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The Effect of Prolonged Exposure to 750°C Air on the Tribological Performance of PM212: Self Lubricating Composite Material

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ABSTRACT

The effect of prolonged exposure to 750 °C air on the tribological performance and dimensional stability of PM212, a high temperature, self-lubricating composite, is studied. PM212, by weight, contains 70% metal-bonded  $\text{Cr}_3\text{C}_2$ , 15%  $\text{BaF}_2/\text{CaF}_2$  eutectic and 15% silver. Rub blocks were fabricated from PM212 by cold isostatic pressing followed by sintering. Prior to tribo-testing, the rub blocks were exposed to 750°C air for periods ranging from 100 to 1000 hours. Then, the rub blocks were slid against nickel-based superalloy disks in a double-rub-block tribometer in air under a 66N load at temperatures from 25 to 750°C with a sliding velocity of 0.36 m/s. Unexposed rub blocks were tested for baseline comparison. Friction coefficients ranged from 0.24 to 0.37 for the unexposed rub blocks and from 0.32 to 0.56 for the exposed ones. Wear for both the composite blocks and superalloy disks was typically in the moderate to low range of  $10^{-5}$  to  $10^{-6}$   $\text{mm}^3/\text{N}\cdot\text{m}$ . Friction and wear data were similar for the rub blocks exposed for 100, 500, and 1000 hours. Prolonged exposure to 750°C air increased friction and wear of the PM212 rub blocks at room temperature, but their triboperformance remained unaffected at higher temperatures, probably due to the formation of lubricious metal oxides. Dimensional stability of the composite was studied by exposing specimens of varying thicknesses for 500 hours in air at 750°C. Block thicknesses were found to increase with increased exposure time until steady state was reached after 100 hours of exposure, probably due to oxidation.

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## INTRODUCTION

Aerospace applications continually demand bearing and seal materials that can operate in a wide range of temperatures and environments. Conditions encountered from below room temperature to 900°C in oxidizing (air), reducing (hydrogen) and inert (argon, nitrogen) atmospheres are not uncommon. Traditional liquid (oil) and solid (graphite and MoS<sub>2</sub>) lubricants do not have the high temperature capabilities needed. These limitations accentuate the need for innovative material systems which are able to provide lubrication over a wide range of temperatures in a variety of atmospheres.

In response to this need, researchers at NASA Lewis Research Center developed the PS200/PM200 class of high temperature, self-lubricating composites.<sup>1</sup> The PS200 series of plasma-sprayed composite coatings and the PM200 series of powder metallurgy composites provide lubrication over a wide temperature spectrum.<sup>2,3</sup> These composites are comprised of a wear resistant, metal-bonded chromium carbide (Cr<sub>3</sub>C<sub>2</sub>) matrix combined with two solid lubricants, silver and barium fluoride/calcium fluoride (BaF<sub>2</sub>/CaF<sub>2</sub>) eutectic. Silver provides lubrication at low to moderate temperatures (up to 500°C) while the BaF<sub>2</sub>/CaF<sub>2</sub> eutectic provides lubrication at high temperatures (above 400°C). The plasma sprayed coatings have performed well as a high temperature cylinder wall coating for a Stirling engine and a backup lubricant coating for gas bearings.<sup>4,5</sup> Applications for the powder metallurgy version include process control valve stem bushings, control surface bearings, and turbine engine bushings.<sup>6</sup>

PM212, a specific composition in the PM200 family of materials contains 70 wt% of the carbide matrix and 15% each of the lubricants and had been studied extensively because it has shown excellent tribological performance as a coating and a free standing composite<sup>2-4</sup>. Two processing techniques, cold compaction followed by sintering and cold compaction followed by hot isostatic pressing (HIPping), have been studied to determine their effects on PM212. Mechanical and thermophysical properties were compared including tensile strength, elastic modulus, thermal expansion coefficient, and thermal conductivity.<sup>7</sup> Fully dense PM212 formed by HIPping was found to be three times stronger in compression than the 78% dense PM212 formed by sintering. The tribological properties of PM212 wear test pins sliding against superalloy disks were tested for a range of temperatures, sliding velocities, and applied loads.<sup>3</sup> A tribological comparison revealed that the HIPped PM212 provided slightly lower friction and wear than the sintered PM212.<sup>8</sup>

Compositional variations of PM212 were also studied to compare their tribological performance with the baseline PM212.<sup>9</sup> Testing in air showed that the composition of PM212 can be altered without significantly affecting friction and wear behavior.

The investigation of PM212 continues with this study to determine the effect of prolonged exposure to 750°C air on the tribological performance of this composite to assess its potential for long term use at high temperatures in such applications as furnace components or turbine engines. The processing route of cold compaction followed by sintering was chosen for this study since the resulting porous structure ( $\approx 80\%$  dense) is more vulnerable to oxidation and may yield accelerated results. Prior to the tribo-testing, rub blocks fabricated from the PM212 composite were exposed to 750°C air for three different periods: 100, 500 and 1000 hours. Then, the rub blocks were slid against nickel-based superalloy disks in a double-rub-block tribometer in air with temperatures ranging from 25 to 750°C at a sliding velocity of 0.36 m/s. Unexposed rub blocks were tested for baseline comparison to the exposed rub blocks. Also, dimensional stability of the composite (an important property for control of bearings clearances) was studied by exposing blocks of varying thicknesses for 500 and 1000 hours in air at 750°C. The composite microstructures were studied using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS).

