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Experimental study on uniaxial time-dependent ratcheting of a polyetherimide polymer*

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Abstract: The uniaxial ratcheting behavior of a polyetherimide (PEI) polymer 'TECAPEI' was studied using stress-controlled cyclic loading at room temperature, including both cyclic tension-compression with non-zero tensile mean stress and tension-unloading tests. The experimental observations were focused on the time-dependent ratcheting of the PEI polymer revealed in cyclic tests at diverse stress rates and with different peak stress holding times. The results showed that the PEI polymer shows obvious ratcheting deformation; i.e., the ratcheting strain accumulates progressively in the tensile direction during stress-controlled cyclic tests with non-zero mean stress. The ratcheting is highly dependent on the applied mean stress and stress amplitude, and is also characterized by a strong time-dependency during the cyclic stressing at diverse stress rates and with different peak stress holding times. The time-dependent ratcheting of the PEI polymer is caused mainly by its remarkable viscosity. A comparison of the ratcheting occurring before and beyond the ultimate stress point of the PEI polymer showed that the ratcheting beyond the ultimate stress point is more significant than that occurring before that point.

Key words: Polyetherimide (PEI) polymer, Uniaxial ratcheting, Time-dependence, Cyclic loading, Viscosity
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1 Introduction

Polyetherimide (PEI) polymer has been widely applied in the manufacture of many structural components in engineering areas including the aerospace, marine, and transportation industries because of its excellent toughness, hardness, and flexural and torsional strength (Ramiro *et al.*, 2006). Such components are often subjected to cyclic loading, especially a kind of cyclic stressing with non-zero mean stress. To better assess the safety and to estimate the fatigue life of structural components made from PEI polymer, it is necessary first to observe and model the cyclic deformation behavior of the PEI polymer materials.

In the last decade, the cyclic deformation of polymer materials including their ratcheting behavior shown during asymmetrical stress-controlled cyclic loading has been studied extensively by many researchers, both experimentally and theoretically. For instance, many previous studies have reported epoxy resins (SP Amperg 20™ and DER332), ultra-high molecular weight polyethylene, Epon 826 epoxy resin, nylon 66 fibers, polytetrafluoroethylene (PTFE) fluoropolymer, high-density polyethylene, polymethyl methacrylate (PMMA), unsaturated polyester resin, vulcanized natural rubber, and isotactic polypropylene (Bardella, 2001; Bergström *et al.*, 2002; Hu *et al.*, 2003; Shen *et al.*, 2004; Chen and Hui, 2005; Xia *et al.*, 2005a; 2005b; Averett *et al.*, 2006; Drozdov and Christiansen, 2007; Liu *et al.*, 2008; Yu *et al.*, 2008; Zhang and Chen, 2009; Kang *et al.*, 2009; Wang *et al.*, 2009; Drozdov, 2010). These studies provided many important results such as the finding that the ratcheting deformation of polymers is dependent on the applied stress level, loading history, loading rate, loading path, and ambient temperature,

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and significant progress was made in solving the problems faced during the design and assessment of engineering components made from polymer materials. However, no data were obtained about the ratcheting of PEI polymers. Previous studies showed that ratcheting behavior differs greatly between different polymer materials. Thus, we cannot directly deduce the ratcheting evolution of untested polymer materials from the results obtained from tests of other polymers. It is essential that investigations of the ratcheting of PEI polymers are carried out so that a reasonable constitutive model can be constructed.

In this study, the uniaxial ratcheting of PEI polymer 'TECAPEI' was investigated using detailed stress controlled cyclic tests at room temperature. The effects of some time-related factors, such as stress rate and peak stress hold on the ratcheting of the PEI polymer were comprehensively addressed. Because of the special mechanical response of polymer materials, the ratcheting was also investigated using cyclic tests performed before and beyond the ultimate strength point of the PEI polymer was reached. The results obtained are of great significance for the construction of a cyclic constitutive model of ratcheting of PEI polymer.

2 Experimental

PEI polymer bars, as received, were machined into cylindrical specimens with a gauge length of 10.0 mm and a cross-sectional diameter of 6.0 mm for uniaxial tests. Test machine MTS809-25KN (MTS Co. Ltd., USA) was used. The loading process was controlled by a Teststar-II control system. A tensile extensometer was used to measure the axial strain. All tests, including monotonic tension, cyclic tension-compression, cyclic tension-unloading, and creep tests, were carried out at room temperature. The tests were performed at diverse loading rates and with or without peak stress hold in order to determine the time-dependent deformation of the PEI polymer.

