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Influence of strain rates on the mechanical behaviors of shape memory polymer

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Abstract

In the last few decades, shape memory polymers have demonstrated their major advantages of extremely high recovery strain, low density and low cost. Generally, the mechanical behavior of shape memory polymers is strongly dependent on the loading strain rates. Uniaxial tensile experiments were conducted on one kind of typical shape memory polymer (epoxy) at several different temperatures (348 K, 358 K, 368 K and 378 K) and true strain rates (0.25% s⁻¹, 1.25% s⁻¹ and 2.5% s⁻¹). Thus, the influence of strain rate and temperature on the mechanical behavior of epoxy, in particular on the post yield stresses and the strain hardening behavior, were investigated through this experimental study. Based on our previous work Guo (2014 *Smart Mater. Struct.* 23 105019), a simplified model which can explain the shape memory effect of epoxy was proposed to predict the strain hardening behavior of the shape memory polymer. Based on the suggestion of Rault (1998 *J. Non-Cryst. Solids* 235–7 737–41), a linear compensation model was introduced to indicate the change in yield stresses with the increase of strain rate and temperature. Finally, the new model predictions for the true strain and stress behavior of epoxy were compared with the experimental results.

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Keywords: shape memory polymer, mechanical behaviors, strain rates

(Some figures may appear in colour only in the online journal)

1. Introduction

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Shape memory polymers (SMPs) are a type of smart material which can recover to its original shape under appropriate stimulus (such as temperature [3, 4], light [5, 6], electric or magnetic fields [7, 8], moisture [9] and water [10]). Compared with other shape memory materials (such as shape memory alloys and shape memory ceramics), SMPs have the advantages of low density, low cost, high shape recovery ratios and excellent processability [11–16]. The thermally activated shape memory effect is shown with changing temperature in figure 1. Depending on the phase transition temperature, the shape memory effect process can be classified into four steps:

- (1) heating and loading the specimen at a high temperature; (2) cooling at a constant strain: (3) removing the external
- (2) cooling at a constant strain; (3) removing the external force; and (4) heating above the phase transition temperature causing the pre-deformed specimen to recover to its original shape. Due their special properties and advantages, SMPs have attracted considerable attention and also have wide applications in the fields of aerospace, smart tensile materials, biomedicine, etc [13, 17–19]. In order to investigate the mechanical behavior of SMPs and their composites, many tension, compression and bending experiments have been conducted and many different kinds of constitutive models have also been developed [20–28]. Based on these models,

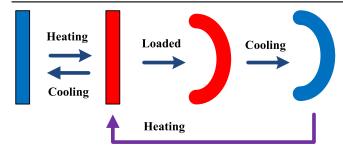


Figure 1. A schematic diagram of the shape memory effect.

the loading and recovering processes of SMPs can be predicted effectively and accurately.

As reported in the literature, a SMP is a typical temperature and rate dependent material, which means that the strain rate also has a significant influence on the yield stress and post yield behavior of the polymer, as does the temperature. Numerous experiments have been conducted to explore and characterize the function between the mechanical behavior and strain rates. During the loading process, strain softening and hardening will emerge, especially at high temperatures, due to the long-chain nature of the material, which is one of the important aspects of the mechanical behavior of polymers [29–32]. During the last few decades, some attention has been paid to the mechanism and mechanical behavior of the strain hardening of polymers and some creative methods for the prediction of the yield stress of polymers have been achieved [33-38]. Some constitutive models have also been developed to describe the nonlinear mechanical behavior of polymers under large tensile or compressive deformation experiments [35–42]. Most of these models are particularly focused on the numerical simulation of the large strain deformation of amorphous, glass polymers. Through taking the strain rate parameter into account, Mohotti [37] developed a new constitutive model to predict the high strain rate behavior of polyurea. In 2014, Cao proposed a finite element model for polycarbonate to better understand its tension deformation [39]. Under the assumption that the local microstructural density is the main reason for the strain non-uniformity fluctuation in glass polymers, Spathis proposed a new model for rate dependent strain hardening [43]. Based on tension and compression experiments at different strain rates ranging from 10^{-4} to 10^4 s⁻¹, the post yield behavior was characterized and a constitutive model was also established in 2006 by Mulliken and Boyce [40]. Warren [44] investigated the dynamics of aging in polymer glass by examining the effect of short temperature square steps and found that a negative temperature step can cause uniform rejuvenation due to reduce aging at a low temperature while a positive temperature step yields a completely different relaxation spectrum. In 2005, Richeton et al proposed a cooperative constitutive model to express the relationship between the temperature and strain rate. In this model the assumptions for above and below the phase transition temperature were discussed [45]. Melick et al studied the influence of network density on the strain hardening behavior of an amorphous polymer and built a equation to

