Flow-Induced Crystallization of XL-HDPE and Instrument for Small Angle Neutron Scattering Study



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Crystallinity

Introduction

In 2014, the annual work were still focused on Flow-Induced Crystallization of XL-HDPE, as well, the preparing of SANS study.

FIC in strain-temperature space under a near-equilibrium system were focused on. A modified stretched network model (SNM) was proposed and it suggests that the transition from lamellar to shish nucleus is determined by critical thickness of nucleus rather than CST at segment level.

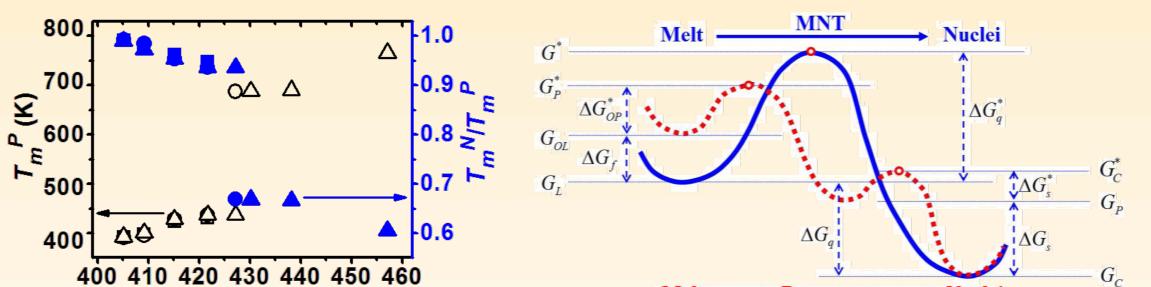
Accordingly, the thermodynamic properties of FIP were discussed and it suggests the existence of different FIPs.

A loading-unloading FIC experiment was conducted in SSRF and shows some interesting results.

The SANS experiments were preparing, and the fist test of PE blend was conducted in Dec 25th, 2014, as a landmark.

Thermodynamic Properties of FIP

It revealed that FIC undergoes two stages, namely, "melt-precursor transition" (MPT) and "precursor-nuclei transition" (PNT). FIP with different inner structures and morphologies can be induced by strain, whose embryo has a shape and structure related to the ones of the corresponding critical nuclei. With the strain-temperature diagrams, the quantitative estimation of the thermodynamic parameters are deduced and it suggest the existence of variant FIPs

