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Effect of target power on the structure and tribological Properties of La-Ti/WS₂ composite films prepared by unbalanced magnetron sputtering

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Abstract

The properties of Physical Vacuum Deposit (PVD) films could be improved by doping rare earth elements and other metal elements. In this paper, La-Ti/WS₂ composite films were prepared by unbalanced magnetron sputtering. The effect of target power on the structure and properties of La-Ti/WS₂ composite films was studied. Scanning electron microscopy (SEM) and x-ray diffraction (XRD) were used to analyze the micro morphology, composition and crystal structure of the film. The hardness and friction properties of the film were tested by nano-indentation and friction and wear testing machine. The results show that the structure and composition of La-Ti/WS₂ composite films prepared by magnetron sputtering are affected by the target power. With the increase of the target power, the diffraction peak of WS₂ (002) was shifted to the low θ value, the crystal surface spacing was decreased, the crystal lattice shrunk, the porous structure of La-Ti/WS₂ composite films was decreased significantly, the coarse columnar crystal was refined significantly, and the film density was improved effectively. When the target power is 20 W, the sliding surface of La-Ti/WS₂ composite film (002) is parallel to the surface, showing lower friction coefficient and better wear resistance; when the target power is 50 W, La-Ti/WS₂ composite film has higher hardness, higher wear rate and poor wear resistance. The density and friction and wear properties of La-Ti/WS₂ composite films can be improved by suitable La-Ti content.

1. Introduction

With the rapid development of aerospace technology, solid lubrication materials have been widely used to solve the lubrication problems of spacecraft in high vacuum, high temperature, high load and other special conditions [1]. WS₂, like MoS₂, has hexagonal crystal structure, weak van der Waals force and low shearing force between layers, it is easy to slide when friction occurs and has excellent lubrication performance, which makes WS₂ have a good development prospect in the field of aerospace [2]. However, the application of this material has been greatly affected because it is easy to absorb moisture and oxidize in humid atmosphere, which lead to failure.

In order to improve the structure compactness of WS₂ film and its bearing capacity, doping Au, Ag, Ti, Pb or other compounds with synergistic effect such as Sb₂O₃ into WS₂ films has become an effective method to improve the properties of the films. Xu et al [3, 4] prepared WS₂/Ag and WS₂-Sb₂O₃/Cu composite films by RF sputtering, which improved the microstructure and mechanical properties of the composite films, and improved the bonding strength and wear resistance of the films in the atmospheric environment. Gang et al [5] prepared WS₂-C/Cu composite film by powder metallurgy hot pressing method, which effectively improved the friction and wear properties of composite film in air. Deepthi et al [6, 7] prepared WS₂/Au and WS₂/Cr composite films by magnetron sputtering, the results show that the doping of metal Au and Cr can improve the
compactness of composite films, reduce the wear rate of composite films and improve their oxidation resistance. Jhonattan et al. [8, 9] prepared WS2/Ti composite films by DC magnetron co-sputtering, the results confirm that the compactness and tribological properties of WS2/Ti composite films were improved, corrosion resistance was enhanced. It has been shown that the compactness, mechanical properties and tribological properties of WS2 films could be improved significantly by doping metals, metal compounds and nonmetals. However, doping two kinds of metal, metal compound or non-metal elements into WS2 film is likely to obtain more excellent properties, which can better meet the engineering requirements of special working conditions.

Rare earth element, as a very active modifier, has been widely used in the field of material surface technology due to its special electronic structure and super chemical activity [10, 11]. Through a small doping amount of rare earth, the microstructure and mechanical properties of the material can not only improve but also have an important impact on the anti-wear and anti-friction properties of the film. However, there were few reported about the modification of WS2 thin film by doping rare earth in the process of magnetron sputtering [12].

In this paper, La-Ti/WS2 composite film with appropriate La and Ti doping was prepared by unbalanced magnetron sputtering. The effect of target sputtering power on the structure and properties of the composite film was studied. The microstructure, morphology, mechanical and tribological properties of the composite film were systematically studied and analyzed.

2. Experimental details

La-Ti/WS2 composite films were deposited by JGP045CA magnetron sputtering system produced by Shenyang Scientific Instrument Company. Because the influence of Ti and La co-doping on the properties of WS2 films is unknown, in order to study the influence of Ti and La co-doping on the properties of WS2 films, WS2 with purity of 99.99% and La-Ti alloy target with atomic ratio of 1:1 were used as sputtering targets (target diameter of 50.8 mm and thickness of 3 mm). Stainless steel and monocrystalline silicon were used as experimental matrix materials. Monocrystalline silicon was used to test the micro morphology and mechanical properties of composite films. Stainless steel was used to test the friction and wear properties of composite films. Before the test, first the substrate was polished and washed in anhydrous ethanol and acetone for 15 min, then dry it with a blower and put it into the vacuum chamber quickly. In order to improve the adhesion of La-Ti/WS2 composite films, 20 min La-Ti transition layer was deposited on the substrate before La-Ti/WS2 composite films were deposited. Then, La-Ti/WS2 composite films were deposited at different target powers. The process parameters are shown in table 1.