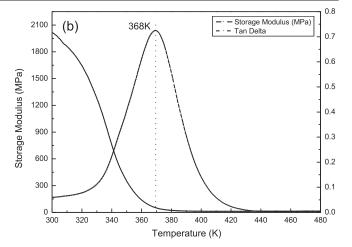


Figure 2. The DMA tests results for the epoxy.

express the variation of modulus [38]. In 2003, Wendlandt *et al* modified the Leonov model using a non-constant strain dependent activation volume, the precision of which was verified through comparisons with experiments [33]. Using a continuum thermo-mechanical framework and molecular dynamics simulations, Jatin *et al* developed a new theory to explain the origin of the stain softening which is the result of the burst of free volume, and strain hardening which is the result of the non-bonded part of the force field [35]. Based on the two-phase representation of semicrystalline polymers, a thermodynamic model which contains a mechanical coupling between these two phases was built in 2008, which was validated by comparison with the tensile, compression and simple shear stresses [46].

In this paper, the rate dependent strain hardening behavior of epoxy is studied based on uniaxial tensile experiments at different temperatures (348 K to 378 K) and true strain rates (0.25% s⁻¹, 1.25% s⁻¹ and 2.5% s⁻¹). The dependence of the strain hardening behavior on the strain rates of epoxy is demonstrated. Based on the work of Rault [2], a linear compensation equation is used to predict the increase of yield stress with the increases of strain rate and temperature. In addition, a three-dimensional model which combines phase transition theory and viscoelasticity is investigated to predict the strain hardening behavior of a SMP, the precision of which was also verified.

2. Experimental results

In this article, a typical SMP—epoxy, the phase transition temperatures of which are listed in figure 2—is introduced to investigate the influence of strain rate on its mechanical behavior. The shape memory effect of epoxy is examined through recovery experiments at a high constant temperature (as shown in figure 3). The plate shaped specimens of epoxy were tensioned at several different temperatures around the phase transition temperature with three different strain rates. From the experimental results, the change in the yield stress and the strain hardening behavior of the SMP with the

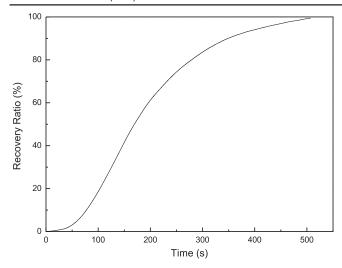


Figure 3. The shape recovery process of the epoxy at $398~{\rm K}$ ($30~{\rm K}$ above the phase transition temperature).

increase of strain rate and temperature can be observed. Finally the corresponding parameters appearing in the new constitutive model are obtained from these experiments.

2.1. Dynamic mechanical analysis tests

Dynamic mechanical analysis (DMA) tests under the tensile mode with constant heating and cooling rates ($\pm 5^{\circ}$ C min⁻¹) were carried out on the epoxy. The samples were cut into pieces $2 \text{ mm} \times 3 \text{ mm} \times 19 \text{ mm}$ in size using a laser cutting machine at a room temperature and the experimental temperature ranged from 293 K to 460 K. From the DMA results (shown in figure 2), we can conclude that the glass transition temperature of epoxy is about 368 K. The storage modulus of these materials decreases rapidly around the phase transition temperature, from near 2000 MPa to 8 MPa during the heating process. Generally, the fastest decline point for the modulus should coincide with the phase transition temperature. However, there are some offsets due to the delayed effect of the polymer, especially at high temperatures. Over the last few years, some constitutive models have been proposed to describe the variation during the heating and cooling processes [1, 22].

