

Low friction mechanism of chlorine-doped amorphous carbon films sliding against an aluminium alloy



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ABSTRACT

The effect of chlorine-doping on the tribological properties of amorphous carbon films was investigated. Chlorine-doped amorphous carbon films were deposited using a plasma-based ion implantation and deposition (PBI&D) method with vaporized tetrachloroethylene as a precursor. During sliding tests with aluminium alloy counter parts, chlorine-doped amorphous carbon films showed lower friction coefficients compared to hydrogenated amorphous carbon films. Both aluminium and a chlorine-based hydrate were observed on the wear track of the carbon films. This hydrate was a tribofilm formed via a tribochemical reaction during sliding between the chlorine-doped amorphous carbon films and aluminium alloy. The viscosity of this tribofilm was similar to polyalpha olefin and we propose that it acted as a lubricant and lowered the friction coefficient.

1. Introduction

Amorphous carbon films are generally composed of sp^2 - and sp^3 -hybridized orbitals of carbon and hydrogen. In recent years, amorphous carbon films have attracted attention in research and industry due to their superior properties, such as optical properties, corrosion resistance, and biocompatibility [1–3]. In particular, amorphous carbon films have extraordinary wear and friction properties, and they have contributed to drastic improvement of the tribological properties of gears, cutting tools, and machine parts [4–6].

Since the seminal report regarding amorphous carbon films was published by Aisenberg and Chabot in 1971 [7], this material has attracted much attention from many research groups. As research on amorphous carbon films has progressed, it has been confirmed that various film properties depend on the structure. Grill proposed that the characteristics and structure of amorphous carbon films are determined by several factors, including the concentrations of hydrogen, sp^3 bonding, and sp^2 bonding in the films. In addition, amorphous carbon films can be classified into four types: amorphous carbon (a-C), hydrogenated amorphous carbon (a-C:H), tetrahedral amorphous carbon (ta-C), and hydrogenated tetrahedral amorphous carbon (ta-C:H) [8]. The a-C:H and ta-C:H films contain up to approximately 50% hydrogen, a-C and ta-C films contain less than 1% hydrogen, and ta-C and ta-C:H consist of a high fraction of sp^3 bonding. Hence, the term “amorphous carbon films”

covers a wide range of carbon-based coatings and the investigation of the relationship between the structure and properties of the films is critical in their application.

Erdemir reported that the hydrogen content of a-C:H films plays an important role in the friction behaviour [9]. a-C:H films contain a large amount of hydrogen, where the terminated hydrogen eliminates most of the dangling σ bonds and π - π interactions and contributes to superlow friction under dry conditions. The low friction of a-C:H films was attributed to the high chemical inertness of films as a result of the surface passivation of the dangling bonds. On the contrary, ta-C films are suitable for oil-lubricated application because of their unterminated dangling bonds inside the films which activate the wear surface. A tribochemical layer caused by friction has been observed to form on the surface of such films [10]. The friction behaviour of these films is strongly dependent on the sliding environment. Hence, it is clear that the optimisation of the film structure for a particular application is critical.

Recent studies have demonstrated doping of amorphous carbon films with different elements (such as Si, F, B, N, Al, Cr, Ti, and W) as a new technique for optimising the structure [11–17]. These efforts contributed to the understanding of mechanisms for improving many properties, for example thermal resistance, optical gap energy, surface energy, relaxation of internal stress, and activation of the tribochemical reaction. Si doping of amorphous carbon films has been shown to improve the high temperature behaviour in comparison to hydrogenated amorphous

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carbon films and also achieve low friction under high humidity due to the formation of SiO_2 particles through a tribochemical reaction [11,12]. In the case of F doping, the surface energy was shown to decrease and the optical band gap improve with increasing F content; the increase in the optical band gap energy induced by F doping was attributed to a modified film structure with a higher fraction of sp^3 bonding compared to non-doped films [13,14]. Doping with metals, such as Al and Ti, has also been shown to contribute to a reduction in the surface energy, as observed for Si doping [15]. The dopant metal atoms inside the films form metal-O covalent bonds, localizing the electrons of the metal; thus, a decrease in dipoles contributes to reducing the surface energy. In addition, metal doping contributes to reducing internal stress and activating the chemical reaction between amorphous carbon films and lubricant additives [16,17].

The aim of this study is to investigate the effect of chlorine doping on the tribological properties of a-C:H films during sliding against an aluminium alloy under non-lubricated conditions. Our hypothesis is that the chlorine inside of the films will contribute to the formation of a chlorinated tribofilm at the sliding interface, in the same way as a reaction with chlorine from additives [18–20], where the chlorinated tribofilm can reduce friction coefficient. In our previous study, we observed a lowering of the friction due to chlorine-doped a-C:H films during sliding with aluminium alloy counter parts [21]. However, the exact mechanism behind this observed low friction was not clarified and hence, the major objective of this study is to reveal the low friction mechanism of such films.

Non-doped a-C:H films and chlorine-doped a-C:H films were deposited using a plasma-based ion implantation and deposition (PBII&D) method using vaporized toluene ($\text{C}_6\text{H}_5\text{CH}_3$) and tetrachloroethylene (C_2Cl_4). Various characterisation methods, including micro-laser Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), Elastic recoil detection analysis (ERDA), and nano-indentation hardness testing were employed to investigate the structure and mechanical properties. The chlorine-doped tribofilm formed through a tribochemical reaction was investigated using surface analysis techniques including Fourier transform infrared spectroscopy (FT-IR) and time-of-flight secondary ion mass spectroscopy (TOF-SIMS).

