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Improved relaxation time coverage in ramp-strain histories

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Abstract Standard methods for deriving relaxation data from measurements invariably involve some form of ramp-type deformation history, the initial portion of which is typically not employed for modulus evaluation. In fact, the "ten-times-rule" or a variant thereof is widely used at the expense of short term data acquisition. This paper suggests a simple if (not) obvious method to extend the range of relaxation data that can be acquired from a single test at a single temperature. The method draws on new computational developments for inverting ill-conditioned systems of equations which allows the determination of relaxation parameters nearly routinely and trouble-free. We demonstrate this process for extraction of relaxation characterization from ramp strain histories through (a) numerical evaluation with a virtual test sequence, as well as through (b) data measured in the laboratory. Limitations regarding the time range over which the relaxation modulus can be extracted from laboratory measurements in terms of equipment resolution and stability are discussed. With these constraints in mind it appears feasible to extend the time range by three to four decades towards shorter times when compared with the application of the "ten-times-rule". Similar treatments apply to the acquisition of creep compliance data.

Keywords Viscoelastic behavior · Relaxation modulus · Relaxation time · Creep · Creep compliance

1 Background

Even though the routine determination of viscoelastic mechanical properties has extended over at least half a century, there exists, apparently, lingering uncertainty regarding the efficient determination of relaxation or creep behavior from laboratory measurements. This statement finds its source in the observation that even though the limitations of routine laboratory procedures are well understood, the improvement of that state of affairs has progressed slowly in spite of the general desire for such an advance. In the sequel we discuss

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this problem in terms of uniaxial relaxation measurements under small strains (linear viscoelasticity). The treatment for shear characterization follows an identical recipe, as does the characterization for creep behavior in shear or uniaxial stress states. In principle, the method is trivial from analytical point of view, but the realities of the laboratory deserve careful attention.

The determination of relaxation data follows ideally from the prescription of a step strain history. In the laboratory, such a prescription is, per force, supplanted by a ramp history wherein the strain increases along a (nearly) constant strain rate path until a predetermined strain is reached, say, at time t_0, after which that strain is maintained constant at that value. In that scenario the material incorporates the effects of the strain history well into the time past t_0 and only after a considerable time does the stress response asymptotically approach the relaxation function. This time, when the relaxation behavior is approximated within an error on the order of a percent by the laboratory history, is on the order of five to ten times the rise time t_0 (sometimes referred to as the *ten-times rule*; we adhere to this nomenclature here)[1]. Given the normally operative time scales imposed by laboratory environments—either in terms of the daily routine or by the electrical or mechanical stability of the test equipment—the loss of data during the initial portion of the strain history imposes a significant restriction on the time scale of meaningful data acquisition. Often data records extend over only two to three decades, but it is clearly desirable to extend that time scale to five or more, which is difficult in general engineering environments unless specially designed and constructed equipment is made available. An exemplary demonstration of the latter approach has been given by Plazek (1968), who recorded data over seven decades at a single temperature with especially carefully constructed equipment that guaranteed stability and measurement precision over the whole, long time range. It is the purpose of this publication to suggest means by which a similarly extensive time range can be accessed from a single test with essentially standard equipment, by drawing on now routinely available computer software.

To extend the time scale of data acquisition we have employed in the past in our laboratory a simple method based on successive approximation as documented by Lee and Knauss (2000) though the method was first implemented by Müller in our laboratory in 1969[2]. Since then Flory and McKenna (2004) have added an alternative view to extend the time range for data acquisition, and a recent publication by Sorvari and Malinen (2006) emphasize the continuing interest in this topic. The special difficulty that arises in this connection is that the solution of the resulting integral equation(s) is not stable under reasonable (experiment-induced) perturbations (Clauser and Knauss 1968; Tschoegl and Emri 1992; Emri and Tschoegl 1993a, 1993b, 1994, 1998). Recent developments in computational capabilities have made it relatively easy, however, to significantly extend the range for determining the time of relaxation characterization in ramp-type tests. Specifically, the arrival of the Trust Region Method a nonlinear, Least-Squares Optimization method introduced by Branch (1999) and the commercially available Matlab code, has virtually removed the numerical instabilities associated with the numerical solution of integral equations—or related large matrix equations—which contain exponential kernels, to allow quick and ready interpretation of laboratory test data. This application is a preferred numerical tool for relaxation characterization, as will be demonstrated below.

[1] The source or first observation of this rule is cloudy. Although the senior writer was familiar with that rule on a "proverbial" basis since the late 1950s, the apparently first reference to it in the literature traces to Kelchner and Aklonis (1971); neither Leaderman (1943), Ferry (1948) nor Tobolsky (1960) refer to it explicitly in their books, though the data reported there must have drawn on that rule.

[2] Kelchner and Aklonis (1971) developed an essentially identical recursive process for accessing shorter time scales in ramp strain histories, as was found through a literature search regarding the ten-times rule.

We divide this paper into roughly three sections: The first topic addresses the question of the range of mathematical material representation if data over a given (logarithmic) time range is provided by measurements.

The second deals with the application of the Matlab routine to ideal data extracted from a real material. To this end a numerically available relaxation modulus is used to compute with high precision the response to a strain-ramp history. The result of this latter computation then becomes input into the determination of a relaxation function by way of the Matlab routine which can then be compared to the original relaxation modulus so as to allow an assessment of the precision of the numerical/mathematical procedure.

The third topic addresses the application of the Matlab routine via an evaluation of relaxation behavior deduced directly from relaxation test data under uniaxial compression in a ramp-strain history. This presentation is intended as a realistic demonstration of how a given ramp-strain history translates into a maximally (or minimally) reliable range of mathematical relaxation characterization.

