Tribological and Microstructural Comparison of HIPped PM212 and PM212/Au Self-Lubricating Composites

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SUMMARY

The feasibility of replacing the silver with the volumetric equivalent of gold in the chromium carbide-based self-lubricating composite PM212 (70 wt% NiCo-Cr3C2, 15% Ag, 15% BaF2/CaF2 eutectic) was studied. The new composite, PM212/Au has the following composition: 62 wt% NiCo-Cr3C2, 25% Au, 13% BaF2/CaF2 eutectic. The silver was replaced with gold to minimize the potential reactivity of the composite with possible environmental contaminants such as sulfur. The composites were fabricated by hot isostatic pressing (HIPping) and machined into pin specimens. The pins were slid against nickel-based superalloy disks. Sliding velocities ranged from 0.27 to 10.0 m/sec and temperatures from 25 to 900 °C. Friction coefficients ranged from 0.25 to 0.40 and wear factors for the pin and disk were typically low 10^-5 mm^3/N-m. HIPped PM212 measured fully dense, whereas PM212/Au had 15 percent residual porosity. Examination of the microstructures with optical and scanning electron microscopy revealed the presence of pores in PM212/Au that were not present in PM212. Though the exact reason for the residual porosity in PM212/Au was not determined, it may be due to particle morphology differences between the gold and silver and their effect on powder metallurgy processing.

INTRODUCTION

The need for systems which provide low friction and wear over a wide temperature spectrum has led to the development of the plasma sprayed composite coating series PS200 (refs. 1 to 3) and the family of powder metallurgy composites PM200 (refs. 4 and 5). PS/PM200 composites are comprised of a wear resistant metal (Ni, Co) bonded chromium carbide (Cr3C2) matrix with the solid lubricants silver and barium fluoride/calcium fluoride (BaF2/CaF2) eutectic. Silver is present as a low temperature lubricant (ref. 6). The BaF2/CaF2 eutectic functions as the high temperature lubricant (ref. 7). The composite materials function by replenishing the sliding contact surfaces with lubricants as wear occurs. In doing so, a film containing the lubricants is formed at the sliding interface. This has been verified by the detection of silver in films formed on superalloys that were slid against the composites (ref. 1). Potential applications for these materials include cryogenic process control valves, variable temperature control surface bearings, high temperature combustion engine cylinder wall coatings, and turbine engine seals and bushings.

The silver in PS/PM200 imposes certain limitations on the use of the composite. Silver has the lowest melting point of the constituents of the composite (961 °C). Thus, the material cannot be used much above 900 °C. Additionally, silver combines directly with sulfur (a constituent typically found in

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petroleum derived fuels) to form silver sulfide, even at low temperature. The possibility for stress corrosion cracking of superalloys due to sulfidation attack is a concern.

PM212 is a specific composite in the PM200 family which contains 70 wt% NiCo-Cr$_3$C$_2$, 15% Ag, 15% BaF$_2$/CaF$_2$ eutectic. It has been shown to provide low friction and wear in sliding against superalloys. Since intended applications of PM212 are in turbine engines which use superalloys, it is desirable to have a composite which does not contain an easily sulfided element, such as silver. Therefore, this study investigates the feasibility of replacing the silver in PM212 with the volumetric equivalent of gold. The new composite is designated PM212/Au and has the following composition: 62 wt% NiCo-Cr$_3$C$_2$, 25% Au, 13% BaF$_2$/CaF$_2$ eutectic.

Gold in its metallic state does not combine directly with sulfur (ref. 8). Thus, the potential for sulfide formation with the low temperature lubricant in the composite is eliminated. In addition, by replacing the silver with gold (MP = 1063 °C), the eutectic has the lowest melting point (1050 °C). Hence, the temperature limit for the composite may be higher than 900 °C for limited periods.

In this paper, the tribological and microstructural properties of two self-lubricating powder metallurgy composites, the original silver-containing PM212 and the gold-containing PM212/Au, are examined. PM212 and PM212/Au are processed using a cold isostatic press (CIP) followed by a hot isostatic press (HIP). This processing route yields parts of higher strength and density, in comparison to a CIP followed by sintering (ref. 5). Hemispherically tipped pins fabricated from the composites are slid against nickel-based superalloy disks in a pin-on-disk tribometer with temperatures ranging from 25 to 900 °C and sliding speeds from 0.27 to 10 m/sec. Scanning electron microscopy, energy dispersive x-ray spectroscopy, optical microscopy, and density measurement are used to compare and characterize the microstructure of the two composites.

