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Experimental and numerical methods for characterization of time-dependent materials

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Abstract: The paper presents a brief overview on experimental approaches accompanied with corresponding numerical procedures for characterization of time-dependent properties of viscoelastic materials. Upgraded experimental procedure is proposed for characterization of material creep and relaxation properties from a single experiment, which are usually measured separately. In addition, newly developed numerical approaches for the construction of unique master curves, and for the analysis of time-dependent materials fatigue properties are presented. The newly developed experimental-numerical methodologies are aimed to optimize process of characterization, and give complete information on time-dependent material behaviour.

Keywords: Viscoelasticity; Time-dependent; Creep; Relaxation; Time-temperature superposition principle; Mechanical spectrum

1. Introduction

When using time-dependent materials for designing of structural elements exposed to different loading conditions such as temperature, pressure, humidity and/or mechanical loading it is important to know how materials' mechanical properties might change during operation of the product.

In the case of time-dependent materials the linear stress-strain relations known from elasto- and plasto-mechanics are replaced by convolution integrals, while material constants (moduli) with time-dependent material functions. There are 21 material functions that describe viscoelastic behaviour of materials in solid state, seven of them are determined by static, and 14 by dynamic methods of measurements. There are two fundamental modes of deformation, bulk deformation (relating to the changes in size), and shear deformation (relating to the changes in shape) that must be considered to be independent of one another [1]. For many time-

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dependent materials their bulk properties change several orders of magnitude less than corresponding shear properties during the same time period [1]. In this respect, shear creep compliance and shear relaxation modulus are, in practice, the most commonly measured time-dependent material functions.

2. Experimental characterization of time-dependent material properties

Time-dependent material functions are defined as a time-dependent response of the material to the step loading in terms of stress or strain. When the step loading is a constant stress the response of the material is strain, which increases in time. The observed process is commonly called as creep of the material. Whereas, when a constant strain is used as a step loading material's stress state will diminish with time. This process is commonly known as relaxation process of the material.

When the loading conditions are given as a constant shear stress, τ_0, the stress-strain relation in the form of a convolution integral simplifies to an algebraic equation from which one may calculate the time-dependent shear creep compliance, $J(t)$, as follows

$$J(t) = \frac{\gamma(t)}{\tau_0}, \tag{1}$$

where t represents time, and $\gamma(t)$ the shear strain response as function of time.

When the loading conditions are given as a constant shear strain, γ_0, simplified integral stress-strain relation yields material time-dependent shear relaxation modulus, $G(t)$, in the form of

$$G(t) = \frac{\tau(t)}{\gamma_0}, \tag{2}$$

where $\tau(t)$ is the shear stress time-dependent response.

The creep compliance and the relaxation function require, in principle, two independent experiments [2]. Alternatively, one of the material functions can be determined experimentally; while the other may be obtained through the interconversion method [1], which requires inverse solution of an integral equation, which is known to be an ill-posed problem.

As an alternative to the traditional way of material creep and relaxation characterization Nikonov et al. [3] presented the theoretical framework for development of a new apparatus, which allows simultaneous determination of the creep compliance and the relaxation modulus from a single experiment. The underlying idea of the experiment is to load the specimen with a Hookean spring which initiates both, creep and relaxation, processes simultaneously. Experiments may be carried out in uniaxial extension, as shown in Fig. 1, or in shear, to obtain the corresponding time-dependent relaxation moduli and creep-compliances,

respectively. Without any loss of generality, we will discuss the physical principle of the experiment for the case of uniaxial extension.

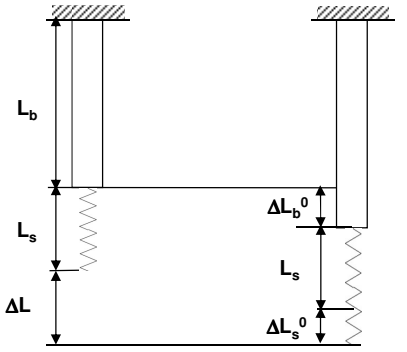

Fig. 1. A viscoelastic bar loaded instantaneously with a spring.

