



**Measurement and modelling of bolted joint stress
relaxation in plastics**

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ABSTRACT

Loss of joint integrity caused by viscoelastic stress relaxation limits the service life of plastic components bolted to dissimilar materials. A technique, incorporating an integral load cell, to measure the stress relaxation properties of bolted polymer components over extended time periods at temperatures of up to 150 °C has been developed. The load retention properties of a phenolic thermostat housing for the motor industry have been tested at relevant operating conditions. Models have been developed to extrapolate the measured stress relaxation, thus providing an estimate of the service life. At elevated temperatures isothermal shrinkage of the polymer can have a significant impact on the load, contrary to what was believed previously. Phenolic thermostat housings undergoing the standard post-cure (seven hours at 170°C) should have service lives of 3 years at operating temperatures of up to 100°C.

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1 INTRODUCTION

Polymers are viscoelastic materials and so undergo stress relaxation. This is of particular importance in the design of bolted joints where the stress in the bolt is required to maintain a sufficient load in the jointed sections. Previously, determination of stress relaxation has been by tightening a bolted joint with a torque spanner, and then measuring the torque to untighten after a chosen conditioning. This measurement can show large scatter, partly arising from frictional effects; and since only one result can be obtained at the end of each test, assessment of time dependence is tedious and inaccurate. Direct stress relaxation tests of bolted joints are more accurate for assessing load decrease with time than either compressive creep or stress relaxation tests on cylinders, in which the effects of geometry, the elasticity of the bolt and other factors are not taken into account.

To enable the rate of relaxation in bolted joints to be investigated two measurement systems have been developed^[1]. The techniques continuously monitor the load on the material by means of an annular load cell which is incorporated into the system. The test rigs have been designed to compensate for the change in elasticity of the joint arising from the introduction of the load cell. In the simpler rig there is partial compensation and in a more elaborate but less practical test rig, the elasticity matches that of the original joint.

This report details the experiments carried out on injection moulded granular phenolic (Vynkier RX613) thermostat housings for use in the motor industry. These replace metal components with consequent savings on weight and assembly costs. In addition, a viscoelastic model to describe the behaviour of the component in a bolted joint is proposed. By fitting a model to the relaxation behaviour it is aimed to reduce the amount of testing required for characterisation. The time taken for the housing to untighten is critical to its performance. This time should certainly be greater than the time between services, and ideally greater than the vehicle's lifetime.

2 DEVELOPMENT OF EXPERIMENTAL TECHNIQUES.

Plastics are viscoelastic materials but they are not always linear viscoelastic, that is, the rate of change of stress can be dependent on the initial stress. For this reason it is important to have similar elasticity in a measuring system to that in the original bolted joint. For the actual joint investigated, Figure 1(a), the sample was 15 mm thick and the elasticity of the system was defined by the behaviour of a 6 mm bolt with an integral washer. This was found to contract 40 μm for a load drop of 10^4N . It was not sufficient just to include a load cell between the sample and either the base or the bolt head, because this increased the length of the bolt and introduced additional elasticity into the system, Figure 1(b), from the bolt and the cell. The Graham and White Instruments model AD-W-50 cell used expands 16 μm for 10^4N load drop and the lengthened bolt contracts 72 μm for 10^4N load drop. So a sample contraction of 88 μm would in this case arise from a 10^4N load drop. In order to measure stress relaxation the extra elasticity must effectively be removed from the measurement. This has been achieved in the two ways described below.

The first measurement rig, Figure 1(c) was designed to regain as much stiffness as possible. A boss was introduced which protruded through the centre of the load cell. The sample was placed onto a washer which distributed the load onto the cell and the assembly was bolted down into the boss. This was termed a type I rig. An additional length of bolt still arose from the thickness of the washer and the small gap left between the top of the boss and the washer. The additional elasticity of the load cell was also present. To partially compensate for these factors the thread of the bolt was not cut right up to the head, but a shank was left. In this way the extension of the bolt under a given tensile load was reduced, thus reducing the overall elasticity of the system. The sample then contracted 61 μm for a 10^4N load drop in a type I rig, but this could not be further reduced to the desired 40 μm .

The second method of measurement excluded the use of the bolt to load the sample and instead compressed the sample, via a stiff spring, through the head of a bolt. This was an unmodified bolt which passed through the sample into a clearance hole in the base plate, Figure 1(d), so that it did not contribute to the elasticity and thread effects of the system. For simplicity the spring consisted of a similar bolt to replicate the elasticity of the original system, screwed into a platen. The length of the screw in the spring could be changed, and by reducing it to less than the thickness of the sample the effect of the elasticity of the load cell was compensated for. The cell (now above the sample) still expanded $16\text{ }\mu\text{m}$ for a 10^4N load drop, and the spring was adjusted so that the whole mechanical system expanded by $40\text{ }\mu\text{m}$ for a 10^4N load drop (the base and loading screw being connected by a rigid frame). Hence a sample contraction of $40\text{ }\mu\text{m}$ was produced by a 10^4N load drop, as required. Advantages of this type II rig include the ability to readily modify the rig to allow for changes in sample thickness without the need for specially manufactured bolts; and the use of standard bolts resting through the sample ensured the correct loading area. This method can also be used for samples of different geometries which do not necessarily have to include a bolt hole. The load is then applied through a platen and not through a bolt head.

For clarity, the above descriptions are given in terms of making load changes; in reality the sample contraction under load produced a decrease in stress in the system. For the type II rig it was possible to exactly correct the elasticity of the system for a sample thickness of about 6mm or more.

3 EXPERIMENTAL RESULTS

3.1 TESTS AT ROOM TEMPERATURE

To aid comparison of measurements with different starting loads it was decided to plot the results as a percentage of the initial load.

Stress relaxation tests were carried out on samples cut from moulded thermostat housings which had 3 bolt holes moulded into their corners, designated A, B and C. B and C have similar geometries, flange A is larger. A number of as-moulded samples were tested to establish the in-batch / inter-batch variability of the mouldings, Figure 2. Each sample was loaded through a torqued bolt to an approximate stress of 75 MPa in a type I rig at 23°C which corresponds approximately to a 7000N load. The decay in load with time was recorded. Similar samples with a standard post-cure treatment of 7h at 170°C were then tested, Figure 3. The material was then tested at three stress levels of 75, 66, and 53 MPa to investigate the effect of initial stress on the viscoelastic behaviour of RX613, Figure 3. To investigate the effect of cure further, a series of different post-cure times was used. Each of the samples, prior to testing, was placed into an oven and the temperature was increased over the period of an hour to 170°C , and was then held at 170°C for a further 2 to 22 hours. Stress relaxation rates were then measured on mouldings with 0, 2, 4, 7, and 22 hours post-cure, Figure 4.

A comparison between the behaviour of the phenolic in the two types of rig was made to check the validity of using the simpler type I rig. Two post-cured mouldings from the same batch were tested in type I and type II rigs, Figure 5. They were both loaded to an initial stress of 75 MPa. No difference between the results from the two rigs was found, hence either rig can be used with confidence.

3.2 TESTS AT ELEVATED TEMPERATURES

Components have to perform above ambient temperature and thus tests have been carried out at 75°C , 100°C and 150°C , Figure 6. It should be noted that the operating temperature

of this particular component is just above 100°C. Tests at 75°C and 150°C are for the purpose of materials characterisation.

Samples were torqued down to a stress of 75 MPa in type I rigs at room temperature and then placed in an oven at an elevated temperature for testing. Samples of the post-cured (Figures 7 and 8) and untreated (Figure 9) phenolic were tested at 100 °C. Figure 7 shows the relaxation of samples cut from different components. Figure 8 shows the relaxation of 3 samples cut from the same component. As-moulded components were torqued to different loads (2.1, 4.6, 7.2 and 9.8 kN), Figure 9.

Following experimental work using DMTA (dynamic mechanical thermal analysis) equipment, Gibbs^[2] found that there was an increase in thermo-mechanical stability of these phenolic mouldings when an additional heat treatment of 1 h at 250°C was given after the standard post-cure cycle. A corner of the same moulding measured by Gibbs, with the further heat treatment, was tested in a type I rig at 100°C and the relaxation was compared to that of mouldings with a standard post-cure at 100°C, Figure 7.

Tests at 150°C were carried out with the post-cured material only. The results are presented in the section on modelling, and in Figure 10.

Stress relaxation curves from tests above ambient temperature exhibit an initial increase in the load due to the greater thermal expansion of the plastic than the steel bolt. To measure the thermal expansion at negligible load the phenolic mouldings have been heated in a newly developed NPL dilatometer and their expansion recorded. The linear expansion coefficient was found to be $25\text{--}33 \times 10^{-6} \text{K}^{-1}$, which was much greater than that of steel at $12 \times 10^{-6} \text{K}^{-1}$. Isothermal shrinkage measurements were also made using this equipment. The samples were heated to a chosen temperature which was then maintained, and isothermal shrinkage measurements were made with time, Figures 11 and 12.

Matched tests with a low initial load (1000 - 2000 N) were made to correct for load independent effects, such as thermal expansion or the sample bedding down. The data from these tests should be dominated by the load independent processes. The viscoelastic relaxation component should be given by subtracting the low load results from the high load data, Figure 13.

4 FINITE ELEMENT ANALYSIS

Finite element analysis has been used to investigate the effects of geometry on the stresses in a sample. The analysis package used was PAFEC and the mesh was made up of eight noded axis-symmetric elements. Typically there were eight meshes in the radial direction and ten in the 'z' direction. For each problem the displacement was specified and the resultant stress distribution calculated. The results of the analysis have been summarised in Figures 14, 15, and 16.

In Figure 14 the relationships between the sample diameter and the load required to produce a given deflection of 0.01mm for two different sample thicknesses are shown. At the interfaces it was assumed that there was no constraint so that slip could occur. There is little effect of increased sample diameter when it is greater than twice the washer diameter through which the load is applied. This is true for both the thick and thin sample, although the load required to attain the same deflection is much greater for the thin sample (eg 4600 N for a 3 mm thick sample and 1700 N for a 14 mm thick sample, for a diameter of 25 mm).

The effect of the diameter of the bolt-hole on the load required for a given indentation is shown in Figure 15, for a sample of thickness 14mm and diameter 25mm. As the hole size is increased the load required is decreased.

Where the surfaces of the sample are in contact with the washer or the base, the sample would either be able to slip or it would be fixed depending on the surface condition and coefficients of friction of the materials. The significance of friction at these surfaces is shown in Figure 16. The results give the upper and lower bounds to the problem as they may not be totally free or fixed surfaces. There is only a very small difference between the upper and lower bounds.

A cylindrical phenolic sample with no bolt hole was tested in a type II rig and the relaxation behaviour was compared to that of the moulded component with a bolt hole, Figure 17. This was carried out in order to assess the effect of sample geometry on relaxation behaviour. The loading conditions for these geometries are shown in Figure 18.

5 ANALYTICAL MODELLING

A viscoelastic model based on the Kelvin model is proposed to describe the stress relaxation behaviour of the moulded phenolic component:

$$\text{Load}(t) = A + B \exp -(\lambda t)^n$$

where load (t) is the load at any time t

A is the final load that the material relaxes to as $t \rightarrow \infty$

A+B is the initial load at $t=0$

λ and n are constants that characterise the material

$1/\lambda$ is the time for the exponential to decline to $1/e$ of its initial value and is called the relaxation time. The exponent n indicates that there are a number of parallel spring and dashpot elements having a distribution of relaxations as opposed to a single element. n determines the shape of the relaxation curve - the higher the value of n the steeper the slope at the relaxation time.

In order to fit the parameters λ and n it is necessary to plot

$$\log (\text{time}) \text{ vs } \log [- \ln \{ (\text{Load}(t) - A)/B \}]$$

Where the value of A is unknown it is taken that the final load is zero.

When this has been plotted a line is fitted using a least squares regression analysis, and from this the slope, n, is measured. The intercept is equal to $n \log \lambda$, and having obtained a value for n, λ is calculated; an example is shown in Figure 19. For two reasons, it was found to be necessary to exclude data points at the start of the curve. Firstly, the model is asymptotic and so as time tends to zero the function calculated, $\log [-\ln \{(\text{Load}(t)-A)/B\}]$, tends to minus infinity. Secondly, there may be the effects of bedding down of the integral washer on the bolt into the surface of the sample, particularly where there is any surface roughness or irregularities; and also bedding down of bolt threads. This would result in a slightly faster load drop at the start of the test. Using the values of n and λ a plot of $\text{Load}(t) = A + B \exp -(\lambda t)^n$ is created and compared to the original data. This has been achieved successfully for phenolic tested at 23°C, Figures 20 and 21.

For relaxation behaviour of the standard post-cured material at 100°C it has been possible to use the same model on results which have been corrected to approximate the stress-dependent relaxation only. To do this, relaxation curves were obtained at the same time on two samples from the same component with one at high initial load and the other at low load. The low load curve represented the expansion effects and non-stress dependent shrinkage, (plus a small but unquantified element of stress relaxation). By taking the difference of the two curves the residual is approximately that of the stress relaxation at 100°C, Figure 13. The model was then fitted to the residual, Figure 22.

For tests carried out on the as-moulded material at 100°C, Figure 23, and the post-cured material at 150°C Figure 10, the model has been unsuccessful, even with the differential method. The occurrence during the test of two mechanisms for decreasing the load, stress relaxation and time dependent shrinkage, have meant that the model to describe the stress relaxation behaviour is inadequate.

The features of the relaxation curves that do not fit the single exponential model suggest a load decrease function having two characteristic timescales and, hence, being better fitted by a model containing two exponentials to represent two Kelvin units with the form:

$$\text{Load}(t) = R_{\infty} + P \exp(-(kt)^a) + Q \exp(-(lt)^b)$$

where R_{∞} represents the residual system load at infinite time
 P, Q are the size of the load change due to each process
 k, l are the characteristic time constants of the material
 a, b account for the distribution of relaxation processes.

The two exponential model can also be stated in a different form:

$$\text{Load}(t) = R_0 - P(1 - \exp(-(kt)^a)) - Q(1 - \exp(-(lt)^b))$$

where R_0 is the initial load and $R_0 - P - Q = R_{\infty}$

The two exponential model was originally applied to the corrected curves determined from the difference between high and low initial load tests. This was found to fit extremely well as shown in Figure 23. It was also established that the two exponential model gave a satisfactory fit to the data from the high initial load tests in most cases. Fits were improved if the first few points were excluded from the fit. As the initial points include non-reproducible effects such as thermal expansion of the rig and the sample bedding down, fitting to the later points will give a more dependable estimate of future behaviour. Figure 24 shows the two exponential model fitted to data not corrected by the low load test (ie the H curves) at times greater than 1000s from the 75MPa curves of as-moulded and post-cured phenolics, the fits are very satisfactory. Not surprisingly, the double exponential model also fits room temperature relaxation curves although the single exponential model is adequate.

At high temperatures the rate of load fall is much higher than at room temperature. This is due in part to the addition of another process arising from the isothermal shrinkage of the sample. However, theoretically the rate of viscoelastic relaxation should rise with temperature. Figure 6 shows this. Information about the viscoelastic properties cannot be directly inferred from the data owing to the effects of isothermal shrinkage.

A quick estimate of the long time behaviour of a bolted material at high temperature can be made in a similar manner to the estimation of the parameters in the single exponential model. By plotting:

$$\log(\text{time}) \text{ vs } \log[- \ln \{ (\text{Load}(t) - A)/B \}]$$

with estimates of A and B obtained from the long time portion of the relaxation curve, n and λ can be found from the linear part of this plot at high $\log(t)$. The exponential function plotted with the values found should fit well to the end of the stress relaxation curve (although the correlation with earlier points may well be poor). B can be taken as the load at the first point of the portion of the curve used. If A is taken as 0 then an estimate of the minimum service life can be made without recourse to computer fit routines.

Figure 25 shows the close correlation between single exponentials fitted to the measured stress relaxation data of as-moulded RX613 at 100°C at times after 10⁶s and a double

exponential fitted to data after 10^3 s. Different values of B (4000 - 7000 N) were used and differences in the values of the load predicted after 10^6 s are small. The extrapolation of the load from the experimental data is insensitive to B provided that the fit is to a portion of the curve significantly after the actual load has decayed below the value of B.

6 DISCUSSION

6.1 TESTS AT ROOM TEMPERATURE

The important feature for characterisation of a relaxation curve is its slope, which gives the rate of relaxation.

A complicating factor in the measurements is the effect of bedding-down of the bolt threads and of the washer into the sample. This should only affect the first part of the relaxation curve where it would slightly increase the rate of load drop. If reproducible, it is partly corrected on taking the difference of high load and low load results.

The stress relaxation rates for as-moulded samples showed limited scatter, Figure 2. Sample number three was an exception to this, as it behaved more like a sample with between 7 and 22 hours post-cure heat treatment. An explanation for this has not been found but DMTA measurements have shown that occasionally mouldings may behave differently to others in a batch. This is believed to be due to inhomogeneity of feed stock or of moulding conditions.

Mouldings with a standard post-cure treatment showed better reproducibility and the rate of relaxation was decreased, Figure 3. This was expected as the number of cross-linking bonds would have increased with the post-cure treatment and further reduced the mobility of the network. Vynkier RX613 phenolic mouldings were shown to be effectively linear viscoelastic at room temperature by the fact that the relaxation test at three different initial stresses gave the same relaxation rate, to within material variation, Figure 3. The effect of increased post-cure time at 170 °C was to reduce the relaxation rate, Figure 4. The decrease was not linear with post-cure time but showed that improvement became less with heat treatment time. From these results the standard post-cure time of 7 h at 170 °C could be seen to be a reasonable heat treatment.

