



Material Behaviour

Effect of relative humidity on uniaxial cyclic softening/hardening and intrinsic heat generation of polyamide-6 polymer

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ABSTRACT

Strain-controlled uniaxial cyclic tests on a polyamide-6 (PA6) polymer with various relative humidity (RH) levels were performed in a room environment. The effects of RH on the cyclic softening/hardening and intrinsic heat generation of the PA6 are discussed by using the experimental data obtained in the cyclic tests with various applied strain amplitudes, mean strains and strain rates. It is observed that the cyclic softening/hardening of PA6 depends greatly on the applied strain amplitude and strain rate, and the cyclic softening observed in the cyclic tests with a larger strain amplitude and at a higher strain rate is mainly caused by the remarkable temperature rise due to the intrinsic heat generation. The variation of relative humidity does not change the cyclic softening/hardening feature of PA6, but does influence the extent of cyclic softening/hardening; an apparent mean stress relaxation occurs in the asymmetrical strain-controlled cyclic tests, and its extent increases with increasing mean strain but decreases with increasing relative humidity.

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1. Introduction

Polyamide-6 (PA6) has been widely used in the automotive industry, electronics and other engineering products as a structural material due to its high specific stiffness, good heat insulation and high toughness, etc. [1–3]. The PA6 components are often subjected to cyclic loading and simultaneously encounter a change of environment humidity. During the cyclic deformation, a significant intrinsic heat generation can occur due to the viscosity of polymer, which will result in a temperature rise in the polymer if the loading frequency or loading rate is high enough. Such an obvious temperature rise greatly influences the cyclic stress-strain response of the polymer, since the cyclic deformation of PA6 is sensitive to the variation of ambient temperature, as mentioned by Shan et al. [4]. On the other hand, PA6 is hygroscopic due to its specific hydrophilic molecular structure, i.e., amide group (–NH–CO–). Thus, it is necessary to study the cyclic deformation of PA6 with different relative humidity (RH) levels by addressing the effect of RH on the cyclic softening/hardening and intrinsic heat generation, so that the design and reliability assessment of PA6 components can be

achieved more reasonably.

In recent decades, many experimental and theoretical researches have been performed to investigate the thermo-mechanically coupled deformation of polymers. The effects of loading mode, loading level, loading rate (loading frequency), ambient temperature and heat conducting medium on the thermo-mechanical responses of polymers have been extensively studied. Tauchert et al. [5] performed torsional oscillation tests to measure the effects of ambient temperature, heat conducting medium, loading rate and torsional-angle amplitude on the temperature rise in polyethylene (PE). Rittle et al. [6–8] investigated the temperature rise occurring in stress-controlled compressive-compressive cyclic tests with relatively high stress levels, and its effect on the uniaxial fatigue failures of polycarbonate (PC) and polymethyl methacrylate (PMMA). Shrestha et al. [9] also conducted uniaxial symmetrical stress- and strain-controlled cyclic tests at different loading rates and with different loading levels to measure the temperature rise in poly (ether-ether-ketone) (PEEK), and they found that the temperature rise played an important role in the fatigue failure of PEEK. Mortazavian et al. [10] studied the effects of the loading frequency and self-heating on the fatigue of thermo-plastic polymers and their composites. Chen et al. [11] studied the intrinsic heat generation occurring in the cyclic deformation of ultrahigh molecular weight polyethylene (UHMWPE) with

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different loading modes and loading levels, as well as at different loading rates, and discussed the effect of temperature rise on the cyclic softening/hardening of UHMWPE. Benaarbia et al. [12,13] performed stress-controlled tension-tension fatigue tests on polyamide-66 (PA66) and investigated the effects of relative humidity and loading frequency on the thermo-mechanical response and ratchetting of PA66. In practical applications, the cyclic softening/hardening of polymers plays an important role in the design and reliability of polymeric components. However, cyclic softening/hardening and intrinsic heat generation of PA6 with different RH levels have not been discussed in the existing literature.

Therefore, in this work, the cyclic softening/hardening of PA6 with different RH levels was investigated by performing a series of strain-controlled uniaxial cyclic tests with various strain levels and at different strain rates. The effects of the applied strain amplitude, mean strain and strain rate on the cyclic softening/hardening and intrinsic heat generation of PA6 were first examined by simultaneously measuring the variation of specimen surface temperature. Then, the effect of RH on the intrinsic heat generation and cyclic softening/hardening of PA6 was addressed. Some significant conclusions were obtained, which are useful to construct a hygro-thermo-mechanical coupled cyclic constitutive model of polymers in future work.

