ESRF	**Experiment title:** Application of time-resolved SAXS/WAXS studies to investigate the effect of D-isomer on the strain-induced crystallisation of poly lactic aciddtions.	**Experiment number**: SC-2053
Beamline: ID02	**Date of experiment**: from: 21/2/07 to: 24/2/07	**Date of report**: 29/08/07
Shifts: 9	**Local contact(s)**: Dr. T. Narayanan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

A Mahendrasingam*, MRM Zin*, DJ Blundell and W Fuller

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

C Konkel*

Innovia Films, Wigton, Cumbria, CA7 9BG

Report:

In this study we have recorded simultaneously WAXS/Strain/Force Applied during uniaxial deformation of poly lactic acid (PLA) with a varying level of D-isomer using the Keele drawing camera. In this report we describe the results obtained during uniaxial deformation of PLA samples 4032D (~1.5% D-isomer) and 4060D (~10% D-isomer) at temperatures 70 ^{o}C, 80 ^{o}C, 90 ^{o}C and 100 ^{o}C at draw rates from 70% min^{-1} to 72000% min^{-1}. Previous studies have suggested that there was no isothermal crystallisation in PLA 4060D. However, we have observed strain induced crystallisation in 4060D for a wide range of draw rates and draw temperatures. Rate of crystallisation in 4060D was much reduced compared to 4032D. In Figure 1 and 2 examples of true stress/ true strain curves and selected WAXS patterns showing the differences in behaviour between 4032D (Figure 1) and 4060D (Figure 2) at the nominal draw rates of 0.375 sec^{-1} and 0.093sec^{-1} for 70^{o}C.

As shown in figure 1 for 4032D, during the initial part of the drawing process, the increase in the stress/strain is uniform until the strain reaches a critical point where the sample begins to crystallise. At the onset of the crystallisation, it can be seen from Figure 1, a sharp increase in stress for a given strain. This clearly illustrate that the strain hardening is clealy linked to crystallisation in PLA. Also it can be seen from Figure 1 that the critical strain required for strain induced crystallisation increases with decrease in draw rates for a given draw temperature. The critical draw ratio required for crystallisation is ~2.7:1 at 0.375sec^{-1} and increases to ~3.2 for 0.093 sec^{-1}. Results from a similar study for 4060D are illustrated in Figure 2. It can be seen from Figure 2 that the stress/train curve for 4060D is almost identical for draw rates of 0.093sec^{-1} and 0.375 sec^{-1} with a very limitted level of crystallisation and strain hardening. For 4060D, the critical draw ratio required for crystallisation is ~4.2:1. This study clearly shows that the rate of crystallisation, the critical draw ratio required for crystallisation and the stress/strain behaviour of PLA can be fine tuned by controlling the amount of D-isomer in the sample. Such experimental information is crucially important for controlling the final properties of PLA films and containers during the industrial processing of PLA samples.

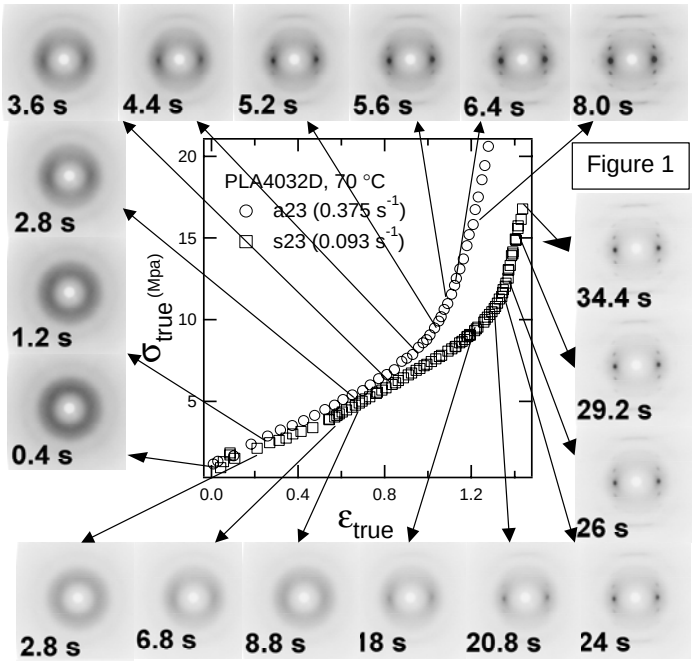

Figure 1

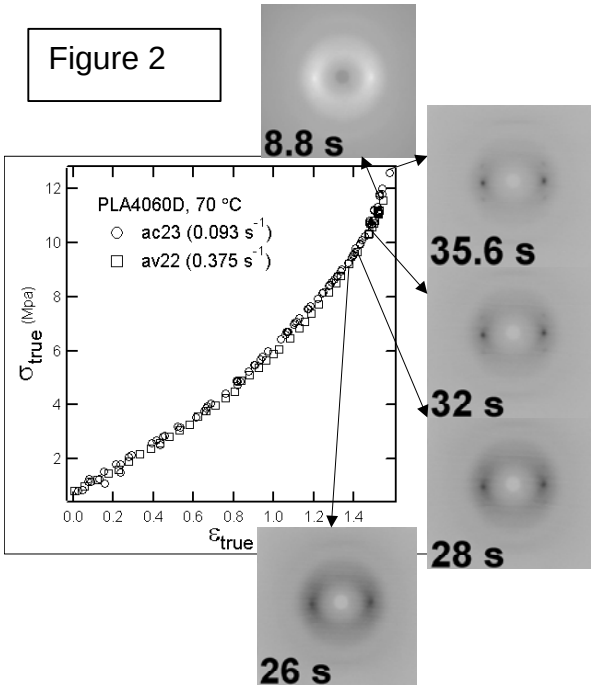

Figure 2