

## A novel design of ultrasonic spinning rheometry

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We introduced grand design of ultrasonic spinning rheometry that utilizes velocity profile measurement by UVP in rotating cylinder flows of complex fluids including multiphase media to overcome problems on common spinning rheometry. Multiple schemes dealing with physical relations through physical values reflecting rheological properties allow evaluation of multiple rheological properties by single measurement. Rheometry along the design was demonstrated on bubble suspension, and effective Newtonian viscosity near the cylinder wall, where the bubbles are largely elongated, and linear viscoelasticity on little inner region were evaluated. Additionally, a novel scheme utilizing Fourier transform for higher frequency spinning tests were proposed.

**Keywords:** Rheometry, Effective viscosity, Viscoelasticity, Rotating flow, Spatio-temporal velocity data

### 1. Introduction

Accurate and rapid evaluation of rheological properties of fluids has great importance in scientific, engineering and industrial fields for multiple purposes. For example, in manufacturing processes dealing with fluid materials, correct estimation of the properties allows accurate control and improvement of the process. Conventional, rotational rheometers that assume ideal Couette flow in a narrow gap between concentric cylinders, a cone and a plate, or concentric disks have satisfied the demands by robustness on the results. But recent broadening demands have exceeded areas covered by the conventional rheometers and have required novel rheometry. One of typical examples is rheometry for multiphase media. If the size of the dispersed phase of a multiphase medium is considerably large in comparison with the test gap of conventional rheometers, assumption of ideal Couette flow is not realized, and thus the measurement results would be unstable. Existence of shear banding and local yield area cause similar problems on the evaluation. To overcome this problem, simultaneous measurement of velocity profiles of fluids filled in wider gaps was performed [1]. Velocity profile measurement also makes possible to evaluate local shear dependent viscosity of flowing material in a pipe [2]. The literatures show that velocity profile measurements have great potential on rheometry for complex fluids.

Our group has developed a novel rheometry utilizing velocity profile measurement by ultrasonic velocity profiling (UVP) and functional oscillation of a cylinder as a vessel of the test media, this is termed ultrasonic spinning rheometry (USR). With USR we performed; (a) rapid estimation of shear dependent viscosity of yoghurt [3]; (b) evaluation of effective viscosity of dispersed bubbly liquid in unsteady shear flows [4]; model-free rheometry for evaluation of rheological properties of unknown media and also for avoiding errors due to miss-selection of rheological model [5, 6]. Attempts to evaluate time-dependent rheological properties of thixotropic fluids are also ongoing [7]. In the individual projects of application of USR introduced above, suitable information of flows is

extracted from spatio-temporal velocity distributions measured using UVP to evaluate different rheological properties. In this study, we would like to propose assembled form of USR to evaluate its applicability and discuss rising problems to be solved for further evolution of the methodology.

### 2. Ultrasonic spinning rheometry

#### 2.1 Configuration of equipment

Measurement configuration, equipment and definition of coordinates for USR through the experiments are explained. Figure 1 shows an illustration of the test cylinder made of acrylic resin with 145 mm in diameter filled with test fluids. The cylinder motion is controlled by a stepping motor and thus functional oscillatory rotations can be realized. The measurement line of UVP is located parallel to the center line of the cylinder with off-center displacement  $\Delta y = 15$  mm; this value is determined empirically to consider both incidence of the ultrasonic wave into the test fluid and reduction of amplification of the measurement error due to inverse projection of velocity vectors. An UVP monitor model Duo (Met-Flow S.A.) was adopted with alternative use of 2 MHz or 4 MHz ultrasonic transducers depending on the attenuation of ultrasonic wave in test media.

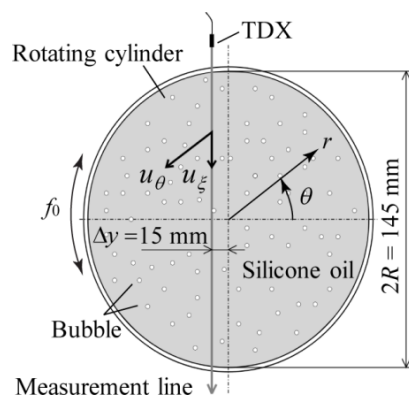


Figure 1: Illustration of a test cylinder, measurement configuration and coordinate system adopted for ultrasonic spinning rheometry [4]

