



Acceleratory and inhibitory effects of uniaxial tensile stress on the photo-oxidation of polyethylene: Dependence of stress, time duration and temperature

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ABSTRACT

The photo-oxidation behavior of high density polyethylene (HDPE) subjected to uniaxial tensile stress is investigated at various temperatures, tensile stress levels and time durations. The effect of stress on the photo-oxidation rate of HDPE is found to be non-monotonous. Tensile stress is found to play distinctly different roles, namely showing acceleratory or inhibitory effect, with the elapse of aging time and at different temperatures. Stress-induced aging inhibition is prone to occur at low temperatures and high stresses within an appropriate aging duration. With elevating temperature, however, this inhibition effect disappears gradually and the stress is found to accelerate the aging process. Microstructural analysis reveals that the effect of stress is closely related to the interplay between the changes in the surface morphology as well as amorphous aggregation structure of materials and the photochemical degradation during aging, which is induced by the variation of molecular orientation and packing state in amorphous region, stress transfer efficiency, thermodynamic and kinetic aspects of photochemical reaction process.

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1. Introduction

Tremendous literature have reported the degradation behavior of polymers under stress. A general understanding is that tensile stress tends to speed up the deterioration of polymers [1–5] while compression stress usually manifests a reverse effect [6–9]. These phenomena are usually explained by the change in the energy barrier of bond dissociation in the presence of stress, which can be quantitatively described by some empirical models such as the Plotnikov equation [10] or Arrhenius-like equations without taking the details of degradation into consideration such as the well-known Zhurkov equation [11]. Particularly, it is found that for some materials, there is a critical stress with a magnitude of 10%–20% of the tensile strength of the material, beyond which stress-induced degradation is prone to take place [3,12]. More interestingly, Tyler et al. found that the aging rate of polymer materials would first increase and then decrease with elevating stress level

[13], which could not be simply explicated with aforementioned knowledge but can be explained by a decreased radical recombination efficiency (DRRE) hypothesis [13]. This hypothesis takes both reaction kinetics and aggregation structure into consideration. In fact, stress not only affects the orientation of polymer matrix [14,15] but also changes the mobility [16] and the conformation [17,18] of polymer chains. As a result, the diffusion behavior of small molecules (such as oxygen and water) and radicals [19] will be altered, resulting in corresponding change in the aging rate or even in the degradation mechanism.

High density polyethylene (HDPE) is widely used as fibers, pipes and structural components in our daily life because of its admirable chemical stability, good mechanical property and excellent dielectric property. In practical use, HDPE molecules usually experience aggressive attack from ultraviolet radiation, heat, oxygen and stress simultaneously [14,20–22]. Therefore, the photo-oxidation of HDPE under stress has received considerable attention in the literature. However, contradictory results are frequently reported. For instance, Busfield and co-workers [3] revealed that oriented HDPE tapes suffered 67–133 MPa (ranging from 47.5% to 95% yield stress of oriented material at 40 °C) underwent more rapid photo-

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degradation owing to the stress-induced decrease of cage recombination of broken molecules. However, stress showed little influence on the creep rate because the drawing process involved mainly lamellar unfolding within the crystal regions. Akay and Tincer [21] found that the resistance to radiation-induced degradation of highly oriented HDPE after cold drawing was enhanced. On the contrary, Ren et al. [23,24] found that stress (ranging from 0 to 10 MPa, which is less than 50% yield stress) showed no significant effect on the photo-degradation of three investigated HDPE but accelerated the creep rate of samples. More complicated degradation behavior of stressed HDPE has also been reported. For example, Evtimova and Peeva [12] demonstrated that low stress (20% of the maximum tensile strength) prolonged the induction period of unstabilized polyethylene and inhibited aging rate while high stress (40% and 60% of the maximum tensile strength) made the oxidation rate rise again. These contradictory results pertaining to the effect of stress on the oxidation of HDPE reflect the complexity of the aging process.

In view of its nonpolar and crystalline molecular nature, HDPE is prone to undergo microscopic deformation and macroscopic creep under stress [25]. As a result, the aggregation structure of HDPE can be changed by stress and it may in turn have a serious and complex impact on the chemical aging process itself. In the present work, we investigated the photo-degradation behavior of injection molded HDPE in the presence of tensile stress at different temperatures with the aging period as a variable parameter. The uniaxial tensile stress was found to play diverse roles, namely inhibitory or acceleratory, on the photo-oxidation rate of HDPE during the aging process. The interplay between the photochemical degradation and change in the surface morphology and the amorphous aggregation structure of HDPE samples was analyzed to provide insight into the underlying mechanism for these interesting phenomena.

