

Prestress and experimental tests on fractional viscoelastic materials

Mario Di Paola¹, Vincenzo Fiore¹, Francesco P. Pinnola¹, Antonino Valenza¹

¹*Dipartimento di Ingegneria Civile, Ambientale, Aerospaziale e dei Materiali (DICAM),*

Università degli Studi di Palermo, Italy

E-mail: mario.dipaola@unipa.it, vincenzo.fiore@unipa.it, francesco.pinnola@unipa.it, antonino.valenza@unipa.it

Keywords: Relaxation test, fractional calculus, viscoelasticity.

Creep and/or Relaxation tests on viscoelastic materials show a power-law trend. Based upon Boltzmann superposition principle the constitutive law with a power-law kernel is ruled by the Caputo's fractional derivative. Fractional constitutive law posses a long memory and then the parameters obtained by best fitting procedures on experimental data are strongly influenced by the prestress on the specimen. As in fact during the relaxation test the imposed history of deformation is not instantaneously applied, since a unit step function may not be realized by the test machine.

Aim of this paper, it is shown that, the experimental procedure, and in particular the initial ramp to reach the constant stress (or strain) strongly influences the best fitting procedure and the coefficients of the power-law.

1 INTRODUCTION

Creep and relaxation tests performed on various materials like polymers, rubbers, biological tissues, wood and so on are will fitted by power-laws with real exponent $\beta : 0 \leq \beta \leq 1$ (1; 2; 3; 4). Based on this observation in the second part of the last century it has been observed that, by using Boltzmann superposition principle, the constitutive law are ruled by fractional operators (5; 6) rather than operators with exponential kernels. With this result in mind a lot of both theoretical (7; 8; 9; 10; 11; 12) and experimental test have been performed (1; 2) confirming the fractional constitutive law to describe viscoelastic behavior.

Recently Di Paola et al. (13; 14; 15) have been shown that the real behavior of viscoelastic materials may be classified as Elasto-Viscous (EV) ($0 \leq \beta \leq 1/2$) those for which the elastic phase prevails over the viscous one. While for $1/2 \leq \beta \leq 1$ the material has been termed as Visco-Elastic (VE) because the viscous behavior prevails over the elastic one. The main motivation for such a different terminology is that the exact mechanical model for former (EV) is an indefinite column of Newtonian fluid sustained by independent springs. While for (VE) material the exact mechanical model is constituted by a shear-type indefinite column resting on a bed of independent dashpots. The law of variation of the viscosity and of the springs decay with a power-law related to β as soon as the distance from the top of the column increase. It has also been shown that during creep (or relaxation) curves exhibit a quite different behavior for the two intervals of β . The aforementioned observations motivate the present paper. As in fact the real creep test is performed by a linear ramp of load in the first few seconds and at a certain time, say \bar{t} it remain constant. While during the relaxation test we have a linear ramp of deformation and then the deformation remain constant. The presence of the initial ramp of load (or the strain) strongly influence the deformation history during the creep or the stress history during the relaxation test especially for material which exhibit high relaxation time. In particular it is shown that for $0 \leq \beta \leq 1/2$ (EV) the initial ramp of load produces a nearly linear response in the first few seconds. While for $1/2 \leq \beta \leq 1$ (VE) the initial ramp of load produces a curve that is quite different from the linear one. On the other hand the real experimental test may not be performed with a unit step function since the perfect unit step is unrealizable from a physical point of view by experimental test machine. Moreover the maximum stress (or strain) is attained, in any

cases, at the time \bar{t} at which the ramp ends, that depends of the rate of the initial ramp. It follows that any identification procedure based upon real experiments are affected by some uncertainties because due to the long-tail memory of power-laws the results in terms of characteristic parameters (amplitude and exponent β) are strongly affected on initial ramp.

In this paper it has been shown that the best way to identify the characteristic values of amplitude and exponent of the power-law is performing the best fitting procedure by taking into account the real load (or strain) history during the creep (or relaxation) test. These results have been archived in two steps. In the first one the ideal test has been performed by considering an assigned power-law for creep (or relaxation). Then the theoretical results have been sampled (like it happens with an experimental test) and then the best-fitting procedure has been performed with the sample curves. With this ideal experiment it has been shown that results in terms of characteristic value are quite different depending of the initial ramp (and then on \bar{t}). Then it has been shown that for for both EV and VE materials by taking into account the real load (or strain) history the results in terms of the characteristic coefficients of the power-law exactly coincide with the ideal model. In the second step real experiments have been performed with different initial ramp by evaluating the differences in terms of characteristic parameters of the power-law. It has been also shown that for VE materials these characteristic coefficients may be also evaluated by sampling the response history in $0 \leq t \leq \bar{t}$.