2 Analytical preliminaries

We choose to represent the relaxation behavior in terms of a Prony series in the form

$$E(t) = E_\infty + \sum E_i \exp(-t/\zeta_i) \tag{1}$$

where the range of the index "i" on the relaxation times "ζ_i" will be discussed and determined subsequently. Let the strain history be defined by the expression(s)

$$\varepsilon(t) = \begin{cases} R \cdot t, & t \le t_0, \\ R \cdot t_0, & t \ge t_0 \end{cases} \tag{2}$$

with "R" denoting the strain rate and "t_0" the experimentally prescribed rise time. Upon invoking the constitutive law for uniaxial deformations in the form of the convolution integral with zero initial conditions

$$\sigma(t) = \int_{0^+}^{t} E(t - t') \frac{d\varepsilon(t')}{dt'} \tag{3}$$

the stress history corresponding to (2) is determined routinely as

$$\sigma(t) = \begin{cases} R\left\{E_\infty t + \sum E_i \zeta_i [1 - \exp(-t/\zeta_i)]\right\}, & t \le t_0, \\ R\left\{E_\infty t_0 + \sum E_i \zeta_i [\exp(t_0/\zeta_i - 1)] \exp(-t/\zeta_i)\right\}, & t \ge t_0. \end{cases} \tag{4}$$

It is clear that once the coefficients E_i (and ζ_i) are determined the full relaxation behavior is known. The left hand sides of (4) are measured in the laboratory for given values of R and t_0. The immediate objective is to determine the material parameters E_i and ζ_i with the aid of the nonlinear optimization scheme.

3 Choice of the relaxation times

The trust region method as implemented in Matlab allows for the determination of all parameters, i.e. all E_i and ζ_i according to its own optimization process, once the parameter range "i" is fixed. In this context it is worth remembering that the choice of the parameters is not unique, and that a multiplicity of parameter sets can provide representations of the experimental data that are equally valid within the range of experimental error. Thus, the investigator has some liberty in prescribing some of the parameters. We have a longstanding experience with fitting relaxation data and find that the choice of the relaxation times is not subject to stringent criteria. We have found that allowing for one relaxation time per decade of data is often satisfactory, though two relaxation times per decade (equally spaced along the logarithmic time axis) render some improvement which is, generally, quite adequate.

A question arises as to how many experimental points per decade are needed to provide for a good definition of a relaxation (creep) function. Current digital equipment provides for high sampling rates with accompanying large numbers of data points. In our experience we found that about 10 or 15 points *per decade* are, generally, quite sufficient for data acquisition purposes. However, due to the stability of the test machine and the associated signal/noise ratio, the best choice for the sampling rate is on the order of 100 points/sec. From such a record the requisite number of points per decade can be readily extracted.

Although the trust region method can provide the relaxation times in addition to the strengths of the spectral lines E_i, the investigator is also free to choose values for the ζ_i according to the time scale represented in the data. We have fitted relaxation data in both ways, i.e. by allowing the Matlab code complete freedom in determining all coefficients after specifying only the number of the parameters or, alternatively, fixing the relaxation times and let the program determine the corresponding moduli (spectral line strengths E_i). While the computer time decreased somewhat—though that was and is not considered to be an issue—the resulting representations for the relaxation modulus could not be differentiated within the plotting uncertainty. It is, therefore, most convenient to initially fix the values of the relaxation times of interest at equal intervals along the logarithmic time scale—one or, preferably, two increments per decade—and then determine the associated coefficients E_i computationally.

Figure 1 shows the result of applying the trust region method (via Matlab) to a master relaxation curve for a polyurea ($T_g \approx -50°C$) via these two processes, namely: 1) the computer code was allowed to choose both the optimal relaxation time and the values of the individual moduli (identified as a "totally automated data fit" in the caption), and 2) the

Fig. 1 Comparison of fitting results with (—) trust region method, totally automated data fit (- - -) simplified function (pre-assigned relaxation times) (○) master curve (Knauss, unpublished data)

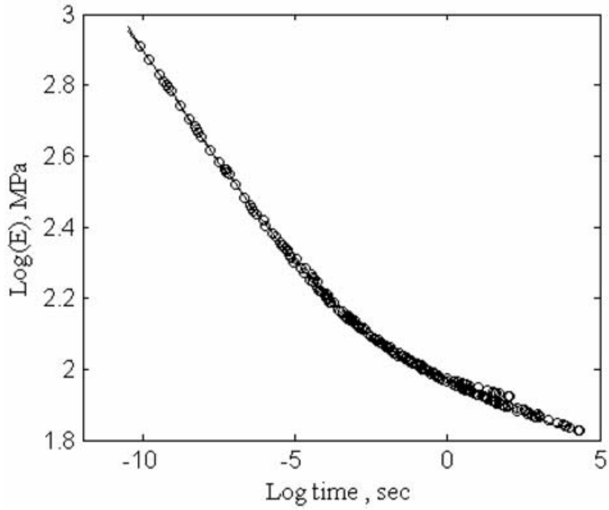


Table 1 Coefficients determined by the full trust region method (Matlab) and by the simplified trust region method

i	Simplified trust region method		Trust region method	
	ζ_i seconds	E_∞ MPa	ζ_i seconds	E_∞ MPa
1	10^{-13}	40	4.481×10^{-12}	90
2	5×10^{-13}	40	9.725×10^{-12}	651.8
3	10^{-12}	40	4.581×10^{-11}	19.81
4	10^{-11}	4.438×10^{-14}	9.415×10^{-11}	207
5	10^{-10}	241.5	4.479×10^{-10}	43.83
6	10^{-9}	191	8.558×10^{-10}	143
7	10^{-8}	126.5	6.978×10^{-9}	136.4
8	10^{-7}	100.4	2.912×10^{-8}	22.45
9	10^{-6}	68.39	1.18×10^{-7}	103.4
10	10^{-5}	46.48	1.44×10^{-6}	66.25
11	10^{-4}	40.5	1.184×10^{-5}	38.46
12	10^{-3}	20.69	10^{-4}	42.14
13	10^{-2}	17.48	1.266×10^{-3}	20.77
14	10^{-1}	10.88	0.01071	16.95
15	1	9.009	0.1657	13.45
16	10	5.388	3.278	10.01
17	10^2	5.975	91.77	6.876
18	10^3	10.4	929.3	10.26
		$E_\infty = 69$		$E_\infty = 69.13$

relaxation times were predetermined at half-decade intervals such that the code computed only the values of the moduli (identified in the caption by "simplified function"). From a practical point of view a distinction does not exist: the two methods produce no discernable difference. The corresponding parameters are listed in Table 1. While the parameters in this table appear to differ measurably, their global results as displayed in Fig. 1 are, essentially, identical.

