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ORIGINAL PAPER

DLC and Glycerol: Superlubricity in Rolling/Sliding Elastohydrodynamic Lubrication

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Abstract

Low friction is one of the most important parameters for the development of machine components and machines with high efficiency. Many of the common machine components of today such as gears, rolling element bearings and cam-followers are defined by their non-conformal contacts leading to high-contact pressures, typically 1–4 GPa. The lubrication of such contacts is usually called elastohydrodynamic lubrication (EHL). Diamond-like carbon (DLC) coatings and glycerol have individually been shown to produce low friction in boundary, mixed and full film lubrication. A few studies have been conducted using both glycerol and DLC-coated surfaces to achieve even lower friction in pure sliding boundary-lubricated contacts. However, the literature is lacking studies of how the combination of glycerol and DLC performs in non-conformal rolling/sliding contacts where many common machine components operate. Such a study is presented in this article where a ball-on-disc test rig is used to investigate the performance of the combination of DLC and glycerol at pressures up to 1.95 GPa at various entrainment speeds and slide-to-roll ratios. The investigation shows that the DLC-glycerol combination provides very low friction values, in some cases, below the superlubricity threshold of 0.01, possibly shown for the first time at such high pressure in a non-conformal rolling/sliding contact. The low friction mechanism in full film lubrication is a combination of the low pressure-viscosity and high temperature-viscosity sensitivity of glycerol in combination with thermal insulation of the DLC coating and is presented as thermally assisted liquid superlubricity.

Keywords Traction · EHL · Coatings, friction reducing · Superlubricity · Friction · Thermal effects in EHL · DLC · Glycerol

1 Introduction

Low friction is one of the most important parameters for the development of machine components and machines with high efficiency. Many of the common machine components of today such as gears, rolling element bearings and cam-followers are defined by their non-conformal contacts leading to high contact pressures, typically 1–4 GPa. At these pressures the surfaces are extensively deformed. Normally, the majority of the deformation is elastic, and the surfaces are mostly undamaged. The lubrication of such contacts is usually called elastohydrodynamic lubrication (EHL). The lubricant film inside of the contact is subjected to approximately the same pressure as the Hertz pressure in an unlubricated contact. At GPa pressure levels, the viscosity of the lubricant typically increases several orders of magnitude and become solid-like. Surface coatings and novel lubricants have been shown to reduce friction in various types of lubricated systems, including EHL.

Lubrication is usually divided into three regimes; boundary, mixed and full film lubrication [1]. Diamond-like-carbon (DLC) coatings have been studied extensively and been shown in many cases to provide friction reductions in boundary lubrication compared to uncoated steel surfaces [2–5]. Several researchers have experimentally observed a reduction in friction with DLC-coated surfaces also in full film EHL [6–12]. The mechanism leading to the reduction in friction in full film conditions is not yet fully understood. One explanation given by several authors is the effect of boundary slip, or solid–liquid interface slip [7, 11, 13]. The present author provided an alternative hypothesis where the friction reduction is explained by thermal softening of the lubricant as a result of thermal

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insulation [8–10] due to the low thermal conductivity of DLC coatings. This has been further validated numerically [14–17] and experimentally by other authors [18].

As an alternative to conventional petroleum-based oils, new types of lubricants have been investigated that show great potential in reducing friction losses. Glycerol has been shown to provide low friction in both boundary and full film lubrication [19–21].

A few studies have been conducted using both glycerol and DLC-coated surfaces to achieve even lower friction in pure sliding boundary-lubricated contacts [22–26]. In some of these studies, friction coefficients below 0.01 is found. Friction below this threshold is often called ultra-low friction or superlubricity. The term superlubricity was introduced in the 1990s when Hirano and Shinjo proposed a theoretical sliding regime in which friction or resistance to sliding nearly vanish [27, 28]. This would be realized if the sliding interface consisted of atomically flat, rigid and incommensurate surfaces which would lead to weak atomic interactions and would only be possible to obtain under high vacuum. This was later called structural (super)lubricity [29]. Experimental work claiming structural superlubricity followed the theoretical work by Hirano and Shinjo [30–32]. More recently, graphene has been used to achieve superlubricity in dry sliding contacts [33–35]. Liquid superlubricity has also been reported in various studies [36–39] where especially the use of ta-C coatings have made it possible to achieve superlubricity in all lubricating regimes. For more information on superlubricity see for instance refs. [40, 41].

