

# Experimental study of thermo-mechanical behavior of a thermosetting shape-memory polymer

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**Abstract** The thermo-mechanical behavior of shape-memory polymers (SMPs) serves for the engineering applications of SMPs. Therefore the understanding of thermo-mechanical behavior of SMPs is of great importance. This paper investigates the influence of loading rate and loading level on the thermo-mechanical behavior of a thermosetting shape-memory polymer through experimental study. A series of cyclic tension tests and shape recovery tests at different loading conditions are performed to study the strain level and strain rate effect. The results of tension tests show that the thermosetting shape-memory polymer will behave as rubber material at temperature lower than the glass transition temperature (Tg) and it can obtain a large shape fix ratio at cyclic loading condition. The shape recovery tests exhibit that loading rate and loading level have little effect on the beginning and ending of shape recovery process of the thermosetting shape-memory polymer. Compared with the material which is deformed at temperature higher than Tg, the material deformed at temperature lower than Tg behaves a bigger recovery speed.

**Keywords** Styrene shape-memory polymers · Loading rate · Loading level · Cyclic loading-unloading test · Free shape recovery

# **1** Introduction

As a typical soft material (Liu et al. 2015), shape-memory polymers (SMPs) (Hager et al. 2015) have exhibited superior applications in engineering. SMPs are able to recover the permanent shape from the predetermined temporary shape after stimulated by temperature, magnetic field, light etc. In comparison with other shape-memory materials like shape-memory alloys and shape-memory ceramics, SMPs exhibit unique properties: high programmable strain, light weight and low cost. These unique properties make SMPs gained extensive research interest in recent years for applications such as in microsystems

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(Fei et al. 2013; Li et al. 2013), biomedical devices (Hearon et al. 2015; Kang et al. 2014), aerospace deployable structures (Tao et al. 2016) and morphing structures (Leng et al. 2011; Meng and Li 2013). The deformation mechanism (He et al. 2015; Molaaghaie-Roozbahani et al. 2016) and constitutive models (Husson et al. 2011; Li et al. 2017; Liu et al. 2006; Yu et al. 2014b) are widely studied to describe the thermo-mechanical behavior of SMPs. Current studies show that many factors such as the recovery time, programmed temperature and heating rate have great influence on the thermo-mechanical behaviors of SMPs. Yu et al. (2014a) provided an approach to predict the shape fixity and free recovery of thermorheological SMPs by making reduced time as a unified parameter. Shape recoveries under different thermo-conditions could be extracted with time-temperature superposition principle. Based on the previous study, Yu and Qi (2014) further studied temperature memory effect in amorphous shape-memory polymers. The research indicated that the characteristic recovery temperature (Tc) is not always equal to the programming temperature (Td). They gained a facile method to optimize the shape-memory performance of SMPs by adjusting their thermo-mechanical working conditions instead of developing new polymer systems. In addition to the discovery of the influence of thermal conditions on SMPs, the mechanical behavior of SMPs was also explored at different mechanical conditions (McClung et al. 2013). Hu et al. (2005) preliminarily studied the dependency of the shape-memory properties of a polyurethane upon thermo-mechanical cyclic conditions. The authors gave the result that the recovery ratios increase with increasing deformation speed and with decreasing maximum strain. Correspondingly, Wang et al. (2013) studied the strain rate effect on the thermo-mechanical behavior of a thermoset shape-memory polymer. In their study, the thermoset SMP was compressed with various programmed strain rates from quasi-static to a high strain rate at temperature lower than Tg. The results revealed a certain strain rate memory effect on thermosetting SMPs: sample programmed at the highest strain rate recovered in a shorter time as it had the smallest amount of strain to recover. Guo et al. (2015) also investigated that both the yield stress and the post-yield behavior of the epoxy SMP with increasing strain rate or decreasing temperature by uniaxial tension experiments. A linear compensation model was established to describe the response of yield stress with the changed strain rate and temperature based on the experimental results. Yu et al. (2016) evidenced that epoxy thermosetting polymer of no visible damage or irreversible softening effect during the deformation still exhibited obvious degradation in the cyclic tension and SM behaviors. They ascribed this phenomenon to SMP's viscoelastic nature. McClung et al. (2012) found that the Young modulus of the Veriflex-E is most sensitive to the strain rate value near Tg by exploring the influence of strain rate on Veriflex-E at a range of temperatures and demonstrated that the Poisson ratio was dependent of the strain magnitude but independent of strain rate. Except for researches on pure SMPs, shape-memory polymer composites (SMPCs) were also studied for its versatile functions compared with pure SMP. Cantournet et al. (2009) synthesized a thermo-responsive shape-memory polymer composite filled by nano-sized carbon black. The tension and shape recovery results showed the stretch induced softening effect, namely the Mullins effect (Diani et al. 2015), can affect the free recovery behavior.

