MAPPING OF FILM THICKNESS IN BOVINE SERUM LUBRICATED CONTACTS

Martin Vrbka*, Tomáš Návrat*, Ivan Křupka*, Martin Hartl*, Jiří Gallo**

The aim of this study is to perform detail experimental mapping of the lubricating film thickness of bovine serum (BS) within the contact between an artificial metal or ceramic femoral head and a glass disc and analyze effect of proteins on the film formation. Mapping of the lubricating film of various concentrations of BS solutions was carried out using an optical test rig. Chromatic interferograms were recorded with a high-speed digital camera and evaluated with thin film colorimetric interferometry. The film thickness was studied as a function of both time and mean speed. The results showed that film thickness increases with time for both the metal and ceramic heads. Films formed at the end of measurements with the metal head were found to be typically in the range of 60–100 nm for all BS solutions and were independent on the amount of proteins in tested fluids. At the beginning of the speed measurements, BS of all concentrations forms a very thin film (1–2 nm) and its thickness increases with increasing mean speed. However, when the speed was decreased, the film thickness did not reduce but increased with decreasing speeds that supports the findings of other researchers. Moreover, it was found that BS supply is sensitive parameter. When the lubricant reservoir below tested head was used then the measured central film thicknesses achieved values only about 20 nm, whereas when the tests were realized without the reservoir, measured central film thicknesses achieved higher values about 100 nm. For both types of the experiments, distribution of the film thickness within the contact zone is not homogeneous and two different film thickness regions can be found; thicker protein film and thinner base film that both increase with time and speed.

Keywords: *artificial hip joint, femoral head, bovine serum, protein formation, lubrication, film thickness, colorimetric interferometry*

1. Introduction

Total hip arthoplasty (THA) is the most effective method for treating severe degenerative, post-traumatic and other diseases of the hip joint. It is estimated that more than 1,000,000 THAs are performed each year globally. More importantly, modelled future projections expect further increase in the need for THAs [1]. There is believed that THA can reliably relieve pain and improve function in the majority of patients for a period of 15 to 20 years or more postoperatively. However, the Kaplan-Meier ten-year revision-free survival estimates for younger patients range from 72 % (95%CI: 67–76) in Finland to 86 % (95%CI: 84.5–88.2) in Sweden [2]. Hence, 14 % to 28 % of such patients on average did not achieve a 10-year THA functioning without revision. The main reason for late failure of THA is aseptic loosening

* doc. Ing. M. Vrbka, Ph.D., Ing. T. Návrat, Ph.D., prof. Ing. I. Křupka, Ph.D., prof. Ing. M. Hartl, Ph.D., NETME – New Technologies for Mechanical Engineering & Faculty of Mechanical Engineering, Brno University of Technology, Czech Republic

** doc. MUDr. J. Gallo, Ph.D., Orthopaedic Clinic, University Hospital Olomouc, Czech Republic

followed by instability of the THA that compromises predominantly the early postoperative period [3].

Aseptic loosening is hypothesised to be the result of a harmful combination of mechanical and biological events destroying the bond between the implant and bone bed. Related to the biological mechanisms the most prominent role plays so-called particle disease associating generation of very small prosthetic particles with chronic periprosthetic inflammation and increased accumulation of osteoclasts at the implant-bone interface both leading eventually to predominance of bone resorption over bone formation.

As a result, a continual production of wear particles is a key problem of THA and its diminishing is of paramount clinical importance. Principally, wear is a conditional functional parameter of articulating bearing surfaces, and not a simple function of bearing biomaterials used for their manufacturing [4–12]. Therefore, the resulting wear rate could be influenced at least partially by modification of tribological parameters. Of them the most important is lubrication regime influencing directly on the coefficient of friction. At present bovine serum (BS) is commonly used as a model of synovial fluid (SF) lubricant for interpretation of wear and friction properties of artificial joints. As well BS film thickness measurements could be expected to provide information about lubrication mechanisms during joint articulation, but at present, there is limited number of publications [13–16].

Mavraki and Cann [13] carried out friction and film thickness measurements on a series of simple protein solutions (simulating SF) and BS under quasi-steady operating conditions. The friction results, performed on the Mini Traction Machine, showed that BS and simple protein solutions demonstrate boundary properties by reducing friction in the slow-speed regime. The central film thickness of the lubricants was measured in a ball-on-disc device under constant load and pure rolling conditions using thin film optical interferometry. The film thickness results with BS showed that BS film thickness initially increased with increasing speed over the range of 5–20 nm. In subsequent speed sweeps, the film thickness became almost independent of speed, although a slight tendency to increase at low speeds was observed (28 nm at 5 mm/s). Moreover approximately 10–18 nm thick residual film was measured at the end of the test. The film thickness results suggest that a thin, solid layer is formed; this could be adsorbed protein or deposited degraded protein.

In the study [14] the same authors analyzed lubricant central film thickness behaviour of BS using ball-on-disc device for various operational conditions. They found that under high pressure rolling tests, BS initially formed thinner films (5–50 nm) over the speed range and in subsequent speed sweeps thicker films (up to 100 nm) were formed at low speeds. This behaviour was imputed to formation of an adsorbed protein layer, which causes high-viscosity film. At the end of each test residual boundary films of 9–19 nm were measured under static loading. Under pure sliding in the high pressure rolling tests, results resulted in much thinner films than obtained under pure rolling conditions. Typically film thickness dropped 70–80 % (from 20–30 nm to less than 5 nm) with sliding. The results at low pressure and pure sliding showed that much thicker films (60–80 nm) were formed over the same speed range and again thicker films were formed at the lower speeds.

