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Preparation and tribological behaviors of DLC/spinel composite film on 304 stainless steel formed by cathodic plasma electrolytic oxidation

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A R T I C L E I N F O

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A B S T R A C T

In this paper, the diamond-like carbon (DLC)/spinel composite film on 304 stainless steel was fabricated by cathodic plasma electrolytic oxidation (CPEO) in glycerin solution, and its microstructure, composition and phase constituents were characterized. The friction performance of the film under dry sliding against ZrO_2 ceramic ball at ambient and high temperature up to 500 °C was evaluated. The thickness of the two-layer structure composite film reaches 18 μm under 260 V for 5 min discharge. The DLC/spinel composite film could significantly reduce the friction coefficient and improve the wear resistance of the 304 stainless steel especially at ambient temperature. The friction coefficients and wear rates of both the 304 stainless steel substrate and the composite film increase with increase of the environment temperature, but the wear process of the composite film was more stable than that of the stainless steel substrate even at high temperature. The wear mechanism of the composite film is adhesive wear at ambient temperature, but the slight plastic deformation along with adhesive could be found at high temperature.

1. Introduction

Diamond-like carbon (DLC) film has great advantages in many aspects such as mechanics, electromagnetics, chemical inertness and biocompatibility [1–3]. However, the high-cost equipment is often needed to achieve vacuum or high temperature environment, furthermore, the excessive internal stress restricts the maximum thickness of the film [4,5]. On the other hand, some Fe-Ni-Cr-O spinel structure oxides, like $NiFe_2O_4$ spinel oxide and $FeCr_2O_4$, not only have good corrosion resistance, heat resistance and wear resistance, but also display good properties of electric magnetic and biological activities [6–8].

Plasma electrolytic oxidation (PEO), namely microarc oxidation (MAO), is generally applied to anodic valve metals such as Al, Mg, and Ti [9–18] and similar ceramic films could also generate on carbon steel [19–21]. When iron and steel are set as a discharge electrode, plasma electrolytic saturation (PES) can be achieved to obtain the hardening layer [22–28]. At present, many papers about plasma electrolytic carburizing (PEC) [22–24], nitriding (PEN) [25], nitrocarburizing (PEN/C) [26], borocarburizing (PEB/C) [27], and borocarbonitriding (PEB/C/N) [28] have been published. Although the PES on anode can also be achieved, PES discharge on cathode draws more attention.

In recent years, we suggested using the method of cathodic plasma electrolytic oxidation (CPEO) to fabricate the oxide films on 304 stainless steel [29] and T8 high carbon steel [30]. The CPEO oxide films had good wear resistance at ambient temperature; however, it is concerned whether the CPEO film can sustain its friction performance at high temperature. Furthermore, it is found that the DLC component was also synthesized in CPEO oxide film on high carbon steel due to the effect of high temperature in discharge envelope around the cathode. CPEO film on 304 stainless steel contains the phases of $FeCr_2O_4$, $NiFe_2O_4$, and Fe_3O_4. It is interesting to fabricate the DLC/spinel oxide composite film.

In this paper, a DLC/spinel composite film was prepared by cathodic plasma electrolytic oxidation (CPEO). Morphology, composition and structure of the film were characterized. Its tribological behaviors at ambient and high temperature up to 500 °C were evaluated, and the wear mechanisms at different temperatures were analyzed.

2. Experimental details

A composite film containing diamond-like carbon (DLC) and spinel oxides on 304 stainless steel was successfully prepared by cathodic plasma electrolytic oxidation (CPEO) in 80 vol% glycerol solutions with

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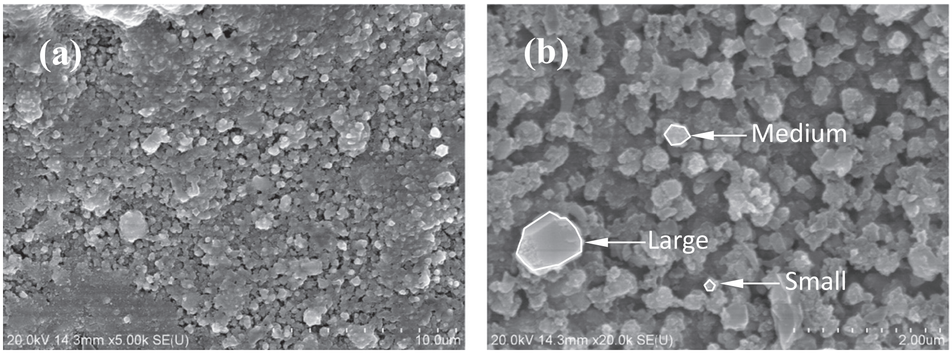

Fig. 1. Surface morphology of DLC/spinel composite film on 304 stainless steel. (b) is the magnified picture of (a).