All above work put more concentration on the behavior of SMPs at high temperatures conditions but did little on the thermo-mechanical performance of SMPs at temperature lower than Tg. Normally the SMPs are loaded at temperature higher than Tg to obtain an expected bigger shape fixity. However, through extensive experimental studies, we find that SMPs can behave as rubber material at temperature lower than Tg under cyclic tension tests, which provide a new view for the deforming of SMPs. Furthermore, the influence of loading rate and loading level on the thermo-mechanical behavior of SMPs is intensively studied

through a series of cyclic loading-unloading tests and shape recovery tests at different loading conditions. The results indicate that strain rate and strain level have little effect on the beginning and the ending of shape recovery process. The material which has been deformed at temperature lower than Tg shows a bigger recovery speed than the material deformed at temperature higher than Tg.

The paper is organized as follows. First, the experimental methods of styrene SMP including the dynamic mechanical tests, tension tests and shape recovery tests are presented. Then the results and discussion as regards the influence of loading rate and loading level are given. Finally, concluding remarks are provided.

# 2 Experimental methods

#### 2.1 Materials and experimental machine

A styrene shape-memory polymer was synthesized by styrene, butyl acrylate, divinylbenzene and benzo peroxide with mass fraction of 60:40:2:2 under a heating condition of 24 hours at 70 °C. The cross-linker in this formulation of SMP is benzo peroxide. The mould is combined with two glass films and an organic glass frame of trapeziform fillister. The solution was mixed for 30 min and stood for 10 min before being injected into the mould through the fillister. The mould was then placed in insulation at 70 °C for 24 hours to cure the styrene SMP. All materials were purchased from Sigma-Aldrich Corporation. Sample size for all tests is 20 mm  $\times$  5 mm  $\times$  0.67 mm. All tests were conducted on DMA (Dynamic mechanical analysis, Model Q800, TA Instruments, New Castle, USA) machine (as shown in Fig. 1).

#### 2.2 Dynamic mechanical analysis

Dynamic mechanical analysis test was performed on a DMA machine to characterize the glass transition behavior of the styrene SMP. This glass transition behavior can be described by the storage modulus and loss factor evolution of the temperature. Samples were vibrated at the frequency of 1 Hz and amplitude of 10  $\mu$ m with the temperature increasing from 0 °C to 90 °C at a rate of 1 °C per minute.

#### 2.3 Cyclic loading-unloading tests at different loading rates

To verify loading rate effect on the mechanical behavior of styrene SMP, uniaxial cyclic loading-unloading tests at different loading rates were conducted at different loading conditions.

Samples were deformed to a strain of 15% and unloaded to zero strain at 40 °C (temperature lower than the glass transition temperature) for four times. The programmed loading rates were 1%/min, 5%/min and 8%/min, respectively.

Another series of experiments were conducted to study loading sequence effect on styrene SMP. One group samples were loaded-unloaded at a sequence of 1%, 4% and 5%, the other group was conducted as the inverse loading-unloading sequence.

## 2.4 Cyclic tension tests of different strain levels of styrene SMP

To demonstrate the effect of strain level on styrene SMP, samples were deformed with a strain rate of 1% per minute to three different strain values: 1% (before yield point), 5% (after yield) and 15% (cold-drawing process) and unloaded to zero strain at 40  $^{\circ}$ C for four times.

**Fig. 1** (a) The overall exploded view of DMA. (b) The tension clamp used in tests





(b)

#### 2.5 Free shape recovery tests

Shape recovery tests were conducted to characterize the free shape recovery properties of styrene SMP. Samples were first deformed at 60 °C with programmed strain rates. Then samples were cooled to 20 °C at 5 °C/min and kept for 5 min to see stress evolution. After cooling process, samples were released the force and reheated to 60 °C with rate of 5 °C/min. Samples were held for 15 min to ensure the shape recovery process was finished. The diagrammatic sketch of complete shape recovery test is shown in Fig. 2. We call this process free shape recovery (FSR).

