



Delineating nature of stress responses during ductile uniaxial extension of polycarbonate glass



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ABSTRACT

We carry out simultaneous mechanical and IR-thermal-imaging-based temperature measurements of tensile extension on untreated, milled (mechanically “rejuvenated”) and melt-stretched bisphenol A-polycarbonate (PC). The extension is found to cause significant buildup of both excess internal energy u_2 and plastic dissipation. The magnitude of u_2 is one to two orders of magnitude higher than the energy involved in rubbery elastic deformation. While the ratio of u_2 to the mechanical work w decreases with increasing rate of extension for untreated PC, milled PC is found to be more dissipative at lower rates. Homogeneous extension of melt-stretched PC in the post-yield regime including strain hardening behavior reveals largely non-dissipative responses, emphasizing the plastic deformation of glassy polymer may not be fully dissipative. The experimental results clearly indicate that a significant component of stress can be intrasegmental leading to the observed buildup of internal energy by distortions of covalent bonds. The glassy polymer physics at the chain level complements the more familiar idea of inter-segmental dissipation as the dominant event during plastic deformation.

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1. Introduction

Mechanical properties are an important characteristic of polymer materials. A core question concerns the nature of mechanical stress arising from polymers of high molecular weight under large deformation, either well above or below the glass transition temperature T_g or crystallization temperature T_c . To delineate the origin of stress in either liquid or solid state, both phenomenological and molecular-level viewpoints have been adopted. The task belongs to the realm of polymer rheology when $T > T_g$ and T_m . In the past many decades a disproportionately large number of studies have concentrated on nonlinear polymer melt rheology [1–4]. Although the explicit molecular picture concerning how chain disentanglement takes place in large deformation is still under active development [5–12], we have achieved a satisfactory level of phenomenological understanding [13,14]. In our view [15], it is key to recognize that such strongly viscoelastic liquids as entangled polymers necessarily undergo yielding, i.e., a transition from elastic deformation to flow at high rates of deformation, and it is crucial to explore the condition for chain disentanglement leading to macroscopic yielding. Stress prior to the yield point is largely rubbery like, arising from stretching of the entanglement network.

The challenge is to describe when the elastic deformation ceases and irreversible deformation begins to dominate.

An appreciable amount of research has been carried out to investigate mechanical responses of amorphous polymers in their glassy state at $T < T_g$ [16–40]. Strain hardening is an important phenomenon [21,24,32–35,41–55], typical of ductile deformation of polymer glasses, involving growing stress with strain in the post-yield (plastic flow) regime. Kramer [56] indicated that the microscopic origin of stress had remained elusive, e.g., the cause of “strain hardening” was unclear. More recent activities searched for and identified a dissipative mechanism to explain strain hardening as predominantly arising from plastic flow associated with inter-segmental processes, based on computer simulations [22–26,50–52], and theoretical formulations [55,57–60].

On the other hand, experiments, based on calorimetric detections [61,62], DSC measurements [63,64] and direct temperature readings [65–67], have revealed appreciable buildup of internal energy in the post-yield regime. Simulations also found a small portion of energetic stress at large deformation while the majority of stress was related to plastic arrangements [22,23,50,51,68].

The present study aims to explore further the nature of stress responses as a function of the deformation rate during either inhomogeneous or homogeneous uniaxial extension of ductile bisphenol A-polycarbonate (PC). In contrast to the suggestion that strain hardening is either associated with reduction of conformational entropy [19] or plastic dissipation [50,55,69], we

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demonstrate that ductile extension of polymer glasses could result in a significant buildup of excess internal (potential) energy u_2 of both inter-segmental and intra-segmental origins that well exceeds contributions due to rubbery elastic stretching of the entanglement network. Specifically, we apply IR thermal imaging to measure the internal (kinetic) energy u_1 increase associated with the temperature rise against the mechanical work density w and find $u_2 = (w - u_1)$ to vary with the applied rate in opposing ways depending on whether or not the polymer glass has undergone pre-treatment, i.e., mechanical “rejuvenation”. Based on the various pieces of evidence from elastic retraction of a necked sample to stress relaxation, a microscopic picture is proposed here to explain how plastic deformation can take place while u_2 builds up during large deformation at different rates.

The paper is organized as follows. After the Experimental Section 2 to describe the material characteristics, we present the experimental results in Section 3 and discuss the microscopic origins of u_2 according to the detailed theoretical analyses that evaluate u_2 based on the simultaneous mechanical and thermal measurements. The paper ends with a conclusion in Section 5.

2. Experimental

Bisphenol A-polycarbonate (PC) under study is Lexan TM 141 111, received from Sabic (GE Plastic). The average weight molecular weight is 63 kg/mol, with a polydispersity of 1.58. Its glass transition temperature T_g is 145 °C, measured by a TA Q2000 DSC at a ramping rate of 10 °C/min. In the present work, we study three “types” of PC that are either untreated, mechanical “rejuvenated” by milling, or melt-stretched.

