The Effect of Composition on the Surface Finish of PS400: A New High Temperature Solid Lubricant Coating

Christopher DellaCorte, Malcolm K. Stanford, Fransua Thomas, and Brian J. Edmonds
Glenn Research Center, Cleveland, Ohio

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Space Administration

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Abstract

A new composite, multi-constituent, solid lubricant coating, NASA PS400, developed for high temperature tribological applications, exhibits a smoother surface finish after grinding and polishing than its predecessors PS200 and PS300. In this paper, the baseline composition of PS400 is modified to investigate each individual constituent’s role on the achievable surface finish through a series of coating deposition, grinding, and polishing experiments. Furthermore, to explore the limits of compositional tailoring for improved tribological performance, several PS400 coatings were doped with additional solid lubricants (graphite, MoS2 and BN) and tribologically tested.

The test results clearly showed that, compared to PS300 coatings, PS400 achieves a smoother surface finish via a reduced lubricant content. Coatings prepared with higher than the baseline level (10 wt%) of lubricants exhibited higher final surface roughness than the earlier generation PS300 coatings. Reducing or eliminating one or both lubricants (fluorides or silver) did not further improve the surface finish suggesting that the current composition of PS400 is near optimal with respect to surface finish. Lastly, attempts to improve the poor initial room temperature tribological behavior of PS400 via the addition of traditional solid lubricants were unsuccessful. Based upon this work and earlier results it is expected that future research will concentrate on developing methods to produce a lubricious glaze on the rubbing surface during “break in” to ensure that low friction and wear are rapidly achieved.

Introduction

NASA has recently developed a new solid lubricant coating, PS400, as an outgrowth of earlier work that resulted in the PS100, PS200 and PS300 families of plasma sprayed coatings (Ref. 1). These four distinct families of coatings were engineered over the last four decades to address specific tribological challenges encountered in various aerospace systems.

The developer of the PS100 family of nickel-glass-solid lubricant-containing coatings pioneered the concept of combining the functions of individual constituents to produce a composite solid lubricant coating (Ref. 2). PS100 was originally formulated to lubricate lightly-loaded oscillatory bearings and bushings for re-entry flight vehicles, like the space shuttle. In this application, high temperature endurance and low friction were paramount. PS200 coatings, which utilized a hard nickel-cobalt-bonded chrome carbide matrix, were tailored for long life to lubricate piston ring-cylinder wall contacts found in Stirling and other heat engine applications (Ref. 4). The PS300 coating system was developed to fit between the earlier coatings in terms of hardness and was tailored specifically to meet the needs for shaft coatings operating against high temperature foil gas bearings (Ref. 4). The general compositions of these three coating families along with their general performance characteristics are given in Table 1.

Due to several drawbacks of PS300, namely the need to undergo a heat treatment for dimensional stabilization and poor initial surface finish, PS400 was invented. PS400 is similar to its sibling coatings in that fluoride and silver lubricants are combined with a hardened metal matrix. PS400, however, differs in that it relies on the matrix material, nickel-aluminum-molybdenum, to enhance oxidative and dimensional stability. Also, the solid lubricant content was lowered from 20 to 10 wt% to lower material costs. Greater detail of PS400’s formulation and baseline tribological performance has been previously reported (Ref. 1).
TABLE 1.—COMPARISON OF THE NASA PLASMA SPRAY (PS) COATING

<table>
<thead>
<tr>
<th>Coating designation</th>
<th>Binder matrix</th>
<th>Hardener</th>
<th>Solid lubricants</th>
<th>General attributes</th>
</tr>
</thead>
<tbody>
<tr>
<td>PS100</td>
<td>NiCr</td>
<td>Glass</td>
<td>Ag+ Fluorides</td>
<td>Soft-high wear</td>
</tr>
<tr>
<td>PS200</td>
<td>Ni-Co</td>
<td>Chrome Carbide</td>
<td>Ag+ Fluorides</td>
<td>Hard-low wear, (abrasive to counter face dimensionally stable)</td>
</tr>
<tr>
<td>PS300</td>
<td>NiCr</td>
<td>Chrome Oxide</td>
<td>Ag+ Fluorides</td>
<td>Moderate hardness, mildly abrasive to counter face, poor dimensional stability-requires heat treatment</td>
</tr>
<tr>
<td>PS400</td>
<td>NiMoAl</td>
<td>Chrome Oxide</td>
<td>Ag+ Fluorides</td>
<td>Excellent dimensional stability and surface finish, poor initial low temperature tribology</td>
</tr>
</tbody>
</table>