#### MATERIALS AND PROCESSING

PM212 powder was made by blending three components— metal bonded  $\text{Cr}_3\text{C}_2$ ,  $\text{BaF}_2/\text{CaF}_2$  eutectic and silver. The metal-bonded  $\text{Cr}_3\text{C}_2$  and the silver powders are commercially available. The  $\text{BaF}_2/\text{CaF}_2$  eutectic was prepared by mixing and prefusing the individual powders in 62/38 wt% proportions, respectively. Following the prefusing step (1100°C in a nitrogen atmosphere) the eutectic was crushed, ball milled, and sieved to the particle size range listed in Table 1. The individual components were weighed out in the proper proportions listed in Table 1 and then mixed in a Vee type blender.

The blended powder was packed into a rubber bag with a 2.5 cm square cross-section and a length of 15 cm. The bag was placed in a chamber which is pressurized to 414 MPa (60 ksi) for 5 minutes. The specimens were removed from the pressure chamber, and then the compacted bars were removed from the rubber bags. This method is referred to as cold isostatic pressing (CIPping). The CIPping method yields PM212 bars with green densities of approximately 75%.

The CIPped specimens were placed into a tube furnace with a dry hydrogen atmosphere. Hydrogen is used to prevent oxidation of the specimens during the sintering process. The furnace was heated at a rate of 10 C°/min up to 1100°C and held at temperature for 30 minutes. The furnace was then cooled at a rate of 10 C°/min down to room temperature before removing the specimens. The sintered PM212 is approximately 80% dense.

The superalloy used for the test disks was Inconel 718. Inconel 718 is a precipitation hardened, nickel-based superalloy with a nominal hardness of  $R_c$  35-40 at room temperature. Table 2 shows the nominal composition of Inconel 718 based on the manufacturer's literature.

## APPARATUS AND PROCEDURE

### Specimen Machining and Preparation

PM212 rub blocks were machined from the CIPped and sintered bars using a diamond slitting wheel. The blocks are 22.23 mm long with a 6.35 mm by 11.11 mm cross-section. Final dimensions were achieved by diamond grinding the blocks using clean water as a coolant. Machining oils were not used so that contamination of the PM212 is minimized. The average surface roughness of the PM212 blocks was 0.45 $\mu$ m CLA. The Inconel 718 disk specimens were 12.7 mm thick with an outside diameter of 35 mm. The disks were ground to an average surface roughness of 0.16 $\mu$ m CLA. The higher surface roughness of the PM212 was primarily due to exposed porosity at the surface.

The PM212 blocks and the Inconel 718 disks were ultrasonically cleaned in 190 proof ethyl alcohol for 5 minutes. Then, the PM212 blocks were heated in a vacuum oven for 3 hours at 150°C and 8 kPa absolute pressure to remove any residues from the processing and machining operations. The blocks and disks were then wet scrubbed with 0.1 $\mu$ m grit size alumina powder, rinsed with distilled water, and dried with clean compressed air.

Metallographic cross-sections of the unexposed and exposed rub blocks were prepared for microscopic examination. The rub blocks were cross-sectioned by diamond slitting and then mounted in a copper specimen holder. Standard metallographic polishing lubricants, such as hydrocarbon oils and aqueous solutions with additives, were found to contaminate the PM212 by infiltrating the pores exposed at the surface and interfering

with the SEM analysis. Therefore, cross-sections were diamond ground followed by wet polishing in water with 180, 400, then 600 grit size SiC paper.

#### Exposure

Four sets of PM212 rub blocks were prepared for tribological testing. One set of 18 blocks was tribotested directly after the machining and preparation steps. The second set of 12 blocks was exposed for 100 hours in 750°C air before tribo-testing. The third set of 18 blocks was exposed for 500 hours in 750°C air before tribo-testing. And the fourth set of 18 blocks was exposed for 1000 hours in 750°C air before tribo-testing.

An additional set of PM212 blocks of varying thicknesses was also thermally exposed to study the effects of thickness on dimensional stability. These blocks were not used for tribo-testing and had thicknesses of 0.8, 2.0, 3.0, 6.0, and 11.1 mm. Three samples of each thickness were exposed to 750°C air to accumulate a total of 500 hours. The samples were removed from the furnace for measurement at the following points of accumulated exposure time: 5, 23, 45, 98, 194, and 500 hours. After cooling to room temperature, thickness and mass changes were measured using a vernier micrometer and microbalance, respectively.

#### Tribological Testing

A double-rub-block-on-disk tribometer was used to evaluate the PM212 rub blocks in sliding against the Inconel 718 disks. In this test configuration shown in Figure 1, the faces (22 x 6.4mm) of the two stationary rub blocks are loaded against the periphery of the rotating disk. A calibrated pressurized bellows is used to apply the load. The disk rotational speed is monitored during the test with a magnetic gear speed pickup. The test specimens are heated with a high-frequency induction coil. The disk surface temperature is measured with an infrared pyrometer, which is calibrated with a contact thermocouple. The friction force is measured using a strain gage transducer which is located outside of the test chamber and is thermally isolated. The transducer output is conditioned and amplified before going to a chart recorder and a computerized data acquisition system.

All tests were run at a sliding velocity of 0.36 m/s with a 66 N normal load. The atmosphere was air with a relative humidity of 30 to 60% at 25°C. These conditions were chosen to simulate a potential bushing applications. A total of 126 one-hour tests were run according to the matrix shown in Table 3.



