

Direct experimental evidence of time-temperature superposition at finite strain for an amorphous polymer network



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ABSTRACT

The time-temperature superposition property of an amorphous polymer acrylate network is characterized at infinitesimal strain by standard dynamic mechanical analysis tests. Comparison of the shift factors determined in uniaxial tension and in torsion shows that both tests provide equivalent time-temperature superposition properties. More interestingly, finite strain uniaxial tension tests run until break at constant strain rate show that the acrylate network exhibits the same time-temperature superposition property at finite strain as at infinitesimal strain. Such original experimental evidence provides new insight for finite strain constitutive modelling of polymer amorphous networks.

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1. Introduction

The mechanical behaviour of amorphous polymers at finite strain is very sensitive to temperature and strain rate, especially in the glass transition temperature range, where a change of temperature affects greatly the molecular mobility. Various studies have reported experimental evidences supporting this fact ([1–7] among others), and several viscohyperelastic models with an account of temperature have been proposed for the representation of the mechanical behaviour of amorphous polymers at finite strain ([1,4,5,8–11] for instance). Despite interesting features, the models of the literature show difficulties to represent accurately the mechanical behaviour of amorphous polymers over a wide range of temperature and strain rate, let alone to predict it. In order to better model the time and temperature dependent behaviour of amorphous polymer networks, useful for shape memory applications for instance, it is proposed to explore the time-temperature superposition property at finite strain. Time-temperature superposition at infinitesimal strain in amorphous polymers is well known [12]. Such a property formulates the idea that time and temperature are dependent parameters. In other words, exposing the material to a high temperature and a short duration is the same as exposing it to a lower temperature over a longer span of time. When looking at the change of the stress–strain responses of amorphous polymers when the

temperature or the strain rate varies, such a property seems also true at finite strain. Some authors showed that master curves could be built for finite strain behaviour quantities such as reduced yield stress [2,3,6] or stretch at break [13]. Nonetheless, finite strain time-temperature equivalence remains to be tested on the entire stress–strain response and its quantitative estimate to be compared to the time-temperature superposition property measured at infinitesimal strain in order to rationalize its introduction in general constitutive equations. For this purpose, an amorphous acrylate network has been synthesized and tested in dynamic mechanical analysis and in conventional uniaxial tension until break. Several finite strain uniaxial tension tests were run at various constant temperatures and constant strain rates in order to show that stress–strain curve superposition exists also at finite strain and to calculate the time-temperature shift factors. The strain rates applied are moderate in order to avoid possible self-heating of the samples.

2. Material and experiments

2.1. Material

An acrylate network was prepared by copolymerization of benzyl methacrylate (BMA) with poly(ethylene glycol) dimethacrylate (PEGDMA) of molar weight 550 g/mol, which was used as crosslinker. 90% molar mass of BMA was mixed at room temperature with 10% molar mass of PEGDMA and 0.5% of 2,2-Dimethoxy-2-phenylacetone (DMPA) used as photoinitiator. Products received from Sigma Aldrich were used without transformation.

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The mix was cured between glass frames in a UVP ultraviolet chamber CL-1000 for 50 min in order to obtain plates of constant thicknesses as required for mechanical testing.

2.2. Experiments

The material was submitted to dynamic mechanical analysis (DMA) in torsion and in uniaxial tension. Dynamic torsion tests were run on a MCR502 rheometer from Anton Paar while dynamic uniaxial tension tests were performed on a DMA Q800 from TA instruments. Rectangular samples of length \times width \times thickness equal to $40 \times 12 \times 1.3 \text{ mm}^3$ were submitted to dynamic torsion and rectangular samples of $30 \times 5 \times 1.3 \text{ mm}^3$ were used for dynamic tension. In order to build the material storage modulus master curve and to calculate the shift factors characterizing the time-temperature superposition at infinitesimal strain, isothermal frequency sweeps at 0.1% strain from 0.01 to 10 Hz were applied from 37°C to 64°C every 3°C in torsion and from 30 to 65°C every 5°C in tension.

