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ARTICLE

Effect of Sputtering Power on High Temperature Tribological Behavior of La-Ti/WS_2 Composite Films

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Abstract: Under high temperature conditions, the crystal WS_2 is easily oxidized to WO_3, which has a great impact on the tribological properties of WS_2 solid lubrication films. In order to improve the tribological properties of WS_2 solid lubricating films at high temperatures, the co-doped La-Ti/WS_2 composite films were prepared by unbalanced magnetron sputtering. The effects of target power on the structure and tribological properties of La-Ti/WS_2 composite films were studied. The micromorphology, composition, mechanical properties and microstructure of the films were analyzed by scanning electron microscopy (SEM), X-ray diffraction (XRD), nano indentation and X-ray photoelectron spectroscopy (XPS). The high temperature tribological properties of composite films were researched by high temperature friction tester. The results show that La-Ti/WS_2 composite films show excellent tribological properties when the target power is 20 W at high temperature. At this time, the H/E value of the composite film is the largest, the friction coefficient is the smallest with the average value of 0.012, and the wear rate is the lowest, which is 1.56×10^{-8} $mm^3/N\cdot m$. This is mainly due to the production of rare earth oxides at the friction interface at high temperatures, which change the friction and wear mechanism of La-Ti/WS_2 composite film, so that WS_2 still has excellent tribological properties when it is damaged at high temperature, further expanding the scope of engineering application of WS_2 composite films.

Key words: rare earth; high temperature; friction; WS_2 composite films

With the rapid development of aerospace technology, bearings, as important rotating parts in aircrafts, often work under extreme conditions such as high/low temperature or high load. In order to solve the problem of bearing lubrication under these extreme conditions, solid lubrication technology has come into being. Transition metal sulfides (WS_2 or MoS_2) have hexagonal layered crystal structure and weak van der Waals force between layers, because of low interlaminar shear force, so it is easy to slide when friction behavior occurs, and has excellent lubrication performance, and thus it has been widely used in the field of aerospace [1–3]. Compared with MoS_2, WS_2 has received special attention because of its better high temperature resistance. However, WS_2 film has poor bearing capacity. In the atmospheric environment, the unsaturated bond on the edge is easy to interact with H_2O to produce WO_3 and H_2S, which greatly restrict the application of transition metal sulfide [4–6].

In order to overcome the shortcomings of WS_2 solid lubrication film and to improve the bearing capacity of WS_2 solid lubrication film, scholars have done a lot of research on the preparation of composite films doped with different elements. These works show that the microstructure and mechanical properties of WS_2 films can be improved by doping metal and non-metallic elements into WS_2 films [7–9]. Moreover, because the doped elements can preferentially interact with O_2, the structure of WS_2 film is protected from being destructed, the tribological properties of WS_2 film have been greatly improved[10–15], and its corrosion resistance in humid environment has been greatly promoted[16–17]. However, facing the complexity of WS_2 thin film engineering applica-

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tion environment, the existing research shows some limitations.

Rare earth elements have special electronic structure and super chemical activity. It is found that doping an appropriate amount of rare earth elements or rare earth compounds in the hard film can strengthen the fine crystal and significantly improve the microstructure of the film[18–21]. Further, the research of Zheng et al[22] shows that adding an appropriate amount of lanthanum oxide into the composites is conducive to the formation of dense friction film and transfer film, which is conducive to improving the tribological properties of the composite materials. Ye et al[23] added a small amount of rare earth compound LaF_3 to MoS_2 solid lubricating films and found that it is helpful to improve the wear resistance of films. The combination doping of rare earth element La and metal element Ti in WS_2 film has great advantages. The author doped rare earth element La and metal element Ti in WS_2 solid lubrication film at the early stage, and confirmed that the tribological properties of lubrication films can be improved[24]. As a solid lubricating material, WS_2 not only has excellent lubricating properties like MoS_2, but also its tribological properties at high temperature have attracted extensive attention [25]. However, most of the existing studies are based on room temperature environment, and there are few reports on the tribological properties of rare earth doped composite films at high temperature [6].

Previous studies have shown that the process parameters have a great influence on the mechanical and tribological properties of both hard and lubricating films prepared by magnetron sputtering[26–28]. The effects of process parameters on the tribological properties of rare earth doped WS_2 films at room temperature have been studied[29]. WS_2 has the characteristics of good high temperature resistance. Studying the influence of process parameters on the structure and tribological properties of rare earth doped WS_2 composite films in high temperature environment can further broaden the application range of WS_2 composite films and make them better serve in extreme working conditions. Undoubtedly, it has important engineering value.

In this study, La-Ti/WS_2 composite films with an appropriate doping amount of La and Ti were deposited by unbalanced magnetron sputtering. The effects of target sputtering power on the structure and high temperature tribological properties of the composite films in atmospheric environment were studied. The microstructure, morphology, mechanical and tribological properties of the composite films were analyzed, and the reasons for the good tribological properties of rare earth doped WS_2 composite films at high temperature were discussed.

