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ScienceDirect
Physics Procedia 18 (2011) 234–239
Physics Procedia

The Fourth International Conference on Surface and Interface Science and Engineering

The effect of Ti content on the structural and mechanical properties of MoS_2-Ti composite coatings deposited by unbalanced magnetron sputtering system

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Abstract

Pure MoS_2 coating and MoS_2-Ti composite coatings with different Ti content (5～15wt.%) have been deposited by HTC 750 unbalanced magnetron sputtering system. The structural and mechanical properties of these coatings as a function of Ti content have been studied. SEM analysis show that the pure MoS_2 coating reveals a typical porous and worm-like surface structure, the MoS_2 -Ti composite coatings appear densified and compact microstructure and coating porosity decrease with an increase of the Ti content. Nanoindentation test show that the hardness and Young's modulus of the coatings increase with an increase of the Ti content. Nano-scratch test show that the Ti doped composite coatings improve their adhesion to the substrate apparently. Friction and wear test results show that the pure MoS_2 coating appears poor tribological behavior which the friction coefficient is about 0.06 and the endurance life is 5850m. MoS_2 -Ti composite coatings not only show low friction coefficient but also low wear rates. The friction coefficient of composite coatings is between 0.02 and 0.04 , and the endurance life of them improve compared to the pure MoS_2 coating.

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PACS: 81.05.-t; 68.55.-a; 68.35.Np; 62.20.Qp.
Keywords: MoS_2 –Ti composite coating; structure; mechanical properties.

1. Introduction

MoS_2 has a 2-D layered crystalline structure[1,2] which provides low shear strength along its basal planes, it's coefficient of friction is low and which can be as low as 0.01[3,4] in vacuum. Because of this characteristic, MoS_2 has already been used as solid lubricant extensively in moving mechanical assemblies of spacecraft such as holding and release mechanisms of solar array, gimbal bearings and precision bearings[5,6].

MoS_2 coatings can be produced by burnishing, bonding and magnetron sputter PVD techniques. Compared to other methods, magnetron sputter MoS_2 coatings can be deposited as thin as 1 μm, shows lower friction coefficients and better adherence, which is suitable for surface lubrication modifying of precise moving parts. However pure

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doi:10.1016/j.phpro.2011.06.087

MoS_2 coatings have low wear resistant life, and MoS_2 can react with O and H_2O in high humid atmosphere to produce MoO_3 and H_2SO_4[7] which lead to degradation of coatings and corrosion of metal. All around the world it is interested in improving the structure and properties of these kind of coatings through doping metals[8-11] and oxides such as PbO[12] and SbOx[13], or using new deposition technologies such as ion beam assisted deposition[14] and unbalanced magnetron sputtering[15-17].

Unbalanced magnetron sputtering deposition technology was developed in 1985[18], and the corresponding equipment was produced since 1990s[19]. In this equipment, a multimagnetron system exist in which unbalanced magnetrons are used in the closed-field arrangement. Here the magnetic field lines from neighboring magnetrons are linked, trapping the plasma, thereby increasing the ion-current density and bombardment intensity to the substrates. This kind of deposition method will densify structure and improve adhesion of coatings.

It has also been found that the properties of MoS_2 coatings can be further improved by the co-deposition of small amounts of metals such as Au, Ag, Pb, Ti, Cr. The content of the doping metals will have important effect on structure and properties of the composite coatings. In this paper the correlation about the Ti contents and structure and mechanical properties of MoS_2-Ti composite coatings is reported.

2. Experimental details

2.1 Deposition

MoS_2-Ti composite coatings were deposited by means of the closed field unbalanced magnetron sputter ion plating technique(CFUBMSIP) by using standard Hauzer HTC-750 equipment on 45 steel and silicon substrates. The arrangement of the magnetrons and target materials within the coating chamber is as shown in Fig. 1. Two MoS_2 targets and one titanium target were used. The procedure starts with the deposition of a 100-nm Ti interlayer, which led to an improvement in coating adhesion. This step is followed by sputtering from two MoS_2 targets and the titanium target simultaneously and the substrates rotated between the three. The amount of titanium content in samples was controlled by the power applied to the target. The main deposition parameters are shown in Table 1.