However, the literature is lacking studies of how the combination of glycerol and DLC performs in non-conformal rolling/sliding contacts where many common machine components operate. Such a study is presented in this article where a ball-on-disc test rig is used to investigate the performance of the combination of DLC and glycerol at pressures up to 1.95 GPa at various entrainment speeds and slide-to-roll ratios. The aim of the work is to investigate if the low friction reported with DLC and glycerol in pure sliding boundary lubricated contacts can be achieved also in rolling/sliding boundary lubricated contacts and if the low friction will be present also in the full film friction regime.

The investigation shows that the DLC-glycerol combination provides very low friction values, in some cases below the superlubricity threshold of 0.01. This is maybe the first time this is shown for a non-conformal rolling/sliding contact under such high pressure and could be presented as thermally assisted liquid superlubricity.

2 Overall Methodology

The following sections cover lubricants, test specimens, coatings, the experimental apparatus and how the experiments were set up and run.

2.1 Lubricants, Test Specimens and Coating

The primary lubricant used in this work is a reagent grade Glycerol (purity ≥ 99%) purchased from Sigma-Aldrich. The water content was checked with a Karl Fisher titration device to be less than 1 wt% before and after each test. A paraffin oil without additives supplied by Statoil Fuel and Retail was used as a reference fluid. A fluid without additives was chosen to minimize additive effects on film thickness and friction.

The absolute viscosities were measured at 40 °C with a Bohlin CVO 100 rheometer. A concentric cylinder geometry was used with a 25 mm inner diameter and a 27 mm outer diameter. In each measurement, the shear rate was controlled at 20 s^{-1}. The viscosities together with other lubricant parameters are summarized in Table 1.

The test specimens for the friction measurements in the ball on disk machine were produced in DIN 100Cr6 (AISI 52100) bearing steel and measured to a surface roughness, RMS of 25 nm for the balls and 35 nm for the disks, which gives a combined roughness of approximately 43 nm. The surface roughness measurements were conducted in a Wyko NT1100 optical profilometer system from Veeco. The measurements were performed using 10× magnification and 1× field of view. The balls are grade 10 with a 13/16 inch (20.63 mm) outer diameter and a hardness of about 60 HRC. The disks have a 4 inch (101.6 mm) outer diameter, a circumferential grind (before polish) and are through hardened to about 60 HRC. Except the steel uncoated reference specimens, the remaining specimens were coated with Tribobond 43, a commercially available hydrogenated amorphous carbon ((Cr+)a-C:H), through plasma-assisted chemical vapor deposition. The coating thickness was measured to 2.8 µm using Calotest. A chromium-based inter layer with a thickness of 0.1–0.3 µm

Table 1 Lubricant properties

Fluid	Glycerol	Paraffin
Kinematic viscosity @ 40 °C [mPas]	270	95
Pressure viscosity @ 40 °C [GPa^{-1}]	5.4 [42]	23.3[a]
Thermal cond. @ amb. pressure [W/mK]	0.29 [43]	0.14[a]
$\delta\eta/\delta T$ @ 40°C [mPas/K]	21[b]	5[a]

[a]Derived from data in [44]

[b]Derived from data in [45]

deposited by magnetron sputtering was used to improve the adhesion. The roughness values of ball an disk were maintained almost the same after the specimens had been coated with DLC.

2.2 Friction Measurements

The experiments were carried out in a Wedeven Associates Machine (WAM) 11, ball on disk test device. The lubricant was supplied at the center of the disk in an oil dispenser that distributes the lubricant across the disk surface. The lubricant was circulated in a closed loop from the oil bath, through a peristaltic pump to the oil dispenser at the center of the disk. The peristaltic pump was delivering approximately 180 ml/min. Two thermocouples were used in the test setup, one located in the oil bath and one in the outlet of the oil supply. A more thorough description of the test rig and its features can be found in previous work [46].

Both ball and disk specimens were cleaned with heptane and ethyl alcohol before starting the experiments for each of the test cases. The tests were performed with two different loads; 80 and 300 N, which corresponds to a maximum Hertzian pressure of 1.25 and 1.95 GPa, respectively. The tests were performed at a temperature of 40 ∘C. Before starting the experiments for each test case, the test device was warmed up to the desired operating temperature for approximately 60 min with lubricant circulation over both ball and disk to ensure thermal stability. When a stable temperature was reached, a 80 or 300 N load was applied and the machine was calibrated for pure rolling by adjusting spindle angle and positioning of the ball to ensure a condition of no spinning. Subsequently, the test procedure was started.

