

**MEASUREMENT METHODS FOR OBTAINING  
VOLUMETRIC COEFFICIENTS FOR HYPERELASTIC  
MODELLING OF FLEXIBLE ADHESIVES**

**by**

**Louise Crocker and Bruce Duncan**

**Project PAJex2 - Flexible Adhesives**

**PAJex2 Report No 3**

**January 2001**

# **METHODS FOR OBTAINING VOLUMETRIC COEFFICIENTS FOR HYPERELASTIC MODELLING OF FLEXIBLE ADHESIVES**

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## **Summary**

In the Performance of Adhesive Joints extension project 'Flexible Adhesives' (PAJex2) a selection of hyperelastic material models are under investigation to determine the suitability of these models for predicting the behaviour of a flexible adhesive in bonded structures. Many flexible adhesives cannot be assumed to be incompressible, as Poisson's ratios are often lower than 0.5 (the case for incompressibility). Hence the compressibility of the material should be included in the model predictions through the volumetric terms in the hyperelasticity equations.

Three methods for obtaining volumetric properties data suitable for incorporation into the hyperelastic models have been investigated. These methods were volumetric compression, uniaxial tension and butt tension. A testing jig has been designed and manufactured in-house to measure the volumetric compression of adhesives. Jig design, specimen preparation and volumetric compression testing have been described. Methods for calculating volumetric compression data have been obtained. It is felt that properties in tension are critical to joint failure and that volumetric behaviour in tension may be more suitable as input data. Details of methods for obtaining volumetric properties from both butt tension and uniaxial tension experimental measurements are presented.

It has been found that, although the volumetric tensile data has more of an effect than volumetric compression data, FE predictions incorporating the volumetric term fail to offer significant improvements over those made without the volumetric term. Of the three methods investigated, the uniaxial tension method is the most practical method for obtaining volumetric properties since these measurements are also required for obtaining other material coefficients.

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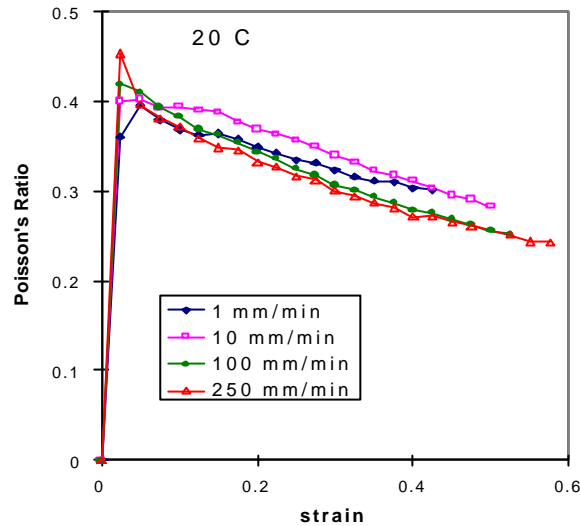
Approved on behalf of Managing Director, NPL, by Dr C Lea,  
Head of NPL Materials Centre

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must be 0.5, and the volumetric terms are set to zero. Uniaxial tensile measurements on bulk specimens of the M70 adhesive have shown that the maximum Poisson's ratio is approximately 0.4, with this value linearly decreasing at increased strains (Figure 1). Therefore the assumption of incompressibility is not valid for the M70 adhesive and it is necessary to allow some compressibility in the material model.



**Figure 1.** Variation of Poisson's ratio with strain

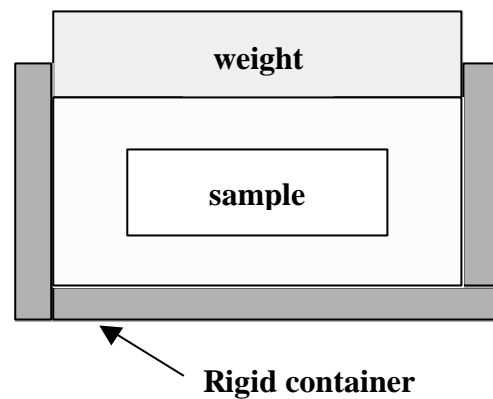
The user manuals for ABAQUS<sup>(2)</sup> recommend that coefficients ( $D_i$ ) required to define the volumetric contribution are calculated from data obtained in pure volumetric compression of a specimen (volumetric strain versus pressure). Rubbers are often used in compressive situations in which case compressive volumetric data will generally be suitable. However, the key deformation mode for failure through rupture of the flexible adhesive is normally tension. Therefore, the properties in tension are critical to joint failure. Thus, volumetric behaviour in tension may be more suitable as input data. Volumetric compression and two tensile methods for obtaining volumetric test data (uniaxial tension and butt tension) are described in this report. Methods for obtaining volumetric properties data for determining volumetric ( $D_i$ ) coefficients are outlined in Sections 2, 3 and 4. Concluding remarks are given in Section 5.

## 2. VOLUMETRIC COMPRESSION METHOD

### 2.1 MEASUREMENT OF VOLUMETRIC COMPRESSION

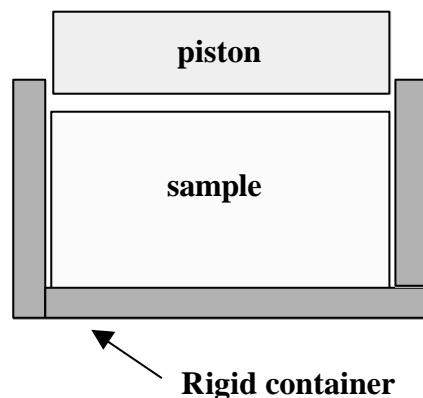
#### 2.1.1 Volumetric Compression Jig

One method of measuring true volumetric compression is a dilatometer. This is an application of true hydrostatic pressure. The stress is applied to a fluid and acts equally on the sample in all directions (Figure 2). Measurements using this method are the most accurate, but the requirement for a liquid to transmit the pressure to the sample limits the pressures range<sup>(5)</sup>.



