In situ study of melting of poly(ethylene terephthalate) (PET) via simultaneous DSC and scattering: effect of annealing

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This demonstration experiment illustrates the use of our differential scanning calorimetry (DSC) sample stage for the study of melting phenomena in polymers. Pastilles of PET material before and after annealing were put between Kapton® windows and loaded on the DSC sample stage. Temperature and heat flow measurements were performed simultaneously with SAXS or WAXS in transmission geometry. The detector consisted of an image plate, which was exposed to the scattered radiation through a 2 mm wide slit. The image plate moved parallel to its surface at a speed of about 1 cm/min. In this manner a series of one-dimensional (1-D) patterns were collected as a function of temperature. The small-angle x-ray scattering (SAXS) data were analyzed via correlation function analysis, and the peaks in the wide-angle x-ray scattering (WAXS) data were fitted using a conventional minimization routine.

Figures 1 to 8 illustrate selected aspects of the data and the results. Results before and after annealing are shown on the left and right columns, respectively. The first four figures show the raw data and results for WAXS, and the last four show corresponding figures for SAXS. The heights of the 0 1 0 and 0 –1 1 peaks (located at distances on the image plate of 6 to 7 cm from the beam center) were chosen to illustrate the WAXS results as most of the fitted peak parameters followed similar trends with temperature. In addition, these peaks are well resolved and are sufficiently removed from the broad amorphous background (for which there is considerably more uncertainty on the correct fitting model). Thus, the chosen peaks are likely to yield results that may be affected to a lesser extent by underlying assumptions (e.g., background or amorphous scattering shape). Figures 3 and 4 show that annealing has a marked effect on the height variation with temperature of these peaks. The as-received sample shows an increase in the height of these peaks in the range of 220 to 245 ºC , while after annealing no such increase is observable. Similarly, the SAXS results in Figures 7 and 8 show an increase in long period (L_B) in the range of 225 to 260 ºC for the as-received sample, but no such increase is observed after annealing. The differences are obviously due to crystallization at the annealing step. The DSC curve in Figure 9 shows a sharper DSC curve for the annealed sample, while the endotherm for the as-received curve is much broader due to the presence of more imperfect cystallites.

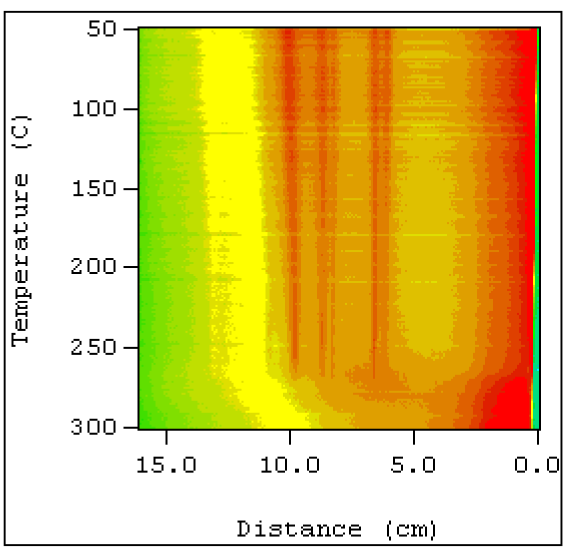


Figure 1: 1-D image plate pattern (WAXS) on as-received sample.