The influence of strain level and strain rate on shape recovery behavior of styrene SMP were studied in three test groups: one test group varied the strain rates of 5%/min, 10%/min, 20%/min and 50%/min at a same loading level of 10%; the second test group differed at the strain level of 10%, 20% and 40% with a same loading rate of 5%/min; the last test group were performed with loading-unloading process at different temperatures before the whole shape recovery test: samples were loaded to 10% strain and unloaded to zero strain with a

Strain

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Fig. 2 The diagrammatic sketch of free shape recovery test on



Temperature

same loading rate of 5%/min for three times at temperature below Tg and at Tg, respectively. A shape recovery test of 15% strain level and 1% strain rate was performed to compare with the sample deformed at temperature lower than Tg.

# 3 Results and discussion

# 3.1 Dynamic mechanical analysis of styrene SMP

With temperature varying, the physical and chemical properties of polymers are correspondingly changed. The glass transition temperature can be defined as the turning temperature of specific volume curve or the sharply changed temperature of volume thermal expansion or the temperature where the internal friction reaches the maximum. The dynamic mechanical analysis of styrene SMP is shown in Fig. 3. To obtain a better understanding of the thermo-mechanical behavior of styrene SMP, Tg is defined as 42.3 °C from the temperature derivative of storage modulus curve E' (as shown in Fig. 4) where the storage modulus changed sharpest.

# **3.2** The influence of loading rates on the mechanical behavior of styrene SMP

From the results of cyclic uniaxial tension test of styrene SMP as shown in Fig. 5, we can see that an obvious softened stress occurred for all tests with cycle numbers increase. When

Fig. 4 Temperature derivative of storage modulus



strain reaches 15%, all samples with different loading rates are undergone yielding. After the first test cycle, all samples undergo a transition from glass to rubber induced by strain, which meant styrene SMP exhibited a rubbery-like property in the rest test cycles in glassy state. styrene SMP can reach the same strain with much smaller stress. This phenomenon could be explained that the reconstruction of the destroyed plastic phase during the enforced high elastic deformation requires much long time to recover. Therefore, the following cyclic tension tests greatly depend on the rubber phase and behave a rubber-like property.

Figure 5(a) shows the strain-stress curve for the loading rate of 1%/min. From Fig. 5(a), we can see that the strain-stress curves are almost the same after the first loading-unloading cycle. The four cycles reach the same stress value at target strain of 15%. With the increase of loading rate, the stress increases (as shown in Fig. 5(b)–(c)), while the stress value at 15% strain at first loading-unloading cycle is bigger than the followed cycles. For loading rate of 5%/min and 8%/min, samples show an obvious softening effect. Besides, the speed of the loading-unloading cycle tending to be equilibrium is lower. We can say that a higher strain rate results in a higher response of stress and a lower speed of equilibrium.

However, the stress is not proportional to the loading rate (as shown in Fig. 6). During the deformation of styrene SMP at strain rates of 5%/min and 8%/min, the enforced high elastic deformation does not develop sufficiently compared to the test at 1%/min, because the relaxation time of the material is large and cannot catch up the velocity of external force. Therefore, a larger strain rate will need more cycle numbers to be equilibrium. There is something in common that the irrecoverable strain of styrene SMP in unloading step is very large and tending to be the same with little relationship of strain rates when the cycle numbers increase (as shown in Fig. 7). With the increase of cycle numbers and strain, the residual strain grows on.

It is obviously that styrene SMP can behavior like rubber at temperature lower than Tg and obtain a high residual strain during the cyclic test. Combined this result with the shapememory property of styrene SMP, applications based on styrene SMP can be deformed to a large strain at temperature lower than Tg with a high shape fixity, which avoids the large deformation due to thermal expansion during temperature increasing. The sample deformed below Tg can recover to its original shape rapidly when the sample is reheated to a temperature higher than Tg. (as shown in Fig. 8).

Figure 9 shows stress evolution with strain at different loading sequences. We can observe that a decreasing loading rate sequence will accelerate the equilibration of Styrene behavior, which means decreasing the cycle of equilibrium (as shown in Fig. 9(a)). Cyclic loading-unloading tests will decrease the stress value and small loading rate will also make a small



stress value. Thus materials behave a larger equilibrium speed and a more obvious softening effect in the cyclic tests with a decreasing loading order. Correspondingly, an increasing loading rate sequence will weaken the softening effect caused by cyclic tests and slower the equilibration of styrene SMP (as shown in Fig. 9(b)). Interestingly, the stress for loading rate of 5%/min shown in Fig. 9(b) is larger than the value in Fig. 5(b) in cycle 3. That is to

**Fig. 6** The stress-strain curve of shape-memory polymer at different strain rates in cycle 4





**Fig. 8** The shape recovery behavior of samples deformed below Tg

say, the strengthening effect on stress induced by the increasing loading rate is larger than the softening effect caused by cyclic tests.

