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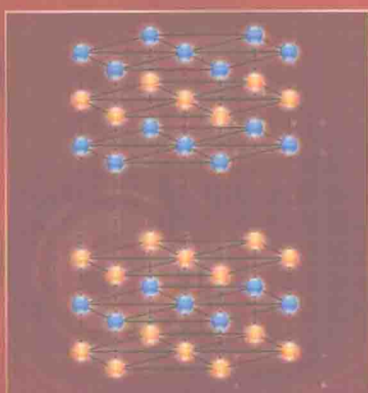
Micro and Nano Sulfide Solid Lubrication

（微纳米硫系固体润滑）

• Haidou Wang Binshi Xu Jiajun Liu •



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With 367 figures

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Preface

In order to cope with the fast development of modern industries, the lubrication of machinery equipment has attracted extensive attention. Although the liquid lubrication is widely used, more and more machine parts are working in the increasingly severe conditions, like high temperature, high load, ultralow temperatures, ultrahigh vacuums, strong radiation, etc. For these working conditions, the commonly used lubricating oils and greases are hardly applicable, so that the solid lubrication is necessary as the supplement for liquid lubrication.

The solid lubrication technique was first applied to the aerospace and military industries, after that, it was gradually propagated to the high-tech and newly developed departments of industry. Its development enlarged the application fields of the traditional lubricating oils and greases, counteracted their shortcomings, and the most important result is the emergence of an increasingly large amount of new lubricating techniques using solid lubricants, which are either new materials, or new techniques, exhibiting more important meanings. The practice has proven that the proper adoption of advanced lubrication materials and techniques can effectively prolong the service life of machinery working under the harsh conditions. Furthermore, saving energy and raw materials is the general and urgent requirement today. The rational application for lubricating materials and techniques are even more important economically and socially.

This book aims to introduce the advanced solid lubrication techniques of sulfur system to a broad range of readers. It presents the recent research successes of the authors. Based on this, the authors, depending on their profound theoretical basis and experiences in the interdisciplinary field covering tribology, materials science, surface engineering, chemistry, physics and so on, explained the solid lubrication of materials and techniques of sulfur system comprehensively and systematically. They involve microstructures, properties, preparation methods, applications and mechanisms of the solid lubrication materials. Hope this book can be useful to those readers who are interested in the research and application for solid lubrication.

The main contents of this book are selected from the authors' basic research work in the recent twenty years. However, the solid lubrication is a new area of tribology and lubricating materials; a special and interdisciplinary subject, most phenomena and mechanisms are still in their early stages. What we have discussed in this book may be incomplete or not one hundred percent correct, for this reason, we welcome different opinions or comments from readers.

The authors would like to thank Dr. Lina Zhu and Dr. Jiajie Kang for their supports. This book wouldn't be finished without their help.

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Haidou Wang　Binshi Xu　Jiajun Liu
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Contents

Chapter 1 Solid Lubrication Materials ···· 1
1.1 Overview of Solid Lubrication ···· 1
1.1.1 Introduction ···· 1
1.1.2 Adhesive Wear and Scuffing of Metals and Methods of Prevention ···· 2
1.1.3 Solid Lubrication ···· 5
1.2 Soft Metal ···· 9
1.2.1 Crystal Structure ···· 9
1.2.2 Physical and Chemical Properties ···· 9
1.2.3 Lubrication Mechanism ···· 11
1.3 Metal Compounds ···· 11
1.3.1 FeS ···· 11
1.3.2 MoS_2 ···· 21
1.3.3 WS_2 ···· 36
1.3.4 ZnS ···· 40
1.4 Inorganic Solid Lubricant ···· 46
1.4.1 Graphite ···· 46
1.4.2 BN ···· 48
1.5 Organic Solid Lubricant ···· 51
1.5.1 Polytetrafluoroethylene ···· 53
1.5.2 Polythene ···· 55
1.5.3 Nylon ···· 56
1.5.4 Polyformaldehyde ···· 56
1.5.5 Phenol Formaldehyde Resin ···· 57
1.5.6 Epoxide Resin ···· 58
1.6 Conclusion ···· 58
References ···· 59
Chapter 2 Solid Lubrication FeS Film Prepared by Ion Sulfuration ···· 61
2.1 The Microstructure of Solid FeS ···· 61
2.1.1 Surface Morphologies of Solid FeS ···· 61
2.1.2 Phase Structures of Solid FeS ···· 62
2.1.3 TEM Morphologies of Solid FeS ···· 63
2.1.4 Analysis of Electron Diffraction ···· 63
2.2 The Formation of Iron Sulfuration Layer ···· 64

