



Microstructure, mechanical and tribological behaviors of MoS₂-Ti composite coatings deposited by a hybrid HIPIMS method

Xiaopeng Qin ^{a,*}, Peiling Ke ^{a,*}, Aiying Wang ^{a,*}, Kwang Ho Kim ^b

^a Ningbo Key Laboratory of Marine Protection Materials, Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, Ningbo 315201, China

^b National Core Research Center for Hybrid Materials Solution, Pusan National University, Pusan 609735, Korea

ARTICLE INFO

Article history:

Received 21 November 2012

Accepted in revised form 17 April 2013

Available online 24 April 2013

Keywords:

Hybrid high power impulse magnetron sputtering

MoS₂-Ti composite coatings

Microstructure

Tribology

ABSTRACT

The MoS₂-Ti composite coatings were deposited by a hybrid high power impulse magnetron sputtering (HIPIMS) source of Ti combined with a direct current magnetron sputtering (DC-MS) source of MoS₂. The composition, microstructure, mechanical and tribological behaviors of the MoS₂-Ti composite coatings were investigated using the various analytical techniques (XPS, SEM, XRD, TEM, nano-indentation, scratch and ball-on-disk test). The results showed that doping Ti using HIPIMS technique enabled MoS₂ coatings to grow in the form of a dense amorphous structure. The crystallization degree of the MoS₂-Ti composite coatings decreased with the increase of doped titanium content. Ti reacting with O to form titanium oxides in the surface inhibited the oxidation of MoS₂. The hardness and adhesion of the composite coatings reached its maximum within a certain range of Ti content. Doped Ti improved the tribological properties of pure MoS₂ coatings in the atmospheric environment. The coefficient of friction (COF) decreased with the increase of Ti content. The lowest average COF at 0.04 and the wear rate at 10⁻⁷ mm³ N⁻¹ m⁻¹ were achieved at the optimum of Ti content at 13.5 at.%. The improved tribological property was discussed in terms of the obtained higher hardness and better adhesion of the composite coatings combined with inhibition of MoS₂ oxidation.

© 2013 Elsevier B.V. All rights reserved.

1. Introduction

Sputtered MoS₂ coating as an excellent solid lubricant has been widely used in the vacuum and space field such as the spacecraft motion components and rolling bearings due to the high wear resistance durability and very low coefficient of friction [1,2]. However, pure sputtered MoS₂ coating generally exhibits the loose structure, low hardness and high chemical activity to oxygen, resulting in the deteriorated wear durability and the corrosion resistance [3]. Recently, doping small amount of metal or ceramic elements into MoS₂ coatings has been attempted to improve the lubricant and corrosion performance of MoS₂ coating [4–9]. It is found that doping the metals such as Al, Au, W, etc., in the MoS₂ coatings by magnetron co-sputtering showed good friction stability in ambient air with long-lasting wear durability [5–7]. In addition, introduction of TiN or TiB₂ was also developed to modify the tribological properties of pure MoS₂ coatings [10,11]. Specially, note that the addition of Ti into the MoS₂ coating in recent years has drawn much attention because of the significant improvement of the oxidation resistance and tribological performance dependent on the humidity in ambient air [12].

Usually, the employed doping metal components are acquired using a conventional direct current magnetron sputtering. However,

the ionization degree of the plasma particles is relatively low, leading to poor coating adhesion to substrate and densification deterioration of coating structure [12,13]. As a consequence, the coating was suffered from the structural degradation due to oxidation causing lubrication failure.

A magnetron sputtering method, which is called high power impulse magnetron sputtering (HIPIMS), has been developed since 1990s, where high density plasma with electron densities about 2–3 orders of magnitude larger than those obtained in conventional magnetron sputtering and high sputtering particle ionization rate may be achieved [14–16]. A great deal of research has been conducted to study the effects of this technique on properties of the deposited coating [17,18], such as densification, changes in structure and properties of coating [19]. From a durability and reliability prospective of MoS₂ coatings in multi-environmental application, if the HIPIMS technique as a metal plasma source is combined with the deposition of MoS₂ coatings, rather than the general used DC-MS and cathodic arc plating hybrid method, one can expect that the structure and properties of coating could be well tailored according to the demanded applications [19].