2 FRACTIONAL CONSTITUTIVE LAW

In this section some fundamentals on fractional hereditary materials are introduced. The constitutive law relating stress $\sigma(t)$ and strain $\gamma(t)$ may be derived starting from the knowledge of the *relaxation function* $G(t)$ representing the stress history for the assigned stress history $\gamma(t) = U(t)$, being $U(t)$ the unit step function. Boltzmann superposition principle leads to

$$\sigma(t) = \int_0^t G(t-\tau) d\gamma(\tau) = \int_0^t G(t-\tau) \dot{\gamma}(\tau) d\tau \quad (1)$$

Eq. (1) is valid for $\gamma(0) = 0$ (virgin material). The inverse of constitutive law (1) may be faced starting from the *creep function* $J(t)$ that is the strain history for the assigned load history $\sigma(t) = U(t)$. The Boltzmann superposition principle for $\sigma(0) = 0$ and virgin material is written as

$$\gamma(t) = \int_0^t J(t-\tau) d\sigma(\tau) = \int_0^t J(t-\tau) \dot{\sigma}(\tau) d\tau. \quad (2)$$

From Eqs. (1) and (2) it may be recognized that $G(t)$ and $J(t)$ are related each another by the following relationship in Laplace domain

$$\hat{G}(s)\hat{J}(s) = \frac{1}{s^2} \quad (3)$$

where $\hat{G}(s) = \mathcal{L}\{G(t); s\}$, $\hat{J}(s) = \mathcal{L}\{J(t); s\}$ and s is the Laplace (complex) parameter.

The relaxation function $G(t)$ (or the creep $J(t)$) is obtained by best fitting procedure obtained by experimental test. Nutting (1) observed that every real materials give a relaxation function well fitted by a power lo of the type

$$G(t) = \frac{C(\beta)}{\Gamma(1-\beta)} t^{-\beta}; \quad 0 \leq \beta \leq 1 \quad (4)$$

where $\Gamma(\cdot)$ is the Euler gamma function and $C(\beta)$ and β are parameters depending of the material at hands.

As soon as we assume for $G(t)$ the power-law decay expressed in Eq. (4), by assuming Eq. (3) the creep function $J(t)$ is readily obtained in the form

$$J(t) = \frac{t^\beta}{C(\beta)\Gamma(1 + \beta)}. \quad (5)$$

By inserting Eq. (4) in Eq. (1) and Eq. (5) in Eq. (2) we get

$$\sigma(t) = C(\beta) \left({}_C D_{0+}^\beta \gamma \right) (t) \quad (6)$$

$$\gamma(t) = \frac{1}{C(\beta)} \left(I_{0+}^\beta \sigma \right) (t) \quad (7)$$

where $\left({}_C D_{0+}^\beta \gamma \right) (t)$ is the *Caputo fractional derivative* defined as

$$\left({}_C D_{0+}^\beta \gamma \right) (t) = \frac{1}{\Gamma(1 - \beta)} \int_0^t (t - \tau)^{-\beta} \dot{\gamma}(\tau) d\tau \quad (8)$$

and $\left(I_{0+}^\beta \sigma \right) (t)$ is the *Riemann-Liouville fractional integral* defined as

$$\left(I_{0+}^\beta \sigma \right) (t) = \frac{1}{\Gamma(\beta)} \int_0^t (t - \tau)^{\beta-1} \sigma(\tau) d\tau. \quad (9)$$

Since the power-law kernels leads to fractional operators we call such materials as *fractional hereditary materials*. A wide discussion on the limitations of β ($0 \leq \beta \leq 1$) is reported in (13), and for $\beta = 0$ the purely elastic materials appears while for $\beta = 1$ a purely Newtonian viscous fluid appears.

Even though the approach here presented is consistent from both a mathematical and mechanical point of view (2; 13; 14) problem on the evaluation of $C(\beta)$ and β from experimental test appears. As in fact in the real relaxation test we never observe an infinite value of the stress in zero and in the real creep test we never observe an infinite slopes in zero. It follows that for small values of the observation time in the real tests performed on real materials a dramatic discrepancy between experimental data and mathematical laws expressed in Eq. (4) and (5) is evidenced. Such observation may leads to reject the conclusion that fractional constitutive laws are approximations of real materials.

