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Anomalous Low Friction Under Boundary Lubrication of Steel Surfaces by Polyols

L. Joly-Pottuz · J. M. Martin · M. I. De Barros Bouchet · M. Belin

Abstract We present here anomalous low friction obtained with highly polished steel on steel hard contact lubricated by glycerol under severe mixed and boundary regimes (λ ratio below 1). We investigated the effects of contact pressure, sliding speed, and temperature on friction coefficient and electrical contact resistance. The mechanism of low friction (typically below 0.02) is thought to have two origins: first a contribution of an ultrathin EHL film of glycerol providing easy shear under pressure, second the chemical degradation of glycerol inside the contact when more severe conditions are attained, generating a nanometer-thick film containing shear-induced water molecules. This new mechanism, called "H-bond Network model", is completely different from the well-accepted "Monolayer" model working with polar molecules containing long aliphatic chains. Moreover, we show outstanding superlubricity (friction coefficient below 0.01) of steel surfaces directly lubricated by a solution of *myo*-inositol (also called vitamin Bh) in glycerol at ambient temperature (25 °C) and high contact pressure (0.8 GPa) in the absence of any long chain polar molecules. Mechanism is still unknown but could be associated with friction-induced dissociation of inositol and H-bond interactions network of water-like species with steel surface.

Keywords Superlubricity · Boundary lubrication · Glycerol · EHL

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1 Superlubricity and Lubrication by Glycerol

Motohisa Hirano and Kazumasa Shinjo first used the term "superlubricity" in 1990 to describe a theoretical sliding regime in which friction or resistance to sliding completely vanishes [1]. Historically, the earliest studies on superlubricity started in the mid-1980s, but the real progress occurred during the 1990s. In particular, theoretical studies by Sokoloff [2] and Hirano predicted the existence of such superlubricious states between weakly interacting, atomically smooth surfaces. It should be noted that superlubricity is theoretically applicable to vanishing of static friction but kinetic friction, which is velocity dependent, can survive. Unfortunately, we are presently unable to measure precisely friction coefficient values below 10^{-3}. More details can be found in a recently published book on superlubricity [3].

Very low friction coefficients under moderate contact pressures (MPa range) in tribological systems are well explained by hydrodynamic (HD) and elastohydrodynamic (EHD) lubrication regimes. It is the case for journal bearings with oil and skiing or skating with water, for example. When pressure is a few thousand times higher, such hydrodynamic films cannot form and friction is typically in the 0.1 range.

According to the Hertzian contact theory (below the elastic limits), and in the sphere-on-plane configuration, the friction coefficient μ depends on three variables as [4]:

$$\mu = S_0\pi\left(3\frac{R}{4}E\right)^{2/3} W^{-1/3} + \alpha \tag{1}$$

where E is the composite elastic modulus of the contacting materials and R is the radius of the sphere. This simplified model assumes that the real contact area

corresponds to the Hertzian zone, as calculated in Eq. 1. Therefore, friction measurements at different normal loads can be useful to determine S_0 and α values in a given tribological system. However, Eq. 1 indicates that the minimum friction coefficient value is α so that the friction cannot vanish completely. For example, friction experiments performed on MoS_2 coatings under different atmospheres [5–9] have led to $\alpha = 0.001$. On the other hand, if the shear strength of the interface is very low, say 25 MPa, μ is predicted to be below the centirange ($\mu < 0.01$), making the measurement of friction very difficult. The accuracy of mechanical devices measuring the friction tangential force and the normal load simultaneously makes it difficult to quantify the friction for $\mu < 0.01$, which we refer to as extremely low friction or superlubricity.

Concerning lubrication by polyhydric alcohols and particularly glycerol $C_3H_8O_3$, selective transfer was the name given in the 1970s to various phenomena involving copper-based materials and ferrous surfaces as friction pairs. It was widely reported to produce extremely low friction in the technical literature of the former USSR. Basically, the mechanism was attributed to the formation of a beneficial metallic film, possibly copper, which has an ability to reduce friction. Later, in the 1990s, the role of colloidal and gel-like products which result from the tribochemical reaction was emphasized and the rheology of the colloidal film was thought to play the key role in the friction reduction. At this point, interest of Western tribologists in these ideas was negligible mainly due to ambiguous definition of the selective transfer mechanism and also low access to Russian literature. More recently, Padgurskas [10] and Ilie [11] published papers on this matter in 2006.

Here, we are mostly interested in the use of pure glycerol to lubricate highly polished steel surfaces in the sphere-on-flat tribometer combination, under severe mixed and boundary lubrication conditions.