**MATERIALS AND PROCESSING**

PM212 powder is made by blending three components; metal bonded Cr$_3$C$_2$, BaF$_2$/CaF$_2$ eutectic and silver. PM212/Au is identical in composition to PM212 except that an equal volume of gold is substituted for the silver. The metal bonded Cr$_3$C$_2$, the gold, and the silver powders are commercially available. The BaF$_2$/CaF$_2$ eutectic is prepared by mixing and prefusing the individual powders in 62/38 wt% proportions. Following the prefusing step (1100 °C in a nitrogen atmosphere) the eutectic is crushed, ball milled, and sieved to the particle size range listed in table I. The PM212 and PM212/Au powder batches are made by weighing out the proper proportions (table I) of the individual components and then mixing in a Vee type blender.

Slugs of the composites are then produced using the following steps:

1. Die-press into cylindrical blanks
2. CIP in rubber molds:
   • 5 min at 414 MPa (60 ksi)
3. Place CIPped slugs in stainless steel cans
4. Vacuum anneal (350 °C), then seal can under vacuum
5. HIP in stainless steel cans:
   • 20 min at 138 MPa (20 ksi) and 1100 °C
   • pressurization-depressurization rates: 0.75 MPa/min-1.5 MPa/min
   • heating-cooling rates: 6 °C/min-12 °C/min

Complete processing details and descriptions of the techniques used can be found in reference 5.

The superalloy used for the test disks is René 41. René 41 is a precipitation hardened nickel-cobalt superalloy with a nominal hardness of $R_e$ 35 to 40 at room temperature. The weight percent composition of the major components of René 41 is as follows: 55% Ni, 19% Cr, 10% Mo, 10% Co, 3% Ti.

APPARATUS AND PROCEDURE

Specimen Machining and Preparation

Hemispherically tipped pins are machined from the HIPped slugs. The pins have a cylindrical diameter of 9.5 mm, a nominal length of 26.0 mm, with 4.76 mm hemispherically radiused ends. Final dimensions are achieved by diamond grinding the slugs using water as a coolant. Machining oils are not used so that contamination of the composite is minimized. The René 41 disk specimens are 12.7 mm thick with an outside diameter of 63.5 mm. The disks are ground and lapped to an average surface roughness of approximately 400 Å CLA.

The composite pins are heated in an oven for 3 hr at 200 °C and 700 mm Hg vacuum to remove any residues from the processing and machining operations. The pins and disks are then cleaned with ethyl alcohol, wet scrubbed with 0.1 μm grit size alumina powder, rinsed with distilled water, and dried with clean compressed air.

Metallographic cross sections of the two composites were prepared by mounting the composites in a polymer followed by diamond sawing and polishing for microscopic examination. In addition, blocks of the PM212/Au composite were fabricated to obtain density measurements by weight and volume measure.

Tribological Testing

The friction and wear behavior of PM212 and PM212/Au pins sliding against René 41 superalloy disks was determined using the pin-on-disk configuration. The test load was 4.9 N and the atmosphere was air with relative humidity of 30 to 70 percent at 25 °C. The 4.9 N load was chosen to facilitate comparison with previous work (ref. 4). The sliding velocity was unidirectional from 0.27 to 10.0 m/sec and the temperature range was 25 to 900 °C. Table II lists the test combinations (speed-temperature-time) performed for this study.

Additionally, two studies were performed to determine run-in effects on wear at the 2.7 m/sec sliding velocity. For PM212/Au, a series of brief tests were performed at 25 °C and 2.7 m/sec. The durations were 1 and 10 min, with two tests performed at each condition. New specimens were used for each test. Pin wear and disk wear were measured for comparison with longer duration tests. For PM212, pin wear was measured after 10, 30, and 70 min for the same set of specimens. This procedure was performed at 25, 350, and 760 °C. A new set of specimens was used for each temperature.
Two different pin-on-disk tribometers were used for this study. One which uses an induction coil for specimen heating was used to perform the 0.27 and 2.7 m/sec tests. The surface temperature of the disk specimen is measured with an infrared pyrometer and a continuous readout of the friction force is made on a chart recorder from a temperature compensated strain gage bridge transducer. The second tribometer, used for the 10.0 m/sec tests uses a resistance heated furnace. This rig is fully described in reference 9. A computerized data acquisition system monitors test conditions, such as load force, friction force, and temperature at several locations in the furnace. The friction force is also recorded on a chart recorder for comparison to the computer acquired data.