The comparison between the simple type I and more elaborate type II rigs, Figure 5, showed that the two rigs gave nearly identical results, well within experimental scatter. Thus for practical purposes with this material the rigs have equivalent elastic properties. However this was for testing linear viscoelastic materials (see below) and for a non-linear viscoelastic sample such as a thermoplastic this factor would need re-checking.

6.2 TESTS AT ELEVATED TEMPERATURES

The load decreases at high temperatures more rapidly than at 23°C. Comparison of the loading behaviour at 100°C of as-moulded and post-cured phenolics is made in Figure 24. As at room temperature, the post-cured material shows slower load fall-off than the as-moulded sample. However, after 3 months even the load on the post-cured sample was less than 50% of the initial value. The actual value of load is dependent on the initial load. Above ambient temperature the strain is not constant due to the increase in sample thickness on heating. When the samples were removed from the oven significant load drops were observed, and again this was due to the difference in expansion coefficient of the rig and sample. The implications of this are discussed below.

The initial load appears to influence the rate of the stress relaxation as shown in Figure 9 which shows the proportion of load lost in samples of as-moulded RX613 at 100°C torqued

to different initial loads (2.1, 4.6, 7.2 and 9.8 kN). The curves do not match. If thermal expansion and isothermal shrinkage are unaffected by stress then these will contribute proportionately more to the load at low stress than at high stress. The increase in load from 1100 to 1900 N at the start of the 150°C low load test (Figure 10) is an extreme example of the effects of thermal expansion in the tests.

Additional post-cure at 250°C for 1 h appears from the plot of the relaxation curve (Figure 7, file 1T100) and the model fit parameters to have made little reduction in the long-term relaxation rate of this material. Relaxation curves at 100°C (Figures 7 and 8) show a large degree of scatter and no significant improvement is observed in relaxation properties following the additional post-cure. The steeper portion of a relaxation curve is associated with a transition. This steepening appears to happen at approximately 10^5 s for both treatments. If the extra treatment had shifted a transition to higher temperatures, then it would be expected that the steeper portion of the curve would occur at longer times, and that is apparently not the case.

The results of the isothermal shrinkage measurements at elevated temperatures, Figures 11 and 12, demonstrate that this phenolic continues to shrink in nominally load-free conditions and at 150°C will shrink to less than the original dimension at 23°C in less than one week. At 100°C isothermal shrinkage rates are slower, but the sample will still shrink to less than its original size within a few weeks. Under load, the isothermal shrinkage will begin to reduce the load applied to the sample as soon as the material shrinks from its maximum expansion. With phenolics, this occurs around 100 minutes after heating. In the period between commencement of shrinkage and final size stability, isothermal shrinkage will contribute significantly to the reduction of load on a bolted joint. The isothermal shrinkage tests completed (Figures 11 and 12) do not show the phenolic stabilising within 3 months, thus shrinkage has long term effects. Thermal expansion/shrinkage of the as-moulded material occurs to a greater extent than in the post-cured phenolic.

Figure 26 shows the load predicted due to the (constricted) differential expansion of the as-moulded and post-cured phenolic (elastic modulus taken as 6 GPa) and bolt in a type I rig at 100°C and 150°C. This shows significant effects on the load, particularly in shrinkage. At long times the measured rate of load reduction measured is approximately equal to that predicted from the shrinkage. This suggests that changes in the size of the sample are more important at long times than the viscoelastic stress relaxation. Isothermal shrinkage occurs over long periods of time and is concurrent with the stress relaxation, and therefore plays a significant part in the reduction of load.

Thermal shrinkage occurs on reducing the temperature of the sample, the zero-load isothermal shrinkage samples showed further contraction on cooling to room temperature. Table 1 (below) summarises the end load of the tests at elevated temperature and the residual load on cooling to room temperature. The load lost on cooling the samples tested at 100°C varied from 700N - 1750N with as-moulded and post-cured materials experiencing similar levels of load loss. In general, the lower the final load at 100°C the smaller the load lost on cooling, suggesting that the thermal contraction may be load dependant. The minimum acceptable load to maintain the joint in applications where the operating temperature is changing (eg motor engine components such as these) will be higher than this to allow for component shrinkage as it cools. It should be noted that the materials here were tested between 23°C and 100°C/150°C, in many practical applications the lower temperature limit will be much less (eg during winter).

Table 1: Loss of load on cooling sample

Temp (°C)	Material	Load (N) at Elevated Temp	Load (N) at 23°C	Loss (N)
75	RX613 PC	6439	5790	649
75	RX613 PC	1269	828	441
100	RX613 PC 1T100 ¹	2730	1668	1062
100	RX613 PC 2T100	3323	2436	887
100	RX613 PC 3T100	3783	2283	1500
100	RX613 PC PT100	3364	1678	1686
100	RX613 PC TE1c	4889	3149	1740
100	RX613 PC TE2c	3006	1954	1052
100	RX613 PC TE3c	3805	2251	1554
100	RX613 AM (9.8kN)	2773	2110	663
100	RX613 AM (7.2kN)	1043	6	1037
100	RX613 AM (4.6kN)	1278	557	721
100	RX613 AM (2.1kN)	369	77	292
150	RX613 PC	1035	193	842
150	RX613 PC	461	0	461

¹ Additional post-cure for 1 hour at 250°C

6.3 FINITE ELEMENT ANALYSIS

The analysis was undertaken to investigate the effect of changing geometry on the behaviour of a bolted joint. The effect of increasing sample diameter is quite small after the diameter exceeds twice that of the washer, Figure 14a. This is due to the constraint of the material that surrounds the loaded section. When the sample has the same diameter as the indenter there is less constraint on bulging of the loaded cylinder. Similarly for a thin sample, Figure 14b, the effect of changing diameter is small. Higher loads are required to attain a given indentation in the thinner sample, largely because as the strain is increased, and with a constant modulus the stress must also increase.

The size of the bolt hole has a relatively greater effect than the diameter of the sample (Figure 15). As the hole diameter increases the load required to attain a given indentation decreases. When the hole is small, a higher load is required, due partly to the increase in area over which the load is applied, and increased constraint.

The presence or absence of slip at the upper surface of the sample in contact with the bolt was negligible (Figure 16). At the lower surface (in contact with the base plate) slip

significantly decreased the load required to indent from 4640 N to 3710 N. The increased effect of slip on the lower surface is due to the larger contact area exerting more constraint.

6.4 ANALYTICAL MODELLING

Analytical models have been fitted to the load against t plots. Normalising to % of initial load against t will change the parameters describing the size of the loads (A , B , R , P and Q) in proportion. However, those describing the rate of relaxation (λ , n , k , l , a and b) will be unchanged.

The viscoelastic model proposed has fitted well to the stress relaxation behaviour of the phenolic component at 23°C, both with and without post-cure heat treatments (Figures 19, 20 and 21). The mean fit parameters (n and λ) of the single exponential model fitted to the stress relaxation of phenolic thermostat housing components are shown in Table 2. The standard 7 hour post-cure changes the time constant of the relaxation process from approximately 10^{-10}s^{-1} to 10^{-13}s^{-1} reducing the rate of relaxation by a factor of 1000 and increasing the service life of the component at 23°C by a similar factor. The mean value of the exponent n similarly falls from 0.20 to 0.13 in the post-cured material, the width of the relaxation processes is thus greater.

Table 2: Mean fit parameters from single exponential fitted by logarithmic method to room temperature relaxation curves

$$L(t) = A + B \exp -(\lambda t)^n, A = 0$$

No of Tests	Post-Cure	$\lambda \text{ (s}^{-1}\text{)}$	n
6	0	$2 (\pm 1) \times 10^{-10}$	0.197 ± 0.02
5	7 hours	$3 (\pm 4) \times 10^{-13}$	0.1346 ± 0.012

In many cases a better fit could be made by allowing the parameters A and B to be variables on a computerised least squares iterative fit. $A+B$ will converge to a value close to the initial load. The estimate of initial test load used in the fits above contains a degree of uncertainty. At the start of a test the load relaxes very quickly both through viscoelastic processes and the bolt/joint settling down, relaxation occurs as the bolt is torqued. Thus the total load applied will be more than the initial load reading noted (the highest load cell reading on finishing torquing). There will also be uncertainties in the times of the earlier readings as the loading takes several seconds and the data-logger reading period is 36s, the relative uncertainty in later readings is much less. More reliable estimates of future behaviour will be made by excluding the early data. The parameters λ and n vary considerably and are, to a great extent, determined by the size of A , the predicted residual. A high predicted residual leads to a faster relaxation and, hence, a larger λ . These residuals are all predicted to occur outwith the duration of the tests. The long time required to reach these predicted residuals (years or more) virtually precludes the use of the current test at room temperature to confirm these predictions. The assumption that the load will tend to zero represents a "worst case" scenario and will give a safety margin in service lifetime predictions.

Table 3: Mean fit parameters for single exponential fitted to room temperature relaxation curves with A and B as variables

$$L(t) = A + B \exp -(\lambda t)^n$$

File	Post-Cure	A	B	$\lambda \text{ (s}^{-1} \text{)}$	n
R0H175	0	3951	3354	1.12×10^{-7}	0.1786
R0H275	0	4702	2376	1.32×10^{-9}	0.1711
R0H375	0	11	6980	2.34×10^{-11}	0.1937
R0H475	0	2	7232	5.65×10^{-11}	0.1565
R0H575	0	1	7428	5.01×10^{-11}	0.2334
R0H675	0	4449	2703	2.30×10^{-7}	0.2561
R7H53s	7	900	4150	1.45×10^{-13}	0.1195
R7H66s	7	2149	4409	2.90×10^{-14}	0.0845
R7H751s	7	5805	1269	9.10×10^{-7}	0.2138
R7H753s	7	6289	1193	3.33×10^{-6}	0.2885

A is the value of the final load at infinite time, and in the preceding section taken as zero in the absence of a better estimate of the final load. Given the finite duration of the tests there is no compelling evidence to support this assumption. In fact stress relaxation tests at 150°C (Figure 10) suggest that a non-zero final value is reached. With A and B as variables, many of the room temperature data sets predict $A > 0$, and in some systems the predicted value of A is more than half the initial load (Table 3).

The single exponential model has proved to give a good fit to the difference between high load and low load data for post-cured phenolics at 100°C (Figures 13 and 23). Other relaxation curves at elevated temperatures are not satisfactorily fitted by the single exponential model. The double exponential model has proved satisfactory for describing the behaviour of the materials tested under all the test conditions. If the early points are excluded from the fit then this model is appropriate to use with high initial load data, removing the need to make a set of matched measurements at low initial load. A single exponential function can be fitted to the long time portion of the test data to give a satisfactory prediction of future behaviour.

Table 4 shows the parameters fitted to data after 1000 s using the two exponential model of stress relaxation data at elevated temperatures.

The relaxation rate parameters (k, l, a and b) fitted to the relaxation curves of the as-moulded phenolic components at 100°C are of similar magnitudes. Only the low load (L) test (2.1 kN) produces a predicted non-zero residual (257 N) but this is less than the load lost when the rig is cooled and, thus, is not significant.

The effect of increasing temperature is to accelerate the relaxation. The parameter k which determines the relaxation time of the largest relaxation component (P) rises with increasing temperature. The other parameters show no significant trends with temperature.

At 100°C the relaxation rate of the as-moulded sample is much faster than the post-cured samples. With the exception of k, there are significant variations in the parameters found for

these post-cured samples. This is not surprising given the large differences between the plotted stress relaxation curves (Figures 7 and 8). Some of the fits for the post-cured components predict non-zero residual loads (up to 2500N) at infinite time.

Table 4: Parameters fitted by two exponential model to stress relaxation data at elevated temperatures

$$L(t) = R_{\infty} + P \exp -(kt)^a + Q \exp -(lt)^b$$

File	R_{∞}	P	k	a	Q	l	b
Temperature = 75 °C							
PC H	0	8319	1.45×10^{-8}	0.4181	887	5.06×10^{-4}	0.749
PC L	772	1687	4.64×10^{-7}	0.6228	131	1.82×10^{-4}	0.620
Temperature = 100°C							
R0L10s AM (2.1kN)	257	2161	2.97×10^{-6}	0.3049	136	2.99×10^{-4}	1.713
RO5KN AM (4.6kN)	0	3617	1.61×10^{-6}	0.3774	1768	9.59×10^{-4}	0.564
R0h10s AM (7.2kN)	0	6685	6.77×10^{-7}	0.2818	1991	8.43×10^{-4}	0.627
R09KN AM (9.8kN)	0	7472	7.79×10^{-7}	0.2434	5932	1.87×10^{-3}	0.510
1T100 PC ¹	94	5647	2.84×10^{-8}	0.6053	2517	8.49×10^{-7}	0.250
2T100 PC	0	4237	2.81×10^{-8}	0.8506	3314	2.98×10^{-7}	0.304
3T100 PC	2229	5567	8.84×10^{-8}	0.5071	257	6.95×10^{-5}	1.149
PT100 PC	2604	3978	2.38×10^{-7}	0.5007	1036	2.52×10^{-4}	0.557
TE1c PC	1235	5681	1.27×10^{-7}	0.4392	198	2.29×10^{-4}	1.085
TE2c PC	2505	3378	2.86×10^{-7}	0.4795	6158	5.21×10^{-1}	0.034
TE3c PC	2459	4314	2.14×10^{-7}	0.4158	114	1.30×10^{-4}	2.318
Temperature = 150°C							
R7HT PC	821	4198	7.32×10^{-5}	0.4390	3695	1.78×10^{-6}	0.769
R7LT PC	432	1220	9.68×10^{-6}	0.9199	289	5.78×10^{-5}	1.643

¹ Additional post-cure at 250°C for 1 hour

The double exponential model does not explicitly separate load loss effects due to the viscoelastic relaxation and the change in size of the sample but rather accounts for the net effects of the two processes. Modelled shrinkage behaviour predicts that the isothermal shrinkage of phenolic components at elevated temperatures will contribute significantly to the decrease of the load on bolted joints. The analysis would be much simplified if the two components, stress relaxation and shrinkage, are additive in nature. A brief comparison of the behaviour of the low initial load tests and the expansion/shrinkage induced load changes

reveals that the increase of load on expansion is approximately that predicted from the thermal expansion of the material but the load reduction rate is less than that predicted from the shrinkage. The low initial load tests seem to be tending towards a non-zero residual whilst the shrinkage predicts a total loss of load within the duration of the test. At high initial load, the load trend is better correlated with the shrinkage model and it is possible to extract an estimate of the relaxation due to viscoelastic properties by subtracting the shrinkage from the test results. This suggests that viscoelastic stress relaxation occurs at a very rapid rate and has little influence at long times. The form of the viscoelastic relaxation is similar to that found in polyester DMCs^[4] which do not undergo isothermal shrinkage.

Sample geometries may influence the rate of relaxation at elevated temperatures. The samples coded TE, whose stress relaxation is plotted in Figure 8, were cut from the same thermostat housing. The sample in the test TE1c was cut from the 'A' corner of the thermostat housing, which has a different geometry to the 'B' and 'C' corners from which the other samples were cut. The corners cut from the thermostat housings have approximately triangular faces with a hole moulded into the centre, the 'A' corner of the housing has a longer base than the 'B' and 'C' corners (which are approximately the same shape and size) and is thus larger. Two of the three tests (files TE2c and TE3c) give similar parameters, their residuals are > 2400N. The residual for the third (TE1c) is less (1200 N). The slope of the relaxation curve of TE1c is much steeper than the those of the other post-cured components. This suggests that sample geometry will have some influence at elevated temperatures. Different flow patterns in the corners of the mould may also be responsible for variations in the relaxation behaviour.

The samples coded T100 were all 'A' corners and show similar relaxation behaviour to TE1c. The inconsistency in the fitted parameters may be due to the variability of the phenolic properties noted earlier.

Table 5: RX613 stress relaxation at 100°C fitted with the assumption that $R_{\infty} = 0$

$$L(t) = P \exp -(kt)^a + Q \exp -(lt)^b$$

File	P	k	a	Q	l	b
1T100 ¹	7674	3.23×10^{-8}	0.4271	236	5.56×10^{-5}	1.948
2T100	4237	2.81×10^{-8}	0.8506	3314	2.98×10^{-7}	0.304
3T100	8004	2.82×10^{-8}	0.4105	1991	1.05×10^{-4}	3.191
PT100	1978	2.42×10^{-7}	0.7676	20239	1.04×10^1	0.033
TE1c	4652	1.37×10^{-7}	0.4938	1416	7.06×10^{-4}	0.033
TE2c	6949	3.12×10^{-12}	0.0331	3214	2.92×10^{-7}	0.487
TE3c	5870	9.90×10^{-11}	0.0424	3297	2.21×10^{-7}	0.475

¹ Additional post-cure at 250°C for 1 hour

6.5 COMPONENT SERVICE LIVES

The fitted models described earlier predict, in many cases, final residual loads of up to 2500N. Clearly a component following this behaviour will remain securely bolted even if a 1500 N load drop on cooling occurs. Confirmation of this residual load would require tests having durations of several years which are impractical. It is therefore prudent to force the

model to fit to a final residual load of zero. This was done by fixing R_{∞} as zero in the computer least squares fit operation. Table 5 shows the values for the fit parameters then obtained with post-cured phenolic materials at 100°C. The values of k for the samples cut from the A corners are similar. The values of the fit parameters for the B and C corners (TE2c, TE3c) are approximately the same.

