



## Effect of Ag content on the microstructure, tribological and corrosion properties of amorphous carbon coatings on 316L SS

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### ABSTRACT

The mechanical durability and stability of a-C:Ag nanocomposite coatings deposited on 316L stainless steel substrates were investigated with respect to Ag content. The coatings were prepared using graphite and Ag targets. The coatings were characterized by Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and transmission electron microscopy (TEM). XPS and Raman spectroscopy confirmed the formation of  $sp^2$  clusters in the a-C coating with 4.46 at.% Ag content. Surface roughness was found to be reduced by increasing the Ag content in a-C coatings as confirmed by AFM analysis. TEM analysis also showed the presence of nanostructured Ag in the a-C matrix. The tribological and corrosion properties of these coatings were also investigated. Hardness and Young's modulus were found to decrease with increase in Ag content. The experimental results revealed that the friction coefficient could be significantly reduced due to inclusion of Ag in the composite coating. In particular, the coating with an Ag content of 4.46 at.% showed the best tribological behavior. Inclusion of Ag also improved the corrosion resistance of the composite coating. It was concluded that Ag content in a-C:Ag nanocomposite coating has a significant effect on the tribological and corrosion properties of the coating. Experimental results revealed that optimum Ag content (4.46 at.%) in a-C:Ag nanocomposite coatings may be effectively utilized to protect 316L stainless steel substrates.

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### 1. Introduction

Amorphous carbon (a-C) coatings are known to have high hardness, good wear resistance, and anti-corrosion behaviors. These coatings are used in various applications, including biomedical and industrial applications. They are often deposited on steel substrates such as 316L SS. Many groups have studied a-C based coatings deposited on 316L SS and found delamination failure to be a disadvantage due to the high hardness and intrinsic stress of the coatings [1–5]. To overcome this problem, metals such as Mo, Cr, W and Ag have been included into the a-C coatings. The inclusion of metals reduces the intrinsic stress and increases the ductility [2,3] of the amorphous carbon coatings, which leads to increased adherence of the coating to the substrate. Metal including in a-C based coatings can also improve their tribological [3–7], electrical [8] and antibacterial properties [9,10].

Various methods are used to fabricate metal:a-C composite coatings, including RF dense plasma method [11], mid-frequency dual-magnetron sputtering [5], plasma enhanced chemical vapor deposition (PECVD) [12], termionic vacuum arc technique [13], ion beam assisted ECR-plasma CVD [14], ion beam deposition [15], and RF magnetron sputtering [3]. Ag is a common metal among many others that can be used to improve the properties of amorphous carbon. Many studies have reported on the effects of Ag on the properties of a-C coatings. Lungu et al. [4] obtained a lower coefficient of friction (COF) by increasing the graphite/Ag target area from 1/9 to 5/5 during ECR-DC deposition. The  $sp^2$  ratio also increased with increased Ag content in diamond-like carbon (DLC) coatings. Wang et al. [5] reported improved hardness, adherence, and mechanical strength in Ag-DLC coatings. Inclusion of Ag was also found to decrease intrinsic stress, leading to good adhesion between the coating and the substrate.

Many studies have investigated the tribological behavior of carbon-based and metal-doped carbon coatings, particularly for mechanical applications [16,17]. Corrosion resistance also plays an important role in the performance of protective coatings [18]. To improve these properties, there is a strong motivation to study the influence of Ag in a-C:Ag nanocomposite coatings. However, only a few reports are available on the tribological and corrosion behaviors of such coatings. Wei et al. [6]

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reported no improvement in the corrosion resistance of Cr incorporated DLC coatings deposited on a magnesium alloy substrate, whereas Morrison et al. [9] observed low corrosion of Ag incorporated DLC films in phosphate-buffered saline [PBS] electrolyte. Thus, the advantage of using Ag in carbon-based coatings has been reported previously. The motivation of this study was to perform a comprehensive investigation regarding the effect of Ag on both the tribological and corrosive properties of a-C:Ag nanocomposite coatings deposited on a 316L SS substrate. a-C:Ag nanocomposite coatings with varying amounts of Ag content were deposited on 316L SS substrates by RF sputtering and their structural, chemical, topographical, tribological, and corrosive properties were investigated.

## 2. Experimental procedure

### 2.1. Substrate cleaning and coating procedure

316L SS substrates having a size of 10 mm × 10 mm were used in all experiments except corrosion tests in which 10 mm × 20 mm substrates were used. Thickness of each substrate was about 0.5 mm and all the substrates were mechanically polished using a standard polishing machine with silicon carbide papers with 500, 1000, and 2000 grits. This was followed by a final polishing procedure using a 0.05 μm alumina powder slurry. The substrates were cleaned with isopropanol, acetone, and distilled water in an ultrasonic agitation bath for 15 min. The cleaned substrates were dried in blowing air.

