Novel In-Situ Observation of the Grease Constituents in Elastohydrodynamic Contacts by Fluorescence Microscopy

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Abstract
Grease is important lubricant for ball bearings as it is used much more often than oil. However, mechanism of the lubrication is not completely clear, especially concerning the role of the thickener in the lubricating process. It was shown that contribution of the thickener to film thickness build-up varies with operating conditions. Also, its influence on the resistive torque of the bearing was proved. However, all studies were in-direct showing its effects. This paper presents new in-situ fluorescence method of the grease constituents’ observation, where both, thickener, and base oil can be observed independently. Two different fluorescence dyes are used to distinguish grease constituents with the use of different microscope setup. Moreover, the new method was tested on two different test rigs. First is typical ball on disc test rig widely used for study of elastohydrodynamic lubrication. Second is ball on ring test rig which more closely represents radial ball bearing conditions. It was shown that thickener engagement is very different for each geometrical configuration.

Keywords EHD · Grease · Thickener · Base oil · Fluorescence

1 Introduction
One of the most typical examples of the elastohydrodynamic (EHD) contact is ball bearing. There are many contacts of this kind in the bearing and to develop better and more efficient bearings or lubricants it is crucial to understand precisely what happens within the contact and in its close surrounding.

One way to study the situation is to describe its behaviour by modelling it theoretically. This method is very often used in case of oil lubrication but has severe limitation in case of grease lubrication as grease has complex rheology which limits its mathematical description. There were some attempts to describe it, but complex satisfactory model was not yet presented.

Another way to study EHD contacts is to simulate them experimentally and observe the behaviour of the lubricant. Most common machine used for this application is ball on disc test rig which was used firstly by Gohar and Cameron [1], Wedeven et. al. [2] And it is often used until nowadays (e.g. 3–10).

Light induced fluorescence method used for observation and evaluation used in this paper was firstly introduced by Smart and Ford [11] who observed the thickness of lubricant film on rotating cylinder. Later, it was pointed out that the use of mercury-lamp-induced fluorescence allows detecting lubricant films down to 30 nm [12]. The principle of the method is based on the intensity of fluorescent emission. It was proved by Azushima [13] that in case of thin films there is a linear dependence between the intensity of emission and thickness of the lubricant layer, so that the intensity can be considered as the non-calibrated film thickness. Fluorescence is currently very frequently used method of thin film observation and evaluation such as study of the oil flow in the bearing done by Chen et.al. [14], Chennaoui et.al. [15] or by Liang et.al. [16, 17].

Unlike theoretical modelling, experimental approach can be also used in the case of grease lubrication, and it is very important because roughly 90% of all operating bearings are lubricated with grease [18]. Grease consists of two main constituents—base oil and the thickener. There is usually also small percentage of the additives, but these two main components are most important from the point of the lubrication. Behaviour and contribution of the oil is already
known and was described extensively such as in review of
the EHL theory provided by Lugt et al. [19].

Behaviour of the grease in the EHD contact was also
studied in detail. Lugt presented complex review on the
grease lubrication [20]. More recent studies are dealing
for example with grease replenishment as studied by Cen
and Lugt [21, 22]. They defined most important parameters
influencing the replenishment performance. Replenishment
process was also modelled with use of CFD by Fisher et al.
[23]. They presented importance of the side reservoirs and
their position. Behaviour of the grease in the ball on ring
configuration was studied by Chen et al. [24, 25]. They pre-
sented migration of the grease in the bearing-like configu-
ration. Liang et al. [26] used the same device to observe
migration of the lubricant in the bearing and influence of
the centrifugal forces [27]. Sakai et al. studied influence on
the thickener type on the lubricant film thickness [28] and
on bearing torque [29]. Similar research was also done by
Cyriac et al. [30], who studied thickener particle geometry
and its influence on the film thickness. Entrainment of the
thickener particles in the EHD contacts at low speed was
observed by Cen et al. [18]. They presented typical V-shape
curve, where film thickness tends to increase with decrease
of the speed from some point.