The surface, cross-section and wear morphology of La-Ti/WS2 film were observed by SEM (Tescan Vega3, Czech), and the composition of the film was analyzed by EDS. The crystal structure of the film was analyzed by x-ray diffractometer (XRD) (D8 Advance, Bruker, Germany) with a Cu Kα radiation. The scanning speed was 2 degree min−1 and the scanning range was from 10–80°. The hardness and elastic modulus of La-Ti/WS2 composite film were analyzed by using the nano-indentation (iNano, Nanomechanics, USA). Berkovich indenter was selected to test the single point hardness on the monocrystalline silicon. In order to avoid the test error, five different positions were tested, the average value of the test results was taken, the test load was 50 mN, and the maximum indentation depth was set to be no more than 1/10 of the film thickness.

The friction and wear properties of La-Ti/WS2 composite film were tested on friction and wear testing machine in the atmospheric environment. The test conditions were as follows: GCr15 steel ball with diameter of 6 mm was selected for the grinding parts, the load was 1n, the grinding time was 8 min, the friction radius was 2 mm, the rotating speed was 336r/min, the friction mode was circular sliding friction under dry friction. The profile of wear tracks of La-Ti/WS2 composite films were measured by white light interference three-dimensional profilometer, the wear area was obtained by integrating the profile, then the wear volume was obtained by multiplying the total length of wear mark, and the wear rate (W) was calculated according to the formula:

\[ W = \frac{V}{F \cdot L} \]

In the formula, W is the wear rate (mm³ · N⁻¹ · m⁻¹), V is the wear volume (mm³), F is the applied normal load (N), and L is the total friction stroke (m). The average value of the wear rate of three friction tests was calculated to reduce the error, and the wear rate was used as a measure of the wear performance of La-Ti/WS2 composite films.
Table 1. Process parameters of WS$_2$ films deposition by magnetron sputtering.

| Background vacuum/Pa | Deposition pressure/Pa | Argon flow rate/Scm | Deposition temperature/°C | WS$_2$ Target power/W | La-Ti Target power/W | Deposition time of transition layer/min | Deposition time of composite film/min |
|----------------------|------------------------|----------------------|---------------------------|-----------------------|----------------------|----------------------------------------|--------------------------------------|
| $5 \times 10^{-3}$    | 1.2                    | 40                   | 300                       | 200                   | 10–50                | 20                                     | 120                                  |
3. Results and discussion

3.1. The characterization of the composite films

As demonstrated in figure 1, the surface morphology of the composite films were characterized by SEM. From figure 1(a), it can be seen that the surface of the composite film has a vermicular structure. It was suggested that the vermicular morphology mainly reflected the dominant orientation and curling effect of the microcrystals of WS$_2$ film, this is consistent with the results of reference [13]. From the cross-section morphology in figure 2(a), it can be clearly observed that La-Ti transition layer, it was a loose columnar crystal structure with many cracks and holes above the transition layer, the thickness of composite film was 7.4 μm. At this time, the growth rate of composite film was faster, as it was found in similar composite films [14]. However, with the increase of sputtering power of La-Ti target, it can be seen from figures 1(b)–(e) that the surface of composite films are granular island structure, the crack between particles were reduced, and the films are denser, it had special electronic layer structure and chemical activity with the rare earth element La, and it was easy to gather and enrich to the grain boundary [15]. It could form stable compounds with the impurity elements such as hydrogen and oxygen, which could inhibit the growth and coarsening of grain and increase the nucleation rate. It also had the purification effect on the grain boundary impurities, which promoted the ordered growth of crystal body,
Figure 3. XRD curves of the composite films deposited at different La-Ti target power.

