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CVD diamond water lubricated tribosystems for high load planar sliding

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ABSTRACT

Water lubricated tribological systems involving chemical vapour deposited (CVD) diamond coatings are appealing to many practical situations, namely in metalworking and fluid handling applications. The motivation behind the present study lies in insufficient knowledge on the behaviour of these water-based lubricated tribosystems especially under high loads. The microwave plasma chemical vapour deposition technique was used to coat dense Si_3N_4 substrates from CH_4–H_2 gas mixtures, setting adequate deposition parameters during 2.5 h, resulting in a film thickness around 15 μm. Self-mated planar reciprocating sliding ball-on-flat wear tests were performed up to 16 h (690 m of sliding distance) with normal applied load ranging from 70 N to 160 N. SEM and AFM characterisation techniques were used to identify the prevailing wear mechanisms. The worn surfaces exhibit a polished appearance resulting from diamond asperities abrasion on the counterface combined with the action of nanometric debris. Some longitudinal wear grooves along the sliding direction are the result of two-body abrasive action of larger debris spawn from the truncation of aggregates of crystals in the first steps of sliding. A steady-state sliding regime sets for almost the test full duration, with a very low friction coefficient in the range 0.04–0.05 and a wear coefficient of about 10^{-8} mm^3 N^{-1} m^{-1}. Partial film wear-out was found at a threshold load of 160 N, however without seriously affecting the tribosystem performance.

1. Introduction

The study of water lubricated CVD diamond tribological systems is important in order to anticipate the frictional and wear response of such highly protective coatings in many practical situations. The intentional use of water-based lubricants is found in metalworking technologies, like wire drawing [1] and cutting or grinding operations [2]. Special applications in fluid handling industry, as in mechanical face seals [3,4], undergo wet functioning after the initial dry running-in regime. Still, information involving CVD diamond-on-diamond water lubricated tribosystems is scarce and, therefore, comprehensive studies on the subject are in the agenda.

In a paper by Grillo and Field [5], a natural diamond stylus was made to slide against a (100) textured hot filament CVD polycrystalline diamond film deposited on Si_3N_4 ceramic substrates. The experiments were performed in water solutions of different pH at low speeds (0.05–0.2 mm s^{-1}) and small applied loads (0.5–2 N). For the neutral de-ionized water solution, sliding speed of 0.2 mm s^{-1} and applied load of 1 N, they observed a reduction of the friction coefficient from its value in air, around 0.1, to 0.07. This slight decrease was attributed to the passivation of surfaces originating from the chemisorption of hydride and hydroxyl groups on the surface. In line with earlier studies of diamond friction in water or moist air, the reduction in friction has been attributed to the formation of thin hydrocarbon waxy surface layers [6,7]. A similar trend has also been observed for CVD diamond coatings by Enomoto et al. [7], who suggested the possible occurrence of a tribochemical wear mode on diamond surfaces reacting with water or moisture.

In the studies indicated above, tests were performed under small applied loads and, generally, constitute more fundamental works concentrated on the friction properties of diamond coatings. As such, a better understanding on both the friction and wear performance of CVD diamond submitted to high applied loads is needed to assess their tribological properties under more intense sliding conditions, better approximating practical use.

The present authors have previously characterized the unlubricated tribological behaviour of homologous pairs of CVD diamond coated Si_3N_4 [8]. The steady-state coefficient of friction displayed values in the range 0.03–0.04, regardless of the applied load (10–80 N). The wear coefficient values for both ball and plate specimens revealed a mild wear regime (10^{-8} mm^3 N^{-1} m^{-1}). Above a threshold applied load of 80 N, it was observed gross film detach-

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ment induced by the tribological action. The present study now deals with the CVD diamond-on-diamond planar sliding water lubricated contact at moderate to high normal loads (70–160 N). The frictional behaviour is related to existing models of polycrystalline diamond friction and predominant wear mechanisms were identified. Results concerning the maximum sustainable load for CVD diamond coated Si_3N_4 ceramics submitted to tribological stress are also presented.

2. Experimental procedure

Dense silicon nitride (Si_3N_4) ceramics were used as the substrate material for the deposition of CVD diamond. Details regarding substrate processing can be found in a previous work by the authors [8]. Geometries used in the present work were as follows: circular discs with 10 mm diameter and 3 mm of thickness, and 5 mm diameter balls. Before diamond deposition, the flat samples were subjected to the following surface pre-treatment: 46 μm grit size diamond wheel grinding, 15 μm diamond polishing on a metallic plate, and manual scratching with 0.5–1 μm sized diamond powder on a silk cloth. Subsequently, the Si_3N_4 discs were rinsed in an acetone ultrasonic bath for 10 min and then in an ethanol bath for 10 min. Concerning the Si_3N_4 balls, these were gradually polished with 15, 6 and 1 μm sized diamond powder suspension followed by microflawing by ultrasonic agitation with diamond suspension (0.5–1 μm) in *n*-hexane to promote diamond nucleation.

