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Prony series calculation for viscoelastic behavior modeling of structural adhesives from DMA data

Cálculo de Series de Prony a partir de datos de DMA para modelado de comportamiento viscoelástico de adhesivos estructurales

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Abstract

In product design is important to choose the correct material for a specific application. Viscoelastic behavior let us know how much energy the material can dissipate on its internal structure or either return it to the surroundings, and the property that describe this is the Complex Modulus G*, it is a complex quantity that can be separated in a real and an imaginary part called G′ storage modulus and iG′ loss modulus respectively. These properties can be measured experimentally from a small material sample easily by performing Dynamical Mechanical Analysis (DMA). In Product Design process there are both, computational and physical validations and there is the need of improving computational studies by understanding the physics of each component. Viscoelastic characteristics of materials can be represented by Prony series, also known as relaxation modulus in function of time. Relaxation modulus can be defined in most of Computer Aided Engineering (CAE) Software. In this article the procedure for calculating Prony Series from DMA data will be explained.
Keywords: Prony series, viscoelasticity, complex modulus, storage modulus, loss modulus, DMA.

Resumen

En el diseño de un producto es importante elegir el material correcto para una aplicación específica. El comportamiento viscoelástico nos muestra qué tanta energía puede disipar un material en su estructura interna, o bien, devolver a sus alrededores; la propiedad que describe esto es el módulo complejo G*, que es una cantidad compleja que se puede separar en una parte real y una imaginaria llamada G′ módulo de almacenamiento e iG″ módulo de pérdida, respectivamente. Estas propiedades se pueden medir experimentalmente de una pequeña muestra de material, aplicando Análisis Mecánico Dinámico (DMA). En el proceso de diseño de un producto existen las validaciones computacionales y las validaciones físicas, por lo que existe la necesidad de mejorar los estudios computacionales mediante el entendimiento de la física de cada componente. Las características viscoelásticas de los materiales se pueden representar por Series de Prony, también conocidas como el módulo de relajación en función del tiempo. El módulo de relajación se puede definir en la mayoría de los paquetes de Software para Ingeniería Asistida por Computadora (CAE). En este artículo se explicará el procedimiento para calcular las Series de Prony de datos obtenidos con DMA.
Descriptores: Serie de Prony, viscoelasticidad, módulo complejo, módulo de almacenamiento, módulo de pérdida, DMA.

Introduction

In this article the method to calculate Prony series from DMA data will be explained with a real example. The Prony series of a piece of structural adhesive used in automotive industry will be obtained showing in detail the considerations and background of each decision taken during the analysis with the purpose of being a good reference for the reader. The motivation for writing this article is the lack of literature available on this subject.

Background on this article analysis is as follows: Automotive body is manufactured from steel or aluminum plates (Davies, 2003). Structural adhesives are used to fill the cavities between these plates and therefore increasing the strength of the structure. Normally they are installed before paint process as it is shown in Figure 1, where they will be cured with the furnace temperature. The installation process consists on place them on the surface of a metal plate in order they adhere by themselves to it. This process is most of the times done manually by an operator. Some of these adhesives when heated liberate gases that will create small bubbles on them, and they will increase their volumes, this is the expansion mechanism that promotes the filling of the closed section in the body structure as shown in Figure 2.

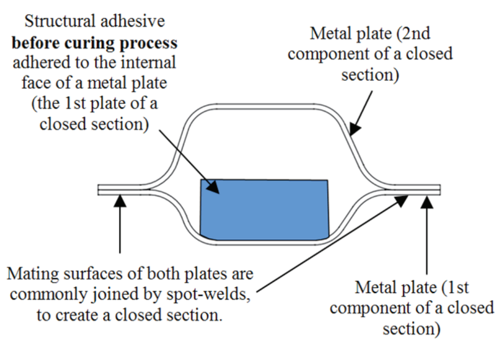

Figure 1. Structural adhesive before curing process

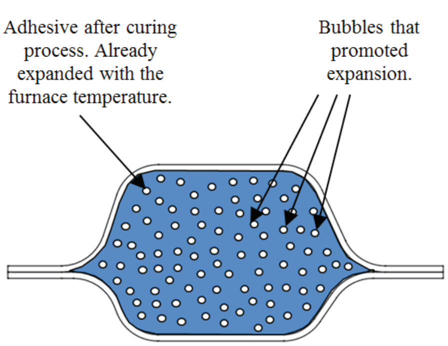

Figure 2. Structural adhesive after curing process

Energy during the impact will be dissipated by the structural adhesives. The properties that describe the energy dissipation capacity of materials are known as dynamic properties. The complex modulus G^* is a complex quantity that can be divided in a real part and an imaginary part, called storage modulus G', and the loss modulus G'', respectively. Their relationship is indicated in equation 1.