Fan et al. [15] studied lubricant film formation with model SF components (proteins) and BS in a sliding test device. The objective was to investigate the role of proteins in the lubrication process. Film thickness measurements at the centre of the contact were made over a speed range of 2–60 mm/s using CoCrMo femoral head as a stationary counterface.

The results for BS showed complex time-dependent behaviour, which was not representative of a simple fluid. After a few minutes sliding BS formed a thin adherent film of 10–20 nm, which was attributed to protein absorbance at the surface. This layer was augmented by a hydrodynamic film, which often increased at slow speeds. At the end of the test deposited surface layers of 20–50 nm were measured. The authors suppose that film formation is dominated by surface deposition and shear-flow aggregation of protein molecules. Protein molecules are aggregated in the inlet shear field and come out of solution to form gel-like deposits in the inlet; this material adheres to the metal surface and periodically passes through the contact forming a much thicker hydrodynamic film. The authors also note fact that protein deposits are formed in the inlet of the contact in the low pressure region.

Myant et al. [16] performed measurements of central film thickness by optical interferometry as a function of time (constant mean speed 0 and 10 mm/s) and variable mean speed (0–50 mm/s) for series of BS and protein-containing (albumin, globulin) saline solutions and for CoCrMo femoral component sliding against a glass disc. The effect of load (5–20 N) on film thickness was also studied. The results showed film thickness increased with time for both the static and sliding tests. In the sliding tests a wear scar rapidly formed on the implant component for the BS and albumin fluids, negligible wear was observed for the globulin solutions. The authors point out that the film thickness decreased rapidly with increasing load for all fluids and also supported the idea of the protein-aggregation lubricated mechanism introduced in [15].

From the cited literature it is apparent that proteins play an important role in the film-forming process. This paper should contribute to amend of research findings presented in the current studies that are mainly focused on experimental analyses of central film thickness in the contact zone. That is why the aim of this study is to perform detail experimental mapping of lubricating film thickness of BS within a whole contact zone between artificial metal or ceramic femoral head and glass disc and analyzed effect of proteins on film formation as a function of time and speed.

2. Experimental method

Mapping of lubricating film of several BS concentrations was observed using an optical test rig (Fig. 1) in which a circular contact is formed between a glass disc and a metal or ceramic head of total hip joint replacement (Fig. 2). BS (Sigma-Aldrich B9433, protein concentration 75.3 mg/ml) and sterile water were used for preparing samples with appropriate w/w concentrations. BS concentrations of 8.3, 25, and 100 per cent with a total protein content of 6, 18, and 75 mg/ml were prepared in volumes of 10 ml and immediately stored in a freezer at −20 °C. The lower surface of the disc was coated with a thin semi-reflective chromium layer and the upper side had an antireflective coating. The both artificial femoral heads AESCULAP NK430K made from Cobalt-Chromium forged alloy (CoCr29Mo / ISO 5832-12) and AESCULAP NK461 made from Aluminum oxide ceramic (Al_2O_3 / ISO 6474) had 28 mm in diameter and were delivered from original package of manufacturer. The artificial heads were rotated by servomotor against the disc to provide pure rolling conditions. The film thickness was studied initially as a function of time within 10 minutes at constant mean speed of 20 mm/s and then as a function of mean speed over a both increasing (labelled UP) and decreasing (labelled DOWN) speed range of 5–40 mm/s (Fig. 3). The residual film thickness was also measured at the end of each test at zero speed.

Experiments were realized at room temperature of 24 °C under steady state load of 5 N corresponding to mean Hertzian pressure of 180 and 190 MPa for metal and ceramic head, respectively. All components which are in the contact with BS (e.g. glass disc, femoral head, shaft, bearings) were cleaned in 1 % w/w sodium dodecyl sulphate, rinsed in distilled water, and then washed in isopropyl alcohol before assembly. This cleaning process was carried out every time between individual measurements. Test fluids were taken out from freezer two hours before measurements and then were supplied to the contact zone through syringe coupled with needle for period of 70 s. A new sample of test fluid was used for each of the measurements. The contact formed between the glass disc and the artificial heads was illuminated by xenon or halogen lamp. Obtained chromatic interferograms were recorded with a high-speed digital camera (CMOS or 3CCD) and evaluated with thin film colorimetric interferometry (Fig. 2). Detailed description of this technique is given in [17–18] and application in [19].

