High-speed optical imaging of liquid film flow and liquid macro-slip over free surfaces with different surface energies

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Abstract

High-speed optical imaging is introduced as a visualization method to investigate the film-flow properties of liquids with different surface tensions and viscosities (water, poly-alpha-olefin oil and glycerol) over free rotating surfaces with different surface energies and polarities (steel, DLC and F-DLC). It was found that the polar surface energy strongly influences the structural dynamics of the liquid film's flow and the film's slip. Namely, a decrease in the polar surface energy results in a less stable film with de-wetting areas and breakups into streams, as well as in a larger amount of film slip, which was most clearly expressed by the F-DLC. It was also found that the combination of a high surface tension and a low viscosity provides the largest amount of liquid slip, with the most obvious breakup of the liquid film being observed with water, which clearly exhibits these properties.

Keywords: Liquid slip, surface energy, surface tension, viscosity, high-speed optical imaging

Introduction

Interactions and phenomena at the solid-liquid interface such as wetting and solid-liquid slip have become popular mechanisms for tailoring and designing lubricated contacts [1-14] that are inspired by biomimetic surfaces showing particular functionalities in nature, such as self-cleaning [15, 16], superhydrophobicity [15-21], strong adhesion [15, 16, 20-25], drag reduction [15, 16, 20, 21, 26, 27] and others [15-17, 24].

There are several tribological studies where it was shown that the friction in lubricated contacts can be tailored by solid-liquid interface interactions and phenomena such as surface energy [1-5], wetting [3, 7-14] and solid-liquid slip [1-4, 7, 8, 11-13, 28-31]. It was thus shown that solid-liquid slip can occur on poorly wetted surfaces in low-loaded [7, 11, 13] as well as in high-loaded contacts [2-4, 12, 31]; however, in most cases, model, non-engineering materials were used [2, 4, 29, 31]. In these studies the solid-liquid slip was determined either theoretically [7] or experimentally [12].

However, in recent years several studies reported that slip can also occur on engineering surfaces such as DLC coatings [1, 3, 11, 12]. In addition, studies showed that DLC coatings provide poorer wetting and solid-liquid interactions with lubricants due to a lower surface energy (especially the polar component) compared to a conventionally used material for the contacts, i.e., steel [32, 33]. What is more, recent tribological studies have shown that DLC coatings significantly reduce the friction in the EHD lubrication regime [1, 3, 11, 12, 14, 34-37]; there are, however, two different explanations for this, both of which are dependent on reduced viscous friction. The first one originates from the lower surface energy of the DLC coatings, and with this the associated poorer wetting that induces solid-liquid slip and thus reduces the viscous friction [1, 3, 11, 12, 14]. The other involves the lower thermal

conductivity of DLC coatings compared to a steel surface that induces a temperature rise in the contact and thus a viscosity decrease, which in turn reduces the viscous friction [34-36].

Due to the discrepancies in the origin of the friction reduction and the fact that neither of these two theories has been proven, here we attempted to analyze the slip behavior in the absence of thermal effects. We introduce the high-speed optical imaging of free-flow films to evaluate only the interactions of the solid-liquid interface. This method is not a direct indication of slip and friction reduction in EHD contacts, because it lacks the high-pressure conditions, as well as the high shear, but it provides another insight into the parameters affecting the wetting and potential slip at the solid-liquid interfaces.

1. Experimental

1.1. Materials and liquids

The testing samples, i.e., the wheels used in this study, were made by turning a steel cylinder (DIN 100Cr6/AISI 52100) with an outer diameter of 72.88 ± 0.02 mm. The outer surface of the wheels was further prepared in a sequence of grinding and polishing steps, based on a mechanical treatment only, using abrasive papers to reach the same level of R_a roughness, i.e., 0.04 ± 0.004 μm , measured with a 3D optical interferometer (Contour GT-K0, Bruker) using white-light interferometry. Two of three wheels had DLC coatings deposited on the outer surface of the wheel. The first DLC coating used was a non-doped, hydrogenated DLC coating (denoted as DLC) with a H content of 34 at.% and an $\text{sp}^3/(\text{sp}^3+\text{sp}^2)$ percentage from 35 % to 40 %. The coating was deposited by a radio-frequency (13.56 MHz), plasma-assisted CVD (RF PACVD) process and had a thickness of 0.8 μm . The Si-based, 1.2- μm -thick layer was used as

an adhesion-promoting interlayer. The second DLC coating used was an F-doped, amorphous, hydrogenated DLC coating (denoted as F-DLC) with a thickness of about 0.8 μm . It was deposited by low-frequency, plasma-assisted CVD and contained 8–12 % of F and 36 % of H. The adhesion-promoting interlayer is the same as for the a-C:H coating, i.e., Si-based and 1.2 μm thick. Some additional details about the mechanical properties of the DLC coatings can be found elsewhere [3, 32], since the same coatings were used in previous work. However, these properties are not directly relevant to the current study.