While researching PS400 it was observed that, in addition to its superior dimensional stability, it achieved a better surface finish compared to PS300. The reasons for this improvement were not known. Earlier research on PS300 aimed specifically at improving the initial surface finish had largely been unsuccessful (Refs. 5 and 6). Only through repeated, high-temperature start-stop sliding could PS300 surfaces become smooth (Ref. 7). The first part of this investigation seeks to explore and understand the reason or reasons for PS400’s superior surface finish. A series of PS400 coatings are prepared in which the major constituent phase composition and proportions are varied and the effect on measured surface finish is examined.

Previous tests with PS300 consistently showed that its friction and wear performance was deficient at room temperature when compared to conventional solid lubricants like graphite, molybdenum disulfide and similar materials. Further, if not previously operated (in sliding) at elevated temperatures during which time a lubricating surface glaze was formed, even markedly higher wear and friction at room temperature was observed. Such behavior was also noted in the first tests with PS400 (Ref. 1). One solution adopted for PS300 was to overlay a thin sacrificial film of solid lubricants onto the coating surface prior to first use (Ref. 5). Such sacrificial “break in” layers are commonly used in machinery systems.

The later part of this investigation seeks to improve the room temperature performance of PS400. To enhance the initial as-installed tribological performance of PS400, several compositional variations were prepared that contain conventional solid lubricants (BN, graphite and MoS2) at varying doping levels. In addition, several tests are done using thin overlay coatings of MoS2 to determine if their presence can reduce initial friction and wear. In the present work, surface roughness measurements were made using stylus and optical profilometry on coated stainless steel coupons. For the tribological evaluations coated disks are rubbed against superalloy pins in a pin-on-disk tribometer.

Materials

PS400 is a plasma sprayed, composite, solid lubricant coating that employs a nickel-molybdenum-aluminum binder to which chrome oxide (Cr2O3) is added as a hardening agent and silver and barium fluoride-calcium fluoride (BaF2/CaF2) eutectic are added as low and high-temperature solid lubricants, respectively. By weight, the baseline PS400 coating contains 70 percent binder, 20 percent hardening phase, and 5 percent each of the solid lubricant phases. The binder phase, by weight percent, contains 90 percent nickel and 5 percent each of Mo and Al. It is available as a commercial plasma spray feedstock and is often used by itself as a wear resistant, high temperature protective coating.

To elucidate the role of the various constituents and their relative proportions on the final achievable surface roughness, several model-coating compositions were formulated. These coatings are not intended or expected to be viable tribological coatings and are not tribologically tested. Rather, they are prepared as small (25 mm diameter, 6 mm thick) coupons coated by plasma spraying the following: a coating designated PS304-NiMoAl in which the NiCr binder normally used for PS300 is replaced by the binder used in PS400; PS400-NiCr in which NiCr replaces the NiMoAl binder normally used in PS400; PS400-A in which the fluoride content is removed; PS400-B in which the silver content is removed;
PS400-C in which the silver lubricant content is increased; and PS400-D in which the fluoride lubricant content is increased. In addition, three PS400 solid lubricant doped coatings were formulated and tribologically tested by adding between 2 and 4 wt% of boron nitride (BN), graphite (C) or molybdenum disulfide (MoS2) to reduce initial room temperature friction. These doped coatings were deposited onto 50 mm diameter, 12.5 mm thick superalloy test disks. Table 2 gives the composition of PS400 along with the variations formulated to determine their effects on surface roughness.