2.2. The shape recovery of epoxy

To verify the shape memory effect, which is a special property of epoxy, three point bending experiments were conducted at a high temperature. Here, the epoxy specimens were cut into a rectangular shape with the dimensions 65 mm × 6 mm × 3 mm. First, the specimens were heated to 398 K (30 K above the phase transition temperature) and loaded with several different strain rates. By keeping the indenter motionless and cooling down to room temperature, the deformation was stored. When raising the temperature, the specimens recovered to their original shape and the shape memory effect of the epoxy was verified (as shown in figure 3). At the beginning of the recovery process, the temperature distribution in the specimen was uniform, which

resulted in the gradual recovery of the epoxy. As time goes on, the specimen will recover rapidly due to the release of the 'storage strain'. Finally, the deformed shape will recover fully and the strain converges to zero, which provides the verification of the shape memory effect of this type of epoxy.

2.3. The influence of strain rate and temperature on the yield stress

The specimens made of epoxy were tensioned uniaxially using ZWICK-Z010 under different temperatures and strain rates, and the specimens were cut into dumbbell shapes with a thickness of 3 mm based on the suggestion of ASTM-D638. Before the loading process, the specimens were heated for about 5 min to release the lower clamp and decrease the impact of thermal expansion on the experimental results. The samples which were clamped on two sides were tensioned until they broke at three different true strain rates, ranging from $0.25\% \text{ s}^{-1}$ to $2.5\% \text{ s}^{-1}$. In addition, the influence of temperature on the mechanical behavior of the epoxy was studied at four different temperatures around the phase transition temperature (348 K, 358 K, 368 K and 378 K). The strain-stress result show the yield stress of the epoxy during the tension experiments (figure 4). As reported in previous articles, the increasing strain rate decreases the molecular mobility of the polymer chain by making the chain stiffer. The first observation is that the yield stress of the SMP increases with increasing strain rates at a certain temperature, while it decreases with the increasing temperature at a specific strain rate. As indicated in figure 4, the yield stresses can be easily obtained at the point where the strain-stress curve shows a local maximum at low temperatures such as 348 K and 358 K, which are both lower than the phase transition temperature. While the maximum values are less defined at higher temperatures, we can use the combined Brereton-Considère approach to calculate them.

As shown in figure 5, the yield stresses are related linearly to the temperature and ln normalized true strain rates. For the material's high modulus at a low temperature, the yield stresses increase much faster as the strain rates increase. However, the slopes of the yield stress show no significant difference for the decreasing of temperature at a constant strain rate. Thus, we can conclude that a decrease in strain rate has the same effect on the yield stress as an increase in temperature.

2.4. Rate and temperature dependent strain hardening behavior

The tension experiments at various strain rates and temperatures indicate that the SMP deformed in a nonlinear manner (including elasticity, yield, strain softening and strain hardening). In addition, the strain rates and temperatures also have a marked effect on the strain hardening behavior, which was another significant observation during the tension experiments. Similarly to the yield stress, an increase in temperature will induce the decrease of the post yield stresses. Due to the decline of the modulus, the elasticity and yield stress of the

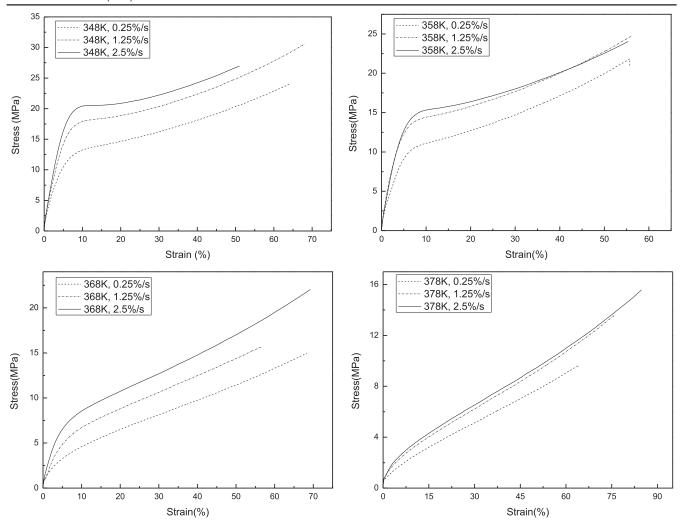


Figure 4. Strain-stress behavior of the epoxy during tensile experiments at different strain rates and temperatures.