However a deeper insight based on the real tests (creep and/or relaxation) show that the aforementioned paradoxes on the behavior of viscoelastic materials in proximity of $t = 0$ disappear and this open the way for the correct evaluation of the coefficient $C(\beta)$ and β from experimental data.

This issue will be addressed in two steps. In the first step we analyzed in detail the problem from a mathematical point of view, with the results obtained from the mathematical aspects, as a second step the experimental tests are revisited opening the way for the correct evaluation of the characteristic values of the viscoelastic materials.

3 EXPERIMENTAL PROCEDURE OF RELAXATION TESTS AND FRACTIONAL CONSTITUTIVE EQUATION

For the full mechanical characterization of viscoelastic material it has to be take into account the time evolution of stress and strain history. Indeed in this case is considered the stress history $\sigma(t)$ and strain history $\gamma(t)$. For this reason the classical tensile test is not able to describe the time-dependent stress-strain relation and we need another type of experimental test. Exist two fundamental tests to characterize the

viscoelastic material, such tests are known as creep and relaxation test. The first of these methods aims to evaluate the time evolution of the strain response due to an imposed stress history which follows a unit-step function. Conversely in the relaxation test is carried out the measurement of the response in terms of stress history due to an imposed strain history which follows a unit-step function. Both the aforementioned tests are idealizations. For this reason the real creep and relaxation tests are different respect to the theoretical mentioned description because the test machines using for these tests aren't able to reproduce a unit step function both in imposed stress and/or strain history, as shown in Figure 1. On the other hand the reference

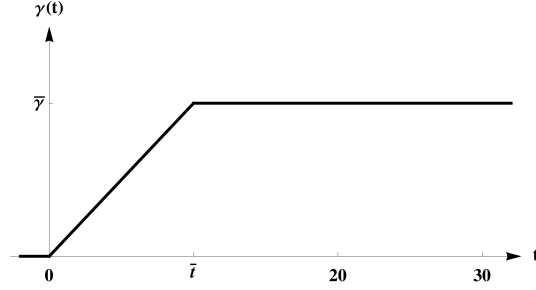


Figure 1: Real deformation history during the relaxation test.

standard (16), for stress relaxation tests for materials and structures give not specific indication regarding to the initial ramp for the initial stress: *The stress application rate in either case should be reasonably rapid, but without impact or vibration, so that any relaxation during the stress application period will be small.* Then the rate of the initial ramp is selected on the basis of the material hand as well as the total strain selected for the relaxation test. The time \bar{t} at which the deformation takes the constant value $\bar{\gamma}$ is strictly related the test machine as well as from the people making the test. Usually the rate of deformation is very high and consequently t is very small. It follows that usually this effect is neglected and it is assumed that $t = 0$ so the unit step deformation history is present. However the hypothesis that $\gamma(t) = \gamma U(t)$, for the power-law case produces an infinite value on the corresponding stress history this causes significant errors on the evaluation of $C(\beta)$ and β . The effect of the initial ramp is studied in detail from a theoretical point of view. Let us suppose that the deformation history, during the relaxation test, is that represented in Figure 1. It follows that the real strain history is

$$\gamma(t) = \begin{cases} 0, & \forall t : t < 0; \\ \bar{\gamma} t / \bar{t}, & \forall t : 0 \leq t < \bar{t}; \\ \bar{\gamma}, & \forall t : t > \bar{t}. \end{cases} \quad (10)$$

By using Eq. (6) we get the stress history in the form

$$\sigma(t) = \begin{cases} 0, & \forall t : t < 0; \\ \frac{C(\beta) \bar{\gamma}}{\Gamma(2-\beta) \bar{t}} t^{1-\beta}, & \forall t : 0 \leq t < \bar{t}; \\ \frac{C(\beta) \bar{\gamma}}{\Gamma(2-\beta) \bar{t}} \left[t^{1-\beta} - (t-\bar{t})^{1-\beta} \right], & \forall t : t > \bar{t}. \end{cases} \quad (11)$$