2.2.1 Experimental Methods ···· 64
2.2.2 Surface Morphologies of Sulfuration Layers ···· 64
2.2.3 Composition on the Sulfurized Steel Surface ···· 66
2.2.4 Phase Structure of Sulfide Layer at Different Sulfurizing Time ···· 67
2.2.5 Formation Mechanism of Sulfurized Layer ···· 68
2.3 Characterization of Ion Sulfurized Layer ···· 69
2.3.1 Characterization of Sulfurized Layer on 1045 and 52100 Steels ···· 69
2.3.2 Characterization of Sulfurized Layer on Four Kinds of Steels ···· 76
2.4 Tribological Properties of Sulfurized Layers ···· 78
2.4.1 Tribological Properties of Sulfurized Layers on 1045 and 52100 Steels ···· 78
2.4.2 Tribological Properties of Sulfide Layer on Four Kinds of Steel ···· 98
2.5 Influencing Factors of the Microstructures and Tribological Properties on Sulfurized Layers ···· 105
2.5.1 Effect of the Substrate State on the Sulfide Layer on 1045 Steel ···· 105
2.5.2 Effect of Environment Temperature on the Sulfurized Layer on 52100 Steel ···· 109
2.5.3 Effect of Wear Conditions on the Tribological Behaviors of Sulfurized Layer on 52100 Steel ···· 112
References ···· 113
Chapter 3 FeS Solid Lubrication Film Prepared by a Two-step Method ···· 115
3.1 Radio-frequency (RF) Sputtering + Sulfurizing Combined Treatment ···· 115
3.1.1 RF Sputtering Technology ···· 115
3.1.2 Process of Preparation ···· 117
3.1.3 Microstructures ···· 117
3.1.4 Tribological Properties ···· 119
3.2 Shot-peening + Ion Sulfuration Combined Treatment ···· 121
3.2.1 Preparation ···· 121
3.2.2 Characterization ···· 122
3.2.3 Tribological Properties of Sulfide Layer ···· 124
3.3 Nitriding + Sulfurizing Combined Treatment ···· 127
3.3.1 1045 Steel Nitriding + Sulfurizing Combined Treatment ···· 128
3.3.2 Gray Cast-iron Nitriding + Sulfurizing Combined Treatment ···· 135
3.4 Nitrocarburizing + Sulfurizing Combined Treatment ···· 138
3.5 Thermal Spraying 3Cr13 Steel Coating + Sulfurizing Combined Treatment ···· 140
3.5.1 Arc Spraying Technology ···· 140
3.5.2 High-velocity Arc Spraying ···· 143
3.5.3 Preparation ···· 143