2 Experimental Part

The sliding tests were performed using a reciprocating sphere-on-flat tribometer equipped with triboscopy analysis and data processing [12] and the lubricant was supplied on the flat before the test by putting a few droplets (5 cl). The sphere (6 or 12 mm diameter) and the flat were made of AISI 52100 steel and were polished with diamond paste (1 μm size) to a composite surface roughness R_a equal to 0.015 μm. The sphere was loaded on the stationary flat by a force ranging from 0.5 to 2.0 N that generated a maximum contact pressure of 800 MPa. The sliding speed can be varied up to 15 mm s^{-1} and the stroke length was fixed to 2.5 mm. The temperature can be varied between ambient (25 °C) and 80 °C. The electrical contact measurement (ECR) is achieved by a dedicated device, developed on purpose. A DC voltage is applied to the two parts of the sliding contact (max voltage: 10 mV). The resulting current is measured with a logarithmic sensitivity. The current in the interface is also limited to a small value (10 mA), so that the electrical power in the contact can be considered as negligible, and is assumed not to affect the shearing process. The bandwidth of the device response is better than 1 kHz, so that spatially revolved measurements are fully relevant. The measured ECR values range from 1 Ω up to 10^6 Ω, and ECR results are presented in log scale.

The glycerol tested in this study is a commercial product from Carlo Erba. Its purity is estimated at 99.5% and its density is equal to 1.257 at 25 °C. The commercial *myo*-inositol is provided in the form of white powder by Acros Organics Company. Its purity is estimated at more than 98%. To carry out the tribological tests, it is diluted in glycerol with a concentration of 1% in mass. Deuterated glycerol was a commercial product from Isotec Co.

3 Results with Pure Glycerol Under Severe Mixed/Boundary Lubrication

3.1 Typical Evolutions of Friction and Electrical Contact Resistance (ECR)

In the testing conditions described above and using a surface composite roughness of about 15 nm, a severe mixed/boundary lubrication regime is thought to be achieved. The value of the film thickness using EHL theory in a sphere-on-plane contact was calculated according to Harmrock and Dowson formulation [13]. More accurately, we calculated the λ ratios (theoretical EHL film thickness divided by the composite surface roughness) at a typical contact pressure of 0.8 GPa, at three temperatures 25, 40, and 80 °C and different sliding speeds up to 5.0 mm s^{-1}. Glycerol has a viscosity of 0.934 Pa·s and a piezoviscosity coefficient of 5.4 GPa^{-1} at 25 °C, 0.283 Pa·s and 5 GPa^{-1} at 40 °C, and 0.028 Pa·s and 4.3 GPa^{-1} at 80 °C, respectively [14]. Figure 1a represents the evolution of the λ parameter as a function of sliding speed for the three temperatures under consideration. For example, using a sliding speed of 1 mm s^{-1} at ambient temperature (25 °C), λ is below 0.6, indicating a severe mixed/boundary lubrication regime with a possible EHL glycerol layer average thickness of about 9 nm. It is to be noticed that there is no abrupt separation between ultrathin EHL, mixed and boundary regimes when surfaces are highly polished. Figure 1b shows also the effect of contact pressure on the λ ratio.

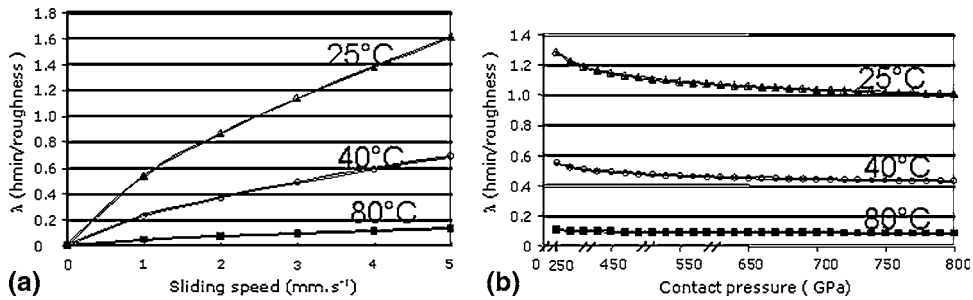

Fig. 1 Evolution of the λ parameter for three temperatures, 25, 40, and 80 °C, as a function of the **a** sliding speed for a contact pressure of 800 MPa and **b** contact pressure for a sliding speed of 2.5 mm s^{-1}

