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APPLICATION NOTE

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Image acquisition with the RheoScope module at high shear rates using a stroboscope light source and contrast enhancing illumination

Author

Fritz Soergel, Fabian Meyer, Philippe Sierro and Cornelia Küchenmeister-Lehrheuer

Thermo Fisher Scientific, Karlsruhe, Germany

Overview

Purpose: During processing and application, shear rates in the order of 10,000 to 100,000 s^{-1} may occur over shorter or longer times, which can lead, e.g., to breaking or coales-cence of emulsions or other unwanted effects, causing a change in morphology or particle size distribution.

Methods: For characterization of the emulsification process and for investigation and optimization of the further processing and/or application of emulsions, simultaneous rheometry and microscopy is a really powerful tool.

Results: Sharp microscopic images can be achieved at shear rates up to 50,000 s^{-1} using a stroboscope instead of a cold light source. For emulsions with low optical contrast, the quality of the images can be improved significantly by adding a half circle diaphragm in the lighting tube.

Introduction

Emulsions play an important role in foods, pharmaceuticals, cosmetics and many other areas. During the emulsification process it is desired to achieve a defined droplet size distribution. In the further processing of emulsions as well as in their application, the material is subject to mechanical shear stresses, which can lead to breaking [1], coalescence (Fig. 2) or other unwanted effects causing a change in morphology or particle size distribution [2, 3]. Furthermore, the stability achieved by proper formulation and processing, can be negatively affected by inappropriate storage conditions.

Using a technique that, on the one hand, simulates and, on the other hand, measures the effects of all these influences, enables designers and manufacturers to optimize formulations and processing as well as product performance.

For a deeper understanding of structure-properties relationships, usually more than one analytical technique is required. However, when samples are tested on two separate analytical instruments, the comparability and reproducibility of the results have certain limitations. Only when exactly the same material is subject to a simultaneous investigation, can these limitations be overcome.

Rheometry delivers an integral mechanical response of the investigated sample (bulk properties) under stress or deformation. The mechanical properties, however, are directly related to the molecular and microscopic structure as well as its changes with time, temperature and mechanical impacts [1, 4-6].

Combining traditional rheometry with other analytical techniques allows for a comprehensive investigation and provides a more complete picture of the sample characteristics. The comparability of all data sets is guaranteed and reproducibility is improved. Efficiency is higher, while sample consumption and lab space requirements are minimized. Finding the right complementary technique is a key factor for maximizing the information gained. Optical as well as spectroscopic methods provide structural information beyond the dynamic-mechanical response and are therefore predestinated for simultaneous application with rheometry.

Simultaneous rheometry and light microscopy

The combination of rheometry with light microscopy (with or without polarization filters) allows the study of the change in a material's structure and properties with shear rate in rotational measurements. The recorded microscopic images reveal the induced structural changes like orientation, deformation, coalescence, aggregation or disaggregation. In addition, stress/strain or temperature dependent changes, e.g., crystallization can be investigated with non-destructive oscillatory measurements [4-6].

The Thermo Scientific™ RheoScope™ module (Fig. 1) is a compact accessory for the Thermo Scientific™ HAAKE™ MARS™ Rheometer (Modular Advenced Rheometer System) integrating an optical microscope, a video camera and a temperature control unit (-5 °C to 300 °C). The typical resolution limit of a light microscope (1 µm) is already reached when the RheoScope module is equipped with a x 20 lens – alternatively x 5, x 10 or x 50 lenses can be mounted. Plate/plate and plate/cone measuring geometries with a diameter up to 60 mm can be used. For improved image quality with transparent samples, rotors with a polished surface are available.

