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ARTICLE

Friction and Wear Behavior of Self-lubricating La-Ti/WS₂ Films by Unbalanced Magnetron Sputtering

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Abstract: The solid lubrication films with high hardness and low friction coefficient should be developed to satisfy the severe working conditions of mechanical rotating parts of spacecraft. The pure WS₂ film, Ti doped WS₂ composite film (Ti/WS₂) and La-Ti doped WS₂ composite film (La-Ti/WS₂) were prepared by unbalanced magnetron sputtering. The micro morphology, composition, hardness and tribological performance of the films were examined. Results show that compared with pure WS₂ films and Ti/WS₂ composite films, the microstructure of La-Ti/WS₂ composite film becomes more compact. Meanwhile, the hardness and elastic modulus of La-Ti/WS₂ composite film also increase significantly. Furthermore, the friction coefficient of La-Ti/WS₂ composite film decreases, and the hardness/elastic modulus (*H/E*) ratio of La-Ti/WS₂ composite film increases, which suggests that the wear rate of La-Ti/WS₂ composite film is reduced. It is indicated that La doping contributes to the formation of stable transfer film on the friction contact surface, and thus improves the wear resistance and carrying capacity of La-Ti/WS₂ composite film.

Key words: La-Ti/WS₂ composite films; unbalanced magnetron sputtering; low friction; wear resistance

In order to meet the lubrication requirements of aerospace industry for severe rugged environment, solid lubrication technology has been widely used in the rotating parts of spacecraft^[1,2]. As a traditional solid self-lubricating material, transition metal sulfides (MoS₂, WS₂) have become one of the key research directions in the field of basic research and industrial application due to their low friction coefficient. However, some W and S atoms at the edge of WS₂ crystal structure can only form bonds with four S atoms and two W atoms, respectively, leading to the dangling bonds. These dangling bonds are very easy to react with oxygen and water in the air to generate WO₃ and H₂SO₄. WO₃ promotes the increase of friction coefficient, while strong acidic H₂SO₄ accelerates the corrosion, making WS₂ very easy to lose its lubrication performance in humid environment^[3]. Moreover, the high porosity and low hardness of WS₂ film make it unable to meet the requirements of high mechanical and tribological carrying capacity.

Some scholars have used the method of single metal doping

to modify WS₂ films, so as to improve the structure, mechanical and tribological properties of WS₂ films. For example, Deepthi^[4,5], Xu^[6,7] et al, prepared the WS₂/Cr, WS₂/Au, WS₂/Cu and WS₂/Ag composite films with different Cr, Au, Cu and Ag contents by magnetron sputtering, which significantly improve the porous columnar structure of WS₂ films, enhance the density of the films, and increase the tribological performance of WS₂ films. Although the method of doping metal in WS₂ film has achieved great success in improving the performance of composite film, the tribological performance of WS₂ film needs to be further improved in order to meet the increasing demand for spacecraft performance under severe conditions. Therefore, more and more researchers paid attention to two element doping method^[8-10].

Rare earth element has been widely used in the field of material surface modification due to its special electronic structure and high chemical activity^[9]. Through a small doping amount of rare earth, the microstructure and mechanical properties of the material were improved, and the wear

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resistance and antifriction performance of the film were also affected. However, there are few reports about the modification of WS₂ films by doping rare earth in magnetron sputtering process, especially the study on improving the performance of composite films by doping rare earth La and Ti in WS₂ films. In this study, pure WS₂ films, Ti/WS₂ and La-Ti/WS₂ composite films were prepared by unbalanced magnetron sputtering. The microstructure, morphology, mechanical and tribological performance of the three kinds of films were studied.

1 Experiment

Pure WS₂ films, Ti/WS₂ composite films and La-Ti/WS₂ composite films were prepared by JGP045CA unbalanced magnetron sputtering system from Shenyang Scientific Instrument Company. WS₂ target (purity of 99.99%) was installed in RF target, La-Ti alloy target (purity of 99.99%) with atomic ratio of 1:1 and Ti target (purity of 99.99%) were installed in two DC targets, and the target size was $\Phi 50.8$ mm \times 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 tribological performance of composite films. Before the test, the substrate was first polished and washed in anhydrous ethanol and acetone for 15 min, then dried with a blower and put into the vacuum chamber quickly. Background vacuum was 5×10^{-4} Pa. Pure WS₂ films, Ti/WS₂ composite films and La-Ti/WS₂ composite films were deposited under a deposition pressure of 1.2 Pa. In order to improve the adhesion, the Ti transition layer of 200 nm was pre-deposited before pure WS₂ film and Ti/WS₂ composite film, and the La-Ti alloy transition layer of 200 nm was pre-deposited before La-Ti/WS₂ composite film. Deposition time of transition layer and composite film was 20 and 120 min, respectively. The process parameters are shown in Table 1.