Table 6: Times (s) predicted for the load to decay to limiting value calculated from fit parameters in Table 5

File	LIMITING LOADS (N)						
	3000	2500	2000	1500	1000	500	10
R0h10	6.6×10^5	1.5×10^6	2.9×10^6	6.0×10^6	1.5×10^7	4.2×10^7	1.1×10^9
R05KN	9.5×10^3	4.2×10^4	1.6×10^5	4.4×10^5	1.2×10^6	3.8×10^6	6.5×10^7
R09KN	7.7×10^5	1.8×10^6	4.0×10^6	9.0×10^6	2.2×10^7	7.5×10^7	3.0×10^9
1T100	2.7×10^7	4.0×10^7	6.0×10^7	9.5×10^7	1.7×10^8	3.2×10^8	2.6×10^9
2T100	1.8×10^7	2.5×10^7	3.5×10^7	4.8×10^7	6.9×10^7	1.1×10^8	1.1×10^9
3T100	3.3×10^7	5.1×10^7	7.9×10^7	1.2×10^8	2.1×10^8	4.2×10^8	3.6×10^9
PT100	2.2×10^7	3.7×10^8	8.0×10^9	$>10^{10}$	$>10^{10}$	$>10^{10}$	$>10^{10}$
TE1c	1.1×10^7	2.0×10^7	4.0×10^7	1.6×10^8	$>10^{10}$	$>10^{10}$	$>10^{10}$
TE2c	1.6×10^9	$>10^{10}$	$>10^{10}$	$>10^{10}$	$>10^{10}$	$>10^{10}$	$>10^{10}$
TE3c	2.8×10^7	2.5×10^8	$>10^{10}$	$>10^{10}$	$>10^{10}$	$>10^{10}$	$>10^{10}$

The fitted models were used to predict the time taken in each 100°C test for the load to reach a preset value (10, 500, 1000, 1500, 2000, 2500 and 3000 N) in order to predict the useful life of the joint. These data are presented in table 6. The additional loss of load on cooling suggests that the higher loads will give a safer estimate of when the joint should be re-tightened to avoid failure of the component. Allowing a 1500 N drop on cooling and assuming that 500N is sufficient to maintain the integrity, a safe load limit of 2000N is estimated. Operating conditions in which the joint is cooled below room temperature may require a higher limit. If 2000N is taken as the safe load limit, even in the worst case of the post-cured phenolics tested here, the load is predicted to take 3.5×10^7 s (or about 1 year) to fall to this limit. The load on the as-moulded samples, even that with the highest initial load (9.8kN), falls to this limit within 2 months. Post-curing gives a far longer service life.

As an additional check on the joint lifetimes, straight lines were fitted to the long time portions of the relaxation curves (which appear linear on a load vs log (time) plot). The slope and intercept were used to calculate the time taken for the load to decay to set values (0, 1000, 1500 and 2000 N), Table 7. The time for the load to fall below 2000N is, for each post-cured phenolic tested here, greater than 5×10^7 s (1.5 years).

Table 7: Times (s) predicted for the load to decay to limiting value from linear extrapolation of load vs log (t) plots (Figures 7 and 8)

File	LIMITING LOAD (N)		
	2000N	1500N	1000N
1T100	6.36×10^7	1.03×10^8	1.67×10^8
2T100	6.70×10^7	1.14×10^8	1.92×10^8
3T100	1.26×10^8	2.13×10^8	3.60×10^8
TE1c	4.93×10^7	7.64×10^7	1.42×10^8
TE2c	2.80×10^8	6.37×10^8	1.45×10^9
TE3c	1.29×10^8	2.90×10^8	6.60×10^9

7 CONCLUSIONS

Use of the new NPL stress relaxation rigs, designed for investigation of bolted joints under stress for long periods of time, has provided the required characterisation of both material and joint behaviour.

Commercial phenolic mouldings are demonstrated to have satisfactory service life, and post-cure of these mouldings decreases the stress relaxation rate and thus increases the safe operating lifetime of a bolted joint significantly.

Post-cured phenolic RX613 components bolted onto metal parts should retain sufficient load to maintain the joint integrity under operating temperatures for at least 1 year.

Increasing the operating temperature of the joint increases the relaxation rate and reduces the service life.

In determining the importance of bolt geometry, finite element analysis has predicted that the effect of sample diameter is less significant than the size of the bolt hole. Also the presence of slip is more significant on the surface next to the base than on the surface in contact to the bolt head. For a linear viscoelastic material the effects of sample geometry can be separately calculated, and geometry does not affect the rate of relaxation (although this would not be the case for non-linear viscoelastic plastics).

Post-cured phenolic RX613 is effectively a linear viscoelastic material at room temperature.

The Kelvin function $\text{Load}(t) = A + B \exp -(\lambda t)^n$ fits well to the relaxation behaviour of phenolic at 23 °C. It is also suitable for modelling some relaxation results at 100°C once allowance for thermal expansion and shrinkage has been taken into account from a low load test.

The proposed single exponential function is not suitable for predicting the stress relaxation behaviour of most of the phenolic samples at high temperatures. This is largely due to the additional effect of isothermal shrinkage during the test period, which has been shown to be appreciable.

A two exponential function ($\text{Load}(t) = R_{\infty} + P \exp -(kt)^a + Q \exp -(lt)^b$) accounts for the additional process. This model is suitable for describing all of the tests to date (if initial

points are excluded), removing the need to make matched high and low load tests.

At elevated temperatures, decay of the bolt load is non-linear and test behaviour of similar samples can vary greatly. Sample geometry may influence relaxation behaviour.

8 FURTHER WORK

Work on a range of polyester dough moulding compounds (DMCs) has been started and tests at 23°C and 100°C are being carried out on these materials. The component is the same as that tested for the phenolic material. Components made from an injection mouldable phenolic DMC have also been made and tested. It will be possible to compare the relaxation behaviour of granular phenolic with phenolic DMC, and of polyester DMC with phenolic DMC.

Measurements of thermal expansion and isothermal shrinkage are also being made on the above materials. The stress relaxation behaviour of these materials will be described in a future report.

9 ACKNOWLEDGEMENTS

The authors wish to express their gratitude to D Ferriss, P Gibbs and K Thomas (all DMM, NPL) for their advice and help pertaining to this report.

10 REFERENCES

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- [2] GIBBS, P.A.J.; Private communication of unpublished NPL work.
- [3] FERRISS, D.; Private communication.
- [4] COOK, A, DAWSON, D and DUNCAN, B. C.; Stress relaxation in dough moulding compounds, *NPL Report DMM(A)110*, July 1993.

Figure Captions

- Figure 1 Test rigs
- Figure 2 Stress relaxation of as-moulded phenolic components at 23 °C
- Figure 3 Stress relaxation of post-cured phenolic components at 23 °C with initial stresses of 53, 66 and 75 MPa
- Figure 4 Effect of post-cure time on stress relaxation behaviour at 23 °C
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- Figure 7 Stress relaxation of post-cured phenolic components cut from different samples at 100°C
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- Figure 24 Double exponential model fitted to high initial stress curves of as-moulded and post-cured phenolic at 100°C
- Figure 25 Single exponential model fitted to high time points of the stress relaxation of as-moulded phenolic at 100°C compared to the double exponential model
- Figure 26 Calculated additional load due to thermal expansion / isothermal shrinkage of phenolic components at elevated temperatures

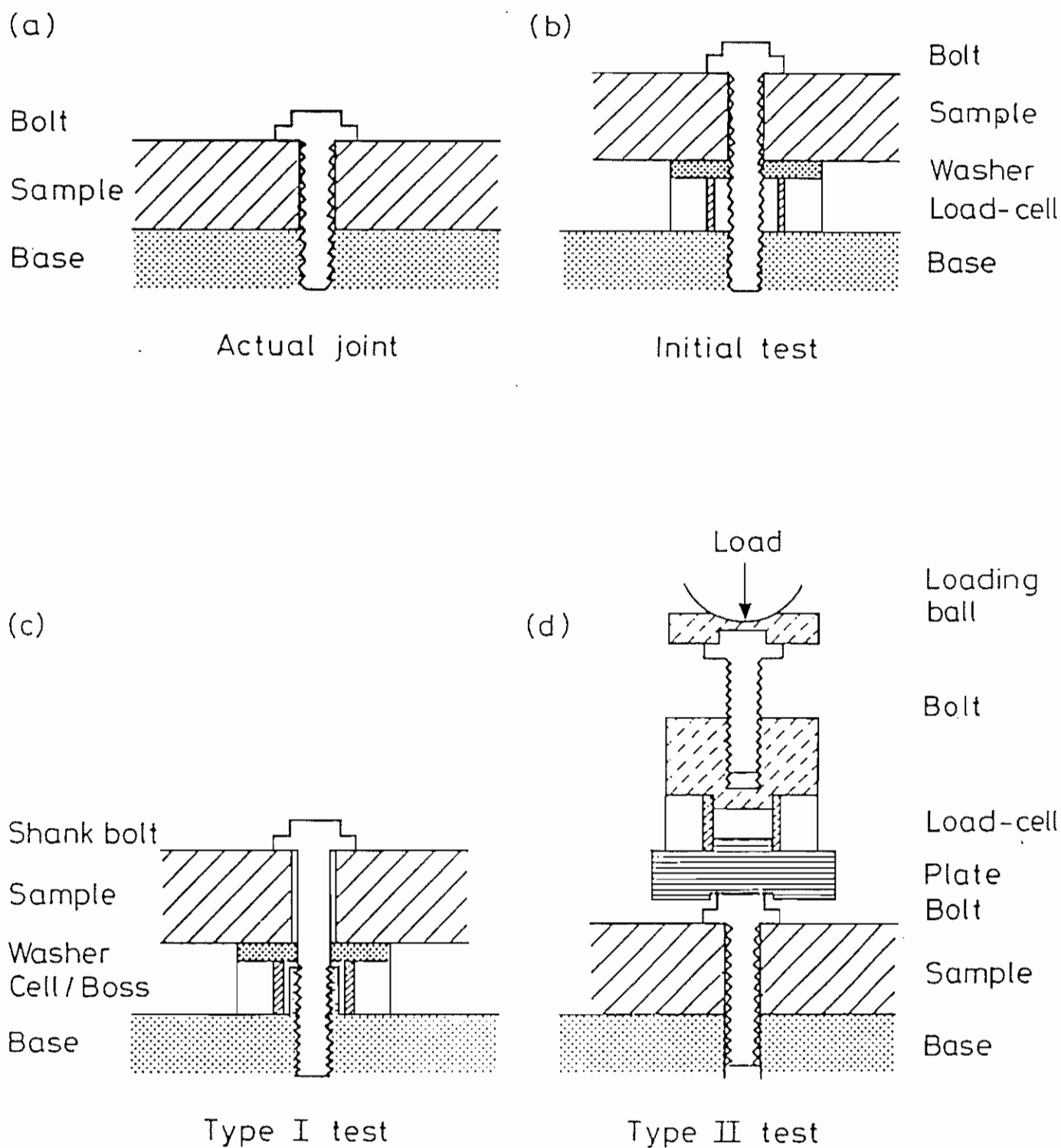


Figure 1 Test rigs

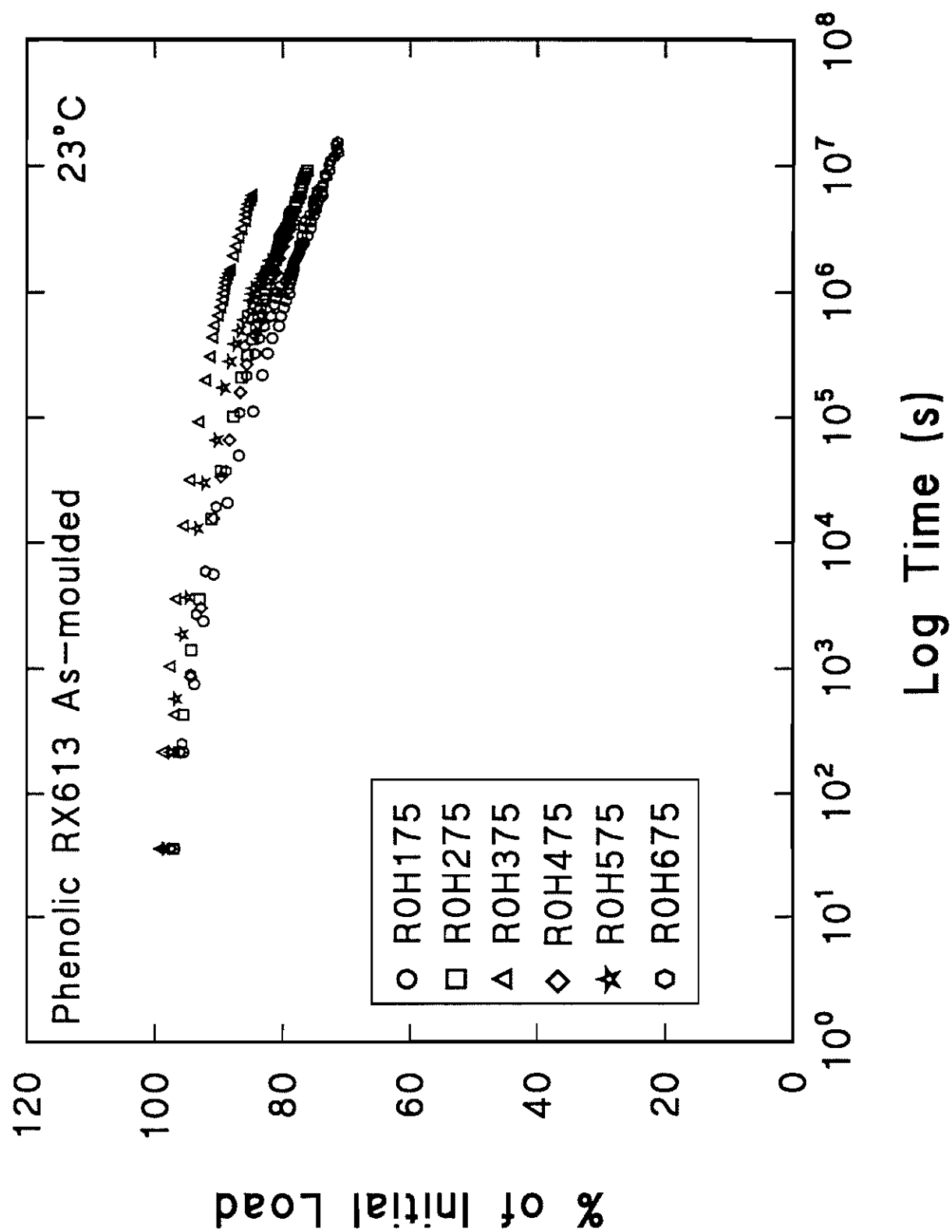


Figure 2 Stress relaxation of as-moulded phenolic components at 23 °C

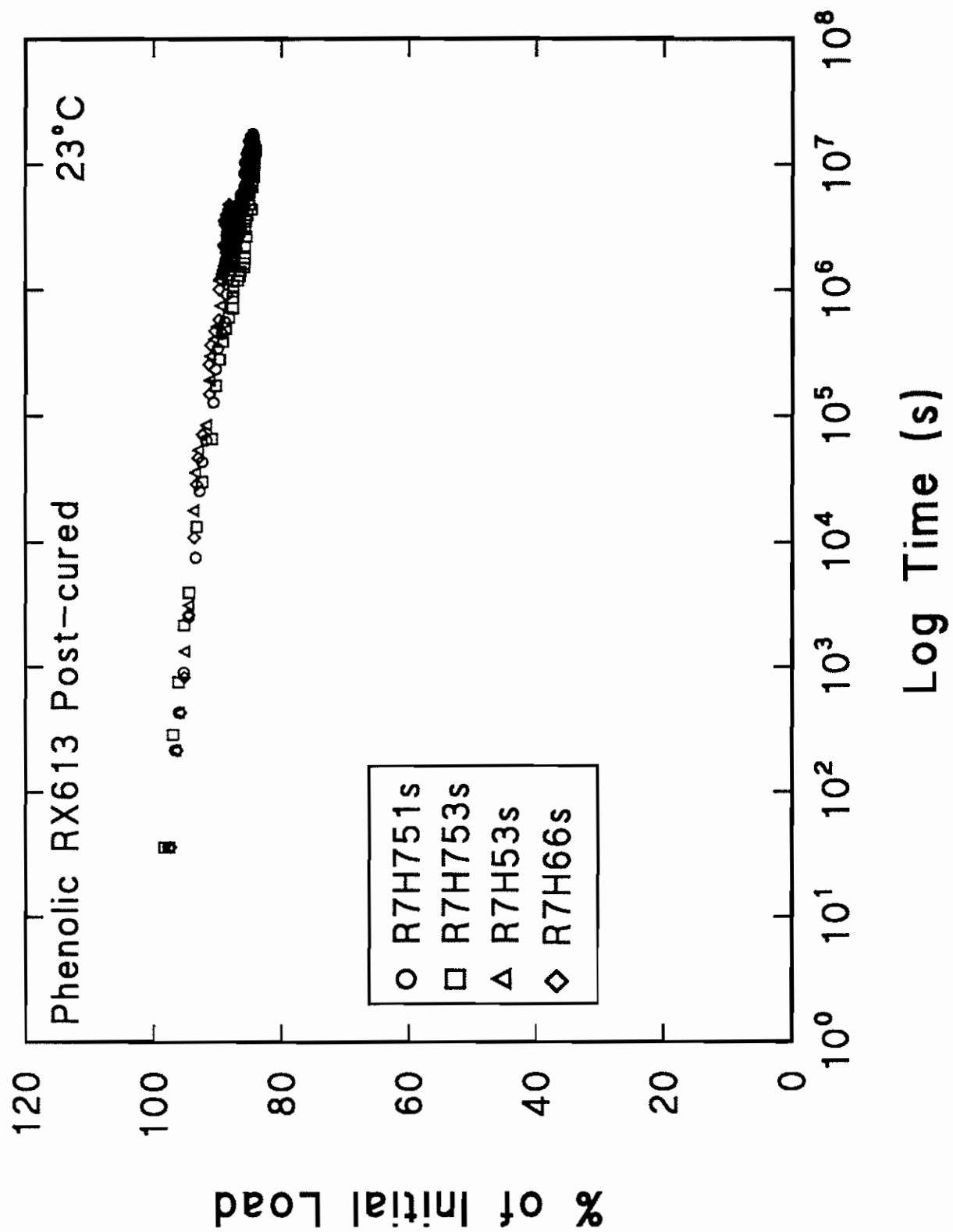


Figure 3

Stress relaxation of post-cured phenolic components at 23 °C with initial stresses of 53, 66 and 75 MPa

