Surface & Coatings Technology 354 (2018) 134–144

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Adhesion and durability of multi-interlayered diamond-like carbon films deposited on aluminum alloy

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ARTICLE INFO

Keywords:
Diamond-like carbon
Aluminum alloy
Interlayer
Adhesion
Wear resistance

ABSTRACT

Aluminum (Al) alloys are light and have good workability; however their low hardness and poor wear resistance are drawbacks limiting their wide application in the automotive industry. Deposition of diamond-like carbon (DLC) films, which exhibit high hardness and good wear resistance, onto the surface of Al alloy substrates can overcome these drawbacks. Because Al alloys and DLC films have low affinity for each other, adhesion between the two is poor. However, the use of an interlayer can improve adhesion. To investigate the effect of multi-interlayers on the adhesion and durability of DLC films on Al substrates, DLC films with Ti, Si-DLC, or Ti/Si-DLC interlayers were deposited onto A2024 substrates using plasma-enhanced chemical vapor deposition (PECVD). Prior to PECVD, an Ar-bombardment treatment was conducted to clean the surface of the substrate. Subsequently, a Ti interlayer was deposited by sputtering for 15 min, a Si-DLC interlayer was deposited using a tetramethylsilane (TMS) and methane gas mixture for 15 min, and DLC was deposited using methane gas for 90 min. The nanohardness of the Ti/Si-DLC multi-interlayered sample reached 33 GPa more than 8 GPa higher than that of the single-interlayered samples. In addition, the Ti/Si-DLC multi-interlayered sample exhibited higher hardness/Young's modulus (H/E) ratios than the single-interlayered samples. Wear tests showed that the wear volumes for the balls and the multi-interlayered samples were smaller than those for the single-interlayered samples. In addition, the delamination distance of the Ti/Si-DLC multi-interlayer sample was 3300 m, more than 1500 m longer than that of single-interlayered samples. This study demonstrates that improving the wear resistance required high plastic index parameter (H/E) values rather than high H values.

1. Introduction

Aluminum (Al) alloys are light and have good workability. Therefore, replacing steel with Al alloys as an automotive material leads to lighter-weight products. However, because of drawbacks such as low hardness and poor wear resistance, Al alloys have limited applications in automotive applications such as transmission gear parts used below 473 K. The deposition of a diamond-like carbon (DLC) film with high hardness and superior wear resistance on the substrate surface can ameliorate these disadvantages. Several types of DLC films are known, including amorphous carbon (a-C), tetrahedral amorphous carbon (ta-C), hydrogenated amorphous carbon (a-C:H), and hydrogenated tetrahedral amorphous carbon (ta-C:H) [1]. In addition, although DLC films exhibited favorable properties such as high smoothness, a low friction coefficient, and good corrosion resistance, they also have some disadvantages, including high residual stress (compressive stress), poor adhesion, and deformation flowability [2–6]. The high residual stress of DLC films is the main reason for their delamination and decreased durability. In addition, because residual stress increases proportionally with increasing film thickness, the deposition of thick films is difficult [7,8]. The residual stress comprises the intrinsic stress of the DLC films and thermal stress generated by the difference between the coefficient of thermal expansion (CTE) of the DLC film and that of the substrate [9]. Wei et al. demonstrated that the mismatch between the CTE of the DLC films and substrates generates thermal stress, thus increasing residual stress [10,11]. The CTE of DLC films (2.3×10^{-6}/K) is substantially different from that of Al alloys (23.5×10^{-6}/K). This mismatch between CTEs is one of the reasons for the difficulties encountered when depositing DLC films onto Al alloys. In addition, the affinity between Al alloys and DLC films is poor, resulting in low adhesion strength [12]. The low adhesion strength results in poor adhesion, low wear resistance, and low durability. The inconsistency between properties such as hardness (H) and Young's modulus (E) for Al alloys and DLC films also results in poor durability [13].

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https://doi.org/10.1016/j.surfcoat.2018.08.094
Received 26 March 2018; Received in revised form 7 August 2018; Accepted 10 August 2018
Available online 05 September 2018
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Many researchers have achieved improved adhesion using interlayers. For example, introducing a metal interlayer has been demonstrated to improve adhesion and decrease the residual stress of the DLC films [14–20]. Further studies describe the use of *Me*-DLC doping in DLC films as an interlayer, where *Me* is a carbide-forming element such as Si, Ti, Cr, and W [14,21–25]. Multilayers also reduce residual stress. Moreover, multilayers change the transition hardness from the top layer to the substrate or deposits of alternating hard and soft films [26–30]. Al alloys have high affinity for Ti and Ti has high affinity for the C in DLC films. Therefore, using Ti interlayers should improve adhesion. Introducing a Me-DLC interlayer also reduces the residual stress and is expected to improve the mechanical properties of Al alloys.

Various techniques have been used to deposit interlayers and DLC films. Metal interlayers have been deposited using physical vapor deposition (PVD) techniques such as sputtering and unbalanced magnetron sputtering [15,30]. In addition, DLC films have been deposited using PVD and chemical vaper deposition (CVD) techniques [31]. Plasma enhanced CVD (PECVD) can be treated at lower temperature [32]. Therefore, PECVD is suitable for coating Al alloys with low melting points and that suffer overaging softening.

