INVESTIGATION of THERMAL PROCESSING on the PROPERTIES of PS304, a SOLID LUBRICANT COATING

Summary of Research Report

Patricia A. Benoy, Ph.D. P.I.

6/15/00 – 6/14/01

St. Louis University
3450 Lindell Blvd.
St. Louis, MO 63103

Grant # NAG3-2469
The effect of thermal processing on PS304, a solid lubricant coating, was investigated. PS304 is a plasma sprayed solid lubricant consisting of 10% Ag and 10% BaF₂ and CaF₂ in a eutectic mixture for low and high temperature lubricity respectively. In addition, PS304 contains 20% Cr₂O₃ for increased hardness and 60% NiCr which acts as a binder. All percents are in terms of weight not volume. Previous research on thermal processing (NAG3-2245) of PS304 revealed that substrate affected both the pre and post anneal hardness of the plasma spray coating. The objective of this grant was to both quantify this effect and determine whether the root cause was an artifact of the substrate or an actual difference in hardness due to interaction between the substrate and the coating. In addition to clarifying past research developments new data was sought in terms of coating growth due to annealing.

The current research project has shown that the apparent post anneal hardness is actually reflecting to a certain degree the hardness of the underlying substrate in the as sprayed condition. Samples spray coated with varying thickness from 0.05” in to 0.20” were hardness tested in this round of research. The thicker the coating the less substrate affect was seen in both the pre and post anneal samples. The hardness appears to approach an equilibrium value in the pre-anneal condition independent of substrate, fig. 1. The results in the post-anneal condition are not quite as clear with apparent coating hardness increasing with increasing thickness on the SS304 while doing the opposite on X750, fig. 1. This anomalous behavior may be due to the coating growth described below. Post-anneal we are probably seeing a combination of the coating hardness, the substrate hardness and the growth layer hardness. This will be investigated utilizing a micro-hardness tester in the next round of testing. All current hardness measurements were made on the Rockwell B scale.

Coating growth was found to be a function of substrate, time at temperature and coating thickness, figs 2 and 3. It is postulated that a diffusion reaction mechanism is occurring. On a substrate of Inconel X750 as the coating thickness increases the % coating growth decreases for all thermal processes. On Stainless 304 substrates the % coating growth is increasing approximately linearly with respect to coating thickness until the annealing temperature is raised to 1200F. At 1200F the coating growth initially increases with coating thickness then falls off as the thickness is increased further. It would appear that there is a minimum temperature for reactant (Oxygen?) diffusion and a maximum diffusion depth based on time at temperature. Additional investigation into these phenomena will also be done during the next round of testing.

In addition to the previously described results thermal processing effects on adhesion were also investigated. Results of this research are contained in NASA Tech Memo 2001-210944. The abstract of this report is attached.

The hardness and coating growth data are being consolidated into a paper that will be submitted for publication in Tribology Transactions and presentation at the STLE Annual Meeting in 2002.

No inventions were developed under the auspices of this research grant.
% Increase in coating thickness

Nominal coating thickness (inches)

