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IDENTIFICATION OF MECHANICAL PROPERTIES OF POLYTETRAFLUOROETHYLENE COMPOSITE BY INVERSE METHOD

Luiz Paulo Camargo Vieira Romano,luiz.paulo@teadit.com.br
Teadit – Av. Pastor Martin Luther King Jr. 8939 CEP:21530012

Lavinia Maria Sanabio Alves Borges,lavinia@mecanica.coppe.ufrj.br
Célio Albano da Costa Neto,celio@metalmat.ufrj.br
UFRJ - Cid. Universitária, Cx. Postal: 68503,CEP: 21941-972

Abstract. *The aim of this work is to characterize and identify the mechanical behavior of a composite material used in industrial gaskets and constituted by PTFE (polytetrafluoroethylene) reinforced with silica particles. The viscoelastic parameters identification fits in the so called model up-date which seeks matching experimental results to analytical modeling. The Maxwell model is adopted for the theoretical description of the problem since it better represents the observed mechanical behavior in the performed controlled deformation tests. By considering the experimental data, numerical results and the Levenberg-Marquard technique, the material properties are identified with a good accuracy.*

Keywords: *viscoelasticity, mechanical properties, composite material, polytetrafluoroethylene, Inverse Method*

1. INTRODUCTION

The PTFE (polytetrafluoroethylene), commercialized by the name of Teflon® , is a polymer formed by long chains of carbon atoms with pendent fluorine atoms. In spite of the processing difficulties due to its high molecular weight and, consequently, its high fusion viscosity, this material is extensively used because of its excellent chemical inertness, its electrical and thermal isolation capacity and its low friction coefficient. Frequently, the PTFE is combined with other materials with the purpose to maximize desired characteristics and minimize undesirable effects. In this work, specifically, a composite material used in industrial gaskets, composed by a PTFE matrix reinforced silica particles is considered. It is important to mention that the viscoelastic behavior of the material is particularly important for the gasket's efficiency.

The objective of this work is to develop a theoretical-experimental model suitable to predict the material's viscoelastic behavior. The experimental results are compared to their counterparts obtained from analytical models and an optimization algorithm is used to fit those analytical models in order to yield the sought viscoelastic constants. The objective function is defined by the difference between the analytical stress and the stress obtained from a controlled deformation hysteresis essay. The Maxwell model is adopted for the theoretical description of the problem since it better represents the mechanical behavior observed in the performed controlled deformation tests. By considering the experimental data, numerical results and the Levenberg-Marquard technique, the material properties are identified with a good accuracy.

Although composite of PTFE are the raw material for gaskets, the aim of this work is not to identify indices of leak or physical characteristic to improve the system's seal but, in fact, to identify properties of viscoelastic material that are fundamental for the material's stress recovery, strength and cold flow.

2. MAXWELL MODEL

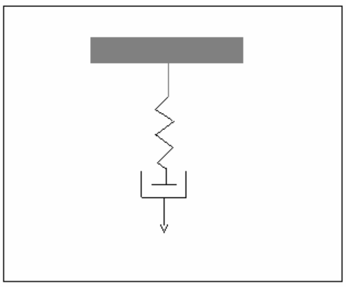

Figure 1- Rheological Maxwell model

The modulus of elasticity and the viscosity are the main parameters that characterize the viscoelastic behavior of materials obeying the Maxwell model. The difficulties to identify polymers properties are justified by their large dependence on the essay's conditions, as for example, the rate of load, temperature and quantity of deformation (Ward ,1985). That is, on the contrary of the elastic material's behavior, the viscoelastic behavior is strongly time dependent. In this section it is described the realized experimental tests and suitable to identify these parameters.

The behavior of Maxwell materials combines the characteristics of a elastic material that obeys the Hooke law with a Newtonian viscous fluid. The rheological sketch suitable in describing Maxwell's models is depicted in the Figure 1. This means that the constitutive relationship, associating the total deformation ε with the stress σ is defined by the following differential equation:

$$\frac{d\varepsilon}{dt} = \frac{1}{E}\frac{d\sigma}{dt} + \frac{\sigma}{\eta}, \tag{1}$$

where η is the viscoelastic coefficient and E is the modulus of elasticity.

This differential equation can reproduce a lot of particular results depending on the boundary conditions and the kind of the load/deformation program. In the following some interesting conditions are analyzed.