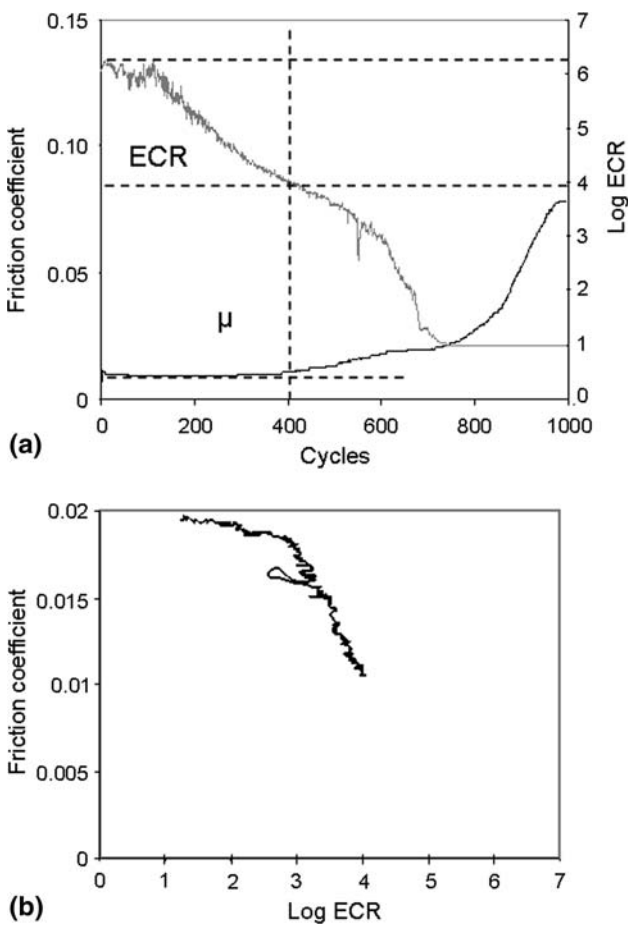

Fig. 2 **a** Friction test performed with pure glycerol at ambient temperature with a sliding speed of 1.0 mm s^{-1} and a contact pressure of 800 MPa. Average evolution of friction coefficient and electrical contact resistance (ECR, in logarithmic scale) are presented. **b** Evolution of the friction coefficient versus the electrical resistance

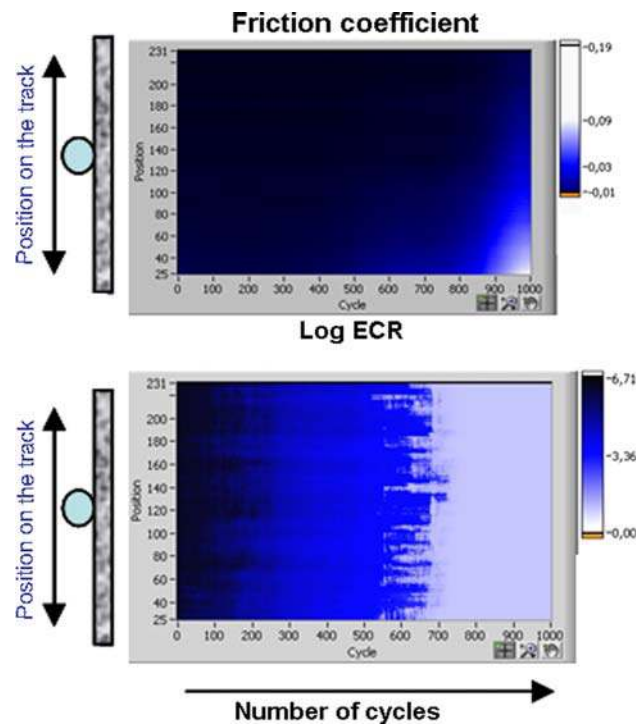

Fig. 3 Spatially resolved data obtained for the friction coefficient and the electrical contact resistance during the friction test presented in Fig. 2. Parts of data corresponding to the extremities of the track have already been removed (25 μm at each extremity). From these data, average values are calculated for each cycle of the test

A typical friction test shown in Fig. 2 was performed at ambient temperature (25 °C), 1.0 mm s^{-1} sliding speed, and a contact pressure of 0.8 GPa (corresponding to λ ratio of 0.6). Figure 3 represents triboscopic images of recordings of raw data for friction and ECR as a function of the duration of the test and the position of the sphere on the wear track. To obtain average evolutions of friction and ECR (logarithmic scale) as a function of the number of cycles (Fig. 2a), we neglected the part of data coming from the extremities of the friction track when the sliding speed is not constant and changes in direction, and we calculated the average value for each passage (one cycle corresponds to two passages). Any details of these evolutions at each passage and position (e.g., fluctuations) can be easily obtained from the retrospective analysis of the raw data when it is needed.

At the beginning of the test, friction is very low (0.01) and the contact resistance is very high (typically 10^6 Ω). During 400 cycles (about 33 min test duration), friction remains low but the contact resistance tends to decrease significantly and progressively, reaching 10^4 Ω after 400 cycles. Then friction starts to increase slightly as ECR decreases drastically. After 800 passes, when the ECR

becomes negligible, then friction suddenly increases to values around 0.1, which is a typical value for boundary lubrication of steel surfaces in presence of polar molecules. For comparison, similar tests previously performed with a PAO base oil gave much higher friction (0.2–0.25) and very low ECR [15]. For each test, we measured the following values: F_{min} corresponding to the lowest value of friction at the beginning, Δ_{ECR} values during this period, and N_c the number of passes with the lowest friction (say below 0.04). ECR is useful to study the presence of an insulating tribofilm inside the contact area. Thus, we correlate the value of friction coefficient to the value of ECR. Figure 2b plots the friction coefficient (linear scale) as a function of ECR values (log scale) during the low friction period. A straight line is obtained which suggests an exponential decay of the contact resistance as a function of thickness of a tribofilm inside the contact area.