# 3.3 The influence of loading levels on the mechanical behavior of styrene SMP

Figure 10 exhibits different stress curves at different strain levels with a same loading rate of 1%/min. Strain levels of 1%, 5% and 15% are selected due to the mechanical behavior of





styrene SMP, which, respectively, belong to the region before yielding, region after yielding and cold-drawing region in tension. After the first loading-unloading cycle, the responses of later cycles loading gradually approach to same in the region before yielding (as shown in Fig. 10(a)). In the region between after yielding and before cold-drawing, the stress undergoes a great decrease after the first cycle but rapidly tended to be equilibrium in the rest cycles (as shown in Fig. 10(b)). When target strain is performed at cold-drawing region, the stress value at the maximum strain is the same for all four cycles. It can be said that if styrene SMP sample is deformed in the after-yield region and cold-drawing region, the sample will rapidly go into an equilibrium state and be independent of the cycle number. These two cases provide a steady work performance for applications. This is an interesting finding for the application of styrene SMP.

With cycle numbers increasing, the stress-strain curve of styrene SMP shows a "Mullins effect" (as shown in Fig. 11), which is the special mechanical property for filled rubber. From Fig. 10, we can realize that the stress curve of 1% strain will be gradually enveloped by the curve of 5% strain with cyclic numbers increase. We also find that the stress curve of 5% strain is enveloped by the curve of 15% strain with cyclic numbers increase. We can deduce that the broken elastic phase during the tension process played the role of filler for the rubbery phase and thus the stress-strain curve of the styrene SMP sample will be dependent of the maximum loading history. Furthermore, the equilibrium cyclic mechanical behavior





of styrene SMP will be decided by the largest strain response with no relationship of whether the programmed strain belongs to the post-yield region, yield region or cold-drawing region.

To evidence the existence of "Mullins effect" in styrene SMP, two group tests based on the tested samples were conducted as follows: one group was deformed at the sequence of 1%, 2% and 3% and the other was the opposite order. Results as shown in Fig. 12 reveal that styrene SMP really presents the "Mullins effect" in temperature lower than Tg. This phenomenon provides a new method to predict the response of smaller strain levels from

**Fig. 11** Comparison of strain-stress curve in the cycle 2, cycle 3 and cycle 4





the stress-strain curve of large strain level. For styrene SMP, we can simplify loading and unloading process as a linear response (as shown by green dash pot in Fig. 11), thus the equilibrium response can be written as

$$\sigma_{\varepsilon} = \frac{\varepsilon}{\varepsilon_0} \cdot \sigma_{\varepsilon_0} \tag{1}$$

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 $\sigma_{\varepsilon_0}$  and  $\varepsilon_0$  are the known strain and stress,  $\varepsilon$  is the required strain and  $\sigma_{\varepsilon}$  is the stress of required strain to solve. For example, if we make the stress curve of 15% strain as the known curve, we can easily predict the stress value is about 0.15 MPa when strain reaches 5% from Eq. (1). The prediction value is near the true stress of 0.167 MPa read in the stress curve of 5% strain in the third cycle in Fig. 9(b).

#### 3.4 The influence of loading level on shape recovery behavior of styrene SMP

In this section, we will talk about the free shape-memory recovery results with different deformation levels. To get a clearer understanding of the shape recovery properties of styrene SMP, we give a definition so that the real-time shape recovery speed is equal to the ratio of the time differential of real-time strain and programmed strain.

$$V_r = \frac{1}{\varepsilon} \cdot \frac{d\varepsilon}{dt} \tag{2}$$

The shape recovery ratio could be defined as

$$R_r = \frac{\varepsilon_t - \varepsilon_r}{\varepsilon_r} \tag{3}$$

 $\varepsilon_t$  is the real-time strain in the recovery cycle and  $\varepsilon_r$  is the fixed strain after unloading cycle.

From Eqs. (2) and (3), we provide the shape recovery ratio of styrene SMP (as shown in Fig. 13) and shape recovery speed of styrene SMP (as shown in Fig. 14) of different strain levels.

Figure 13 shows the shape recovery ratio on styrene SMP with different strain levels. At the beginning of free shape recovery, recovery ratio of styrene SMP increases slightly due to the thermal expansion and then decreases. At the end of the recovery process, the larger deformation level exhibits a merely larger recovery ratio but needs more recovery time to be equilibrium. The larger strain level makes the material a smaller free volume which means a larger restriction for shape recovery, so the recovery speed evolution in the last stage of FSR together in Fig. 14. The results show that the shape recovery speed increase first and then decrease with the temperature increasing. We define the starting recovery temperature of styrene SMP as the temperature where shape recovery speed begins negative and shape recovery temperature as where shape recovery reaches the biggest. At the same loading



and heating condition, all three strain levels behave a nearly same characteristics of starting temperature and characteristics of shape recovery temperature. That is to say, the loading level has little effect on the shape recovery behavior of styrene SMP at the same loading rate and heating rate.