Three different liquids with distinctively different viscosities, polarities and surface tensions were used, i.e., highly polar demineralized water, a moderately polar water mixture of glycerol (90% glycerol + 10% water) and a non-polar, synthetic, poly-alpha-olefin oil (PAO). The main properties of the liquids used in this study (at an operating temperature of 22 ± 2 °C) are summarized in Table 1. A water mixture of glycerol was used to decrease the viscosity to ensure adequate flow during the experiment; this is further denoted as glycerolIM.

1.2. Surface energy

The surface energy of the used surfaces was calculated using the OWRK model [38] by measuring the contact angles at the surfaces, wetted by model liquids with well-defined properties, in particular the surface tension, the polar and the dispersive components. Prior to the above-described, contact-angle measurements for the determination of the surface energy with model liquids, the samples were cleaned with high-purity ethanol, wiped with a cloth and then dried in a stream of hot air. The contact-angle measurements were performed using a contact-angle optical goniometer (CAM 101, KSV Instruments, Helsinki, Finland). For

each contact-angle determination at least 10 measurements were made, and a statistical evaluation was subsequently performed to determine the average value and the standard deviation.

1.3. Experimental setup

A wheel was mounted onto the shaft of an electric motor and was rotated at a constant frequency of 12 Hz, which corresponded to a surface velocity of 2.75 m/s. Liquid was dispensed onto the top of the running surface through a supply system consisting of a reservoir mounted on a pipe with a regulating valve. Due to the different liquid viscosities the valve was adjusted separately for each liquid to ensure a constant liquid flow of 2.5 mL/s. To attain a liquid film of uniform thickness, a horizontal plate was mounted in such a way as to provide a fixed gap relative to the wheel runner's surface of 250 μm , and the liquid was able to move only under the plate's lower edge at a fixed film thickness defined by the gap. The testing apparatus used in the experiment is presented in Figure 1.

The region of interest was focused on a segment of liquid film downstream and just below the plate (Figure 1). The liquid film's flow dynamics was investigated using a high-speed visualization setup. For this purpose, a high-speed camera (Fastec Hispec 4 mono 2G) was used with a 50-mm Nikkor lens set at an aperture of 2.0. In each of 9 different experimental setups (3 surfaces and 3 liquids), 10,000 consecutive grayscale images of 384×226 pixels were acquired at 2000 frames per second and with a 498- μs shutter time. To improve the visibility of the liquid film, the region of interest was illuminated by a LED fitted with a 25-mm lens and covered by opaque glass. Such a lighting setup produced a coaxial beam of diffuse light that made the film-flow structures clearly visible (see Figure 2a for a sample image). To accurately

monitor the rotational frequency of the wheel, reflective markers were added to the edge of its surface for the optical measurements (Figure 1b).

1.4. Image processing and film dynamics

After their acquisition the visualization images were saved to a computer's hard drive for further processing. For the purpose of a qualitative analysis, the images were enhanced by average elimination and color inversion (Figure 2b).

In addition to the qualitative analysis, two different parameters were used to quantify the liquid film's flow properties: (a) liquid slip measurement and (b) flow structure dynamics. The liquid slip (s) was calculated using Equation (1) after measuring the liquid film's velocity (v_f) and the wheel's circumferential velocity (v_0).

$$s = \frac{v_0 - v_f}{v_0} \quad (1)$$

The wheel's circumferential velocity v_0 can be calculated from the rotational frequency f_0 :

$$v_0 = 2\pi f_0 R \quad (2)$$

The rotational frequency f_0 was maintained at 12Hz, leading to a theoretical circumferential speed of 2.75 m/s. However, due to the liquid's friction against the vertical plate and the slight oscillations of the electric motor's voltage, the actual circumferential velocity v_0 of the wheel was slightly different to the desired value and so the value was taken from the actual measured data. To calculate the slip as accurately as possible, the actual value of the wheel's circumferential velocity v_0 was obtained using Equation (2), whereas the rotational frequency

f_0 was determined by measuring the time between the marker's passages in the visualization images.