The following general steps are taken to prepare the PS400 and its variant coatings. First, powders of the individual components, as listed in Table 2, are weighed and mixed in a suitable container. The powders in the container are first hand mixed then poured into the rotary drum of a commercial plasma spray powder feeder. In this device, the rotating motion of the drum continuously mixes the powder blend before and during the spray deposition process. To deposit a coating, the powders are injected into an argon gas carrier stream and fed into a plasma spray gun. Inside the gun, an electric field ionizes inert gas to provide the high temperatures (~10,000 °K) needed to melt the particles. The molten particle-inert gas stream is projected onto the substrate where they form splats that solidify and adhere to form a coating. Many passes are made to build up a thick, dense coating approximately 300 μm thick. Table 3 gives the nominal plasma spray parameters used in this process.

The plasma spray process results in a rough surface that must be ground smooth to the desired coating thickness and dimensions.

Typical acceptable coating thicknesses range from about 200 to 400 μm. Thicker coatings can be made but often suffer from poor strength and residual stresses. Coatings thinner than 125 μm often lack

### TABLE 2—COATING COMPOSITION SUMMARY

<table>
<thead>
<tr>
<th>Coating designation</th>
<th>Binder (wt%)</th>
<th>Hardener (wt%)</th>
<th>Low temp lubricant, (wt%)</th>
<th>High temp lubricant, (wt%)</th>
<th>Additional solid lubricant, (wt%)</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>PS304 NiCr /60%</td>
<td>Cr2O3/20%</td>
<td>Ag/10%</td>
<td>Fluorides/10%</td>
<td>-----</td>
<td>Baseline</td>
<td></td>
</tr>
<tr>
<td>PS400 NiMoAl / 70%</td>
<td>Cr2O3/20%</td>
<td>Ag/5%</td>
<td>Fluorides/5%</td>
<td>-----</td>
<td>Baseline</td>
<td></td>
</tr>
<tr>
<td>PS304-NiMoAl NiMoAl /60%</td>
<td>Cr2O3/20%</td>
<td>Ag/10%</td>
<td>Fluorides/10%</td>
<td>-----</td>
<td>Binder substitution</td>
<td></td>
</tr>
<tr>
<td>PS400-NiCr NiCr /70%</td>
<td>Cr2O3/20%</td>
<td>Ag/5%</td>
<td>Fluorides/5%</td>
<td>-----</td>
<td>Binder substitution</td>
<td></td>
</tr>
<tr>
<td>PS400-A NiCr /68%</td>
<td>Cr2O3/17%</td>
<td>Ag/5%</td>
<td>Fluorides/10%</td>
<td>-----</td>
<td>Reduced silver</td>
<td></td>
</tr>
<tr>
<td>PS400-B NiMoAl /68%</td>
<td>Cr2O3/17%</td>
<td>Ag/10%</td>
<td>Fluorides/5%</td>
<td>-----</td>
<td>Reduced fluoride</td>
<td></td>
</tr>
<tr>
<td>PS400-C NiCr /68%</td>
<td>Cr2O3/22%</td>
<td>Ag/0%</td>
<td>Fluorides/10%</td>
<td>-----</td>
<td>Additional fluorides</td>
<td></td>
</tr>
<tr>
<td>PS400-D NiMoAl /68%</td>
<td>Cr2O3/22%</td>
<td>Ag/10%</td>
<td>Fluorides/0%</td>
<td>-----</td>
<td>Additional silver</td>
<td></td>
</tr>
<tr>
<td>PS400-BN NiMoAl /68.6%</td>
<td>Cr2O3/19.6%</td>
<td>Ag/4.9%</td>
<td>Fluorides/4.9%</td>
<td>BN/2%</td>
<td>Added BN</td>
<td></td>
</tr>
<tr>
<td>PS400-G NiMoAl /67.9%</td>
<td>Cr2O3/19.4%</td>
<td>Ag/4.85%</td>
<td>Fluorides/4.85%</td>
<td>C/3%</td>
<td>Added graphite</td>
<td></td>
</tr>
<tr>
<td>PS400-M NiMoAl/68.95%</td>
<td>Cr2O3/19.7%</td>
<td>Ag/4.93%</td>
<td>Fluorides/4.93%</td>
<td>M3S2/1.5%</td>
<td>Added M3S2</td>
<td></td>
</tr>
</tbody>
</table>