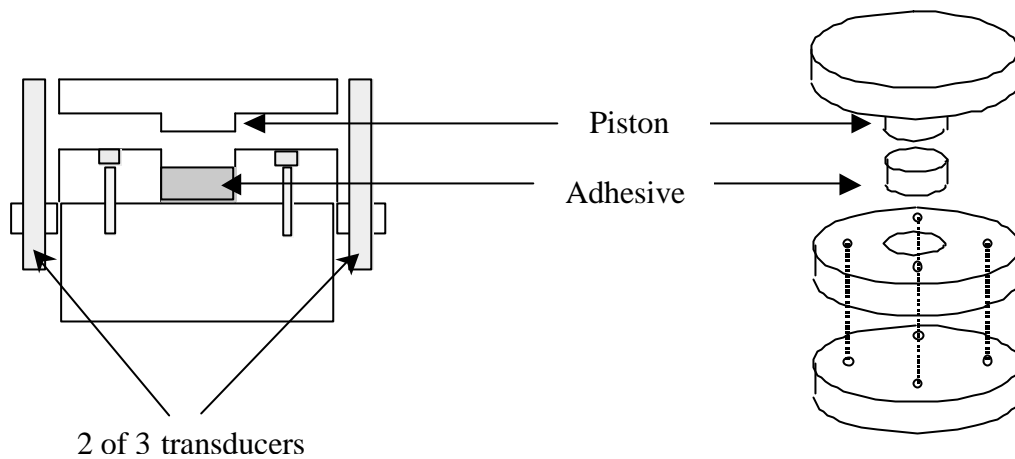
**Figure 2.** Idealised method of applying true hydrostatic compression

A simpler but more approximate way of conducting a volumetric compression test involves applying stress to the sample in one direction only. The sample is a small cylinder of material inside a rigid container. As the surface of the sample is compressed the sides try to move outwards in response to the stress, but are restrained by the rigid container (Figure 3). Stresses are thus generated on the sides as well as the top and bottom of the sample, although the stress field is only quasi-hydrostatic as the stresses on the sides are lower than those at the top and bottom. Although both volumetric and deviatoric deformations are present, the deviatoric stresses will be several orders of magnitude smaller than the hydrostatic stresses (because the bulk modulus is much higher than the shear modulus) and can be neglected. The limitation of this type of apparatus is the stiffness and yield strength of the container.



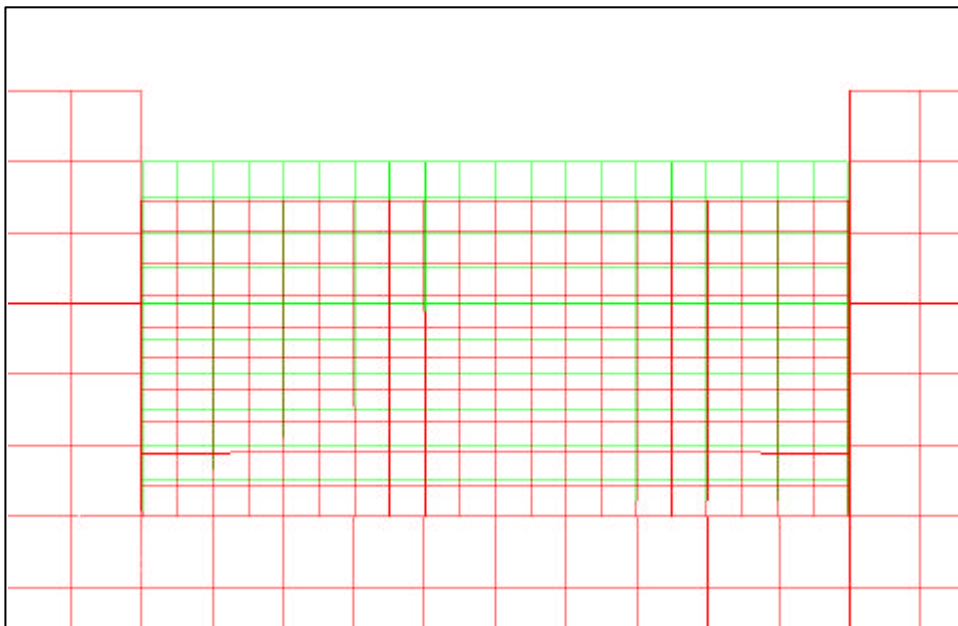
**Figure 3.** Idealised method of applying quasi-hydrostatic compression

A volumetric compression jig was designed and manufactured in-house (Figure 4). The jig consisted of three main parts. The base plate is a 35 mm deep cylindrical piece with 3 clamps spaced equally around the circumference. These three clamps hold displacement transducers during testing. A thinner cylindrical piece is bolted to the base plate. This cylinder has a depth of 12 mm, an outer diameter of 100 mm and an inner diameter of 20 mm. The hollow cylindrical centre holds the sample. An upper piece with a cylindrical protrusion fits on top of the central part of the jig and acts as the piston when pressure is applied.



**Figure 4.** Volumetric Compression Jig

The thick cylindrical walls of the central part of the jig were designed to prevent any deformation of the jig during testing. To verify this, an FE analysis of the jig was carried out. Figure 5 shows both the undeformed and deformed mesh of the sample and jig and as expected, there is no significant deformation of the thick cylinder walls.