In the next step, the liquid film's velocity v_f was obtained using a manual correlation method as follows. At each operating point, the vertical position (y) of a clearly visible flow disturbance was taken at two different times, t_0 and t_1 , as marked in Figure 3. Then, the liquid film's velocity v_f was calculated using Equation (3):

$$v_f = K \frac{y(t_1) - y(t_0)}{t_1 - t_0} \quad (3)$$

$$K = \frac{L}{y(t_1) - y(t_0)} \quad (4)$$

In Equation (3) the correction factor $K > 1$ (Equation (4), Figure 3) was introduced to compensate for the effect of the flow-disturbance projection from the curved wheel surface to the vertical viewing plane of the camera. K is defined as the ratio between the actual disturbance path L on the wheel surface, and its vertical projection ($y(t_1) - y(t_0)$).

In addition to the liquid-slip calculation, the flow structural dynamics was assessed with a gray-level analysis. The image's gray level $0 \leq G \leq 1$ was acquired in a profile transversal to the liquid film (Figure 4). The profile consisted of 35 square cells (8×8 pixels) so that the central 31 cells covered the complete width W of the film below the plate. Transversal coordinates were defined both dimensionally (x) as well as non-dimensionally ($p = x/W$). The left- and right-hand edges of the vertical plate correspond to $p = 0$ and $p = 1$, respectively.

In every cell of the profile defined in Figure 4 the mean gray level within the cell was calculated for every individual (i -th) image. This procedure was repeated for 4000 consecutive images, forming 35 time series (one for every profile cell), each 2 seconds long. Let G_m be the 1D time

series for the m -th profile cell. Now, the gray level's standard deviation at the m -th cell, $\sigma_{G,m}$, can be defined as:

$$\sigma_{G,m} = std(G_m) = \sqrt{\frac{1}{N-1} \sum_{i=1}^N \left| G_{m,i} - \frac{1}{N} \sum_{i=1}^N G_{m,i} \right|^2} \quad (5)$$

To eliminate the effect of the non-uniform illumination and the varying reflectivity between the different wheel surfaces, the term $\sigma_{G,m}$ was normalized to $0 \leq \sigma N_{G,m} \leq 1$, Equation (6). The 1D profile of the normalized standard deviation is denoted as σN_G .

$$\sigma N_{G,\mu} = \sigma_{G,m} / \max(\sigma_{G,m}) \quad (6)$$

2. Results

2.1. Surface energy

The surface-energy results are presented in Figure 5. It is clear that the F-DLC coating provides a much lower surface energy, i.e., approximately 25 mJ/m², than the steel and a-C:H. The F-DLC also provides an almost 2-fold lower dispersive component of the surface energy compared to the steel and a-C:H (i.e., approximately 24 mJ/m²), and its polar component is almost negligible, i.e., around 1 mJ/m². On the other hand, the steel and a-C:H coating exhibit a much higher surface energy than the F-DLC, i.e., between 48 mJ/m² and 49 mJ/m². However, these two surfaces are distinguished by their surface-energy components: the a-C:H has a higher dispersive and a lower polar component than the steel surface. The polar component for the a-C:H coating is thus 24 % lower than for the steel surface.

2.2. Liquid slip

Figure 6 shows processed visualization images for different combinations of liquids and wheel surfaces. The dark areas denote flow disturbances such as liquid bulges, air bubbles etc., while the lighter areas present regions with a thin uniform film, or no film (a de-wetted area, i.e., a dry surface). Note that the surface energy in Figure 6 increases in the order F-DLC < DLC < steel, while the liquid surface tension increases in the order PAO < glycerol < water.

In the case of the water film on the steel surface (the highest surface energy) a stable liquid film with some flow disturbances (Figure 6a) and the lowest amount of slip (Figure 7) was formed. In the case when the DLC coating was used, intermittent breaking of the film into individual liquid streams occurred (Figure 6b) as well as an increase in the percentage of slip (Figure 7). When a wheel with the F-DLC coating (the lowest surface energy) was used, there was an even more significant increase in the liquid slip (Figure 7), with substantial film de-wetting so that very little liquid remained on the wheel in the region of interest (Figure 6c). During this particular operating condition, most of the liquid was flung off the wheel surface long before it could form a stable film.

Similar, though less pronounced, differences between the liquid films on the three surfaces were observed for the glycerol solution (Figures 6d-f). Although the slip increases were the lowest on the steel and the highest on the F-DLC (Figure 7), film breakup was only observed on the F-DLC surface. However, a large fraction of the film remained on the wheel (Figure 6f), much more than with the water.

