

Measurement Methods for Time-Dependent Properties of Flexible Adhesives

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Project PAJ1: Failure Criteria and their Application to Visco-Elastic/Visco-Plastic Materials.

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SUMMARY

Flexible adhesives are visco-elastic materials. Therefore, creep will occur under any load. The challenge for the engineer is to design the bonded structure to restrict creep to acceptable levels. Finite Element Analysis can be used to predict the creep behaviour of adhesive joints under load. Time-dependent property measurements - creep compliance or stress relaxation - are required for accurate predictions. These measurements can be made in tension using bulk test specimens. However, such measurements can be difficult to perform and analyse accurately. Modern oscillatory rheometers have been shown to have the capability for performing time-dependent property measurements on flexible adhesives. Stress relaxation methods are likely to give more reliable time-dependent properties for FEA of flexible adhesives than creep measurements. This is because it is easier, in relaxation measurements, to identify and separate the 'elastic' response from the time-dependent response. However, relaxation measurements will not yield information on failure whereas creep measurements can.

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1. INTRODUCTION

Prediction of joint performance throughout the service life is one of the important issues facing the designer of adhesively bonded structures. Moisture absorption, thermal cycling, fatigue and interface degradation are some of the processes that act to compromise the long-term strength of adhesive joints. Also very important for long-term performance are the time-dependent mechanical properties such as creep⁽¹⁾.

Flexible adhesives differ from common structural materials, such as metals, in creep behaviour. They are visco-elastic materials. Thus, there is not an elastic limit below which creep will not occur. Time-dependent deformation will happen at all levels of loading. The challenge for the engineer is to design structures where the creep in service is limited to acceptable deformations. Finite Element Analysis (FEA) software packages, such as ABAQUS⁽²⁾, have the capability to carry out analysis for creep deformations. However, for predictions to be accurate, time-dependent properties of flexible adhesives are needed. In ABAQUS either creep measurements or stress relaxation measurements are acceptable for modelling time-dependent visco-elastic properties.

In project PAJ1 of the Performance of Adhesive Joints programme, a study of the time-dependent properties of flexible adhesives is being undertaken. This is aimed at investigating methods for predicting the properties of joints under continuous loading. The failure of these specimens as a result of creep should help to establish failure criteria for the adhesives. Methods for measuring the creep of metals and rigid plastics⁽³⁾ are well developed. However, the measurement of creep of flexible materials is a less well developed field. Creep and stress relaxation can be measured in tension using bulk test specimens. Methods for carrying out such measurements are described in the current report.

As part of project PAJ1, an oscillatory rheometer has been developed and evaluated for measuring the mechanical properties of flexible adhesives^(4, 5). Modern rotational rheometers also have the ability, through their software control and data analysis packages, to perform measurements for time-dependent properties of fluids⁽⁵⁾. This report seeks to describe how such instruments can be used to study the creep and stress relaxation properties of flexible adhesives. The advantages and problems of these rheometry techniques are compared with the bulk specimen tests for flexible adhesives. Future reports will compare rheometry data with tensile data and evaluate FE predictions of adhesive joints under creep loading.

2. EXPERIMENTAL METHODS

Time-dependent properties can be investigated using creep and stress relaxation measurements. In this work two different types of apparatus for performing such measurements - bulk tension tests and shear rheometers - are assessed.

In creep tests the time-dependent deformation of a specimen subjected to a constant stress is measured. Measurement of the local strain in the specimen is best for determining the creep compliance properties but this may not always be possible. For example, accurate contacting extensometers are unlikely to have sufficient range to measure the extension of bulk tensile specimens manufactured from flexible adhesives. In such situations, measurement of the movement of the grips may be more convenient. The tensile creep compliance, $J(t)$ is defined as:

$$J(t) = \frac{\epsilon(t)}{\sigma}$$

where $\epsilon(t)$ is the creep strain and σ is the applied stress. In shear, the shear strain, γ , and torque, τ , replace ϵ and σ respectively in the calculation of compliance. The stress should be constant at all times for this definition to be valid. Where grip movement is measured, a relationship between strain and displacement is needed to calculate compliance.

For the FE analysis, compliance data are normalised with respect to the instantaneous modulus of the material⁽²⁾. Hence, it is not strictly necessary to determine the creep compliance for ABAQUS input data provided that there is a linear relationship between total measured displacement, $d(t)$, and strain. In practice, normalisation can be carried out with respect to the initial, elastic displacement (d_0) measured after applying the force. The creep input data, $C(t)$, are calculated:

$$C(t) = \frac{d(t)}{d_0}$$

The times (t) are those after application of the load. $C(t)$ will be 1 at time = 0 and will increase with time.

In stress relaxation measurements, a constant deformation is applied and the time-dependent force is measured. The relaxation modulus, $E(t)$ is calculated:

$$E(t) = \frac{\sigma(t)}{\epsilon}$$

where $\sigma(t)$ is the relaxation stress and ϵ is the applied strain. $J(t)$ and $E(t)$ are not simply inverse functions of each other. The compliance and relaxation modulus are related through a convolution integral:

$$\int_0^t E(t-u)J(u)du = t$$

In general, $J(t) \neq 1/E(t)$ but in the limits of $t \rightarrow 0$ and $t \rightarrow \infty$ then $J(t) \rightarrow 1/E(t)$.

It has been shown for adhesives⁽⁶⁾ that relaxation modulus of a visco-elastic material at time (t) can be related to a dynamic modulus at frequency (f) where t and f are related:

$$t \approx \frac{0.1}{f}$$

Stress relaxation data for ABAQUS input are normalised with respect to the initial, elastic modulus⁽²⁾. Consequently it is not strictly necessary to know the strain as this is eliminated from the expression by the normalisation. The relaxation input data, $R(t)$, are calculated from the ratio of measured force, $F(t)$, to initial force, F_0 :

$$R(t) = \frac{F(t)}{F_0}$$

In any creep or relaxation test measurement it is critical that the test conditions remain constant throughout the test. The test temperature must be maintained at a constant level during the measurements. Any transducers used should have long term stability under the test conditions.

2.1 TENSILE CREEP MEASUREMENTS

Creep measurements on bulk specimens of flexible adhesives (or rubbers) are difficult to perform. The low stiffness of the specimens means that loads required to produce representative levels of stress are small. The likely deformations of the specimens will be large. Consequently, the creep test methods for engineering plastics⁽³⁾ (incorporating accurate, low-range, clip-on extensometers) are unsuitable for flexible adhesives. Alternative methods developed to study these materials are described below.