Table 1 Main deposition parameters of MoS_2-Ti composite coatings

Targets	MoS_2 (99.0%)	Ti (99.9%)
Target power (kW)	5	0-5
Base pressure (Pa)	1×10^{-3}	
Ar pressure (Pa)	0.5	
Deposition temperature (℃)	~100	
Substrate bias (V)	-100	
Unbalanced coil currents (A)	5	
Deposition time (min)	60	

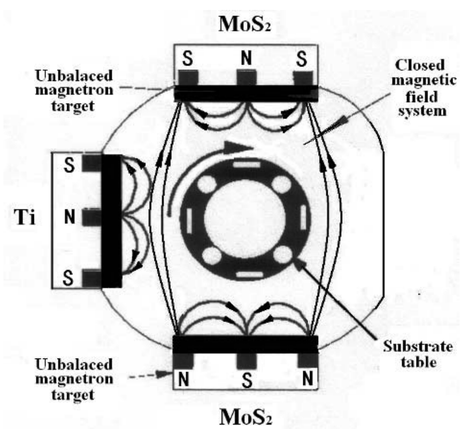


Fig. 1. Schematic representation of coating chamber.

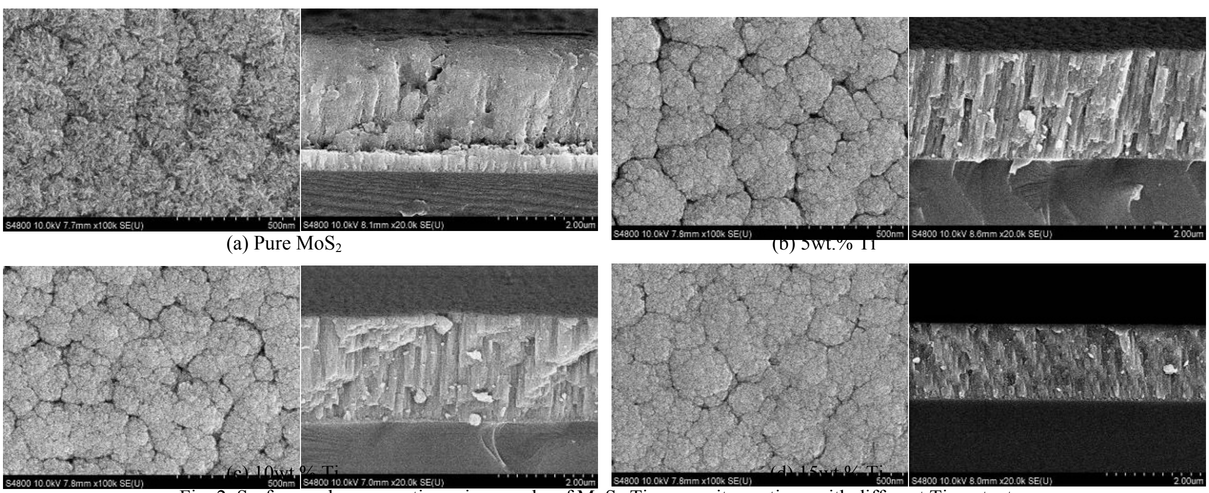
2.2 Analysis and testing

SEM analysis was carried out using a S-4800 equipment operated at 10 kV. XRD measurements were performed using Philips X′Pert PRO equipment with Cu Kα radiation and glancing angle of 1° in order to enhance the signal from the coating. XRD patterns were collected between 10° and 70° 2θ angles. Nanoindentation tests were performed using a Berkovich diamond (20μm radius) with an indentation depth of 150 nm. An average value for nanohardness and Young's modulus was calculated from fifteen measurements. Scratch tests were performed using a CSEM nano-scratch equipment with a Rockwell-C diamond (2μm radius) which was drawn across the surface of the coating at a constant linear velocity of 10mm min^{-1} while increasing the load linearly from 1 to 110 mN (equipment's up limit). The critical load Lc, the load at which the coating undergoes adhesive failure, is given by the first maximum peak in a plot of the first derivative of the friction against load curve and by optical examination. The tribological properties of coatings have been tested by ball-on-disc friction and wear tester at humidity between 30 and 50% in air. The tests were performed at 5 N at diameters of 24 mm. The sliding speed was 1.257 m s^{-1}(1000 rpm). A 8 mm diameter 440C stainless steel ball was used as the counterpart. The friction coefficient (μ) was continuously monitored during the tests. The wear life of the coatings(τ) was defined to be the distance of passes at which the μ rose to 0.2.

