Laser-induced fluorescence for film thickness mapping in pure sliding lubricated, compliant, contacts.

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ABSTRACT

A laser-induced fluorescence (LIF) technique has been used to measure fluid film thickness in a compliant, sliding contact under low-load/low-pressure conditions. The soft contact between an elastomer hemisphere and a glass disc is lubricated by a liquid containing fluorescent dye. The contact is then illuminated with 532 nm laser light through the glass disc, and viewed with a fluorescence microscope. From the intensity of emitted radiation, film thickness maps of the contact are determined. Previous calibration procedures have used a separate calibration piece and test specimen with possible errors due to differences in reflectivity between the calibration and test specimens. In the work reported in this paper a new calibration process is employed using the actual test sample, thereby avoiding such errors.

Results are reported for a sliding contact between PDMS and glass, lubricated with glycerol and water solutions under fully flooded and starved conditions. It was found that, for glycerol, the measured film thickness is somewhat lower than numerical predictions for both lubrication conditions. It is suggested that a combination of thermal effects and the hygroscopic nature of glycerol may cause the lubricant viscosity to drop resulting in thinner films than those predicted for fully flooded contacts. Starvation occurs above a critical entrainment speed and results in considerably thinner films than predicted by fully-flooded I-EHL theory. A numerical study has been carried out to determine the effect of the observed starvation on film thickness. Predicted, starved film thickness values agree well with those obtained experimentally.

Keywords: Tribology, Lubrication, Film thickness, Soft contact, Compliant contact, Isoviscous-Elastic, Fluorescence, Starvation.

1. INTRODUCTION

In many practical engineering and biological applications one or both of the bodies in the contact has a low elastic modulus and the prevailing lubrication mode is Isoviscous-Elastohydrodynamic Lubrication (I-EHL). In this regime, the contact pressure is large enough to cause significant elastic deformation of one or both of the interacting solids, but the pressure within the contact is low and insufficient to cause any substantial change in the fluid viscosity [1]. Typical example applications are windscreen wipers [2], rubber o-ring seals [3-6], synovial joints [7] and tongue-palate contact during oral processing of foodstuffs and beverages [8-13],

Although the term “soft-EHL” is widely used, the word “soft” in this context is perhaps inappropriate, since in engineering it implies a low yield point rather, than, as is characteristic of soft-EHL, a low elastic modulus. In this paper the word “compliant” will generally be used in preference to “soft”.

Most previous experimental investigation into I-EHL has focussed on the frictional characteristics in lubricated compliant contacts. By mapping Stribeck curves one is able to obtain a clear indication of the prevalent lubrication regime. Within the foodstuffs industry a large amount of research has been carried out in this area [13-17]. This has shown how lubrication regimes can be controlled by varying contact parameters such as load or elastic modulus [18], surface roughness [19] or wetting characteristics [19]. However without knowing the lubricant film thickness it is difficult to determine the shear rate experienced by the fluid film or the true tribological mechanisms controlling the lubrication regime boundaries. Film thickness measurements would also enable a deeper knowledge on the behaviour of confined thin films and deposition mechanisms.

To date there has been little work published on film thickness measurements in I-EHL contacts. This is probably because the two main methods used to measure film thickness in “hard”, metallic contacts, optical interferometry and electrical capacitance, are difficult to apply to “soft”, elastomeric contacts. There have, however, been a number of theoretical studies of I-EHL and equations for film thickness in I-EHL contacts have been developed [20,21] although these have not yet

been experimentally verified. Perhaps the best known of these is the Hamrock and Dowson equations for film thickness in elliptical contacts [20];

$$H_c = 7.32(1 - 0.72e^{-0.28k})\bar{U}^{0.64}\bar{W}^{-0.22} \quad [1]$$

$$H_m = 7.43(1 - 0.85e^{-0.31k})\bar{U}^{0.65}\bar{W}^{-0.21} \quad [2]$$

where H_c and H_m are the dimensionless central and minimum film thicknesses respectively, defined by h/R' . k is the ellipticity parameter, which reduces to unity for the circular contact of interest in the current study. Therefore for a ball on flat contact;

$$h_c = 3.3\bar{U}^{0.64}\bar{W}^{-0.22}R' \quad [3]$$

$$h_m = 2.8\bar{U}^{0.65}\bar{W}^{-0.21}R' \quad [4]$$

where h_c and h_m are the central and minimum film thickness respectively and the dimensionless operating parameters are:

$$\text{dimensionless speed parameter, } \bar{U} = \frac{U\eta}{E'R'} \quad [5]$$

$$\text{dimensionless load parameter, } \bar{W} = \frac{W}{E'R'^2} \quad [6]$$

where U is the entrainment speed, W the applied load, η the lubricant dynamic viscosity, R' the reduced radius of curvature in the entrainment direction, and E' the effective elastic modulus. The latter two terms are defined by $1/R' = 1/r_{x1} + 1/r_{x2}$ and $2/E' = (1 - \nu_1^2)/E_1 + (1 - \nu_2^2)/E_2$, respectively, where r_{x1} , r_{x2} , E_1 , E_2 , ν_1 , and ν_2 denote the radii in the entrainment direction, the Young's moduli, and the Poisson's ratios of the two contacting bodies.