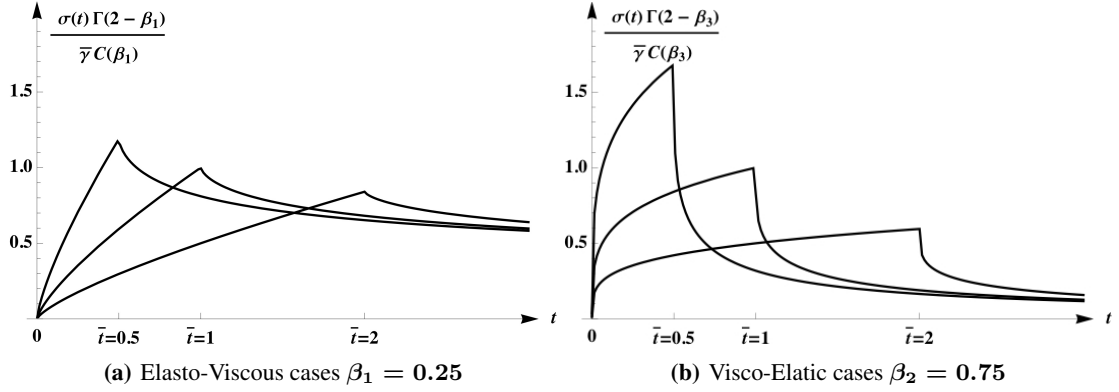


Figure 2: Stress history for the deformation history in Figure 1 for different value of \bar{t} and β .

Or in compact form Eq. (10) may be rewritten as

$$\sigma(t) = \frac{C(\beta) \bar{\gamma}}{\Gamma(2 - \beta) \bar{t}} \left[t^{1-\beta} - (t - \bar{t})^{1-\beta} U(t - \bar{t}) \right], \quad \forall t \geq 0. \quad (12)$$

The maximum stress is attained at $t = \bar{t}$ and it is obviously the value $\sigma(\bar{t})$ remains finite and is given as

$$\sigma(\bar{t}) = \frac{C(\beta) \bar{\gamma}}{\Gamma(2 - \beta)} \bar{t}^{-\beta}. \quad (13)$$

In Figure 2 the stress history corresponding to the deformation history in Figure 1, for assigned $\bar{\gamma}$ and various values of \bar{t} and different values of β , is depicted. From these figures it may be observed that for $\beta < 0.5$ in which the elastic phase prevails the response for $t < \bar{t}$ is nearly linear, and for $t > \bar{t}$ the classical relaxation curve appears that follows a power-law decay. Moreover, according to Eq. (13), the maximum value $\sigma(\bar{t})$ depends of the rate of deformation and on \bar{t} .

A different scenario appears for $\beta > 0.5$ in which the viscous phase prevails. In fact for $t > \bar{t}$ the Visco-Elastic behavior is present and the power-law is manifested, according to Eq. (12). Now the question is: how this distortions at the origin influence the value of $C(\beta)$ and β when the relaxation is assumed to be that given in Eq. (4) by disregarding the initial ramp? This question is of primary importance because usually the relaxation test is performed with the effective strain history given in Figure 1 and the best-fitting is performed on the entire stress history without taking into account the initial ramp. That is the best-fitting of experimental data performed by excluding the results in the interval $0 \leq t \leq \bar{t}$. With this way to obtain characteristic values $C(\beta)$ and β . In Table 1 the results of $\tilde{C}(\beta_i)$ and $\tilde{\beta}_i$ with $i = 1, 2, 3$ are reported by a best-fitting assuming the initial ramp is not present, for various values of \bar{t} . The total observation time t^* is 100 sec, 1000 sec, 3600 sec. The effective values of $C(\beta)$ and β are $C(\beta) = 1$, $\beta_1 = 0.25$, $\beta_2 = 0.50$, $\beta_3 = 0.75$. The percent error $\eta(\cdot)$ in the estimation procedure is defined as

$$\eta(v) = \left| \frac{v - \tilde{v}}{v} \right| 100 \quad [\%] \quad (14)$$

where v is the real value and \tilde{v} is estimated value. This error, which is related to the parameters β and $C(\beta)$, is also reported in Table 1 for the various case.

