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Friction properties of DLC/DLC contacts in base oil

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ABSTRACT

Diamond-like carbon (DLC) coatings are promising surface coatings in terms of friction and wear performance and are beginning to be introduced in internal combustion engines, mainly because of the need to reduce friction and because of compositional constraints on engine lubricants. This paper compares the friction and wear behaviour of twelve different types of DLC coating lubricated with an API Group III base oil. A clear dependence of friction and wear on DLC type has been observed. ta-C coatings provide lower boundary friction than the other types. W-DLC coatings experience more wear whereas Si-DLC and a-C:H coatings show very little wear.

1. Introduction

The first use of diamond-like carbon (DLC) coatings was reported in 1971 by Aisenberg and Chabot [1], who showed that they possessed good resistance to scratch and corrosion. In recent years, DLC coatings have drawn much attention because of their excellent tribological properties, including very low friction, high hardness and resistance to wear, as well as, for biotribological applications, their bio-inertness. Many of these properties make DLC coatings appear attractive for automotive parts. In practise there are several different types of DLC coating, classified based on the percentage of sp^3 content in the film and the hydrogen and dopant content [2].

A number of researchers have evaluated the tribological performance of DLC coatings [3–21]. However, in general, these studies have tended to focus on just one or two coatings, with the exception of Ref. [20]. This means that, in view of the wide range of different available types of DLC, it is difficult to assess whether the tribological properties measured are characteristic of DLCs in general, the type of DLC examined or, indeed, just the particular DLC coating being studied. General rules are needed for the tribological behaviour of the various types of DLC available, especially if these coatings are to be used in liquid lubricated systems. This is because formulators of lubricants rarely choose the DLC coatings that their lubricants will encounter, so they need to know the effectiveness of their base oil and additives with all types of DLC coatings. This paper describes the friction and wear behaviour of a large number of different DLC coatings, obtained from several different suppliers, when lubricated with an additive-free base oil. This is part of a larger study, which looks at the behaviour of different types of DLC coatings with the various components of an engine lubricant, including base oil, individual additive solutions and fully formulated oils.

2. Test methods and materials

Friction tests were carried out using a minitraction machine (MTM). This is based on a ball-on-disc configuration, where a 19 mm diameter DLC-coated ball is loaded and rubbed in rolling–sliding conditions against a DLC-coated disc immersed in lubricant solution. The friction test conditions used in this study were: applied load = 31 N, corresponding to a contact pressure of about 1 GPa; entrainment speed = 0.1 m/s, slide–roll ratio = 0.5, temperature = 100 °C and test duration = 2 h. The entrainment speed is defined as $(u_b+u_d)/2$, where u_b and u_d, respectively, are the speed of the ball and disc with respect to the contacting surfaces, while the slide–roll ratio SRR is defined as the ratio of sliding speed ($|u_b-u_d|$) to entrainment speed. The calculated lambda ratio (ratio of calculated elastohydrodynamic lubricant film thickness to composite surface roughness) was of order 0.3, so the operating regime was mixed-boundary lubrication.

An initial Stribeck curve was taken prior to beginning of prolonged rubbing, by measuring friction while varying entrainment speeds from 0.007 to 3.5 m/s at a fixed slide–roll ratio of 0.5. Then prolonged slow speed rubbing was carried out at the test conditions mentioned

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Table 1
Details of DLC coatings investigated, H and W are in atomic%.

No.	DLC	H (%)	W (%)	sp^3 (%)	No. of layers	Interlayer/inclusion*	R_a (nm)	Hardness (HV)	Elastic modulus (GPa)	Deposition technique
1	a-C:H:W	15	21	20	2	–	15	1190 ± 60	118 ± 6	PVD/PECVD
2	a-C:H:W	15	18	15	4	*Graphite	12	1318 ± 35	165 ± 3	PVD/PECVD
3	a-C:H:W	15	12	20	3	Cr	12	1183 ± 80	135 ± 5	PVD
4	a-C:H:W	15	14	25	3	Cr	14	1250 ± 80	140 ± 7	PVD
5	Si-DLC	20	–	30	1	Ti	24	1315 ± 130	90 ± 3	PVD/PECVD
6	ta-C	< 1	–	40	1	Ti	42	4510 ± 620	407 ± 45	PVD
7	ta-C	< 1	–	75	1	–	22	6793 ± 350	473 ± 25	PVD
8	a-C:H	17	–	35	2	Cr	10	2365 ± 87	197 ± 5	PECVD
9	a-C:H	18	–	35	2	Cr	16	2500 ± 76	200 ± 6	PVD/PECVD
10	a-C:H	25	–	38	1	Si	10	2372 ± 140	172 ± 7	PECVD
11	a-C:H	22	–	35	3	Si	10	2500 ± 60	183 ± 2	PECVD
12	a-C:H	20	–	35	1	–	10	2460 ± 145	175 ± 7	PECVD
13	AISI 52100 Steel	–	–	–	–	–	10	760 ± 10	210 ± 5	

above. Periodically, after 5, 15, 30, 60 and 120 min, the slow speed test was halted and a Stribeck curve was taken. In this study, only Stribeck curves taken initially and after 2 h rubbing are shown. The use of mixed sliding–rolling meant that both surfaces move with respect to the contact, with neither surface being in contact continuously, as is the case in pure sliding. After testing, the discs were rinsed in cyclohexane before the wear track was examined by a series of techniques, including AFM (Veeco Explorer in contact mode), SEM/EDX (Hitachi S3400 VPSEM and Inca EDX system), Raman spectroscopy (Renishaw 1000 confocal system) and an optical microscope.