We next discuss the determination of the range of relaxation times in relation to the range of the experimental data. Two issues are of interest here: One concerns the question of the "influence range" of a single relaxation time as represented by a Maxwell element (say the ith element in a Prony series), the other establishes how far experimental data may be extrapolated in a mathematical representation beyond either side of the experimental data range. With respect to the first question we present in Fig. 2 the function

$$Mod = \sum_{i=1}^{i=7} 10^{5-i/2} \exp[t/10^{i-2}] \tag{5}$$

as the "upper envelope" along with the seven individual summands; From this it is apparent that each term covers a time scale of about a decade, and that the influence of a particular term decreases rapidly as that range is exceeded. Conversely, relaxation times which are inside a range of available data or are of interest in a problem solution will not depend significantly on the values of the moduli far outside of that range, though inclusion of relaxation time outside of the data range may yield improved representations, as demonstrated later.

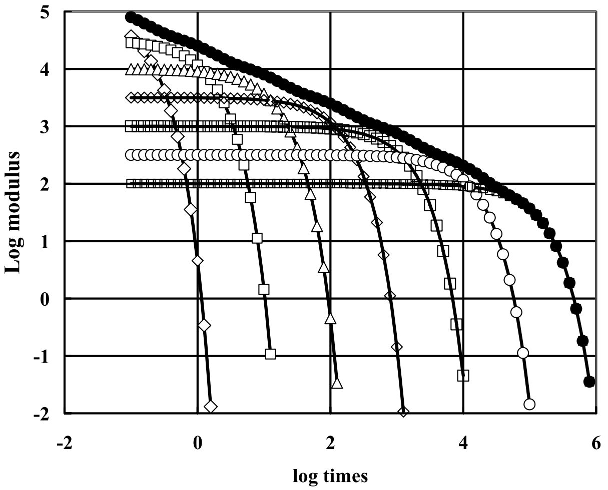


Fig. 2 Construction of a relaxation function via an exponential series, showing contributions of individual summands (one relaxation time per decade)

For example, if a problem involves response times in the range of 1 to 1000 seconds, then the moduli corresponding to relaxation times shorter than 0.1 sec and longer than 10^4 sec will not matter significantly. For this example time range the time dependence will be represented accurately if the moduli for

$$1 \text{ sec} < \zeta_i < 10^3 \text{ sec} \tag{6}$$

are prescribed. On the other hand, the smoothness of relaxation functions in general (low curvature on a log–log plot) allows one to safely extend the range (6) on the short and long sides by one half to one decade so that determining the moduli in the range

$$0.1 < \zeta_i > 10^4 \text{ sec} \tag{7}$$

from data in the range (6) will represent the relaxation modulus for the range of interest very well.

It is now clear that the range of the relaxation times for the fitting process is about a decade or two larger than the complete range of the available data with values for the ζ_i's fixed at, say, half a decade before and after the nearest end points of the data range. These are typically determined at the short-time end by the resolution of the timing equipment or by the dynamic response of the test frame, and, at the long-time end, by the operator's patience or the electrical and environmental stability of the test set-up. This aspect will be discussed further in Sect. 6.

4 Notation

Because data fitting will be governed by both the time range of the data and by the choice of the range for the relaxation times, as well as by their increments, it is useful to adopt here a short-hand notation. To this end we employ the convention that $[x_1 : x_2 \mid y_1 : y_2 \mid z]$ identifies x_1 and x_2 as establishing the range of the experimental data (base-10 logarithms); similarly, y_1 and y_2 define the range of the chosen relaxation times (base-10 logarithms), and $z = 1$ or $z = 1/2$ signifies whether the log-time increments of "y" are whole or half decades, respectively.

5 Example computations

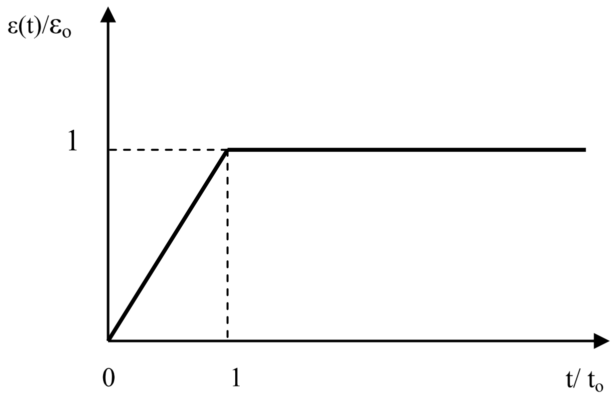
To illustrate the recipe for determining a mathematical representation for the relaxation modulus and to demonstrate the precision that is available mathematically—if not in the laboratory—we first use the data represented in Table 1 under “Simplified trust region method” to compute the ideal response for a ramp strain history. We next use this ideal, computed strain history and treat it as experimental data, from which the relaxation modulus may then be determined. The degree to which this latter determination tracks the original modulus specification is then a measure of how reliable the proposed method can be analytically.

For now, we use the strain history in Fig. 3 and use initially the values $t_0 = 1$ sec and $\varepsilon_0 = 0.04$ for computations. For orientation purposes it is instructive at this point to visualize the limitations imposed by the ten-times-rule on the range of recoverable relaxation data. Figure 4 shows the first 16 seconds of the responses to step and ramp strain, and Fig. 5 the difference between the two responses. The latter shows more clearly than Fig. 4 that a data exclusion time of $10t_0$ is very conservative, leading to a potential error of only 0.2%, while exclusion of the data up to $5t_0$ ($= 7/10$ of a decade) places the initial error at 0.5%. But even this observation still leaves about two to three decades of shorter times unaccounted for. This understanding cannot be arbitrarily generalized for all measurements since this result depends to some degree on the rapidity with which the relaxation progresses in any specific material.

5.1 Evaluation of data from the ramp portion

We next use the stress response in Figs. 4 and 6 (though not discernable there) to “evaluate” the relaxation modulus in the time ranges $t < t_0$. Choose first the time range[3] 0.01 sec $\leq t \geq 1$ sec for the constant rate portion to compute the relaxation modulus for that range. As mentioned in Sect. 3, it is not important for this purpose to have available a very large number of data points. The determination of the relaxation function in this range then follows the first part of (4). Figure 7 shows the result for different choices of relaxation time ranges, the relaxation times being spaced logarithmically at either one or half decade intervals as indicated in the figure.

