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Short communication

Structural origin of fast yielding-strain hardening transition in fluoroelastomer F2314

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ABSTRACT

The mechanical response of fluoroelastomer F2314 under uniaxial extension and its relation with strain-induced structural change are investigated by mechanical analysis, in-situ small angle X-ray scattering (SAXS) and wide angle X-ray scattering (WAXS). Yielding of samples followed by strain hardening is found without necking. Meanwhile, the strain range of strain softening is short, which is 0.77 at 25 °C and 0.3 at 60 °C. Microscopically, solution-cast F2314 shows microphase separation and low crystallinity. Results of WAXS and SAXS indicate strain-induced formation of fibrillar crystal during strain hardening. Based on these findings, it is supposed that the fast relay between destruction of microphase separated domains and formation of fibrillar crystal is the origin of the unique mechanical response.

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1. Introduction

Fluoroelastomer is a class of copolymer of fluorinated alkenes, which has excellent resistance to solvents, chemicals and heat. The unique performance makes it widely used in sealing ring and fuel hose. In these applications, the mechanical property of fluoroelastomer becomes important, particularly for the case of large deformation to failure [1,2]. In the past decades, the mechanical property of other elastomers like rubber has been extensively investigated [3–6]. However, the glass transition temperature (T_g) of fluoroelastomer can be relatively high, e.g. near to room temperature [1,7,8]. Also the difference in monomer polarity results in microphase separated structure [9]. These characters make fluoroelastomer different from natural rubbers and thermoplastic elastomers. How the unique structure of fluoroelastomer will affect its mechanical response under deformation is an interesting question and needs more effort.

On the other hand, deformation induced structure change has feedback on the imposed stress or deformation, which is important in material design [10–12]. Take natural rubber as an example. It behaves as an elastomer in the rubber-elastic region, where the typical stretch ratio is around 4 [5]. After that, strain-induced crystallization will happen and formed crystal will toughen rubber itself. Propagation of cracks or further deformation can be thus inhibited [13,14]. This is the widely known mechanism of self-toughening of natural rubber. More generally, materials can resist deformation or failure through structure changes can be defined as adaptive materials. How to design such materials needs information on structural change induced by external field, which may be mechanical force, temperature and other factors.

In this work, we aim to reveal the structural origin of mechanical response of fluoroelastomer F2314 under uniaxial extension. The structural evolution was investigated by simultaneous SAXS and WAXS. The stress response was also recorded with help of homemade extensional apparatus. It is found that microphase separation and strain-induced crystallization are dominant factors controlling the mechanical response. Moreover, faster crystallization under strain is helpful to shorten strain softening, which may prevent rapid failure after yielding.

2. Experimental section

2.1. Material and sample preparation

The fluoroelastomer (F2314) was supplied by Chengdu Research Institute of Chemical Industry. F2314 is a random copolymer of

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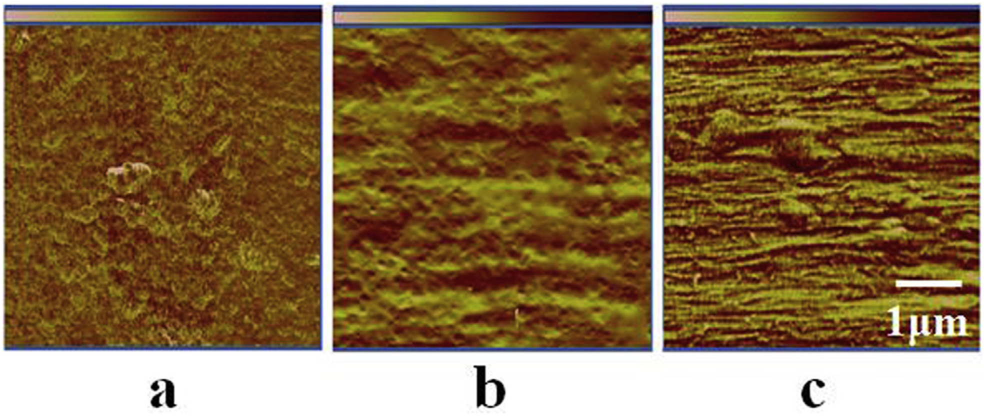

Fig. 1. AFM phase images of F2314. Images a-c correspond to samples before stretch, after stretch at 25 °C and after stretch at 60 °C, respectively. The stretch direction is horizontal.

vinylidene fluoride (VDF) and chlorotrifluoroethylene (CTFE) with a molar ratio of VDF:CTFE = 1:4. The weight-average molecular weight and number-average molecular weight are 11.3×10^4 g/mol and 5.3×10^4 g/mol, respectively.