Figure 4 shows three consecutive tests performed in the same conditions as in Fig. 2, but with different durations (number of cycles). After stopping the test, we observed the wear scars on the sphere by optical microscopy. During the low friction period (below 200 cycles), the wear track is nearly not visible on both the sphere and the flat. However, when friction drastically increases after 500 cycles, the wear scar is clearly visible on the sphere, indicating a collapse of the glycerol tribofilm and metal contacts. A wear process is occurring when the friction coefficient increases.

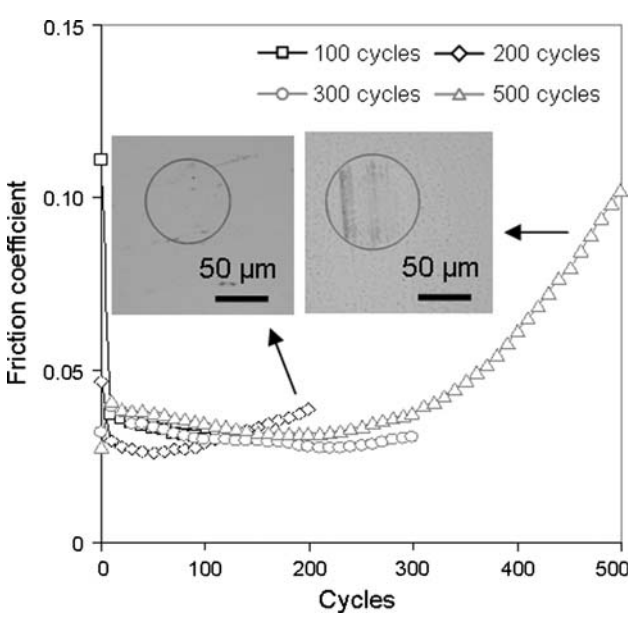

Fig. 4 Evolution of the friction coefficient during short duration friction tests with glycerol. Optical images of the wear scar after a 200 cycles friction test and a 500 cycles friction test show that no wear is observed after 200 cycles. But after 500 cycles, when the friction has increased, wear is observed

3.2 Influence of Temperature, Sliding Speed, and Pressure on Friction with Glycerol

Figure 5 shows several tests performed at 0.8 GPa contact pressure, 2.5 mm s^{-1} sliding speed and for different temperatures (20, 40, 50, 65, and 75 °C). It is clearly seen that the length of the low friction period N_c decreases with increasing temperature. At 65 °C, friction coefficient does not decrease below 0.08. The effect of sliding speed in the speed range of 0.25–5 mm s^{-1} is shown in Fig. 6. For these experiments, the contact pressure was fixed to 800 MPa and the temperature was adjusted to 25 °C. It is

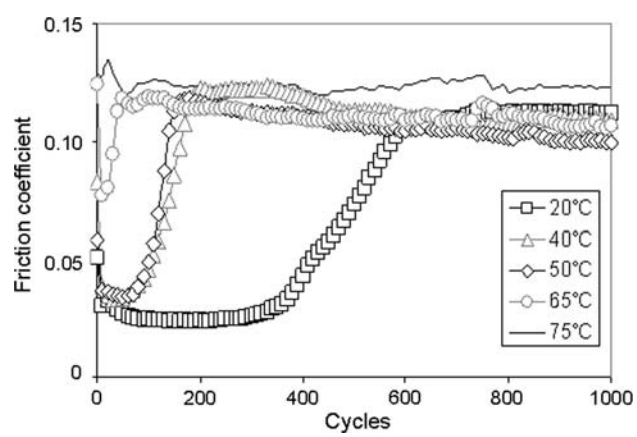

Fig. 5 Evolution of the tribological performances of glycerol as a function of temperature. The higher the temperature, the lower the N_c (number of cycles when the friction is below 0.04)

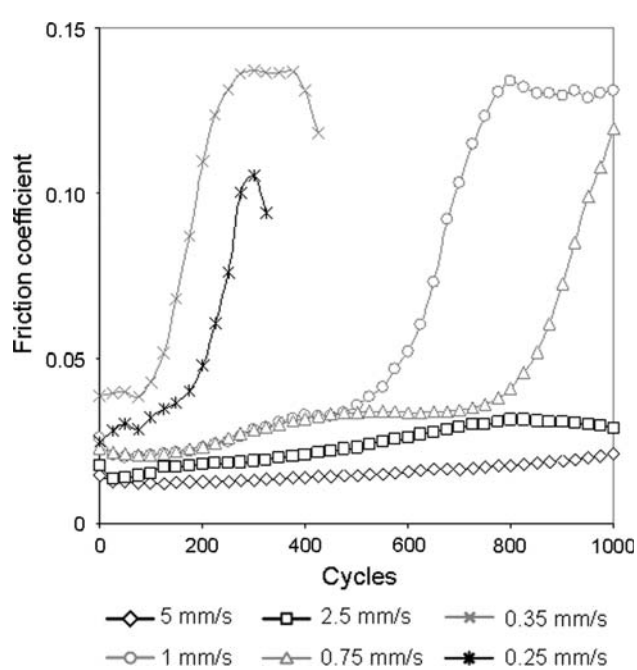

Fig. 6 Friction coefficients obtained with glycerol at several sliding speed, from 0.25 up to 5.0 mm s^{-1}. Lower friction is obtained at higher sliding speeds

