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Plasma-Assisted Physical Vapor Deposition Surface Treatments for Tribological Control

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PLASMA-ASSISTED PHYSICAL VAPOR DEPOSITION SURFACE TREATMENTS

FOR TRIBOLOGICAL CONTROL

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Abstract

In any mechanical or engineering system where contacting surfaces are in relative motion, adhesion, wear, and friction affect reliability and performance. With the advancement of space age transportation systems, the tribological requirements have dramatically increased. This is due to the optimized design, precision tolerance requirements, and high reliability expected for solid lubricating films in order to withstand hostile operating conditions (vacuum, high-low temperatures, high loads, and space radiation). For these problem areas the ion-assisted deposition/modification processes (plasma-based and ion beam techniques) offer the greatest potential for the synthesis of thin films and the tailoring of adherence and chemical and structural properties for optimized tribological performance. The purpose of this paper is to review the present practices and new approaches of applying soft solid lubricant and hard wear-resistant films to engineering substrates. The ion bombardment treatments have increased film adherence, lowered friction coefficients, and enhanced wear life of the solid lubricating films such as the dichalcogenides (MoS₂) and the soft metals (Au, Ag, Pb). Currently, sputtering is the preferred method of applying MoS₂ films; and ion plating, the soft metallic films. Ultralow friction coefficients (<0.01) have been achieved with sputtered MoS₂. Further, new diamond-like carbon and BN lubricating films are being developed by using the ionassisted deposition techniques.

Introduction

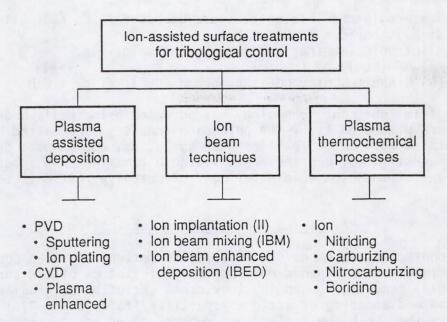
When contacting surfaces are in sliding, rolling, or oscillating motion, surface interactions through friction, wear, and adhesion control the performance and reliability of the functional mechanical or engineering system. In general, a mechanical or engineering system consists of three basic components: the material surfaces in contact; the lubricant; and the environment. The tribological requirements for improved performance, longer life, and high reliability have increased dramatically in line with the advanced operational systems. For instance, the many spacecraft and satellite moving mechanical assemblies and components (solar array drives, antenna pointing and control systems, despin mechanisms, and rack and pinion gears) have to operate under severe environmental conditions such as high-low temperatures, high vacuum, and nuclear or space radiation. In many of these spacecraft mechanisms, low-vapor-pressure oils and greases have been used. But there are numerous devices, such as optical devices or sliding electrical contacts, where liquid lubricants have to be avoided. The limiting factors influencing the use of fluids includes their possible decomposition/ outgassing and their viscosity variation with temperature. Further, most mechanism designs involving liquid lubrication require oil reservoirs, creep barriers, and molecular seals. These factors introduce added complexity to designs, when compared to solid lubricants.

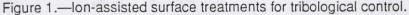
The advances in solid lubrication technology in the past two decades have been generated primarily by the demands to apply solid lubricating films to the tribomechanical components (bearings, gears, gimbals, splines, etc.). These precision triboelement surfaces, because of their optimized design and precision tolerances, require only very thin (0.2 to 0.4 μ m) adherent lubricating films. The plasma-assisted deposition processes (sputtering and ion plating) have offered great promise for satisfying the current needs in terms of long operational durability (5 to 10 years) and high reliability, and have been widely used and investigated.

Extensive research and development has been performed on sputtered MoS₂ and ion-plated Ag and Au Pb films (refs. 1 to 5). Sputtering and ion plating processes have not only produced thin, continuous, reproducible films, but also permit tailoring the structural, morphological, and chemical properties and improve film adhesion for tribological control. Recently an entirely new class of thin film materials, the diamond-like carbon films, have been discovered by utilizing the ion-assisted processes which use plasmas or hot filaments. These films have a potential to display outstanding lubricating and wear-resistant properties and are presently extensively studied. The objective of this paper is to summarize the state of the art of obtaining sputtered and ion-plated tribological films which display optimized lubricating properties and wear resistance for space applications (i.e., vacuum environment). When thin solid lubricating or wear-resistant films are used, it is essential to investigate and understand the process-structureproperty-performance interrelationships for optimized tribological control.