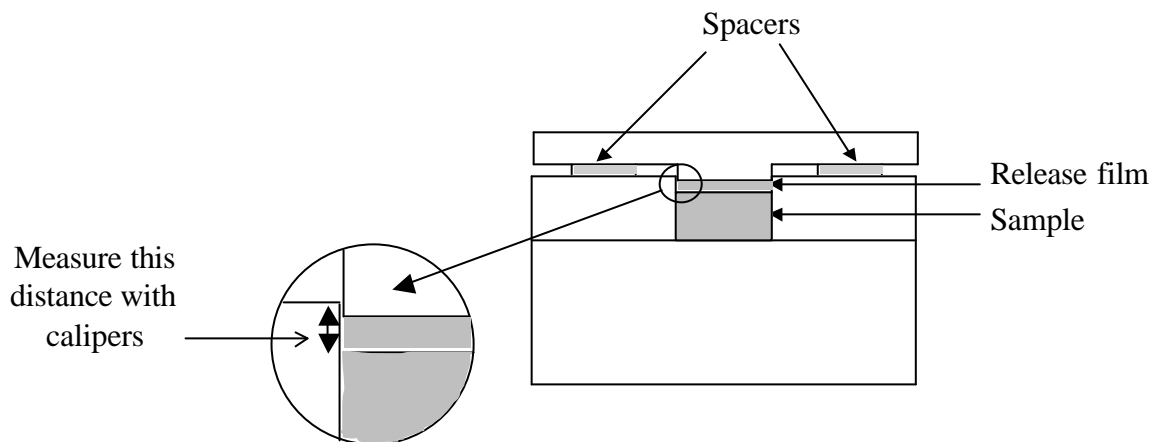
**Figure 5.** Undeformed and deformed mesh of compression jig and sample

### 2.1.2 Sample Preparation and Testing

The adhesive samples were prepared and cured in the compression jig, hence only one sample can be prepared at any one time. Measurements of temperature profiles during heating of the jig were used to devise a cure schedule that gave the compression sample a similar state of cure to both the M70 joint samples and bulk sheets previously prepared for the project. The M70 adhesive is highly viscous and consequently it is difficult to dispense the adhesive into the jig, making the presence

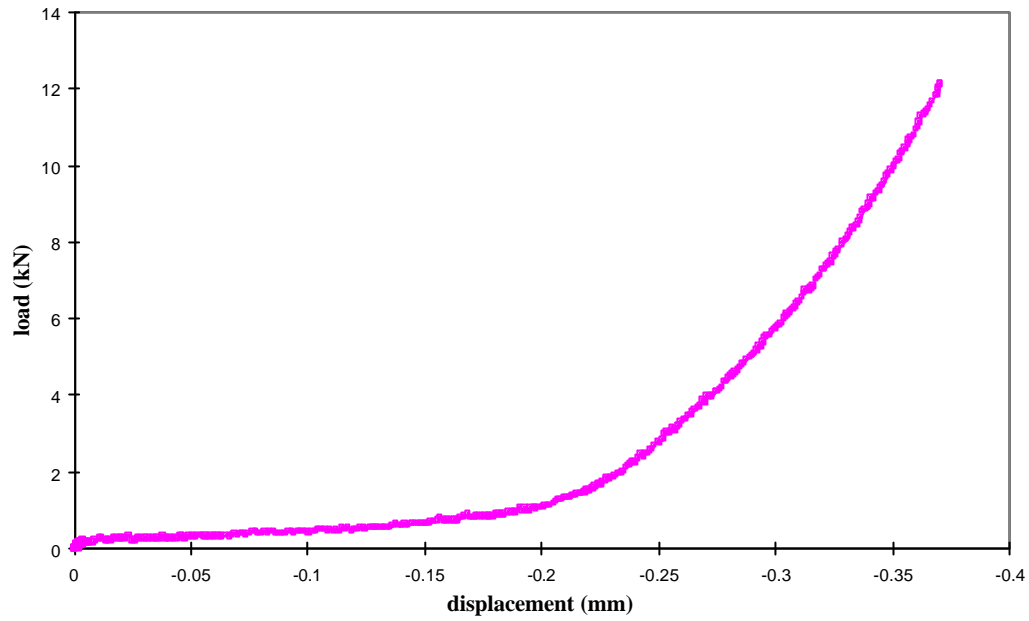
of voids likely. It has been shown<sup>(6)</sup> that the contribution of the voids to volumetric strain is significant at low loads. Ideally the specimen needs to have a smooth, flat top surface with minimal air bubbles throughout the bulk of the sample. In an attempt to achieve this, the following method of sample preparation was used.

Adhesive was dispensed from the cartridge onto a piece of release film. The adhesive was smoothed out slightly with a spatula to remove any air bubbles, and then 'spooned' into the cavity in the jig. Care was taken to minimise incorporation of air bubbles. Enough adhesive was added to fill approximately three quarters of the cavity. A small circle of release film that fitted tightly inside the cavity was placed carefully on top of the adhesive. The piston was then put in place using spacers of a size such that slight pressure was applied to the specimen to compress the adhesive (Figure 6). The complete jig was then placed in a preheated oven to cure the sample. After curing, the piston was taken off the jig and the disc of release film removed. Callipers were used to measure the distance from the top of the sample to the top of the central section of the jig as shown in Figure 6. The sample height can be calculated by subtracting this height from that of the central section.



**Figure 6.** Diagram of sample preparation

The volumetric compression jig fits between two flat plates on an Instron test machine. Lowering the piston imposes a compressive stress. Compressive displacements were measured using three sensitive displacement transducers equally spaced around the circumference of the jig. The transducers were clamped to the lowest part of the jig (see Figure 4), the piston displacement is measured via the transducers touching the Instron top plate (i.e. level with the top of the piston). These measurements also serve as a check on the axially of the compression. The load was output from the Instron. An example of the data obtained is shown in Figure 7.



**Figure 7.** Typical force-displacement response from an M70 volumetric compression test

The volumetric strain in the compression sample can be computed from the piston displacement as follows:

$$\mathbf{e}_v = \frac{V_0 - V}{V_0} = \frac{\mathbf{pr}^2 h_0 - \mathbf{pr}^2 h}{\mathbf{pr}^2 h_0} = \frac{h_0 - h}{h_0} \quad (4)$$

where  $V_0$  is the original volume,  $V$  is the current volume,  $h_0$  is the original sample height and  $h$  is the current sample height ( $h_0$ -piston displacement).

After testing, the two main parts of the jig can be disassembled and the sample pushed out of the jig. Samples were examined visually after slicing with a scalpel. In general, there appeared to be a fair number of small air bubbles and few large air bubbles.

## 2.2 CALCULATION OF VOLUME RATIO AND FE COEFFICIENTS

The coefficient ( $D_i$ ) required to define the volumetric contribution can be calculated from data obtained in pure volumetric compression of a specimen.