Table 1 Process parameters of film deposition by magnetron sputtering

| Argon flow rate/mL·min ⁻¹ | Deposition temperature/°C | WS ₂ target power/W | La-Ti/Ti target power/W |
|--------------------------------------|---------------------------|--------------------------------|-------------------------|
| 40 | 300 | 200 | 20 |

The surface, cross-section and wear morphology of three kinds of films were observed by SEM (Tescan Vega3, Czech), and the composition of the films were analyzed by EDS. The crystal structure of the films was analyzed by X-ray diffractometer (XRD, D8 Advance, Bruker, Germany) with a Cu K α radiation. The scanning speed was 2°/s and 2 θ scanning range was 10°~80°. The hardness and elastic modulus of three kinds of films were analyzed by 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 selected, and the average value of the test results was taken; test load was 50 mN, and the maximum indentation depth was set to be no more than 1/10 of the film thickness^[11].

The tribological performance of three kinds of films was tested on tribometer (HT-1000, Lanzhou Zhongke Kaihua Technology Development Co., Ltd) in the atmospheric environment. The test conditions were as follows: GCr15 steel ball (HRC ~60, roughness R_a ~0.10 μ m) with diameter of 6 mm was selected for the grinding parts, the load was 1 N, the grinding time was 8 min, the friction radius was 2 mm, the rotating speed was 336 r/min, and the friction mode was circular sliding friction under dry friction. The profile of wear tracks of three kinds of films was 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 Eq.(1):

$$W = \frac{V}{FL} \quad (1)$$

where 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 films.

2 Results and Discussion

2.1 Chemical composition and microstructure

As demonstrated in Fig.1, the surface morphologies of pure WS₂, Ti/WS₂ and La-Ti/WS₂ composite films are characterized by SEM. From Fig.1a, it can be seen that the surface of the pure WS₂ film has a “vermicular” loose porous structure

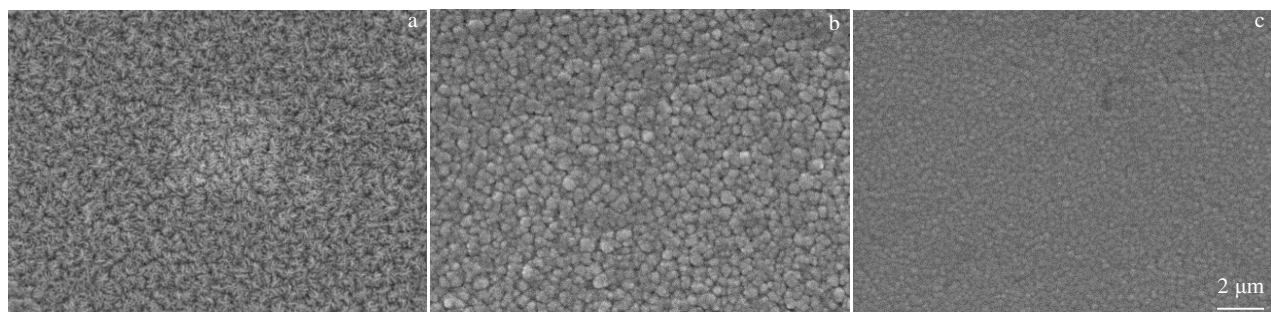


Fig.1 SEM images of surface morphology of films: (a) pure WS₂, (b) Ti/WS₂, (c) La-Ti/WS₂

with many cavities and pores. The surface roughness R_a measured by white-light interfering profilometer is about 21 nm, and the density is weak. The density of the film can be significantly improved by doping Ti in WS_2 film (Fig.1b), but the grain size is still coarser, and there are still pores between them; the surface roughness R_a is about 27 nm. However, Fig.1c shows that the La-Ti/ WS_2 composite film doped with rare earth La presents island growth mode, and the surface presents "hillock cellular" morphology structure; porosity is reduced, arranged compactly with uniform structure, and the grain size is smaller than 100 nm; no obvious defects can be observed, and surface roughness is about $R_a=16$ nm.