1 Experiment

1.1 Preparation of composite films

In this study, La-Ti/WS_2 composite films were deposited onto stainless steel and monocrystalline silicon by JGP045CA unbalanced magnetron sputtering system from Shenyang Scientific Instrument Company. WS_2 target (purity 99.99%) was installed in RF target, La-Ti alloy target (purity 99.99%) with atomic ratio of 1:1 and Ti target (purity 99.99%) were installed in two DC targets, and the target size was Φ50.8 mm×3 mm. Mono-crystalline silicon and stainless steel were used to test the mechanical and tribological properties of the films, respectively. In order to improve the adhesion between the film and the substrate, the substrate was pretreated and deposited with an interlayer. In the deposition process, the worktable rotates between three targets at the speed of 20 r/min. For La-Ti/WS_2 composite film, WS_2 target and metal targets were used at the same time. Background vacuum was 5×10^{-4} Pa. Deposition pressure was 1.2 Pa. The deposition temperature was 300 ° C. The parameters for deposition process of the La-Ti/WS_2 composite films were: WS_2 target power 200 W and La-Ti target power 0–50 W.

1.2 Characterization of film structures and properties

The surface and cross-section morphology of La-Ti/WS_2 composite films were investigated by scanning electron microscopy (SEM, Sigma300, ZEISS), and the composition of the films was determined by energy dispersive spectrometer (EDS). The crystal structure of the films was analyzed by X-ray diffractometer (XRD, SmartLab X, Rigaku, Japan) with a Cu Kα radiation and the scanning range was 10°–90°.

The hardness and elastic modulus of La-Ti/WS_2 composite films were analyzed by the nano-indentation (iNano, Nanomechanics, USA) with a diamond Berkovich indenter tip on the monocrystalline silicon. The maximum indentation depth was set to be no more than 1/10 of the film thickness. The indentations of each composite films were measured five times and the average value was calculated to reduce the test error.

The chemical composition of wear marks and transfer films was tested by X-ray photoelectron spectroscopy (XPS, Escalab 250xi, Thermofischer). The vacuum degree of the analysis chamber was 4×10^{-7} Pa, the excitation source adopted Al Kα ray (HV=1486.6 eV), the working voltage was 14.6 kV, the filament current was 13.5 mA, and the signal accumulation of 20 cycles was carried out. The passing energy was 20 eV with a step of 0.1 eV, and the charge correction was carried out with C 1s=284.8 eV as the binding energy standard. Before the measurement, the surface of each sample was cleaned for 5 min by Ar+ion beam with an energy of 3 keV to remove surface contaminants.

The tribological performance of La-Ti/WS_2 composite films under atmosphere environment was tested on tribometer (HT-1000, Lanzhou Zhongke Kaihua Technology Development Co., Ltd). Friction tests were performed at 500 °C under a constant load of 1 N, the circular sliding friction under dry friction was applied at 6 Hz, and the rotation radius was 2 mm. The counterpart was a GCr15steel ball with a diameter of 6 mm, a hardness of 6 GPa and a surface roughness of 0.1 μm. The grinding time was 8 min. After friction test, the profile of wear marks of La-Ti/WS_2 composite films was measured by white light interference three-dimensional profilometer, and the wear volume of each wear mark was calculated for three times to obtain the average value. The wear rate (W) was calculated from their wear volumes using the following formula:

$$W = \frac{V}{FL} \tag{1}$$

where V is the wear volume (mm^3), F is the normal load (N), and L is the total friction stroke (m). The wear rate was used as a parameter to measure wear resistance.

2 Results and Discussion

2.1 Composition and structure

The SEM images of surface morphology and cross-section morphology of the composite films are shown in Fig. 1 and Fig.2, respectively. From Fig.1a and Fig.2a, it can be seen that the surface of the pure WS_2 film has willow leaf structure, the cross-section morphology is loose sheet structure, and there are many gaps in the film. The research shows that the willow leaf morphology mainly reflects the dominant orientation and curling effect of WS_2 film microcrystals[30]. However, it can be observed from Fig. 1b−1e that with the doping of La-Ti, the microstructure of pure WS_2 film can be improved, especially with the increase in target sputtering power, the willow leaf structure on the surface of the composite film becomes granular island structure, the gap between particles decreases, and the film becomes denser. It can also be seen that its cross-section morphology presents a columnar structure and the thickness of film is significantly reduced. In particular, when the sputtering power is 30 W, the film thickness is the smallest and the cross-section morphology is dense. This is because the rare earth element La has special electronic layer structure and extremely active chemical properties, which is conducive to grain boundary segregation and inhibits grain growth. La is easy to form stable compounds with O, S and other impurity elements, the damage of impurity elements to the film structure is reduced, and the porosity of the films is diminished[31]. The doped Ti element will preferentially occupy the active site of WS_2 edge, playing a role of passivating the active site, and the preferential growth trend of WS_2 (100) edge is effectively block, and thus the grain size is reduced and the film thickness is decreased[4]. Due to the improvement of film compactness, the oxidation resistance and bearing capacity are enhanced, and its friction coefficient and wear resistance are elevated[32]. The research shows that the energy of the incident ions is affected by the target power. With the further increase in the target power, on the one hand, the composition and structure of the film will change due to different sputtering rates of different elements; on the other hand, the concentration of particles splashed from the target in the vacuum chamber increases, and the number of collisions received when moving toward the workpiece surface increases, resulting in small kinetic energy when reaching the workpiece surface, and the weakening of the re-sputtering effect. The compactness of film is reduced, and its thickness is increased.

XRD patterns of composite films with different target powers are shown in Fig.3. When the La-Ti doping power is 0 and 10 W, four diffraction peaks of WS_2, i. e. (002), (100), (110) and (200), appear at 14°, 33.8°, 60° and 71°, respectively, indicating that WS_2 is polycrystalline in the film[33]. In addition, no XRD signals of Ti and La are observed, which may be due to their low content. With the increase in La-Ti power, the XRD pattern of the composite films shows a broadened diffraction peak between 10° and 15°, the width of the peak is increased, and the peak moves to low θ direction. At 38.8°, the broadened diffraction peak of Ti (002) is observed, indicating that Ti exists in polycrystalline form. By comparison, it is found that the diffraction peaks of (100), (110) and (200) in this region gradually disappear, indicating that the crystalline state of the composite film is changed, making it tend to be amorphous state.

