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# The correlation between the surface energy, the contact angle and the spreading parameter, and their relevance for the wetting behaviour of DLC with lubricating oils



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## ABSTRACT

In this study we investigate the correlation between the wetting, the contact angle, the spreading, the surface energy and the surface tension for five types of DLC coatings (doped, non-doped, hydrogenated and non-hydrogenated) and steel, wetted with synthetic, non-polar, polyalphaolefin (PAO) oils with two distinctly different viscosities. We show how these parameters correlate with each other and how and why they change when different DLC or steel surfaces come into contact with the oils. Several previously reported inconsistencies in the wetting and the surface-energy properties of DLC coatings are discussed. The spreading parameter (SP) presented in this work provides the necessary information to reveal the actual wetting properties of the DLC coatings and the steel with oils, instead of the contact angle, which is the parameter that is normally considered, but was found here to be inappropriate. A correlation between the polar surface energy and the DLC's hydrogen content is clearly observed. The results further indicate that the polar surface energy is a key parameter for tailoring the DLC wetting and the related surface tribochemical characteristics. In addition, the influence of various models for the surface-energy calculation is described.

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## 1. Introduction

Surfaces are very important for every tribological system as they are the first parts to come into contact as well as being a direct connection between the material and the environment. Several parameters are generally agreed to be important for tribological materials and surfaces, such as the hardness, the roughness, the elastic/plastic properties, etc., and are typically taken into consideration for the design of tribological systems. However, the wettability, the hydro/oleo-phobicity and/or the hydro/oleo-philicity and the surface energy are also very important for solid-liquid properties, which are therefore relevant for all lubricated tribological conditions. In spite of this obvious correlation, these parameters are typically neglected in tribological studies, and they are not yet considered in most of the engineering tribological models and theories.

Recent studies have, however, shown that DLC surfaces can offer specific beneficial oil/water–DLC interface properties that can significantly affect the friction and other tribological behaviours [1–3]. For example, the slip of the oil/water at the DLC interface, which is affected by surface-liquid interactions, was suggested to

reduce the friction in the (elasto)hydrodynamic regime [1–3], and a ready-to-use diagram-based model to consider this effect was proposed [1]. In fact, interfacial slip, which reduces the friction and drag force, was already proposed, based on theoretical analyses [4,5], experiments at the nano-scale [6–8], and by employing model lubricants and surfaces [6–9]; however, any verification with typical engineering materials and lubricants was missing until recently. Therefore, the lack of conventional engineering contact conditions and common engineering materials, as well as lubricants, did not allow this concept to develop more significantly in tribological practice. Accordingly, an important step forward, i.e., the implementation of slip as a relevant tribological theory in interface behaviour, and an available proposed method or “tool” [1] to implement it, could have a significant influence on the applicability and use of DLC coatings in several applications.

However, the exact physico-chemical background for slip and/or friction reduction to occur is still unclear and a number of suggestions for the slip requirements were given so far, although sometimes they were also contradictory: low wetting/high contact angle [1,2,9], good wetting [8,10], viscosity decrease [4,5], viscosity increase [7], molecular slip [11,12], formation of air pockets [13], existence of air nanobubbles [14], low surface energy [3], hydrophobic surfaces [4,5], hydrophilic surfaces [8], high-roughness surfaces [15,16] and low-roughness surfaces [17–19]. Clearly,

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## Nomenclature

$\theta$	contact angle, deg
$\gamma_S$	total surface energy, $\text{mJ}/\text{m}^2$
$\gamma_S^D = \gamma_S^{LW}$	dispersive component of surface energy, $\text{mJ}/\text{m}^2$
$\gamma_S^P$	polar component of surface energy, $\text{mJ}/\text{m}^2$
$\gamma_S^-$	electron donor part of polar component of surface energy, $\text{mJ}/\text{m}^2$
$\gamma_S^+$	electron acceptor part of polar component of surface energy, $\text{mJ}/\text{m}^2$

$\gamma_L$	total surface tension, $\text{mN}/\text{m}$
$\gamma_p^D = \gamma_L^{LW}$	dispersive component of surface tension, $\text{mN}/\text{m}$
$\gamma_L^P$	polar component of surface tension, $\text{mN}/\text{m}$
$\gamma_L^-$	electron donor part of polar component of surface tension, $\text{mN}/\text{m}$
$\gamma_L^+$	electron acceptor part of polar component of surface tension, $\text{mN}/\text{m}$
$\gamma_{SL}$	solid–liquid interfacial tension, $\text{mN}/\text{m}$
$W_a$	work of adhesion (solid–liquid interface), $\text{mJ}/\text{m}^2$
$W_c$	work of cohesion (liquid), $\text{mJ}/\text{m}^2$

a wide spreading and weak definition of these phenomena, especially related to slip, are established today.