HIPIMS requires higher excitation voltage, then the ionized particles may be draw back by target itself because of the high negative voltage of the target surface, causing low film deposition rate. To solve the problem, a modified HIPIMS power supply coupled a DC unit with the high power pulse unit has been employed in the

* Corresponding authors. Tel.: +86 574 86685036.

E-mail addresses: kepl@nimte.ac.cn (P. Ke), aywang@nimte.ac.cn (A. Wang).

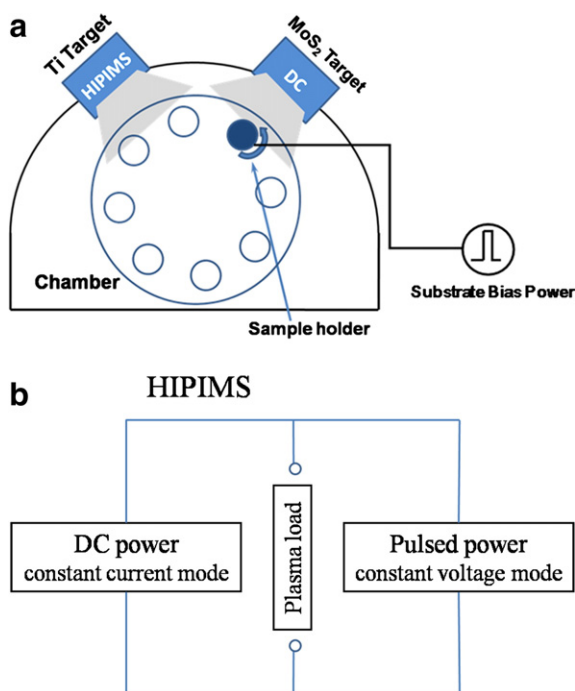


Fig. 1. Schematic diagrams: (a) the hybrid HIPIMS deposition system of MoS₂-Ti composite coatings and (b) a parallel connection operation mode of HIPIMS power supply.

present work. On the one hand, a high deposition rate can be obtained by the coupled DC unit; on the other hand, a DC unit could optimize pulse glowing and plasma stabilization through pre-ionization [11,20]. During the deposition, the power supply was able to deliver both pulses and DC, where a DC unit was also used to easily control the doped Ti content in the coatings. In addition, a high power impulse could produce plasmas with highly ionized metallic species with high ion energy. In the preliminary research work, we obtained the optimized parameters of high power pulse part, pressure and bias voltage, which were fixed in the process and were beneficial to enhanced mechanical and tribological behaviors of MoS₂-Ti composite coatings.

MoS₂-Ti composite coatings with different Ti contents were thereafter deposited by the co-sputtering of the hybrid HIPIMS system for Ti and a DC magnetron sputtering unit for MoS₂. Different Ti doping contents in the coatings were obtained by varying the target current. The influence of Ti contents on microstructure, mechanical properties and tribological behaviors in atmospheric environment was investigated.

2. Experimental details

Hybrid high power impulse magnetron sputtering system was employed to deposit the pure MoS₂ and MoS₂-Ti composite coatings onto mirror-finished high speed steel (HSS) discs and silicon P-(100) substrates. The system was combined with a Ti target connecting to high power impulse power supply with a MoS₂ target