The axial ratcheting strain ε_r was determined by

$$\varepsilon_r = \frac{\varepsilon_{max} + \varepsilon_{min}}{2}, \tag{1}$$

where ε_{max} and ε_{min} are the measured maximum and minimum axial engineering strains respectively in each cycle. The ratcheting strain rate is defined as the increment of ratcheting strain ε_r after each cycle (Kang, 2006; Kang *et al.*, 2006; 2009).

3 Results and discussion

3.1 Monotonic tension

The PEI polymer was first tested using displacement controlled monotonic tension to determine its basic mechanical properties and to set suitable stress levels for ratcheting tests. The loading conditions were set as follows: (1) one specimen was first tensioned up to an engineering strain value of 14.5%, and then unloaded to zero-stress point where it was maintained for 30 min (Fig. 1a); (2) three specimens were stretched at displacement rates of 0.5, 0.05 and 0.005 mm/s respectively until they fractured (Fig. 1b).

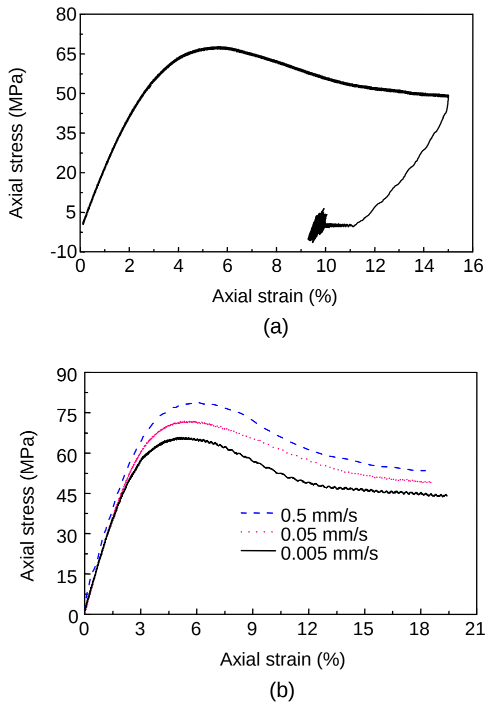

Fig. 1 Tensile stress-strain curves of the PEI polymer
(a) One specimen was first tensioned up to an engineering strain value of 14.5%, and then unloaded to zero-stress point where it was maintained for 30 min (displacement rate 0.005 mm/s); (b) Three specimens were stretched at different displacement rates until they fractured

The PEI polymer shows a tensile stress-strain curve typical of amorphous polymers, i.e., a linear response phase followed by a nonlinear hardening phase before the ultimate strength point is reached, and an apparent continuous softening phenomenon beyond the ultimate strength point and up to the point of failure of the polymer (Fig. 1a). During the unloading, a nonlinear elastic response occurs. After unloading to zero-stress point, the residual strain changes gradually from 11.2% to 9.7% over 30 min as a result of the reversible viscoelastic deformation of the PEI polymer. Fig. 1b implies that the polymer deforms rate-dependently and its stress-strain response at a higher loading rate is greater than that at a lower loading rate. This can be attributed mainly to the intrinsic rate-dependent response of macromolecular chains in polymers to external loading, as explained by Bicakci and Cakmak (2002) for PEI polymers and by Ramiro *et al.* (2006) for other polymers.

3.2 Ratcheting behavior

Two types of ratcheting tests were conducted in view of the distinctive hardening and softening behaviors shown by the PEI polymer tested before and beyond its ultimate stress point. One test involved stress-controlled tension-compression cycling with a peak stress lower than the ultimate stress. The other test involved first tensioning up to a strain of 7% by displacement-controlled loading, unloading to zero-stress point where it was maintained for 2 min, and then testing by stress-controlled cyclic tension-unloading to observe the ratcheting of the polymer beyond its ultimate stress.

3.2.1 Ratcheting before ultimate stress point

First, the ratcheting behavior of the PEI polymer before its ultimate stress point and its dependence on applied stress levels are discussed. The experimental results obtained in the cyclic tests with constant stress amplitude and various mean stresses or with constant mean stress and various stress amplitudes are illustrated in Figs. 2a, 2b and 2c, respectively.