The pure WS_2 film prepared by unbalanced magnetron sputtering has poor density and obvious porous structure (as shown in Fig.1a). The structure makes it expand the contact area with external oxygen and water vapor, reduces its carrying capacity and easily leads to WS_2 film failure, which is similar to the analysis conclusion in Ref.[12]. When Ti is doped into the composite film, the distribution of Ti in the composite film is uniform, which plays a role in refining the grains. As a result, the voids and pores of the composite film are reduced, the grain size is decreased, and the density is improved. However, after La doping, the grain size of Ti/ WS_2 composite film becomes smaller, the microstructure of La-Ti/ WS_2 composite film is more uniform and compact, and the density of film is greatly improved. It is speculated that due to the doping of La, it is easy to concentrate at the grain boundary, inhibiting the growth and coarsening of grain, increasing the nucleation rate, and promoting the orderly growth of crystal arrangement. Thus fine crystal structure is

obtained and the density of composite film is improved^[10,13].

Fig.2 exhibits the sectional morphology of three kinds of films. The La-Ti transition layer can be observed obviously in pure WS_2 film, but the upper part shows loose and porous coarse columnar crystal structure. The boundary is clear, and the thickness of film is 6.48 μm (Table 2). It can be seen from Fig.2b that the Ti/ WS_2 composite film shows a columnar crystal structure with good density, the thickness and the growth rate of the composite film are reduced. However, the thickness of La-Ti/ WS_2 composite film is decreased to 2.58 μm , suggesting that the growth rate of La-Ti/ WS_2 composite film is slow, while the columnar grains of this composite film are significantly refined. This is obviously related to the doping of La, which slows down the growth rate of the columnar crystal structure of WS_2 film, leading to uniform and compact microstructure of the composite film. The oxidation resistance and carrying capacity of the composite film can be enhanced due to its compact structure^[14].

The XRD patterns of the three kinds of films are shown in Fig.3. The XRD patterns show that there are different intensity WS_2 (002) diffraction peaks for WS_2 film and Ti/ WS_2 composite film near $2\theta=14^\circ$. At the same time, there are different intensities of (100), (110), (200) diffraction peaks in the XRD patterns. The appearance of different intensity multiple diffraction peaks indicates that WS_2 is polycrystalline in the film. Compared with WS_2 film, the width and height of (002) diffraction peak of Ti/ WS_2 composite film are increased significantly, while the height of (100), (110) and (200) diffraction peak is decreased significantly, which indicates that the grain size of film is decreased, and the film grows

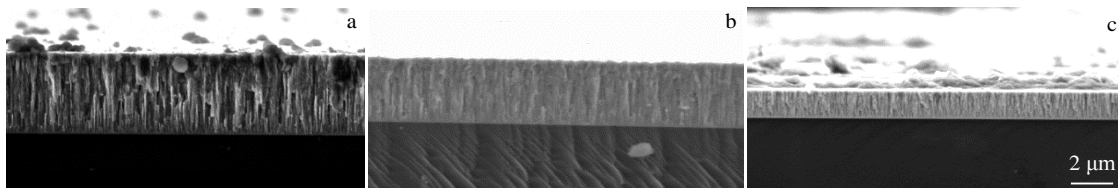


Fig.2 Cross-sectional morphologies of films: (a) pure WS_2 , (b) Ti/ WS_2 , (c) and La-Ti/ WS_2

along the preferred orientation of WS_2 (002) crystal surface. This is due to the doping of Ti element in WS_2 film, which is consistent with the analysis of the micro morphology in Fig.1. However, after La doping into Ti/ WS_2 composite films, the XRD patterns of the composite films show a broadened diffraction peak at $10^\circ \sim 15^\circ$, and the peak is moved to the direction of low angle; there is an unobvious broadened diffraction peak between $30^\circ \sim 40^\circ$. Through comparison, it is