Ion-Assisted Surface Processes

The ion-assisted surface deposition/modification processes encompass a wide range of thin film growth methods, and they can be conveniently divided into plasma based, ion beam based, and plasma thermal based as shown in Figure 1. All the these processes depending on ion energy, ion flux, and the arrival rate ratio of ions to condensing atoms can be further classified into two distinct treatments as they modify the resultant surface. The process produces either distinct overlay coatings (plasma based) or forms no discrete coatings but modifies the surface without any dimensional change (ion beam and plasma thermal based) as shown in Figure 2. In all these processes,





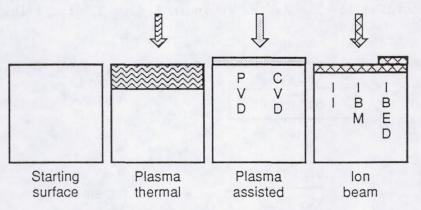


Figure 2.—Surface treatment technologies.

ions transfer energy, momentum, and charge by varying degrees depending on the deposition method. The resultant material properties can be selectively modified in terms of structure, morphology, and composition. In this paper, the process-property-performance interrelationships affecting the tribological behavior of surfaces deposited by the physical vapor deposition (PVD) techniques are addressed, since only these processes are presently used in space tribology.

Potential Solid Tribomaterials

Materials which are used to reduce friction and/or wear in a tribological contact are either solid lubricant films or hard, wear-resistant films. With the soft lubricating films, the aim is to confine friction losses to a thin film of low shear strength interposed between the contacting surfaces; whereas films affording protection against wear by virtue of their inherent high hardness and wear resistance are designated as hard films. Thus surface modification technology for tribological control can employ both soft and hard films. Some common potential materials used as tribological coatings can be classified as follows:

- (1) Solid lubricants
 - (a) Layer lattice compounds (dichalcogenides): MoS₂, WS₂, NbSe₂
 - (b) Soft metals: Au, Ag, Pb
 - (c) Inorganics: PbO, TiO₂, CaF₂, BaF₂

- (2) Wear-resistant intermetallics: carbides (TiC, WC), nitrides (TiN, Si₃N₄), borides (CrB₂, TiB₂)
- (3) Allotropic lubricant/wear-resistant materials:
 - (a) BN (hex), BN (cubic)
 - (b) Graphite, diamond-like carbon (DLC): i-c, α -C:H

The solid film lubricant technology has advanced principally because of the high demands placed on it by the aerospace industry. Sputtered MoS_2 films and ion-plated soft metallic films such as Au, Ag, and Pb have been widely used and investigated, and they have filled a great need in space tribology in the areas of momentum wheels for control-moment gyros, ball bearings, etc.

Principles of Solid Film Lubrication

When two contacting surfaces are in relative motion, or tribo-contact, the resulting interaction depends upon the characteristics of the surfaces, the environmental conditions, and the lubricant. Friction originates in the deformation and shearing of surface asperities (refs. 6 and 7). Adhesive wear occurs when both the surface and subsurface interact. According to the adhesion theory of friction in the absence of ploughing, the frictional force F is determined by the shear strength s and the real area of contact A according to F = As, as shown in Figure 3. For friction to be

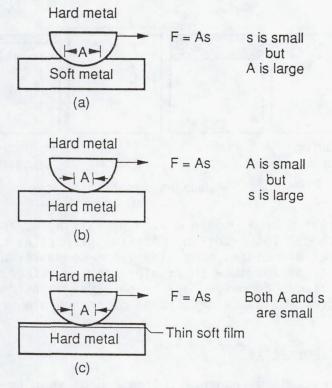


Figure 3.—Surface behavior of tribocontacts to metal hardness.

low, either A and s or both must be small. This means that the most suitable materials must have high hardness and low shear strength. However, this generally is not achievable with monolithic materials.