The PEI polymer showed obvious ratcheting deformation during the stress cycling even if the applied peak stress was lower than its ultimate strength. The ratcheting strain increased progressively with the number of cycles, while the ratcheting strain rate decreased quickly after 10–15 cycles, and then reached a nearly stable state with a constant deformation rate. The ratcheting of PEI polymer also depends on the applied stress levels. The ratcheting strain produced in the stress cycling with higher mean stress or stress amplitude was larger than that obtained with lower mean stress or stress amplitude (Figs. 2b and 2c). These results are similar to those obtained in studies of other polymers reviewed in Section 1.

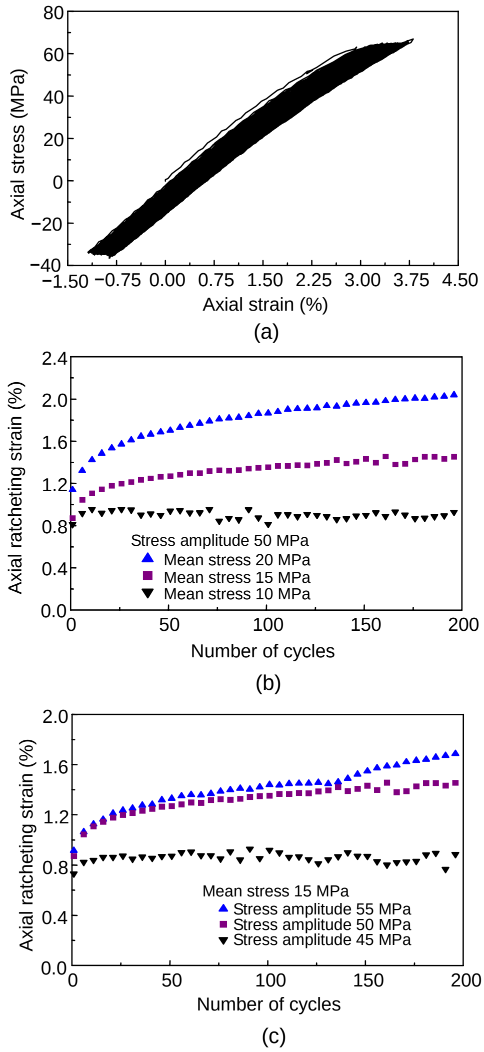

Fig. 2 Ratcheting behavior before the ultimate stress point (all carried out at a stress rate of 30 MPa/s)
(a) Hysteresis loop with a mean stress of 15 MPa, stress amplitude of 50 MPa; (b) Ratcheting strain vs. number of cycles with various mean stresses; (c) Ratcheting strain vs. number of cycles with various stress amplitudes

Comparison of the results (Figs. 2b and 2c) shows that increasing the mean stress cause more significant ratcheting in the polymer than increasing the stress amplitude, if the increment of stress is the same. This result is similar to that observed by Kang *et al.* (2009) for unsaturated polyester resin.

Previous observations of Shen *et al.* (2004), Chen and Hui (2005), Zhang and Chen (2009), and Kang *et al.* (2009) for other polymers showed a strong time-dependency in the ratcheting of polymer materials. Therefore, it is important to investigate the effects of some time-related factors, such as stress rate and the presence or absence of peak stress hold, on the ratcheting of PEI polymer. The results obtained from cyclic loading with the same stress levels (i.e., mean stress of 15 or 32.5 MPa and stress amplitude of 50 or 32.5 MPa, respectively), but at different stress rates and with different holding times at the peak stress point are shown in Figs. 3a and 3b. The PEI polymer also presents a remarkable time-dependent ratcheting behavior, and the ratcheting strains produced at lower stress rates and with longer peak stress holding times are much larger than those obtained at higher stress rates and with shorter holding times, as found for other materials by Kang (2006) and Kang *et al* (2006; 2009).