Creep measurements have been performed on two types of adhesives (3M DP609, a 2-part polyurethane and Evode M70, a single-part epoxy-butadiene) at 20 °C, 40 °C and 60 °C. These tests were carried out under different levels of initial stress. The test specimen used was dumb-bell shaped with a 5 mm wide central parallel section. To maintain comparability with the material in joint specimens, the bulk specimens were machined from 1-2 mm thick flat sheets prepared by casting the adhesive paste. Specimen temperature was maintained using Climatic Systems temperature cabinets fitted to the creep machines. These are capable of maintaining temperatures from 10 °C below ambient to 60 °C above ambient with fluctuations of less than 0.2 °C. These influence of these fluctuations on the results tends to be insignificant

2.1.1 Dead Weight Loading

The original lever arm creep machines were adapted to load the specimen in a dead weight mode. The specimen was mounted between a jaw-faced grip and a load hanger as shown in Figure 1. The top jaw-faced grip was clamped securely to the rigid creep machine. The lower jaw-faced grips previously used were replaced with load hangers as shown in Figure 1. Weights hung from this hanger gave the required initial stress in the test. Movement of the grip hanger was measured using a long travel linear variable differential transformer (LVDT) displacement transducer as shown in Figure 2. The outputs from the LVDTs were logged onto a PC using a data acquisition programme specifically written for the application.

The recorded displacements had a resolution of around 15 µm. This was acceptable as initial displacements of several mm were expected. The weight of the load hanger was equivalent to 2N and additional weights could be added at 1 N increments.

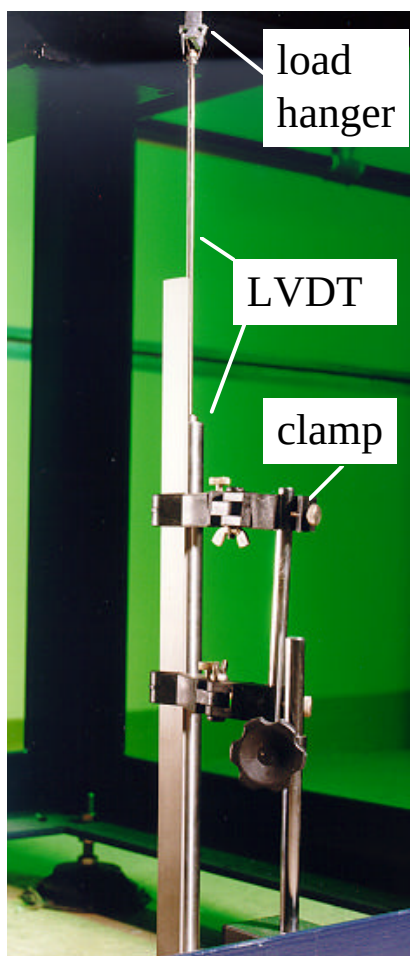


Figure 2: Dead Weight Creep Test

starting position is somewhat indeterminate. Subsequent creep measurements can not be relied upon to better than ± 1 mm. The zero force displacement is unknown as the mass of the load hanger is supported by the specimen prior to application of the test load.

A further problem is the tendency of the specimen to twist following loading. This alters the stress distribution (no longer uniaxial). It also introduces errors in the displacement measurement. The gripping band of the hanger led to premature failure of the specimen in the grips during some of the tests. This was partly corrected by bonding aluminium tabs onto the ends of the specimens or by using a layer of glass paper between the grips and the specimen. Even with these precautions, some specimens still failed in the grips.

Due to these problems, the dead weight loading technique was unsuitable for carrying out creep measurements on the adhesives to an acceptable level of accuracy and repeatability. Therefore, creep machines were converted back into the lever arm configuration.

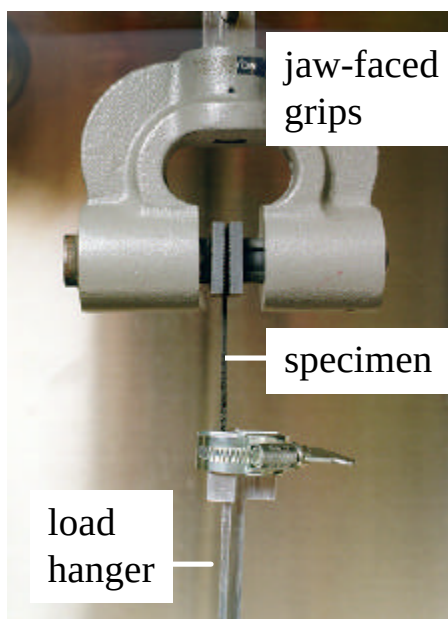
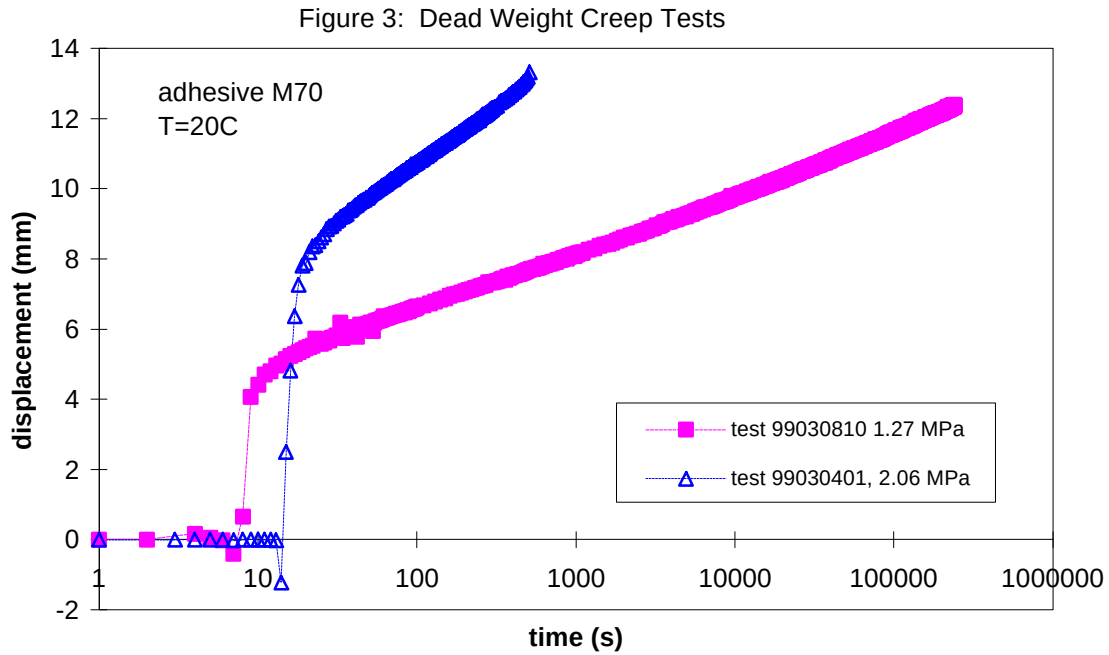