The first and second deviatoric strain invariants shown in Equation 1 are defined as:

$$\bar{I}_1 = \bar{I}_1^2 + \bar{I}_2^2 + \bar{I}_3^2 \quad (5)$$

$$\bar{I}_2 = \bar{I}_1^{-2} + \bar{I}_2^{-2} + \bar{I}_3^{-2} \quad (6)$$

where the deviatoric stretches  $\bar{I}_i = J^{-1/3} I_i$ ,  $J$  is the total volume ratio and  $\lambda_i$  are the principle stretches.

In a pure volumetric compression test the principle stretches ( $\lambda_i$ ) are equivalent, i.e.  $\lambda_1 = \lambda_2 = \lambda_3 = \lambda_v$ ; therefore  $\overline{I_1} = \overline{I_2} = 3$  and  $J = \lambda_v^3 = V/V_0$  (the volume ratio). Using the polynomial form of the strain energy potential, the total pressure on the sample is:

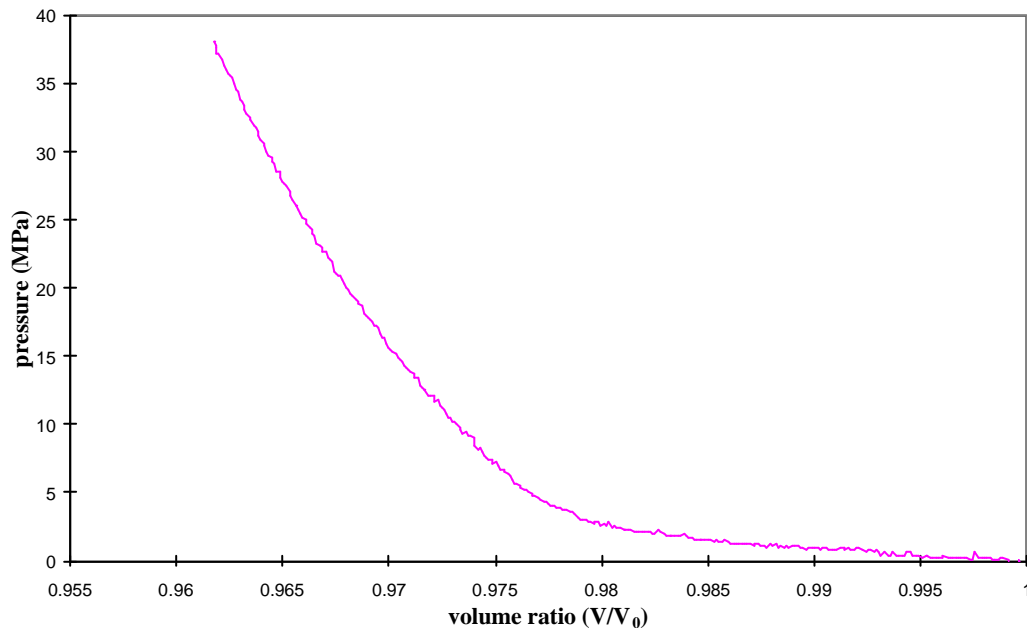
$$p = -\left(\frac{\mathbf{s}_1 + \mathbf{s}_2 + \mathbf{s}_3}{3}\right) = -\sum_{i=1}^N 2i \frac{1}{D_i} (I_v^3 - 1)^{2i-1} \quad (7)$$

This equation can be used to determine the  $D_i$  values from volumetric test data.

For the calculation of  $D_i$  the following volumetric test data are required; the pressure,  $P$ , and the corresponding volume ratio,  $J$  (current volume/original volume) throughout the volumetric compression test. The compressive stress imposed by the piston is effectively the pressure data. The volume ratio,  $J$ , is calculated as follows:

$$J = \frac{V}{V_0} = \frac{\mathbf{p}r^2h}{\mathbf{p}r^2h_0} = \frac{h_0 - \text{piston\_displacement}}{h_0} \quad (8)$$

A typical plot of pressure versus volume ratio is shown in Figure 8.



**Figure 8.** Pressure versus volume ratio for an M70 volumetric compression test

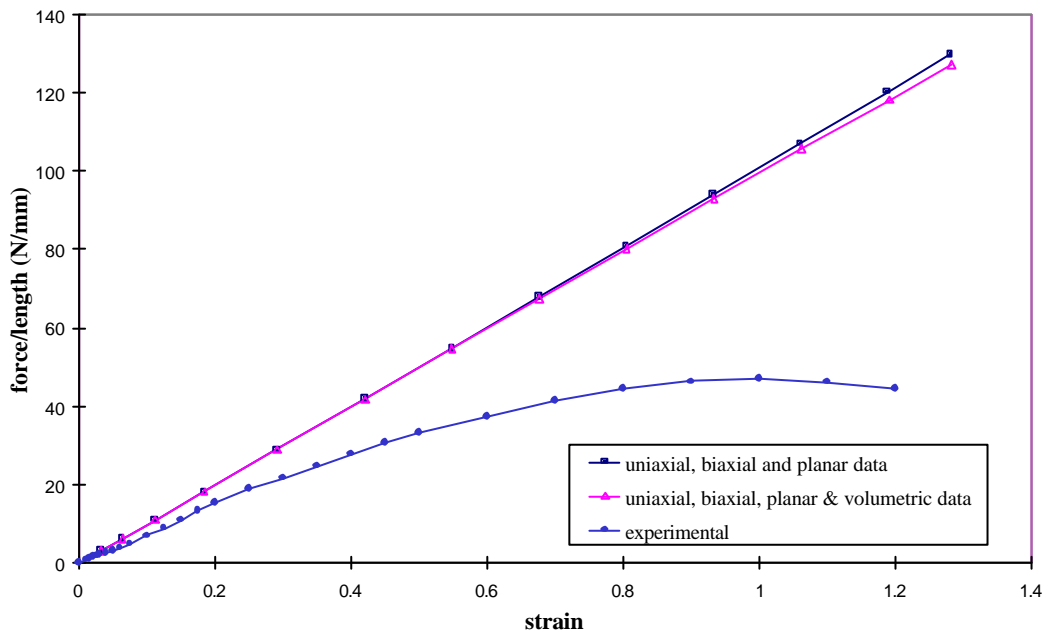
After entering this volumetric data into an input file, together with uniaxial, biaxial and planar data, ABAQUS computes the volumetric term  $D_i$  along with the Mooney-Rivlin coefficients  $C_{10}$  and  $C_{01}$ . The  $C_{10}$ ,  $C_{01}$  and  $D_i$  coefficients obtained are shown in Table 1.