$$G^* = G' + iG'' \tag{1}$$

The storage modulus G' describes the capacity of a material of storing and returning energy to the surroundings, and the loss modulus G'' describes the capacity of a material of dissipating energy on its internal structure. A graphical explanation of these concepts are often explained with the Figure 3.

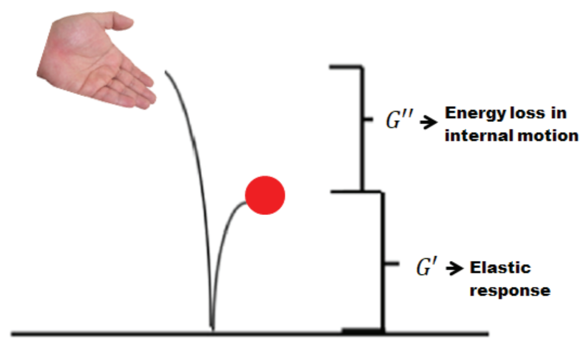

Figure 3. Concept explanation of storage & loss moduli (Saba *et al.*, 2016)

Dynamical mechanical analysis

The dynamical properties are obtained by an experimental method called dynamical mechanical analysis, DMA from now on. DMA is performed in a laboratory using a Dynamic Mechanical Analyzer, a device commonly used in the study of polymers rheology. Dynamical properties are studied by applying a cyclic stress into a material sample (Sepe, 1998), it can be applied in different modes: tension, bending, cantilever, dual cantilever, torsional, etc. Often the results are shown as frequency response diagrams, also named frequency sweep, where in the vertical axis are shown the values of Storage Modulus G' and loss Modulus G'', and in the horizontal axis are shown the frequencies where the analysis was performed. Most of the times horizontal axis is presented in a logarithmical scale since it is important to show high and low frequency values. An example of a frequency scan can be observed in Figure 4.

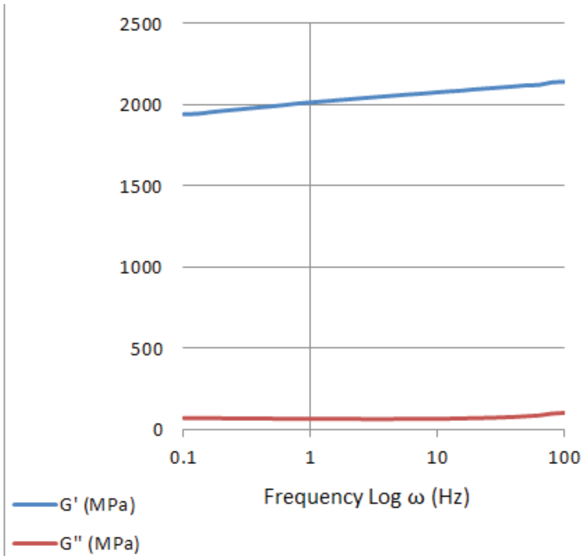

Figure 4. Example of a frequency sweep

The Dynamical Mechanical Analyzer is a device that has movable and fixed clamps. The fixed clamps remain static while the movable clamp will be displaced up and down by a driveshaft deforming the material in an oscillation pattern as illustrated in Figure 5. The amplitude and frequency of the oscillation is programed based on the characteristics of each material. The mathematical background for the calculation of G′ and G′′can be found in (Ferry, 1980).