Estimation values of Elasto-Viscous case $\beta_1 = 0.25$ and Visco-Elastic case $\beta_2 = 0.75$

\bar{t}	t^*	$\tilde{C}(\beta_1)$	$\eta(C_1)$	$\tilde{\beta}_1$	$\eta(\beta_1)$	$\tilde{C}(\beta_2)$	$\eta(C_2)$	$\tilde{\beta}_2$	$\eta(\beta_2)$
0.5	100	0.912	8.8 %	0.230	7.9 %	0.460	54.0 %	0.612	18.4 %
	1000	0.947	5.3 %	0.241	3.2 %	0.482	51.8 %	0.637	15.1 %
	3600	0.962	3.8 %	0.245	1.9 %	0.490	51.0 %	0.645	14.0 %
1	100	0.868	13.2 %	0.220	11.9 %	0.354	64.6 %	0.568	24.3 %
	1000	0.919	8.1 %	0.238	5.0 %	0.375	62.5 %	0.603	19.6 %
	3600	0.941	5.8 %	0.242	3.0 %	0.384	61.6 %	0.615	18.0 %
2	100	0.813	18.7 %	0.206	17.2 %	0.265	73.5 %	0.522	30.4 %
	1000	0.883	11.7 %	0.232	7.4 %	0.285	71.5 %	0.568	24.2 %
	3600	0.914	8.6 %	0.239	4.5 %	0.294	70.6 %	0.584	22.1 %

Table 1: $C(\beta)$ and β estimation for different values of \bar{t} and of observation time t^* .

From these results it may be asserted that for \bar{t} small, $\beta \leq 0.5$ and large observation times t^* the best-fitting performed with Eq. (4) are in accordance each another. Moreover for $\beta > 0.5$ in any case the influence of the ramp is very important and best-fitting performed with Eq. (4) leads to uncorrected values of both $C(\beta)$ and β . On the other hand if for Visco-Elastic material the best-fitting is performed in the initial ramp ($0 \leq t \leq \bar{t}$), always the exact values of $C(\beta)$ and β are identified. Similar way for the identification of $C(\beta)$ and β for $0 \leq \beta \leq 0.5$ may not be pursued because the initial ramp is nearly linear and the results obtained by real experimental tests may be affected on the errors produced by the machine. That is the characterization of Elasto-Viscous materials the discrepancy from the linear ramp is small and then the errors in the evaluation of $C(\beta)$ and β are consistent.

Once all these aspects on the presence of the initial ramp have been highlighted some conclusions may be withdrawn: i) The presence of the initial ramp influence significantly the estimation of characteristic parameters $C(\beta)$ and β of the material; ii) The larger is the observation time the smaller is this influence; iii) In any case in the experimental test the time \bar{t} has to be reported since in some circumstances this value is very important and may not be disregarded.

For the creep test the results are very similar and are not here reported for shortness sake's. In the next section results based on real experimental test will be presented.

4 EXPERIMENTAL RESULTS AND CHARACTERIZATION OF CONSTITUTIVE LAW

In this section both the manufacturing procedure of the epoxy resin analyzed and all experimental creep tests hereinafter. Among the various viscoelastic materials, an epoxy resin has been chosen since nowadays this type of thermoset polymer finds application in several fields: e.g. coatings, electrical and electronic insulation, adhesives, construction and as matrix for composite materials widely used in automotive, nautical, aerospace and also civil fields (17; 18; 19). The material characterized was an epoxy resin obtained mixing a diglycid ether of bisphenol-A (DGEBA) epoxy monomer (SP 106, supplied by Gurit) with its own amine based curing agent (5 : 1 mix ratio by volume). As reported in the supplier datasheet, Table 2 shows the main properties of the epoxy resin cured for 28 days at 21 °C.

In this work, all the samples were prepared by pouring the epoxy monomer-curing agent blend onto the cavities of silicon rubber mold so as to get dog-bone specimens with thickness 5 mm, width of narrow section 6 mm, length of narrow section 33 mm, distance between grips 65 mm. Then, the curing process of

Tg [°C]	$\Delta H \left[\frac{J}{g} \right]$	Moisture Absorption [%]	Density $\left[\frac{g}{cm^3} \right]$	Lin. Shrinkage [%]	Barcol Hardness
64.8	13	1.578	1.188	1.7	30

Table 2: Properties of testing epoxy resin.

the thermoset material was carried out at room temperature for 24 h followed by a post cure treatment at 50°C for 6 h. Tensile and relaxation tests were performed by employing an Universal Testing Machine (UTM) mod. Z005 by Zwick Roell, equipped with a load cell of 1 kN. From tensile tests, carried out according to ASTM standard (20) on five samples setting the rate of deformation equal to 2 mm/min, average values of ultimate stress (44.9 ± 4.9 MPa), strain ($3.88 \pm 0.36\%$) and deformation (2.52 ± 0.24 mm) have been derived. After this, relaxation experiments were performed imposing in the first period a linear ramp of deformation up to reach a constant value correspondent to 40% of Δl_u (i.e. 1 mm) at a certain time, say \bar{t} . After this, the deformation was keeping constant for 24 h, an interval time that may be considered as sufficiently representative but these other tests are describe in the next section.

