PLASMA SPRAYED, SELF-LUBRICATING COATINGS FOR USE FROM CRYOGENIC TEMPERATURES TO 870°C (1600°F)

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This information is being published in preliminary form in order to expedite its early release.
A plasma-sprayed coating with good lubricating properties, over a wide temperature range is described. The coating, designated NASA LUBE PS101, contains silver, nichrome, calcium fluoride, and an oxidation protective glass. Oscillating tests of self-aligning, plain cylindrical bearings, in which the bore was lined with 0.025 cm (0.010 in.) thick coatings of PS101, were conducted at a radial load of $3.5 \times 10^7$ N/m$^2$ (5000 psi) in nitrogen gas at $-107^\circ$C ($-160^\circ$F), in vacuum at room temperature and in air from room temperature to $870^\circ$C ($1600^\circ$F). Friction coefficients were less than 0.25 in all cases and wear rates were low. The coating is not brittle, and it has adequate oxidation resistance in air to at least $870^\circ$C.

Sliding contact bearings, which are serviceable over a wide range of temperatures are needed in many areas of technology. A specific example is the need for airframe bearings, which can withstand an unusual combination of conditions, for use on the Space Shuttle Orbiter. The self-lubricating coating composition described in this paper was formulated with the requirements for the payload bay door bearings of the Orbiter in mind. These conditions are estimated to be:

1. Ground checkout - Oscillating, high load, atmospheric temperature and pressure.
2. Door operation in orbit - Oscillating, moderate load, vacuum, $-110^\circ$ to $+149^\circ$C ($-167^\circ$ to $+300^\circ$F) bearing temperature.
3. Reentry - Orbital conditions to sea level air and $760^\circ$C ($1400^\circ$F) bearing temperature.

Therefore the bearings must be operable under normal sea level atmospheric conditions and in vacuum from $-110^\circ$ to $149^\circ$C ($-167^\circ$ to $300^\circ$F) but, in addition, must be made of materials which are thermally and oxidatively stable in air at temperatures up to $760^\circ$C ($1400^\circ$F) if the bearing is to be reusable for future missions.
The types of bearing under consideration are plain cylindrical and plain spherical oscillating bearings. The sliding surfaces are lubricated with a composite liner material which contains a solid lubricant. A class of composite materials currently being evaluated by the Shuttle prime contractors contains the solid lubricant molybdenum disulfide (MoS$_2$). Certain of these MoS$_2$ composites appear to be suitable for ground checkout and orbital conditions but can be expected to oxidize in the hot, oxidizing environment during reentry. Therefore, the bearings may have to be replaced after every mission.

It was the objective of the research reported in this document to develop self-lubricating bearings which could be reusable for repeated missions of the Shuttle vehicle. This requires a lubricant which not only has good friction and wear properties in ground checkout and in orbit but also has good high-temperature oxidation resistance.

It was previously reported that composites containing the high-temperature solid lubricant calcium fluoride (CaF$_2$) and glass in a nichrome metal matrix were oxidation resistant and provided effective lubrication from 260° to 900°C (500° to 1650°F) (ref. 1). However, they were not especially good lubricants below 260°C (500°F). In the present work, silver additions to the CaF$_2$ composites were investigated as a possible means of improving low temperature friction and wear without causing severe adverse effects on the good high-temperature properties.

A promising material resulting from this research is a plasma-sprayed coating whose chemical composition in weight percent is 30 silver (Ag) - 30 nichrome (NiCr) - 25 calcium fluoride (CaF$_2$) - 15 sodium free glass. The coating is designated NASA-LUBE PS101, where PS indicates plasma-sprayed and 101 is an arbitrary code number to designate the composition.