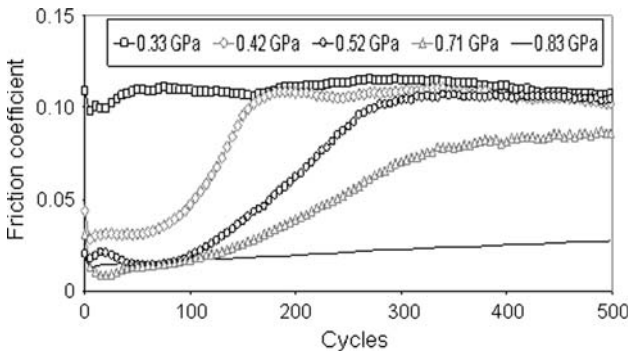

Fig. 7 Tribological performances of glycerol at several contact pressures (between 0.33 and 0.83 GPa)

clearly observed that higher sliding speeds give better friction reduction by increasing the low friction period N_c. For values of speed of 2.5 and 5 mm s^{-1}, friction coefficient stays below 0.03. Figure 7 shows the effect of contact pressure at ambient temperature at a sliding speed of 2.5 mm s^{-1}. High contact pressures provide the better result in friction reduction. At 800 MPa, friction coefficient does not increase. But, at lower contact pressures, an increase of the friction coefficient above 0.1 is observed.

3.3 Lubrication Mechanism of Steel by Glycerol

The origin of the contact resistance observed in Fig. 2 can be attributed to the existence of a homogeneous glycerol layer in the Hertzian contact area, the diameter of which being about 50 μm. Pure glycerol, like pure water, is not an electrical conductive fluid. When it is anhydrous, its electrical conductivity is about 5.5×10^{-6} S/m at 20 °C [16]. If we consider the ohmic resistance of a cylinder of glycerol (50 μm diameter and 6 nm thick) and taking the value of the electrical resistivity of glycerol at ambient temperature and atmospheric pressure, the calculated value of the electrical conductivity is about 10^{-7} Ω, in good agreement with our measurement. However, the explanation for the simultaneous decrease of ECR and increase of friction is not straightforward. This can be attributed either to a decrease of the film thickness or to a chemical degradation of glycerol inside the contact area. If we plot the friction coefficient (linear scale) as a function of ECR values (log scale) during the superlow friction period (see Fig. 2b), we obtain a straight line in the low friction regime, suggesting an exponential decay of the contact resistance as a function of thickness. This suggests the existence of a tunnelling conductivity in the Hertzian contact when the film thickness becomes below 5 nm. This behavior was already observed in the literature by Mansot [17], for example, using a similar friction test with steel surfaces and an ester lubricant.

The existence of a thin EHL glycerol layer separating completely the two metallic surfaces in the very low friction regime is confirmed by the results observed in Fig. 4. At the beginning of the test, no wear scar is observed. But after the increase of the friction coefficient, wear is observed, suggesting that the protective glycerol layer progressively collapses. This thin layer of glycerol itself is found able to give an anomalous low value of friction. This is in agreement with a previous work by Vergne [14], who found similar traction values in full EHL lubrication of steel surfaces with glycerol. Actually, the viscosity of glycerol is high enough to allow an EHL film to be established, but its very low piezoviscosity coefficient prevents high traction values, at the opposite of paraffin, for example, having a solid-like behavior in an EHL contact.

Results observed in Figs. 5, 6, and 7 can be examined by using the λ parameter. At 80 °C, friction coefficient measured remains equal to 0.12 all along the test. At this temperature, the λ parameter is below 0.1 and the glycerol film thickness is certainly not able to separate completely the steel surfaces to avoid metallic contacts. As shown in Fig. 1a, glycerol film thickness depends strongly on the sliding speed. At high sliding speed, film thickness increases avoiding contact between metallic surfaces. Thus friction coefficient remains low (Fig. 6). These results suggest that low friction coefficient is obtained when a glycerol film is present inside the contact area. Results observed in Fig. 7 are not in agreement with the contact pressure effect on the film thickness shown in Fig. 1b. For a sliding speed of 2.5 mm s^{-1} and the three temperatures under consideration, the λ parameter is slightly higher at lower contact pressure.

We have no clear explanation why friction increases after a certain time (or cumulated slid distance), which is not negligible (typically a few hundred of cycles or some tens of minutes duration). Probably, the glycerol molecules are progressively dissociated inside the contact under the effect of pressure and shear. We previously observed some corrosion patterns in the Hertzian contact area when the test was stopped after a certain time [18]. We attributed this fact to the degradation of glycerol into water molecules and acids or aldehydes fragments and this was further confirmed by MD calculations in the case of DLC-coated surfaces [18]. It is known that mixtures of glycerol and water become rapidly less viscous, even at a few percent water concentration.

