A Sliding Plate Microrheometer for Monitoring Structure Evolution in Self-Assembling Peptide Solutions and Other Complex Fluids

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INTRODUCTION

The conformational and dynamical behaviour of viscoelastic polymer melts and solutions close to solid boundaries is of great interest for the polymer processing industry. In particular, when the gap approaches microscopic scales, boundary effects such as wall-slip [1], cohesive [2] and adhesive failure [3] occur on the same scale as the overall deformation of the bulk sample and their impact on the measured rheological properties can no longer be neglected. These effects are especially important when structural elements (such as those encountered in self-assembling biopolymer solutions, microgels and emulsions) dominate the viscoelastic properties of the fluid system or when the characteristic microscopic length scales such as the inter-chain separation or the mesh spacing of a gel approach the characteristic dimension of the probe volume.

Previous microrheometer designs such as the Surface Force Apparatus (SFA) [4], imbedded probe particles [5] or AFM techniques [6] focus mainly on the studies of thin films in the nanometer range. On the other hand many industrial processing operations as well as the emerging field of microfluidics lead to flows on an intermediate or 'meso-scale' range that cannot be readily probed with either bulk rheometry or nanoscale measurements of apparent viscosity or surface friction. With the exception of the work of Granick and co-workers [7] there are few established experimental techniques that are capable of measuring on the meso to micro-scales.

The aim of this paper is therefore to introduce a new design of a Flexure-based Microgap Rheometer (FMR) that allows the determination of the viscometric properties of small fluid samples (<10μl) in adjustable gaps that cover meso-scale dimensions of 200 μm down to micro-scale dimensions of 1 μm.

FLEXURE-BASED MICROGAP RHEOMETER (FMR)

The Flexure-based Microgap Rheometer (FMR) is a miniaturised sliding plate rheometer and generates a Plane Couette shearing flow between two optical flats (polished flat to within λ/20, or 30nm and coated with a semi-reflective layer of 100nm TiO_2). Two separate three-point nano-positioning stages using piezo-stepping motors are used to control the orientations of the upper and lower surfaces. A compound flexure system [8] is used to hold the test fixtures and fluid sample between a drive spring and an independent sensor spring as shown in Figure 1. The compound flexure system allows for a purely linear relative translation of the surfaces over distances of several millimetres with an orthogonal displacement of less than 1 nm.

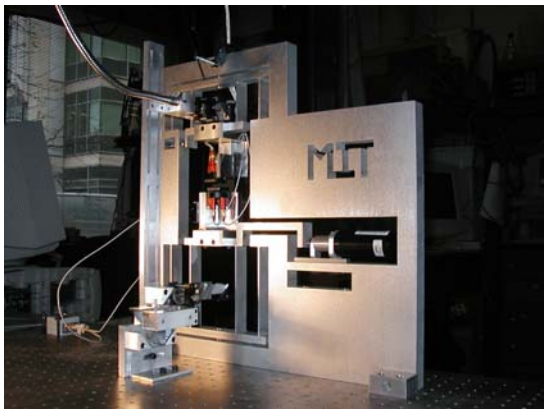

Figure 1. Flexure-based Microgap Rheometer (FMR)

Alignment fidelity, device orthogonality and total error stack-up are all optimized by machining the entire instrument frame from a single monolithic aluminum block using CAD design plus water-jet and EDM technology. The dynamic force range of the instrument can be varied greatly by changing the length and/or thickness of the compound flexures. Displacements in the sensing flexure are detected using an inductive proximity sensor with a resolution of ±3 nm allowing the detection of maximum loads of up to 6 N with an accuracy of 3 mN. Calibrations give a standard deviation from linearity of the sensor spring of less than 2%. The lower plate is attached to a drive flexure which is driven by an 'inchworm' motor with a maximum displacement of 6 mm and a resolution of 0.1 nm.

WHITE LIGHT INTERFEROMETRY

The parallelism of the shearing surfaces with respect to the shearing direction as well as the absolute gap separation are both controlled using white light interferometry [9, 10]. Only integer wavelengths are passed through the semi-reflective cavity of the gap. Orthogonal 1D optical crosscuts of the shearing surfaces are dispersed by a spectrometer as shown in Figure 2. Image processing of the fringe pattern allows the determination of the tilt angle of the surfaces and simultaneous adjustment to parallelism with software-controlled nanopositioning stages (tilt angle resolution < 0.003º). The gap is calculated from the wavelength of the fringes and can

be adjusted with a single nanopositioning stage with a step resolution of < 30nm.