Grease has been studied vastly in the past decade, how-
ever, in most studies, presence of the thickener in the grease
at certain points in the contacts and its surrounding is proved
only indirectly by the change of the thickness or the torque
(friction). Grease was considered usually as one-constitu-
ent compound when fluorescence was used in past studies
because only oil is usually dyed. Therefore, only oil can be
observed.

Aim of the presented study is to provide direct observa-
tion of the thickener in the contact inlet region and contact
itself.

2 Method

Only experimental method were used in this study. Contact
simulators are based on ball on disc type and light induced
fluorescence microscopy is used to observe thickener
behaviour.

2.1 Test Rigs—Simulators

Two different simulators have been used within this study.
Both of them provides unique possibilities. First test rig is
typical ball on disc used thoroughly in the history of EHD
research while second is Ball on ring type which is new
design and provides new possibilities of study due to its
configuration. Both are described separately.
bearing 6314. This deep groove ball bearing was selected based on the diameter of the ball, which is 25.4 mm—same as commonly used sample in ball on disc. Therefore, ball has diameter of 25.4 mm and is made of 100Cr6 steel. Ring is made of BK7 borosilicate glass and has inner groove with diameter 26.4 mm. Combination of these two bodies provides conformity of 0.52 which is similar to real bearing. Ellipticity of the created contact is 8. Test rig can be also equipped with cage, however, it was not used within this study to not interfere in the comparison with ball on disc (it has no cage). Ring is driven by servo drive. Ball on the test rig is supported by two shafts that are shaped in the way to not interfere with the lubricant layer on the ball. They are supporting the ball mainly on the sides and enabling free rotation. It was also checked for sliding and none was measured, so the contact operates under assumption of pure rolling. Load is created similarly as on ball on disc—dead weight in combination with lever.

2.2 Fluorescence Microscopy

Observation method used in this paper is fluorescence induced microscopy. It was used because of its properties that enables to separately observe specific compounds. Simple scheme is shown in Fig. 3. Monochromatic LED or mercury lamp was used to illuminate the sample. Specific excitation filter to isolate the desired wavelengths was used to excite the sample in the simulator. Excited lubricant is due to stoke’s shift emitting light of slightly longer wavelength that is transmitted via dichroic mirror and isolated by emission filter. Only this light is afterwards captured by 16-bit monochromatic sCMOS camera. Sample is using two different dyes, therefore filter-set and illumination needs to be changed accordingly to observe each dye. Specific parameters are listed in Table 1.

### Table 1 Fluorophores parameters

| Fluorophore | Pyrene | Coumarin 6 |
|-------------|--------|------------|
| Excitation peak | 365 nm | 415 nm |
| Emission | 471 nm | 495 nm |
| Excitation filter | 350/50 nm | 420/40 nm |
| Mirror | 387 nm | 455 nm |
| Emission filter | 460/50 nm | 465lp nm |

2.3 Grease Sample Composition

One grease was prepared for this study to achieve separate observation of the thickener and base oil. General multipurpose grease with Lithium complex thickener and PAO8 base oil was prepared. PAO oil was selected as it has no fluorescence properties, therefore only artificially added dyes can be observed without interference from other compounds. Viscosity of this base oil is 46.2 cSt at 40 deg C. NLGI grade of the produced grease is 2.

Two dyes were used within this grease—Coumarin and Pyrene. Coumarin was used to track thickener and pyrene to observe oil. Both dyes are not purely attached to only one grease constituent, but there is preferable one. Coumarin can be found mostly on the thickener, but weak signal comes also from the oil. Contrary, pyrene is mostly found in the oil but partially also on the thickener. Ratio is explained in greater detail in Sect. 2.5. Content of the coumarin 6 was 200 ppm and pyrene 1 mass %. Behaviour and spectra based on the concentration of the pyrene was already studied and presented previously by Kostal et al. [31].