With periodic oscillations of the cylinder, flows inside the cylinder can be assumed as axisymmetric. And conditions with frequency of 0.5 Hz to 2.0 Hz and amplitude 90 degrees, it was confirmed that flows of a viscous silicone oil agree with ideal one-directional flow driven by cylinder rotation in comparison between exact solution with assumption of the one-directional flow and spatio-temporal velocity distributions measured UVP [4]. The velocity component along the measurement line,  $u_\xi$ , can therefore be assumed as projection of the azimuthal velocity  $u_\theta$  and thus  $u_\theta$  is simply calculated from  $u_\xi$  at each radial position with  $\Delta y$  (hereafter we remove  $\theta$  from  $u_\theta$  for simplicity). For measurements, quasi-neutrally buoyant particles with  $O(10 \mu\text{m})$  were dispersed into the test fluid as tracer particles.

## 2.2 Assembled design of USR

Figure 2 shows block diagram of design of USR by assembling previous attempts for various test fluids, where italic words represent rheological properties evaluated by USR from analysis on spatio-temporal velocity distributions  $u(r, t)$  via different process. Words in the boxes indicate physical values or physical relations reflecting the rheological properties. The parameters that can be set are oscillation speed  $U$  and frequency  $\omega$  for sinusoidal oscillation cases, and type of functions for the cylinder motion, steady rotation, rapid start and sudden stop are chosen depending on the purpose. The individual schemes to evaluate the rheological properties are summarized below.

**Shear dependent viscosity and Flow curve:** Radial velocity profile of fluid having shear-dependent viscosity against steady rotation of the cylinder takes a shape depending on the viscosity. Fitting the velocity profile estimated from “suitable” rheological model, for example power law,

$$\tau = k\dot{\gamma}^n = k\left(\frac{\partial u}{\partial r} - \frac{u}{r}\right)^n \quad (1)$$

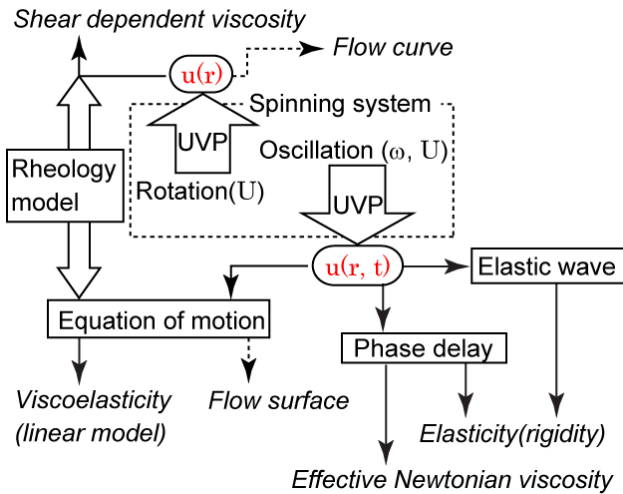


Figure 2: block diagram of assembled design of ultrasonic spinning rheometry: italic words represent rheological properties that can be evaluated and words in boxes indicate physical values or physical relations to extract the properties utilizing spatio-temporal velocity data,  $u(r, t)$

where  $k$  and  $n$  represent the properties, then we can evaluate the viscosity. This scheme was already established well [1] and we do not mention details of this.

Combination with torque measurement allows having general relation between shear stress and strain rate, so-called flow curve. This does not require rheological model and achieves model-free rheometry [5].

**Effective Newtonian viscosity:** Assuming Newtonian viscosity on test fluids, propagation of the cylinder oscillation into the fluid has to be described by phase delay  $\phi(r)$  of the azimuthal velocity against the cylinder wall in the radial direction. With comparing the phase delay obtained from  $u(r, t)$  with that of ideal exact solution for representative viscosity [4], we can estimate the viscosity of the test fluid. This scheme is applicable to liquid with dispersion phase because of relatively large measurement volume of UVP, and thus ‘effective’ viscosity in the measurement volume is obtained [4].