Figure 1: Specimen Clamping

It became apparent that there were a number of problems with this measurement technique. This test arrangement was prone to excessive movements due to the low modulus and cross-section of the specimen. It was difficult to apply the weights without altering the initial position of the hanger. As Figure 3 shows, there are fluctuations in the displacement readings while the load is applied to the specimen. Thus, the initial



It could be possible to improve the dead weight loading test. Suggestions for this include:

1. Addition of guides for the load hanger to prevent twisting. Linear bearings could be used to reduce friction.
2. Clamps to support the load hanger while specimen is clamped and the weights are applied. This would help to reduce the uncertainty in the starting position.

2.1.2 Lever Arm Loading

In this arrangement, loads are applied via a lever whereas, in the dead weight configuration the specimen supports the load directly. Loads are applied to weight carriage on one end of the lever. The lever is supported on a low friction pivot. The force is transmitted via the lever to the upper grip. The bottom grip is fixed to the base of the creep machine. Both grips are of the jaw-face type. Specimens rarely failed in these grips. As shown in Figure 4, the LVDT displacement transducer is used to measure the movement of the upper grip.

The lever is constrained from off-axis movement so the specimen does not twist. As shown in Figure 5, this arrangement proved to be much less susceptible to disturbance during the application of load than the dead weight loading arrangement. The measurements made seemed to have better repeatability. The lever is counter-balanced so that the unloaded force is zero. This contrasts with the dead weight configuration where the load hanger has a finite mass and the initial, zero-load extension is not known.

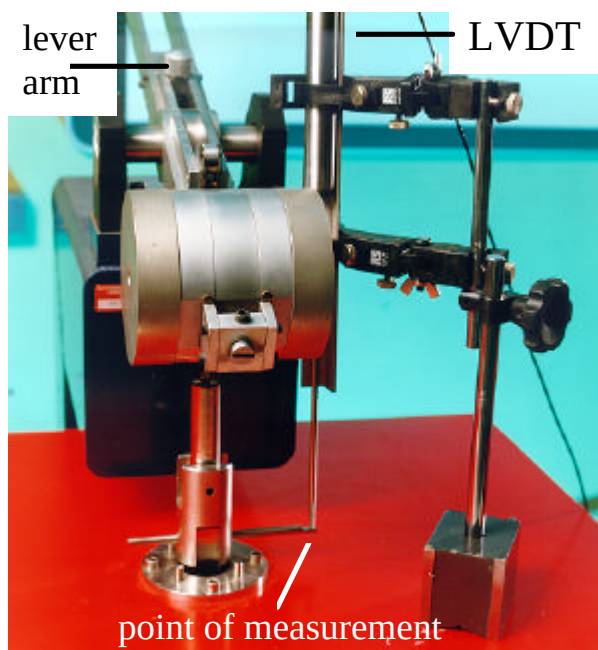
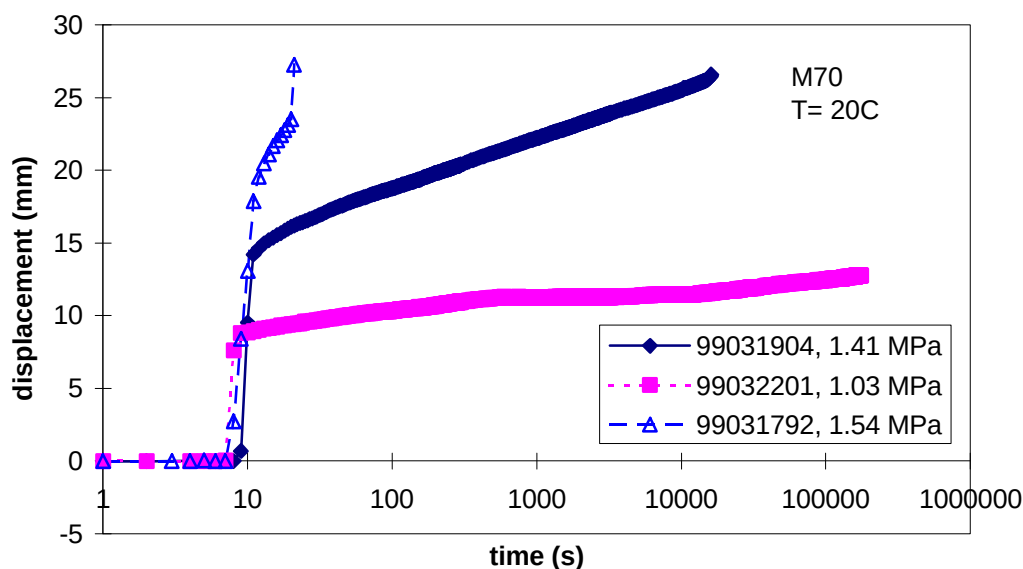


Figure 4: Displacement Measurement Using Lever Arm

adhesives in constant strain rate tensile tests. Thus, in some of the tests (e.g. at 1.5 MPa in Figure 5) the time to failure was extremely low as the short time tensile strength of the material was exceeded. Smaller lever arm ratios (e.g. 1:1) would allow smaller load increments and would be more suitable for the flexible adhesives.