Fig. 2 Coating growth on stainless 304
Fig. 3 Coating Growth on X750
<table>
<thead>
<tr>
<th>4. TITLE AND SUBTITLE</th>
<th>Thermal Processing Effects on the Adhesive Strength of PS304 High Temperature Solid Lubricant Coatings</th>
</tr>
</thead>
<tbody>
<tr>
<td>6. AUTHOR(S)</td>
<td>Christopher DellaCorte, Brian J. Edmonds, and Patricia A. Benoy</td>
</tr>
</tbody>
</table>
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) | National Aeronautics and Space Administration  
John H. Glenn Research Center at Lewis Field  
Cleveland, Ohio 44135–3191 |
| 8. PERFORMING ORGANIZATION REPORT NUMBER | E-12798                                                                                     |
| 9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) | National Aeronautics and Space Administration  
Washington, DC 20546-0001 |
| 10. SPONSORING/MONITORING AGENCY REPORT NUMBER | NASA TM—2001-210944                                                                        |
| 11. SUPPLEMENTARY NOTES | Christopher DellaCorte and Brian J. Edmonds, NASA Glenn Research Center; and Patricia A. Benoy, St. Louis University, St. Louis, Missouri 63103. Responsible person, Christopher DellaCorte, organization code 5960, 216–433–6056. |
| 12a. DISTRIBUTION/AVAILABILITY STATEMENT | Unclassified - Unlimited  
Subject Category: 23  
Distribution: Nonstandard  
Available electronically at http://gtrs.nasa.gov/GTRIS  
This publication is available from the NASA Center for AeroSpace Information, 301–621–0390. |
| 12b. DISTRIBUTION CODE |                                                                                               |
| 13. ABSTRACT (Maximum 200 words) | In this paper the effects of post deposition heat treatments on the cohesive and adhesive strength properties of PS304, a plasma sprayed nickel-chrome based, high temperature solid lubricant coating deposited on stainless steel, are studied. Plasma sprayed deposition coating samples were exposed in air at temperatures from 432 to 650 °C for up to 500 hr to promote residual stress relief, enhance particle to particle bonding and increase coating to substrate bond strength. Coating pull off strength was measured using a commercial adhesion tester that utilizes 13 mm diameter aluminum pull studs attached to the coating substrate interface. Pull off force was automatically recorded and converted to coating pull off strength. As deposited coating samples were tested as a baseline. The as-deposited (untreated) samples either delaminated at the coating-substrate interface or failed internally (cohesive failure) at about 17 MPa. Samples heat treated at temperatures above 540 °C for 100 hr or at 600 °C or above for more than 24 hr exhibited strengths above 31 MPa, nearly a two fold increase. Coating failure occurred inside the body of the coating (cohesive failure) for nearly all of the heat-treated samples and only occasionally at the coating-substrate interface (adhesive failure). Metallographic analyses of heat-treated coatings indicate that the Nickel-Chromium binder in the PS304 appears to have segregated into two phases, a high nickel matrix phase and a high chromium precipitated phase. Analysis of the precipitates indicates the presence of silicon, a constituent of a flow enhancing additive in the commercial NiCr powder. The exact nature and structure of the precipitate phase is not known. This microstructural change is believed to be partially responsible for the coating strength increase. Diffusion bonding between particles may also be playing a role. Increasing the heat treatment temperature, exposure time or both accelerate the heat treatment process. Preliminary measurements indicate that the heat treatment also results in a one time, permanent coating thickness increase of about 3 percent. Based upon these results, the incorporation of a heat treatment prior to final finishing has been incorporated in the application process of this coating technology. |
| 14. SUBJECT TERMS | Coatings; Lubrication; Solid lubricants |
| 15. NUMBER OF PAGES | 17 |
| 16. PRICE CODE | 298-102 |

**ABSTRACT**

In this paper the effects of post deposition heat treatments on the cohesive and adhesive strength properties of PS304, a plasma sprayed nickel-chrome based, high temperature solid lubricant coating deposited on stainless steel, are studied. Plasma sprayed deposition coating samples were exposed in air at temperatures from 432 to 650 °C for up to 500 hr to promote residual stress relief, enhance particle to particle bonding and increase coating to substrate bond strength. Coating pull off strength was measured using a commercial adhesion tester that utilizes 13 mm diameter aluminum pull studs attached to the coating substrate interface. Pull off force was automatically recorded and converted to coating pull off strength. As deposited coating samples were tested as a baseline. The as-deposited (untreated) samples either delaminated at the coating-substrate interface or failed internally (cohesive failure) at about 17 MPa. Samples heat treated at temperatures above 540 °C for 100 hr or at 600 °C or above for more than 24 hr exhibited strengths above 31 MPa, nearly a two fold increase. Coating failure occurred inside the body of the coating (cohesive failure) for nearly all of the heat-treated samples and only occasionally at the coating-substrate interface (adhesive failure). Metallographic analyses of heat-treated coatings indicate that the Nickel-Chromium binder in the PS304 appears to have segregated into two phases, a high nickel matrix phase and a high chromium precipitated phase. Analysis of the precipitates indicates the presence of silicon, a constituent of a flow enhancing additive in the commercial NiCr powder. The exact nature and structure of the precipitate phase is not known. This microstructural change is believed to be partially responsible for the coating strength increase. Diffusion bonding between particles may also be playing a role. Increasing the heat treatment temperature, exposure time or both accelerate the heat treatment process. Preliminary measurements indicate that the heat treatment also results in a one time, permanent coating thickness increase of about 3 percent. Based upon these results, the incorporation of a heat treatment prior to final finishing has been incorporated in the application process of this coating technology.
NASA GRANTEE
NEW TECHNOLOGY SUMMARY REPORT