PAO was the only non-polar liquid studied and it is clear that the structure of the PAO film (Figures 6 g-i) was very different to the above-described water and glycerol. Again, the liquid

slip increases with a decrease in the surface energy and the polar component (i.e., from steel towards F-DLC), but most of the liquid flow is concentrated in one or two thick circumferential streams, while the rest of the film is relatively thin (Figures 6g-i). Similar to the glycerol, the PAO film partly disintegrates, but only on the wheel with the F-DLC coating (Figure 6i).

2.3. Structural dynamics of the liquid film

Figure 8 shows the transversal distribution of the gray-level normalized standard deviation (σ_{N_G}), as defined by Equations (5) and (6).

For the water film (Figure 8a), the steel surface results in a relatively uniform distribution of σ_{N_G} in the region directly below the plate, which indicates a uniform flow-disturbance distribution in the transversal direction. A similar σ_{N_G} profile is also seen for the DLC coating, but there is a more pronounced bias towards the right-hand side of the plate. When the F-DLC coating is used, the liquid film breaks into separate streams, which fluctuate around their central positions, reflected in several distinctive peaks in the value of σ_{N_G} .

The σ_{N_G} profiles for the glycerol (Figure 8b) are significantly different from those of the water. All the types of surfaces produce a similar distribution of σ_{N_G} , with relatively small fluctuations at the center of the film ($p = 0.5$) and a more unsteady flow closer to the left- and right-hand edges of the film ($p = 0$ and $p = 1$, respectively). However, the relative amplitude of the σ_{N_G} peaks near the edges seems to increase with a decrease in the polar surface energy (see the F-DLC curve), suggesting that the film-breakup process is more intense in this region.

In the case of the PAO film, the transversal distribution of σ_{N_G} values is highly non-uniform. Distinct peaks in the steel and DLC surfaces (Figure 8c) are consistent with the findings from the images' visual inspection, where thick, isolated streams of liquid were found on the film.

These streams exhibit more disturbances than the surrounding thinner film, resulting in a higher standard deviation of the gray level. Nevertheless, if the F-DLC coating is used, the film intermittently disintegrates into a larger number of smaller streams, resulting in a more complex shape of the σ_{NG} profile with several strong peaks.

3. Discussion

Several aspects, defined mainly by the strength of the solid-liquid interactions, were found to be important for the behavior of the liquid film under dynamic conditions.

(a) The first one relates to liquid slip. As shown in Figure 7, liquid slip was the lowest for the steel, larger for the DLC and the largest for the F-DLC. In this order, the surface energy of the surfaces decreases, especially their polar components (Figure 5). In order to present the role of the surface polarity more clearly, the percentage of slip was plotted as a function of the polar surface energy (Figure 9). It is clear that an increase in the polar surface energy results in a decrease in the percentage of slip for all three liquids used. This can be explained in terms of the permanent polar interactions that are more pronounced on the surfaces with a higher polar surface energy. It has already been reported that more polar surfaces decrease the slip [3], which agrees with the bonding energy of the polar forces being up to 10 times greater than the dispersion forces, leading to a stronger adhesion [39]. A higher polar surface energy thus inhibits the liquid slip.

Furthermore, the difference in slip percentage between the different liquids is less pronounced for the more polar surfaces (note the very similar results for the steel surface and the large differences for the F-DLC in Figure 7). Thus, the slip percentages on the steel surface (polar surface energy of 8.8 mJ/m^2) are 0.19–1.48 % and for DLC surface (polar surface energy of 6.7 mJ/m^2) 1.14–2.81 %. Much larger differences in the percentage of slip between the

different liquids are observed for the F-DLC surface (that has a very low polar surface energy of 1.15 mJ/m^2), i.e., 2.30–13.06 %. The absence of polar interactions, reflected in a very low polar surface energy, thus results in the highest percentage of slip as well as larger differences in the amount of slip between the different liquids. This agrees with several previous studies that showed a decisive role of the polar surface energy in tailoring the wetting of different liquids on various engineering surfaces [32, 33] and also the friction of the lubricated contacts [3, 6, 12].

