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Study of tribological performance of ECR–CVD diamond-like carbon coatings on steel substrates
Part 2. The analysis of wear mechanism

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Abstract

This paper describes the tribological performance of diamond-like carbon (DLC) coatings deposited on AISI 440C steel substrates by electron cyclotron resonance chemical vapor deposition (ECR–CVD) process. A variety of analytic techniques were used to characterize the coatings, such as Raman spectroscopy, atomic force microscopy (AFM) and nano-indentation. The sliding wear and friction experiments were carried out by the conventional ball-on-disk tribometry against 100Cr6 steel counterbody at various normal loads (1–10 N) and sliding speeds (2–15 cm/s). All the wear tests were conducted under dry sliding condition in ambient air for a total rotation cycle of 1×10^5 (sliding distance ∼2.2 km). Surfaces of the coatings and the steel balls were examined before and after the sliding wear tests. The DLC coatings that had been tested all showed relatively low values of friction coefficient, in the range of 0.1–0.2 at a steady-state stage, and low specific wear rates (on the order of 10^{-8} mm^3/Nm). It was found that higher normal loads or sliding speeds reduced the wear rates of the coatings. Plastic deformation became more evident on the coating surface during the sliding wear test at higher contact stresses. The friction-induced transformation of the coating surface into a graphite-like phase was revealed by micro-Raman analysis, and the flash temperature of the contact asperities was estimated. It was suggested that the structural transformation taking place within the wear tracks was mainly due to the formation of compact wear debris layer rather than the frictional heating effect. On the other hand, an adherent transfer layer (tribolayer) was formed on the counterface, which was closely related to the steady-state friction during sliding and the wear mechanisms. Fundamental knowledge combined with the present tribological study led to the conclusion that adhesive wear along with abrasion was probably the dominant wear mechanism for the DLC/steel sliding systems. Additionally, fatigue processes might also be involved in the wear of the coatings.

Keywords: Diamond-like carbon (DLC); Steel; Electron cyclotron resonance (ECR); CVD; Tribology; Friction; Wear mechanism

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1. Introduction

Diamond-like carbon (DLC) coatings have attracted much attention in tribological applications due to their unique combination of properties such as high hardness, low friction coefficient, high wear resistance, and chemical inertness [1–3]. Currently different methods including PVD and chemical vapor deposition (CVD) are available for the production of DLC coatings on various substrate materials [4–6]. From the vast number of investigations of their mechanical and tribological properties, it appears that DLC coatings are promising for industrial applications as wear resistant solid lubricants on tools or mechanical components.

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Generally, the friction and wear behaviors of DLC coatings depend largely on the deposition method, processing parameters, and operating conditions, etc. [7]. The friction coefficients of DLC coatings usually span a range of 0.1–0.4 in ambient air, and are strongly governed by tribochemical effects [8]. Therefore, tribochemical reactions in various types of environment have also attracted special attention, although DLC coatings usually exhibit good chemical inertness. Additionally, the coatings also demonstrate high wear resistance and protect the rubbing surface of counterpart from extensive wear. Up to now various theoretical mechanisms have been proposed to explain the tribological performance of DLC coatings [6]. The observed low values of friction coefficient have been associated with the amount of hydrogen present in the coatings and the graphitization of the surface during sliding wear [9–11]. It was found that the low friction coefficients of DLC coatings in the steady-state after a long-distance sliding were due to the formation of a carbon-rich transfer layer on the counterface [12]. Liu et al. [13] showed with evidence that the transfer layer contained a fine distribution of graphite nanoparticles in a distorted diamond-like structure, and a graphitization process took place within the wear track region of the coatings under thermal and strain effects from the repeated friction. The low wear rates were thus related to the formation of the graphitized layer with low shear strength between the two surfaces in contact. Some investigations have also confirmed that both friction and wear performance of DLC appeared to be controlled by this interfacial transfer layer that had a lubricating effect in the contact surface [14,15]. For hydrogenated DLC coatings, it was postulated that the surface hydrogen atoms could desorb during friction or heating to 300–400 °C, and the formation of the transfer layer could be enhanced by hydrogen [16,17]. However, Voevodin et al. [18] suggested that the sp^3 to sp^2 phase transformation (graphitization process) was probably induced by stresses from the friction contacts rather than by the frictional heating effect. Jiang et al. [19,20] have correlated the low wear rates with the formation of compact layer from wear debris particles. The generation of wear debris particles could probably related to a micro-fatigue process [20]. Therefore, the physical and/or chemical interactions between two bodies in contact during sliding wear are rather complicated. The proposed mechanisms for the friction and wear performance of DLC are still subjected to controversy and have not been elucidated as yet [13,21–24].