Both tribometers generate 51 mm diameter wear tracks on the rotating disk while holding the pin in a fixed position. After the test run, a photomicrograph is taken of the pin wear scar to determine the volume removed. A surface profilometer is used to measure the wear track on the disk. Typically, four measurements of the wear track area are averaged. The area of the wear track is multiplied by the track circumference to determine the disk wear volume. Figure 1 shows a pin wear scar and a disk wear track profile scan for one of the tests performed.

The average coefficient of friction, $\mu$, for each test run is determined by averaging a sampling of data points recorded throughout the test. A sample standard deviation about this average, $s(\mu)$ represents the typical scatter that was present in the test. When five or more tests were run at similar conditions, an overall average coefficient of friction is computed, and the standard deviation about the overall average represents the test to test variation.

Wear factors were computed for both the pin ($K_{\text{pin}}$), and the disk ($K_{\text{disk}}$). Where several tests were performed, an average wear factor was determined with a standard deviation representing the test to test scatter. Wear factors are detailed in several of the references (refs. 1, 2, and 4). Briefly, the wear factor is equal to the volume removed due to wear during sliding divided by the normal load and the total sliding distance. Wear factors greater than $10^{-4}\text{mm}^3/\text{N-m}$ are considered high and those less than $10^{-6}\text{mm}^3/\text{N-m}$ are considered very low.

Wear surface morphology was examined on the pins and disks after testing using optical microscopy and scanning electron microscopy (SEM) in select cases.

**Microstructural Analysis**

Metallographically polished surfaces of the two composites were examined using optical microscopy, SEM, and energy dispersive x-ray spectroscopy (EDS). The EDS system is an integrated part of the SEM. The analyses help to compare and characterize the microstructures based on elemental distribution and surface topography.

Settings used on the SEM were 20 kV accelerating voltage, 0.6 nA probe current, and 39 mm (15 mm for backscatter) working distance. The backscattering mode of the SEM was used to illustrate the elemental distribution differences. Brighter areas correspond to higher atomic number elements. Key elements were identified using x-ray mapping with the EDS system. All SEM work was performed at an original magnification of 500X. The secondary electron image (SEI) micrographs from the SEM are a close representation of an optical view. Optical micrographs were obtained using vertical illumination on a metallographic microscope at two magnifications (original: 50X, 400X).

The density of PM212/Au was estimated based on measured dimensions of the machined blocks and their respective masses. Measured density was compared to theoretical density to determine the porosity
level present after processing. Density and porosity levels of PM212 have previously been established using liquid porosimetry and volume techniques (ref. 5). In addition, a sample of the gold powder used was metallographically prepared for analysis. The section was investigated using optical microscopy and SEM to determine morphology and structure.

RESULTS

Tribological

Table III summarizes the friction coefficients and the pin and disk wear factors for the composites at the speed-temperature combinations studied. For the 2.7 m/sec speed, five or six tests at each speed-temperature combination were performed. Thus, the friction coefficients and wear factors represent the overall averages.

Figure 2 contains plots of friction coefficient versus temperature for the three test speeds. Error bars (one standard deviation in each direction) for PM212 at the 2.7 m/sec speed are based on the test to test variation, whereas all others are based on the variation within a single test. The dashed lines are present to suggest a trend and aid in visualizing the relationship between the data points. Tests were not performed at intermediate temperatures. The friction coefficient was typically between 0.25 and 0.4 (fig. 2). PM212 at 0.27 m/sec and 25 °C was the exception with a friction coefficient of 0.5. In several cases, the friction coefficients of the two composites were within data scatter of each other. The largest test to test variation was 0.08 whereas variations within a single test were typically on the order of 0.03.

