Optical Coherence Tomography Velocimetry with Complex Fluids

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Abstract. We present recent results obtained with an Optical Coherence Tomography Velocimetry technique. An optical interferometer measures the velocity of a sheared fluid at specific depths of the sample using the coherence length of the light source. The technique allows the dynamics of 3 pico liter volumes to be probed inside opaque complex fluids. In a study of opaque starch suspensions, classical bulk rheology experiments show non-linear shear thickening, whereas observations of the velocity profiles as a function of distance across the gap show Newtonian behavior. The ability of the technique to measure velocity fluctuations is also discussed for the case of polyacrylamide samples which were observed to display shear banding behavior. A relationship between the viscoelasticity of the sample and the size of the apparent fluctuations is observed.

1. Introduction
When attempting to understand complex fluid behavior rheometers are only capable of telling us information about the bulk of the fluid, when in reality a range of phenomena can be occurring on a hierarchy of different length scales. More detailed information is available if microrheology measurements are performed on a specimen [1]. A particularly useful microrheology measurement is to calculate the velocity profiles of a sample under flow (a variety of non-linear microrheology). Particle imaging velocimetry (PIV) is the most commonly used technique for measuring velocity profiles, but this technique has its limitations, mainly in terms of the thickness and concentration of the samples that can be probed [2]. Optical Coherence Tomography (OCT) Velocimetry is a relatively new technique and has been previously demonstrated to be a powerful tool for measuring complex flows and rheological behavior in a wide range of materials [3]. OCT is a new type of photonics technique that adds to the growing list of available rheo-optical methods [4].

2. Overview of OCT Velocimetry
OCT is a technique that was originally developed for imaging in ophthalmology, since it allows the retina to be imaged relatively independently of the shape of the cornea. OCT has seen a wide range of applications and developments [5, 6]. In OCT Velocimetry a Doppler signal is measured using an interferometer for light scattered off a sheared fluid. High resolution measurement with this technique is achieved by using a light source with a small coherence length, which means
that the back scattered interference signal can be associated with a thin layer of the sample at a specific depth [3]. The infra-red light source currently used has a coherence length and therefore an axial resolution of 9 \( \mu \text{m} \), which factoring in the spot size of the focused beam, gives a probe volume of 3.4 pico liters. The Doppler frequency is measured in the form of a peak in the Power Spectrum of the light intensity recorded on a fibre coupled detector, and this in turn can be converted into a velocity, given the wavelength and angle of incidence of the light source. OCT has a penetration depth of the order of a few millimeters in opaque materials (in the case of our OCT Velocimetry system the depth of focus is 1.2 mm, so that the entire rheometer gap is in focus and no refocusing is required during an experiment), which is greater than in most conventional microscopy techniques, and is facilitated by the use of an infra-red super luminescent diode light source, with a wavelength of 1.3 \( \mu \text{m} \).

Figure 1 gives a list of velocimetry methods that can be used to probe the velocity of complex fluids in a rheometer. OCT Velocimetry is competitive in terms of temporal resolution, spatial resolution and price. Our current realization of the technique requires samples to have relatively low flow birefringence. However this restriction can be lifted if a polarization sensitive detector is used [7].

**Figure 1.** Comparison of different commonly used velocimetry techniques to probe velocity profiles in rheometers. Adapted from [2]

| Technique                      | Spatial resolution | Temporal resolution | System requirements | Advantages                                      | Disadvantages                                      |
|--------------------------------|--------------------|---------------------|---------------------|------------------------------------------------|---------------------------------------------------|
| **OCT shear rheometer**        | 9 \( \mu \text{m} \) slices, 3 \( \mu \text{L} \) volume | 4 \( \mu \text{s-1000 s} \) dynamics, \( \sim 1 \text{s per point} \) | Source of speckle | High spatial and temporal resolution, Opaque materials | Would require polarization sensitivity to probe liquid crystals¹ |
| **Standard photon correlation spectroscopy (PCS) velocimetry** | 100 \( \mu \text{m} \) slices | 1 s per point | Weak scattering, No turbidity | Reasonable spatial and temporal resolution | No opaque materials |
| **Ultrasonic velocimetry (UT)** | 40 \( \mu \text{m} \) slices | 20 ms-2 s per point | Acoustic contrast | Reasonable spatial and temporal resolution, Opaque materials | Can require seeding |
| **NMR**                        | 50-100 \( \mu \text{m} \) slices | 1 s per profile | Correct nuclei | Opaque materials | Costly, Low resolution |
| **Particle tracking velocimetry** | 1-10 \( \mu \text{m} \) slices | 2.5 ms-0.1 s per profile | Tracer particles and optically transparent | Excellent spatial and temporal resolution | Requires seeding and transparent systems. Small depth of field. No opaque materials |