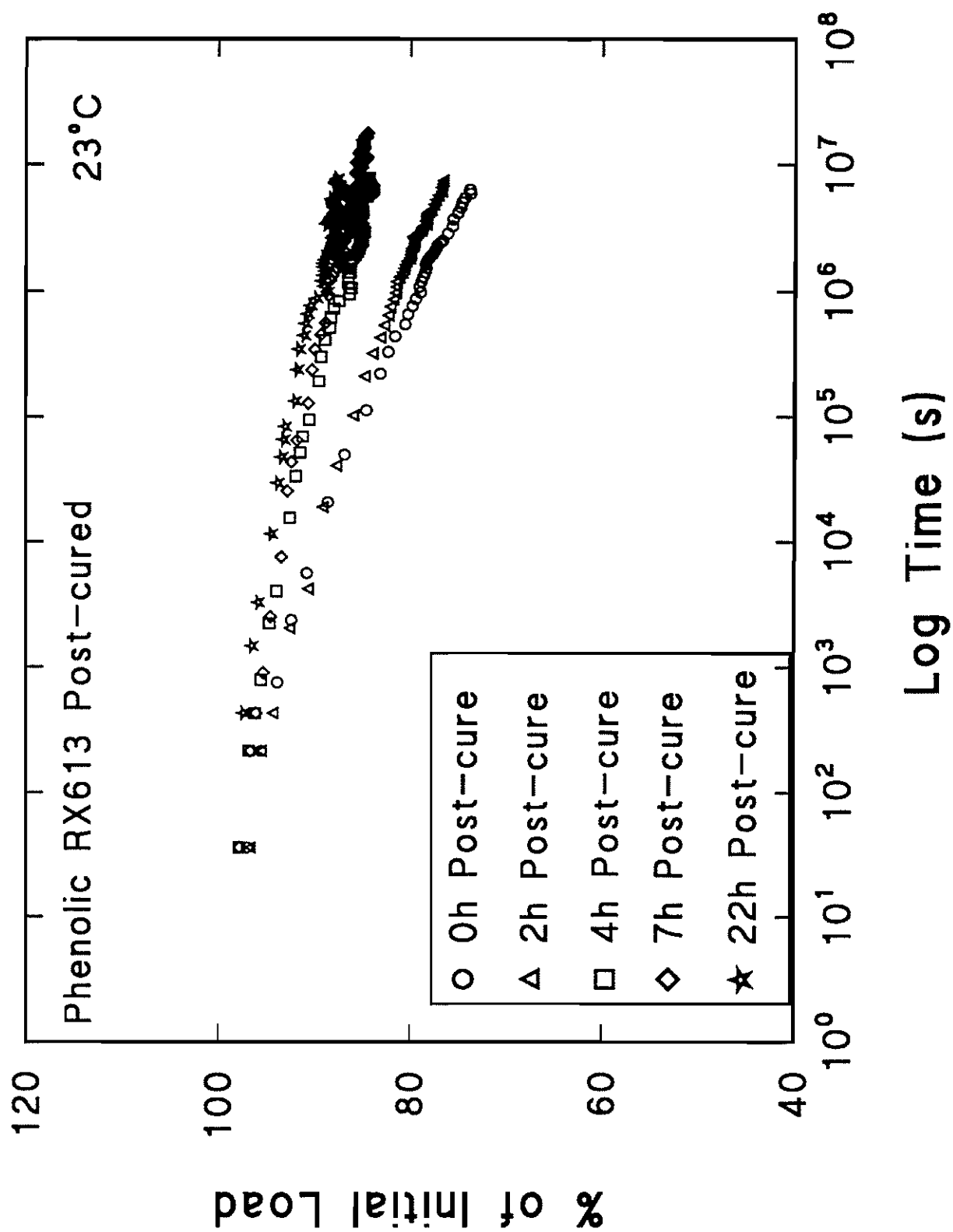


Figure 4 Effect of post-cure time on stress relaxation behaviour at 23 °C

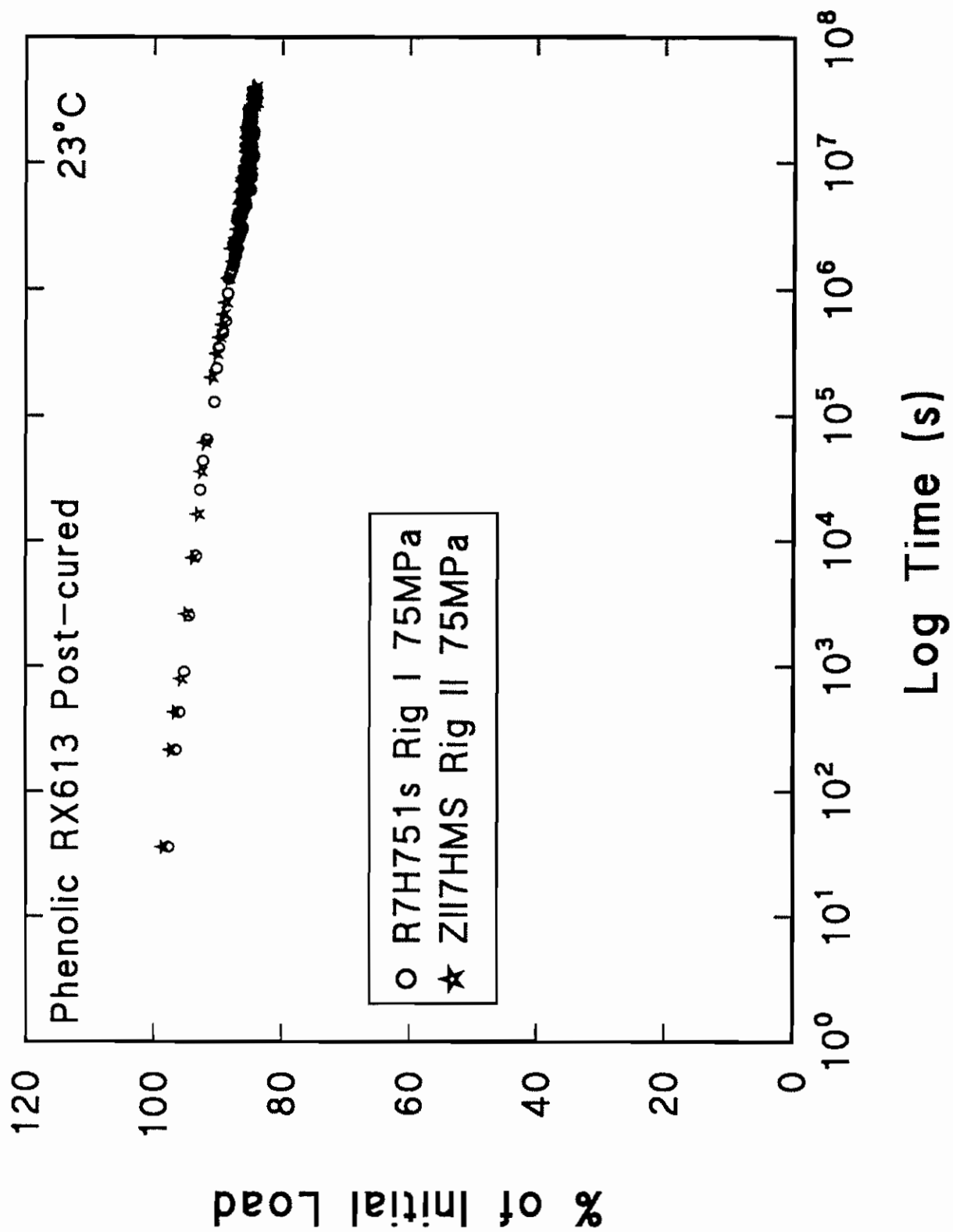


Figure 5

Comparison of stress relaxation of post-cured phenolic at 23 °C in type I and type II rigs

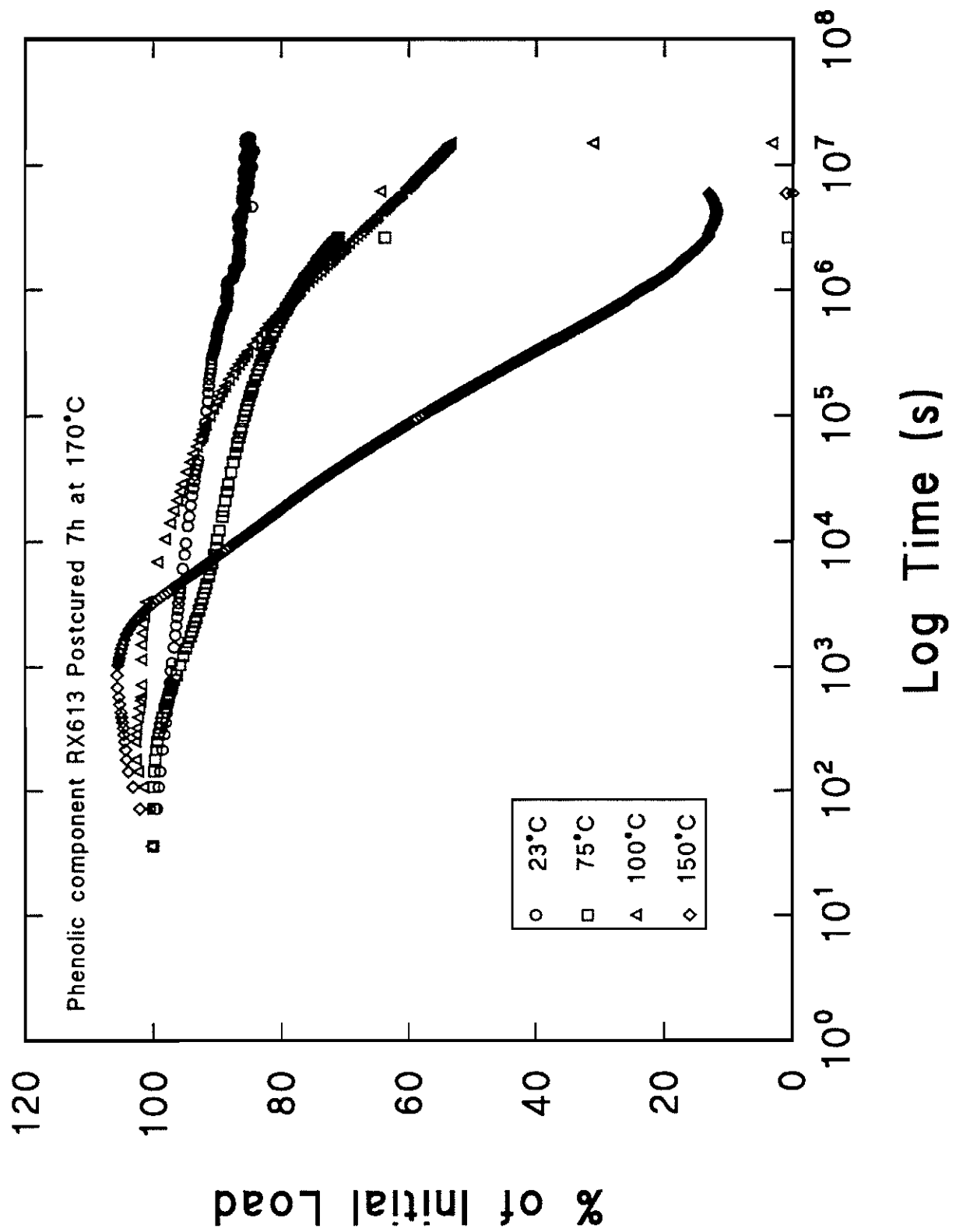


Figure 6

Stress relaxation of post-cured phenolic components at different temperatures

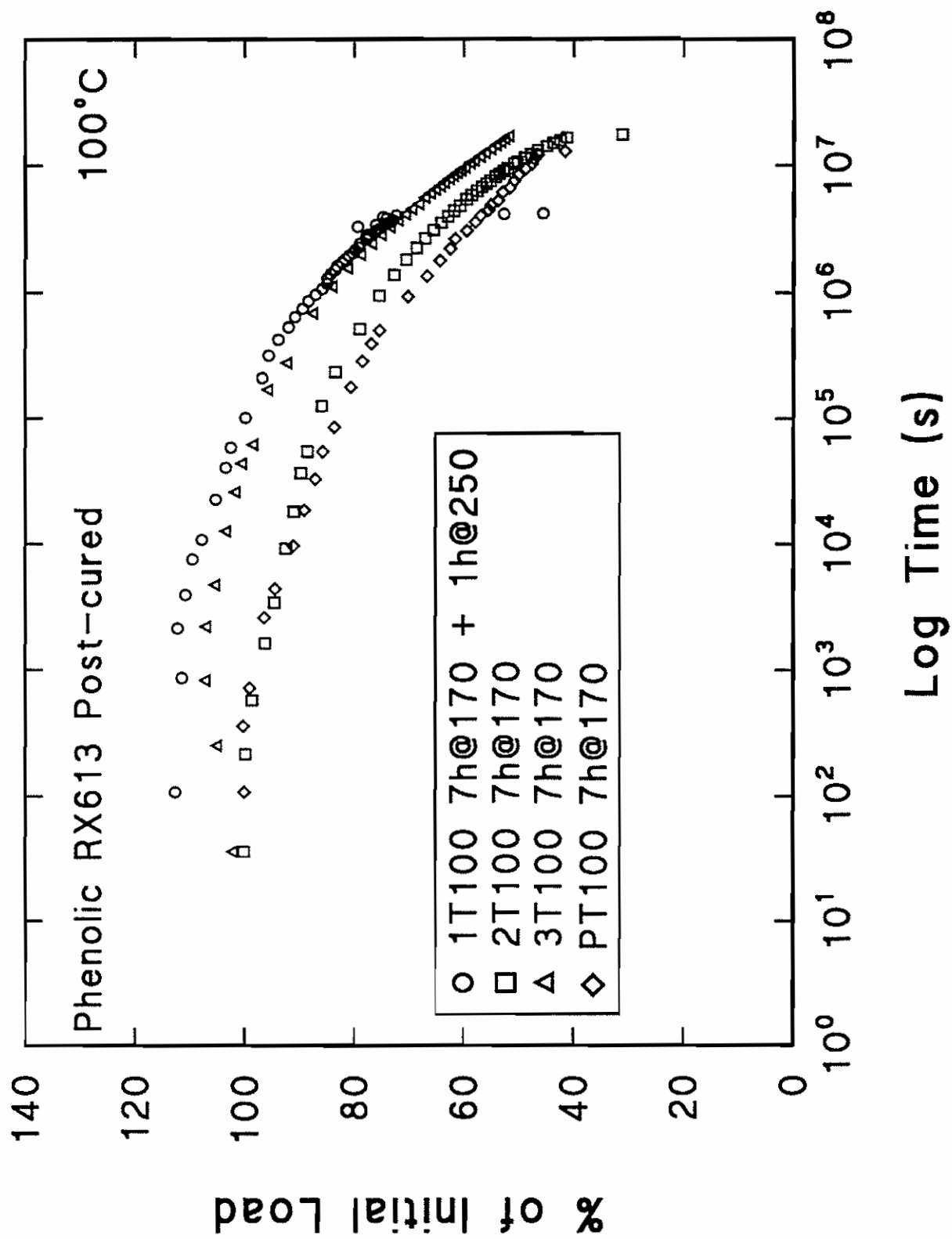


Figure 7 Stress relaxation of post-cured phenolic components cut from different samples at 100°C

