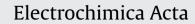
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Electrochemical behavior of diamond-like-carbon coatings deposited on AlTiC (Al₂O₃ + TiC) ceramic composite substrate in HCl solution

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ARTICLE INFO

ABSTRACT

Article history: Received 8 October 2010 Accepted 9 November 2010 Available online 18 November 2010

Keywords: DLC Composites Thin films Corrosion EIS Ceramic materials PECVD FCVA The electrochemical characterization/corrosion behavior of diamond-like carbon thin films is worthwhile to study and needed in the field (as there has been limited comprehensive evaluation of this across all types of DLC in the literature). In this paper, newly developed tetrahedral amorphous carbon (ta-C) and hydrogenated amorphous carbon (a-C:H) films, prepared by filtered cathodic arc deposition (FCVA) and plasma enhanced chemical vapor deposition (PECVD) respectively were deposited over AITiC ($Al_2O_3 + TiC$) ceramic composite substrate. Electrochemical impedance spectroscopy (EIS) and polarization measurements have been used to evaluate the coating performance in 2 M HCl solution. This ceramic substrate is used widely for the hard disk drives and read and write heads in computer. The memory of the hard disks can be increased by improving the surface quality and decreasing the pinholes. The DLC coatings were modified under different preparation conditions by changing the nitrogen-doping ratios as an attempt for improving the surface distribution and minimizing the surface coating defects.

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1. Introduction

Carbon films with very high hardness, high resistivity, and dielectric optical properties, are now described as diamond-like carbon or DLC. The pioneer study for the preparation of amorphous carbon thin films with diamond properties using ion-beam deposition (IBD) technology was reported by Aisenberg and Chabot in 1971 [1]. Since that time, the research in DLC films has increased sharply from experimental, theoretical and technological view points.

DLC coatings have some of the valuable properties of diamond. In pure form these diamond coatings offer extraordinary protection against abrasive wear and attack from atmospheric moisture and chemical vapors. DLC coatings have unique characteristics such as high hardness, low friction, electrical insulation, chemical inertness, optical transparency, biological compatibility, ability to absorb photons selectively, smoothness, and resistance to wear. They can also be deposited on all materials that can be put in a vacuum. They are certified to use in food processing and can be prepared without toxic byproducts or harmful waste. Therefore, these economically and technologically attractive properties have drawn the interest towards these coatings [2–4]. A rapidly growing variety of applications for DLC coatings are being found in tooling components, magnetic storage devices, mechanical engineering and bio-medical implants. Many researchers deposited DLC films on a wide range of metals, ceramics, glasses, and plastics by chemical vapor deposition [5–8], laser ablation [5], ion beam assisted deposition (IBAD) [9,10], IBD [11–13], dual ion-beam sputter [14], ion-beam sputter [12–15], magnetron sputtering [16], ion implantation [17–19], plasma enhanced chemical vapor deposition (PECVD) and filtered cathodic arc deposition (FCVA) methods [2,3].

Until recently, the work on DLC worldwide has not yielded the expected industrial benefits in the field of wear resistance and general mechanical performance. Most of the success has been in applications for magnetic storage media and optical coatings. The reasons for this are:

- 1. Only relatively thin coatings (<1 μ m) have been used.
- 2. The economical aspect associated with most of the deposition routes.
- 3. The difficulty to deposit coatings of good adhesion performance to metallic substrates.
- 4. Poor corrosion resistance of the deposited coatings.

The hardest amorphous carbons to date are tetrahedral amorphous carbon (ta-C) prepared by filtered cathodic vacuum arc deposition (FCVA) [20,21] and pulsed laser deposition (PLD). However, these films suffer from high internal stress. Therefore, softer,

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^{0013-4686/\$ -} see front matter © 2010 Elsevier Ltd. All rights reserved. doi:10.1016/j.electacta.2010.11.021

but less stressed, hydrogenated amorphous carbon (a-C:H) films prepared by plasma enhanced chemical vapor deposition (PECVD) [22] are still attracting the research and technological interest. These coatings are widely applied in magnetic storage materials such as AlTiC (Al₂O₃ + TiC) ceramic composite substrate. Such ceramic substrate is the base material used in magnetic head devices [23]. DLC is employed in hard disk drives to protect the flying head element against wear, which may occur during disk start-up or shut down at the landing zone [2].

Tetrahedral amorphous carbon (ta-C) with a thickness of 10 nm and hydrogenated amorphous carbon (a-C:H) films with a thickness of 50 nm, were previously prepared by filtered cathodic arc deposition (FCVA) and plasma enhanced chemical vapor deposition (PECVD) respectively. Several studies investigated the morphology of such coatings and evaluated the mechanical properties. The (ta-C) films showed a better hardness, adhesion and wear resistant compared with (a-C:H) [2,3]. These coatings have been successfully applied as anti-wear and anti-sticking layer. However, the reported data about the anti-corrosion behavior of such DLC films over ceramic composite substrate are very scarce.

There are a lot of factors affecting the corrosion resistance of DLC in chloride solution.