**Elasticity (rigidity):** Two different schemes were proposed for elasticity analysis. One is from propagation of elastic wave dominated by equation of waves,

$$\frac{\partial^2 \Theta}{\partial t^2} = c^2 \frac{\partial^2 \Theta}{\partial r^2}, \quad c = \sqrt{\frac{G}{\rho}}, \quad (2)$$

where  $\Theta(r, t)$  represent azimuthal oscillation of media and  $c$  speed of elastic wave. Sudden start of the rotation, for example, induces the elastic wave in viscoelastic bodies and calculating  $c$  from  $u(r, t)$  as propagation speed of waves tells us modulus of rigidity  $G$ . Another scheme assumes situation where the quasi-viscous body connects continuously viscoelastic body. At positions nearby the boundary, phase lag of the velocity fluctuation due to the cylinder oscillation  $\phi(r)$  is affected by elasticity and is decreased from pure viscous body with  $\delta$  as

$$\phi(r + \Delta r) = \phi(r) + \left. \frac{d\phi}{dr}(\mu) \right|_r \Delta r - \left. \frac{d\delta}{dr}(E, \mu) \right|_r \Delta r \quad (3)$$

where  $r + \Delta r$  is neighboring measurement volume for  $r$ . The 2nd term represents increase of the phase lag due to the original viscous effect. Assuming linear viscoelasticity,  $\delta$  is estimated as influence of elasticity relative to the viscosity. Then it is represented with storage modulus  $G'$  and loss modulus  $G''$  by

$$\frac{\pi}{2} - \delta = \tan^{-1} \frac{G''}{G'} = \tan^{-1} \frac{E}{\omega \mu} \quad (\text{for Maxwell model}) \quad (4)$$

Here we adopted Maxwell model for linear viscoelasticity. According to Eq. (4), elasticity  $E$  is obtained from  $\delta$  and viscosity  $\mu$ .

**Viscoelasticity and flow surface:** If the strain on the fluid motion is small enough, we can evaluate linear viscoelasticity as rheological properties of viscoelastic bodies. Assuming axisymmetric one-directional flow in the cylinder, equation of motion of fluids is reduced into

$$\rho \frac{\partial u}{\partial t} = \frac{\partial \tau}{\partial r} + \frac{2\tau}{r}. \quad (5)$$

Measured  $u(r, t)$  has to satisfy this equation. And

adopting Maxwell model to represent ‘linear’ viscoelasticity,

$$\tau + \frac{\mu}{E} \frac{\partial \tau}{\partial t} = \mu \left( \frac{\partial u}{\partial r} - \frac{u}{r} \right), \quad (6)$$

viscosity and elasticity can be evaluated as values minimizing cost function

$$\begin{aligned} \min_{\tau, \mu, E} \int_r \int_t \left[ \tau + \frac{\mu}{E} \frac{\partial \tau}{\partial t} - \mu \left( \frac{\partial u}{\partial r} - \frac{u}{r} \right) \right] dt dr, \\ \text{s.t.: } \rho \frac{\partial u}{\partial t} - \frac{\partial \tau}{\partial r} - \frac{2\tau}{r} = 0. \end{aligned} \quad (7)$$

In difference form, it is described as

$$\begin{aligned} \min_{\tau, \mu, E} \sum_i \sum_j \left[ \tau_{i,j} + \frac{\mu}{E} \frac{\tau_{i+1,j} - \tau_{i,j}}{\Delta t} - \mu \left( \frac{\partial u}{\partial r} \Big|_{i,j} - \frac{u_{i,j}}{r_j} \right) \right]^2, \\ \text{s.t.: } \rho \frac{\partial u}{\partial t} \Big|_{i,j} - \frac{\tau_{i,j+1} - \tau_{i,j}}{\Delta r} - \frac{2\tau_{i,j}}{r_j} = 0. \end{aligned} \quad (8)$$

Similar to the flow curve explained above, relation of stress, strain, and strain rate can represent general viscoelastic properties without adopting rheological model. This representation is not restricted in linear regime, and is termed ‘flow surface’ by our group as a novel model free rheometry [6]. Obtaining this relation requires additional torque measurement simultaneously with velocity profile measurement by UVP.

### 3. Demonstrations and results of USR

The USR is demonstrated on evaluation of rheology of bubbly liquid. Evaluation of the effective viscosity on bubbly liquid has long history and some evaluation schemes were proposed for steady [8] and unsteady shear flows [9]. But the scheme still has imperfection for general situations.

