

The effect of temperature on the tribological mechanisms and reactivity of hydrogenated, amorphous diamond-like carbon coatings under oil-lubricated conditions

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Abstract

In this work we present the wear and friction behaviour of boundary-lubricated, hydrogenated, amorphous, diamond-like carbon coatings (a-C:H), in self-mated a-C:H/a-C:H contacts, at three different testing temperatures: 20, 80, 150 °C. We present results from Auger electron spectroscopy, X-ray photoelectron spectroscopy and Raman analyses relating to the chemical and structural changes in the diamond-like carbon coatings during sliding in the presence of mineral oil, with and without additives. We show, that chemical reactions between the a-C:H coatings and the oil additives take place, which are dependent on the temperature, on the presence of additives and the type of additives used. At high temperatures the extreme pressure additive interacts with the diamond-like carbon surface and forms a tribochemical layer with a four-times lower sulphur/phosphorous ratio than the additive formulation. In the absence of additives, however, graphitisation of the coating occurs under these conditions, which results in high-wear and low-friction behaviour. Another result from this study is that a-C:H coatings can oxidise during room-temperature experiments, suggesting that some interactions and adsorptions are also possible at lower temperatures.

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1. Introduction

Until recently, most of the understanding of the tribological behaviour of diamond-like carbon (DLC) coatings was focused on their performance in environments like dry or humid air, vacuum and different gas atmospheres [1–5], with much less emphasis being placed on the oil-lubricated conditions. However, due to great improvements in the properties of coatings and their ability to operate under more severe conditions, it has become obvious that for the appropriate and broad implementation of DLC coatings in various machine components they also need to operate under lubricated conditions, remembering that these conditions are often poor and/or starved, i.e. in the boundary regime. For this reason, an increased number of recent studies have focused on the properties and mechanisms of

boundary-lubricated DLC coatings using different lubricants, coatings and experimental conditions.

For example, studies were performed using synthetic lubricants, usually poly-alpha-olefins and specific additives that are typical for automotive applications [6–11]. In contrast, some other studies [12–14], and our own research in recent years [15], were mainly focused on mineral base oil, since most of the lubricating oils that are used in many industrial applications, transmission systems, hydraulics, etc., are mineral based. Mineral oils possess better lubricating properties, and they are also better able to dissolve additives. However, they have a lower oxidation stability than synthetic oils, among other characteristics [16–18]. Therefore, because of the chemistry and performance differences observed in conventional metal/steel systems, mineral and synthetic lubricants most probably have different boundary-lubrication abilities in combination with DLC coatings, too. So far, by employing mineral base oils we have studied the effect of the type of oil additives and the different doping elements in self-mated DLC/DLC [19,20] and

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steel/DLC [20,21] contacts, and we have also investigated the effects of the polarity and the saturation of different base oils with the same additives being employed [22].

In these, and most other reported studies, additives significantly affected the wear and friction of boundary-lubricated DLC contacts; however, the extent of the change depends on the type of oils, additives, coatings, counter-bodies, etc. Such an effect is to be expected for the contacts with steel as the counter-body, where at least the steel surfaces interact with the additives in accordance with conventional theories [16,17]. And this was indeed observed in all studies, irrespective of the other specific conditions. Such behaviour can even be anticipated for doped-DLC/doped-DLC contacts, where the metal and other doping elements can provide sites for the additive interactions even without a steel counter-body [15,19]. However, more surprisingly, in our work, we have found by using different types of oils and several coatings that pure, non-doped, hydrogenated, diamond-like coatings (a-C:H) in self-mated contacts, without any interference from other counter-bodies or doping materials/elements, also interact with additives [19,20,22]. In agreement with this favourable response of a-C:H coatings to oil additives, some other studies that compared a-C, a-C:H and Ti-DLC coatings under lubricated conditions (self-mated and against various counter-bodies) also noted that a-C:H coatings experience the largest changes in tribological performance among these coatings when additives are used in the oil, in particular reducing the wear of the coating [7,8,12], like in our work. However, despite our efforts to perform different surface-sensitive analyses [15], we have not yet succeeded in obtaining clear evidence for chemical reactions between these selected pure a-C:H coatings and the additives.