3.5.4 Microstructures · · · 144
3.5.5 Tribological Properties · · · 146
3.6 Thermal Spraying FeCrBSi + Sulfurizing Combined Treatment · · · 148
3.6.1 Preparation · · · 149
3.6.2 Microstructures · · · 149
3.6.3 Tribological Properties · · · 151
3.7 MIG (metal inert-gas) Welding + Sulfurizing Combined Treatment · · · 160
3.7.1 MIG Welding Technology · · · 160
3.7.2 Preparation · · · 160
3.7.3 Structures · · · 161
3.7.4 Tribological Properties · · · 162
3.8 Mechanism of FeS Film Prepared by Different Methods · · · 165
References · · · 168
Chapter 4 FeS Solid Lubrication Layer Prepared by Other Methods · · · 169
4.1 High-velocity Flame Sprayed FeS Coating · · · 169
4.1.1 High-velocity Flame Spraying Technology · · · 169
4.1.2 Preparation · · · 175
4.1.3 Characterization · · · 175
4.1.4 Tribological Properties of FeS Coating · · · 178
4.1.5 Lubrication Mechanism of Sprayed FeS Coating · · · 183
4.2 Plasma Sprayed FeS and FeS_2 Coatings · · · 183
4.2.1 Plasma Spraying Technique · · · 183
4.2.2 Preparation of FeS (FeS_2) Coating · · · 186
4.2.3 Characterization of FeS (FeS_2) Coating · · · 187
4.2.4 Tribological Properties of FeS (FeS_2) Coating · · · 188
4.3 Plasma Sprayed Nano-FeS and FeS-SiC Composite Coating · · · 192
4.3.1 Plasma Sprayed Nano-FeS Coating · · · 193
4.3.2 Sprayed FeS-SiC Composite Coating · · · 193
4.4 Comparison of the Tribological Properties of Ion Sulfurized Layer and Plasma Sprayed FeS Coating · · · 194
4.4.1 Experimental Method · · · 194
4.4.2 Microstructure and Tribological Properties · · · 194
4.5 Sol-gel FeS Coating · · · 196
4.5.1 Preparation · · · 196
4.5.2 Characterization · · · 197
4.5.3 Tribological Properties · · · 199
4.6 FeS Film Prepared by S-ion Implantation · · · 203
4.6.1 Ion Implantation Technology · · · 203

4.6.2 Tribological Properties of Sulfur-implanted Steel ··· 205
References ··· 205
Chapter 5 Micron-nano MoS_2 Solid Lubrication Film ··· 207
5.1 MoS_2 Film ··· 207
5.1.1 MoS_2 Sputtering Film ··· 207
5.1.2 MoS_2 Film Prepared by Two-step Method ··· 209
5.1.3 Thermal Spraying MoS_2 Film ··· 226
5.1.4 Bonded MoS_2 Film ··· 227
5.1.5 Inorganic Fullerene-like Nano MoS_2 Film ··· 234
5.2 MoS_2/metal Co-deposition ··· 235
5.2.1 MoS_2/Ni Composite Film ··· 236
5.2.2 MoS_2/Ti Composite Film ··· 248
5.2.3 MoS_2/Au Co-sputtered Film ··· 250
5.3 MoS_2/Metal Compound Composite Film ··· 251
5.3.1 MoS_2/TiN Composite Film ··· 251
5.3.2 MoS_2/Pb_2O_3 Composite Film ··· 255
5.3.3 MoS_2/LaF_3 Composite Film ··· 255
5.3.4 MoS_2/FeS Multilayer Film ··· 256
5.4 MoS_2/graphite Sputtered Coating ··· 261
References ··· 263
Chapter 6 Micron-nano WS_2 Solid Lubrication Film ··· 265
6.1 WS_2 Film ··· 265
6.1.1 Characterizations of the Synthetic WS_2 Film ··· 266
6.1.2 Tribological Properties of the Synthetic WS_2 Film ··· 267
6.2 WS_2/Ag Composite Film ··· 273
6.2.1 Structures of WS_2/Ag Composite Film ··· 273
6.2.2 Tribological Properties of the WS_2/Ag Composite Film ··· 275
6.3 WS_2/MoS_2 Multilayer Film ··· 279
6.3.1 WS_2/MoS_2 Co-sputtered Film ··· 279
6.3.2 WS_2/MoS_2 Multilayer Film Prepared by Combined Treatment ··· 281
6.4 WS_2/CaF_2 Composite Coating ··· 287
6.5 Ni-P-(IF-WS_2) Composite Film ··· 288
References ··· 290
Chapter 7 Micron-nano ZnS Solid Lubrication Film ··· 291
7.1 ZnS Film Prepared by High Velocity Arc Spraying + Sulfurizing Treatment ··· 291
7.1.1 Preparation ··· 291
7.1.2 Characterizations ··· 291

7.1.3 Tribological Properties · · · 293
7.1.4 Lubrication Mechanisms of the Zn/ZnS Composite Layer · · · 295
7.2 ZnS Film Prepared by Nano-brush Plating + Sulfurizing Treatment · · · 295
7.2.1 Preparation · · · 296
7.2.2 Morphologies · · · 296
7.2.3 Friction Coefficient · · · 297
7.2.4 Worn Morphologies · · · 298
7.2.5 Energy Spectrum Analysis · · · 299
7.3 Conclusion · · · 303
Index · · · 305

