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Multiaxial deformation and strain-induced crystallization around a fatigue crack in natural rubber

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ABSTRACT

The study of fatigue crack propagation in elastomers is an essential prerequisite to improve the service life of tire products. Natural rubber is a key compound in tires, because of its unique mechanical properties and more particularly its remarkable resistance to fatigue crack growth as compared to synthetic rubbers. To explain this resistance, the literature often mentions the phenomenon of strain-induced crystallization which takes place at fatigue crack tips in natural rubber and then reinforces it. In the present study, an original experimental set-up that couples synchrotron radiation with a homemade mechanical fatigue machine is developed to investigate both strain-induced crystallization and deformation multiaxiality around fatigue cracks in natural rubber. During uninterrupted fatigue tests, recording of wide-angle X-ray diffraction patterns is performed in the crack tip region providing the two-dimensional spatial distribution of both crystallinity and principal strain directions. In particular, the influence of loading conditions on the size of the crystallized zone is investigated and related to fatigue crack growth rates. Finally, measurements of deformation multiaxiality, i.e. principal strain directions and change in thickness, obtained by this method are successfully compared with digital image correlation results.

1. Introduction

Because of its outstanding mechanical properties, particularly its fatigue resistance [1–3], Natural rubber (NR) is a key material in many industrial applications such as tires. In literature, its ability to crystallize under deformation (strain-induced crystallization, SIC [4]), in particular in the vicinity of crack tips where the material is largely strained [5–7], is classically invoked to explain longer service life and better fatigue crack growth resistance than synthetic elastomers. More precisely, authors argue that the crystallized rubber around the crack tip slows down crack growth [3]. However, the influence of this crystallized zone at crack tip on fatigue crack propagation remains still unproven.

In this context, the present paper aims to relate fatigue crack growth rates in NR samples with the characteristics of the crystallized zone around crack tip, and then to explain the macroscopic fatigue properties. In this way, three experimental methods are developed and their results are compared:

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Nomenclature

c	crack length
dc/dn	crack growth rate
h	thickness of the deformed PS sample
h_0	thickness of the undeformed PS sample
I_{200}	intensity of the X-rays diffracted by the (200) plane
$I_{amorphous}$	intensity of the X-rays diffracted by the amorphous part of the rubber
l	height of the deformed PS sample
l_0	height of the undeformed PS sample
n	number of cycles
T	tearing energy (or energy release rate)
w	strain energy
W_0	strain energy per unit undeformed volume
χ	index of crystallinity
Φ	angle of rotation of the principal strain direction
λ	stretch ratio in the tension direction for the PS sample
λ_3	stretch ratio in the out-of-plane direction (thickness) for the PS sample

Acronyms

CB	carbon black
DIC	digital image correlation
IR	isoprene rubber
IR50	isoprene rubber filled with 50 phr of carbon black
NR	natural rubber
NR50	natural rubber filled with 50 phr of carbon black
phr	parts per hundred of rubber
PS	pure shear
SIC	strain-induced crystallization
WAXD	wide-angle X-ray diffraction

- first, classical fatigue crack propagation tests are performed on different pre-cracked "pure shear" (PS) samples of NR filled with carbon black in order to evaluate crack growth rate;
- second, the strain field around the crack tip is evaluated by digital image correlation (DIC) measurements;
- third, in situ fatigue tests are conducted at the French national synchrotron facility SOLEIL in order to measure in real-time the crystallinity in the vicinity of the crack tip. The method consists in mapping the crack tip neighbourhood while recording wide-angle X-ray diffraction (WAXD) patterns; see [8] for details on its development. It permits the measurement of both SIC distribution and deformation state, i.e. principal strain directions and change in thickness, around the crack during uninterrupted fatigue tests.

2. Experimental method

2.1. Material and sample

The fatigue experiments are conducted with NR samples filled with 50 phr of N347 carbon black (CB). Vulcanization is carried out with 1.6 phr of sulphur together with CBS (N-Cyclohexyl-2-benzothiazole sulfonamide) that acts as an accelerator. Each blend also contains zinc oxide (ZnO) and stearic acid. 6PPD (N-(1,3-dimethylebutyl)-N′-phenyl-p-phenylenediamine) is used as an antioxydant. In addition, a synthetic isoprene rubber (IR) filled with 50 phr of CB N347 and with exactly the same amounts of additives than NR compound, is synthesized. As in this IR (89% of CIS configuration) SIC is considerably reduced compared to NR, the corresponding measurements are helpful to discuss SIC influence on fatigue crack growth properties. The formulation of NR and IR materials are given in Table 1. In the following, these two compounds will be denoted NR50 and IR50.

The geometry of the samples is a classical "pure shear" geometry (also known as planar tension samples), commonly employed for fatigue crack growth tests [9–11]. The dimensions are reported in Fig. 1.

2.2. Fatigue crack growth rate tests

Fatigue crack growth measurements are based on the energy balance approach of Rivlin and Thomas [12], who extended the concept of energy release rate of Griffith [13] to the case of non-linear hyperelastic materials. Considering thin planar