NASA requires each research grantee, research contractor, and research subcontractor to report new technology to the NASA Technology Utilization Office. The required reports and corresponding schedules are as follows:

<table>
<thead>
<tr>
<th>Title of Report</th>
<th>Form Number</th>
<th>Timetable</th>
</tr>
</thead>
<tbody>
<tr>
<td>New Technology Disclosure</td>
<td>NASA 1679</td>
<td>The grantee discloses each discovery of new technology individually, at the time of its discovery.</td>
</tr>
<tr>
<td>NASA Grantee New Technology Summary Report (checkmarked “Interim”)</td>
<td>NASA C-3043</td>
<td>For multi-year grants, the grantee summarizes the previous year's disclosures on an annual basis. The first Interim New Technology Summary Report is due exactly 12 months from the effective date of the grant.</td>
</tr>
</tbody>
</table>

Grantee Name and Address

Report Submitted by: Patricia A. Benoy
Telephone Number: (812) 372 - 8448

NASA Grant Title: Investigation of Thermal Processing on the Properties of PS 304, a Solilo Warrant Coating
NASA Grant Number: NASA 3-2469
NASA Grant Monitor: J. C. Frank Williams
Grant Completion Date: 6/14/01
Today's Date: 1/15/02

New technology may be either patentable or non-patentable. NASA defines a new technology item as any invention or discovery conceived or first reduced to practice during the performance of a NASA grant, contract, or subcontract; items must be disclosed as they are discovered.

Although grantees are not required to disclose non-patentable new technology, all disclosed NON-PATENTABLE and PATENTABLE new technology items are automatically evaluated for publication as NASA Tech Briefs. If an item is selected for publication as a NASA Tech Brief, a $350 check payable to the grantee innovator is awarded.

PLEASE COMPLETE THE REVERSE SIDE OF THIS FORM AND MAIL TO THE FOLLOWING ADDRESS:

NASA Lewis Research Center
Attn: Kathy Kerrigan
Commercial Technology Office; Mail Stop 7-3
Cleveland, Ohio 44135

NASA C-3043 (6-98) Page 1 of 2
I. General Information

1. Type of Report: ( ) Interim ( ) Final

2. Size of Business: ( ) Small ( ) Large ( X ) Nonprofit Organization

3. Have any nonpatentable new technology items resulted from work performed under this grant during this reporting period? ( ) yes ( ) no

4. Have any patentable new technology items resulted from work performed under this grant during this reporting period? ( ) yes ( ) no

5. Are new technology items (nonpatentable or patentable) being disclosed with this report? ( ) yes ( X ) no

II. New Technology Items

Please provide the title(s) of all new and previously disclosed new technology items conceived or first reduced to practice under this grant.

<table>
<thead>
<tr>
<th>Title</th>
<th>Internal Docket Number</th>
<th>Patent Appl. Filed</th>
<th>Patentable Item</th>
<th>Nonpatentable Item</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

III. Subcontractors

Please complete the following section listing all research subcontractors participating to date. Include each subcontractor's name, address, contact person, and telephone number.

________________________________________________________________________
________________________________________________________________________
________________________________________________________________________
________________________________________________________________________
________________________________________________________________________

IV. Certification

I certify that active and effective procedures ensuring prompt identification and timely disclosures of reportable new technology items have been followed. Furthermore, I certify that all new technology items required to be disclosed and conceived during the period identified on this form, have been disclosed to NASA.

[Signature]

Name and Title of Authorized Official

[Signature]

Signature and Date

NASA C-3043 (6-98) Page 2 of 2