3. Results and discussion

3.1 SEM analysis

The morphology of studied Coatings' surface and cross-section are shown in Figure 2. It can be seen that the morphology of pure MoS_2 coating is entirely different to those of MoS_2-Ti composite coatings. The pure MoS_2 coating reveals a typical worm-like surface structure, densified Ti interlayer can be seen apparently in the cross-section picture, porous and loose pure MoS_2 coating deposited on the Ti interlayer. The surface of MoS_2-Ti composite coatings are made of many small bulges, and the dimension of these bulges reduce in scale when Ti content increase. When Ti content is 5wt.%, the size of bulges is about (300～500) nm, when Ti content increased to 15wt.%, the size of bulges is about 200 nm. As the Ti content increasing, the coatings become more dense and smooth. The cross-section morphologies of MoS_2-Ti composite coatings with different Ti content are similar, all appear in dense coherent column structure and the Ti interlayer can't been found.



(a) Pure MoS_2 (b) 5wt.% Ti (c) 10wt.% Ti (d) 15wt.% Ti

Fig. 2. Surface and cross-section micrographs of MoS_2-Ti composite coatings with different Ti content

3.2 *XRD analysis*

XRD patterns of pure MoS_2 and MoS_2-Ti composite coatings with different Ti contents are shown in Figure 2. All of the coatings deposited on silicon samples . It can be seen that the pure MoS_2 coating is crystalline and the strongest peak is found in the (002) plane then the (101) and (103) plane in turn. None of these peaks occur on analysis of the MoS_2-Ti composite coatings, only revealed two broad band pattern at 2θ 10～18º and 30～45º. It would appear that the doping of Ti into the coatings inhibits the formation of crystalline MoS_2.With increasing of Ti content, the intensity of broad band at 2θ 10～18º which corresponding to the MoS_2(002) plane decrease and the broad reflection peak lines shift to lower diffraction angles . At the same time,the intensity of another broad band at 2θ 30～45º which corresponding to the Ti(002) plane and Ti(101) plane increase. The results show that MoS_2 -Ti composite coatings are at least quasi-amorphous and incorporation Ti in the composite coatings is most likely in the space between the S planes or making junctions between MoS_2 lattices. When too much Ti is doped in the coating, some new crystallite like Ti(002) and Ti(101) can produce.

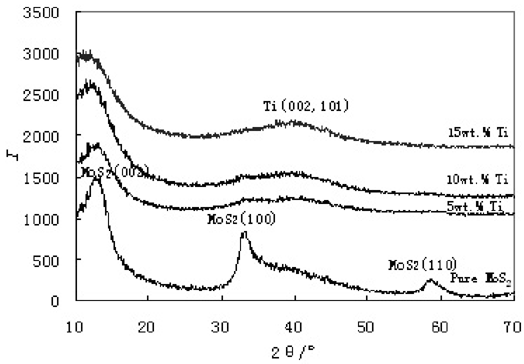


Fig. 3. XRD patterns of MoS_2-Ti composite coatings

3.3 *Nanoindentation*

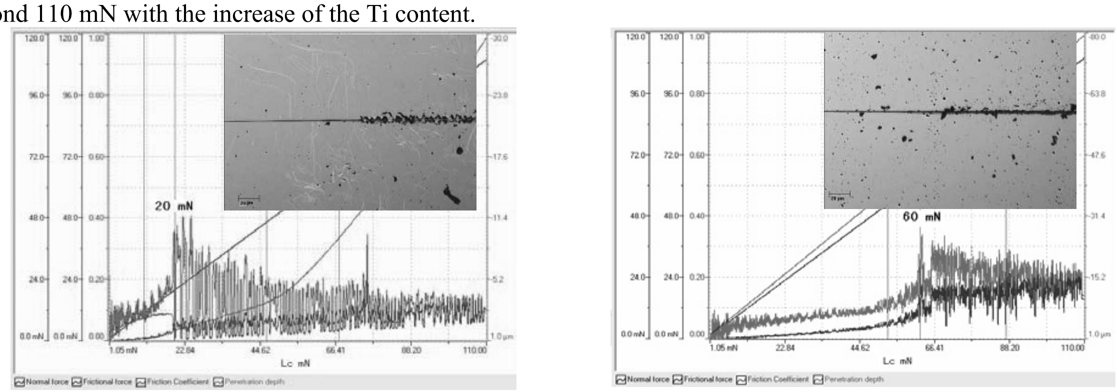
The relationship between Ti contents and the nanoindentation results of the coatings are shown in table 2. The results show that the nanohardness and Young's modulus of the coatings increase with an increase of the Ti content. The nanohardness value of the pure MoS_2 coating is only 1 GPa .When doped Ti in the coating ,the nanohardness of the film increased quickly and which can arrive at 5.7 GPa when Ti content is 15wt.%. The Young's modulus values of the pure MoS_2 coating is 27 MPa, and the values of composite coatings can increase from 44 to 100 MPa. It can be supposed that the improved mechanical properties of the Ti doped composite coatings come from densified microstructure and crystal lattice deformation.

