Study on Synthesis and Tribological Properties Characterization of MoS$_2$-Ti$_L$/MoS$_2$-Ti$_H$ Nano Multilayer Coating

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Abstract. Benefitting from its novel structure, MoS$_2$-Ti$_L$/MoS$_2$-Ti$_H$ nano multilayer coating possesses excellent tribological properties. The multilayer coating was prepared by varying the power of Ti target alternatively using magnetron sputtering technology. To investigate its tribological properties, more extensive analyses have been carried out on a reciprocating friction and wear testing machine under different loads. Then, wear trace morphology was studied by a confocal microscope. The friction coefficient is very stable around 0.1~0.2 at a load smaller than 20 N. Correspondingly, wear trace is narrow and the wear loss is very small under a small load. With a larger load, wear traces become wider and the lubricant only lasted for a short time. Whereas, compared to MoS$_2$-Ti composite coating, the multilayer coating has the advantage apparently on wear resistance and lubrication.

1. Introduction

MoS$_2$ based composite coatings with small amount of oxides such as PbO, Sb$_2$O$_3$ $^{[1,2]}$ or metals such as Ti, Au, Nb, Pb, Cr, Zr, Ni, W $^{[3-11]}$ have drawn much attention as excellent solid lubricants due to their potential application in ambient air. Special attention has been paid on the addition of Ti into MoS$_2$ coating because of the significant improvement of the oxidation resistance and wear resistance performance in humid environment $^{[7-13]}$. With the addition of Ti, the structure of MoS$_2$-Ti coating became more compact, the surface smoother, the binding force with substrate stronger and the coating harder. As well as, TiO$_2$ generated on the coating surface could prevent oxygen diffuse into the inside coating $^{[9-13]}$. As a consequent, the performance of oxidation resistance and wear resistance got improved.

However, the lubricating properties of MoS$_2$-Ti coating would deteriorate with more Ti content. It could result in too much TiO$_2$ content in the coating, which could reduce the lubricating component and cause lubrication failure finally $^{[10]}$. Meanwhile, densification deterioration of coating structure caused by the columnar growth of the deposition coating $^{[10, 14]}$ and the low ionization degree of magnetron sputtering $^{[15]}$ also limited its application in humid ambient air. In recent years, several methods have been developed to focus on making the coating structure more densification and improving its environmental adaptability $^{[14-16]}$. One of them enhanced wear properties of MoS$_2$-Ti coating by using MoS$_2$-Ti$_L$/MoS$_2$-Ti$_H$ nano multilayer structure $^{[16]}$. Such novel structure not only made...
the coating denser and more compact, but also could prevent the column structure growth effectively. Accordingly, the multilayer coating possessed lower friction coefficient, longer wear life and narrower wear trace [16].

Here, more extensive description on synthesis of MoS$_2$-Ti$_L$/MoS$_2$-Ti$_H$ nano multilayer coating and tribological properties characterization were presented. The nano multilayer coating was deposited using magnetron sputtering technology by co-sputtering MoS$_2$ target and Ti target with the power of Ti target varied alternatively. After deposition, friction and wear behavior tests were carried on a reciprocating friction and wear testing machine. Then, the wear traces were characterized by a confocal microscope.

2. EXPERIMENTAL

MoS$_2$-Ti$_L$/MoS$_2$-Ti$_H$ nano multilayer coating was prepared on single crystal silicon wafer and mirror-finished stainless steel disc by using magnetron sputtering technology with the power of Ti target varying alternatively. A Ti target (99.9 %) connecting to DC power and a MoS$_2$ target (99.9 %) connecting to RF power were adopted in the sputtering system. Argon (99.9 %) was input into the chamber as the working gas and the working pressure was kept at 8 mTorr. During depositing the nano multilayer coating, the power of MoS$_2$ target was kept constantly at 100 W. While, as shown in Fig. 1, the power of Ti target was varied from 50 W to 100 W respectively at intervals of 10 min for depositing MoS$_2$-Ti$_L$ and MoS$_2$-Ti$_H$ nanolayer thin film respectively. The whole deposition was carried out under ambient temperature for 240 min. To enhance coating adhesion with the substrates, a Ti interlayer about 100 nm thickness was constructed in advance before depositing the nano multilayer coating. Structure diagram of MoS$_2$-Ti$_L$/MoS$_2$-Ti$_H$ nano multilayer coating is shown in Fig. 2.