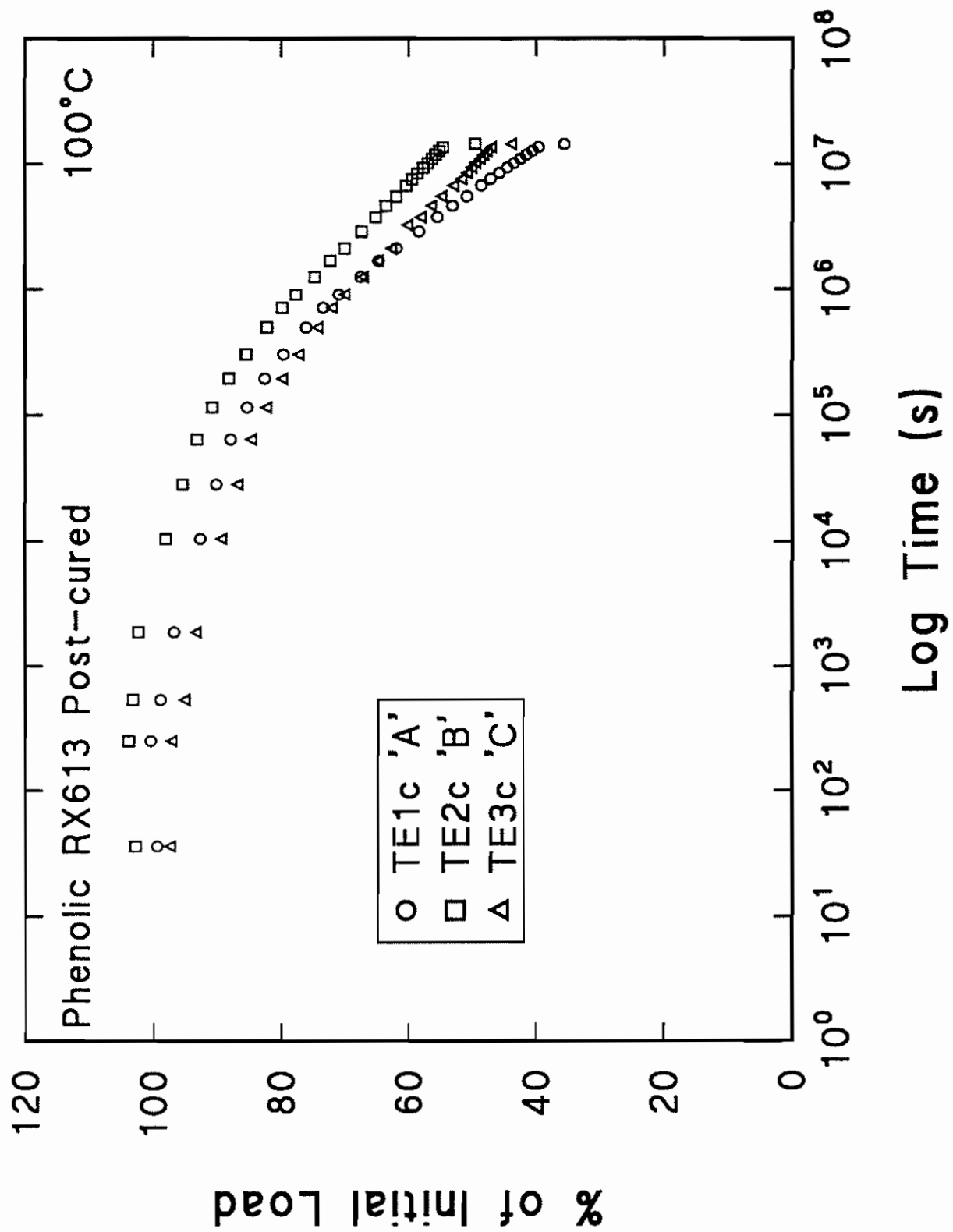


Figure 8

Stress relaxation of post-cured phenolic components cut from the same sample at 100°C

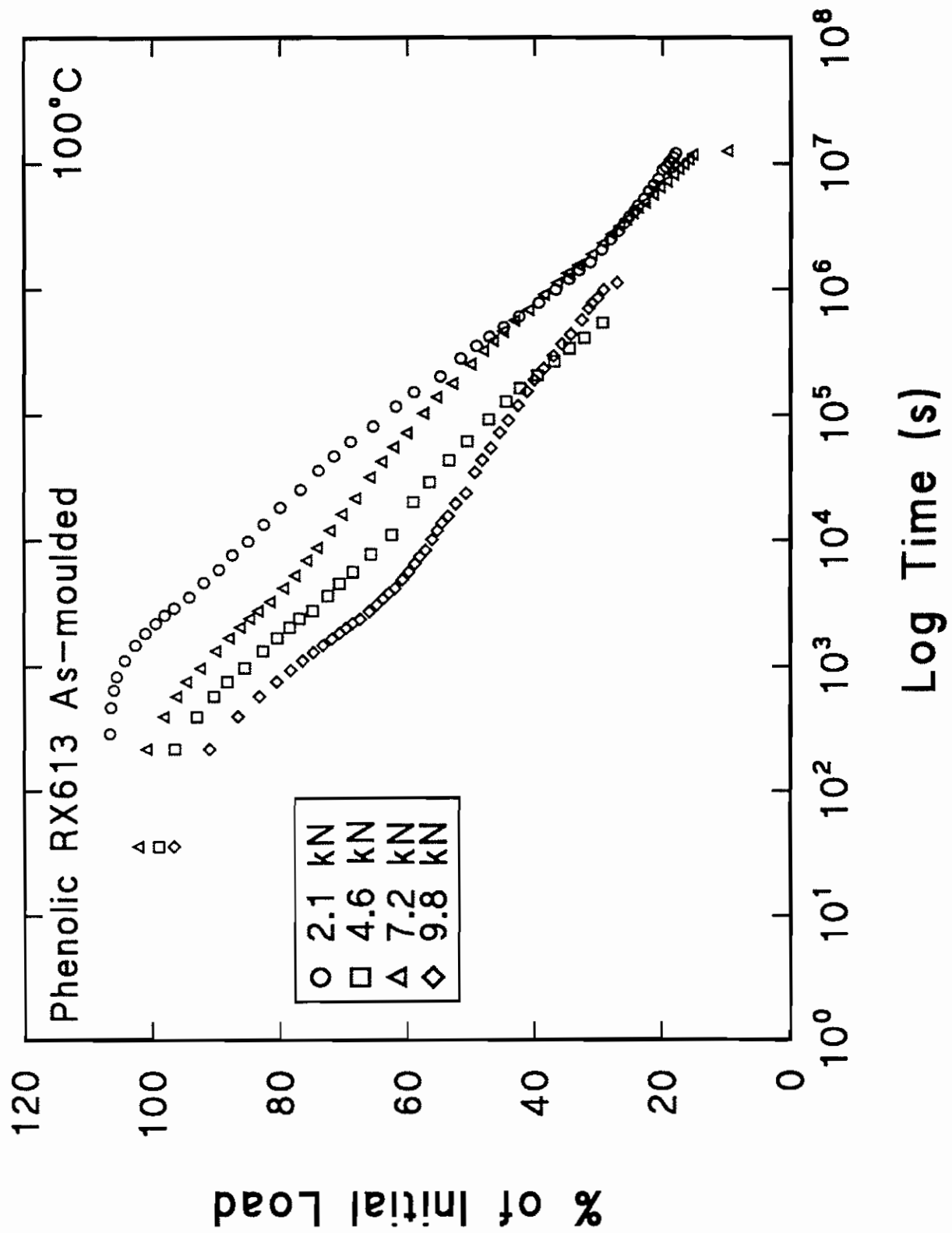


Figure 9 Stress relaxation of as-moulded phenolic components with different initial stresses at 100°C

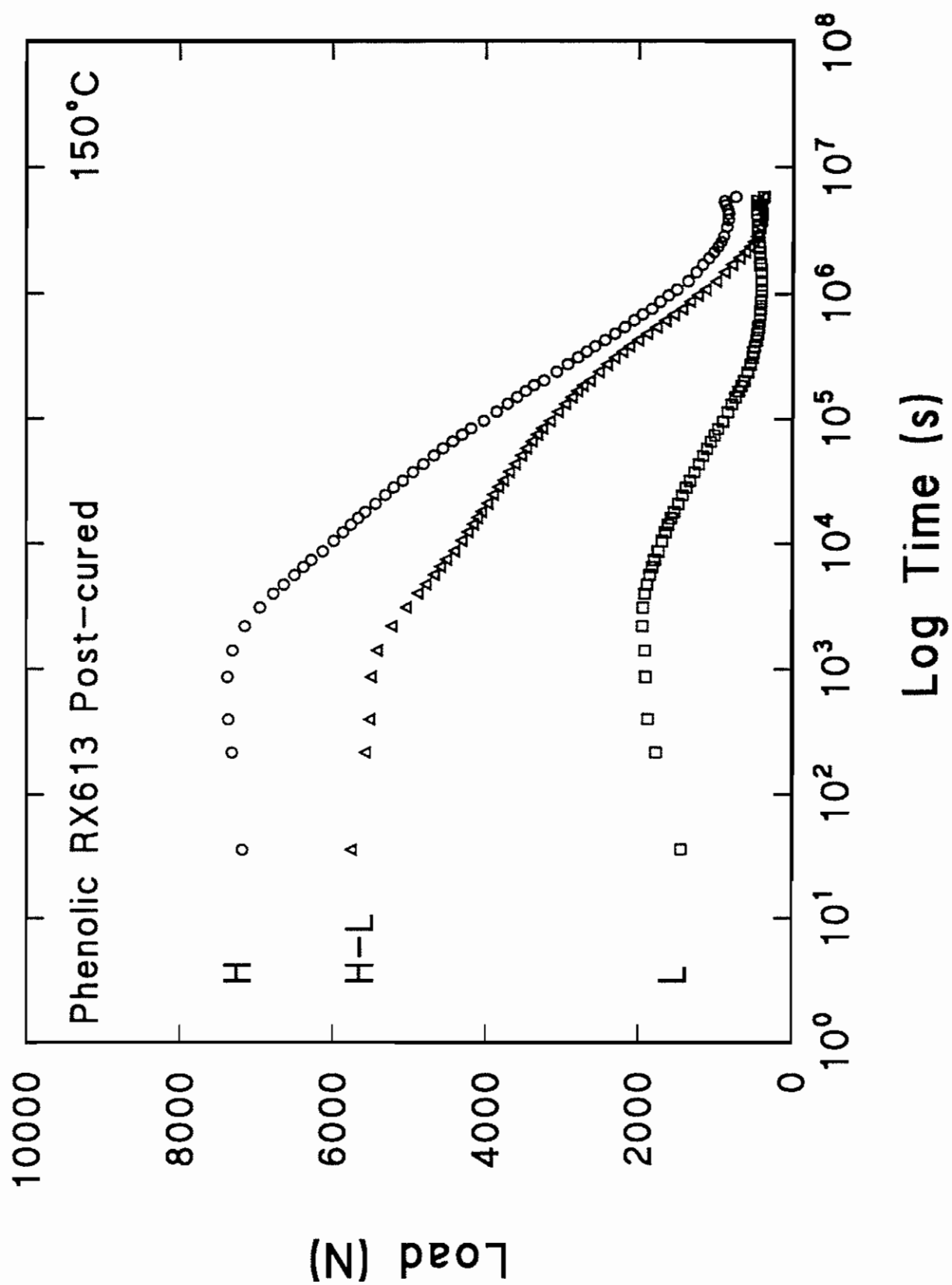


Figure 10

Stress relaxation of post-cured phenolic components at 150°C, H is the curve relating to a high initial load of 7000 N, L is the curve for a low initial load of 2000 N and H-L is the difference between them

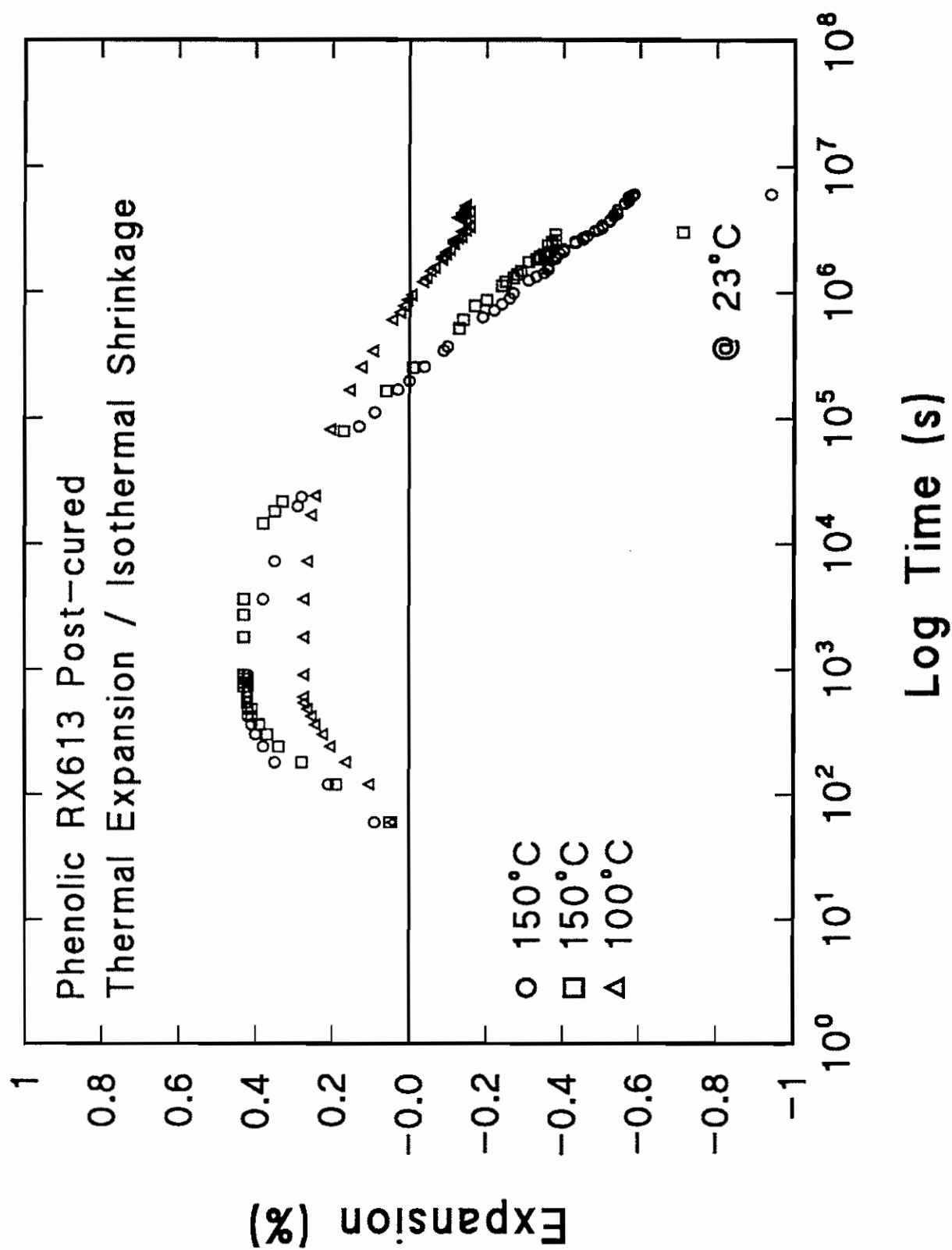


Figure 11

Thermal expansion / isothermal shrinkage of post-cured phenolic components at 100°C and 150°C and the shrinkage after cooling to 23 °C

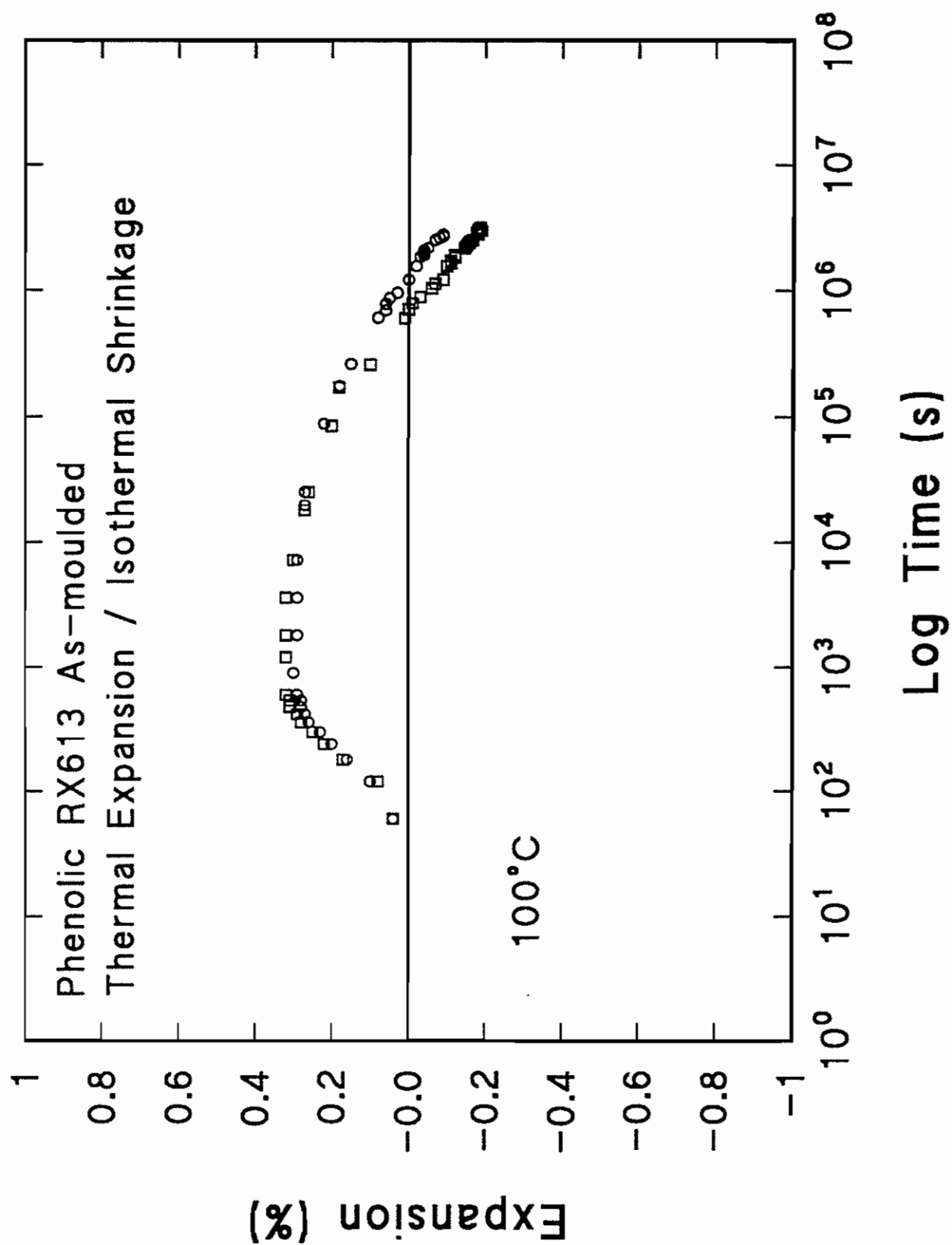


Figure 12 Thermal expansion / isothermal shrinkage of as-moulded phenolic components at 100°C