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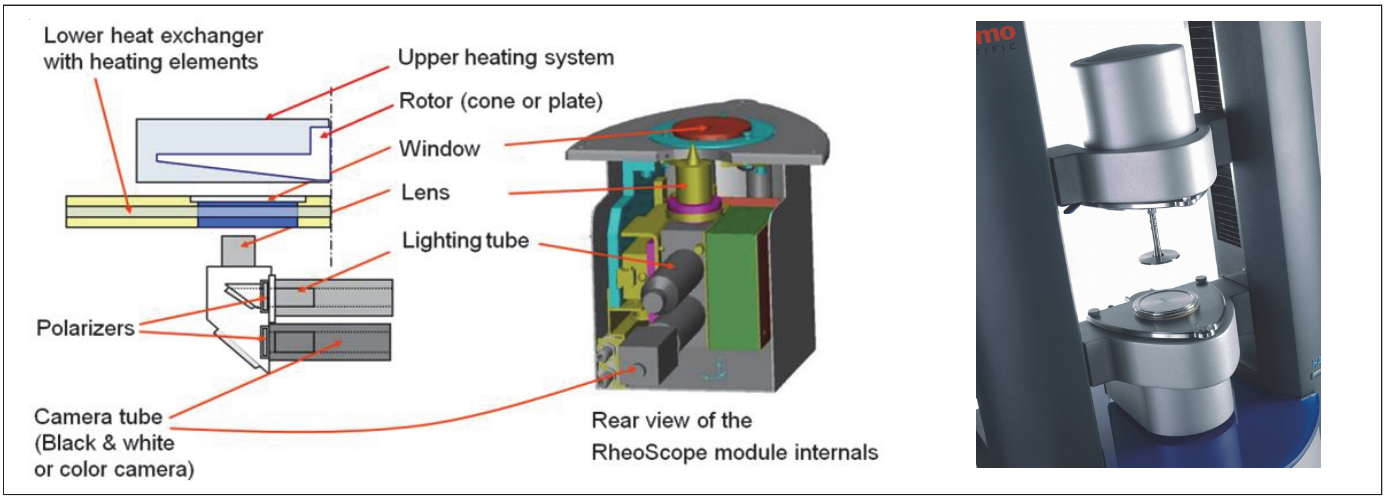

Fig. 1: RheoScope module: Schematic drawing (left: side view, center: rear view) and mounted in the HAAKE MARS Rheometer (right).

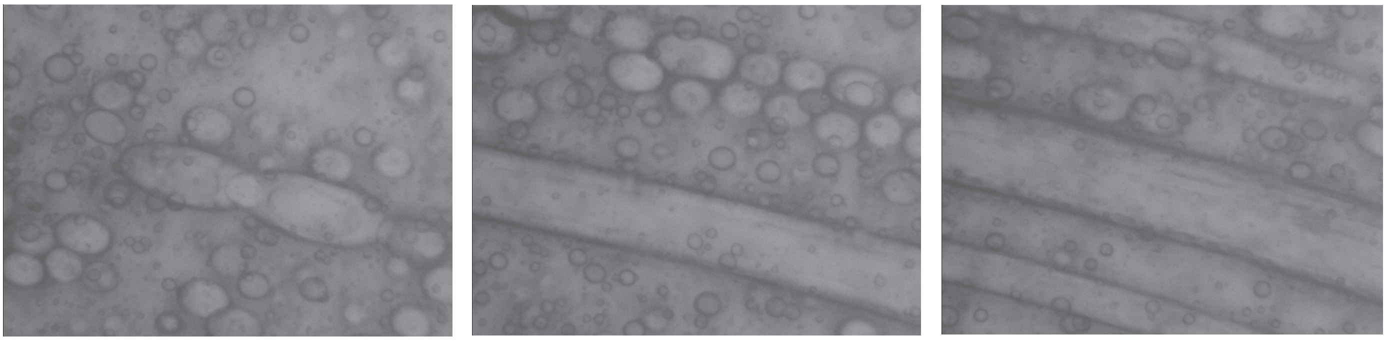

Fig. 2: Coalescence: at 500 s^{-1} applied for 0.3 s (left) and for 5.4 s (center) as well as at 1000 s^{-1} applied for 4.8 s (right).

