25</td>
<td>3.8</td>
<td>3.5</td>
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<td>5.5</td>
<td>3.81</td>
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<td>1.87</td>
<td>5.0</td>
<td>4.13</td>
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<td>1.87</td>
<td>4.6</td>
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<td>1.87</td>
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</tr>
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<td>750</td>
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<td>1.87</td>
<td>3.9</td>
<td>5.3</td>
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<td>900</td>
<td>2.95</td>
<td>1.87</td>
<td>3.8</td>
<td>5.26</td>
</tr>
<tr>
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<td>1000</td>
<td>3.0</td>
<td>1.25</td>
<td>2.6</td>
<td>5.34</td>
</tr>
</tbody>
</table>
The following table shows measurements made on loss angle of $K^*$ for rubber samples W203 C & E. The first column shows the first measurement made the subsequent values were obtained after moving from the measuring frequency then returning to it some interval later.

<table>
<thead>
<tr>
<th>W203 C</th>
<th>Frequency (Hz)</th>
<th>Loss angle/repeats (degrees)</th>
<th>W203 E</th>
<th>Frequency (Hz)</th>
<th>Loss angle/repeats (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>100</td>
<td>8</td>
<td></td>
<td>25</td>
<td>8/10</td>
</tr>
<tr>
<td></td>
<td>200</td>
<td>3</td>
<td></td>
<td>50</td>
<td>12/10</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>4</td>
<td></td>
<td>75</td>
<td>6/9</td>
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<td>400</td>
<td>9/8/8</td>
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<td>100</td>
<td>10/10</td>
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<tr>
<td></td>
<td>420</td>
<td>10</td>
<td></td>
<td>250</td>
<td>14/11</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>10/4/12</td>
<td></td>
<td>500</td>
<td>10/20/14</td>
</tr>
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<td>14/10</td>
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<td>750</td>
<td>13/16</td>
</tr>
<tr>
<td></td>
<td>700</td>
<td>14/13</td>
<td></td>
<td>900</td>
<td>17</td>
</tr>
<tr>
<td></td>
<td>800</td>
<td>12</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>820</td>
<td>14/14</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>840</td>
<td>12/12/19</td>
<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td>860</td>
<td>15/20/22</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>880</td>
<td>8/14</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>900</td>
<td>16</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>950</td>
<td>16</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>980</td>
<td>28/26</td>
<td></td>
<td></td>
<td></td>
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<td>1100</td>
<td>10</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1150</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
TABLE III

Preliminary study using nitrile rubber to investigate the effect of pressure and refractive index of submerging fluid. All obtained using cylindrical sample.

<table>
<thead>
<tr>
<th>Frequency (Hz)</th>
<th>Superimposed Static Pressure (bar)</th>
<th>Bulk Modulus $K$ GN/m²</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>2.0</td>
<td>1.75</td>
<td>Submerged in glycerine</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.0</td>
<td>1.65</td>
<td>Submerged in transformer oil</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>3.25</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>4.5</td>
<td>2.1</td>
<td>Static tests carried out on Instron test machine</td>
</tr>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20.0</td>
<td>3.5</td>
<td>using method outlined(1)</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>442.0</td>
<td>8.8</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>50.0</td>
<td>20</td>
<td>3.48</td>
<td>Dynamic results obtained from samples submerged in glycerine</td>
</tr>
<tr>
<td>100.0</td>
<td>&quot;</td>
<td>3.03</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>&quot;</td>
<td>3.37</td>
<td></td>
</tr>
<tr>
<td>800</td>
<td>&quot;</td>
<td>3.48</td>
<td>(Fringe data treated with Gladstone Dale)</td>
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<td></td>
</tr>
<tr>
<td>80</td>
<td>&quot;</td>
<td>3.3</td>
<td>Dynamic results obtained using samples submerged in transformer oil</td>
</tr>
<tr>
<td>500</td>
<td>&quot;</td>
<td>3.23</td>
<td></td>
</tr>
<tr>
<td>900</td>
<td>&quot;</td>
<td>3.06</td>
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</tr>
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</table>
Static tests using method outlined by Holownia (40) using Instron test machine