To prepare untreated PC sheets, PC pellets were placed into a 100 mm × 100 mm × 0.8 mm compression mold with two sheets of Kapton HN polyimide film at the surface of both sides in a 50-ton Dake hydraulic press. The pressing temperature was set at 200 °C. PC was first pre-heated between two plates for 30 min, and then pressed with 25 tons of force for another 30 min. The sample was then removed from the press to allow cooling down to room temperature. Dog-bone shaped samples were obtained by a punch press at room temperature with a dog-bone mold (ASTM D-412) involving an effective length of $L_0 = 39$ mm and a width of $W_0 = 3.30$ mm.

The milled samples were prepared by placing an untreated PC sheet with a dimension of 100 mm × 100 mm × 2 mm into a gap between two identical counter-rotating (at 10 rpm) rolls of 150 mm in diameter. Such a milling procedure involves a thickness reduction of 0.1 mm in each passing. After many passes, we achieved an accumulated thickness decrease of 30%. Such milled sheets were then cut into dog-bone (ASTM D-412) shaped with a thickness of $H_0 =$ ca. 1.4 mm, a length of $L_0 = 39$ mm and a width of $W_0 = 3.30$ mm.

To obtain melt-stretched samples, dog-bone shaped PC sheets with middle dimensions of 80 mm × 35 mm × 0.5 mm were first pressed using the same procedure as that adopted to prepare untreated PC. We then fixed the sample onto Instron 5567 and allowed it to relax at 160 °C for 15 min to reach the thermal equilibrium. Uniaxial extension of PC was then carried out at a cross-head speed $V_0 = 6$ mm/s to a draw ratio of 2.5. At the end of melt stretching, icy water was sprayed onto the sample. Melt-stretched samples were obtained by cutting such samples in the stretching direction by a dog-bone mold (ASTM D-638) with an effective length of $L_0 = 13.7$ mm, a width of $W_0 = 3.30$ mm and a thickness of 0.3 mm.

All the uniaxial extension tests of three different PC were carried out using Instron 5543 at around 23 °C. *In situ* measurements of the specimens' temperature were performed by an infrared camera

(FLIR SC325) operating at 60 Hz to record the whole time-dependent temperature profile on a video clip.

To determine the convective heat transfer coefficient h , we preheated a comparable dog-bone shaped sample with a thickness of 1.2 mm and used the IR camera to record the temperature decrease as a function of time due to the air cooling. Fig. 1 shows the temperature drop $\Delta T = (T - T_a)$ as a function of time t . From the energy balance equation, we have

$$\rho c_p \frac{dT}{dt} = -\frac{2h}{H} \Delta T, \quad (1a)$$

where H represents the thickness of sample. Approximating the specimen as a thin sheet, we have

$$\Delta T = (T_i - T_a) \exp\left(-\frac{2h}{H\rho c_p} t\right) \quad (1b)$$

where T_i is the initial temperature and T_a is the ambient temperature. For PC, the density is $\rho = 1200$ kg/m³, and the specific heat capacity is $c_p = 1200$ J/(kg·K). By fitting the temperature data to the exponential function of eq (1), we obtain the decay time constant to be 33 s, from which h is estimated to be $h = 25$ W/(m²·K).

3. Results

3.1. Untreated sample

The elastic yielding phenomenon [71] suggests to us that significant energy storage (i.e., U_2) takes place during neck formation. The structure of the neck front is sketched in Fig. 2. To characterize the nature of the stress response during uniaxial extension of untreated PC, we carried out theoretical analysis in Appendix A. As shown in Appendix A.1, we can gain more insight about the nature of ductile deformation of polymer glasses by determining the ratio U_2/W according to either eq (A.16) or eq (A.9), depending on whether the extension speed is high or low and whether air cooling contribution needs to be incorporated or not. Fig. 3a shows the IR thermal imaging of shear yielding upon startup extension at a low speed of $V_0 = 3$ mm/min. See supporting information for two movies with $V_0 = 3$ and 300 mm/min respectively. Fig. 3b displays the temperature profile at the neck front during its steady

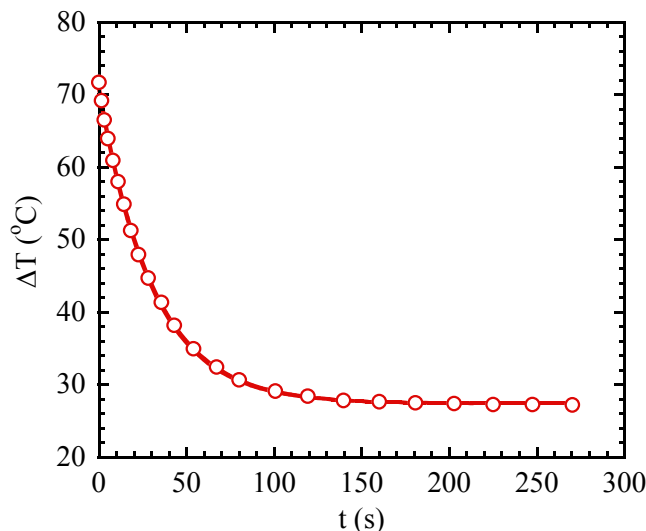


Fig. 1. IR camera measurement of the surface temperature of a heated PC that drops due to air cooling according to eq 1a-b.

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