In order to demonstrate that the presence of the initial ramp of deformation strongly influence the stress history during the relaxation test, different tests were performed by varying the rate of initial ramp. In particular, keeping constant the value of deformation (i.e. 1 mm) at which the initial ramp ends, the relaxation test were performed by setting the rate of deformation equal to 2mm/min ($\bar{t} = 30$ s) and 0,2 mm/min ($\bar{t} = 300$ s) respectively.

The experimental test carried out to study of the influence of the initial ramp have been performed on three different specimens for two different performed tests. By using the Eq. (12) for the best fitting of the experimental data it is taking into account the pre-load ramp and the estimated parameters for the six tests are reported in the Table 3. The parameters reported in Table 3, have been obtained by the best fitting by using

\bar{t}	specimen #	$C\beta$	β
30	1	2588.89	0.0425
	2	2341.58	0.0427
	3	2973.67	0.0427
300	4	3370.38	0.0530
	5	2710.34	0.0425
	6	2695.81	0.0425

Table 3: Estimated parameters of tested epoxy resin taking into account the pre-load ramp

different value of observation times $t^* = 200, 2000, 7200$ sec and no appreciable differences between $C\beta$ and β have been evidenced. Moreover experimental results in dotted lines of Figure 3 and 4, are contrasted with the theoretical ones obtained by using Eq. (12). Moreover in Table 4 the estimated parameters of tested epoxy resin are reported by using the best fitting procedure by using directly Eq. (4), that is by disregarding the initial ramp and for different values of observation times. As we aspect the larger the observation time is, the results in term of $C(\beta)$ and β tends to the values obtained by corresponding the initial ramp.

In Figure 3 and 4 the results in terms of relaxation function obtained by the parameters in table 4 are plotted in solid line by using Eq. (4), that is by disregarding the effect of the initial ramp. It may be observed that the theoretical results differ from the experimental one in a sensible way, especially of small value of t . As a concluding remark on the basis of both theoretical and experimental results we may assert that the

		$t^* = 200$ sec		$t^* = 2000$ sec		$t^* = 7200$ sec	
\bar{t}	specimen #	$\tilde{C}(\beta)$	$\tilde{\beta}$	$\tilde{C}(\beta)$	$\tilde{\beta}$	$\tilde{C}(\beta)$	$\tilde{\beta}$
30	1	2234.36	00150	2409.66	0.0308	2593.42	0.0418
	2	2003.29	0.0150	2163.18	0.0311	2327.04	0.0421
	3	2546.81	0.0139	2783.17	0.0328	2965.80	0.0425
300	4	2596.51	0.0103	2871.21	0.0314	3169.49	0.0464
	5	2195.62	0.0089	2341.28	0.0223	2474.14	0.0306
	6	2169.76	0.0083	2319.33	0.0220	2453.23	0.0304

Table 4: Estimated parameters of tested epoxy resin not taking into account the pre-load ramp

influence of the initial ramp for the best fitting parameters and the correct interpretation of the parameters may be obtained may be obtained from both the initial ramp and the subsequent decay especially for VE materials.

