Experimental report

11/08/2022

Proposal: 9-11-1983 **Council:** 4/2020

Title: Equilibration and reversibility inpolymer/fullerene nanocomposites; continuation proposal

Research area: Soft condensed matter

This proposal is a continuation of 9-11-1903

Main proposer: Anthony M. HIGGINS

Experimental team: Valeria ITALIA

Local contacts: Philipp GUTFREUND

Samples: Polystyrene
Bis-PCBM

Instrument	Requested days	Allocated days	From	To
D17	3	3	11/06/2021	14/06/2021

Abstract:

Organic photovoltaics (OPVs) are candidates for the large-scale capture of solar radiation, due to the potential to process these materials in large areas at low cost. However, considerable challenges exist in terms of lifetime and robustness of performance. This proposal forms part of a wider effort in which our motivation is to complement device optimisation strategies with in-depth studies of model systems, aimed at increasing the fundamental understanding of the materials science within polymer nanocomposite thin-films.

This continuation proposal seeks to confirm preliminary results pointing to non-monotonic equilibrium behaviour as a function of temperature. Measurements will be performed on polystyrene (PS)/bis-adduct phenyl-C60-butyric acid methyl ester (bis-PCBM) bilayers. We will also repeat and extend measurements on the approach to equilibrium from different starting positions in phase space. Accessing the buried interface between the separated phases at elevated temperature via in-situ reflectivity measurements is the key to these experiments, allowing the probing of composition profiles at and on the way to equilibrium, and also enabling the testing of reversibility.

Experimental report

Experiment 9-11-1983; Equilibration and reversibility in polymer/fullerene nanocomposites; continuation proposal; 3 days on D17 (June 2021)

This experiment was carried out remotely, and involved thermal annealing under vacuum of fullerene/polystyrene bilayers. Due to problems due to covid19, no experimental team members were able to attend the experiment. Therefore all experimental work on site was carried out by Philipp Gutfreund. As well as the three days of experiment, which involved sample changes every few hours, this included extensive temperature calibrations, both before and after the experiment. We are very grateful to Philipp for his dedicated help and support under these difficult circumstances. As a result, the experiment was successful, and produced some interesting findings, which we are in the process of writing up. We also thank IT support at ILL for facilitating remote access to the instrument, which worked well.

We were able to perform a series of measurements in which we thermally annealed model polymer/fullerene bilayer samples *in-situ* within the neutron beam. Significant mass-transfer of fullerene occurred on heating to sufficiently high temperatures (several tens of degrees above the glass transition temperature of the components). Figure 1 shows typical reflectivity curves and fits for a sample, before and after thermal annealing. This sample initially had a bottom layer that was pure fullerene and a top layer that was pure polystyrene (PS). The sample was annealed at a range of temperatures up to around 180 °C. Using a relaxed resolution, we were able to obtain this data using only 10 minutes total acquisition time (using two incident angles of 0.8 ° and 3.0°). The fits shown in figure 1 correspond to simple bilayers (two uniform SLD layers with Gaussian roughness at the buried interface and sample surface). The relatively rapid measurement time, allowed us to examine the temporal and thermal behaviour of mixing in a systematic set of samples, using several different PS molecular weight standards.

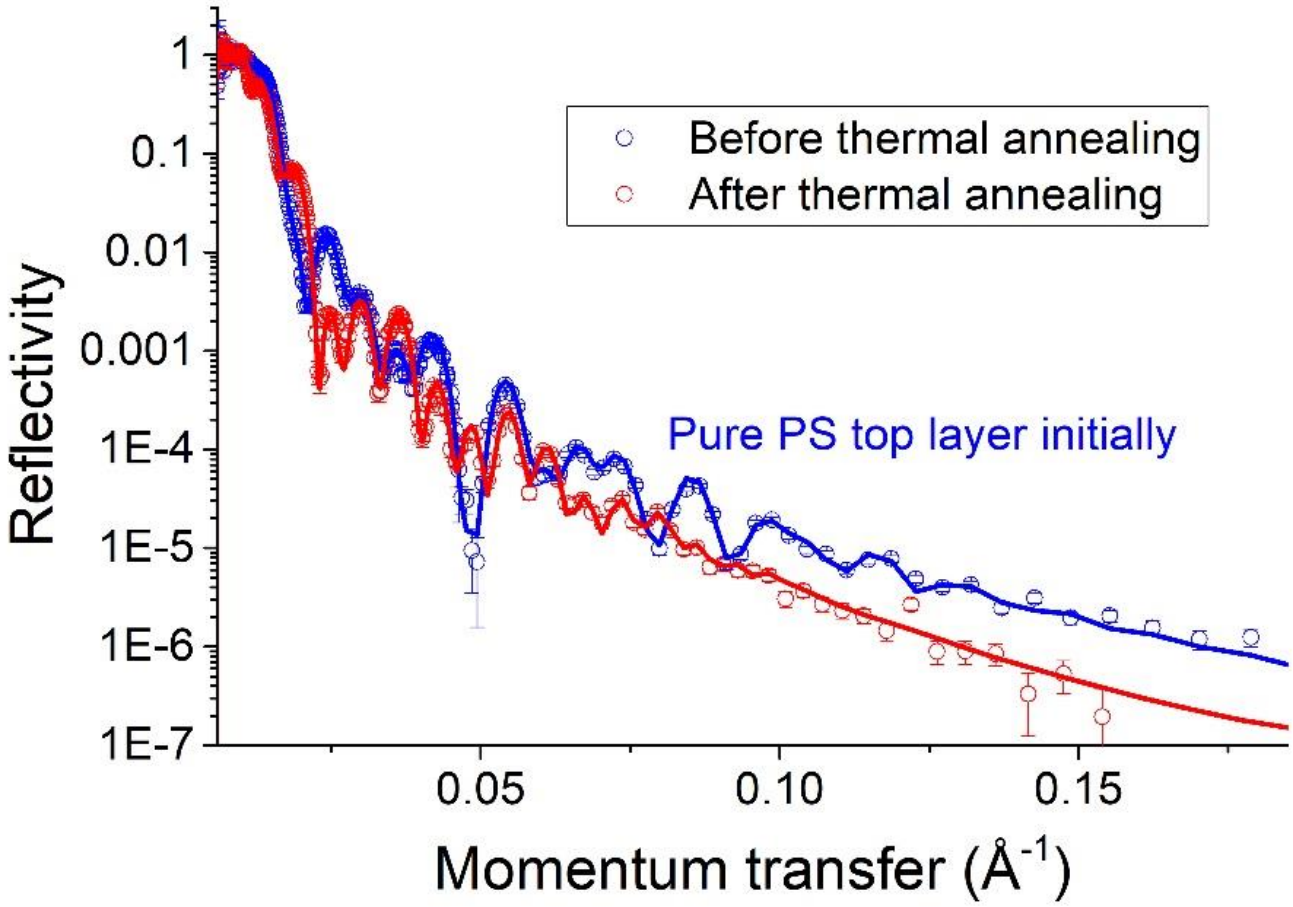

Figure 1; Neutron reflectivity curves and fits for a PS/fullerene bilayer sample before and after thermal annealing (temperature cycling) at elevated temperature. These measurements were carried out at 80 °C.