Fig. 3 Ramp strain history



[3]The choice of the lower limit will be discussed in more detail in connection with the laboratory data.

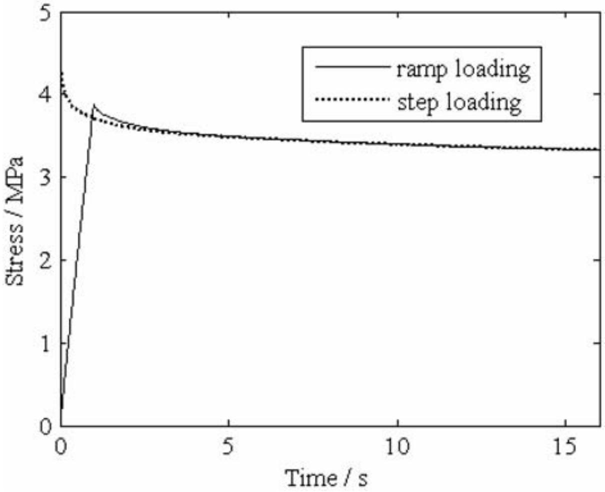


Fig. 4 Responses to step and ramp loadings with $\varepsilon_0 = 0.04$; Initial part only

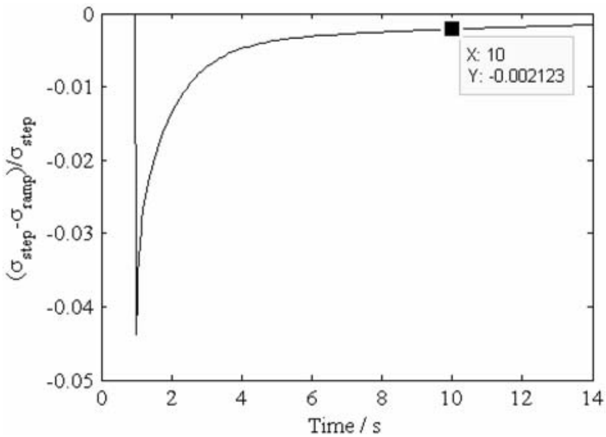


Fig. 5 Fractional stress difference between ramp and step loadings corresponding to Fig. 4

We are well aware at this juncture that this somewhat idealized situation may not yield the same results as laboratory test data might. In fact, our experience tells us that laboratory data is not as free of noise as the presently generated data set. Rather than deal with that issue at this point, we defer its discussion until the laboratory data prompts it directly.

To examine the effect of how the choice for the range of model parameters affects the data acquisition we expand, in Fig. 8, for illustration purposes the long-time portion of Fig. 7. Similar considerations prevail for the lower end of the time range exhibited in Fig. 7. For all representations shown the data range encompasses the two decades of time $0.01 < t < 1$ sec. We note first that terminating the model representation at $\log t = 0$ produces a marked deviation from the master curve beyond that time frame. On the other hand, including relaxation times as high as $\log \zeta = 1$ renders considerably improved adherence to the master curve data. Amongst the latter cases one discerns a slight improvement by choosing the whole range to cover the interval $0.001 < t < 10$, which we attribute simply to the fact that a larger number of parameters is available for fitting the data though the difference is hardly meaningful in terms of the normally expected data scatter derived from direct laboratory measurements.

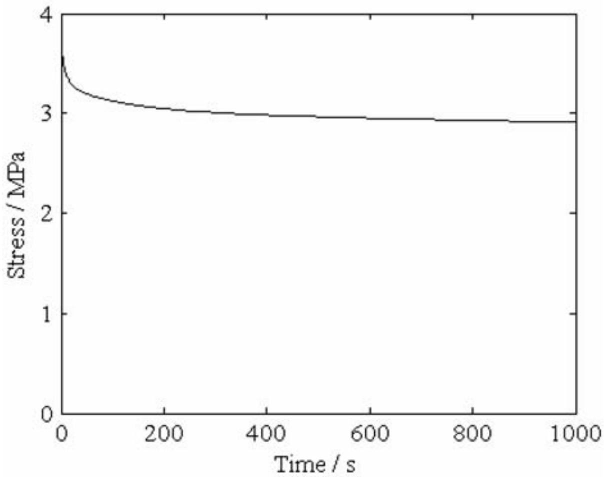


Fig. 6 Complete stress history for the ramp strain in Fig. 3 as the basis for the simulation computations

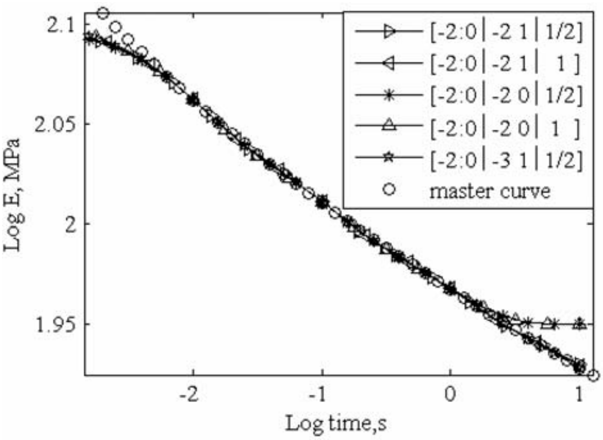


Fig. 7 Relaxation data at 0°C and a strain rate of 0.04/sec derived from two decades of data in the ramp portion of the strain history, using different model representations

It is noteworthy that in Figs. 7 and 8 the representations employing one or two relaxation times per decade over the range 0.01 sec $< t <$ 10 sec render the same results within plotting precision, and that the representation of 0.001 sec $< t <$ 10 sec improves slightly on that result even in the long-time (see Fig. 8). However, a distinct difference appears if one uses the range 0.01 sec $< t <$ 1 sec depending on whether one uses one relaxation time per decade or two; relaxation times spaced at half a decade render results nearly indistinguishable from the previous results for the wider range choice for the relaxation times.

