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Vibrating wire rheometry

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A B S T R A C T

We describe the design and operation of a novel vibrating-wire rheometer. Our device consists of a tungsten wire under tension and immersed in a fluid in a magnetic field. When an alternating current is passed through the wire it vibrates at the driving frequency, and we measure the voltage induced across the wire as a function of frequency. The resonant frequency of the wire is of order 1000 Hz, and can be tuned by varying its length and the applied tension. We modify an analytic expression for the induced voltage, previously derived for Newtonian fluids, to include a complex viscosity, and determine the viscous and elastic modulus of complex fluids by fitting this expression to our data. Our device gives excellent results for the viscosity of Newtonian fluids and the viscoelastic moduli of aqueous polymer solutions, at frequencies higher than those accessible using a conventional shear rheometer. Because the amplitude of the wire's vibrations is on the order of a few microns, it can be used to probe the microrheology of fluids that are heterogeneous on that scale. We illustrate this by measuring the micron-scale moduli of a viscoplastic suspension of Laponite clay as it gels.

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1. Introduction

The viscoelastic properties of complex fluids are commonly measured using rotational shear rheometers. These instruments are typically limited to frequencies \leqslant 100 Hz due to the inertia of the rheometer tool [\[1,2\].](#page--1-0) Knowledge of the rheological properties of complex fluids at higher frequencies is important for understanding their dynamical properties, microstructure, and interactions between the components of the fluid. For example, the highfrequency behavior of the viscoelastic moduli contains information about inter-particle forces in a concentrated colloidal suspension [\[3\].](#page--1-0) Very fast relaxation processes in polymer solutions can only be measured using high-frequency methods [\[4\].](#page--1-0) In some cases, the high-frequency properties can be inferred by employing timetemperature superposition or time-concentration superposition [\[5\],](#page--1-0) but there are many fluids for which these techniques cannot be applied due to effects such as evaporation or freezing of the solvent or temperature-induced phase transitions. There is thus a need for new experimental methods that will enable the exploration of the high-frequency dynamics of complex fluids.

Devices based on the vibrations of a wire in a fluid have frequently been used to measure viscosity and density. A brief review is provided in Ref. [\[6\].](#page--1-0) Viscosity damps the vibrations. It can be determined either in the time domain, by measurements of

<http://dx.doi.org/10.1016/j.jnnfm.2016.06.007> 0377-0257/© 2016 Elsevier B.V. All rights reserved. the damping time, or in the frequency domain, through measurements of the wire's resonance curve [\[6\].](#page--1-0) In 1850, Stokes analyzed the problem of an infinite cylinder oscillating perpendicular to its axis in an infinite fluid [\[7\].](#page--1-0) In 1964, Tough et al. [\[8\]](#page--1-0) constructed a vibrating-wire viscometer which they used to measure the viscosity of liquid helium. Retsina et al. [\[9\]](#page--1-0) solved the Navier–Stokes equations for a finite cylindrical rod vibrating in an infinite Newtonian fluid. They developed a set of working equations that relate the motion of the wire to the fluid properties and discussed the effects of the physical dimensions of the device on the accuracy of the viscosity measurements. Kandil and Marsh [\[10\]](#page--1-0) constructed a vibrating-wire device to measure the viscosity of a range of Newtonian fluids. Forced oscillations were produced by passing an alternating current through a wire under tension in a constant magnetic field. They expanded upon the theory of Retsina et al. [\[9\]](#page--1-0) to derive an expression for the voltage induced in the wire due to its motion in the magnetic field and its dependence on fluid properties. Many other devices of this type have been reported, for use in applications ranging from the petroleum industry to bedside measurements of blood viscosity and under a broad range of environmental conditions [\[6\].](#page--1-0)

Vibrating objects have been used for rheometrical measurements in non-Newtonian fluids in the past. A patent for a vibrating-wire rheometer was issued in 1996 [\[11\],](#page--1-0) but we are unaware of any published description of this instrument, nor of any results obtained using it. Fritz et al. [\[1\]](#page--1-0) explored the high frequency dynamics of several complex fluids using torsional

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resonators. They used cylindrical and double-dumbbell-shaped torsional resonators to perform measurements at discrete frequencies on the order of 10 kHz. Using a simple model of mechanical resonators and comparing measurements in the fluid to reference measurements in air, the properties of several complex fluids were determined. Although Fritz et al. successfully measured viscous and elastic moduli using this method, it requires calibration of the device, operates at a single discrete frequency, and the geometry is too complex for a complete theoretical description. Wang et al. recently introduced a method for performing measurements over a range of frequencies around the resonance peak of a torsional oscillator [\[12\]](#page--1-0) and successfully applied it to determine the viscoelastic moduli of polymer solutions at frequencies from 0 to 400 Hz [\[13\].](#page--1-0)

Measurements at higher frequencies require smaller devices with higher resonant frequencies. Lemaire et al. have investigated the use of vibrating microstructures to measure density and complex viscosity [\[14\].](#page--1-0) They fabricated two devices, a microcantilever and a U-shaped vibrating wire, which operated at frequencies in the range from 10^3 to 10^5 Hz. By estimating the hydrodynamic force on the structures and taking the fluid viscosity to be complex (see Eq. (8)), they were able to measure the viscous modulus *G*^{$\prime\prime$} to within 20% of the accepted value. Using nanofabrication techniques, Riesch et al. fabricated a viscometer consisting of a suspended plate 100 \times 100 μ m in size with a resonance frequency of 16 kHz [\[15\].](#page--1-0) While this device was successfully used to measure viscosity, its use was not extended to complex fluids.