**Table 1.** Mooney-Rivlin coefficients obtained using uniaxial, biaxial, planar and volumetric compression data

$C_{10}$	$C_{01}$	$D_1$
$4.713647 \times 10^{-2}$	2.13972	$7.442617 \times 10^{-3}$

### 2.3 FE PREDICTIONS USING VOLUMETRIC COMPRESSION DATA

The calculated coefficients were used to predict the behaviour of the M70 lap joint modelled in previous work<sup>(7)</sup>. It was found that, although the M70 has a Poisson's ratio much lower than the 0.5 assumed for an incompressible material, the addition of volumetric data to the Mooney-Rivlin analysis had only a small effect on the prediction of stress/strain for the M70 lap joint. This can be seen in Figure 9, which shows a comparison of predictions made using uniaxial, biaxial and planar data with and without volumetric data. The corresponding experimental result is also shown in the plot.

**Figure 9.** Comparisons of M70 lap joint predicted behaviour with and without volumetric compression data

The prediction made using the added volumetric term only begins to deviate from the original prediction at about 0.7 strain. The difference between the two curves is very small. In both cases, the predictions match the experimental result only at very small strains. At larger strains, the forces predicted greatly overestimate those seen experimentally. These results are discussed in a further report<sup>(7)</sup>. As the addition of volumetric compression data has only had a very slight effect of the FE predictions, the volumetric compression method may not always be suitable for obtaining volumetric data for adhesives.

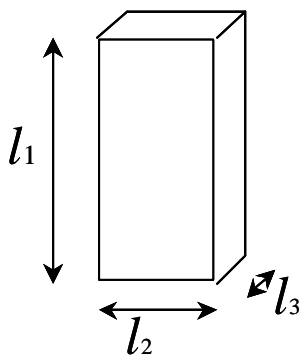
### 3. UNIAXIAL TENSION METHOD

#### 3.1 SAMPLE PREPARATION AND TESTING

Tensile specimens were obtained from bulk adhesive sheets. Bulk sheets were prepared in the following manner. A sheet of adhesive was cast<sup>(8)</sup> onto flat metal plates covered with PTFE release film. The adhesive was applied with a spatula and scraped to an even layer thickness taking care to remove any visible voids. A sheet of PTFE film was placed over the cast adhesive and was pressed down using a roller to force out entrapped air. A second metal plate was then placed on top of this sheet. Strips of plastic 1.0 mm thick were used to control the distance between the two plates and hence, the specimen thickness. The sheet was cured in an oven (pre-heated to 200°C) for 45 minutes. Half sized ISO 3167<sup>(9)</sup> multi-purpose test specimens were punched from the bulk sheet using a shaped cutter. Tensile tests were carried out using an Instron 4505 test machine. Tensile and transverse strains were measured using a Messphysik video extensometer.

#### 3.2 CALCULATION OF VOLUME RATIO

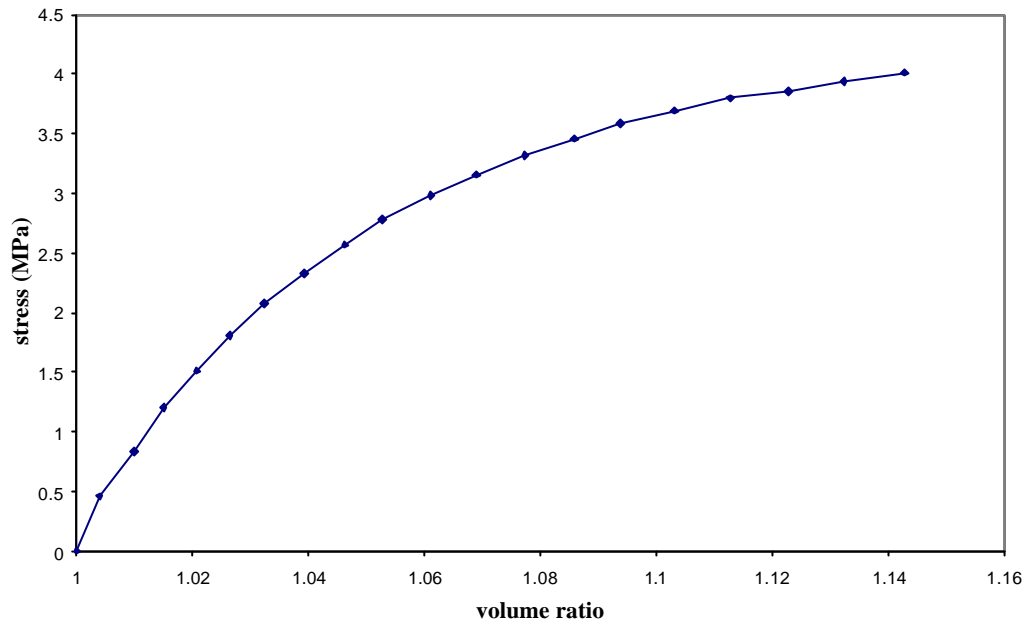
Bulk specimens of the adhesive M70 were tested in uniaxial tension. During testing both the axial and lateral strains were recorded along with the load. The stress required by ABAQUS can be calculated from the measured load. From the strain measurements it is possible to obtain a form of volumetric tensile data, making the assumption that the through-thickness strain is the equivalent to the lateral strain (i.e.  $\epsilon_3 = \epsilon_2$ ). The original and current volumes are calculated as follows:



$$\text{Original volume; } V_0 = l_1 l_2 l_3 \quad (9)$$

$$\text{Current volume; } V = l_1(1 + \epsilon_1) l_2(1 + \epsilon_2) l_3(1 + \epsilon_2) \quad (10)$$

Where  $l_1$  is the extensometer gauge length,  $l_2$  is the specimen width,  $l_3$  is the specimen thickness,  $\epsilon_1$  is the axial strain and  $\epsilon_2$  is the lateral strain. A typical plot of stress versus volume ratio is shown in Figure 10. It can be seen that the stresses do not become as high as those seen in volumetric compression. This is due to the highly constrained state of the specimen at large displacements in volumetric compression testing.