Plots of $K_{\text{pin}}$ versus temperature for the three test speeds are found in figure 3. The wear factor is plotted on a log scale. The pin wear behavior for the two composites were similar to each other except at the 10.0 and 2.7 m/sec tests where at higher temperature, the pin wear for PM212/Au was notably higher than that of PM212. The pin wear was typically in the low $10^{-5}$mm$^3$/N-m range, with a few cases in the low $10^{-6}$mm$^3$/N-m range. $K_{\text{pin}}$ for PM212/Au at elevated temperature-higher speed conditions was high $10^{-5}$mm$^3$/N-m. A notable reduction in $K_{\text{pin}}$ occurred at 760 °C, 0.27 m/sec speed to low $10^{-6}$mm$^3$/N-m (fig. 3(c)).

Several cases produced a "positive" disk wear. That is, a transfer from the pin to the disk caused a buildup on the disk. Table III indicates at which cases this occurred by the negative sign. $K_{\text{disk}}$ was low $10^{-5}$mm$^3$/N-m or less except for the 0.27 m/sec tests where the disk wear exceeded $1\times10^{-4}$mm$^3$/N-m in a few cases.

The results of the run-in study for PM212 are plotted in figure 4(a). At 25 and 350 °C, the pin wear factors were nearly constant at 1 to $2\times10^{-5}$mm$^3$/N-m. $K_{\text{pin}}$ decreased at 760 °C from $1\times10^{-6}$mm$^3$/N-m at 20 min to $5\times10^{-7}$mm$^3$/N-m at 70 min. This indicated that the run-in process was essentially completed within the first 10 min. Figure 4(b) shows that higher wear (both pin and disk) occurred in the initial sliding for PM212/Au. The 1 min test revealed a wear factor of approximately $1\times10^{-4}$mm$^3$/N-m for both the pin and disk. The 10 min test revealed wear factors in the low $10^{-5}$mm$^3$/N-m range. The pin and disk wear factors further decrease as the 70 min data points illustrate. A definite run-in mode with higher wear was observable with the 1 min test. This run-in was also observable in the friction behavior. Tests typically began at an elevated friction which decreased within 2 min.
Microstructural

The theoretical densities of PM212 and PM212/Au are 6.61 and 7.48 g/cm$^3$, respectively. PM212 achieves near full density, with a remaining porosity of 0.2 percent. PM212/Au achieved a density of 6.34 g/cm$^3$, with a remaining porosity of 15.2 percent.

Figure 5 contains micrographs obtained with the SEM. The backscatter images present an elemental view of the composites. To identify the major components, x-ray mappings of nickel, chromium, barium/calcium, silver for PM212, and gold for PM212/Au were obtained. The mappings show a similar distribution of the major components for the two composites.

Figure 6 contains low magnification optical and high magnification SEM (secondary electron image, SEI) micrographs of the two composites. The main observation regarding the optical micrographs is that PM212/Au contains a greater percent of dark area. It has previously been observed (ref. 4) that dark area represents eutectic all pore space under vertical illumination. Distinct pores in the PM212/Au microstructure are clear in the SEM image. The distinct pores in PM212/Au were discernable due to the depth of field possible with the SEM. Since the two composites had the same volumetric percentage of eutectic, PM212/Au possesses more pore space than PM212. An examination of PM212 at higher magnification revealed only minute pores in comparison to those observed at higher magnification in PM212/Au. At higher magnification on the optical microscope, the gold appeared as an interconnected, fine web-like structure whereas the silver appears in distinct pockets. A subtle shade difference between the gold and the carbide matrix made reproduction of the micrograph for illustration impractical.

The gold powder particle cross sections were examined for structure and morphology. They were found to be solid as seen from the cross section micrograph in figure 7. Figure 7 also includes samples of the starting silver and gold powders. It is noted that the predominant size of the silver was near the high end of the sieve range ($\approx 150 \mu$m) whereas the predominant size of the gold was near the low end ($\approx 50 \mu$m).

DISCUSSION

From the pin-on-disk test results, it is clear that the friction and wear characteristics of the two composites were similar. In addition, both composites provided low friction and wear over the range of speeds and temperatures studied. It is also noted that the behavior of the composites in sliding against René 41 was exceptionally steady after a brief run-in period lasting less than 2 min.