2. Experiments

2.1. Material and sample preparation

A commercial grade high density polyethylene (HDPE 5000 S, Lanzhou Petrochemical Corp., China) was considered. It has a weight average molecular weight of 117 kg/mol and a melt flow index (MFI) of 1.12 g/10 min (2.16 kg, 230 °C, ISO 1133). To study the effect of orientation structure formed during injection molding on the photo-oxidation of polyethylene, dumbbell-shaped tensile sample bars with a size of 150 mm × 10 mm × 4 mm (gauge length: 110 mm) were prepared from the virgin HDPE pellets by using a PS40E5ASE precise injection-molding machine (Nissan, Japan) without adding any additional light or thermal stabilizer. The temperature profile from hopper to nozzle was 190, 200, 200 and 170 °C. The initial 10 samples were rejected to ensure all parts of the injection-molding machine being in a balanced condition.

2.2. Aging experiments

The exposure experiments under uniaxial stress were performed in a homemade aging oven equipped with forced air circulation apparatus and an Iodine-Gallium lamp (500 W) with a maximum intensity at 365 nm. The irradiation intensity was $5 \pm 0.2 \text{ W/m}^2$ and the temperature was controlled at $30 \pm 1 \text{ }^\circ\text{C}$, $40 \pm 1 \text{ }^\circ\text{C}$ and $50 \pm 1 \text{ }^\circ\text{C}$, respectively. The sample surfaces contacting fixed module plate during injection molding were exposed to UV light. The tensile direction was parallel to that of melt flow. According to the mechanical test results with an Instron universal testing machine at a rate of extension of 50 mm/min at 23 °C, the yield stress of neat HDPE sample bars is 24.2 MPa. To avoid conspicuous deformation damage, four different uniaxial tensile stress

levels (0 MPa, 2.5 MPa, 5 MPa and 7.5 MPa), which were no more than 35% yield stress, were applied to tensile specimens. The maximum aging time was 144 h for 30 °C, 120 h for 40 °C and 50 °C.

2.3. Characterization methods

All the characterizations are performed on the center part of exposed specimens allowing for the uniformity of irradiation and the reproducibility as well as comparability of data.

2.3.1. FTIR analysis

Fourier transform infrared spectra (FTIR) of aged specimens were recorded by using a Nicolet 6700 spectrometer (Thermo Fisher Scientific) in attenuated total reflection (ATR) mode from 16 scans between 700 and 4000 cm^{-1} with a resolution of 4 cm^{-1} . Then, spectra of HDPE were normalized by using the reference peak integrated intensity at 2912 cm^{-1} , which is resulted from asymmetric stretching vibration of carbon hydrogen bond and is almost not affected by aging [26]. Five samples were used and the error bars were determined by standard deviation. According to infrared spectra, carbonyl index (CI) was calculated by

$$CI = \frac{A_{C=O}}{A_{CH_2}} \quad (1)$$

where $A_{C=O}$ is the integrated area of carbonyl peak at 1712 cm^{-1} and A_{CH_2} represents integrated intensity of carbon hydrogen bond at 2912 cm^{-1} . The vinyl index (VI) was calculated by

$$VI = \frac{A_{C=C}}{A_{CH_2}} \quad (2)$$

where $A_{C=C}$ is the integrated absorption intensity of vinyl group (908 cm^{-1}) and A_{CH_2} has the same meaning as defined in Equation (1).

2.3.2. DSC characterization

The melting crystallization behavior of aged samples was studied by using differential scanning calorimetry (DSC Q20, TA instrument). The indium standard was used for temperature calibration. The sample was collected by milling powder away from the exposed surface of specimens with a depth of ca. 0.1 mm and a weight of 3–4 mg. Three samples were considered for reproducibility. The experiments were carried out by heating sample powder from 40 °C to 200 °C and holding at 200 °C for 5 min, and then cooled to 40 °C without isothermal process, subsequently, subjected to a second heat from 40 °C to 200 °C. A heating and cooling rate of 10 °C/min was used while the flow rate of nitrogen as a purge gas was 50 ml/min. The crystallinity was calculated by Equation (3) using data obtained from thermograms,

$$X_c = \frac{\Delta H}{\Delta H_0} \times 100\% \quad (3)$$

where ΔH_0 is the melting enthalpy of 100% crystal with a value of 293 J/g according to a previous study [27]. ΔH is the enthalpy of fusion of samples obtained from thermogram.

2.3.3. SEM analysis

The surface morphology of aged HDPE was observed by a scanning electron microscope (Phenom Pro, Phenom World). Rectangular samples with the size of approximate $10 \times 5 \times 4 \text{ mm}$ were cut from the center part of dumbbell-shaped sample bars, then were scanned after gold sputtering treatment for 100 s prior to characterization.

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