Table 2 Chemical composition and thickness of three kinds of films

| Film | S/W | Ti content/at% | La content/at% | Thickness/ μm |
|---------------|------|----------------|----------------|--------------------------|
| Pure WS_2 | 2.08 | | | 6.48 |
| Ti/ WS_2 | 1.93 | 10.23 | | 4.94 |
| La-Ti/ WS_2 | 1.40 | 4.49 | 7.29 | 2.58 |

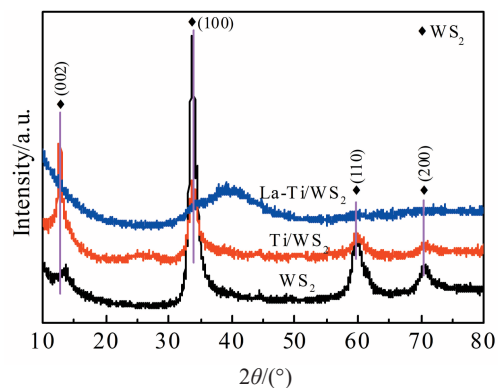


Fig.3 XRD patterns of pure WS_2 film, Ti/ WS_2 and La-Ti/ WS_2 composite film

found that the (110) and (200) diffraction peaks disappear, which shows that the crystalline state of the composite film is changed, and a small number of (002) crystal planes perpendicular to the substrate surface are suppressed, so it tends to amorphous state^[15]. Comparing the XRD patterns of the three kinds of films, it can be seen that the crystalline state of the films is changed due to the doping of La, which leads to the dominant orientation of the films. The (002) plane of WS₂ in the film is arranged parallel to the substrate. The diffraction peak of WS₂ (002) is further widened due to the doping of La, which reflects the further reduction of the film particle size. Therefore, by doping rare earth La, the long-range ordered arrangement of WS₂ molecules is effectively blocked, the growth of (002) crystal plane perpendicular to the substrate is inhibited, the growth of (002) slip plane parallel to the substrate surface is promoted, and the micro crystallization of the film is produced, making the composite film more conducive to lubrication^[16]. No diffraction peak of dopants is found in the XRD pattern, which indicates that the composite film is not a multilayer structure, but tends to be a solid solution^[17].

Table 2 lists the chemical composition and thickness of the three kinds of films. It can be seen that the S/W atomic ratio of WS₂ film and Ti/WS₂ composite film is approximately close to the WS₂ normal atomic ratio, while the S/W atomic ratio of La-Ti/WS₂ composite film is reduced to about 1.40. It can be seen that the doping of La has a significant effect on the reduction of S/W atomic ratio in the film. The reason is that the sputtering deposition of S element is inhibited by the doping of La, and the effect of desulfuration purification is produced. As a result, the S/W atom ratio in the film is decreased and the content of W element in the film is higher, which is beneficial to improve the density and hardness of the film.

2.2 Hardness and elastic modulus

The doping of La has a great influence on the microhardness of the composite films. Table 3 exhibits the hardness (H), elastic modulus (E) and H/E ratio of the three kinds of films. The hardness of WS₂ film is the lowest, while the hardness of La-Ti/WS₂ composite film is the highest, which is 22 times higher than that of WS₂ film and 12 times higher than that of Ti/WS₂ composite film, and the elastic modulus are also improved by La doping. The reason is that the grain size and microstructure of WS₂ film are changed by doping metal elements. In addition, the hardness and elastic modulus are increased more obviously due to the doping of rare earth La. Due to the doping of La, the impurities in the grain boundary are further removed, the defects between the crystals are made

Table 3 Microhardness, elastic modulus and H/E ratio of three kinds of films

| Film | Hardness, H /GPa | Elastic modulus, E /GPa | H/E ratio |
|-----------------------|--------------------|---------------------------|-------------|
| Pure WS ₂ | 0.21 | 29.66 | 0.007 |
| Ti/WS ₂ | 0.366 | 31.22 | 0.012 |
| La-Ti/WS ₂ | 4.597 | 84.58 | 0.054 |

up, and the area of grain boundary becomes larger; meanwhile, lattice distortion is caused, the resistance of dislocation movement is increased, the dislocation slip deformation in the grains is hindered, so the hardness and carrying capacity of the film are increased^[8].