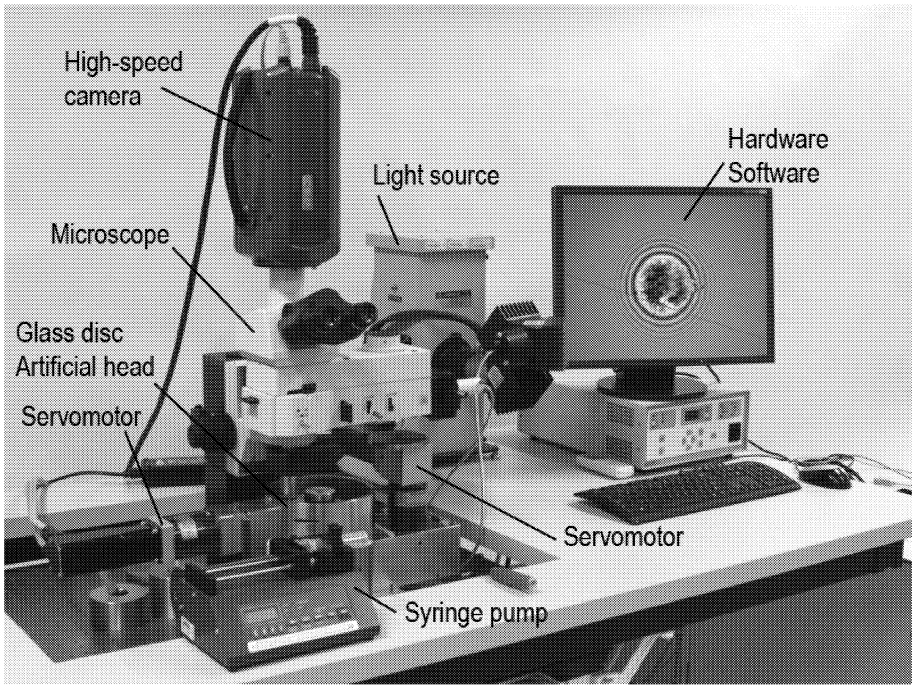

Fig.1: Optical test rig for lubricant film thickness measurements

3. Results and discussion

3.1. Film thickness as a function of time

At first the film thickness was studied as a function of time within 10 minutes at constant mean speed of 20 mm/s for metal artificial head and for concentrations of BS 8.3 %, 25 % and 100 %. During these experiments, the test fluid passing through the contact zone was trapped in a reservoir placed below tested head (Fig. 2) so that the trapped lubricant was partially returned by the head again to the contact. Radial clearance between the reservoir and head was 1 mm. The contact zone was recorded with a high-speed CMOS digital camera with a frequency 24 Hz during 10 minutes. During this time a number of 14,400 interferograms

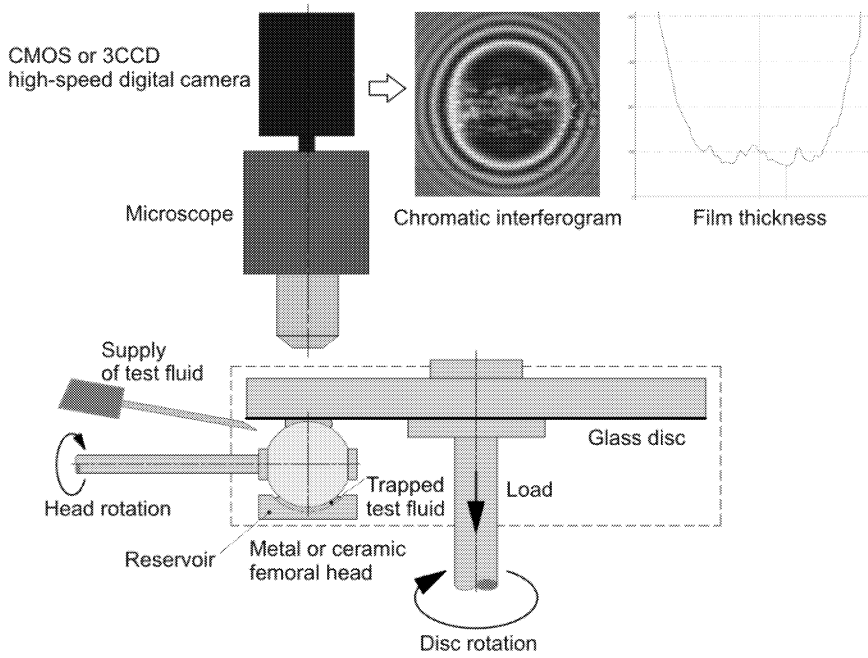

Fig.2: Schematic diagram of film thickness mapping

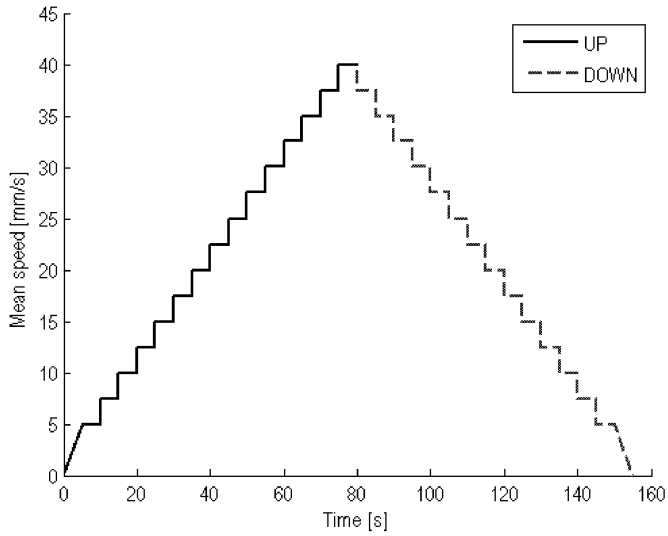

Fig.3: Increasing (labelled UP) and decreasing (labelled DOWN) distribution of mean speed in dependence on time applied for measurement of film thickness as a function of speed at constant load

were recorded for each BS concentration. From each performed measurement number of 21 interferograms (going at intervals of 30 seconds) was selected and then the film thickness was evaluated. Film thicknesses measurements as a function of time for all BS concentrations are displayed in the Figs. 4–7. Individual points in graphs in the Figs. 4–7 correspond to

average film thickness from central area of the contact zone and marked regions represent minimal and maximal film thickness within the contact zone.

