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# Oscillatory and steady shear viscosity: The Cox–Merz rule, superposition, and application to EHL friction

Scott Bair<sup>a,\*</sup>, Tsuyoshi Yamaguchi<sup>b</sup>, Ludwig Brouwer<sup>c</sup>, Hubert Schwarze<sup>c</sup>,  
Philippe Vergne<sup>d</sup>, Gerhard Poll<sup>e</sup>

<sup>a</sup> Georgia Institute of Technology, Center for High-Pressure Rheology, George W. Woodruff School of Mechanical Engineering, Atlanta, GA 30332-0405, USA

<sup>b</sup> Department of Molecular Design and Engineering, Graduate School of Engineering, Nagoya University, Furo-cho B2-3(611), Chikusa, Nagoya, Aichi 464-8603, Japan

<sup>c</sup> Technische Universität Clausthal, Institut für Tribologie und Energiewandlungsmaschinen, Leibnizstraße 32, 38678 Clausthal-Zellerfeld, Germany

<sup>d</sup> Université de Lyon, INSA-Lyon, CNRS, LaMCoS UMR5259, F-69621 Villeurbanne, France

<sup>e</sup> Leibniz Universität Hannover, Institut fuer Maschinenkonstruktion und Tribologie, Postfach 6009, D-30060 Hannover, Germany

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## ABSTRACT

The new quantitative approach to elastohydrodynamic lubrication requires a description of the steady shear dependent viscosity for calculations of film thickness and friction. This property can be obtained from measurements in pressurized thin-film Couette viscometers. However, frequency dependent viscosity can be obtained from a torsionally vibrating quartz crystal viscometer at high pressure or a relatively simple ambient pressure measurement with a shear impedance spectrometer. Here it is shown for squalane and for a cyclic hydrocarbon and for a diester that both the steady shear dependent viscosity and the frequency dependent viscosity obey time–temperature–pressure superposition with the simplest shifting rule over the range of conditions investigated. Flow curves shift along a constant steady stress path or a constant complex modulus path. The Cox–Merz rule has been confirmed only for squalane and then only near the transition. The EHL friction for squalane at low pressure may be predicted with fair accuracy from the frequency dependent viscosity measured at ambient pressure. It appears that the Cox–Merz rule only applies to low-molecular-weight liquids when the molecule is composed of a long chain.

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## 1. Introduction

The prediction of film thickness and friction in the elastohydrodynamic regime of lubrication (EHL) requires the knowledge of the steady shear dependence of the viscosity. The approach to modeling of the temperature, pressure and shear rate dependence of viscosity,  $\eta(T, p, \dot{\gamma})$ , in EHL has recently undergone a revolutionary change. Traditionally, viscosity has been employed as an adjustable parameter to reconcile experimental measurements of film thickness [1] and friction [2] with simple, but inaccurate, assumptions regarding  $\eta(T, p, \dot{\gamma})$ . The use of viscometers had become controversial as these contradicted the adjusted viscosity [3]. Often, different assumptions were required for film thickness and as for friction. With the successful prediction of both the film

thickness and the friction from a single  $\eta(T, p, \dot{\gamma})$  relation using the viscosity measured in viscometers [4,5], the field has moved quickly toward the more realistic approach.

The measurement of the limiting low shear viscosity as a function of temperature and pressure  $\mu(T, p)$  has always been relatively easy. The capability of reaching EHL pressures in a viscometer has existed for almost ninety years [6]. The limiting low shear viscosity,  $\mu$ , is the shear-dependent viscosity,  $\eta$ , in the limit of zero shear rate,  $\dot{\gamma}$ . (Outside of EHL, the subscript “0” is usually applied to  $\eta$  to indicate the low shear value; however, in EHL this subscript has been reserved for the zero pressure value.) In practice, it is only necessary to measure the viscosity at low shear stress,  $\tau < 100$  Pa, to assure terminal (Newtonian) response in lubricants and such low stress can be applied by falling bodies. Using gravity to apply the shear stress assures that the stress is repeatable and removes the complication of mechanically driving an experiment through a thick-walled pressure chamber.

The measurement of the steady shear dependence of the viscosity has been more challenging. To be useful, measurements must substantially exceed the Newtonian limit which requires that  $\gamma\lambda_s \gg 1$ , where the time “constant”,  $\lambda$ , employed in the empirical

\* Corresponding author.

E-mail addresses: [scott.bair@me.gatech.edu](mailto:scott.bair@me.gatech.edu) (S. Bair),

[tyama@nuce.nagoya-u.ac.jp](mailto:tyama@nuce.nagoya-u.ac.jp) (T. Yamaguchi),

[brouwer@itr.tu-clausthal.de](mailto:brouwer@itr.tu-clausthal.de) (L. Brouwer),

[schwarze@itr.tu-clausthal.de](mailto:schwarze@itr.tu-clausthal.de) (H. Schwarze),

[philippe.vergne@insa-lyon.fr](mailto:philippe.vergne@insa-lyon.fr) (P. Vergne), [poll@imkt.uni-hannover.de](mailto:poll@imkt.uni-hannover.de) (G. Poll).