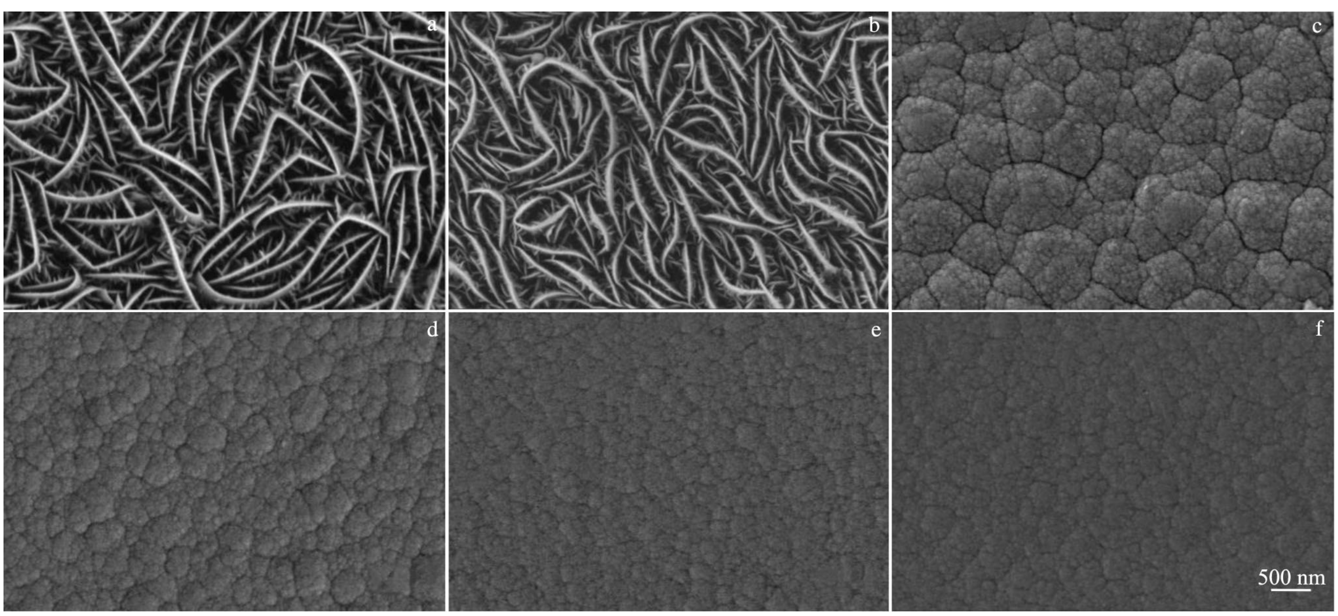

Fig.1 Surface morphologies of La-Ti/WS_2 composite films with different target powers: (a) 0 W, (b) 10 W, (c) 20 W, (d) 30 W, (e) 40 W, and (f) 50 W

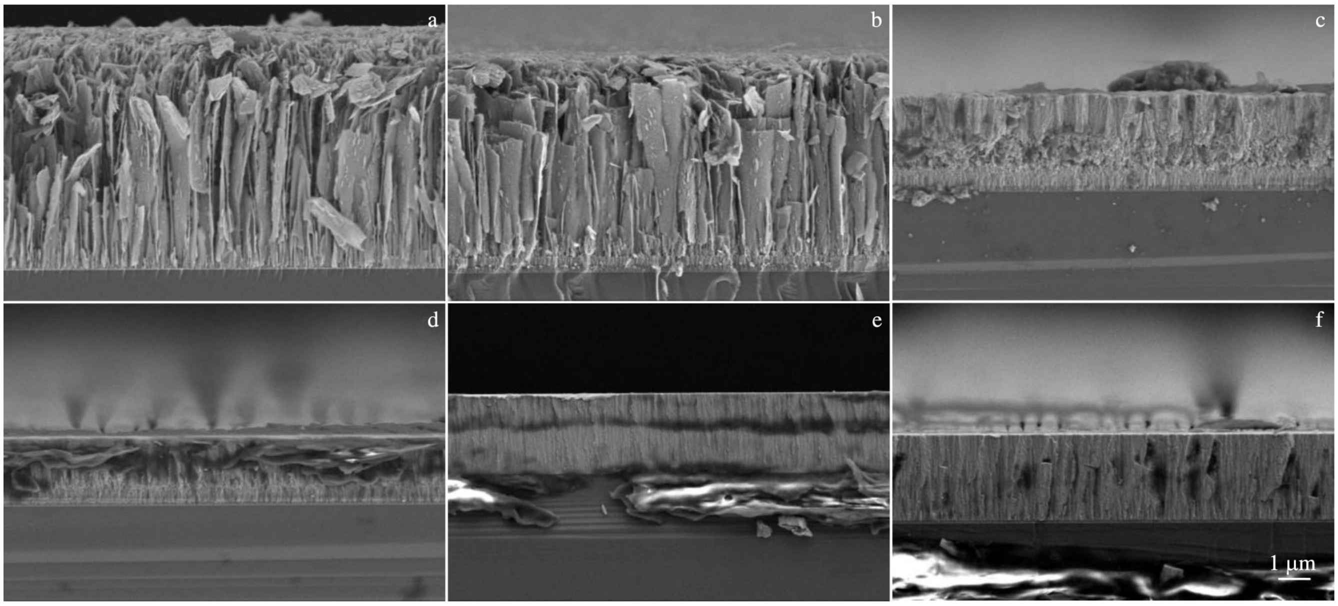

Fig.2 Cross-sectional morphologies of La-Ti/WS_2 composite films with different target powers: (a) 0 W, (b) 10 W, (c) 20 W, (d) 30 W, (e) 40 W, and (f) 50 W