Two different test procedures were used, hence producing two types of friction curves, traction-curves and stribeck-curves. For the traction-curves, two different entrainment speeds were used, 1.6 and 6.1 m/s. The slide to roll ratio (SRR) were varied form the lowest value of 0.002 to the highest value of 1.05. SRR is defined as the ball surface speed minus the disk surface speed, divided by the entrainment speed. All tests in this investigation were hence conducted with the ball having a higher surface speed than the disk.

For the stribeck-curves, the SRR were held constant at two different levels, 0.1 and 1.0. The entrainment speed was then varied from the highest value of 6.1 m/s to the lowest value of 0.013 m/s. For both test procedures the temperature of the oil bulk and fluid adhered to the disk surface was typically deviating less than ±1 °C from the target temperature of 40 °C during testing.

A summary of the test conditions is found in Table 2. All tests were performed at least five times to ensure repeatability.

Table 2 Friction test conditions

Test procedure	Stribeck	Traction
Lubricant	Glycerol and paraffin	Glycerol and paraffin
Temperature	40 °C	40 °C
Contact load,	80 and 300 N	80 and 300 N
Maximum Hertzian pressure	1.25 and 1.95 GPa	1.25 and 1.95 GPa
Entrainment speed, U_e	6.1 to 0.0013 m/s	1.6 and 6.1 m/s
Slide to roll ratio, SRR	0.1 and 1.0	0.0002 to 1.05

Table 3 Film thickness and thermal correction

Fluid	Glycerol	Paraffin
Isothermal film thickness		
H_{min} @ 1.25 GPa, 1.6 m/s (nm)	363	366
H_{min} @ 1.95 GPa, 1.6 m/s (nm)	330	332
H_{min} @ 1.25 GPa, 6.1 m/s (nm)	903	908
H_{min} @ 1.95 GPa, 6.1 m/s (nm)	820	825
Gupta thermal correction factors		
1.25 GPa, 1.6 m/s	0.77	0.84
1.95 GPa, 1.6 m/s	0.76	0.83
1.25 GPa, 6.1 m/s	0.38	0.5
1.95 GPa, 6.1 m/s	0.35	0.47
Thermally corrected film thickness		
H_{min} @ 1.25 GPa, 1.6 m/s (nm)	281	309
H_{min} @ 1.95 GPa, 1.6 m/s (nm)	250	276
H_{min} @ 1.25 GPa, 6.1 m/s (nm)	340	451
H_{min} @ 1.95 GPa, 6.1 m/s (nm)	287	390

3 Results and Discussion

The traction curves obtained for glycerol and the paraffin oil are shown in Fig. 1. First of all, it should be mentioned that all of the results shown in Fig. 1 were measured in full film conditions with no asperity interactions. At the lowest entrainment speed of 1.6 m/s, the highest SRR of 1.05 and the highest contact pressure of 1.95 GPa, the thermally corrected Hamrock & Dowson equation gives a minimum film thickness of 276 nm for the paraffin and 250 nm for glycerol. In both cases significantly more than three times the combined roughness of the specimens, approximately 130 nm.

The isothermal minimum film thickness of the fluids at 40 °C and 1.25 and 1.95 GPa pressure levels at 1.6 and 6.1 m/s were calculated using the Hamrock & Dowson equation [47] and the results are shown in Table 3. To account for inlet shear heating a thermal correction factor for film thickness in circular contacts by the work of Gupta et al. [48] were employed using the values in Table 1. The