From the graphs displayed in Figs. 4–7 it is possible to observe that central film thickness is increased with time and distribution of the film thickness in the contact is not uniform due to great differences between minimal, maximal and central film thickness values. At the end of measurement (at 10 minutes) the central film thicknesses achieve values 103 nm, 60 nm and 70 nm for BS concentrations 8.3 %, 25 % and 100 %, respectively. Formation of the lubricant film thickness for all tested BS concentrations is quite similar and is not influenced by the amount of proteins in tested fluids.

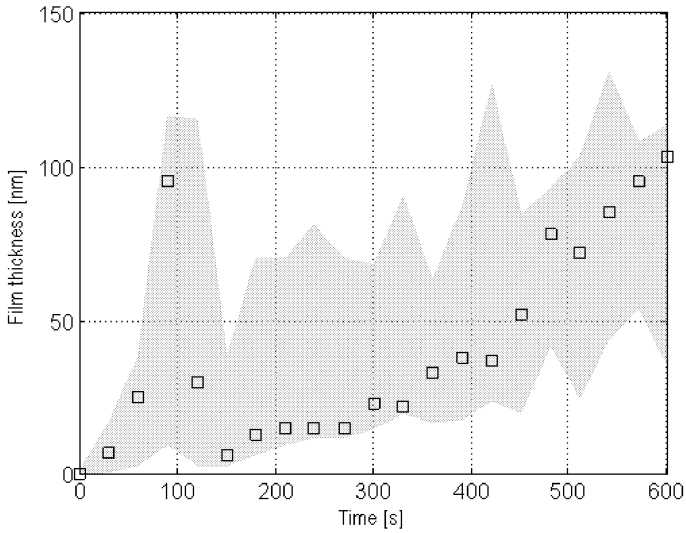

Fig.4: Film thickness plotted as a function of time for BS concentration of 8.3 %

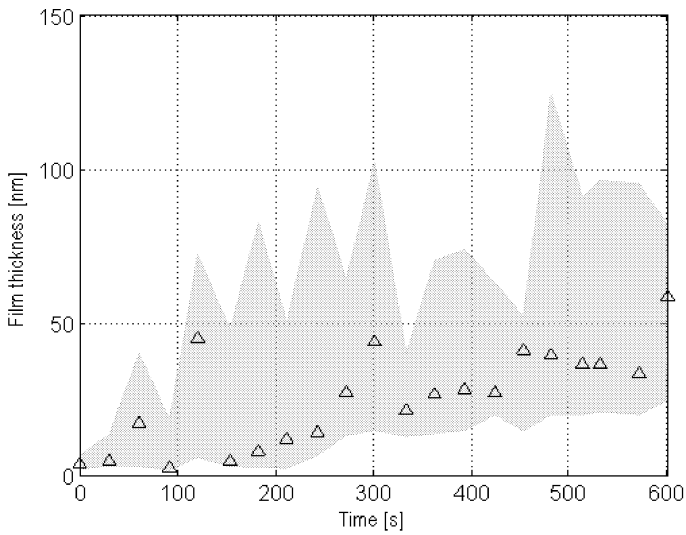

Fig.5: Film thickness plotted as a function of time for BS concentration of 25 %

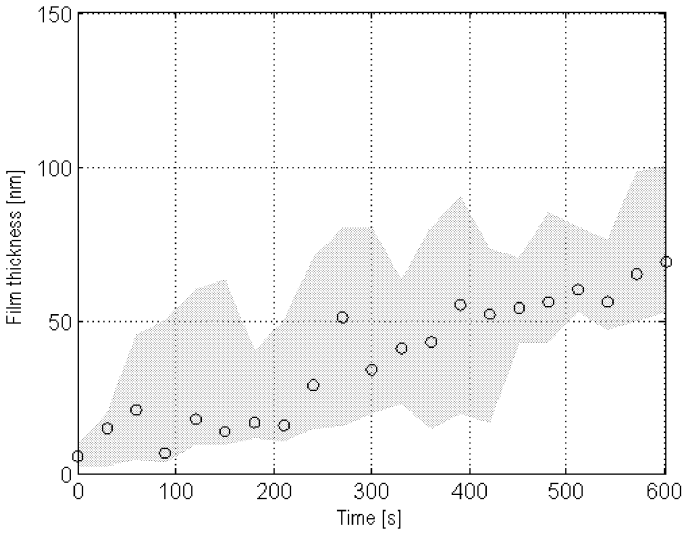

Fig.6: Film thickness plotted as a function of time for BS concentration of 100 %

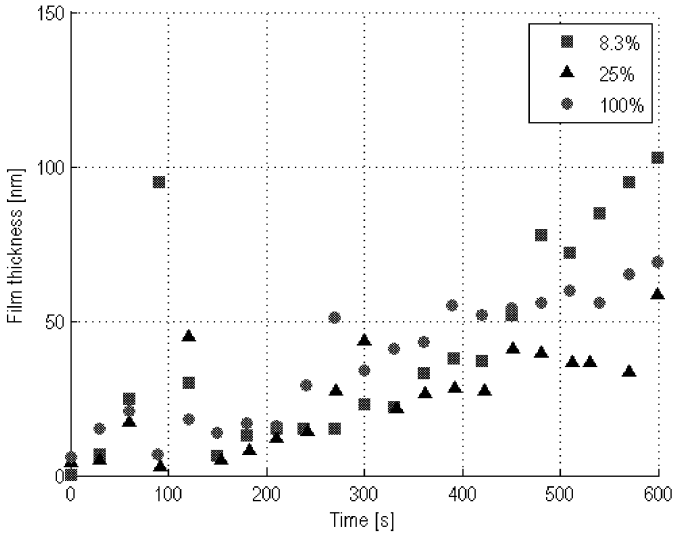

Fig.7: Film thicknesses plotted as a function of time for BS concentrations of 8.3 %, 25 % and 100 %