**Figure 10.** Stress versus volume ratio for an M70 uniaxial tension test

The calculation of  $D_1$  values in ABAQUS is designed for the inclusion of volumetric compression data where the compressive stress is deemed positive. When calculating the compressive volume ratio, the current volume is always smaller than the original volume. Hence the volume ratio ( $J$ ) starts at  $J=1$  (zero stress) and decreases with increasing compressive stress. When considering tensile volumetric data, the current volume is larger than the original volume, hence  $J=1$  at zero stress and increases with increasing tensile stress. If the compressive stress has a positive value, then the tensile stress should be regarded as negative. An FE analysis was carried out using tensile data in the form  $P = -\sigma$  and  $J \geq 1$ . During the data checking stage ABAQUS generated the following warning message “The nominal strain is opposite in sign to the stress. This is not physically reasonable” as the data was not in the expected format. The presence of a warning message does not cause failure of the analysis, and a  $D_1$  value was successfully calculated.

An alternative approach is to calculate the inverse volume ratio giving  $J \leq 1$  and using positive pressure values. In this case  $J=V_0/V$  (original volume/current volume). A  $D_1$  value was calculated without the generation of a warning message. The  $D_1$  values obtained from both methods were comparable.

### 3.3 FE PREDICTIONS USING VOLUMETRIC TENSION DATA

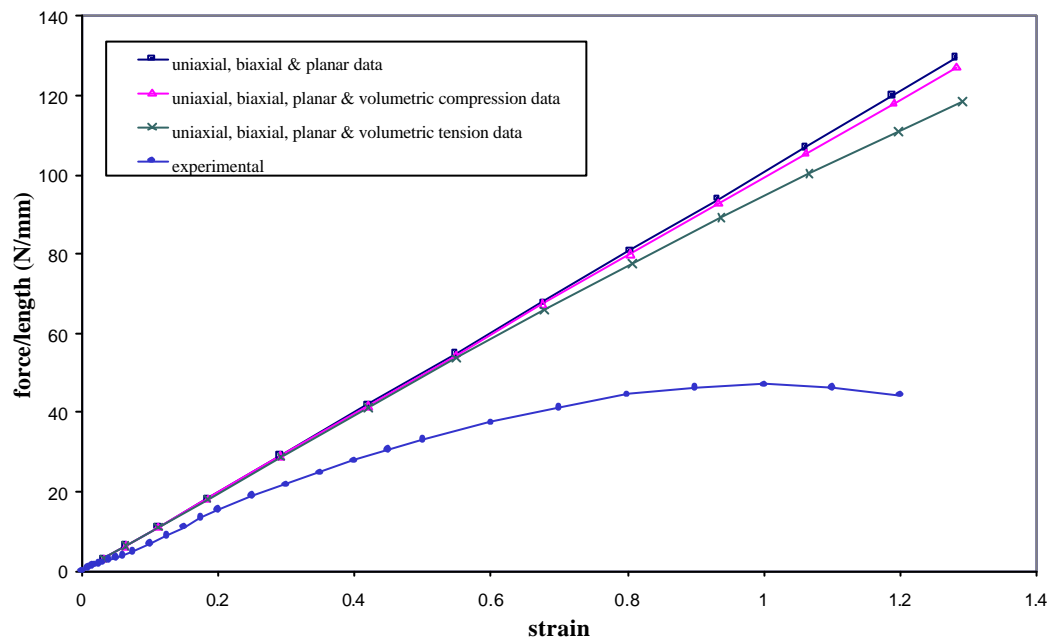
After entering the tensile volumetric data into an ABAQUS input file, along with uniaxial, biaxial and planar data, ABAQUS computed the volumetric term  $D_1$  and the Mooney-Rivlin coefficients  $C_{10}$  and  $C_{01}$ . The three coefficients obtained are shown in Table 2.

**Table 2.** Mooney-Rivlin coefficients obtained using uniaxial, biaxial, planar and volumetric tension data

$C_{10}$	$C_{01}$	$D_1$
$4.713647 \times 10^{-2}$	2.13972	$4.739247 \times 10^{-2}$

The Mooney-Rivlin  $C_{ij}$  coefficients are based purely on the uniaxial, biaxial and planar data, so these remain unchanged from Table 1. The  $D_1$  value obtained using volumetric tension data is over a factor of 5 larger than that obtained using volumetric compression data and so should have more of an effect on the FE predictions.

The coefficients shown in Table 2 were used to predict the behaviour of the M70 lap joint. Figure 11 shows the prediction obtained using volumetric tension data, along with the prediction obtained using volumetric compression.



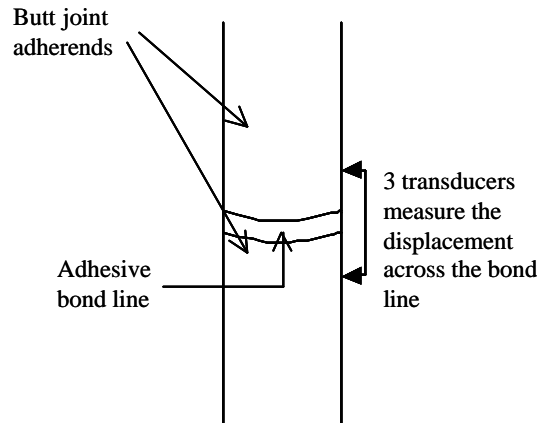
**Figure 11.** M70 lap joint predictions obtained using volumetric tension data compared to those obtained previously

As expected, the  $D_1$  value calculated from volumetric tension did lower the predicted loads further; improving on the predictions obtained using volumetric compression. It can be seen that the predictions still greatly overestimate the experimentally observed loads.