A stylus surface profilometer was used to measure wear on the rub blocks and disk. The wear scar width of each block was averaged from three measurements to calculate the block wear volume using the equation derived in Appendix A. Wear scar width rather than wear scar profile was used because the wear depth usually exceeded the dynamic range of the stylus profilometer. Four wear scar profiles of the disk wear track were measured and averaged to determine the wear track area. This area was multiplied by the disk circumference to calculate the disk wear volume. Wear factors were computed for both the block ( $K_b$ ), and the disk ( $K_d$ ). Briefly, the wear factor is equal to the wear volume removed during sliding divided by the product of the normal load and the total sliding distance.

#### Wear Surface and Microstructural Analyses

Wear surfaces of the block and disk were analyzed using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS). The disk wear tracks were examined for transfer films formed from sliding against the PM212 blocks. Additionally, the surface morphology of the wear tracks were examined. Settings used on the SEM were 20kV accelerating voltage, approximately 0.6 nA probe current, and 39mm working distance.

## RESULTS

### Exposure

The mass and thickness changes due to exposure in 750°C air for the blocks of various thicknesses are shown in Figures 2a and 2b, respectively. For each exposure set, the average change in mass was found to increase with increasing block thickness and increasing hours of exposure. But as exposure hours increased, the rate of mass change decreased for decreasing block thickness with the thinnest blocks approaching a steady state. For all block thicknesses, the thickness changes were found to initially increase with increasing exposure time. By 100 hrs, of exposure, the thickness changes appeared to have reached a steady state. No distinct relationship was exhibited between block thickness and thickness increase with hours of exposure.

### Tribological

The friction and wear results for the PM212 unexposed and exposed rub blocks are summarized in Table 4 and are shown in Figure 3. Average friction coefficients for the rub blocks are compared in Figure 3a. Prior to thermal exposure, friction coefficients were observed to range from  $0.24 \pm 0.04$  at 540°C to  $0.37 \pm 0.08$  at

750°C. After thermal exposure, the friction was observed to range from  $0.32 \pm 0.03$  at 540°C for the blocks exposed 100hrs to  $0.56 \pm 0.05$  at 25°C for the blocks exposed 500 hrs. The friction coefficients of the exposed blocks (100, 500, and 1000 hrs) were within scatter of each other at each test temperature. Increasing the test temperature caused the friction coefficients of the exposed blocks to converge within scatter of those of the unexposed blocks. Within each rub block set, the lowest friction coefficient occurred at the intermediate test temperature of 540°C.

Average wear factors of the PM212 rub blocks are compared in Figure 3b. Unexposed block wear ranged from  $6.0 \times 10^{-6} \text{ mm}^3/\text{N-m}$  at 540°C to  $1.1 \times 10^{-4} \text{ mm}^3/\text{N-m}$  at 25°C. After thermal exposure, block wear ranged from  $4.3 \times 10^{-6} \text{ mm}^3/\text{N-m}$  at 540°C for the blocks exposed 100 hrs to  $3.07 \times 10^{-4} \text{ mm}^3/\text{N-m}$  at 25°C for the blocks exposed 500 hrs. The rub block wear factors of the exposed blocks (100, 500, and 1000 hrs) were within scatter of each other at each test temperature. Increasing the test temperature caused the block wear of the exposed blocks to converge within scatter of those of the unexposed blocks. Within each rub block set, the lowest block wear occurred at the intermediate test temperature of 540°C. Average wear factors of the Inconel 718 disks slid against the PM212 rub blocks are compared in Figure 3c. With the unexposed rub blocks, disk wear ranged from  $5 \times 10^{-6} \text{ mm}^3/\text{N-m}$  at 540°C to  $1.01 \times 10^{-4} \text{ mm}^3/\text{N-m}$  at 25°C. After thermal exposure of the rub blocks, disk wear ranged from  $2.8 \times 10^{-7} \text{ mm}^3/\text{N-m}$  at 750°C to  $9.3 \times 10^{-5} \text{ mm}^3/\text{N-m}$  at 25°C, both for the blocks exposed 100 hrs. The disk wear factors with each rub block set (unexposed, exposed 100, 500, and 1000 hrs) were within scatter of each other at each test temperature. Also, disk wear was found generally to decrease with increasing temperature.

The average friction coefficient is plotted as a function of time at each test temperature for the unexposed and exposed rub blocks in Figure 4. Friction behavior was similar for all the exposed blocks at each temperature. As the test temperature increased, the friction performance of the exposed blocks converged with that of the unexposed blocks. At each test temperature the average standard deviations of the friction coefficients were similar for all the rub blocks. The largest deviations, given in Table 4, were exhibited at the 750°C tests for all the rub blocks.

The block wear factor is plotted as a function of time at each test temperature for the unexposed and exposed PM212 rub blocks in Figure 5. After thermal exposure, block wear generally decreased with time at

each test temperature. Also, as the sliding time increased the wear factors of the exposed blocks converged with those of the unexposed blocks at each test temperature.

### DISCUSSION

The results from Figure 2 show that PM212 composite blocks are physically affected by prolonged exposure to 750°C air. Figures 3a and 3b show, respectively, that block mass and thickness increases with increasing exposure time until a steady state is reached. Thickness increases reached a steady state after 100 hours, at which point a decrease in block thicknesses was observed with no corresponding decrease in mass reflected by Figure 3a. This unexpected decrease is probably due to measurement error as no other decrease was observed. Nonetheless, increasing a dimension does not have a strong influence on increasing its dimensional growth. This suggests that the exposure causes oxidation and dimensional growth in the surface region rather than through the bulk of the composite.