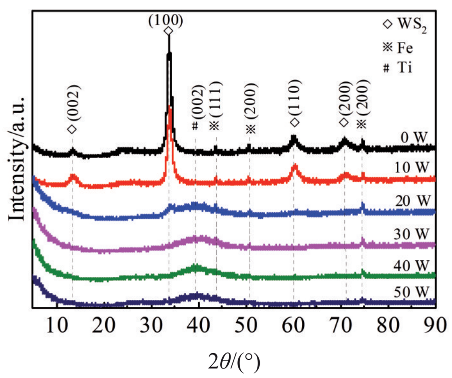

Fig.3 XRD patterns of La-Ti/WS_2 composite films with different target powers

Comparing the XRD curves of different La-Ti power composite films, it can be seen that the films have a dominant orientation after La-Ti doping. When the target power is 0 and 10 W, the (100) crystal orientation peak is the strongest, indicating that there is a preferred orientation in the growth process of the films. When the target power is further increased, the (100) diffraction peak gradually weakens, and WS_2 in the films are mainly arranged in the way that the (002) base plane is parallel to the substrate. With the increase in La-Ti power, the diffraction peak of WS_2 (002) is continuously widened, which reflects that the particle size of the film is continuously reduced. Therefore, due to the doped La-Ti, the long-range ordered arrangement of WS_2 molecules is effectively blocked, resulting in microcrystallization of the film, which is conducive to improving the wear resistance and oxidation resistance of the film[34].

The composition of La-Ti/WS_2 composite films is analyzed by EDS, as shown in Table 1. With the increase in target power, the content of Ti ranges from 0at% to 5.82at%. The appearance of O is due to the residual gas in the vacuum chamber. With the change of La-Ti power, the S/W ratio decreases first and then increases. At 20 W, the S/W ratio is the smallest. The S/W ratio of the films is less than the ideal stoichiometric ratio of WS_2 due to the doped elements. The analysis shows that the reason for the decrease in S element in the film is that, on the one hand, S element is preferentially combined with residual oxygen or water vapor in the vacuum chamber and then extracted by the pumping system during the film deposition, on the other hand, the sputtering deposition of S element is inhibited due to the addition of La element, resulting in the removal of S purification. However, the reason for the increase in S/W atomic ratio with the increase in power is speculated to be the increase in the number of collisions during the movement of target sputtering particles, the anti-sputtering effect on the substrate film is weakened, and the loss of S element in the film is decreased[35]. The content of hard-phase W in the film will increase due to the decreased S element, which will affect the tribological properties of the film.

Table 1 EDS results of La-Ti/WS_2 composite films with different target powers

La-Ti power/W	0	10	20	30	40	50
W content/at%	30.42	31.29	35.01	34.8	33.22	32.17
S content/at%	62.48	57.83	50.41	50.58	50.71	52.56
La content/at%	-	3.22	6.29	5.77	7.4	5.65
Ti content/at%	-	2.16	3.49	4.25	4.57	5.82
O content/at%	7.1	5.5	4.8	4.6	4.1	3.8
S/W ratio	2.05	1.84	1.44	1.45	1.52	1.63

2.2 Hardness and elastic modulus of composite films

Fig. 4 exhibits the hardness and elastic modulus of composite films with different La-Ti target powers. It can be seen that the hardness and elastic modulus of pure WS_2 films are the lowest, which are 0.263 and 25.48 GPa, respectively. The micromorphology analysis of the film shows that doping La-Ti can improve the compactness of the film. Therefore, with the increase in target power, the hardness and elastic modulus of the composite film are increased gradually. When the target power is 30 W, the composite film has the highest hardness and elastic modulus, which are 7.91 and 95.26 GPa, respectively. On the contrary, the target power is increased to 40 and 50 W, and the hardness of the composite films decreases slightly. It is speculated that this is due to the increase in plasma density in the vacuum chamber, more particles are sputtered on the substrate surface, and the particle bombardment and secondary sputtering on the workpiece surface are weak, which will reduce the loss of S element in the film, increase the S/W ratio in the film, and reduce the content of hard W element in the film, and the hardness and structure density of the films become worse. It can be seen that doping La-Ti has an obvious effect on improving the hardness of the film. The results show that the elastic modulus of the film doped with excessive Ti and other metal elements can be improved, but the brittleness of the film will be increased. In addition, the ceramic phase formed during friction will play the role of abrasive particles, which is not conducive to the formation of transfer film and increased wear[35–37]. Further comparing the H/E value of the film under different powers (Table 2), it can be found that the H/E value of the film is the largest at 20 W which reflects the best wear resistance of the film[38]. However, the H/E value decreases with the increase in power, which proves that La doping can improve the compactness of the film, but excessive doping of La is also harmful.