![Power of Ti target](image1.png)

Fig. 1. Diagram of the power of Ti target varied from 50 W to 100 W alternatively.

![Structure diagram](image2.png)

Fig. 2. Structure diagram of MoS$_2$-Ti$_L$/MoS$_2$-Ti$_H$ nano multilayer coating.

After deposition, the surface and cross section morphology of the coating were studied by SEM (S4800, Hitachi). The friction and wear behavior tests of coatings deposited on the stainless steel discs
against GCr15 steel balls were performed on a reciprocating friction and wear testing machine (UMT-3, CETR). The diameter of GCr15 steel balls was 3mm, and all tests were carried out at room temperature with a relative humidity of about 60% under a sliding velocity at 20 mm/s. The forces loaded on the coating were 2 N, 5 N, 10 N, 15 N, 20 N, 25 N, and 30 N respectively. And the total testing time was 20 min for every test. Then, a confocal microscope (CSM700, Axio) was employed to study wear traces. The abrasive loss of each wear traces was also analyzed. For contrast, the friction and wear behavior tests of MoS2-TiL monolayer composite coating (MoS2-TiL) under a load of 2 N and 5N was also performed.

3. Results and discussion

SEM images of surface and cross section of MoS2-TiL/MoS2-TiH nano multilayer coating are shown in Fig. 3 a, b respectively. As shown in Fig. 3 a, the coating is very dense and smooth. From the cross section morphology, it can be clearly seen that the multilayer coating appears a denser and compact structure with MoS2-TiL, MoS2-TiH nanolayer alternately. Especially, unlike MoS2-Ti composite coating, the column structure hasn’t been found in MoS2-TiL/MoS2-TiH nano multilayer coating according to SEM images of cross section shown in Fig. 3 b. All these improvements can be attribute to the nano multilayer structure. As this novel structure could not only make the coating denser and more compact, but also prevent the column structure growth effectively [16-18]. Consequently, the multilayer coating had more excellent wear resistance compared to MoS2-Ti composite coating benefitting from its novel structure [16].

Fig. 3. SEM images of (a) surface and (b) cross section morphology of MoS2-TiL/MoS2-TiH nano multilayer coating.

To investigate tribological properties of the multilayer coating, ball-on-disc friction tests were carried out on a reciprocating friction and wear testing machine against steel balls with different loads under ambient air. Tribological property of MoS2-Ti composite coating performed under a load of 2 N was also provided for comparison in our previous work. As shown in Fig. 4, MoS2-TiL/MoS2-TiH multilayer coating appears low friction coefficient and excellent tribological properties towards the test. Whereas, MoS2-Ti monolayer coating doesn’t have same excellent wear properties as MoS2-TiL/MoS2-TiH nano multilayer coating. The friction coefficient of multilayer coating is low and stable during the whole test. While, MoS2-Ti composite coating possesses low friction coefficient only at the beginning of the test. The friction coefficient of the monolayer coating becomes large suddenly at the test time around 200 sec and worse toward the following test. The illustration of Fig. 4 presents wear trace morphology of each coating. As can be seen, the width of wear trace of MoS2-Ti composite coating is about twice as broad as the multilayer one. Clearly, tribological properties have gotten improved remarkably by the multilayer structure.
As mentioned above, nano multilayer structure has improved tribological properties effectively. To make clear of the improved extent, tribological properties tests under a load of 2 N, 5 N, 10 N, 15 N, 20 N, 25 N, and 30 N were carried out on MoS$_2$-TiL/MoS$_2$-TiH nano multilayer coating respectively. Fig. 5 shows friction coefficient of coatings as a function of testing time under different loads, and Fig. 6 presents the corresponding wear trace morphology. In Fig. 6, a and b is the wear trace morphology of MoS$_2$-Ti composite coating under 2 N and 5N. In addition, c, d, e, f, g, h and i is the one of MoS$_2$-TiL/MoS$_2$-TiH nano multilayer coating under 2 N, 5 N, 10 N, 15 N, 20 N, 25 N, and 30 N respectively. As shown in Fig. 5, the nano multilayer coating presents excellent wear properties. The coefficient of friction remains stable and small around 0.1 ~ 0.2 at a load smaller than 20N and becomes worse accompanied by a brief begin with small value at a load larger than 25N. That means the lubrication failure after a short time under such large load. From wear trace morphology presented in Fig. 6, it can be clearly seen that the ones under a load of 25 N and 30 N are more width then any other. Moreover, the substrate is exposed clearly which means wear resistance is bad under a heavy load. While, it hasn’t been found the solid lubrication flake off under a light load from the wear trace morphology for the multilayer coating. Consequently, the coating can keep lubricate well.
Fig. 5. Friction coefficient of MoS$_2$-Ti$_{II}$/MoS$_2$-Ti$_{III}$ coatings under different loads.

