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INFORMATION BRIEF

Title: A Review of Tribological Coatings for Control Drive Mechanisms for Space Reactors

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Summary: Tribological coatings must provide lubrication for moving components of the control drive mechanism for a space reactor and prevent seizing due to friction or diffusion welding to provide highly reliable and precise control of reflector position over the mission lifetime. Several coatings were evaluated based on tribological performance at elevated temperatures and in ultrahigh vacuum environments. Candidates with proven performance in the anticipated environment are limited primarily to disulfide materials. Irradiation data for these coatings is nonexistent. Compatibility issues between coating materials and structural components may require the use of barrier layers between the solid lubricant and structural components to prevent deleterious interactions. It would be advisable to consider possible lubricant interactions prior to down-selection of structural materials. A battery of tests was proposed to provide the necessary data for eventual solid lubricant/coating selection.

Introduction: The control drive mechanism for reflector sliders must provide highly reliable and precise control of reflector position in order to control the reactor power level over the mission lifetime. The control drive mechanism will be required to make fine adjustments to slider position intermittently following long, stationary periods. It is imperative that the power output (torque) required to move the sliders remains constant to ensure that precise control is maintained. Friction in the sliding components of the control drive mechanism could lead to increased loads on the control motors and imprecise or ineffective slider positioning. As a worst case, seizing of the mechanism due to static welding of metallic components could result in subcriticality and mission failure. Solid lubricant/anti-seize coatings will be necessary to provide highly reliable, low-friction operation of the control drive mechanism.

Table 1 details the critical factors affecting tribological coating selection. Tribological coatings for the control drive mechanism must provide reliable performance in a high temperature, ultra-high vacuum environment. The coating must be stable at elevated temperatures and must not volatilize in the vacuum of space. Solid lubricants that rely on oxide film formation or adsorbed species are not appropriate for space applications, given the lack of an atmosphere. The coating cannot react with the structural material. Reaction products may interfere with coating performance and degrade structural properties. Concern for interdiffusion between the substrate and coating may be important, especially in the hotter regions of the control drive mechanism. Interdiffusion of coating components may have deleterious effects on structural components and may degrade the tribological properties of the coating. Diffusion barriers between the structural material and coating may be necessary to prevent interdiffusion effects. The diffusion barrier would slow or negate interdiffusion between the coating and the substrate. Candidate diffusion barriers for tribological coatings will require further research should their application prove necessary.

The control drive mechanism will remain stationary for approximately one thousand hours between movements. The reflectors must move precisely when adjustments are required. Operating in an ultra-high vacuum at elevated temperatures (near 1000K) could cause diffusion welding. The coating cannot diffusion weld when stationary for long durations of time. Any degree of bonding would increase the amount of torque necessary to move the reflectors if they could, in fact, be broken loose and moved after self welding. The likely result would be an imprecise movement of the reflectors.

The tribological coating will be exposed to a significant neutron flux that could also contribute to degradation of the coating. The radiation environment could also lead to increased interdiffusion between the coating and substrate. The interdiffusion may lead to the formation of intermetallics phases that could embrittle the control drive materials. The potential for transmutation of coating components must be considered. Transmutation products may degrade coating performance or lead to compatibility issues with structural materials.

The vibrations and loads (fretting) that will be experienced at launch by the tribological coating also must be taken into account when choosing a coating. Performance over the intended life of the coating could be adversely affected by fretting occurring early in life. There most likely is no way to determine how the coating will be affected by these conditions without prototypical testing.

Table 1: Critical factors influencing tribological coating selection.

Critical Factors	Concerns
Tribological properties	Coefficient of friction Wear rates Coating life
Performance in space environment	Ultrahigh vacuum of space High temperature near reactor Radiation effects
Compatibility with control drive structural materials	Interdiffusion Reactivity
Resistance to interdiffusion and diffusion welding	Diffusion rates
Deposition of coating	Complex geometry High aspect ratio holes

Tribological Coatings Currently Used in Industry

Dichalcogenides

Dichalcogenides are used as solid lubricants and exhibit good life and low coefficients of friction. They are good candidates for space applications due to there properties under vacuum conditions and ability to operate at elevated temperatures. The dichalcogenides only decompose at temperatures above their maximum service temperatures. Table 2 presents a list of various dichalcogenides, their maximum service temperatures, and their decomposition temperatures.

MoS_2

Molybdenum disulfide is widely used as a solid lubricant in vacuum and inert gas atmospheres [1] and increasingly is being used for ball-bearings for space applications [2]. There has been a significant amount of research done on MoS_2 and its applications and properties are well understood. The properties and life of MoS_2 degrade quickly in air and humid atmospheres. It has been found that $MoSe_2$ has similar properties to MoS_2 but has better durability in air and humid atmospheres [1]. In a vacuum atmosphere MoS_2 can self heal from wear damage [3]. The tribological performance for MoS_2 is dependent on sputtering conditions when applied as a coating by sputtering. The surface roughness of the coating is reported to affect the durability of the MoS_2 [4].