Cross-sections of PM212 rub blocks are shown in SEM micrographs of Figure 6, which shows the microstructural differences near the surface of an unexposed block and one exposed for 500 hours. After exposure, porosity which was present in the unexposed material appears to have been filled, presumably with oxidized material, and the fine lamellar structure of the eutectic is no longer readily observed. The matrix material appears largely unaffected. The corresponding EDS spectra shown in Figure 7 shows more oxygen to be present in the exposed block near the surface. However, the core of the block exposed for 500 hours appears unaffected and microstructurally similar to the unexposed block core in the micrographs and spectra of Figures 8 and 9, respectively. Figure 10 shows this gradient in microstructural appearance from the core to the surface for the block exposed for 500 hours. Cross-sectional micrographs of rub blocks exposed for 100 and 1000 hours revealed similar microstructural characteristics with no significant changes after 100 hours of exposure. This suggests that dimensional stabilization of bearings can be achieved by suitable heat treatment in air prior use in an application requiring small clearances.

The exposure results indicate that the observed changes to the PM212 composite occur during the first 100 hours of exposure and that exposure beyond 100 hours (at least to 1000 hours) does not significantly affect the material further. This conclusion is also supported by the tribological results from Figure 3. At all test temperatures, friction and wear is similar for the rub blocks exposed 100, 500, and 1000 hours. At room

temperature the exposed rub blocks had higher friction and wear than the unexposed blocks, but disk wear was similar for all blocks. However, increasing the test temperature caused the tribological performance of all the rub blocks to converge. This behavior could be explained by the formation of lubricious metal oxides on the superalloy disks at high temperatures. This phenomenon was observed in the tests of Reference 10 wherein ceramic rub blocks were slid against superalloy disks in air at temperatures from 25 to 800°C. A decrease in friction at high temperatures was attributed to the formation of these oxides whose presence in the block wear tracks was inferred by EDS analyses. The presence of metal oxides (e.g., chrome oxide, nickel oxide) could only be inferred from the EDS analyses because this technique can only detect the presence of oxygen and metals not how they are combined. Furthermore, the analysis system used is not able to detect fluorine reliably due to its low energy. In the current study, EDS analyses shows the presence of oxygen in both the block and disk wear tracks increases for the high temperature tests. Representative EDS spectra showing this oxygen increase are shown in Figures 11 and 12, which compare the spectra for a rub block exposed for 500 hours and its corresponding disk after a room temperature test and a 750°C test, respectively. It seems reasonable that for these tests lubricious metal oxides are contributing to lower friction and wear at elevated temperatures.

Lubricant transfer from the block to the disk also increases at higher temperatures as shown in Figures 11 and 12, which reveal an increase in the amount of silver and the Ba/Ca-fluorides present in the disk wear track at the 750°C test. This increase in lubricant transfer may explain why the tribological performance converges at high temperatures. The SEM micrographs of Figure 13 show representative wear track surfaces of a rub block exposed for 500 hours and its corresponding disk after a room temperature test. Therefore, while prolonged exposure to 750°C air increases friction and wear of the PM212 rub blocks at room temperature, their triboperformance remains unaffected at higher temperatures.

#### RESULTS SUMMARY AND CONCLUSIONS

1. The thickness and mass of PM212 blocks were found to increase with increasing hours of exposure to 750°C air. However, these changes approached a steady state at 100 hours, after which time prolonged exposure to 1000 hours did not significantly affect the material further.
2. Prolonged exposure to 750°C air caused microstructural changes that affected the PM212 composite

blocks near the exposed surface but not at the core. These surface region changes include apparent densification and loss of lamellar structure of the eutectic while the matrix material appears largely unaffected.

3. After thermal exposure for 100 hours or more, the friction and wear of the PM212 rub blocks increases at room temperature, but their triboperformance remains unaffected at higher temperatures, probably due to the formation of the metal oxides whose lubricating mechanism dominates at these temperatures.

4. Based upon its good high temperature tribo performance after long thermal exposure, PM212 has potential as a long life, high temperature bearing material.

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APPENDIX A  
RUB BLOCK WEAR VOLUME

The double-rub-block tribometer produces a wear scar on the rub blocks worn by the periphery of the rotating disk. For each block the wear volume can be calculated by multiplying the rub block thickness,  $t$ , by the cross-sectional area worn out by the disk,  $A_{\text{Total wear}}$ . This cross-sectional wear area can be related to the disk radius,  $R$ , and the block wear scar width,  $W$ , through the block wear depth,  $d$ , and the following geometry in Figure A1.

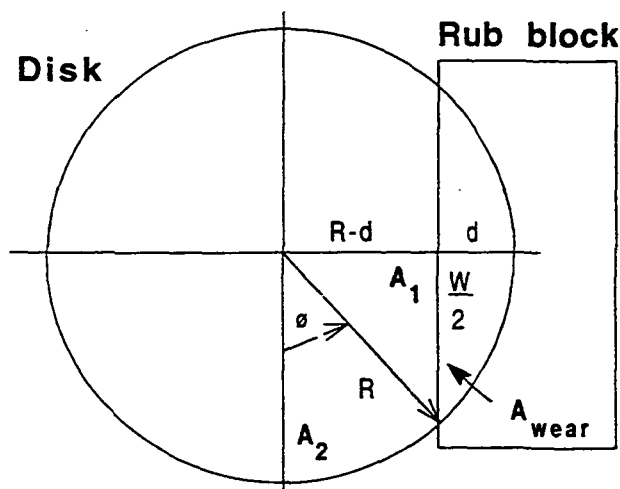


Figure A1.— Rub block on disk test geometry.