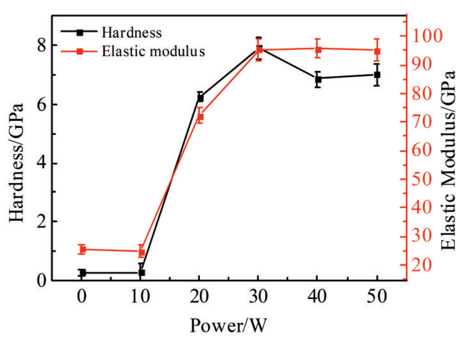

Fig.4 Hardness and elastic modulus of La-Ti/WS_2 composite films with different target powers

Table 2 H/E value of La-Ti/WS_2 composite films with different target powers

La-Ti power/W	0	10	20	30	40	50
H/E	0.01	0.011	0.086	0.083	0.072	0.074

2.3 Tribological properties of composite films at high temperature

The friction coefficient curves of composite films with different La-Ti target powers are illustrated in Fig. 5. The friction coefficient of pure WS_2 films is unstable, and it gradually increases during the friction process. When the La-Ti target power is 10 W, the friction coefficient enters the stable stage after a short running in. In contrast, when the target power of La-Ti is 20 W, the friction coefficient curve is the most stable, and the average friction coefficient is 0.016. When the power is above 30 W, the friction coefficient is unstable and tends to increase gradually. Further analysis of the wear rate of the composite film with different target powers (Fig.6) shows that the wear rate of the composite film is the smallest when the La-Ti target power is 20 W, which is 1.56×10^{-8} $mm^3/N\cdot m$, and the wear rate is the largest when the La-Ti target power is 0 W. This shows that the wear resistance of the composite film is the best when the target power of La-Ti is 20 W.

In order to study the wear mechanism of composite films at high temperature, the wear marks and spots of composite films are observed. The wear marks and wear spots of La-Ti/WS_2 composite film after high temperature friction test are presented in Fig.7 and Fig.8, respectively.

It can be seen from Fig. 7 and Fig. 8 that the wear mark

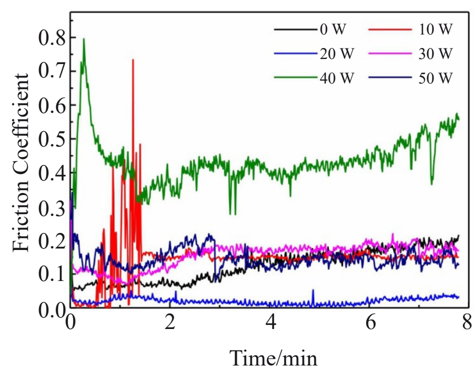

Fig.5 Friction coefficient curves of La-Ti/WS_2 composite films with different powers

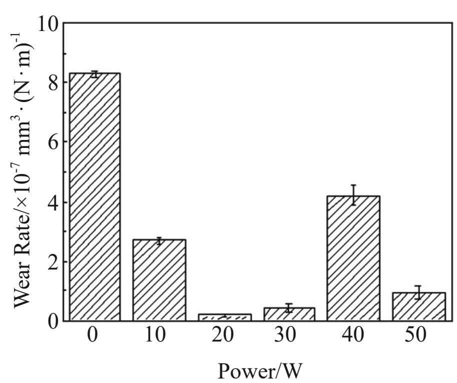

Fig.6 Wear rate of La-Ti/WS_2 composite films with different target powers

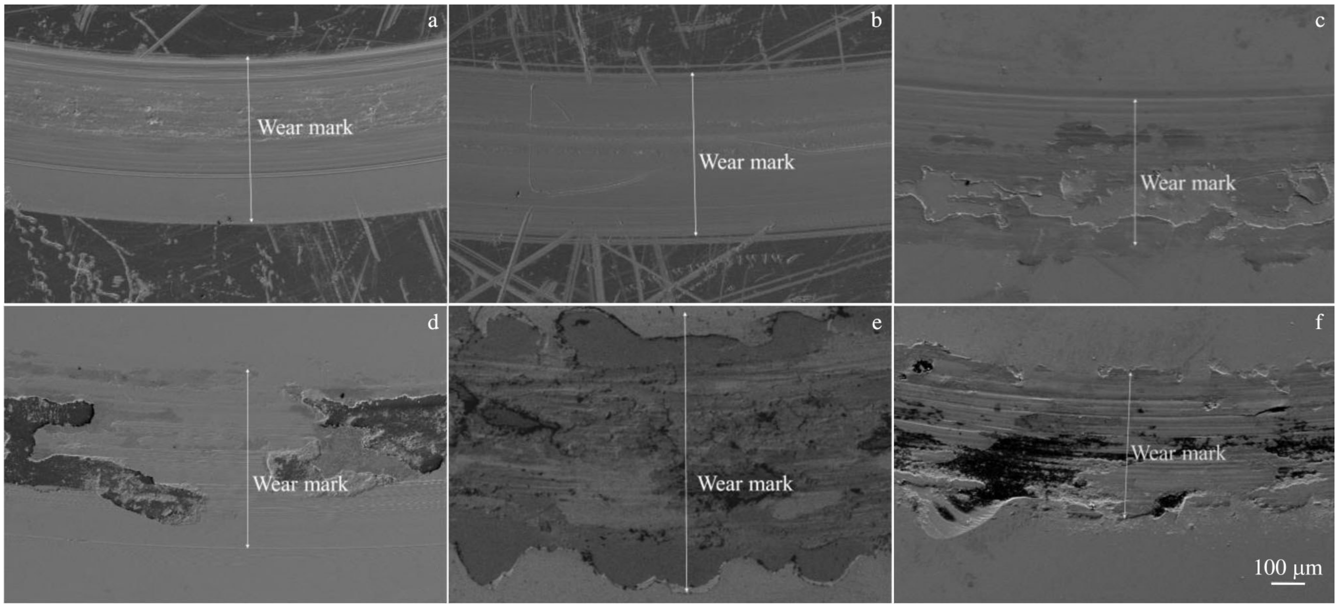

Fig.7 Wear marks of La-Ti/WS_2 composite films with different target powers: (a) 0 W, (b) 10 W, (c) 20 W, (d) 30 W, (e) 40 W, and (f) 50 W