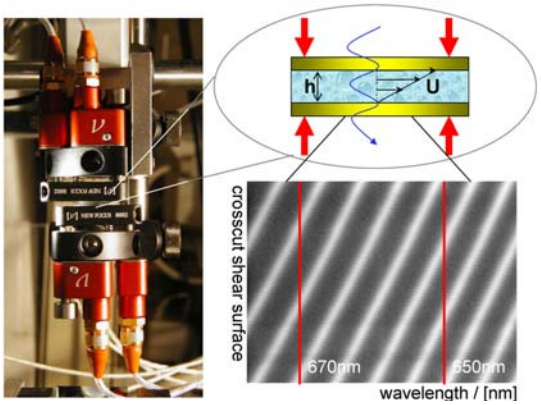

Figure 2. White light interferometry: the fringe pattern of a dispersed crosscut over the shearing surfaces

The convenient optical access perpendicular and parallel to the shearing surfaces enables the simultaneous detection of dichroism, birefringence and optical microscopy during the imposed shearing deformation.

RESULTS

Comparison of shear experiments for different gaps over a range of $2 \leq h \leq 170$ μm with two constant viscosity fluids are shown in Figure 3. In the present configuration, the FMR instrument is capable of determining shear stress over a range of 2×10^2 – 1×10^5 Pa (determined by the stiffness of the flexure spring). The results show very close agreement with the values determined using a conventional torsional rheometer. At the lower resolution limit of the flexure-based sensor there is a progressive deviation from linearity. In a non-dustfree environment the minimum gap size is limited to $h \sim 4$ μm. Below this critical separation, trapped dust particles cause a nearly constant frictional stress when the shear rate and therefore the stress drop below a critical value.

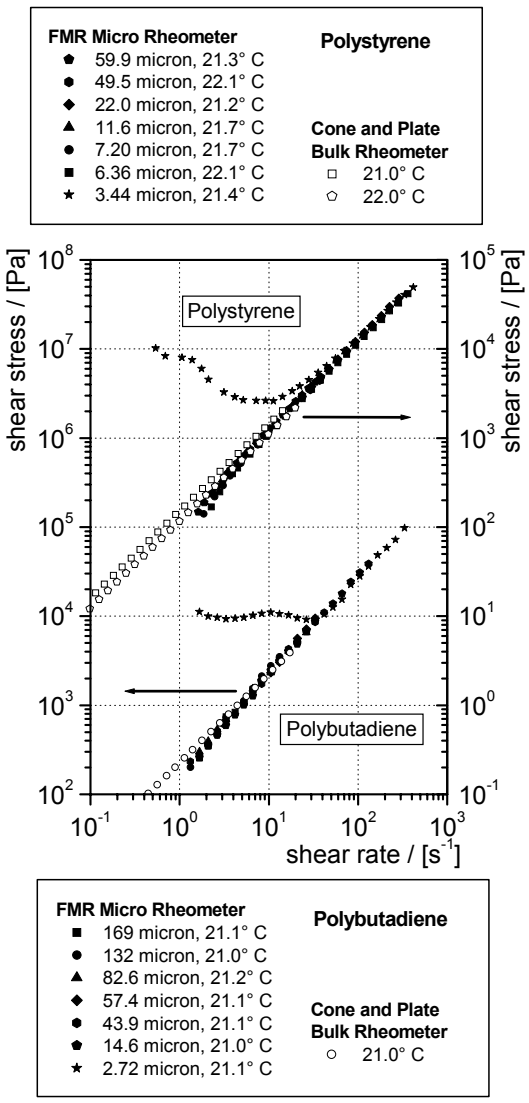

Figure 3. Comparison of the shear stress as a function of the shear rate measured with the FMR micro-rheometer for different gap separations and with a conventional cone & plate rheometer for a polybutadiene melt ($M_w = 1600$ g/mol) and a polystyrene Boger fluid ($M_w = 2 \times 10^6$ g/mol, c = 0.05wt%)

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