It is assumed that the disk radius,  $R$ , is essentially constant (i.e. disk wear depth is small compared to the radius) and the wear track width,  $W$ , is uniform.

From the radial symmetry of the disk, half of the wear area can be calculated from its corresponding quarter-section of the disk. The wear area of this section can be determined from the total area of this section by the following relation:

$$A_{\text{wear}} = \frac{\pi \cdot R^2}{4} - (A_1 + A_2) . \quad [1]$$

The area of the triangular region,  $A_1$ , can be calculated from the following equation:

$$A_1 = \frac{1}{2} \cdot (R - d) \cdot \frac{W}{2} . \quad [2]$$

By applying the Pythagorean Theorem to this same triangle, the following quadratic equation is obtained:

$$(R - d)^2 + (W/2)^2 = R^2 .$$

The quadratic formula can be used to solve this equation for the variable wear depth,  $d$ :

$$d = R - \frac{(4 \cdot R^2 - W^2)^{1/2}}{2} . \quad [3]$$

By substituting [3] in [2], the following relation for  $A_1$  is obtained:

$$A_1 = \frac{W \cdot (4 \cdot R^2 - W^2)^{1/2}}{8} . \quad [4]$$

The area of the sector,  $A_2$ , is related to the central angle,  $\phi$  (in radians), by the equation:

$$A_2 = \frac{1}{2} \cdot R^2 \cdot \phi . \quad [5]$$

The angle,  $\phi$ , can be determined from its relation to the sides of triangle,  $A_1$ , by the following:

$$\phi = \cos^{-1}(W/2 \cdot R) . \quad [6]$$

By substituting [6] in [5], the following relation for  $A_2$  is obtained:

$$A_2 = \frac{R^2 \cdot \cos^{-1}(W/2 \cdot R)}{2} . \quad [7]$$

Finally, by substituting [4] and [7] in [1],  $A_{\text{wear}}$  is related to  $R$  and  $W$  by the equation:



$$A_{\text{wear}} = \frac{\pi \cdot R^2}{4} - \frac{W \cdot (4 \cdot R^2 - W^2)^{1/2}}{8} - \frac{R^2 \cdot \cos^{-1}(W/2 \cdot R)}{2} .$$

The total wear area for one block is twice the above value. Therefore, the total wear area is:

$$A_{\text{Total wear}} = \frac{\pi \cdot R^2}{2} - \frac{W \cdot (4 \cdot R^2 - W^2)^{1/2}}{4} - R^2 \cdot \cos^{-1}(W/2 \cdot R) . \quad [8]$$

Multiplying [8] by the rub block thickness,  $t$ , yields the block wear volume. Thus, the rub block wear volume can be determined by substituting the known disk radius,  $R$ , the known rub block thickness,  $t$ , and the measured block wear scar width,  $W$ , into the following equation:

$$V_{\text{Block Wear}} = t \cdot \left\{ \frac{\pi \cdot R^2}{2} - \frac{W \cdot (4 \cdot R^2 - W^2)^{1/2}}{4} - R^2 \cdot \cos^{-1}(W/2 \cdot R) \right\} . \quad [9]$$

For the tests described in this paper, the Inconel 718 disk radius of  $R=17.465$  mm and PM212 rub block thickness of  $t=6.35$  mm were used to calculate values for the rub block wear volume,  $V_{\text{Block Wear}}$ , as a function of block wear scar width,  $W$ , using equation [9]. These values along with the block wear depth,  $d$ , which was calculated from equation [3], are shown in Table A1 for block wear scar widths ranging from 0.0 to 20.

Table A1.— Rub block wear volume as function of wear scar width  
for 17.465 mm radius disk and 6.35 mm thick rub block.

Wear scar width, W [mm]	Wear scar depth, d [mm]	Rub block wear volume, $V_{\text{block wear}}$ [mm <sup>3</sup> ]
0.000	0.000	0.000
0.500	0.002	0.004
1.000	0.007	0.030
1.500	0.016	0.102
2.000	0.029	0.243
2.500	0.045	0.474
3.000	0.065	0.820
3.500	0.088	1.303
4.000	0.115	1.947
4.500	0.146	2.775
5.000	0.180	3.811
5.500	0.218	5.079
6.000	0.260	6.603
6.500	0.305	8.409
7.000	0.354	10.520
7.500	0.407	12.964
8.000	0.464	15.764
8.500	0.525	18.949
9.000	0.590	22.544
9.500	0.658	26.578
10.000	0.731	31.078
10.500	0.808	36.074
11.000	0.889	41.596
11.500	0.974	47.673
12.000	1.063	54.337
12.500	1.157	61.621
13.000	1.255	69.558
13.500	1.357	78.183
14.000	1.464	87.532
14.500	1.576	97.642
15.000	1.692	108.551
15.500	1.814	120.301
16.000	1.940	132.934
16.500	2.071	146.494
17.000	2.208	161.026
17.500	2.350	176.581
18.000	2.497	193.209
18.500	2.651	210.964
19.000	2.810	229.904
19.500	2.975	250.089
20.000	3.146	271.585

Table 1.— Composition of PM212.

(a) Major components' proportions and particle sizes.