Chapter 1
Solid Lubrication Materials

Solid lubrication is an important component of lubrication. It overcomes some inherent drawbacks for liquid lubrication. Solid lubrication can be adopted in very harsh conditions such as high temperature, heavy load, ultralow temperature, ultrahigh vacuum, strong oxidation, and intense radiation, etc. In above conditions, liquid lubrication will lose its lubricating function. Solid lubrication materials are divided into four categories, i.e., soft metals, metal compounds, inorganic and organic materials. They have different lubricating mechanisms and can be applied to different situations.

1.1 Overview of Solid Lubrication

1.1.1 Introduction

Friction leads to the loss of a large quantity of mechanical energy, while wear is an important reason of the failure of machinery parts. According to incomplete statistics, about 1/3 of the energy was consumed by friction; and about 80% failure of machine parts was caused by wear. For a highly industrialized country, the annual economic loss caused by friction and wear almost accounts for 2% GDP value of the national economy. Therefore, the research on friction and wear is a long term subject, which has important social and economic benefits.

From time immemorial, friction and wear have been accompanying human life and production, and the efforts that people try to control friction and reduce wear have never been stopped. With the development of industry, especially in modern industry and technology, ever-increasing demands are put forward for friction and wear in the harsh working environment such as high speed, heavy load, high vacuum, high and low temperature, nuclear radiation. Such a background provided strong impetus for the development of the interdisciplinary subject of industrial tribology. In recent years, the application of surface engineering techniques for improving the friction-reducing and wear-resistance of materials has played a very important role in elevating quality of products, decreasing cost and saving energy. Surface engineering technology can modify the materials surface of machine parts, greatly improve their performance and significantly extend their service life through preparing various coatings and films. Meanwhile, it can be also used for repairing the failed machine parts. So, surface engineering technology will be one of the key technologies in manufacturing and remanufacturing of mechanical equipment, which will master the industrial development in the 21st century. The rapid development of surface engineering provided a very good opportunity for the R&D of solid lubrication materials, in other words, the great progress of solid lubrication in recent years is promoted by the development of surface engineering technology.

1.1.2 Adhesive Wear and Scuffing of Metals and Methods of Prevention

1.1.2.1 Essence of adhesive wear and scuffing

When two surfaces come into contact, load is actually born by many asperities; the real contact area of rough surfaces is usually far less than the nominal contact area. Therefore, the local high stress is easily generated at the asperity peaks. When the surfaces are sliding with each other, the local melting caused by friction heat will lead to the adhering or welding between asperities, which can induce the macro adhesion and scuffing, or even seizure of friction surfaces. Adhesive wear is a common wear mode of the friction-pair in machinery equipment. Due to high surface energy and large chemical activity, clean metal surfaces are prone to adhesion, thus the friction coefficient is large, and the wear is severe. Under oil lubrication condition, adhesion can occur if the oil film is damaged; otherwise, the friction coefficient and wear can maintain a relatively low level. According to the degree of adhesion, adhesive wear can be divided into four categories. When soft metal material transfers to hard metal surface, and the transfer film is very thin, it is known as smearing. When shearing occurs within the subsurface layer of soft metal, and hard surface is also scratched, it is known as scratching. When shearing occurs in the deep layer of substrate metal, it is known as scuffing. Cold welding caused by plastic deformation and molecular absorption is known as the first type of scuffing, while hot welding caused by the rise of surface temperature is known as the second type of scuffing. When external force can not overcome the bonding strength of interface, the relative movement is forced to be stopped, which is known as seizure.

Under low-load condition, oxidation wear will happen firstly on the friction surface, the generated oxide film can prevent the occurrence of adhesion; when the load is increased, the oxide film will be destroyed, the lubrication effect is lost, and the adhesive wear will be present. The existence of liquid or solid lubricating films can effectively prevent the development of adhesive wear. There are many factors influencing the adhesive wear, in general, including two aspects: one is the own characteristics of the friction-pair materials, like intersolubility of metals, crystal structure, microstructure, and hardness, etc.; the other is working conditions, such as load, sliding speed, working temperature, and lubricating condition, etc.