### TABLE 3—PLASMA SPRAY PARAMETERS

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Current</td>
<td>600 A</td>
</tr>
<tr>
<td>Voltage</td>
<td>30 to 32 V</td>
</tr>
<tr>
<td>Stand-off distance</td>
<td>80 to 100 mm</td>
</tr>
<tr>
<td>Argon arc gas flow</td>
<td>~35 standard l/min</td>
</tr>
<tr>
<td>Powder flow</td>
<td>~1 Kg/hr</td>
</tr>
<tr>
<td>Powder gas flow</td>
<td>~0.4 m³/hr</td>
</tr>
</tbody>
</table>

The following general steps are taken to prepare the PS400 and its variant coatings. First, powders of the individual components, as listed in Table 2, are weighed and mixed in a suitable container. The powders in the container are first hand mixed then poured into the rotary drum of a commercial plasma spray powder feeder. In this device, the rotating motion of the drum continuously mixes the powder blend before and during the spray deposition process. To deposit a coating, the powders are injected into an argon gas carrier stream and fed into a plasma spray gun. Inside the gun, an electric field ionizes inert gas to provide the high temperatures (~10,000 °K) needed to melt the particles. The molten particle-inert gas stream is projected onto the substrate where they form splats that solidify and adhere to form a coating. Many passes are made to build up a thick, dense coating approximately 300 μm thick. Table 3 gives the nominal plasma spray parameters used in this process.
adequate bond strength. Figure 1 shows a typical cross-section sample of a PS400 coating in which the splat-type morphology from the plasma spray process can be readily seen.

Grinding, using silicon carbide or diamond abrasive wheels, is usually the preferred finish process for PS400. Using standard grinding practices, surface root-mean-square (rms) finishes of about 0.25 μm are typical for PS400. Following machine grinding, selected coupons were further finished using varying grades of silicon carbide abrasive paper and water. Figure 2 shows a photograph of a turbine engine shaft that has been prepared for service with a ground and polished PS400 coating.

Figure 1.—PS400 cross-section control coating. Morphology is typical of plasma sprayed coatings and shows splat nature of constituent phases.

Figure 2.—Turbine engine shaft coated with PS400 after grinding.
Two types of coating specimens are fabricated for use in the present investigation; small round wafer coupons made from stainless steel and large superalloy test disks. These stainless steel wafers are 25 mm in diameter and 6 mm thick and are coated on one face with PS400 or one of the variants. These are utilized for the surface roughness measurements. The test disks are 63 mm in diameter and 12.5 mm thick and are made from Inconel X-750. These disks are coated on one face with PS400 which is ground to a smooth surface and a final coating thickness of about 250 μm. Several standard PS400-coated disk specimens were overlayed with a thin layer (~5 μm thick) of MoS₂ after grinding to reduce initial friction using the procedure outlined in Reference 5. For the pin-on-disk testing, hemispherical-tipped pins are made Inconel X-750. Figure 3 shows a sketch of the test rig and specimen configuration.

Test Apparatus and Procedure

Surface Roughness Measurements

The surface roughness coupons were evaluated primarily using stylus surface profilometry techniques. The wafer coupons were first plasma spray coated and then ground with SiC grinding wheels (400 grit) and polished using SiC abrasive paper and water (400 and 600 grit). The polished surface was subjected to repeated profilometry scans, 10 mm in length, at varying locations and directions across the entire coupon surface. Fifteen scans were obtained for each coated sample to get a representative roughness value. At least five coupons were prepared for each coating composition. The primary roughness parameter measured and recorded was the arithmetic mean, Ra, value. The scan speed was 0.5 mm/s giving a total scan time of 20 ces. This speed was found to be sufficient to minimize
measurement time while still preventing dynamic measurement problems such as stylus skipping. Typical variation in the roughness measurements of repeated samples was about 20 percent.