Results obtained with this coating in oscillating bearings are reported. Conditions include moderate vacuum of 0.05 torr, atmospheric air, and temperatures from cryogenic to 870°C (1600°F).
COATING PREPARATION

The coatings were applied by co-deposition of mixed powders of silver (Ag), nichrome (NiCr), calcium fluoride (CaF$_2$), and glass with a plasma spray gun. Coating preparation consisted of three basic procedures: (1) preparation of powders for plasma spraying, (2) plasma spraying, and (3) machining and surface finishing operations.

Powder Preparation

Glasses have been used as oxidation protective coatings for metals (refs. 2, 3). The glass used in our composites was based on one of the formulations originally developed under a NASA contract (ref. 2) to explore protective coatings for gas turbine blades. The composition of the mill batch or starting material from which the glass used in our work was made, and the calculated final compositions of the glass are given in Table 1. The difference in mill batch and glass compositions are caused by the loss of carbon dioxide from the carbonates and water from the hydrated materials used during preparation of the glass.

The mill batch was melted in a nickel crucible at 1370°C (2500°F). About 10 minutes were allowed for complete decomposition of the carbonates and hydrated compounds. The melt was then poured into water to form readily pulverized, shotlike particles of glass frit. The frit was pebble-milled dry to a powder which would pass through a U.S. Standard sieve No. 120. (Particle sizes less than 125 micrometers.)

The powdered glass was then mixed with powdered nichrome metal, powdered silver, and with CaF$_2$ to the desired composition for plasma spraying. The composition of sprayed powder was by weight percent: 30Ag 30NiCr 25CaF$_2$ 15 glass.

Plasma Spraying Procedure

The substrate surfaces were grit blasted with coarse alumina grit. The composites were sprayed to a thickness of about 0.050 centimeters (0.020 in.) and subsequently machined back to a thickness of 0.025 centimeters (0.010 in.)

During spraying, argon was used as the carrier gas and the arc gas. An arc current of 350 amperes was used. The spray distance was maintained between
approximately 7.5 to 10 centimeters (3 to 4 in.) The positive displacement (roto-feed) powder feed was used to minimize the possibility of segregation of the mixed powders in transit from the powder hopper to the spray nozzle. No visible segregation of the powder occurred in the spray pattern. (When severe segregation occurs in a plasma spray pattern, it can be visually observed because of the difference in brightness of the flowing metal and ceramic powders as they emerge from the arc.) Semi-quantitative x-ray analyses were also performed on selected specimens to confirm that no substantial change in composition occurred during the coating process.

Machining the Plasma-Sprayed Coatings

The recommended machining procedure is very simple, but must be done correctly to prevent excessive smearing of the metal over the machined surface. It is very important that the surface areas occupied by the nonmetallic components of the composite are not diminished by metal smearing during machining. The following procedure has produced the best surfaces to date with the least amount of smeared and folded metal.

1. Machine dry
2. Use a single-point carbide tool
3. Machine at low speed of 9 to 12 M/min (30-40 ft/min)
4. Remove no more than 0.010 centimeter (0.004 in.) per cut

Post-machining Surface Treatments

Any machining smears that do occur can be removed and surface finish can be improved by wet sanding with sand paper progressing from 150 grit through at least two intermediate grades to 600 grit.

Test Bearings

The design of the test bearing is illustrated in Fig. 1. The general design is that of a rod end spherical bearing. However, in this program, the spherical element was not fastened to the journal but was allowed to float. The self-lubricating composite layer was plasma-sprayed on the bearing bore and one thrust surface. The coatings were machined to a thickness of 0.025 centimeter (0.010 in.), then sanded to remove machining marks.

All bearing elements (except the self-lubricating layer in the bore) were made of René 41, a precipitation hardening nickel alloy. The bearings were hardened
to Rockwell C-32. The lined bearing bore was 1.537 centimeters (0.605 in.)
diameter and 1.9 centimeters (0.75 in.) long. The spherical diameter was
2.92 centimeters (1.51 in.) The clearance between the journal and the com-
posite lined bore was 0.013 centimeters (0.005 in.), and the ball/outer race
clearance was 0.008 centimeter (0.003 in.)