However, the lever arm method introduces other measurement problems. The ratio of the lever arm used was fixed at 5:1. This meant that the effective loads on the specimen were 5 times the applied load on the weight carriage. The minimum weight increment of 1 N gives a minimum force of 5 N. This corresponds to a stress of 0.5 MPa for a 2 mm thick specimen. The smallest stresses used were closer to 1 MPa since few of the specimens were prepared with a thickness greater than 1.5 mm. Even these low stresses are a significant proportion of the failure stresses of some flexible

Figure 5: Overslung Creep Test Results



2.1.3 Strain Determination

The basic creep measurement methods outlined above measure the creep deformation from the movement of the grips. This is only suitable for determining the shape of the creep functions that are used in the FE analysis. However, the local strains in the test specimen that are needed for investigating failure criteria or creep compliances are not determined. Strain is not simply the ratio of measured grip movement to initial grip separation as the width of the dumb-bell shaped specimen is not constant. The

measured grip movement also includes extension or slippage of the specimen in the grips. In an attempt to determine a relationship between strain and grip movement, tests were performed where measurements were made simultaneously of strains in the specimen gauge section using a video extensometer and of grip movements using one of the LVDTs.

The displacement and strain measurements could not be recorded simultaneously on the same device. This meant that further analysis to combine two separate data records was needed. This introduced a level of uncertainty into the correlation. The lowest sampling interval used to when logging the LVDT output was 1 s. This was much larger than the interval between measurements from the video extensometer (ca. 0.08 s). Therefore there is a larger uncertainty in the 'start' of the test in the LVDT data. It could lie anywhere between the last point of 'zero' displacement and the first point of 'non-zero' displacement. When correlating the LVDT and video extensometer outputs there is a large uncertainty in the low displacement/strain points. However, there are lower levels of uncertainty in the points at longer times. Approximate relationships between strain and displacement were determined from the slopes of straight lines fitted to the strain-displacement curves. Typical fits are shown in Figure 6.

In each test, at large displacements, there is a reasonable linear relationship between strain and displacement. However, at low strains the uncertainties are large. There is relatively large scatter between the slopes of the different strain-displacement fits. The mean slope calculated from 20 tests was 0.0184 with a standard deviation of 0.022. This could lead to uncertainties (of around 10% of the measurement) in strains determined from grip displacement measurements alone.

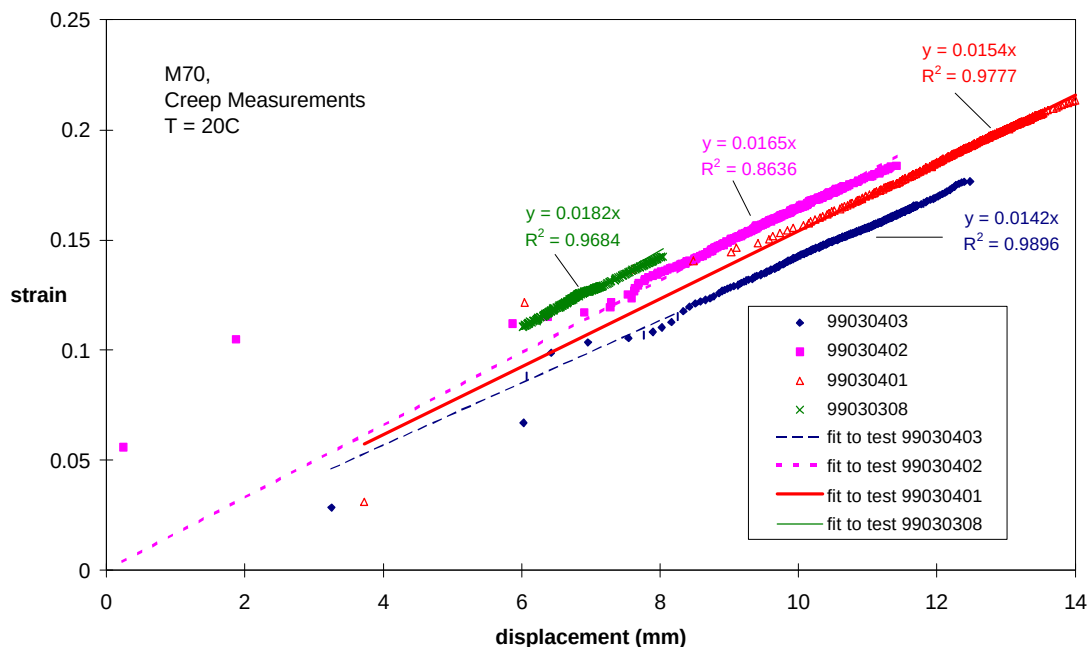


Figure 6: Strain-Displacement Fits

Video extensometry is a relatively costly strain measurement method. It was not economically feasible to purchase and dedicate such equipment to each creep machine

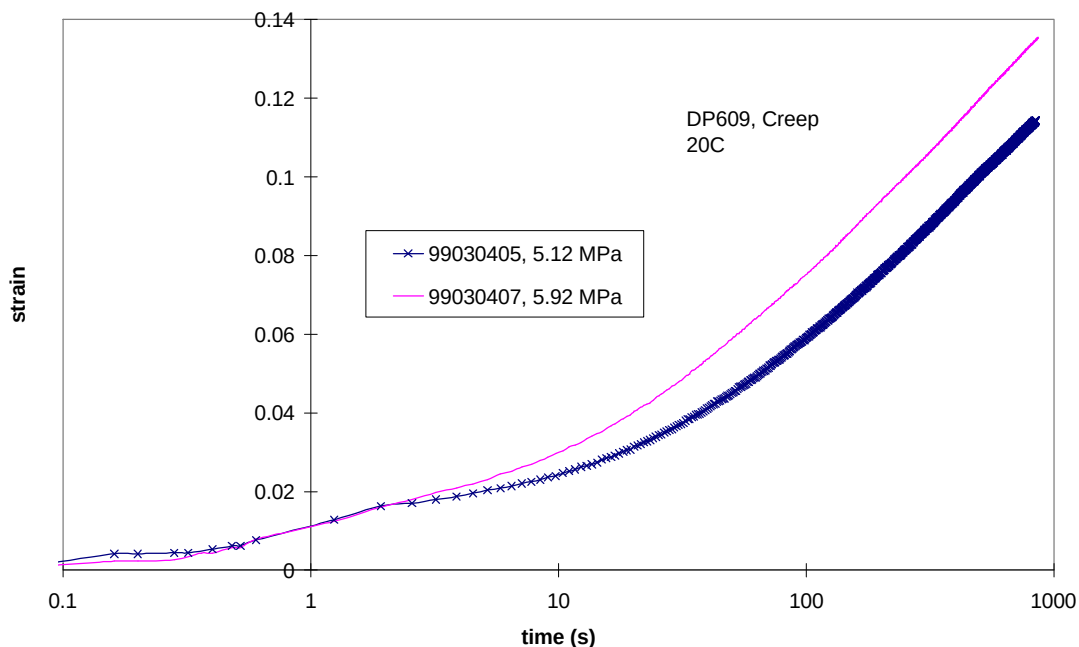
(up to 6 tests were running simultaneously). Therefore, most of the creep measurements were made using the LVDT transducers to monitor displacement of the grips.