2.2. Cold Flow or Creep test

Here it is considered the solution of equation (1) when the material is submitted to the constant stress σ_0. This is the condition imposed to a creep test. Then, in this case

$$\frac{\boldsymbol{d}\sigma}{\boldsymbol{dt}} = 0 \qquad \Rightarrow \qquad \frac{\boldsymbol{d}\varepsilon}{\boldsymbol{dt}} = \frac{\sigma_0}{\eta} \tag{2}$$

Consequently, the solution of the equation is given by:

$$\varepsilon = \frac{\sigma_0}{\eta}\boldsymbol{t} + \boldsymbol{C_o} \tag{3}$$

In this model, the combination of a linear spring in series with a linear damp, produces at the initial time an instantaneous elastic deformation. This initial condition allows determining C_0, the constant of integration that appears in equation (3). That is

$$\boldsymbol{C_o} = \frac{\sigma_0}{\boldsymbol{E}} \tag{4}$$

and the function that defines deformation along the time is described by the equation:

$$\varepsilon = \frac{\sigma_0}{E}\left(\frac{t}{t_\eta} + 1\right), \tag{5}$$

where t_η represents the relaxation time, being defined by :

$$t_\eta = \frac{\eta}{E} \tag{6}$$

The Figure 2 shows the creep response of Maxwell material by considering different relaxation times. It is important to observe that, with the increase of the relaxation time, this viscous material has a tendency to behave as an elastic linear material, or equivalently, the deformation remains constant along the time and equal to the initial instantaneous deformation.

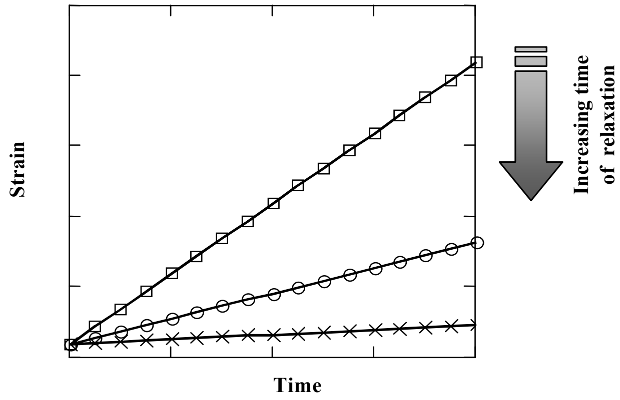

Figure 2. Maxwell model - creep response for different relaxation times

2.3. Prescribed deformation test

This sub-section describes the Maxwell material behavior when it is submitted to a controlled deformation process, as the defined by the equations (7) and (8) and shown in Fig 3:

$$\varepsilon(t) = \varepsilon_m \frac{t}{t_m} \qquad 0 \le t \le t_m \tag{7}$$

$$\varepsilon(t) = -\frac{(t - t_m)}{t_f - t_m}\varepsilon_m + \varepsilon_m \qquad t_m \le t \le t_f \tag{8}$$

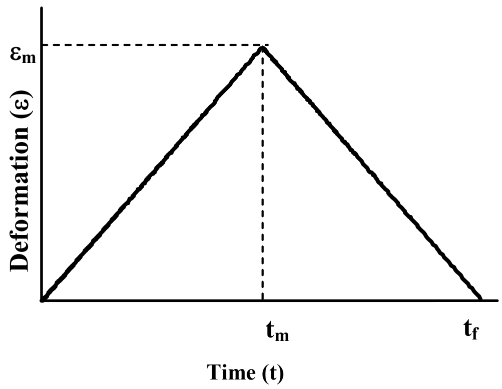

Figure 3. Controlled Deformation Program

In this case, the solution of the differential equation (1) with initial condition $\sigma(0) = 0$, gives :

$$\sigma(t) = E\,\varepsilon_m \frac{t_\eta}{t_m}\left(1 - e^{\frac{t}{t_\eta}}\right) \qquad 0 \le t \le t_m \tag{9}$$

$$\sigma(t) = E\,\varepsilon_m \frac{t_\eta}{t_m}\left[\left(\frac{t_f}{t_f - t_m}\right)e^{\frac{t_m - t}{t_\eta}} - e^{\frac{t}{t_\eta}} + \frac{t_f}{t_f - t_m}\right] \qquad t_m \le t \le t_f \tag{10}$$

The resulting stress versus strain function is depicted in Figure 4 by considering different values of t_η and E.

