Experimental report

22/05/2016

Proposal: TEST-2522 **Council:** 4/2015

Title: Influence of the polymer-surface interaction on the entaglement dynamics

Research area:

This proposal is a new proposal

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Samples: Al2O3+(C5H10)n+(C5D10)n
Al2O3+(C5H8)n+(C5D8)n
C5H8)n+(C5D8)n
C5H10)n+(C5D10)n

Instrument	Requested days	Allocated days	From	To
IN15	10	10	16/10/2015	26/10/2015

Abstract:

Experiment report – TEST-2522_154_in15

The experiment on IN15 was performed at ILL between October 16 and 26, 2015. The experiment is focused on the influence of the polymer-surface interaction on the entanglement network, in particular at strong confinement condition. Two different polymers, polyisoprene (PI) and polyethylene-alt-propylene (PEP) were confined inside Alumina cylindrical pores. This study provided a direct microscopic measurement of the entanglement distance *d* and led to the first microscopic experimental evidence of a dilution of the total entanglement density in a polymer melt under confinement.

Both polyisoprene (PI) with molecular weight 350 kg/mol under strong confinement conditions R_{ee}/D_{pore}=2.5 (pore diameter 25 nm) and polyethylene-alt-propylene (PEP) with a molecular weight of 50 kg/mol and R_e/D_{pore}=1 have been investigated. With the proposed experiment we want to analyze the influence of the confinement on the entanglement network in absence of specific adsorption of the polymer segment on the Alumina surface, i.e. weak or neutral polymer-surface interaction. It is expected that in absence of specific adsorption the dynamical properties of the polymer chain will be only influenced by confinement.

For PI we measured at Q=0.05, 0.08, 0.1,0.15 and 0.22 $Å^{-1}$ for τ up to 250 ns in standard modus and up to ~1 ns with the shorty modus. For PEP we measured at Q=0.08, 0.15 and 0.22 $Å^{-1}$ for τ up to 250 ns only in standard modus. In both cases only one wavelength has been used: 12 Å. From existing literature for similar systems a plateau (signature for a confinement) is expected at τ~50 ns, e.g. for Q=0.12 $Å^{-1}$.

The samples were in Al container. Due to the specific structure of the pores and in order to avoid extra contributions from the Bragg peaks we tilted all the samples (i.e. confined polymers, bulk polymers, references and empty cells) of -34° for PI and -30° for PEP(in agreement with previous experiments at SNS-NSE); the tilting-angle (the angle between the incoming vector k_i and the orientation of the sample) was fixed for all Q's; the samples were tilted in the opposite rotation direction w.r.t. that of the detector. Active carbon was used as reference for Q>0.2 $Å^{-1}$, otherwise graphoil. No mask was used, but the same area of illumination for all samples (of the same art) is assured due to correct preparation of blender (left unchanged during all the experiments for PI and PEP) and alignment of the sample.

We measured: PI in confinement (Al cell) + PI as bulk (Nb cell), both at temperature ~393 K (orange cryostat)+ empty Al cell; PEP in confinement (Al cell) + PEP as bulk (Nb cell), both at 410-420 K (orange cryostat) + empty Al cell, different geometry from that of PI.

The picture below shows the intermediate scattering function (y) vs. fourier time in ns (x) for both PEP and PI (after background subtraction). The measurement have been performed aiming at a statistics of ~3%, exept for longer fourier times

at 0.22 Å^{-1}, which would have required to much time. A small but systematic slow down of the dynamics of the polymer in confinement can be observed.

Remarks: ours was one of the first experiments after the refurbishment of the coils and the first mounting the small echo (shorty modus); the tuning of the small echo took some of the beam time, for τ>0.6 ns one should maybe try with the standard modus; no empty Nb cell measured because not available.