2.1.4 Analysis for Creep Strains

Classical theories of creep assume that the test can be split into two sections - an instantaneous elastic response to the load followed by creep deformation. The creep strain is the difference between the total measured strain $\epsilon(t)$ and the initial, elastic strain ϵ_0 . For many materials, the determination of ϵ_0 in the creep tests is straight-forward. The elastic strain, ϵ_0 , is the strain where there is a discontinuity in the strain-time curve between the high strain rate, elastic loading stage and the low strain rate creep. Often it is simply the point at which two straight lines intersect.

In visco-elastic adhesives, near to or above T_g , the determination of ϵ_0 is not so straight-forward. The properties of the material are strongly time-dependent. The concepts of an elastic response followed by creep deformation are not strictly applicable. However, these data are required since this seems to be the data format for FE analysis. The strain-time curve, even during the initial loading stage, is curved. Therefore, it is difficult to identify the end of the elastic loading and the start of creep deformation. Errors in the value assumed for ϵ_0 will give rise to significant errors in the creep strain. Similarly, uncertainties also occur in the determination of the elastic displacement (d_0) used to determine the FE creep input function. This initial elastic displacement will be as difficult to determine accurately as the elastic strain.

Figure 7: Tensile Creep Strain Measurements



The measurements shown in Figures 3 and 5 indicate some of the difficulties in precisely locating the elastic strain (or displacement). The transition between 'static' loading and creep is assumed to occur where the rate of extension slows. This is in the region of the plots with the largest degree of curvature.

Figure 7 shows a set of data from which calculation of creep strains would be extremely problematic. The 3M DP609 polyurethane adhesive has a T_g close to the test temperature (20 °C). There will be significant time-dependence of the material properties near T_g . At times less than a few seconds, the material will behave as a 'glassy' polymer with a high modulus. The initial deformations are small. However, as the material relaxes, the modulus decreases significantly. A large extension rate is then measured. From these curves it is extremely difficult to separate the elastic response from the creep response. The load would have been fully applied somewhere between 1 s and 10 s.

2.1.5 Creep Stress

Creep tests are assumed to be carried out under conditions of constant stress. Obviously, in a tensile test the specimen will narrow as it is extended. Hence, a constant load will produce an increasing stress. For stiff materials, where the total strain is small, the change in dimensions of the specimen will be small. Increases in stress may be neglected. However, in low modulus materials the extensions of the specimens may be large. Therefore, contraction of the specimen may give significant increases in stress during the test. For example, using a material with a Poisson's ratio of 0.5 (the normal assumption for rubbers), a strain of 20% gives a 23.5% increase in stress above the initial value. If the strain then increased by a further 5% then the stress would be 31% above the initial value. A further 5% strain would lead to a stress of 38% above the initial value. The stress could be recalculated to compute the true stress. However, if significant creep takes place then the stress will continue to increase. The increase in stress will feed back into increased creep rate.

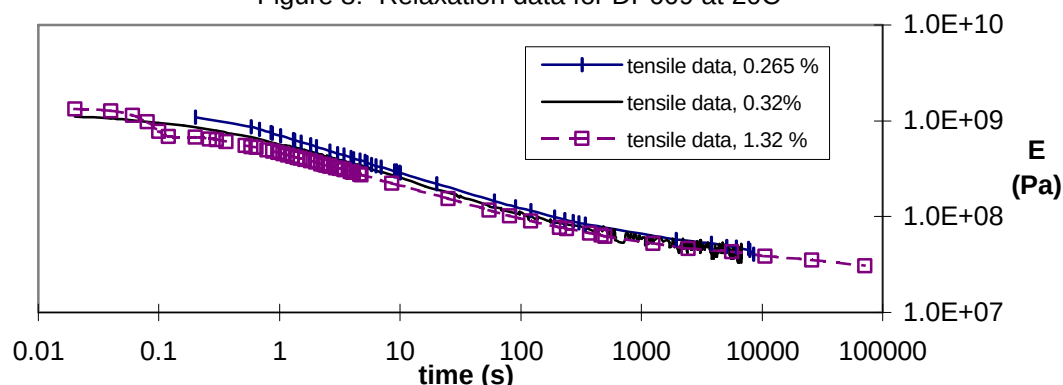
2.2 TENSILE STRESS RELAXATION

Tensile stress relaxation measurements were performed on an Instron 4505 universal test machine. This was fitted with a 1000 N capacity load cell. The specimens tested were the manufacture in the same way as the specimens used for the tensile creep measurements. The test temperature was set and maintained using a Climatic Systems temperature chamber and temperature conditioning unit. Temperatures between -10 °C and 60 °C can be maintained to within 0.2 °C.

The tests were performed by deforming the specimen at 50 mm/min until a pre-set load limit (10-30 N) was exceeded and the test machine automatically stopped. The extension was then held constant. Forces were recorded using Instron Multi-Axis Acquisition software. Strains were measured separately using a video extensometer. These were used in the calculations of relaxation moduli.

Since the specimens have relatively small dimensions and the materials low moduli, the pre-set stopping loads were small. Stopping loads of 25 N were typically used to give 5-10 % strains. These are small in comparison to the 1000 N load cell capacity. The resolution of the force cell is 0.025 N. Therefore, the sensitivity is around 0.1 % of the initial force. This limits the accuracy of the measurement.

Figure 8: Relaxation data for DP609 at 20C



Despite the measurement limitations outlined above, stress relaxation measurements tended to be reasonably accurate and repeatable. Figure 8 shows relaxation measurements made on a polyurethane adhesive (3M DP609). It was indicated earlier that relaxation times can be equated to dynamic frequencies. Dynamic frequencies of 0.1, 1 and 10 Hz correspond to relaxation times of 1 s or less. The low time modulus values of 500 MPa to 1000 MPa are similar to modulus values measured using dynamic measurement methods⁽⁵⁾.

