

CREEP OF FLEXIBLE ADHESIVE JOINTS

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Summary

Commercial finite element analysis (FEA) software packages are used extensively for the prediction of the performance of adhesive joints. The accuracy of any prediction will depend on the validity of the material model employed and the reliability of the test data input into the chosen model. The aim of this work was to carry out an initial investigation into the creep properties of flexible adhesives.

FE software packages incorporate visco-elastic models for describing time-dependent material properties. Data are required from either creep tests or stress relaxation in order to calculate the relaxation constants for the models. Creep measurements have been carried out on bulk specimens to derive input data for the FE models and to investigate the failure of such materials under long-term loads. Despite the scatter in the data there is some evidence to suggest that there may be simple rules to predict long-term performance. In particular, constant rate tests performed at elevated temperatures may give information relating to the long-term failure criteria for the joint.

FE predictions of the behaviour of lap joints under constant loads have been compared with experimental measurements. The relaxation model used in the FE software relies on normalised input data. This means that the long-term FE predictions are critically dependent on the accuracy of the initial 'elastic' extension of the joint under load. Since the joint is rarely loaded at the same rate at which the elastic properties are obtained the accuracy can be poor. This is an area requiring further research. More suitable models making use of actual material properties such as compliance and incorporating stress dependence of the visco-elastic function are likely to increase the reliability of the predictions.

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1. INTRODUCTION

As design philosophies change, flexible adhesives are becoming considered for bonding applications where structural performance is important. However, the development of design tools for flexible adhesive joints has lagged behind those for structural adhesives. Project PAJ1 of the DTI Materials Metrology Programme “Performance of Adhesive Joints” seeks to address some of the design issues relating to flexible, visco-elastic adhesives. For flexible adhesives to be used in structural applications the durability of the joints must be considered as it would for structural adhesive bonds. Since the maximum strength of the flexible adhesives is often small, creep problems may become significant at low levels of load. The joints need to be designed to sustain long-term structural loads in addition to short-term or transient loads. The aim of this report is to describe how methods for measuring and modelling the creep performance of flexible adhesive joints could be implemented.

Finite Element Analysis (FEA) is a powerful tool for predicting the performance of adhesive joints. However, to make reliable analyses, suitable materials models and accurate materials property data are required. Flexible adhesives have received less attention than structural adhesives. Consequently, reliable methods for modelling flexible adhesives are not available. Other work in project PAJ1⁽¹⁾ has indicated that the simple Elastic and Elastic-Plastic material models are unsuitable for characterising flexible adhesives. The hyperelastic models that characterise rubber elasticity may offer a reasonable approximation to the flexible adhesives. However, there are still outstanding issues regarding the acquisition of input data and implementation of the models still to be overcome.

Creep is handled in FE software through visco-elastic models⁽²⁾. These models apply time dependency to the materials parameters such as the elastic modulus or the hyperelastic coefficients. This time dependency is then implemented through either dynamic analyses (e.g. ABAQUS/EXPLICIT) or pseudo-dynamic analyses (e.g. using the *VISCO function in ABAQUS/STANDARD). The input data used to define the visco-elastic model (*VISCOELASTIC) are derived by fitting an exponential decays (Prony) series with one or more relaxation terms to creep or stress relaxation test data. ABAQUS also contains a plastic creep model (requiring the creep strain rate as input data). This was not investigated in the current study as the concept of hyperelasticity excludes plastic deformation.

Stress relaxation is probably the more reliable of the two methods for obtaining time-dependent mechanical properties^(3, 4). However, relaxation measurements reveal very little about how the material will rupture. Creep tests can be used to obtain failure hence their widespread use. Rubber theories suggest that rupture criteria are related to the strain energy in the material. This may also apply to flexible adhesives, although the initial data are inconclusive.

2. CREEP MEASUREMENTS

2.1 BULK SPECIMEN TESTS

Tests were performed on two adhesives; 3M DP609, 2-part polyurethane, and Evode M70, 1-part elastomer. Bulk specimen creep tests were carried out in tension using the test methods described in an earlier report⁽³⁾. Analysis of the data produced using the underslung loading technique showed that the uncertainties in these data were large⁽³⁾. Consequently, most of the data reported here were obtained from overslung lever tests.

The creep displacements were measured from the movement of the loaded arm or of the weight carriage. The creep strain was calculated using a correction factor (1 mm extension = 0.0184 strain) determined from an analysis of strain measurements obtained using a video extensometer. These strains have large uncertainties as the measured extension includes non-repeatable effects such as movement of the specimen in the grips.

Creep tests were performed at a variety of temperatures and stress levels. Due to the limitations of the size of the cross-section of the test specimen (typically 5 mm x 1 mm), and the available load increments (5 N), the minimum stress applied to the specimens was often a significant proportion of the failure stress of the adhesive (summarised in Table 1). Times to failure were often quite low. Only a limited number of tests could be conducted due to time and equipment limitations. Tests where the specimen had not failed after approximately one week were terminated to make way for another test.

Table 1a: Uniaxial Tensile Data for DP609

Temperature	Maximum Stress (MPa)	Maximum Strain %
-20	45.7 ± 2.4	4.0 ± 0.7
-10	39.6 ± 3.7	9.4 ± 6.5
0	34.5 ± 2.7	6.2 ± 2.4
10	26.5 ± 1.8	86.7 ± 8.8
20	20.7 ± 3.6	103 ± 13
30	15.1 ± 1.3	77.5 ± 3.0
40	7.9 ± 0.5	53.9 ± 1.8
50	7.8 ± 0.4	42.7 ± 2.0
60	4.2 ± 0.3	27.9 ± 4.3
70	4.3 ± 0.5	25.5 ± 3.7
80	3.5 ± 0.4	18.8 ± 3.0

Table 1b: Uniaxial Test Data for M70

Temperature	Maximum Stress (MPa)	Maximum Strain %
-20	3.99 ± 0.41	38 ± 8
-10	3.03 ± 0.21	47 ± 6
0	1.79 ± 0.28	58 ± 8
10	1.75 ± 0.13	60 ± 10
20	1.61 ± 0.28	48 ± 13
30	1.55 ± 0.26	44 ± 12
40	1.21 ± 0.20	31 ± 5
50	1.23 ± 0.18	38 ± 10
60	1.25 ± 0.23	37 ± 6
70	1.02 ± 0.21	31 ± 1
80	0.97 ± 0.24	32 ± 13

Creep tests were performed on the two adhesives (i.e. 3M polyurethane DP609 and Evode elastomer M70) at four different temperatures (20 °C, 40 °C, 60 °C and 80 °C). The data for different initial stresses are shown in Figures 1 and 2. The strains were determined from tests in which the extension of the specimen was determined from the movement of the top grip in an overslung lever arrangement⁽³⁾. Strains were calculated using the correction factor of 0.0184 strain per mm that was previously estimated.

The measurements made on DP609 at 20 °C (close to the glass transition temperature, T_g , where the visco-elastic nature of the material is most pronounced) differ significantly from the other temperatures. In many tests, there are no readily distinguishable differences between the initial 'elastic' stage as the specimen is loaded and the creep stage. For most of the tests on M70 and DP609 at other temperatures there is an initial rapid rise in displacement as the load is applied followed by a slower extension under creep.

To analyse the data independently of the applied stress, the strains were converted to compliances, $J(t) = \epsilon(t)/\sigma$, where $\epsilon(t)$ is the time-dependent strain and σ is the applied stress. It should be noted that in tension the stress is not constant. The reduction of the specimen cross-section as it extends will lead to an increasing stress with time. The compliance data for DP609 and M70 are shown in Figures 3 and 4 respectively. The graphs suggest that the compliance is greater for higher stresses. This is expected given the curvature of the constant strain-rate stress-strain curves determined from tests such as the uniaxial tensile test. Figures 3 and 4 also show that there is significant scatter between curves measured at similar stresses. This may be partially due to variability of the adhesive specimens but may also indicate the presence of measurement uncertainties. The plots show little differences between the compliance values determined for DP609 over the temperature range from 40 °C to 80 °C. Similarly, within the experimental scatter, there appears to be little difference between M70 compliance values over the range 40 °C to 80 °C.

ABAQUS requires input data in the form of either creep compliances or stress relaxation moduli (both normalised by the 'elastic' value) to model time-dependent behaviour. To obtain these data, an elastic strain was determined for each creep curve - using the point where the strain rate decreases abruptly. These and subsequent strains were normalised through dividing by this elastic strain. Thus, the normalised creep compliance at the beginning of the creep phase (creep time = 0) is 1.0. Figures 5 and 6 show normalised compliance data calculated from the creep tests. It is noticeable that the curves can be exceedingly scattered making it difficult to select a single representative curve at each temperature to use in the FE model. Some of this scatter will be due to uncertainties in the elastic strains used. This is likely to be most significant for DP609 at 20°C - the difficulties in determining 'elastic' strains from these data were mentioned earlier. In many cases it was not possible to select an 'elastic' strain from the curves and the strain at 10 s after loading was used. This set of data gave the most scatter amongst the normalised compliance curves - for instance at 1000 s the normalised compliance values vary between 1.5 and 8. In contrast, at the other temperatures normalised compliance values rarely exceeded 1.5.

2.2 JOINT SPECIMEN CREEP TESTS

Lap joint specimens were prepared using 25 mm wide by 3 mm thick steel strips with an overlap length of 12.5 mm as previously reported⁽¹⁾. The adherends were grit blasted and solvent degreased prior to bonding. Creep tests were performed using an overslung leverarm machine. The jaw faces of the grips were offset to align the bondline parallel to the axis of loading. Initial tests where only the movement of the grips was measured showed that the deformation of the adhesive bondline was unlikely to account for all of the compliance of the system.

Figure 7 shows an alternative displacement measurement technique that was used to determine the extension of the adhesive. A displacement transducer was attached to the adherend that was clamped in the lower, fixed grips. The head of the transducer contacted the bottom of the top, movable grip. The movement of the top grip relative to the lower adherend was thus measured. This arrangement compensated for most of the compliance of the system. It is still sensitive to movement of the specimen within the top grip. An arrangement where the head of the transducer is fixed to the upper adherend would remove the uncertainties due to movement of the sample in the top grip. The testing schedule did not allow for many tests exceeding one week and many specimens were only tested for approximately 1 hour.

Creep tests were performed on DP609 and M70 lap joints at two temperatures (20 °C and 40 °C) under various levels of stress. The total extensions measured from lever arm movement in tests on DP609 and M70 performed at 20 °C are shown in Figures 8 and 9 respectively. Qualitatively, the graphs look much like most of the bulk tensile creep tests showing an initial 'elastic' displacement followed by creep. This shape of the creep curve even occurs with DP609 at 20 °C (which is significantly different from the form of the DP609 tensile creep curves at 20 °C). The size of the measured initial displacement shows no relationship to the size of the applied stress. It is thought that the measured deflection includes other contributions besides the shear extension of the adhesive layer in the lap joint. One obvious contribution is movement of the specimen

within the grips. Since the bondline thickness was approximately 0.5 mm, the strains implied by these extensions of the specimen (often > 1 mm) are physically unrealistic. Where there was little movement of the system, in addition to the extension of the bondline, the low resolution of the long travel LVDT displacement transducer led to poor accuracy.

Measurements from the displacement transducer attached between the lower adherend and the upper grip are shown for DP609 and M70 in Figures 10 and 11. These indicate substantially lower extensions. However, in most of the measurements, the logarithmic creep rate measured by this transducer is very similar to the creep rate determined from the total extension. There are still large variations in the initial, 'elastic' extension values determined in the tests. The extensometer measured the movement of the top grip relative to the bottom adherend. This not only includes the extension of the bondline, but any movement of the specimen in the top grip.

3. CREEP FAILURE RESULTS

3.1 BULK SPECIMEN DATA

The time available for tests was limited. Therefore the bulk specimen creep data measured in this work are relatively short-term data. Since the aim was to generate data on the failure of the adhesives, relatively high levels of creep stress were applied. Most of the tests were removed after approximately 250,000 s (3 days). Only a few tests were allowed to reach 1,000,000 s (12 days).

3.1.1 DP609 Results

Figure 12 shows a plot of maximum strain against applied stress for DP609 specimens that failed during the creep tests. The datasets show results for 20°C, 40°C, 60°C and 80°C tests. The data all appear to lie on the same general curve. The mean values for failure stress and failure strain (in Table 1a) determined from uniaxial tension tests performed at constant strain rate are also shown. These give a curve similar to the creep results.

The levels of applied stress in the creep tests were normalised with respect to the failure stress from the constant rate tests (F_s) at each temperature. Figure 13 shows a plot of these normalised applied stress values vs time to failure. Many tests were performed at normalised stress levels above 0.75 F_s .

- The times to failure above 0.75 F_s are extremely scattered. However, virtually all these tests failed within 10,000 s (3 hours).
- Only at the higher test temperatures did specimens above 0.75 F_s survive 10,000 s.
- Trends in the data are more recognisable at lower levels of normalised stress.
- The normalised creep strength retention of the specimens at constant time declines with temperature as the material approaches T_g .
- The curve for DP609 at 20 °C lies below the other temperatures. At 0.5 F_s , failure typically occurs within 1,000 s.
- Only below 0.2 F_s do the specimens at 20 °C survive beyond 100,000 s.
- In contrast, 60°C and 80°C tests at 0.5 F_s tend to survive beyond 100,000 s.

The data shown in Figure 12 suggest that the effect of a constant load is to increase the effective 'temperature' of the material leading to failure at stress and strain levels associated with higher temperatures. This may be due to some de-ageing mechanism due to stress increasing the mobility of the polymer chains and, hence, reducing the glass transition temperature of the adhesive. However, the concept of the age of the specimens influencing the time-dependent material properties is normally applied to glassy polymers at temperatures below their T_g . Above T_g , the mobility of the chains is assumed to be sufficient that molecules rearrange rapidly in response to applied stresses.

The concept of stress shifting the effective temperature of the adhesive may be useful in drawing up simple design rules for adhesive joints under creep. The levels of stress and strain at which the bulk specimens failed were analysed in conjunction with the constant rate data to determine the increase in effective temperature in the test. These were then plotted as a function of creep time (Figure 14). The data are scattered. The effective temperature shifts for the 20 °C test specimens tend to be much larger than for specimens tested at higher temperatures. However, the trend is that the temperature shift increases with creep time. Therefore, the knowledge of how the effective temperature shifts with time under stress could be combined with information on the temperature dependence of strength for constant rate tests to produce a method for predicting the de-rating of joint strengths due to time under constant load

3.1.2 M70 Results

The M70 has a lower tensile strength than DP609. However, the tensile strength was less sensitive to temperature. Unfortunately, the tensile strength of M70 is comparable with the lowest stress levels that could be applied using the overslung leverarm test method. Consequently, there are few results for specimens tested at normalised stress lower than 0.5. The majority of the tests were conducted at normalised stress levels above 0.75. As Figure 15 shows, there are few obvious trends in the failure strain-failure stress plots. Most of the specimens failed at strains below the average failure strains measured in constant rate uniaxial tension tests. There appears to be only a small dependence of time to failure on the normalised applied stress (Figure 16). The times to failure for similar stress levels can be widely scattered. Many specimens survived beyond 100,000s at normalised stress levels greater than 0.8. However, other specimens at similar levels of stress failed within 100s. The specimens contained varying levels of voids and this may partially account for the scatter in the times to failure.

3.2 JOINT SPECIMEN TESTS

Most of the joint creep tests were performed at 20 °C. Only a limited number of specimens were tested at 40 °C. The number of lap joint specimens that failed during the creep tests was few as many tests were only run for a short duration. In many tests analyses of the output attached to the specimen indicated problems with the measurement (such as poor attachment to the specimen or transducer saturation) leading to the omission of these extension results (the times to failure measured were still valid for analysis). The data from the second extensometer (a long travel LVDT

measuring the total movement of the lever arm) were still available from these tests. However, the reliability of the initial extensions given by this method is extremely suspect and, thus, the failure extensions cannot be relied upon.

At 20 °C, few DP609 specimens failed at applied stress levels below 1 MPa. At higher stress levels (1-2 MPa), the incidence of failure increased although the times to failure were scattered (Figure 17). This probably indicates variability in the quality of the joints. These DP609 specimens were prepared from adherends that had been grit blasted and degreased. The stress and strain at failure of such specimens in constant strain rate tests⁽¹⁾ were 4.9 ± 0.5 MPa and 0.32 ± 0.05 MPa respectively. Figure 18 shows a plot of extension at failure (as determined from the extension measured by the transducer attached to the specimen) against applied stress. It is interesting to note that the extension at failure decreases with applied stress. This is the opposite of the bulk specimen results. The higher applied stress levels may cause more localised growth of stress concentrations near the ends of the joint leading to 'premature' peel failures. The qualitative finding of the bulk specimen data that only specimens experiencing creep stress levels below 0.2 of the constant rate strength had a high probability of surviving beyond 100,000 s also appears to apply to the joints.

