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Property Modelling

Fading memory of deformation history in carbon black-filled thermoplastic elastomers

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ABSTRACT

Observations are reported on a carbon black–reinforced thermoplastic elastomer in multistep uniaxial tensile cyclic tests with a mixed deformation program (oscillations between maximum elongation ratios k_{max} and various minimum stresses σ_{min} with k_{max} monotonically increasing with number of cycles n). Fading memory of deformation history is demonstrated: when specimens are subjected to two loading programs that differ along the first $n-1$ cycles of deformation and coincide afterwards, their stress–strain diagrams become identical starting from the nth cycle. A constitutive model is developed in cyclic viscoplasticity with finite deformations, and its adjustable parameters are found by fitting the observations. Ability of the stress–strain relations to describe the fading memory phenomenon and to predict the mechanical response of polymer composites in multi-step cyclic tests with large strains is confirmed by numerical simulation.

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1. Introduction

This paper is concerned with experimental investigation and constitutive modeling of the mechanical behavior of a carbon black–reinforced thermoplastic elastomer (TPE) subjected to multi-step tensile cyclic deformation with finite strains.

Thermoplastic elastomers belong to an important class of polymers that combine mechanical properties of rubbers (ability to withstand large deformations without noticeable changes in their structure) with high-speed processability and recyclability of thermoplastics [1]. This study focuses on the viscoplastic response of composite Thermolast V TV5LVZ (Kraiburg TPE GmgH, Germany), a thermoplastic elastomer reinforced with a large amount (60–70 wt%) of carbon black. Its matrix consists of a hydrogenated styrene block copolymer (HSBC)-based thermoplastic elastomer with addition of polypropylene segments. Due to chemical structure of the matrix and high concentration of filler, Thermolast V demonstrates strong thermal and chemical resistance, which makes it suitable for use as a sealing material in low temperature proton exchange membrane fuel cells.

The mechanical behavior of TPEs has attracted substantial attention in the past decade ([2–11], to mention a few). The time- and rate-dependent responses of TPEs reinforced with carbon black have recently been analyzed in Refs. [12–14]. Previous studies in cyclic viscoelasticity and viscoplasticity of particle-reinforced TPEs concentrated on conventional loading–unloading tests with a mixed program: oscillations between a fixed maximum strain and the zero minimum stress [15]. The objective of this work is to examine mechanical properties of TPE composites subjected to multistep cyclic tests with monotonically growing maximum strains and partial unloading (in each cycle of oscillations tensile stress is reduced to positive values that differ from cycle to cycle). An unusual feature of the mechanical response observed in these tests is fading of memory of deformation history: when elongation ratio k at the nth cycle exceeds maximum elongation ratio $k_{max}(n-1)$ at the $(n-1)$th cycle, the stress–strain diagram along the nth cycle for a preloaded specimen coincides with that for a virgin sample [not suffered previous $(n-1)$ cycles of oscillations]. Moreover, this property is independent of stresses $\sigma_{min}(k)$ $(k=1,\ldots, n-1)$ down to which unloading is performed: the stress–strain curves for two specimens subjected to cyclic deformation with various sets of minimum stresses $\{\sigma_{min}(1), \ldots, \sigma_{min}(n-1)\}$ coincide along the nth cycle. The fading memory phenomenon is typical not only for thermoplastic elastomers, but for a wider class of polymers as well. The effects of the residual orientation and high rate of cooling in injection-molded specimens have been

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investigated in experiments on semicrystalline polymers and nanocomposites with semicrystalline matrices. The results demonstrate that the mechanical response of annealed samples under cyclic deformation do not differ pronouncedly from that of virgin samples, which confirmed that the fading memory effect is not induced by residual stresses [16].

In some sense, this phenomenon resembles the Mullins effect [17,18]: when a sample subjected to loading–unloading with a fixed maximum strain is stretched above this strain, its stress–strain curve rapidly reaches that of a virgin (not subjected to cyclic preloading) specimen. The difference between our findings and observations on the Mullins phenomenon is that not only loading paths, but also retraction paths coincide of the stress–strain diagrams.

Experimental data provide a challenge for constitutive modeling: on the one hand, they reveal fading of entire memory of deformation history in cyclic tests with increasing maximum elongation ratios, while on the other, they show that under a cycle of loading–retraction down to the zero stress, substantial (comparable with maximum strain under stretching) irreversible strains are observed, which implies that some memory is preserved in TPE composites. This implies that conventional stress–strain relations for the description of Mullins' effect [19–30] are inapplicable for the analysis of fading memory, and more sophisticated constitutive equations are required. The aim of this study is to demonstrate that the fading memory effect can be described quantitatively by (i) distinguishing the loading, unloading, and reloading paths of the stress-strain diagram and (ii) presuming different mechanical responses along these paths.