Wear tests were conducted using the ball-on-disc MTM operating in disc-reciprocating mode. The ball was held stationary and the disc reciprocated, resulting in a pure sliding contact. Test conditions were: applied load = 31 N, corresponding to a contact pressure of about 1 GPa; frequency = 10 Hz; stroke length = 4 mm; temperature = 100 °C; test duration = 4 h. After wear tests, the tested samples were rinsed in cyclohexane and the tribolayers (if any) were removed by solvents before measuring the wear volumes of the tested ball and disc using an optical white light interferometer (WYKO NT 9100). The wear rates of individual components, *i.e.* ball and disc, were calculated from their respective wear volumes using the following equations:

$$k_b = \frac{V_b}{2 \cdot \Delta x \cdot n \cdot W}, \quad k_d = \frac{V_d}{2 \cdot \Delta x \cdot n \cdot W}$$

where, V_b and V_d are the wear volumes of ball and disc, respectively, k_b and k_d are the ball and disc wear coefficients, respectively, W = normal load, Δx = stroke length and n = number of cycles.

DLC coatings were deposited on the AISI 52100 steel balls and discs normally used with the MTM. Before coating, these balls and discs had hardness = 760 HV and root mean square, R_q, roughness = 10 nm. Twelve different types of DLCs were studied, including four tungsten-doped hydrogenated amorphous carbons (a-C:H:Ws) with varying tungsten concentrations, one silicon-doped DLC, two tetrahedral amorphous carbons (ta-Cs) and five hydrogenated amorphous carbons (a-C:Hs) with varying hydrogen concentrations. The thickness of the DLC coatings studied ranged between 3 and 4 μm. The properties and chemical composition of the DLC coatings are listed in Tables 1 and 2, respectively. Fig. 1 shows the AFM topography of all the deposited DLC coatings. All the tests were made on DLC on DLC-coated tribopairs. Although in practise a DLC-coated surface will normally be rubbed against a metallic one, the use of a DLC/DLC tribopair enabled the impact of the DLC type on friction and wear, and the effect of possible interactions of lubricant with DLC, to be observed unambiguously.

The base fluid used in this study was an additive-free API Group III oil having density of 0.78 g/cm^3, viscosity of 3.32 cP and effective pressure viscosity coefficient of 16.3 GPa^{-1} at the test temperature of 100 °C.

Table 2
Chemical composition (atomic%) of as-deposited DLC coatings.

As-deposited	C	O	Si	Ti	Cr	Fe	Co	Ni	Ag	W
W-DLC										
1	77	4.7	–	–	–	–	3.3	–	–	15
2	76	8.4	–	–	–	0.6	2.0	–	–	13
3	85	–	–	–	–	0.8	–	2.2	–	12
4	90	–	–	–	–	–	–	2.0	–	8
Si-DLC										
5	91	4	4.5	0.5	–	–	–	–	–	
ta-C										
6	98	–	–	1.1	–	0.9	–	–	–	–
7	97	0.9	–	–	0.1	2.0	–	–	–	–
a-C:H										
8	95	–	–	–	4.8	0.2	–	–	–	–
9	98	–	–	–	1.9	–	–	–	0.1	–
10	94	–	1.0	–	–	1.0	–	–	0.1	–
11	98	–	1.9	–	–	0.1	–	–	–	–
12	99	–	1.0	–	–	–	–	–	–	–

3. Results

3.1. Hardness and elastic modulus measurements

The hardnesses and elastic moduli of all the DLC coatings were measured using a microindentation hardness testing system, Fischerscope HM2000, operated according to ISO 14577-1. This work was carried out at Fischer Instrumentation (GB) Ltd., Lymington, UK. All measurements were carried out at 5 mN load and 0.2 μm indentation depth. The indentation depth was chosen such that it should be less than 10% of the total layer thickness of the coatings to avoid influence of the substrate material [17]. Six measurements were taken in order to obtain a good statistical representation. The mean and their standard deviations values of hardness and elastic modulus are presented in Table 1. ta-Cs and in particular DLC 7 showed high hardness and modulus compared to the other coatings.

3.2. Hydrogen and tungsten content measurements using RBS/EBS/ERD/PIXE

Rutherford backscattering spectrometry (RBS) is an accurate, thin film depth profiling technique typically carried out with 2 MeV He