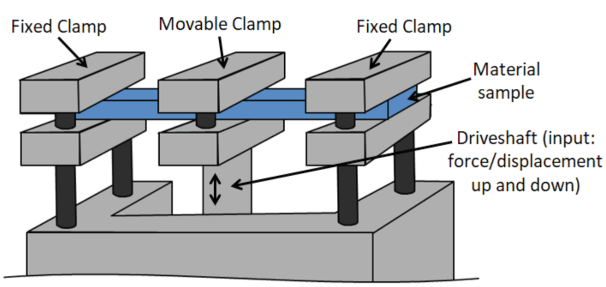

Figure 5. Dynamical Mechanical Analyzer

In this work a sample of cured structural adhesive (epoxy) with low expansion capability (around 15 % of volume increase) was evaluated. Frequency sweep from 0.1 to 100 Hz was carried out with a Q800 TA Instrument DMA device using a dual cantilever clamp configuration as is shown in Figure 5. An oscillation amplitude of 5 mm was applied at a constant temperature of 30 °C to obtain the experimental viscoelastic properties. Structural adhesive sample dimensions for ω dual cantilever DMA test were 35.0x13.1x3.7 mm. These samples were prepared cutting the sample of epoxy adhesive with the required dimensions and cured in a convection furnace at 120 °C for 2 hours. Structural adhesives can go from a 15 % to a 300 % expansion ratio depending on the application and performance required. DMA is performed in order to obtain the values of G', G'' and tan that is defined as:

$$\tan\delta = \frac{G''}{G'} \qquad (2)$$

In a DMA experiment where a cyclic force is applied on a material sample, it can be noticed a phase lag between the applied load curve and the strain curve, as shown in Figure 6. This phase lag is an angle named δ. tan δ is a quantity often used in polymers rheology, also known as damping factor and it is related to the energy that the material is dissipating on its structure.

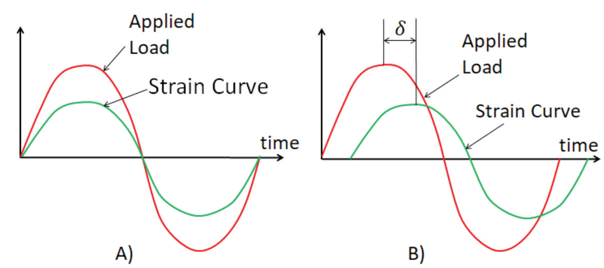

Figure 6. Explanation of tan δ concept: a) shows the plots of an ideal 100 % elastic material, where both curves are in phase, b) shows the plots of a viscoelastic material where there is a phase lag between both curves

The method used to get the dynamical properties of the structural adhesive was the following: the imposed deformation mode was dual cantilever, and the size of the sample was 35.0x13.1x3.7mm. The analysis was performed in a range of frequencies that goes from 0.1 to 100 Hz, with 10 measurements on each frequency decade. A decade must be understood as an order of magnitude, for example; from 0.1 to 1 we have 1 decade and from 1 to 10 we have another one. This frequencies range was selected based on vibrations commonly detected in automotive bodies due to the input of pavement irregularities through vehicle’s suspension. That input typically goes from 1 to 2 Hz, as mentioned in (Spinola, 2012), nevertheless it was decided to measure in a wider range in order to have the necessary data to make the curve approximation of G′ or G′′ as functions of the frequency ω as it will be exposed later in this work.

It is desirable to have a wide range of measurements along 3 or more frequency decades for any material under study to have a good material characterization. Also it is recommended that the common usage input frequency of the product that is under study is included in this range (in this case the structural adhesive used in automotive body). The advantage of perform testing in the frequencies domain is that it requires less time than carry out them on time domain, since the frequency is an inverse quantity with regards of time. This characterization in the frequencies domain will allow a good curve fitting that will let us know the numerical values that will be finally substituted in the Prony series that are functions in the time domain.

The deformation applied to the sample depends on the nature of the material. One method is to impose a small deformation (less than 1 % of the sample size) and keep increasing it until a constant value of the moduli is obtained.

The measured values obtained by performing DMA to this sample are shown in the Table 1.

Models of viscoelastic behavior

Most of all materials have a viscoelastic behavior and in the case of adhesives it is remarkable due to their polymeric nature. Elastic behavior is modeled by Hooke law that can be represented by the following equations:

$$\sigma = E\varepsilon \tag{3}$$

In the case of tensile stress, and:

$$\tau = G\gamma \tag{4}$$

In the case of shearing stress; where:

σ = tensile stress
E = Young's modulus
ε = tensile strain
τ = shear stress
G = shear modulus
γ = strain by shear

Viscous behavior is modeled by the Newton's law:

$$\tau = \eta\dot{\gamma} \tag{5}$$

Where:

τ = shear stress
η = viscosity
$\dot{\gamma}$ = strain rate

In the case of viscoelasticity of polymers, a more complex model is needed because it depends on several facts: the size of the molecules, if they are crosslinked or not (Ferry, 1980), and also stress relaxation due to polymeric chains re-ordering with time. By these reasons, the equation that models viscoelasticity uses a modulus that is a function of time, as follows:

$$\tau = G(t)\dot{\gamma} \tag{6}$$

Where $G(t)$ is the stress relaxation modulus of the material.