The decrease in bulk viscosity of glycerol due to water generation could lower too much the film thickness, and metallic contacts would begin to occur. Moreover, corrosion of steel by acids can change the surface roughness and this can give an additional effect on the λ ratio. However, the effect of in situ water formation in the contact could also be an advantage, decreasing the shear value of glycerol at high contact pressure. In this regard, the effect of pressure could provide more shear-induced water molecules in the contact area only and consequently better

friction reduction. It is expected that this low quantity of water produced inside the contact area during a test would certainly not affect immediately the water concentration and the viscosity in the bulk lubricant volume providing the EHL film thickness. To study this point in more detail, additional tribological tests were performed with glycerol containing 10 wt% of water (not shown here). A friction coefficient of 0.05–0.06 was obtained during 100 cycles, afterwards it rapidly increases to 0.12, indicating that the presence of water in glycerol reduces drastically N_c.

To summarize, glycerol is certainly dissociated in the contact area at high pressure, generating confined water which could be a benefit for low friction achievement but water is progressively released in glycerol at the outlet of the contact, lowering its bulk viscosity and then the EHL film thickness. Probably, acids and/or aldehydes fragments can be neutralized by glycerol when they leave the contact zone. An alternative but not exclusive mechanism is the increase of steel surface roughness due to the generation of aggressive species and the subsequent corrosion of the surface, leaving carbide grain as asperities.

The chemical composition of steel surfaces can be studied accurately by Angle Resolved XPS (ARXPS). Rossi et al. [19] used ARXPS to investigate the nature and thickness of iron oxides and hydroxides layer at the top surface of steel samples. Figure 8 represents a schematic view of these layers. We can see that two kinds of oxides are present near the steel substrate, first Fe_2O_3 and then Fe_3O_4. A hydroxide layer is located at the top (probably FeOOH) and even some water molecules are detectable on the hydroxide layer. The total thickness of the passivation layer does not exceed 4 nm. Of course, a 2-nm-thick layer of adventitious carbon is always present on the top, even in UHV conditions of the XPS analysis. We tried to investigate the effect of glycerol on this steel surface using Static SIMS surface analysis and we used deuterated pure glycerol for this particular experiment. For deuterated glycerol, we substituted ^2H for all hydrogen atoms of hydroxyl group only. Figure 9 shows positive ions SIMS spectra obtained inside the wear track of the flat in a test stopped in the superlow regime. The first spectrum (a) shows surface species before argon etching and the spectrum (b) is recorded after 10 s etching (typically corresponding to 1 nm thickness of material removed). On the top surface, $^2H^+$ ions are detected and also O^2H^+ ions are visible together with typical contamination contribution of carbon species coming from the environment. After etching, mainly the contribution of the oxide layer is visible (O^+ ions). We can deduce from this SIMS analysis that any solid-like tribofilm which could form in the wear scar has a thickness not exceeding 1 nm. Second, we show that elements from glycerol (deuterium) are present on the surface. Importantly, we do not observe any higher mass in the SIMS spectrum, coming from polymerization. Tentatively, we propose a schematic representation of the lubricating interface in presence of glycerol molecules including some water molecules formed by the degradation of glycerol (Fig. 10a).

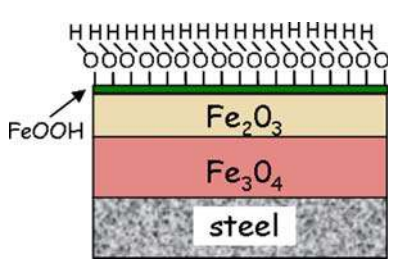

Fig. 8 Model of the composition of steel top surfaces proposed by Rossi et al. [19] from ARXPS analyses. Two kinds of oxides are present at the top surfaces of steel: 1.3 nm of Fe_2O_3 and 1.8 nm of Fe_3O_4