Table 2 Nanoindentation results of MoS_2-Ti composite coatings with different Ti content

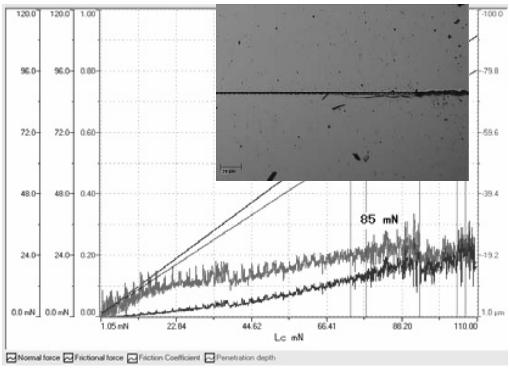
Results	Ti content (wt.%)			
	0	5	10	15
Hit (GPa)	1.0	2.8	3.5	5.7
Eit (MPa)	27	44	69	100

3.4 *Scratch testing*

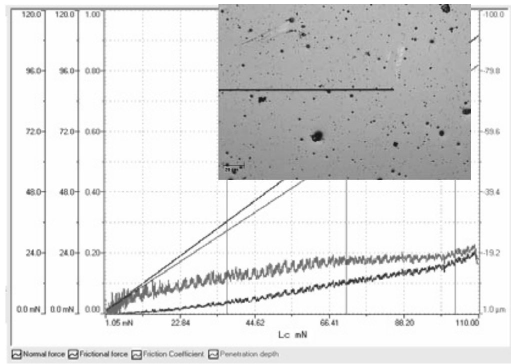
The scratch test images of MoS_2-Ti coatings with different Ti content are shown in Figure 4. The results show that the adhesion of the pure MoS_2 coating to the substrate is poor, and the ultimate failure occurs at an average load of 20 mN. Ti doped composite coatings improve their adhesion to the substrate apparently, Ti concentration appears to play an important role in the adhesion behavior of the coatings, the critical loads increase from 60 mN to beyond 110 mN with the increase of the Ti content.



(a) Pure MoS_2 (b) 5 wt.% MoS_2



(c) 10 wt.% MoS_2

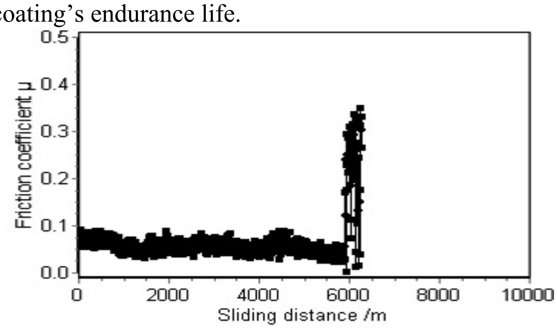


(d) 15 wt.% MoS_2

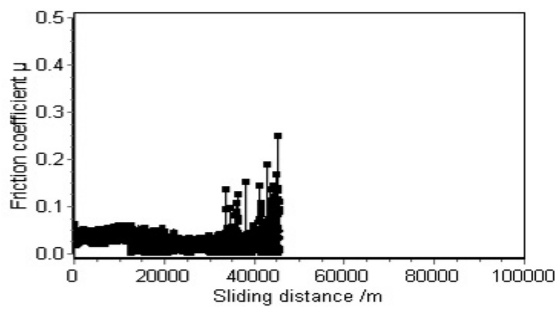
Fig. 4. Scratch test results of MoS_2-Ti composite coatings with different Ti content