Samples were prepared through solution casting of 5 wt% butyl acetate solution. Then solvent was evaporated at 50 °C for 48 h. The obtained film has a thickness of around 220 μm.

2.2. Uniaxial tensile

A homemade extensional machine was used to impose extension [15]. The dimension of samples was 23 mm × 6 mm × 0.22 mm. The stretching speed was kept constant 0.1 mm/s in this work.

2.3. In-situ X-ray scattering measurement

Small angle X-ray scattering (SAXS) and wide angle X-ray scattering (WAXS) were carried out at BL16B1 in Shanghai synchrotron radiation facility. The wavelength was 0.124 nm. Two-dimensional (2D) SAXS pattern was collected by Mar165 CCD detector with pixel size $80 \times 80\ \mu m^2$. 2D WAXS pattern was collected by Pilatus 200 K detector with pixel size $172 \times 172\ \mu m^2$. Distance between sample and detector was 2080 mm and 214 mm in 2D SAXS and WAXS measurements. Modulus of scattering vector q is integrated as $q = 4\pi sin\theta/\lambda$ in SAXS, where 2θ and λ are scattering angle and wavelength, respectively.

The crystallinity index (CI) in WAXS is calculated by the following formula:

$$CI = \frac{A_c}{A_c + A_a} \tag{1}$$

where A_c and A_a are the area of crystalline and amorphous peak, respectively.

2.4. Atomic force microscopy

Atomic force microscopy (AFM) phase images were acquired on the DI Multimode V in tapping-mode at room temperature in USTC.

3. Results

Fig. 1 shows AFM phase images before and after deformation at 25 °C and 60 °C. Before stretching, the surface shows isotropic morphology while regions with different brightness can be found. This is possibly related to microphase separation between VDF and CTFE segments [16]. After stretching at 25 °C, highly oriented fibrillar structure is observed with a strain of 5.4, as shown in Fig. 1b. The contrast is much more evident than that of the initial state. When stretched at 60 °C, the fibrillar structure becomes denser in space, indicating formation of more crystal.

Fig. 2 presents engineering stress-strain curve, 2D-WAXS and 2D-SAXS patterns during stretching at 25 °C. The stretching direction is horizontal. The engineering stress-strain curve can be divided into three regions, namely linear elasticity (I), yielding and strain softening (II) and strain hardening (III). The transition points locate at strain around 0.23 and 1.0, respectively. The occurrence of yielding is different from common behavior of rubber and elastomers with crystalline structure during extension [17–20]. Representative 2D-WAXS and SAXS patterns in the three regions are shown in Fig. 2b and c. Around the yielding point, the initial isotropic scattering from lamellar crystal changes into four-point pattern in SAXS. Further increasing strain, a streak along equator direction appears. Meanwhile, new scattering maximum along

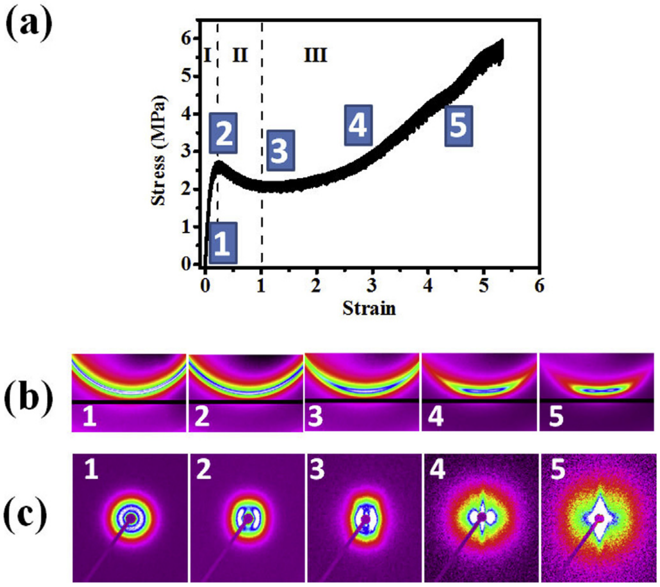

Fig. 2. (a) Engineering stress-strain curve, (b) selected 2D WAXS patterns and (c) selected SAXS patterns of sample stretched at 25 °C. According to the stress response, stretching process is divided into three regions (I-III), as shown by dashed line. Stretching direction is horizontal. Numbers from 1 to 5 represent corresponding strain in the stress-strain curve.

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