At higher temperatures, the ability of DP609 joints to withstand creep loads declines. Failures occur at stress levels less than 1 MPa at 40 °C. No constant rate data were determined at these higher temperatures for joint specimens that had been prepared using grit blasting and degreasing. Hence, comparable constant rate failure stress and strain data are not available for comparison with the creep tests. However, for degreased only DP609 joints (not tested in creep) the constant strain rate strengths at 40 °C and 60 °C were 70 % and 50 % respectively of the 20 °C strength. These relative decreases in strength with temperature may also apply to the lap joints tested in creep.

The mode of failure in the DP609 creep tests was peel of the adhesive from one adherend. This is unchanged from the constant rate tests.

The test results for the M70 joints indicate that at 20 °C, failure of joints below 1 MPa applied stress was extremely rare. At stresses of 1 MPa and above, specimen failures were nearly always achieved within a reasonably short duration (Figure 17). This is similar to the performance of the DP609 joints. The constant rate tests on M70 lap joints at 20 °C gave typical failure strengths and extensions of 1.2 MPa and 0.9 mm respectively. M70 joints thus appear to be able to withstand creep under reasonably high proportions of their constant rate strengths. This is in broad agreement with the trends in the bulk specimen results. At stress levels above the constant rate strength failure is almost instant. Extensions at failure were in the range 0.7-1.0 mm, close to the constant rate failure extensions. Specimens that did not fail in creep did not reach these extensions. A few tests at 40 °C were performed at stress levels of 0.5 MPa or less. No failures were seen to occur under these conditions. The constant rate strength at 40 °C is around 0.9 MPa. The results are consistent with the supposition that M70 joints fail in creep only at relatively high fractions of their constant rate failure loads.

Those joints that did fail in creep did so through cohesive failure through the centre of the adhesive layer. This is the mode of failure found in constant rate tests.

3.3 FAILURE OF CREEP SPECIMENS

There was neither the time nor a suitable range of the applied stress increments to obtain sufficient data for long-term creep to make definite conclusions regarding the long-term performance of the adhesives. However, a number of points relevant to joint design were noted.

- The mode of failure of joint specimens is the same in creep tests as it is in constant strain rate tests.
- Stress retention (in relation to the constant rate strength) appears to be worse near the glass transition temperature.
- The stress-strain values characterising failure under creep for bulk specimens of one of the adhesives appear to fall on the stress-strain curve determined for failure in constant strain rate tests at different temperatures. Hence, there seems to be a correlation between load duration and 'effective' temperature (a time-temperature 'superposition'). Time under load may increase the 'effective' temperature of the adhesive.
- Using the 'superposition' idea it may be possible to relate the load at which a joint fails under creep (within a specified duration) to the failure load in a constant rate test at a higher temperature. Thus, it may be possible to determine sustainable service loads from elevated temperature tests.

4. FE MODELLING

4.1 FE MODEL

The FE mesh used for the creep analysis of the lap joint was virtually identical to the one used for the analysis of the monotonic loading case described in a previous report⁽¹⁾. The only difference was the use of plane stress elements (CPS8R - cubic, plane stress, 8-noded, reduced integration) rather than plane strain elements (CPE8R - cubic, plane strain, 8-noded, reduced integration). This was necessary as plane strain elements cannot be used in ABAQUS for hyperelastic material models incorporating visco-elasticity.

The Mooney-Rivlin model was used for the elastic, time-independent properties of the adhesives. The input coefficients determined in the earlier work were used. The D (volumetric) parameter was assumed to be zero (i.e. incompressible). However, it is known from Poisson's ratio measurements that the assumption of incompressibility is unlikely to hold.

Analyses were run with constant static loads applied to the free end of the joint specimen. The analyses consisted of two steps. The first step was either used *STATIC (time-independent) or a *VISCO (5 s duration, creep allowed) to apply the load (*CLOAD). The second step used the *VISCO function to model the joint under a constant load. Displacement predictions were recorded at the loaded end and two gauge points either side of the bondline. The results showed few differences between

the extension of the gauge points and the movement of the free end. This is expected as the stiffness of the adhesive layer is insignificant in comparison to the steel adherends. The extension of the steel adherends under the low levels of force is negligible.

4.2 EFFECT OF INPUT VISCO-ELASTIC DATA

4.2.1 *VISCOELASTIC Model

Time-dependent properties of a material can be specified in ABAQUS using the *VISCOELASTIC option. In the simple elastic case, the dimensionless relaxation modulus $g_r(t)$ is defined by the a Prony series⁽²⁾:

$$g_r(t) = 1 - \sum_{i=1}^N g_i^{-p} (1 - e^{-t/\tau_i})$$

where N is the number of terms, g_i^{-p} is a material constant and τ_i is the relaxation time that characterises the rate of decay of the stress. The relaxation modulus, $G(t)$, is obtained by multiplying the instantaneous ‘elastic’ modulus G_0 by $g_r(t)$. In the case of hyperelastic materials the relaxation function is applied to the constants that define the strain energy function. The long time modulus G_∞ is defined equal to:

$$G_\infty = G_0 \left(1 - \sum_{i=1}^N g_i^{-p} \right) \quad (1)$$

Visco-elasticity is incorporated in the hyperelastic models by applying the same relaxation function to the hyperelastic coefficients. If volumetric test data are available then a (separate) relaxation function can be applied to the volumetric coefficients.

The time-dependent properties (relaxation constants, characteristic relaxation times) can be directly input into the material property definition. However, normally these are not available and material data in the form of stress relaxation or creep compliance data from which ABAQUS calculates the Prony coefficients. These data must be normalised to the instantaneous modulus or compliance. The normalisation is performed by dividing the time-varying creep compliance or relaxation modulus by the initial, ‘elastic’ value. Thus the normalised creep compliance increases from one and the normalised relaxation modulus decreases from one. ABAQUS fits the relaxation coefficients to relaxation data. Therefore, the creep compliance data (J) have to be converted to relaxation modulus data (G) using numerical integration of the convolution integral:

$$\int_{u=0}^t G(t-u)J(u)du = t \quad (2)$$

This is potential source of error in the derivation of the relaxation coefficients. However, the computed relaxation moduli are not available to the user. Therefore, the reliability of the data conversion cannot be verified.

The maximum number of terms (or order), N, of the Prony series may be specified. Alternatively, ABAQUS selects the order of Prony series to restrict fit errors below a specified value (set as **errtol** in the ABAQUS input line). Normally N will be in the range 1 to 3. The quality of the fit of the model to the relaxation data will influence the accuracy of the predictions.

ABAQUS assumes that the material is linear elastic - the relaxation function is independent of the level of applied strain (relaxation tests) or stress (creep tests). The test data presented earlier show that this is not always the case. However, generalised models that account for non-linearities in the visco-elastic properties are not implemented in ABAQUS. There is also the assumption that the relaxation function determined using uniaxial data applies to multi-axial data.

Figure 19 shows creep predictions for a DP609 lap joint under 0.25 MPa stress at a temperature of 20 °C. This adhesive and temperature were the most 'challenging' of the various combinations to model accurately. The tensile creep data are variable, the elastic extension is difficult to determine accurately and the material properties are very sensitive to many variables including strain rate, temperature, specimen age, moisture content and thermal/cure history.

Many of the models predict that the extension will reach a stable 'long-term' value within a finite time, as shown in Figure 19. This is not observed experimentally. It is an artefact of the fit function as the sum of the g_i terms determines a maximum relaxation magnitude that cannot be exceeded. It is worrying to note that least squares fitting routine often gives a solution where the relaxation timescales can be much lower than the times covered by the creep input data. Extrapolation of short-term data to long-term service predictions is potentially highly inaccurate. A limiting long-term creep compliance or relaxation modulus can be specified in ABAQUS using the **shrink** operator in the input file. This, of course, requires knowledge of the long-term material properties.

4.2.2 Sensitivity to Input Data

The predicted creep displacements are very sensitive to the time-dependent data input into ABAQUS. Predictions of the lap joint behaviour under the same creep conditions (0.25 MPa stress, 20 °C) using different test data are shown in Figure 19.

The two sets of creep test data, set A (6.13 MPa, initial strain 3.18 %, 'elastic' modulus 172 MPa) and set B (3.01 MPa, initial strain 5.5 %, 'elastic' modulus 55 MPa) represent typical high creep rate and low creep rate behaviour respectively in Figure 5. The period of load application in creep tests was typically 1-5 s. The maximum sampling rate of the data logger used was 1 point per second. Therefore, very small time properties of the materials could not be determined from these tests.

The stress relaxation data, case C, (strain 0.275 %, initial stress 3.7 MPa, 'elastic' modulus 2500 MPa) were determined following a rapid application of the strain. A high sampling rate recorder was used. Consequently, relaxation moduli were determined at very small times (<0.05 s). This difference in the effective minimum loading time probably accounts for the large differences between the 'elastic' moduli determined in the two types of test. The material properties are not determined under the same conditions. The stress relaxation modulus over the first few seconds decreases by a factor of 5-10. This gives an 'elastic' modulus closer to the creep data.

The stress relaxation data used in the FEA included the very small time results. The rate of change in the normalised stress relaxation in the first few seconds of the data is very large. This is modelled as a high creep rate. This modelled rate is much faster than the measured creep rate. Hence, the predictions in Figure 19 showing a much higher degree of creep when the stress relaxation data was used to calculate the visco-elastic relaxation constants. The differences between the creep set A and the creep set B results reflect the different creep behaviour of the input files. All these predictions were made using 1st or 2nd order Prony series. The FEA used two *VISCO steps, allowing creep during the load application.

None of the predictions agree with the experimental data also shown in Figure 19. The failure of the predictions is likely to be due to several factors:

- The loading in the creep test is around an order of magnitude faster than in the constant strain rate tests used to determine the time-independent hyperelastic material properties. Therefore, it is likely that the initial extension of the joints predicted from these properties will be overestimated. The 'elastic' tensile modulus estimated from the measured initial extension of the lap joint was ≈ 37 MPa. The tensile modulus (E_0) determined from the Mooney-Rivlin coefficients ($E_0 = 6(C_{01} + C_{10})$) is 22.5 MPa. This probably accounts for the differences between the initial 'elastic' extensions (arbitrarily assumed to be at 1 s) found for the measured and predicted curves. The larger 1 s extension predicted using the stress relaxation data (set C) is due to the large degree of initial relaxation/creep occurring in the initial *VISCO stage. Using an initial *STATIC step brings the 'elastic' extension into agreement with the other predictions, but does not alter the long-term behaviour significantly.
- The relaxation behaviour is likely to be stress sensitive. None of the input data for the visco-elastic relaxation model were determined under applied stress levels close to 0.25 MPa. Development of the tensile creep apparatus to allow smaller load increments would be needed to obtain these low stress data. It is encouraging to note that the predictions incorporating creep measurements made at the lowest level of stress are closest to the experimental data.
- The shapes of the creep curves suggest poor fits to the visco-elastic relaxation model. The time constant of the predicted relaxation is much lower than the experimental data or the input bulk creep data. The sigmoidal shape of the creep prediction overestimates the creep rate for short time intervals. It also erroneously predicts a constant, 'long-term' extension within finite times.

4.2.3 Effect of Different Order Prony Series

ABAQUS models time-dependent visco-elasticity as the product of the instantaneous material property and a sum of exponential relaxations. The more relaxation terms in the sum the better the fit to the experimental data. As the data in Figure 20 show, the number of terms in the Prony relaxation series has a significant effect on both the shapes of the predicted creep curve and the long-term extension predicted. Generally, the larger the order (N) the longer is the duration of the relaxation event and the greater the predicted creep. Increasing the number of relaxation terms from $N = 1$ to 3 in the predictions using the set A data leads to a broadening of the duration of the

predicted creep and an increase in the magnitude of the long-term plateau extension. The differences seem greater than would be expected from the RMS fit errors which were ca. 13.86 % for $N = 1$ and 13.26 % for $N = 3$.

The effects of using 2 and 3 relaxation terms on predictions using the set C relaxation data are extremely dramatic. The rate of creep is extremely rapid reaching extensions in excess of 0.8 mm within ca. 100s (at which point the analyses failed to converge). There was no indication of any tendency towards a maximum extension. The RMS fit errors to the relaxation data were small in comparison to those for the creep data (at 7.5%, 1.86% and 0.7% for $N = 1, 2, 3$ respectively).

Figure 21 shows a comparison between the measured relaxation data and the fit predictions for $N=1$ and $N=3$. It is obvious that the $N=3$ fit is much better than the $N=1$ fit. The $N=1$ fit shows a sigmoidal shape with an asymptote at 0.086 (this is equivalent to a long-term normalised creep compliance of ca. 12). The actual relaxation data tend towards 0.017 and the $N=3$ asymptote is 0.022 (equivalent to normalised compliance of ca. 45). The large ratio of the long-term normalised relaxation moduli for $N=1$ to that for $N=3$ explains the dramatic differences between the creep predictions. It is not possible to make this sort of comparison for constants determined from creep data as the intermediate 'relaxation' data calculated by the software are not available to the operator.

Earlier work⁽³⁾ indicated that the long-term stress relaxation modulus measured in tension was approximately the reciprocal of the long-term tensile creep compliance. This suggests that it should be possible to use the data interchangeably to predict long-term creep properties. However, ABAQUS uses normalised data to model the time-dependent visco-elastic properties. These are critically dependent on the initial values selected for the normalisation. As this work shows, using a very short time relaxation moduli as the normalisation factor can lead to severe errors in the predicted creep response.

One way of using relaxation data more reliably would be to select a relaxation modulus at a longer relaxation time (e.g. 5 s) as the normalisation factor. This can be rationalised on the basis that it is the long-term performance of the joint that is of interest not the short-term performance. This modification was performed on the set C relaxation test data. The 5 s modulus was set as the 'instantaneous' modulus and used for normalisation. All data before 5 s were deleted. Even after this modification, the long-term normalised compliance was tending towards 0.1. This implies that substantial creep deformations are still predicted (Figure 22). A further option is to use hyperelastic constants that give an initial stiffness comparable to the 'elastic' relaxation modulus in the stress relaxation data. To explore this concept the adhesive was modelled as perfectly elastic with a modulus of 2500 MPa and a Poisson's ratio of 0.35. The FE creep prediction used the full stress relaxation data. As shown in Figure 22, the use of this high initial stiffness leads to extremely low deflections. Even though the predicted creep strain in the adhesive is in excess of 30 times the elastic strain the deflections are only a small fraction of the experimental creep results.

4.2.4 Alternative Models for Time-Dependent Properties

Much of the data presented above suggest that the visco-elastic model available in ABAQUS may not be suitable for flexible adhesives such as the DP609 polyurethane. One of the problems with obtaining accurate analyses is that the magnitude of the creep or relaxation is calculated from normalised data. Thus, the accuracy of the initial ‘elastic’ datapoint is crucial. There is evidence to suggest that this point is sensitive to strain rate and immediate strain rate history. Errors in the prediction of this point will lead through to large errors in long-term performance. Even the **shrink** parameter that can be used to specify the long-term relaxation modulus as a constraint to the fitted visco-elastic constants is a normalised term and, thus, sensitive to the initial data point.

A model where the visco-elastic properties (whether relaxation modulus or creep compliance) can be specified in engineering units may give more robust predictions of long-term performance. Previous work by NPL⁽⁵⁾ on the time-dependent properties of plastics focussed on models for the time-dependent creep compliance based on a power law:

$$J(t) = J_0 \exp\left(\frac{t}{t_0}\right)^\gamma \quad (3)$$

where J_0 is the initial compliance, t_0 is a characteristic relaxation time and γ is the power law constant defining the width of the relaxation.

Models based on this function were developed to incorporate physical ageing and stress dependence.

$$J(t) = J_0 \exp\left(\int_0^t \frac{du}{t_0(u, \sigma)}\right)^\gamma \quad (4)$$

where J_0 and γ are as before but the relaxation time (t_0 constant now depends on the stress (σ) and physical age (t_e):

$$t_0(t, \sigma) = \left(A^2(\sigma) t_e^{2\mu} + C^2(\sigma) t^{2\mu} \right)^{\frac{1}{2}} \quad (5)$$

$$A(\sigma) = A_0 \exp(-\alpha \sigma^2), \quad C(\sigma) = A_0 \exp(-\beta \sigma) \quad (6)$$

The constants A_0 , α and β are found from fits to creep compliance data determined under different conditions. Procedures for doing this were outlined⁽⁵⁾.