The spring is instantaneously deformed by a constant tensile deformation, ΔL . The lower end of the spring is then kept in a fixed position. Due to the applied load the viscoelastic bar will elastically deform to the initial extension, ΔL_b^0 , while leaving the initial deformation of the spring to be ΔL_s^0 . The applied deformation of the bar will initiate the relaxation process in the material. Whereas, the remaining deformation of the spring will impose a force acting upon the viscoelastic bar (specimen), causing its time-dependent deformational process, i.e, creep process. Hence, both, creep and relaxation process run in parallel. Over time the creep process will decrease the deformation of the spring, which, in return, will decrease the force acting on the bar, etc., until the material reaches the equilibrium state.

The key issue which enables the simultaneous determination of creep and relaxation behaviour is the fact that the force on the viscoelastic bar is linearly related to the deformation of the bar. Measurement of force over a period of time yields inverse problem for creep, while measurement of deformation yields equivalent inverse problem for relaxation. Furthermore, it is not necessary to measure both force and deformation, since they are linearly related to each other. The inverse problems for creep and relaxation may be solved analytically by means of Laplace transform.

3. Numerical approaches for characterization of time-dependent material properties

Presented approaches were developed by the research group of the Centre for Experimental Mechanics, Faculty of Mechanical Engineering, University of Ljubljana, and serve as additional tool for post-processing of experimentally determined creep and/or relaxation data. Developed methodologies may

significantly contribute to the complete characterization of time-dependent materials.

3.1. CFS method for constructing master curve of time-dependent material property

In order to obtain complete information on time-dependent creep compliance or relaxation modulus, properties must be measured over a very long time interval (of several years). Since this is not acceptable from the practical and economical point of view, material characterization is usually done through measurements of response function segments at selected temperatures and/or pressures. Measured segments are then superposed into a master curve by using the time-temperature-pressure superposition principle [1, 4]. Each segment is measured within a certain time interval, the so called experimental window. Upper time limit of the experimental window is for practical reason usually limited to $10^4\,s$, which is approximately $3h$.

By applying the time-temperature superposition principle, one can shift measured segments along the logarithmic timescale to generate a complete creep compliance or relaxation modulus master curve at a selected reference temperature, T_{ref}. In practice, the shift factors are commonly determined by so-called manual shifting performed with an aid of a spreadsheet program. This approach is not unique and strongly depends on a person who performs the shifting. Hence, two different persons will most certainly obtain two different results.

Recently, Gergesova et al., [5], developed a new closed form methodology for automated construction of master curves, called the Close Form Shifting (CFS) algorithm. The CSF algorithm constructs an unique master curve, and, therefore, completely removes weaknesses of other shifting methods. The proposed mathematical formulation of the shifting procedure takes into account that material functions measured at two different temperature and/or pressure boundary conditions represent behaviour of the material at two different thermodynamic states, which differ in the corresponding Gibbs free energy $\Delta W = V\,dP - S\,dT$, where W denotes Gibbs free energy, V volume of the specimen, S the internal entropy of the material, while (T_0, P_0) and (T_k, P_k) represent two selected equilibrium thermodynamic states at which the corresponding segments of the material function have been measured. The rate at which mechanical energy is absorbed per unit volume of a viscoelastic material at a given boundary conditions (T_k, P_k) is equal to the stress power, i.e., the rate at which work is performed. The stress power at time t is

$$\frac{dW(t,T_k,P_k)}{dt} = \sigma(t,T_k,P_k)\frac{d\varepsilon(t)}{dt}\,. \tag{3}$$

The absorbed mechanical energy causes material inherent structural (molecular) rearrangements during the relaxation or creep experiment. Thus, any two segments of the material function, measured at the reference state (T_0, P_0), and any other

selected state (T_k, P_k), that need to be superimposed (shifted) into a master curve should have the same stress power at all points of the superimposing interval. This criterion may be expressed as

$$\frac{dW(t, T_0, P_0)}{d\log t} = \frac{dW(t/a_k, T_k, P_k)}{d\log t} \quad . \tag{4}$$

The condition in Eq. (3) is fulfilled when overlapping area A between the two segments is equal to zero, as shown in Fig. 2.

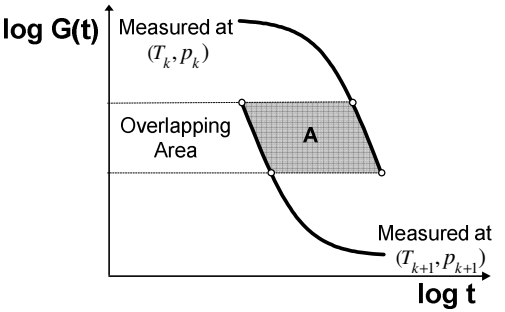

Fig. 2. Schematics of the CFS shifting procedure.