2.3 Tribological properties

The friction coefficient curves of the three kinds of films are illustrated in Fig. 4. Obviously, the friction coefficient of Ti/WS₂ composite film is the largest. It can be seen that the hardness of the composite film is improved due to the doping of Ti, but the friction performance of the composite film may be lower than that of WS₂ film due to the inappropriate content. However, the tribological properties of La-Ti/WS₂ composite films are improved effectively after the doping of La, and the friction coefficient is stable and the minimum value is 0.071. The reason is that after La and Ti are doped in WS₂, the structure of composite film is compact, the adhesion of film and substrate is improved, the carrying capacity is enhanced, and it is not easy to worn out in the process of friction.

The wear rate comparison of the three kinds of films is presented in Fig. 5. The wear rate and friction coefficient of the composite films have the similar change rule. The wear rate of Ti/WS₂ composite film is the largest, reaching $8.78 \times 10^{-7} \text{ mm}^3/(\text{N} \cdot \text{m})$. This is because although the density and hardness of the film are improved by the doping of Ti, there are still small pores, which make it as easy to react with water and oxygen in the atmosphere as WS₂ film, leading to the lubrication failure. In addition, the composite film shows great brittleness, which is not conducive to the formation of high-quality transfer film. The wear rate of La-Ti/WS₂ composite film is the lowest, which is $2.45 \times 10^{-7} \text{ mm}^3/(\text{N} \cdot \text{m})$. Research suggests that the wear resistance of the film is better with the increase of the ratio of H/E ^[18]. From the H/E ratio in Table 3, it can also be seen that the H/E ratio of the composite film is increased significantly after rare earth doping, which shows that the wear resistance of the film is strengthened^[19-21]. At the same time, the (002) slip surface of composite film is parallel to the surface of substrate, which also plays a good lubrication role.

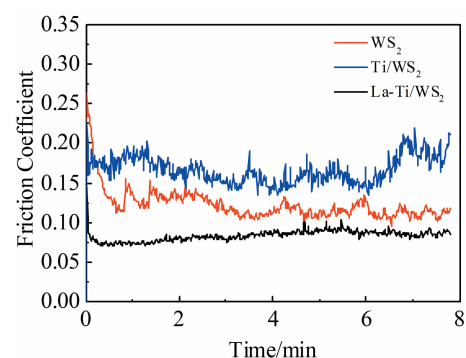


Fig.4 Friction coefficient curves of pure WS₂ film, Ti/WS₂ and La-Ti/WS₂ composite film

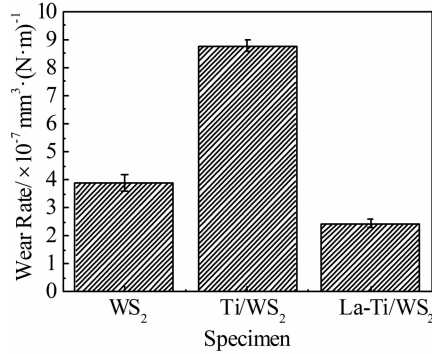


Fig.5 Wear rate of pure WS₂ film, Ti/WS₂, and La-Ti/WS₂ composite film

The two-dimensional profiles of wear tracks of three kinds of film are shown in Fig. 6. The wear track of La-Ti/WS₂ composite film with the narrowest width and the shallowest depth is noticed, as shown in Fig.6c. The wear tracks of WS₂ film and Ti/WS₂ composite film present burrs at the edge, which indicate the wear characteristics of three body abrasive wear. Due to the doping of rare earth element La, the hardness and wear resistance of the film are increased^[22].