In the current work, a laser-induced fluorescence (LIF) technique has been developed to study fluid film thickness in lubricated, compliant contacts. Fluorescence is an optical phenomenon whereby incident radiation is absorbed and reradiated at a longer wavelength. As described by Haugland [22], fluorescence can be used to characterize

any scalar that affects the fluorescence of the dye, including film thickness. Measured fluorescence intensity is a function of the dye characteristics, the dye concentration, the exciting light intensity, and the scalar being measured. Once a particular dye and concentration are selected, the fluorescence dependence on these factors is constant [23].

In this study, a compliant contact is produced between a PDMS elastomer hemisphere and an uncoated glass disc and is located beneath a fluorescence microscope. A low concentration of fluorescent dye is dissolved in the lubricant. The contact is then illuminated with a 532 nm laser light so that, when lubricant is entrained into the contact, a fluorescent image of the contact is produced. This image is captured using an image-intensified camera. Fluorescence intensity is proportional to film thickness so fluorescent intensity maps obtained in this way can be converted to maps of film thickness using a simple calibration. In addition to its robustness regarding the optical properties of the specimen materials, a further advantage of this technique is the wide range of film thicknesses that can be measured. A range of *ca* 200 nm to 25 μm is demonstrated in this paper. At high dye concentration (or thick films) there is a danger of saturation occurring, but theoretically the upper range can be controlled by simply altering the dye concentration.

Results are reported for a pure sliding contact between PDMS and glass lubricated with glycerol and water solutions over a range of entrainment speeds. Contact profile shapes and film thickness maps are compared to theoretical models and discussed.

2. BACKGROUND

Currently there exist a number of techniques for measuring lubricant film thickness within lubricated contacts. These include electrical techniques, e.g. electrical capacitance and resistance measurement and electromagnetic radiation-based techniques; e.g. optical interferometry, Raman spectroscopy, X-ray transmittance, laser fluorescence, as well as techniques such as ultrasonics and direct displacement. An overview of in-situ tribological techniques is given by to Spikes [24]. Many of the above approaches are well-suited to electrically-conducting and reflective materials such as metals. Far fewer are suited to measure film thickness in contacts where one

or both surfaces are an elastomer or tissue material. However, optical interferometry [25-28], magnetic resistance [5] and Raman spectroscopy [29] have been used successfully to measure lubricant film thickness within such contacts.

In a series of pioneering studies in the late 1960s, Roberts and Tabor [30] showed how optical contact interferometry could be applied to measure film thickness in compliant, rubber on glass contacts. They used monochromatic light to obtain interference images from a lubricated contact between a flat glass plate and a hemisphere or hemicylinder of smooth rubber. Using this method, Roberts *et al* [25], were able to achieve film thickness measurements down to *ca* 20 nm. Early compliant contact work was restricted to static loading and studied fluid film flow squeezed from the contact zone over time [30-32]. A small, lateral, sinusoidal motion was introduced by Roberts [26] and film thickness results were reported over a small entrainment speed range. More recently Kaneta *et al* [3,4] used a monochromatic optical interferometric technique to investigate the lubricant film thickness in a reciprocating, compliant, line contact. Investigations were made into the behaviour of o-ring seals by forming contacts between a nitrile rubber specimen of D-shaped cross section and a sinusoidally-oscillating, glass plate. Film thickness profiles were reported at a range of positions within the stroke.

In an attempt to measure film thicknesses up to a hundred microns within compliant, engineering seal contacts, Poll *et al* [5] used an approach involving magnetic flux measurement. Magnetite particles were dispersed within the lubricant, using surfactant molecules to protect against oxidation and coagulation. The particle size (average diameter of 10 nm and maximum of 80 nm) were claimed to be too small to influence film formation or to cause wear. A magnetic circuit was built such that magnetic flux was directed through the seal contact. Due to the high permeability of magnetite, the inductivity and impedance of the coil providing the magnetic potential could be calibrated to give the amount of fluid present. Using this method, film thickness was successfully measured within a rotary lip seal. The advantage with this technique is that no optical window is required; however it suffers from the fact that a magnetic fluid is required. This clearly limits the range of lubricants tested and puts a lower limit on the measurable thickness.

Bongaerts *et al* [29] successfully demonstrated the use of confocal Raman spectroscopy to measure lubricant film thickness within a compliant, tribological contact lubricated with simple Newtonian fluids and stabilized oil/water emulsions. The system has the added advantage of revealing lubricant composition within the contact and can be used to study emulsions and opaque lubricants, *e.g.* shampoos. However, the approach has some limitations. Water has a poor Raman signal, so aqueous solutions need an additional bulk solvent component, making low viscosity aqueous solutions difficult to study. Currently the method is also time-consuming and values can only be obtained at single locations, making lubricant film thickness maps difficult and tedious to acquire.