**Nomenclature**

$f$	cyclic frequency, Hz
$K$	Dyson parameter
$k$	geometric parameter, kg <sup>2</sup> /m <sup>4</sup>
$M$	molecular mass, kg/kmol
$n$	power-law exponent
$p$	pressure, Pa
$p_H$	Hertz (maximum) pressure, Pa
$R$	universal gas constant, 8314 J/kmol K
$t$	time, s
$T$	temperature, K
$\gamma$	shear rate, s <sup>-1</sup>

$\Gamma$	Newtonian limit, Pa
$\eta$	rate-dependent steady shear viscosity, Pa s
$\eta'$	oscillatory shear viscosity in-phase with shear rate, Pa s
$\eta''$	oscillatory shear viscosity in-phase with shear strain, Pa s
$\eta^*$	modulus of complex shear viscosity, Pa s
$\lambda$	time “constant”, s
$\mu$	limiting low-shear viscosity, Pa s
$\rho$	mass density, kg/m <sup>3</sup>
$\tau$	shear stress, Pa
$\omega$	angular frequency = $2\pi f$ , rad/s

viscosity functions is found to be approximately proportional to  $\mu$ . The proportionality follows from time–temperature–pressure superposition. Since  $\gamma\lambda_s \propto \gamma\mu$ , the shear stress is critical to shear-thinning regardless of the temperature and pressure. Then the measurement of steady shear-thinning in lubricants requires that a high shear stress be applied to the liquid and that the liquid respond without sufficient temperature change to influence the viscosity [7] and that liquid failure such as shear cavitation [8] or shear localization [9] or wall slip not occur. All of these requirements may be met by high pressures. To mitigate thermal softening the most simple strategy is to increase the viscosity so that the shear stress,  $\gamma\eta$ , can be large while the viscous power,  $\gamma^2\eta$ , is kept reasonably small. Viscosity grows rapidly with pressure. The tensile principal normal stress which causes cavitation [8] cannot appear at high pressures and if  $\gamma\eta/p <$  about 0.12 the shear deformation will not localize [9]. For these reasons, high pressure is essential to the observation of steady shear-thinning in low-molecular-weight liquids.

The capillary viscometer, which is often employed to characterize the shear-dependent viscosity of polymers, has a fundamental limitation to the shear stress which may be generated by the pressure gradient in the flow direction. As the stress is increased, a singularity in the flow rate equation appears for a piezoviscous liquid and the flow will be choked [10]. The pressurized, thin-film Couette viscometer [11] has become the instrument of choice for investigations of steady shear-thinning in low-molecular-weight liquids.

Shear-frequency dependence also appears for the viscosity,  $\eta^*(\omega)$ , measured in small-strain oscillatory shear, known as dynamic measurements of viscosity [12]. This  $\eta^*$  is the amplitude of the shear stress divided by the amplitude of the shear rate. Shear-frequency dependence, in the case of oscillatory shear, presents when the angular frequency,  $\omega$ , exceeds a critical value which depends on temperature and pressure. Analogous to the steady shear response, shear relaxation begins for  $\omega\lambda_d > 1$ , where  $\lambda_d$  is a relaxation time.

Measurements of the shear-frequency dependence of viscosity in oscillatory shear have certain advantages over steady shear experiments because of the small strains. Here, the shear relaxation is independent of the shear rate amplitude,  $\gamma_0$ . First, notice that the time-averaged viscous power, which is less than  $\gamma_0^2\eta^*/2$ , can be made small by utilizing small  $\gamma_0$  without affect for the shear-frequency dependence. Thus, thermal softening will not influence the result. Second, the maximum shear stress,  $\gamma_0\eta^*$ , can be made to be much less than the pressure by utilizing small  $\gamma_0$  without affect for the shear-dependence. Thus, shear cavitation and shear localization may be avoided even at atmospheric pressure. Given the obvious advantages of these dynamic viscosity measurements, it may be asked whether they can contribute

information regarding the steady shear dependence that is required for EHL calculations.

The linear viscoelasticity of oils was invoked many years ago to explain the initial, linear portion of a traction curve [13]. However, the value of the shear modulus was considerably lower than that obtained from primary measurements [14]. Comparing the traction slope with and without a liquid film shows that the elastic creep of the rollers is responsible for the initial slope at high pressures [15].

Another application of oscillatory shear to viscometry is the torsionally vibrating crystal viscometer [16]. This technique is readily applied at high pressures. An alternating voltage applied to a suspended quartz crystal produces shear waves in the surrounding liquid. The viscous damping is determined from the electrical impedance of the crystal. This method is being investigated for precise measurements of the low-shear viscosity under pressure [17].

Promise for the application of frequency-dependent shear-thinning to problems in EHL comes from the analogy between steady and oscillatory shear known as the Cox–Merz rule [18]. This article examines the relationships between viscosities measured in steady and oscillatory shear for three organic liquids with similar dynamic properties to lubricants. These are squalane, DCMP and DEHP; 2,6,10,15,19,23-hexamethyltetracosane, 2,4-dicyclohexyl-2-methylpentane and di(2-ethylhexyl)phthalate, respectively. These liquids have significance to EHL since squalane is a reference liquid for EHL with well-characterized properties (see for example [19,20]), DCMP is the base oil for the traction fluids, Santotrac, and DEHP is a diester often used in EHL experiments and has been proposed as a high pressure viscosity standard by Harris [19]. The aim is to provide an alternative to high pressure viscometry for EHL property characterization and to provide insight into the molecular scale mechanism of shear-thinning in low-molecular-mass liquids.

## 2. The Cox–Merz rule

Suppose that a liquid is sheared between parallel plates so that the shear rate varies with time as  $\gamma = \gamma_0 \cos(\omega t)$  and the measured shear stress is given by  $\tau = \eta'(\omega)\gamma_0 \cos(\omega t) + \eta''(\omega)\gamma_0 \sin(\omega t)$ . The modulus of the complex viscosity is  $\eta^*(\omega) = \sqrt{\eta'^2 + \eta''^2}$ .

An analogy between steady and oscillatory shear has been experimentally observed in polymer melts and solutions [12,18].

$$\eta(\gamma) = \eta^*(\omega), \quad \gamma = \omega \quad (1)$$

Other similar analogies between steady and transient shear response exist. For example, the viscosity may be measured as a function of time following the start of shearing of a quiescent

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