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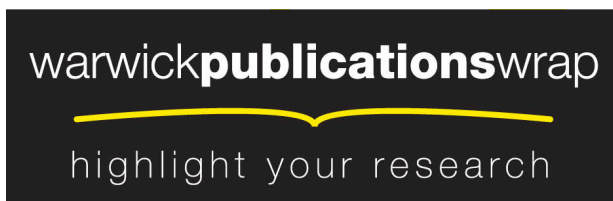
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# **A non-destructive method for the measurement of residual strains in semi-crystalline polymer components**

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## **ABSTRACT**

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 1 mm. 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.

## **KEYWORDS**

Residual strain, Polymer components, Synchrotron X-rays, Non-destructive testing

## **1. INTRODUCTION**

During the production of thermoplastic engineering products, a form is generally extruded or injection moulded from a molten stage followed by non-uniform cooling. A consequence of irregular cooling is the formation of macroscopic residual stresses and strains in the final product. Stresses that are classified as residual are ones that are *locked-in* after production, after

the removal of all external loads. The actual stress experienced by the component in-service, is a combination of the applied and residual stresses. This summative nature of the applied and residual stresses implies that not all residual stresses are negative in terms of material performance. Indeed, residual stress modification in metallic components is often used at the design stage; for example where compressive residual stresses are imparted to enhance resistance to fatigue crack growth [1, 2].

Methods utilised to assess the magnitude of residual stress in polymer components, fall into three distinct categories - non-destructive, destructive and predictive. Often, techniques applicable to residual stress analysis in metals are not suitable for similar analysis in polymers. Non-destructive experimental methods, though limited, generally measure relaxation of the locked-in stress; for example holographic interferometry can be used to visualise physical relaxation of a component during heating and calculate the initial stress state [3]. Destructive techniques rely on measuring deformation following a cut (or hole) as the stresses relieve to zero at the new surface. Hole drilling has been employed [4, 5], where the local relaxation is determined by strain gauges or photoelastic sheets. A number of variations of layer removal [6], ring slitting and combinations of such [7] have been used. However these techniques require meticulous sample preparation, use of suitable strain gauge adhesives, detailed analysis, are impractical for samples with complex geometry and often only provide surface data. Finally, computer simulation of the cooling process, allowing prediction of residual stresses has been used [8], however for a model system to be trustworthy it must be validated against reliable experimental data.

## **2 MEASUREMENT OF RESIDUAL STRAIN IN POLYMERS WITH SYNCHROTRON X-RAYS**

Non-destructive diffraction techniques have been employed to study structure development (crystallisation/orientation) in semi-crystalline polymers [9] where spatial resolution is simply determined by sample thickness. Recently, both neutrons and synchrotron X-rays have been used for the measurement of residual strains in metallic components [10, 11]. The technique effectively uses the crystal lattice as an ‘atomic strain gauge’; changes in macroscopic strain cause deformation at a crystal level resulting in a change of diffraction peak position. The technique relies upon definition of a small ‘gauge-volume’ from where an average measurement of lattice strain can be made. In metals, the diffracting units are small grains. Movement of the sample through the gauge volume allows spatial mapping of local residual strains.

The application of diffraction techniques in measuring localised residual strain in semi-crystalline polymers has not previously been attempted. However, it could be expected that similar methods used in metals could be applied to these materials. Regions of crystallites in polymers should allow the local elastic strain state to be evaluated. There are however, a number of experimental conditions specific to polymers to consider. Neutron diffraction methods are not generally applicable because of the high incoherent scattering of hydrogen. The requirements for the measurement of strain in polymer components using X-rays include:

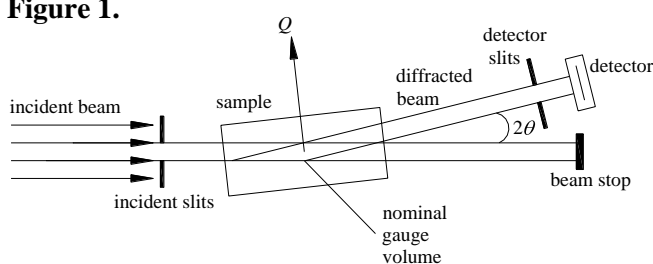
- precise definition of a gauge volume with incident and diffracted beam optics;
- reliable sample translation for spatial mapping;
- high instrumental resolution of the diffraction peak, allowing accurate determination of peak-centre variation as a response to strain;
- choice of appropriate energy giving adequate penetration for bulk investigation.

Modern third-generation synchrotron X-ray sources are able to meet these requirements, delivering high photon flux at wide energy ranges, low divergence and outstanding beam brilliance. Here, we report on the use of synchrotron X-ray scattering to investigate the residual strain in polymer materials.

### 3 EXPERIMENTAL METHODOLOGY AND MATERIALS

Measurements were made on beamline ID31 at the European Synchrotron Radiation Facility

**Figure 1.**



(ESRF), Grenoble, France. ID31 is a high-resolution X-ray powder diffractometer [12]. The layout of the modified instrument for strain measurement in polymers is shown in Figure 1. An incident monochromatic parallel beam of

X-rays is collimated via the use of precise motorised slit optics. Similarly, slits are positioned in front of the X-ray detector, defining a three-dimensional nominal gauge volume (NGV). The only X-rays recorded at the detector come from diffraction within the NGV. A combination of the hard X-rays needed for penetration and the X-ray atomic form factor necessitates a low diffraction angle ( $2\theta$ ). Thus, the NGV is an elongated ‘diamond’ shape being significantly longer in one direction (along the incident beam direction). Diffraction peaks were fitted using a