The purpose of the present investigation is to understand the wear mechanisms for DLC coatings deposited by electron cyclotron resonance chemical vapor deposition (ECR–CVD) process in unlubricating sliding against steel balls. A physical model for the wear process during sliding is also presented based on the detailed analysis of the contact surfaces, and the experimental results for the wear tests are explained accordingly.

2. Experimental details

DLC coatings were deposited on the polished surfaces of AISI 440C steel substrates ($R_a \sim 0.03\,\mu$m) by ECR–CVD process from a gas mixture of acetylene (C_2H_2) and Argon (Ar) at a flow ratio of 0.6. The microwave power was fixed at 400 W. The preparation procedure has been described in detail in Part 1 of this paper. During deposition, a bias voltage was applied to the substrate holder using a 13.56 MHz rf power supply. It has been shown that the mechanical properties of the coatings are sensitive to the rf-induced dc self bias voltage [25], which determines mainly the energy of ion species impinging on the growing films. The DLC coatings used in this study were approximately 1 μm thick and deposited at a bias of −180 V. Under this optimum condition, the hardness, and Young's modulus of the deposited DLC coatings were 25 and 220 GPa, respectively, as determined by nanoindentation.

The friction and wear tests of the DLC coatings were carried out with a CSEM ball-on-disk tribometer under ambient environment (20–25 °C, 45–55% RH). Only dry sliding wear against 100Cr6 steel balls (6.0 mm in diameter) was performed in this experiment. The hardness of the steel balls was 62–65HRC, and their surface roughness was around 8 nm centerline average. The applied normal loads were selected to be 1, 2, 5, and 10 N, creating initial Hertzian stresses of 0.7, 0.9, 1.2, and 1.5 GPa, respectively. At the same time, the sliding speed was varied between 2 and 15 cm/s. The flash temperature of the contact asperities during sliding depends strongly on the sliding speed and the normal load. All the wear tests were conducted for a total rotation cycle of 1×10^5(corresponding to a sliding distance of ∼2.2 km), and the frictional plot against sliding distance could be recorded continuously. After the wear testing, the worn surfaces of the coatings and the counterpart were examined by optical microscopy. In fact, atomic force microscopy (AFM) is a very useful and powerful tool for observing a surface structure with three-dimensional (3D) images under ambient conditions, and has an advantage of extremely high space resolution. For this reason, in the present study, AFM (Park Scientific Instruments) was also used to identify the morphological characteristics of the contact surfaces both before and after the sliding wear test. The AFM imaging was performed in a contact mode, and the scan size was $10\,\mu m \times 10\,\mu m$ in most cases, from which the root mean square (rms) roughness could be determined. The wear rates of the coatings and the balls were measured by surface profilometry and optical microscopy, respectively, as has been described previously [25].

Micro-Raman spectroscopy was used to investigate the bonding structure of as-deposited coatings as well as wear tracks, transfer layers, and wear debris particles. The spectra were recorded at room temperature over a range of 1000–2000 cm^{-1}, using a Renishaw 2000 spectrometer with an Ar-ion laser (λ = 514 nm) operating at a power of 20 mW.