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IN-SITU MONITORING OF POLYMER CRYSTALLIZATION BY OPTICAL COHERENCE TOMOGRAPHY (OCT)

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The use of Optical Coherence Tomography (OCT), a novel interferometric method, is demonstrated for assessing the progress of polymer crystallization in-situ during a shear experiment. Unlike conventional online measurement methods - e.g. birefringence - OCT analyses the backscattered light of evolving structures and therefore is able to work in advanced states of crystallization also, where the intensity of transmitted light goes down tremendously; thus it is now possible to investigate more severe shearing conditions and to get closer towards industrial demands. The set-up consists of a single screw extruder with a slit die, which has two windows at the die exit to get the laser directed through the crystallizing polymer. To that die an optical system is attached, consisting of the OCT unit to measure reflected light on the one side and a transmission unit to measure the transmitted light on the other side. Both optical units are extended by polarizers and beam splitters to detect not only the intensity, but also the polarization state of the light, thus being able to draw conclusions on the birefringence of the pertinent structures. Experiments are performed with iPP quenched from the melt to 140 ℃ and after thermal equilibration sheared for up to 150 s by resuming extrusion for a short time. After the shear period isothermal crystallization is optically monitored. The solidified sample is then cooled to room temperature, extracted and analysed under the polarisation microscope. 3D depth-resolved images produced by OCT as well as ex-situ microtome cuts in the microscopic analysis show the development of the well-known highly oriented outer layers, exhibiting strong birefringence in the polarisation images. Structures down to 20 µm can be resolved. A quantitative