Rheo-Microscopy: Simultaneous Structural and Rheological Information

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ABSTRACT

A modular optical accessory kit which can be mounted onto a standard research rheometer allowing microscopic investigations simultaneously to rheological measurements is described. Details of the Rheo-Microscopy devices as well as various application examples such as emulsions, starch gelation, or crystallization processes are presented.

INTRODUCTION

Rheological methods reveal information on macroscopic material properties. However, the mechanical material properties are strongly dependent on the underlying microstructure. Therefore information on the microstructure is often valuable for a better understanding of the rheological behavior. Simultaneous use of rheological and optical techniques, i.e. of rheo-optical methods, is helpful to gain a better understanding of the dependencies between the microstructure and the mechanical properties of complex fluids, like food products.

Microscopic techniques are a widely used for investigations of micrometer-sized structures. In contrast to scattering methods a microcopy image shows individual structure elements. The combination of a microscope setup with a rheometer offers the possibility to follow structural changes simultaneous to the rheological flow behavior. Typical applications of Rheo-Microscopy are all kinds of complex fluids like surfactant solutions, colloidal suspensions, emulsions, polymer solutions, and polymer blends.

To facilitate such measurements with a high quality of the microscopic images as well as the rheological data a special modular optical accessory kit has been developed for the use in combination with the Anton Paar MCR rheometer platform. Different temperature control systems based on Peltier or electrical resistance heating as well as different optical techniques are available.

RHEO-MICROSCOPY SETUP

The microscopy setup as sketched in Figure 1 consists of a CCD camera, a microscopy tube and a long working distance objective. The illumination is integrated in the microscopy tube and illuminates the sample from the bottom side. Due to the modular design the light source as well as the CCD camera and the objectives can be exchanged. In order to obtain the highest optical quality a special glass thickness corrected objective is used. To provide a good mechanical stability and a good temperature control the bottom glass needs a certain thickness. Standard microscope objectives are designed for observing objects through very thin glass slides with neglect able thickness. The use of a standard objective in combination with

glass plates with a thickness of several millimeters lead to a significant blurring of the image and a reduction in the resolution. The microscope is movable in y- and z-direction for focusing and selecting a certain observation area. The rheological measurements take place at the top of a glass plate heated by Peltier elements. An additional Peltier heated hood can be used for assuring an accurate temperature control and a uniform temperature distribution throughout the sample in the temperature range from -20° up to 200°C. At low temperatures a flow of dry air is used to avoid condensation and ice formation at the bottom of the lower glass plate. The parallel-plate measurement geometry is made of glass as well to prevent unwanted reflections.

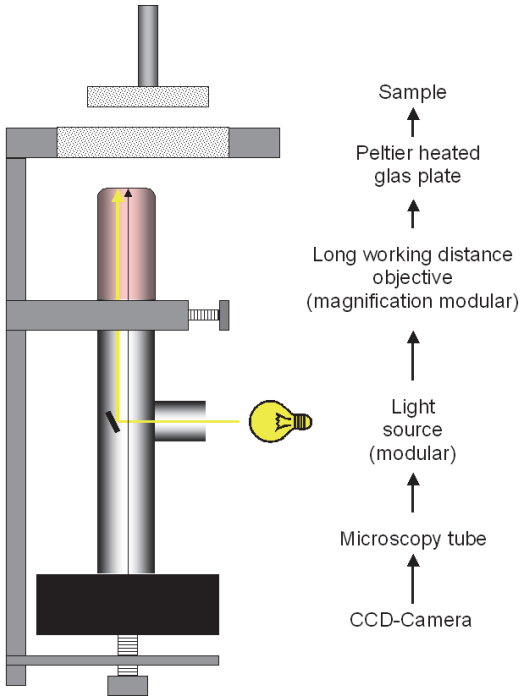

Figure 1. Principle of the Rheo-Microscope setup.

The microscope can be equipped with polarizers in order to be able to measure under the condition of crossed polarizers.

For measurements at higher temperatures a system based on electrical resistance heating is available providing measurement conditions up to temperatures of 300°C.

RESULTS

As a first example an immiscible polymer blend with domains of polyisobutylene (PIB, 1%) in a polydimethylsyloxane (PDMS) matrix is shown in Figure 2. The measuring geometry was a parallel-plate system with a diameter of 43 mm and a gap of 0.3 mm. As expected the PIB domains are spherical for the PIB/PDMS sample at rest and stretched and oriented along the flow direction for the sheared state. A more quantitative investigation reveals that the droplets start to be deformed at the shear rate where the zero-shear viscosity region is finished and the shear thinning region begins.