The complete control of the optical components is fully integrated into the measuring software, including radial positioning of the lens and focus adjustment. Both polarization filters can be moved into and out of the optical paths as shown in Fig. 1. Additionally, the polarization can be crossed by angular adjustment. Contrast, brightness, gamma value and (auto) exposure time of the camera can be set. Complete sets of all these parameters can be saved and loaded, e.g., for using different parameter sets for different states of a sample within one measuring job. Rheological data and microscopic images are one-to-one correlated and can be viewed during measurement and analysis.

Coalescence can occur in a salad dressing even at lower shear rates and within a short time. Fig. 2 depicts such an experiment: First a constant shear rate of 500 s^{-1} was applied in CR mode (controlled rate), followed by a shear stress of 0 Pa for 10 s in CS mode (controlled stress). Then twice the initial shear rate was applied (1000 s^{-1} in CR mode). The RheoScope module was equipped with a x 20 lens (images size 640 x 480 µm). A C60/1° Ti rotor with a polished surface was used.

High shear imaging

Particle and droplet sizes as well as their size distributions can be determined, even at (very) high shear rates using a stroboscope (Fig. 3) instead of a cold light source, connected to the same light guide. Swapping requires no tools and takes only a few seconds. Sharp microscopic images can be achieved up to 50,000 s^{-1} with the stroboscope, while with a cold light source (depending on the individual sample) images up to a shear rate of 2000 s^{-1} can be taken. This means that the use of a stroboscope with the RheoScope module extends the measuring range by a factor of 25 or more [7].

Thanks to the stroboscope, for example the behavior of paints and inks during the coating process or the coalescence of emulsion droplets under (very) high shear rates can be investigated with the RheoScope module. Shear-induced orientation and deformation in a sample as well as the structural disaggregation and recovery under defined temperature and shear conditions are further examples of its capabilities.

At lower shear rates, moreover, the stroboscope can improve the quality of the microscopic images due to a shorter exposure time and a higher light intensity [7]. For direct comparison, microscopic images of an oil-in-oil dispersion of silicone oil in mineral oil were recorded at three different shear rates using a cold light source (Fig. 4) or a stroboscope (Figs. 5-6). A x 20 lens and a plate/plate measuring geometry with a diameter of 35 mm and a measuring gap of 50 µm were used; the surface of the rotor was polished.

Significant differences were already visible at shear rates of 1290 and 2520 s^{-1}. The images taken with the stroboscope with a flash frequency of 20 Hz (Fig. 5) had a signi-

Fig. 3: Stroboscope (BVS-II Wotan, Polytec GmbH), equipped with a Xenon flash lamp, maximim flash frequency 20 Hz (200 Hz with internal trigger).

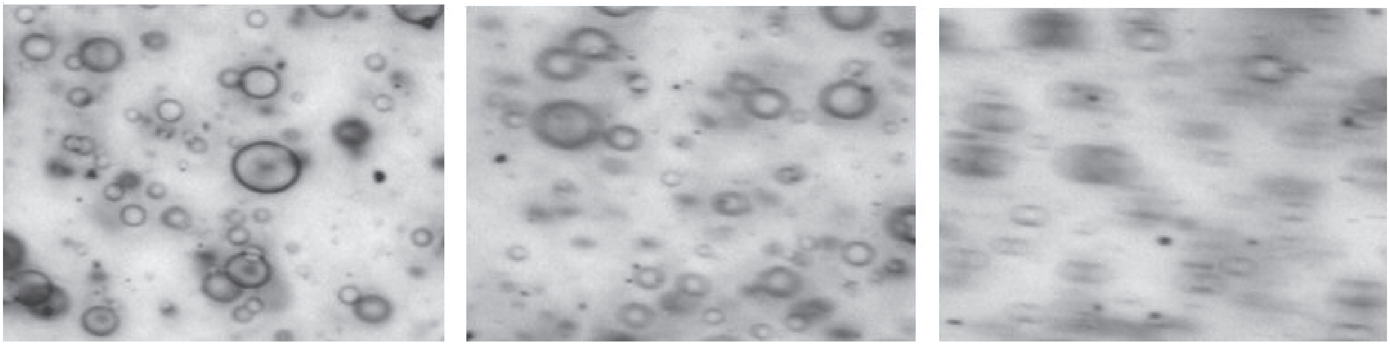

Fig. 4: Images taken at different shear rates using a cold light source: 630 s^{-1} (left), 1290 s^{-1} (center) and 2520 s^{-1} (right).

