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Flow measurements of a polyphenyl ether oil in an elastohydrodynamic contact

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Abstract
A novel methodology, based on the use of phosphorescence imaging, is applied to determine the local through-thickness velocity profile of lubricant in an elastohydrodynamic contact. The technique has spatial and temporal resolutions of 40 μm and 340 μs respectively and thus allows lubricant rheology to be investigated at conditions close to service conditions. The capability of the newly-developed method is verified by examining the flow of 5P4E polyphenyl ether, a lubricant base fluid used in very high temperature applications and is well-known for its high viscosity-pressure coefficient. Experimental results highlight the effect of the contact pressure on the velocity profile of this fluid in lubricated contacts. At low pressures, the velocity profile of 5P4E is close to linear, characteristic of Couette flow. As the local pressure increases, its velocity profile progressively deviates from a Couette profile and shear banding is evident at high pressure.

Keywords: lubricant flow, shear banding, novel in situ measurement, elastohydrodynamic lubrication

(Some figures may appear in colour only in the online journal)
application of common velocimetry techniques, such as particle imaging velocimetry (PIV), unsuitable. For microscopic flows, molecular tagging velocimetry techniques (MTV) have proved useful in various applications, including the development of chemical and biological devices based on the investigation of the flow in cylindrical microcapillaries [12] and the development of micronozzles for aerospace applications [13]. When a molecule in a flow is tagged in this setting, it can be identified within the rest of the fluid in the flow. The molecules constituting the flow can be tagged using a laser beam by photobleaching, phosphorescence or fluorescence [12, 14, 15]. The subsequent flow of tagged molecules can then be related to the flow of the fluid. In an EHD contact, MTV has been implemented to measure the average speed of lubricant flow [16]. Very recently, the authors have developed MTV based on fluorescence photobleaching and successfully obtained local flow profiles of lubricants in EHD contacts [17]. The effects of pressure [18] and surface chemistry [19] on flow profiles have been investigated with a spatial x- and y-resolution of 30 μm and 3 μm respectively and a temporal resolution of 30 ms.

The technique used in this work is a new molecular tagging velocimetry technique that supersedes the resolutions of the method previously developed in [17]. It allows investigation of the rheology of model, low viscosity lubricants at conditions much closer to those present in real machine components. Briefly, the new method, (molecular) Phosphorescence Imaging Velocimetry (mPIV), tags a small volume of fluid within an EHD contact using phosphorescence. The temporal evolution of the shape of the tagged volume is recorded. Then a numerical reconstruction scheme is used to iteratively deduce the unknown, through-thickness velocity profile.

The aim of this work is to demonstrate the capability of mPIV in obtaining through-thickness flow profiles in an EHD contact at conditions and resolutions unattainable by other methods. The model lubricant used in this study is 5-phenyl-4-ether (5P4E). This synthetic fluid is employed in some aerospace applications due to its very high thermal and oxidative stability and has been widely used in EHD experimental studies [20–25] because of its very strongly pressure-dependent viscosity. The flow curve for 5P4E, obtained with a pressurized Couette viscometer [26], shows a Newtonian regime at low shear rates with shear-thinning occurs at higher shear rates, followed by a constant shear stress regime that is characteristic of shear-banding. The present study applies mPIV to investigate the flow of 5P4E in an EHD contact. Complex flow phenomena are observed and shear-banding of 5P4E is evident in its through-thickness velocity profiles at high pressure conditions.

Experimental

Materials

The lubricant used in this study is commercial 5P4E (GBR Technology). The lubricant is doped with a phosphorescent dye, tris(dibenzoylmethane) mono(1,10-phenanthroline) europium(III) (Sigma-Aldrich) (referred to as Eu dye in this work), at a concentration of 1.8 mM. The complete dissolution of the dye in 5P4E is achieved by magnetic stirring for at least 2 h at 60 °C. At ambient conditions, 5P4E has a dynamic viscosity μ = 2.10 Pa·s and pressure-viscosity coefficient α = 45.9 GPa−1 [27].