Scuffing is a serious wear mode, it usually appears in the sliding wear; its definition is defined as “severe wear with characteristic of forming the partial weld spot between sliding surfaces”. It indicates that it was generally accepted that the essence of scuffing is belonging to adhesive wear, but the subsequent researches proved that adhesive wear is only one of the mechanisms of scuffing and mostly occur in the case of dry friction. Under oil lubrication condition, the scuffing mechanism is mainly strain fatigue wear present in the form of delamination. When load and sliding speed increase to a certain extent, the boundary lubricating film will be damaged, or it is insufficient to form boundary lubricating film; the scuffing mechanism will convert from fatigue wear to adhesive wear.

1.1.2.2 Surface modification

In order to improve the wear-resistance of machine parts, a wide range of surface modification technologies are employed, such as surface quenching, surface chemical heat treatment, electric plating, laser modification, thermal spraying, superhard film depositing, ion implantation, etc. Surface modification can change the structures and properties of friction-pair surfaces, weaken the mutual solubility between materials, increase hardness and strength of surface, as a result, the deformation of surface can be reduced, the adhesion and scuffing of friction-pair are mitigated or avoided. But for surface hardening materials, when the center hardness is high, and hardened layer is thick, the shallow flaking will happen; when the center hardness is low, and hardened layer is thin, the whole hardened layer will peel off. The mechanical conditions leading to the two kinds of flaking are shown in Figure 1.1. For stress fatigue wear, crack initiation is the dominant process; thus the higher the material hardness, the more difficult the crack initiation, and the longer the fatigue wear life. For the strain fatigue wear, the propagation of fatigue crack is the dominant factor to determine the fatigue life. The increase of hardness must lead to decrease of the material toughness and accelerating the propagation rate of cracks, which makes scuffing happen easily. Therefore, for resisting adhesive wear and fatigue wear, high hardness of material surface is not always necessary. Many researches have proved that the high carbon steel in the normalizing state possesses better anti-scuffing performance than that in the quenching state. Therefore, in the case for improving the anti-scuffing property of friction surface as the main purpose, the modification techniques of establishing a solid lubrication coating (soft coating) on the hard substrate should be adopted, but not to increase the surface hardness blindly.

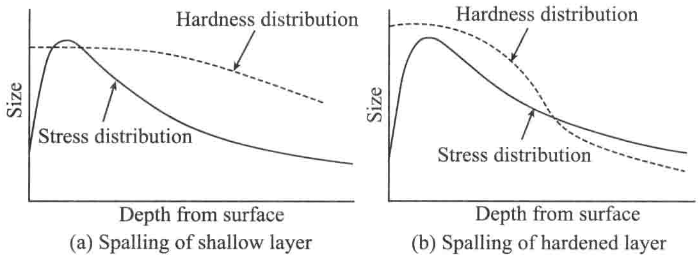


Fig.1.1 Mechanical conditions leading to the contact fatigue damage

Figure 1.2 shows the principle scheme how the solid lubrication coating can protect the material surface from strain fatigue wear (delamination).

1.1.2.3 Liquid lubrication and additive

Under the condition of hydrodynamic or elastohydrodynamic lubrication, the lubricating oil film will become thinner if its viscosity decreases, or the load is increased or speed is decreased. When the thickness of oil film is less than a certain value, asperities come into contact; the friction-pair will enter the state of boundary lubrication. In this case, the active elements in the oil can react with the metal surface to generate chemical reaction film under the effect of friction heat. Figure 1.3 shows the