This type of model, although not available in ABAQUS, may offer a more general solution to creep problems. The capability of handling stress dependence would be more suitable for analyses of complex joint geometries where stress levels may differ significantly within the adhesive bond.

4.3 COMPARISON BETWEEN PREDICTIONS AND EXPERIMENTS

The sensitivity of the FE predictions of the DP609 adhesive at 20 °C to the accuracy of the input data, and the consequent inability of the FE analyses to produce predicted displacements remotely corresponding to the experimental data, meant that there was little point in detailed analysis of the FE results. In contrast to DP609, the M70 adhesive appeared more amenable to the visco-elastic modelling. The bulk specimen data is more repeatable. The shape of creep compliance curves is less sensitive to stress. Additionally, the 'elastic' extension is more straightforward to determine from the bulk specimen creep curves. Therefore, there are fewer uncertainties in the normalised relaxation data used to calculate relaxation constants.

Figure 23 shows a comparison between FE predictions of creep and experimental measurements on a lap joint specimen at 20 °C (stress levels of 0.25 MPa, 0.5 MPa, 0.75 MPa and 1.0 MPa). FE predictions were made using creep data measured at a stress level of 1.03 MPa. All analyses were performed using a *VISCO step for load application and a second *VISCO step of 100,000 s for creep under a constant force. The visco-elastic constants were determined using a third order (N=3) Prony series.

The qualitative agreement between the measured and predicted creep response of the joints is much better than for the DP609 joints discussed in earlier sections. The main difference between the curves appears to be due to the 'elastic' extensions predicted using the time-independent hyperelastic model coefficients. Earlier work has indicated that the hyperelastic model can be relatively poor at describing the deformation of lap joints in constant strain rate tests. Also, the rate of initial loading in the creep test is considerably higher than in the constant rate bulk specimen from which the hyperelastic coefficients were derived. It is likely that the modulus of the adhesive is rate dependent. Thus the faster loading rate would correspond to a higher modulus. The joint would be 'stiffer' than expected from constant rate tests and, hence, the initial extension would be overestimated.

Some predictions of the variation of stress in the joint are shown in Figure 24. The time dependence of stress components in three elements (two corner elements and a central element) is shown. Contour plots of stress and strain distributions showed that most of the adhesive in the joint experiences the same stress and strain. This is represented by the central element (no. 83). The ends of the joint are under different stress states. The extremes of which occur near the corners of the bond (elements 32, free adherend, and 160, static adherend). The simulation failed to converge satisfactorily (with a creep increment **cetol**, of 2 %) at ca. 0.84 mm. This is common to the analyses run on both of the adhesives using any of the visco-elastic input data.

The shear stress (s12) in the centre of the specimen and in the corner of the bond attached to the static adherend are predicted to decrease as the joint creeps. In contrast, the shear stress in element 32 increases with time. The tensile stress (s11) is surprisingly high and increases significantly with time in all the elements. The tensile stress becomes the largest component of stress towards the end of the simulation. It is likely that the deformation of the bondline predicted in the simulation (and encountered in the experimental measurements) is too great for the assumption that the stress is predominantly simple shear to hold. Peel stresses in the elements 83 and 160 are either

negligible or compressive throughout the simulation. The peel stress in element 32 is significantly larger. It is comparable with the shear stress. This peel stress is predicted to increase slightly as the specimen creeps then decrease as extremely large extensions are reached.

Predictions of strain and strain energy in the joint are shown in Figure 25. Shear strain (e12) in elements 83 and 160 is predicted to increase at first but decline at higher times (and extensions). The shear strain in element 32 increases with time in the FEA output. The tensile strain (e11) is initially close to zero but is predicted to increase significantly with time in all three elements. The results for peel strain (e22) are unexpected. In all 3 elements the peel strain is predicted to become more compressive with time. Intuitively, an increase in tensile peel strain would be expected to occur in the joint (particularly at the corners).

The final plot in Figure 25 shows the total strain energy density in the three elements. In elements 83 and 160 the total strain energy is more or less constant throughout the duration of the creep prediction, only increasing slightly at long times. In element 32 the strain energy increases throughout the creep prediction.

Bearing in mind the limitations of the accuracy of the FE model (including the assumption that the adhesive is incompressible), it is possible to make some comments on the trends in the predictions of stress and strain values in the adhesive during creep. These show that the strain energy increases most in the corner of the bondline where the initial peel stresses are greatest. However, the FEA predicts that the peel stresses and the peel strains in this corner, and elsewhere in the joint, actually decline (even becoming compressive) at long durations. Intuitively, reduced peel suggests a stronger joint. However, the creep is accompanied by large increase in the tensile stress and strain along the axis of the bond. This is likely the cause of the increased strain energy and, thus, likely to lead to rupture of the adhesive.

4.4 FAILURE AND DESIGN CRITERIA

Strain energy has been postulated as a possible failure criterion for flexible adhesives. However, as with the constant extension rate results reported previously, the significant differences between the FE predictions of extension and the measured test extensions in the joint specimens make it difficult to draw any conclusion regarding the validity of this criterion. Accurate predictions of force-extension performance of the joint are required before it is sensible to make correlations between strain energies in the adhesive joint and those in the bulk test specimens.

Failures in both the bulk specimen and joint specimen creep tests appear to be related to failure conditions in constant rate tests. For both adhesives the mode of failure of the lap joints under creep is the same as in constant rate tests. If a function describing the degradation of failure strength with time under load can be found then simple design rules based upon the de-rating of the constant rate joint strength could be used to predict service lives. However, additional low stress, long duration creep tests would be needed to validate this approach.

The failure stress/strain values of the adhesives under creep appear to correlate with the values for stress/strain at failure in constant rate tests performed at elevated temperatures. This suggests that constant rate test results at elevated temperatures could be used to identify safe long-term loads.

5. CONCLUDING REMARKS

Some aspects of the performance of flexible adhesive bonds under constant loads have been investigated. The ability of FE methods to predict long-term creep performance is critically dependent on the input data. The 'elastic' properties of the adhesive must be known at strain rates approximating the loading rate to have any chance of correctly predicting the crucial initial extension of the joint. As the subsequent creep strains predicted are proportional to this extension any errors in this extension will be magnified at longer times. Research to improve the determination of constants for the hyperelastic models and their sensitivity to strain rate will be continued in the PAJ extension project, PAJex2 Flexible Adhesives.

The visco-elastic model in ABAQUS used for describing the time-dependent material properties may not be particularly suitable for flexible adhesives where large normalised compliances may be encountered. The model relies on normalised data. Therefore, the accuracy of the initial 'elastic' datapoint is critical to the accuracy of the entire prediction. This initial 'elastic' datapoint may be difficult to determine in the input data. Also, it may not be accurately predicted in the FEA. The visco-elastic model is also unable to incorporate stress dependence of the visco-elastic properties. A visco-elastic model, capable of incorporating stress dependence, in terms of unnormalised material properties is likely to be more reliable for predictions of long-term properties.

The data presented suggest that it may be possible to derive simple design rules for safe loads for long-term service. This might be achieved from a function relating the reduction in strength with time under load or from tests performed at elevated temperatures. However, further data is needed (particularly long time, low stress tests) to validate this approach. The test arrangement used in this work is difficult to adapt to the low forces required to achieve long-term creep loading in bulk specimens of such compliant materials.

Some recommendations for good practice in the measurement and modelling of creep are outlined below:

- Bulk test specimens of flexible adhesives will experience large extensions under creep. Therefore, it is vital that (i) any extensometry used has sufficient range; and (ii) where local strain measurement methods are not employed some method of relating total specimen extension to local strain is available.
- Creep tests should be performed under closely controlled temperatures.
- It is essential to determine creep properties under different proportions of the static failure stress in order to obtain (i) an indication of the relationship between time to failure and stress; and (ii) data on the stress dependence of the creep function.

- Repeat tests are needed to obtain statistically significant conclusions regarding the failure of the flexible adhesives since the test specimens may be of variable quality.
- Elevated temperature tests may accelerate the creep 'ageing' of the specimen and enable extrapolation of short term results to long term performance. However, caution should be used when doing this since this approach has yet to be validated.
- As the implementation of the FEA creep model requires that input creep data are normalised by an initial 'elastic' creep compliance, it is critical that this is determined accurately. It is recommended that (i) the unloaded position is accurately known and stable; (ii) the sampling rate over the initial 'loading' stage of the test is sufficiently high to minimise uncertainties in the location of the end of the stage; and (iii) the resolution of the extensometry used is appropriate for the displacements applied.
- When modelling the initial, elastic loading stage of the creep process the accuracy of the prediction of the initial elastic deformation of the adhesive is critical to the reliability of the predictions of creep response. Therefore, the analysis should be supplied with elastic properties at strain rates appropriate to the loading rates experienced by the joint.
- Potentially modelling of the creep of flexible, visco-elastic adhesive joints may lead to severe errors. It is important that some validation tests, even if only relatively short term, are performed to assess the accuracy of the analyses.
- For accurate FEA predictions of a joint or component performance, the input data should be determined using test specimens that are in the same cure state as the adhesive in the bonds being modelled. Where the final cure state of the adhesive is sensitive to the thermal history during cure, extra care needs to be taken in selecting a cure regime for the test specimens to ensure comparable properties.

6. ACKNOWLEDGEMENTS

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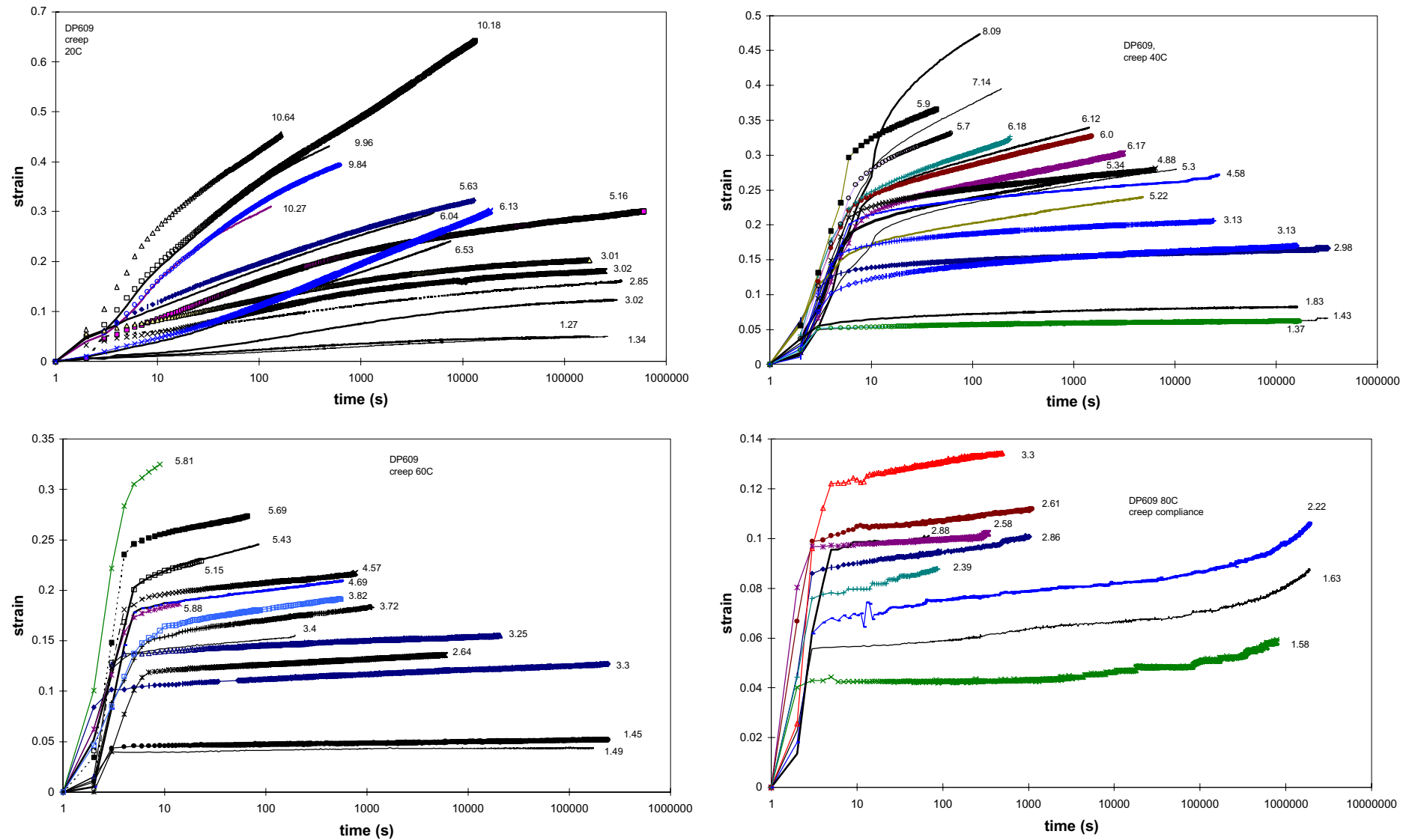


Figure 1: Creep Test Data for DP609 Showing Time-Dependent Strains at Different Applied Stress Levels and Temperatures