As a tool to measure lubricant film thickness, fluorescence has several advantages over other, more established techniques. A limiting factor to optical interferometry is the coherence of the illuminating light which is reduced as the separating gap is increased. Interference image quality reduces proportionally with reducing coherence and eventually film thickness measurements are no longer obtainable. This critical point is dependent on the interference system in use; *i.e.* reflecting surfaces, light source *etc.* and generally occurs at thicknesses around 2 μm . Theoretically LIF has the capability to measure far greater values of film thickness. Another significant advantage of fluorescence techniques is that they require no reflective coatings on the contacting surface. Such coatings are expensive and are prone to wear, especially for rough surfaces. LIF also has the potential to measure very low levels of fluorescence emission down to as little as that emitted from one molecule. This could be achieved with development of the experimental equipment and the use of modern visible light photon detectors, which are extremely sensitive.

There has been very little application of fluorescence to EHL contacts, probably because optical interferometry already provides an accurate measurement tool. Sugimura *et al* [33] used LIF to measure thin lubricant films in hard ball-on-disc contacts. This early investigation was restricted by poor image equipment and optical interference, inherent in all illuminated hard/metallic contacts and few experimental film thickness results were presented. However since this work, photon detection equipment, as well as fluorescent dye technology has advanced significantly. Poll *et*

al [6] successfully used fluorescence to investigate lubricant film thickness in rubber rotary lip seals. Few results were given, but sub-micron film thickness measurements were achieved across a narrow entrainment speed range ($10 - 150 \text{ mms}^{-1}$). Poll also presents a clear explanation of the LIF technique. Hidrovo *et al* [23] used a dual dye technique to eliminate optical interference effects and reduce background noise created by the image system and illuminating light source. High quality images were presented but the lower and upper limits of the system were not proven, nor was the technique used on a realistic tribological contact.

3. EXPERIMENTAL SETUP

3.1. Overall Arrangement:

The setup used in this study employs an adapted EHL optical interferometry rig (PCS-Instruments, UK). The standard rig employs a steel ball supported on a bearing carriage, to allow free rotation of the ball. For the current study, the ball and carriage are replaced by a hemispherical elastomer specimen, elevated to a similar height of the ball by a spacer. The elastomer specimen is glued to the spacer, allowing tests to operate in pure sliding conditions. Lubricant is entrained into the contact by rotation of the glass disc and to ensure this the bottom surface of the disc is immersed in lubricant.

As shown in Fig. 1, the tribological contact consists of a stationary elastomer hemisphere pressed against a transparent optical window. The optical window is a plain BK7 glass disc (PCS instruments, UK), which is naturally hydrophilic and was used as supplied. No reflective coatings were used on either surface of the glass disk. The glass disc has a Young's modulus of 65 GPa, Poisson's ratio of 0.24 and refractive index of 1.517.

The test sample is mounted on a platform within the sample pot and loaded against the glass disc from below. The existing displacement load system designed for hard/metallic contacts is employed but at low loads ($W < 1 \text{ N}$) the system is not sufficiently sensitive or responsive to correct fluctuations in load created by mechanical vibrations. The real applied load for each test was therefore determined

by fitting JKR theory to the load-dependent contact area obtained from captured images of a static contact. This load was then used for film thickness calibration and the subsequent operating load when a lubricant film is present was taken to be the static load minus the adhesive force.

3.2. Optical Equipment:

The fluorescence intensity images were observed with an Axiotech Vario microscope (Zeiss, Germany), adjustable along the vertical axis. The mechanical rig was placed on X and Y platforms, allowing image capture of the tribological contact to take place on all three axis of interest. A magnification of 3X was found to be convenient for observing both the Hertzian region and inlet/outlet lubrication across the range of loads tested. No eyepiece was needed for the experimental setup as a live image display is provided on the camera's operating PC. Excitation was provided using a solid-state, diode-pumped pulsed laser which generates a wavelength 532 nm (Laser2000 Ltd, UK). For further details on the optical setup the reader is referred to [34].

Images were captured *via* a Rolera MG1 B/W EMCCD camera (QImaging, UK). This camera was operated using a separate PC from that controlling the mechanical components. Images were captured at a series of entrainment speeds, beginning at a low value and increasing in stages. A computer-processing technique was then used to analyse captured images, pixel by pixel, and create film thickness maps based on grey scale intensity.