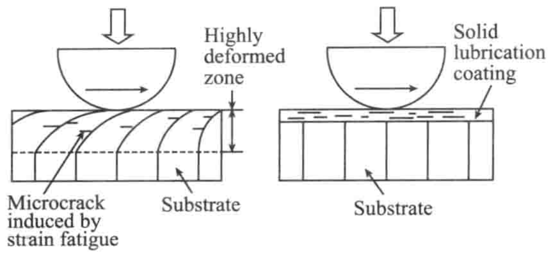
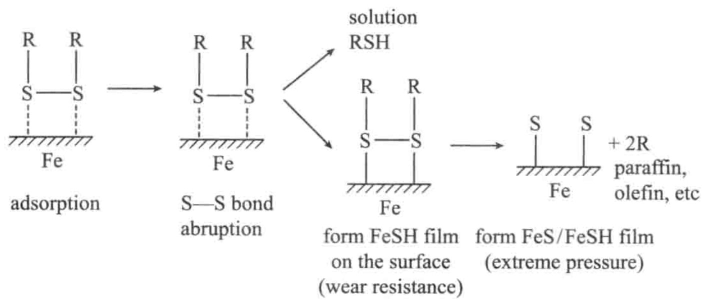


Fig.1.2 Principle scheme how the solid lubrication coating can protect the material surface from strain fatigue wear (delamination)

schematic diagram of the formation of FeS reaction film. The thickness of FeS reaction film is very small not more than 0.2μm, but the bonding strength between the film and substrate is enough high. In addition, the reaction is completely non-reversible and has greater stability compared with physical adsorption and chemical adsorption; therefore, adding various additives to base oil is an important method to achieve excellent lubrication effect. In a large number of wear-resistant and extreme pressure additives, sulfur and organic sulfide are a very important kind of additives. Batchelor has pointed out that when the hexadecane containing 0.75wt% sulfur is deposited on the fresh steel surface, a 75Å thick sulfide film can be generated within 3×10^{-2}s, and it is enough to make the adhesion of surface decrease to a safe range. As the formation time of ion sulfide film is short, when the lubricating oil contains sulfur, the scuffing is hard to occur even in very poor lubrication condition. At 100—170°C, the reaction of organic disulfide and iron on the friction surface is as follows:



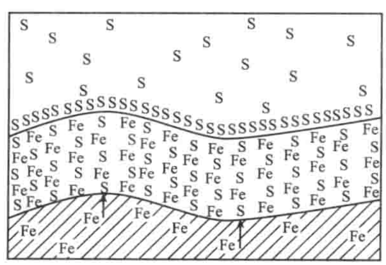


Fig.1.3 Schematic diagram of the formation of FeS reaction film by sulfur and iron

Disulfide is absorbed on the metal surface, and S—S bond disconnects to form mercaptan iron film, which can effectively protect the surface if the friction condition is not very serious. Under extreme pressure and heavy load, C—S bond disconnects to form iron sulfide, which can protect the surface as well.

Organic sulfide as boundary lubricating additive can play a good role only with the existence of air (oxygen). The researches of Baldwin have shown that under the extreme pressure condition, the balance product on the friction surface is $FeSO_4$, but it usually can not be obtained, as a result, FeS is present. For each kind of sulfur-containing additive, the formed boundary film always contains iron sulfide and iron oxide, among them iron oxide plays the role of catalysis to make the active sulfur atoms dissociated from sulfide and reacted with iron on the friction surface. Sulfur atoms can be replaced easily by oxygen atoms when the iron sulfide is in oxygen atmosphere; however, the sulfide can not play an effective role without the existence of oxygen.

Although liquid lubrication has a wide range of application, with the development of industry and technology, more and more machine parts are applied in the very harsh conditions, such as high temperature, heavy load, ultralow temperature, ultrahigh vacuum, strong oxidation, and intense radiation, etc. the general liquid and grease lubrications have lost their function in above conditions. To this end, solid lubrication can be adopted only.

1.1.3 Solid Lubrication

1.1.3.1 Definition and significance

According to the tribological terminology formulated by the Organization of Economic Cooperation and Development (OECD), the definition of solid lubrication is "use of any solid powders or films, which can protect the surfaces in relative motion from damage and reduce the friction and wear". In the course of solid lubrication, the physical and chemical reactions will happen between the solid lubricant and surrounding medium to reduce friction and wear [1].

Solid lubrication technology was first applied in military industry, and then applied to some high-tech fields such as artificial satellite, spacecraft and electronic industry. It had solved many problems, which could not be solved by liquid lubrication, and had been successfully applied in a variety of special conditions. Therefore, more and more attentions have been paid to solid lubrication technology. On the other hand, the global energy resources have been getting tense at present, thus the voices of that solid lubrication will replace liquid lubrication get more and more rising. So that the theoretical studies on the lubrication mechanism are increasing day by day; the effectiveness of solving the lubrication problems by solid lubricant is increasingly significant.