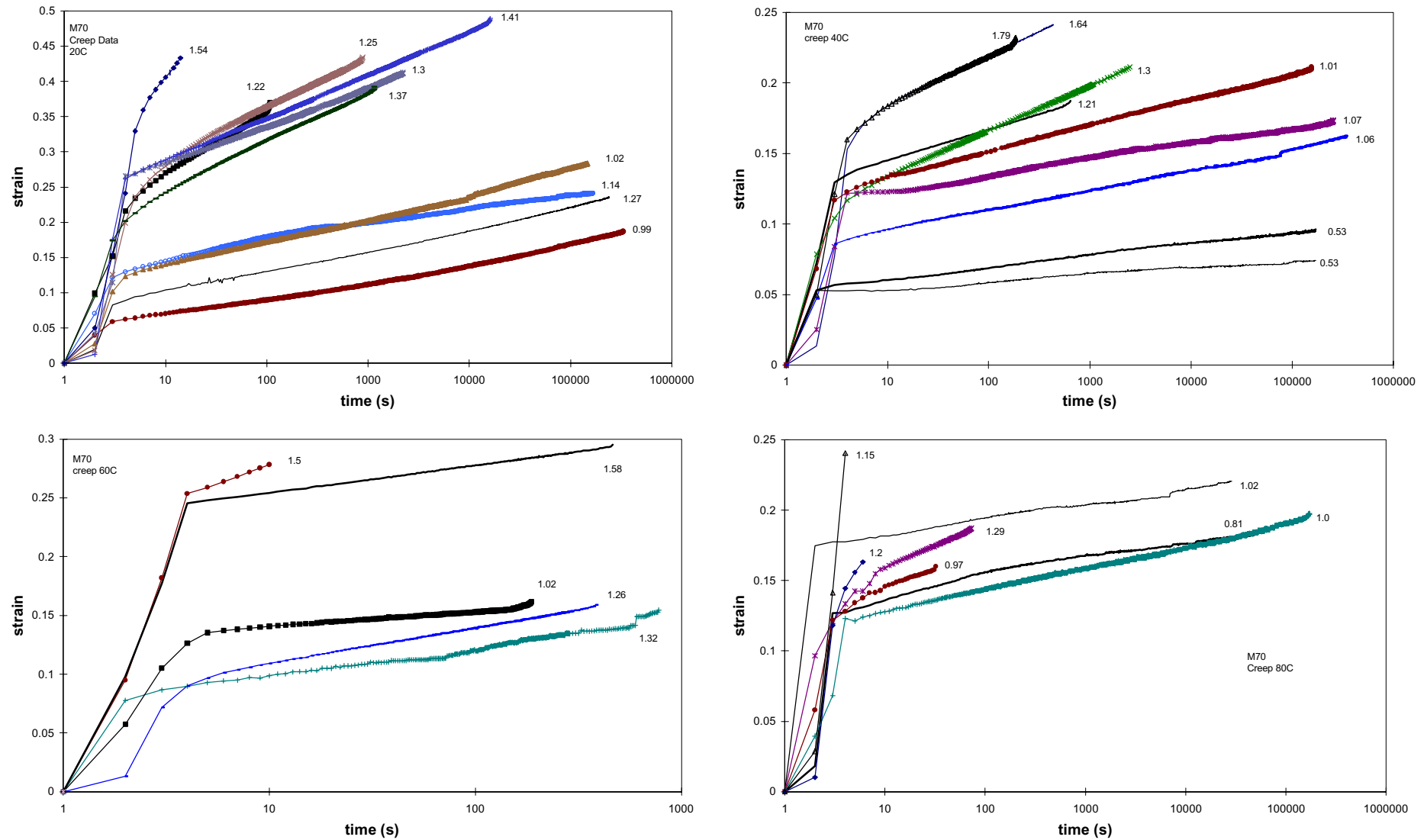


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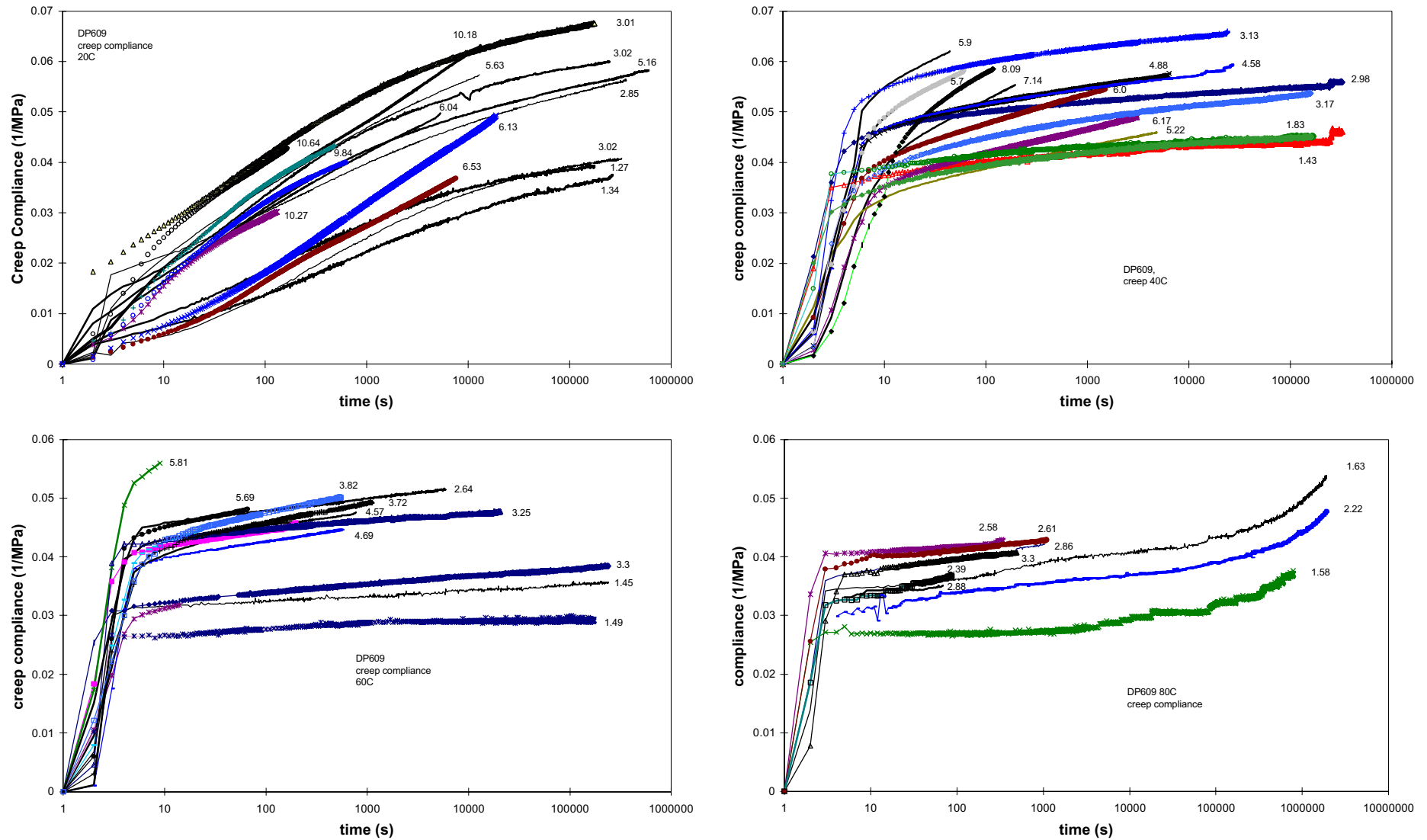


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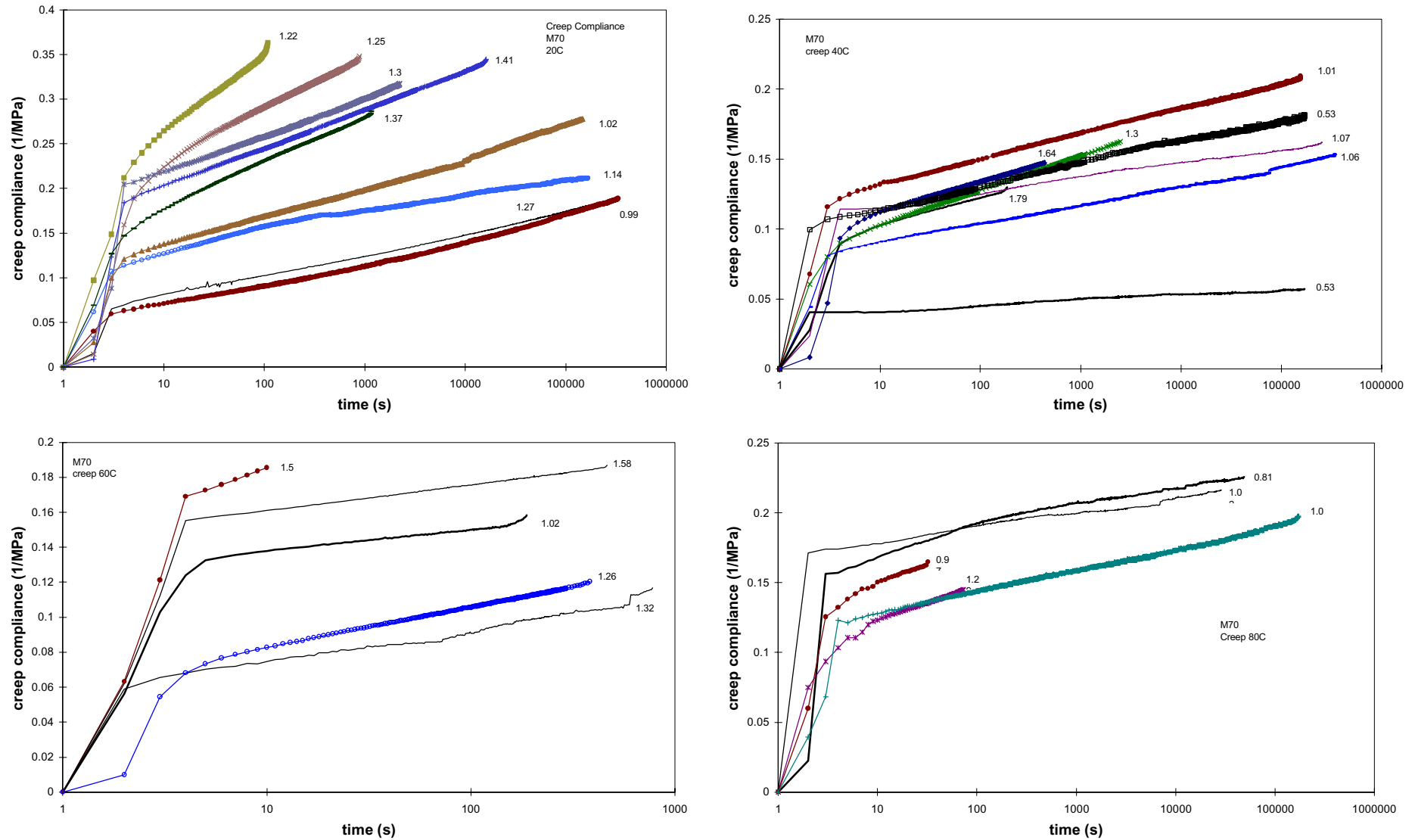


Figure 4: Creep Test Data for M70 Showing Compliances at Different Applied Stress Levels and Temperatures

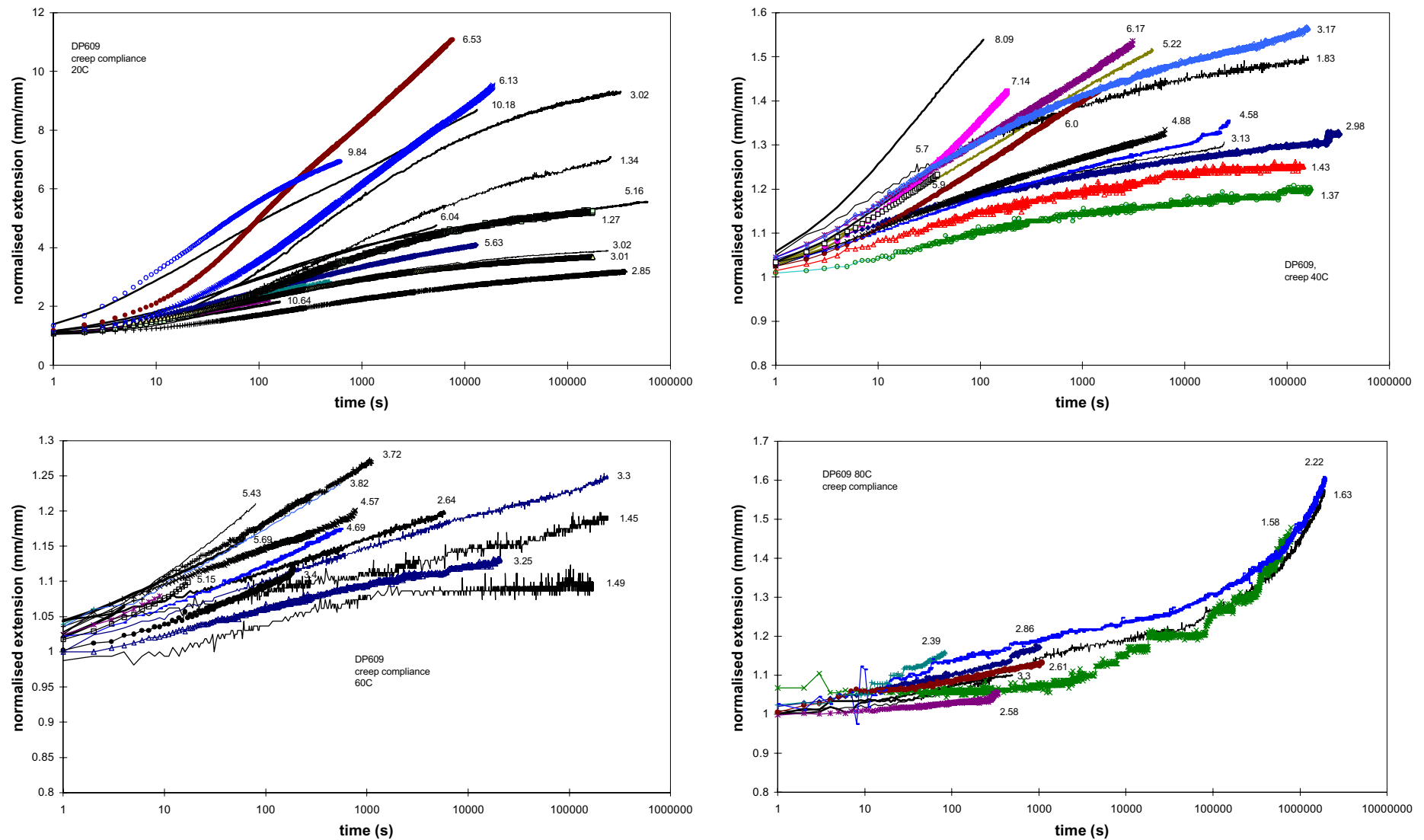


Figure 5: Creep Test Data for DP609 Showing Normalised Extension Values at Different Applied Stress Levels and Temperatures as may be used for input visco-elastic properties in FE analysis.

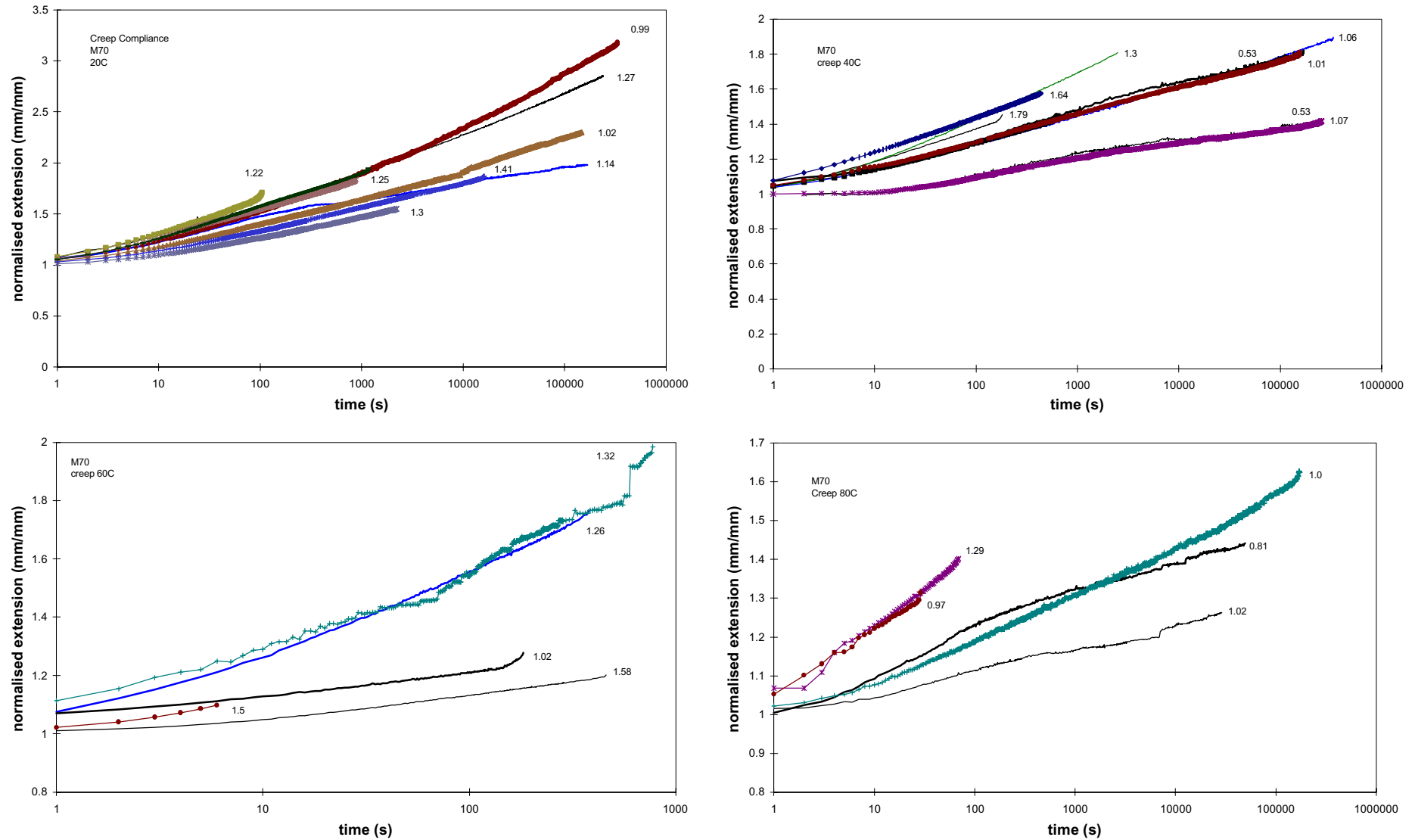
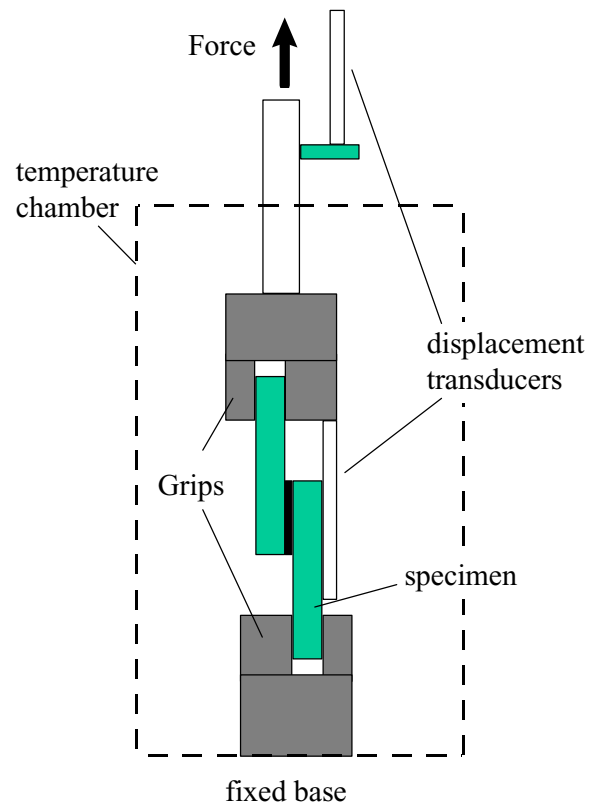
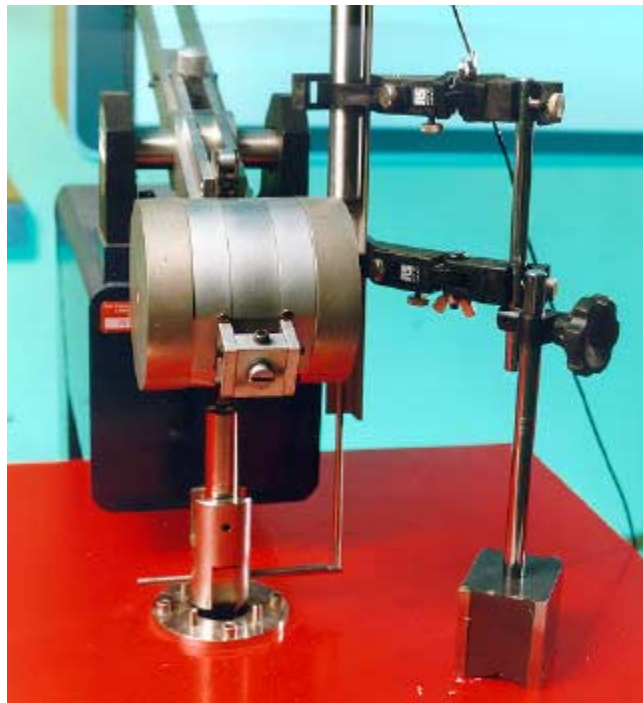


Figure 6: Creep Test Data for M70 Showing Normalised Extension Values at Different Applied Stress Levels and Temperatures as may be used for input visco-elastic properties in FE analysis.



