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Effect of 110 keV electrons on the deformation mechanisms of low density polyethylene



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ABSTRACT

The deformation mechanisms of low density polyethylene (LDPE) irradiated by 110 keV electrons with different fluences are discussed basing on the deformation behaviors and the structural evolution during uniaxial tensile deformation. The structural evolution is in-situ obtained from small angle X-ray scattering (SAXS) and wide angle X-ray diffraction (WAXD). It is found that the 110 keV electrons with low fluence change the transition strains of different regions (strain-hardening region, strain-softening region, plateau in the strain-softening region and sub-stages in the second strain-hardening region and quick break of the samples. From SAXS and WAXD analyses, it is known that the 110 keV electrons have no effect on the disintegration of the original lamellae and the rotation of the lamellae, only slow down the formation of the new crystals. It is concluded that the deformation behaviors of LDPE.

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1. Introduction

Polyethylene (PE) is a plastic material of popular demand owning to its superior properties, availability and low costs [1]. Noticeably, due to its unique properties, such as the good toughness, high resilience, high strength-to-weight ratio and the ability to be moulded, PE is widely used in space applications, such as thermal blankets, circuit boards and insulation films [2].

When used in space, its space environment survivability is an important factor. The charged particles radiation is an important environment in space. When PE is exposed to a radiation environment, it will favor crosslinking. The crosslinking sites produced by the radiation are located primarily in the amorphous phase and along the lamellar amorphous interphase [3,4]. It is of importance to evaluate the sensitivity of PE to space radiations, such as electrons, protons and heavy ions [5,6]. Many investigations have been reported on the effect of irradiation on the structures and properties of PE [7,8]. The effect is related to the nature of the materials and the radiation sources. The changes in properties of materials could affect the spacecraft reliability. Especially, the deformation

http://dx.doi.org/10.1016/j.polymdegradstab.2014.07.006 0141-3910/© 2014 Elsevier Ltd. All rights reserved. mechanisms of PE under irradiation are worth to study when it is used as a structural material.

Various techniques can be applied to investigate the deformation mechanisms of PE. Some researchers have examined the deformation mechanisms of the pristine and the irradiated PE by the electron microscopy, infrared spectroscopy, pulsed nuclear magnetic resonance and laser profilometry [9–18]. These methods could only reveal the deformation mechanisms by deduction from the final state of samples. They are lack in investigating the structural evolution of samples in-situ. It is urgency to find a method that can reveal the deformation mechanisms by investigating the structural evolution in-situ during the deformation process. Modern X-ray scattering beam lines allow researchers to follow in-situ process of PE, thus be able to study the deformation mechanisms [19–29] and the crystallization and melting behaviors [30,31] of PE. The deformation mechanisms of PE with different conditions, such as cold drawing, compression, tension and elevated temperature, are efficiently revealed in-situ by a combined small angle X-ray scattering (SAXS) and wide angle X-ray diffraction (WAXD) setup. The synchrotron radiation technique with a combined SAXS and WAXD setup can provide detailed information on both the molecular and supramolecular length scales. However, up to now, there are few works to explore deformation mechanisms of the irradiated PE in-situ by means of SAXS and WAXD.

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In our previous work [32], by SAXS and WAXD, it is found that 1 MeV electrons slow down the strain-induced melting and the strain-induced recrystallization of high density polyethylene (HDPE) at low strains. Now we focus on the deformation mechanisms in the whole deformation process. In this paper, the structural evolution of LDPE irradiated by 110 keV electrons with different fluences is in-situ studied by SAXS and WAXD. The effect of 110 keV electrons on the deformation mechanisms of LDPE is discussed basing on the deformation behaviors and the structural evolution.

2. Materials and methods

2.1. Materials preparation

LDPE was provided by National center for Nanoscience and Technology of China. The LDPE was prepared using a screw extruder with a screw speed of 80 rpm at a barrel temperature of 140 °C. Samples with a thickness of 0.25 mm are compressed and moulded under 25 MPa at 150 °C for 5 min warming up and 5 min pressure-keeping, and following by a natural cooling down.

