Optimized Rheo-optical Measurements Using Fast Fourier Transform and Oversampling

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Abstract:
Rheo-optics is a method that allows the analysis of optical properties, like birefringence and dichroism under steady and oscillatory shear. It is possible to correlate macroscopic mechanical responses with induced microscopic changes in the material. We describe how this method was improved several fold and implemented on a commercially available setup. However, the here presented ideas are applicable to any rheo-optical setup, based on modulation of the laser light. Additionally it does not need a lock-in amplifier and therefore reduces the costs of the setup.

Zusammenfassung:
Die Rheo-optik erlaubt eine Analyse der optischen Eigenschaften Doppelbrechung und Dichroismus unter dem Einfluss konstanter oder dynamischer Scherfelder. Es ist daher möglich die makroskopische mechanische Antwort mit den durch die Scherung induzierten mikroskopischen Veränderungen im Material zu korrelieren. In diesem Artikel wird diese Methode an einem kommerziell verfügbaren Apparat vielfach bezüglich der Zeitauflösung und der Sensitivität verbessert. Die hier vorgestellten Ideen können prinzipiell in jeder rheo-optischen Apparatur implementiert werden, die auf der Modulation von Laser-Licht beruht. Zusätzlich benötigt der experimentelle Aufbau keinen Lock-In Verstärker, dies reduziert darüber hinaus die Kosten.

Résumé:
La Rhéo-optique est une méthode qui permet l'analyse des propriétés optiques, telles que la biréfringence et le dichroïsme sous cisaillement stationnaire et oscillatoire. Il est possible de corréler des réponses mécaniques macroscopiques avec des changements microscopiques induits dans le matériau. Nous décrivons comment cette méthode a été améliorée à plusieurs reprises et mise en application sur un appareillage disponible dans le commerce. Cependant, les idées présentées ici sont applicables à n'importe quel appareillage rhéo-optique basé sur la modulation de la lumière émise par un laser. Par ailleurs le système ne nécessite pas d´amplificateur Lock-in ce qui par conséquent réduit les coûts de l´installation.

Key words: rheo-optics, birefringence, oversampling, fast Fourier transformation.

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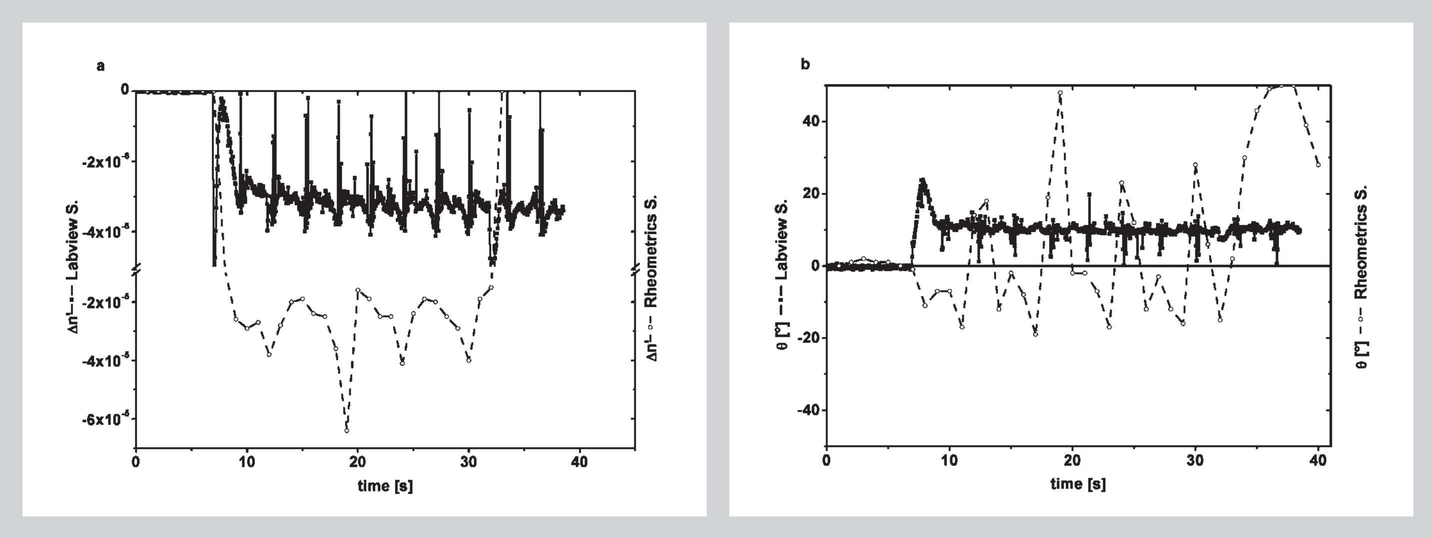