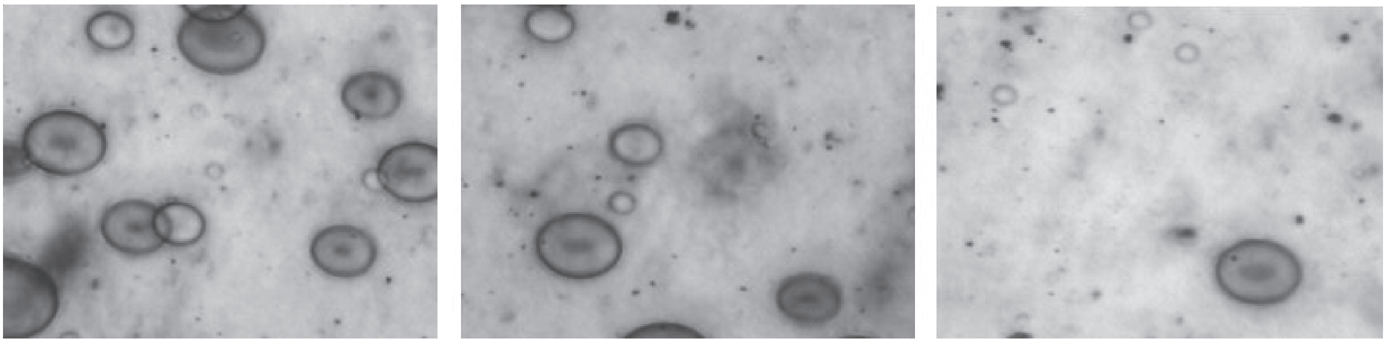

Fig. 5: Images taken at different shear rates using a stroboscope: 630 s^{-1} (left), 1290 s^{-1} (center) and 2520 s^{-1} (right).

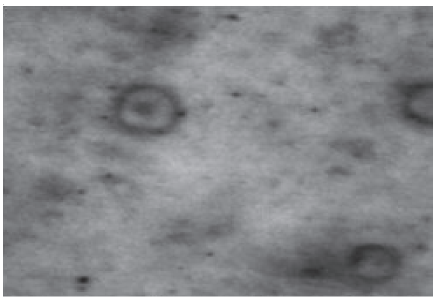

Fig. 6: Images taken at a shear rate of 44,000 s^{-1} using a stroboscope.

ficantly higher quality than those obtained with a cold light source (Fig. 4). With the same sample, evaluable microscopic images could be obtained up to a shear rate of 44,000 s^{-1} (Fig. 6).

Contrast enhancing illumination

Samples with low optical contrast contain different components having similar refractive indices, e.g., oil-in-oil emulsions or chocolate melt/spread containing different kinds of fat. The quality of the these microscopic images can be improved significantly by adding a half circle diaphragm in the lighting tube [8].

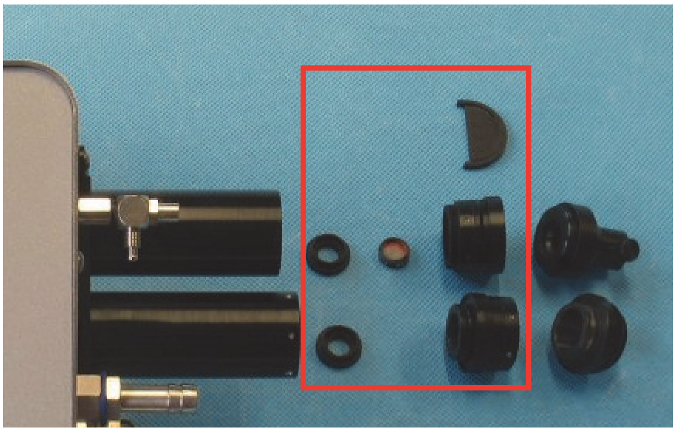

Fig. 7: Optional segment universal holder (red box) for RheoScope module lighting tube (center left) and/or camera tube (bottom left), offering a slit to mount a half circle diaphragm (top) as well as a mounting ring for a standard filter with a 12.5 mm metric thread (center).