5.2 Evaluation of data from the constant strain portion

We turn next to the evaluation of the post-ramp portion of the stress history, which by Fig. 6, extends to 1000 seconds. We concentrate on fitting the relaxation modulus to the "data"

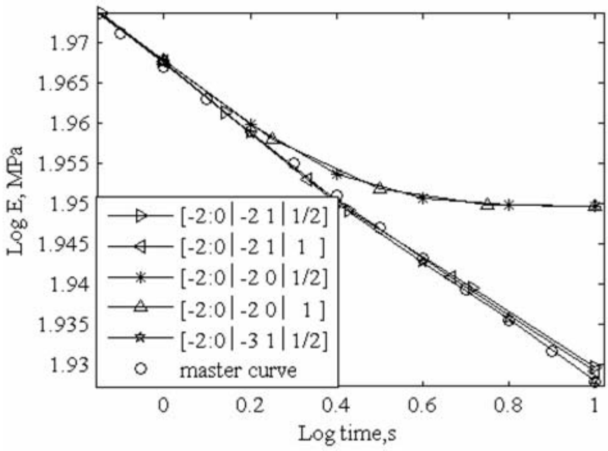


Fig. 8 Enlarged segment of Fig. 7

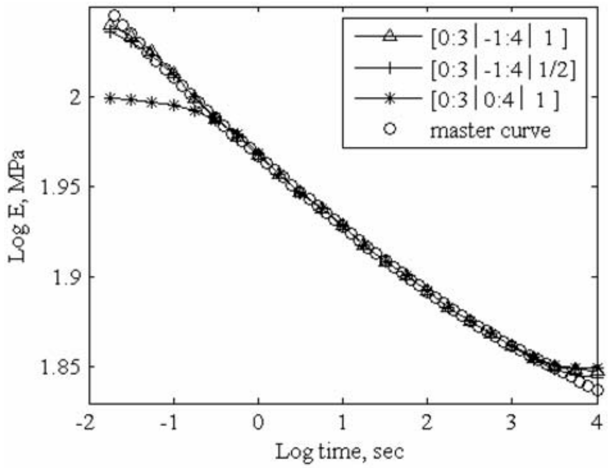


Fig. 9 Relaxation modulus determined from constant strain portion using different ranges for the relaxation times

covering the range 1 sec $< t <$ 1000 sec[4], but select various sets of model parameters to evaluate the Matlab code via the "virtual data" for this case. The results are shown in Fig. 9 in comparison with the master curve. Similar to the comparison for the ramp portion of the strain history, we experience again that expanding the range of relaxation times beyond the range of the "physical data" stretches the time over which the modulus can be fitted. By comparison, the choice of one or two relaxation times per decade brings relatively small changes, though a more detailed analysis than the cursory inspection of Fig. 9 shows that a half-decade spacing of the relaxation times improves the fit by a small amount.

Figure 10 summarizes the results of Sects. 5.1 and 5.2 and we note that even for an exclusion of $1/10$ of the ramp time at start-up ($t > 0.1t_0$ used) the logarithmic time range for the modulus has been approximately doubled when compared to that available from a straight application of the ten-times rule. The doubling is based on the fact that only roughly

[4]For ease of reference recall that and 8 hour day corresponds to 4.45 second-decades so that exclusion of the first five rise times (five seconds) results in a data record over 3.75 decades and the exclusion of the first ten rise times (ten seconds) of only ~3.5 decades.

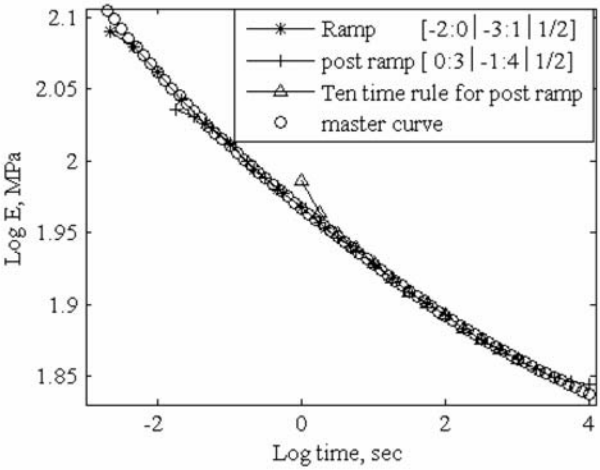


Fig. 10 Summary of the modulus extraction by the proposed method as compared to the ten-times-rule

three decades of data were available. Should the time scale for the constant strain portion of the history be extended to, say, four and a half decades (~8 hours) the gain in short-time extraction is still on the order of three decades so that a total modulus history of about 7 decades could be established from a single test.

6 Application to laboratory data

Having explored the proposed data reduction process in term of a predetermined material description and demonstrated its satisfactory utility, we turn now to applying the same approach to data determined physically in the laboratory. The data reduction process applies equally well to tension or compression data: the following data were acquired in uniaxial compression on specimens measuring $14 \times 14\ \text{mm}^2$ in cross section and 30 mm in length. Tests (MTS test system) invariably showed data irregularities in the start-up phase, though a ramp history with a 10 sec ramp time instead of one of 1 sec duration improved the recovery of the relaxation modulus. Two paradigms were employed to reduce errors and noise effects. Because some of the force transients were traced to the *gradual* compression and seating between specimen and testing machine platens, both the specimen and the platens were lapped and polished—the specimen in a special holding fixture—to achieve surfaces that were parallel to within 0.01 mm per cm (0.00011 rad = 0.064 degrees) across the specimen diameter. Moreover, to minimize the last vestiges of such undesirable errors the specimens were pre-compressed through a displacement of 0.02 to 0.04 mm, with a subsequent rest period of at least one hour to allow sufficient time for the associated relaxation to a steady stress before imposing the ramp history (as the dominant, additive load history). To assure acceptable data consistency commensurate with the small displacements corresponding to the small strains, the oil in the test frame was allowed to achieve a constant temperature of at least 40°C during two hours of warm-up and the temperature control unit was allowed to reach its pre-set temperature during at least a one-hour start-up period followed by a one hour thermal equilibration of the specimen. To eliminate any potential thermal preloading the relative displacement between the load-free end of the test frame were monitored with an LVDT, so that preloading would occur only after that relative displacement was found to be non-detectable. During testing, the temperature varied within a range of no more than ±0.5°C.

6.1 Ramp-portion of the deformation history

We begin with the initial ramp portion of the deformation and Figs. 11 and 12 show the relevant experimental records. The material is nominally the same as that represented in Fig. 1, though the specimen employed in the current study came from a different production batch.

