COUPLED THERMAL AND VOLUME CHANGE MEASUREMENTS DURING STRETCHING OF FILLED EPDM RUBBERS

Nicolas Candau[a], Jean-Luc Bouvard[a], Edith Peuvrel-Disdier[a], Rudy Valette[a], Christophe Pradille[b], Noëlle Billon[a]

[a]MINES ParisTech, PSL Research University, CEMEF - Centre de Mise en Forme des Matériaux, UMR CNRS 7635, CS 10207, 06904 Sophia-Antipolis, France, nicolas.candau@mines-paristech.fr, jean-luc.bouvard@mines-paristech.fr, edith.peuvrel-disdier@mines-paristech.fr, rudy.valette@mines-paristech.fr, noelle.billon@mines-paristech.fr

[b]Mat-xper, 06560 Valbonne, France, christophe.pradille@mat-xper.com

Keywords: EPDM, strain field, volume change, VIC-3D, thermomechanical analysis

Introduction

Ethylene-propylene-diene-monomer rubber (EPDM) has been widely studied in the recent years. Thanks to its high resistance to ageing and mechanical properties, it is one of the best candidates for the automotive industry. For a better understanding of its mechanical properties, a suitable characterization of its thermomechanical behavior is mandatory. For this purpose, the combination of infrared thermography and strain field measurement is commonly used in literature [1]. Over the last decade, image correlation approach has been widely improved. It now allows precisely estimating the volume change during the deformation [2]. However, to the authors' knowledge, no study was already devoted to the combined analysis of both thermal and volume change. This approach should be useful to identify the respective contributions of the reversible and irreversible mechanisms associated with the deformation of elastomers.

Objectives and Methodology

The present work proposes an experimental approach for the combined measurement of both volume and thermal changes during stretching of elastomers. This is done thanks to the set-up of a specifically dedicated device which consists of a four camera stereovision system and an infrared camera (figure 1a). The stereovision system is made up of two independent camera pairs – allowing neglecting the out of plane displacements – which are positioned to measure the complete strain displacement field (front and side faces of the samples) and thus the volume change of the sample. A speckle pattern is put on the surface sample and Digital Image Correlation is used to analyze the displacement field. The IR camera is switched 2h before tensile tests to stabilize its internal temperature. To reduce the impact of external heat sources, a black box surrounding the sample is used during each test. The studied rubbers are carbon black filled EPDM obtained by sulphur vulcanization of the gum. They contain 80phr of carbon black (80 g of fillers per 100 g of rubber). EPDM samples are stretched uniaxially with an INSTRON tensile test machine at room temperature with the strain rate $1.s^{-1}$ up to four different maximum strains (figures 1b, 1c and 1d). Mullins type tests are also performed. They consist of four series of three cycles with an increasing maximum strain.

Results and analysis

Figures 1b-d present the evolution of the volume change and self-heating during stretching of EPDM rubbers at various maximum strains. Volume and temperature increase during loading and decrease during unloading. At a given strain, volume and temperature are found higher during the unloading phase respectively due to the remaining open cavities and the accumulation of heating on

the surface sample. Following Martinez et al. [3], temperature rise in filled elastomers is mainly due to entropic elasticity and viscosity but damage mechanisms are assumed to be non-negligible. With an appropriate treatment of thermomechanical equations (i.e. taking into account the non-adiabatic conditions), the impact of damage on the mechanical and the thermal response could be quantified. This point is of matter of importance concerning the Mullins effect, whose physical meaning is not totally understood [4]. During such a test, the accumulation of cycles at a fixed strain is associated with a progressive decrease of the stress (so-called Mullins effect). Our results show that the main volume change is observed at the end of the first cycle, but still progressively increases with the number of cycles, suggesting that irreversible damages still occur. We think that the present work would be interesting to identify the contributions of reversible and irreversible mechanisms associated with the deformation of elastomers. This approach will be improved by testing different experimental conditions such as various strain rates or loading conditions. as well as EPDM obtained for different , i.e. different sulfur and carbon black contents.

