Rheology-Raman Spectroscopy: Tracking Polymer Crystallization with the Combination of a Rheometer and a Raman-Microscope

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The use of a coupled rheometer and Raman spectrometer for obtaining comprehensive insight into a materials behavior is presented. Rheology is the analytical method of choice to correlate the absolute flow and deformation characteristics of a given product with its behavior towards a certain processing or application step. However, Rheology as an integral method only yields answers on the bulk of the investigated sample. It does not give any insights into what is actually happening on the molecular level during a certain processing step. Raman spectroscopy has shown its ability as a powerful, effective and non-invasive method for chemical analysis. Coupling a rheometer with a Raman spectrometer provides direct information about the molecular structure and the mechanical properties. This is extremely useful for studying the crystallization behavior of polymer melts during processing. It can also provide insight for in-situ characterization and monitoring which can be challenging when working with on-line techniques as only relative flow fields are characterized. In this contribution we present results on the melting and crystallization behavior, two common phase transitions that are critical to the flow properties of various complex fluids. These temperature-sensitive transitions are often indicated via changes in molecular conformation, while optical measurements provide direct observation of structural characteristics. However, measurements performed on separate instruments are often challenging to correlate due to variations between samples, processing history, and temperature control. To demonstrate the capabilities of the rheo-Raman microscope we provide simultaneous Raman, rheological, and optical measurements on high density polyethylenes during crystallization as well as on a commercial cosmetic emulsion during melting and phase separation. The experimental setup represents a novel integration of commercial instrumentation: a Raman microscope (Thermo Scientific DXR) and rotational rheometer (Thermo Scientific HAAKE MARS III) are coupled through an optically transparent base modified from the Thermo Scientific RheoScope Module.