## 4. BUTT TENSION METHOD

### 4.1 SAMPLE PREPARATION AND TESTING

The butt-joint specimen consists of two 25 mm diameter hardened steel rods bonded together with the adhesive at end faces (Figure 12). An alignment jig was used during bonding to ensure accurate alignment of the adherends. The number of alignment jigs available limits the number of samples prepared at any one time. Prior to testing an extensometer, consisting of an assembly of three displacement transducers equally spaced around the circumference of the joint, was attached to the sample. Collet grips are used to provide rigid clamping of the adherends. An alignment jig with Instron ALIGNPRO software is used to align the collets. The grips maintain the specimen alignment throughout the test and help prevent bending of the specimen. During testing, the extensometer determines the axial deformation of the adhesive bond over a gauge length of 17 mm straddling the bond line. The measured displacements were corrected to remove the contribution from the steel adherends, so that the corrected data gave the extensions of the adhesive layer. The adhesive is highly constrained between the adherend faces so little contraction occurs in the radial direction.



**Figure 12.** Schematic diagram of the butt joint specimen

### 4.2 CALCULATION OF VOLUME RATIO

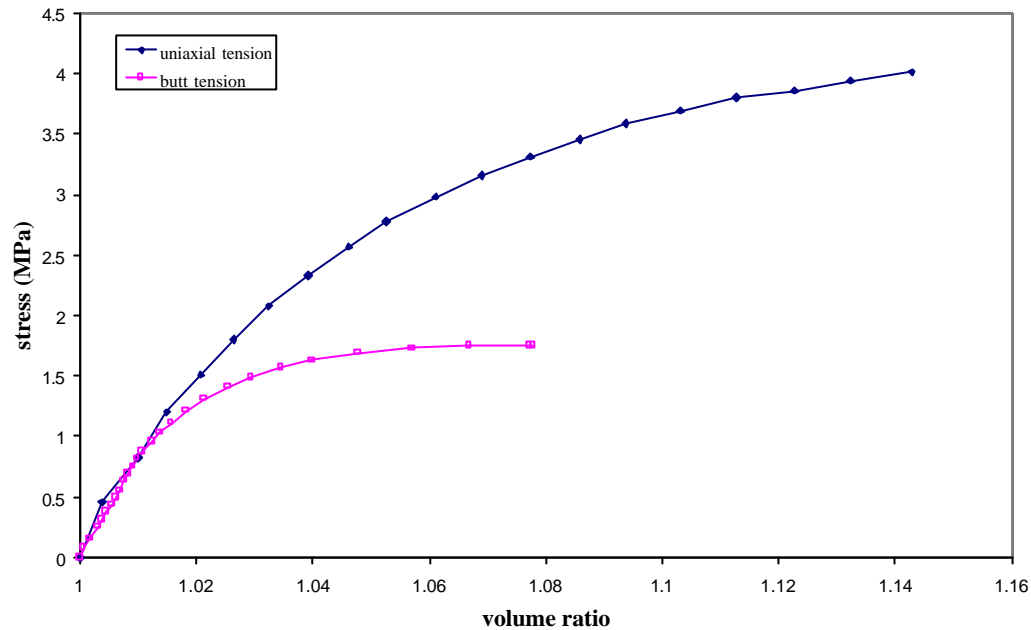
The volume ratio for the butt tension data was calculated as follows:

$$J = \frac{V}{V_0} = \frac{\pi r^2 (h_0 + a)}{\pi r^2 h_0} = \frac{h_0 + a}{h_0} \quad (11)$$

where  $J$  is the volume ratio,  $V_0$  is the original volume,  $V$  is the current volume,  $r$  is the adherend radius,  $h_0$  is the original adhesive thickness and  $a$  is the corrected displacement (i.e. extension of the adhesive).

A typical plot of stress versus volume ratio calculated from butt tension data is shown in Figure 13. For comparison, the uniaxial tension data is also shown in the plot. Initially, the volume ratio calculated from butt tension data matched well with the data. Both tensile methods give a similar stress versus volume ratio plots although, at

smaller volume ratios, the butt tension data has a slightly lower stress than that for the corresponding uniaxial tension volume ratio.



**Figure 13.** Stress versus volume ratio calculated using butt tension data, and compared to that from uniaxial tension data

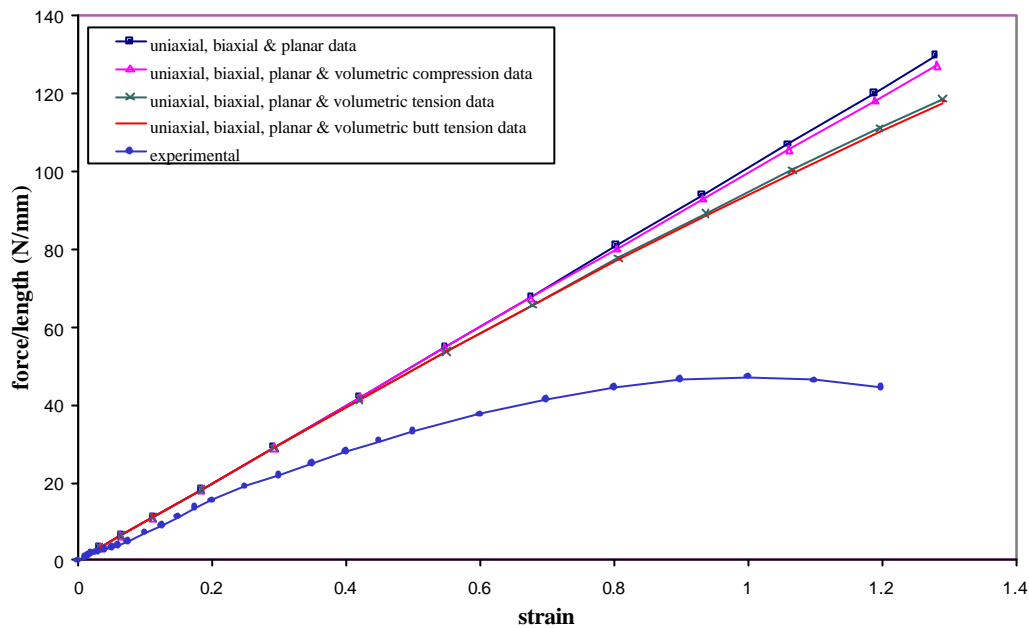
#### 4.3 FE PREDICTIONS USING VOLUMETRIC BUTT TENSION DATA