2. Experimental procedure

The PA6 used in this work was TECAST L, with density, glass transition and melt temperature of 1150 kg/m³, 45 °C (in the dry state) and 220 °C, respectively. Dumb-bell shaped specimens with a gauge length of 12 mm and section diameter of 6 mm were machined from the as-received PA6 bars with a diameter of 20 mm. The test machine was a MTS858 Bionix-5kN. The test data were collected by a Flex-Text 40 control system. An MTS axial extensometer (634.31F-24) with a gauge length of 10 mm was used to measure the axial strain, the measurable strain range of which is from –20% to 40%. The temperature variation on the surface of specimen was measured by a FLIRA655sc infrared thermometer at an ambient temperature of 25 °C controlled by an air conditioner (the fluctuation was less than 1.5 °C).

Strain-controlled monotonic tensile and cyclic tensile-compressive tests were performed on PA6. In the monotonic tensile tests, the specimens were first loaded to an axial nominal strain of 25% under strain-controlled loading at a strain rate of 0.005 s⁻¹; and then were unloaded to zero stress under a stress-controlled mode at a stress rate of 1.8 MPa/s (50 N/s). Finally, they were held at zero stress for one hour to assess the recoverable part of the residual strain after the unloading. In the cyclic tensile-compressive tests, including the symmetrical and asymmetrical ones, the cyclic softening/hardening and intrinsic heat generation of PA6 were investigated. Four strain amplitudes (i.e., 1.0, 1.75, 2.5 and 3.0%) and four strain rates (i.e., 0.005, 0.02, 0.05 and 0.1 s⁻¹) were selected for the symmetrical cyclic tests; three mean strains (i.e., 1.0, 1.5 and 2.0%) and a constant strain amplitude of 1.5%, as well as a constant stress rate of 0.02 s⁻¹ were prescribed for the asymmetrical ones. Furthermore, the strain-controlled cyclic tests were also conducted on PA6 with various RH levels, i.e., 0, 1.26 and 2.58%. It should be noted that the humidity is not actually uniform in the sample section since the diffusion of water molecules in the sample requires a certain gradient. However, the sample's section diameter is only 6 mm, and the samples with RHs of 1.26% and 2.58% are immersed in water for a long time (e.g., 50 and 233 h, respectively). Hence, the humidity gradient in the samples is relatively small and can be neglected.

To illustrate the experimental results more clearly, some parameters used in this work are defined as following:

(1) Relative humidity level RH in samples:

$$RH = \frac{m_t - m_0}{m_0} \times 100\% \quad (1)$$

where, m_0 is the mass of dry sample, and m_t is the mass of the sample after it is immersed in water for certain time t . It should be noted that the dry sample (i.e., with RH = 0) was obtained by putting it in a desiccator at room temperature for enough time to reach constant mass.

(2) Responding stress amplitude σ_a and mean stress σ_m :

$$\sigma_a = \frac{\sigma_{\max} - \sigma_{\min}}{2} \quad (2)$$

$$\sigma_m = \frac{\sigma_{\max} + \sigma_{\min}}{2} \quad (3)$$

where, σ_{\min} and σ_{\max} are the minimum and maximum nominal axial stress per cycle, respectively.

(3) Percentage of relative stress amplitude (PRSA):

$$PRSA = \frac{\sigma_{aN} - \sigma_{a1}}{\sigma_{a1}} \times 100\% \quad (4)$$

where, σ_{a1} and σ_{aN} are the responding stress amplitudes obtained in the first and N -th cycles, respectively.

3. Results and discussion

3.1. Monotonic tension

The monotonic tensile tests on PA6 with different RH were carried out first. The temperature variation on the surface of the specimen was measured by a FLIRA655c infrared thermometer. The results are shown in Fig. 1.

It can be concluded from Fig. 1 that: (1) the responding stress of PA6 decreases with increasing RH, as shown in Fig. 1a. From a chemical point of view, the water molecules will penetrate into the molecular chain structure of PA6, which can weaken the intermolecular forces and make the molecular segments more flexible [14]. Also, slight strain softening is observed for the PA6 with RH = 0% and RH = 1.26% after yielding. That is, the responding stress slightly drops when the applied strain increases from about 3.5% to 25%. However, the feature of strain softening becomes weaker with increasing RH, and finally disappears at RH = 2.58%, as shown in Fig. 1a. (2) The temperature on the specimen surface decreases slightly with increasing applied strain at the first stage of monotonic tension but, when the applied strain becomes high enough, it monotonically increases with increasing strain, as shown in Fig. 1b, which is similar to that observed in other polymers, e.g., UHMWPE and PA66 [11,13]. The decreased temperature of the specimen at the first stage of tensile deformation is caused by the standard thermo-elastic effect firstly observed by Lord Kelvin [15]. However, with increasing deformation, the intrinsic dissipated energy gradually accumulates. When the dissipated energy becomes larger than the elastic energy, i.e., when the applied strain becomes large enough, the temperature on the specimen surface will increase with increasing strain, as shown in Fig. 1b. It was also found from Fig. 1b that, although the highest surface temperature in the monotonic tensile tests of PA6 apparently decreases, the transition strain, where the temperature changes from a decreased mode to an increased one, increases with increasing RH. (3) As for the stage of zero stress hold after unloading, the residual strain partially

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