An EHD contact is created with a 19 mm borosilicate glass ball (PCS Instruments) loading against a glass slide (VWR). The arithmetic mean roughnesses (Rz) of ball and slide surfaces have been measured by a Talysurf profilimeter (Form Talysurf Series 2) and an optical profilimeter (Veeco, Wyko NT9100) and are 16 nm and 0.9 nm respectively. The applied normal load is varied from 1 to 17 N, leading to a maximum Hertzian pressure Pmax between 140 and 360 MPa. A soft-EHD contact with low peak pressure Pmax = 232 kPa was also obtained by loading a glass ball against a glass slide coated with a 1 mm layer of polydimethylsiloxane (PDMS) (Sylgard 184, Dow Corning). The glass ball rotates at a fixed peripheral speed of Uθ = 144 mm s−1, while the slide remains stationary Ud = 0. This gives an entrainment speed Ue = (Uθ + Ud)/2 of 72 mm s−1. The lubricant is contained on the slide by a PDMS well to ensure fully flooded conditions (see figure 1). All experiments were carried out at room temperature.

The lubrication regimes for these operating conditions are identified in Esfahanian and Hamrock’s map [28] as shown in figure 2. Most conditions applied in this work lie in the piezoviscous-elastic (EHD) region, where extreme pressures and deformations are present. The exception is Pmax = 232 kPa, it is in isoviscous-elastic (soft EHD) regime and has been used to validate the developed technique in a soft-EHD contact. Assuming isothermal condition, the pressure induced effects

Figure 1. Schematic of the phosphorescence imaging setup.
in the soft EHD contact are expected to be negligible, such that a Couette velocity profile is expected.

Phosphorescence imaging velocimetry

In this work, through-thickness velocity profiles are determined using phosphorescence imaging velocimetry (mPIV). Previously the authors have published photobleached fluorescence imaging velocimetry (PFIV) [17] to obtain through-thickness velocity profile of polybutene (PB) in EHD contacts by tagging the fluorescently-doped PB using photobleaching. The main limitation of PFIV is temporal resolution. In PFIV, the tagged volume is observed as a low intensity volume against a bright background. Thus the requirement of a high relative photobleaching rate and the inherent low signal-to-noise ratio mean that only conditions of low entrainment speeds can be investigated. This in turn limits the choice of model lubricant to highly viscous lubricants and precludes the study of lubricant normally used in engineering practice. In the present study, a laser pulse is used to tag the phosphorescently-doped fluid; as such the tagged volume is bright due to phosphorescence emission. The velocity profile is then inferred by following the evolution of the shape of this tagged volume against a dark background. As only a short laser pulse is needed to create the tagged volume, flow of low viscosity lubricants at higher speeds, as detailed in this study, can now be investigated.

The use of phosphorescence emission for velocimetry requires further discussion. Fluorescence and phosphorescence are both examples of photoluminescence. In both cases, light of short wavelength is absorbed and then re-emitted at a longer (less-energetic) wavelength. The relaxation of unstable excited electrons to the ground state can take multiple pathways. Emission of a photon from a singlet excited state to a singlet ground state, or between any two energy levels with the same spin, is called fluorescence. This process usually occurs on a timescale of nanoseconds to microseconds. In a phosphorescent process, excitation of electrons to a higher state is accompanied with the change of a spin state. Relaxation is then a slower process since it involves energy state transitions. Emission lifetimes in cases of phosphorescent processes are in the range of microseconds to seconds. Both fluorescence and phosphorescence will be emitted by an excited molecule, with the former having much higher intensity than the latter. If phosphorescence emission is of interest, care must be taken to remove signal from fluorescence emission. In this study, this is done by incorporating a time delay between tagged volume creation and image acquisition. This time delay is longer than the fluorescence lifetime of the dye.