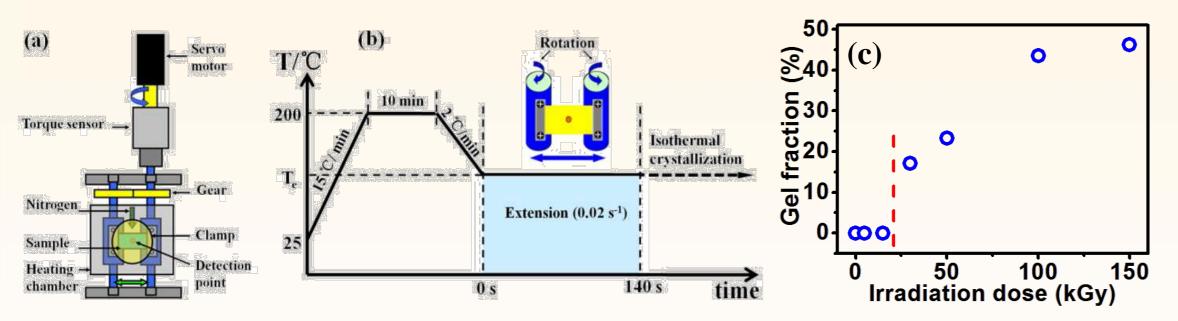


Melt Precursor Nuclei

PNT

MPT

Experiments and Samples

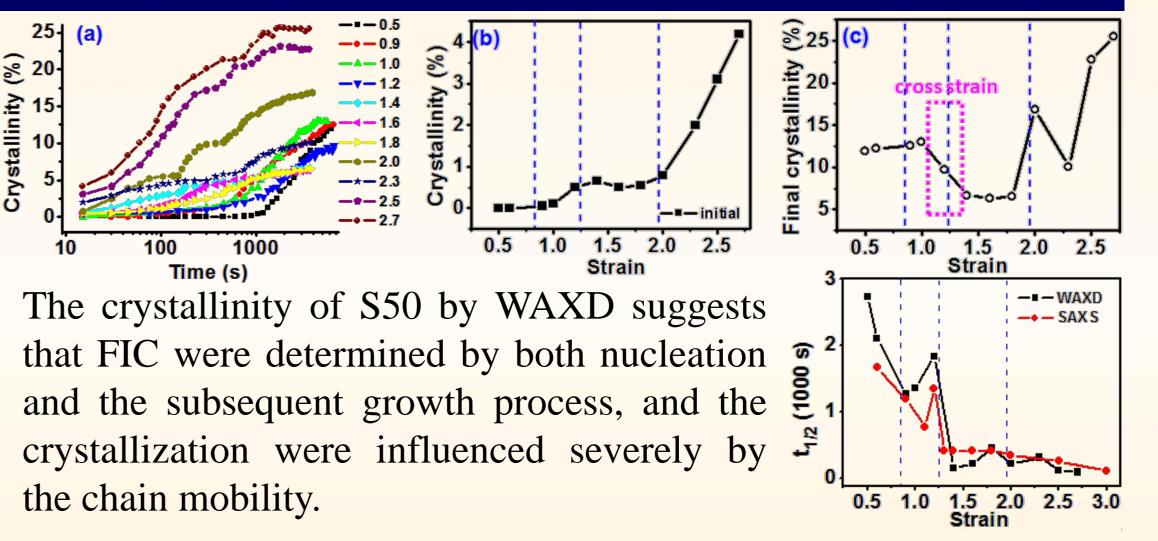


(a) The schematic drawing of the home-made two-drum extensional rheometer device. (b) One example (slow extensional) of the experimental procedures used in the *in-situ* experiment. (c) Gel fraction of XL-HDPE samples.

FIC under Near-Equilibrium Conditions

Extension FIC under near-equilibrium condition has been studied. According to crystal morphology and structure, four regions were defined in strain-temperature space, namely "orthorhombic lamellar crystal" (OLC), "orthorhombic shish crystal" (OSC), "hexagonal shish crystal" (HSC) and "oriented shish precursor" (OSP), respectively. This indicates that flow not only induces entropic reduction of initial melt, but also modifies the free energies of the final states, which is overlooked in the classical stretched network model (SNM) for flow induced crystallization (FIC). Incorporating the free energies of various final states, a modified SNM is developed and employed to analyze strain-temperature equivalence on nucleation in FIC, which reveals that the critical nucleus thickness l^* at different regions leads to a natural transition from lamellar to shish nuclei. The results suggest that classical nucleation theory is still valid for FIC under near-equilibrium condition provided that the free energy changes of initial melt and final states induced by flow are taken into account.

Nucleation and Growth of FIC

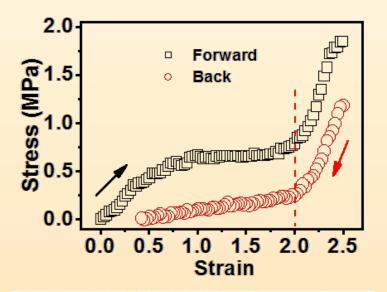


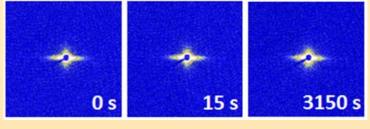
FIC of Loading-Unloading Procedure

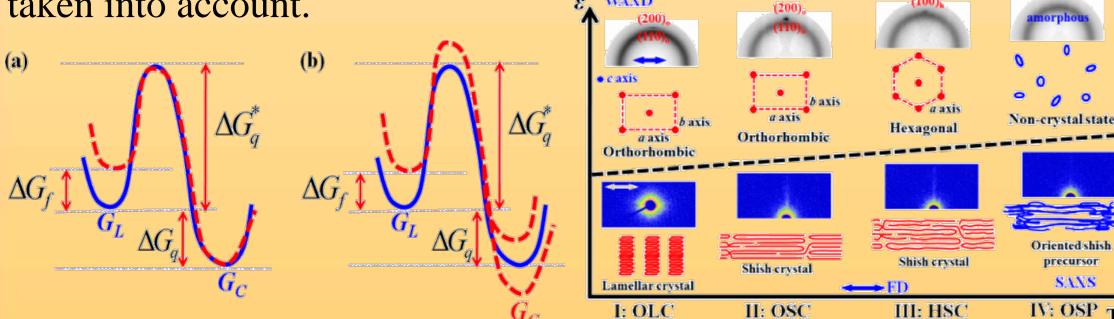
The loading-unloading procedure of FIC were designed and conducted with variant loading strain, unloading strain, and temperature.

Temperature (K)

The SAXS result shows interesting result of "Shuriken" shape patterns within some conditions. It indicates ordered some structures with intense contrast.

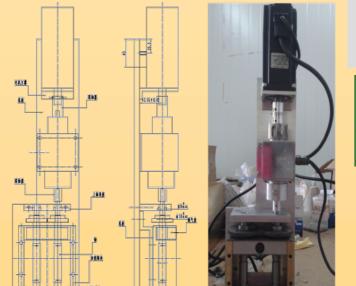


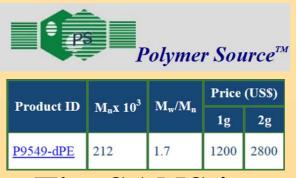


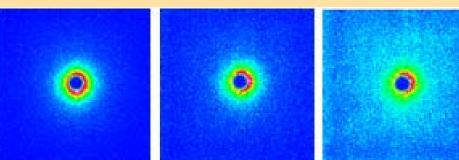


Further and solid evidences are needed before any conclusion to be drawn.

Development of SANS Instrument and SANS Experiment (with Haoran)







The SANS instrument was developed; The deuterated PE was purchased; SANS tests of dPE/hPE were conducted;

Publication or Other Achievement

Dong Liu, et al. *Macromolecules* 2014, 47, 6813–6823 Dong Liu, et al. SCIENCE CHINA Chemistry, Accepted



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