Optical profilometry was also used on selected samples but was found to give widely variable roughness results. When pits were part of the analyzed area, the roughness was inordinately skewed yielding much higher values not truly representative of the surface. With stylus profilometry, artifacts such as pits cause less effect on roughness because the relatively large probe tip radius (2 \( \mu \)m) glides over the voids effectively filtering them from the measurement. Only when the selected analysis area was free of deep pits (exposed porosity) did the optical roughness measurements correlate well with those from stylus profilometry. For these reasons, only stylus profilometry results are presented and used to compare coating roughness.

**Pin-On-Disk Tribotesting**

Figure 4 shows the high temperature pin-on-disk test rig is used to evaluate the friction and wear properties of PS400 and its variants. In this rig, coated disks were rotated against stationary metal pins to evaluate the effect of solid lubricant additives to the baseline PS400 coating. A hemispherical tipped superalloy pin is loaded against a coated disk under a 4.9 N dead weight load. Three disk-pin specimen sets are tested to assess repeatability. Each specimen set is run for a total of nine 30-min tests; three at 25 °C followed by three at 500 °C and then three at 650 °C in ambient air. Selected specimen sets were additionally tested at 25 °C following the standard sequence of nine tests to assess the possible formation of lubricious surface glazes during high-temperature sliding. An infrared pyrometer is used to measure disk surface temperature just ahead of the sliding contact and heating is accomplished using a low-frequency induction-heating coil located around the disk specimen. Sliding velocity is 3 m/s and the pin generates a 51 mm diameter wear track on the disk surface. Friction is measured using a load cell and wear is measured after each 30 min test using optical microscopy (pin wear) and stylus profilometry (coating wear) methods as described in the literature (Refs. 3 and 4).

![Figure 4](image.png)

Figure 4.—Typical surface roughness profile for ground PS400. Note occasional deep pits on surface from exposed voids and preferentially removed soft phases.
Results and Discussion

Surface Roughness Measurements

Table 4 shows the roughness measurement results from the stylus profilometry tests. Figure 4 shows a representative stylus roughness profile for PS400 and Figure 5 shows an area profile derived from optical profilometry measurements. Both methods reveal a fairly uniform surface texture with occasional deep pits typical for thermal spray coatings of this type. The roughness values show significant data scatter and overlap, yet deeper examination reveals some interesting trends and patterns.

<table>
<thead>
<tr>
<th>Coating designation</th>
<th>Hard phase content (wt%)</th>
<th>Soft phase content (wt%)</th>
<th>Surface roughness Ra (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PS304</td>
<td>80</td>
<td>20</td>
<td>0.61 ± 0.12</td>
</tr>
<tr>
<td>PS400</td>
<td>90</td>
<td>10</td>
<td>0.43 ± 0.08</td>
</tr>
<tr>
<td>PS304-NiMoAl</td>
<td>80</td>
<td>20</td>
<td>0.92 ± 0.18</td>
</tr>
<tr>
<td>PS400-NiCr</td>
<td>90</td>
<td>10</td>
<td>0.37 ± 0.07</td>
</tr>
<tr>
<td>PS400-A</td>
<td>85</td>
<td>15(5 Ag + 10 FL)</td>
<td>0.32 ± 0.06</td>
</tr>
<tr>
<td>PS400-B</td>
<td>85</td>
<td>15(5 FL + 10Ag)</td>
<td>0.37 ± 0.07</td>
</tr>
<tr>
<td>PS400-C</td>
<td>90</td>
<td>10 (Fl only)</td>
<td>0.30 ± 0.06</td>
</tr>
<tr>
<td>PS400-D</td>
<td>90</td>
<td>10 (Ag only)</td>
<td>0.24 ± 0.05</td>
</tr>
</tbody>
</table>

Figure 5.—Typical optical profile of ground and polished PS400 coatings showing deep voids and pits as dark regions.
For instance, the roughness measurements of the PS304 and PS400 coatings in which the matrix compositions have been reversed demonstrate that the matrix has little effect on the roughness. The roughness of PS304 and PS304-NiMoAl are high and similar, about 0.7 μm. Roughness of PS400 and its variant PS400-NiCr are also similar but lower, about 0.4 μm. Clearly the improved surface finish of PS400 compared to PS304 is not solely driven by the change in binder from NiCr to NiMoAl. When examining these four compositions (PS304, PS304-NiMoAl, PS400 and PS400-NiCr), however, one can observe a different correlation.