Based on these considerations the CFS algorithm for calculation of time-temperature shift factors was derived. The maximal error that CFS shifting contributes to the response function master curve is at least 10 times smaller than the underlying experimental error.

3.2. Windowing algorithm for mechanical spectrum calculations

Material functions are transfer functions that correspond to a selected excitation. From this perspective, the relaxation spectrum, $H(\tau)$, and the retardation spectrum, $L(\lambda)$, represent two transfer functions, which represent structural rearrangements at molecular level caused by the step-like strain and stress excitations, respectively. Linear viscoelastic theory reflects physical intuition in that it demands the spectra to be line spectra rather than continuous functions because it stands to reason that viscoelastic behaviour should consist of a superposition of independent mechanisms [6]. Discrete spectra, as well as continuous spectra cannot be determined experimentally. It can only be determined from the experimental responses by using proper computer algorithm. Once a line spectrum is available, any response curve within the same representational group (i.e., within relaxation or creep behaviour) can be generated readily. Discrete spectrum is an excellent form in which to store the results of an experiment performed to fully characterize the linear viscoelastic behaviour of a given material [6].

Calculations of mechanical spectrum from a material response function requires solving of mathematically ill-posed problem. One of the efficient algorithms for calculating mechanical spectrum was proposed by Emri and Tschoegl [7, 8], i.e., the "Windowing Algorithm" (WA). WA recognizes that in the calculation of a spectrum line from a given response function only certain regions of that function contribute to the instabilities that may affect the calculation of the line, while not even being essential for the computation. The algorithm localizes and excludes the trouble-causing regions without impairing the determination of the spectrum. The advantage feature of the algorithm is that it avoids negative intensities of spectrum lines, which are not acceptable from the physical point of view. Besides this, it calculates each spectrum line from a subset of the entire set of experimental data. This subset is the 'window', through which the algorithm scans the data set and it is from this window that the spectrum lines are obtained. Generated windows effectively constitute band-pass filters that move along the abscissa. "Windowing" is the feature of the algorithm by which it gets around the problem of ill-posedness.

3.3. Strain accumulation approach for analysis of fatigue of time-dependent materials

Time-dependent behaviour of viscoelastic materials strongly affects their functionality and durability. When viscoelastic materials are cyclically loaded, there are two basic competitive mechanisms that can cause material failure; for higher frequencies the problem of local heating occurs due to the production of hysteresis energy during fatigue, while at lower frequencies fatigue failure is essentially caused by localized strain accumulation process that leads to the crack initiation, followed by its propagation. Within this subsection we are presenting brief overview of the methodology for analyzing the strain accumulation process.

Developed strain accumulation model considers cyclic loading conditions in terms of a shear stress, $\tau(t)$, as schematically presented in Fig. 3a. Figure displays loading conditions within one cycle that consist of loading phase with the shear stress, τ_0, followed by the unloading phase. Fig. 3b shows the time-dependent response of the viscoelastic material in one loading cycle under specified loading conditions.

At certain conditions, that are implied by the frequency of cyclic loading and ration between the time of loading and unloading phase, the retardation process between two loading cycles cannot be fully completed to a strain-free state. Therefore, material enters the next loading cycle with a strain residual. Strain starts to accumulate and consequently leads to the hardening of material, crack formation, and ultimately to its failure.