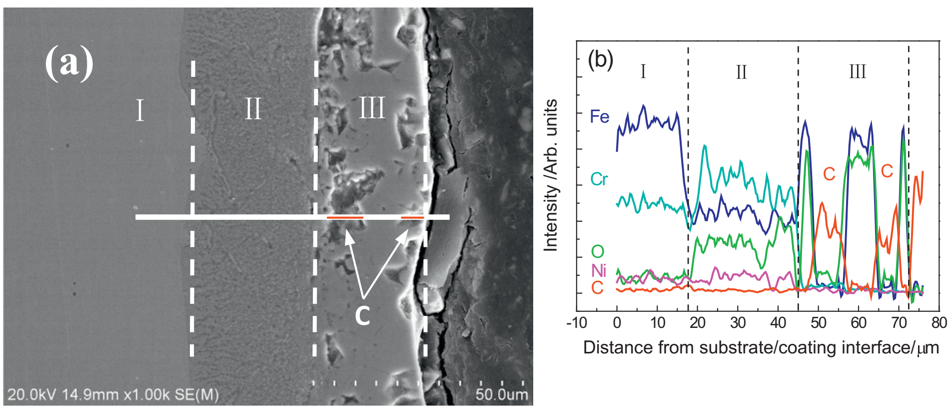

Fig. 2. Cross-section microstructure and composition profiles of the DLC/spinel composite film on 304 stainless steel. I 304 stainless steel substrate, II inner layer, III outer layer.

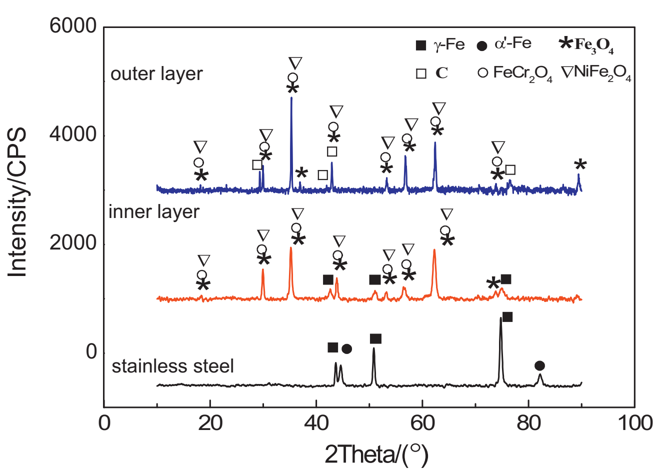

Fig. 3. XRD patterns of bare 304 stainless steel and DLC/spinel composite film.

KCl additive. Rectangular coupons with dimension 40 mm × 17 mm × 1 mm of 304 stainless steel (C: ≤0.07, Si: ≤1.0, Mn: ≤2.0, Cr: 17.0–19.0, Ni: 8.0–11.0, S: ≤0.03, P: ≤0.035, Fe rest) was mechanically polished up to 1000-grit emery paper to achieve a surface roughness $R_a \approx 0.1\,\mu m$, then cleaned in running water and dried. These coupons were set as cathode. The film was fabricated by a WHYH-100 kW electrolytic plasma unit as a DC power supply in a stainless steel container equipped with stirring and cooling systems. After achieving a stable plasma discharge envelope by initially setting the negative bias voltage of 140 V, the voltage was adjusted to 260 V for 5 min stable discharge, and a hardened film containing diamond-like carbon and spinel phases was formed.

The surface morphology and cross-sectional microstructure of the film on 304 stainless steel was studied by scanning electron microscopy (SEM, Hitachi S-4800), and the composition was analyzed by energy dispersive X-ray spectrometer (EDS). The phase constituents of film and the chemical state of elements were determined by X-ray diffraction (XRD, Philips PW-1830) with Cu K_α irradiation, Raman spectroscopy

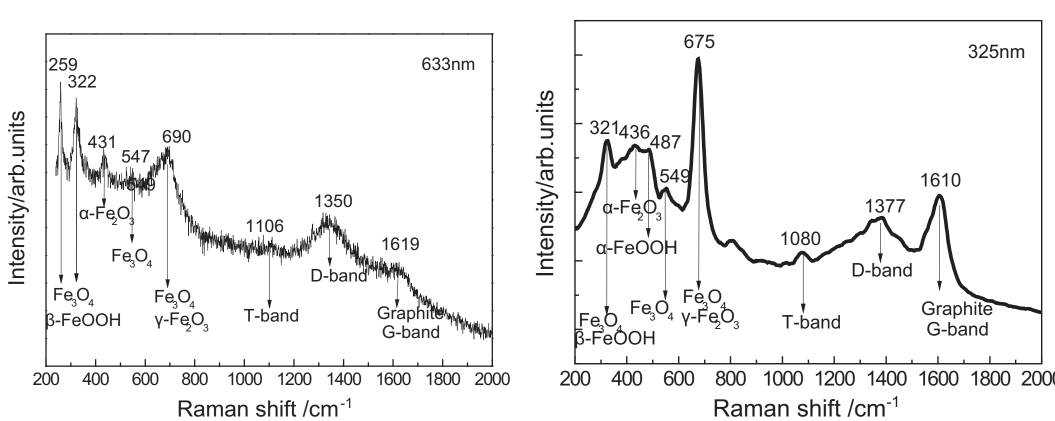

Fig. 4. Raman spectra of DLC/spinel composite film on 304 stainless steel with different laser excitation wavelength. (a) at 633 nm, (b) at 325 nm.