3.3. Test Specimens:

The elastomer samples were moulded in a plano-concave lens (Edmund Optics, UK), radius of 12.7 mm, and were made from PDMS (Sylgard 184, Dow Corning, UK). A filler was added to the PDMS to reduce background scattering and absorb laser light, since light transmitted through the PDMS might confuse intensity readings due to irregular reflectivity from within the specimens or from the sample mounting. The filler was carbon black (CB) (Fluffy, Cabot, UK), which was added to the PDMS at 0.5 wt. %. The elastic modulus of the resulting elastomer was $E = 3.8$ MPa, obtained

using dynamic mechanical analysis (DMA, Triton, UK). The concentration of filler has a large effect on the resultant elastic modulus. This has led to a number of elastic moduli of PDMS being reported in the literature; 2.4 MPa by Bongaerts *et al* [19] when no filler was used and 4.1 MPa by Vicente *et al* [14] when MgO pigment was present in the PDMS. The value of 3.8 MPa is used in all theoretical calculations in this paper.

Surface analysis of the CB-filled PDMS specimens was carried out using a Wyko optical interferometer (Veeco, UK). This showed that the CB-filled PDMS had an optically smooth surface finish of $R_a = 10.24$ nm, and a peak to valley height of 373.5 nm. The latter was due to CB particles. SEM images of the PDMS surface are shown in Fig. 2 and at 5000X magnification the CB particles can be seen to have agglomerated during the curing process and to protrude from the PDMS surface. It is believed that these protruding CB particles are easily rubbed off the surface.

A new elastomer sample was used for each test and was cleaned by successively rinsing in sodium dodecylsulphonate and distilled water, followed by immersion in isopropanol in an ultrasonic bath for three minutes and then rinsed in distilled water. The elastomer was naturally hydrophobic and was used in this state. The glass disc was cleaned using lens cleaning solution (Dallos safetyTM), followed by acetone. All tests were carried out at room temperature ($T = 22 \pm 2$ °C).

4. TEST LUBRICANTS

Glycerol and water were used as the fluid components for the majority of testing, since their polar nature allows them to dissolve a range of commercially-available fluorescent dyes. For this work, the dye Eosin was dissolved at a concentration of 0.4 wt % in all test lubricants. Eosin was chosen since its absorption peak coincides with the wavelength of the laser excitation (532 nm) and its quantum yield is sufficiently high to result in bright, clearly-defined images of the contact. Three lubricants were used; pure glycerol (GLY), 50% wt. glycerol solution (GLY50) in distilled water and distilled water. The dynamic viscosities, η , of lubricants employed were 1.16, 0.0055, 0.00089 Pas, respectively. Lubricant viscosities were measured prior to testing using a

Stabinger Viscometer (Anton Paar, UK). The viscosity for distilled water was obtained from Douglas *et al* [35].

A small amount of fluorescent dye was found to attach to both PDMS and glass; however the outputted intensity from this was very small compared to the fluorescent intensity created by the high concentration of dye used in the test solutions. Therefore, any build up of dye attached to the surfaces during testing was insignificant and had negligible effect on film thickness measurements.

5. NORMALIZATION OF FLUORSCENCE IMAGES

The laser used in the current study has a Gaussian intensity distribution. The illuminating light will also have intensity irregularities and speckle, which is caused by the ND filters, lenses or dust and damage on any of the lens/filter surfaces [36]. The overall effect of the intensity irregularities is to induce noise in the emission intensity, while the Gaussian distribution will skew the emission intensity, and thus film thickness measurements, around the Gaussian curve. To eliminate these effects, all images were normalized against a background, non-contact image, captured prior to testing. Figure 3.a) shows an intensity image from a typical, loaded tribological contact of interest, under static conditions, prior to normalization. Some of the intensity variation observed is a result of the varying laser intensity distribution rather than variations in dye quantity. Figure 3.b) shows the laser distribution from a non-contact image. Fig 3.c) shows the resultant contact image after normalization of the image in Fig. 3.a) using Fig. 3.b). It can be seen that the noise is greatly reduced by this normalization.

To further reduce the effect of background noise present in the imaging system and of fluctuations in illuminating light, smoothing of intensity values was also carried out. This was done by smoothing three successive times using a simple weighted average in which the intensity at each pixel, $P_{(i,j)}$, was determined by the following equation:

$$P_{(i,j)} = (8P_{(i,j)} + P_{(i+1,j)} + P_{(i-1,j)} + P_{(i,j+1)} + P_{(i,j-1)} + P_{(i-1,j-1)} + P_{(i+1,j-1)} + P_{(i-1,j+1)} + P_{(i+1,j+1)})/16; \quad (7)$$

where the last 8 terms are the intensity values of the immediate neighbours.

Figure 4 shows intensity plots taken from Fig. 3. a), b) and c) taken through $y = 250$ (*i.e.* along the mid-line through the contact). The non-contact image profile shows the intensity irregularities across the image. It can be seen that the contact profile is clearly improved by the normalization process. In particular, outside the contact area the profile more closely resembles a Hertzian ball-on-flat out-of-contact shape.

6. CALIBRATION

In previous fluorescence work calibration has been achieved by plotting a known film thickness *versus* fluorescent intensity. This has been done in a number of ways. Sugimura *et al* [33] employed film thickness values measured using optical interferometry and compared them to intensity curves. For compliant contacts, where extensive film thickness investigations have yet to be carried out, no comparable calibration was possible. Hidrovo *et al* [23] and Poll *et al* [6] used an assumed geometry of a calibration wedge or cylinder, respectively. Hidrovo has remarked on the effect of reflectivity on emission intensity, and pointed out that any difference between the calibration piece and the test specimen will introduce an error when converting intensity to film thickness.