The application of solid lubricants has already a long history. Graphite, molybdenum disulfide, plumbum salt, soft metal powders and other solid lubricants have been applied well in industries, for example the polytetrafluoroethylene powder has been applied successfully in lubricating oil and grease as additive.

1.1.3.2 Role of solid lubricant

The solid lubricants overcome some inherent drawbacks for liquid lubricant. For instance, lubricating oil and grease are easy to evaporate, and the vapor pressure is high; thus they can not be used for a long time in the high vacuum of more than 10^{-1}Pa. However, in the outer space at the height of 1000 km, the vacuum degree is very high; the absolute pressure can achieve 10^{-3}—10^{-2}Pa. Therefore, artificial satellite needs the solid lubricant with low vapor pressure.

For the rocket carrying satellite, the kerosene and liquid hydrogen are used for propellant; liquid oxygen is used for oxidant. As the liquid hydrogen and oxygen are ultralow temperature fluid, with the boiling points of −253°C and −183°C respectively, the lubricating oil can not be used when the pressured transmission from tank to the turbopump support shaft of combustion chamber is conducted, especially once the liquid oxygen and lubricating oil contact, explosion will happen. Therefore, the bearings on the turbopump can be lubricated only by solid lubricant.

When liquid lubricant bears heavy load, the oil film will be damaged and lose the lubricating function at high temperature, while solid lubricant possesses high bearing capacity and can resist high temperature.

1.1.3.3 Application conditions of solid lubricants

1. The conditions, where solid lubricant can replace lubricating oil and grease

Under the following conditions, solid lubricants can replace lubricating oil and grease to lubricate friction surface.

(1) Solid lubricants can be used in various special conditions such as high temperature, low temperature, vacuum and heavy load, etc. The common lubricating oil and grease can not meet their requirements.

(2) Solid lubricants can be used in conditions where lubricating oil and grease are easily polluted or washed away by other liquids (such as water, seawater). They can also be used in the moist environment and solid impurities (such as silt, dust) existing conditions.

(3) Solid lubricants can be used in the conditions where lubricating oil and grease are difficult to be supplied to some components and friction-pairs, it is difficult to approach or load and unload during installations, and it is difficult to operate maintenance periodically.

2. To improve the properties of lubricating oil and grease

Solid lubricant can be added to lubricating oil and grease with following purposes:

(1) To improve the load-carrying capacity of lubricating oil and grease;

(2) To improve the ageing properties of lubricating oil and grease;

(3) To improve the high-temperature properties of lubricating oil and grease;

(4) To make the lubricating oil and grease form tribological polymer film.

3. Harsh running conditions

(1) Lubrication under a wide range of temperature.

The service temperature of lubricating oil and grease is about −60—350°C, while solid lubricant can be used under the temperature ranging from −270°C to above 1000°C.

The solid lubrication under ultra-low temperature is the key of ultra-low temperature technology. Solid lubricants like PTFE, and plumbum, etc. still possess lubricating properties under this temperature.

The ceiling service temperatures of high molecular material polyimide and plumbum oxide are 350°C and 650°C respectively, while that of the mixture of calcium fluoride and barium fluoride is 820°C. The operating temperature of hot rolling of steel products can reach more than 1200°C. In this condition, graphite, glass, and all kinds of soft metal films can be used as effective lubricant.

(2) Lubrication under a wide range of speed.

A variety of solid lubricating films can be used under a wide range of sliding speed. For instance, the motion of machine tool guideways belongs to low speed motion, the lubricating oil containing solid lubricant can reduce crawling, and the solid lubrication dry film made from high molecular material by spraying method can reduce wear. The soft metal plumbum film can be applied to sliding surfaces of low speed motion. The bearings applied in the condition of low speed and heavy load can be made from sliding materials in the form of mosaic structure. For the bearings used in the high speed condition, when titanium carbide film with thickness of 2—5μm is deposited on their surface, the wear is slight even if they are operating at a speed of 24000r/min for 25000h.