For instance, when a hard metal slides tangentially on a soft metal, the friction force is a function of a real area, which is largely due to deformation as shown in Figure 3(a). Where two hard surfaces are in contact (e.g., SiC against SiC) as shown in Figure 3(b), the friction force can increase because the shear strength can increase due to the higher surface

energy (higher interfacial strength) of the contacting metal. However, by using thin layers of soft, low-shear-strength solid materials on hard, very smooth surfaces, friction and usually wear as well can be reduced as shown in Figure 3(c). The real contact area in Figure 3(c) is determined by the hardness or elastic modulus of the substrate. The solid film lubricating mechanism is based on the thin, low-shear-strength film in combination with the high modulus/high hardness substrate. The combination of the low shear strength of the film and the small contact area produces a low friction. Since s is strictly the property of the lubricant, A depends on the deformation properties of the contact materials. If the contacts are elastically loaded, which is normally the situation in bearing technology, the contact area is given by the Hertzian equation

$$A = \pi \left(\frac{3 WR}{4E}\right)^{2/3}$$

where

R radius of sphere

- E effective elastic modulus
- W applied load

This last combination of a soft layer on a hard surface has been widely explored and used not only in metal-metal tribocontacts, but increasingly in studies of ceramic and other tribocontacts. These thin film lubrication concepts in practice are further discussed in this paper.

Solid Lubricant Coating Developments

Sputtered MoS₂ Films

The unique characteristic of ${\rm MoS}_2$ (natural molybdenite) is its highly anisotropic crystal layer structure. It is composed of "sandwich" layers each of which comprises a plane of Mo atoms arranged in hexagonal array situated between two hexagonal layers of S atoms as shown in Figure 4. The interlamellar (layer) attractions between the adjacent lamellae are weak and consist basically of weak van der Walls forces. However, the bonds between Mo and S atoms within the lamellae are covalent and, therefore, strong. As a result the weak interlamellar bonding contributes to the low shear strength during sliding, which in turn, is reflected in the low coefficient of friction and the excellent lubrication properties. Further, the anisotropic layer structure also exhibits an anisotropic crystal adsorption of water vapor or oxygen. The layer surfaces (basal planes) are basically inert to gas adsorption or interaction, whereas their edges are chemically reactive to oxidation (refs. 8 and 9). As a result, MoS_2 films are highly sensitive to moisture and oxidize easily. The deterioration of the films starts from the edge and progresses toward the center. This effect is accelerated if the films are maintained at high relative humidity. Therefore, MoS₂ films should not be used in tribo-systems which are exposed to high humidity. MoS₂ films exhibit their best lubrication under vacuum conditions and, therefore, they are of great interest and are the primary candidate materials for space tribology applications.

Optimized, sputtered, MoS_2 films display ultralow friction coefficients, 0.01 or less, under sliding conditions in vacuum. This represents an uncommonly low level of friction. In nature, such low friction values are only approached during rubbing of ice on ice at 0 °C, where a friction coefficient of 0.025 is observed (ref. 6). The tribological properties of sputtered MoS_2 films are extremely sensitive to the sputtering conditions.

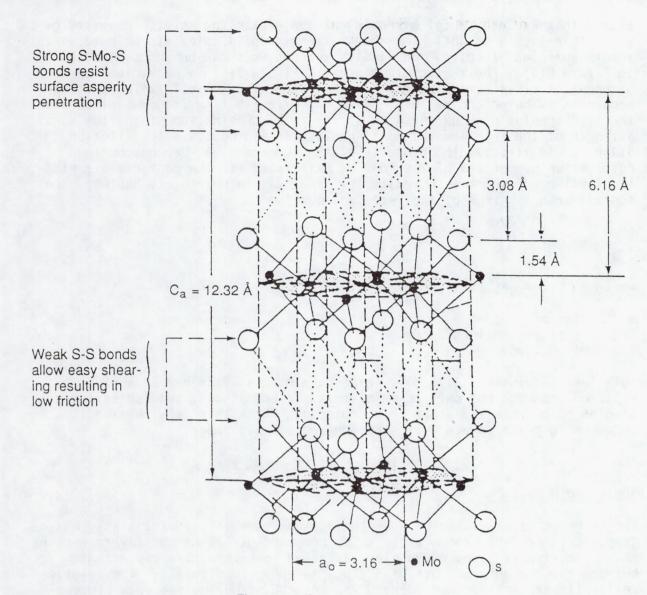


Figure 4.—Structure of MoS2.

