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Study of stress relaxation in polytetrafluoroethylene composites by cylindrical macroindentation

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ABSTRACT

In the present work, the time-dependent mechanical behavior of three materials from the fluorocarbon family, including PTFE without filler, 15 vol.% regenerated graphite particles-filled PTFE, and 32 vol.% carbon and 3 vol.% graphite particles-filled PTFE, was investigated using cylindrical macroindentation. Indentation relaxation experiments with a cylindrical flat-tip indenter having a diameter of 1 mm were performed at three different strain levels. The generalized Maxwell model in terms of the Prony series and a time-dependent solution based on the power-law creep equation were used to model the viscoelastic response and to extract the time-dependent properties. The stress relaxation properties were found to improve with the addition of fillers. The unfilled PTFE exhibited the lowest stress–relaxation properties, whereas the 32 vol.% carbon and 3 vol.% graphite particles-filled PTFE composite showed the highest stress–relaxation properties.

1. Introduction

Polymers and polymer-based composites exhibit a wide range of mechanical behaviors from that of an elastic solid to that of a viscous liquid, depending on the loading and environment conditions. In order for manufacturers to have confidence in their products made of polymer-based composites, they need to know how various loading and working conditions affect the performances over a period of time.

Creep and stress relaxation are the main time-dependent deformation mechanisms for polymers and polymer-based composites that can cause concern [1,2]. Unlike in metals, in polymeric materials relaxation is observed even at small strains while creep can develop at relatively low stress levels even at room temperature. Therefore, the problem of predicting the time-dependant properties of structural components is of main concern for the polymer manufacturing industry.

In order to predict the time-dependent behavior of polymers and polymer-based composites one should determine the viscoelastic material functions. Traditionally, the creep compliance and relaxation functions are modeled by using the Prony series [3–6], which gives a straightforward fitting approach to the experimental data while offering exact numerical conversion to other linear viscoelastic material functions. To reduce the number of material parameters associated with the Prony series and to improve the prediction for the polymer behavior, different approaches were also developed, for example: the fractional derivative models [7], the direct inversion of the Fourier integral [8], and the direct estimation for fast relaxation [9,10]. Moreover, since creep and stress relaxation are both aspects of the same time sensitive behavior of materials, interrelations of creep and stress relaxation were also proposed in order to predict the creep from stress relaxation and the other way around. For example, Arutyuyan [11] proposed a model in which the nonlinear creep is written in the form of a non-linear differential equation, Findley et al. [12] used a multiple integral formulation in order to interrelate creep and relaxation, while Ashby and Jones [13] and Xu and Hou [14,15] proposed interrelations based on the power-law creep equation.

The properties of polymers and polymer-based composites are very sensitive to manufacturing methods, and, generally, the manufacturing conditions for polymeric parts as well as the geometry differ from that of laboratory specimens. Moreover, due to the complex geometry of the parts, they have locally different properties which can affect the deformation behavior of the overall parts. Thus, in many situations, for accurate predictions, it is recommended that the material properties that define the time-dependent behavior of polymeric parts to be determined directly on the final parts instead of on the laboratory specimens. However, linking the laboratory properties to the properties of the manufacturing parts is a subject of great complexity.

The conventional creep or relaxation testing methods cannot be applied on small size samples or directly on the final components.

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Moreover, they are usually time consuming and require the preparation of numerous samples. As an alternative to conventional creep and relaxation tests (uniaxial tension or compression tests) one can consider the instrumented indentation technique [16].

An indentation test is a method used to evaluate material properties based on the relationship between load and displacement [16]. The instrumented indentation test has the following advantages: (i) it is a nondestructive method and allows local properties measurements; (ii) it requires small samples with simple geometry for the analysis; (iii) it can be applied when the analyzed materials are not available in the form of conventional tests, i.e., insufficient study material, special environment testing conditions or when the material cannot be firmly clamped as is the case of gels.

From micro- and nanoindentation tests, one can extract the hardness and elastic modulus of both hard and soft materials, including substrates and thin films [16–19], the yield strength and strain-hardening exponent [20], and the time-dependent properties of polymeric materials [21–25].

However, applications of sharp micro- and nanoindentation techniques (Vikers, Knoop, or Berkovich tips) to polymeric materials which possess viscoelastic and also viscoplastic properties under load can lead to errors when they are applied to extract bulk properties. Generally, the method used to extract the hardness and elastic modulus from an indentation test is based on the Oliver and Phar method [26] which takes into account the changing contact area during unloading. This technique provides higher values for bulk properties than what will be found by conventional methods. When sharp indentation is performed on polymeric materials, the most important drawback is related to the fact that this analysis is time-independent assuming that the unloading response is purely elastic while the mechanical behavior of polymers is time-dependent [27–29]. Another drawback when using sharp (pyramidal and conical) indenters is the determination of the true contact depth and the corresponding true contact area. The true contact area is influenced by the pile-up and sink-in which is primarily affected by the plastic deformation of the material during the loading stage. Thus, the knowledge of the relationship between the indentation load and the true (projected) contact area is essential to extract mechanical properties by instrumented indentation.