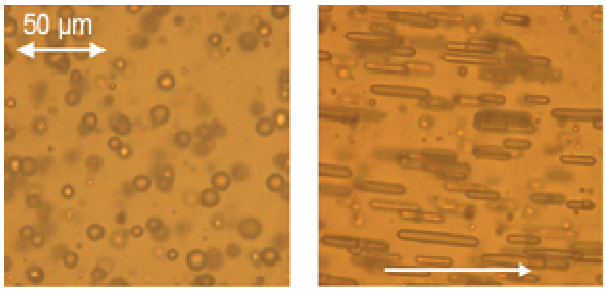

Figure 2. Microscope images of the PIB/PDMS sample at rest (left) and at shear rate 50 s^{-1} (right). The arrow in the right side indicates the flow direction.

In Figure 3 the crystallization of a palm oil sample is depicted. Microscopy images were taken simultaneously to the application of a constant shear rate of $1s^{-1}$ while the sample was cooled down constantly from 60°C to 20°C. During the measurement the viscosity value increases strongly indicating a cross over from a liquid like to a solid like sample. In the liquid phase individual

crystals occur whereas in the solid phase the sample is almost completely crystalline.

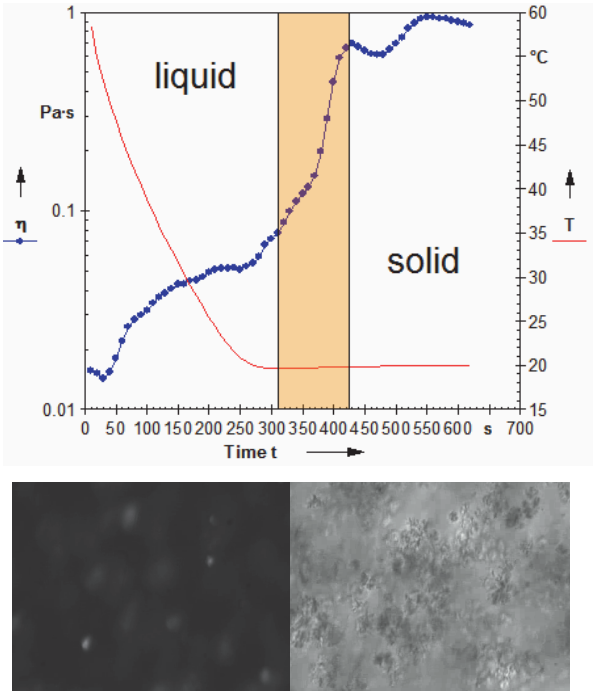

Figure 3. Viscosity of a palm oil during cooling (top) and microscope images in the liquid phase (bottom left) and the solid phase (bottom right).

Two different starch samples have been measured in a constant oscillation mode with at a strain of 0.1% and an angular frequency of 10 rad/s after a temperature step form ambient to 90°C. As can be seen in Figure 4 the gelation process was much slower for the pure starch sample compared to a starch and sugar mixture. For both samples the process from the initial starch granules, which start to swell after some time, to the bursting of the granules and the gelation of the starch can be followed with the microscopic images. For the starch and sugar mixture images before and after the strong increase in the moduli are shown in Figure 4.

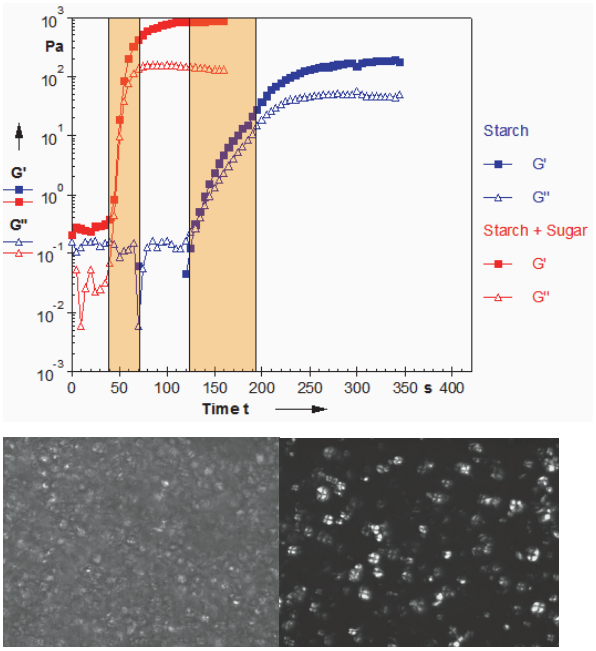

Figure 4. Storage and loss moduli for two starch samples after a temperature step to 90°C (top) and microscope images before (bottom left) and after strong rise in the moduli (bottom right) for the starch and sugar sample.

CONCLUSIONS AND OUTLOOK

Rheo-Microscopy is a very helpful tool to visualize structural changes on a micrometer length scale simultaneously to rheological measurements which in turn provide macroscopic material functions. For a thorough understanding of the processes involved only the combination of a microscope system with a research rheometer provides the full information on the structure and the rheology. Further possible enhancements of the setup include the integration of fluorescence and UV-microscopy to extend the application range of the technique.