The tribological properties of MoS_2 are very good in vacuum. The coefficient of friction has been measured at 0.01 or less [4]. However, if exposed to air prior to service in vacuum the coefficient increases during initial cycling and then self heals to an approximate coefficient of friction of 0.05. When spalling occurs, the coefficient of friction has been found to increase to approximately 0.36 [3]. The MoS_2 coating has a long life and low wear rates in ultra-high and normal vacuum [4]. The wear rates and life of the coating is dependent on the operating temperature.

Sulfide coatings may react with some structural materials at the intended operating temperatures in the Prometheus system. Both MoS_2 and WS_2 react with tantalum and beryllium to form stable sulfides at 900 K. The reaction of structural materials with sulfur could be detrimental to the properties of numerous structural materials of interest. The diffusion of sulfur into nickel base superalloys could substantially affect mechanical properties.

WS_2

Tungsten disulfide performance is similar to MoS_2 but it displays wider operating parameter windows than MoS_2. The oxidation resistance and thermal stability of WS_2 is superior to MoS_2, and the maximum operating temperature is about 100°C higher. The coefficient of friction for WS_2 is less than 0.1. The manufacturing and processing of WS_2 is critical since its properties are very process dependent. Laser annealing is recommended for WS_2 coatings. The scatter in the coefficient of friction values decreases, the wear life increases by a factor of ten, and the wear rate is decreased by 30 times after laser annealing of WS_2 [5].

Other Dichalcogenides

Selenide and telluride coatings tend to exhibit high vapor pressures under vacuum at the temperatures of interest. Both molybdenum and tungsten selenides have vapor pressures on the order of 10^{-3} atm at 900 K. For comparison, sulfides have vapor pressures on the order of 10^{-7} atm at comparable temperature. The higher vapor pressures of selenides will likely eliminate them from consideration for space applications. Tellurides do not have sufficient thermal stability to be used at 900 to 1000 K.

Table 2: Maximum Temperature for Dichalcogenides in Vacuum [20]

Material	**Maximum Service Temperature (K)**	**Thermal Decomposition Temperature (K)**
MoS_2	923	1203
WS_2	1003	1143
$MoSe_2$	1033	1033
WSe_2	1033	973
$MoTe_2$	813	973

PS304

PS304 is the trade name for a plasma sprayed coating. The coating is made up of four parts, each with a specific function. Sixty weight percent NiCr is used as a ductile binder. The next constituent is 20 weight percent Cr_2O_3 which is the constituent providing wear resistance. The low temperature lubricant is 10 weight percent Ag. The final component is 10 weight percent CaF_2/BaF_2 eutectic for high temperature lubrication [6].

PS304 is a transfer coating that needs to be "broken in" before the coefficient of friction reaches its ideal level [6]. The coating is made for applications in air and has not been tested under vacuum conditions, which makes this a concern for space applications. PS304 is made to operate up to temperatures of 1073K (800°C) [7]. The coating has been tested on various substrates and fails before the coating is "pulled off". Therefore, adhesion does not seem to be an issue with PS304 [8]. The adhesion strength will increase when heat treated [9]. A significant concern with the application of PS304 is consistently obtaining a satisfactory surface finish. The ideal surface roughness (Ra) is 0.05 to 0.1 μm [10], which must typically be attained by surface grinding.

The properties of PS304 have been characterized for in air applications and are temperature dependent. The coefficient of friction for PS304 ranges from 0.4 to 0.5 up to 900K (625°C) [11] and 0.23 to 0.31 at 1073K (800°C) [7]. The wear coefficient ($\times 10^{-4}$ mm^3/Nm) up to 900K (625°C) is 1.5 to 3 [11] and at 1073K (800°C) is 0.5 [7]. The life of PS304 is approximately 100,000 start/stop cycles up to 925K (650°C) [6].

The coefficient of thermal expansion for PS304 is 12×10^{-6} °C^{-1} [10]. It is unknown how the interdiffusion of coating components, especially silver and, perhaps, fluorides, will affect structural materials or if diffusion welding could occur during stationary periods since prior experience is largely limited to continuous sliding contact applications.