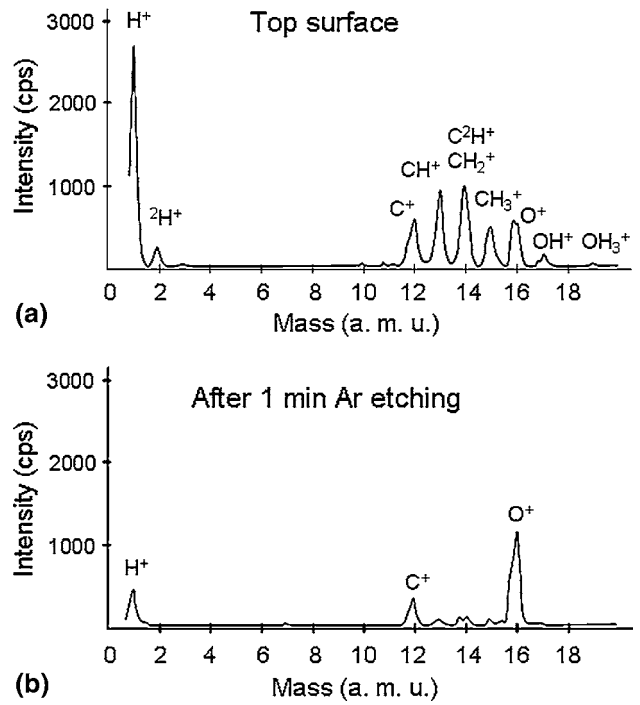

Fig. 9 SIMS analysis of the wear scar after friction test with deuterated glycerol: **a** top surface and **b** after an etching of 1 min (corresponding to the removal of 1 nm of material). No higher mass are detected

Since the pioneering work of Tabor in the 1950s, the friction reduction mechanism under boundary lubrication has always been attributed to the effect of long chain polar (amphiphilic) molecules. In the so-called "Self-Assembled Monolayer (SAM)" or "Monolayer" model, the polar extremity of the molecule is strongly chemisorbed on the native oxide layer present on the steel surface while the paraffinic moiety extends outside the metal surface. The amphiphilic molecules must have at least 10 carbons in

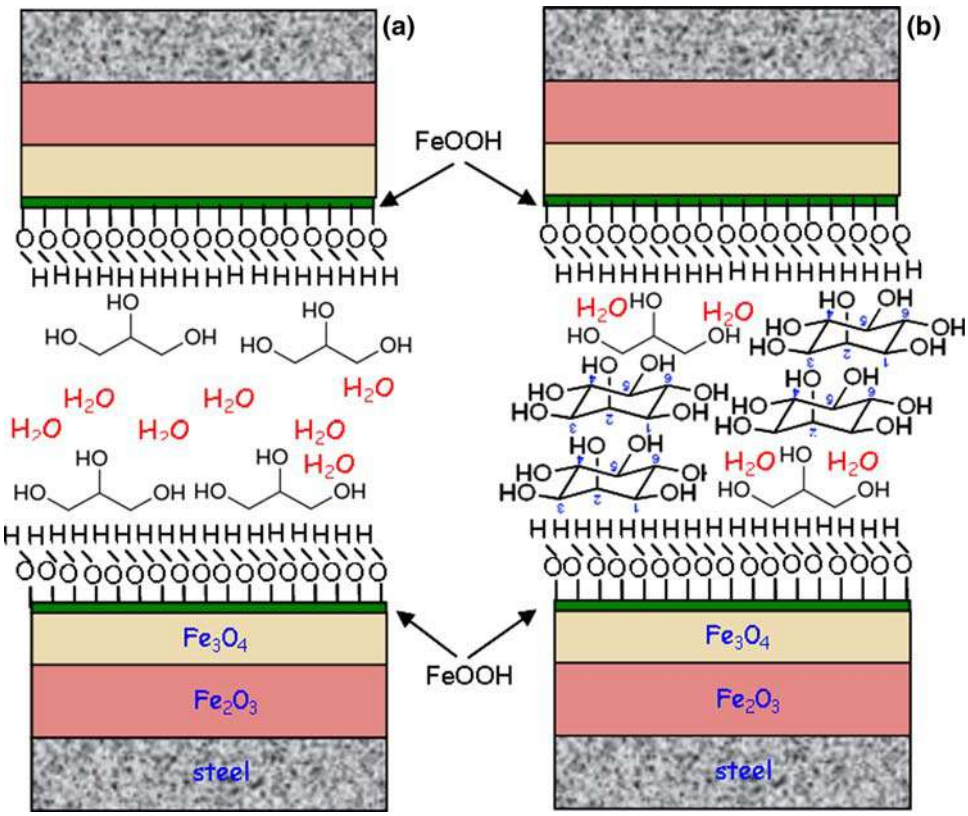

Fig. 10 Possible schematic model of the tribofilm inside the contact area: **a** with pure glycerol and **b** with glycerol containing inositol. Acids or aldehydes fragments have not been represented here

their aliphatic chain in order to promote the formation of a crystal-like structure in the monolayer. Then, low friction is generally attributed to the easy sliding of methyl groups of SAMs over each other, as described by Molecular Dynamics simulations of Harrison [20] for example. The drawback of this situation is that the monolayer has a limited durability and molecules generally desorb or polymerize at elevated temperature. Another problem is that superlow friction values (below 0.01) are only attained at very low contact pressures (e.g., below 10 MPa in the surface force apparatus SFA). At several hundred MPa contact pressures or more in macro-scale situations, friction hardly drops down below 0.04. In our experiments, the SAM model cannot be applied since glycerol molecule has not such a long aliphatic chain and does not polymerize. Then there is an opportunity for another lubrication mechanism here. The new model, which we call "H-Bond Network" model, has been recently published in the literature [18] in the case of DLC-coated surfaces.