The three repeat measurements have good agreement at relaxation times above 10 s. There are small differences between these data at low times. These are thought to be due to imprecise determination of the time of the start of the relaxation. The relaxation data points at extremely low times (< 0.1 s) have relatively large uncertainties in the time. Additionally, the modulus at low times will depend strongly on the loading history of the specimen to reach the constant test strain. Small differences in loading history may lead to different initial stresses. There is also a suspicion that the test machine may ‘back-off’ a small amount of extension when it stops suddenly.

The stress relaxation method has a number of advantages over creep tests for the acquisition of time-dependent materials properties. The stress relaxation test takes place at constant deformation. Thus, unlike the creep test, the dimensions of the specimen remain constant during the test. There are fewer difficulties in identifying the start of the relaxation than in identifying the start of creep. The start of relaxation is taken as the point where the load starts to decrease.

2.3 CREEP MEASUREMENTS IN A SHEAR RHEOMETER

Controlled stress rheometers such as the Carrimed CSL500 from TA instruments are able to apply the constant load needed to perform creep measurements. The displacement can be measured to a resolution of 10^{-5} rad (or about 0.005 %). The CSL500 is not suitable for stress relaxation measurements.

The rheometer used had been modified to increase the stiffness of the instrument. These modifications have been described in previous reports^(4, 5). The stiffness of the rheometer has been determined using ‘infinitely’ stiff specimens to be around 610 Nm rad⁻¹. Therefore, when the maximum torque of 0.05 Nm is applied, the shaft will deform by about 10^{-4} rad (or about 0.05 % strain with the measurement geometry

used). This is purely an elastic strain in creep measurement and can be subtracted from the measured strain to obtain the true sample strain. The significance of the correction depends on the stiffness of the sample. For a material with a shear modulus of 600 MPa, the displacement in the sample would equal the displacement in the shaft of the machine (i.e. the measured displacement would be twice the actual sample displacement). The adhesives studied here had shear modulus values lower than this. The compliance correction should become less significant in relation to the total displacement as the creep strain increases.

Creep measurements were performed using a parallel plate measurement system. In the parallel plate geometry both stress and strain depend on the distance from the centre of the plate⁽⁷⁾. Both stress and strain are zero at the centre and at a maximum at the rim. The shear strain at the rim (γ) is calculated from the angular displacement (ω , in radians), the plate radius (r) and the gap between the plates (s):

$$g = \frac{r}{s} w$$

The shear stress (τ) at the rim is calculated from the torque (T):

$$t = \frac{2}{\pi r^3} T$$

The stress depends on the radius cubed and will be very sensitive to this dimension.

The measurement geometry used was the 10 mm diameter system with a 1 mm gap described previously⁽⁵⁾. The maximum torque of 0.05 Nm available from the rheometer applies a maximum stress of 0.25 MPa to the specimen. This is lower than the minimum stress levels used in the tensile creep tests. Specimens were prepared and cured in the rheometer (thereby ensuring good alignment and the correct measurement gap). However, this is a slow method of specimen preparation that requires exclusive use an expensive instrument. If the cure time is significantly less than the test duration then preparation in the instrument may not significantly increase the cost of testing. Alternatively, studs could be clamped and bonded outside the rheometer using Unex grips. Excess adhesive was removed from the bondline prior to curing. This was done to control the specimen radius to that of the stud.

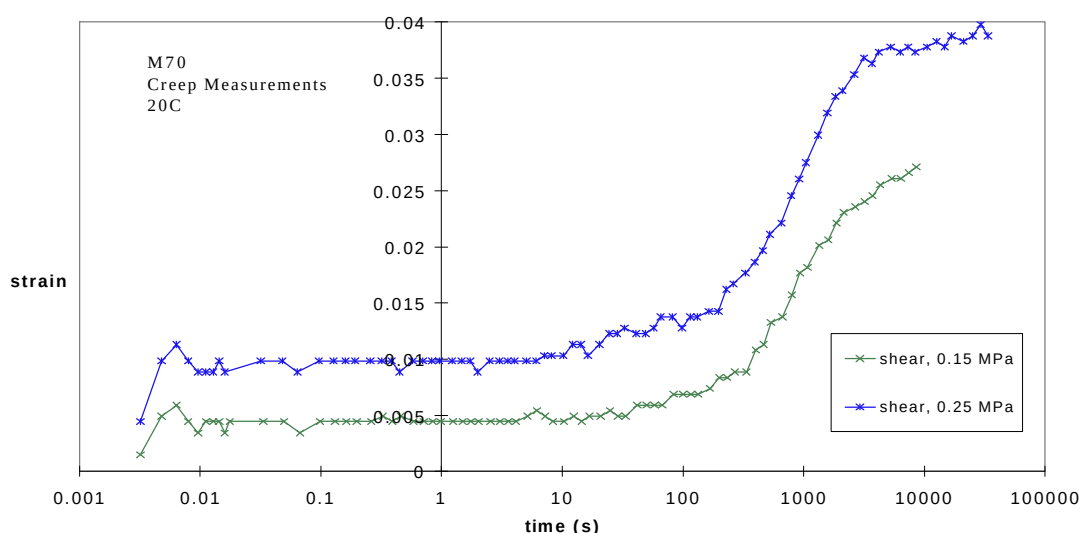
Temperature could be controlled on the Carrimed rheometer using the extended temperature module (ETM)⁽⁵⁾. This arrangement is designed to allow both extremes of cryogenic and high temperatures. Temperatures are controlled via the heating coil that fits over the sample geometry and liquid nitrogen that is pumped through the top and bottom plates. In this arrangement the stud is bonded directly to the bottom plate making the removal of excess adhesive difficult. The temperature sensor that controls this system is located in the bottom plate. At close to ambient temperature, the heating/cooling system tends to be unstable overshooting the control setting through over-heating or over-cooling.

Better temperature control can be obtained through the Peltier plate temperature control system. A significant advantage of this system is that specimens can be produced using Unex grips. Excess adhesive around the bond-line can be easily removed. The Peltier system, however, has a smaller temperature range than the ETM system. It cannot be used much beyond ambient temperature. Furthermore, the Peltier

plate is positioned some distance from the specimen. Consequently, the temperature in the sample differs from the control temperature.