PS/PM 212

PM212 is a trade name for a powder metallurgy coating and PS212 is a plasma sprayed version of the same coating. The coating is made up of four parts like PS304 but has a different wear resistant hardener. The PM/PS 212 is made up of 70 weight percent metal bonded chromium carbide in which the metal binder is NiCr. The low temperature lubricant is 15 weight percent Ag. The final constituent is 15 weight percent CaF_2/BaF_2 which is the high temperature lubricant [12]. PS/PM 212 is a more manufacturing intensive coating than PS304 since it is harder to spray than PS304 [7]. A post-application heat treatment stabilizes the coating reducing the swelling. Coating performance has been characterized in air up to 700°C [13]. There is no data available for application of PM/PS 212 in vacuum at elevated temperatures.

Most properties of PM/PS 212 are similar to those of PS304. The wear rates of PM/PS 212 are reported to be comparable [7], and the coefficient of friction is dependent on substrate, load, and operating temperature, ranging from 0.25 to 0.60 [12]. The coefficient of thermal expansion for PM/PS 212 varies from 12.7 to 13.8 $\times10^{-6}$ °C^{-1}, slightly decreasing with temperature [13].

$CrC \bullet CaF_2$

The $CrC \bullet CaF_2$ tribological coating is a two layer coating that is used in air up to 700°C. The CrC is the bottom layer and CaF_2 is the top layer. When $CrC \bullet CaF_2$ coatings were tested against each other, chipping occurred in various regions of the coating. Also scaling occurs at the CaF_2 to CrC interface and tends to increase in relation to thickness. The scaling is due in part to thermal stresses [14]. There is no data, quantitative or qualitative, on $CrC \bullet CaF_2$ in vacuum. Testing under vacuum conditions would be required.

Tribaloy 700

Tribaloy 700 is the trade name for a tribological coating that is Ni based. The coating is a Ni-base hard facing alloy that has been used in liquid metal cooled reactors. The composition of the coating is 50 weight percent Ni, 32 weight percent Mo, 15 weight percent Cr, and 3 weight percent Si. This coating forms a low friction oxide in the presence of oxygen and its performance is dependent upon film formation and retention [15]. Obviously, a high vacuum environment could preclude maintenance fo the requisite oxide film.

Ni Titanate

Nickel titanate is a high temperature tribological coating that is used in air. The microstructure of the coatings tested by Taylor were nickel titanate crystals and rutile. The coatings are generally uniform, flat and show good adhesion to the substrate.

The current use of the Ni titanate coating is to decrease the coefficient of friction between TiC and Hastelloy. The coating shows good wear properties and has a coefficient of friction that is approximately 0.09 at 800°C. The coating is combinations of Ni and Ti particles implanted into Si nitride against TiC [16]. Nickel titanate has not been tested under vacuum conditions; therefore its performance in environments similar to space is unknown.

HfO_2

Hafnium oxide is a good tribological coating for use at temperatures up to 1550K. The coefficient of friction is significantly higher than the coatings previously discussed. The dynamic coefficient of friction is 1.5 and the static coefficient of friction ranges from 2 to 5.5. There are some self-welding issues with HfO_2 and the rate controlling mechanism for self-welding is plastic flow. Hafnium oxide was considered for use on the safety rod, safety rod thimble and reflector drive mechanism on SP-100 [17].

Diamond-Like Carbon (DLC)

Diamond like carbon is used in N_2 atmospheres up to 500°C. At temperatures above 500°C a gasification process occurs and the coating breaks down [18]. The wear rates for DLC are higher than those for MoS_2 [4]. The DLC coatings are amorphous; when used under vacuum conditions, the coatings failed immediately due to high friction [18]. DLC coatings require a chemically active atmosphere for low coefficients of friction. The low coefficient of friction arises from the surfaces being covered by adsorbed surface species [19].

Coating Deposition

The process used to deposit tribological coatings will depend on the type of coating selected. Dichalcogenide coatings (disulfide, etc.) can be deposited by burnishing, air spray application, physical vapor deposition (PVD), chemical vapor deposition (CVD), and inorganic or resin-bonded processes. Given that tenacious films are required, it seems likely that PVD or CVD would provide the best chances of successfully depositing a suitable quality film. Other coatings, particularly PS304 type and oxide coatings are deposited by plasma spraying or PVD. DLC can be deposited using a CVD process. Given the complex geometries and high aspect ratio holes present in the control drive design, it is likely that a non-line of sight process such as CVD would be most appropriate. CVD of disulfide coatings has been demonstrated; however, this is not a mature technology.

Testing

Extensive testing will be required to select coating materials and properties suitable for assurance of component performance over the mission lifetime. Testing will be needed to establish coefficient of friction, wear rate, diffusion for coating-substrate combinations and coating lifetime. In performing these tests, environmental conditions must also be considered. These considerations include high temperature, vacuum, and irradiation environments. Coatings must also withstand vibrational loading, i. e., fretting, during launch. Additionally, coating substrate combinations must be chosen with consideration of the long term performance required.