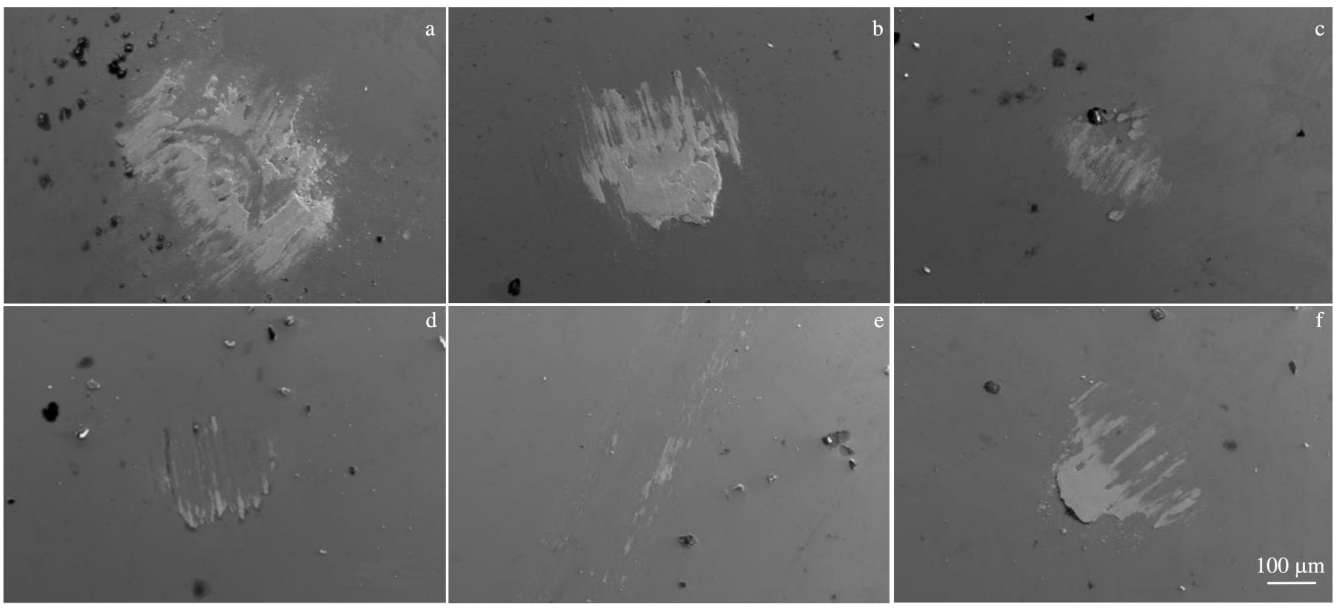

Fig.8 Wear spots of La-Ti/WS_2 composite films with different target powers: (a) 0 W, (b) 10 W, (c) 20 W, (d) 30 W, (e) 40 W, and (f) 50 W

surface of pure WS_2 film is rough with obvious furrows, and there are many pits inside. Although the transfer film is formed in the center of the corresponding wear spot, it is seriously worn, and its wear mechanism is mainly abrasive wear. There are a small amount of furrows and debris accumulation on the wear mark surface with La-Ti target power of 10 W, indicating that hard particles form during the friction process, resulting in abrasive wear, and its friction coefficient fluctuates continuously. A transfer film forms in the center of the corresponding wear spot, which reduces the wear of the film in the later stage. When the La-Ti target power is 20 W, the wear mark is relatively smooth, and there is no loose debris accumulation on both sides. A dense transfer film with good shape forms in the center of the corresponding wear spot, which indicates that the wear mechanism of the composite film has changed and the friction and wear has been greatly reduced. When the La-Ti target power is 30 and 50 W, the composite film peels off, the substrate is exposed, the transfer film is not formed in the corresponding wear spot center, and only a small amount of wear debris is scattered around. When the La-Ti target power is 40 W, the wear mark of the composite film is the widest, the film falls off, and no transfer film is formed in the center of the corresponding wear spot, indicating that the wear mechanism is mainly adhesive wear. The results show that with the increase in target power, the brittleness of the films increases due to the large content of La and Ti in the films, and its bearing capacity decreases. At the same time, there are a large internal stress on the surface of the films, which leads to defects and damage on the surface of the film during the friction process, resulting in serious adhesive wear, and adversely affecting the wear resistance of the film. It is the most obvious when the power is 40 W.