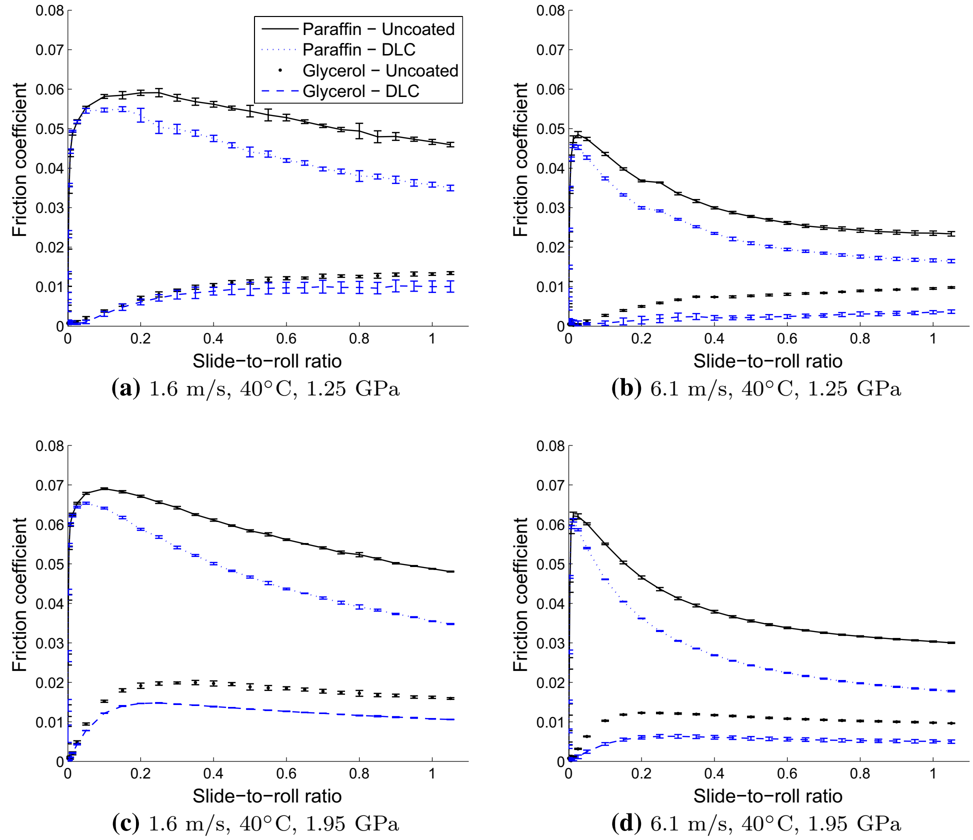


Fig. 1 Traction curves comparing glycerol and paraffin oil with uncoated and DLC coated specimens including standard deviations

(a) 1.6 m/s, 40°C, 1.25 GPa **(b)** 6.1 m/s, 40°C, 1.25 GPa **(c)** 1.6 m/s, 40°C, 1.95 GPa **(d)** 6.1 m/s, 40°C, 1.95 GPa

gupta thermal correction has recently been shown to offer rather good agreement with film thickness measurements up to 20 m/s, albeit at 0.5 GPa pressure for mineral oil, PAO and an ester oil [49]. The resulting thermal correction factors and minimum film thicknesses for the glycerol and paraffin at 40 °C and 1.25 and 1.95 GPa pressure levels at 1.6 and 6.1 m/s with a SRR of 1.05 is also presented in Table 3. The thermal correction gives significantly reduced film thickness values, especially at the higher entrainment speed. Furthermore, due to its higher temperature-viscosity sensitivity, the film thickness reduction is larger for glycerol than for paraffin. The application of an insulating coating will further reduce film thickness, but not to a great extent. Data from [9] show that with the coating thickness used in the present experiment at 1.95 GPa pressure, 6.1 m/s and a SRR of 1.05 would give a difference in minimum film thickness between coated and uncoated specimens for squalane oil of 10 %.

It is obvious that glycerol provides significantly lower friction than the paraffin oil at all investigated operating conditions with uncoated specimens, black lines in Fig. 1a–d. The difference being larger at lower speed and higher pressure. The low friction provided by glycerol in rolling/sliding full film lubrication is in line with previous studies [20, 21], although this time shown for significantly higher pressures, speeds and SRRs. The low friction obtained with glycerol in full film EHL is not yet fully understood. The main reason for the low friction generation with glycerol is due to differences in viscosity trends. Glycerol which is a hydrogen-bonded liquid does not increase as much in viscosity with pressure as van der Waals liquids such as paraffin oil [50]. Therefore, glycerol has a low pressure-viscosity coefficient, 5.4 GPa^{-1} [42] at 40 °C compared to around 23 GPa^{-1} for a typical paraffin oil [44] at the same temperature. Due to the low pressure-viscosity response of glycerol, the viscosity in the high-pressure region of the contact where friction is governed will be low, thus contributing to low friction. Glycerol also has a higher temperature-viscosity sensitivity than the paraffin oil which would not only contribute to film thickness reduction at higher speeds and SRRs but also give rise to a larger viscosity reduction in the high pressure-zone and therefore also reduce friction.