BEARING TEST MACHINE

A drawing of the bearing test rig is given in Fig. 2. The test bearing is
mounted in an induction-heated metal plate; bearing temperatures of 900°C
(1650°F) can be readily achieved. The oscillating journal is taper-mounted
into a drive shaft which is supported at both ends by cylindrical roller
bearings. Radial load is applied to the test bearing by a pneumatic actuator.
Capability for axial loads is also provided but was not employed in this
program. The journal is oscillated by a reversible hydraulic actuator and
 crank assembly fastened to the rear of the drive shaft.

The test bearing chamber is enclosed by a large, stainless steel bellows
which allows: (1) air evacuation to about $10^{-2}$ torr with a mechanical vacuum
pump or (2) atmosphere control by purging with controlled humidity air or
other gas as required. Bearing temperatures as low as -107°C (-160°F) are
achieved by flowing cold nitrogen gas through the chamber. The gas is pre-
cooled by passage through copper tubing submerged in liquid nitrogen. Bearing
temperatures are measured by thermocouples and by infrared pyrometry.

BEARING TEST PROCEDURE

The tests consisted of beginning bearing oscillation under a nominal unit
radial load of $4.5 \times 10^5$ N/m² (65 psi) then increasing the unit load in $7 \times 10^6$
N/m² (1,000 psi) increments (2 minutes at each increment) up to the test load
of $3.5 \times 10^7$ N/m² (5,000 psi). Unit radial load is defined as the total radial
load per unit projected area of the bearing bore where projected area is
obtained by multiplying bore diameter by bore length. Journal oscillation was
$\pm 15^\circ$ at a frequency of 1 Hertz.
RESULTS

Bearing tests were run in the following environments: vacuum at room temperature, cold nitrogen gas, -107°C (-160°F), and air from 25° to 870°C (75° to 1600°F). Radial unit load unless otherwise noted was 3.5x10^7 N/m^2 (5,000 psi). Bearing performance in the various environments is described below.

Vacuum

The lowest friction was obtained in vacuum where the friction coefficient was typically 0.15. Friction coefficients and wear as a function of test duration are given in Fig. 3. Wear rates expressed as increase in bearing diametral clearance per oscillating cycle decreased with test duration. This is seen in Fig. 3(b) where increase in clearance (y) as a function of oscillating cycles (n) is plotted. A curve fit equation y = 2.3x10^-4 n^0.35 centimeters coincides with the experimental data. This extrapolated curve gives an increase in bearing clearance of only 4.5x10^-3 centimeters (1.8 milliinches) for 5,000 oscillating cycles.

Cold Nitrogen Gas

Friction and wear trends in cold nitrogen at a bearing temperature of -107°C (-160°F) are given in Fig. 4. Friction coefficients are in the range of 0.21 to 0.23. Increase in radial clearance was linear with test duration and yields a value of 3.8x10^-3 centimeter (1.5 milliinches) after 5,000 oscillating cycles.

Air, 25° to 870°C (75 to 1600°F)

Friction and wear at various temperatures in air are given in Fig. 5. Friction coefficients were in the range of 0.19 to 0.24 with the highest friction coefficient of 0.24 at room temperature. Wear after 5,000 oscillating cycles was in the range of 2.5x10^-3 to 7x10^-3 centimeters (1 to 2.8 milliinches) within the indicated temperature range. Data for all the bearing tests are summarized in Table II.

DISCUSSION

The plasma-spray coating NASA-LUBE PS101 described in this paper appears to have good self-lubricating characteristics under the atmospheric and temperature conditions forecast for the Shuttle bay door hinge bearings. This coating has a high metal content. Metallic silver provides good low temperature
friction and wear and nichrome metal enhances strength. Both metals lend ductility so that the composite coating is not as brittle as most other self-lubricating composites. The coating also contains the high-temperature solid lubricant calcium fluoride. This compound is not considered a good lubricant below 260°C (500°F); but, in combination with silver, lower friction and wear are obtained at low temperatures than with either material alone. A special sodium ion-free glass is also incorporated to enhance oxidation resistance of the composite coating.