A schematic of the optical setup is presented in figure 1. A 349 nm laser (Spectra Physics Explorer) operating at 1000 Hz is used to create a phosphorescent column of lubricant within the contact. The laser beam is expanded onto the back of an objective (20 × 0.5 NA). The full width at half maximum (FWHM) of the laser beam on the focal plane of the objective is deduced from the image of a focused phosphorescent spot created in a thin film of doped lubricant and is about 10 μm. A tagged volume is generated by exposing the phosphorescently-doped lubricant to a short laser pulse (FWHM <5 ns). The tagged volume can be considered as a through-thickness cylinder with uniform cross-sectional area and intensity. The uniformity of the cross-section of the tagged column through the film thickness is verified in the next section (see figure 5).

The phosphorescence emission from the tagged column is then collected through the objective and passes through a 488 nm long pass filter to remove the residual signal from the laser. Images of the shape evolution of the tagged column against time are recorded in the x–y plane using an intensified CCD camera (Hamamatsu Orca-R2).

A first image is recorded 2 μs after the laser pulse excitation. The 2 μs delay was determined to be sufficient to ensure that only phosphorescence emission from the tagged volume will be collected. Then a sequence of 17 images is recorded with an exposure time of 5 μs. The exposure time is sufficiently small compared to the speed at which the phosphorescent volume is travelling to avoid blurring of images (the distance that the tagged column travels during a frame is actually smaller than a pixel). There is a delay of 20 μs between consecutive images. Hence the observation time for each image sequence is 342 μs. Image sequences obtained directly from the experiment without any correction are referred as raw sequences.

Flow profile determination

Raw sequences contain phosphorescence signals from the Eu dye in the doped lubricant and also, potentially, from background phosphorescence of rubbing surfaces and 5P4E itself, \( I_b, I_p \) is captured by obtaining sequences of 20 images with non-doped 5P4E in identical conditions. It is about 5% of the total signal collected and has a lifetime of about 14 μs. The background phosphorescence sequence (figure 3(b)) is then subtracted from the raw sequence (figure 3(a)) to give the background corrected sequence (see figure 3(c)). As \( I_b \) is low, the difference between raw sequences and background corrected sequences is small (see figures 3(a) and (c)).

![Figure 2](image-url)
The phosphorescence lifetime of a dye is affected by experimental conditions, such as its solvent, pressure and temperature. The lifetime of Eu dye in 5P4E in an EHD contact has been inferred from the integration of the temporal intensity distribution at room temperature. It decreases from about 110 μs to 10 μs for peak pressure $P_{\text{max}}$ from 151 MPa to 313 MPa.

Note that the phosphorescence lifetime of the glass sphere and glass slide is much shorter than the phosphorescence lifetime of the Eu dye in 5P4E at low pressure.

Each background-corrected image of a sequence is normalised by its total intensity to eliminate the temporal decrease of phosphorescence. This gives the normalised sequence and is used to obtain through-thickness velocity profiles $u(z/h)$. An iterative reconstruction scheme similar to one detailed in [17] is used to determine $u(z/h)$ from the spatiotemporal intensity distribution in the $x$-direction, averaged in the $y$-direction over five pixels. The reconstruction is complete when the simulated spatiotemporal intensity distribution from the reconstructed $u(z/h)$ matches the experimental spatiotemporal intensity distribution.

The determination of through-thickness flow profile assumes that a through thickness tagged volume is created and is cylindrical (while the intensity distribution is Gaussian). Hence, assuming negligible side flow at the centre of the contact, the width of the stretched tagged volume (in the $y$-direction) should remain constant irrespective of $x$ position. In order to check the uniformity of the cross-section of the tagged volumes within the film thickness created in this study, intensity profiles along the $y$-direction have been determined from the normalised background corrected image recorded 302 μs after the laser pulse at different positions (A), (B) and (C) along the $x$-direction under Couette flow conditions (figure 4(a)). This corresponds to the lowest pressure case in this study. Figure 5 shows that the geometry (both width and shape) of the intensity distribution of the tagged volume along the $y$-direction does not vary with $x$-position. Hence the cross-section of the initial tagged column of fluid is uniform within the film thickness (i.e. in the $z$-direction). In addition, $U_b$ and $U_d$ determined from the flow profile (figure 4(c)) based on figure 4(a) match...
the actual applied speeds (i.e. \( u/U_b = 1 \) and 0 at \( z/h = 1 \) and 0 respectively), confirming that a through-thickness cylinder is created between the glass slide and the glass ball even under the conditions that give the thickest lubricant film investigated in this work.