Therefore, it is essential to establish, for a particular sputtering system, the optimized sputtering conditions, since film adherence, structure (morphology) and stoichiometry are directly interrelated with the tribological behavior. In thin film lubrication, it is important to understand the relationships between the sputtering conditions, the resultant film properties, and the friction and wear behavior.

Strong film adherence is the key to achieving extended endurance lives; therefore, the preparation of the substrate surface prior to film deposition has a major effect on the degree of adhesion. To some extent, it can also influence the nucleation and growth characteristics of the film, which determine the packing density of the columnar film structure. The most commonly used surface pretreatment prior to deposition is sputter-etch cleaning, which is accomplished by negatively biasing the substrate in the presence of the glow discharge for a preselected time. Sputtered MoS₂ films on 440C or 52100 bearing steel surfaces have been most widely investigated, and these substrates display an excellent adherence (refs. 10 and 11). Presently extensive studies are performed to investigate the interfacial modifications (chemical or mechanical bonding) which determine the degree of adherence. The structure of sputtered MoS_2 films can change from crystalline to amorphous by simply changing the substrate temperature during sputtering. It is generally known that the grain size of a thin film decreases by lowering the substrate temperature. Sputtered MoS_2 films at ambient or elevated temperature display the characteristic morphology of a dense, columnar crystallite microstructure with the basal planes of the crystallites perpendicular to the substrate but randomly oriented (refs. 12 and 13). However, when sputtered on cold substrates (7 °C or less) an amorphous structure is formed (ref. 14). These amorphous films are very brittle and do not display any lubricating properties but are essentially abrasive.

Upon sliding, the as-sputtered crystalline films, which have perpendicular basal plane orientation, reorient with the basal planes parallel to the sliding direction as shown in Figure 5. It is still unclear whether shear during sliding occurs between basal planes within crystallites or between the crystallites themselves. Other factors, such as film stoichiometry, can also affect tribological behavior.

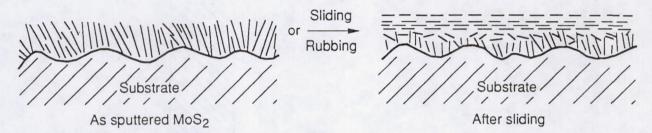
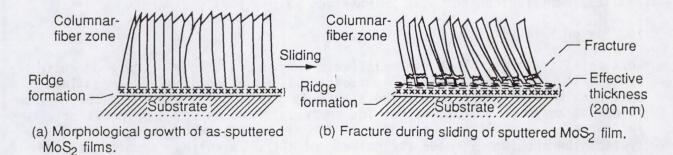
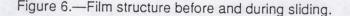


Figure 5.—Schematic of sputtered MoS₂ crystallite orientation before and after sliding.

The stoichiometry of sputtered MoS_2 films can vary widely from being sulfur deficient to sulfur rich (S/Mo = 1.1 to 2.2). It should be noted that the basic molybdenite structure prevails even for films with S to Mo ratios as low as 1.1 (ref. 15). Oxygen is normally the major contaminant in the sputtered films and can be present in a variety of chemical forms (refs. 16 and 17). The most commonly found MoO₃ phase is detrimental to film performance, in terms of both friction and endurance. Recently, it was suggested that oxygen can substitute for sulfur in the MoS₂ lattice, thus forming a solid-solution type of structure of $MoS_{2-x}O_x$ (refs. 18 and 19). It should be emphasized that the coefficient of friction is very sensitive to these stoichiometric changes. Numerous independent studies have shown that the optimized, sputtered MoS₂ films generally have a S to Mo ratio of 1.8.

In addition to the three critical material properties (adherence, structure, and stoichiometry), film thickness can also directly effect the tribological performance, primarily in undesirable debris generation This is of critical importance when coating the components (bearings, gears, and gimbals) for





precision space mechanisms. Previous studies (refs. 20 and 21) have shown that during sliding the crystallites deform easily and fracture. The separated film is wiped away, thus generating wear debris. A residual film, generally 0.2 μ m thick, is left behind; it has strong adherence and is essentially responsible for the lubrication. This film fracture mechanism is illustrated in Figure 6. Therefore, the deposited film thickness should be limited to a thickness where film fracture is minimized. This fracture is generally dependent on the morphology and packing density of the sputtered MoS₂ film.