Component	Density [g/cm <sup>3</sup> ]	Weight %	Volume %	Particle Size [mesh]	Particle Size [μm]
Bonded Cr <sub>3</sub> C <sub>2</sub>	7.0	70.0	66.2	-200 +400	37 to 74
Eutectic	4.1	15.0	24.4	-200 +325	44 to 74
Silver	10.5	15.0	9.4	-200 +325	44 to 149

(b) Composition of metal bonded  
Cr<sub>3</sub>C<sub>2</sub> component.

Element	Weight %
Chromium	48
Nickel	28
Cobalt	12
Carbon	6
Molybdenum	2
Aluminum	2
Boron	1
Silicon	1

Table 2.— Composition of Inconel 718 based on manufacturer's literature.

Element	Weight %
Nickel (plus Cobalt)	50.00-55.00
Chromium	17.00-21.00
Iron	Balance
Columbium (plus Tantalum)	4.75-5.50
Molybdenum	2.80-3.30
Titanium	0.65-1.15
Aluminum	0.20-0.80
Cobalt	1.00 max.
Carbon	0.08 max.
Manganese	0.35 max.
Silicon	0.35 max.
Phosphorous	0.015 max.
Sulfur	0.015 max.
Boron	0.006 max.
Copper	0.30 max.

Table 3.—Test matrix for tribo-testing of PM212 rub blocks against Inconel 718 disks.

Block Exposure <sup>a</sup> [hours]	Test Temperature [°C]	Number of Specimen Sets <sup>b</sup>	Number of Tests per Specimen Set <sup>c</sup>	Total Number of Test hours
Unexposed	25	3	4	12
	540	"	"	"
	750	"	"	"
100	25	2	3	6
	540	"	"	"
	750	"	"	"
500	25	3	4	12
	540	"	"	"
	750	"	"	"
1000	25	3	4	12
	540	"	"	"
	750	"	"	"

<sup>a</sup>Rub blocks were exposed in air at 750°C.

<sup>b</sup>Specimen set was comprised of 2 PM212 rub blocks and 1 Inconel 718 disk.

<sup>c</sup>Each test was run for 60 minutes before taking wear measurements.

Table 4.—Tribological data summary for PM212 rub blocks slid against Inconel 718 disks.

Block Exposure [hours]	Test Temperature [°C]	Friction Coefficient and Variation <sup>a</sup>	Wear, Factor $K_{\text{block}}^b$ and (scatter) [10 <sup>-5</sup> mm <sup>3</sup> /N-m]	Wear, Factor, $K_{\text{disk}}^c$ and (scatter) [10 <sup>-5</sup> mm <sup>3</sup> /N-m]
Unexposed	25	0.28±0.05	11.0 (7.1 to 13.0)	10.1 (-2.9 to 17.0)
	540	0.24±0.04	0.6 (0.008 to 1.5)	0.5 (-0.03 to 1.2)
	750	0.37±0.08	1.5 (-0.7 to 13.0)	0.9 (-0.7 to 7.0)
100	25	0.52±0.03	20.0 (16.0 to 28.0)	9.3 (5.8 to 11.0)
	540	0.32±0.03	0.43 (-0.12 to 1.4)	0.55 (0.15 to 0.72)
	750	0.41±0.08	8.4 (2.0 to 12.0)	0.028 (-1.3 to 1.5)
500	25	0.56±0.05	30.7 (19.0 to 47.0)	9.1 (3.2 to 14.0)
	540	0.35±0.04	1.4 (-0.003 to 9.1)	0.9 (-0.08 to 2.3)
	750	0.35±0.09	4.3 (0.7 to 11.0)	0.2 (-0.6 to 1.0)
1000	25	0.52±0.05	25.7 (18.0 to 42.0)	8.1 (2.8 to 11.0)
	540	0.34±0.04	0.6 (-0.09 to 2.6)	0.5 (-0.3 to 1.7)
	750	0.34±0.09	3.1 (-0.2 to 8.7)	0.05 (-0.09 to 0.8)

<sup>a</sup>Averages with standard deviation.

<sup>b,c</sup>Averages with minimum and maximum values in parentheses.

Negative sign indicates material transfer or buildup.

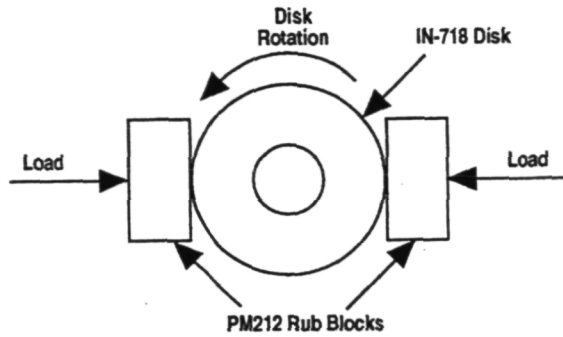


Figure 1.— Double rub block friction and wear apparatus.