The spatial resolution of this technique in the \( x \)-direction is governed by the size of the initial phosphorescence spot as the tagged column has to travel at least a distance which is equivalent to its own size during the acquisition of the images for the determination of the velocity profile. While the FWHM of the phosphorescence spot is 10 \( \mu \text{m} \), the distance travelled by the spot and hence the resolution in \( x \)-direction are set at approximately 40 \( \mu \text{m} \). The required acquisition time is about 340 \( \mu \text{s} \). It depends on test speed and reduces with increasing test speed. The spatial resolution in the \( y \)-direction is about 2 \( \mu \text{m} \) due to the averaging of the intensity profile over five pixels. The resolution in the \( z \)-direction is governed by several factors including the roughness of the surfaces and the reconstruction scheme. It has been shown in [29] that the reconstruction scheme resolves the velocity profile to within 10% of the film thickness, due to the limitation of resolvable relative shear rates. Since the thickness of lubricant films investigated in the present study is larger than roughness of rubbing surfaces, the restrictive factor is here the limitation of the reconstruction scheme.

How the spatial resolution compares with the contact diameter requires special attention. Since normal pressure within an EHD contact resembles the Hertzian contact pressure distribution, the normal pressure exerted onto the lubricant is position dependent and lubricant flow may differ locally. This consideration is especially critical in piezoviscous conditions. As the obtained profile represents the average flow of lubricant within the measurement distance, the effect of averaging will depend on the rate of change of pressure with respect to \( x \)-position \( dP/dx \). As \( dP/dx \) diminishes as we move closer to the centre of the contact, so does the effect of averaging. \( dP/dx \) around the centre also diminishes as the applied pressure and hence the contact area increases.

In this study, through-thickness velocity profiles have been investigated at the centre of the contact, meaning that the tagged column of the lubricant travels from position \( x = 0 \) \( \mu \text{m} \) to \( x = 40 \) \( \mu \text{m} \) during the acquisition of the images needed for flow profile reconstruction. To estimate the effect of averaging at various loading conditions, the size of the contact area has been measured using laser induced fluorescence (LIF) [30]. The measured contact diameters for peak pressures in the piezoviscous-elastic regime are in good agreement with approximated theoretical calculations using Hertzian contact mechanics [31] and are 165 \( \mu \text{m} \) and 293 \( \mu \text{m} \) for the peak pressures 201 MPa and 362 MPa respectively in a glass/glass contact. Within this distance, the variation in the local pressure is about 12% for the lowest pressure investigated at \( P_{\text{max}} = 201 \) MPa and drops to 4% for \( P_{\text{max}} = 362 \) MPa. Hence the \( x \)-resolution is sufficient at high pressure to determine reliable velocity profiles in shear banding conditions. When the pressures decrease the viscosity of 5P4E becomes less sensitive to pressure. Thus the 12% local variation in pressure within the measurement distance for \( P_{\text{max}} = 201 \) MPa is unlikely to affect significantly the velocity profile.

The \( x \)-resolution can be improved by using higher numerical aperture objective, which would make the diameter of the initial phosphorescent spot smaller and would therefore reduce the distance needed to completely distinguish two spots when shear banding occurs.