A microwave plasma reactor was used for the production of CVD diamond coatings on both the flat and ball specimens. The deposition parameters of the MPCVD process were as follows: microwave power = 3 kW; chamber total pressure = 1.2×10^4 Pa; H_2/CH_4 gas flow = 400/25 standard cubic centimetre per minute (sccm) and 400/16 sccm for the flat and ball specimens, respectively. The selected growth conditions were previously reported to lead to similar film thicknesses for both geometries, around 12–14 μm, after 2.5 h of deposition [8].

Planar sliding wear tests were performed in a ball-on-flat reciprocating configuration. Prior to each tribotest, both plate and ball specimens were cleaned with alcohol for 10 min in an ultrasonically agitated bath. The diamond coated balls were mounted as the upper sample and the disc shaped flat specimens were fixed to the oscillating table. The contact region remained immersed in a pool-type container filled with distilled water. The tests were performed at constant stroke (6 mm) and a frequency of 1 Hz, corresponding to an average linear velocity of 9 mm s^{-1}, during 16 h (690 m of sliding distance). The normal applied load varied in the range 70–160 N. The friction coefficient was determined from measurements of the tangential force exerted on a load cell attached to the upper sample holder. The wear coefficient (k) of the ball specimens was estimated from the diameter (d) of near-circular wear scars, the ball radius (r), the applied normal load (W) and sliding distance (x) following Archard's law of wear [9]:

$$k = \frac{\pi d^4}{64 r W x}$$

The surface topography of the as-deposited and worn films was characterized by atomic force microscopy (AFM), using tapping mode. The surface roughness parameter R_q (RMS), as determined from AFM $50 \times 50\ \mu m^2$ scans, was used to quantify surface degradation. The coatings morphology of pristine and damaged specimens was observed by scanning electron microscopy (SEM), which also allowed direct measurements of the radius of the ball wear scars and wear debris.

3. Results

Tribotesting of CVD diamond coated Si_3N_4 ceramics was performed using two ranges of applied normal load magnitudes hereafter labelled as moderate and high. The first group of experiments involved applied load values in the range 70–90 N and were selected based on experimental data from former studies using self-mated CVD diamond systems under dry sliding conditions [8]. To also assess the maximum sustainable load of deposited CVD diamond coatings under planar sliding, i.e. prior to delamination by gross film detachment or film wear-out, the second group of tests involved harsher conditions using heavier loads in the range 110–160 N.

3.1. Moderate loads (70–90 N)

In Fig. 1, SEM micrographs of as-deposited and worn CVD diamond coated Si_3N_4 films are depicted. Fig. 1a and b show the surface morphologies of both the as-deposited plate and ball spec-

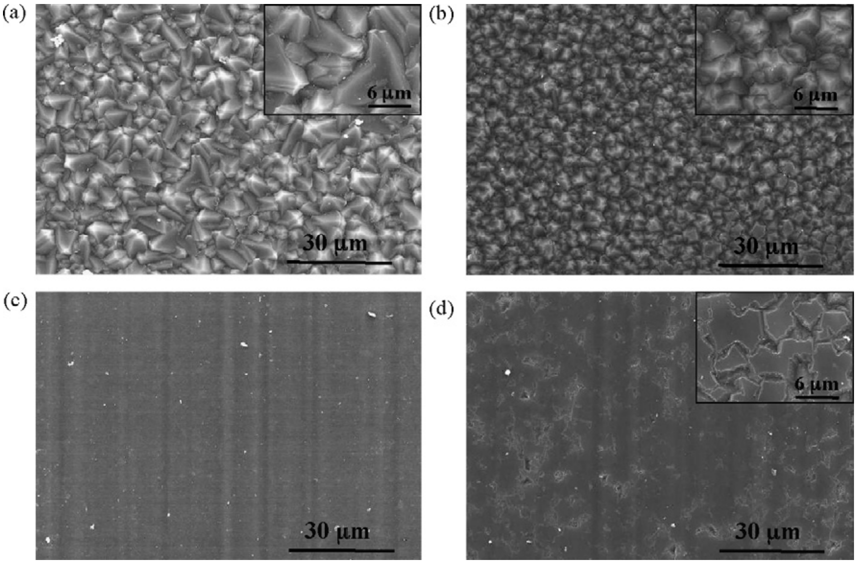

Fig. 1. SEM micrographs of as-deposited CVD diamond coatings: (a) on the ball; (b) on the plate. Worn surfaces after self-mated testing under 70 N: (c) ball; (d) plate. High magnifications are shown on the insets.