Figure 3 shows pictures of bubble deformations in the oscillating cylinder with difference conditions, (a) steady, oscillation with (b) 0.5 Hz and 2.0 Hz in frequency, where the amplitude of oscillation is 90 deg. for the both cases and volume fraction of bubbles is  $\alpha \sim 2\%$ . The bubbles are largely deformed near the cylinder in oscillation conditions, and seem to keep original spherical shape at the inner part. It is suggested that the surface

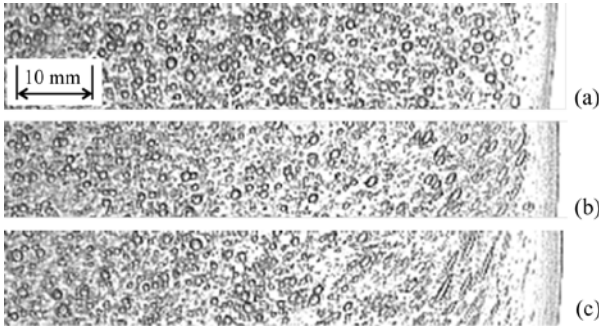


Figure 3: Pictures of bubble deformations in the oscillating cylinder; (a) stationary, (b) 0.5 Hz and (c) 2.0 of the cylinder oscillation

tension of bubbles that is origin of elasticity on bubbly liquid is already lost in the former case and still effective in the latter case. So rheology in the former case would be able to be evaluated as effective Newtonian viscosity and one in the latter as linear viscoelasticity.

#### 3.1 Effective Newtonian viscosity

Effective Newtonian viscosity analysis is thus adopted in near wall region of the cylinder. As mentioned in Sec. 2.2, effective Newtonian viscosity is estimated from phase lag of the velocity fluctuations against the cylinder wall, where the phase is calculated from Fourier analysis of the velocity fluctuation at each radial position as phase of the most dominant frequency component. Figure 4 shows profiles of the phase lag measured from 1000 cSt silicone oil without/with bubbles, where the profile estimated from analytical solution for 1000 mm<sup>2</sup>/s in the kinematic viscosity is displayed for comparison [4]. The variation for the pure oil seems to follow that of the analytical solution. On the other hand, the variation in bubble suspension has displacement from the analytical solution and it becomes wider toward the cylinder center. We evaluated gradient of the phase delay  $\Delta\phi/\Delta r$  at each radial position to estimate local effective viscosity and averaged them in a range of 0.1R to reduce the noise. Comparing the  $\Delta\phi/\Delta r$  of bubble suspension with that of analytical solution with various value of the kinematic viscosity, the local effective viscosity  $\nu^*$  is determined as a viscosity providing most similar value of  $\Delta\phi/\Delta r$ .

Table 1 summarizes results of the estimation of effective Newtonian viscosity  $\mu^*$  for different frequency condition and at  $r/R = 0.95$  and 0.85, where the viscosity is in style of relative viscosity against the original viscosity of silicone oil  $\mu$ ,

$$\eta = \frac{\mu^*}{\mu} = (1 - \alpha) \frac{\nu^*}{\nu}. \quad (9)$$

The relative viscosity larger than unity means increase of viscosity by bubble dispersion and vice versa. Near the cylinder wall, the viscosity increases for 0.5 Hz and decreases for larger frequencies. As shown in Fig. 3(c), bubbles near the wall for 2.0 Hz are largely elongated and have very small effort on the momentum propagation. This is reason why the viscosity decreases, and value of

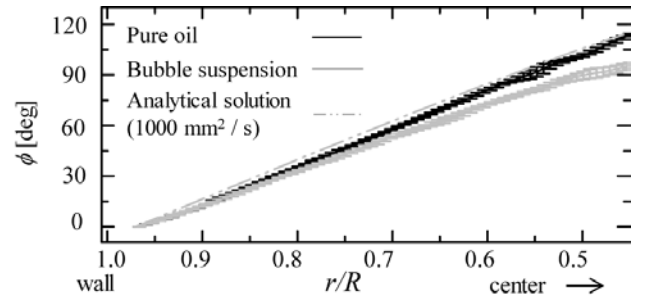


Figure 4: Phase lag of the velocity fluctuation against the cylinder oscillation for pure oil, bubble suspension and estimated value from analytical solution for 1000 mm<sup>2</sup>/s in kinematic viscosity [4]

the decrease, around 17 %, is much larger than estimation from the previous schemes [9]. In the inner block at around  $r/R = 0.85$ , the viscosity increases with 10 %. This is also considerably larger than the evaluation according to the previous scheme [9].

