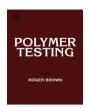
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Property modelling

# Nanoindentation creep of nonlinear viscoelastic polypropylene



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#### ABSTRACT

Uniaxial tensile creep tests at various applied stresses were carried out to demonstrate that PP is nonlinear viscoelastic. A novel phenomenological model consisting of springs, dashpots, stress-locks and sliders was proposed to describe the nonlinear viscoelasticity. Indentation creep tests at different applied load levels were also performed on nonlinear viscoelastic PP. It was found that the shear creep compliance varies with the applied load level when the applied load is less than 5 mN, which means the indentation creep behavior was nonlinear. To find the real reason for the nonlinearity in indentation creep tests, the elastic modulus at various indentation depths was measured using continuous stiffness measurements (CSM). By analyzing the variation of elastic modulus with indentation depth, the nonlinearity of indentation creep behavior was proved to be caused by the non-uniform properties in the surface of the specimen rather than nonlinear viscoelasticity.

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#### 1. Introduction

Since polymeric materials and polymer-matrix composites are widely used as load-carrying components in many structural applications, their mechanical properties, which are very important for product design, should be characterized by reliable approaches. With the development of micromachining processes, polymeric components and structures become smaller and smaller. The conventional testing techniques face great challenge when applied to such products due to the difficulty of specimen preparation. Nanoindentation, where tests are conducted in a small region (typically 1  $\mu$ m - 10  $\mu$ m in diameter) on the surface of actual components with little specimen preparation, is an efficient and convenient tool for probing the

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local mechanical properties of small volumes of materials such as polymeric thin films deposited on substrates, and small structures such as Micro-Electro-Mechanical-Systems (MEMS).

A large body of literature has described the analysis of the nanoindentation response for polymers whose behavior is typically time-dependent [1–10]. In the literature, the majority of the researchers assumed that polymers are linear viscoelastic, and the shear creep compliance is usually used to characterize the time-dependent response [1–7]. The shear creep compliance J(t) is defined as the change in strain as a function of time under instantaneous application of a constant stress, or

$$J(t) = 2(1+\nu)\frac{\varepsilon(t)}{\sigma_0} \tag{1}$$

where  $\nu$  is the time-independent Poisson's ratio of the sample. The shear creep compliance can be measured directly via conventional tension or compression creep

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tests [11]. Based on the viscoelastic contact solution derived by Lee and Radok [12], Hunter [13], Graham [14] and Ting [15], researchers have increasingly proposed instrumented (conical and spherical) indentation methods to determine the shear creep compliance [1–9]. For the case of a conical indenter indenting the sample with an instantaneously applied and constant load,  $F_0$ , the shear creep compliance can be calculated by

$$J(t) = \frac{4 \tan \alpha}{\pi (1 - \nu) F_0} h^2(t)$$
 (2)

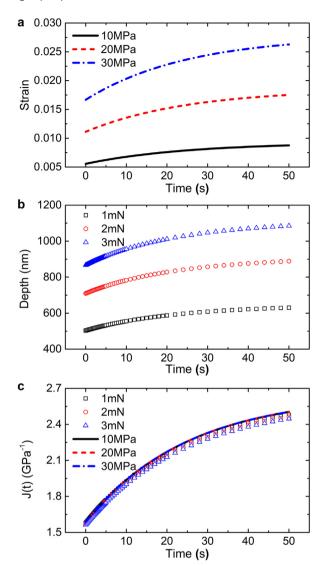
where  $\alpha$  is the included half-angle of the conical indenter; h(t) is the indentation depth. In the linear viscoelastic regime, the shear creep compliance is independent of test method and conditions. To validate this, tensile creep tests at various applied stresses, and indentation creep tests at various applied loads were carried out on an ideal linear viscoelastic solid in the commercial finite element program ABAQUS. The computational results shown in Fig. 1 illustrate that the shear creep compliance is indeed invariant with applied stress in tensile creep tests and applied load in indentation creep tests.

For nonlinear viscoelastic solids, however, the shear creep compliance varies with the applied stress. Jazouli et al. [16] studied the nonlinear creep behavior of polycarbonate (PC) via uniaxial tensile creep tests at different stress levels and found that the creep compliance increases with the applied stress. Tweedie et al. [9] observed that the assumption of linear viscoelasticity breaks down for several polymers (including polycarbonate and polypropylene) when creep compliance is measured via conical indentation. Oyen [17] discussed the geometrical nonlinearity and material nonlinearity for indentation of nonlinear viscoelastic solids. These researchers did great work but did not reveal the real reason for the nonlinearity in indentation creep tests.

In the present work, a phenomenological model composed of springs, dashpots, stress-locks and sliders is proposed to describe the stress-dependent shear creep compliance. Both uniaxial tensile creep tests at three different stress levels and indentation creep tests at seven different load levels were carried out on polypropylene (PP) in order to find the real reason for the nonlinearity in conical indentation creep tests.

### 2. Phenomenological model

For nonlinear viscoelastic polymers, the creep compliance increases with the applied stress. Generally, four main types of deformation, i.e. elastic, plastic, viscoelastic and viscoplastic, exist during indentation. To describe these features and the nonlinear viscoelasticity, a phenomenological model shown in Fig. 2 was proposed. In this model, a novel element called "stress-lock" was adopted. The property of stress-lock is characterized by a critical stress,  $\sigma^*$ , which can be regarded as the stress-key to the stress-lock. When the applied stress is equal to or less than  $\sigma^*$ , the stress-lock element remains locked and no deformation is allowed; when the applied stress is greater than  $\sigma^*$ , the stress-lock element is unlocked and deformation occurs



**Fig. 1.** Linear creep behaviors for an ideal linear viscoelastic material that constructed in ABAQUS. (a) The strain-time curves measured by uniaxial tensile creep tests at applied stress of 10 MPa, 20 MPa and 30 MPa deviate from each other. (b) The depth-time curves for indentation creep tests at applied load of 1 mN, 2 mN and 3 mN also deviate from each other. (c) The shear creep compliance determined by uniaxial tensile creep tests and indentation creep tests coincides well with each other.

freely. Thus, the locked Voigt-Kevin unit (a stress-lock, a spring and a dashpot in parallel) in the phenomenological model only works when the applied stress is greater than the corresponding stress-key,  $\sigma_i^*$ . As shown in Fig. 2, the Maxwell unit (a spring in series with a dashpot) is able to describe elastic deformation and stress relaxation; the Voigt-Kevin unit (a spring and a dashpot in parallel) is able to describe linear viscoelastic deformation; the viscoplastic unit (a slider and a dashpot in parallel) is able to describe viscoplastic deformation (the strain hardening after yielding is neglected here); the locked Voigt-Kevin units in series are able to describe nonlinear viscoelastic deformation because they can be equivalent to different viscoelastic

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