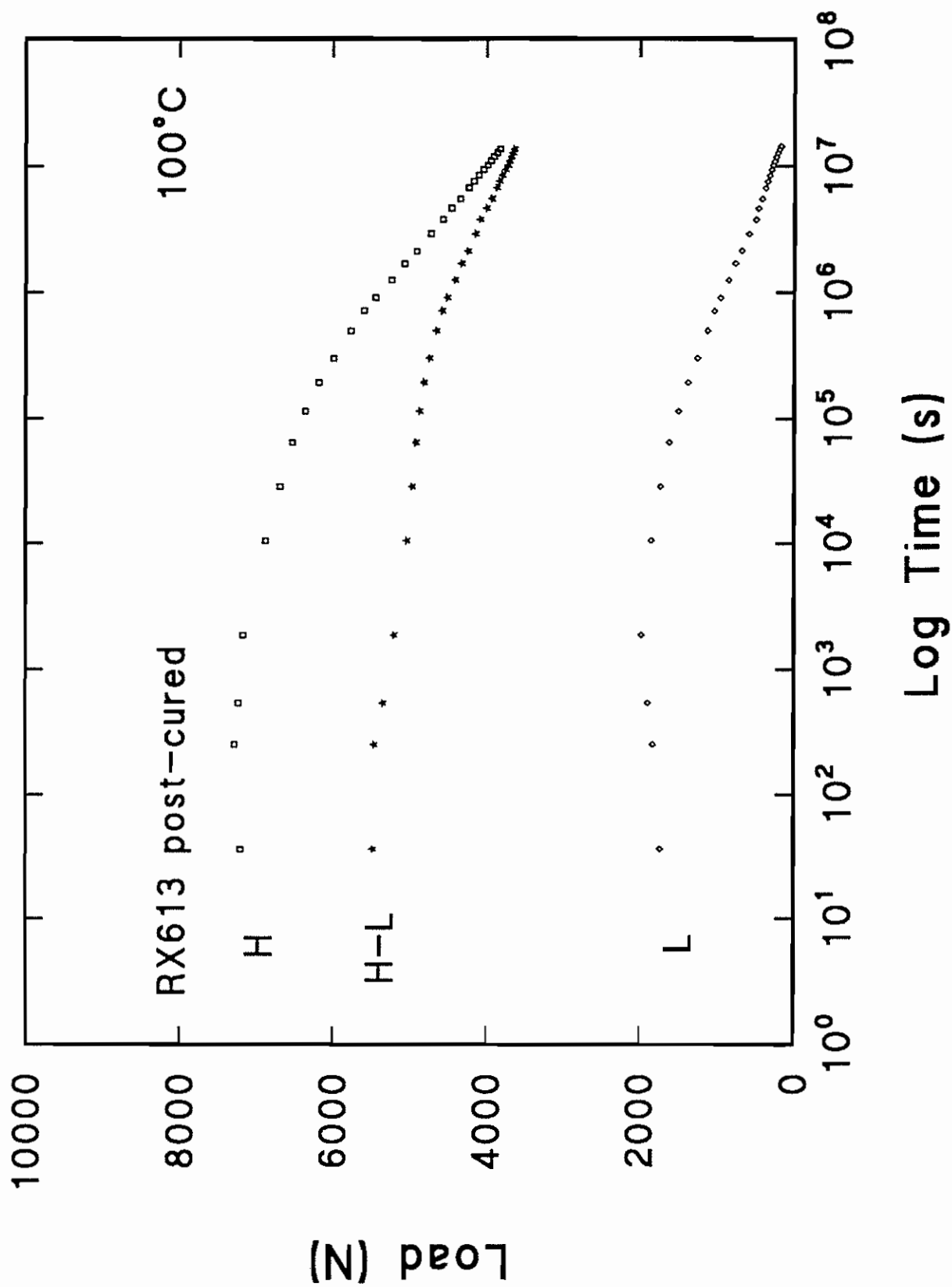
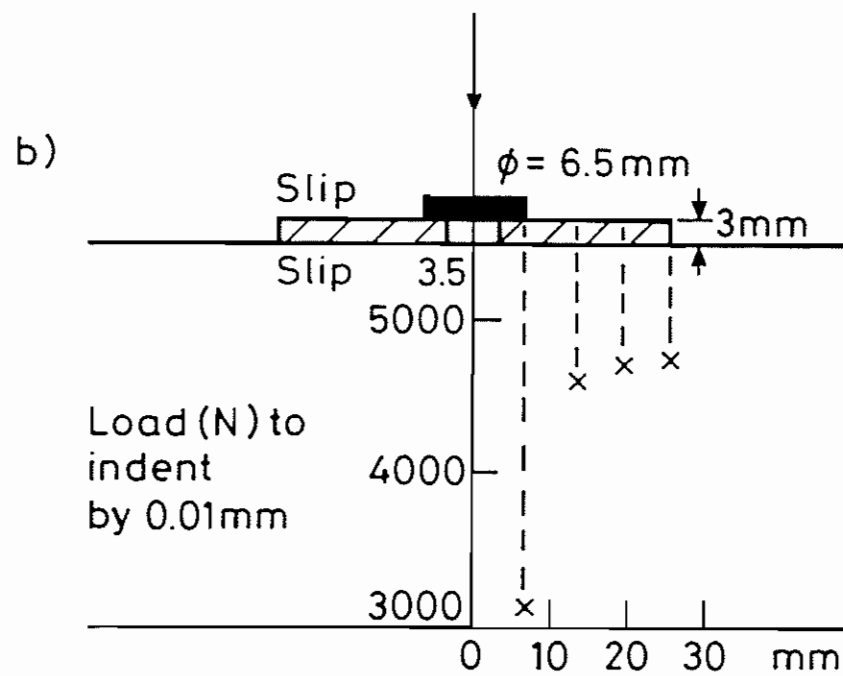
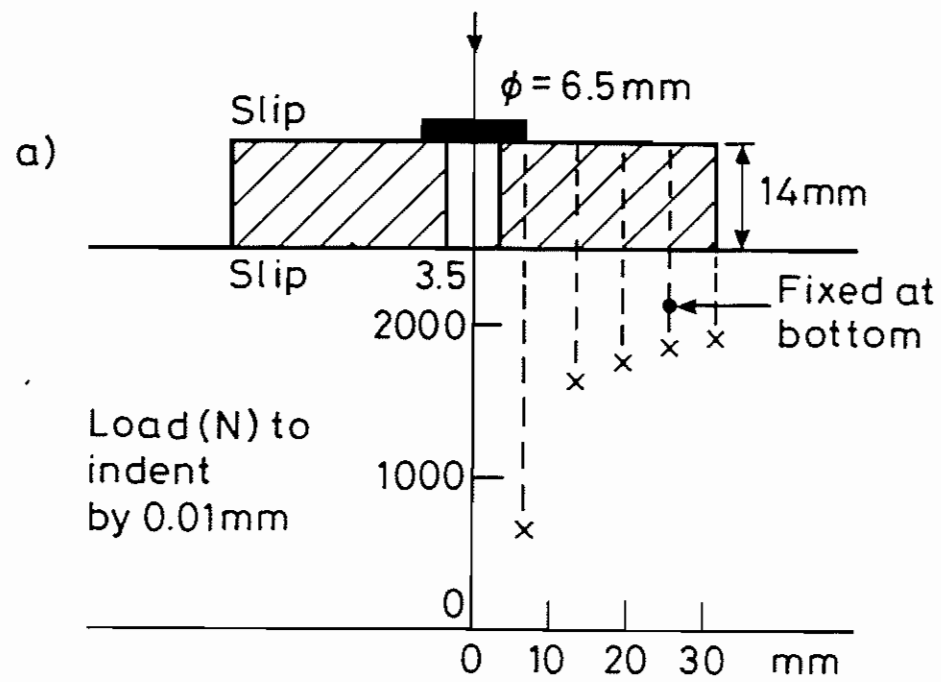
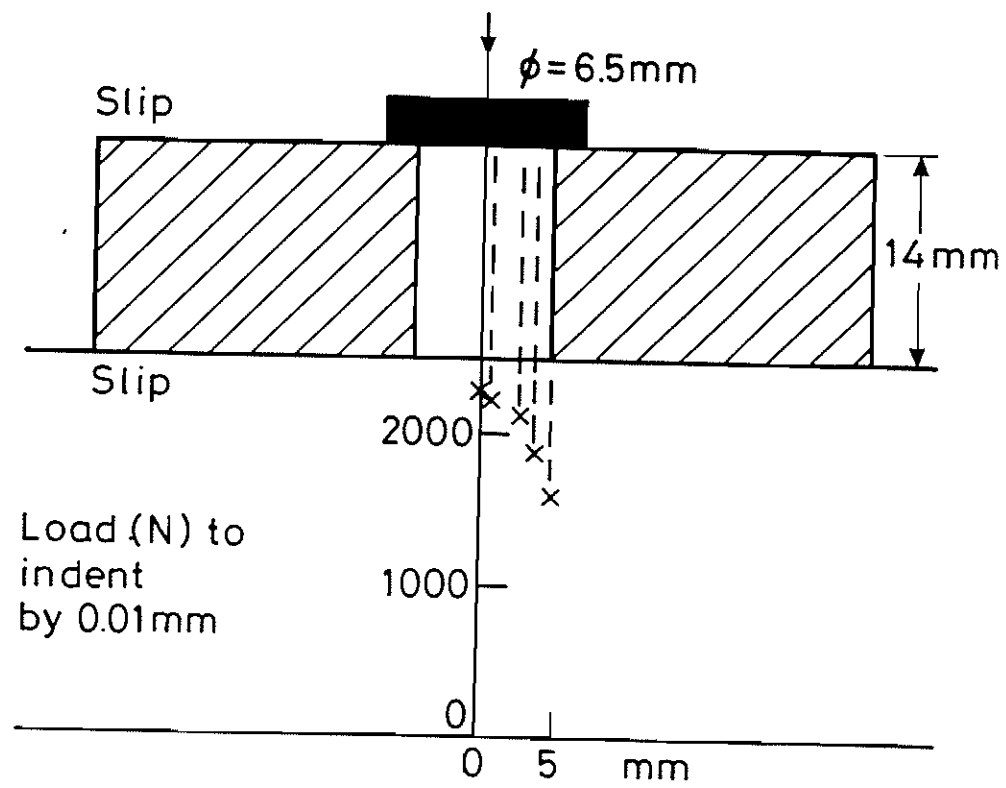


Figure 13 Stress relaxation of post-cured phenolic components at 100°C at high and low initial stresses (H and L) and corrected for load independent effects (H-L)