To achieve the required level of testing in the pre-launch time frame, several levels of testing will be needed. Basic tribological testing will be used to limit the number of possible coatings. This testing will establish base material properties and provide the knowledge needed to make informed preliminary selection decisions. Further component testing will be needed to determine coating quality (durability, uniformity, etc.) and accuracy of property models in usable component geometries. Final system level testing will be useful to verify coating application on complex component shapes and durability of the coatings under exposure to the mission conditions; however, time limitations may limit the extent of this type of testing.

Tribometer Testing

Basic tribometer testing will provide data on coefficient of friction, wear rates and lifetime (in regards to number of cycles). These tests can be modified to take into consideration certain environmental conditions including temperature and vacuum. Lifetime testing can be done very rapidly in tribometer test setups by using a high cycle rate. This type of lifetime testing fails to take into account the very low expected cycle rates or long times spent in static contact.

For this system a pin-on-disk test configuration would be recommended in order to most accurately simulate the sliding motion anticipated for the components. The pin-on-disk tribometer consists of a stationary pin with known diameter and hardness loaded against a rotating disk coated with the material under consideration. The frictional force displaces the pin to some degree, which can be measured and used to calculate the coefficient of friction [4]. Wear rates can be measured either by measuring the depth of any trough left by the pin or, given a large wear rate, by measuring a net decrease in the disk mass. Literature suggests that these systems can be modified to operate in elevated temperatures and vacuum regimes.

The pin-on-disk tribometer configuration allows for fast turnaround time when testing multiple materials in varying environmental conditions. Additionally, the tests require minimal sample preparation. The combination of these factors ensure that tribometer testing will be the lowest cost option for reducing the number of coatings that require further, more expensive testing.

Component Testing

Component testing would provide data on coating performance when deposited in the geometries necessary for the reactor. This testing would be necessary to ensure practicability of coating processes and of achieving coating uniformity that meets requirements. This type of testing would involve significantly more sample preparation and testing time, leading to a much higher cost per sample tested. For this reason it would be recommended only for testing of coatings previously identified by tribometer testing as having favorable properties for mission parameters.

Component tests typically use angular contact bearings and operate in vacuum. The tests can be performed at elevated temperatures as needed. Typically, component tests are not accelerated beyond normal system parameters, indicating long test durations necessary to accurately predict performance over the projected mission lifetime. This testing would allow for a much better prediction of actual coating performance under mission conditions but at a high cost in terms of both time and money.

Some additional component or system level testing will be needed to ensure that diffusion welding will not occur. To do this at least one mock-up of a slider and drive mechanism must be fully assembled and tested in a high-temperature, vacuum environment. The system must be held static for 1000 hours then moved a short distance. This type of activity will be typical of the demands on the sliders throughout the mission lifetime and assurance of its reliability is one of the main functions of the coatings in question. However, the expense of this type of testing based on both time and complexity will dictate that only a limited number of coatings can be tested.

Diffusion Testing

Diffusion testing will be required to ensure that the applied coatings will not diffuse into the substrate material or surrounding environment and adversely affect either the substrate or coating properties. These considerations are particularly important considering the elevated temperatures and long mission lifetime involved. Coatings considered include various sulfides, carbides, oxides, and metals (Ni, Cr, Ag, etc.). Significant impurities of sulfur, carbon, and oxygen could significantly degrade properties of the substrate materials under consideration. It is not clear what, if any, effects metallic impurities will have on structural components. For this reason it is especially critical that diffusion be taken into account when selecting wear coating materials.

Testing parameters need to be carefully chosen based on number of samples and complexity of testing setups. The key parameters that will need to be evaluated are test time and irradiation effects. Test duration must be considered carefully for such a long life mission. Early testing on a large number of samples should be limited to relatively short time; however as the number of possible materials decreases longer term testing will be required. This will provide critical data for predictions regarding diffusion over the life of the product. Irradiation testing should also be performed to evaluate the effect of irradiation on tribological performance. This should be initiated as early as practical in order to permit timely completion of post-irradiation examinations.

Diffusion couples would have been used to determine interdiffusion rates of coating materials into structural components and vice versa. Testing procedures were to be developed based on material selection and processing. Analysis of the diffusion couples was expected to be detailed in order to identify new phases formed, changes in volume fraction of various phases and diffusion distances. Appropriate analysis techniques would be used to determine the compositions of new phases, changes in compositions of the phases present before diffusion, and concentration gradients across the coating(s) and substrate where diffusion occurred. A microprobe or similar analysis might be preferred in order to obtain concentration profiles over the thickness of the coating and ascertain diffusion depths into the substrates.

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