¹ Polarization sensitive OCT techniques with static samples have been demonstrated in the literature, and polarization sensitive detectors are commercially available [7].
3. Velocimetry of starch suspensions

Starch is a well studied colloidal material due to its importance as a food stuff and also as a raw material (e.g. paper production). It is an example of a non-Newtonian complex fluid due to its shear thickening properties [8]. In the case of starch suspensions, shear thickening is caused by jamming processes, and it is therefore reasonable to expect that non-linear velocity profiles across the rheometer gap might be observed for suitably high shear rates and concentrations [9]. Therefore aqueous corn starch suspensions of between 40 and 50 percent by weight were examined with a bulk rheometer and were found to display shear thickening behavior in viscosity measurements as shown in Figure 2.

![Figure 2](image)

**Figure 2.** Viscosity plotted as a function of shear rate measured in a bulk rheometer for a range of concentrations of corn starch for a rheometer gap of 300 µm, showing shear thickening behavior at high shear rates (>10 s⁻¹) for concentrations above 45% w/w.

Figure 3 shows velocity profiles as a function of position in the rheometer gap at three different concentrations of corn starch, 40% w/w which showed no shear thickening, and 45% w/w and 50% w/w which showed significant shear thickening [10, 11].

\[
\dot{\gamma} = \frac{\sigma}{\eta} = \frac{dv}{dz}
\]

(1)

Surprisingly, all three profiles in Figure 3 show linear Newtonian profiles, with no apparent slipping on either plate, conforming to Equation 1, where the shear rate of the fluid, \( \dot{\gamma} \), is equal to the velocity gradient across the rheometer gap, \( dv/dz \), and is related to the bulk viscosity of the sample, \( \eta \), and stress applied to it, \( \sigma \). A comparison between the velocity profiles for different shear rates for the 50% w/w suspension, which showed the greatest amount of shear thickening, is shown in Figure 4. All of the velocity profiles are again linear.

4. Study of velocity fluctuations

Another useful aspect of the OCT Velocimetry method is its ability to quantify fluctuations in the flow velocity. This should prove to be useful in studying the onset of turbulence, since characteristic velocity fluctuations have been observed previously [12]. The velocity of the fluid can be calculated by fitting to a peak in the Power Spectrum, with a central frequency position...
Figure 3. Velocity profile as a function of depth for opaque 40% w/w, 45% w/w and 50% w/w suspensions of corn starch with a parallel plate geometry and steady shear rate of 80 s$^{-1}$. Linear Newtonian behaviour is observed in all cases, Equation 1.

Figure 4. Velocity profiles as a function of depth for an opaque 50% w/w corn starch sample at a range of shear rates, showing Newtonian behaviour in all cases as described by Equation 1.

$\omega$, which is then used to calculate the velocity at which complex fluids move within the 3 pico liter probe volume using Equation 2,

$$v = \frac{\lambda \omega}{2 \sin \theta}$$

where $\lambda$ is the wavelength of the light source and $\theta$ is the angle at which the light is incident upon the sample. This peak is approximately Gaussian with a finite width ($\delta \omega$) which will be greater for volumes of fluid where the velocity is not constant. Also as the velocity of fluid within the probe sample varies over time, the position of the peak will also fluctuate over time. The size of velocity fluctuations can thus vary at specific depths within the sample ($\delta v$). An understanding of the velocity fluctuations can give greater insight into turbulent or unstable regions of flow. An example of different Power Spectra with varying peak widths is shown in Figure 5.

The OCT Velocimetry method has been previously used in a study of polyacrylamide samples, which displayed a range of flow phenomena with dynamic phase transitions that depended on the molecular weight and the concentration of the polyacrylamide, Figure 6 [13]. For the lowest molecular weight samples that were analyzed (1.5k and 10k MW), the velocity profiles across the gap were found to be always linear, regardless of the concentration of polymer in the sample. However for medium molecular weights (5M MW), shear banding occurred as the concentration of polymer was increased, and for high concentrations wall slip became more apparent. Similar results were obtained for higher molecular weights (18M MW), however the onset of shear banding and wall slip was observed for much lower concentrations. This shear banding behavior may be associated with the entanglement of the polymer chains and is also referred to as elastic turbulence in the literature [14, 15, 16].