The required universal holder can be mounted easily as an additional segment either in the lighting tube and/or in the camera tube (Fig. 7) of any RheoScope module. Moreover, these holders can be used for individual measuring set-ups with standard filters with a 12.5 mm metric thread, e.g., for wavelength-dependent investigations using color filters, long pass or short pass filters [8].

Fig. 8 shows microscopic images (lens x 20) of a dispersion consisting of silicone oil S1000 (< 5 %) in mineral oil E6000 (> 95 %). A 35 mm plate/plate measuring geometry was used with a polished rotor surface and a measuring gap of 100 µm. Comparing the images without (left) and with half circle diaphragm (right), a significant contrast enhancement can be seen – particularly for smaller droplets. This contrast enhancement also improves the accuracy of a subsequent particle size distribution determination, using an image analysis software, e.g. SPIP™ software [3]).

Conclusion

Shear rates of 10,000 s^{-1} and more can occur over shorter or longer times during processing and application of materials in coatings, foods, pharmaceuticals and cosmetics and can lead for example to breaking or coalescence of emulsions or other unwanted effects causing a change in morphology and particle size distribution. The HAAKE MARS Rheometer with RheoScope module in combination with a stroboscope light source facilitates recording of sharp microscopic images at (very) high shear rates for formulation development, processing optimization and stability testing. By adding a half circle diaphragm in the lighting tube, the contrast of the images can be enhanced significantly.

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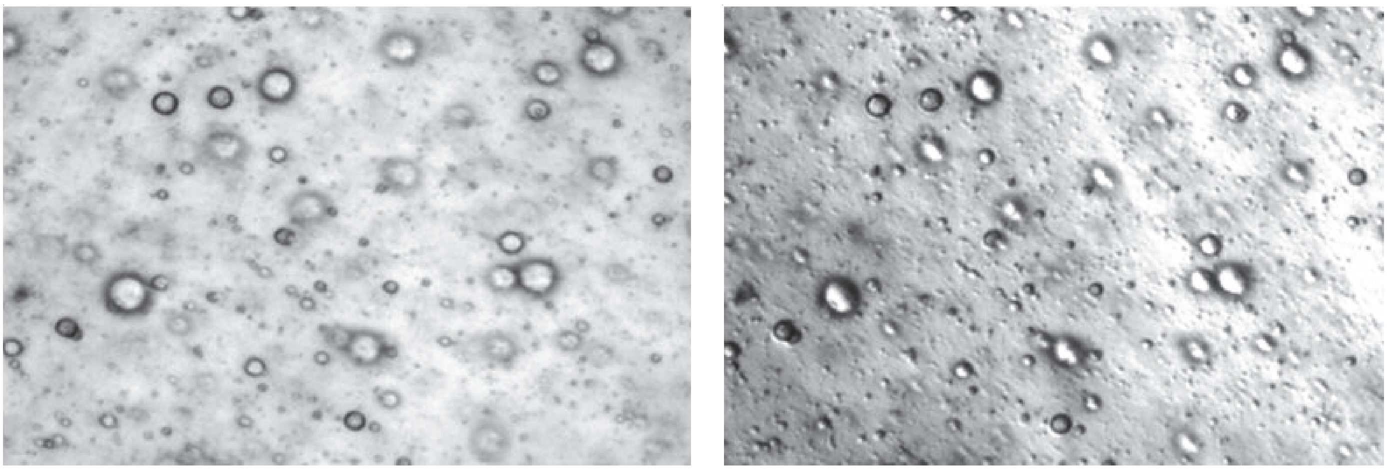

Fig. 8: Images taken on an oil-in-oil emulsion without (left) and with half circle diaphragm (right).

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