Formations and evolutions of the film thickness in the whole contact zone in dependence on time are illustrated by interferograms displayed in Fig. 8 for the metal head and BS concentration of 8.3 %. Figure 9 shows the distribution of the film thickness in the direction of rolling evaluated from interferograms recorded in time 0 s, 60 s and 570 s. At the beginning (Figs. 8a, 9-section1) very thin film with thickness about 1 nm was measured, however at 60 s (Figs. 8b, 9-section2) the film thickness increased in the high pressure contact zone only. Afterwards fairly thick film subsequently enlarged to the whole contact zone (Figs. 8e–f, 9-section3). From these interferograms some spots with very thick film can be observed. Moreover these spots are randomly distributed and thereby cause the variability in the film thickness distribution.

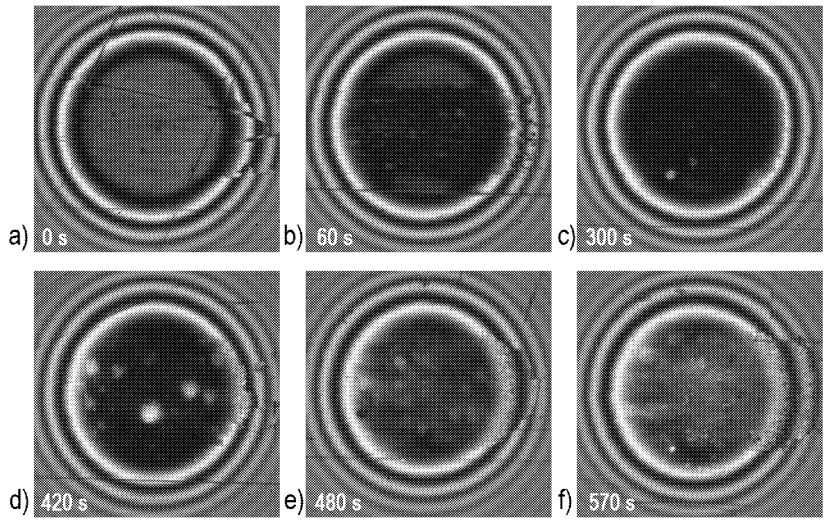

Fig.8: Chromatic interferograms recorded within 10 minutes and corresponding to metal head and concentration of BS 8.3 %

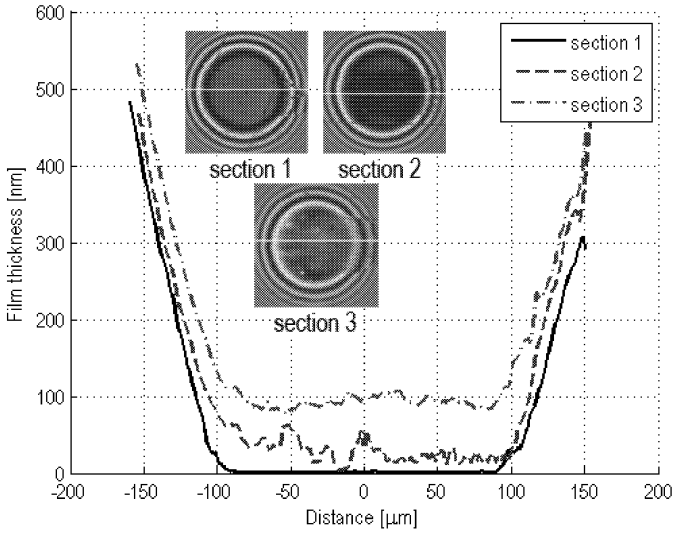

Fig.9: Distribution of the film thickness in direction of rolling evaluated from interferograms recorded in time 0 s (Fig. 8a), 60 s (Fig. 8b) and 570 s (Fig. 8f)

The variable formation of lubricant film thickness can be also observed from selected interferograms recorded in the sequence with the time step of 0.042 s for all BS concentrations (see Figs. 10, 12 and 13). Interferogram in the Fig. 10a contains several spots with very thick lubricating film. However, such spots can be hardly observed in consecutive interferograms (Figs. 10b, c). This random process of the formation of such spots was observed during all measurements. It is evident that measured film is not homogeneous so that two different film thickness regions can be found within the contact zone; thicker protein film and thinner base film and both increase over the time (see Figs. 4–6). For better clarity of the graph, Fig. 11 shows differences in distribution of the film thickness in direction of rolling evaluated

from both interferograms displayed in the Figs. 10a and 10c. During the test the fairly thick protein film was measured up to 200 nm (Fig. 11).

