ESRF	**Experiment title:** Residual stress in HDPE pipeline using synchrotron XRD: a new tool for in polymer engineering	**Experiment number**: MA930
Beamline: ID31	**Date of experiment**: from: 19 November 2009 to: 21 November 2009	**Date of report**: 1 September 2010
Shifts: 6	**Local contact(s)**: Caroline Curfs	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

D.J. Hughes – WMG, Warwick University*

E.L. Heeley – The Open University*

C. Curfs – ESRF*

Outcomes to date (1 Sept 2010)

D.J.Hughes, E.L. Heeley and C. Curfs. A non-destructive method for the measurement of residual strains in semi-crystalline polymer components. Submitted to Materials Letters (Aug 2010)

Report

Abstract of submitted paper: "A new non-destructive tool is presented enabling the measurement of *locked-in* residual strains in semi-crystalline polymer-based components. The technique employs high-energy synchrotron X-rays to probe the variation of diffraction angle within a well-defined 'gauge volume' with a spatial resolution of the order of 1mm. Lattice strain is calculated from the diffraction angles.

An overview of the experimental methodology and underlying principles involved in the non-destructive evaluation of residual strain in polymer-based components is given. Preliminary results show that synchrotron X-rays can be used successfully to measure the internal elastic residual strain field in polymer components, being potentially applicable to other materials. The method was used successfully to measure residual strains in a commercial high density polyethylene gas pipeline sample."

Experimental details: The sample measured was a commercial high density polyethylene (HDPE) pipe provided by Lyondell Basell (Table 1). The length was cut to 120 mm because of sample environment restrictions. It was assumed that the principal stress directions lay along the radial (R), hoop (H) and axial (A) directions in the sample. A through-thickness line of measurement points was chosen at mid-length of the pipe in order to avoid any relaxation effects near the cut edges. Measurements were made with translator steps of 0.5 mm from the inner to the outer surface of the pipe wall.

Figure 1 shows the orientation of the pipe sample for all three sets of strain measurements. For the axial strain measurements, the pipe was mounted vertically - in order to scan the sample through the NGV from inner to outer wall, the pipe was translated horizontally (perpendicular to the beam). For the radial measurements, the pipe was mounted horizontally and translated vertically. The same sample orientation was used for the hoop direction, however the sample was translated horizontally (parallel to the beam).

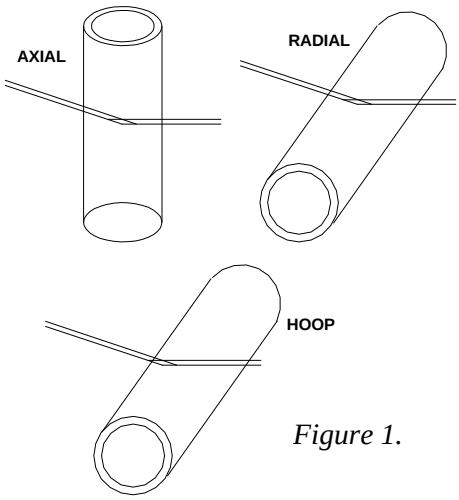

Figure 1.

Table 1: Properties of the HDPE pipe sample	
Outer diameter/mm	110.0
Wall thickness/mm	12.0
Material grade	CRP 100 black
Density/g cm^{-3}	0.959
Tensile modulus at 23 °C /MPa	900
Carbon black content /%	2.25
Crystallinity/%	72

In certain measurement positions, the X-ray path length became significant (≈45 mm) thus an X-ray wavelength of λ = 0.354 Å was used. Figure 1 shows that the dimensions of the NGV vary according to the X-ray slit apertures. A key experimental consideration was the elongation of the NGV in the X-ray beam direction. For measurements in the axial and radial directions the NGV was defined by 1 × 1 mm^2 slits at the incident and diffracted beam position, defining an NGV as 1 mm wide by 1 mm high but elongated 11 mm along the X-ray beam direction. Although the elongation of the NGV reduces the spatial resolution of the measurements, in the axial and radial strain measurements, this elongation is along a tangent of the pipe cross-section. Due to the large diameter of the pipe, the spatial resolution achieved is determined by the 1 mm NGV height. However, for the hoop strain direction, the elongation of the NGV inevitably reduces the spatial resolution achievable. To minimise this effect, the vertical slit heights were reduced to 0.125 mm, reducing the horizontal dimension of the NGV to ~1.5 mm, requiring increased data collection times. Hence, the actual spatial resolution achieved across the pipe wall was similar in all three strain directions.