The two coatings with 20 percent soft phases (solid lubricant content) and 80 percent hard phases (binder and hardener content) have higher roughness than the coatings with 90 percent hard phases and only 10 percent soft phases. This suggests that the softer phases are preferentially removed during grinding and polishing leaving low spots, pits and depressions that contribute to a higher measured surface roughness. Such roughness can degrade performance in selected applications like gas bearings (Ref. 7). Furthermore, since the soft phases are solid lubricants, there is a loss in lubrication performance. Thus their absence or depletion leads to higher friction and wear during sliding until the wear process can remove the surrounding hard phases revealing fresh lubricants to the surface (Ref. 8).

The science and art of metallographic polishing is faced with preferential material removal challenges on a regular basis (Ref. 9). Many techniques are available that may improve the surface finish of the PS series of coatings such as oil-based lapping compounds, reduced polishing rates and the use of structurally-rigid lapping plates. These techniques, however, are not necessarily practical or possible for a manufactured coating for which contamination and costs must be minimized and complex shapes are often involved. To explore the effects of soft-phase concentration on surface finish, four alternative compositions were prepared PS400-A, PS400-B, PS400-C, and PS400-D.

PS400-A and PS400-B both contain 85 percent hard phases and 15 percent soft phases. The primary difference is that PS400-A has more fluoride than silver and PS400-B has more silver than fluoride. In terms of roughness, PS400-A achieved a slightly smoother finish than PS400-B though the data for the two coatings overlap considerably. This is not surprising since the fluorides and silver are soft and are susceptible to removal during finishing to a similar extent. The smoothest surfaces were achieved for the remaining two model coatings, PS400-C and PS400-D, which contained only fluoride or silver lubricants, respectively. The silver containing coating, PS400-D, achieved the lowest roughness of all, perhaps due to silver’s high ductility allowing it to resist micro-fracture type wear during polishing. However, it may also simply be experimental scatter since there is considerable data overlap.

The results of the surface finish experiments point to the existence and extent of soft phases as the cause for high finished surface roughness. Since the lubricants are needed to mitigate friction and wear their presence cannot be avoided. Efforts to develop more effective polishing techniques would be beneficial as would the infusion or overlaying of the finished surface with solid lubricants. Another approach is to add small quantities of highly effective solid lubricants to retain good tribological properties in the event that some soft phase depletion occurs during finishing. These approaches are addressed by the tribology experiments discussed below.

**Tribology Results**

Five coatings are evaluated, the baseline PS400, PS400 doped with several weight percent of BN, graphite or MoS₂ and one PS400 coating onto which a thin layer of pure MoS₂ was applied after finish grinding and polishing was completed. The intent of the pin-on-disk testing was to determine if room-temperature friction and wear behavior of the baseline PS400 coating could be enhanced through the addition of traditional solid lubricants. Table 5 displays the tribological results obtained in this research. The results clearly show that this approach is not successful.
TABLE 5.—FRICTION * AND WEAR ** SUMMARY