(a) Schematic of test configuration with transducer attached to specimen



(b) Photo of LVDT transducer measuring total movement of the leverarm

Figure 7: Lap Joint Test Configuration

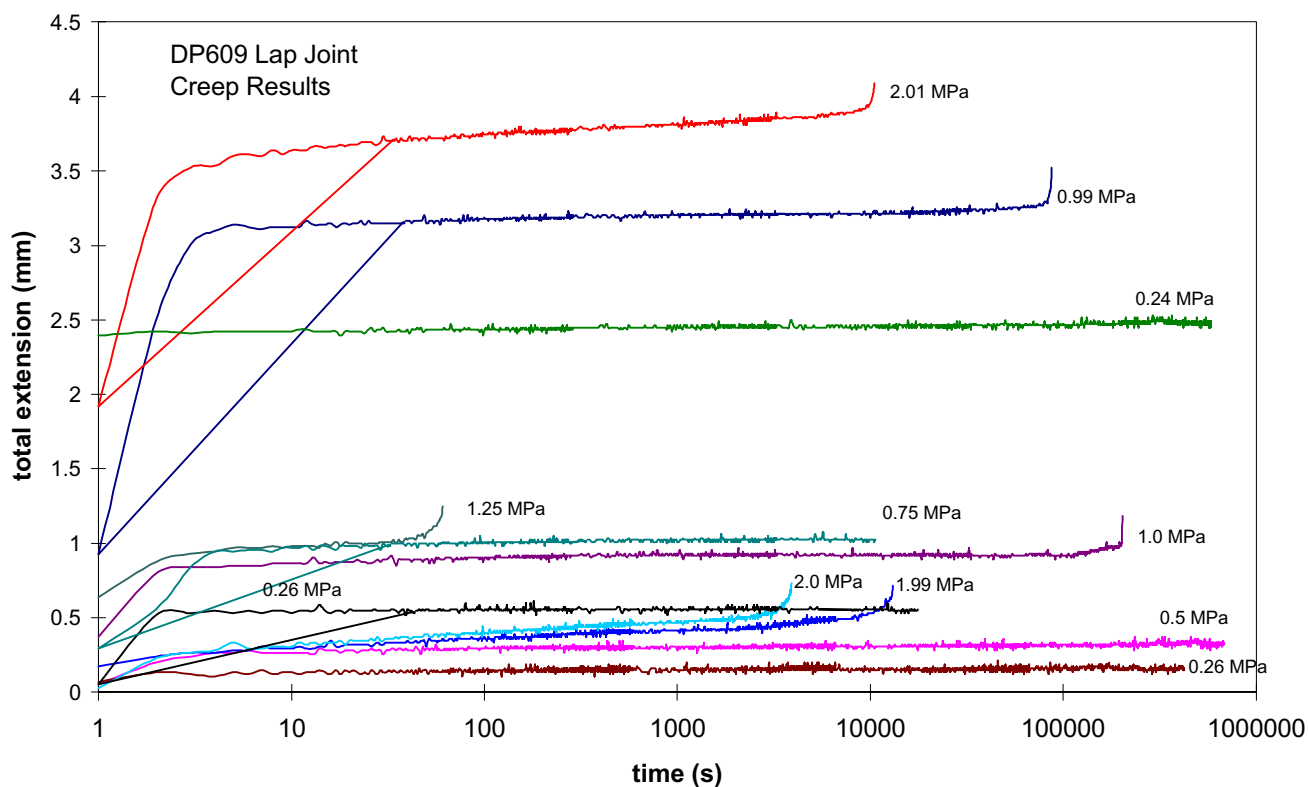


Figure 8: DP609 Lap Joint Creep Results

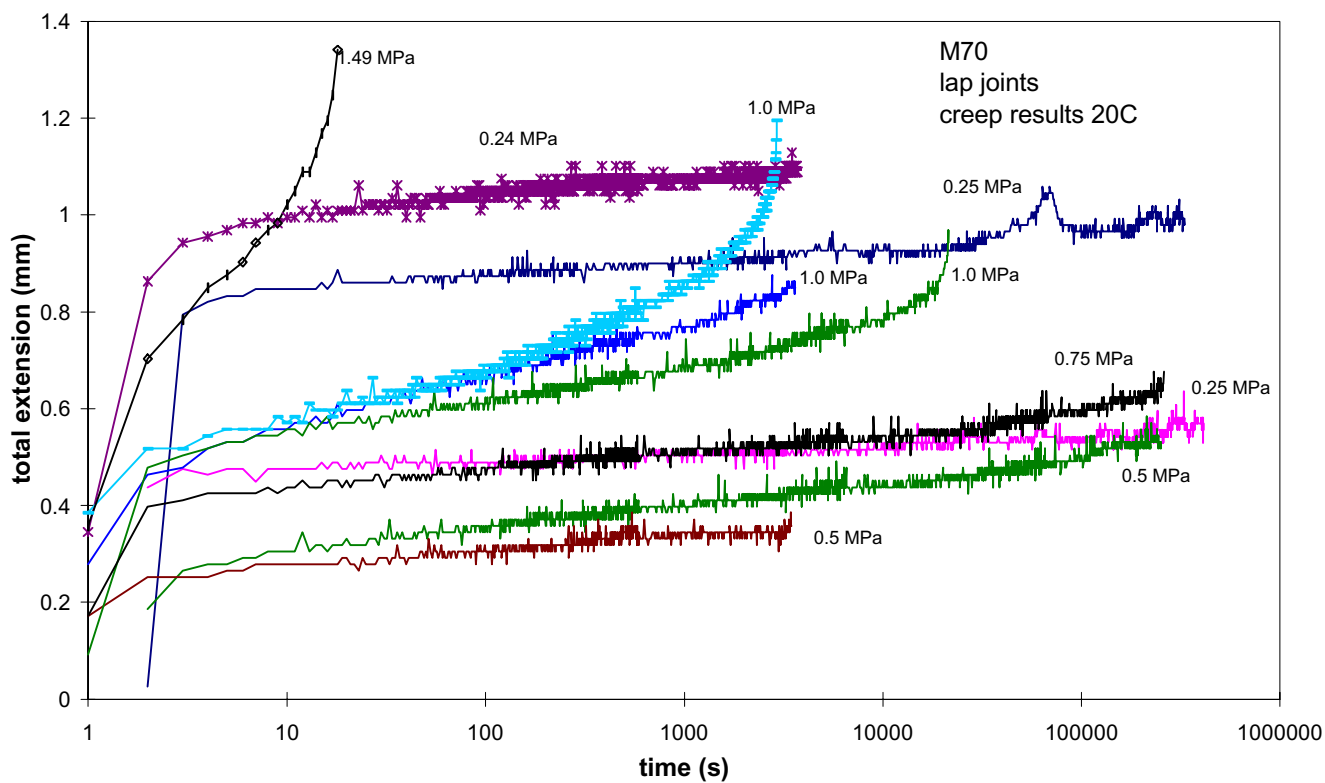


Figure 9: M70 Lap Joint Creep Results

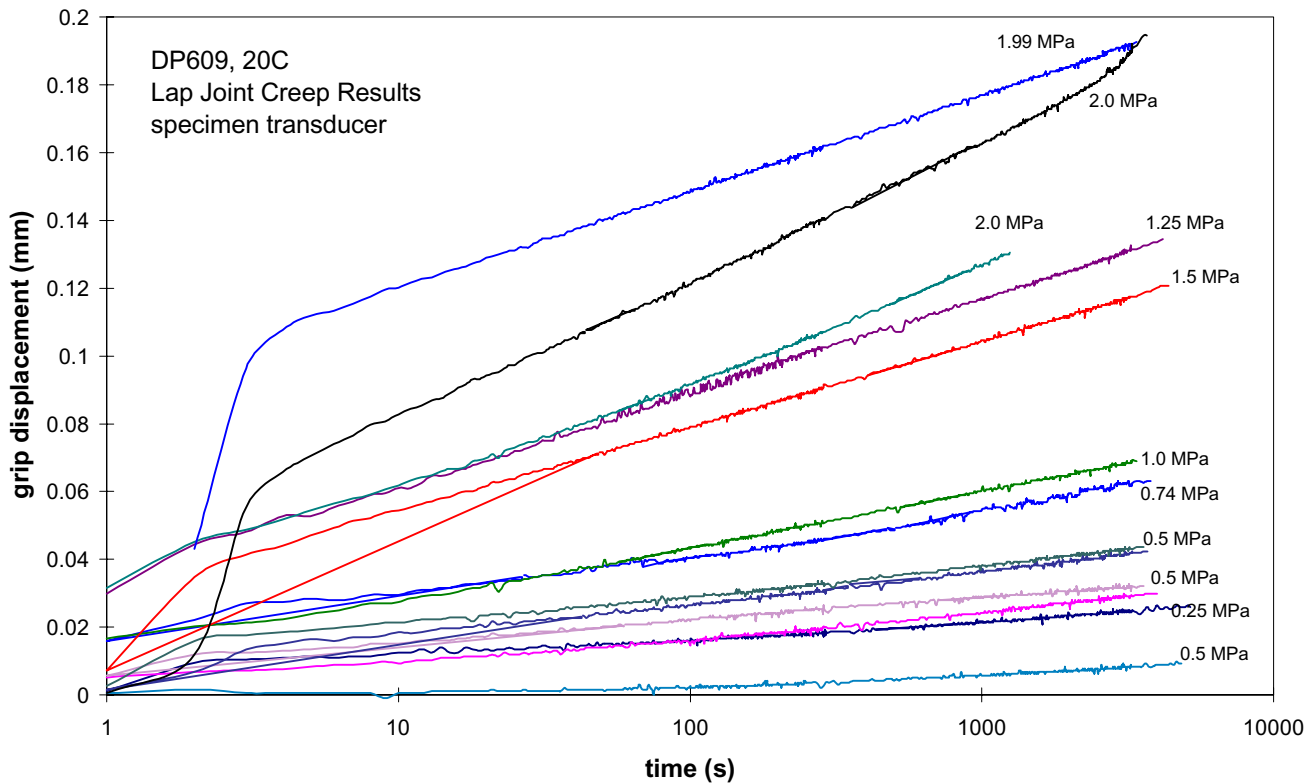


Figure 10: DP609 Lap Joint Creep Results Determined Using Transducer Attached to Fixed Adherend

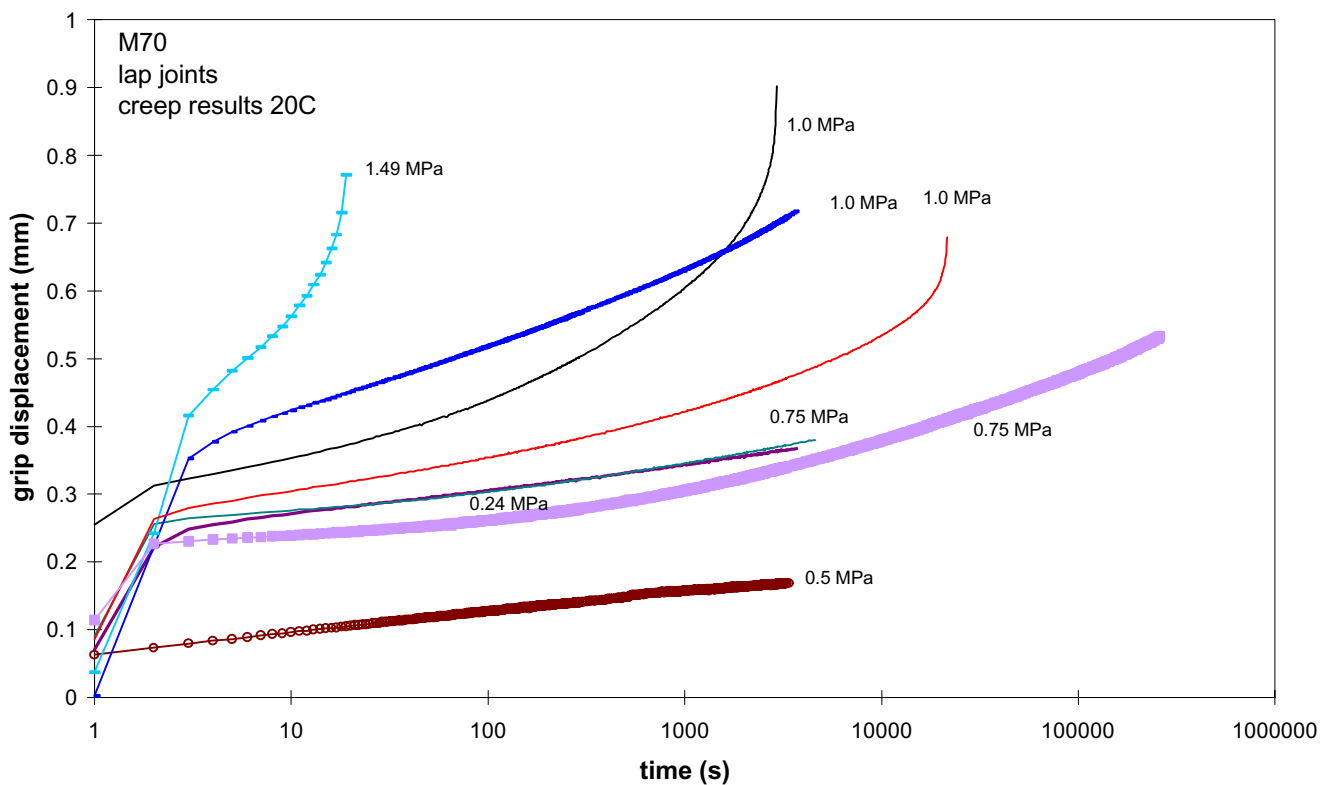


Figure 11: M70 Lap Joint Creep Results Determined Using Transducer Attached to Fixed Adherend