Figure 9 shows typical creep data measured using the rheometer. Temperature was controlled using the Peltier system. The stress was applied to the specimen very quickly (within 0.01 s). This led to a quick, initial step in the strain. These initial strains are consistent with a shear modulus value of 15-20 MPa. This is comparable to modulus measurements made on the M70 adhesive⁽⁵⁾. There is a small initial oscillation as the instrument seeks to control the stress. The noise in the data at larger times is thought to be due to temperature instability. Fluctuations in the plate temperatures of ± 0.75 °C were measured. Long term temperature stability appeared to be a problem with this instrument. The temperature control system would need to be modified or replaced if the rheometer was to be used seriously for creep measurements.

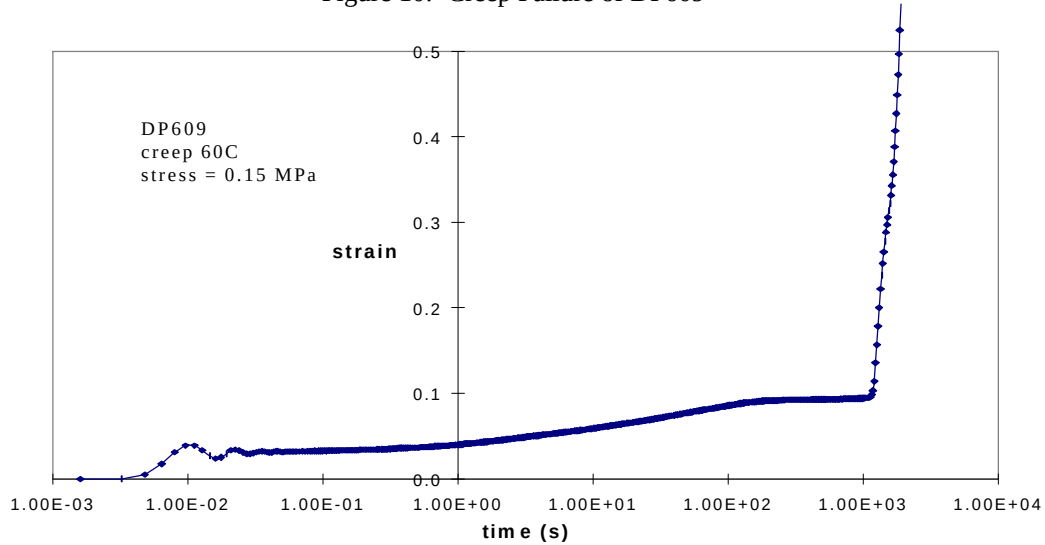
Figure 9: Creep Measurements from the Rheometer



These measurements show an increase in the rate of creep (against log(time)) after approximately 100 s. This appears to contrast with the tensile creep measurements. In tension, after the initial displacement on applying the load, the rate of extension appears linear with log(time). The shear creep measurements were made using applied stresses of approximately one tenth of those in the tensile tests. A future report will directly compare creep compliance and relaxation moduli obtained from the two techniques.

In shear tests, the specimen dimensions should not change with strain. Therefore, the increase in stress with increasing strain found in the tensile test will not occur. However, the stress and strain distribution in the sample will not be uniform. Both stress and strain will be zero in the centre of the plates and increase to a maximum at the rim. If the applied stress influences the relaxation behaviour of the material then the properties of the adhesive will vary along the radius of the plates.

Figure 10: Creep Failure of DP609



In some of the creep tests carried out on the DP609 adhesive using the rheometer (such as Figure 10), it was noticed that the strains increased rapidly to large values. This was ascribed to failure at the adhesive-plate interface. No surface preparation was used apart from cleaning the plates using a solvent. To achieve optimum bonding of DP609 onto steel the surfaces should be primed.

2.4 SHEAR STRESS RELAXATION USING A RHEOMETER

Stress relaxation measurements were made using a Bohlin VOR rheometer⁽⁸⁾. This instrument is shown in Figure 11. Strain step functions were input via the operating software. Strains were applied in around 0.01 seconds. The resulting load was monitored and the stress was calculated. Temperature control was maintained through blowing heated/chilled air into the measurement cell. This seemed to give adequate temperature control according to the instrument. However, independent measurements would be advisable to check the actual sample temperature.

The stud sample geometry could not be used with this instrument as the studs were marginally too small to fit the clamps (measurement geometries for the Bohlin have 10.04 mm shafts). Some initial tests were made using the studs before this was realised. These gave apparent relaxation moduli orders of magnitude lower than expected. Much of the applied displacement was taken up by slippage between the stud and clamp. This system did not give reliable measurements. Therefore, subsequent relaxation measurements were made using a 25 mm parallel plate system. The plate separation was 1 mm. The largest torque that can be measured is 0.02 Nm. Therefore, the largest stress available, in this geometry, is 815 Pa. This is much lower than the CSL500's capabilities.



Figure 11: Bohlin VOR Rheometer

The initial problems encountered with this instrument could have been detected easily if a compliance check had been run. In essence, the compliance of the instrument is checked through testing an 'infinitely' stiff sample. In this case, this would be a single stud. Clamping the same stud into both clamps mimics the measurement system without the compliant specimen. Clamping problems would become apparent if unrealistically high compliances for the system were measured. Compliance checks on the Bohlin VOR showed that the stiffness of the instrument was 246 Nm

rad⁻¹. This is slightly less than half that of the modified Carrimed CSL500.