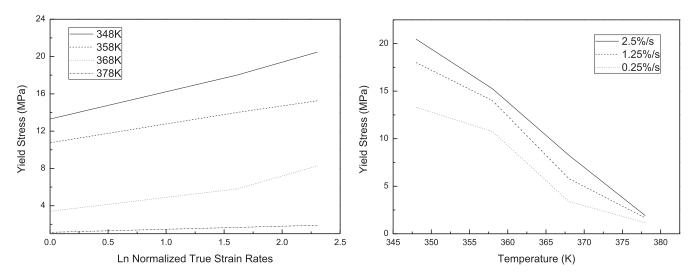


Figure 5. Variation of the yield stress of epoxy with increasing strain rate and temperature.

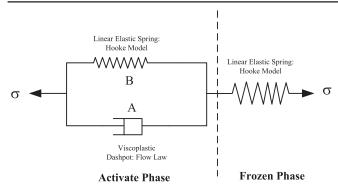


Figure 6. Three-dimensional representation of the SMP deformation model.

epoxy at a high temperature are not obvious compared to those at a low temperature. Moreover, the stresses (including yield stresses) also increase and the curves of the true strain– stress for different strain rates are nearly parallel with the increase of strain rates.

3. The constitutive model and simulation

It has been established that strain rate and temperature all have the same effect on the mechanical behavior of the polymer. Obviously, the yield stress is an important feature when the effect of the strain rate on the mechanical behavior of a SMP is discussed or investigated. For simplicity, we just use a linear compensation model provided by Rault in 1998 [2] which combines the effects of stress and temperature together to predict the variation of yield stress:

$$\sigma_{y} = \sigma_{0} - \frac{\sigma_{0}}{T_{g}}T + \frac{kT}{V}\ln(\dot{\varepsilon}/\dot{\varepsilon}_{0})$$
 (1)

where $T_{\rm g}$ and T are the phase transition and absolute temperature, respectively. k is the Boltzmann constant and V is the active volume. The corresponding parameters $(\sigma_0/T_{\rm g})$ and kT/V appearing in (1) can be measured from the experiments.

To describe the strain hardening behavior of the SMP, we introduce a three-dimensional rheological representation of the viscoelasticity model (figure 6). In this model, the SMP is decomposed into two phases: an active phase and a frozen phase. As has been reported in previous papers [1, 22], the phase transition between the frozen and active phase is the

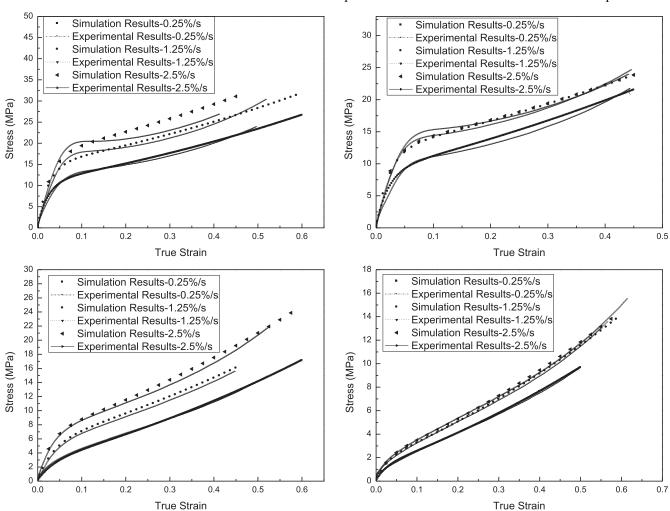


Figure 7. Comparison between the simulation and experimental results.