2. Experimental setup

2.1. Deposition of amorphous carbon films using PBII&D

A PBII&D system (PBII-R1000/Kurita Seisakusho) was employed to deposit the non-doped a-C:H films and chlorine-doped a-C:H films on an aluminium alloy (ISO-AlMg1SiCu) disk with a diameter of 30 mm. Nano-indentation hardness of substrate was 1.7 GPa. Fig. 1 shows a schematic diagram of the PBII&D system used in this study. In this method, the substrate itself is used as an RF antenna and a high-frequency power supply generates a plasma from the source gas around the substrate; then a negative charge is applied to the substrate. Using this process, plasma ions can be implanted into the substrate surface to form the desired film. This method has various advantages; for example, it is possible to form a uniform film on a complex surface at deposition temperatures of less than 473 K. Note that the films were deposited via a hybrid process of simultaneous chemical reaction and ion implantation, which was accelerated by the applied voltage. Prior to film deposition, all substrates were cleaned by Ar bombardment to remove contaminants and activate the surface. After Ar bombardment, Si interlayer was deposited using hexamethyldisiloxane (HMDSO) gas on the substrate surface. Table 1 shows the different chlorine and hydrogen contents of five types of samples prepared in this study. A non-doped a-C:H films were prepared using only toluene gas, while the chlorine-doped a-C:H films were deposited using two different gases, a mixture of toluene and tetrachloroethylene ($\text{C}_6\text{H}_5\text{CH}_3 + \text{C}_2\text{Cl}_4$; #1, #2, #3, and #4) or pure C_2Cl_4 (#5). When using the mixed gas, the flow ratio of C_2Cl_4 to $\text{C}_6\text{H}_5\text{CH}_3$ was increased in order of #1 to #4; the other

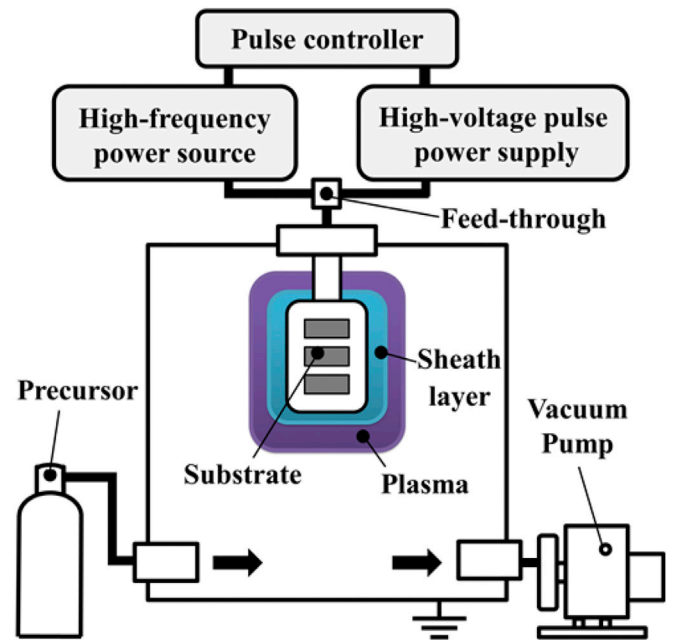


Fig. 1. Schematic diagram of the plasma-based ion implantation and deposition system used in this study.

Table 1

Deposition parameters of non-doped and chlorine-doped a-C:H films.

		Cl/(C + Cl) ratio	Hydrogen content [atm %]
Non-doped a-C:H films		0.0	21.9
Chlorine-doped a-C:H films	#1	0.013	20.3
	#2	0.037	10.1
	#3	0.083	4.8
	#4	0.108	3.1
	#5	0.118	<3.0

deposition parameters, such as the applied voltage of 5 kV and RF power of 500 W were the same for all samples. In Table 1, the hydrogen content and the presence ratio of chlorine relative to the total number of carbon and chlorine $\text{Cl}/(\text{C} + \text{Cl})$ of each film were measured using ERDA and XPS, respectively. ERDA was used to determine the hydrogen content inside of each film at a depth of 50 nm from the film surface. XPS was used to measure the $\text{Cl}/(\text{C} + \text{Cl})$, where the $\text{Cl}/(\text{C} + \text{Cl})$ was estimated from the relative areas of the chlorine peaks (Cl 2p) and the carbon peaks (C 1s). The surface roughness parameter R_a of these amorphous carbon film were about 0.022–0.026 μm and film thickness was above 1.0 μm .

2.2. Structure and mechanical properties

Raman spectroscopy and XPS analyses were used to investigate the structure of the films. Specifically, the effect of chlorine-doping on the internal structure of the films was analysed by Raman spectroscopy and the effect on the sp^3/sp^2 ratio of the films was calculated from XPS data. The Raman spectrometer (In Via Reflex/Renishaw) was equipped with a YAG laser with a wavelength of 532 nm. The laser power, spot size, and spectral range were 0.25 W, 5.0 μm , and 800–2000 cm^{-1} , respectively. An XPS apparatus (Quantera/Ulvac-Phi) was used to analyse chlorine in the films and measurements were undertaken after a sputtering pre-treatment using Ar^+ . The detection range of the carbon and chlorine peaks were 275–300 eV and 190–220 eV, respectively. The X-ray source, X-ray power, and target emission were $\text{AlK}\alpha$, 10kV25W, and 3.0 mA. The $\text{sp}^3/(\text{sp}^3 + \text{sp}^2)$ bonding ratio of carbon was investigated by analysing the

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