Figure 11 portrays the deformation history with the insert illustrating the detail at small times. Because of the start-up transient a least-squares straight line fit determined the ef-

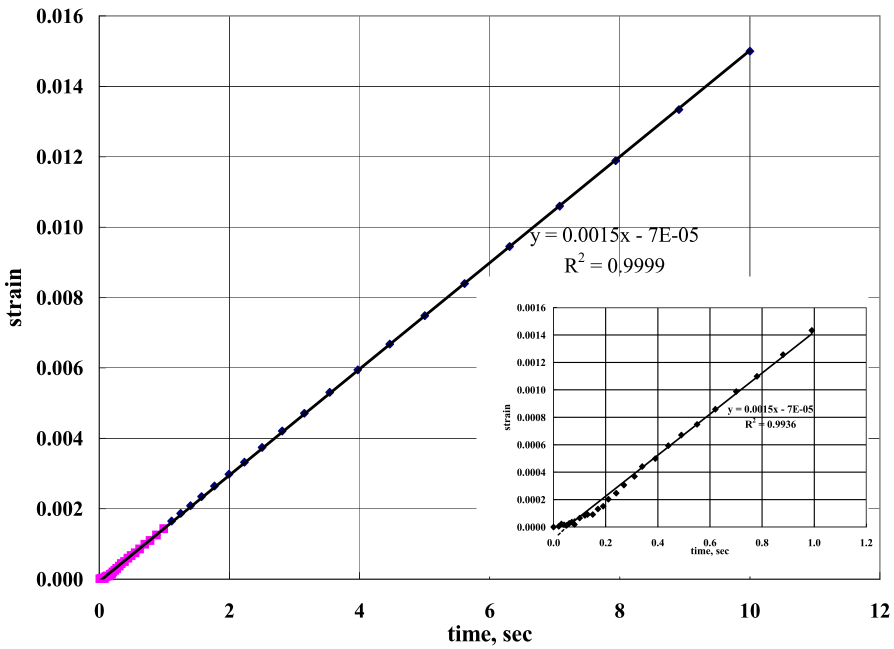
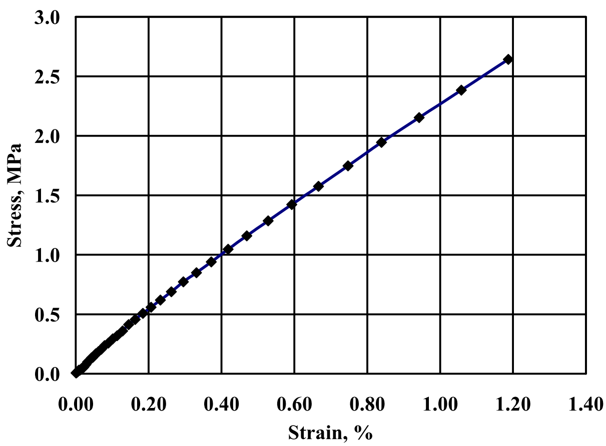


Fig. 11 Strain history of the ramp portion. Inset illustrates lack of precision during the initial, transient phase

Fig. 12 Stress–strain history derived from a ramp strain portion ($t_0 = 10$ sec) recorded at $-34°C$



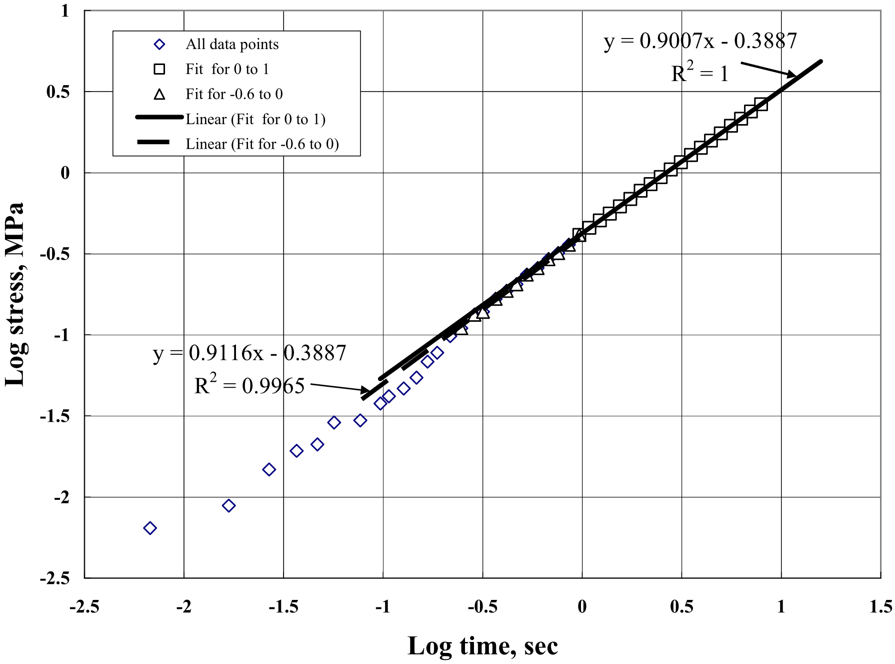


Fig. 13 Log–log plot of the stress history during the ramp

fective zero-time offset (0.0466 sec) by extrapolation to the (zero) displacement axis[5]; all time records were then adjusted by the resulting time-offset. We found it sufficient to perform this displacement-related adjustment in time without having to worry separately about the corresponding stress history, though the same time-adjustment was applied to the stress record.

Figure 12 illustrates the stress history as a function of strain ($\dot{\varepsilon} = 0.0015/\text{sec}$ as determined from the least-square fit in Fig. 11). The small curvature of the trace in Fig. 12 is indicative of the relaxation/creep behavior. While it is not very pronounced in this plot it does provide the relevant material characterization for the time frame of the ramp deformation. Though the trace appears smooth on the whole, the insert in Fig. 11 as well as the logarithmic plot in Fig. 13 demonstrate that considerable error exists at small strains, which errors arise, in part, from the displacement control of the test frame—dynamics of the test machine- and in part from the resolution capability of the load cell. It is clear that the range of obvious data scatter cannot provide meaningful data for modulus evaluation. Bearing this data limitation in mind we address next the extraction of relaxation behavior from this response portion.