Fig. 7 gives out wear trace cross section of MoS$_2$-Ti$_{II}$/MoS$_2$-Ti$_{III}$ multilayer coating under different loads. For contrast, the ones of MoS$_2$-Ti composite coating under a load of 2N, 5N are presented in this figure too. From the figure, it can be seen that the depth is extremely small under a light load and becomes larger along with the increasing of the load for the multilayer coating. As shown in the figure, the depth of wear trace of MoS$_2$-Ti$_{II}$/MoS$_2$-Ti$_{III}$ multilayer coating is much smaller compared to MoS$_2$-Ti composite coating at a load of 2N and 5N. Wear trace morphology in Fig. 6 a, b, c and d shows that the width of wear trace of the nano multilayer coating is only about one half of the MoS$_2$-Ti composite coating one at the same load respectively. Which means the wear loss of MoS$_2$-Ti$_{II}$/MoS$_2$-Ti$_{III}$ multilayer coating is much lower. Accordingly, it can be gotten the conclusion that the multilayer coating possesses more excellent wear resistance. Corresponding to wear trace morphology shown in Fig. 6 h and i, it can be seen that these two wear traces are very deep from Fig 7 h and i. Consequently, lubrication failure is mainly attribute to the fact that the lubricate coating has been worn off quickly under a large load. Which means such coating cannot work under so large load.
4. Conclusion
MoS$_2$-Ti$_{IL}$/MoS$_2$-Ti$_{IH}$ nano multilayer coating has been successfully synthesized by changing the power of Ti target alternatively using magnetron sputtering technology. Such nano multilayer possesses excellent tribological properties due to the novel multilayer structure. Compared to MoS$_2$-Ti composite coating, the multilayer coating has longer wear life and better wear resistance according to the friction and wear behavior tests. These improvements can be contributed to the novel nano multilayer structure which could make the coating denser and more compact, as well as prevent the column structure growth. More extensive analyses have been carried out on the multilayer coating under different loads. The results show that the coefficient of friction is very stable around 0.1 ~ 0.2 at a load smaller than 20 N. However, the lubricant only lasted for a short time under a larger load. Correspondingly, wear trace is narrow under a small load and becomes wider along with the increasing of the load. The wear loss is very small according to the shallow cross section of wear trace under a load small than 10 N. While, the deep wear traces under the load of 25 N and 30 N indicate that the corresponding wear loss is very large and multilayer coating has been worn off entirely. Accordingly, the coating loses lubricant so quickly under such large load. Whereas, compared to MoS$_2$-Ti composite coating, wear resistance of the nano multilayer coating has been gotten improved effectively anyhow.
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