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Effect of gold oxide incorporation on electrochemical corrosion resistance of diamond-like carbon

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ABSTRACT

Gold oxide nanoparticles were incorporated into diamond-like carbon (DLC) films in order to improve protection of AISI-1020 from electrochemical corrosion. The AuO_x:DLC films were prepared by plasma enhanced chemical vapor deposition and were subsequently characterized by scanning electron microscopy, Raman spectroscopy and electrochemistry measurements. The electrochemical corrosion performance of the AuO_x:DLC coating was contrasted to AISI-1020 and DLC without AuO_x coating. The electrochemical techniques that were utilized for this investigation were potentiodynamic and electrochemistry impedance spectroscopy. The electrochemical analysis indicated that AuO_x:DLC films presented superior corrosion resistance as compared to DLC. This resulted in 99.8% and 96.8% protection efficiency respectively, when compared to AuO_x:DLC and DLC coatings.

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1. Introduction

In the coastal regions of Brazil and Africa, a significant fraction of petroleum and natural gas world reserves are trapped in rocks that exist in the pre-salt layer [1]. These natural resources lie beneath deep layers of salt and rock under the Atlantic Ocean [2]. Drilling through this barrier to extract these highly valuable natural resources is still exceedingly costly and requires special drill bits to cut the rock [3]. To date these drill bits have been made of steel and diamond. Diamond-like carbon (DLC) films have been especially attractive due to their high adherent coatings with exceptional wear and corrosion resistance [4,5]. These outstanding properties and recent low production costs have encouraged DLC application as protective “shield” for drill bits [6,7]. The DLC corrosion resistance is significantly dependent on the film composition and structure, which are strongly related to deposition technique, precursor gas and incorporated metals [8,9]. The incorporation of metals into the DLC matrix has shown to be effective for the reduction of film stress, improved hardness, toughness and corrosion resistance by increasing sp^3 content [10–17].

Morrison et al. showed the incorporation of silver and platinum nanoparticles into diamond-like carbon matrix prepared using a multi-component target pulsed laser deposition process [10]. These DLC films possessed hardness and Young's modulus values of about 37 GPa and 331 GPa respectively and exhibited low corrosion rates at the open-circuit potentials in a saline electrolyte. Marciano et al. showed that DLC films have higher wear resistance towards oxygen attack when silver nanoparticles are incorporated during plasma enhanced chemical vapor (PECVD) deposition [11]. Chen and co-workers reported that the incorporation of Si_3N_4 and SiO_x into DLC films produced similar hardness, however with significantly different compressive stresses, 1.5 vs 11 GPa, respectively [12]. Pal et al. presented the incorporation of amorphous chromium (Cr) nanoparticles into DLC by a technique known as “plasma-assisted magnetron sputtering” [13]. Compared to DLC, without Cr, the Cr-DLC films reduced the intrinsic compressive stresses significantly while increased its hardness. Wan and collaborators incorporated anatase titanium dioxide (TiO_2) nanoparticles into DLC films that effectively made the surface rougher and surprisingly increased the sp^3-hybridized carbon bonding [14]. Zanin et al. improved the charge transfer properties of DLC films by incorporating multi-walled carbon nanotubes (MWCNT) by a PECVD technique [15]. They observed that the incorporation of MWCNT increased the sp^2 content in the DLC coating and reducing adherence. Wei et al. investigated the incorporation of copper (Cu) and titanium (Ti) into DLC deposited by a sequential pulsed laser ablation of two targets. They observed higher fractions of sp^3 bonded carbon with immense wear resistance properties [16]. Paul et al. studied the effect of field emission of DLC

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with gold nanoparticles that were prepared by a capacitively coupled plasma-chemical vapor deposition technique [17]. They observed that the modification of the surface with the incorporation of gold nanocrystallites and an associated modulation of sp^2/sp^3 ratio in the films culminated improved field emission properties.

Actually, gold is the most resistant noble metal, with its nanophase found as Au_2O_3 or Au_2O. Au_2O_3 is a semiconductor and is more stable than Au_2O, which is a metastable endothermic system [18]. The higher stability of Au_2O_3 is attributed to a greater hybridization of Au 5d and O 2p states throughout the valence-band region as compared to Au_2O [18]. Heating to around 450 K, Au_2O_3 decomposes into Au_2O [19]. Preparation of oxide films, AuO_x, with varying oxygen concentrations, $0.2 < x < 1.5$, has also been reported where the resistivity increases with oxygen content, reaching a maximum at $x = 1.5$, corresponding to Au_2O_3 [18]. These gold oxide nanoparticles could improve DLC electrochemical corrosion resistance, which by itself is fascinating subject.

To our best knowledge, this is the first time that gold oxide nanoparticles have been incorporated into DLC coatings and having these coatings evaluated for their electrochemical corrosion resistance. Our results reveal that the new hybrid material is even more resistant to electrochemical corrosion than DLC without gold oxide nanoparticles, which is very promising for corrosion protective coating research.