It can be concluded that the anisotropy (structure) of MoS_2 films controls both shear behavior and reactivity of the film to the environment as shown by the changes in friction coefficient in Figure 7. Further, the ultralow

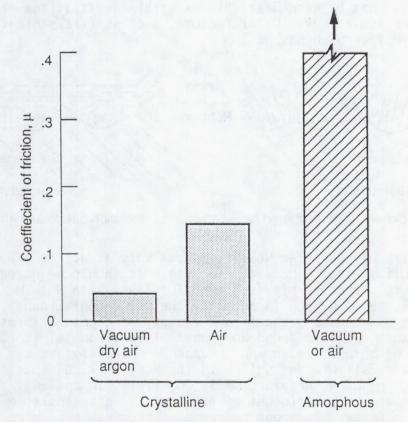


Figure 7.—Coefficient of friction of sputtered MoS₂ films 0.2 µm thick during sliding at ambient temperatures.

friction, which is responsible for optimized tribological control presently is acceptable over a friction range of 0.01 to 0.04 in vacuum and about 0.15 in humidity as shown in Figure 8. The lowest friction value of 0.007 has been derived for MoS_2 films sputtered on silicon nitride, since here the smallest Hertzian contact area is observed as previously discussed.

Ion-Plated Soft Metallic Films

Soft metallic films, which are relatively inert, can also provide good solid lubrication. The soft metallic lubricating films of gold, silver, and lead have been primarily applied by the simple dc-diode configuration in the thickness range of 0.2 μ m, either for spacecraft mechanical components or for terrestrial applications (refs. 22 to 24). In the basic ion plating system, the specimen is made the cathode of the high-voltage dc circuit; the evaporation source (resistance heating) is made the anode. It should be emphasized that in the basic ion plating system the ionization efficiency is

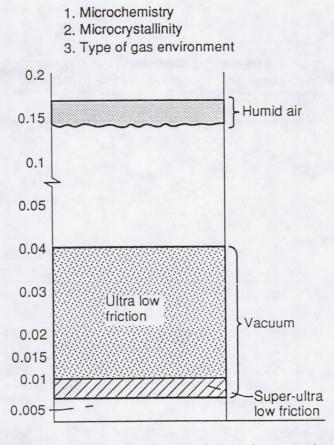


Figure 8.—Frictional variation of sputtered MoS₂ films.

not more than 0.1%. It has been estimated that typically less than 10% of the total energy arriving at the cathode is carried by ions and the 90% by energetic neutrals (ref. 25). For instance, when 2 kV is applied to the cathode and argon pressure is 10^{-2} torr, the average energy of the arriving particles is about 100 eV (ref. 25). The two important features of the process are (1) the flux of high-energy ions and neutrals, which causes exceptionally strong adherence between the film and the substrate and the favorable nucleation and growth sequence of the film and (2) the high throwing power, which provides the three-dimensional coverage to coat complex shapes.

The excellent adherence is caused mainly by atomistic mixing, which generates a graded interface (i.e., one in which there is a gradual transition between the properties of the substrate and the coating). This has been verified by using X-Ray Photoelectron Spectroscopy (XPS) depth profiling (ref. 26). The interface formation can also be identified by making micro-Vickers measurements. The microhardness of an ion-plated gold film, graded gold-nickel interface, and nickel substrate as a function of distance from the surface is shown in Figure 8(a). The gold was gradually removed by argon ion sputtering prior to the microhardness determinations. Initially, the hardness was relatively low in the gold film but gradually increased in the interface region and finally decreased again as the nickel substrate was reached. The higher hardness in the interface was due to alloying. The vapor-deposited gold film on nickel shown in Figure 9 exhibited constant hardness, which is indicative of a sharp interface.

