



Friction and wear behavior of plasma assisted chemical vapor deposited nanocomposites made of metal nanoparticles embedded in a hydrogenated amorphous carbon matrix

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ARTICLE INFO

Article history:

Received 2 September 2011

Accepted in revised form 28 December 2011

Available online 3 January 2012

Keywords:

Nanocomposite coatings

Hydrogenated amorphous carbon

Metallic nanoparticles

Reciprocating sliding

Tribology

Plasma CVD

ABSTRACT

Nanocomposite coatings consisting of preformed silver or chromium nanoparticles embedded into a hydrogenated amorphous carbon matrix (a-C:H) were synthesized by Electron Cyclotron Resonance plasma assisted Chemical Vapor Deposition (ECR-CVD). In a first step, the nanoparticles were distributed on silicon substrates by dipping in an ethanol suspension. In a second step, the ECR-CVD deposition of the a-C:H layer was done. The effect of the incorporation and the concentration on the friction and wear behavior was derived from unlubricated reciprocating sliding tests performed in ambient air. A decrease in the coefficient of friction, more intense with Cr incorporation, is induced by the preferential metal interaction with environment. In addition, for both metals, the coefficient of friction becomes lower as the metal concentration increases. A gradual increase in the coefficient of friction is detected for increasing the number of sliding cycles, which is attributed to the combined effect of surface smoothing and oxidation in the sliding contact. In conclusion, the valuable protective properties of the fullerene-like a-C:H coatings are enhanced by metal addition. As a consequence, a considerable reduction of the surface roughness and the volume loss in the wear tracks is especially noticeable for 10,000 cycles tests.

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1. Introduction

Hydrogenated amorphous carbon (a-C:H) spans a wide range of carbon materials with properties that are strongly dependent on the hydrogen content and bonding structure of the amorphous carbon network. In particular, the development of hard carbon coatings such as diamond-like carbon (DLC) has attracted a lot of research interest over the last three decades. DLC combines unique properties (high elastic modulus, high mechanical hardness and chemical inertness), which makes it a valuable material for numerous applications [1–6]. Casiraghi et al. [7] based the classification of a-C:H materials on the relative percentage of tetrahedral sp³, trigonal sp² and bonded hydrogen in the amorphous structure. Four types of a-C:H materials have been distinguished [7], namely polymerlike (PLCH), graphitelike (GLCH), diamondlike (DLCH or DLC) and tetrahedral (TACH) hydrogenated amorphous carbon.

Alloying of the a-C:H material allows a controlled change of basic physical properties such as hardness, thermal expansion coefficient, electrical properties, chemical reactivity, elasticity, oxidation behavior and internal stress which alter the field performance [3]. The incorporation of metals in the insulator carbon matrix introduces changes in several characteristics of a-C:H films, like electrical and thermal conductivity, magnetic properties, and film adhesion [8–11]. Nano-alloyed or doped-DLC films are capable of providing superior mechanical, tribological, thermal and electrical properties [12–14]. Adding Si, Ti or W into DLC films results in a lower friction and higher wear resistance under lubricated sliding conditions, and in a high scuffing resistance under severe contact pressures [15,16]. Also, the improvement of the adhesion of the films by chromium addition is well documented and is commonly associated with the matrix relaxation. Chromium is an easy carbide former and is able to stabilize the structure and to relieve the internal stress [10]. The introduction of Cr in the carbon film also improves the corrosion and wear resistance of the final coating. Likewise, silver provides a lubricious behavior in fretting tests [17] and also exerts a bactericide role [18].

The wear of carbon-based coatings is generally very complex and both intrinsic (materials properties) and extrinsic parameters (such as test environment and applied load) are key factors determining the overall coating performance. Traditionally two types of wear mechanisms are considered for amorphous carbon films: physical

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mechanisms (adhesive, abrasive and surface fatigue) and chemical mechanisms (corrosive wear) [19]. Among the chemical wear mechanisms usually proposed, graphitization is often pointed out as a possible mechanism of low friction in a-C:H layers [20,21], and consists in the promotion of sp^3 to sp^2 hybridizations, favored by the increase in contact temperature during sliding. The graphitization hypothesis is contradictory to some extent with the fact that graphite needs to absorb water molecules for becoming lubricious [22], while on the contrary, an increasing relative humidity generally gives rise to an increasing coefficient of friction of a-C:H coatings [23,24]. Recently, the passivation of dangling bonds in the surface by H atoms has been proposed for explaining the ultralow friction of carbon materials [12,25]. The tribochemistry of sliding interfaces involving a-C:H coatings is complex, since the carbon films interact or react with the chemical species present in the surrounding as well as with the counterpart materials [12,26]. The formation of a third body transfer layer which results from the interactions between the base materials, the debris, and the surrounding atmosphere is often proposed as guiding the wear mechanism of both undoped and doped a-C:H materials [12,27]. The incorporation of certain alloying elements (including various metals) into DLC films induces a significant effect on the tribochemical processes on these films [12,28]. The reactivity of such elements with oxygen or water vapor during sliding may induce higher inertness of the carbonaceous network [12].