<table>
<thead>
<tr>
<th>Coating designation</th>
<th>Test temperature, °C</th>
<th>Friction Kpin</th>
<th>Friction Kdisk</th>
<th>Pin wear Kpin</th>
<th>Disk wear Kdisk</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>25 °C</td>
<td>500 °C</td>
<td>650 °C</td>
<td>25 °C</td>
<td>Retest</td>
</tr>
<tr>
<td>PS400</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.80</td>
<td>0.16</td>
<td>0.21</td>
<td>0.31</td>
<td>14.1×10⁻⁶ mm³/Nm</td>
</tr>
<tr>
<td></td>
<td>NM</td>
<td>0.21×10⁻⁶</td>
<td>0.89×10⁻⁶</td>
<td>118×10⁻⁶ mm³/Nm</td>
<td></td>
</tr>
<tr>
<td>PS400-BN</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.30</td>
<td>0.35</td>
<td>0.36</td>
<td>0.40</td>
<td>135×10⁻⁶ mm³/Nm</td>
</tr>
<tr>
<td></td>
<td>230</td>
<td>7.8</td>
<td>1.7</td>
<td>0.60</td>
<td>9.9×10⁻⁶ mm³/Nm</td>
</tr>
<tr>
<td>PS400-M</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.064</td>
<td>0.45</td>
<td>0.32</td>
<td>0.40</td>
<td>2.5×10⁻⁶ mm³/Nm</td>
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<tr>
<td></td>
<td>172×10⁻⁶</td>
<td>40.8</td>
<td>9.5</td>
<td>NM</td>
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<tr>
<td>PS400-MoS2</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>0.20***</td>
<td>0.40</td>
<td>0.22</td>
<td>0.40</td>
<td>2.5×10⁻⁶ mm³/Nm</td>
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<td></td>
<td>2.71</td>
<td>1703</td>
<td>2570×10⁻⁶</td>
<td>NM</td>
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</table>

Notes:
* Data scatter for friction typically ± 20 percent nominal
** Data scatter for wear (Kpin, Kdisk) typically ± 50 percent nominal; all values are ×10⁻⁶ mm³/Nm
*** Friction coefficient for PS400 – MoS₂ overlay represents first 10 min of sliding only

The baseline PS400 coating exhibits low friction coefficients (0.16 to 0.21), low pin wear coefficients (~10⁻⁶ mm³/Nm) and low coating wear coefficients (~7×10⁻⁶ mm³/Nm) at elevated temperatures. If room temperature testing commences before previous sliding at high temperatures, friction and wear are excessive. This has been attributed to the absence of lubricious glazing on the tribological surfaces that only form during high-temperature sliding. For this reason, the PS400 data at room temperature was collected after the high temperature tests were complete. That data shows a low friction coefficient (~0.3), low pin wear coefficient (~14×10⁻⁶ mm³/Nm) and a moderate coating wear coefficient (118×10⁻⁶ mm³/Nm).

The goal of the alternate composition studies discussed below was to improve the room temperature performance and provide low friction coating without the need to first run at high temperature. The addition of BN to PS400 caused degradation in performance in every respect. The friction coefficient at room temperature exceeded 1.0. Pin wear and coating wear increased by at least an order-of-magnitude and the wear couple could be described as abrasive. When the coating was tested following high temperature operation, friction was high (0.4), pin wear was excessive and disk wear could not be characterized due to the excessively rough nature of the wear track. In these tests, the addition of BN had deleterious effects on the tribological performance of PS400.

The addition of graphite to the PS400 coating also did not improve the performance. While not as damaging to the behavior of the baseline PS400 as BN, graphite was unsuccessful in significantly lowering friction and wear during initial room-temperature sliding. Friction was high (0.64) and pin and coating wear coefficients were both in the 10⁻⁴ mm³/Nm range. Friction and wear at elevated temperature suffered also, especially coating wear which ranged from 3×10⁻³ to 4×10⁻³ mm³/Nm at 500 and 650 °C, respectively. Pin wear at the highest test temperature, 650 °C, was negative indicating gross material transfer from the disk. Clearly, adding graphite to the PS400 coating was not an improvement.

MoS₂, considered among the best solid lubricants, was added to the PS400 coating in two ways. As a constituent, MoS₂ was not able to reduce room-temperature friction significantly from the baseline case (0.76 versus 0.80). Both pin wear and coating wear were in the moderate 10⁻⁵ mm³/Nm ranges. At 500 °C, friction was twice as high as the baseline coating and pin wear was about 4 times higher. Coating wear was excessive (10⁻³ mm³/Nm). At 650 °C, friction was comparable to the baseline case as was pin wear.
but coating wear remained high ($10^{-3}$ mm$^3$/Nm). During the room temperature re-test, following high-
temperature sliding, friction was moderate, and pin wear remained low ($10^{-6}$mm$^3$/Nm), but the coating
wear track became roughened and was not measurable (NM). These results show that adding MoS$_2$ as a
coating constituent is not an acceptable path to improved tribological performance.

