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AlTiN layer effect on mechanical properties of Ti-doped diamond-like carbon composite coatings

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ABSTRACT

Ti/Ti-doped diamond-like carbon (DLC) and Ti/AlTiN/Ti-DLC composite coatings were deposited by magnetron sputtering on W18Cr4V high speed steel substrates. The effect of the AlTiN support layer on the properties of these composite coatings was investigated through microstructure and mechanical properties characterization, including hardness, elastic modulus, coefficient of friction and wear properties measured by scanning electron microscopy, Raman spectroscopy, scratch and ball-on-disk friction tests. Ti and AlTiN interlayers have a columnar structure with 50–80 nm grains. The hardness and elastic modulus of Ti/Ti-DLC and Ti/AlTiN/Ti-DLC coatings is 25.9 ± 0.4, 222.2 ± 6.3 GPa and 19.3 ± 1, 205.6 ± 6.7 GPa, respectively. Adhesion of Ti-DLC, Ti/AlTiN/Ti-DLC and AlTiN/Ti-DLC coatings expressed as the critical lateral force is 26.5 N, 38.2 N, and 47.8 N, respectively. Substrate coefficient of friction without coatings is 0.44, and it is 0.1 for Ti/Ti-DLC and Ti/AlTiN/Ti-DLC coatings. Wear resistance of Ti/AlTiN/Ti-DLC composite coatings is much higher than Ti/Ti-DLC coatings based on the wear track width of 169.8 and 73.2 μm, respectively, for the same experimental conditions.

1. Introduction

With the fast development of coating technology, single layer coatings no longer satisfy industrial requirements, thus double or combination coatings and multilayers have been developed as a new generation of protective coatings [1–3]. Diamond-like carbon (DLC) is one of the best coatings with high hardness, optical transparency in the visible and infrared regions, high electrical and thermal conductivities, high wear resistance and excellent biocompatibility [3–5]. All of these properties allow for important applications and encourage research works to develop techniques and methods to deposit this kind of films. DLC coatings usually consist of amorphous material with a combination of graphite-like sp^2 and diamond-like sp^3 bonds. However, the problem of low adhesion resulting from a high level of internal stress in DLC films and thermal expansion mismatch when grown on substrates such as high speed and stainless steels, has restricted their use. Adhesion improvement routes have been studied along with the problem of coating thermal stability. Adding a metal interlayer between DLC coating and the substrate, and doping DLC with metal have been utilized to solve this problem [6–8]. Metal-containing diamond-like carbon (Me-DLC) is a DLC film in which nano-scale metal clusters are dispersed homogeneously helping to solve adhesion problems. Metal clusters are remarkably effective for decreasing the stress in DLC films. Tribological properties of Me-DLC films in dry sliding wear using a ball-on-disk tribometer have been reported [9,10]. The average friction coefficient strongly depends on the test duration [11–13]. The wear rate correlates with both hardness and with the intrinsic nanometer-scale roughness of Me-DLC [11]. Friction and sliding characteristics of Me-DLC have been studied [14–16], with a possibility of non-lubricated sliding. However, DLC thin film hardness generally drops with an increased amount of metal inclusions, and film damage occurs rather than delamination.

Another way to solve adhesion problems is to add an interlayer between the substrate and the Me-DLC coating. AlTiN coating has been proven to increase the cutting performance in heavy duty machining of hardened or austenitic stainless steels. These advantages are observed in different cutting operations like milling, turning, or drilling. Strong improvements of operational properties are directly connected with smooth surface, high hardness, and very good adhesion. For applications where low friction is favorable, AlTiN can be combined with a Ti–C:H top layer [17].

In this work we studied and compared properties of Ti-DLC and Ti/AlTiN/Ti-DLC coatings deposited on the high speed steel substrates (HSS). Using specific analytical methods, microstructure and mechanical properties, including hardness, elastic modulus, and coefficient of friction, and wear properties were investigated by scanning electron microscopy, Raman spectroscopy, and scratch and ball-on-disk friction tests.