Figure 12: Shear and tension relaxation data, M70 at 40C

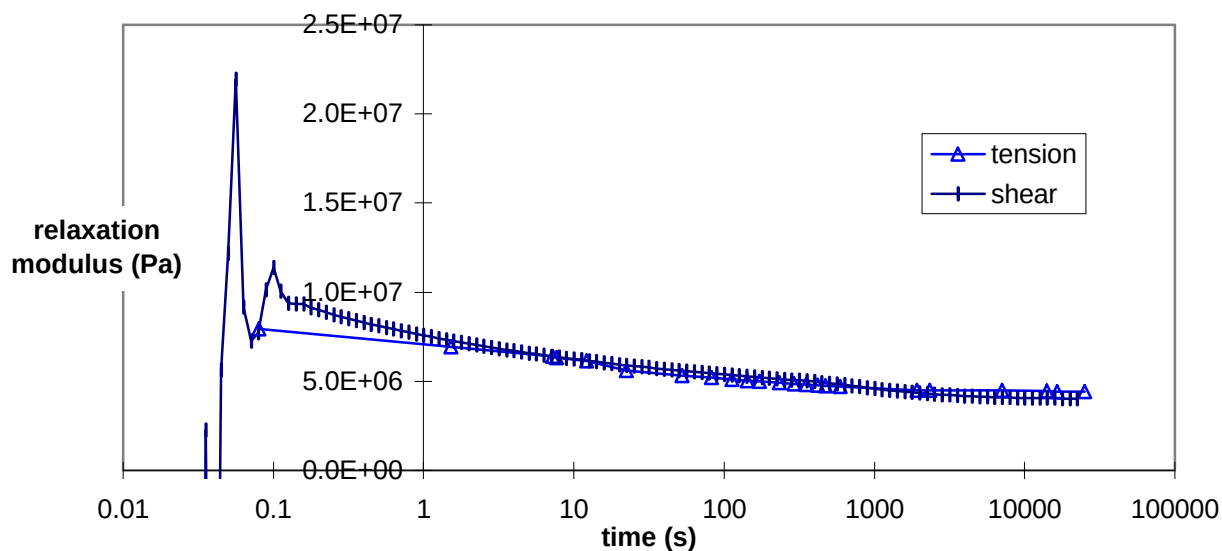


Figure 12 shows a comparison between stress relaxation measurements made in shear and in tension. At low times there is considerable noise in the shear data. This may be a consequence of dynamic oscillations in the torque sensor following the rapid application of the strain. Above 1 second, the shear and the tension relaxation moduli are comparable. This is contrary to expectations. Shear moduli would be expected to be approximately one third of the tensile moduli. However, the tensile relaxation measurement was performed at a nominal strain of 13.1 % strain whereas the shear

relaxation measurement was at 1.1 % strain. Additionally, the tensile strain was applied in 7.7s. The shear strain was applied in 0.02 s. The secant modulus to the constant rate stress-strain curve will decrease with strain. Thus, larger strains will lead to a lower initial relaxation modulus.

3. TESTS FOR TIME-DEPENDENT PROPERTIES

3.1 CREEP VERSUS RELAXATION

Either creep compliance or stress relaxation data can be used as time-dependent visco-elastic properties of a flexible adhesive in the ABAQUS FE solver. Creep measurements are converted to stress relaxations using the convolution integral. Prony series relaxation constants are then determined⁽²⁾. Initial experiences with creep analyses using ABAQUS suggest that relaxation data give fewer problems in the data conversion stage.

Separating the elastic response from the creep response of the flexible adhesives is difficult. The strain (or displacement) versus time plots are normally curved. Identification of the completion of the elastic deformation is difficult. Unfortunately, this elastic deformation is needed to calculate the normalised creep compliance needed for the FE. In theory, the elastic deformation could be calculated from the stress-strain behaviour measured in constant strain rate tests. However, the properties are sensitive to rate. Predictions of the elastic deformation made using data not pertaining to rate of extension when applying the load could contain significant errors.

In contrast, the initial stress required for normalising the stress relaxation data is easily determined from the maximum load applied. There may be some initial uncertainty over the start of the relaxation but these will become less significant at longer times.

If the creep extension of the specimen is large then the contraction of the specimen gauge section will become significant. The assumption that the local stress applied to the material is constant throughout the test will not hold as the load bearing area will contract. The specimen will experience increasing stress as well as increasing strain. In stress relaxation measurements the deformation is constant and only the stress will vary.

For the purpose of determining time-dependent visco-elastic properties of flexible adhesives the stress relaxation test has many advantages. However, if information on how the material or adhesive bond may rupture under load is required then creep measurements have to be made. Test specimens will not normally fail in relaxation tests unless they fail during the initial extension of the specimen.

3.2 SELECTION OF APPARATUS

Selection of the test method for determining the time-dependent properties of a visco-elastic material is determined partly by the data required and partly by the economics of the testing. Long term test measurements are expensive. Test conditions must be maintained throughout the measurement. Expensive equipment must be dedicated to measurement of single samples. Therefore, it is rare for long term creep or relaxation

tests to be carried out on expensive test equipment such as mechanical test machines or rotational rheometers. Such equipment may be used for short term creep or relaxation tests (generally less than 1 day). Longer term tests normally use simpler, less costly apparatus.

For these reasons creep tests are commonly performed. Creep tests also give information on the failure of the material or bond. The temperature can normally be maintained adequately by room control or individual temperature chambers. The loading frames and displacement transducers are relatively inexpensive.

Stress relaxation tests can also be carried out for long term properties. The requirements are adequate temperature control, a jig for applying (and maintaining) the strain and a force transducer that has long term stability. An example of a simple relaxation test method is the bolted joint stress relaxation test⁽⁹⁾. A simple jig holds the sample and load washer. Compressive strain is applied through tightening the bolt. The force measured by the load washer is logged. The relaxation in the applied force is measured. However, the geometry of the test specimen leads to a non-uniform stress distribution. The strain on the specimen is not measured. Therefore, relaxation moduli are not calculable.

4. CONCLUSIONS

Rheometers are versatile instruments. The more sophisticated rheometers are capable of performing creep or stress relaxation measurements on low modulus materials such as flexible adhesives. However, these instruments are limited to low stress measurements owing to the low power of the drive motors and the limited range of the force transducers. Temperature stability appears to be poorer than in the temperature cabinets used for bulk tension specimen tests. This could limit the accuracy of the measurements. Rheometers are expensive instruments and it may not be economically feasible to dedicate one to a few long term tests. Multiple sets of apparatus are normally required to complete a series of long term tests within an acceptable period.

A tensile creep test has been developed for bulk specimens of flexible adhesives. Dead weight loading is not a suitable test configuration. The lever arm loading configuration gives better quality data. However, there are problems with calculating strains from the measured grip movement. The shape of the material response curves makes the separation of the elastic and creep portions of the data, needed for FE analysis, difficult.

Stress relaxation is a more reliable technique for determining time-dependent, visco-elastic properties for FE analysis. However, data on failure of the material cannot be obtained through this method.

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