This uncertainty and lack of understanding of the relations between these parameters, as well as their exact effect on the tribological properties, is also clearly demonstrated in the oil-lubrication studies of DLC coatings. This relates primarily to the effect of wetting and surface energy, especially in engineering contacts. For example, sometimes the surface energy [3,20–22], sometimes the wetting [1,2,6,9,25] and sometimes [23,24] both properties (surface energy and wetting) are proposed as being responsible for the same effects, such as slip that reduces the friction/drag force or for low adhesion. This implicitly assumes that wetting and surface energy have the same effect or meaning. However, it is very clear that wetting and surface energy are not “the same” parameters and cannot be simultaneously—and equally—modified and tailored in an oil–DLC system. Moreover, the very low surface energy of DLC coatings compared to steel is often also implied by their relatively high inertness compared to steel [1,26–32], but this was typically not supported and evidenced by surface-energy results.

Accordingly, in order to be able to tailor and optimise the oil–DLC interface in terms of wetting and surface energy, it is necessary that the actual key interface parameters affecting the friction of the DLC coatings and the steel under oil-lubricated conditions are identified. While in another companion study we reported on which of the above parameters correlates the best with the tribological behaviour, and we presented a tentative model for it [33], in this study we investigate the correlation between the wetting, spreading, contact angle, surface energy and surface tension for five types of DLC coating and steel, wetted with synthetic, non-polar, polyalphaolefin (PAO) oils with two distinctly different viscosities. The spreading parameter (SP), derived by us, was also introduced and proved to provide key data to reveal important differences between the DLC coatings and the steel, especially for their wetting with oils. Additionally, the influence of various models for the surface-energy calculation is presented, which is typically not discussed as a relevant “parameter” when it comes to influencing such results.

## 2. Experimental

### 2.1. Materials and oils

In this work we use five different DLC coatings and steel, tested under ambient conditions. The steel DIN 100Cr6 (AISI 52100), which is frequently used in tribological studies, was selected. This also serves as a relevant reference engineering material. The steel surfaces that were thermally treated to a hardness of  $62 \pm 1$  HRC were further prepared in a sequence of grinding and polishing steps, through a mechanical treatment only, using abrasive discs to reach the same level of  $R_a$  roughness, which was  $0.05 \pm 0.015 \mu\text{m}$ ,

measured with a stylus-tip profilometer (T8000, Hommelwerke GmbH, Schwenningen, Germany). Some of these samples were later used to deposit coatings.

Five different DLC coatings were selected in such a way as to have different structural properties, and thus, presumably, also wettability and surface energy. We used two, non-doped, DLC coatings: hydrogenated amorphous DLC (denoted as a-C:H) and non-hydrogenated tetrahedral amorphous DLC (denoted as ta-C). We also used three doped DLC coatings: a hydrogenated amorphous diamond-like carbon doped with silicon, denoted as Si–DLC, and the same coating that, in addition, contains fluorine, denoted as F–DLC. Another coating employing N as a doping element was also investigated (N–DLC).

The a-C:H coating used was typical standard non-doped hydrogenated amorphous DLC coating with H-content of 30 at% and the  $sp^3/(sp^3+sp^2)$  ratio from 35% to 40%. Coating was deposited by radio frequency (13.56 MHz) plasma-assisted CVD (RF PACVD) process and has a thickness of  $1.1 \mu\text{m}$ . Si-based,  $0.6 \mu\text{m}$  thick layer was used as an adhesion promoting interlayer. The second non-doped coating and the only non-hydrogenated coating used in this work was tetrahedral amorphous ta-C coating. Coating was deposited on steel samples by using arc evaporation, i.e. physical-vapour-deposition with the same thickness range of about  $1.2 \mu\text{m}$ , similar to other CVD coatings in this study. The Si-doped Si–DLC coating having  $1.5 \mu\text{m}$  coating thickness was deposited by using a low-frequency plasma-assisted CVD and contains 5–10% of Si depending on gas flow management during deposition. Adhesion promoting interlayer is the same as for a-C:H coating, i.e. Si-based and is  $0.6 \mu\text{m}$  thick. Another doped DLC coating was F-doped coating F–DLC, which is a variant of Si–DLC coating, produced with the same deposition process. As for the Si–DLC coating, the F dopant is part of a gas phase with the same concentration share. The functional coating thickness was about  $1 \mu\text{m}$ . The interlayer for improving the adhesion performance was also employed and is about  $1.8 \mu\text{m}$  thick. N-containing DLC coating N–DLC was prepared under the same conditions as a-C:H coating, i.e. by RF PACVD deposition process. The functional coating thickness is about  $1 \mu\text{m}$ . This coating has also an adhesion promoting interlayer, based on Si, in total thickness of  $0.4 \mu\text{m}$ . The key properties of these DLC coatings are presented in Table 1.

Synthetic polyalphaolefin (PAO) base oils (Chevron Philips, Texas, USA) with two distinctly different viscosity grades, i.e., 19 and  $56 \text{ mm}^2/\text{s}$  at  $40^\circ\text{C}$ , were used. They are denoted as PAO4 and PAO9, respectively. No additives were included in the base oil. The main properties of these two oils are presented in Table 2.

### 2.2. Surface energy

The surface energy can be calculated via different theoretical models using the known wetting properties of selected materials with particular “model liquids”. Accordingly, to determine the surface energy of our materials, the contact angles at the surfaces

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