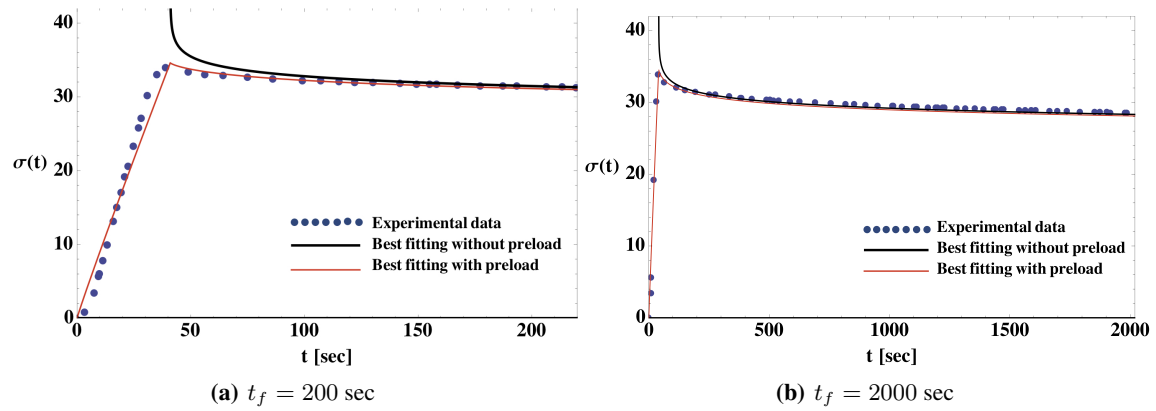


Figure 3: Stress history with $t^* = 31$ sec for different observation time t_f .

If such a procedure is performed then the influence of the total of the total observation time (provided $t > t_f$) does not produce sensible variation on $C(\beta)$ and β . It follows that the experimental time consuming for the characterization of the VE or EV materials may be strongly reduced provided the effective strain (or stress) history is known. As in fact how it has been shown in Table 3 with only 200 sec of experimental test the correct coefficient $C(\beta)$ and β are in good agreement with those evaluated for very long t^* .

5 CONCLUSIONS

The viscoelastic behavior of real materials is usually identified by a creep and/or relaxation test and subsequent best fitting procedure with proper assigned laws. Creep and relaxation presume that the stress or the strain imposed is a unit step function. However the real experimental tests are made by an initial ramp that may be assumed linear and then the stress (or strain) remain constant for the remainder time of observation. The initial ramp, especially for polymeric materials with very high relaxation time, strongly influences the entire stress (or strain) history. It follows that the best fitting procedure for such materials has

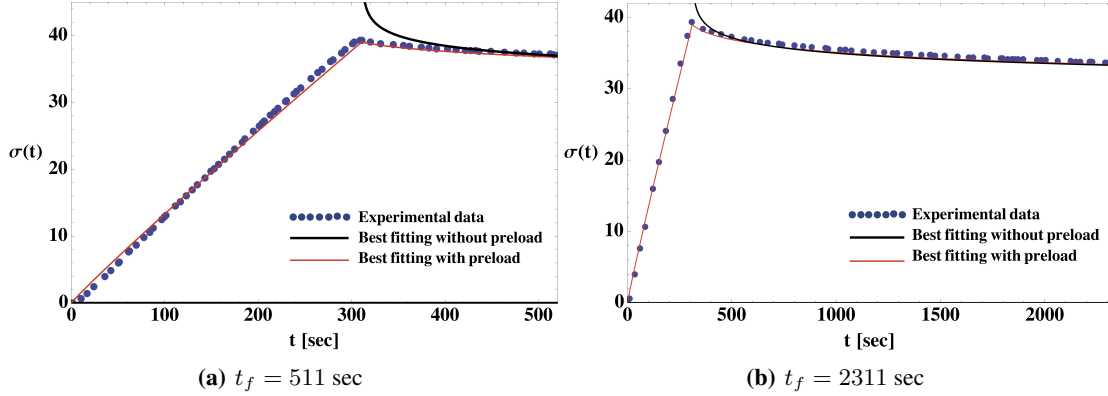


Figure 4: Stress history with $t^* = 411$ sec for different observation time t_f .

to take into account also the initial ramp.

In this paper it has been shown that the best fitting performed by taking or not into account the initial ramp gives very different parameters and then the constitutive law is strongly influenced by the real experimental test. It is also shown that by accounting for the effective strain (or stress) history leads to an impressive matching between experimental tests and results obtained by using Boltzmann superposition principle and power law as candidate for the best fitting procedure. As a concluding remark, we may assert that in every experimental test the rate of the initial ramp or the time at which the unitary (constant value of) strain (or stress) is attained must be always declared.

Moreover from the theoretical results presented in the paper and from the experimental test performed on an epoxy resin is assessed that if the initial ramp is taken into account then the influence of the total observation time does not alterate sensibly the parameters estimation in terms of $C(\beta)$ and β . The latter observation allow us to reduce the time consuming of the experimental test.

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