Several tests were run with PM212 at the 2.7 m/sec speed. It was observed that the test to test variation of the friction coefficient was typically twice as large as the variation within a single test. In several cases, the performance of the two composites were within data scatter. In general, the steady state performance of the two composites exhibited a friction coefficient of 0.3 and a wear factor in the low $10^{-5}$mm$^3$/N-m range.

An unusually high friction coefficient of 0.5 for PM212 at 0.27 m/sec and 25 °C was measured. It has been observed that brief run in periods (1500 m) at elevated speeds (2.7 m/sec) often exhibit lower friction coefficients ($\approx 0.3$) at low sliding velocities.

High friction and wear were typically encountered during a brief run-in period at the beginning of the test. This run in period typically lasted less than 2 min. Near steady state conditions were achieved
in 10 min as shown by the run-in study for PM212/Au. The high friction and wear during run-in is attributable to the high contact stresses associated with a hemisphere on flat, and the time required to develop a lubricating surface film at the contact. Previous work with PS200 coatings have also exhibited a run-in effect (ref. 10).

Typically the composite wear lowers as temperature rises from 25 to 760 °C. The reduction may be from lubricous oxides formed on the superalloy (ref. 11). As temperature nears 900 °C, the wear typically increases. The increase in wear at 900 °C may come from the weakening of the composite with increased temperature (ref. 5). At higher temperature and higher speed PM212/Au showed higher wear than PM212. This may be due to the fact that full density was not achieved with PM212/Au, because the material strength is notably lowered by increased porosity at higher temperatures (ref. 5).

The tribological results for HIPped PM212 and PM212/Au were very close to results previously achieved with sintered PM212 (ref. 4). In general, sintered PM212 had friction coefficients of 0.3 to 0.4 and wear factors in the $10^{-5}$ to $10^{-6}$mm$^3$/N-m range. The HIPped composites did encounter lower friction in some case. Notably, neither HIPped PM212 nor HIPped PM212/Au experienced an increase in friction coefficient after 8 km of sliding at 350 °C that the authors of reference 4 observed for sintered PM212.

The wear scar in figure 1 was representative of typical pin surfaces after testing. There was very little difference between the wear surfaces for PM212 and PM212/Au pins. This was also true for the disk wear surfaces. This was not surprising due to the microstructural similarity of PM212 and PM212/Au. The use of EDS analyses on the SEM were selectively used in this study. Since the compositions of René 41, PM212 and PM212/Au contain many of the same elements, the spectra could not easily distinguish pin material from disk material and thus an accurate transfer film analysis was not possible. However, the presence of silver was detected in wear tracks from PM212 and gold in those from PM212/Au. This indicated that a transfer film was indeed developed for both composites.

It was a surprising result that a PM212/Au composite of near full density was not achieved by HIPping since it had consistently been achieved with PM212. It should be recalled that the two composites contain the same volume percentages of the three major components. The only difference is PM212 contains silver and PM212/Au contains gold as the low temperature lubricant.

The examination of the microstructures provided few, if any, answers to why full density was achieved with PM212 and not with PM212/Au. Initially, the reason was believed to be related to the higher melting point of gold. The processing temperature (1100 °C) is above both the melting point of gold (1063 °C) and silver (961 °C). Thus, as planned, liquid phase processing of the lubricants should occur. Yet the fine web-like structure of the gold observed at high optical magnification suggested that the gold may not have liquified. This would have been attributed to an error in the HIPping unit's temperature which was not the case. Yet, when the composites were examined on the SEM, the distribution of the gold and silver, as well as all other components appeared almost identical (fig. 5).

Finally, the structures of the gold and silver powders were examined. Since the gold particles were found to be solid, no air would have been carried into the compact inside the gold powder. A remaining possibility lies in the particle morphology difference. Figure 7 shows that the gold particles are spherical and the silver particles are granular. All other powders used in the composites have a granular morphology. It is possible that the spherical gold particles somehow permitted the formation of sealed air pockets in the composite during the CIPping operation which were not outgassed during the vacuum annealing and sealing processes. Thus, air space could have been carried into the HIPping process which would have been trapped in the sealed can, yielding parts with porosity in the microstructure. Using gold
powder with a granular morphology instead of spherical would determine if this speculation is valid. Additionally, processing the PM212/Au at a higher temperature and/or pressure might determine if the porosity was due to insufficient HIPping temperature and/or pressure.