(3) Lubrication under heavy load.

The oil films of common lubricating oil and grease can sustain only relatively low loads. Once the load exceeded the limited value, the oil film will be damaged, and the seizure may occur. However, solid lubrication film can bear the average load over 10^8Pa. For instance, MoS_2 film with thickness of 2.5μm can bear a contact pressure of 2800MPa and operate at a high speed of 40m/s. PTFE film can bear a pressure of 10^9Pa, while the load-carrying capacities of metal-based composite materials are even higher. For the pressure working of metals such as rolling, extruding, or stamping, the load is very high. In this condition, oil-based or water-based lubricant containing solid lubricant or solid lubrication dry film can be used.

(4) Lubrication under vacuum.

Under vacuum or high vacuum conditions, common lubricating oil and grease have relatively large evaporability, which easily destroys the vacuum condition, and affects the properties of other components. In this condition, metal-based and high molecular polymer-based composite materials are commonly used.

(5) Lubrication under radiation.

Under the radiation condition, general liquid lubricants will be polymerized or decomposed, and lose their lubrication properties. Solid lubricants have better radiation resistance. For instance, the radiation resistance of metal-based composite materials is more than 10^8Gy; no detectable change happens after graphite is radiated by strong neutrons of $10^{20}/cm^3$. Under the radiation condition, layered solid lubrication materials, polymer-based and metal-based composite materials can be used.

(6) Lubrication for conducting sliding surfaces.

For the friction between conducting sliding surfaces, such as electric brush, conductive slider, solar slip rings on the man-made satellite working in vacuum, and sliding electrical contact, carbon-graphite, metal (Ag)-based composite materials, or composite material composed of metal and solid lubricant can be used.

4. Formidable environment conditions

The transmission parts of transport machinery, construction machinery, metallurgy machinery, and mining machinery, etc. are working mostly in the formidable environment, such as dust, silt, high temperature and moisture, etc., solid lubricants can be used in this case.

5. Clean environment conditions

The transmission parts of machinery in the electron, textile, food, medicine, paper making, and printing industries, etc. should avoid pollution and require clean environment. They can be lubricated with solid lubricants.

6. Maintenance-free conditions

Some transmission parts do not need maintenance, or the maintenance frequency for some transmission parts should be reduced to save expenditure. In these conditions, it is rational and convenient to use solid lubricant.

(1) Unmanned and maintenance-free conditions.

For the supporting of large and medium bridges and transmission parts of large and heavy-duty equipment, it is difficult to maintain. In order to reduce maintenance expense, prolong the service life of machines, and extend the effective operating period under service-free conditions, solid lubricant can be used. For long-term storage and easy maintenance firearms and instantaneous products, solid lubricating dry films can be used.

(2) Frequent dissembling and maintenance-free condition.

When the fasteners, screws, and nuts, etc. are covered by solid lubricating dry films, they are easily loaded and unloaded; meanwhile, the fretting wear of fasteners can also be prevented.

1.1.3.4 The employed methods for solid lubricant

1. Usage as integral parts

As some engineering plastics, such as polytetrafluoroethylene, polyacetal, polyoxymethylene, polycarbonate, polyamide, polysulfone, polyimide, chlorinated polyether, polyphenylene sulfide, and poly p-phenylene dicarboxylic acid vinegar have low friction coefficient, good formation ability and chemical stability, excellent electric insulating property and strong shock resistance, they can be made into integral parts. Their comprehensive property will be better if these plastics are strengthened by glass fiber, metal fiber, graphite fiber and boron fiber, etc. These engineering plastics are widely applied to gears, bearings, cams, rolling bearing cages, etc.

2. Usage as composite materials or combination of materials

As the physical and chemical properties and shapes of solid lubricating materials are different and immiscible, the composite materials with better performances can be obtained by combining these solid lubricating materials.

3. Usage as solid lubricating powders

The load-carrying capacity and boundary lubricating conditions can be improved when proper quantities of solid lubrication powders (such as MoS_2) are added to lubricating oil and grease.

4. Usage as surface layers

Solid lubricants can be applied to form films or coatings with a certain self-lubricating properties on the friction surface. This is a commonly used method.