Fig. 7 shows the worn surface morphologies of pure WS₂ film, Ti/WS₂ composite film and La-Ti/WS₂ composite film. It is found in Fig.7a that for the pure WS₂ film, there are obvious furrows and wide wear track on the worn surface, indicating severe abrasive wear in the sliding direction, and the friction coefficient fluctuates continuously (Fig. 5). By comparison,

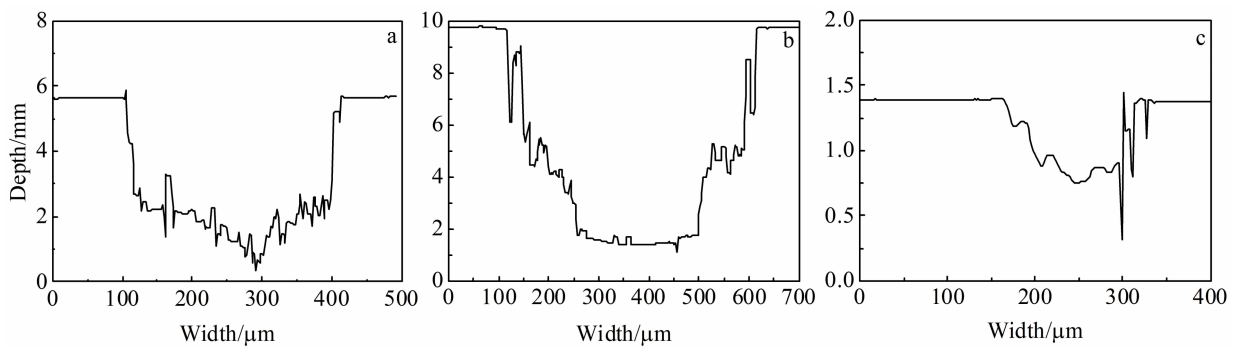


Fig.6 Two-dimensional profiles of wear tracks for different films: (a) pure WS₂ film, (b) Ti/WS₂ composite film, and (c) La-Ti/WS₂ composite film

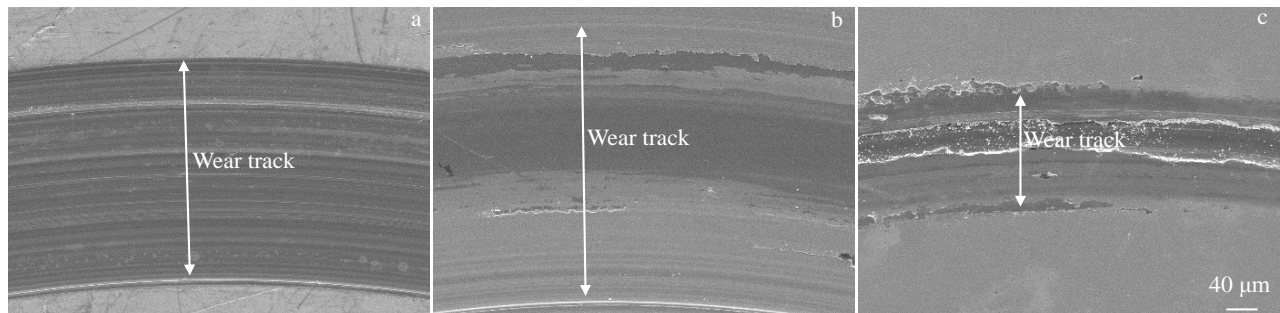


Fig.7 Worn surface morphologies of pure WS₂ film (a), Ti/WS₂ composite film (b) and La-Ti/ WS₂ composite film (c)

the Ti/WS₂ composite film exhibits worse wear resistance than the pure WS₂ film, since the wider wear track is still noticed on the worn surface of Ti/WS₂ composite film, as shown in Fig.7b. Also, it can be seen from Fig.6b that the depth of the wear tracks exceeds the total thickness of the Ti/WS₂ composite film. Whereas, the width and depth of wear track of the La-Ti/WS₂ composite film are rather small, as seen in Fig.7c. It suggests that the La-Ti/WS₂ composite film exhibits better wear resistance when sliding against the steel ball, as recording in Fig.6c.

The morphology of wear scars on the surface of steel ball with three kinds of films is shown in Fig. 8. As can be seen

from Fig.8a, a large number of wear debris of pure WS₂ film are adhered in the center of the wear scar, and the wear debris is scattered around. It shows that the adhesion of the pure WS₂ film is weak, which is not conducive to the formation of stable transfer film. The morphology of wear scar in Fig.8b is similar to that in Fig.8a. Due to the poor structure compactness of the Ti/WS₂ composite film, the film is easy to oxidize and the stable transfer film cannot be formed at the friction interface. However, the transfer film is formed on the surface of the steel ball with La-Ti/WS₂ composite film (Fig. 8c). The transfer film can play a lubricating role in the friction process, and the wear rate is reduced. The reason is that the structure of