CONCLUSION

HIPped PM212 and PM212/Au provide moderate friction and moderate to low wear over a wide range of sliding velocities (0.27 to 10.0 m/sec) and temperatures (25 to 900 °C). Friction coefficients were typically between 0.25 and 0.40 and wear factors for both the pin and the disk predominated in the low $10^{-5}$mm$^3$/N-m range. In several cases, the friction and wear of the two composites in sliding against René 41 were within scatter of each other. Thus, the silver in PM212 can be replaced with gold and still retain good tribological properties. Density measurements indicated a residual porosity of 15 percent was present in the HIPped PM212/Au composite whereas PM212 was processed to full density. The presence of pore space was verified in PM212/Au through the use of optical and scanning electron microscopy. The microstructures of the composites were similar except the gold in PM212/Au appeared as a finely distributed web-like structure whereas the silver in PM212 appeared as discrete pockets. The residual porosity in PM212/Au may be caused by gas entrapment during CIPping by the spherical gold particles.

Gold has been shown to be a suitable substitute for the silver in PM212. Sulfide formation that is possible with silver is not possible with gold. Thus, PM212/Au is recommended for critical applications requiring chemical stability.

REFERENCES


TABLE I. - COMPOSITIONS OF THE TWO COMPOSITES

<table>
<thead>
<tr>
<th>Material</th>
<th>Component</th>
<th>Density, g/cm³</th>
<th>Wt%</th>
<th>Vol%</th>
<th>Particle size, mesh</th>
<th>Particle size, μm</th>
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<td>Silver</td>
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<td>9.6</td>
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TABLE II. - TRIBOLOGICAL TEST CONDITIONS

<table>
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<th>Sliding velocity, m/sec</th>
<th>Composite</th>
<th>Temperature, °C</th>
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<tr>
<td>10</td>
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<td></td>
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<td>PM212</td>
<td>900</td>
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<td>Temperature, °C</td>
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a Overall average for 2.7 m/sec tests, single test average for others.
bK_{avg} for 2.7 m/sec tests with PM212, s(K) as uncertainty.
cNegative sign indicates material buildup during test.
dFor this condition, range was 0.05×10^{-5} to 1.7×10^{-5}. 
Figure 1. Wear measurements of pin and disk specimens.
Figure 2. Friction coefficient versus temperature for the two composites based on 70 min tests.

Figure 3. Pin wear factor versus temperature for the two composites based on 70 min tests.
Figure 4. Run-in effects on wear for the two composites.
Figure 5. SEM images illustrating surface typography and elemental distribution.
Figure 6. Low magnification optical micrographs and high magnification SEM micrographs of the composites.

(a) Silver particles.  (b) Gold particles.

(c) Cross section of gold powder.

Figure 7. Gold and silver powder morphology.
**Tribological and Microstructural Comparison of HIPped PM212 and PM212/Au Self-Lubricating Composites**

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The feasibility of replacing the silver with the volumetric equivalent of gold in the chromium carbide-based self-lubricating composite PM212 (70 wt% NiCo-Cr₃C₂, 15% Ag, 15% BaF₂/CaF₂ eutectic) was studied. The new composite, PM212/Au has the following composition: 62 wt% NiCo-Cr₃C₂, 25% Au, 13% BaF₂/CaF₂ eutectic. The silver was replaced with gold to minimize the potential reactivity of the composite with possible environmental contaminants such as sulfur. The composites were fabricated by hot isostatic pressing (HIPping) and machined into pin specimens. The pins were slid against nickel-based superalloy disks. Sliding velocities ranged from 0.27 to 10.0 m/s and temperatures from 25 to 900 °C. Friction coefficients ranged from 0.25 to 0.40 and wear factors for the pin and disk were typically low 10⁻⁹mm³/N-m. HIPped PM212 measured fully dense, whereas PM212/Au had 15 percent residual porosity. Examination of the microstructures with optical and scanning electron microscopy revealed the presence of pores in PM212/Au that were not present in PM212. Though the exact reason for the residual porosity in PM212/Au was not determined, it may be due to particle morphology differences between the gold and silver and their effect on powder metallurgy processing.