The objective of this work is threefold:

1 To report experimental data in uniaxial tensile cyclic tests with finite deformations (maximum strains reach 90%) that demonstrate the fading memory phenomenon.
2 To develop constitutive equations in cyclic viscoplasticity of polymer composites and to find adjustable parameters in the stress–strain relations by fitting the observations.
3 To confirm ability of the model to describe the fading memory phenomenon and to predict the mechanical behavior of carbon black-reinforced TPE in multi-step cyclic tests by numerical simulation.

To develop a constitutive model in cyclic viscoplasticity of polymer composites, a two-step approach is applied. At the first step, stress–strain relations are derived for an individual cycle of loading–retraction by means of the Clausius–Duhem inequality. These equations involve several adjustable parameters that are treated as constants along each cycle of oscillations. At the other step, some of these parameters are allowed to evolve with number of cycles, and phenomenological relations are introduced to describe their changes driven by damage accumulation. As a measure of damage, specific plastic work is employed.

The novelty of our approach to constitutive modeling consists in the following:

1 Deformation gradient for plastic deformation is split into two components that describe irreversible deformations in the polymer matrix and filler clusters.
2 Strain energy density of a composite is treated as the sum of mechanical energy stored in the matrix (this energy depends on principal invariants of the Cauchy–Green tensor for elastic deformation) and the energy of interaction between polymer chains and filler particles (that depends on principal invariants of the Cauchy–Green tensor for plastic deformation in the matrix).
3 Under multi-step cyclic tensile deformation with increasing maximum elongation ratios $k_{\max}(n)$, the stress–strain diagram is split into three groups of segments corresponding to (i) stretching (elongation ratio k increases and engineering stress σ exceeds maximum stress $\sigma_{\max}(n-1)$ at the previous cycle), (ii) unloading (elongation ratio decreases), and (iii) reloading (k increases, but σ remains lower than $\sigma_{\max}(n)$). Plastic deformations in the matrix and inclusions of filler under stretching, unloading and reloading are described by the same differential equations, but with different coefficients.

The exposition is organized as follows. Observations in multi-step uniaxial tensile cyclic tests with finite strains are reported in Section 2. Constitutive equations in cyclic viscoplasticity of polymer composites are derived in Section 3. Adjustable parameters in the stress–strain relations are found in Section 4 by fitting the experimental data. Results of numerical simulation are presented in Section 5. Concluding remarks are formulated in Section 6.

2. Experimental procedure

Carbon black–reinforced thermoplastic elastomer Thermolast V TV5LVZ (density 1.05 g/cm^3, melt flow index 12 g/10 min at 230 °C, elongation at break 520%) was purchased from Kraiburg TPE GmgH (Germany). Dumbbell specimens for uniaxial tensile tests (ASTM standard D–638) with cross-sectional area 10.2 mm × 3.4 mm were molded by using injection-molding machine Arburg 320C.

Mechanical tests were conducted at room temperature by means of universal testing machine Shimadzu AG-X (Shimadzu Corp., Japan) equipped with an optical extensometer. Tensile force was measured by 10 kN load cell. Engineering stress σ was determined as the ratio of axial force to cross-sectional area of undeformed specimens.

The experimental program involved four series of cyclic tests. Each test was performed on a new specimen and repeated twice. Observations reveal good reproducibility of measurements: deviations between stresses measured on different specimens did not exceed 3%.

In the first series of tests, specimens were stretched with cross-head speeds $\dot{d} = 1,\ 10$ and 100 mm/min (these speeds correspond to rates $\dot{k} = 1.7 \cdot 10^{-4}, 1.7 \cdot 10^{-3}$ and $1.7 \cdot 10^{-2}\ \mathrm{s}^{-1}$, where k stands for elongation ratio) up to maximum elongation ratio $k_{\max} = 1.9$ and unloaded with the same cross-head speeds down to minimum stress $\sigma_{\min} = 0.1$ MPa (this value was chosen to avoid buckling of specimens under retraction).

Experimental stress–strain diagrams in these tests are reported in Fig. 1. The following conclusions are drawn: (i) at all cross-head speeds, loading and unloading paths are strongly nonlinear and differ pronouncedly from each other, (ii) growth of cross-head speed $\dot{d}$ induces an increase in stress σ under tension and weakly affects stress under retraction, (iii) residual strain (estimated as the tensile strain reached after unloading down to the zero stress) is independent of cross-head speed. An increase in temperature can affect the mechanical response of thermoplastic composites. However, its role in our experiments is negligible. Hysteresis energy at the highest strain level was calculated as the area between loading and unloading paths in Fig. 1. Because of the energy dissipation, the increase in of temperature is less pronounced (due to the heat exchange through the lateral surface of the sample), which means that deformation of a sample can be treated as isothermal.

The other series involved three cyclic tests with a mixed deformation program. In each test, a specimen was stretched with cross-head speed $\dot{d} = 100$ mm/min up to elongation ratio $k_{\max}(1) = 1.3$, unloaded down to stress $\sigma_{\min}(1) = \sigma_{\min}$, reloaded up to elongation ratio $k_{\max}(2) = 1.6$, unloaded down to the same stress

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