Since the boundary lubrication of DLC coatings is in a very early stage of research, it is understandable that the actual boundary-lubrication mechanisms and the interactions with additives are still poorly understood. The conditions under which the conventional additives can interact with the pure non-doped DLC coatings, especially without the presence of any doping elements or steel counter-body, remain to be determined. Therefore, in order to elucidate the effect of the temperature, which is a parameter known to detrimentally influence the additive's reactivity with metals [16,17], we have investigated the tribological behaviour of lubricated self-mated a-C:H/a-C:H contacts at three different testing temperatures. Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS) and Raman analyses were employed to verify the chemical and structural changes of the DLC coatings in contacts with mineral base oil with and without anti-wear (AW) and extreme pressure (EP) additives. The results show that DLC coatings already oxidise at room temperature and that chemical reactions between the DLC and the additives are highly temperature-dependent, as well as depending on the type of additives used.

2. Experimental details

The tribological tests were performed using the ball-on-flat testing geometry, using balls and flats made from DIN 100Cr6 steel. All the balls and discs initially had the same mechanical,

thermal and surface characteristics. The steel balls were standard, commercially available balls with a diameter of 10 mm; they had a hardness of 850 HV (corresponding to 8.3 GPa), measured using a micro-hardness tester (Leitz Miniload, Wild Leitz GmbH, Germany). The surface roughness was measured using a stylus-tip profilometer (T8000, Hommelwerke GmbH, Germany) and the R_a value was better than 0.03 μm . The steel flat samples were cut from a rod into $\phi 24 \text{ mm} \times 7.9 \text{ mm}$ discs, and were heat treated to the same hardness as the balls. The steel discs were ground and polished in several steps to a final roughness R_a of 0.05 (± 0.01) μm . All the steel balls and discs were then coated with a single-layer "pure" hydrogen-containing amorphous diamond-like carbon (a-C:H) coating (without any doping elements) with a thickness of 2.67 (± 0.04) μm . A hybrid plasma vapour deposition/chemical vapour deposition (PVD/CVD) process was employed for the deposition of the coating, and a TiN interlayer was employed to improve the adhesion of the coating. This adhesion was investigated with a scratch tester (Revetest, CSM Instruments SA, Switzerland), and the average Lc1, Lc2 and Lc3 values [23] were determined to be 16.6 N, 28.0 N and 29.5 N. The hardness and the Young's modulus of the coating were measured using the depth-sensing indentation technique (NanoTest 600 instrument with Berkovich indenter, Micro Materials Limited, UK). The hardness of the coating was 25.6 (± 3.1) GPa, and the Young's modulus was 187.9 (± 16) GPa. The surface roughness, R_a , of the balls and discs, was measured after the coating deposition using the same stylus-tip profilometer, and the average value was about the same, or only slightly higher, i.e. by less than 0.01 μm . Table 1 summarizes some of the most important material properties.

The experiments were performed under two sets of conditions: using only a base oil, and using a mixture of a base oil and 1% of a single wear-reducing additive (Table 2). A paraffin base oil (denoted as M) of viscosity grade ISO VG 46 was used for the tribological tests under selected conditions. Two different additives that provide wear protection under a variety of possible conditions in mixed and boundary-lubrication conditions and are frequently used for conventional steel surfaces were employed. One of the additives was an ashless, multifunctional, mild, anti-wear/extreme pressure (AW/EP) additive, i.e., a mixture of amine phosphates, having about 4.8% and 2.7% of P and N, respectively. The second additive used was a typical, strong, extreme pressure (EP) additive, i.e., dialkyl dithiophosphate, containing 9.3% of P and 19.8% of S.

Table 1
Some important characteristics of the DLC-coated ball and disc samples

Hybrid PVD/CVD process, a-C:H single-layer, TiN interlayer				
Hardness (GPa)	Young's modulus (GPa)	Thickness (μm)	Roughness R_a (μm)	Scratch-test loads (N)
25.6	187.9	2.67	0.03 (Ball)	Lc1=16.6
(± 3.1)	(± 16)	(± 0.04)	0.05 (Disc)	Lc2=28.0
			(± 0.01)	Lc3=29.5

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