Figure 5: Comparing birefringence data as measured with the commercial Rheometrics Software (lines with empty circles) and using the home-built Labview software (lines with filled squares) see Figure 5a. The sample is PS in DOP at 20°C with shear rate of 20 s^{-1}. The background compensation (see text) was used for all the measurements irrespective of home written or commercial software. The home written Labview software acquires more data per time and the values of the birefringence correspond well to earlier measurements [18] in contrary to the commercial software. It is furthermore possible to detect the overshoot during the onset of shear. Comparison of the orientation angle θ, data measured with the commercial Rheometrics software (lines with open circles) and with the home written Labview program (lines with filled squares) see Figure 5b. The Labview software acquires more data per time and the values of the orientation angle have the correct sign. It is further possible to detect the overshoot during the onset of shear.

3 RESULTS

In the following experiments the background birefringence was minimized using a variable retarder. In Figure 5 the measured birefringence and orientation angles acquired by the home written Labview software are compared with the original Rheometrics software. To compare two different hardware setups, measurements were performed on the same PS solutions. Experiments were performed in steady shear with start-up and cessation of flow. The applied shear rate was 20 s^{-1}. The time resolution of the Labview data is significantly improved and the sign of the orientation angle is correctly determined [18]. The birefringence values as computed by the Rheometrics Software are at least 10 times too large, while the orientation angle seems to fail to give the proper values. Using the modified setup an early overshoot during the start up of the shear is detected in the optical signal (see Figure 5a, b), whereas this overshoot is not visible using the commercial software. The periodically appearing peaks in the Labview data in Figure 5a, b are caused by an air inclusion present in the sample and crossing the light beam at each rotation of the cup. The stress optical coefficient $C = 4 \cdot 10^{-9}$ m^2/N as derived from the Labview data is in close agreement with the value found earlier, $C = 5 \cdot 10^{-9}$ m^2/N, by Hilliou [18].

4 CONCLUSIONS

Our self-developed Labview software enables us to simultaneously acquire optical and mechanical data with substantial higher resolution in time and in detectable birefringence respective dichroism. The software is using an on-the-fly FFT and oversampling to determine the oscillation frequency of the spinner motor. Due to the modified design this setup works without a lock-in amplifier, therefore increasing simplicity and reducing cost. Principally at the modulation frequency we work (1600 Hz) a lock-in amplifier is not better in the accuracy than our setup. If a different setup with a higher modulation frequency is used, it is important to check if the ADC-card is fast enough to monitor that specific frequency according to the Nyquist theorem. The rheo-optical birefringence signal is disturbed by the residual birefringence, that originates from e.g. misalignments in the optical train, non-ideal optical elements, and temperature induced strains. Using a variable retarder to compensate the residual birefringence it is possible to minimize the influence of the residual birefringence. It was also possible to increase the resolution in the time domain by oversampling. Furthermore it could be shown that a vectorial representation of the residual birefringence signal is helpful to understand the influence of the residual birefringence on the measurement of the birefringence signal. Analyzing the signals of a rotating birefringent object then allowed to verify this effect, and birefringence down to $\Delta n'_{min} = 10^{-8}$ could be detected using a variable retarder. A further reduction of the noise can be achieved by an insulation of the optical train to stabilize against thermal fluctuations. The experimental improvements were validated on two different setups measuring equivalent results. It is emphasized that this method is not only useful for the commercial Optical Analysis Module II setups provided from TA instruments (formerly Rheometric Scientific), but also in any other rheo-optical setup based on similar modulation techniques.

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