



<b>Experiment title:</b> Combination of high spatial and high time resolution in the study of crystallisation kinetics during drawing and annealing of poly(ethylene terephthalate) (PET).	<b>Experiment number:</b> SC-133	
<b>Beamline:</b> ID13	<b>Date of experiment:</b> from: 19.09.96 to: 21.09.96	<b>Date of report:</b> 28.08.97
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**Report:**

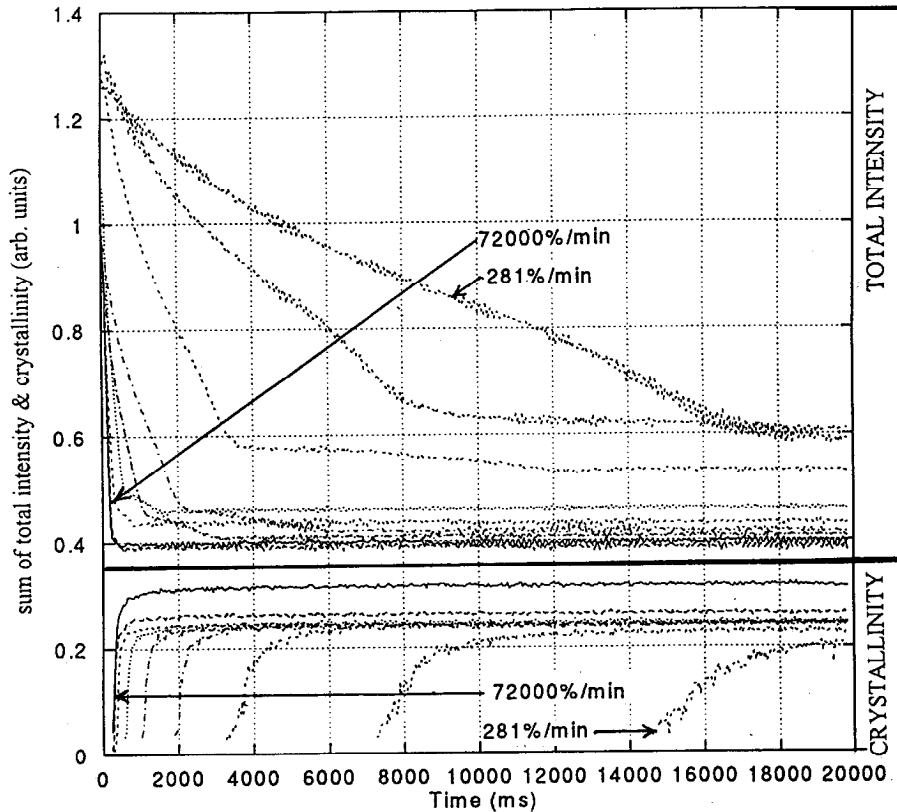
Understanding the mechanism of strain induced crystallisation in polymers is fundamentally important for industrial polymer processing. Strain induced crystallisation enables high orientation to be obtained by preventing chain slippage. It also stabilises orientation after draw and prevents shrinkage above  $T_g$ .

In the present study, we have investigated the strain induced crystallisation of PET as a function of draw rate, draw ratio and draw temperature. Samples were drawn at 90°C with a nominal draw ratio of 3.6: 1. The draw rates were varied from 281%  $\text{min}^{-1}$  to 72000%  $\text{min}^{-1}$ . In each experiment 496 diffraction patterns were recorded using a Photoic Science CCD camera linked to an i860 based Synoptics frame grabber with an exposure time of 40 msec. The analytical methodologies used for determining the crystallisation kinetics for each experiment have been previously reported [ 1]. Results from the present analyses are shown in Figure 1 and a detailed account has been submitted for publication [2].

The graphs in the top part of Figure 1 indicate the progress of the drawing process, which is monitored by integrating the total scattering in the wide-angle diffraction pattern. As the sample is drawn the total scattering decreases to a minimum, which indicates the end of draw. From these graphs it is possible to determine the local draw ratio, draw rate and end

of draw for the region of the specimen from which the diffraction pattern is recorded, within the accuracy of the diameter of the x-ray beam. In these experiments this was 30 microns.

The graphs in the bottom part of Figure 1 illustrate the crystallisation kinetics for various draw rates. It can be seen from these graphs that at faster draw rates crystallisation begins at the end of the draw. As the rate decreases to  $1125\% \text{ min}^{-1}$ , crystallisation begins just before the end of the draw. It also evident from these graphs that the rate of crystallisation decreases with draw rate.



**Figure 1:** Variation of total scattered intensity and crystallinity with time for a series of experiments in the range  $281\% \text{ min}^{-1}$  to  $72000\% \text{ min}^{-1}$ .

### References

- [1] Blundell,D.J., MacKerron,D.H., Fuller,W., Mahendrasingam,A., Martin C., Oldman,R.J., Rule,R.J. and Riekel,C., *Polymer*, (1996), 37, 3303-3311.
- [2] Mahendrasingam,A., Martin,C., Fuller,W., Blundell,D.J., Oldman,R.J., Harvie,J.L., MacKerron,D.H., Riekel,C. and Engström, P., *submitted to Polymer*.