In order to deeply understand the differences of high temperature friction experiments of composite films with different La-Ti target powers, the composition of the transfer film was analyzed by EDS. The EDS analysis results of wear spots of La-Ti/WS_2 composite films after high temperature friction test are shown in Fig.9. As can be seen, both pure WS_2

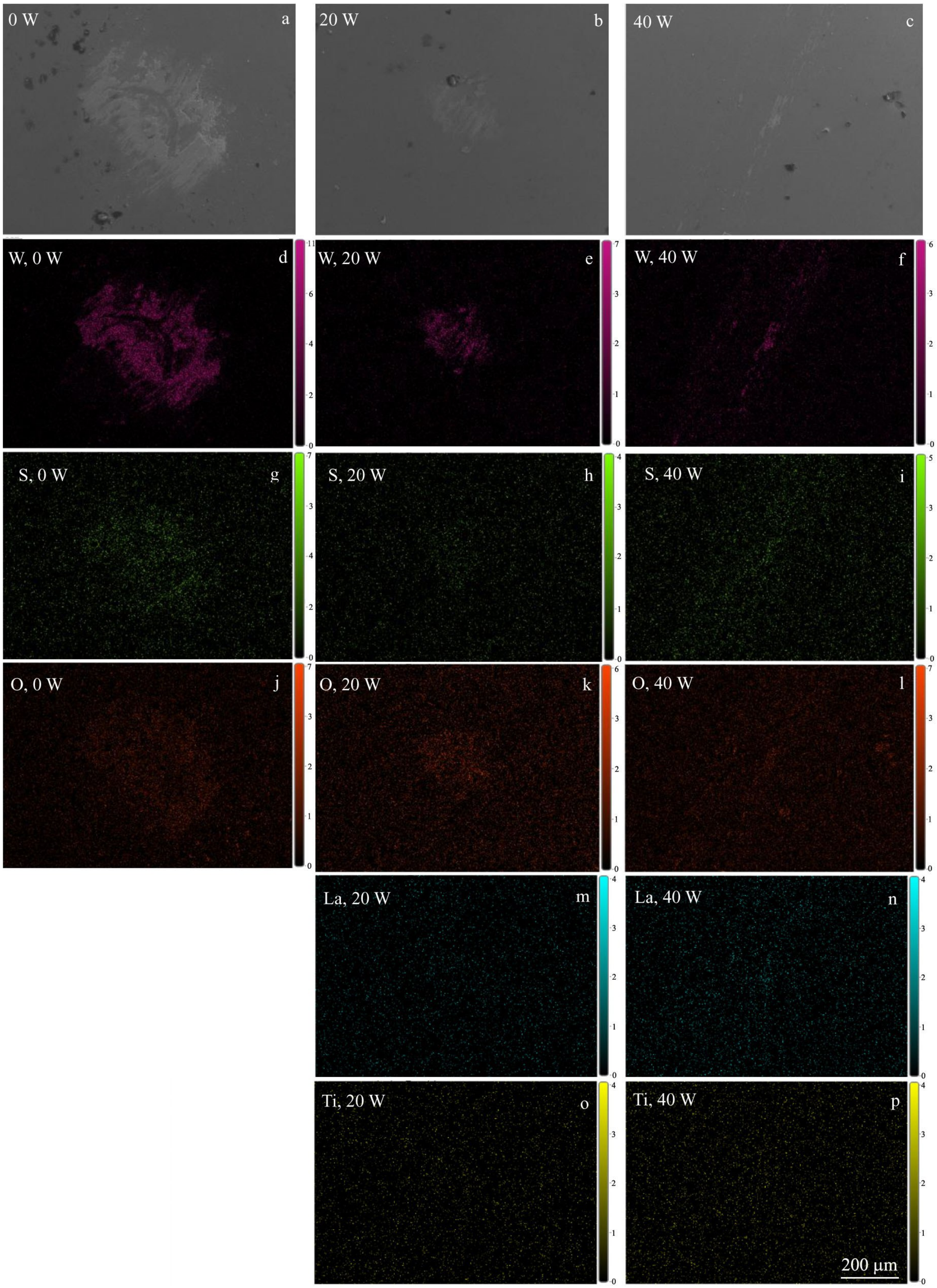

Fig.9 SEM images (a–c) and EDS mappings (d–p) of wear spots of La-Ti/WS_2 composite films at different target powers