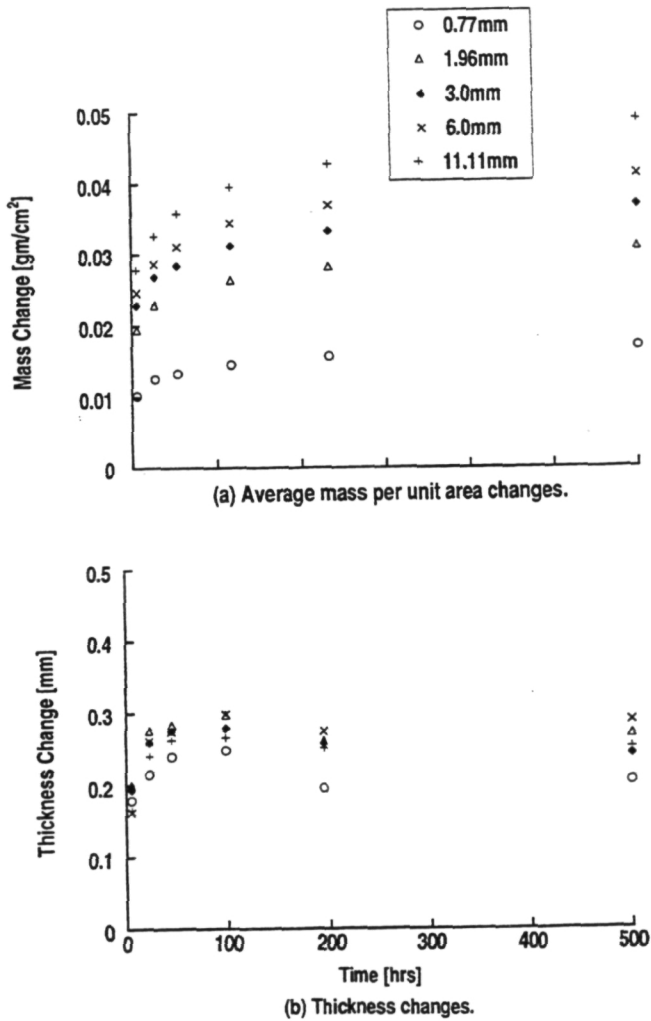


Figure 2.—Average mass per unit area and thickness changes due to exposure in 750 °C air for blocks of various thicknesses.

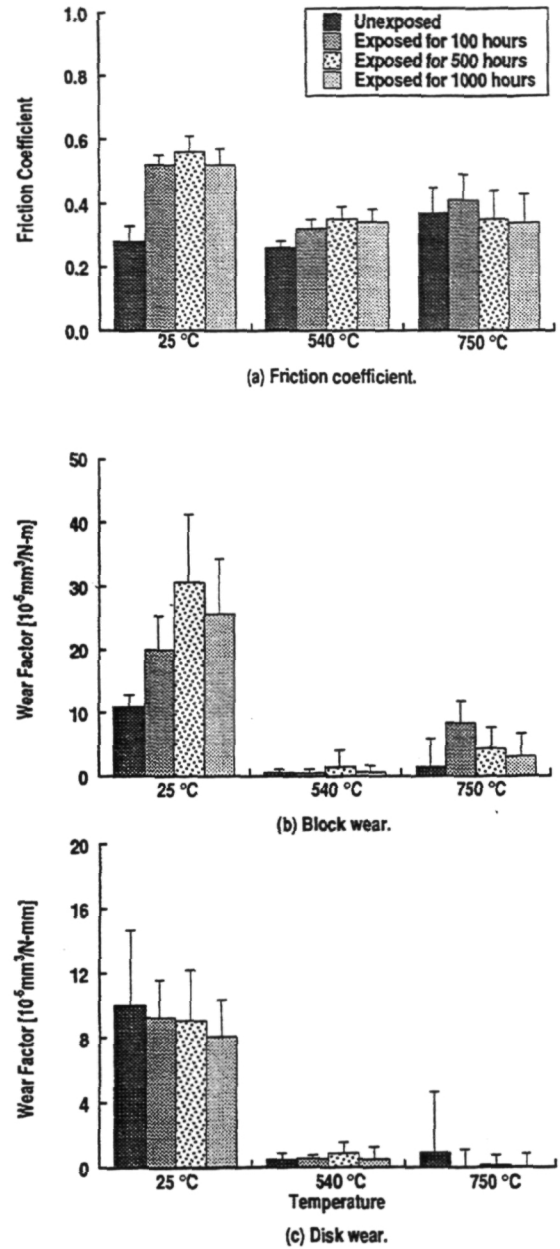
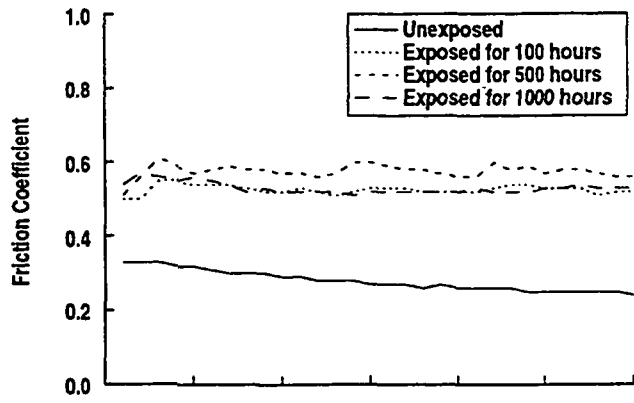
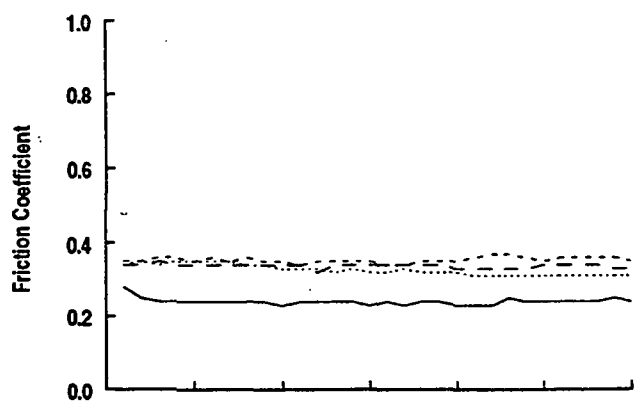