Pure a-C and a-C:Ag nanocomposite coatings were prepared by RF ( $\nu = 13.56$  MHz) sputter using graphite and Ag targets under a constant flow of argon at room temperature. The deposition chamber was pumped down to a base pressure using rotary and turbomolecular pumps. MKS mass flow controllers were used to regulate the flow rate of high purity argon (99.999%). All depositions were carried out a constant working pressure and a constant thickness of about 200 nm was maintained by adjusting the deposition time relative to the rate of deposition. No substrate bias was applied during the deposition process. Pre-sputtering of each target was performed for 5 min before deposition. Various coating parameters such as base pressure, argon flow rate, working pressure, film thickness, etc., are shown in Table 1.

### 2.2. Characterization

The surface topography and surface roughness of the pure a-C and a-C:Ag nanocomposite coatings were investigated using an AFM (Seiko SPA400). Transmission electron microscope (TEM) analysis was performed using a JEOL TEM 2100 operated at 200 kV. Chemical bonds were analyzed by X-ray photoelectron spectroscopy (XPS) using an angular resolved electron analyzer with a monochromated Al K $\alpha$  source (Theta Probe, Thermo Fisher Scientific). Raman spectroscopy measurements were carried out under backscattering geometry with a liquid-nitrogen-cooled CCD detector (Labram HR). The spectra were collected under ambient conditions using the 514.5 nm line of an argon-ion laser.

**Table 1**

Deposition parameters of a-C and a-C:Ag nanocomposite coatings.

Parameters	Condition
Technique adopted	RF sputtering
Target material	Graphite and silver
Substrate	316L SS
Substrate temperature	Room temperature
Graphite target power	400 W
Silver target power	16, 20, 24 and 28 W
Base pressure	0.003 Pa
Argon flow rate	50 sccm
Working pressure	0.75 Pa
Film thickness	200 ± 5 nm

The surface morphology of coatings on pure a-C and a-C:Ag nanocomposite coatings was investigated using a scanning electron microscope (SEM; JEOL 6210) equipped with an energy-dispersive X-ray spectroscopy system (EDS; OXFORD INCA Energy). The diameter of the analyzed area of the XPS spectrometer was varied from 30 μm (inside of the wear track) to 400 μm (large-area averaging). XPS spectra were deconvoluted using the Gauss–Lorentz function with a shape factor of 0.6. Measurements were performed inside of the wear track as well as outside at a location close to the boundary of the wear track.

### 2.3. Tribological tests

The friction and wear properties of the coating were investigated using a commercial reciprocating tribo-tester. An Al<sub>2</sub>O<sub>3</sub> sphere with a diameter of 1 mm was used as the counter surface after a standard cleaning procedure. All tests were conducted under the following conditions: room temperature, relative humidity of about 40%, applied load of 10 mN, and sliding speed of 2 mm/s with a 4 mm stroke. The COF and number of sliding cycles were recorded automatically through a data acquisition system. The tests were repeated five times for each specimen to obtain an average friction value. The mechanical properties of the coatings were assessed using a commercial ultra-nano hardness tester (CSM UNHT). A standard diamond Berkovich tip was used in the indentation tests. Max indentation load applied was 0.3 mN. At this load, the maximum indentation depth was ~45 nm, which is less than 25% of the film thickness.

#### 2.3.1. Wear rate calculation

The standard wear equation proposed by Archard [19] was used to obtain the wear rate using the following equation:

$$\text{WearRate} = \frac{V}{WL} \quad (1)$$

where  $V$  is the wear volume,  $W$  is the applied load, and  $L$  is the sliding distance. The wear volume was estimated from the cross-sectional area of the wear track below the horizontal line of the specimen surface.

### 2.4. Electrochemical corrosion analysis

Electrochemical corrosion studies of uncoated and coated 316L SS substrates were carried out in 3.5% NaCl solution by potentiodynamic polarization techniques and electrochemical impedance spectroscopy (EIS) measurements. The anodic and cathodic polarization curves were found by changing the electrode potential from –250 mV to +800 mV relative to the open circuit potential (OCP) with a constant sweep rate of 2 mVs<sup>-1</sup>. The corrosion rates were evaluated using the Tafel plot analysis and the results were presented in mils per year (mpy). The electrochemical impedance measurements were recorded over a frequency range of 10<sup>4</sup> to 1 MHz, with an acquisition of 10 points per decade and a signal amplitude of 10 mV at OCP. The impedance spectra analysis was carried out by fitting the experimental results to equivalent circuits using the non-linear least-square fitting procedure. The quality of fitting to equivalent circuits was judged by the  $\chi^2$  value (i.e., the sum of the square of the difference between the theoretical and experimental points) and then by limiting the relative error in the value of each element in the equivalent circuit to 5%. All measurements were repeated at least three times and good reproducibility of the results was observed.

## 3. Experimental results

### 3.1. HRTEM analysis

By increasing the RF power on the Ag target while maintaining constant power on the graphite target (400 W), the Ag content in the

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