Polymer Testing 37 (2014) 138–147

Contents lists available at ScienceDirect

Polymer Testing

journal homepage: www.elsevier.com/locate/polytest

Test method

Viscoelasticity mapping method based on the contact resonance of a piezoelectric cantilever

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ARTICLE INFO

Article history:
Received 18 April 2014
Accepted 27 May 2014
Available online 5 June 2014

Keywords:
Viscoelastic properties
Polymers
Piezoelectric cantilever
Contact resonance

ABSTRACT

In this work, we propose a novel viscoelastic property mapping method/system for polymers based on the contact resonance of a piezoelectric unimorph cantilever. The viscoelastic properties of materials are derived from the contact resonance frequency (CRF) and quality factor (Q-factor) of the cantilever-sample system by using a cantilever vibration model. The performance of this system is examined by testing on a composite specimen made of nitrile rubber (NBR) and epoxy. It is shown that the dynamic modulus of the NBR is smaller than that of the epoxy, while the loss factor of the former is larger than the latter, which is consistent with the measurement results by using dynamic nanoindentation. The proposed method is very promising for *in situ* viscoelasticity mapping, which can be very useful in discrimination of unknown mixtures, interface characterization or elastography of superficial organs for medical diagnosis.

1. Introduction

Viscoelasticity, which relates to the molecular mobility and internal friction in polymers, is one of the most important properties and has attracted more and more attention in recent years [1–4]. Accurately determining the viscoelastic properties of polymers can widen their applications, such as for artificial valves [5]. At the same time, it can help understand internal physical processes such as phase transformations or defect motion in materials [6]. Currently, the most widely used viscoelastic property measurement methods and instruments include the rod resonance method [7], mass loaded method [8], sound or wave method [9], cantilever beam technique [10], optimized inversion technique [11] and dynamic mechanical analyzer (DMA) [12]. However, in all the above mentioned methods, the test specimen should be homogeneous and regular shaped (typically in one or two dimensions and symmetrical), and the measurement result is the averaged viscoelastic properties of the whole specimen [10]. Up to now, it is still a challenge to measure the distributed viscoelastic properties of inhomogeneous materials at macroscopic scale. Also, it would be very helpful if an *in situ* viscoelasticity measurement method could be developed.

On the other hand, at micro or nanoscale, methods for local dynamic viscoelastic property mapping based on contact vibration have been developed in recent years, i.e., dynamic nanoindentation (NI) [13] and contact resonance force microscopy (CR-FM) [14]. In dynamic NI, the local viscoelastic properties are obtained by recording both the amplitude and phase responses of the indenter to an excited load which consists of a static bias load of several millinewton and an alternating load of several to tens micronewton. The CR-FM, which is based on atomic force microscopy (AFM), maps the viscoelastic properties by scanning the sample and tracking the dynamic vibration

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http://dx.doi.org/10.1016/j.polymertesting.2014.05.013

characteristics of the AFM cantilever-sample contact resonance system [15]. Both dynamic NI and CR-FM can map the viscoelastic properties at very small scales, such as in a cell or a single fiber [16]. Recently, it was reported that CR-FM has less relative measurement errors than dynamic NI, which may indicate that CR-FM is more suitable for viscoelasticity mapping at the microscale [17]. However, several common disadvantages limit the application of both these methods. As in microscale or nanoscale testing methods, the geometry parameters of the indenters or tips are very difficult to determine exactly and always remain unclear, while these parameters play important roles in the mechanical model to derive the viscoelastic properties [13,18]. What is worse, the indenter's shape would be changed after long time contact vibration due to heavy wear [19]. Furthermore, both these methods are very sensitive to environmental vibration, and thus not suitable for *in situ* measurement. Intuitively, it would be very helpful for viscoelasticity measurement if these two methods could be extended to the macroscopic scale, but such works are still lacking.

