ESRF	**Experiment title:** Temporally and spatially resolved simultaneous micro-SAXS and stress / strain measurements of single struts of an open-celled elastomeric polyurethane foam.	**Experiment number**: SC-762
Beamline: ID13	**Date of experiment**: from: 23.09.00 to: 26.09.00	**Date of report**: 26.02.01
Shifts: 9	**Local contact(s)**: Dr C. Riekel	*Received at ESRF:*

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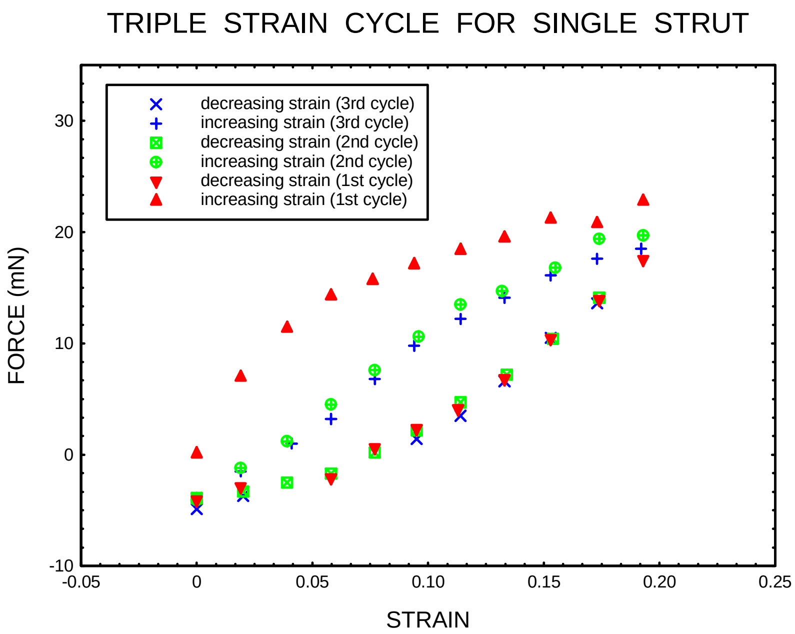
Report:

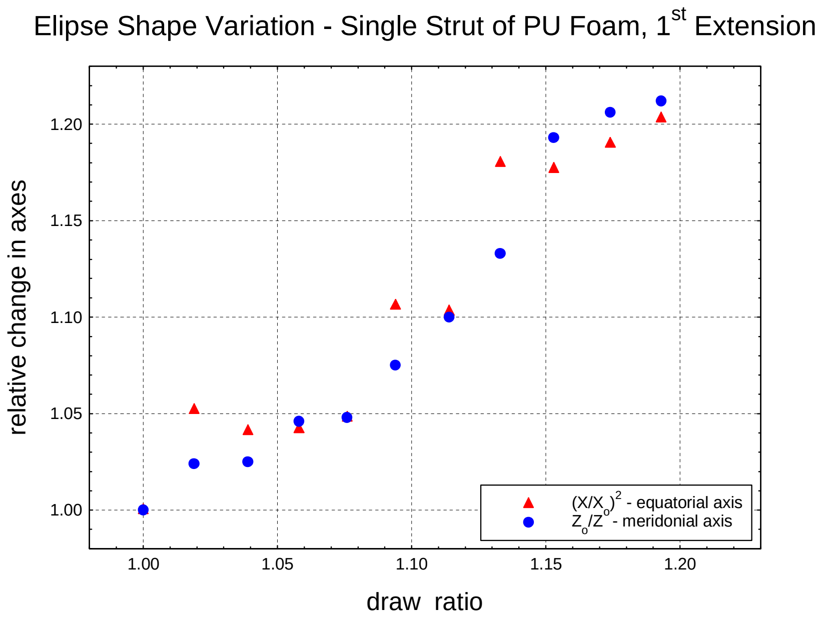
Exploitation of the micro-SAXS techniques developed on ID13 in the physical characterisation of micron-sized samples is demonstrated in their application to single struts of an elastomeric polyurethane (PU) foam. In parallel with the implementation of micro-SAXS optics by beamline staff, we have developed novel micro-deformation instrumentation[1]. This instrumentation has the capability of the precise measurement of strain and force applied to struts of a PU foam simultaneously with micro-SAXS data. Typical strut dimensions were of the order of 50-100 microns in sectional width and 500-1000 microns in length.

In a unique series of time-resolved experiments, cyclical strain patterns were applied to struts extracted from a commercial MDI-based foam with a hard segment volume fraction of 25%. In these experiments the applied strain was increased in a series of steps separated by 1 minute intervals. SAXS data were collected at each step for 15 seconds on a *MAR-CCD* detector, simultaneously with strain and force data. A Young's Modulus of 56 MPa was measured from the stress-strain curve of the first strain cycle. This is similar to values calculated for similar bulk PU films[2], which gave confidence in the techniques applied. Analysis of multiple full strain cycles allowed the hysteresis characteristics of the struts to be measured. See Figure 1. In addition, it has been possible to quantify the stress-relaxation time-constants associated with soft and hard segment relaxation mechanisms. For example, these quantities were approximately 5 and 27 seconds respectively for a strain of 0.05 on the first tensile deformation cycle.



<u>Figure 1</u>: *A force / strain plot for a triple-cycle deformation experiment illustrating the hysteresis characteristics of the foam strut.*

Techniques are being developed in order that the micro-SAXS data from this type of experiment can be appropriately analysed to determine the nature of the deformation characteristics of single struts of PU foam. These techniques will be equally applicable to data collected from other polymeric materials. In this, comparison with well-characterised samples of bulk PU films, which are intrinsically much easier to study, will be significant. The experiments described here have enabled us to demonstrate that at strains up to about 0.2 the PU foam struts deform in an affine manner. See Figure 2. This type of information, though as yet limited in its scope, is crucial to a formal description of the kinetics of the deformation characteristics of these economically significant materials.



<u>Figure 2</u>: *Plot from the analysis of SAXS pattern ellipse shape variation during deformation up to a strain of about 0.2. The approximately linear relationship between the draw ratio and the relative change in major and minor axes suggests that the deformation, within this strain range, is affine in manner.*

Having established the techniques and analytical techniques required to execute these demanding experiments, we propose a further series of experiments to extend their range in order to permit a full description the kinetics of their deformation.

<u>References</u>:

[1] Martin, C, Eeckhaut, G., Mahendrasingam, A., Blundell, D.J., Fuller, W., Oldman, R.J., Bingham, S.J., Dieing, T. and Riekel, C., (2000), *J. Synchrotron Rad.*, **7**, pp245-250.

[2] Stanford. J.L., (1998), in *Polymer Networks* ed. by R.T.F Stepto, Ch. 5, London, Wiley.