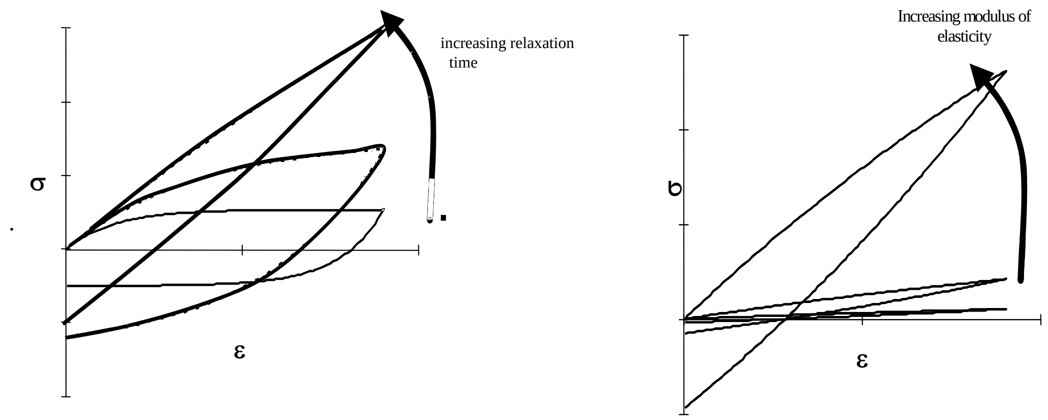

Figure 4. Maxwell Model – Stress x Strain

3. INVERSE METHOD - LEVENBERG-MARQUARDT

This section describes the optimization technique adopted in order to estimate the viscoelastics parameters of the composite material. The technique is based on the adjustment of coefficients in an optimization process in which the objective function is minimized with respect to the viscoelastic constants. Through the Levenberg-Marquardt method (Özisik and Orlande , 2000), the values are sought for the viscoelastic parameters that provide the best agreement between the stress obtained from experimental procedures and those ones assessed from the analytical model, here defined by the Maxwell model.

The objective function is defined by the sum of squared relative differences between each experimental stress and its corresponding theoretical prediction, or

$$\mathbf{S}(E,\eta) = \sum_{i=1}^{n} \left(\sigma_{\exp}(t_i) - \sigma_a(t_i, E, \eta)\right)^2 \quad , \tag{11}$$

where $\sigma_{\boldsymbol{exp}}(t_i)$ represents the experimental stress values at each instant of the controlled deformation test. The variable $\sigma_{\boldsymbol{a}}(t_i,E,\eta)$ is the value of stress obtained from the analytical model and, here, defined by the equations (9) and (10) .

The minimum values in (11) are those ones that satisfy the following optimality condition:

$$2\left[-\frac{\partial \mathbf{S}}{\partial \mathbf{P}}\right]^T [\boldsymbol{\Delta\sigma}] = 0 \quad , \tag{12}$$

where **P** is a vector in which the components are *E* and η. The vector $\boldsymbol{\Delta\sigma}$, has *n* components representing the difference $\sigma_{exp}(t_i) - \sigma_a(t_i,E,\eta)$, calculated at the time t_i. It means

$$\mathbf{P} = \begin{bmatrix} E \\ \eta \end{bmatrix} \qquad \boldsymbol{\Delta\sigma}(E,\eta) = \begin{bmatrix} \sigma_{\exp}(t_1) - \sigma_a(t_1, E, \eta) \\ \vdots \\ \sigma_{\exp}(t_n) - \sigma_a(t_n, E, \eta) \end{bmatrix} \qquad \frac{\partial \mathbf{S}}{\partial \mathbf{P}} = \begin{bmatrix} \dfrac{\partial \mathbf{S}(t_1)}{\partial E} & \dfrac{\partial \mathbf{S}(t_1)}{\partial \eta} \\ \vdots & \vdots \\ \dfrac{\partial \mathbf{S}(t_n)}{\partial E} & \dfrac{\partial \mathbf{S}(t_n)}{\partial \eta} \end{bmatrix}$$

Therefore, the Jacobian matrix's components are obtained by

$$\mathbf{J}(\mathbf{P})_{i,j} = \left[\frac{\partial \mathbf{S}(\mathbf{P})_i}{\partial \mathbf{P}_j}\right] = \left[\frac{\sigma_a(t_i, E, \eta)}{\partial P_j}\right] \quad . \tag{13}$$