As found previously with PS304 coatings for foil gas bearings, thin overlay coatings of MoS$_2$
deposited onto PS400 provided a temporary reduction in friction and wear. Testing was conducted only at
room temperature. The friction coefficient was low, about 0.20, and was comparable to that obtained for
the baseline coating at elevated temperatures. After 10 min of sliding, the friction gradually increased
eventually reaching about 0.4 by the end of the 30-min test period. Pin wear was low, $10^{-6}$mm$^3$/Nm,
and coating wear was too low to reliably measure for such a short test and is estimated to be below
$10^{-6}$mm$^3$/Nm. Subsequent tests at elevated temperatures yield performance comparable to baseline PS400
coatings indicating that the MoS$_2$ overlay is a benign, short-term solution that can function as a break-in
lubricant. This behavior corroborates experience with PS304 coatings and gas foil bearings in which the
overlay coatings are employed as a “one-time” use coating to prevent machine damage during initial
running (Ref. 7).

Summary Remarks

The goals of this research were to elucidate the factors affecting the achievable surface roughness
levels of the newly developed PS400 coating and to investigate methods to improve its friction and wear
performance especially during initial sliding contact at room temperatures. A series of coatings were
prepared at the coupon level specifically to determine surface roughness effects. Modified PS400
coatings, doped with different solid lubricants, were prepared on tribological test disks to evaluate the
potential for improved friction and wear performance. Based upon the results in this effort the following
conclusions are drawn:

- The improved final surface roughness of PS400, compared to its predecessor PS304, is largely
due to the reduction in soft phase content, the solid lubricants silver and fluorides. Altering the
coating matrix has no significant effect on surface finish.
- Standard grinding and polishing techniques appear to preferentially remove the softer lubricant
phases at the finished surface resulting in high surface roughness and possibly high initial friction
and wear. Enhanced techniques for finishing are needed to reduce roughness and improve initial
tribological performance.
- The addition of solid lubricants graphite, BN and MoS$_2$ did not improve any of the tribological
properties of PS400. In general, observed friction and wear increased dramatically when these
solid lubricants were added to the coating.
- Overlay coatings of MoS$_2$ sprayed over the PS400 surface provided low friction and wear for a
limited duration. This behavior corroborates earlier experience with such overlay coatings used
on foil bearing shafts. Such an approach is likely an engineering solution to using PS400 in
engines and other machinery.

References

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The Effect of Composition on the Surface Finish of PS400: A New High Temperature Solid Lubricant Coating

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A new composite, multi-constituent, solid lubricant coating, NASA PS400, developed for high temperature tribological applications, exhibits a smoother surface finish after grinding and polishing than its predecessors PS200 and PS300. In this paper, the baseline composition of PS400 is modified to investigate each individual constituent’s role on the achievable surface finish through a series of coating deposition, grinding, and polishing experiments. Furthermore, to explore the limits of compositional tailoring for improved tribological performance, several PS400 coatings were doped with additional solid lubricants (graphite, MoS2 and BN) and tribologically tested. The test results clearly showed that, compared to PS300 coatings, PS400 achieves a smoother surface finish via a reduced lubricant content. Coatings prepared with higher than the baseline level (10 wt%) of lubricants exhibited higher final surface roughness than the earlier generation PS300 coatings. Reducing or eliminating the one or both lubricants (fluorides or silver) did not further improve the surface finish suggesting that the current composition of PS400 is near optimal with respect to surface finish. Lastly, attempts to improve the poor initial room temperature tribological behavior of PS400 via the addition of traditional solid lubricants were unsuccessful. Based upon this work and earlier results it is expected that future research will concentrate on developing methods to produce a lubricious glaze on the rubbing surface during “break in” to ensure that low friction and wear are rapidly achieved.