In the current study, film thickness calibration was achieved based on intensity images of the contact in static steady state ($U = 0$) conditions. At the start of each test, the PDMS hemisphere was loaded against the glass disc and intensity profiles taken through the centre of the contact. These intensity profiles were then plotted against the Hertzian equation for the gap outside the central contact region [37].

$$h = \frac{a \cdot p_{\max}}{E'} \left[- \left(2 - \frac{r^2}{a^2} \right) \cos^{-1} \frac{a}{r} + \left(\frac{r^2}{a^2} - 1 \right)^{\frac{1}{2}} \right] \quad (8)$$

where the maximum pressure is defined as $p_{\max} = \frac{3W_s}{\pi a^2}$, r is the distance from the centre of the contact, a the contact radius and W_s is the total load present in the static contact.

There are two potential problems in applying this approach to low load, soft contacts. One is that there will be a significant contribution to the load from adhesive surface forces in the low load, static contact, i.e. $W_s = W_{\text{appl}} + W_{\text{adh}}$. This needs to be taken into account in solving Eq. (8). However it will not be present when the surfaces are separated by a lubricant film. The second is the effect of adhesion in the static contact may change the separation profile outside the contact so that Eq. (8) is no longer applicable. A third complication in the current study was that the actual value of applied load, W_{appl} was not directly controlled due to inadequacies in the loading system.

To determine W_s and W_{appl} the following procedures were adopted. The contact radius was determined from the static intensity image. Using JKR theory for a ball on flat, this is related to the applied load and the surface energy by:

$$a_{\text{JKR}} = \left(\frac{3R}{4E'} \left(W_{\text{appl}} + 3\pi R \Delta\gamma + \left(6\pi \Delta\gamma R W_{\text{appl}} + (3\pi R \Delta\gamma)^2 \right)^{1/2} \right) \right)^{1/3} \quad (9)$$

where $\Delta\gamma$ is the specific energy of adhesion between the two surfaces and R the radius of the ball. The specific energy of adhesion for the PDMS samples on glass was determined by carefully placing a PDMS sample on the top surfaces of a plain glass disc. The contact was then viewed from the underside of the disc, and the applied load was simply the weight of the PDMS sample. An optical interferometric technique employing polarised white light and quarter wavelength plates, developed by Eguchi *et al* [38] was used to accurately capture the contact area and measure the contact radius. By applying Eq (9), $\Delta\gamma$ was found such that the calculated JKR contact radius matched the observed image contact radius. $\Delta\gamma$ was found to be 0.0356 J m^{-2} .

This value of $\Delta\gamma$ could then be used in Eq. (9) at the start of each test to obtain from the static contact radius the value of applied load, W_{appl} and also the total static load,

W_s for use in Eq. (8). The latter is simply the sum of the four terms in the second bracket in Eq. (9). During the test the contact operates in the I-EHL regime where there should be no surface adhesion forces so the operating load equals W_{appl} .

The applied load was found to be $W_{appl} = 40 \pm 2$ mN for all tests.

Figure 5.a) compares the intensity profile across the centre of the contact with the gap predicted from Eq. (8). Zero film thickness is assumed within the central contact region. For a compliant contact such as the one used in the present study, rapid approach of the surfaces traps a ‘bell’ of liquid in the centre of the contact and the entrapped lubricant is squeezed out over time under static conditions [30,31]. To achieve an accurate calibration the contact was therefore left for 10 minutes before the intensity image was taken. The intensity *versus* film thickness calibration graph is shown in Fig.5.b). At low film thickness, a difference in thickness of one micron is represented by *ca* 500 intensity counts, which means that theoretically a difference in thickness of 2 nm can be detected. However, this value is likely to be far higher, as indicated by the intensity noise present at low film thickness in Fig 5.a). This noise will create a minimum measurable film thickness, which is later shown to be *ca* 300 nm.

The calibration shown in Fig. 5 assumes the theoretical gap outside the central contact regions can be obtained from Hertz theory (Eq. 8), *i.e.* there is no significant change resulting from adhesion forces in the static contact. Greenwood and Johnson [39] have derived a numerical solution for the gap profile outside of the contact area between a ball and a flat contact influenced by surface adhesion forces. They used a ‘double-Hertz’ model which overlaps the initial Hertzian contact zone with a larger out-of-contact zone, over which the adhesive force acts. In the current work it was found that the difference in film thickness between the Hertz and double Hertz models over the gap height range of interest was less than 5 %. Based on this, the simple Hertz model with total load modified to include an adhesive contribution was considered to be adequate for calibration purposes.