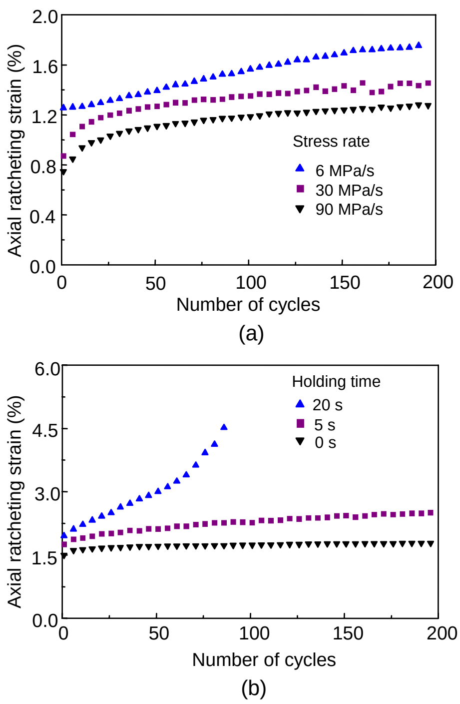

Fig. 3 Time-dependent ratcheting before the ultimate stress point

(a) Ratcheting strain vs. number of cycles at various stress rates (mean stress of 15 MPa, stress amplitude of 50 MPa); (b) Ratcheting strain vs. number of cycles with different peak stress holding times (mean stress of 32.5 MPa, stress amplitude of 32.5 MPa, and stress rate of 30 MPa/s)

From the viewpoint of physical analysis, the viscosity of a polymer is the key factor determining such time-dependence (Bicakci and Cakmak, 2002; Guillemenet *et al.*, 2002; Ramiro *et al.*, 2006). Thus, the higher viscous deformation is produced during cyclic loading at a lower stress rate and with longer holding time, which then results in a larger ratcheting strain, similar to the case of SS304 stainless steel at high temperature (Kang *et al.*, 2006). Note that apparent ratcheting occurs in the cyclic tension-unloading without peak stress hold even if there is no reverse compression (Fig. 3b). This differs from the results obtained for many steels at room temperature, e.g., for SS304 stainless steel by Kang *et al.* (2006), where no obvious ratcheting occurs because of the lack of plastic strain accumulation in the subsequent cyclic tension-unloading. Such a difference is likely to be caused by the significant viscosity of PEI polymer and the much weaker viscosity of steels at room temperature.

Furthermore, decreasing the stress rate or increasing the holding time at the peak stress point not only makes the value of the ratcheting strain larger, but also changes the evolution features of the ratcheting deformation (Figs. 3a and 3b). During cyclic loading at a stress rate of 6 MPa/s, the ratcheting strain increased almost linearly with the increasing number of cycles. With a holding time of 20 s, the ratcheting strain rate increased quickly after a certain number of cycles, and then resulted in the failure of the specimen within a much smaller number of cycles.

3.2.2 Ratcheting beyond ultimate stress point

Fig. 4 shows the experimental results of ratcheting obtained in stress-controlled cyclic tension-unloading tests of the PEI polymer beyond its ultimate stress point; i.e., the second type of ratcheting tests described in Section 2.

The results showed that apparent ratcheting occurred during the cyclic tension-unloading beyond the ultimate stress point, and that it depended greatly on the applied stress level and stress rate. The previous deformation produced in the monotonic tension up to a strain of 7% did not restrain the occurrence of ratcheting in the subsequent cyclic tension-unloading. As in the case of tests carried out before the ultimate stress point was reached (subsection 3.2.1), the time-dependent ratcheting of the polymer beyond its ultimate stress point (Fig. 4c) was also caused mainly by its viscosity (Bicakci and Cakmak, 2002; Guillemenet *et al*, 2002; Ramiro *et al.*, 2006).