In this study, we deposited DLC films, introduced Ti interlayers using sputtering, deposited Si-DLC interlayers using PECVD, and added Ti and Si-DLC multi-interlayer DLC films on A2024 Al alloy substrates. The effects of the composition and number of adhesion layers on wear resistance and the durability of the DLC film were investigated.

2. Experimental

2.1. Materials

A2024 Al-Cu-Mg alloy disk (ϕ25 mm × 5 mm), A2024 strips (50 × 5 × 0.5 mm^3), and Si wafers (2 × 2 × 0.5 mm^3) were used as substrates. The A2024 samples were polished with #2000 abrasive paper and 1.0 μm diamond paste.

2.2. PVD and CVD coatings

DLC films were deposited onto an A2024 Al alloy substrates (disk and strips) and onto Si wafers using the PECVD technique. A schematic of the PECVD apparatus is shown in Fig. 1. Before deposition, samples were ultrasonically cleaned in acetone for 10 min. Sputtering and PECVD were conducted at a 1×10^{-5} Pa base pressure and 443 K. Samples were cleaned for 10 min using Ar plasma generated at a −380 V applied bias and a 0.70 Pa working pressure. Before deposition of the Ti interlayer, pre-sputtering was carried out for 5 min using Ar plasma generated at a −100 V applied bias with a 0.70 Pa working pressure. Details of the deposition conditions for the Ti and Si-DLC interlayers and DLC films are presented in Table 1. In this study, the samples with no interlayer, with a Ti interlayer, with a Si-DLC interlayer, and with both Ti and Si-DLC interlayers are denoted as Al/DLC, Al/Ti/DLC, Al/Si-DLC/DLC, and Al/Ti/Si-DLC/DLC, respectively.

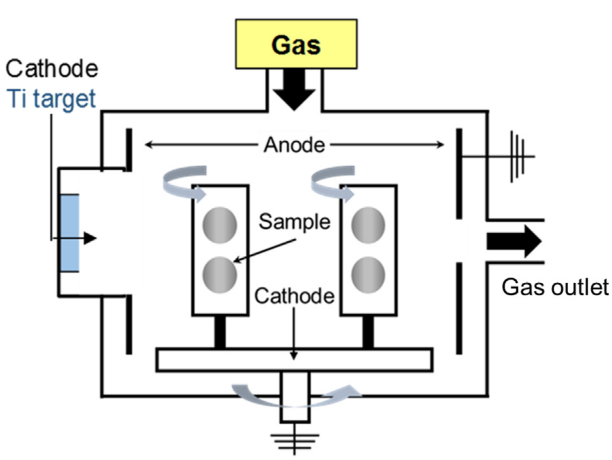

Fig. 1. Schematic of the PECVD and PVD apparatus.

Table 1
Conditions during the PVD and CVD processes.

	Ar bombardment	Ti interlayer	Si-DLC interlayer	DLC
Gas	Ar	Ar	CH_4 + TMS	CH_4
Gas pressure/Pa	0.70	0.70	1.2	1.0
Gas flow/sccm	40	40	100 + 10	100
Applied bias/V	380	200	400	400
Time/min	10	15	15	90
Temperature/K	443	443	443	443

2.3. Cross-sectional morphology

The cross-sectional morphology and thickness of the films were analyzed by scanning electron microscopy (SEM, JEOL, JSM-6060LV). The accelerating voltage was 15 kV.

2.4. Surface roughness

The roughness of the A2024 substrate, interlayers, and DLC films, as well as the traces of the wear tracks after the wear test, were examined using a profilometer (Surf-corder, Kosaka-Lab, SE 300). In this test, the arithmetic average roughness (*R*a) and maximum roughness valley depth (*R*v) were used. Moreover, the surface morphology of the DLC films on the Si wafers was examined using atomic force microscopy (AFM, SHIMADZU, SPM-9600). The average roughness (*R*c) was determined by AFM.

2.5. Residual stress

The residual compressive stresses of the DLC film were determined using Stoney's Eq. (1):

$$\sigma = \frac{E_s \cdot h_s^2}{3h_f \cdot R(1 - \nu_s)} \tag{1}$$

where E_s is the Young's modulus for the A2024 substrate (73.5 GPa), ν_s is the Poisson's ratio for the substrate (0.34), h_s is the thickness of the substrate (0.5 mm), h_f is the thickness of the thin film, and R is the radius of curvature of the reed-shaped sample [33]. R before and after deposition were measured using a profilometer.

2.6. Raman spectroscopy

The chemical bonds in the DLC films were investigated using Raman spectroscopy (Nanophoton, RAMAN Touch). The Raman spectra of the films consist of two peaks at approximately 1350 and 1580 cm^{-1}, which correspond to the disordered band (D-band) and graphite band (G-band), respectively [34].

2.7. XPS and ERDA

Also the chemical bonds in the films were investigated using X-ray photoelectron spectroscopy (XPS, ULVAC, PHI 5000). The X-ray source of the X-ray photoelectron spectrometer was an Al (Kα = 1486 eV) anode operated at an emission voltage of 15 kV and current of 2 mA. The detector pass energy was 23.5 eV. The baseline pressure within the XPS chamber was approximately 2×10^{-6} Pa.

The hydrogen content of the a-C:H films was determined by elastic recoil detection analysis (ERDA) performed on rutherford backscattering spectrometry (RBS) system (Nagaoka University of Technology, Japan, NHV; NT-1700HS accelerator) [35]. The detection of recoil protons was performed at an angle of 30°.

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