Figure 3 showed the XRD curves of composite films with different target powers. At 10 W power, there are (002), (100), (110) and (200) diffraction peaks on the XRD curves, indicating that WS2 has a polycrystalline structure in the films. With the increase of La Ti power, the XRD curves of composite films show a broadened diffraction peak between 10° and 15°. With the increase of La-Ti power, the peak width increases, and the peak top were shifted toward lower values. No obvious broadening diffraction peak package were found between 10° and 40°. The results show that the highest position of the (100) diffraction peak package were moved toward the right with the increase of La-Ti power, while the (110) and (200) diffraction peaks disappear, indicating that the crystalline state of the composite film has changed, which made it tend to the amorphous state. Compared with the XRD curves of different La-Ti power composite films, it can be seen that the doping of La-Ti results in the dominant orientation of the films. The (002) plane of WS2 in the film was arranged parallel to the substrate, and the diffraction peak of WS2 (002) broadens with the increase of La-Ti power, which shows that the particle size of the films decreases continuously. WS2 molecules were blocked by La-Ti doping in a long-range ordered arrangement, which promoted microcrystallization of the film. Obviously, due to the addition of rare earth La, the growth of (002) crystal plane perpendicular to the substrate was inhibited, the growth of (002) slip plane parallel to the substrate surface was promoted, which made the composite film more conducive to lubrication. Due to the improvement of the compactness, the oxidation resistance and bearing capacity were enhanced, and the friction coefficient were reduced and wear resistance were improved.

EDS results of the composite films deposited at different La-Ti target power were shown in figure 4. It can be seen that the S/W atomic ratio of the composite film was the highest at 10 W, reached 1.94 (See table 2), and the content of Ti and La was the lowest. The atomic ratio of S/W was the lowest at 20 W, which was 1.4. With the increase of the target power, the S/W atom ratio in the film increased, but the S/W atom ratio was less than 2, and the content of Ti and La increased. It was believed that the loss of S element in the film may be caused by the combination of S element and residual oxygen or water vapor in the vacuum chamber, which is easily extracted by the air extraction system, resulting in the decrease of S element in the film composition. The inhibition of sputtering deposition of S-element and the purification of S-element are related to the addition of La. With the increase of sputtering power, the S/W atom ratio in the film also increased, this is because the sputtering power increased, the target sputtering particles were collided more in the process of motion, the anti-sputtering effect on the substrate film were decreased, and the loss of S element in the film was reduced.
3.2. Mechanical properties

The hardness and elastic modulus of the composite film under different target power were illustrated in figure 5. As seen in figure 5, the hardness and elastic modulus of the composite film increased gradually with the increase of target power [24]. The composite film deposited at target power of 50 W exhibited the highest hardness and elastic modulus, which shows that La-Ti doping has obvious effect on improving the hardness of the film, the rare earth element La played an important role in removing the impurities in the grain boundary, hindering the dislocation sliding deformation in the grain, making up the defects between the crystals, reducing the pores and

![Figure 4. EDS results of the composite films deposited at different La-Ti target power: (a) 10 W, (b) 20 W, (c) 30 W, (d) 40 W, (e) 50 W.]

Table 2. Composition of WS₂ composite films samples with different target power.

| Target power /W | Ti mass fraction/% | La mass fraction/% | S atomic fraction/% | W atomic fraction/% | S/W atomic ratio |
|-----------------|--------------------|-------------------|--------------------|-------------------|-----------------|
| 10              | 1.56               | 5.95              | 61.83              | 31.79             | 1.94            |
| 20              | 2.23               | 10.5              | 51.41              | 36.81             | 1.40            |
| 30              | 3.61               | 8.88              | 51.48              | 35.50             | 1.45            |
| 40              | 2.82               | 12.46             | 51.21              | 34.72             | 1.47            |
| 50              | 3.61               | 9.91              | 53.57              | 33.17             | 1.61            |
strengthening the grain boundary, increasing the hardness and strength of the film, and enhancing the carrying capacity of the film. Compared with 20 W power, the hardness and elastic modulus of the composite film decreased at 30 W power, and the mass percentage of Ti in the composite film increased at this power, while the mass percentage of La was decreased. The decrease of La content of rare earth caused the decrease of coupling effect, the increase of columnar crystal and the poor compactness of the composite film, which was also confirmed by the micro morphology of the film. The increase of hardness caused by doping elements was generally considered to be the result of the increase of film compactness, dispersion strengthening of metal elements and lattice deformation caused by metal doping [10]. Therefore, the composite films with different hardness and elastic modulus can be obtained by controlling the content of Ti and La in the films.

3.3. Tribological properties

Figure 6 exhibited the friction coefficient curve under different target power. It can be seen from figure 6 that the friction and wear performance of the composite film were significantly impacted by the target power. The film prepared by La-Ti target power at 0 W is pure WS2 film, the friction coefficient of pure WS2 film is stable and small. The friction coefficient of the composite films decreased first and then increased with the increase of the target power. The friction coefficient stabilized in target power of 10W–30W. Then it demonstrated a lowest friction coefficient with a minimum value of 0.071 when the target power of La-Ti reached 20 W, while the friction coefficient unstabled at 40 W and 50 W, and increased rapidly at 50 W, lost the lubricating effect. The friction coefficient was larger while the target power is 10 W, which implied that the composite film grew from the (002) crystal surface orientation. This kind of film structure expressed loose columnar growth structure, with
poor bearing capacity. In the process of friction, it was easy to oxidize with water and oxygen in the atmosphere, which affected the friction performance of the composite film, resulting in a large friction coefficient \[14\]. When the target power reached 40 W and 50 W, the content of La-Ti in the composite film was large, the composite film might form a multilayer structure with the largest hardness and elastic modulus. The composite film structure had a tendency of amorphization and showed a large brittleness, which was not conducive to the formation of high-quality transfer film, and the friction coefficient increased rapidly and decreased its lubricating effect. The composite film possessed higher hardness and the structure was compact at the target power of 20 W. La-Ti was a highly uniform solid solution in WS\(_2\), which was conducive to the formation of high-quality transfer film, showed a low and stable friction coefficient \[25\].

