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Pulsed DC magnetron sputtered MoS_x–TiN composite coating for improved mechanical properties and tribological performance

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ABSTRACT

This work reports on the morphological, mechanical and wear properties of MoS_x–TiN composite films, deposited by closed-field unbalanced magnetron sputtering (CFUBMS) process applying pulsed DC power supply simultaneously to the targets and substrate bias. The structure of the coating was studied using grazing incidence X-ray diffraction (GIXRD), field emission scanning electron microscopy (FESEM) and energy dispersive X-ray (EDX) microanalysis. EDX analysis showed presence of around 30% Ti in the composite matrix. Presence of MoS_x peak in GIXRD profile confirmed the presence of MoS_x as a compound, not as elemental Mo and S. One improvement of the composite architecture was attempted by combining the composite architecture with hard TiN underlayer and soft MoS_x top layer. The adhesion, hardness and wear properties were studied by scratch test, Rockwell and Vickers indentation and pin-on-disc test respectively. Adhesion was found to be improved in the composite architecture compared to pure MoS_x and TiN coating. The composite architecture also showed superior tribological performance as compared to pure MoS_x and TiN coatings.

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1. Introduction

Surface engineering or surface treatment by applying a thin layer of coating on the substrate has been one of the most widely applied methods to improve mechanical and tribological performance of a component. Conventionally, thin hard coating is applied on the component to improve the wear characteristics, hardness etc. but being hard and brittle they often fall short in performance. The friction coefficient of the hard coatings is also on the higher side which limits its use in tribological applications. Even in cutting tool applications hard grains of the worn coating may cause rapid failure due to three-body abrasion instead of showing the 'wear pad' effect expected from them. Sometimes they are used along with environmentally harmful liquid lubricant to reduce the temperature and friction force.

Use of solid lubricants (e.g. MoS_2, Graphite) can possibly circumvent the environmental problem and they are capable of reducing friction between two contacting surfaces [1]. Sputtered MoS_x ($x \approx 1.2$–2) is often used in space or vacuum application, where they have shown very low friction coefficient and long lifetime [2]. In 1993, Rechberger et al. first introduced the concept of soft 'physical vapour deposition' (PVD) coating in machining by applying MoS_2 coating on HSS cutting tools [3]. However, the poor abrasion resistance along with rapid oxidation under humid condition limits its wide use [4]. Spalvins [5] showed that when the thickness of MoS_2 layer becomes more than 80 nm (i.e. after a thin layer at the surface) porous columnar structure starts to grow which is prone to oxidation due to higher porosity. Buck [6] reported formation of Type I, Type II and amorphous MoS_2 coatings with increasing humidity in the deposition chamber. Type II shows desired orientation where c plane is parallel to substrate whereas in Type I film c plane is perpendicular to substrate.

It is evident that the combination of hard and soft layer in the coating architecture can provide the desired combination of properties. This was initially tried out by putting MoS_x top layer over hard underlayer of TiCN and TiAlN. This double coating system was found to be effective in machining sticky materials like aluminium [7]. However, due to poor wear resistance of the soft MoS_x top layer, it often gets consumed in the break-in wear phase while put into machining application [8]. As one of the remedies, inclusion of metal dopants into MoS_x matrix was attempted. This leads to some improvement in tribological performance. The possible causes are sacrificial gettering of oxygen by the metal during the wear [9], higher amount of elastic recovery by the metal doped coating [10], more densified coating structure [11] and more basal oriented structure compared to pure MoS_2 coating [12]. MoS_2/Ti composite coating developed by Teer Coatings Limited has given excellent results in many cutting and forming applications [13,14]. Another method is the formation of nano-scaled MoS_x/metal multilayer structure [15] which causes structural improvement in MoS_x by stopping the columnar growth [16].

The more recent method is the inclusion of MoS_x in some hard matrix (e.g. TiN,). Gilmore et al. first adopted this route by using a composite target [17] and in a separate work using combined arc evaporation and DC magnetron sputtering [18]. Later Rahman et al.

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worked on this architecture using DC magnetron sputtering with or without Ti/TiN_x graded interlayer for better graduation of properties between substrate and coating [19,20]. Effect of plasma nitriding the surface prior to deposition of TiN–MoS_x coating on its mechanical properties was also studied [21]. However in all such endeavours, TiN was the dominant phase (90–92 mol%) and a separate MoS_x phase could not be detected. No detailed investigation of surface morphology of this proposed architecture was carried out.

TiN, in addition to its higher hardness and load bearing capacity [22], forms lubricious rutile transfer layer in the presence of environmental humidity [23] which, while substoichiometric often leads to the formation of lubricious magneli phases [24]. Hence, the architecture which combines both MoS_x and TiN is expected to be hard as well as lubricious. Pulsed power supply at the target can reduce the problem of micro arcs and consequent target poisoning whereas pulsing the power at substrate bias circuit makes the plasma field more energetic compared to DC power supply and significant increase in ion current density can be obtained at the same bias voltage [25]. Application of pulsed DC magnetron sputtering has thus become an effective technology for deposition of various hard and soft coatings with improved properties [26–28]. Similar benefit should be reasonably expected for MoS_x–TiN composite coating with or without TiN underlayer which has not been reported so far.