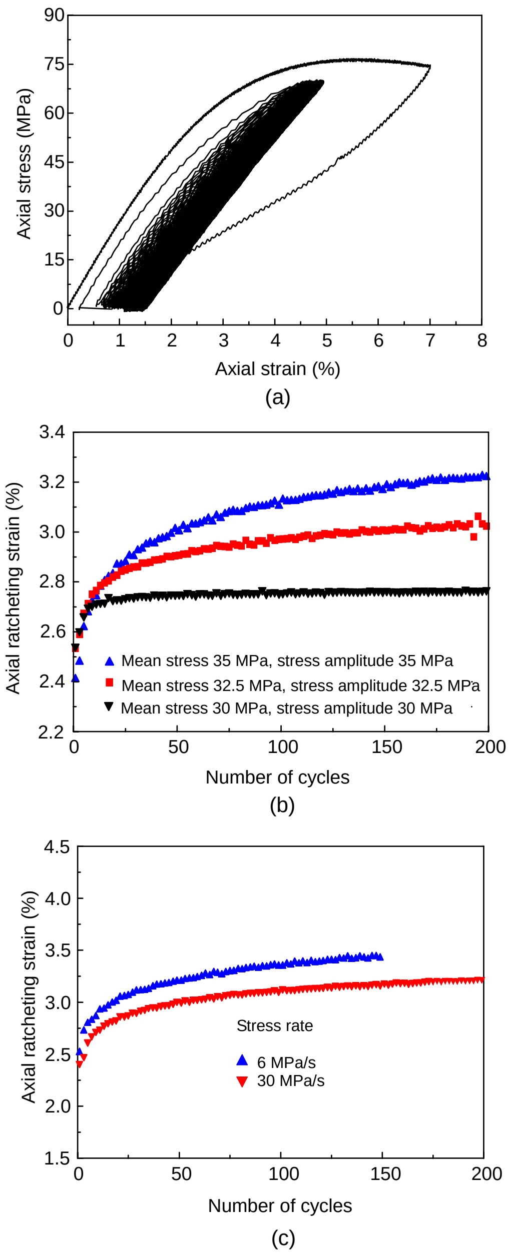

Fig. 4 Ratcheting of the PEI polymer beyond its ultimate stress point
(a) Cyclic stress-strain curve with mean stress of 35 MPa and stress amplitude of 35 MPa at a stress rate of 30 MPa/s; (b) Ratcheting strain vs. number of cycles under different stress levels (stress rate 30 MPa/s); (c) Ratcheting strain vs. number of cycles at two stress rates (mean stress 35 MPa, stress amplitude 35 MPa)

3.3 Further discussion

A direct comparison of the ratcheting behaviors of the PEI polymer produced in the cyclic tests before and beyond its ultimate stress point is shown in Fig. 5.

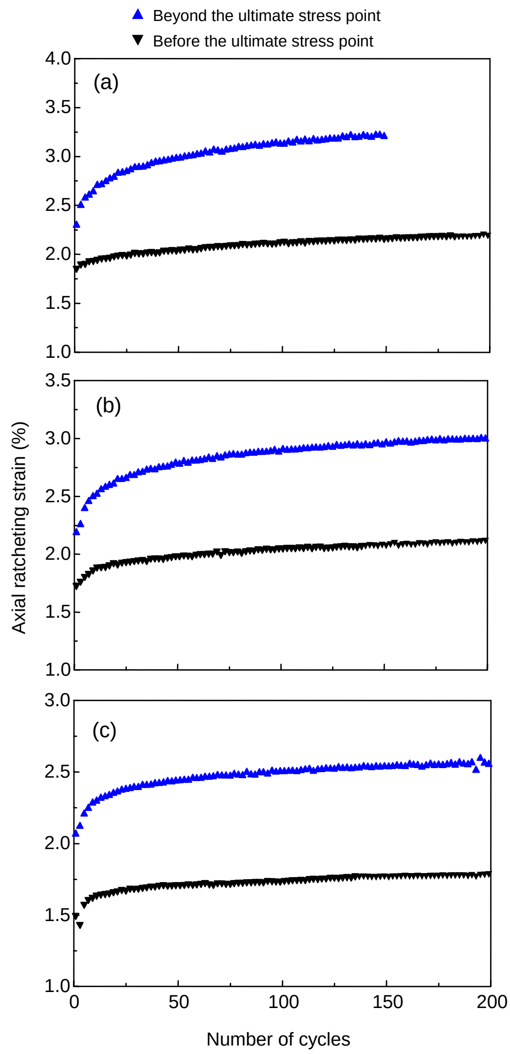

Fig. 5 Comparsion of the ratcheting before and beyond the ultimate stress point
(a) At a stress rate of 6 MPa/s, mean stress of 35 MPa, and stress amplitude of 35 MPa; (b) At a stress rate of 30 MPa/s, mean stress of 35 MPa, and stress amplitude of 35 MPa; (c) At a stress rate of 30 MPa/s, mean stress of 32.5 MPa, and stress amplitude of 32.5 MPa