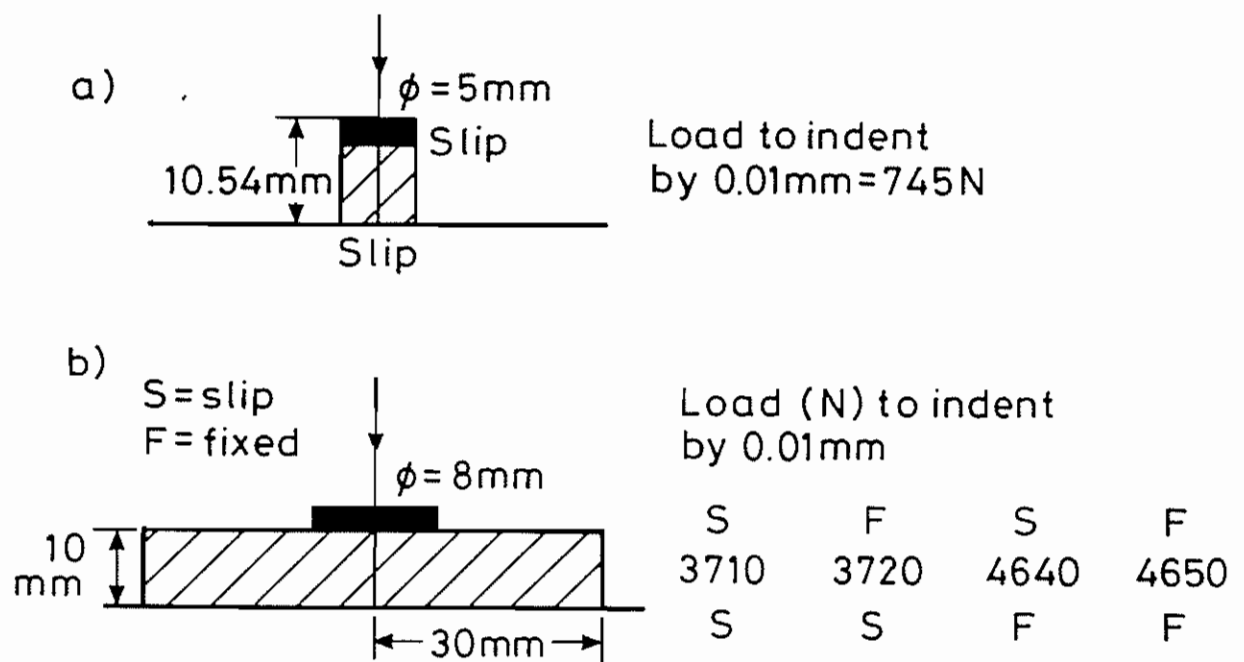
Effect of sample diameter

Figure 14 Finite element analysis of the effect of sample diameter



Effect of bolthole diameter

Figure 15 Finite element analysis of the effect of bolt hole size



Effect of slip at surfaces

Figure 16 Finite element analysis of the effect of slip at surfaces

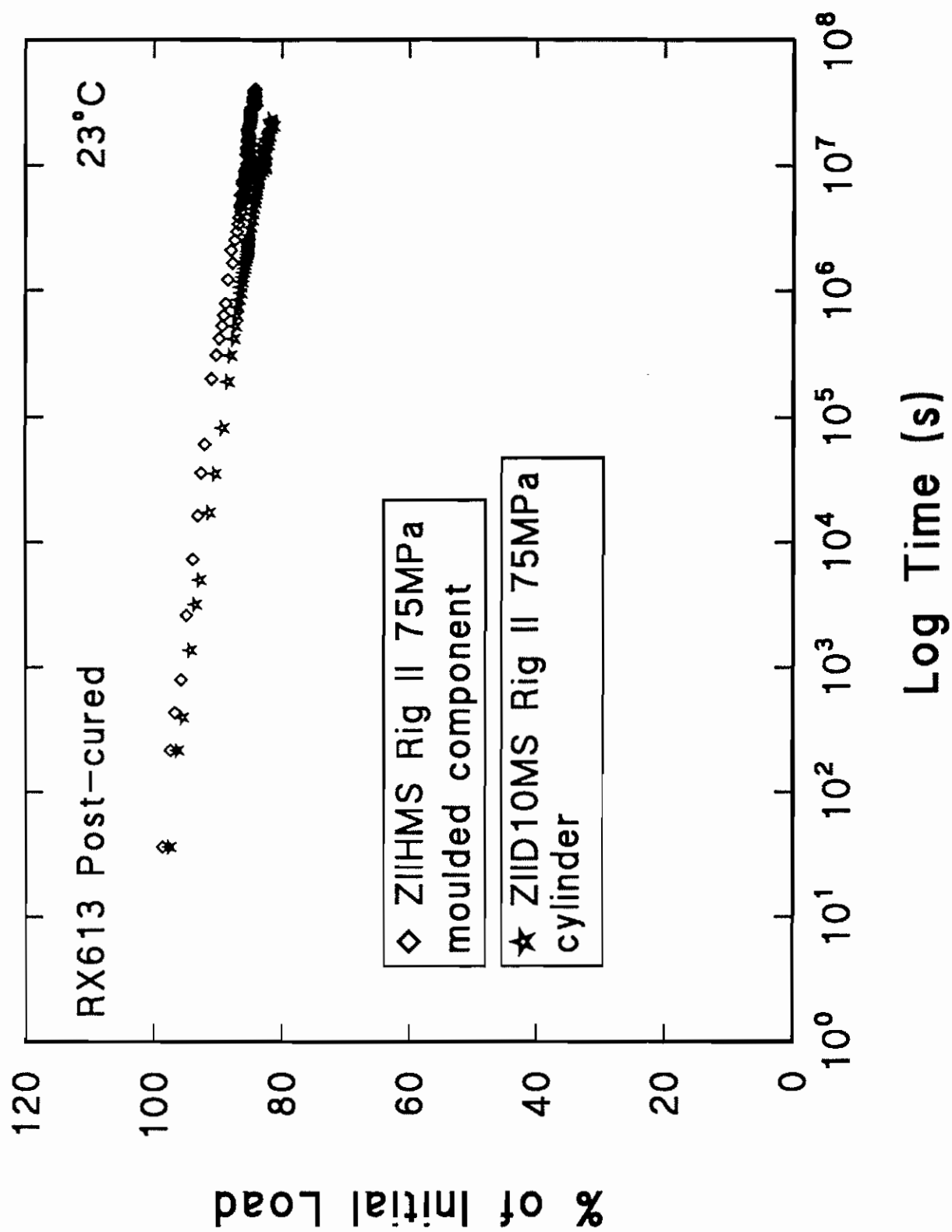
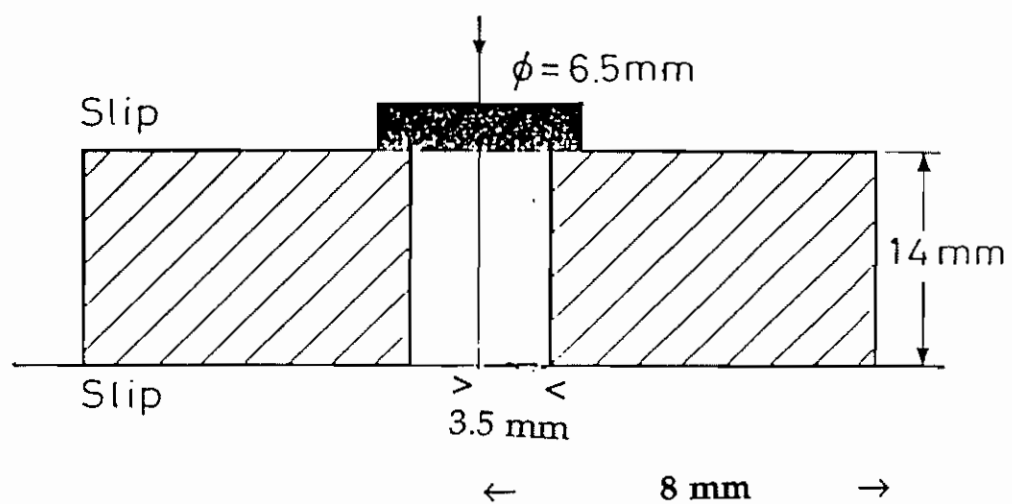


Figure 17 Effect of geometry on stress relaxation of post-cured phenolic at 23°C



Load (N) to
indent
by 0.01 mm
= 1078 N

Figure 18 Loading conditions corresponding to Figure 17

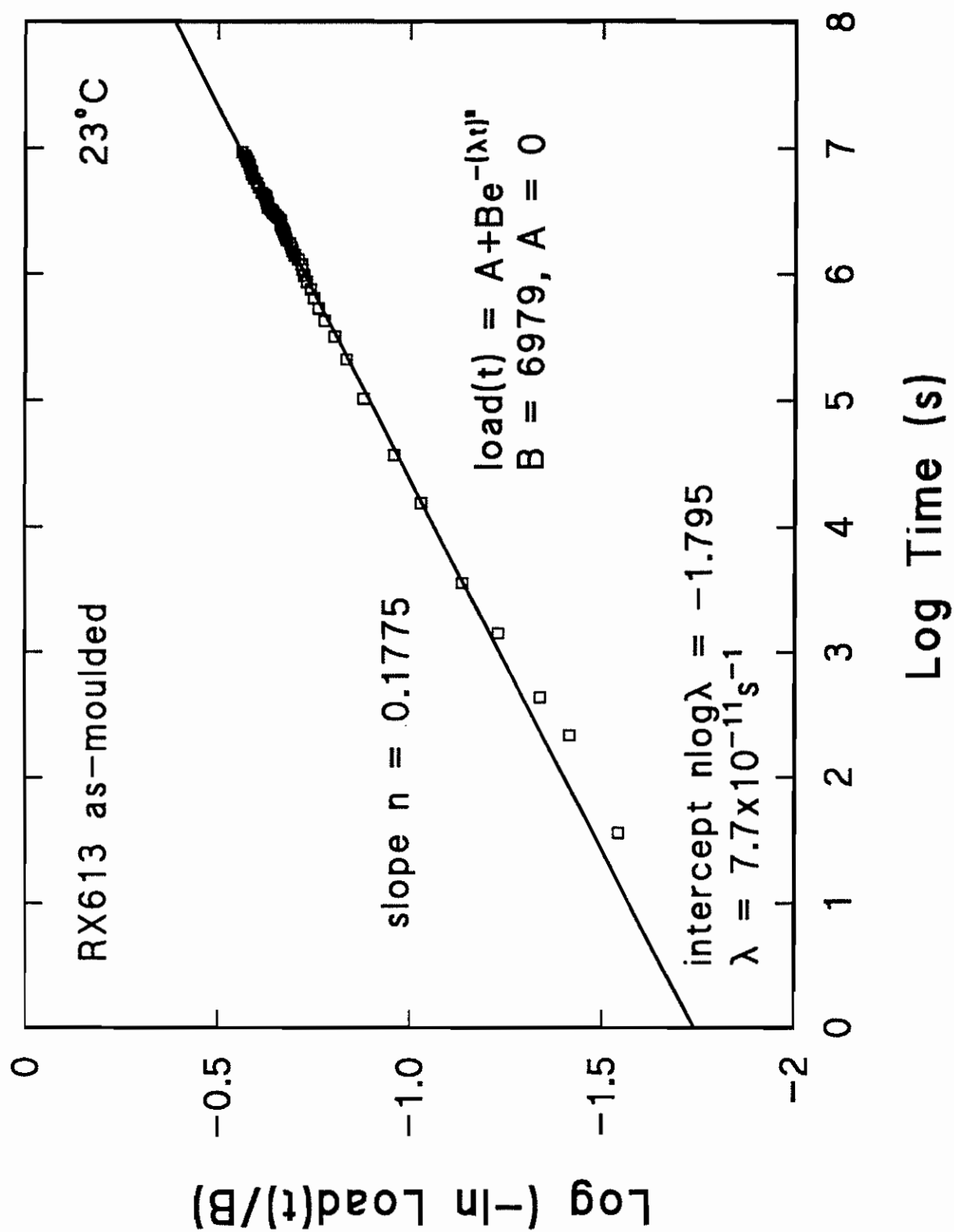


Figure 19

Plot to determine the variables n and λ for as-moulded phenolic stress relaxation at 23°C

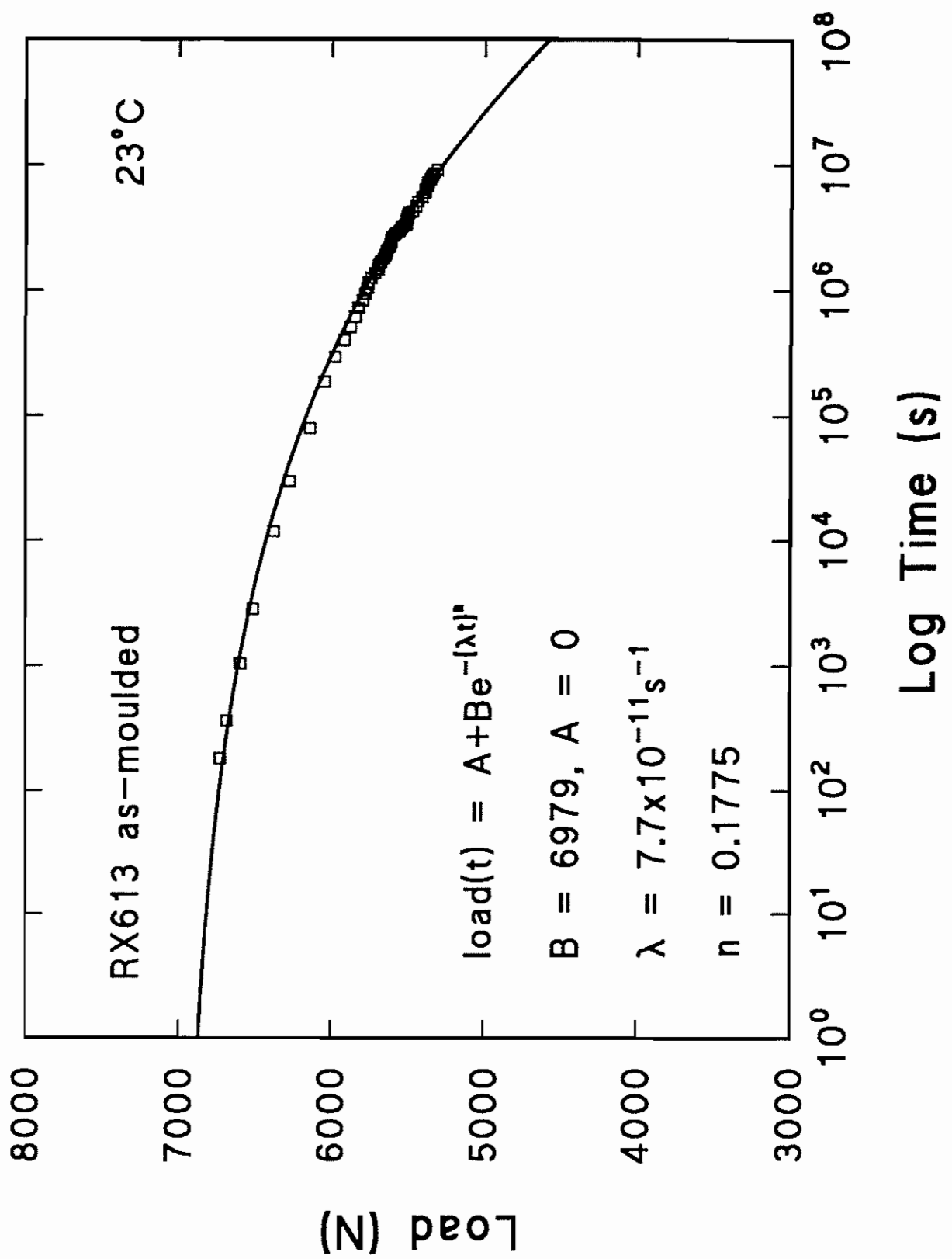


Figure 20 Fit of Kelvin model to experimental results for as-moulded phenolic at 23°C

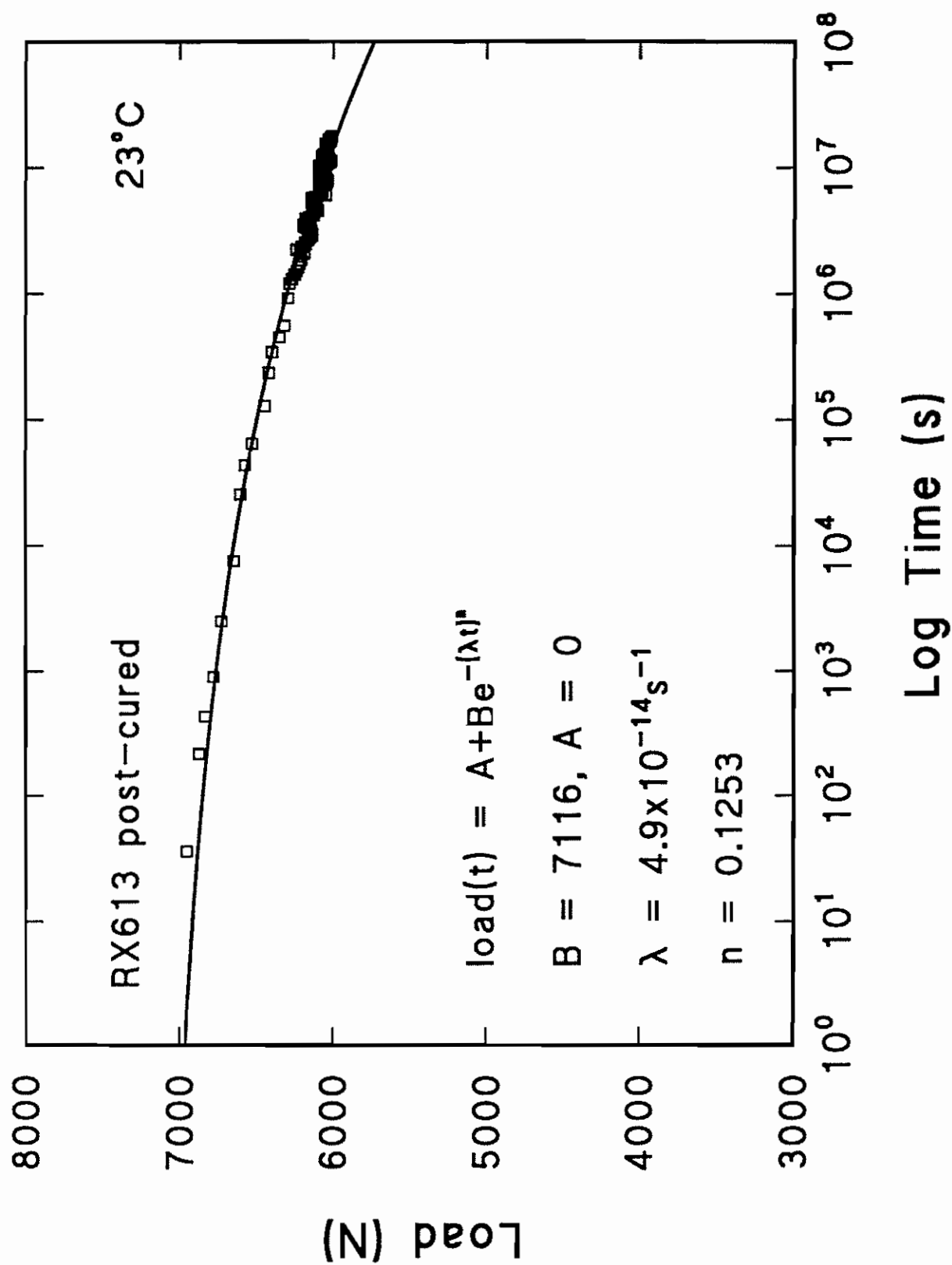


Figure 21 Fit of Kelvin model to experimental results for post-cured phenolic at 23°C

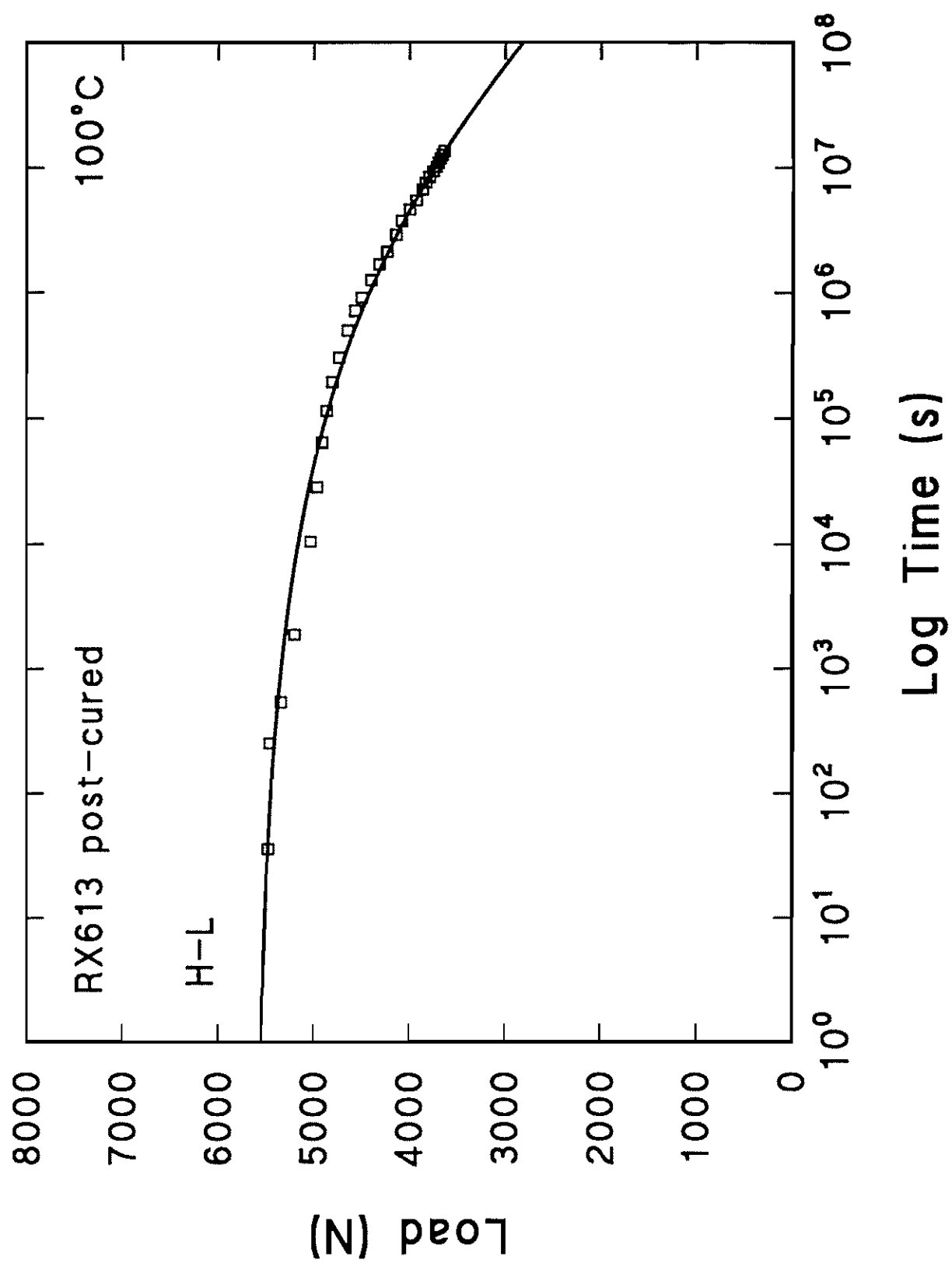


Figure 22 Fit of Kelvin model to experimental results for post-cured phenolic at 100°C