By considering the Taylor's expansion for the analytical stress function written as

$$\sigma_a(\mathbf{P}^{k+1}, t_i) = \sigma_a(\mathbf{P}^k, t_i) + \sum_{j=1}^{2} \left[\mathbf{J}^k\right]_{i,j} (\mathbf{P}_j^{k+1} - \mathbf{P}_j^k) \quad , \tag{14}$$

by substituting (14) at (12) and solving the resulting linear system, the expression that defines the interactive process is obtained

$$\mathbf{P}^{k+1} = \mathbf{P}^k + \left[(\mathbf{J}^k)^T \mathbf{J}^k\right]^{-1} (\mathbf{J}^k)^T \left[\Delta\sigma(\mathbf{P}^k)\right] \tag{15}$$

The viability of the method depends on the non-singularity of the matrix J^TJ. This condition is verified if the objective function is convex. As the convexity of objective function can not be guaranteed in the practical applications, Levenberg-Marquardt proposed the disturbance of the matrix J^TJ to assure its positiveness and, consequently, the existence of the inverse matrix. So the new formula for defining the iterative process is

$$\mathbf{P}^{k+1} = \mathbf{P}^k + \left[(\mathbf{J}^k)^T \mathbf{J}^k + \mu^k \mathbf{\Omega}^k\right]^{-1} (\mathbf{J}^k)^T \left[\mathbf{\Delta\sigma}(\mathbf{P}^k)\right] \quad , \tag{16}$$

where μ^k is a small positive parameter, established in the beginning of the iterative process. The Ω^k matrix is diagonal and, in this work, defined by

$$\mathbf{\Omega}^k = diag[(\mathbf{J}^k)^T \mathbf{J}^k] \tag{17}$$

4. PARAMETERS IDENTIFICATION

As mentioned before, to completely characterize the Maxwell material, it is necessary to identify the modulus of elasticity E and the viscosity η. In order to validate the proposed inverse method two classical experiments, the tension and the creep testing, were performed before the controlled strain essay. The values obtained in these preliminary tests were adopted as initial values for E an η in the iterative identification process.

The experiments samples's were made with a PTFE sheet reinforced with silica particles with 2.09 mm of mean thickness and density equal to 2.21 g/cm^3. Ten specimens with 12.7 mm of length were tested in a controlled environment room at 25^{o}C and 53% of relative humidity. The tests were carried out in an INSTRON® universal materials testing machine, presenting 116 mm spacing between jaws and drive at 300 mm/min.

4.1. Tension testing

In order to determine the modulus of elasticity from the tension testing, only strains values lower than the maxim deformation strain predicted at controlled strain test was considered, this means approximately 1.7% (Fig 5).

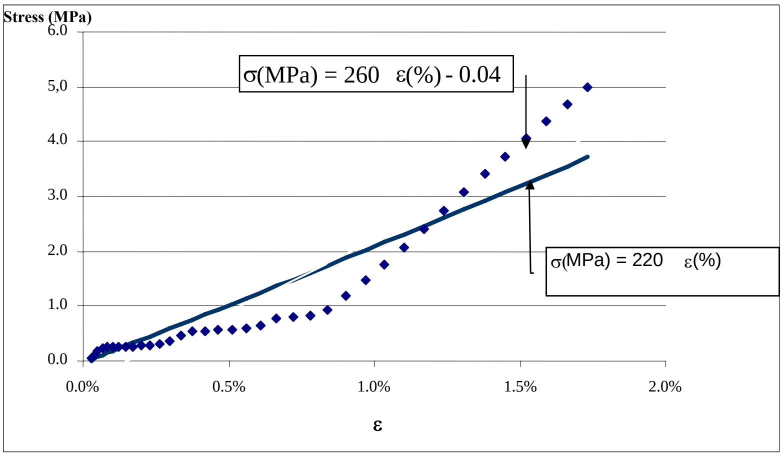

Figure 5. Stress x Strain (ε < 1.7%)

The tensile stress shows the initial estimation (between 223 MPa and 259 MPa) is reasonable modulus of elasticity to initiate the interactive process.

4.2. Creep test

The initial value for viscosity was estimated by considering the Maxwell model defined by equation 5, an experimental cold flow stress 7.2 MPa and the modulus of elasticity 240 MPa (average of the data tensile stress test, calculated at 4.1) and $\varepsilon_0 = 3\%$. It is important to mention that the results from creep tests did not allow to accurately assessing the relaxation time due to the high degree of uncertainties observed in the strain's measures during the creep tests.