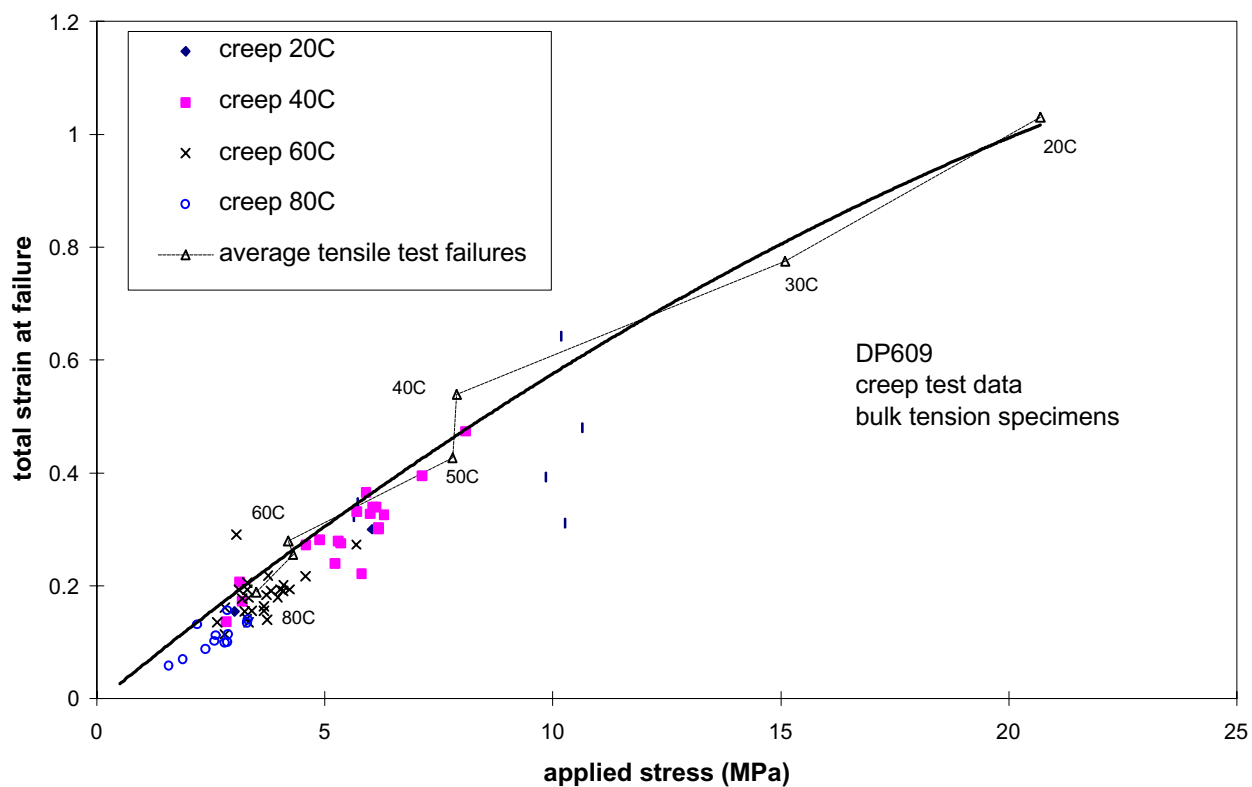


Figure 12: Failure Parameters for Bulk DP609 Specimens

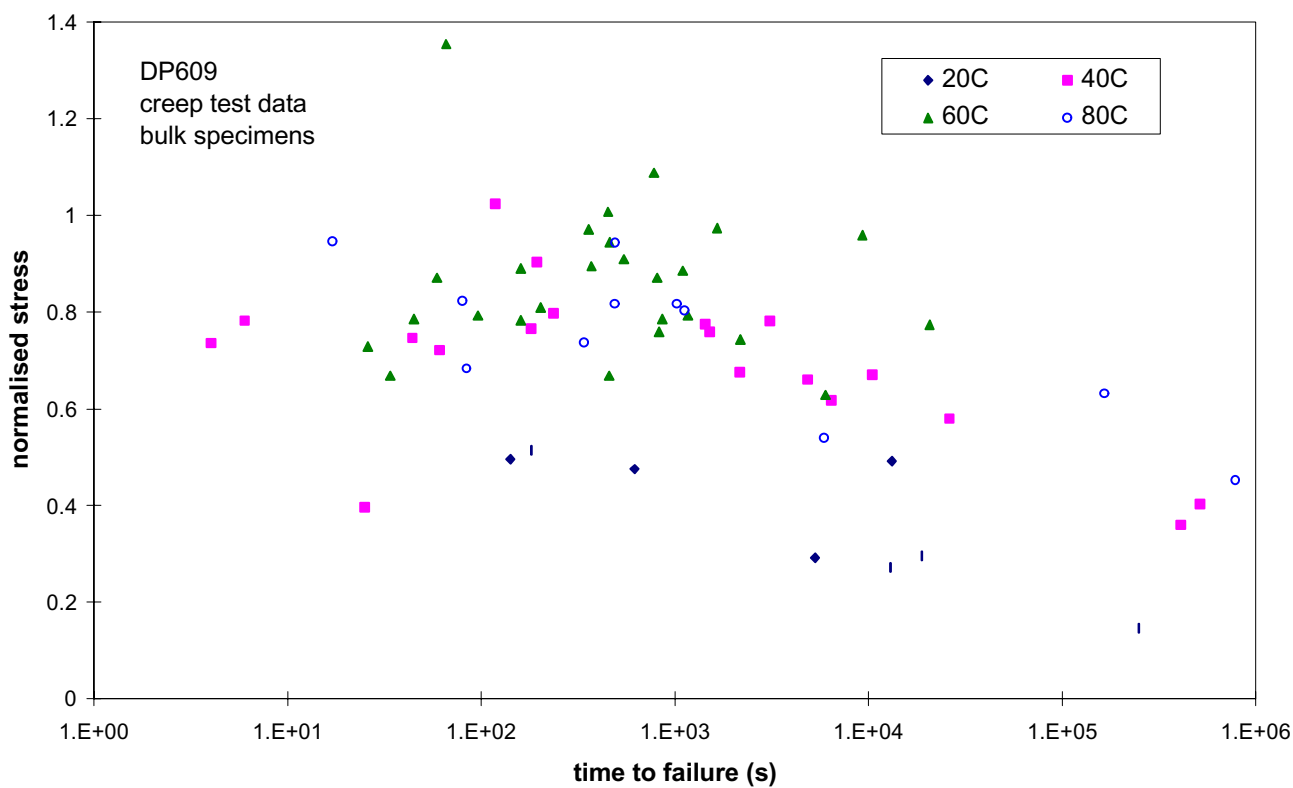


Figure 13: DP609 Time to Failure as a Function of Applied Stress at Different Temperatures

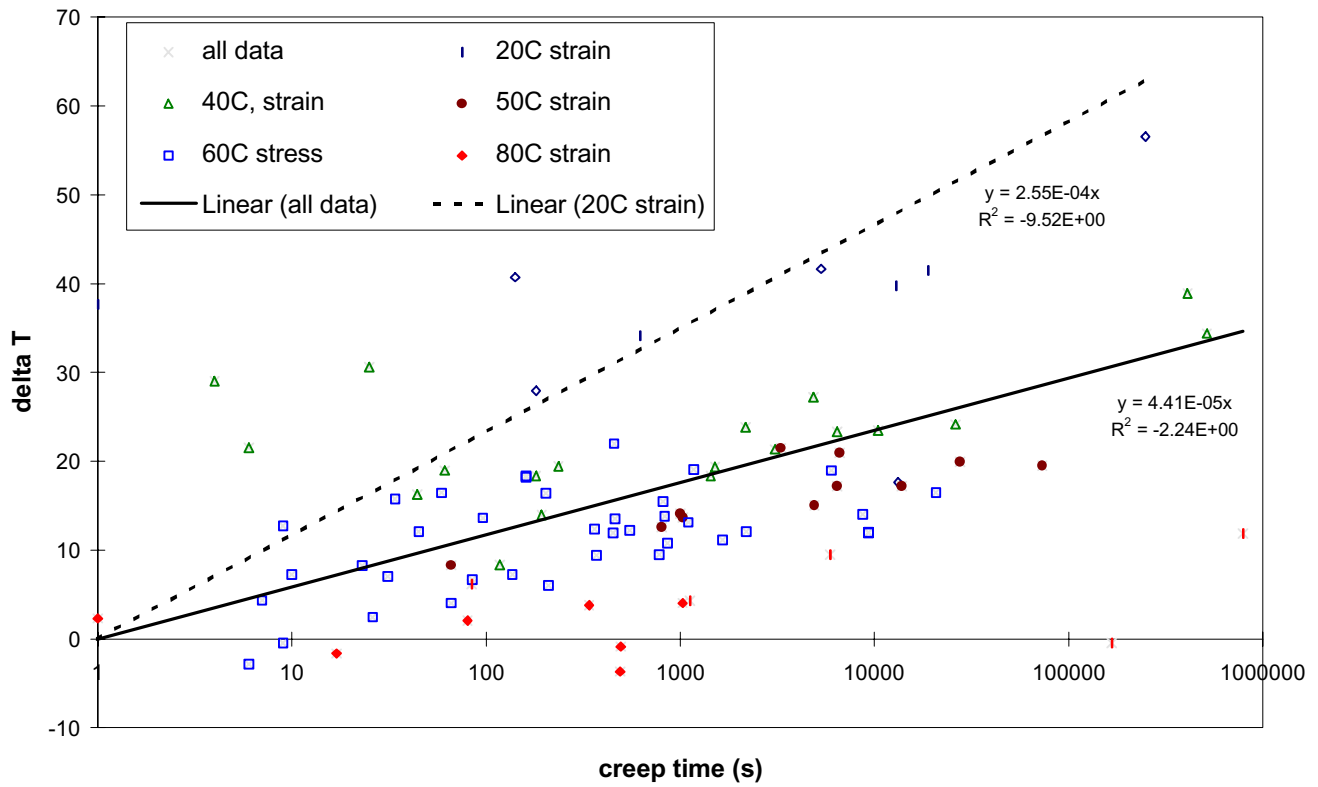


Figure 14: Shift in 'Effective' Temperature of DP609 Specimens Due to Time Under Load

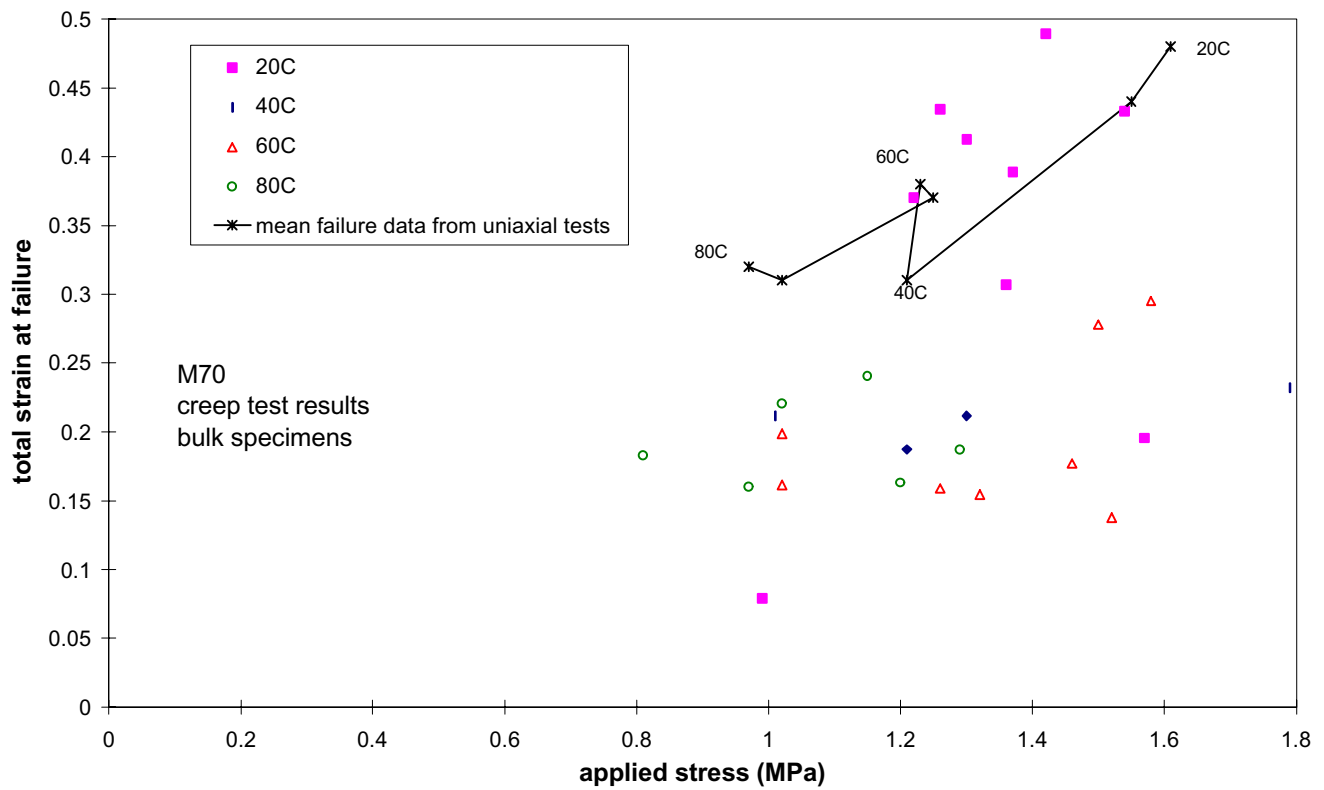


Figure 15: Failure Parameters for Bulk M70 Specimens

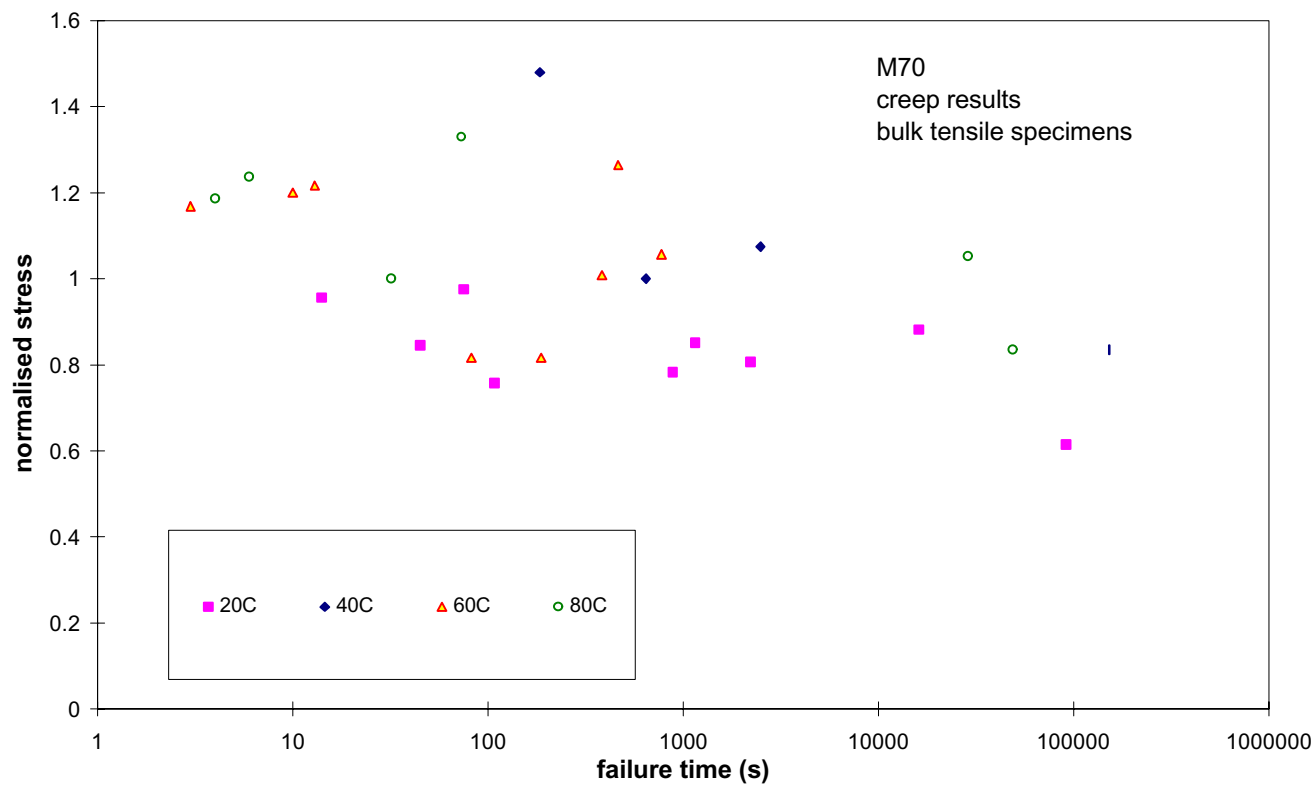


Figure 16: M70 Time to Failure as a Function of Applied Stress at Different Temperatures