#### 3.5 The influence of loading rate on shape recovery behavior of styrene SMP

Figures 15 and 16 show the shape recovery behavior at different loading rates. It is observed that the deformation of styrene SMP recovers more with a larger strain rate. In rubbery state, the molecular chain of styrene SMP will change the conformation of segment to adapt the action of external force. In loading step, a larger loading rate will result in a higher stiffness of molecular chain. The stress of styrene SMP increases with the increasing loading rate (as shown in Fig. 17(a): from the 0.9 MPa with 5%/min strain rate to 0.12 MPa with 50%/min strain rate). Once the temperature is decreased, the motion of segment and free volume are frozen. The stress decreases first and then increases for the combined action of relaxation and the frozen chain segment. And a larger strain rate behaves a larger stress (as shown in Fig. 17(b)). We can see that a stress of 0.13 MPa of 50% strain rate and a 0.11 Mpa of 5% strain rate at the last of cooling step. With temperature increasing above Tg, the motion of molecular chain is activated again and the elasticity stored in molecular chain will provide the energy to recover to its original shape. So it is clear how to explain that an increasing







strain rate stores more elasticity in a molecular chain, which provides more driving force for shape recovery.

In rubbery state, the molecular chain obtains enough motion energy and free volume. Thus, the relaxation time of chain segment is small enough which can be compatible with the loading rate. Therefore the increased loading rate does not induce an obvious increase



of stress. A larger strain rate will result in a larger recovery ratio but the increase of the recovery ratio is not obvious.

Time(min)

# **3.6** The influence of preconditioning loading-unloading treatment on shape recovery behavior of styrene SMP

Figures 18 and 19 show three types of free shape recovery behaviors at different loading conditions. Compared with sample without loading-unloading treatment, the sample experiences a repeat of loading-unloading treatment with strain rate of 5% for three times exhibit a better shape recovery property. During the loading-unloading process at temperature lower than Tg and at Tg, sample yields and stress softens by developing the super-molecular structure. The super-molecular structure is regrouped by the physical cross-linking of large molecular chain to favor the deformation. Once the temperature is heated above Tg, the regrouped super-molecular structure will be released due to the motion of molecular chain. From the results in Fig. 18, we can deduce that the super-molecular structure does not release completely during heating. So the shape recovery test with prior cyclic loading-unloading test obtains the larger shape recovery ratio. While the super-molecular structure is easier to develop at low temperature during yielding, the sample loaded-unloaded at temperature lower than Tg can obtain the highest recovery ratio.

70

60

40

30

20

10

emperature(°C 50



### 3.7 The comparison of styrene SMP deformed at temperature lower than Tg and deformed at temperature higher than Tg

Figure 20 shows the comparison of samples which are deformed at two temperatures (T > Tg and T < Tg). The results reveal that the sample deformed at temperature lower than Tg has a larger recovery speed and a smaller recovery time. We can see a similar recovery behavior after reaching the shape recovery temperature, while the shape recovery behavior is quite different before reaching the shape recovery temperature. In the glass state, the deformation of styrene SMP is the forced high-elastic deformation which is provided by the molecular elongation. Once the temperature is increased above Tg, the chain segment is activated and coil again. Sample rapidly recovers to its original shape. Sample deformed at high temperature has high compliance and the deformation state is a high-elastic deformation. The free volume decreases due to the molecular motion. When the temperature is decreased lower than Tg, the molecular will be more tightly packed than the sample which has no heating and cooling process. Therefore, the sample deformed at temperature lower than Tg has smaller restriction to recover its initial shape. The sample deformed at temperature higher than Tg needs more time and higher temperature to recover.

#### 4 Conclusion

In this paper, we demonstrated the influence of loading level and loading rate on the thermomechanical behavior of styrene shape-memory polymer. The experimental results show that loading level and loading rate have great influence on the mechanical behavior of thermosetting shape-memory polymer at temperature lower than Tg but little on the shape recovery behavior. A prior cyclic loading-unloading treatment will make styrene SMP obtain a better shape recovery property due to the formation of super-molecular structure. A cyclic loadingunloading test will make styrene SMP enter into rubbery state at low temperature and show a better shape recovery behavior. We hope all these new findings would provide guidelines for SMP applications.

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