connecting to DC power supply. The distance between the target and the substrate was 11 cm. The schematic diagram of the system is shown in Fig. 1(a), and the parallel connection of HIPIMS power supply is shown in Fig. 1(b), in which the pulsed power by the constant voltage mode is adjusted to achieve the high impulse power and the DC power by the constant current mode was regulated to obtain the different content of doped Ti. Before loading into the vacuum chamber, all the substrates were ultrasonically cleaned in acetone and ethanol for 15 min, respectively, and then dried in air. Thereafter, the cleaned substrates were mounted on the rotated substrate holder in the chamber. Prior to deposition, the chamber was pumped down to less than 3×10^{-5} Torr, and the substrates were cleaned in the argon plasma for 30 minutes. The Ti interlayer (~100 nm) was first constructed to enhance the coating adhesion to the substrate. During the deposition of top MoS₂-Ti composite coatings, DC magnetron sputtering current applied onto the MoS₂ target was fixed at 1.0 A, and HIPIMS power with various currents (0.5, 0.8, 1.0, 1.5 and 2.0 A) was supplied to the Ti target magnetron sputtering unit to control the doped Ti content in the coatings. The process parameters are shown in Table 1. A negative pulsed direct current bias, with a frequency of 350 kHz and reverse time of 1.1 μ s, was applied to the substrates during the coating deposition.

The thicknesses of the deposited coatings were measured by a surface profilometer (KLA-Tencor, Alpha-Step IQ) through a step between the coatings and the Si wafers covered with a shadow mask. Surface morphology of the coatings was studied by a field emission scanning electron microscope (S4800, Hitachi). The composition and chemical bonds of the deposited coatings was analyzed by X-ray photoelectron spectroscopy (XPS, Axis ultraDLD) with Al (mono) K α irradiation at the pass energy of 160 eV. Before taking the measurement, an Ar⁺ ion beam with the energy of 3 keV was used to etch the sample surface for 5 min to remove contaminants. X-ray diffraction (XRD) measurements were performed by AXS D8 Advance diffractometer (Bruker). High-resolution transmission electron microscopy (TEM) of the coatings was carried out on a Tecnai F20 electron microscope (FEI), which was operated at 200 KeV with a point-to-point resolution of 0.24 nm. The specimens for TEM analysis, with thicknesses of about 50 nm, were deposited directly on freshly cleaved single-crystal NaCl wafers and then were peeled off through dissolving the NaCl wafers in the deionized water.

Mechanical properties of the coatings were tested by the nano-indentation technique (MTS NANO G200) in a continuous stiffness measurement mode using a Berkovich diamond tip. The characteristic hardness was chosen in a depth of around 1/10 of the coating thickness where the contribution of Si substrate to the results could be ignored. The adhesion of the coatings on the HSS substrate was performed by a CSM scratch tester with a Rockwell-G diamond

Table 1
Process parameters for MoS₂-Ti composite coatings deposition.

Deposition parameters	Ti interlayer	MoS ₂ -Ti composite coatings
Ar (sccm)	40	50
Bias voltage (V)	−100	−300
Pulse width (μ s)	100	100
Pulse frequency (Hz)	100	100
Ti target pulse voltage (V)	500	500
Ti target direct current (A)	2.0	0.5, 0.8, 1.0, 1.5, 2.0

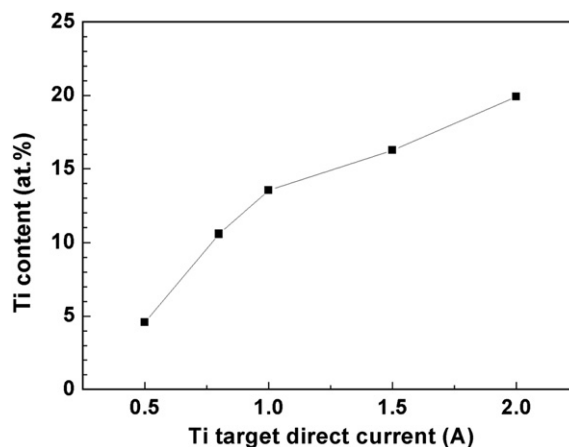


Fig. 2. Ti content of MoS₂-Ti composite coatings with different Ti target direct currents.

Download English Version:

<https://daneshyari.com/en/article/8029959>

Download Persian Version:

<https://daneshyari.com/article/8029959>

[Daneshyari.com](https://daneshyari.com)