The required coefficients are calculated as before and are presented in Table 3. The Mooney-Rivlin coefficients remain unchanged, while the volumetric term  $D_1$  has increased only slightly from that obtained using uniaxial tension data. Therefore, it is likely to give similar predictions to those obtained previously.

**Table 3.** Mooney-Rivlin coefficients calculated using uniaxial, biaxial, planar and volumetric butt tension data

$C_{10}$	$C_{01}$	$D_1$
$4.713647 \times 10^{-2}$	2.13972	$5.349298 \times 10^{-2}$

The coefficients shown in Table 3 were used to predict the behaviour of the M70 lap joint. Figure 14 shows the prediction obtained using volumetric butt tension data, along with the prediction obtained using volumetric tension. As anticipated from the similarity of the volumetric terms, there is little difference between the predictions obtained using volumetric butt tension data and volumetric tension data.



**Figure 14.** M70 lap joint prediction obtained using butt tension volumetric data compared with predictions using other volumetric data

## 5. DISCUSSION

Flexible adhesives cannot always be assumed to be incompressible. In many cases Poisson's ratio is less than 0.5. Hence the compressibility of the material needs to be taken into account when making design predictions. Volumetric compression, uniaxial tension and butt tension methods for obtaining volumetric data suitable for use with the FE package ABAQUS have been investigated with the aim of improving predictions of lap joint behaviour. The relative merits and disadvantages of these methods are described below and final conclusions are drawn about the applicability of these methods. It was found that the inclusion of volumetric data in hyperelastic models has some effect on the FE predictions of joint performance, but does not significantly improve the accuracy.

The ABAQUS manuals recommend using volumetric compression data for hyperelastic modelling when the assumption of incompressibility is incorrect. A volumetric compression jig was designed in-house, and experiments conducted. Although sample preparation itself was relatively easy, it was very hard to eliminate air bubbles/voids from the adhesive in the jig. This could have a major effect on the volumetric compression data obtained, especially at low loads. The majority of voids occurred within the centre of the sample. These could only be seen after testing by cutting the specimen into pieces. Air bubbles on the top surface of the sample were visible after curing. Testing was straightforward, and the output converted easily into the required pressure stress and volume ratio. One major disadvantage of this volumetric compression method is that samples can only be made and tested one at a time. Hence it is very time consuming to obtain a reasonable number of test results.

The addition of volumetric compression data only had a limited effect on the predictions when compared to predictions obtained assuming incompressibility (i.e.  $D_1=0$ ).

Butt tension specimens are straightforward to prepare with few voids apparent, but the number of alignment jigs available limit the number of butt joints prepared in one day. The alignment of the adherends is very important and an alignment jig is necessary to align the collet grips that clamp the sample. Without this rigid clamping, sample bending can occur causing divergence of the three transducers outputs. As a result butt tension testing is more complex and time consuming than volumetric compression. The butt tension data obtained requires further manipulation prior to calculation of the volume ratio. The butt tension volumetric data produced the highest volumetric coefficient of all three methods. But this only gave slight improvement in the predictions.

Uniaxial tension specimens are cut from bulk sheets of adhesive, with multiple samples being cut from one sheet. The sheets need to be prepared carefully to eliminate the presence of voids. Uniaxial tension testing is routine, and little manipulation of the data is required to obtain the volume factor. One great advantage of using uniaxial tension data rather than volumetric compression or butt tension data to obtain the volumetric data is that uniaxial tests are already required for obtaining the Mooney-Rivlin deviatoric coefficients  $C_{10}$  and  $C_{01}$ . Hence, these same tests can be utilized to obtain volumetric data, removing the need to carry out further sample preparation and testing of either volumetric compression samples or butt joints. The calculated volumetric coefficient was comparable to that obtained using the butt tension data, and hence gave similar predictions of lap joint behaviour. Since tension data are required for calculating the other coefficients, this is the most efficient method for obtaining volumetric properties.  $D_1$  volumetric coefficients determined from M70 tension specimens tested at a number of strain rates and temperatures<sup>(7)</sup> were found to vary between  $2.5 \times 10^{-2}$  and  $6 \times 10^{-2}$ . This variability in the value of  $D_1$  was found to have little significant effect on FEA predictions<sup>(7)</sup>.

## 6. RECOMMENDATIONS

When using hyperelastic material models to predict the behaviour of flexible adhesives with a Poisson's ratio of less than 0.5, it is prudent to account for the materials compressibility. This is achieved by including a volumetric term (calculated from volumetric data) along with the hyperelastic coefficients.

For the following reasons it is recommended that volumetric data be obtained from uniaxial tension tests:-

- Uniaxial tests are already required for obtaining the deviatoric hyperelastic coefficients making this the most efficient method for obtaining volumetric properties
- Uniaxial tension testing is routine and sample preparation is straightforward

- Little data manipulation is necessary to obtain data in the form required by ABAQUS
- The critical region in a bond where the adhesive fails will experience tensile rather than compressive stress components and, therefore, tensile data may be more appropriate for modelling this region
- FE predictions obtained using uniaxial tension volumetric data were an improvement over those obtained using volumetric compression data
- FE predictions obtained using uniaxial tension volumetric data were similar to those obtained using butt tension volumetric data

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