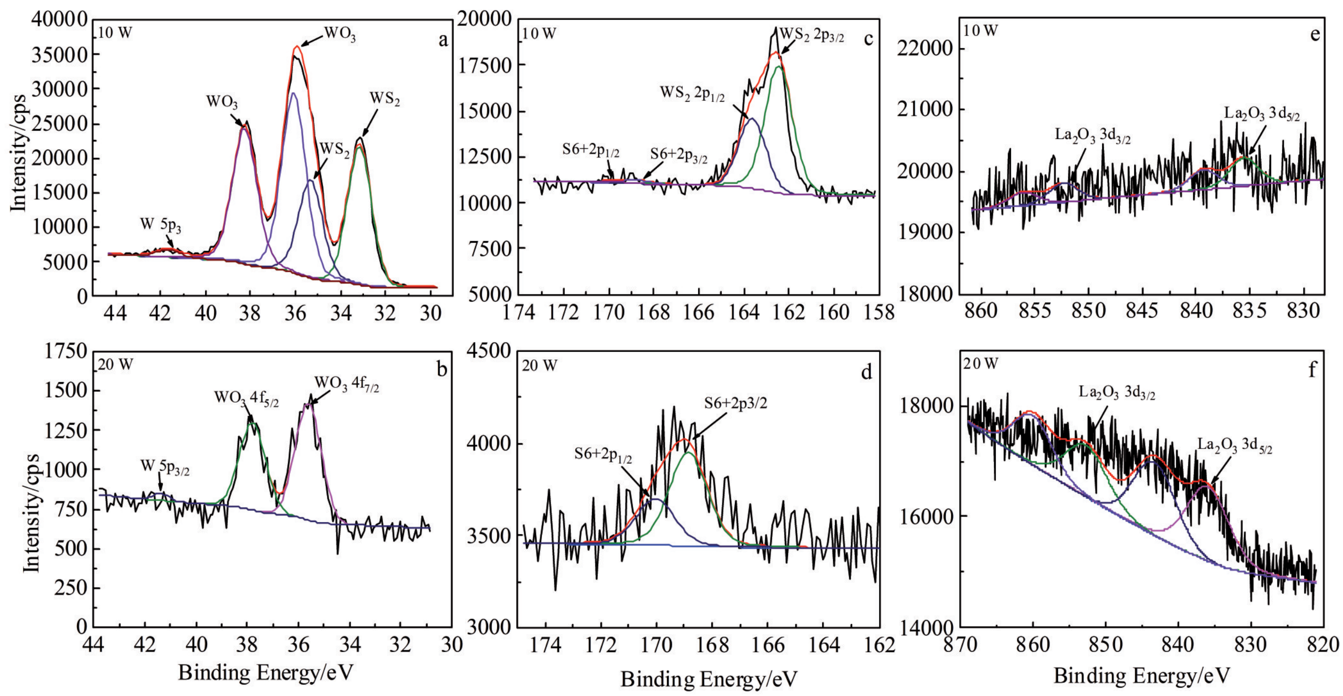

Fig.10 XPS spectra of La-Ti/WS_2 composite films with different elements of W (a, b), S (c, d), and La (e, f) at 10 W and 20 W

film and La-Ti/WS_2 composite films contain W and S elements at the center of wear spots, which indicates that WS_2 is transferred to the wear spot surface. Compared with the case at 20 W, the content of La in the transfer film is higher at 40 W. By observing the distribution of O element, it is found that the enrichment of O element occurs in the three transfer membranes, and the content of O element decreases with the increase in power. This is because the metal element plays the role of oxygen collector and preferentially reacts with H_2O and O_2, which can protect the structure of WS_2 from being damaged[13, 35].

In order to further confirm the composition of the transfer film, the wear spots of the composite films after the high-temperature friction test were analyzed by XPS. Since no obvious transfer elements of Ti and La are found in the grinding balls at 30, 40 and 50 W, 10 W is selected as a comparison. The XPS spectra of the wear spots of the composite films at 10 and 20 W are shown in Fig.10. With the change of power, the peak shape of all samples is changed, indicating that the chemical bond of elements in the film has changed significantly. Compared with 20 W, the spectral intensity of WS_2 at 10 W is significantly higher, but the spectral intensity of S6+2p is significantly reduced. And there is a weak La_2O_3 spectral peak at 20 W, while there is no obvious La_2O_3 spectral peak at 10 W. This also proves that the content of La element in the transfer membrane is less. From the peak splitting results, the transfer films mainly exist in the chemical state of WO_3, which is the result of obvious oxidation reaction of WS_2 at high temperature in the atmospheric environment, and will lead to the loss of lubrication performance of the composite films. However, with the increase in La-Ti power, on the one hand, a small amount of La and Ti doping can also improve the oxidation resistance of the transfer film, on the other hand, an appropriate amount of Magnéli phase La_2O_3 is generated at the friction interface, while La_2O_3 has a low shear force during the friction process, and the shear term and adhesion term in the friction process are reduced, which may change the wear mechanism of La-Ti/WS_2 composite films at high temperature, resulting in the decrease in friction coefficient, thus showing excellent tribological properties.

3 Conclusions

1) With the doping of La and Ti, the microstructure of WS_2 film is improved, and the density of La-Ti/WS_2 composite films is obviously enhanced. Its hardness and elastic modulus are gradually increased, and the H/E value first increases and then decreases.

2) Although WS_2 will be destroyed at high temperatures to form WO_3 which is not conducive to lubrication, La-Ti/WS_2 composite film has the largest H/E value at 20 W, and still shows the best friction coefficient and the lowest wear rate, which is due to the synergistic effect of rare earth oxide formed during the process of high temperature friction and WS_2 film.

3) An appropriate amount of rare earth doping is of great significance to solve the disadvantage of WS_2 composite film which is easy to fail under high temperature environment.

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溅射功率对La-Ti/WS_2复合薄膜高温摩擦学行为的影响

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摘　要：高温条件下WS_2易于氧化生成WO_3，导致WS_2固体润滑薄膜的摩擦学性能受到较大影响。为改善WS_2固体润滑薄膜在高温条件下的摩擦学性能，采用非平衡磁控溅射技术制备了共掺杂La-Ti/WS_2复合薄膜，研究了靶功率对磁控溅射La-Ti/WS_2复合薄膜结构和高温摩擦学性能的影响。利用扫描电镜（SEM）、X射线衍射仪（XRD）、纳米压痕仪和X射线光电子能谱仪（XPS）分析了薄膜微观形貌、成分、力学性能、微观结构。利用高温摩擦磨损试验机研究了复合薄膜的高温摩擦学性能。结果表明，高温环境下，靶功率为20 W时La-Ti/WS_2复合薄膜表现出优异的摩擦学性能。此时，复合薄膜*H*/*E*值最大，摩擦系数最小，平均为0.012，磨损率最低为1.56×10^{-8} mm^3/N·m，这主要归因于高温下摩擦界面产生的稀土氧化物，促使La-Ti/WS_2复合薄膜的摩擦磨损机制发生了改变，使得WS_2在高温受破坏的情况下仍然具有优异的摩擦学性能，从而进一步拓展了WS_2复合薄膜的工程应用范围。

关键词：稀土；高温；摩擦；WS_2薄膜

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