



Effect of a hydrogenated interface on the wear behavior of a diamond-like carbon film in a water environment

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ABSTRACT

Hydrogenated diamond-like carbon (DLC) films or DLC films with hydrogenated interlayers tend to undergo early stage failure in water environments for reasons that are not fully understood. In this paper, a hydrogenated interface was prepared between a hydrogen free DLC film and a CoCrMo substrate, and the effects of the hydrogenated interface on the film structure, hardness, adhesion, wear behavior and corrosion resistance were investigated. The hydrogen free DLC films on the hydrogenated and the hydrogen free substrates exhibited similar structures and mechanical properties. The DLC films on both the hydrogenated and the virgin CoCrMo substrates exhibited good adhesion. The adhesions of the DLC films on both the substrates were not deteriorated after 150 days of immersion in 0.9% NaCl solution. The wear resistance of the DLC film on the hydrogenated CoCrMo substrate was worse than that on the virgin CoCrMo substrate in water. The presence of hydrogen improved the activity of the interface, restrained the formation of a passivation layer, and promoted the corrosion of the interface, which caused early stage failure of the DLC film in water. Therefore, the hydrogenated interface has a negative effect on the wear behaviors of DLC films in water environments.

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1. Introduction

Artificial joints have been successfully used to treat arthritis-related disabilities in the last century. The first generation of metal-on-metal (MOM) type artificial joints was proposed in the 1960s; however, their clinical applications showed undesirable wear behavior due to inaccurate geometrical arrangements. The second generation of CoCrMo MOM type artificial joints is considered to be ideal for young and active patients due to their improved wear resistance and potential to design them with large femoral heads. Yet, the applications of CoCrMo MOM type artificial joints are still under scrutiny, because their products of degradation from wear and corrosion may cause tissue inflammation and osteolysis [1].

Diamond-like carbon (DLC) is a promising material for reducing the wear and corrosion of MOM joints, due to its low friction, wear resistance, and chemical inertness [2]. Based on the presence of hydrogen, DLC films can be divided into hydrogenated DLC films and hydrogen-free DLC films, respectively. The hydrogen in hydrogenated DLC films can passivate dangling bonds via the formation of C–H bonds, leading to low friction in vacuum and inert atmospheres [3]. Hydrogenated DLC films have been used in clinical applications to modify artificial joints, but the results were less than ideal. In 2001, knee joints coated

with hydrogenated DLC (a SiO_x-containing hydrogenated DLC film) were sold by the Swiss company Implant Design AG, but they were later banned by the Swiss Federal Office of Public Health in the same year due to increased wear, partial film delamination, and loosening of the implant [4]. In 2003, Taeger et al. published data regarding the clinical use of hydrogenated DLC (deposited by ion beam) coated Ti6Al4V femoral heads [5]. Their report reveals that 45% of the DLC coated implants had to be replaced within 8.5 years. Pits and delamination were present on the retired femoral heads, indicating failure based on corrosion, fatigue, and film embrittlement. The hydrogenated DLC films mentioned in the above cases suffered early stage failure when they are used in vivo. In some vitro studies, hydrogenated DLC was also reported to be vulnerable in water-lubricated conditions [6]. Even DLC films with hydrogenated interlayers exhibited catastrophic failure in liquid environments [7,8]. It is considered that the liquid will penetrate the interface or the interlayer through film defects or pinholes, causing dissolution of the interface or the interlayer [9,10]. However, this does not fully explain the sensitivity of some hydrogenated DLC films to aqueous environments, and the stability of hydrogen-free DLC films under such conditions.

Bonded and unbonded hydrogens are present in hydrogenated DLC films. The bonded hydrogen atoms affect the structure of the DLC film, while the unbonded hydrogen atoms influence the film stress and mechanical properties [11]. In this study, a hydrogenated interface was intentionally fabricated between a hydrogen-free DLC film and a CoCrMo

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alloy in order to investigate the influence of the unbonded hydrogen atoms at the film–substrate interface on the stability of the DLC film in water environments, excluding the influence of hydrogen in the DLC film. The effect of the hydrogenated interface on the wear of the DLC film in water environments was also explored.

2. Experimental

2.1. Sample preparation

Biomedical CoCrMo alloy (Cr: 28.02%, Mo: 6.11%, Ni: 0.2%, C: 0.062%, Co: bal.; widely used as MOM joint material) was used as the substrate. CoCrMo discs (thickness 1.2 mm, Φ 14 mm) were ground and polished to a roughness of 2–10 nm. Prior to the deposition of the DLC film, the vacuum chamber was pumped down to a base pressure of 1.0×10^{-3} Pa. All the CoCrMo substrates were then sputter cleaned by Ar plasma at a DC bias voltage of 1000 V. After the Ar plasma sputter cleaning, the hydrogenated CoCrMo interface was prepared by hydrogen pulse glow discharge. Hydrogen gas was introduced into the chamber and the pressure was set to \sim 8.0 Pa. A pulse voltage (2.0 kV, 40 kHz, 20%) was then applied to the substrate. The hydrogen gas was ionized to generate hydrogenated surfaces on the CoCrMo substrates. After this process, hydrogen-free DLC films were prepared on the hydrogenated surfaces by the filtered cathode vacuum arc method [12]. High purity graphite was used as the carbon source, and the DLC film was deposited to a thickness of \sim 400 nm. The DLC film with the hydrogenated interface is labeled as CoCrMo/H/DLC as shown in Fig. 1a. For a control sample, the same DLC film was deposited on virgin CoCrMo substrate with no hydrogenated interface, and labeled as CoCrMo/DLC (Fig. 1b). The virgin CoCrMo substrate after the Ar plasma sputter cleaning is labeled as CoCrMo. The CoCrMo substrate with the hydrogenated surface is labeled as CoCrMo/H.