In this work, encouraged by the principle of CR-FM, we propose a novel macroscopic viscoelasticity mapping method/system for polymers. A self-exciting and self-sensing piezoelectric unimorph cantilever is used to measure viscoelasticity based on the cantilever-sample contact resonance. The viscoelastic properties are derived from the contact resonance frequency (CRF) and quality factor (Q-factor) of the first mode contact vibration of the cantilever-sample system. Performance of the proposed method/system is examined by testing on a composite specimen made of nitrile rubber (NBR) and epoxy, and the viscoelasticity measurement results are consistent with that obtained using other commercially available methods. The proposed viscoelasticity mapping method is very promising for inhomogeneous materials testing or *in situ* measurement, such as under mechanical loading or high temperature.

2. Experimental

2.1. Design of the piezoelectric cantilever

In CR-FM, the cantilever-sample contact system is excited by the piezoelectric stage beneath the sample, and viscoelastic properties are derived by recording the displacement response of the cantilever using a four-quadrant PSD and an optical lever [20]. Here, we use a similar principle to measure the viscoelasticity on the macroscopic scale, in which a self-exciting and self-sensing piezoelectric unimorph cantilever is used to "sense" the sample's response during contact vibration.

A diagram of the designed piezoelectric unimorph cantilever is shown in Fig. 1. A short lead titanate zirconate (PZT) ceramic piece, poled in the thickness direction, is bonded to a steel cantilever near the clamped end, and a high sensitivity strain gauge is bounded on the top surface of the cantilever to monitor the strain, thus a self-exciting and self-sensing detection cantilever is formed. A cone-shaped steel tip is fabricated onto the free end of the cantilever, which can ensure tight contact with various samples. The cantilever design and the use of strain gauges make this method/system especially suitable for *in situ* measurement, and also applicable under a harsh environment or high temperature. In addition, the strain signal always consists of two parts, i.e. the AC signal and the DC signal. The AC signal denotes the dynamic vibration of the cantilever, from which the frequency response curve can be tracked to extract the sample's viscoelastic properties. The DC signal denotes the cantilever's static deformation, from which the pressing force applied to the sample can be monitored and controlled.

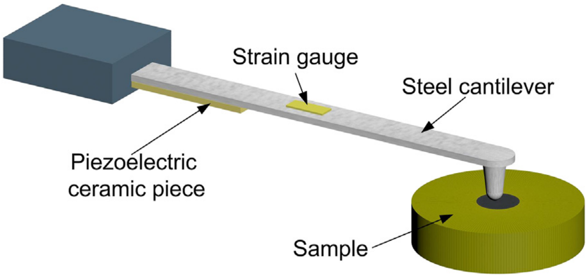

Fig. 1. Diagram of the piezoelectric unimorph cantilever. Excitations are generated by the thickness poled piezoelectric ceramic piece and the dynamic response of the cantilever is monitored by the strain signal using a high sensitivity strain gauge.

2.2. Principle of the contact resonance based viscoelasticity mapping method

In this section, we will briefly introduce how to extract the sample's viscoelastic properties by tracking the CRF and Q-factor of the cantilever-sample system. During testing, the cantilever tip is pressed to keep tight contact with the sample under a controllable force, and then the cantilever is excited by the short PZT piece using an AC voltage. Previous studies of a piezoelectric unimorph cantilever indicates that the excitation of PZT piece can be regarded as a linear moment applied to the cantilever, and the reacted contact force at the tip is proportional to the applied voltage [21]. Thus, the excitation of the PZT piece can be represented as an equivalent harmonic force excitation at the tip.

Details of the mathematical works for the cantilever's dynamics can be found elsewhere [22,23] and here we only present the cantilever's response to the harmonic force excitation. As the PZT piece is much shorter than the cantilever, the dynamic model of the piezoelectric cantilever can be simplified to a uniform Euler-Bernoulli beam. As shown in Fig. 2, a linear spring-dashpot system with stiffness k^* and damping factor of c is used to simulate the contact interaction between the cantilever and the testing sample at the tip position of $x = L_1$; $w(x,t)$ is the distributed displacement of the beam relative to its initial deflection, and L_2 is the length from the tip to the free end. As the parameters in the equivalent Euler-Bernoulli beam model cannot be measured directly, here we use a self-consistent approach to determine them [23].

The governing equation for the flexural displacement of the beam under a harmonic force of $Fe^{i\omega t}$ applied at $x = L_1$ can be expressed as