The mapping of lubricant film was also performed for the ceramic artificial head for the BS concentrations of 8.3 %, 25 % and 100 %. Nevertheless the quantitative evaluation of the film thickness from recorded interferograms was not possible because the reflectivity of

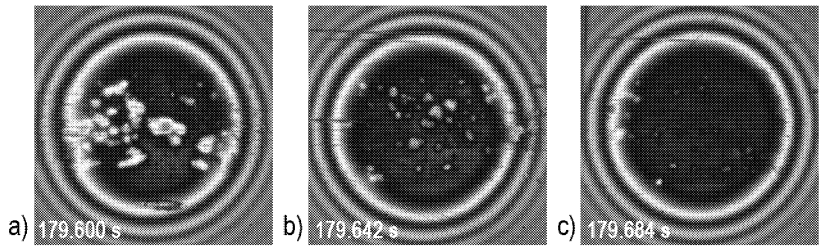

Fig.10: Selected chromatic interferograms recorded in sequence with time step of 0.042 s and corresponding to metal head and concentrations of BS 8.3 %

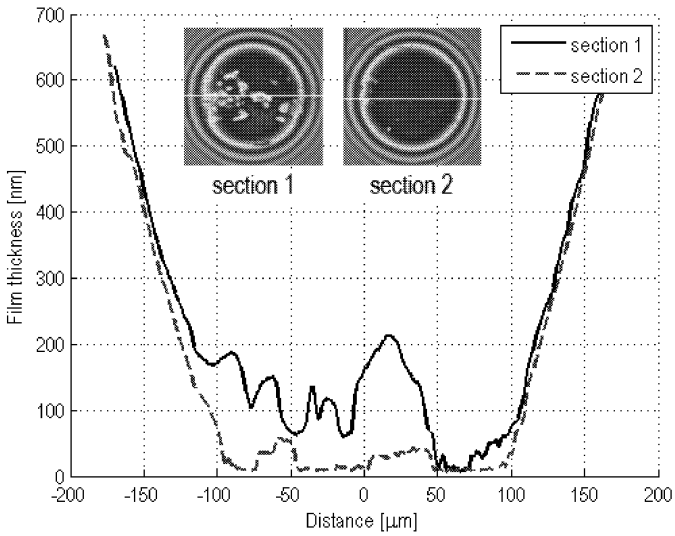

Fig.11: Differences in distribution of the film thickness in direction of rolling evaluated from both interferograms displayed in the Figs. 10a and 10c; formation of proteins causes a significant increasing of the film thickness up to 200 nm

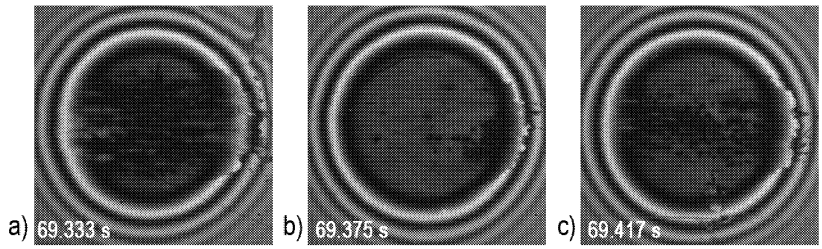

Fig.12: Selected chromatic interferograms recorded in sequence with time step of 0.042 s and corresponding to metal head and concentrations of BS 25 %

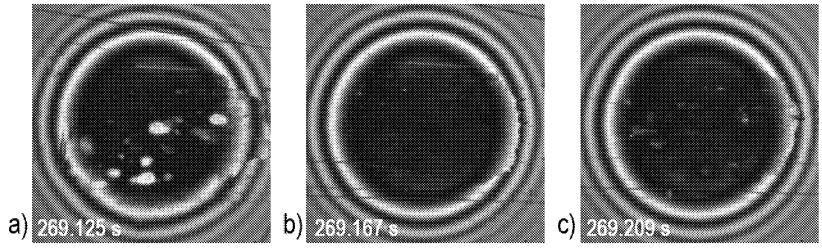

Fig.13: Selected chromatic interferograms recorded in sequence with time step of 0.042 s and corresponding to metal head and concentrations of BS 100 %

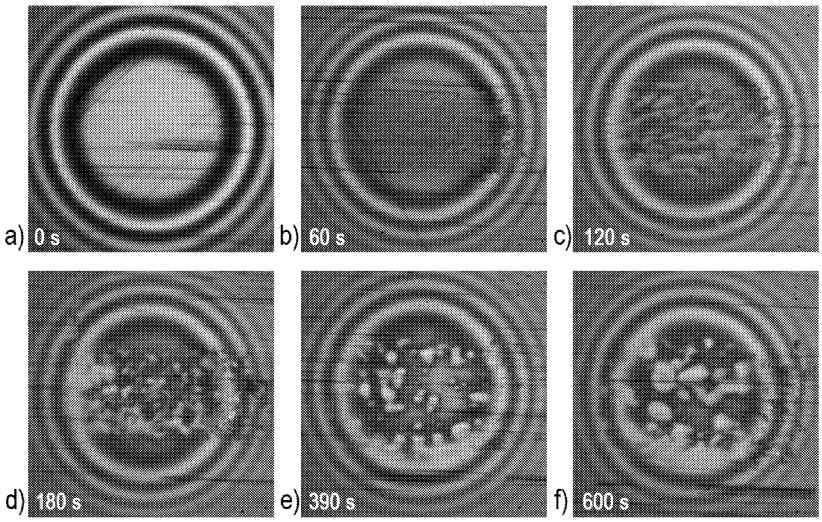

Fig.14: Chromatic interferograms recorded within 10 minutes and corresponding to ceramic head and concentration of BS 8.3 %

the ceramic head was approximately three times lower comparing the metal head thereby interferograms did not have sufficient contrast. Nevertheless, the qualitative assessment of protein film formation was performed. Figure 14 displays the formation of the protein film for the BS concentration of 8.3 % which is very similar to those obtained with the other BS concentrations (25 % and 100 %). From individual interferograms displayed in Fig. 14 it is evident that proteins form a thick lubrication film which is increased with time. It was observed that the use of the ceramic head causes the formation of more local film thickness peaks (more protein spots) which are subsequently enlarged to the whole contact zone (Fig. 14e,f). This effect is stronger than in the metal head and in addition the proteins aggregations were observed on all interferograms within the test duration. It suggests that the formation of proteins in the contact is sensitive to the rubbing surfaces properties.

