

**Experiment title:**

Microstructural analysis of thin polymer-metal laminates.

Experiment**number:****SC-167****Beamline:**

ID13

Date of experiment:

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Shifts:

6

Local contact(s):

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*Received at ESRF:***29 AUG 1997****Names and affiliations of applicants** (* indicates experimentalists):

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Report:

In our previous studies, we have demonstrated the use of the microfocuss beamline to investigate structural variations in spherulites of the organic polymer Biopol [1] and across the wall of a container fabricated from poly(ethylene terephthalate) (PET) [2].

In the present study, we have investigated structural variation adjacent to a polyester-aluminium interface using wide-angle diffraction techniques with an x-ray beam of 2 micron diameter. The polymer component of the laminate was produced by the co-extrusion of polyester and polyester based co-polymers which were then rolled onto an aluminium substrate. The use of plastics with and without aluminium backing as protection for food or other consumable items is demanding an ever increasing degree of technical sophistication in order to meet an increasing range of applications.

Crucial to the success of these experiments was the availability of (i) the computer controlled X/Y stage on ID13, which allowed the specimen to be tracked in two dimensions perpendicular to the X-ray beam with an accuracy of about 0.1 micron, and (ii) a Photonics Science CCD detector linked to an i860 based Synoptics framegrabber and dedicated computer system, which allowed diffraction patterns to be recorded with exposure times as short as 40 milliseconds and to be displayed in real time.

Cross-sections of the polyester/aluminium laminate (embedded in epoxy resin) were scanned with a 2 micron diameter x-ray beam in a direction perpendicular to the film surface through the two polyester layers. The total thickness of the polymer component was 12 microns and diffraction patterns were recorded in 1 micron steps from the aluminium across the interface and through the complete polymer layer. A selection from this sequence of wide-angle diffraction patterns is shown in Figure 1. In this figure, the top left pattern is from the PET layer from which it can be seen that the sample is well crystalline and oriented. As we move towards the polymer-aluminium interface, the diffraction pattern (top right) indicates decreasing crystallinity. The bottom left diffraction pattern is from the copolymer layer is largely unoriented and amorphous. The bottom right pattern shows increasing diffraction from the aluminium surface and even less polymer orientation. It is possible that this polymer layer with these characteristics may enhance adhesion to the aluminium surface while the crystalline PET layers may enhance the barrier property of the laminate.

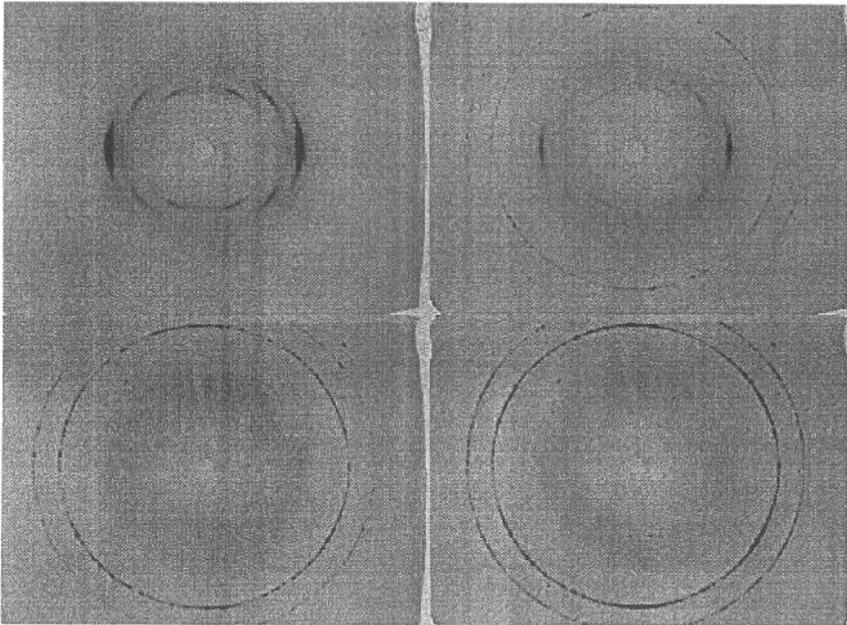


Figure 1: Selected wide-angle diffraction patterns from a PET/copolymer/aluminium laminate

References

- [1] Mahendrasingam,A., Martin,C., Fuller,W., Blundell,D. J., MacKerron,D.H., Rule,R.J., Oldman,R.J., Liggat,J., Riekel, C. and Engström,P., *J. Synchrotron Rad.*, (1995), 2, 308-311.
- [2] Martin,C., Mahendrasingam,A., Fuller,W., Harvie,J.L., Bludell,D. J., Oldman,R. J., Whitehead,J., Riekel,C. and Engström, P., *J. Synchrotron Rad.*, (1997), 4, 223-227.