2. Experimental details

Ti/AlTiN/Ti-DLC and Ti/Ti-DLC composite coatings were deposited by magnetron sputtering (Ti/AlTiN) and plasma-enhanced chemical

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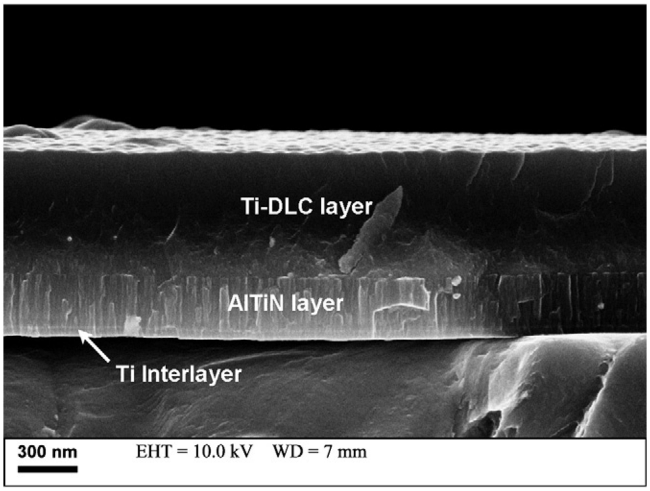

Fig. 1. Cross-section SEM micrograph of Ti/AlTiN/Ti-DLC composite coatings.

vapor deposition (Ti-DLC) on the high speed steel W18Cr4V, which is widely used in die and cutting tools (C: 0.7–0.8%, W: 17.8–19%, Cr: 3.8–4.4%, V: 1.0–1.4%, Si: ≤0.4%, Mn: ≤0.4%, Mo:≤0.3%). Ti and metallic compound targets (Al/Ti at.% ratio = 33:67) with a purity of 99.9% were used. One of the two sources was used to deposit the Ti interlayer between the substrate and the main layer to improve adhesion. The second AlTi target was employed to deposit the AlTiN layers. An ion source was utilized to enhance ion intensity. The rotation speed of the substrate holder was 30 rpm. The target current was 1 A, the base pressure was 2×10^{-4} Pa, and the flow rate of Ar with 99.99% purity was 45 standard cubic centimeters per second (sccm), while the reactive gas was N_2 with 99.99% purity. After Ti/AlTiN deposition, C_2H_2 was introduced into the chamber for Ti-DLC deposition with a self bias of −400 V.

Prior to sputtering, substrates were cleaned in acetone and ethanol for 10 min, respectively, and subjected to 10 min in-situ Ar plasma cleaning at RF power of 100 W in order to remove any contaminants on the substrate surface and to activate the surface. Ti interlayer was deposited on the substrates for 5 min with a DC current of 1 A, and then N_2 gas was introduced into the sputtering chamber.

Microstructure of composite coatings was characterized by cross-section scanning electron microscopy (Zeiss Supra™ 55) with a 10.0 kV operating voltage and Raman spectroscopy (Jobin-Yvon HR-800). Hardness and Young's modulus of the coatings were characterized using Hysitron Triboindenter with a Berkovich diamond indenter tip. Hardness and modulus values presented are maximum average values from 9 indentations on each sample in the 70–80 nm depth range. Hardness values were obtained by analyzing the unloading portion of the load-displacement curves using the Oliver–Pharr method varying with indentation depth.

Adhesion and tribological properties of composite coatings were evaluated by means of scratch and ball-on-disk tests using a Micro-Tribometer model UMT-2 at room temperature in air, made by the Center for Tribology Inc. Normal load was continuously increased at a rate of 1 N/s, while the conical diamond tip (120° angle, 200 μm tip radius) was moving at a constant velocity of 0.05 mm/s for adhesion test. GCr15 steel ball with high carbon and chrome contents (C: 0.95–1.05%, Mn: 0.25–0.45%, Si: 0.15–0.35%, S: ≤0.025%, P: ≤0.025%, Cr: 1.40–1.65%, Mo: ≤0.10%, Ni: ≤0.30%, Cu: ≤0.25%, Ni + Cu: ≤0.50%) was used to characterize coating wear properties in air. A normal load of 10 N was applied to the coating surface for 1 h. The circular wear track developed on the coating had a radius of 6 mm, and the ball rotational speed was 600 rpm.