In the present work, a composite coating architecture was developed where TiN was incorporated into soft matrix of MoS_x with or without hard and wear resistant TiN underlayer. This was achieved by co-deposition using pulsed DC CFUBMS technique. The relative effects of composite coating architecture in comparison to pure MoS_x and TiN coating on crystallographic orientation, morphology along with mechanical properties like hardness, adhesion, friction and wear characteristics have been investigated.

2. Experimental

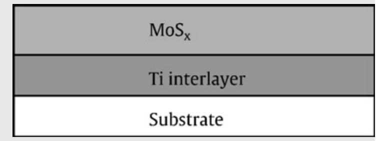
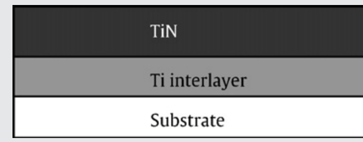
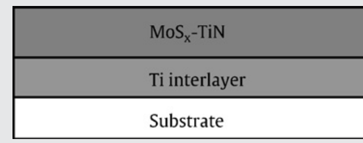
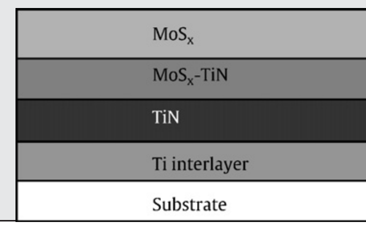
2.1. Coating deposition

IS C40 (AISI 1040/DIN 1.1186) steel substrate was selected for tribological analysis and M2 grade high speed steel (HSS) substrate was considered for other physical and mechanical characterisation tests. The compositions of these materials are given in Table 1. Four different coating architectures, as shown in Table 2 along with their corresponding coating thickness, were studied in the present investigation. The substrates were polished to a roughness value of $R_a = 0.05$ µm and then ultrasonically degreased with trichloroethylene and isopropyl alcohol prior to deposition. All the coatings were deposited by a dual cathode closed-field unbalanced magnetron sputtering system (TOOL COATER, VTC-01A) manufactured by Milman Thin Films Pvt. Ltd., India. The system consisted of one Ti and one MoS_2 target (with the dimension of 254 mm × 127 mm × 12 mm and 99.9% purity). Substrates placed in a substrate holder were rotated between two targets at a speed of 3 rpm (2-fold rotation). Target to substrate distance was approximately 130 mm. The schematic representation of the coating deposition set up is shown in Fig. 1. Both the targets as well as the substrate bias were powered with three pulsed DC power supplies, each of 10 kW with variable voltage and current controllers. The power supplies were operated with a pulse frequency (f) of 35 kHz and pulse on time (T_{on}) of 25 µs both for the cathodes as well for the substrate bias throughout the entire deposition cycle and for all the coating architectures. The chamber was first pumped down to a base pressure of approximately 2×10^{-4} Pa followed by pre-sputtering of the targets in argon atmosphere with a chamber pressure of 0.2 Pa with shutters closed. In order to reduce water vapour content in the vacuum chamber, Ti was sputtered under argon, during ion cleaning of the substrates prior to deposition with pulsed substrate bias of −400 V ($f = 35$ kHz, $T_{on} = 25$ µs). Initially, Ti interlayer of around 100 nm thickness was deposited over the substrate in order to promote improved film-substrate adhesion and minimize residual stress in the coating [29]. Ti target was activated for 5 min. with a pulsed DC power of 620 W during this operation. This was followed by the deposition of coatings of different architectures as shown in Table 2. The applied pulsed DC power to MoS_2 target was 200 W (for specimens A1, A3 and A4) and to Ti target was 620 W (for specimens A2, A3 and A4) during the depositions of coatings. Ar and N_2 gas flow were individually controlled by mass flow controllers (EL-FLOW, Bronkhorst). During deposition of MoS_x as a top layer of specimens A4 and A1, power to Ti target was switched off. All coating architectures were deposited at a working pressure of 0.2 Pa, substrate temperature of 200 °C and pulsed substrate bias voltage of −50 V.

Table 1
Chemical compositions of substrate materials.

Composition (%)	M2 grade HSS	IS C40
C	0.76	0.4
W	6.3	–
Mo	4.3	–
Cr	3.95	–
V	1.64	–
Mn	–	0.7
Si	–	0.2
P	–	0.04
S	–	0.05
Fe	Rest	Rest

Table 2
Different coating architectures along with corresponding layer thickness.

Specimen	Coating architecture	
A1	Ti / MoS_x(1.5 µm)	
A2	Ti / TiN (3 µm)	
A3	Ti / (MoS_x–TiN) (1 µm)	
A4	Ti /TiN (1 µm)/MoS_x–TiN (800 nm)/MoS_x (50 nm)	

2.2. Coating analysis

Surface morphology of the coatings was studied by a high resolution Carl Zeiss, Supra 40 field emission scanning electron microscope (FESEM). The composition of the as-deposited films was determined by energy dispersive X-ray (EDX) microanalysis at 20 keV coupled with the FESEM. Film thickness was also determined by observing the coating fractographs under FESEM. Grazing Incidence X-ray Diffraction

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