Both dyes were dissolved in the PAO base oil at elevated temperature before the production of the grease. After complete dissolve, the grease was formulated in the ordinary way. It was observed that dyes are separated to grease constituents as explained before.

2.4 Fluorescence Setup

Fluorescence setup enabling separate observation consists of different filter sets on the microscope and different illumination. Pyrene need to be excited in the UV part of the spectra, while slightly longer wavelength can be used for Coumarin as can be seen in Fig. 4. Parameters of the light and filters are listed in Table 1. Due to slight overlap of the spectra, only qualitative observation is currently possible, however, calibration attempt was done as explained later.
2.5 Calibration, Verification

Glass ring mentioned in the previous chapter can create optical distortion as it is curved on the outer and inner surface. Therefore, verification measurements were done to find if any of the distortions is significant to the obtained images. Steel ball with specific roughness was selected and picture of the roughness taken. The same ball was inserted in the contact with glass ring as it was in EHD contact. Both images were compared from the point of mutual differences in the positions of roughness features (grooves). No significant distortion was measured. Tested surface is shown in Fig. 5.

Another series of tests was done to prove what is the ratio of the fluorescence emission from coumarin and pyrene, e.g. from thickener and from oil. This is rather complicated to found exactly, but quantitative results were obtained. First, sandwich setup with 50 μm gap described in previous paper [31] was used to measure original sample of the grease with composition of 86% of oil vs. 14% of thickener. This grease was already augmented with 1% of pyrene and 0.02% of coumarin. Results from this part are shown in Fig. 6a, b. Image a is with pyrene’s emission, while b is coumarin’s emission. Given different concentrations and different strength of the dyes, signal from pyrene was amplified roughly by 4.8 times to obtain similar strength of the signal as of pyrene. This is considered as calibration, because sum of the fluorescence intensity obtained from pyrene and coumarin together is now composed from 84% from pyrene and 16% from coumarin—equal to composition of the grease. Same amplification of the pyrene’s emission was done in further images.

Next experiment was done in the same sandwich setup, but without precision shim separating surfaces so that lubricant was strongly squeezed in narrow gap between glass surfaces. Lubricant flowing in the gap was observed to be mostly oil. Several areas on the edge of the sample in the fluorescence images such as marked in Fig. 6c, d, where only oil was found were investigated. This was used to verify the separation of the fluorescence dyes together with grease constituents. Hypothesis is that only pyrene should be observed in the bled oil as coumarin is attached to thickener which remains closely to the centre during squeeze. Emission from this test showed that fluorescence from coumarin was only 5% from total signal in average in the selected areas. Remaining 95% is from pyrene.
Last experiment was done to obtain signal from thickener only. Results can be seen in last pair of images Fig. 6e, f, where thickener-rich area was selected and both emission from pyrene and coumarin were recorded. Several places similar to this one were measured and average value is that emission from pyrene is 65%, while 35% is from coumarin. This does not mean that this area is covered solely by thickener, however it is evidence that this area is richer for the thickener than the average grease shown in Fig. 6a, b. It was expected that it will be found also pyrene’s emission in this area, and it is hard to estimate the percentage of thickener/oil as thickener is porous and can contain a lot of oil in its structure. However, results observed during this test can be close to realistic distribution of the grease.

Tests described above proved that coumarin is almost purely attached to the thickener as it is not found in bled oil. Only around 5% of its emission remains in the oil. Areas rich for the thickener on the other hand showed stronger emission from coumarin and weaker from pyrene which proves that pyrene is distributed mostly in the oil. This method can be used to calibrate quantitative measurements in the future research.
Image shown in Fig. 7 shows the real fluorescence of the individual dyes dissolved in the grease captured in static EHD elliptical contact. Colours on the images correspond to the real emission of the individual dyes. These images were captured by colour DSLR camera to see the difference. Main difference between individual images can be seen especially in the central area of the Hertzian contact. Lubricant film in this area in compressed between surfaces and oil tends to leak out while thickener attached to the surfaces remains. It is observed that grease rich for the thickener remains in this area. Spot marked with the arrow on the top part of Fig. 7 is showing dark areas in the centre, which means that there is the least thickness of the pyrene (base oil). Contrary, on the bottom part of Fig. 7 can be seen strong fluorescence in the same region. This emission comes from the coumarin (thickener) which represents higher thickness of the thickener in the region.