Ferry (1980) shows the same constitutive equation with the following notation:

$$\sigma_{ij}(t) = \int_{-\infty}^{t} G(t-t')\dot{\gamma}_{ij}(t')dt' \tag{7}$$

Where:

σ_{ij} = stress tensor
$\dot{\gamma}_{ij}$ = strain rate tensor
t' = all the past times were the integration must be carried out over all of them, this must be considered due to the memory of the material during deformation.

Two basic mechanical models that predict viscoelastic properties of materials are Voigt and Maxwell elements, that are represented by springs and dashpots connected between them, as illustrated in Figure 7. The materials behavior can be described as combinations of Maxwell and Voigt elements connected between them. One simple model is the generalized Maxwell model that is just a set of Maxwell elements connected in parallel.

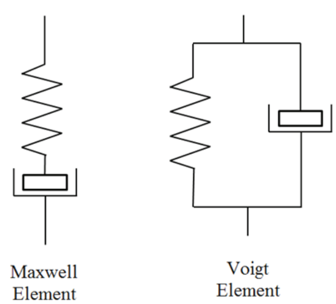

Figure 7. Basic viscoelasticity modeling elements

In generalized Maxwell model, the contribution in relaxation modulus of each Maxwell element is given by the equation 8:

$$G_i(t) = G_i e^{-t/\tau_i} \tag{8}$$

Where G_i is the spring constant and τ_i is the relaxation time of the element, defined in equation 9.

$$\tau_i = \eta_i / G_i$$

Where η_i is the dashpot constant. The relaxation modulus of the complete model is the sum of the relaxation moduli of all Maxwell elements connected in parallel, as follows:

$$G(t) = \sum_i G_i e^{-t/\tau_i} \tag{10}$$

When $t \to \infty$ in the case of materials that have a solid-like behavior, $G(t)$ approaches a finite value G_e. For liquid-like materials, $G(t)$ approaches zero (Ferry, 1980), this is why in the case of structural adhesives, that become solid-like materials after the curing process, the equation 4 can be rewritten as:

$$G(t) = G_e + \sum_i G_i e^{-t/\tau_i} \tag{11}$$

The equation 11 is also known as the Prony Series (Tschoegl, 1989) of the material, and is useful to describe viscoelastic properties in CAE Software (ABAQUS, 1998).

Using DMA data to get Prony series of structural adhesives

As mentioned previously, it is possible to obtain the values of storage modulus G' and loss modulus G'' in function of the frequency by performing DMA to a sample of material. Also, as exposed by Ferry (1980) & Tschoegl (1989), these quantities are represented as functions of frequency in the following equations:

$$G'(\omega) = \{G_e\} + \sum_i \frac{G_i \omega^2 \tau_i^2}{1+\omega^2 \tau_i^2} \tag{12}$$

$$G''(\omega) = \sum_i \frac{G_i \omega \tau_i}{1+\omega^2 \tau_i^2} \tag{13}$$

This means that is possible to make a curve approximation in order to know the values of the G_i and τ_i terms, depending on the number of terms desired for the sum. One theoretical method is exposed by Baumgaertel & Winter in (1989). For practical uses, this can be done using software to approximate the curve to these custom equations. The first step is to decide the number of terms. For this work, we will approximate a curve to the first two terms of the sum shown in equation 12.

$$G'(\omega) = \{G_e\} + \frac{G_1 \omega^2 \tau_1^2}{1+\omega^2 \tau_1^2} + \frac{G_2 \omega^2 \tau_2^2}{1+\omega^2 \tau_2^2} \tag{14}$$