PS101 has higher friction coefficients than MoS₂-based composites, but the friction coefficients are below 0.25 under all conditions tested. Advantages of PS101 are greater ductility than MoS₂ composites and good oxidation resistance in air to at least 870°C (1600°F).

CONCLUSION

NASA-LUBE PS101 is a plasma-sprayed, self-lubricating composite coating which has a combination of lubrication, strength, and oxidation-resistant properties that recommend it as a promising candidate for external airframe bearings on the Space Shuttle. More generally, the coating should be considered for any sliding contact application involving extreme conditions of temperature and reactive atmospheres.

REFERENCES

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<th>Initial Mill Batch Composition</th>
<th>Final Glass Composition</th>
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<tr>
<td>SiO$_2$</td>
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<td>BaO</td>
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<td>21.2</td>
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<td>9.5</td>
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<td>K$_2$CO$_3$</td>
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TABLE II

PERFORMANCE SUMMARY FOR OSCILLATING PLAIN SLIDING BEARINGS
SELF-LUBRICATED WITH A PLASMA-SPRAYED COATING IN VARIOUS ATMOSPHERES

PS101 COATING: 30Ag 30NiCr 25CaF$_2$ 15Glass
0.025 cm (0.010 in.) THICK
3.5x10$^7$ N/m$^2$ (5000 psi) UNIT LOAD, +15° OSCILLATION AT 1 HERTZ

<table>
<thead>
<tr>
<th>Bearing Temperature</th>
<th>Ambient Atmosphere</th>
<th>Typical Friction Coefficient</th>
<th>Increase in Radial Clearance cmx10$^3$, (milliinches)</th>
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<tr>
<td>°C  °F</td>
<td></td>
<td></td>
<td>After 100 Cycles After 5000 Cycles</td>
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<tr>
<td>room room</td>
<td>Vacuum 5x10$^{-2}$ Torr</td>
<td>0.15</td>
<td>1.3 (0.5) 4.5 (1.8)</td>
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<tr>
<td>-107 -160</td>
<td>Nitrogen</td>
<td>0.22</td>
<td>0.3 (0.1) 3.8 (1.5)</td>
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<tr>
<td>room room</td>
<td>Air 760 Torr</td>
<td>0.24</td>
<td>0.5 (0.2) 7.0 (2.8)</td>
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<tr>
<td>540 1000</td>
<td>&quot;</td>
<td>0.19</td>
<td>0.5 (0.2) 6.0 (2.4)</td>
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<tr>
<td>650 1200</td>
<td>&quot;</td>
<td>0.21</td>
<td>0.3 (0.1) 2.5 (1.0)</td>
</tr>
<tr>
<td>870 1600</td>
<td>&quot;</td>
<td>0.23</td>
<td>0.3 (0.1) 2.5 (1.0)</td>
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</table>
Fig. 3  Bearing friction and wear in vacuum of 5x10^-2 Torr and room temperature NASA-Lube coating. PS101, 0.035 cm (0.010 in) thick, 3.5x10^3 N/m^2 (5000 psi) load, 215° oscillation at 1 Hertz.
FIG 4 BEARING FRICTION AND WEAR AT -107°C (-160°F)
NASA LUBE PS 101 COATING .025 CM (0.10 IN) THICK
3.5 x 10^7 N/m² (5000 PSI) LOAD
± 15° OSCILLATION AT 1-HERTZ
FIG 5  BEARING FRICTION AND WEAR IN ROOM AIR AT VARIOUS TEMPERATURES
NASA-LUBE COATING, PS101, 0.025 CM (.010 IN) THICK, 3.5 x 10^3 N/m^2 (5000 PSI) LOAD
± 15° OSCILLATION AT 1 HERTZ