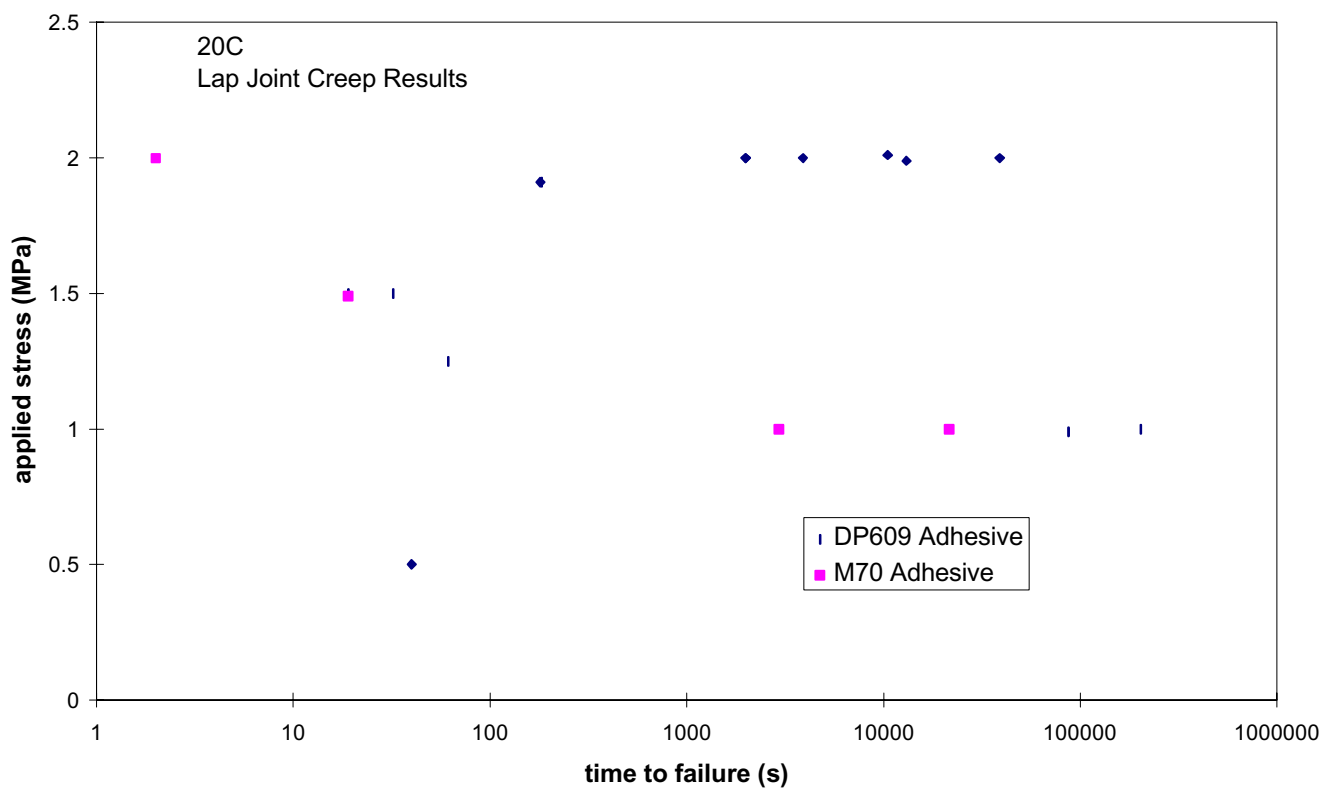


Figure 17: Time to Failure as a Function of Applied Stress for DP609 and M70 Lap Joints

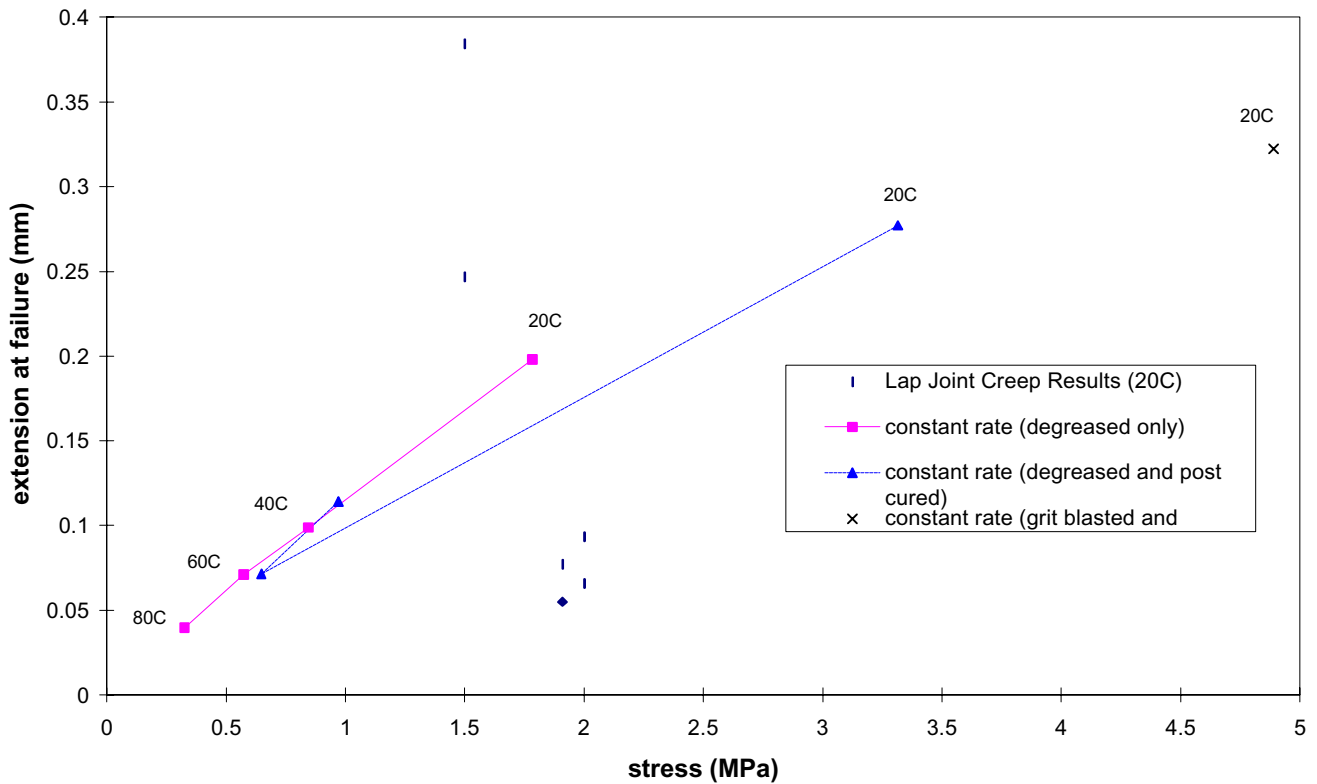


Figure 18: Failure Parameters for DP609 Lap Joints

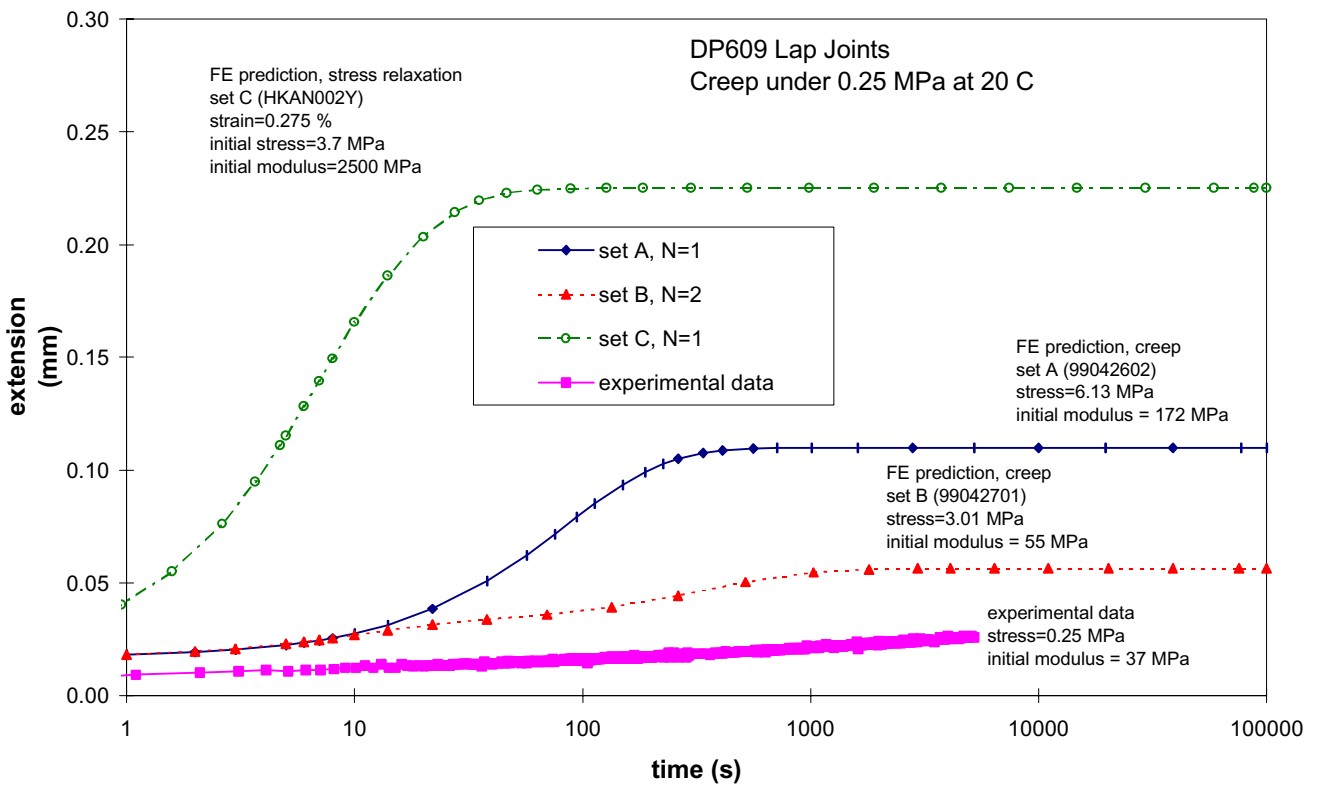


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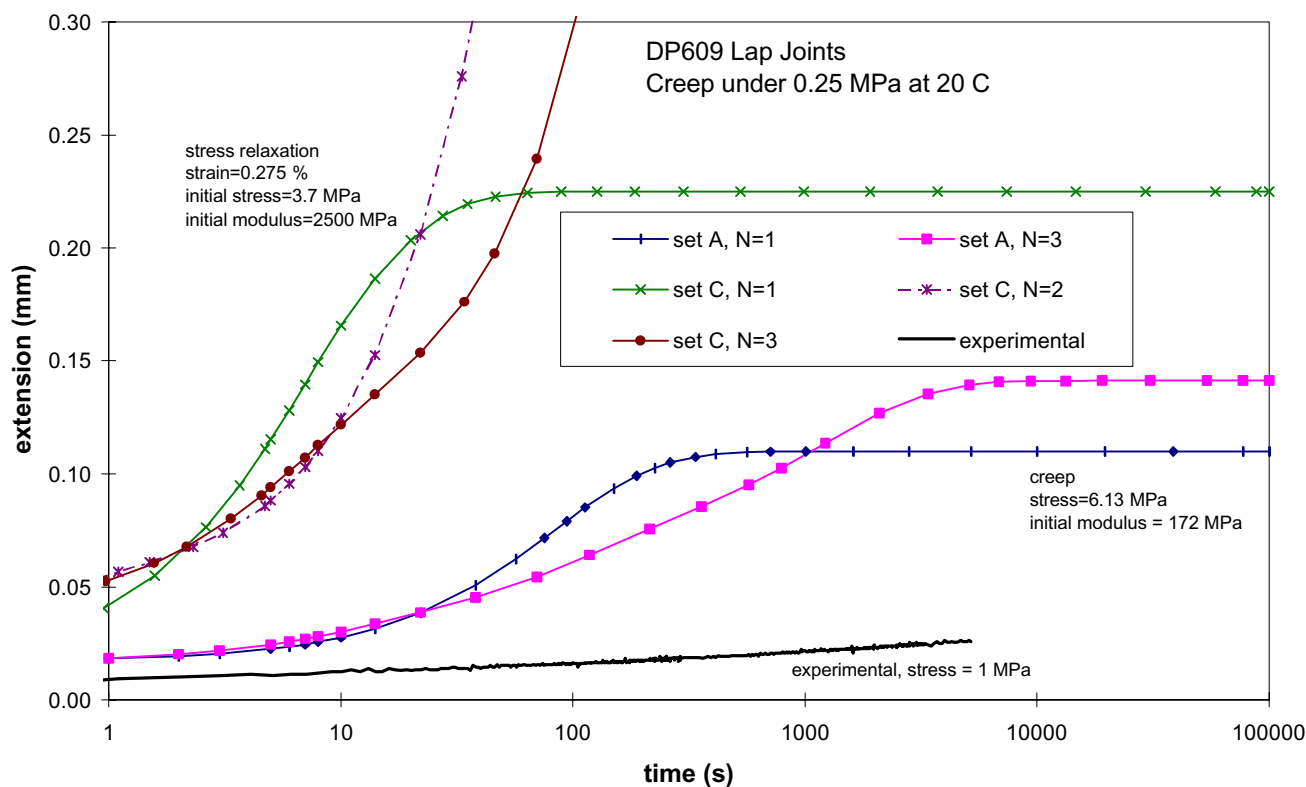