2. Experimental

The AISI-1020 substrate surfaces (10.0 mm × 10.0 mm × 1.0 mm) were prepared by mechanically polishing them to a mirror-like finish cleaned using acetone in an ultrasonic bath for 15 min and then dried in a nitrogen atmosphere. In the PECVD chamber (vacuum base pressure of 1.3 mPa), the substrates were additionally cleaned by an argon discharge (1 sccm gas flow at 11.3 Pa working pressure with a discharge voltage of −700 V) for 10 min prior to deposition. The adhesion of the DLC films to the metallic surface was enhanced using a thin amorphous silicon interlayer (thickness around 200 nm). The silicon interlayer was deposited using silane as the precursor gas (1 sccm gas flow, 11.3 Pa for 12 min and a discharge voltage of −700 V) [11]. The DLC film deposition was initiated using methane as the feed gas to a thickness of around ~0.5 μm (1 sccm gas flow at 11.3 Pa for 30 min and a discharge voltage of -700 V). After the DLC deposition using methane was complete, toluene was used to complete the film growth to a thickness around 1.0 μm (at 10.7 Pa for 5 h and a discharge voltage of −700 V).

The gold oxide nanoparticles (AuO_x) were prepared by gold chloride reduction using sodium citrate in a molar ratio of 0.026, which based on Lee and Meisel methodology [20]. The AuO_x nanoparticles were centrifuged three times for 3 min at 1300 rpm. The supernatant was removed and the AuO_x was suspended in toluene at 1.4 μ mol L^{-1} following ultrasonic agitation for 10 min. This dispersion replaced the toluene during the DLC deposition to produce AuO_x:DLC films. The DLC films without any AuO_x present were grown using similar conditions to contrast the electrochemical corrosion performance.

All reagents were purchased from Sigma Aldrich with the highest purity available and the ultrapure water was collected from a Milli-Q Integral Water Purification System.

The atomic arrangement of the films was analyzed by Raman scattering spectroscopy using a Renishaw 2000 system with an Ar^+-ion laser ($\lambda = 514$ nm) in backscattering geometry. The laser had a power rating of ~0.6 mW and a spot diameter of 2.5 μm which was used to irradiate the sample. The Raman shift was calibrated in relation to the diamond peak at 1332 cm^{-1}. All measurements were carried out in air at room temperature.

The dispersion of Au nanoparticles in DLC films was analyzed using scanning electron microscopy (SEM — ZEISS EVO MA10) and a Wyko NT1100 optical profiler was used to characterize film morphology and roughness values.

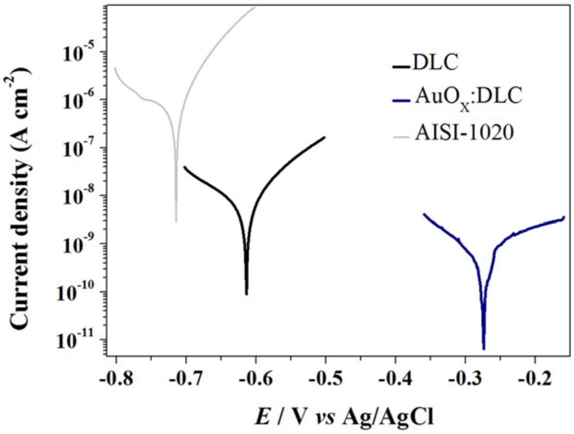

Fig. 1. Potentiodynamic polarization curves from AISI-1020 coated by AuO_x:DLC or DLC, contrasting with uncoated AISI-1020.

The electrochemical tests were performed using a standard three-electrode electrochemical cell. In the setup, the saturated Ag/AgCl (KCl 3 mol l^{-1}) was used as a reference electrode, pure platinum coiled wire as a counter electrode and the working electrodes were the AISI-1020, DLC on AISI-1020 and AuO_x:DLC on AISI-1020. The working electrode had an exposed surface area of 0.057 cm^2. The electrolyte solution was a 0.17 mol l^{-1} sodium chloride (NaCl) aqueous solution, which was not stirred and was naturally aerated. Potentiodynamic tests were performed by the polarization of samples in the anodic direction from 100 mV before and after the equilibrium potential 30 min after the immersion of samples into the electrolyte solution. The potential sweep rate was 1 mV s^{-1}. The corrosion current densities (i_{corr}) and corrosion potentials (E_{corr}) are obtained from the polarization curves. The protection efficiency (P) was calculated by $P = 100\left(1-\frac{i_{corr}}{i_{corr}{}^{o}}\right)$, where i_{corr} and $i_{corr}{}^{o}$ indicate the respective corrosion current densities in the presence and absence of the coating [21].

Electrochemistry impedance spectroscopy (EIS) measurements were also implemented in a 0.17 mol l^{-1} NaCl aqueous solution. The electrochemical impedance spectra (EIS) were obtained over the 100 kHz to 10 mHz frequency range at an open circuit potential with an alternating current (AC) excitation of 10 mV. The impedance spectra were acquired after 6 and 24 h immersions. All experiments were conducted at room temperature and a Kramers–Kronig routine was performed to ensure the linearity, causality and stability of the system through the measurements [22].

3. Results and discussion

Potentiodynamic polarization tests were used to investigate the corrosion resistance of the coating. The greatest E_{corr} of −0.274 mV was measured from the AuO_x:DLC coating. The negative OCP values for the DLC and AuO_x:DLC coatings may be caused by the penetration of the electrolyte [23,24]. The electrochemical parameters obtained from the potentiodynamic polarization curves (Fig. 1) are summarized in Table 1. The corrosion current density (i_{corr}) of AuO_x:DLC samples

Table 1
Electrochemical parameters extracted from potentiodynamic polarization curves (Fig. 1).

Samples	E_{corr} (mV)	i_{corr} (nA cm^{-2})	Protection efficiency (%)
AuO_x:DLC	−0.274 ± 0.030	0.11 ± 0.09	99.8%
DLC	−0.614 ± 0.059	1.58 ± 0.53	96.8%
AISI-1020	−0.715 ± 0.054	50.10 ± 4.52	–

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