3.2. Film thickness as a function of speed

Second series of experiments were focused on the mapping of lubricant film thickness as a function of mean speed over its increasing and decreasing range within 5 and 40 mm/s. Mean speed distribution in the dependence on time used for all measurements is shown in Fig. 3. The speed was changed with an increment of 2.5 mm/s and during the time period

of 5 s three interferograms were recorded. The results are labelled UP for the speed increase and DOWN for the subsequent speed decrease. The measurement was performed for each BS concentration two times with/without lubricant reservoir placed below tested head (Fig. 2). At the end of each test, the residual film thickness was measured under static loading of 5 N.

Typical results measured for all BS concentrations are shown in Fig. 15, where central film thickness is plotted against mean speed. Figure 16 also shows how the central film thickness is distributed during time period in which the mean speed is changed. Results

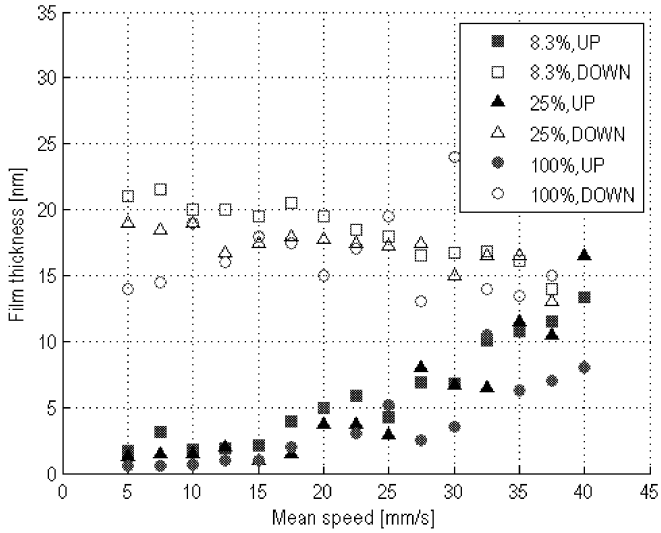

Fig.15: Central film thicknesses plotted as a function of mean speed for BS concentrations of 8.3 %, 25 % and 100 % (with using of the lubricant reservoir)

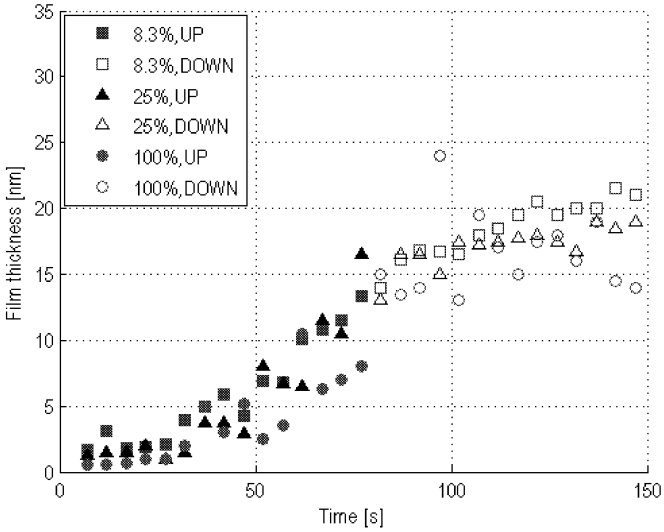

Fig.16: Central film thicknesses plotted as a function of time for BS concentrations of 8.3 %, 25 % and 100 % and for increasing/decreasing speed range (with using of the lubricant reservoir)

shown in Figs. 15 and 16 are plotted for the case when the lubricant reservoir is placed below tested metal head. At first, the BS of all concentrations forms a very thin film (1–2 nm) and its thickness increases with increasing mean speed (UP). However, when the speed was decreased (DOWN) the film thickness did not reduce but had tendency to increase at the lower speeds. At the lowest speeds, thickest film (over 20 nm) was measured for the BS concentration of 8.3 %. Generally, various BS concentrations have not substantially influenced the distribution of the central film thickness over the whole speed range; therefore the BS having protein concentration of 8.3 % is sufficient for the formation of fairly thick lubrication film. From the observation of recorded interferograms, it is obvious that after some time the film thickness starts to fluctuate as proteins start to form highly viscous film on the rubbing surfaces. This film is not homogeneous so that two different film thickness regions can be found within the contact; thicker protein film and thinner base film. During measurements the formation of proteins were observed primarily within the higher pressure contact zone. At the end of the tests (at speed of 0 mm/s) the residual films under static load of 5 N were measured. The central film thicknesses were 17 nm, 10 nm and 7.5 nm for BS concentrations of 8.3 %, 25 % and 100 %, respectively.