7. FULLY FLOODED RESULTS

In the first set of experiments, GLY, GLY50 and Water are used as test lubricants to demonstrate the film thickness measurements capabilities of the LIF technique under fully flooded, steady state conditions. Figure 6 shows central film thickness, h_c , results for all three test lubricants. The theoretical central film thicknesses for all three lubricants, from Eq. (3), are also plotted as solid black lines. In hard, metallic contacts h_c normally lies within a flat plateau region bounded by a horseshoe-shaped constriction, making it a significant value as it describes a large proportion of the contact. For compliant contacts h_c is less clear since the contact generally forms a hydrodynamic wedge shape without a central plateau. Therefore the position of h_c , was simply defined as the middle of the detected contact map [28].

For GLY50 and distilled water, a large amount of scatter is observed at low entrainment speeds and low film thickness. The noise indicates a minimum detectable film thickness of *ca* 300 nm for the current set-up. As entrainment is increased good agreement between theoretical prediction and experimental values is observed.

For GLY it can be seen that the experimental results are considerably lower than the numerical predictions for the measured dynamic viscosity of $\eta = 1.16$ Pas. However using a value of $\eta = 0.15$ Pas a good agreement is achieved. Similar work carried out by the authors using optical interferometry [28] and work carried out by Bongaerts *et al* [29] using Raman spectroscopy to measure film thickness in compliant contacts, observed a similar disparity between theoretical and experimental values. It should be noted that Eq. (3) is a best fit obtained to a series of I-EHL numerical solutions obtained by Dowson and Hamrock [20]. In their study a range of values of $\bar{U} = 5 \times 10^{-9}$ to 5×10^{-8} and $\bar{W} = 0.2$ to 2×10^{-3} was used. In the current experimental measurements the range of values of $\bar{U} = 7 \times 10^{-12}$ to 1×10^{-6} and $\bar{W} = ca 2 \times 10^{-5}$ was covered so $\bar{W}$ is somewhat smaller than the dimensionless load parameters used by Hamrock and Dowson. This may account for the large disagreement between experimental and theoretical plots.

Bongaerts *et al* [29] suggested that the discrepancy between the measured and lower operating viscosity might be due to either an increase in lubricant temperature or to

the glycerol adsorbing excess water from the atmosphere. One possibility is heating by the illuminating laser light. The maximum impact of such heating can be estimated by relating the laser power, P_R , to the change in temperature, ΔT , using the relationship:

$$P_R = \frac{m.c_p \Delta T}{t} . \quad (10)$$

where c_p is the specific heat capacity of glycerol, m the mass of lubricant volume affected ($m = \pi a^2 h_a \rho$) and t the time taken for fluid to pass through the contact ($t = \frac{2a}{U}$) where ρ is the density of glycerol, a is the contact radius and h_a the average film thickness. For a density and specific heat of glycerol of 1250 kg/m^3 and $2400 \text{ J kg}^{-1} \text{ K}^{-1}$, $a = 0.42 \text{ mm}$ and assuming an average film thickness of $5 \text{ }\mu\text{m}$ at $U = 10 \text{ mms}^{-1}$, as indicated in Fig 6. The temperature rise at the given laser power of 0.4 mW would be *ca* $4 \text{ }^\circ\text{C}$.

It is also possible that shear heating of the lubricant in the contact might also result in a temperature increase and consequent reduction in effective viscosity since. Due to its high viscosity, an increase of $10 \text{ }^\circ\text{C}$ in the test temperature will, for glycerol concentrations between 100 and 90 %, roughly half the lubricant viscosity [40]. An upper-bound estimate of the effect of such heating on temperature rise can be estimated from a simple heat balance between the heat generated by shear and that removed by convection, assuming no heat conduction, *i.e.*

$$\dot{q} = \mu W u_s = m c_p \frac{\Delta T}{t} \quad (11)$$

where μ is the friction coefficient and u_s the sliding speed. Rearrangement gives;

$$\Delta T = \frac{\mu W u_s t}{\rho h A c_p} = \frac{2 a \mu W S R R}{\pi a^2 \rho h c_p} \quad (12)$$

where SRR is the slide-roll ratio (ratio of sliding speed to entrainment speed = 2 for pure sliding). The friction coefficient, which is derived from Couette forces, can be calculated using the equation [21]:

$$\mu_{Couette} = SRR(3.8\bar{U}^{0.71}\bar{W}^{-0.76} + 0.96\bar{U}^{0.36}\bar{W}^{-0.11}) \quad (13)$$

Assuming the viscosity at the inlet, where the majority of the Couette friction arises, is the measured viscosity of 1.16 Pas, Eq. (13) gives $\mu = 0.25$. From Eq (12) the subsequent temperature rise predicted for $U = 10 \text{ mm s}^{-1}$, is *ca* 2 °C. However the actual value for temperature rise will probably be considerably less than this due to (i) heat conduction, (ii) the fact that equation (12) calculates the rise at the contact exit rather than the inlet where entrainment is established and (iii) the laser power is lower than that quoted due to it illuminating a larger area than the contact, so the power intensity over the contact is lower, and (iv) the power is diminished by *ca* 4 % at each interface the laser passes through (*e.g.* the lens surfaces). Even so, a temperature change of a few degrees within the contact seems possible, but insufficient to cause the observed discrepancy.