2.2. Characterizations of substrate and DLC film

The hydrogen content at the CoCrMo/H surface was measured by the elastic recoil detection analysis (ERDA) method (9SHD-2, China). The structural properties of the DLC films were characterized by Raman spectroscopy (Renishaw Invia, UK) using a 514 nm Ar⁺ laser. The mechanical properties of the substrates and the DLC films were evaluated using a DUH-211S dynamic ultra-micro hardness tester (SHIMADZU, Japan). The indentation hardness and the modulus were calculated from the load-unload curves under a 20 mN peak load, as described in the literature [13].

2.3. Adhesion stability of DLC films

The adhesion stability of the DLC films was evaluated by static immersion experiments and scratch tests. The CoCrMo/DLC and the CoCrMo/H/DLC samples were immersed in 0.9% NaCl solution at room

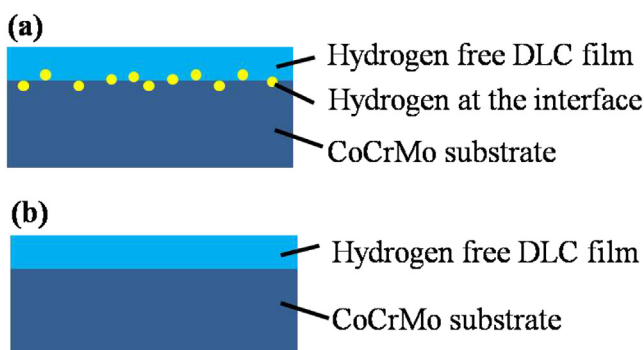


Fig. 1. Schematic diagrams of (a) CoCrMo/H/DLC and (b) CoCrMo/DLC.

temperature for 150 days. The adhesion of the as-deposited and the immersed DLC films was then measured by an MFT-2000 scratch tester (Lanzhou, China). An Al₂O₃ ball (3 mm diameter) was used to scratch the DLC films under a progressive load from 0 to 100 N. The loading speed was 100 N/min and the total scratch length was 5 mm. After the scratch test, the morphologies of the scratch traces were observed using an optical microscope (OM; Axio Lab.A1, ZEISS, Germany).

2.4. Wear testing

The wear behaviors of CoCrMo/DLC and CoCrMo/H/DLC in air and pure water were investigated using a ball-on-disc reciprocating tribometer (CSEM, Switzerland). Al₂O₃ balls with 6 mm in diameter were used as the friction pairs. The sliding velocity and the sliding distance were 2.5 cm/s and 5 mm, respectively. The applied load and the total number of cycles for each sample were 2 N and 20,000 cycles, respectively. After the wear test, the OM and a profilometer (XP2, AMBIOS, USA) were used to characterize the morphologies of the wear tracks.

2.5. Electrochemical testing

The corrosion behaviors of CoCrMo and CoCrMo/H were determined from the open circuit potential (OCP) and potentiodynamic polarization curves measured using an electrochemical workstation (IM6, Zahner, Germany). The OCP was recorded in 0.9% NaCl solution at 37 °C for 3 h. The potentiodynamic polarization curves were obtained in the voltage range of -0.5 to 1.0 V in 0.9% NaCl solution at 37 °C, at a potential scan rate of 0.1 mV/s.

3. Results

3.1. Hydrogen content, film structure and mechanical properties

The hydrogen content at the CoCrMo/H surface was detected in the depth direction by ERDA. As shown in Fig. 2, the maximum hydrogen content at the CoCrMo/H surface is \sim 35%, confirming that a hydrogenated surface was successfully prepared on the CoCrMo substrate. The structures of the CoCrMo/DLC and the CoCrMo/H/DLC samples were characterized by Raman spectroscopy, as shown in Fig. 3. The Raman spectrum of CoCrMo/DLC was similar to that of CoCrMo/H/DLC. The Raman curve can be fitted using the Gaussian function to yield D and G peaks; the I_D/I_G value is related to the number and the size of sp² clusters. The I_D/I_G value of CoCrMo/DLC was similar to that of CoCrMo/H/DLC, indicating that the structure of CoCrMo/DLC is similar to that of

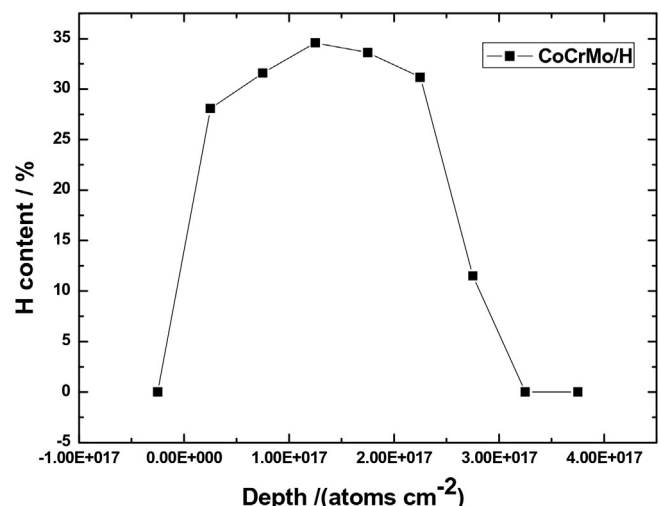


Fig. 2. Variation in the hydrogen content along the depth in CoCrMo/H.

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