ESRF

ESRF	**Experiment title:** Time-resolved SAXS/WAXS studies of strain-induced crystallisation in Poly(lactide) under industrial processing conditions	**Experiment number**: SC-1513
Beamline: ID02	**Date of experiment**: From: 2/3/2005 to: 5/3/2005	**Date of report**: 16/2/2006
Shifts: 9	**Local contact(s)**: Dr. T Narayanan	*Received at ESRF:*

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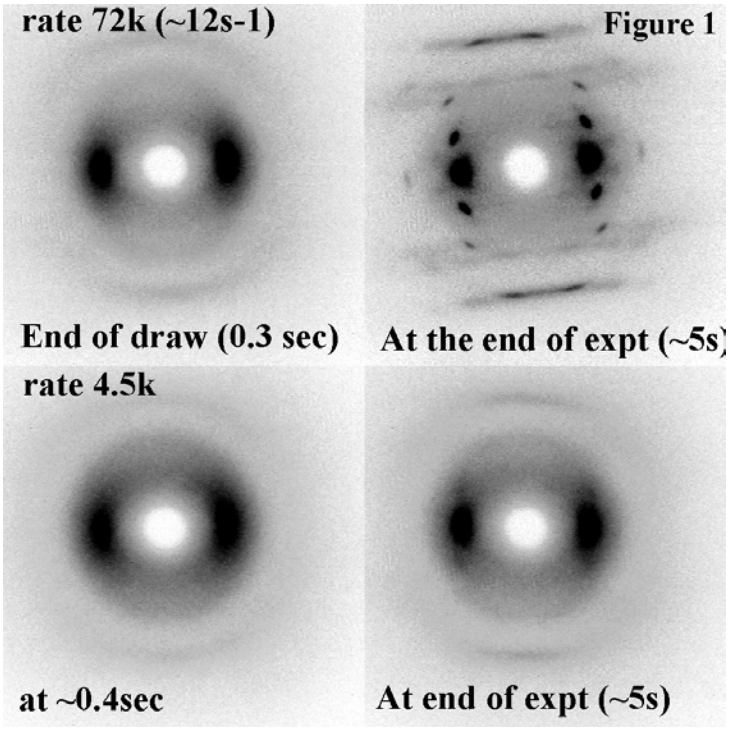
Report: The purpose of this investigation was to exploit the high-brilliance of the ID02 beam-line and advances in the time-resolved x-ray diffraction techniques to investigate the strain-induced crystallisation in Poly (Latic-Acid) (PLA) under industrial processing conditions.

In this study we have recorded simultaneously WAXS/Strain developed/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 uniaxial deformation of PLA at temperatures 70^{o}C, 80^{o}C, 90^{o}C, 100^{o}C and 110^{o}C at draw rates from 2250% min^{-1} to 72000% min^{-1}. WAXS data was recorded using a Photonics Science CCD detector with a time resolution of 40ms and sample to film distance of 60mm.

A selection of WAXS data recorded during the deformation of PLA samples at draw rate 72000% min^{-1} (top) and 4500% min^{-1} (bottom) at 70°C is shown in figure 1. Top left of the WAXS patterns indicates that the sample is oriented amorphous at the end of draw (0.3 sec) and it is partially crystalline at the end of the experiment (after ~5 seconds). Bottom left of the WAXS patterns indicates that the sample is oriented amorphous at the end of draw (~0.4sec) and its remains amorphous at the end of the experiment (after ~5 seconds). These results clearly show that the rate of crystallisation reduces with the decrease in the draw rate.



We have also determined the rate of crystallisation for samples drawn at a rate of 72000% min^{-1} at temperatures from 70°C to 110°C (figure 2). These results show that the rate of crystallisation increases with the temperature up to ~80°C, but that further increase in the temperature up to 110°C reduces the rate of crystallisation. These results are crucially important for refining industrial processing conditions to maintain dimensional stability of the film during the industrial processing of PLA.

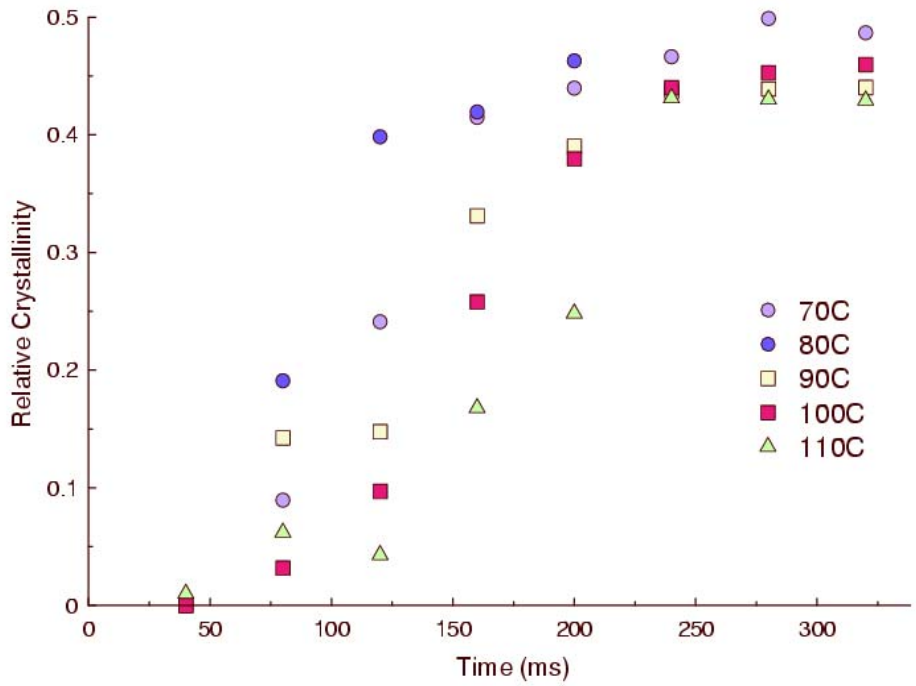


Figure 2: Relative crystallinity within a sample for a range of experimental temperatures, all at a draw rate of $12s^{-1}$.