3.5 *Friction and wear test*

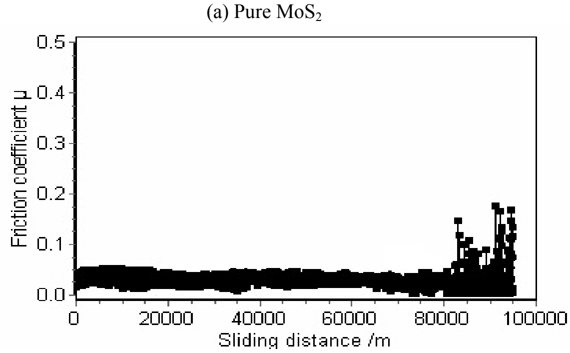
The results of coating's friction and wear test are shown in Figure 5. When tested in air, temperature varied from 18 to 25℃ with humidity is below 50%. The results show that the pure MoS_2 coating appears very poor tribological behavior , the friction coefficient of the coating is higher than those of the composite coatings and the value is about 0.06 , the endurance life of the coating is shorter than those of the composite coatings which is 5850m. MoS_2 -Ti composite coatings not only show lower friction coefficient but also show longer endurance life. The friction coefficient of the composite coatings is similar and varies between 0.02 and 0.04 , and the endurance life of them improve apparently when compared to the pure MoS_2 coating. The best endurance life of the MoS_2 -Ti composite coating can reach 80000 m, which is about 13 times longer than that of pure MoS_2 coating. It can be supposed that when composite coatings worked in air environment, Ti in the composite coatings may react with O elements to produce Ti oxide, which will be helpful to improve coating's wear resistant properties. On the other hand, Ti doped in the composite coatings can densify coating's microstucture, which can also contribute to increase coating's endurance life.



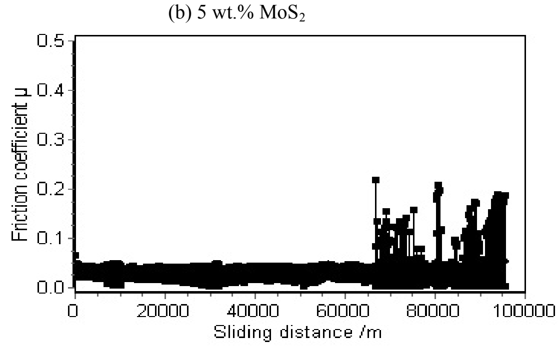
(a) Pure MoS_2



(b) 5 wt.% MoS_2



(c) 10 wt.% MoS_2



(d) 15 wt.% MoS_2

Fig. 5. Ball-on-disc test results of MoS_2-Ti composite coatings with different Ti content in air environment

4. Conclusions

Pure MoS_2 coating reveals a typical porous and worm-like surface structure, the MoS_2 -Ti composite coatings appear densified and compact microstructure and coating porosity decrease with an increase of the Ti content. Pure MoS_2 coating is crystalline and the strongest peak is found in the (002) plane then the (100) plane and (110) plane in turn. MoS_2 -Ti composite coatings reveal only a broad reflection peak between 10-20° 2θ scattering angle range peaked at about 13° which corresponding to the (002) plane, and the broad reflection peak lines are shifted to lower diffraction angles as the Ti content is increased. The results show that MoS_2 -Ti composite coatings are at least quasi-amorphous and incorporation Ti in the composite coatings is most likely in the space between the S planes or making junctions between MoS_2 lattices. The hardness and Young's modulus of the coatings increase with an increase of the Ti content. The hardness values of the coatings increase from 1 to 5.7 GPa , and the Young's modulus values increase from 27 to 100 MPa. The scratch tests show that pure MoS_2 coating fail at an average load of 20 mN, Ti doped composite coatings improve their adhesion to the substrate apparently, Ti concentration appears to play an important role in the adhesion behavior of the coatings, the critical loads increase from 60 mN to beyond 110 mN with the increase of the Ti content. The ball-on-disc friction and wear test show that the pure MoS_2 coating appears poor tribological behavior which the friction coefficient is about 0.06 and the endurance life is 5850m. MoS_2 -Ti composite coatings not only show low friction coefficient but also low wear rates. The friction coefficient of composite coatings is between 0.02 and 0.04 , and the endurance life of them improve apparently compared to the pure MoS_2 coating.

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