Next series of measurements (see Figs. 17 and 18) were performed under the same operational conditions, but the test rig configuration was changed. The lubricant reservoir commonly placed below tested metal head (Fig. 2) was removed so that the trapped lubricant was not returned again to the contact zone. It was found that the use of the lubricant reservoir had significant effect on the development of the film thickness in these speed measurements. When the lubricant reservoir was used then the measured central film thicknesses achieved values of 20 nm (Figs. 15, 16) whereas when the tests were realized without the reservoir, the measured central film thicknesses achieved higher values about 100 nm (Figs. 17, 18). From graphs displayed in Figs. 16 and 18 it can be observed that in the time period of 0–40 s the film thickness increases very slightly for both test rig configurations (with/without lubricant reservoir). Nevertheless in the time period of 40–150 s the film thickness increases sharply for test rig configuration devoid of the lubricant reservoir. In both configurations it is possible

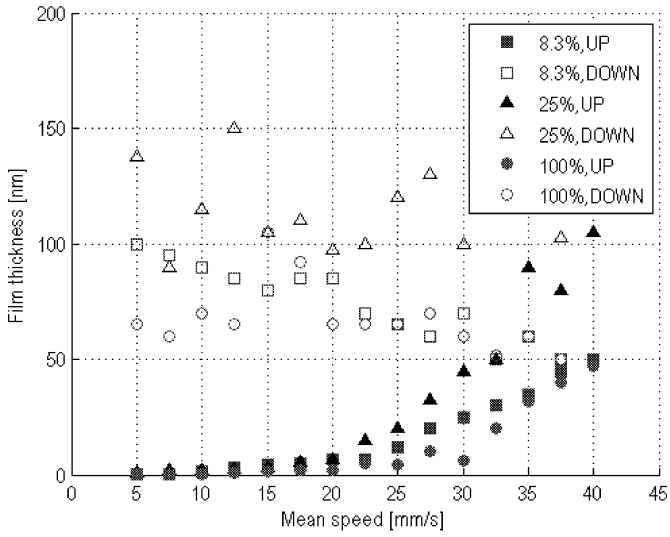

Fig.17: Central film thicknesses plotted as a function of mean speed for BS concentrations of 8.3 %, 25 % and 100 % (without using of the lubricant reservoir)

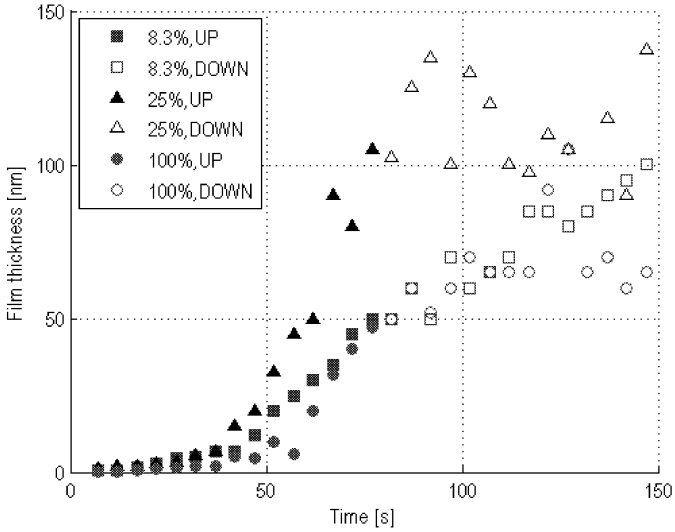

Fig.18: Central film thicknesses plotted as a function of time for BS concentrations of 8.3 %, 25 % and 100 % and for increasing/decreasing speed range (without using of the lubricant reservoir)

observe that film thickness always increases even if the supply of the BS was finished (after time of 70 s).

4. Conclusions

Lubricating film formation between the artificial femoral head (metal and ceramic) and the glass disc was observed. Film thickness measurements were made as a function of time and as a function of mean speed over a both increasing and decreasing speed range. The following conclusions may be drawn from obtained results.

1. The central film thickness between the metal head and the glass disc increases with time. At the end of measurements central film thicknesses achieve values 103 nm, 60 nm and 70 nm for BS concentrations 8.3 %, 25 % and 100 %, respectively. Formation of the lubricant film thickness for all tested BS concentrations is quite similar and is not influenced amount of proteins in tested fluids. The distribution of the film thickness within the contact zone is not homogeneous which shows that two different film thickness regions can be found; thicker protein film and thinner base film that both increase over time. In some tests the formation of proteins caused a significant increasing of the film thickness up to 200 nm.
2. Qualitative assessment of the film formation with ceramic head for all BS concentrations was performed. Results showed that proteins form thick lubrication film which increases with time. It seems that using of the ceramic head causes formation of more local film thickness peaks which are subsequently enlarged to the whole contact zone. This effect is stronger than in the metal head and in addition the proteins aggregations were observed on all interferograms within the test duration.
3. Various BS concentrations have not substantial influenced the distribution of central film thickness over the whole speed range; therefore the BS having protein concentration of 8.3 % is sufficient for formation of fairly thick lubrication film.

4. Lubricant supply has significant effect on development of the film thickness in the speed measurements. When the lubricant reservoir was used then the measured central film thicknesses achieved values of 20 nm, whereas when the tests were realized without the reservoir measured central film thicknesses achieved higher values about 100 nm.

Although ball-on-disc test configuration used in this study is different from conditions occurring in artificial hip joints where contacting surfaces are highly conformal and working under rolling/sliding conditions, transient speed and loading cycles, results obtained in this study clearly show that proteins formation plays an important role in the lubrication processes of artificial joints of the human. The formation of lubricating film thickness can be affected by a lot of other factors such as type and temperature of the BS, transient loading and speed, rolling/sliding conditions and especially conformity of the contact surfaces. Some of those are currently under investigation at present time.

Acknowledgments

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