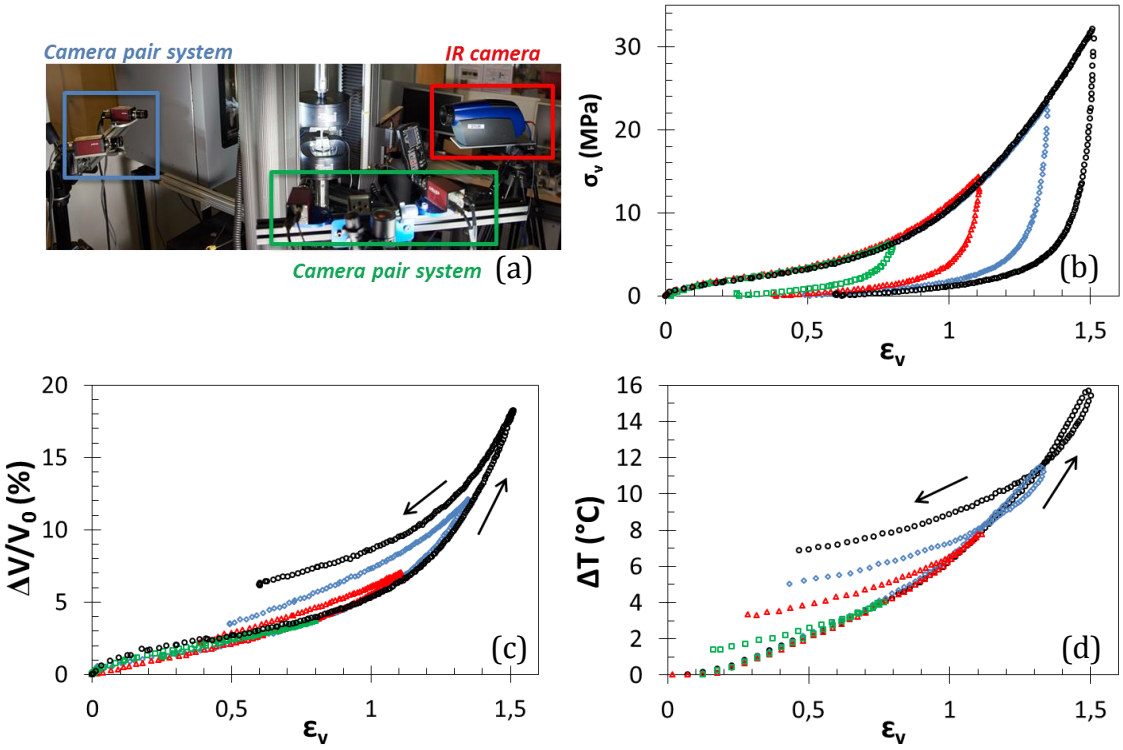


Fig.1. Experimental set up (a). Stress-strain curves for cyclic tests at room temperature and the strain rate $1.s^{-1}$ (b). Volume change (c). Self-heating (d).

References

[1] A. Chrysochoos, V. Huon, F. Jourdan, J.M. Muracciole, R. Peyroux, B. Wattrisse. Use of Full-Field Digital Image Correlation and Infrared Thermography Measurements for the Thermomechanical Analysis of Material Behaviour. *Strain*, *46*(1): 117-130 (2010)

[2] J. De Crevoisier, G. Besnard, Y. Merckel, H. Zhang, F. Vion-Loisel, J. Caillard, D. Berghezan, C. Creton, J. Diani, M. Brieu, F. Hild, S. Roux. Volume changes in a filled elastomer studied via digital image correlation. *Polymer Testing*, *31*(5), 663-670 (2012)

[3] J.S. Martinez, J.B. Le Cam, X. Balandraud, E. Toussaint, & J. Caillard. Mechanisms of deformation in crystallizable natural rubber. Part 2: quantitative calorimetric analysis. *Polymer*, *54*(11), 2727-2736 (2013)

[4] J. Diani, B. Fayolle, & P. Gilormini. A review on the Mullins effect. *European Polymer Journal*, *45*(3), 601-612 (2009)