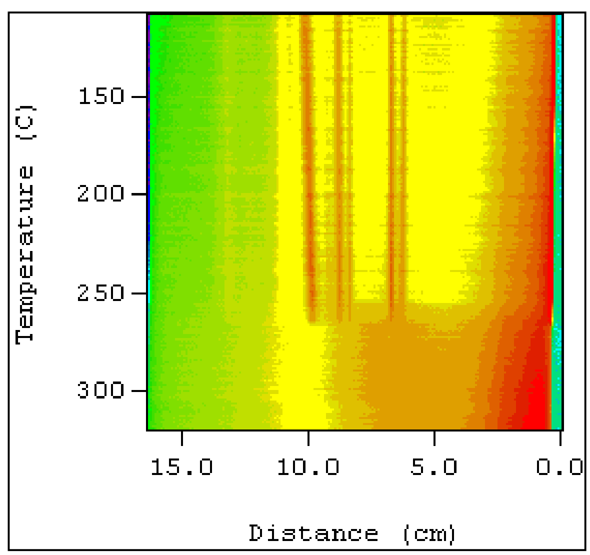
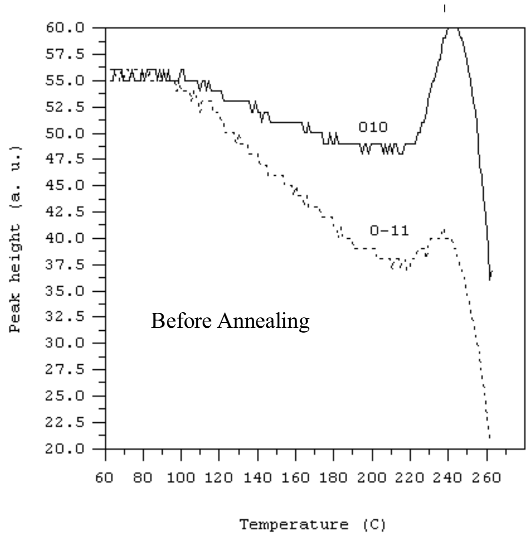


Figure 2: 1-D image plate pattern (WAXS) on sample after annealing.



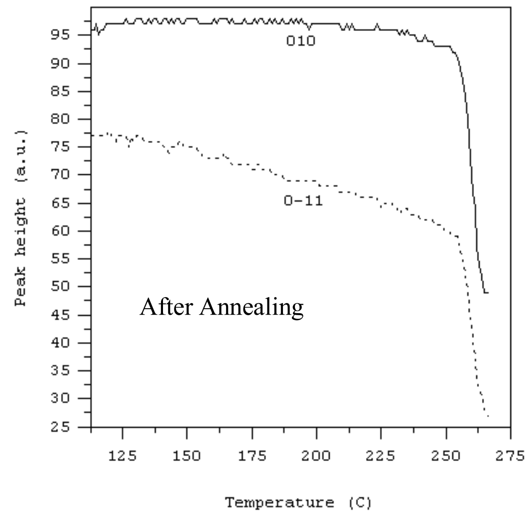


Figure 3: WAXS data from as-received sample.

Figure 4: WAXS data from sample after annealing.

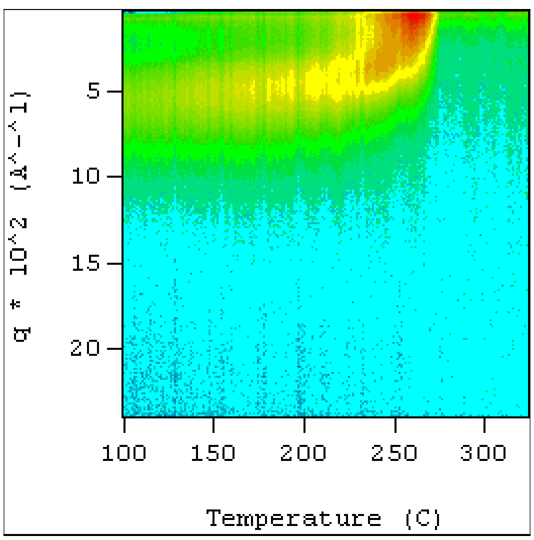


Figure 5: 1-D image plate pattern (SAXS) on as-received sample.

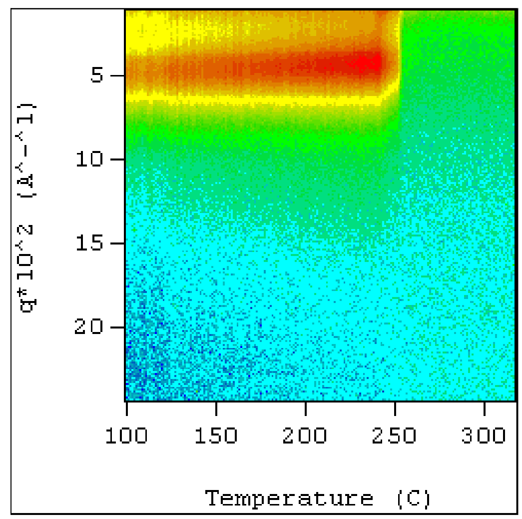


Figure 6: 1-D image plate pattern (SAXS) on sample after annealing.