Notwithstanding the discussion in Sect. 5 associated with (4), there are several ways by which the relaxation behavior can be deduced from the stress response during the ramp history. The most direct way is readily derived from (3) by noting that for $\varepsilon = Rt$ that relation leads to

$$E(t) = \frac{\partial \sigma}{\partial \varepsilon} = \frac{1}{R}\frac{\partial \sigma}{\partial t}. \tag{8}$$

[5]Displacement of the preloading and at the beginning of the test sequence.

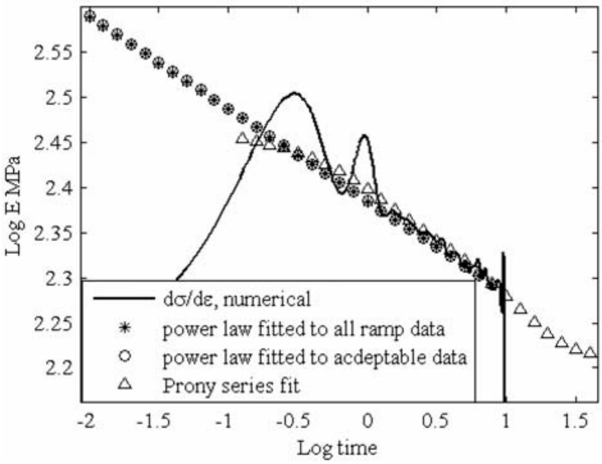


Fig. 14 Relaxation data derived from the ramp portion by indicated means

Consequently, the relaxation modulus may be obtained by numerical differentiation, though that process usually does not apply well to experimental data without further processing. Such processing may involve smoothing of the data and invoking the MATLAB code to perform the differentiation, or, vice versa, performing the differentiation followed by data smoothing. Alternatively, one may fit the data so obtained with a power law representation to arrive at an evaluation. As a further recourse one may assume a power-law representation, approximately valid over a limited number of decades in the form

$$E(t) = A\left(\frac{t}{\zeta}\right)^{-\alpha} \tag{9}$$

from which follows the stress

$$\sigma(t) = \frac{A\zeta}{1-\alpha}\left(\frac{t}{\zeta}\right)^{1-\alpha} = Ct^{\beta}. \tag{10}$$

Plotted on a log–log basis, this latter equation renders a straight line, the slope β and coefficient C of which may be readily determined. These different processes are demonstrated in Fig. 14. It is apparent, as already illustrated less dramatically in Fig. 13, that the data before $t = 1$ sec $= t_0/10$ region for 1 sec $< t <$ 10 sec appears to be of questionable value.

6.2 Post-ramp relaxation

If one decomposes, for didactic purposes, the ramp history of Fig. 3 into the two linear functions illustrated in Fig. 15 it is in principle clear that the data, immediately following achievement of the ramp time t_0, contains the same short term relaxation response as the initial ramp; and one might argue, therefore, that the post-ramp data contains as much short-term information as does the initial ramp data. This observation is only partially correct since one notes that this start-up data associated with the second (subtractive) history is superposed on the relaxation behavior associated with the previous ramp-history. Consequently, it falls, to some degree, into the data noise of the larger relaxation signal, regardless

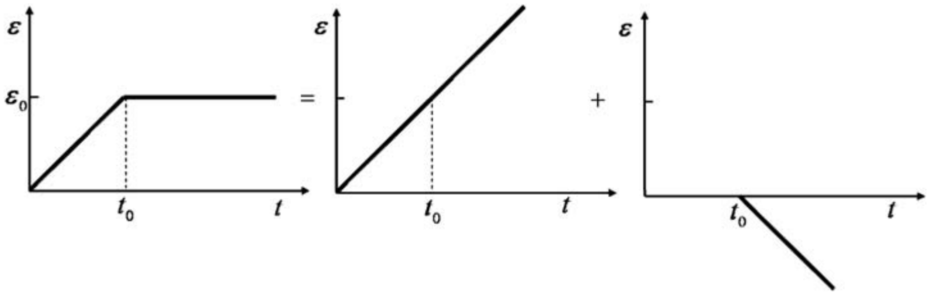
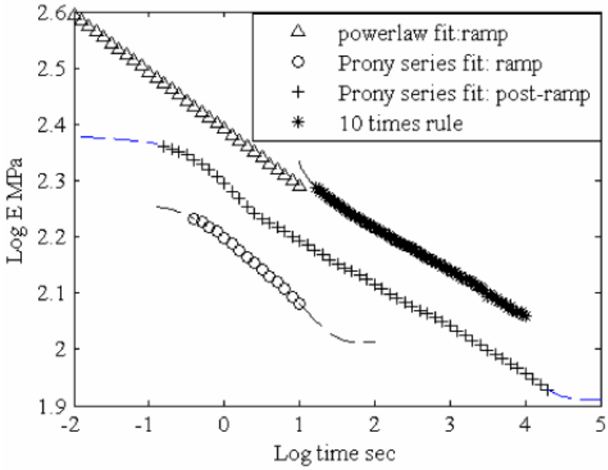


Fig. 15 Superposition of linear functions to generate a ramp

Fig. 16 Modulus fit for the post-ramp portion (*central symbols*) along with segments of the ramp data fits. The various segments have been displaced a tenth of a decade up and down, respectively. The *dashed portions of the curves* are judged not to be physically meaningful



of the physical fact that the test frame cannot provide the idealized deformation history because of some, always present, inertial response of the test machinery. It was for primarily this reason that we chose to work with a ten-second ramp-time rather than one of one-second duration, so as to reduce the inertial consequences of a rapidly changing deformation rate.

Figure 16 shows the exponential fit (1) for the second of (4), and identifies the range where the computed relaxation modulus is acceptably free of experimental exigencies. This data is supplemented, for complete comparison purposes, by some of the data derived from the ramp-portion as discussed in Sect. 6.1. The power-law parameters are identified as $C = 0.4102$ MPa/sec$^{\beta}$, $\beta = 0.8977$.

Table 2 lists the model parameters for this fit.