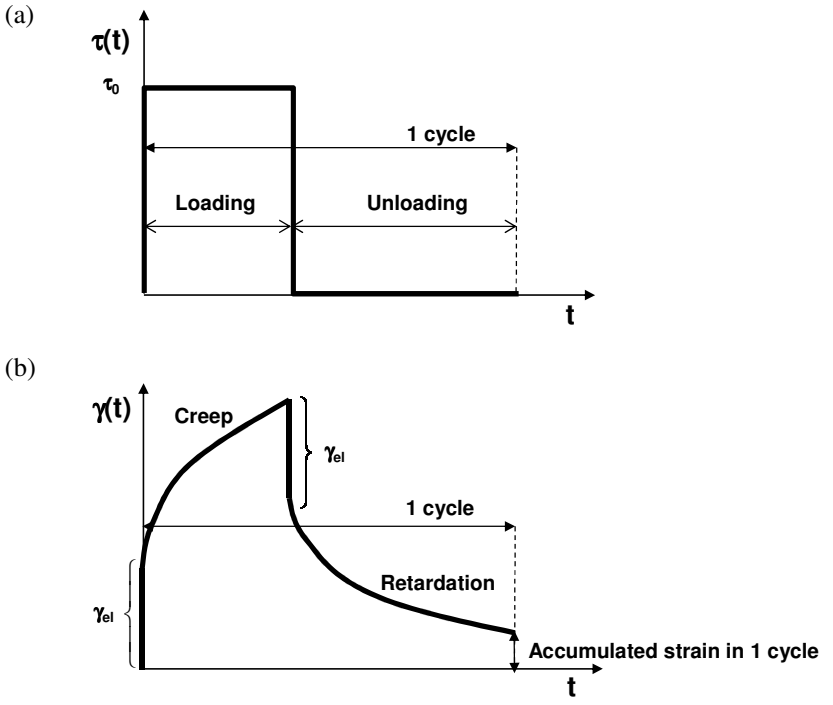

Fig. 3. Schematics of (a) loading conditions in terms of the shear stress, and (b) time-dependent response of viscoelastic material in terms of the shear strain within one cycle of cyclic loading.

As it was shown in the papers [9, 10], there exist critical cyclic loading conditions at which the strain accumulation process in the material is the most intensive. Critical loading conditions strongly depend on the time-dependent material behaviour expressed through mechanical spectrum. Due to this fact mechanical spectrum of viscoelastic material, from which the product is made of, presents the most important material function for analysing the process of strain accumulation. Proposed model takes in consideration the time-dependent response of the material by introducing material spectrum, and enables determination of critical loading frequencies, that cause the most intensive strain accumulation process. In this manner it may serve as numerical tool for predicting durability of cyclically loaded viscoelastic materials.

4. Conclusions

Paper presents an overview of main characteristics of material viscoelastic properties by pointing out newly developed approaches for gaining more comprehensive information on time-dependent behaviour of materials. It is important to emphasize that presented methodologies are based on valid physical laws, and have in this perspective strong advantage to give reliable results within the limits of governing physics. Therefore, they may be utilized as additional tools and

guidelines for preparation of newly-establishing standards for characterization of time-dependent materials.

References

[1] Tschoegl N.W., *The Phenomenological Theory of Linear Viscoelastic Behavior*, (Springer-Verlag, Berlin, 1989). ISBN 3-540-19173-9.

[2] Ferry J..D., *Viscoelastic properties of polymers, 3rd ed*., (Wiley, New York, 1980). ISBN 0-471-04894-1.

[3] Nikonov A., Davies A.R., Emri I., "The determination of creep and relaxation functions from single experiment," *Journal of Rheology*, **49**(6), pp. 1193-1211 (2005). ISSN 0148-6055.

[4] Aklonis J. J., MacKnight W. J., *Introduction to Polymer Viscoelasticity, 2nd ed.*, (John Wiley and Sons, New York, 1983). ISBN 0-471-86729-2.

[5] Gergesova M., Zupančič B., Saprunov I., Emri I., "The closed form t-T-P shifting (CFS) methodology," *Journal of Rheology*, **55**, pp. 1-17 (2011). ISSN 0148-6055.

[6] Knauss W. G., Emri I., Lu H., "Mechanics of polymers: viscoelasticity," in *Handbook of experimental solid mechanics*, William N., ed. (New York: Springer, cop. 2008, pp. 49-95). ISBN 978-0-387-26883-5

[7] Emri I., Tschoegl N. W., "Generating Line Spectra from Experimental Responses. 1. Relaxation Modulus and Creep Compliance," *Rheologica Acta*, **32**(3), pp. 311-321 (1993). ISSN 0035-4511.

[8] Emri I., Tschoegl N. W., "Determination of Mechanical Spectra from Experimental Responses," *International Journal of Solids and Structures*, **32**(6-7), pp. 817-826 (1995). ISSN 0020-7683.

[9] Emri I., Kramar J., Hribar A., Nikonov A., Florjančič U., "Time-dependent constitutive modeling of drive belts - I. The effect of geometry and number of loading cycles," *Mechanics of Time-dependent Materials*, **10**(3), pp. 245-262 (2006). ISSN 1385-2000.

[10] Zupančič B., Emri I., "Time-dependent constitutive modeling of drive belts – II. The effect of the shape of material retardation spectrum on the strain accumulation process," *Mechanics of Time-dependent Materials*, **13**(4), pp. 375-400 (2009). ISSN 1385-2000.