Bongaerts' other suggestion is that the glycerol in the test chamber, which is hygroscopic, will absorb water vapour from the atmosphere, lowering the fluid viscosity. To produce the apparent reduction in viscosity by dissolved water, alone would imply a change on glycerol composition from 100% glycerol to *ca* 90% glycerol [40]. This seems unlikely. However, a combination of temperature rise and water absorption may be a possible cause of the observed discrepancy between the experimental and theoretical results for glycerol.

8. STARVED RESULTS

A second set of experiments was carried out investigated the onset of starvation in a compliant contact. To encourage the onset of starvation, tests were carried out in which a small amount of lubricant was smeared onto the underside of the glass disc rather than fully-immersed conditions. The maximum entrainment speed was also increased to 1100 mm s^{-1} .

Figure 7 shows a series of fluorescence intensity images obtained with increasing entrainment speed. Lubricant flows from right to left along the x axis. A ‘horseshoe’ constriction at the outlet of the contact is formed whenever lubricant is entrained, although it can only be seen from $U = 14.6 \text{ mm s}^{-1}$ and above, due to the colour scale chosen for these images.

It should be emphasised that, unlike optical interferometry, LIF does not measure the separation of the surfaces but rather the amount of fluorescent dye between the surfaces. Thus if starvation or cavitation occurs, this will be reflected in a lower intensity than there would be with a full film between the surfaces.

From the calibrated fluorescence images, central and minimum film thickness values were obtained. Central film thickness values were taken at the position x and $y = 0$. The results for pure glycerol are shown in Fig. 8 plotted against the entrainment speed, U . Also shown is the theoretical central film thickness plotted as a solid black line. This is based on the effective viscosity of 0.15 Pas found in the fully flooded tests.

It can be seen that film thickness values for h_c are below the theoretical fully-flooded values, even though the latter are based on the effective viscosity of 0.15 Pas which best fitted the fully-flooded measurements. At high speeds, a rapid divergence is recorded. Since this reduction in film thickness occurs when the lubricant supply is limited it is most likely to result from starvation.

Figures 9.a) and (b) shows measured film thickness profiles along the entrainment direction (at $y = 0$) and transverse to the entrainment direction (at $x = 0$) respectively. These were obtained from the images shown in Fig. 7. As U increases, profiles along the sliding direction change from being close to Hertzian to forming an almost linear wedge. The constriction near the contact exit can be seen but is partially obscured by noise in the profile. This noise is not believed to indicate a real feature such as surface roughness or debris within the lubricant, but rather is due to noise within the illuminating light [36].

In Fig. 10 the measured film thickness profiles for GLY, along the midline in the entrainment direction ($y = 0$), for $U = 14.6, 54.8$ and 210 mms^{-1} , are compared to numerical solutions for compliant EHL developed by de Vicente *et al* [21]. The theoretical profiles under fully flooded conditions (using $\eta = 0.15 \text{ Pas}$) are shown and also solutions assuming starved conditions. The solution method is described in [21]. In the starved cases the inlet fluid boundary was taken to be the positions “inlet S ” shown in the figures while in the fully flooded conditions it was taken to be $4.5a$ in front of the centre of the contact where a is the Hertzian contact radius. The minimum film thickness position was used as a universal reference point to compare the plots.

It can be seen that the experimental results at high speeds can only be made to match predictions if severe starvation is assumed with the inlet approaching very close to the Hertzian radius. Under these conditions it is also clear that there is far less fluid present upstream of this starved inlet than would be required to fill the gap between the surfaces. By contrast, at low speeds, only mild starvation of $S = 2a$ is required to fit the results and, indeed, the fluorescence results show that the inlet remains full of fluid out the maximum measureable distance of $2a$ in front of the Hertz inlet.

Starvation under elastohydrodynamic conditions has been extensively investigated for ‘hard’, metallic contacts and is now well understood [37,41-43]. Wedeven *et al* [44] showed that the film thickness within the Hertzian contact region is a function of the lubricant supply immediately upstream. Wedeven derived a dimensionless expression for the starved central film thickness, h_s :

$$\frac{h_s}{h_f} = \left[\frac{S}{S_f} \left(2 - \frac{S}{S_f} \right) \right]^{\frac{1}{2}} \quad (14)$$

where S and S_f are the distances between the lubricant boundary edge of the inlet lubricant reservoir and leading edge of the Hertzian contact for a starved and just fully

flooded cases respectively and h_f is the central film thickness under fully-flooded conditions. Wedeven has also provided an empirical expression for the fully-flooded inlet distance for a ball on flat contact, $S_f = 3.52(R'h_f)^{2/3}a^{-1/3}$.