At finite strain, dog-bone shape specimen of 1.3 mm thickness, 4 mm width and 20 mm gage length were submitted to isothermal uniaxial tension tests on an Instron 5881 testing machine equipped with a thermal chamber. Since crosshead displacements provide inaccurate measures of strain when dealing with finite strain, local strain was measured by video extensometry. The resulting force was recorded with a 1 kN cell. Some tests were run at constant crosshead speed of 1 mm/min. In order to run tests at constant strain rate, our experimental setup required to use constant piecewise displacement speed. The calculation of the strain rate afterwards proved that it remained close to constant during these tests. The strain rate was estimated by taking the derivative with respect to time of the logarithmic strain, $\varepsilon = \ln(l/l_0)$, with l and l_0 being the current and the initial distances between two markers painted on the free surface of the sample and followed by the video extensometer. Therefore, the strain rate was calculated according to $\dot{\varepsilon} = \frac{\dot{l}}{l/l_0} = \dot{l}/l$. Note that \dot{l}/l_0 is the engineering strain rate. It was observed that tests run at constant crosshead speed do not provide access to either constant engineering strain rate tests or constant strain rate tests due to non-homogeneous strain within the sample resulting from the large strain applied. In order to satisfy to a commonly used representation of material behaviour in conventional uniaxial tension, the material response will be plotted in terms of the engineering stress (F/S_0) with respect to the engineering strain $(l-l_0)/l_0$, with l and l_0 being obtained from the video extensometer.

Each test was performed three times in order to validate the experimental reproducibility.

3. Time-temperature superposition at infinitesimal strain

The amorphous acrylate network was submitted to standard frequency sweeps in torsion at constant temperature. From the measured storage modulus G' and damping factor, master curves were built in order to verify the time-temperature superposition property (Fig. 1). The latter property is expressed mathematically by introducing a reduced time τ defined at the reference temperature T_0 , and writing the relationship between the reduced time and the actual time at temperature $T(t)$ through the shift factor a_{T_0} according to:

$$d\tau = \frac{dt}{a_{T_0}(T(t))} \quad (1)$$

The material linear viscoelasticity was also characterized in uniaxial tension. Isothermal frequency sweep tests were carried out in order to determine the storage modulus E' and its associated damping factor. The master curves characterizing the material frequency dependence in tension are shown in Fig. 2.

The comparison of the shift factors directly obtained from the master curve building procedure is shown in Fig. 3. The material presents similar shift factor values in uniaxial tension and in torsion.

Comparing the storage modulus and the damping factor obtained in uniaxial tension to those measured in shearing is possible but demands to make some assumptions. For linear viscoelastic isotropic materials, the complex moduli satisfy to the same relationship as the elastic moduli for elastic isotropic materials [14]. Therefore, denoting $G^* = G' + iG''$ and $E^* = E' + iE''$ the complex shear modulus and the complex Young modulus respectively, one may write:

$$G^* = \frac{3K^*E^*}{9K^* - E^*} \quad (2)$$

with K^* the complex bulk modulus. It is very difficult to reach K^* experimentally. Moreover, the storage bulk modulus is expected to drop by a factor of 2–3 only when temperature rises above the glass transition [15]. Therefore, a first assumption consists in assuming the bulk modulus as elastic ($K^*=K$) and constant. Doing so and choosing a value of 1150 MPa to reach a Poisson ratio of 0.41 in the glassy state and 0.499 in the rubbery state, complex shear storage modulus values may be calculated from the values of E' measured

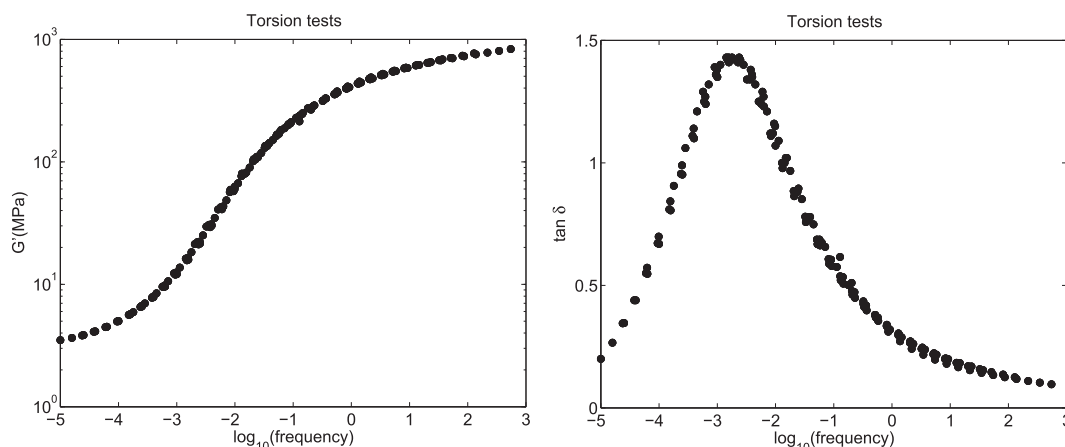


Fig. 1. Master curves for the storage modulus and damping factor measured in torsion for a reference temperature of 45°C .

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