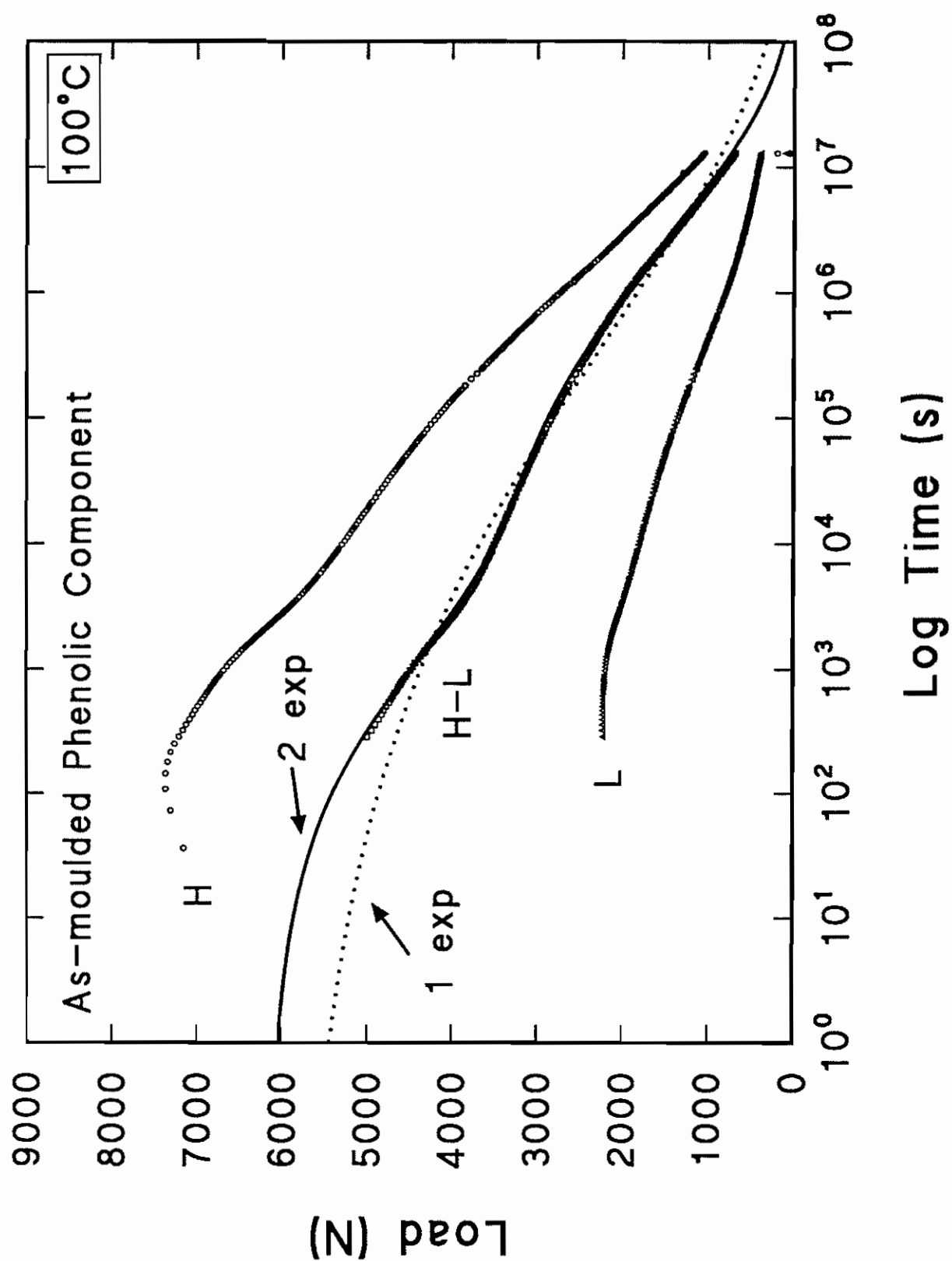


Figure 23 Stress relaxation of as-moulded phenolic components at high and low initial stresses (H and L), corrected for load independent effects (H-L) at 100°C. Fitted by single and double exponential model

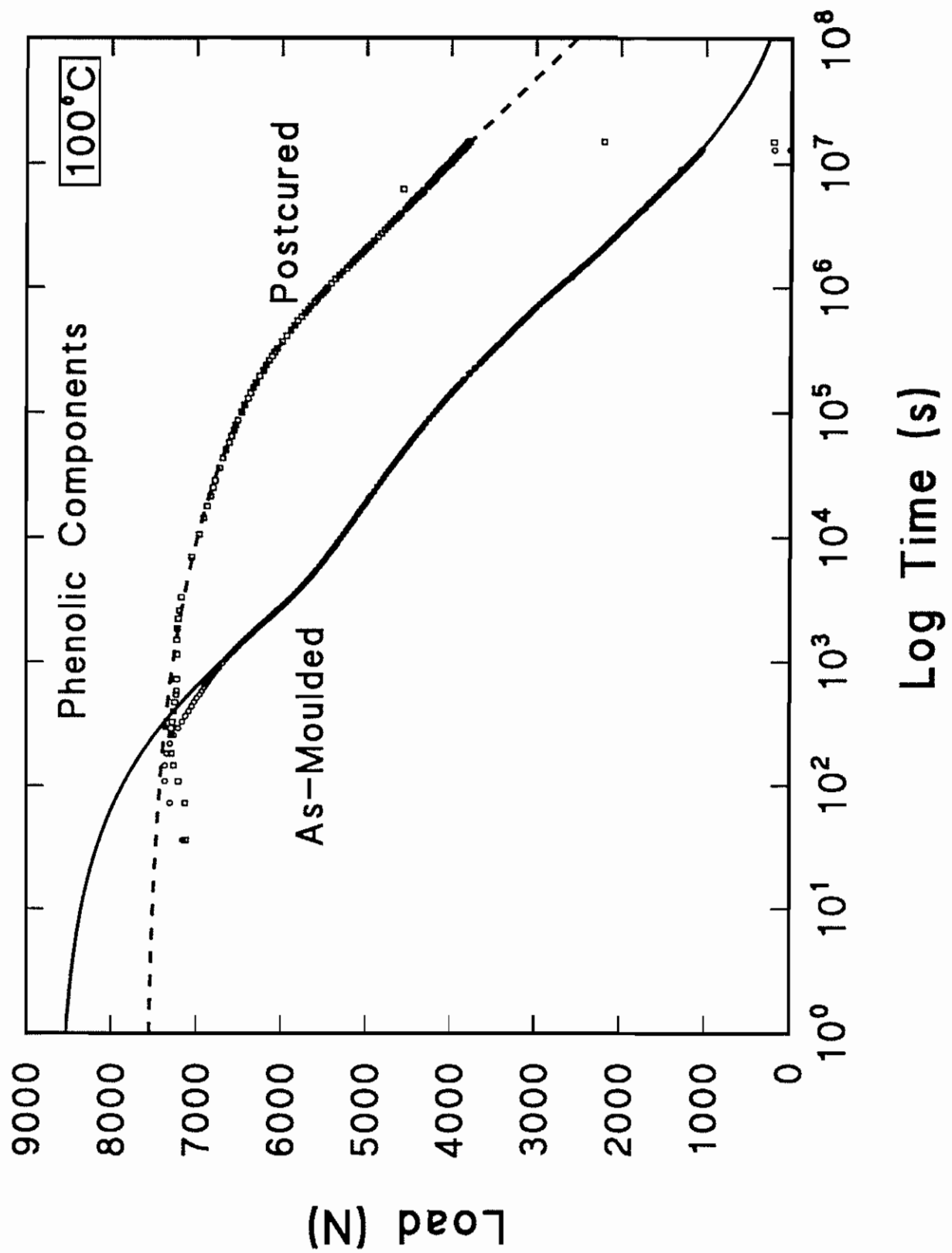


Figure 24 Double exponential model fitted to high initial stress curves of as-moulded and post-cured phenolic at 100°C

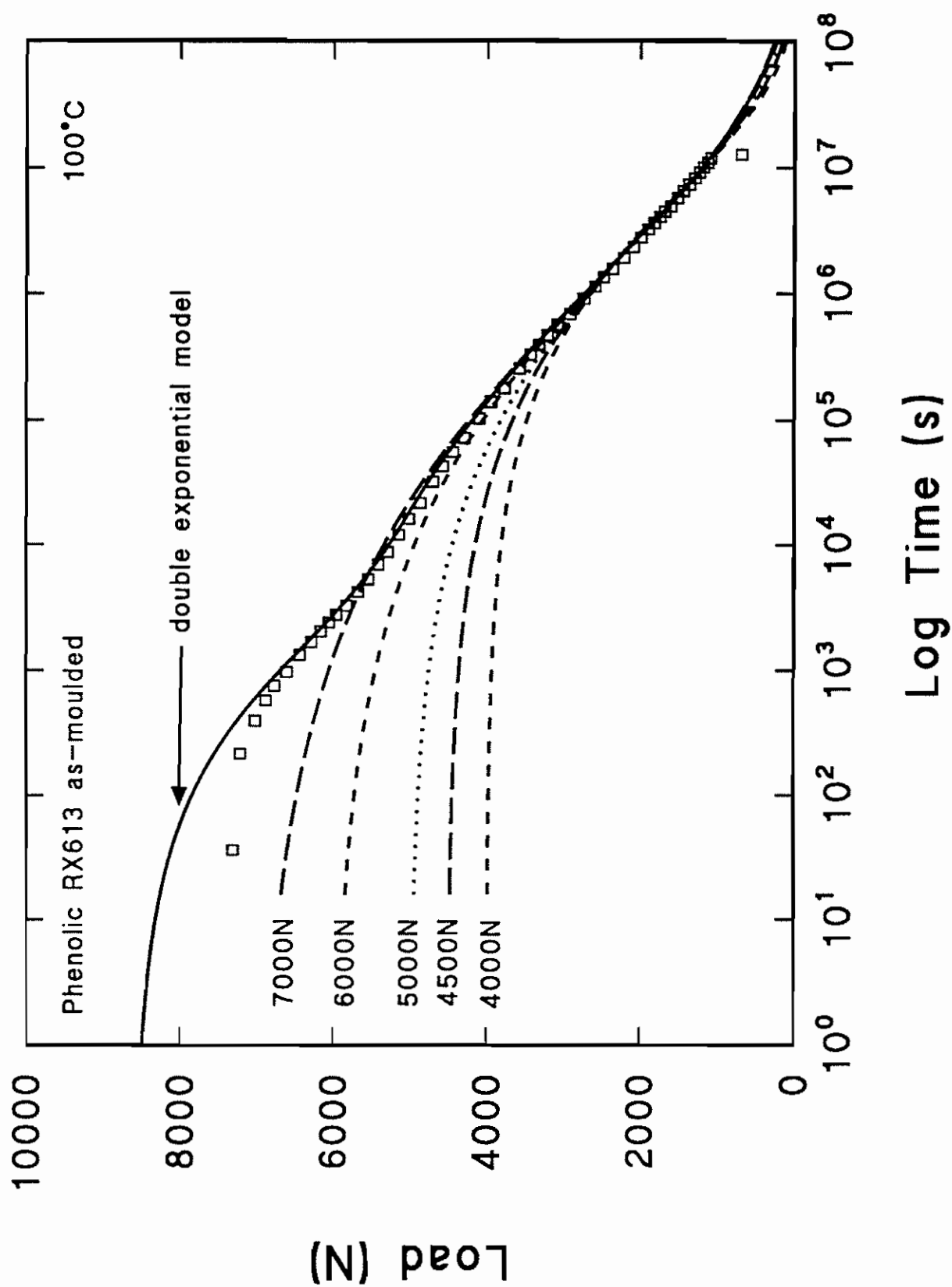


Figure 25

Single exponential model fitted to high time points of the stress relaxation of as-moulded phenolic at 100°C compared to the double exponential model

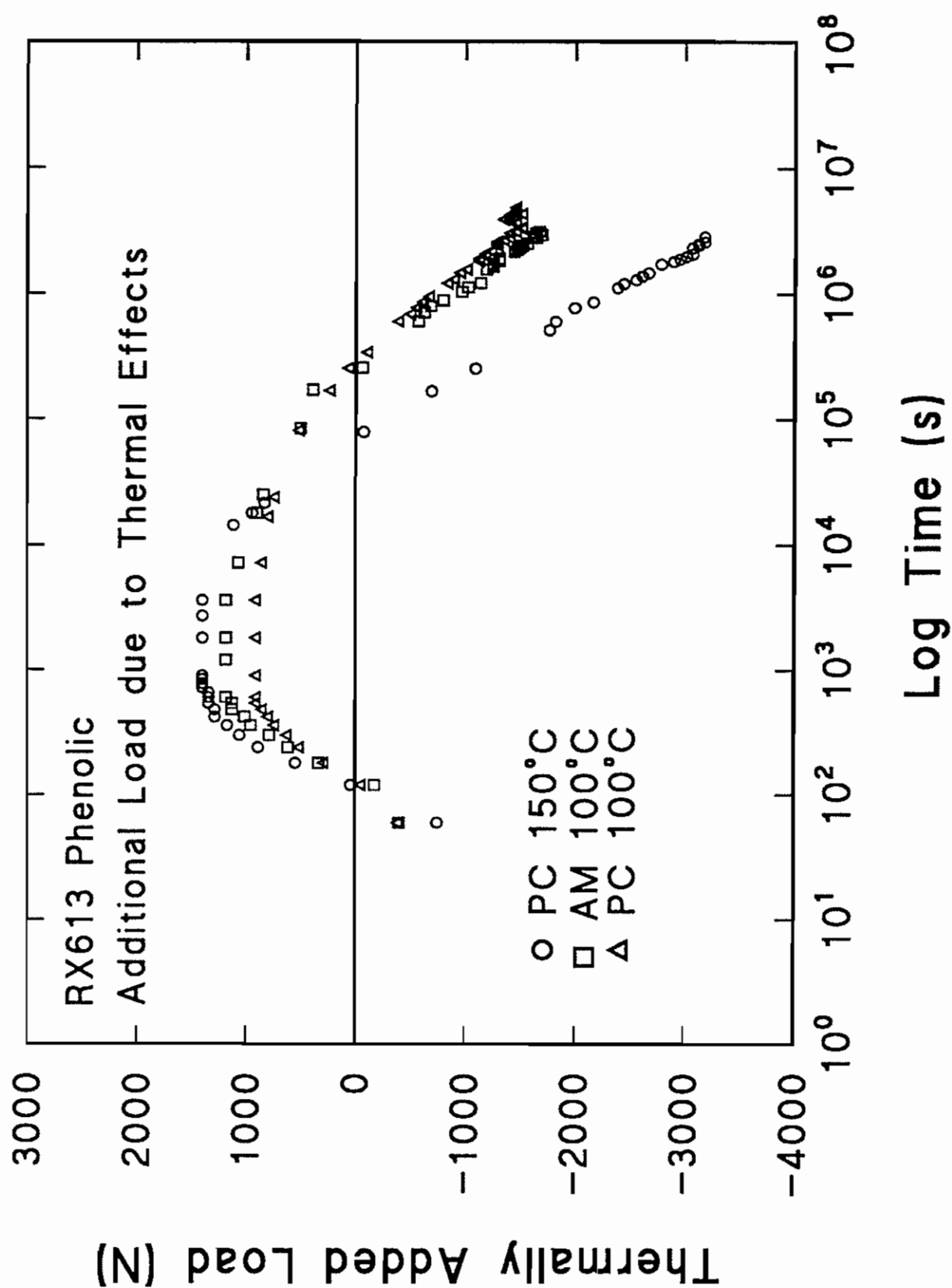


Figure 26

Calculated additional load due to thermal expansion / isothermal shrinkage of phenolic components at elevated temperatures