In equation 14 there are five unknown values: G_e, G_1, G_2, τ_1 and τ_2, these values will be approximated by fitting the curve with the form of equation 14 to the data obtained by DMA on the "Storage Modulus" column in Table 1. Software such as Matlab® can be used for a fast procedure capable to meet the Automotive Industry demands on quick analysis. The command used in Matlab® to make the curve approximation is "cftool" (curve fitting tool). This command will display an interactive fitting tool where the column vectors previously created with the information of the columns "Storage Modulus" and "Frequency" in the Table 1 can be loaded as vertical and horizontal axis respectively. In the case of solid-like materials is convenient to use the storage modulus instead of the loss modulus, for example, in this material it was possible to make the approximation of the curve with 30 points over 3 decades, but in the case of loss modulus the experiment should be made in a wider range of frequencies to get a better approximation of loss modulus curve. The loss modulus values are small in comparison to storage modulus values (by a factor of 30 times approximately). This also can be noticed in tanδ values that approach zero. By this reason it is very difficult to get a good approximation with loss modulus curve, since dominant behavior is described by storage modulus. Only storage modulus curve approximation will be discussed in this work.

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Table 1. Data obtained from DMA

Time	Temperature	Frequency	Storage Modulus	Loss Modulus	Tan Delta	Angular Strain
min	°C	Hz	MPa	MPa		%
7.49	30.26	0.1	1941	70.08	0.03611	0.08203
16.95	30.19	0.13	1944	70.48	0.03626	0.082
24.06	30	0.16	1953	69.81	0.03574	0.082
25.81	30	0.2	1961	68.64	0.035	0.08202
27.8	30	0.25	1968	68.17	0.03463	0.08202
32.92	30	0.32	1976	67.41	0.03411	0.08201
34.66	30.01	0.4	1983	67.03	0.03379	0.08202
35.74	30	0.5	1990	66.37	0.03335	0.08202
45.19	30	0.63	1998	65.99	0.03302	0.08201
56.9	30	0.79	2007	65.61	0.0327	0.08201
59.52	30	1	2014	65.14	0.03234	0.08203
60.5	30	1.3	2021	64.65	0.03199	0.08202
61.7	30	1.6	2027	64.54	0.03184	0.08202
62.08	30	2	2033	64.26	0.0316	0.08203
62.32	30	2.5	2039	64.04	0.0314	0.08203
63.33	30	3.2	2046	63.56	0.03106	0.08202
63.7	30.01	5	2058	64.16	0.03118	0.08203
64.68	30	6.3	2064	64.53	0.03127	0.08203
65.88	30	7.9	2069	64.9	0.03136	0.08203
66.26	30	10	2076	65.59	0.0316	0.08203
66.78	30	12.6	2082	66.4	0.0319	0.08203
67.4	30	15.8	2087	67.69	0.03243	0.08203
67.65	30	19	2093	68.91	0.03293	0.08203
67.8	30	25	2100	71.17	0.03389	0.08203
68.32	30	31.6	2106	73.72	0.035	0.08203
68.94	30	39.8	2112	76.72	0.03632	0.08203
69.19	30	50	2119	81.4	0.03842	0.08203
69.34	30	63	2121	85.96	0.04053	0.08203
69.59	30	79.5	2137	97.34	0.04556	0.08203
69.76	30	100	2141	102.3	0.04779	0.08203

The obtained values from storage modulus $G'(w)$ curve approximation are the following: G_e = 1947.00, G_1 = 85.79, G_2 = 97.63, τ_1 = 0.0659, τ_2 = 1.6950 replacing these values in equation 14 it is obtained a function of the frequency ω only, as is shown in equation 15:

$$G'(w) = 1947.00 + \frac{85.79w^2(0.0659)^2}{1+w^2(0.0659)^2} + \frac{97.63w^2(1.695)^2}{1+w^2(1.695)^2} \quad (15)$$

By plotting the equation 15 as a function of the frequency ω is obtained the chart in Figure 8.

