ESRF

ESRF	**Experiment title:** Time-resolved SAXS/WAXS studies of conformational transitions in poly (L-lactide)	**Experiment number**: SC-1823
Beamline: ID02	**Date of experiment**: From: 22/2/2006 to: 25/2/2006	**Date of report**: 18/2/2007
Shifts: 9	**Local contact(s)**: Dr. T Narayanan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

A Mahendrasingam*, W Fuller, DJ Blundell* and M Parton*

School of Chemistry and Physics, Keele University, Staffs, ST5 5BG, UK

Dr. John Rasburn*
Innovia Films, Wigton, Cumbria CA7 9BG, UK

Report: The purpose of this investigation was to exploit the high-brilliance of the ID02 beam-line and the advances in time-resolved x-ray diffraction techniques to investigate conformational transitions in poly (lactic acid) (PLA).

In this study we have recorded simultaneously WAXS/SAXS/Strain and force applied during uniaxial deformation of PLA using the Keele drawing camera. The camera allows samples to be drawn uniaxially and to be thermally annealed at elevated temperatures with an accuracy of 1.0^{o}C. In this report we describe the results obtained during step annealing of a PLA sample, which was previously drawn uniaxially at 70^{o}C. WAXS data was recorded using a Photonics Science CCD detector with a time resolution of 40ms and sample to film distance of 60mm.

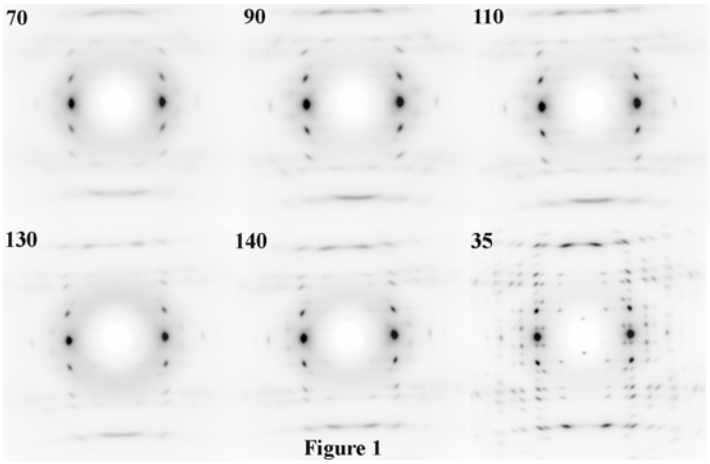
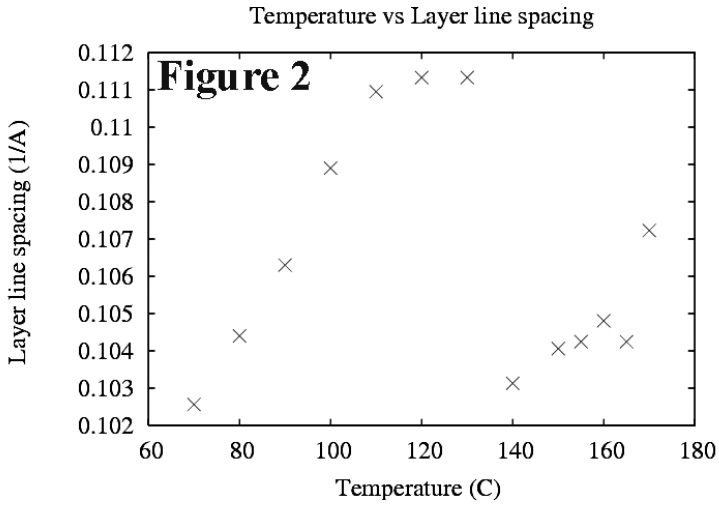


Figure 1

A selection of WAXS data recorded during the step annealing of a PLA sample at temperatures 70°C, 90°C, 110°C, 130°C, 140°C and 35°C is shown in figure 1. WAXS pattern at 70°C indicates that the sample at the end of draw is oriented and partially crystalline. The crystallinity of the sample substantially increased in subsequent step annealing up to 140°C. Further step annealing up to 170°C reduces the crystallinity substantially to a poorly oriented almost amorphous state with only a small fraction of the highly oriented crystalline form remaining. From these diffraction data we have measured the changes in the layer line spacing during the annealing, which is shown in figure 2. It can be seen that there is a gradual change in the layer line spacing up to 130°C and followed by a abrupt change at 140°C suggesting a solid state phase transition at ~140°C.



Subsequent annealing up to 170°C and cooling to 35°C of this sample resulted in a highly oriented and crystalline state as shown in bottom right of figure 1. During the above annealing process the changes in the layer line spacing was also determined and are shown in figure 3. It can be seen from figure 3, there is very little change the pitch of the helix during the annealing. This observation suggests that once a stable crystalline form (alpha form) is formed there is very little change in the pitch of the helix. Prior to this, the change in helical pitch suggests the polymer is highly polymorphic within a partially crystalline state.