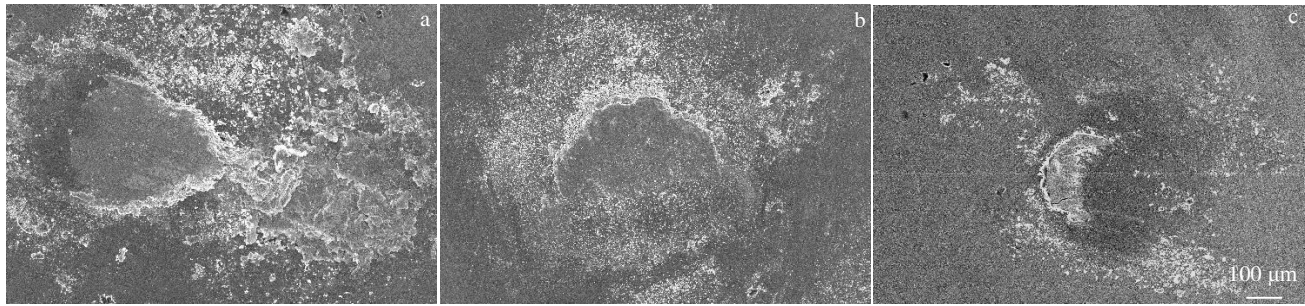


Fig.8 Morphologies of wear scars on the surface of steel ball with pure WS₂ film (a), Ti/WS₂ composite film (b) and La-Ti/WS₂ composite film (c)

the composite film is changed due to La doping, the density of the composite film and the adhesive strength with the substrate are enhanced. Furthermore, the transfer film formed in the process of friction effectively prevents the direct contact between the composite film and steel ball, and thus the delamination and ploughing phenomenon of the film are weakened. Besides, due to the low friction coefficient of the friction interface, the wear resistance of the film also tends to be strengthened by compaction with the continuous friction^[23]. To sum up, it can be concluded that the tribological performance of the La-Ti/WS₂ composite film is largely determined by integrative action of surface morphology, microstructure and microhardness of the films.

3 Conclusions

1) Rare earth La is applied in WS₂ composite films prepared by magnetron sputtering. The pure WS₂ film, Ti/WS₂ and La-Ti/WS₂ composite films can be successfully fabricated on stainless steel and monocrystalline silicon substrates by magnetron sputtering process.

2) Compared with that of pure WS₂ film and Ti/WS₂ composite film, the density of La-Ti/WS₂ composite film is obviously enhanced. The La-Ti/WS₂ composite film exhibits higher hardness than pure WS₂ film and Ti/WS₂ composite film.

3) The superior tribological performance of La-Ti/WS₂ composite film is attributed to the formation of WS₂ transfer films at contact area and good mechanical properties, which can provide a better carrying capacity.

4) La-Ti/WS₂ composite film shows compact structure and high carrying capacity, which make the film have great engineering application potential.

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非平衡磁控溅射 La-Ti/WS₂ 自润滑薄膜的摩擦磨损行为

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摘要: 要满足航天器机械转动部件在恶劣工况下的工作, 需研制高硬度、低摩擦系数的固体润滑薄膜。采用非平衡磁控溅射法分别制备了纯 WS₂ 薄膜、Ti 掺杂 WS₂ 复合薄膜和 La-Ti 掺杂 WS₂ 复合薄膜。分析了薄膜的微观形貌、成分、硬度和摩擦学性能。结果表明, 与纯 WS₂ 薄膜和 Ti/WS₂ 复合薄膜相比, La-Ti/WS₂ 复合薄膜的微观结构更加致密。La-Ti/WS₂ 复合薄膜的硬度 H 和弹性模量 E 也显著提高。此外, La-Ti/WS₂ 复合薄膜的摩擦系数减小, 并且 H/E 比值增大, La-Ti/WS₂ 复合薄膜的磨损率降低。结果表明, La 的掺杂有助于在摩擦接触表面形成稳定的转移膜, 提高 La-Ti/WS₂ 复合薄膜的耐磨性和承载能力。

关键词: La-Ti/WS₂ 复合薄膜; 非平衡磁控溅射; 减摩; 耐磨

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