key factor to explain the shape memory effect. Thus the relationships of stress and strain in this model are given as:

$$\sigma_{f} = \sigma_{a} = \sigma_{A} + \sigma_{B};$$

$$\varepsilon = f_{f} \varepsilon_{f} + f_{a} \varepsilon_{a};$$

$$\varepsilon_{A} = \varepsilon_{B};$$

$$f_{f} + f_{a} = 1;$$
(2)

where σ_f , σ_a , σ_A and σ_B are the stress tensors of the frozen phase, active phase, part A and part B, respectively, and ε_f , ε_a , ε_A and ε_B are the corresponding strain tensors. The volume fractions of the active and frozen phases ϕ_f and ϕ_a , which are a function of temperature, are given using a normal distribution equation with change in temperature:

$$\phi_{\rm f} = \int_{T_{\rm s}}^{T} \frac{1}{S\sqrt{2\pi}} e^{-\frac{\left(T - T_{\rm g}\right)^2}{2S^2}} dT \tag{3}$$

where T and T_s are the environmental temperature and starting phase transition temperature, respectively. T_g is the phase transition temperature while S is the variance of the normal distribution function. During the loading process at a constant temperature, the strain rate can be given as the function of volume phase fraction, total stress and its rate:

$$\dot{\varepsilon}_{ij} = S_{ijkl}\dot{\sigma}_{kl} + \phi_{a} \left(\frac{(1+v)\sigma_{ij} - v\sigma_{kk}\delta_{ij}}{u} - \frac{(1+v)\varepsilon_{ij} - v\varepsilon_{kk}\delta_{ij}}{\lambda} \right)$$
(4)

where u, λ and v are the viscosity time, retardation time and Poisson ratio of the active phase, respectively. Here, '·' denotes the differential form of mechanical strain in the active phase to time σ_{ij} is the total stress during the tension experiments and S_{ijkl} is the elastic compliance tensor of the whole polymer. From (4), we can easily conclude that the viscoelasticity will play a more important role due to the increase of the active volume fraction at higher temperatures, which can explain the decline of the elasticity stage and the extension of strain hardening behavior during the uniaxial tensile experiments. Thus, the applied stress during uniaxial tension or compression experiments can be finally expressed as:

$$\sigma = -\frac{u\dot{\varepsilon}}{\phi_{\rm a}} e^{-\frac{\phi_{\rm a}E}{u}t} + \frac{u}{\lambda}\varepsilon + \frac{u}{\phi_{\rm a}}\dot{\varepsilon}$$
 (5)

Due to the constant true loading strain rates, transitions between the engineering strain (or strain rate) and true strain (or strain rate) are necessary. So a modified form of (5) is given by:

$$\sigma(t) = C e^{-\phi_a E t/u} + \frac{E \dot{\varepsilon}_T + \phi_a E/\lambda}{\dot{\varepsilon}_T + \phi_a E/u} e^{\dot{\varepsilon}_T t} - u/\lambda \tag{6}$$

in which \dot{e}_T is the true strain rate during the loading process. The parameter C can be calculated under the initial boundary condition of $\sigma(0) = 0$. Equation (6) consists of two exponential parts: the first part represents the influence of

temperature on stress while the second part indicates the effects of strain rates. With increasing strain rate, the internal variables E, u and λ , which are functions of the strain rates, can be described by exponential equations [47]. Figure 7 shows the simulation results from (6), the precision of which was also verified though comparison with the experiments. The model constructed in this paper can describe the mechanical behavior of epoxy (including the elasticity, yield stress, strain softening and hardening) precisely, especially at high temperatures.

4. Conclusion

In this paper, the effect of strain rate and temperature on the mechanical behavior of an SMP (epoxy) were investigated based on uniaxial tension experiments. Both the yield stress and post yield behavior of the epoxy increase with increasing strain rate or decreasing temperature. Similarly to the experiments conducted by Wang [42], all of the specimens will almost recover to their original shape due to their shape memory effect. When the specimens were stretched at a high strain rate, most of the deformation was afforded by the linear spring, which resulted in a smaller recovery strain and shorter recovery time. Through the uniaxial tension experiments, we measured the yield stresses of the epoxy at different strain rates and temperatures, the relationships of which were expressed using a linear compensation model. Moreover, the three-dimensional constitutive model proposed in this paper, based on phase transition and viscoelasticity theories which may explain and describe the shape memory effect, precisely illustrates the influence of strain rates and temperatures on the mechanical behavior of epoxy.

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