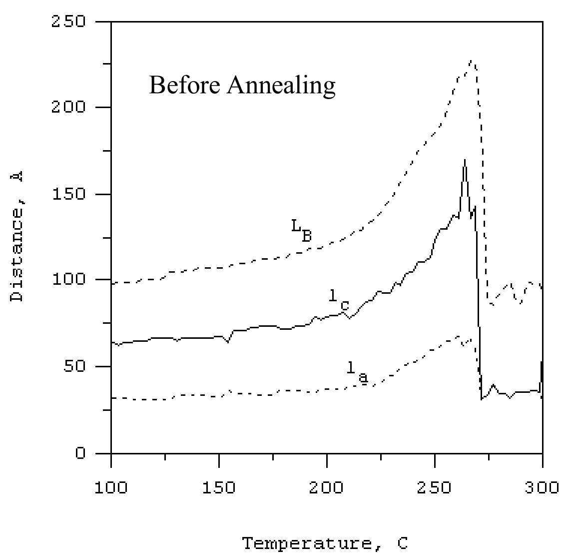


Figure 7: SAXS data from as-received sample.

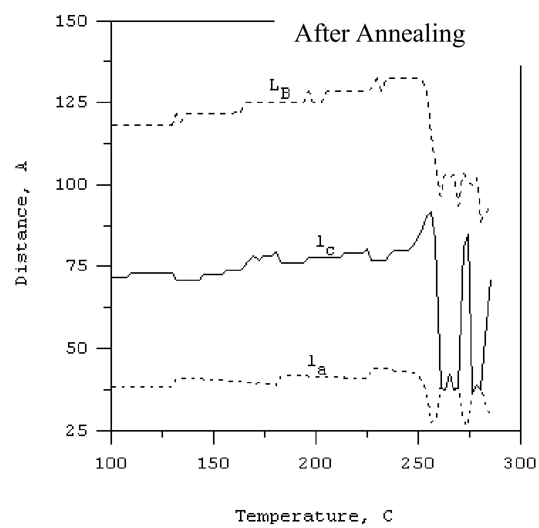


Figure 8: SAXS data from sample after annealing.

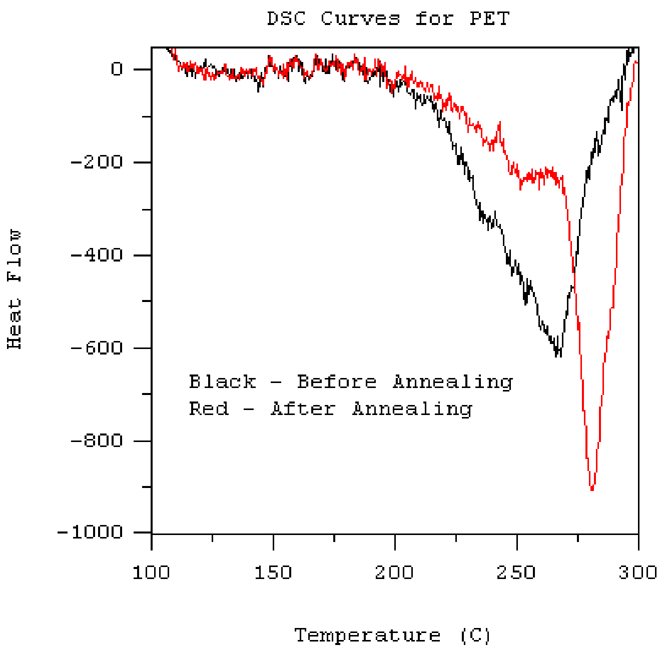


Figure 9: DSC curves for PET before and after annealing.