The addition of metals in a-C:H coatings has usually been carried out by physical deposition methods, such as plasma immersion ion implantation [29], reactive magnetron sputtering [30,31], or by combination of hybrid plasma enhanced chemical-physical methods [10,32]. In this study, we have implemented a new metal incorporation strategy taking advantage of the Electron Cyclotron Resonance Chemical Vapor Deposition (ECR-CVD) process to obtain the desired nanocomposites. Cr and Ag nanoparticles are deposited in a first step as aggregates on silicon substrates by dipping them in a liquid suspension. In previous studies in publication process, we demonstrated that during thin film growth by ECR-CVD individual nanoparticles are efficiently dispersed in the a-C:H matrix material, yielding Me-C:H nanocomposite coatings (Me = Ag, Cr). The undoped coatings were previously characterized by pin-on-disk tests [23], where the contact area is exposed to the ambient air for most of the cycle/test duration. Preliminary sliding tests using a pin-on-disk geometry showed a significant improvement in the durability for Me-C:H with different contents of Cr nanoparticles [33]. However, no clear trend could be detected for the Ag nanocomposites [34]. Since the counterpart pin is held stationary and the Me-C:H sample rotated during pin-on-disks tests [35], the main test characteristic is the large period of exposure of the wear track area to the surrounding atmosphere. The aim of this study is to analyze the effect of metal incorporation with a technique selected to reduce the exposure time of the wear track and possible atmospheric effects (such as water adsorption and material oxidation) during the test experiment. The wear and friction behavior of Cr-C:H and Ag-C:H nanocomposites are investigated under reciprocating sliding at a relatively small displacement amplitude of 200 μm and a contact frequency of 5 Hz. In particular, the effect of the concentration on the friction and wear behavior of Me-C:H nanocomposites has been analyzed in detail. The obtained results give new insights into the tribological performance of Cr-C:H and Ag-C:H nanocomposite coatings and reveal the potential of these low friction resistant coatings.

2. Experimental

Me-C:H (Me = Ag, Cr) nanocomposite coatings were synthesized on double side polished (100) p-type silicon in a two-step process. In a first step, partially oxidized commercial Cr ($\varnothing = 50$ nm, measured by single-point BET – Brunauer Emmett Teller surface area

analysis) or Ag ($\varnothing = 35$ nm, measured by TEM – transmission electron microscopy) nanoparticles were deposited as aggregates on silicon (100) substrates by dipping in a liquid suspension of these nanoparticles. Subsequently, the deposition of a-C:H coating was carried out by ECR-CVD to produce Me-C:H nanocomposite coatings.

Me-C:H coatings with three different metal contents were prepared for both types of metal using ethanol suspensions containing either 5000, 300 or 150 ppm nanoparticles. The particle concentration of the suspensions participates, and thus controls the final metal content in the composites [34]. The nomenclature of the samples is based on the concentration of nanoparticles suspended, namely as Me-C:H(5000), Me-C:H(300) and Me-C:H(150) respectively (Table 1). A probe sonication was applied for 3 min to break up the agglomerates that nanoparticles naturally tend to form, and to obtain a well dispersed suspension. Dipping of the substrate was done for less than 1 s and the seeded substrates were subsequently dried in ambient air. The surface coverage and distribution by metallic nanoparticles was estimated by Image J software [36] applied on scanning electron microscopy (SEM) images.

The carbon matrix of the Me-C:H nanocomposite films was synthesized by ECR-CVD (ASTEX, Model: AX 4500) with a 2.45 GHz microwave power generator operating at 200 W. The bonding structure and composition of the carbon matrix was controlled by the direct current bias voltage (-300 V) applied to the substrate [23,37–39]. A gas flow ratio methane/argon of 15:35 sccm was kept constant at a pressure of $1.2 \cdot 10^{-2}$ mbar during 1 h growth process, giving rise to 1.0 to 1.3 μm thick layers as measured by profilometry (DEKTA 150). The temperature during the growth process was below 120 $^{\circ}\text{C}$, caused by plasma heating of the substrate. The a-C:H matrix obtained by ECR-CVD in this work can be referred to as fullerene-like hydrogenated carbon (FLCH), since it contains self-formed C_{60} inclusions and fullerene-like curved carbon structures that significantly improve the mechanical and tribological properties of the carbonaceous network [37,40]. The a-C:H matrix displays a medium H content (~ 30 at.%) and good mechanical properties [23,37]. The growth conditions used are comparable to those commonly applied for the production of hard hydrogenated carbon, generally referred to as DLC.

Reciprocating sliding tests performed at a relatively small displacement amplitude [27,41] were carried out on the Me-C:H coatings. A corundum ($\alpha\text{-Al}_2\text{O}_3$, producer: Ceratec (NL), $\varnothing = 10$ mm) ball counterbody, vibrating at a frequency of 5 Hz and a peak-to-peak displacement amplitude of 200 μm were selected. A new corundum ball was used for each test performed at 21 $^{\circ}\text{C}$ at a relative humidity of 50%, using an experimental setup described in detail elsewhere [27]. Two test series were conducted in which: 1) the normal load was varied from 2 N, to 5 N, and 10 N for 10,000 cycles; 2) the number of cycles was varied from 10,000 to 50,000 and 100,000 cycles keeping the normal load constant at 2 N. During all the sliding tests, the linear contact displacement, the normal contact force, and the corresponding friction force were measured as a function of time during the determined sliding cycles [27].

An Atomika A-DIDA 3000-30 Secondary Ion Mass Spectroscopy (SIMS) system evaluated the metal distribution in the Me-C:H coatings, using a 12 keV accelerated O^{2+} molecular ion beam. SEM (Nova Nano

Table 1
Surface coverage of the silicon substrates by the Cr and Ag nanoparticles after the dipping in ethanol suspensions with various concentrations of metal nanoparticles.

Metal	Concentration of solution (ppm)	Nomenclature	Surface coverage (%)
Chromium	150	Cr-C:H(150)	0.5
	300	Cr-C:H(300)	1.5
	5000	Cr-C:H(5000)	6.0
Silver	150	Ag-C:H(150)	0.2
	300	Ag-C:H(300)	0.4
	5000	Ag-C:H(5000)	2.7

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