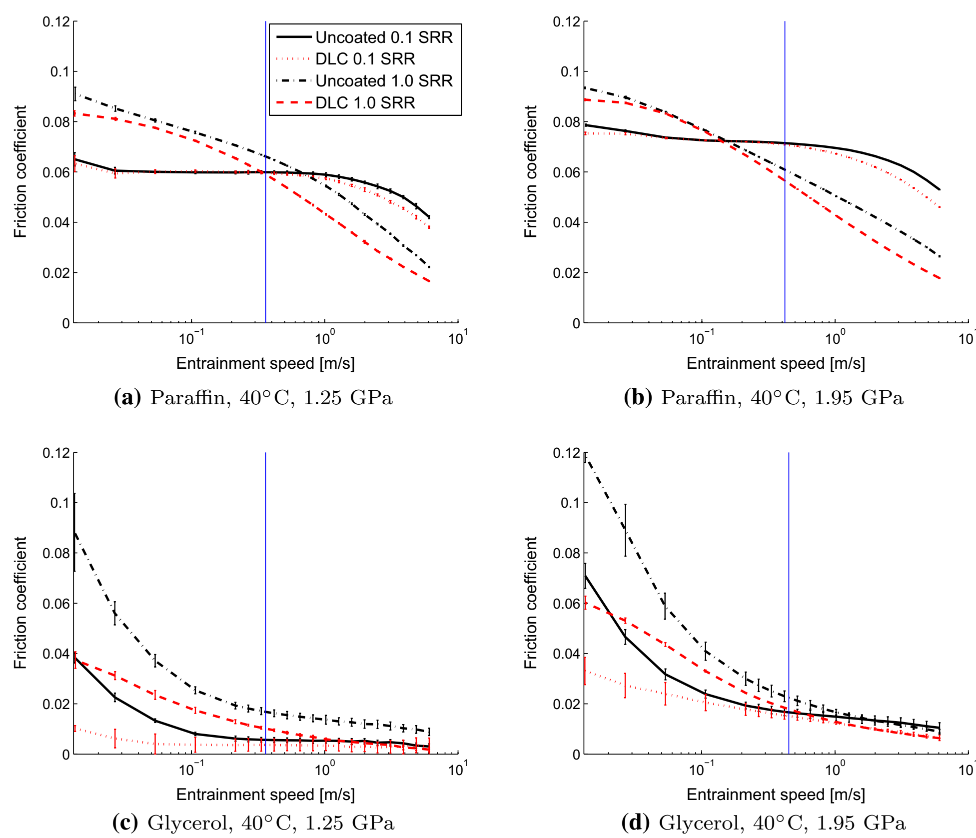
Another reason for the low friction of glycerol in EHL is believed to be related to the formation of aldehydes or organic acids and water molecules in the contact due to tribodegradation of glycerol under the combined effects of pressure and shear [19, 20, 24]. The water formation leads to lower local viscosity and thus lower friction.

The paraffin oil shows the classical regimes of friction in EHL starting with a linear increase in friction at low SRRs, followed by a non-linear increase where shear thinning is taking place. At even higher SRRs follows a region where friction is governed by the limiting shear stress of the lubricant, here a plateau can also be present, followed by a decrease in friction due to thermal softening of the lubricant [51, 52]. Glycerol, on the other hand, shows a different behavior. Due to its low molecular weight (92 kg/kmol), glycerol has a high Newtonian-limit, and the low sensitivity to pressure makes it likely that limiting shear stress will not be operative. For the lower pressure case in Fig. 1a and b calculations using data from [50] and thermally corrected film thicknesses gives a value above 1 for Na/Wi^2, where Na is the Nahme-Griffith number and Wi^2 the Weissenberg number. A value above 1 indicates that thermal softening will overwhelm the shear-dependence of viscosity [53, 54]. Consequently, these friction curves would, therefore, be governed by thermal softening of the lubricant. However, for the higher pressure case in Fig. 1c and d Na/Wi^2 gives a value below one, suggesting that non-newtonian effects cannot be excluded.

