



Friction and wear behaviour of Mo–W doped carbon-based coating during boundary lubricated sliding



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ABSTRACT

A molybdenum and tungsten doped carbon-based coating (Mo–W–C) was developed in order to provide low friction in boundary lubricated sliding condition at ambient and at high temperature. The Mo–W–C coating showed the lowest friction coefficient among a number of commercially available state-of-the-art DLC coatings at ambient temperature. At elevated temperature (200 °C), Mo–W–C coating showed a significant reduction in friction coefficient with sliding distance in contrast to DLC coatings. Raman spectroscopy revealed the importance of combined Mo and W doping for achieving low friction at both ambient and high temperature. The significant decrease in friction and wear rate was attributed to the presence of graphitic carbon debris (from coating) and ‘in situ’ formed metal sulphides (WS₂ and MoS₂, where metals were supplied from coating and sulphur from engine oil) in the transfer layer.

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1. Introduction

Metal-free and metal-doped diamond-like-carbon (DLC) coatings are extensively used as tribological coatings for engine parts due to their excellent combination of low friction and improved wear resistance properties. Engine components such as piston rings, piston pins, cam followers, cam shafts, rockers, gears and tappets are often coated with DLC and involve interfacial contacts with either steel or DLC coated surfaces in the presence of lubricant (formulated engine oil). But it is quite challenging to use DLC on the components, which are operated at high temperature and pressure conditions and at high sliding velocity (such as piston-cylinder and valve-train assembly, where maximum operating temperature typically is in the range of 300 °C and 150 °C respectively [1]).

It is well understood that DLC coatings show low friction and high wear resistance at ambient temperature due to formation of graphitic tribolayer at the asperity contacts [2,3]. The tribological

behaviour of these coatings however is strongly influenced by the operational environment. Hydrogenated DLC coatings for example show higher friction with increasing humidity whereas lower friction is observed for hydrogen-free DLC (a-C and ta-C) coatings due to termination of the dangling bonds. Moreover with increase of the test temperature (100–300 °C), both graphitisation and oxidation of DLC coating degrade the coating properties leading to a substantial increase in both friction and wear coefficients [4]. However DLC coating can survive comparatively higher temperature in the presence of lubricant, which isolates the coating from the hostile environment of the surroundings and acts as a coolant. The tribological performance at high temperature mostly depends on the test temperature, reactivity of the lubricant with the sliding surfaces (such as steel, DLC-coated, metal-doped DLC-coated, etc.), coating architecture and the coating deposition procedure. Therefore in high temperature applications (200 °C and above), it is imperative that the coatings possesses qualities like high thermal stability, strong coating–substrate adhesion, low coefficient of friction and high wear resistance. Furthermore it is important to understand that the coating and the surrounding ambient are two parts of one special tribo-system where the interplay between these two parts defines the overall tribological performance. Several approaches have been explored to achieve enhanced performance.

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One such approach is the exploitation of the tribolayer formation process during lubricated sliding. It is well known that the tribological performance of the DLC coatings can be successfully manipulated by the chemistry of the lubricants used. For example, some of the state-of-the-art formulated engine oils contain anti-wear (AW) additives such as Zinc dialkyl dithiophosphate (ZDDP), extreme-pressure (EP) additives and friction modifier such as molybdenum dithiocarbamate (Mo-DTC). During sliding, Mo-DTC is thermally decomposed forming tribolayer containing both MoS₂ and MoO₃ [5–8]. The friction is significantly reduced due to formation of MoS₂; however it has been shown that MoO₃ acts as a third body abrasive media, which increases the wear rate of the DLC coating [9]. An optimised concentration of ZDDP in the engine oil can promote the formation of MoS₂ over MoO₃ and reduce but not completely remove the negative effect of the MoO₃ [10,11]. It is accepted in general that, formation of MoS₂-containing tribolayer reduces the friction when steel or DLC-coated surfaces are used as counterfaces.

When the engine oil is free of friction modifier, the MoS₂-containing tribolayer cannot be formed leading to higher friction coefficient. Fairly high friction coefficient ($\mu \sim 0.1$ – 0.3) was reported for amorphous DLC (a-C:H) coating in the temperature range of 20–200 °C, when lubricant was free from Mo-DTC but composed of base oil (such as polyalphaolefin or mineral oil) and additives (AW and EP) [12,13]. Unlike amorphous DLC, significantly low friction coefficient is observed for tungsten-doped DLC coatings. Research showed that the chemical reactions occurring between the coating and the EP additives of the engine oil produced WS₂-containing tribolayer. The WS₂ compound played the role of friction reducer due to its crystallographic structure. Formation of WS₂-containing tribolayer was well documented for W-DLC coatings during lubricated sliding at ambient temperature [14] as well as in the temperature range of 50–200 °C [13,15–19]. For example, reactive magnetron sputtered WC-doped hydrogenated DLC coating (having multilayer structure of WC and a-C:H) showed $\mu \sim 0.055$ at ambient temperature due to formation of WS₂-containing tribolayer [14]. In another study, reactive magnetron sputtered W-DLC coating maintained similar friction coefficient ($\mu \sim 0.1$ – 0.3) in the temperature range of 50–200 °C due to formation of WS₂-containing tribolayer [13,19], but it started to decompose at 200 °C leading to high wear rate of the counterpart [13]. In all the cases discussed above, the formation of low-friction tribolayer during lubricated sliding plays an important, critical role for maintaining low coefficient of friction.