In addition to the polar surface energy as a solid-surface property, an important role in tailoring the amount of liquid slip is also played by two properties of the liquids, i.e., the surface tension and the viscosity. The results show that the highest amount of slip, regardless of the solid surface used, always occurred with water (1.48–13.10 %), followed by PAO oil (0.40–5.63 %) and glycerol (0.19–2.30 %), Figure 7. This is expected since water exhibits the highest surface tension among the liquids used (Table 1). A higher surface tension means stronger interactions within the liquid and poorer wettability of the solid surface, enhancing the conditions for liquid slip. On the other hand, although glycerol has a much higher surface tension compared to PAO oil (Table 1), PAO oil always exhibits a larger amount of slip than glycerol. This discrepancy in the liquid slip for PAO oil and glycerol based on the surface tension can be explained in terms of the liquid viscosity. Namely, the liquid viscosity (Table 1) shows a significant difference for glycerol and PAO oil: for glycerol it is more than 35-times higher than for the PAO oil, and 200-times higher than for water. This suggests that highly viscous liquids inhibit slip, which has also been reported before [40]. In the case of glycerol, its high viscosity thus prevails over the relatively high surface tension and suppresses the liquid slip, which would otherwise be expected according to its surface-tension property.

(b) The influences of the polar surface energy, the surface tension and the viscosity are also reflected in the structural dynamics of the liquid films. Since PAO oil has the lowest surface tension, its cohesion is the lowest, thus the forces at the wheel (centrifugal, gravitational, spinning, irregularities at the surface, etc.) can cause the largest breakup of the film into smaller streams, as clearly seen through several individual streams (peaks) for PAO in Figure 8c. Based on this, water, with the highest surface tension that keeps the liquid together, should provide the smallest number of separate streams. In fact, there is only one obvious stream (Figure 8a). However, in contradiction with the above explanation, glycerolM has no obvious separate streams (Figure 8b), indicating that film breakup did not occur to the extent anticipated. The reason lies in the very high viscosity of the glycerolM, which can resist the external forces that tend to spread out the fluid film, so providing an extra cohesive effect and thus a more uniform glycerol film. The phenomenon of viscous forces slowing down and reducing the film-breakup process was observed before [41].

However, the dynamic behavior of the films also depends on the adhesion between the liquids and the surface, where wetting plays a crucial role. Accordingly, even though PAO oil has the lowest cohesion, its adhesion to all the surfaces is high compared to water, which was already determined and discussed above in terms of its high slip. Therefore, in spite of the several thin and separate streams of fluid (affected by low cohesion), a larger portion of the surface will be covered by PAO than water due to its better adhesion to the surfaces. In other words, high-surface-tension water will maintain thicker streams, but they cannot remain at the surface due to the poor wetting (high slip) and poor adhesion, so the fluid will be removed from the surfaces, causing de-wetting.

4. Conclusions

High-speed imaging was successfully used to investigate liquid film flow over free surfaces with different surface energies and polarities and to determine the liquid slip.

1. The lowest amount of slip was always observed on the steel surface, which provides the highest surface energy and polarity, resulting in good wetting and solid-liquid interactions. On the other hand, the lowest amount of slip was observed on the F-DLC coating, which provides the lowest surface energy and is almost non-polar, resulting in poor wetting and solid-liquid interactions.

2. Viscosity decreases the slip and increases the wetting of surfaces under dynamic conditions.

3. The largest degree of liquid-film breakup into smaller streams was observed for PAO oil with a low surface tension (due to the lower cohesive forces) that also covers a large portion of the surface with a liquid film due to the better wetting. On the other hand, water, with the highest surface tension, provides fewer individual liquid film streams, but also poorer wetting of the surface with many de-wetted areas.

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Table 1: The properties of the liquids used in the study (at operating temperature of 22 ± 2 °C).

Liquid	Dynamic viscosity, Pa·s	Density, kg/m ³	Surface tension, mN/m
Demineralized water	0.0009	997	72.80
glycerolM	0.2087	1239	64.88
Poly-alpha-olefin	0.0059	798	28.00

Figure 1: Experimental set-up for liquid film visualization: (a) front view, (b) side view.

Figure 2: An example of image enhancement for qualitative analysis (left: raw image; right: processed image). The edge of the vertical plate is visible in the top section of the image.

Figure 3: Flow disturbance vertical displacement and correction factor definition.

Figure 4: Gray level acquisition profile (with cell numbers $m = 1...35$) and the transversal coordinate definition.

Figure 5: Surface energies of used surfaces determined by the OWRK method.

Figure 6: Liquid film topological structure depending on the type of liquid and surface. Dewetted areas are marked with dashed line.

Figure 7: Liquid slip depending on the type of liquid and surface.

Figure 8: Transversal distribution of the gray level normalized standard deviation (σN_G) for different liquids and surfaces.

Figure 9: Percentage of slip as a function of the polar component of the surface energy for all liquids used in the experiments.