Table 1: Estimated relative effective viscosity for different frequency condition and at  $r/R = 0.95$  and  $0.85$  [4]

Frequency [Hz]	$\eta$ at $r/R = 0.95$	$\eta$ at $r/R = 0.85$
0.5	1.109	1.169
1	0.979	1.154
2.0	0.831	1.062

### 3.2 Linear viscoelasticity analysis

We performed linear viscoelastic analysis according to the scheme detailed in Sec. 2.2. As a preprocessing, we performed POD (proper orthogonal decomposition) filtering on the original spatio-temporal velocity distribution (Fig. 5(a)) to reduce measurement errors that are largely enhanced by process of differences in calculation of Eq. (8). Figure 5(b) shows the results constructed from 1<sup>st</sup> and 2<sup>nd</sup> POD modes and the distribution seems to smooth enough. Evaluated value of viscosity and elasticity for 1.0 Hz at  $r/R = 0.7$  are  $\mu = 1.05$  Pa·s and  $E = 69.40$  Pa. Corresponding absolute value of complex viscosity,

$$|\mu^*| = \frac{\mu}{\sqrt{1 - (\omega\mu/E)^2}} \quad (10)$$

is 1.045 Pa·s.

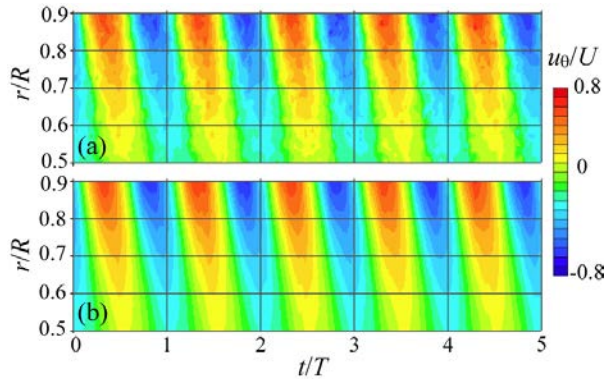


Figure 5: Spatio-temporal distribution of azimuthal velocity component for 1.0 Hz in the oscillation frequency; (a) raw data and (b) filtered data by POD, where 1<sup>st</sup> and 2<sup>nd</sup> modes are used

### 4. Ongoing attempts

Each scheme in the assembled design has not been fully optimized yet and has area to be improved. Here we introduce ongoing attempts to reduce calculation noise on linear viscoelastic analysis.

For high frequency testing on USR, sampling frequency of UVP would become insufficient to resolve fluid motion. And it may enhance measurement error in the process of differences of spatio-temporal velocity distribution for viscoelastic analysis. To overcome this

problem, we propose frequency domain analysis using Fourier transform. Applying Fourier transform converts Eqs. (5) and (6) into

$$\hat{\tau} + i\omega \frac{\mu}{E} \hat{\tau} = \mu \left( \frac{\partial \hat{u}}{\partial r} - \frac{\hat{u}}{r} \right), \quad (11)$$

$$\hat{\tau}(r, \omega) = \mathcal{F}[\tau(r, t)], \hat{u}(r, \omega) = \mathcal{F}[u(r, t)].$$

$$i\omega \rho \hat{u} = \left( \frac{\partial}{\partial r} + \frac{2}{r} \right) \hat{\tau}. \quad (12)$$

The problem is converted into minimizing cost function,

$$G(r; E, \mu) = \int_0^\Omega \left[ i\omega \rho \hat{u} - \left( \frac{\partial}{\partial r} + \frac{2}{r} \right) \hat{\tau} \right]^2 d\omega, \quad (13)$$

where

$$\hat{\tau}(r, \omega) = \mu \left( \frac{\partial \hat{u}}{\partial r} - \frac{\hat{u}}{r} \right) \left( 1 - i\omega \frac{\mu}{E} \right) \left[ 1 + \left( \omega \frac{\mu}{E} \right)^2 \right]^{-1}. \quad (14)$$

Applicability of this scheme has not evaluated yet, but the equations tell us it can avoid temporal differences in the calculation.

### 6. Summary

We proposed assembled desing of ultrasonic spinning rheometry for wider use of the methodology on general complex fluidss. According to the design we can evaluate same rheological properties by different schemes and to increase robustness of the estimated value. The design is still in progress and also each scheme still requires improvement. The present methodology has great potential and demonstration on analysis of bubble suspension was performed.

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