The values obtained in the curve approximation can also be replaced in the equation 11, in this case for the two first terms of the series, obtaining:

$$G(t) = 1947.00 + 85.79^{e-t/0.0659} + 97.63e^{-t/1.695} \quad (16)$$

The terms of equation 16 are the Prony series of the structural adhesive, also known as the relaxation modulus of the material in function of time. In Figure 9 is showed a plot of equation 16 as a function of time t, it is possible to observe the behavior of the relaxation modulus, where can be noticed that it's value when a load

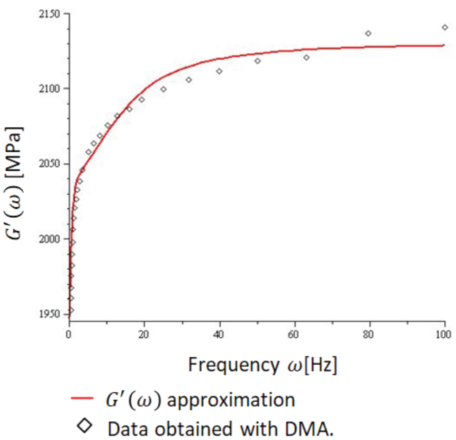

Figure 8. Curve approximation of storage modulus to data obtained by DMA

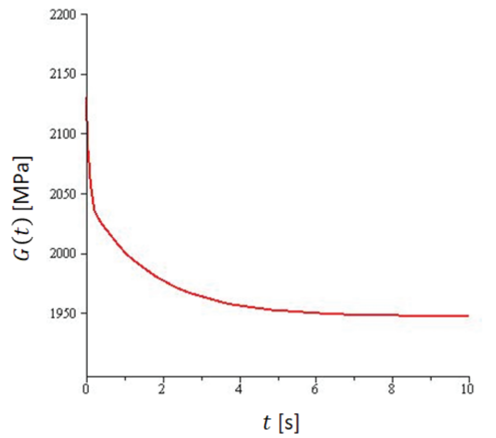

Figure 9. Relaxation modulus G(t) in function of time

have been applied for a long time tends to the value of $G_e = 1947.00$ *MPa*, as mentioned previously.

It can be confirmed the accuracy of the model since the approximated values of G' (ω) calculated with equation 15 have less than 1 % of error with regards of the data obtained by experimental measurement through DMA experiment. Table 2 shows the error calculation for each DMA data point *vs.* the calculated value with the approximated function.

These results allow us to confirm that model showed in equation 15 is a good prediction for G' (ω) since the error values are below 1 %, and this assure that $G(t)$ values will have good correlation with real behavior.

CAE modeling using Prony series

As mentioned previously, the Prony Series of equation 16, are also known as the discrete relaxation modulus in function of time. When equation 16 is substituted in equation 7, the constitutive equation is completely defined, the resulting is in equation 17.

$$\sigma_{i,j} = \int_0^t [1947.00 + 85.79e^{-(t-t')/0.0659} + 97.63e^{-(t-t')/1.695}]\dot{\gamma}_{i,j}(t')dt' \quad (17)$$

CAE makes possible to incorporate the geometry of the product under analysis by dividing a complex geometry in small elements through the creation of a mesh. Equation 17 will be discretized and solved for each one of the elements of this mesh, since σ_{ij} is the stress tensor of an infinitesimal element as ilustrated in Figure 10.

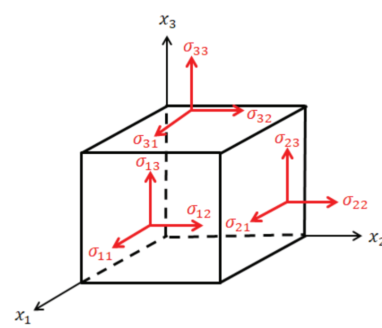

Figure 10. Stress tensor components

The definition of Prony Series may be different on each commercial CAE software package. In the case of Abaqus® when the model is viscoelastic and defined in the time domain, the software displays a table similar to Table 3, where the requested parameters are: g_i, k_i, and t_i. The g_i terms are the normalized Prony coefficients for shear (deviatoric) behavior, the k_i terms are the normalized Prony coefficients for volumetric behavior, and the t_i values are the relaxation times of Prony series. Abaqus® assumes that the frequency dependence of g_i and k_i is independent. In this work the volume changes in the material will not be considered, therefore the column for k_i values will be left blank. The changes in shape are described by g_i the deviatoric behavior, described by g_i terms, they are defined as follows (Chen, 2000):

$$g_i = \frac{G_i}{G_0} \quad (18)$$

Where the G_i values are the coefficients of the Prony series, and G_0 is the relaxation modulus $G(t)$ evaluated in $t = 0$.