4 Results with Glycerol Plus Inositol Under Severe Mixed/Boundary Lubrication

Myo-inositol crystals were dissolved in glycerol to reach a concentration of 1% by weight after heating at 60 °C for 5 h. The solution was very clear and limpid. The viscosity of glycerol was practically not affected by the addition of inositol. The same tribological conditions were used in order to compare the two lubricants (namely glycerol and glycerol + inositol). Figure 11 shows the results obtained at different temperatures, at a contact pressure at 800 MPa and a sliding speed of 1.0 mm s^{-1}. Table 1 represents the critical values F_{min} and N_c measured for the two lubricants, respectively. First of all, we observe a significant increase of N_c, the number of cycles with a friction below 0.04 when inositol is added to glycerol. Second, we observe a smaller

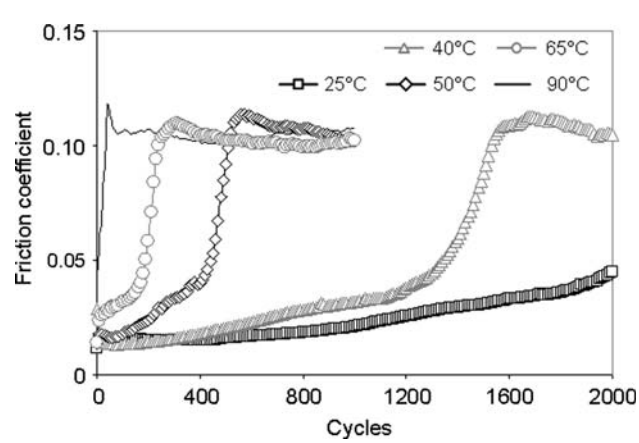

Fig. 11 Friction performance of glycerol containing 1 wt% of *myo*-inositol at several temperatures (from ambient temperature to 90 °C)

Table 1 Values of F_{min} (lowest friction coefficient obtained) and N_c (number of cycles below 0.04) for pure glycerol and glycerol + 1 wt% inositol

	F_{min}	N_c
Glycerol	0.01	875
Glycerol + 1 wt% inositol	0.01	1900

value of F_{min} when inositol is present in glycerol. The friction coefficient in the presence of inositol is near 0.01, a critical value to reach the superlubricity regime [18]. The origin of the better performance in the presence of inositol is not yet understood. A possible explanation could be due to the degradation mechanism of the inositol molecules confined in the interface region which could not form aggressive species like acids. Actually, the accumulated inositol molecules in the contact zone could completely transform into water and quinone [18]. However, we observed that inositol is rapidly consumed and that the addition of a new drop of lubricant was necessary to keep the superlow regime. Then it appears that the decrease of performance of glycerol in steel lubrication is mainly attributed to the corrosion process and not to the decrease of bulk viscosity due to water formation. In the presence of inositol, a schematic representation of the operating interface is presented in Fig. 10b. This result shows that the excellent lubricating properties of glycerol can be even enhanced by adding particular polyhydric cyclic alcohols.

5 Conclusion

Approaching superlow friction in the 0.01 range was obtained for a significant sliding distance using highly polished steel on steel surfaces lubricated by pure glycerol at ambient temperature. Afterwards, friction increases when the electrical contact resistance decreases. Glycerol is not an electrically conductive fluid but is a very efficient lubricant in EHL lubrication because of its low piezoviscosity coefficient and relatively high viscosity. From our results we can assume that the very low friction coefficient obtained at the beginning of the test is due to the presence of a few nanometer-thick glycerol nanolayer inside the contact area.

The influence of sliding speed and the temperature on the tribological performances of glycerol confirms this point since temperature has an influence on viscosity and then on the thickness of the film, in agreement with the EHL theory. Thus, at ambient temperature, the film thickness is thicker and the friction coefficient is lower. Film thickness also depends on sliding speed. The higher the sliding speed, the thicker the film.

The study of the influence of pressure indicates that higher contact pressures give lower friction coefficients. This result is not consistent with the EHL model because the film thickness should decrease slightly with the pressure. Thus the lubrication mechanism by glycerol could have another origin: the formation of beneficial shear-induced water molecules inside the contact area due to the tribo-degradation of glycerol molecules. Another possible advantage is the decrease of glycerol viscosity due to water content. A drawback of this situation is that tribo-degradation of glycerol leads also to the formation of aldehydes or acids fragments, which can be aggressive toward steel counterparts in the contact zone. However, this is not the case with carbon surfaces and especially DLC coatings.

Addition of *myo*-inositol to glycerol decreases the value of the friction coefficient at the beginning of the test (F_{min}) and increases significantly the number of cycles with friction below 0.04. These better performances can be explained by the fact that the tribo-degradation of *myo*-inositol could lead to the formation of less aggressive fragments toward steel and not to a decrease of viscosity. However, we observed that inositol is rapidly consumed by the tribochemical reaction. More experiments are necessary to fully understand these unexpected performances and make that polyols could lubricate steel for longer time in the superlow regime.

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