For the conditions in Fig. 10.b), the theoretical Hertzian radius is 0.42 mm. Assuming the central contact position is located at *ca* 0.3 mm, the theoretical central fluid film thickness for a fully flooded case (using $\eta = 0.15$) is *ca* 10 μm . This gives a value for the fully-flooded inlet distance, $S_f = 1.18$ mm. S is taken to be the distance from the contact edge on the measured profile to the point where the measured profile deviates from the theoretical. Taking S to be approximately 0.1 mm, so $S/S_f = 0.085$. Based on this ratio, Eq. (14) predicts a starved central film thickness of 4.01 μm , which is reasonably close to the measured value of *ca* 5.7 μm . The same process fails for $U = 210 \text{ mm s}^{-1}$, Fig 10.c), as the value for S appears to be negative which results in a complex number for Eq. (14). It should be recognised that this is a very approximate analysis since Eq. (14) was derived from piezo-viscous EHL theory and is unlikely to be valid for the I-EHL contact studied. EHL contacts remain close to Hertzian in shape under heavy starvation while the compliant contacts studied in this paper appear to adopt a truncated wedge shape. The analysis above also requires knowledge of the position of the centre of the contact, which can only be approximately estimated. But the estimate does lend some further credence to the hypothesis that starvation is occurring in the sliding contacts studied.

6. Conclusion:

This paper has shown that fluorescence microscopy can be used to study film thickness in lubricated, compliant contacts. There are a number of benefits to this technique:

- No reflective coatings are required to either contacting surface.
- Obtaining film thickness maps is fast compared to alternative film thickness techniques such as monochromatic optical interferometry [28] and Raman spectroscopy [29] which tend to be time-consuming and suffer from technical difficulties.

- The ease of film thickness mapping in compliant contacts should be of particular value when investigating the lubricating properties shear thinning and viscoelastic solutions.
- The method can be used to validate theoretical models, in particular for I-EHL contacts.

Film thickness data were obtained for fully flooded conditions. There was good agreement for low viscosity fluids but film thickness was lower than predicted values for high viscosity ones. This was tentatively attributed to a lowering of the viscosity due to thermal effects and the hygroscopic nature of glycerol. The film thickness reduced further below predicted values with the onset of inlet starvation. A numerical solution of the point contact, starved I-EHL problem has been obtained for comparison with experimental results. This shows good agreement with the measured values.

A detectable minimum film thickness limit was indicated in experimental results to be *ca* 300 nm. Through careful selection of dye concentration it may be possible to reduce this limit. For future work the use of a two-dye LIF ratiometric [23] system should allow for high quality imaging and could provide a means of measuring very low film thicknesses. This system also allows for temperature mapping and it may also be possible to measure multiphase lubricants by adding a separate dye to each phase.

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LIST OF FIGURES

Figure 1. Tribological contact and sample pot.

Figure 2. SEM images of CB filled PDMS test specimens. Image on the left is taken at 2500X magnification, image on the right at 5000X.

Figure 3. Intensity images used for normalization process; (a) typical tribological contact of interest prior to normalization: (b) non-contact image: (c) image (a) after normalization.

Figure 4. Intensity plots of non-contact, contact and normalized contact image, taken through $y = 250$.

Figure 5.a) Line profile of the fluorescence intensity from calibration image for the tribological contact lubricated with pure glycerol, and predicted film thickness profile across the calibration contact from Eq. (8), plotted as solid and dashed lines respectively.

Figure 5.b) Intensity *versus* film thickness calibration curve.

Figure 6. Central film thickness for the tribological contact of interest under $W = 40$ mN, lubricated with GLY, GLY50 and water. Numerical predictions from equation (3) are shown as solid lines for each lubricant using the measured viscosity. The predicted film thickness for $\eta = 0.15$ Pas is shown as a dashed line.

Figure 7. Film thickness maps of the tribological contact, lubricated with GLY. Film thickness is expressed as RGB intensity given in the colour bar scale on the right of the figure. Inlet is on the right of each image. Images are *ca* 1.5x1.5 mm in size.

Figure 8. Central film thickness for the tribological contact of interest under $W = 25$ mN, lubricated with GLY. Numerical predictions from equation (3) are shown as a solid line, using the lowered viscosity ($\eta = 0.15$).

Figure 9.a) Film thickness profile plots in the YZ plane at selected entrainment speeds. Fluid flows left to right.

Figure 9.b) Film thickness profile plots in the XZ plane for selected entrainment speeds.

Figure 10. Measured and numerically predicted film thickness profiles for the tribological contact of interest, lubricated with GLY, under $W = 25$ mN, at (a) $U = 14.6$, (b) 54.8 and (c) 210 mm s⁻¹. Fluid flows from right to left. Plots have been reconciled at the minimum film thickness values, h_m .

Figure 1.

Figure 2.

Figure 3.

Figure 4.

Figure 5.(a)

Figure 5.(b)

Figure 6.

Figure 7

Figure 8

Figure 9.(a)

Figure 9.(b)

Figure 10