**Results and discussion**

The low pressure contact with \( P_{\text{max}} = 232 \) kPa corresponds to the case of a soft EHD where pressure effect is small and a Couette flow profile is expected. The normalised background-corrected image sequence (figure 4(a)) shows that the tagged volume is stretched in the \( x \)-direction with time. Based on figure 4(a), the averaged intensity distribution over five pixels in the \( y \)-direction is shown in figure 4(b) (circles). The averaged intensity distribution is used to determine the lubricant’s flow profile (figure 4(c)) and a Couette flow profile with stick boundary condition is indeed obtained. The numerical intensity distribution (figure 4(b) (lines)) based on the determined flow profile matches the experimental results (figure 4(b) (circles)) well (with an average goodness of fit \( R^2 = 0.99 \)). This shows that the newly-developed phosphorescence-based technique is capable of determining through-thickness flow profile accurately at high entrainment speed, which is at least two orders of magnitude higher than our previous work. This capability is crucial for the future study of low viscosity lubricant.

Results presented hereafter focuses on the behaviour of 5P4E under piezoviscous conditions. They are obtained at the centre of the contact \((x = 0 \text{ and } y = 0)\). Central film thickness,
The transition from Couette flow to non-linear flow profile has been observed at low shear rates, followed by a shear thinning region and then by a limiting shear stress region. In fact shear banding behaviour in 5P4E is fully developed at the highest pressures investigated in this work. The observation of an asymmetric flow profile is consistent with [32] where 5P4E is seen to be divided into two layers of different thickness in an EHD contact. The three types of 5P4E flow profiles observed are summarised schematically in figure 8.

Both the shape of the flow profile and the position of the highly sheared region are affected by the applied pressure, as shown in figures 7 and 8. While a pressure-induced transition from Couette flow to non-linear flow profile has been observed in polybutene (PB) [18], PB gives a flow profile which has been described as partial plug flow, i.e. the two boundary layers have low viscosity and experience high shear rate while the centre of the fluid flows like a plug. Hence 5P4E behaves very differently to PB, with the growth of the upper viscous boundary layer forming next to the moving surface as pressure increases. Such difference may stem from their rheological sensitivity to pressure and temperature. The increase in applied shear rate in this work as compared to the conditions applied to PB can also affect the flow of lubricant.

Shear-banding of 5P4E at high pressure has been previously observed by Bair et al [33, 34] using flow birefringence in a high-pressure flow visualization cell where the deformation behaviour of 5P4E was investigated within a shearing gap of several tens of micrometres. The transition from linear Newtonian behaviour to rate-independent behaviour has also been illustrated by flow curves in [26, 34] obtained using a pressurised Couette viscometer. A Newtonian regime has been observed at low shear rates, followed by a shear thinning region and then by a limiting shear stress region. In fact shear banding behaviour has been attributed to the existence of a limiting shear stress at which the response becomes strain-rate independent [23]. In [34], a constant limiting shear stress

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**Figure 7.** Reconstructed velocity profiles at different contact pressures (\(U_b = 72 \text{ mm s}^{-1}\)) (dashed line corresponds to a theoretical Couette flow).

**Figure 8.** Three types of flow profiles of 5P4E observed in an EHD contact.
region has been found to appear at shear rates higher than about 3,000 s\(^{-1}\) for the following conditions: \(P = 0.11\) GPa and \(T = 20^\circ C\). Hence shear banding of 5P4E may occur at pressures typical of EHD contacts. Note similar conditions have been applied in this work although higher shear rates have been achieved. To find out if our test conditions have reached the limiting shear stress region, estimation based on Eyring traction map in Evans and Johnson [35] has been used to identify the rheological regime our experiments correspond to. The test conditions applied lie in either the Eyring regime or viscoelastic regime. Due to the shear rate applied, the Eyring equation as shown in equation (1),

\[
\dot{\gamma} = \frac{\sigma_0}{\mu(P, T)} \sin h \left( \frac{\sigma}{\sigma_0} \right)
\]  

(1)

where \(\dot{\gamma}\) is shear rate, \(\sigma\) is shear stress and \(\sigma_0\) is the Eyring stress of the fluid, can be applied to both regimes. This suggests that shearing banding of 5P4E observed in this work occurs before a limiting shear stress is reached. This would be possible when a fluid with temperature and/or pressure sensitive viscosity is used as such through-thickness temperature/pressure variation can give rise to viscosity heterogeneity for shear localisation. A true limiting, shear rate independent, shear stress regime can only be achieved if 5P4E actually ‘breaks’. This is referred as mechanical shear banding [26].