To prove the concept of measuring changes in lattice strain as a response of macroscopic strain, a test using a HDPE three-point bent bar was used. The sample was fitted with an extensometer at the surface measuring the change in lattice strain under different load conditions.

Results and discussion- Figure 2 shows a 2θ scan at the mid-position in the pipe (axial direction). The data show a series of narrow peaks relating to the orthorhombic unit cell of HDPE (a = 7.42 Å, b = 4.95 Å, c =2.55Å) superimposed on a broad amorphous peak. Diffraction peak positions were obtained from the Gaussian fitting procedure with an average full width half maximum of ~0.05 °. Initial measurements were made using both the 110 and 200 peaks, but, for the rapid mapping of strain, it was necessary to select only a single diffraction peak (110).

Figure 3 shows the variation of calculated residual strain ε through the pipe wall (0.5 mm steps). The uncertainty in strain arising from peak fitting is $< \pm 10 \times 10^{-6}$ for the axial and radial data and $\sim \pm 60 \times 10^{-6}$ in the hoop direction (due to the reduced gauge volume). The 'strain-free' reference d_0 was determined from a single small piece of material, assumed to be free of macroscopic stress. A clear variation in lattice strain for the three strain directions through the wall of the pipe is observed. The hoop and axial strains are of a similar magnitude being tensile at the internal face (0 mm) and compressive at the external surface (12 mm). The magnitude of the radial residual strain is higher, particularly showing strong tensile strain approaching the external surface.

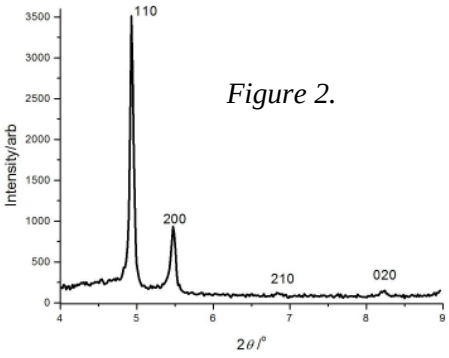

Figure 2.

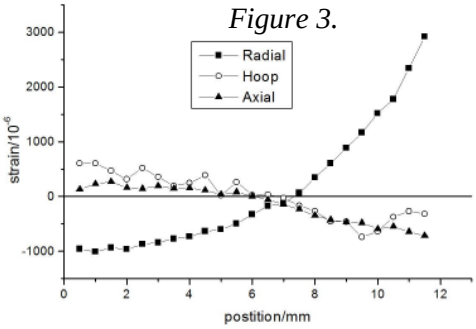

Figure 3.

Based on the calculated residual strain measurements, an attempt was made to determine the residual stresses using the assumptions discussed earlier. However, the results proved to be inconsistent since certain boundary conditions *must* be met (e.g. normal stress at a surface must be zero). There are several possible explanations for this which require further work. Firstly, it is possible that the use of a single reference parameter is inappropriate, due to a change in strain-free lattice parameter across the wall. Although this type of variation is seen in metals, it seems unlikely to be causal in this sample. More likely, assumptions made that the principal stress directions coincide with the geometrical (R, H, A) directions are false. The principal stress direction will be largely determined by the extrusion and cooling process where significant variations in morphology are possible. Further measurements are planned to investigate the texture variation and the possibility of a strain-free lattice change from wall-to-wall.

Conclusions- The new technique allows the non-destructive measurement of residual strains in bulk samples of semi-crystalline polymers. Measurements are feasible in samples of complex geometries, with consideration of the effects of NGV elongation. Although the technique inherently relies upon diffraction from a crystalline phase, there is applicability for low- and non- crystalline polymers, for example via the mixing of a small volume of metal powder at fabrication stage. Of particular interest is the relevance to the study of residual and applied strains in polymer matrix composite materials which to-date, has proven difficult to determine experimentally.

Further work- We are currently preparing a second manuscript for publication. In order to fully understand the pipe data we require information relating to the texture through the pipe wall. Further experiments are planned to study the texture and also establish the applicability of the method to stresses in polymer-based composites.