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Table 2. Error calculation of values measured with DMA vs. values calculated with the fitted function $G'(\omega)$ (equation 15)

Frecuency	Calculated storage modulus with equation 15	Measured storage modulus	$Error = \frac{\lvert measured\ value - calculated\ value \rvert}{measured\ value}$
Hz	MPa	MPa	%
0.1	1949.7303	1941	0.4498
0.13	1951.5271	1944	0.3872
0.16	1953.6981	1953	0.0357
0.2	1957.0780	1961	0.2000
0.25	1961.882	1968	0.3107
0.32	1969.2311	1976	0.3426
0.4	1977.8047	1983	0.2620
0.5	1987.9030	1990	0.1054
0.63	1999.1612	1998	0.0581
0.79	2009.9052	2007	0.1448
1	2019.7899	2014	0.2875
1.3	2028.5758	2021	0.3749
1.6	2033.8793	2027	0.3394
2	2038.2660	2033	0.2590
2.5	2041.7259	2039	0.1337
3.2	2045.0396	2046	0.0469
5	2051.6141	2058	0.3103
6.3	2056.2783	2064	0.3741
7.9	2062.2139	2069	0.3280
10	2070.0282	2076	0.2877
12.6	2079.1046	2082	0.1391
15.8	2088.7098	2087	0.0819
19	2096.4331	2093	0.1640
25	2106.6909	2100	0.3186
31.6	2113.6681	2106	0.3641
39.8	2118.8206	2112	0.3229
50	2122.4477	2119	0.1627
63	2124.9605	2121	0.1867
79.5	2126.6367	2137	0.4849
100	2127.7134	2141	0.6206

Table 3. Dimensionless Prony series coefficients input in Abaqus®

g_1	k_1	τ_1
0.0403	-	0.0659
0.0458	-	1.695

Calculating the values of G_0, g_1 and g_2:

$$G_0 = G(0) = 2130.42\ MPa$$

$$g_1 = \frac{G_1}{G_0} = \frac{85.79\ [MPa]}{2130.42\ [MPa]} = 0.0403$$

$$g_2 = \frac{G_2}{G_0} = \frac{97.63\ [MPa]}{2130.42\ [MPa]} = 0.0458$$

g_1 and g_2 are also known as the dimensionless Prony series coefficients.

Table 3 shows the input of values in Abaqus®. This table can have as many rows as known terms of the Prony series are available. By filling this table, the viscoelastic characteristics of the material under study are completely defined.

The method to calculate the relaxation modulus in function of time presented in this work has many application possibilities. Examples of other applications in automotive industry that used Prony series to describe viscoelastic behavior are : Prediction of damping of railway sandwich type dampers (Merideno *et al.*, 2014), Modeling of viscoelastic parameters for automotive seating applications (Deng *et al.*, 2003), Stress concentration analysis near holes in viscoelastic bodies (Levin *et al.*, 2013), Analysis of adhesion of thermoplastics to steel (Golaz *et al.*, 2011), Finite element analysis of seat cushion and soft-tissue materials focused on passenger-vehicle occupants comfort (Grujicic *et al.*, 2009), Modeling of composites viscoelastic behavior (Machado *et al.*, 2016; Ishikawa *et al.*, 2018).

Conclusions

Viscoelastic properties of materials applied on CAE studies can provide more accurate results since it is a better approximation to real behavior of the material. The relaxation modulus of the analyzed structural adhesive showed a significant dependence with time since modulus values change from the maximum value in $G_O = 2130.42\ MP_a$ to the value of $G_e = 1949.00\ Mp_a$ due to stress relaxation. This shows that for vehicle CAE impact evaluation that occurs at a high speed the modulus will have also a high value since the stress relaxation will not occur at this speed. In the case of durability CAE evaluation, where the load is applied in long periods of time or in a time dependant load variation the stress relaxation will occur, therefore the modulus value will achieve the G_e value. Viscoelastic properties of structural adhesives can be incorporated to CAE by estimating Relaxation modulus in function of time through Prony series by fitting a model to DMA experimental data in function of frequency.

Model fitting for G' (ω) with respect to experimental DMA data showed an error smaller than 1 % using only two terms of the Prony series.

Through this methodology, parameters required to model viscoelasticity behavior in function of time in CAE software like Abaqus® can be obtained from the information of a single DMA experiment performed to a small material sample.

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