Another approach for further reduction of the friction is doping DLC with different metals. For example, the RF magnetron sputtered Ti-DLC and Mo-DLC coatings showed much lower friction ($\mu \sim 0.03$ and $\mu \sim 0.05$ – 0.1 respectively) compared to metal-free DLC coating ($\mu \sim 0.2$) when SAE 5W-20 formulated engine oil (contains polyalphaolefin, ZDDP and Mo-DTC) was used as lubricant. The low friction of Ti-DLC and Mo-DLC coatings was attributed to the formation of high strength metal carbides in the defective parts of the carbon network and formation of MoS₂-containing tribolayer due to thermal decomposition of Mo-DTC [20]. Similar to ambient temperature, formation of MoS₂-containing tribolayer in the temperature range of 50–100 °C decreases the friction ($\mu \sim 0.02$ – 0.14) of various metal-doped (such as Si-doped, Ti-doped and W-doped) DLC coatings under different test conditions [10,21].

The addition of suitable doping elements into the DLC coating delays the graphitisation process and therefore is seen as a highly resourceful approach for preservation of coating properties to higher temperatures. It has been found that doping with Si, Mo or W increases the thermal stability of DLC coating up to ~ 500 °C [22–25]. The formation of metal carbide phases during annealing stabilises the diamond-like structure and increases the thermal stability compared to metal-free DLC coating. Both the Mo-DLC and

W-DLC coatings provide low friction at ambient condition. Depending on the presence of friction modifier in the lubricant, they are also able to provide reduced friction in elevated temperature.

However, with further increase in test temperature (200 °C and above) all the coatings discussed above significantly increase their friction and wear rate due to degradation of their properties. Therefore, these coatings cannot be classified as suitable for components used in high temperature applications and further research is needed to provide solution for this demand.

The aim of this research is to develop a new generation metal-doped carbon-based coating which is simultaneously doped with Mo and W. It is expected that the Mo–W doped carbon-based coating (Mo–W–C) will be able to produce “in situ” a low-friction tribolayer during lubricated sliding via tribochemical reactive process taking place at the asperity contacts, which will allow operation at higher temperatures.

Further aim is to produce coating with highly dense microstructure and strong coating–substrate interfacial adhesion which will prevent coating delamination and therefore withstand more effectively wear during sliding. DLC research is well established and large number of vacuum based deposition techniques have been used over the years for the production of the various individual coatings belonging to this family. Most of these techniques suffer from poor adhesion, lower density and high residual stress. We report first time the application of the high power impulse magnetron sputtering (HIPIMS) as a technology for production of Mo–W doped carbon-based coatings. The high ionisation degree of the plasma generated in the HIPIMS discharge provides conditions for deposition of highly dense, strongly adherent and very smooth (low growth defect density) coatings [26–28] which is of paramount importance for any tribological coating. This study represents the most in-depth research so far in the understanding of microstructure formation and wear mechanism in the system.

2. Experimental details

2.1. Sample preparation and coating deposition

Prior to coating deposition, mirror polished (average $R_a \sim 0.01$ μm) M2 grade HSS disc samples ($\varnothing$ 30 mm $\times$ 6 mm) were cleaned in an industrial sized automated ultrasonic cleaning line using alkali water solutions to remove surface impurities and then dried in a high temperature vacuum drier before loading into the coating chamber. The Mo–W doped carbon-based coating (Mo–W–C) was deposited using combined HIPIMS and UBM techniques in an industrial sized Hauzer HTC 1000–4 PVD coating machine enabled with HIPIMS technology. A 200 nm thick base layer was deposited in reactive Ar + N₂ atmosphere in order to enhance the coating–substrate adhesion, followed by the deposition of ~ 2.2 μm thick Mo–W–C coating in non-reactive Ar atmosphere. The coating architecture consisted of a HIPIMS engineered interface, Mo–W–N base layer and a Mo–W–C top layer. The coating showed high adhesion with substrate (scratch adhesion test critical load $L_c \sim 80.8$ N) and moderate hardness (~ 1677.5 HV). More details about the deposition of Mo–W–C coating and the mechanical properties of the as-deposited coating were provided elsewhere [29].

The tribological properties of the Mo–W–C coating during boundary lubricated sliding at ambient temperature were compared with a range of commercially available state-of-the-art diamond-like carbon (DLC) coatings. The lowest friction coefficient was shown by DLC(Cr/Cr–WC/W:C–H/a:C–H) when compared to other DLC coatings namely DLC I, DLC II and DLC III. The DLC(Cr/Cr–WC/W:C–H/a:C–H) coating is a well-established state-of-the-art coating which has a special architecture containing a Cr base layer,

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