



	Experiment title: Simultaneous SAXS/WAXS/Rheology during crystallization of polyethylenes	Experiment number: SC869
Beamline:	Date of experiment: from: 23/11/01 to: 25/11/01	Date of report:
Shifts: 8	Local contact(s): Dr Pierre Panine	<i>Received at ESRF:</i>
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Report:

During polymer processing the crystallization of a material gives rise to its mechanical and aesthetic properties. Studying the relationship between crystallization and structure development in materials is an important and major aspect of the polymer processing industry. Thus, gaining a deep understanding of the process allows greater control and so aiding the development of new application specific materials in the future. Following the crystallization of semi-crystalline polymers has been easily performed using simultaneous SAXS/WAXS techniques and Rheology. However, following the rheological responses of a polymer system during crystallization simultaneously with SAXS/WAXS has not been attempted until now and is usually done separately using off-line Rheometers and SAXS/WAXS at synchrotron X-ray sources where it is difficult to link the time resolution and validity of data collected.

Here, a system has been developed on ID02 where simultaneous SAXS/WAXS/Rheology of the crystallization of Polyethylenes has been performed. This has enabled the long range structural ordering from SAXS with unit cell/crystallinity information from WAXS to be coupled with the rheological data to be collected simultaneously for the crystallization of model polyethylene systems. Experimentally, a Couette geometry system has been used controlled by a Haake Rheometer, which records the rheological responses during crystallization being synchronized with the recording of SAXS/WAXS data from two CCD detectors. The experimental set-up is shown in figure 1. Figure 2, for example, details some of the data taken from the crystallization of a mono-disperse sample of hydrogenated polybutadiene (hPBD50k) blended with a similar sample containing long chain branching (comb10). Here, the sample was crystallized at

105°C having been quenched from the melt. The SAXS/WAXS and Rheology was recorded throughout the crystallization process.

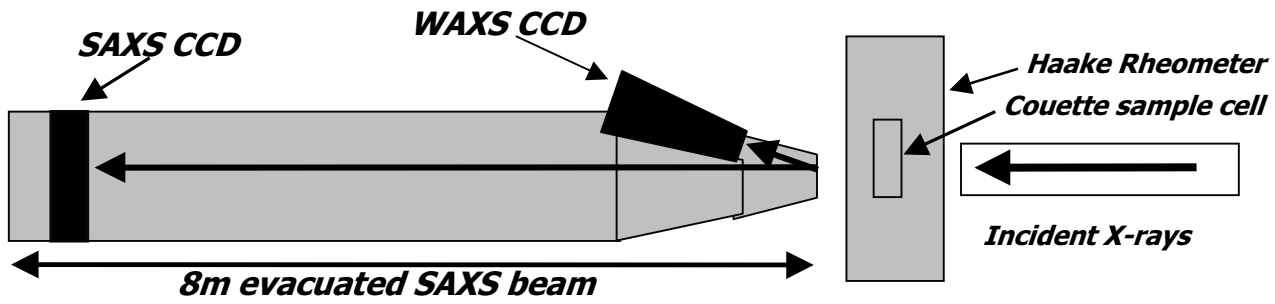


Figure 1, SAXS/WAXS/Rheology experimental set-up on ID02

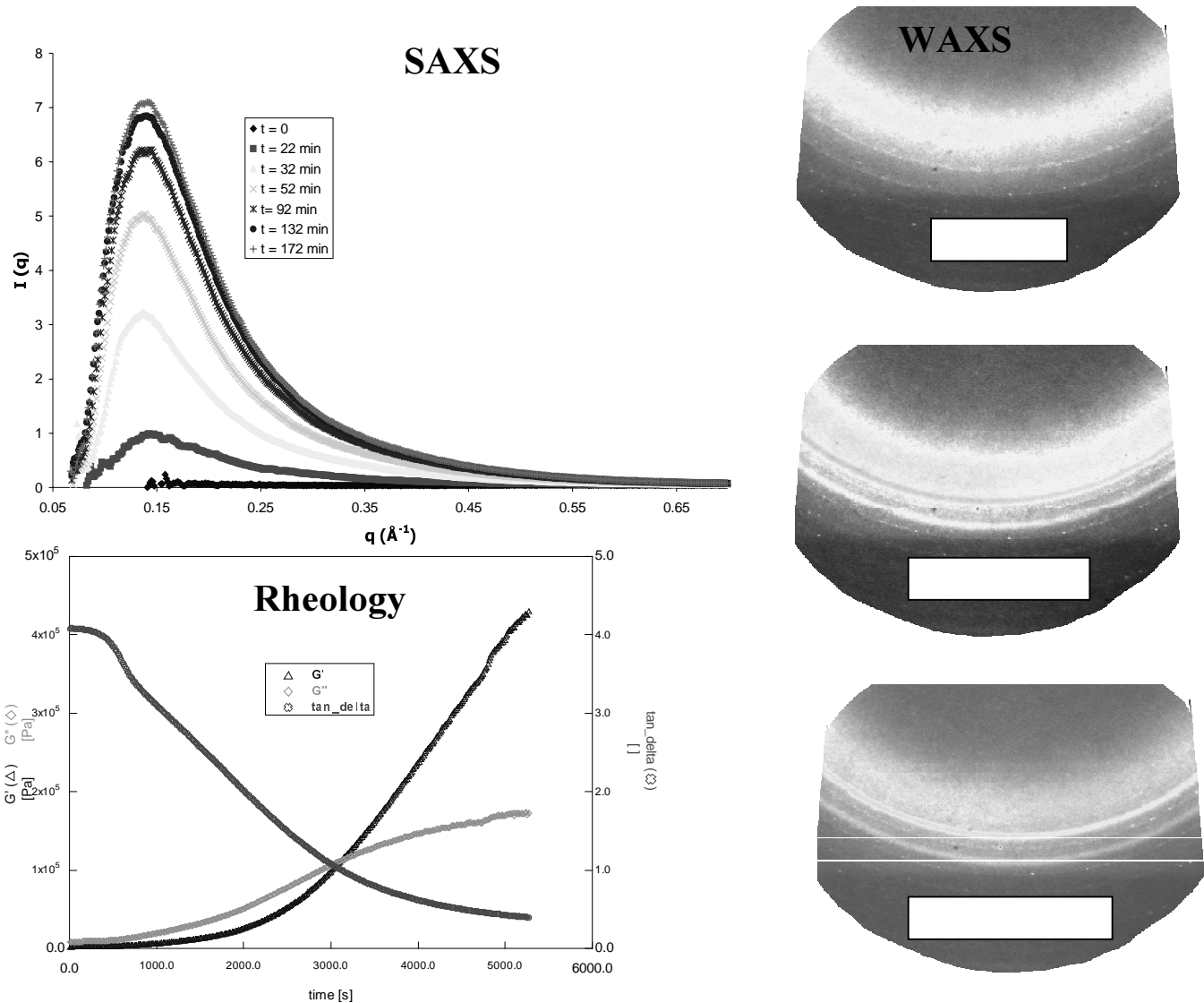


Figure 2, SAXS/WAXS Rheology during crystallization of hPBD50k + 10% comb10 at 105°C

This experimental set-up has enabled crystallization studies of specially synthesised polyethylenes to be followed. The great advantage here, is that the samples which have differing architectures can have profound effects upon the rheology of the materials thus, recording the rheology simultaneously with the scattering allows valuable processing information to be gained and the effects that unique architectures have on the macro- and micro-structure as well as the overall crystallization kinetics.