In that case, trying to estimate the initial value to the iterative algorithm adopted in identification process, the model was analyzed by imposing different values to the relaxation time and observing the resulting stress-strain curve (Fig 6). The identification test was carried out with the three initial values presented in the graph, or better, 30 h, 0.03 h and 0.0003 h.

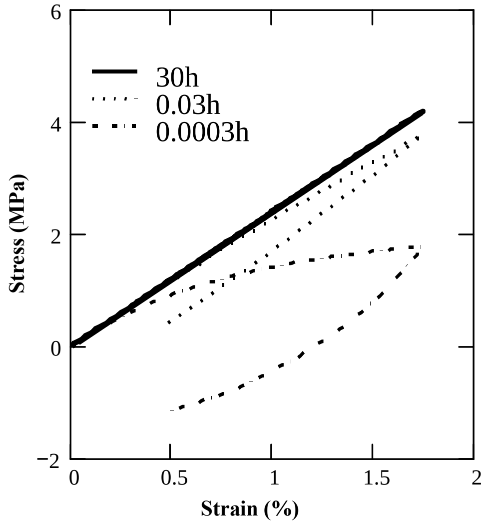

Figure 6. Initial viscosity estimated by Maxwell model from the creep test.

3.5. Final parameters identification - E and η

The Levenberg-Marquardt interactive procedure, described at section 3, was performed by considering the initial values obtained as described in the previous section. The best results was achieved with E=240 MPa e t_η=108s (0.03h). The algorithm converged in 6 iteractions and the following values were obtained:

$$E = 218\,MPa \qquad \eta = 13GPa.s$$

For these values, the time for relaxation time can be estimated as

$$t_\eta = 59s$$

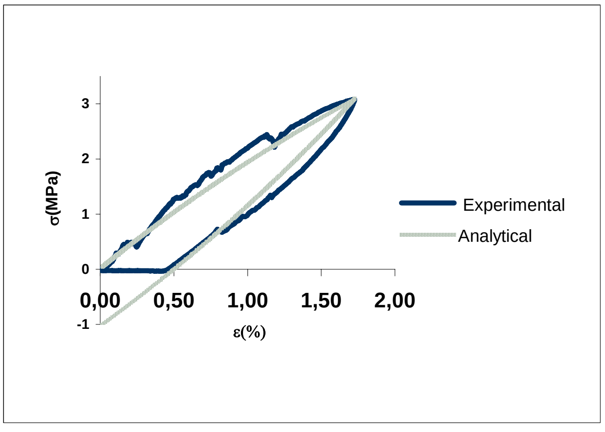

Figure 7. Stress x Strain - Analytical solution after identification process

In order to confirm the quality and validate the results it is important to verify the graphics in figures 7 and 8, where the experimental results were compared to the analytical results.

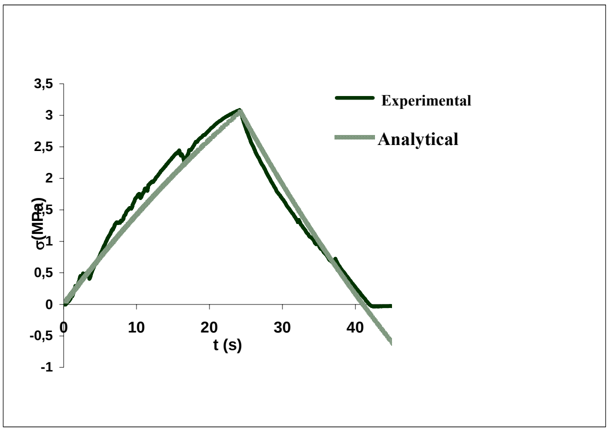

Figure 8. Stress x time - Analytical solution after identification process

6. CONCLUSION

The results of elasticity modulus and the viscosity estimated by the proposed methodology are within the limits presented in literature and are comparable with that one obtained with other experimental results. It was observed that Maxwell model represents appropriated behavior for this material.

The proposed methodology was successful in identifying the consistent values for properties of the viscoelastic material studied. It also proved to be a helpful, simple and reliable tool to analyze the mechanical behavior of viscoelastic materials. Nevertheless, the efficient convergence of Levenberg-Marquardt technique relies on the choice of initial parameters. They must be coherent with the order waited for the searched parameters, mainly the initial value of the viscosity parameters .

It is important to carry out stress relaxation test to confirm the accuracy of the results, mainly in the relaxation time. Additionally, it necessary to perform new experiments considering other strain rate and environment conditions so as to characterize the material definitely.

7. ACKNOWLEDGEMENTS

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