Polymer 55 (2014) 6345-6353



Polymer

journal homepage: www.elsevier.com/locate/polymer

Thermomechanical analysis of the crack tip zone in stretched crystallizable natural rubber by using infrared thermography and digital image correlation

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ARTICLE INFO

Article history: Received 23 April 2014 Received in revised form 22 September 2014 Accepted 5 October 2014 Available online 12 October 2014

Keywords: Strain induced crystallization Crack tip Quantitative calorimetry

ABSTRACT

This paper provides the first characterization of heat source field in the crack tip zone in carbon black filled natural rubber (NR). It focuses more especially on the calorific effects of strain induced crystallization (SIC). For this purpose, full thermal and kinematic fields are measured simultaneously. Initial image processing based on motion compensation enables us to track the temperature of any material point at the specimen surface. A second image processing stage, based on the heat diffusion equation, enables us to obtain the fields of heat sources produced and absorbed by the material during the test. The heterogeneity of the stretch states is analyzed from the kinematic measurements. In terms of heat production, crystallization acts in two opposite ways in the crack tip zone: the crystallization process produces additional heat, but crystallites act as fillers, which increases material stiffness in the crack tip zone. Moreover, the heat sources in the crack tip zone remain positive and small during unloading. This phenomenon is due to the production of mechanical dissipation and probably a continuation of the crystallization process. The results attained are compared with those recently obtained in non-crystallizing carbon black filled styrene butadiene rubber (SBR50).

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1. Introduction

Fracture mechanics in rubber is classically addressed through phenomenological approaches, especially through the strain energy release rate, also named tearing energy [1]. The tearing energy is the total energy necessary to create a unit fracture surface by crack propagation. To account for the physical phenomena involved in crack growth, the expression of the tearing energy can be modified, for instance by weighting it with a dissipative energy function to model the effects of dissipation at the crack tip [2]. Despite this, such approaches remain a description of fracture on the global scale and do not enable us to satisfactorily predict crack

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propagation paths or to understand how changes in the microstructure affect crack growth. Consequently, the investigation of physical mechanisms that drive crack growth is an essential prerequisite to improving fracture mechanics theories.

This explains why, over the last two decades, the state of the matter in the crack tip zone has increasingly been investigated. For instance, Refs. [3–6] have used wide-angle x-ray diffraction (WAXD) to map the crystallinity in the crack tip zone in natural rubber. Cavitation has also been identified as a phenomenon of paramount importance in the crack growth mechanism [7,8]. More recently, the interaction between cavitation and SIC has been highlighted and investigated in Ref. [9]. Finally, several authors have proposed mechanisms of crack growth [7,10,11]. Even though the results obtained have increased our knowledge of crack growth mechanisms, it is currently not possible to update constitutive models in order to account for all these phenomena and their interactions. Therefore, between a global description of fracture with continuum quantities (for instance tearing energy) and the





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description of the physical mechanisms at the local scale, we think that a trade-off can be found by measuring continuum quantities at the scale at which phenomena take place, *i.e.* in the crack tip zone. This should be all the more interesting since the continuum quantities measured are energies (making the link with tearing energy).

In our previous work, we investigated the thermomechanical response and calorific effects during the homogeneous deformation of rubber, using full-field measurement techniques. We highlighted the calorimetric signature of various phenomena involved during deformation: entropic and isentropic elasticity [12], viscosity due to filler adding [13], SIC [14,15] and the Mullins effect [16]. Then we studied heterogeneous deformations by developing a new data processing methodology in the case of a large displacement of points at the observed surface [17]. This methodology was applied to the determination of the kinematic and thermal fields in the crack tip zone of filled styrene butadiene rubber (SBR) to evaluate the effects of the mechanical dissipation of the total heat source¹: see Ref. [18]. In the present study, the effects of SIC on the full kinematic, thermal and heat source fields are investigated in the crack tip zone by using such methodology. The objective is therefore to link the change in the microstructure with variation in the heat sources, which are quantities originating in continuum mechanics.

Section 2 of the paper describes the experimental setup. Section 3 presents the image processing used to assess heat sources from temperature fields measured at the specimen surface in the case of large heterogeneous deformation. Section 4 gives the results and the analysis of the full kinematic, thermal and heat source fields and compares the results with those obtained in SBR material, which is not crystallizable under strain.

2. Experimental setup

2.1. Material and specimen geometry

The material used in the present study was a crystallizable natural rubber filled with 50 parts per hundred of rubber in weight (phr) of carbon black. It is denoted NR50 in the following. Table 1 summarizes its chemical composition. It was cured for 22 min at 150 °C.

The geometry of the specimens is presented in Fig. 1. The width, height and thickness were equal to 80 mm, 13 mm and 2 mm, respectively. This corresponds to classic Pure Shear (PS) geometry. The specimens were notched at one of their sides using a razor blade prior to testing. The initial crack length was about 8 mm. This undeformed state geometry was considered as the reference configuration in the present study.

2.2. Loading conditions

The mechanical test corresponded to uniaxial tensile loading. One load-unload cycle was applied under imposed displacement using a 500 N INSTRON 5543 testing machine (see Fig. 2). The signal shape was triangular in order to ensure a global constant strain rate during loading and unloading. The maximum global stretch ratio λ_g was equal to 1.5. The global loading rate and the global strain rate were equal to ± 200 mm/min and ± 0.26 s⁻¹, respectively.

Table	1
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Chemical composition.

Ingredient	Amount (phr)
Natural rubber NR	100
Carbon black	50
Antioxidant 6PPD	1.9
Stearic acid	2
Zinc oxide ZnO	2.5
Accelerator CBS	1.6
Sulfur solution 2H	1.6



Fig. 1. Specimen geometry. a) Side view. b) Front view.

2.3. Displacement field measurements

The kinematic fields were measured using the Digital Image Correlation (DIC) technique, which consists of correlating the grey levels between two different images of an observed region of the specimen surface named "Region Of Interest" (ROI) (see for instance Refs. [19-21]). Images were recorded using a cooled 16-bit PCO-Edge sCMOS camera. The sensor of the camera features 2650×2160 pixels. The acquisition frequency f_a was set to 50 Hz. Uniform lighting of the specimen surface was ensured by a set of three fixed cold lamps in order not to heat the specimen and not to induce external radiations. In order to improve the contrast of the images, white talc was deposited on the surface of the specimen before the measurements. Particular attention was paid to these preliminary settings for a maximum use of the dynamics of the camera. The software employed for the correlation process was SeptD [22]. A set of sub-images was considered to determine the displacement field of a given image with respect to a reference image. Each sub-image is referred to as a "Zone of Interest" (ZOI). A bilinear correlation function was employed to calculate the displacement of the center of a given ZOI between the two images. Correlation parameters were chosen to reach a spatial resolution of 10 pixels, corresponding to 101 µm.



Fig. 2. Experimental device.

 $^{^1}$ 'Heat source' is used in this paper to designate the heat power density (in W/m³) that is produced or absorbed by the material. Note that 'heat source' and 'heat' must be distinguished: heat (in J/m³) is the temporal integration of heat source.

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