The ion-plated metallic films also exhibit a distinct nucleation behavior as shown in Figure 10. The nuclei formed during ion plating exhibit these distinct characteristics: the nuclei are considerably smaller (150 Å) and have

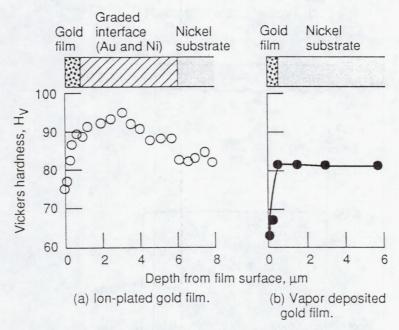
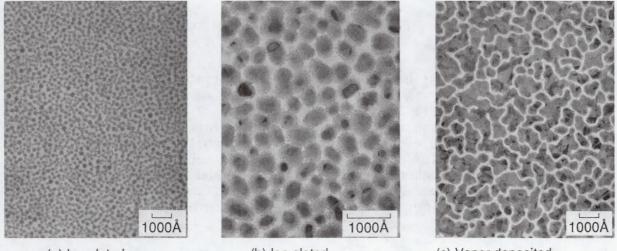


Figure 9.—Hardness depth profiles for gold ion plated and vapor deposited on nickel substrate. Hardness measuring load, 0.1 N.



(a) lon plated.

(b) lon plated.

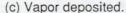
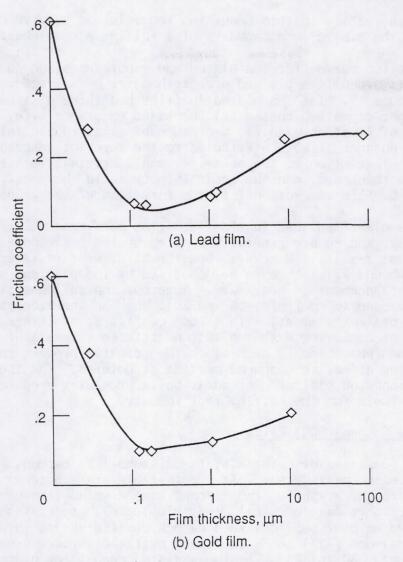
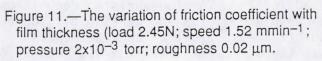


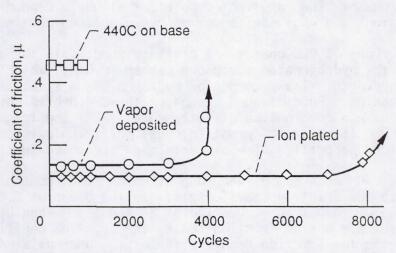
Figure 10.—TEM micrographs of lead during nucleation.

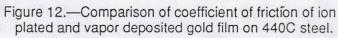
a high density and a uniform distribution. As deposition continues, the nuclei remain rounded with less than a 20% increase in size. Consequently, continuous films are formed in the 250 Å thickness range with uniform grain structure, high packing density, and minimum lattice misfit. It is, there-fore, clear why ion-plated metallic films display favorable morphological properties.

In thin film lubrication, as previously discussed, the film thickness has a very pronounced effect on the coefficient of friction as shown in Figure 11 for ion-plated Au and Pb films. The effective or minimum film thickness for Au and Pb films was about 2000 to 2500 Å with minimum coefficients of friction of 0.1 and 0 085, respectively (ref. 23). It has been suggested for Pb films that under Hertzian contact the as-deposited film flows plastically until a thin film (<100 Å) remains and then elastically deforms with the









substrate (ref. 27). In this condition the film can survive contact loads approaching the static load capacity of a rolling element bearing.

Typical friction curves for ion-plated and vapor-deposited Au films 2000 Å thick as determined in a pin and disk tribometer under vacuum conditions are shown in Figure 12. The ion-plated Au films had three distinct improvements over the vapor-deposited ones: (1) increased endurance life, (2) lower coefficient of friction, and (3) avoidance of catastrophic failure. The increased endurance life is attributed to the superior adherence, the lower coefficient of friction to the cohesive, small crystallite size and the optimum film thickness, and the gradual increase in the coefficient of friction.

It should be clarified that lubricating films made from relatively easily melted Au, Ag, and Pb are generally applied by the basic dc-diode discharge with excellent results. However, when the hard, wear-resistant intermetallic compound films have to be deposited by ion plating, some form of ionization enhancement is necessary. Numerous ionization-assisted process layouts have been tried (refs. 28 and 29), but the thermionically supported (resistance-heated filament) with triode configuration offers the most versatile means of achieving high ionization efficiency levels of about 10%. This enhanced ionization is crucial to the reactive process in synthesizing and depositing effective compound coating structures. The thermionically assisted triode ion plating configuration is presently used to produce TiN coatings on tools for the cutting tool industry.