Gaussian profile with the LAMP software package [13] in order to obtain peak parameters. The peak positions were converted to lattice spacing  $d$  using Bragg's law:

$$\lambda = 2d \sin \theta \quad (1)$$

The lattice strain  $\varepsilon$  may be calculated from:

$$\varepsilon = \frac{d - d_0}{d_0} = -\cot \theta \Delta \theta \quad (2)$$

where  $d_0$  is the unstrained (reference) lattice spacing,  $\lambda$  is the X-ray wavelength and  $\theta$  the Bragg angle. It is important to note from Figure 1, that the strain measured is along the scattering vector ( $Q$ ), which bisects the incident and diffracted beams. Often the determination of the residual stress state, rather than simply the residual strains is the objective. Generally, six independent strain directions are needed to calculate stress, however usually the principal stress directions are known, thus it is sufficient to measure strain along each of these directions [14]. For example, the stress in a principal direction  $X$ , is given by:

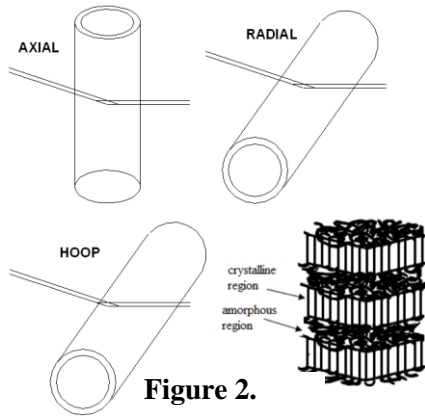
$$\sigma_x = \frac{E}{(1+\nu)(1-2\nu)} [(1-\nu)\varepsilon_x + \nu(\varepsilon_y + \varepsilon_z)] \quad (3)$$

where  $\varepsilon_x$ ,  $\varepsilon_y$ ,  $\varepsilon_z$  are the strains along the principal directions  $X$ ,  $Y$  and  $Z$ ;  $E$  is the elastic constant and  $\nu$  the Poisson ratio respectively.

Outer diameter/mm	110.0
Wall thickness/mm	12.0
Material grade	CRP 100 black
Density/g cm <sup>-3</sup>	0.959
Tensile modulus at 23 °C /MPa	900
Carbon black content /%	2.25
Crystallinity/%	72

The sample measured was a commercial high density polyethylene (HDPE) pipe provided by Lyondell Basell (Table 1). Figure 2 (bottom right) includes a schematic showing typical crystal structure in semi-crystalline polymers. The length was cut to 120 mm because of sample environment height 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 2.**

Figure 2 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).

In certain measurement positions, the X-ray path length became significant ( $\approx 45$  mm) thus an X-ray wavelength of  $\lambda = 0.354 \text{ \AA}$  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  $1 \times 1 \text{ mm}^2$  slits at the incident and diffracted beam position, producing a NGV 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  $\sim 1.5$  mm, requiring increased data collection times. Hence, the actual spatial resolution achieved across the pipe wall was similar in all three strain directions.

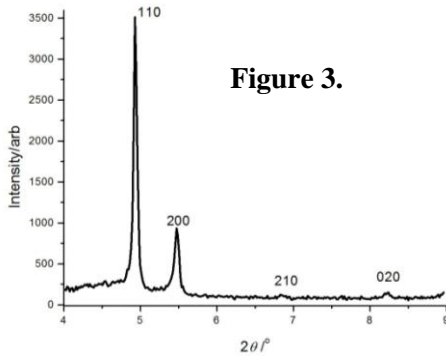
To prove the concept of measuring changes in lattice strain as a response of macroscopic strain, a test using a HDPE three-point bent bar was used. The sample was fitted with an extensometer at the surface measuring the change in lattice strain under different load conditions. The results will be presented elsewhere [15].

#### 4 RESULTS AND DISCUSSION

Figure 3 shows a  $2\theta$  scan at the mid-position in the pipe (axial direction). The diffraction data show a series of narrow peaks relating to the orthorhombic unit cell of HDPE ( $a = 7.42 \text{ \AA}$ ,  $b = 4.95 \text{ \AA}$ ,  $c = 2.55 \text{ \AA}$ ) superimposed on a broad amorphous peak. Diffraction peak positions were

obtained from the Gaussian fitting procedure with an average full width half maximum of  $\sim 0.05^\circ$ . 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 4 shows the variation of calculated residual strain  $\varepsilon$  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



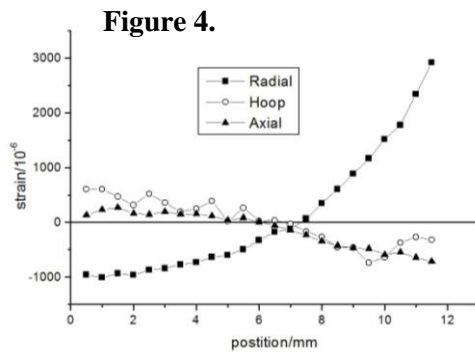
**Figure 3.**

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.

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



**Figure 4.**

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 [16].

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 [17, 18]. Further measurements are planned to investigate the texture variation and the possibility of a strain-free lattice change from wall-to-wall.

## 5 CONCLUSIONS

The new technique presented here 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 [19].

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## FIGURE CAPTIONS

Figure 1: General layout of ID31 instrumental set-up for strain measurements (in horizontal plane).

Figure 2: Measurement positions of pipe sample in X-ray beam: (top left) axial direction, (top right) radial direction and (bottom left) hoop direction. Inset (bottom right) is a schematic showing typical layering of crystal and amorphous layers in semi-crystalline polymers.

Figure 3: A preliminary  $2\theta$  scan of HDPE in the axial direction, with peaks indexed.

Figure 4: Variation of the residual strain of the sample in the axial, radial and hoop directions (zero – inner surface: 12 – outer surface).