- 1- The sp²/sp³ ratio which indicates the conductivity/insulating properties of the DLC sample.
- 2- The hydrogen inclusion factor. It was reported that more hydrogen makes the structure porous and hence less corrosion resistant.
- 3- The type of the substrate material can affect the corrosion behavior. Despite the attractive mechanical, electrical and thermal properties of ceramic composite materials, the corrosion resistance of such materials is very poor in chloride containing environments due to the galvanic action between the alloying elements and compounds. That is why we used only one type of material which is AlTiC (Al₂O₃ + TiC) ceramic substrates in this study.

This paper aims at studying the electrochemical behavior of DLC layers prepared by PECVD and FCVA onto AlTiC ($Al_2O_3 + TiC$) ceramic composite substrates as a function of nitrogen content. The effect of hydrogen on DLC corrosion resistance was investigated by comparing nitrogen-doped hydrogenated amorphous carbon (PECVD) coatings with nitrogen-doped un-hydrogenated amorphous carbon (FCVD) coatings. The electrochemical impedance spectroscopy (EIS) and polarization measurements were used to evaluate the coating performance over two weeks of immersion in 2 M HCl solution. The optimum conditions under which such coatings can provide good corrosion protection to the ceramic composite substrate were determined. The surface morphologies of the coated samples were investigated using optical microscope with an indenter to follow the surface defects before and after corrosion in HCl over two weeks.

2. Experimental

2.1. Materials

The substrate was AlTiC ($70 \text{ wt\% Al}_2O_3 + 30 \text{ wt\% TiC}$) ceramic composite substrates which is the base material used in magnetic head devices.

2.2. Deposition of the DLC coatings

Nitrogen-doped DLC coatings of 10 and 50 nm thick were prepared by PECVD (for hydrogenated amorphous carbon a-C:H) and FCVA (for tetrahedral amorphous carbon a-C) deposition systems. Nitrogen-doped DLC (both hydrogenated and un-hydrogenated) samples were deposited over AlTiC ($Al_2O_3 + TiC$) ceramic composite substrates at different nitrogen contents. The nitrogen gas content values are ranging from 0% to 14.24%.

2.2.1. PECVD coatings

Hydrogenated amorphous carbon (a-C:H) DLC coatings were prepared by PECVD. More details about this system have been described earlier by several researchers [2,3,27]. A -500 V self bias voltage with 20 sccm/min of C₂H₂ at a pressure of 0.55 Pa was used. Five groups of coated substrates were prepared under the following nitrogen contents.

Sample no.	PECVD 1	PECVD 2	PECVD 3	PECVD 4	PECVD 5
Nitrogen %	14.24%	8.57%	8.39%	5.06%	0%

2.2.2. FCVA coatings

Tetrahedral amorphous carbon (a-C) were prepared using the FCVA Nanofilm Technologies International Pte Ltd. system equipped with an off-plane double bend filter to screen the macro and micro-particles. To limit the internal stress, most films were deposited at the floating potential (Vs 0) with the ion energy ranging from 20 to 30 eV at an arc current of 70 A, and the RF (13.56 MHz). Five groups of coated substrates were prepared under the following nitrogen contents.

Sample no.	FCVA 1	FCVA 2	FCVA 3	FCVA 4	FCVA 5
Nitrogen %	14.24%	8.57%	8.39%	5.06%	5.06%

FCVA 5 coating sample is a modified version of FCVA 4 where an arc current of 80 A instead of 70 A was used with the same nitrogen content. More experimental details about this coating system have been described previously [2,3].

2.3. Methods

2.3.1. Corrosion measurements

The corrosion behavior of the previous specimens was monitored using electrochemical impedance spectroscopy (EIS) and DC polarization techniques over two weeks of immersion in 2 M HCl solution. EIS technique was successfully applied previously to analyze the corrosion behavior of some DLC films on ceramic composite substrates [24–26].

A three-electrode set-up was used with impedance spectra being recorded at the corrosion potential *E*_{Corr}. A saturated calomel electrode (SCE) was used as the reference electrode. It was coupled capacitively to a Pt wire to reduce the phase shift at high frequencies. Polarization curves and impedance spectra were measured with a Gamry ac/dc spectroscopy system. All measurements were normally conducted by immersion the samples at the open circuit potential for 30 min in 2 M HCl solution open to air and at room temperature, followed by electrochemical impedance spectroscopy investigations for up to two weeks, followed by potentiodynamic polarization experiments at the end of the two weeks immersion time. EIS spectra were acquired at the open circuit potential in the frequency range $10^6 - 10^2$ Hz, with an AC excitation amplitude of 20 mV. Potentiodynamic polarization was carried out at a scan rate of 1.66 mV s⁻¹. After rinsing with deionized nano-pure water, the samples were examined by the optical microscopy. The exposed coating area was 4 cm². All curves and data were normalized to $1\,\mathrm{cm}^2$.

2.3.2. Surface morphology

An indentor of a DM-400F LECO, Hardness Tester was used to mark definite areas that contain some surface defects before corroDownload English Version:

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