Through-thickness viscosity variation can be temperature induced. If a thermal gradient does exist, the moving ball would be cooler than the stationary surface. Thus the very viscous boundary layers of lubricant formed close to both surfaces at high pressure may potentially be due to thermal effects. To investigate if temperature gradient is responsible for the observed shear banding in 5P4E, estimations of the rise in temperature due frictional heating of the film itself and of both stationary and moving surfaces have been respectively performed using the approach of Olver et al [5] and Jaeger [36] for various peak pressures in the range of 176 MPa to 362 MPa. Results show that the average surface temperature rise above the inlet temperature is 30 °C for \(P_{\text{max}} = 176\) MPa and increases to 70 °C for \(P_{\text{max}} = 362\) MPa. The average temperature rise in 5P4E film is however only about 2 °C above the surface temperature in all cases. This suggests that the existence of through thickness thermal gradient in the film is unlikely.

While through-thickness thermal gradient may be small in the 5P4E film, the film is nevertheless warm. The glass transition pressure of 5P4E would increase from 200 MPa in ambient temperature to 400 MPa at 70 °C, and reaches 600 MPa at 90 °C under static conditions [37]. The applied \(P_{\text{max}} = 362\) MPa would thus be insufficient to induce 5P4E to undergo glass transition in our applied test conditions. This supports that the shear bands observed are not mechanical shear bands.

The application of shear in an EHD contact can promote alignment of 5P4E with the degree of alignment increases with increasing test speed [38, 39]. Alignment of 5P4E can reduce its viscosity. Partial alignment can potentially occur if a thermal gradient exists, giving rise to viscosity heterogeneity in the film [40]. Further work is on the way to explore this possibility.

While the temperature of 5P4E film in the contact increases with pressure, the shear banding becomes more localised. The evolution of 5P4E profiles suggests that a very viscous layer form initially at the top, moving surface. This layer grows into the remaining of the lubricant towards the lower surface. The remaining fluid obeys Couette flow despite experiencing increasing shear rate due to the formation of thicker upper boundary layer with pressure. The origin of such pressure dependence is currently unclear. Since the shape of the film is governed by the applied normal stress, it is likely that this in turn affects the through-thickness stress distributions within 5P4E. As 5P4E has a high pressure-viscosity coefficient, small variation of pressure distribution can greatly affect viscosity distribution in the film, resulting in the observed dependence.

**Conclusion**

For the first time, through-thickness velocity profiles in an EHD contact at high speed have been obtained by phosphorescence imaging. The developed technique supersedes a previous technique based on fluorescence photobleaching and has allowed investigation of flow property of lower viscosity lubricants at conditions closer to actual service conditions than our previous work. The technique has been validated for a lubricant film of 5P4E under Couette flow obtained at low pressure and then applied to high pressure contacts. The latter shows clearly the development of shear localisation in 5P4E as the contact pressure increases. This emphasizes that linear velocity profile cannot be assumed in models for predicting friction and wear in engineering applications.

Since pressure affects the velocity profile of 5P4E in an EHD contact, it is likely that the velocity profile of 5P4E changes as 5P4E travels across the contact due to EHD pressure distribution. Thus further investigations will consist of determining both film thickness profiles and velocity profiles within the contact area from inlet to outlet. Also, as the film thickness has been shown to depend on the slide-to-roll ratio [41], further work will focus on the effect of the slide-to-roll ratio on the velocity profiles and the shear banding behaviour of 5P4E in EHD contacts.

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