Behaviour of the thickener was observed to be very different when results from two test rigs mentioned in the method were compared. Results from two test rigs are presented further separately—ball on disc test rig provides circular contact, while ball on ring provides elliptical contact, while both devices are using same rolling element. Fluorescence intensity is translated to colour model “Fire”, where darker area represents thin film while bright white areas represent thicker films.

All results presented from this point further were captured as movies and processed to individual figures or sequences. Original movie is attached as supplementary material to the paper and accessible online. Authors recommend viewing the video as the behaviour is much more obvious from it than from the figures.

### 3.1 Ball on Disc

Behaviour of the fresh and worn-out grease can be studied simply on the ball on disc test rig. Because fluorescence provides limited light, direct observation is limited to low speed. Further images were captured at 0.2 mm/s with framerate 40 fps. Field of the view for shown image is 1.69 mm horizontally and 1.41 mm vertically. Contact pressure was 0.5 GPa.

Passage of the bigger thickener particle from the fresh of-shelve grease is shown in Fig. 8 on individual frames selected from the recording. Specific particle can be traced as it passes through the contact pressure area—one selected particle is enclosed by the dashed white circle as it is squeezed by the pressure of EHD contact. Method in this stage provides direct observation of the milling bigger chunks of thickener particles from the fresh grease to the smaller ones.

Another effect observed during the experiment is rapid backflow, where mixture of oil and thickener rapidly changes velocity vector and avoids contact pressure in the inlet region. One of the experimental results was evaluated with particle image velocimetry (PIV) method to obtain velocity vectors and speeds. PIV algorithm used for evaluation was in MATLAB toolbox called PIVLab developed by Thielicke and Sonntag [32]. Results are presented in Fig. 9 where there is original fluorescence image at the top and velocity magnitude and direction in the bottom for the same situation. Shown speed is equal to the speed components in rolling direction. Backflow up to 0.8 mm/s was observed for rolling speed of 0.2 mm/s.

Completely different picture can be seen when grease is slightly more worn-out—this was achieved by several hundreds of revolutions on ball on disc at speed 1 m/s. Speed was decreased again to 0.2 mm/s seconds and conditions shown...
in Fig. 10 were observed. Depleted rolling track is visible in the central region, while inlet region includes meniscus. However, meniscus clearly consists of pure oil as no particles of thickener can be observed. Small particles of the thickener can be traced in Fig. 10b, d—marked with white dashed ellipse. They are being dragged from the top side reservoir and are passing through the contact thus helping to support film in the contact, although their content is rare compared to previous Fig. 8.

Meanwhile, some particles are already squeezed onto the rolling surfaces and are passing through the contact without any change of the trajectory or shape. This situation is designated by one example particle group in Fig. 10 in part d-f, marked with dashed white circle.
3.2 Ball on Ring

Results from ball on ring test rig are presented here to be compared with ball on disc. First, similar situation with fresh grease is shown. Contact is due to the geometry very wide with ellipticity around 8. Wider contact is also larger, therefore smaller magnification of the objective was used (5 × compared to 2 ×). Field of view is expanded compared to ball on disc results—8.3 mm horizontally and 7.0 mm vertically. Contact pressure was kept constant at 0.5 GPa to be comparable to ball on disc. Speed at which images were captured was 1.42 mm/s due to the limitation of the test rig, which is designed rather for higher speeds.

Passage of the rolling element through new layer of fresh grease looks very similar to ball on disc. Only thickener particles are shown smaller due to larger scale but lubricant is the same.

One particle is traced in Fig. 11 and in Fig. 12 zoomed. It can be clearly seen that particle is being compressed and its size is increasing during entrainment into contact zone.

