



**Experiment title:** Time-resolved x-ray diffraction studies of simultaneous and sequential biaxial deformation of poly lactic acid under industrial processing conditions.

**Experiment number:**  
SC-2183

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<b>Shifts:</b> 9	<b>Local contact(s):</b> Dr. T. Narayanan	

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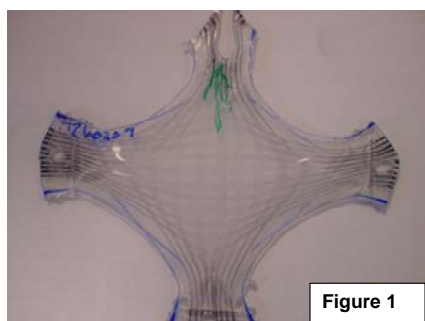
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## Report:

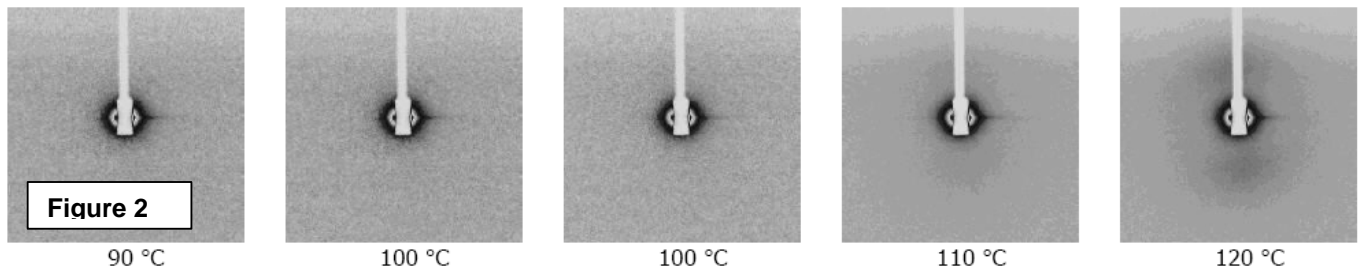
We have recorded simultaneously SAXS/WAXS/Strain/Force Applied during biaxial deformation of poly lactic acid (PLA) using the Keele biaxial camera. In this report we describe the results obtained during simultaneous biaxial deformation of PLA samples 4032D (~1.5% D-isomer) and 4060D (~10% D-isomer) at 90 °C with the draw rate of 9000% min<sup>-1</sup>. One of the major challenges involved in biaxial deformation studies is to determine suitable sample geometry for a given polymer sample. In the present study, we have successfully



adopted a modified version of a sample geometry which has been successfully developed and used in the studies of poly ethylene terephthalate. A typical image of a simultaneously and equibiaxially deformed PLA sample at 90°C with a draw rate of

9000 min<sup>-1</sup> is shown in figure 1. Figure 1 shows that the centre part of the sample was

uniformly deformed where the SAXS/WAXS data was recorded. PLA sample 4032D was simultaneously and equibiaxially drawn at  $90^{\circ}\text{C}$  with a draw rate of  $9000\text{min}^{-1}$  and was



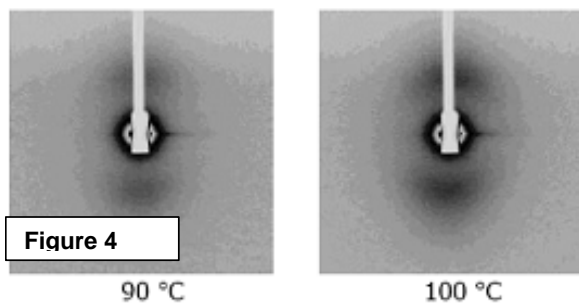
subsequently step annealed up to  $120^{\circ}\text{C}$ . During the annealing process, simultaneous

SAXS/WAXS data was recorded. A SAXS data is shown in figure 2 and the

WAXS data for the final temperature ( $120^{\circ}\text{C}$ ) is shown in figure 3. There is no

evidence of SAXS pattern at the end of the draw and up to an annealing

temperature of  $100^{\circ}\text{C}$  (Figure 2). The SAXS pattern begins to develop  $\sim 110^{\circ}\text{C}$ .



SAXS data recorded during a similar biaxial

deformation study of the PLA sample 4060D is

shown in figure 4 and the WAXS data for the final

annealing temperature ( $110^{\circ}\text{C}$ ) is shown in figure 5.

In our previous studies, under similar drawing

conditions, crystallisation has been observed in a uniaxially drawn 4060D sample.

Furthermore, unexpectedly the 4060D sample (biaxially drawn) shows a relative strong

SAXS pattern at the end of draw and it further developed during the annealing process. This

suggests a development of a two phase system while there is no evidence of crystallisation

from the WAXS data (figure 5). These observations require further

systematic investigations to identify the development of the structure/

morphology during the biaxial deformation of PLA as a function of amount

of D-isomer in PLA samples.