In this paper we describe a vibrating-wire rheometer for the measurement of viscoelastic moduli in low- to moderate-viscosity fluids at frequencies around 1 kHz. The construction of the device, experimental setup, and data analysis method are discussed. Measurements in Newtonian fluids and a homogeneous viscoelastic polymer solution are presented to demonstrate the efficacy and accuracy of the device. We also show how the device can be used to probe the time-dependent changes in microstructure of an aging suspension of Laponite clay [\[16,17\].](#page--1-0)

2. Theory

2.1. Newtonian fluids

Theoretical descriptions of the motion of a wire vibrating in a Newtonian fluid have been presented in [\[7–10\].](#page--1-0) This earlier work was extended by Kandil [\[18\]](#page--1-0) to consider the voltage induced in a current-carrying wire vibrating in a viscous Newtonian fluid in a magnetic field as a function of fluid parameters and vibration frequency. The amplitude of the wire's oscillation, and therefore the induced voltage, is a maximum at a resonance frequency. The induced voltage is given by Kandil [\[18\]](#page--1-0)

$$
V = a + bf + ci + dfi + \frac{\Lambda ft}{f_0^2 - (1+\beta)f^2 + (\beta' + 2\Delta_0)f^2i},
$$
 (1)

where the first four terms are due to the electrical impedance of the stationary wire and any background voltage; *a, b, c*, and *d* are adjustable parameters determined by curve fitting. The last term is the motional electromotive force and is a function of the fluid properties. *i* is the imaginary number $\sqrt{-1}$, Λ is the voltage amplitude, f_0 is the resonance frequency of the vibrating wire in vacuum, Δ_0 is the self-damping of the wire, β is the added mass arising from fluid displacement, and β' is the viscous damping term.

The density and viscosity of the fluid are contained within β and β' . Retsina et al. [\[9\]](#page--1-0) studied the fluid mechanics of a vibrating wire of density ρ_w in a fluid of density ρ and determined β and β' to be given by

$$
\beta = k \frac{\rho}{\rho_w},\tag{2}
$$

 $\beta' = k' \frac{\rho}{\rho_w}$, $\hspace{2.6cm} (3)$

respectively, where *k* and *k* are

$$
k = -1 + 2\mathfrak{A}(\mathbf{A}),\tag{4}
$$

$$
k' = 2\Re(\mathbf{A}).\tag{5}
$$

Here \Im and \Re represent the imaginary and real components, respectively. **A** is a complex quantity given by

$$
\mathbf{A} = \left(1 + \frac{2K_1(\sqrt{\Omega i})}{\sqrt{\Omega i}K_0(\sqrt{\Omega i})}\right) i,\tag{6}
$$

where

$$
\Omega = \frac{2\pi f \rho R^2}{\eta}.\tag{7}
$$

 K_0 and K_1 are the modified Bessel functions of zeroth and first order, and Ω is a dimensionless quantity related to the Reynolds number which characterizes the flow around a cylindrical wire of radius *R* in a fluid with viscosity η . η is determined by fitting the experimentally-measured voltage across the vibrating wire to Eq. (1).

2.2. Viscoelastic fluids

To extend the above theory to viscoelastic fluids, we replace the Newtonian viscosity η in Eq. (7) by a complex viscosity η^* [\[5\]:](#page--1-0)

$$
\eta \to \eta^* = \eta' - i\eta'' = \frac{G' + iG''}{i\omega},\tag{8}
$$

where G' and G'' are the elastic and viscous moduli respectively and $\omega = 2\pi f$ is the angular frequency. This substitution is exact in the limit of linear response $[5]$. In general G' and G'' will be functions of frequency, although we will assume them to be constant over the limited frequency range of our experiments, as discussed below. In our experiments, described below, we measure the induced voltage $V(f)$ and determine G' and G'' by fitting our data to Eq. (1) with this substitution.

3. Experiment

3.1. Vibrating-wire rheometer

We constructed several vibrating-wire devices designed to have different resonant frequencies; they differ only in the active length of the wire. One is shown in the left panel of [Fig.](#page--1-0) 1 and drawn schematically in the right panel. The operation of the device is similar to that described by Kandil $[10,18]$. A tungsten wire is held under tension by two stainless steel clamps. The steel clamps are mounted on a borosilicate glass backbone. This wire holder is placed inside a vial containing the sample fluid, and the vial is immersed in a temperature controlled housing as shown in the center panel of [Fig.](#page--1-0) 1. To ensure wall effects are negligible, the ratio of the radius of the fluid container R_c to the radius of the wire R_c must be large. Retsina et al. determined that a ratio $R_c/R = 33$ contributes an error of less than \pm 0.05% to the measured viscosity [\[9\].](#page--1-0) In our case the distance to the glass backbone yields a ratio of 67, while the fluid vial yields a ratio greater than this. Two permanent magnets are mounted on the sides of the housing to produce a magnetic field. An alternating current of frequency *f* and amplitude ∼1 mA is passed through the wire, and the resulting Lorentz force causes the wire to vibrate at the driving frequency.

Tungsten wire of diameter 0.15 mm (Goodfellow, Huntingdon, UK) was chosen for the vibrating wire because of its relatively

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