Samples, placed between two glasses with smooth surface, were put into a vacuum oven under 95 °C for 30 min after moulding. Then, the samples were cooled down to room temperature with a speed of 0.1 °C/min. In order to release electric charge on the surface and inside samples, a "short circuit" treatment was done. The samples were placed between two mirror surface copperplates connected by wires, and were put into a vacuum oven under 80 °C for 24 h. Then the samples were cooled down to room temperature under 0.1 °C/min.

The electron irradiation experiment was performed in vacuum using an accelerator in Harbin Institute of Technology, China. The samples were perpendicularly irradiated by 110 keV electrons at given flux of 3 \times 10¹¹ cm⁻² s⁻¹ with fluences of 5 \times 10¹⁴, 1 \times 10¹⁵, 5 \times 10¹⁵ and 1 \times 10¹⁶ cm⁻², respectively. For the sake of convenience, the pristine LDPE and the LDPE irradiated by 110 keV electrons with fluences of 5 \times 10¹⁴, 1 \times 10¹⁵, 5 \times 10¹⁵ and 1 \times 10¹⁶ cm⁻² are abbreviated to LDPE-y, LDPE-e1, LDPE-e2, LDPE-e3 and LDPE-e4, respectively.

2.2. Tensile tests

The length, width and thickness for the dog-bone-shaped samples are 8, 4 and 0.25 mm, respectively. Uniaxial tensile deformation was performed with a home-made tensile apparatus with a speed of $2 \,\mu$ m/s at room temperature in air. The initial strain rate is 0.015 min⁻¹. As a result of extension, the strain rate varies throughout the drawing but is always no more than 0.015 min⁻¹. The stress and strain mentioned through the article are all engineering stress and engineering strain.

2.3. SAXS and WAXD measurements

SAXS and WAXD were used to in-situ characterize the structure evolution of HDPE samples during tensile deformation. SAXS and WAXD tests were performed on beamline BL16B1 of the Shanghai Synchrotron Radiation Facility (SSRF). The measurements are separate SAXS and WAXD measurements with same detector moved to different positions. Moreover, the SAXS and WAXD measurements are performed separately using two similar samples. The incident X-ray wavelength λ is 0.124 nm. The size of X-ray beam is 0.5 mm \times 0.5 mm. A detector with dimension of 2048 pixel \times 2048 pixel (80 µm/pixel) was used. The distances from sample to detector are 5.15 m (SAXS) and 0.15 m (WAXD). SAXS and WAXD images were taken immediately when the required strain



Fig. 1. Engineering uniaxial stress-strain curves for all the samples.

values had been reached during the deformation process. The intensity profiles were recorded with a two-dimensional imaging plate at room temperature. All the X-ray scattering data were corrected for the background and air scattering, the beam fluctuations and sample thickness variations. A camera focusing on the sample side was set up to record the change of sample thickness real-time. Using image-processing software, the real-time sample thickness could be calculated from the initial sample thickness. The scattering intensity is proportional to the real-time sample thickness, thus in order to compare signals of different thicknesses, the scattering intensity must be corrected by real-time sample thickness.

3. Results and discussions

3.1. Stress-strain curves

Fig. 1 shows the engineering uniaxial stress—strain curves for all the samples. Some important information can be obtained from Fig. 1, as follows:

(1) For LDPE-y, LDPE-e1 and LDPE-e2, the stress—strain curves show three regions which are characterized by strainhardening, strain-softening and secondary strainhardening, and are named with region I, region II and region III, respectively. In region I, the stress increases with increasing strain from the elastic region up to the ultimate point, showing an obvious strain-hardening phenomenon. In region II, the stress decreases with strain gradually, demonstrating a characteristic of strain-softening. In region III, the stress increases with strain again, which indicates that a secondary strain-hardening occurs. In addition, it is noticed that a plateau shows in region II, and there are two substages with different slopes in region III. The terminal

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The ranges	of different	regions	for all	samples

Sample	Fluence (cm ⁻²)	Region I	Region II	Region III (sub-stage I)	Region III (sub-stage II)
LDPE-y LDPE-e1 LDPE-e2 LDPE-e3 LDPE-e4	$- \\ 5 \times 10^{14} \\ 1 \times 10^{15} \\ 5 \times 10^{15} \\ 1 \times 10^{16}$	0-20% 0-20% 0-20% 0-20% 0-20%	20–100% 20–80% 20–50% –	100–500% 80–330% 50–180% –	500-880% 330-500% 180-320% 20-55% 20-22.5%

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