Same condition as in the case of ball on disc was also tested—several hundred revolutions at 1 m/s was done to create starved condition and to worn-out the grease. After this fast rolling, speed was again decreased to observe behaviour of the thickener. It was observed that inlet meniscus rich for thickener particles was formed almost immediately after slowing down. Lubrication of observed contact was provided in combination with oil and also thickener with much larger scale compared to ball on disc (Fig. 13).

All the pictures above were captured by observation of the coumarin, thus mostly emission from the thickener. To compare the same situation with the emission from the pyrene thus oil Fig. 14 is shown. It can be clearly seen that gap around the contact is much more homogeneously filled with oil (upper image) than with thickener particles (bottom image). Also, the track has weaker signal in case of pyrene, that would suggest that track is covered with more thickener than oil.

4 Discussion

Comparison between the results of the starved lubrication captured with the same approach on the ball on disc and ball on ring shows different results. Both results side by side can be seen in Fig. 15. Much stronger signal from the inlet meniscus means that lubricant present in this area is much richer for thickener. Reason of the thickener presence can be in the difference of geometrical configurations of these two test rigs. Ball on disc provide flat to ball surface with low conformity. Conformity in the case of ball on ring is much better as it was designed specifically to be like real bearing. Capillary forces acting in the diverging gap in the direction perpendicular to rolling direction are much stronger and capable of replenishing the lubricant from further distance from the contact. In contact replenishment is stronger in this case.
Another reason is that ball on disc test rig has fixed rotational axis for both bodies. Therefore, depleted track created in the lubricant layer cannot change spontaneously. On the other hand, ball in the ball on ring test rig is freely rotated on the supports and there can be slight spin possible. It can also cause better replenishment as fresh lubricant might be attracted to contact with each over-rolling.

Further difference is in the direction of the inertial forces. In both cases, surfaces were rotated at high velocity (1 m/s) for certain time. Inertial forces are already quite important at this velocity. However, much more favourable case from the point of replenishment due to inertial forces is in the case of ball on ring. Inertial forces are pushing lubricant (including thickener) to the central area of the rolling track due
Fig. 11  Thickener on ball on ring—fully flooded
to its circular shape. This can also help to achieve flooded conditions.

There is also some restriction to the method used in this study. Filter setup used to observe grease constituents was found to be optimal, although not ideal. It was found that during observation of the coumarin, very little pyrene emission can be included. However, when observing pyrene significant portion of coumarin emission can be included in the signal. This is also due to the fact that coumarin emission is much stronger than pyrene’s. It was measured to be roughly 10–20x stronger depending on the concentration and properties of the lubricant. This effect needs to be overcome to allow quantitative description of the grease constituents and this will be part of future work.

5 Conclusions

Conclusion from this paper can be drawn in two ways. First is the benefit of the new method of observation—method was developed to be able to distinguish grease constituents during in-situ observation of EHD contacts. The method proved to be useful and provides unique point of view on the grease lubrication with great possibilities for further research as it enables to observe behaviour of the thickener and base oil separately and to measure thickener concentration in the film.

Second point is that this new method was used to observe behaviour of the grease and its component in the two different geometrical configurations. First is typical ball on disc vastly used throughout the history and second is newly designed ball on ring that should more closely represents radial deep groove ball bearings. Method proved that behaviour of the thickener is different in both cases. Much more thickener is being dragged into the contact and is passing through pressure zone in case of ball on ring than in case of ball on disc. Therefore, conformity of the contact must be considered carefully whenever are tribometer results used to describe behaviour of the bearing.
Fig. 13  Thickener on ball on ring—starved
Fig. 14 Comparison between signal from oil (top) and thickener (bottom)

Fig. 15 Comparison of thickener amount for starved contact on ball on disc (top) and ball on ring (bottom)
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Declarations

Conflict of Interest  The authors have no relevant financial or non-financial interests to disclose.

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