Cylindrical samples 25.4mm dia 15mm long

<table>
<thead>
<tr>
<th>Sample Ref:</th>
<th>K(GN/m²)</th>
<th>E(MN/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W203 A</td>
<td>2.57</td>
<td>3.16</td>
</tr>
<tr>
<td>W203 C</td>
<td>2.86</td>
<td>4.5</td>
</tr>
<tr>
<td>W203 E</td>
<td>3.01</td>
<td>7.03</td>
</tr>
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</table>

Static and dynamic ..................
Bulk and uniaxial results for W203 C obtained from optical technique.

<table>
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<tr>
<th>Freq.Hz</th>
<th>K(GN/m²)</th>
<th>E(MN/m²)</th>
<th>Loss Angle</th>
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<tbody>
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<td>0.49989</td>
<td>1.3</td>
</tr>
<tr>
<td>50</td>
<td>3.9</td>
<td>0.49957</td>
<td>10.0</td>
</tr>
<tr>
<td>100</td>
<td>3.8</td>
<td>0.49941</td>
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<td>0.4986</td>
<td>32.3</td>
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<td>400</td>
<td>3.86</td>
<td>0.49845</td>
<td>36.9</td>
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<td>500</td>
<td>3.88</td>
<td>0.49815</td>
<td>43.0</td>
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TABLE V

Measurements of the bulk modulus of submerging fluids.

Results for glycerine obtained by the method outlined in (1)

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<th>Frequency Hz</th>
<th>Superimposed Pressure Bar</th>
<th>Bulk Modulus K GN/m²</th>
<th>Comments</th>
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<td></td>
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</tr>
<tr>
<td>0.0</td>
<td>13.4</td>
<td>4.3±0.3</td>
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</tr>
<tr>
<td>10.0</td>
<td>-</td>
<td>5.34</td>
<td></td>
</tr>
<tr>
<td>31.0</td>
<td>-</td>
<td>5.62</td>
<td>-indicates unknown pressure</td>
</tr>
<tr>
<td>50.0</td>
<td>20</td>
<td>4.7</td>
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</tr>
<tr>
<td>80</td>
<td>20</td>
<td>5.3</td>
<td>Additional</td>
</tr>
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<td>100</td>
<td>20</td>
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<td>Freq:</td>
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<td>5.13</td>
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<td>-</td>
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Hydraulic oil - results obtained by method outlined in (1)

\[ \text{Ko} = 1.68 \times 10^9 \text{ N/m}^2 \text{ @ mean pressure 15 bar} \]
\[ \text{Ko} = 1.52 \times 10^9 \text{ N/m}^2 \text{ @ mean pressure 25 bar} \]

(values obtained using Lorentz - Lorenz equation for glycerine + 14%)
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Sample supplied in tile form. Test specimen was cut from tile and glued into sample holder.
TABLE XIII

Temperature dependence of $k^*$ for Norsorex (as cast)

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APENDIX II

PUBLICATIONS AND PRESENTATIONS

Optical Metrology at Loughborough University.
P Montgomery and A C Rowland.
Advanced Study Institute, Viano Do Castelo, Portugal.
NATO, July 1984.

Dynamic Measurement of Volume Contraction and Phase Change of Submerged Viscoelastic Bodies using E.S.P.I.
A C Rowland and B P Holownia.
Optics and Lasers in Engineering, 6, 165-177, 1985.

E.S.P.I. Fringe Manipulation.
A C Rowland.
Optics in Engineering Measurement, Cannes, France.

A C Rowland and F Mendoza.
Optical Engineering 25, 7, 865-870, 1986.

Measurement of Dynamic Bulk Modulus and Phase Angle using ESPI.
B P Holownia and A C Rowland.