The wear rate under different target powers were displayed in figure 7. The wear rate displayed a similar discipline with friction coefficient. The as-prepared film deposited at target power of 10 W exhibited the highest wear rate of \(7.01 \times 10^{-6} \text{mm}^3 \text{N}^{-1} \text{m}^{-1}\) while it possessed a relatively low content of Ti and La, this phenomenon suggested the poor adhesive strength between the film and substrate. With the increase of target power, the wear rate of the composite film increased gradually. The wear rate reached the lowest value of \(5.9 \times 10^{-7} \text{mm}^3 \text{N}^{-1} \text{m}^{-1}\) at the target power of 20 W. The wear rate of films increased to \(1.56 \times 10^{-6} \text{mm}^3 / \text{(Nm)}\) and \(1.41 \times 10^{-6} \text{mm}^3 / \text{(Nm)}\) as the target power of La-Ti increased to 40 W and 50 W. However, when the power is 20W-50W, the wear rate is lower than that of pure WS\(_2\) film.

The SEM images of wear tracks were presented in figure 8. The wear surface of pure WS\(_2\) film has obvious furrow, it has a wide wear mark and wear resistance is poor. With the increase of power, the wear tracks of the composite film appeared brittle peeling, showed peeling wear characteristics. In the process of friction, the external force would induce microcracks at the wear tracks. When the cracks extend to the critical length, the film would peel off from the substrate. In combination with the wear rate in figure 7, the wear rate was higher at target power of 40 W and 50 W, the wear tracks was wider, and the content of La and Ti was larger, which indicated that there was internal stress between the transfer film formed on the dual surface and the dual material during the friction process, and the internal stress increased with the increase of the transfer film thickness, and the large internal stress caused the local peeling of the film surface, result in serious adhesive wear. At this time, the wear is severe heavy, poor wear resistance \[5\]. The composite film deposited at target power of 20 W owned the lowest wear rate and the narrowest wear tracks. It was clear that the structure of the composite film were changed due to the appropriate content of La and Ti doping, enhanced the adhesive strength between the composite film and the substrate, and the rare earth element La has a large atomic and ionic radius, which is easy to fill the defects on the grain surface and hinder the grain growth. The hardness and wear resistance of the film were improved due to the fine grain strengthening and dispersion strengthening effect of rare earth \[26\]. At the same time, the high-quality transfer film formed in the friction process effectively prevented the direct contact between the two pairs, and the peeling off and furrow phenomenon of the film were weakened. La doping played an important role in forming a WS\(_2\) transferred film \[11, 27\]. Obviously, the wear resistance life of the composite film was very sensitive to the content of La-Ti doping.
4. Conclusions

The novel La-Ti/WS$_2$ composite films were successfully fabricated at different La-Ti target power via unbalanced magnetron sputtering method. Their morphologies, structures and tribological performances were systematically characterized. The main results were summarized as follows:

a. By doping rare earth La and Ti element into WS$_2$ based films, the growth of coarse columnar crystal could be inhibited obviously, especially the fine grain strengthening effect of rare earth elements La reduced the deposition rate, decreased the porosity and defects, and increased the structural compactness.

b. For as-prepared La-Ti/WS$_2$ composite films, the atom ratio of S/W firstly decreased from 1.94 to 1.40 as the target power of La-Ti increased from 10 to 20 W, and then it gradually increased to 1.61 as the target power of La-Ti continued to increase. The content of Ti increased with the simultaneous increase of target power while the content of La exhibited a different discipline. The hardness and compactness of the composite film were improved, and the thickness was obviously reduced.

c. Compared with pure WS$_2$ film, La-Ti/WS$_2$ composite films deposited at La-Ti target power of 20 W could possess superior mechanical property and tribological performances. The results show that the friction and wear properties of the composite films were significantly improved by appropriate La-Ti doping amount.

d. The results show that the properties of WS$_2$ films could be improved by La and Ti co-doping, but in the later study, different La and Ti ratios should be further considered, and better composite films may be obtained.

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