

Abstract

# Measuring In-Situ X-ray Scattering of Natural Rubber Biaxial Deformation: A New Equipment for Polymer Studies <sup>†</sup>

Daniel P. da Silva <sup>1</sup>, Barbara Lamolinara <sup>1</sup>, André Costa <sup>1</sup>, Fábio Gameiro <sup>1</sup>, Paula Pascoal-Faria <sup>1</sup>,  
Artur Mateus <sup>1</sup>, Juan Carlos Martinez <sup>2</sup>, Pranee Phinyocheep <sup>3</sup>, Taweechai Amornsakchai <sup>3</sup>  
and Geoffrey R. Mitchell <sup>1,\*</sup>

- <sup>1</sup> Centre for Rapid and Sustainable Product Development, Polytechnic of Leiria, 2430-080 Marinha Grande, Portugal; daniel.p.silva@ipleiria.pt (D.P.d.S.); barbara.lamolinara@ipleiria.pt (B.L.); andre.a.costa@ipleiria.pt (A.C.); fabio.a.gameiro@ipleiria.pt (F.G.); paula.faria@ipleiria.pt (P.P.-F.); artur.mateus@ipleiria.pt (A.M.)
- <sup>2</sup> NCD Beamline, Alba Synchrotron Light Source, Cerdanyola del Vallès, 08290 Barcelona, Spain; guilmar@cells.es
- <sup>3</sup> Department of Chemistry, Faculty of Science, Mahidol University, Rama 6 Road, Ratchathewi, Bangkok 10400, Thailand; pranee.phim@mahidol.ac.th (P.P.); taweechai.amo@mahidol.edu (T.A.)
- \* Correspondence: geoffrey.mitchell@ipleiria.pt; Tel.: +351-962426925
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**Abstract:** Understanding biaxial deformation is essential for a more realistic evaluation of rubber elasticity compared to the more usual uniaxial deformation. To study crystallisation occurring during biaxial deformation of natural rubber films, a new simple equipment has been designed and assembled. The equipment, mounted in the beamline of ALBA synchrotron light source facility, allowed the in-situ measurement of X-ray scattering of natural rubber during biaxial deformation. This work provides, for the first time, quantitative information on crystallisation during biaxial extension.

**Keywords:** natural rubber; biaxial deformation; uniaxial deformation; crystallisation; SAXS; WAXS



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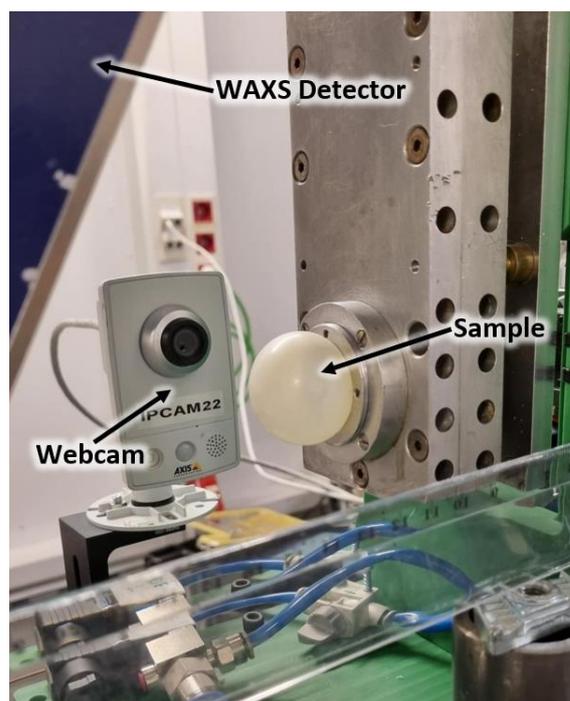
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The biaxial deformation test, compared with widespread uniaxial deformation systems, is a more comprehensive analysis with which to investigate polymer film properties during deformation. In fact, uniaxial deformation is not fully representative of the complex types of stress applied to polymer films in common application areas. As a consequence, the study of the behaviour of polymer films during biaxial deformation was of interest to carry out. The objective of this work was to contribute to the development of a full understanding of crystallisation during the biaxial deformation of natural rubber films and explore how this differed from crystallisation during uniaxial deformation.

Moreover, we evaluated how the process changed with different deformation symmetry. To achieve this, a new experimental stage system (Figure 1) was designed and tested to collect in situ X-ray scattering data during the biaxial deformation of natural rubber films. The equipment was mounted and aligned on the beamline of the ALBA Synchrotron Light Facility in Barcelona. Natural rubber films, in the form of discs, were mounted in the stage and deformed as a bubble using air pressure. The stage was designed so that the incident X-ray beam passed through the pole of the bubble, where the strain was purely biaxial. Small and wide-Angle X-ray scattering (SAXS/WAXS) data were recorded at different air pressures to continuously deform the rubber film. Both WAXS and SAXS showed different scattering features for the uniaxial and biaxial cases.



**Figure 1.** Biaxial deformation stage, with natural rubber air pressured bubble mounted on the NCD-SWEET Beam line at the ALBA Synchrotron Light Source. The incident X-ray beam enters from the right hand-side behind the green vertical panel. The wide-angle X-ray scattering detector is shown on the left-hand side, slightly inclined towards the sample. The small-angle X-ray scattering detector is set more ten 6 metres to the left.

This work demonstrated that this bubble biaxial system worked successfully as an in-situ X-ray stage, providing high quality data for both uniaxial and biaxial deformation on the same rubber samples. Both loading and unloading were investigated. The new system provided, for the first time, quantitative information on crystallisation during biaxial extension. It represents a promising suitable experimental geometry for biaxial deformation research of different polymer films. Further studies in different areas and materials are expected—for example, in biomaterials such as skin tissue or the bladder wall, both of which involve biaxial deformation and in which high-quality, small-angle X-ray scattering could provide excellent input to aid in the understanding of deformation at the molecular level.

**Supplementary Materials:** The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/materproc2022008136/s1>.

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**Conflicts of Interest:** The authors declare no conflict of interest.