On the other hand, the measurement of the viscoelastic function by sharp indentation is impeded by the time-independent instantaneous plastic deformation and the time-dependent irreversible deformation developed under the indenter during the loading stage which results in an increase in indentation depth (creep) or in a decrease in load (stress relaxation).

Unlike sharp indenters, cylindrical indenters rigorously provide constant contact area; thus, the stress linearly increases with indentation displacement [30–34]. The pile-up or sink-in around the indenter has no effect on the indentation stress; therefore, the mechanical properties, including time-dependent properties, can be accurately extracted from the load–displacement–time relationship.

Given the advantages of cylindrical indentation, macroindentation tests using cylindrical flat-tip indenter could be an alternative to conventional creep or stress relaxation tests for determining the viscoelastic material functions [30–32].

In this paper, the applicability of the cylindrical indentation technique to measure the stress relaxation behavior of filled and unfilled polytetrafluoroethylene is explored. The indentation stress relaxation tests are carried out using universal testing machine equipped with a cylindrical flat tip indenter, having a tip diameter of 1 mm. The results of the indentation relaxation experiments are analyzed using the generalized Maxwell model and the power-law steady-state creep equation in order to derive the relaxation functions.

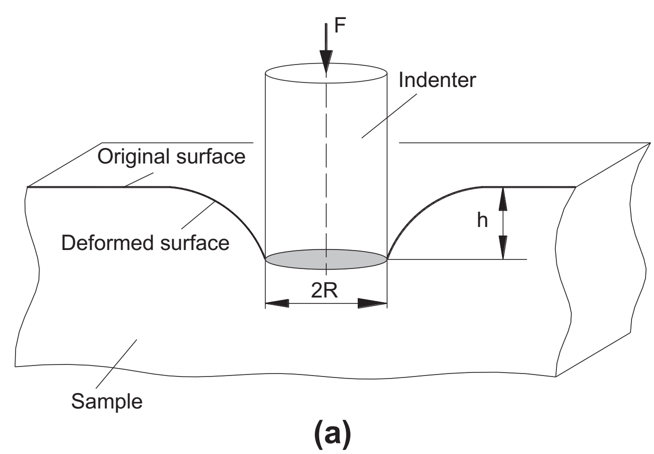
2. Analytical modeling and basic assumptions

2.1. Indentation using a cylindrical flat-tip indenter

In general, indentation tests are based on the local deformation produced by an indenter upon application of a given load [16,35,36]. During the indentation test the indentation displacement versus load is continuously measured and, based on the load–displacement curve, various mechanical properties may be extracted. During the indentation with a cylindrical flat-tip indenter (to which we refer simply as "cylindrical indenter"), the contact area remains constant (see Fig. 1a) and the contact pressure increases linearly with the applied load producing a steady-state rate of penetration as shown in Fig. 1b. Moreover, the pile-up and sink-in around the indenter has no effect on the indentation stress.

The load–displacement curve obtained by cylindrical indentation exhibits several characteristic stages of deformation, as shown in Fig. 1b: (i) the first linear region dominated by elastic deformation where the load varies linearly with the displacement, (ii) a transient region which corresponds to the yield point, and (iii) the second linear region, the so-called hardening region, dominated by plastic deformation. The slope of the first linear region of the indentation curve defines the so-called indentation modulus. The load level at which the material loses its linear behavior defines the critical transition force, F_Y. This point can be related to the load at which plastic deformation initiates, i.e. yield stress.

The tensile yield stress, σ_{YS}, may be related to the mean indentation stress (p_m) or hardness (H) of the material through the following



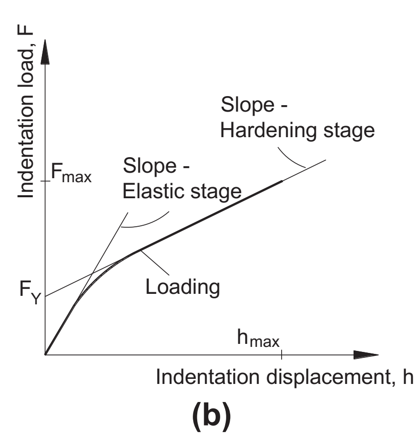


Fig. 1. Cylindrical flat tip indentation scheme (a) and load–displacement curve (b).