By analyzing the degree of broadening of the peaks in the data that produced the velocity profiles, we can gain further insight into the role of polymer concentration in the determination of the flow behaviour. Figure 7 shows the reduced velocity fluctuations across the rheometer gap for a range of different concentrations of low molecular weight polyacrylamides and Figure 8 shows similar data for high molecular weight samples.
Figure 5. Three different Power Spectral Densities measured using OCT for different shear velocities of probe spheres embedded in a polyacrylamide solution [13]. The lines in bold show Gaussian fits. The fitted curves give shear velocities of $1.06 \pm 0.20 \text{ mms}^{-1}$, $1.86 \pm 0.32 \text{ mms}^{-1}$ and $2.73 \pm 0.77 \text{ mms}^{-1}$ for the blue, red and green curves respectively.

Figure 6. Examples of velocity profiles as a function of distance across the plate-plate geometry ($z/z_0$) for different concentrations and molecular weights of Polyacrylamide samples displaying Newtonian (red), Shear Banding (black) and Wall Slip behavior (blue and pink).

Figure 7. Velocity fluctuations ($\delta v/v$) as a function of distance across the plate-plate geometry ($z/z_0$) for low molecular weight (10k MW) polyacrylamide solutions at varying concentrations.

The reduced velocity fluctuations are plotted in order to compensate for the relative scaling of the fluctuations at higher shear velocities. In both cases the flow of the system is apparently unstable close to both of the plates of the rheometer, however we are currently unsure of the origin of this instability. It could be due to irregularity in the control loop of the rheometer (although the instability is also near the stationary plate), broadening due to the roughness/asymmetry of the plates (although the lower plate is highly polished glass), an artifact resulting from the limited ability of the instrument to measure low shear velocities or a real intrinsic hydrodynamic effect. Further away from the surface of the plates, the
change in fluctuation size for different concentrations is small and most noticeable when plotted logarithmically. For the low molecular weight samples, the velocity fluctuations decrease for higher concentrations of polymer. This makes logical sense as the viscosity of the sample increases with concentration, and this effectively dampens any instability more than a less viscous sample. The opposite however seems to be the case for high molecular weights as the fluctuations increase with concentration, Figure 8. The velocity fluctuations in this case may be driven by the non-linear elastic instability that drives the non-Newtonian velocity profiles, Figure 6[17]. The elasticity of the samples at higher concentrations (they are reptating and above the entanglement concentration) may help to drive the instability.

Figure 8. Velocity fluctuations as a function of distance across the plate-plate geometry \( (z/z_g) \) for high molecular weight (18M MW) polyacrylamide solutions at varying concentrations. The arrow indicates the increase in the velocity fluctuations that we associate with elastic turbulence and the onset of wall slip.

5. Future directions
There are a wide range of potential samples that can be investigated using OCT Velocimetry that are known to display complex flow behavior including; live bacteria, blood cells, food stuffs such as biopolymers and milk products, petroleum based products, surfactants and colloidal suspensions. The OCT Velocimetry method should in principal function with other types of rheometer such as concentric cylinders (a Couette cell), channel flow and also polymer melt rheometers, which will allow a wider range of materials to be studied. We are in the process of developing a new OCT Velocimetry design using an Electro Optical Modulator that we expect will provide improved resolution on the velocity fluctuations.

6. Conclusions
OCT Velocimetry allows quantitative analysis of complex flow systems both in terms of velocity profiles and velocity fluctuations. This has been demonstrated in a study of opaque starch suspensions which have been shown to display Newtonian behavior across the plate/plate geometry despite displaying shear thickening in conventional bulk rheometry measurements. Also polyacrylamide samples are examined in which the velocity fluctuations are a sensitive function of polymer concentration. However the trends observed for the velocity fluctuations differ depending on the molecular weights of the polymer chains in question. For the shear banding (elastic turbulence) specimens, velocity fluctuations increase with polymer concentration, whereas the opposite is true for specimens with Newtonian profiles.
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This technique is patented [18], but for further information, including detailed descriptions on how to build a non-commercial OCT Velocimeter, please go to http://www.personalpages.manchester.ac.uk/alex.malm or contact Alex Malm directly: alex.malm@manchester.ac.uk

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