Table 1
Ti-DLC layer elemental composition.

Element	Weight %	Weight % error	Atom %	Atom % error
C	81.91	±2.71	94.45	±3.12
Al	1.79	±0.18	0.92	±0.09
Ti	14.37	±0.55	4.15	±0.16
Fe	1.93	±0.31	0.48	±0.08
Total	100.00		100.00	

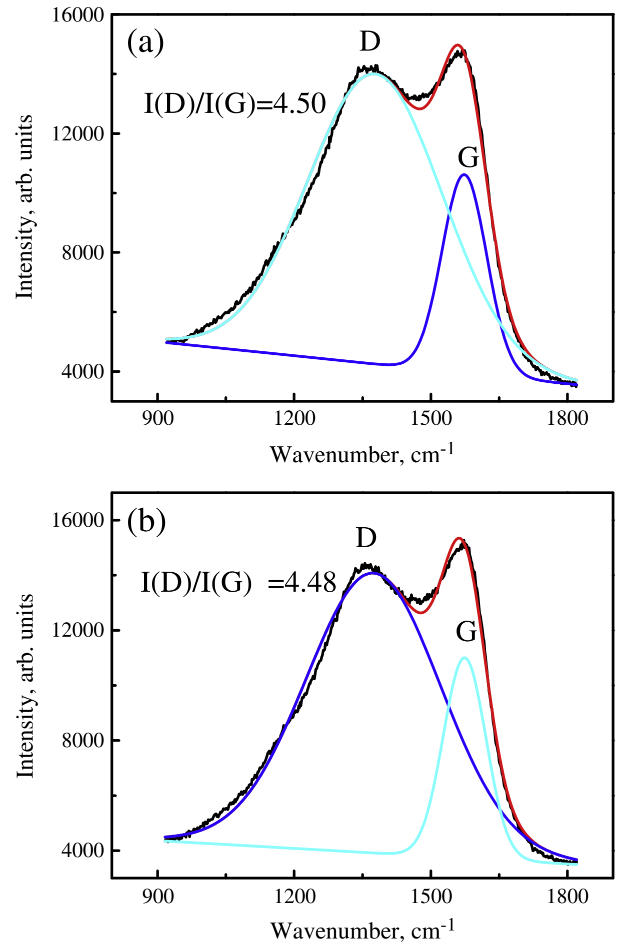

Fig. 2. Raman spectrum of the DLC layer of (a) Ti/Ti-DLC and (b) Ti/AlTiN/Ti-DLC coatings.

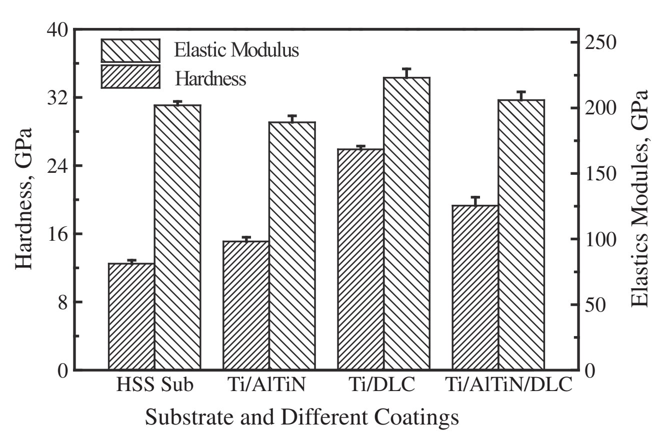

Fig. 3. Hardness and elastic modulus of the steel substrate, Ti/AlTiN, Ti/Ti-DLC and Ti/AlTiN/Ti-DLC coatings.