Figure 20: Effect of Different Order Prony Series on Creep Predictions

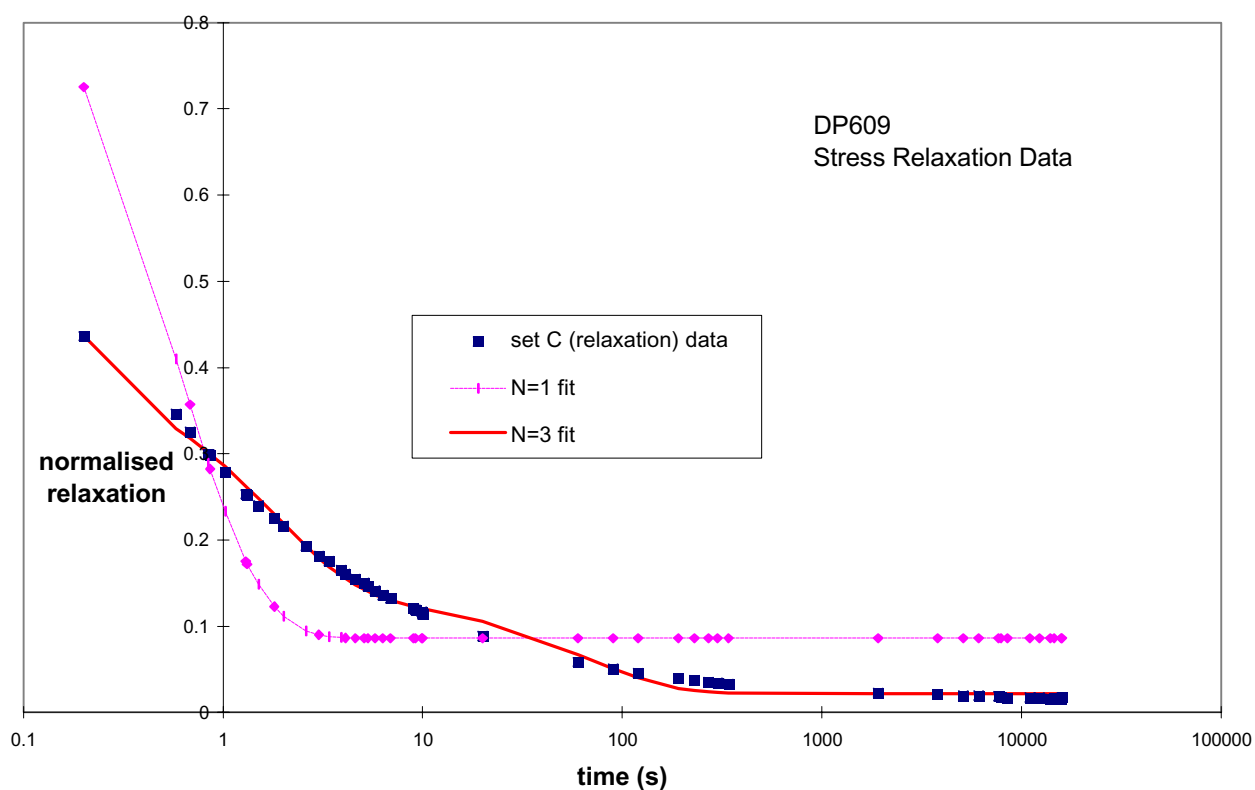


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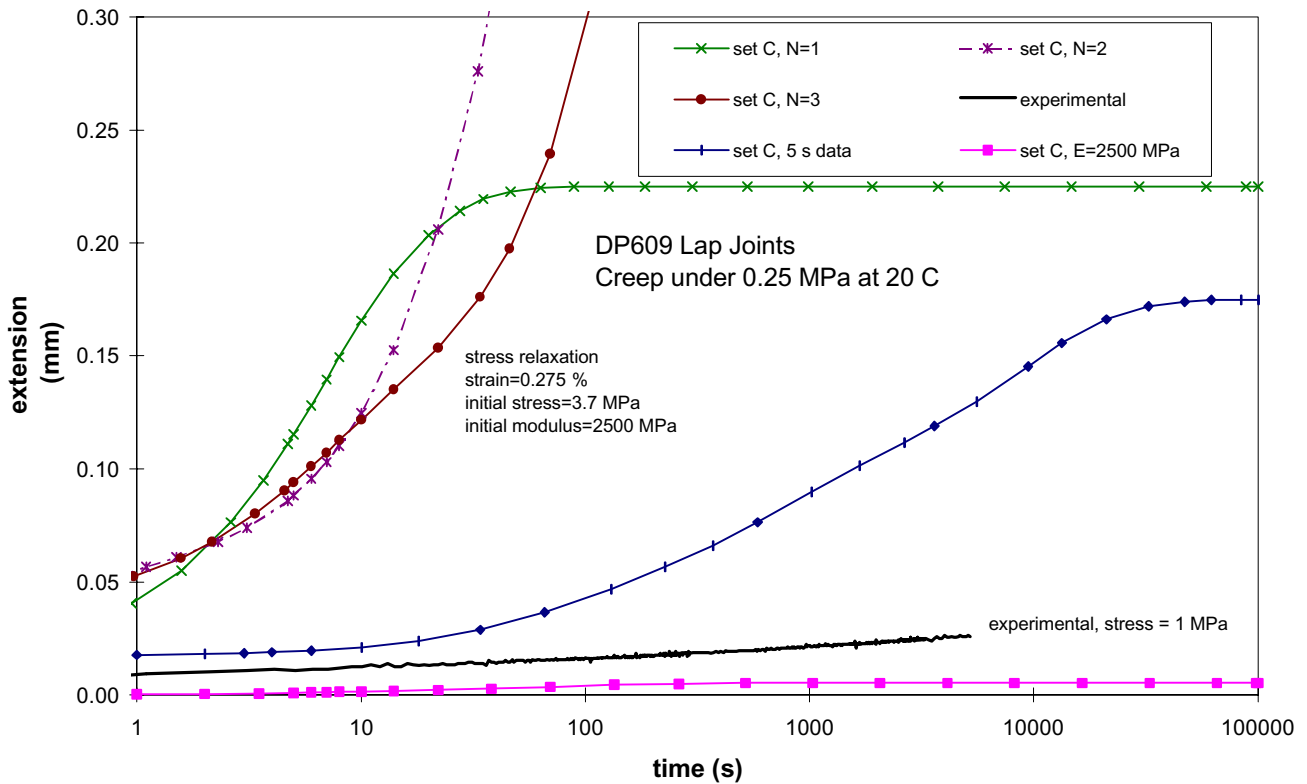


Figure 22: Effect of Different Presentation of Stress Relaxation Data

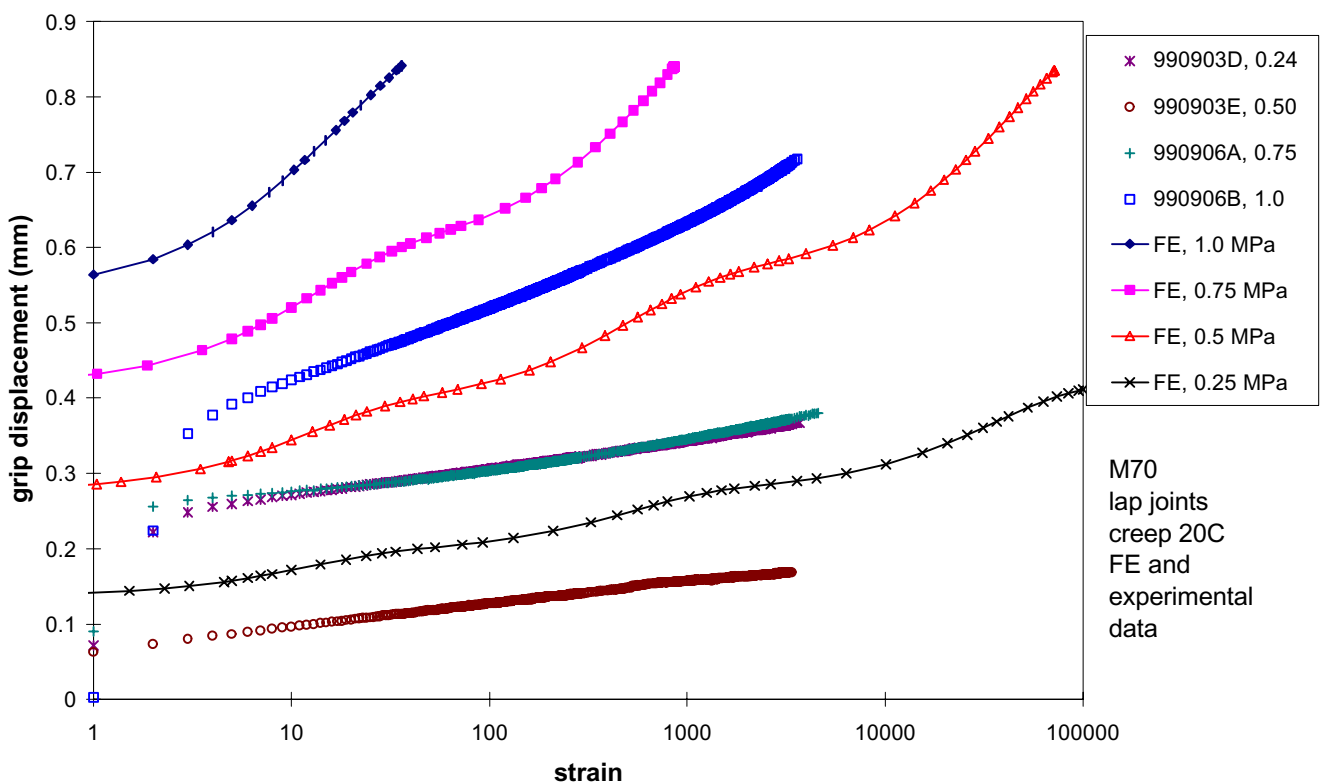
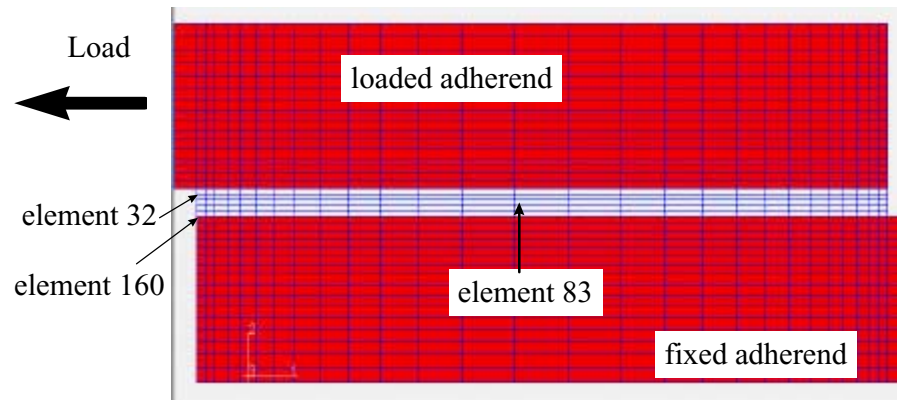


Figure 23: Comparison of FE Predictions and Experimental Data for M70 Joints



Elements in FE model of the bondline

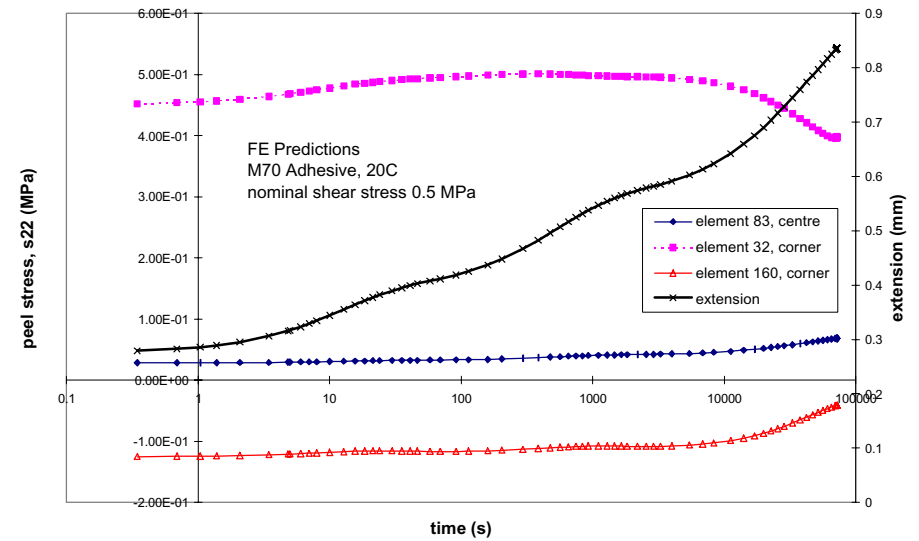
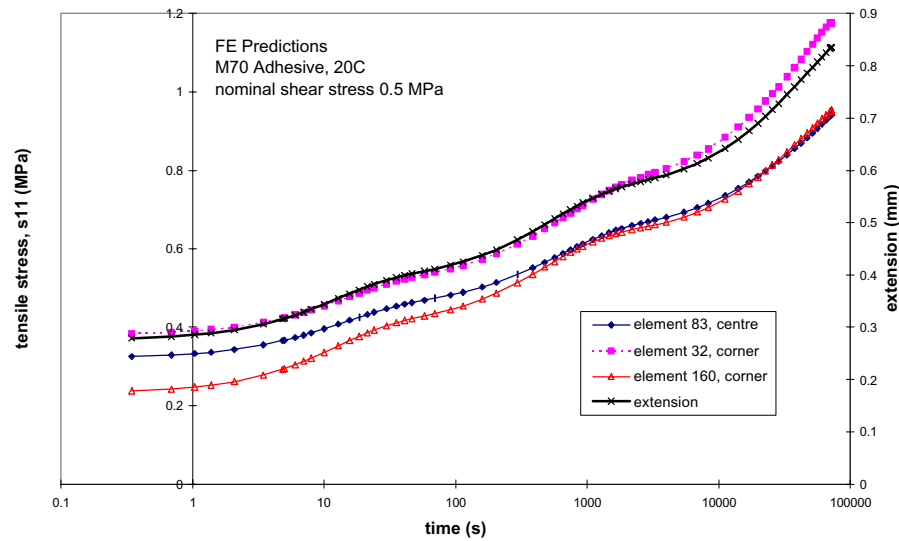
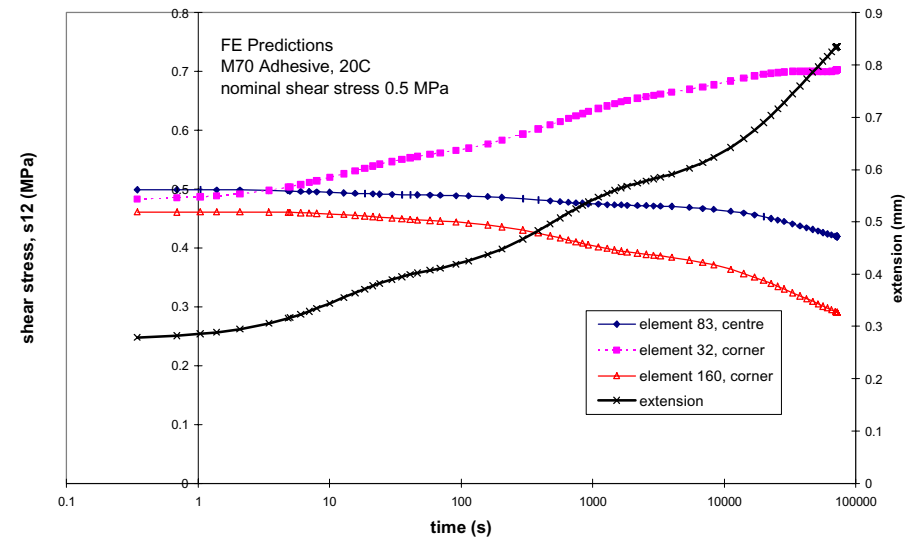


Figure 24: FEA predictions of stress in lap joint experiencing creep

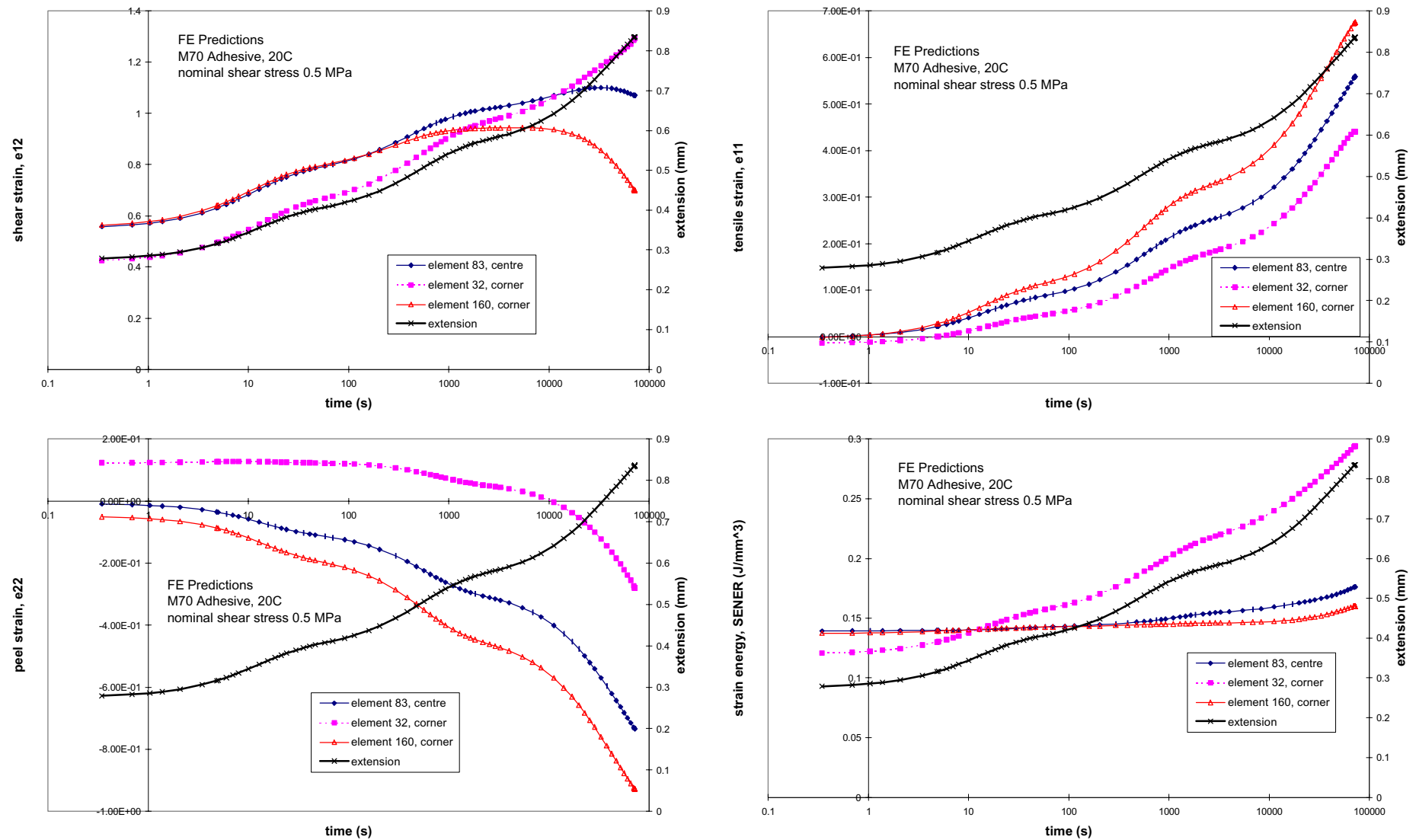


Figure 25: FEA predictions of strain and strain energy density in M70 lap joint under creep.