What is also clear from the results in Fig. 1 is that the addition of a DLC coating reduces friction under all tested operating conditions, both for paraffin and glycerol, blue lines in Fig. 1a–d. This friction reduction is consistent with previous studies of the effect of DLC coatings on EHL friction [6, 8–11, 55] and most likely an effect of thermal insulation due to the low thermal conductivity of the DLC coating [8–10]. In case of glycerol this effect will be enhanced by the higher temperature-viscosity sensitivity compared to paraffin.

The already low friction with glycerol is brought down to very low friction levels with DLC-coated specimens. In most of the tested conditions the DLC-glycerol combination provides friction values below 0.01, which is described as ultra-low friction or superlubricity in pure sliding contacts. This is quite remarkable and possibly the first time it has been shown for a high-pressure rolling/sliding contact in a wide range of operating conditions. This friction regime could be presented as thermally assisted liquid superlubricity.

The stribeck curves obtained for glycerol and the paraffin oil are shown in Fig. 2. The curves span both full film, mixed and boundary lubrication. Vertical lines are inserted at entrainment speeds calculated to give a minimum film thickness 3 times the combined RMS roughness of the specimens. This line is an approximation of the transition between full film and mixed lubrication at a value 3 of the film parameter lambda [1]. Entrainment speeds above this limit gives full film lubrication and entrainment speeds below this limit will lead to more and more asperity interactions. The minimum film thickness was approximated with the Hamrock & Dowson equation [47] and compensated for inlet shear heating with a thermal correction factor for film thickness in circular contacts by the work of Gupta et al. [48] using the lubricant parameters in Table 1.



(a) Paraffin, 40°C, 1.25 GPa

(b) Paraffin, 40°C, 1.95 GPa

(c) Glycerol, 40°C, 1.25 GPa

(d) Glycerol, 40°C, 1.95 GPa

Fig. 2 Stribeck curves comparing glycerol and paraffin oil with uncoated and DLC-coated specimens including standard deviations. Blue vertical lines are inserted at entrainment speeds calculated to give a minimum film thickness three times the combined RMS roughness of the specimens, approximately 130 nm. This line is an approximation of the transition between full film and mixed lubrication at a value three of the film parameter lambda [1]. Higher speeds gives full film lubrication while lower speeds gives increased mixed and boundary lubrication. (Color figure online)

Comparing the paraffin curves with uncoated specimens, black lines in Fig. 2a and b to the glycerol curves with uncoated specimens, black lines in Fig. 2c and d show both similarities and differences. In all tested cases, the high SRR (1.0) yields the highest friction coefficients at the lowest entrainment speeds. This is to be expected since the higher sliding velocities gives harsher conditions in the contact with increased asperity interactions.

At higher entrainment speeds, when the contact moves into mixed and full film lubrication, the paraffin oil shows a transition where the high-SRR cases provides lower friction than the low-SRR cases. This is also expected since the higher SRR leads to increased thermal softening of the lubricant and thus a decrease in viscosity and friction. This can also be seen for the paraffin in Fig. 1 where at a constant entrainment speed and load, an increase in SRR after a certain level decreases the friction.

The same cannot be observed for glycerol. Here, at the lower pressure, Fig. 2c the high SRR gives higher friction in the whole entrainment speed range. This is most likely due to the fact that at this friction and pressure level, thermal softening is not as pronounced. At the higher pressure, Fig. 2d, a crossing point in the friction curves for the uncoated case can be seen at a very high entrainment speed. Here, it is likely that the heat generation in the contact is sufficient to induce significant thermal softening of the lubricant, thus leading to lower friction at high entrainment speeds for the high SRR case.

Distinct differences can also be seen between paraffin and glycerol with the addition of the DLC coating, blue lines in Fig. 2 . While the DLC coating reduces friction significantly for paraffin at higher entrainment speeds, the reduction at lower entrainment speeds dominated by boundary lubrication is very small or negligible. As long as hydrodynamic effects dominates the friction behaviour friction is reduced due to the thermal insulating effects of the coating [8–10]. When the contact moves into boundary lubrication where friction is no longer governed by hydrodynamic effects, the friction reduction is negligible or very small. This is in line with previous studies where mineral oils without additives seem to perform on the same level with a-C:H coated surfaces compared to uncoated steel surfaces [2, 3, 7].