The process of deducing relaxation behavior from this two-portion fitting method has been repeated for data obtained at several temperatures as shown in Fig. 17 according to the tests/temperatures listed in the inset of that figure. The construction of the master curve from these thermal segments is shown in Fig. 18. Two sets of data were obtained, each one for a specific material specimen. The corresponding (average) shift factor is shown in Fig. 19. For orientation purposes WLF shift factors are included for glass transition temperatures of -45 and $-59°$C.

Table 2a Relaxation parameters of polyurea at −34°C for ramp portion

i	λ_I seconds	E_i MPa
1	0.01	0.0525
2	0.1	0.0036
3	1	53.9533
4	10	74.5333
		163.0667

Table 2b Relaxation parameters of polyurea at −34°C for constant strainportion

i	λ_I seconds	E_i MPa
1	1	78.3033
2	10	36.5947
3	10^2	30.2619
4	10^3	22.8388
5	10^4	30.4048
6	10^5	0.0001
		$E_\infty = 102.6$ MPa Not physically meaningful

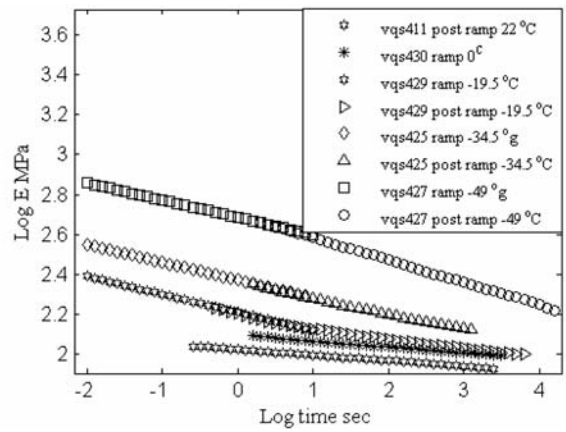


Fig. 17 Relaxation behaviors at different temperatures ("vsq" refers to different test runs)

7 Concluding remarks

We have demonstrated that, in contrast to the traditional way of obtaining relaxation data from ramp-type deformation tests, extended time coverage may be deduced from such tests in a fairly routine manner. This process has been greatly aided by the advent of new numerical-analytical tools incorporated into a commercially available code which has wide access in the scientific community. Even without the exploitation of the ramp portion of the deformation history, the evaluation of the complete constant-strain portion allows an extension of the time frame by about two decades. The addition of the ramp portion extends this range further by an amount that depends on the measurement precision and experimental

Fig. 18 Master curves for two specimens generated from the same material batch

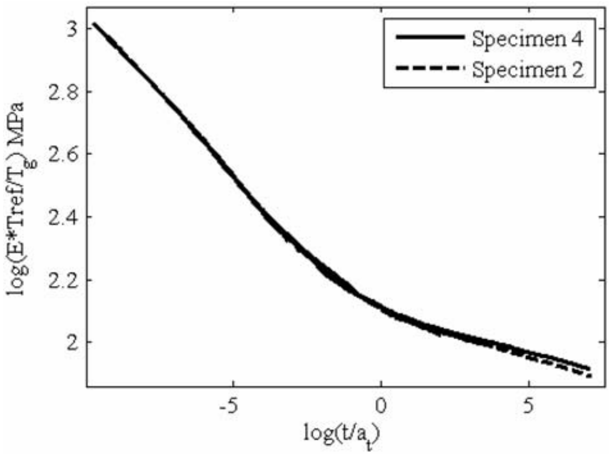
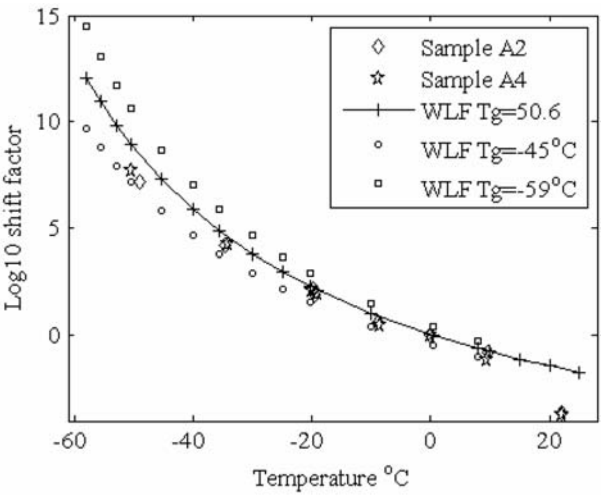


Fig. 19 Shift factor as a function of temperature



care and, to some extent, by the subjective evaluation tempered by the curvature of the relaxation function (log–log plot): A fairly precise evaluation may be achieved by a power-law fit which is bounded by the data noise during the start-up transients. Extrapolation to shorter times must be tempered by the curvature of the relaxation function in the time range considered. We have been successful in deriving relaxation over six decades-plus of time from a single relaxation test, where the "normal" method of employing the ten (five)-times rule would have yielded fewer than three decades.

The range of recoverable relaxation behavior depends clearly on the precision of the measurements. Lack of care in defining the deformation history, such as by allowing too much of an influence of transients—start-up or change from constant rate to constant strain—will impair the reliability and jeopardize the data extraction process. An analysis of the measured stress and strain histories is essential to establish the range of the measured input and output in the tests. If "inadequate" care is exercised in this regard it may be just as efficient to rely on a greater number of temperature-controlled tests and the ten (five)-times rule than to increase the time range at the expense of fewer test temperatures to generate data for a master curve[6]. This observation simply reflects the well known adage “You don’t get something for nothing”.

Beyond this observation we remark that the method presented here clearly relies on computational evaluation of test data beyond the dimensional parameters. To the extent that such a data reduction can and will introduce (numerics-based) errors the final result will incorporate these. If such errors are not acceptable there seems to be no alternative but to employ the data acquisition via the classical ten-times rule.

Acknowledgements This study is part of an investigation into the application of standard viscoelastic master curves derived from quasistatic, low-rate laboratory measurements to dynamic (wave propagation) problems conducted under the auspices of the Office of Naval Research through Grants N00014-05-1-0548 and N00014-05-1-0624. The authors wish to expressly thank Dr. Roshdy S. Barsoum for his support and guidance during this endeavor.

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[6]To achieve definitive overlap of various temperature segments we would not recommend collecting data over less than three decades and to allow at lest one and a half, or better, two decades of overlap in the shifting process.