It can be concluded that the ratcheting occurring beyond the ultimate stress point is more significant than that occurring before. The ratcheting strain and its rate are both higher in the cyclic tests beyond the ultimate stress point than in those before the ultimate stress point is reached (Figs. 5a, 5b, and 5c). This implies that previous tensile deformation up to a strain of 7% (i.e., beyond its ultimate stress) will accelerate the development of ratcheting for a PEI polymer. This corresponds to the softening behavior of the polymer shown in the monotonic tension test at the stage beyond the ultimate stress point. This response is different from that of metal materials, where previous plastic deformation produced by overloading greatly restrains the occurrence of ratcheting in subsequent cyclic loading with lower stress levels because of strong strain hardening. For a PEI polymer, during deformation beyond its ultimate stress point, a remarkable strain softening occurs, even if the polymer can continue to deform stably till the failure of the material, because of its special molecular structure (Bicakci and Cakmak, 2002; Guillemenet *et al*, 2002; Ramiro *et al.*, 2006). The strain softening feature and the viscosity of the polymer result in increases in the ratcheting strain and its rate during cyclic loading beyond the ultimate stress point.

Previous explanations attributed the strong time dependence of the ratcheting to the viscosity of the PEI polymer at room temperature. But what else contributes to the time-dependent ratcheting of this polymer? Is the creep strain produced during peak stress hold and/or the accumulation of inelastic strain caused by cyclic loading? To answer these questions, a creep test was performed and compared with a cyclic tension-unloading test with peak stress hold. The evolution of creep strain at room temperature is shown in Fig. 6. The holding time at constant peak stress (65 MPa) was 1 h. An obvious creep deformation occurred for the PEI polymer even at room temperature. The creep strain increased with the increasing holding time, but the strain rate decreased rapidly at the onset of the creep. After some time, a steady creep stage with a very low strain rate was reached. The tertiary creep stage was not observed in the creep test with the prescribed stress of 65 MPa and within the holding time of 1 h. Comparison of the strain values produced in the cyclic loading and creep tests with the same peak stress shows that the strain value obtained in the cyclic loading with peak stress hold (4.69% in the case of holding for 20 s, Fig. 4c) is much higher than that produced in the creep strain (3.36%), even if the total holding time of the former (about 2000 s) is shorter than that of the latter (about 3600 s). This means that the creep strain produced during the peak stress hold in the cyclic loading is only one cause of time-dependent ratcheting, and accumulation of inelastic strain (viscoelastic or viscoplastic strain) caused by cyclic loading is also an important contributor.

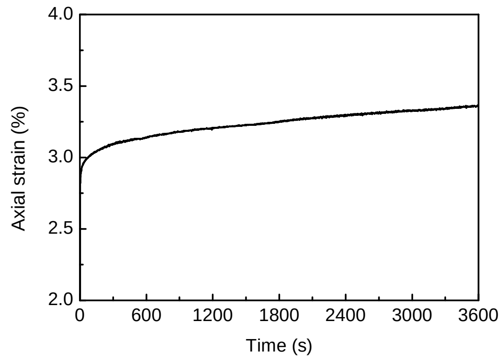

Fig. 6 Creep curve of the PEI polymer with a constant stress of 65 MPa at room temperature

4 Conclusions

1. Polyetherimide (PEI) polymer shows a strongly rate-dependent deformation because of its viscosity at room temperature. A stable deformation with strain softening occurs beyond its ultimate stress point.

2. PEI polymer shows an obvious ratcheting deformation under stress-controlled cyclic loading with a peak stress lower than its ultimate stress. The ratcheting depends greatly on the applied stress level, and the time-dependent ratcheting strain at lower stress rates or with longer peak stress holding times is greater than that at higher stress rates or with shorter holding times.

3. Time-dependent ratcheting also occurs in the PEI polymer during the cyclic stressing beyond its ultimate stress point, and this also depends on the stress level and stress rate. The ratcheting beyond the ultimate stress point is more significant than that before because of the stable strain softening that occurs during deformation beyond the ultimate stress point of the polymer.

4. Time-dependent ratcheting deformation cannot be attributed only to the creep strain produced at lower stress rates and with peak stress holds. Total ratcheting strain results from a combination of cyclic accumulated viscoelastic or viscoplastic strain and creep strain.

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