For glycerol on the other hand, the friction reduction in boundary lubrication with the DLC coating is significant. Especially for the low pressure and low SRR case as shown in Fig. 2c the DLC coating keeps the friction at a very low level in the whole entrainment speed range. Previous tests in pure sliding contacts with glycerol and a-C:H coatings are inconclusive in terms of friction reduction compared to uncoated steel surfaces in boundary lubrication. In a reciprocating pin-on-flat disc tribometer at a pressure of 270 MPa, a-C:H-coated specimens showed no better performance than uncoated steel specimens [23]. However, in another study, a Cameron-Plint tribometer with a cylinder on flat configuration in reciprocating movement at a maximum Hertzian pressure of 0.8 GPa showed a significant friction reduction with glycerol and a-C:H-coated specimens compared to glycerol and uncoated steel specimens [56]. It is difficult to draw any conclusions from those tests in relation to the present study, but it may indicate that the combination of glycerol and a-C:H-coated specimens perform better than uncoated steel specimens at GPa-pressure levels. However, both these pure sliding-reciprocating tests were quite different to the rolling/sliding GPa-level tests performed in the present investigation. It has been tentatively proposed following SIMS measurements that glycerol forms a tribo-film on AISI 52100 steel surface in friction testing not exceeding 1 nm in thickness [19]. The authors have ruled out a "self-assembled monolayer" mechanism and are instead proposing what they call "H-bond network" model [24]. This model has been used to explain the low friction achieved with glycerol and tetrahedral amorphous carbon (ta-C) DLC coatings in pure sliding boundary lubrication with even lower friction as a result compared to a-C:H-coated specimens [22–26] Whether this mechanism is responsible for the friction reduction in the present investigation is not known, follow-up analysis involving time-of-flight secondary ion mass spectroscopy (ToF-SIMS) needs to be performed. It is however reasonable to believe that part of the low boundary friction obtained in the present a-C:H-glycerol case is related to the formation of a glycerol-based tribofilm. Lower SRRs and lower contact pressure permits a higher degree of film formation and thus lower friction, which may also explain why the a-C:H-glycerol combination is more effective in lowering friction in the boundary lubrication regime in rolling/sliding contacts compared to pure sliding contacts. Hydrogenated DLC coatings have also shown low friction in boundary lubrication due to a repulsion between the two surfaces [57], but it is doubtful that this has any effect in the highly loaded cases presented here.

Overall, the findings in this study indicates the possibility to achieve ultra-low friction or thermally assisted liquid superlubricity in high pressure, non-conformal contacts in conditions similar to common machine components such as gears, rolling element bearings and cam followers.

However, it should be mentioned that glycerol has many practical drawbacks as a lubricant such as being very hygroscopic, having low Viscosity Index etc.

4 In Conclusion

Rolling/sliding ball on disc friction measurements were conducted at 1.25 and 1.95 GPa with glycerol and paraffin with uncoated and DLC-coated specimens in a wide range of operating conditions spanning both boundary, mixed and full film lubrication. The results show that glycerol provides significantly lower friction than paraffin in most operating conditions with uncoated steel specimens. DLC-coated specimens reduces friction for both fluids in the mixed and full film regime while providing a significant friction reduction also in boundary lubrication for glycerol. The glycerol-DLC combination in some cases gives a friction coefficient below 0.01, often called ultra-low friction or superlubricity in sliding contacts, possibly shown for the first time at such high pressure in a non-conformal rolling/sliding contact. The explanation for the low friction is believed to be a combination of the low pressure-viscosity and high temperature-viscosity sensitivity for glycerol. The low pressure-viscosity sensitivity will lead to low viscosity in the high pressure region of the contact, thus reducing friction. The high temperature-sensitivity will lead to increased thermal reduction of the viscosity in the high pressure region of the contact also contributing to low friction. Finally, the low thermal insulation of the DLC coating will further enhance the thermal viscosity reduction leading to ultra-low friction, or thermally assisted liquid superlubricity.

More work is needed to further understand the low friction behaviour of glycerol and glycerol/DLC combinations in EHL. It would for instance be useful to perform film thickness measurements at pressure levels similar to the 1.25 and 1.95 GPa where friction has been studied in the present manuscript and study the effect of entrainment speed and sliding. It would also be interesting to perform full thermal EHL calculations on both film thickness and friction to in detail investigate the thermal effects. This would require additional work on high-pressure rheology for glycerol. For accurate modeling of friction in glycerol, the reported water formation inside the contact may need to be taken into account. Finally, additional work is required to investigate tribofilm formation on the surfaces in high pressure rolling/sliding contacts with and without coatings.

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