Diamond-Like Carbon (DLC) Films

An entirely new class of materials, the diamond-like carbons (DLC), has been discovered in the past decade. As a metastable state between the naturally occurring carbons, graphite, and diamond, the diamond-like carbons display extraordinary physical, chemical, and tribological properties. DLC films can be formed on most surfaces when carbon containing gas (hydrocarbons) at low concentrations (<1%) in H₂ is flowed over the surface near an electric discharge (refs. 30 to 32). The ion-assisted deposition processes such as dc, rf, or microwave plasmas, low-energy (100 eV) ion beams, or thermionically emitting hot filaments provide the super thermal energy required to synthesize/deposit the DLC films. All the DLC deposition processes are characterized by the interactions of energetic ions with the surface of the growing film. The diamond-like carbon films can be completely amorphous or they can contain small portions of diamond or graphite crystals dispersed in the amorphous phase. The resultant properties strongly depend on the preparation conditions and upon the amount of hydrogen incorporated in the film.

Basically two types of DLC phases are distinguished: (1) the amorphous carbon (α -C) and the hydrogenated amorphous carbon (α -C:H), which can contain up to 30% of hydrogen. The presence of hydrogen has a stabilizing effect for the SP³ bonding. Pure diamond possesses the SP³ hybrid covalent bonds; the DLC films have a combination of tetrahedral (SP³) and trigonal (SP²) bonding. The DLC films are of great interest for tribological uses because of their combined properties of wear resistance and solid lubrication.

The DLC films (α -C and α -C:H) when deposited on steel and ceramic surfaces have shown very low friction coefficients: in the order of 0.1 in air (ref. 33) and 0.01 to 0.1 in vacuum (refs. 34 and 35). When DLC films are exposed to moist air during friction tests, the friction coefficient increases and the low-friction behavior is lost. Numerous studies to overcome the moisture degradation problem have been performed by modifying the deposition process parameters (ref. 33). Presently the synthesis/deposition of DLC films is very actively pursued in research laboratories, since these films are of crucial interest in the magnetic recording industry for magnetic storage devices. The triboproperties of these films are presently under development, and their potential use has not been widely verified.

BN Films

BN exists in a soft, lubricious hexagonal phase, similar to graphite, as well as the super-hard cubic phase, similar to diamond. The great interest in preparation of cubic-BN films stems from the fact that these films are second only to diamond in hardness. They are more oxidation resistant, because they form a thin, protective layer of boron oxide that is stable in vacuum to at least 1300 °C. In addition, cubic-BN has virtually the same lattice constant as diamond, making a highly compatible dual layer of the two materials an interesting possibility for investigation.

The BN films have been produced by a wide variety of the plasma-based and ion-beam-based processes, but generally most of the present depositions tend to produce amorphous or hexagonal phase films. However, it has been reported that the amorphous or hexagonal matrix also contains small crystallites of cubic-BN phase, but to produce these coatings with ultrahard adherent properties requires an innovative process technology.

The BN films display a low friction of 0.1 against 440C stainless steel and Si_3N_4 counterfaces (refs. 36 to 38). The friction coefficient generally increases with an increase in the B/N ratio and also with a higher hydrogen content. Presently it is essential to fully exploit the most suitable ion-assisted deposition process which will promote the cubic phase in the growing film.

Concluding Remarks

The improvements in the integrity and durability of solid lubricating and wear-resistant films and the manufacture of new classes of these films have been mainly performed by using the presently available and the newly emerging ion-assisted processes. The plasma-based and the ion-beam-based processes not only have produced thin adherent and uniform films, but are capable of tailoring interfaces, desirable crystalline/morphological structures, and compositional properties for improved tribological control. The potential of these ion processes offers great promise which has yet to be fully exploited. Presently, sputtering is the preferred method of applying MoS₂ films which display ultralow friction coefficients with long endurance lives when used in vacuum or dry air. The soft metallic lubricating films have achieved a high degree of maturity when applied by ion plating. The recent developments in the diamond-like carbon and BN films offers a new class of film materials whose tribological potentials are gaining great interest and experimental investigation, but their use has yet to be verified. Many of these thin film developments are highly relevant to space tribology.

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