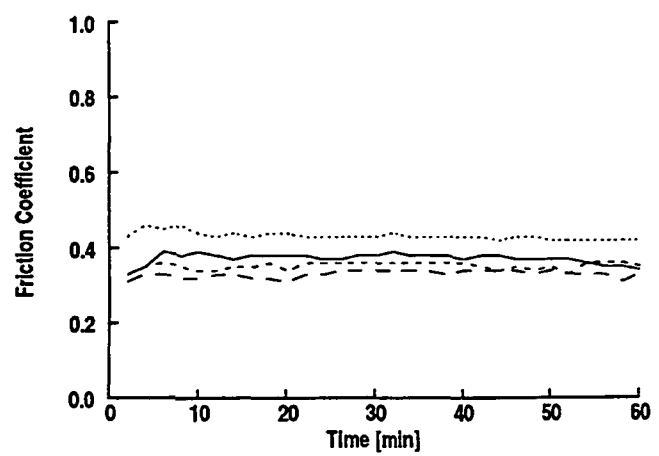
Figure 3.—Averages of friction and wear performance with standard deviations represented by error bars.



(a) 25°C test temperature.

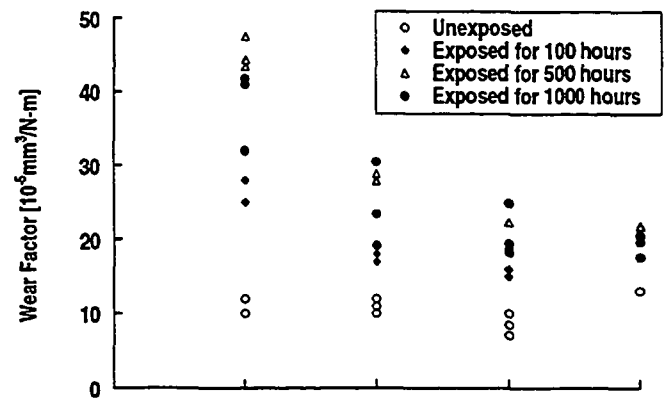


(b) 540°C test temperature.

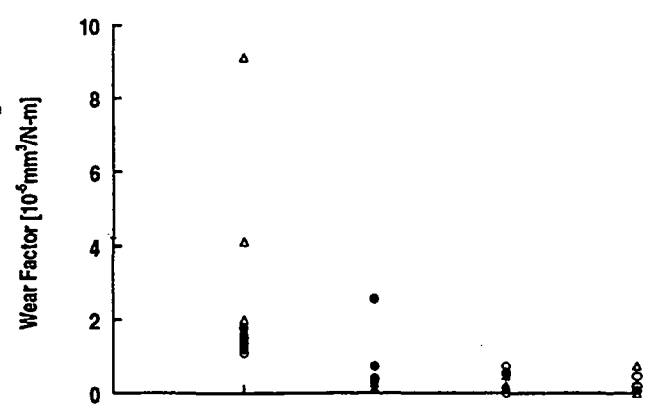


(c) 750°C test temperature.

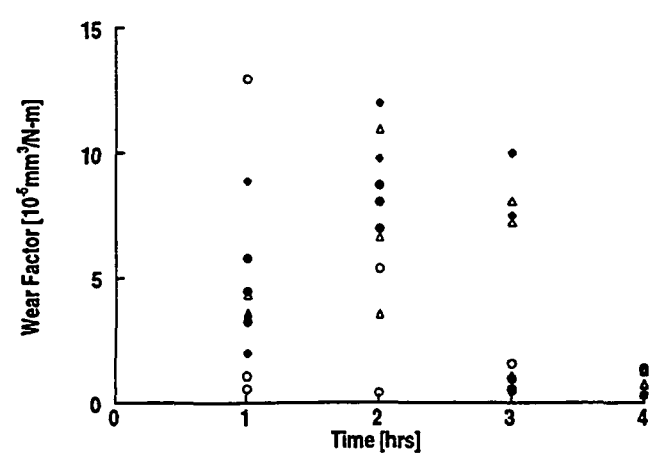
Figure 4.—Comparison of average friction coefficient versus time for all rub blocks at the different test temperatures.



(a) 25°C test temperature.

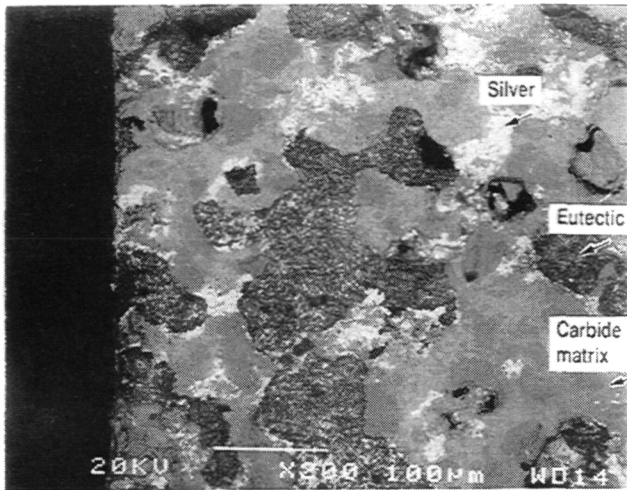


(b) 540°C test temperature.

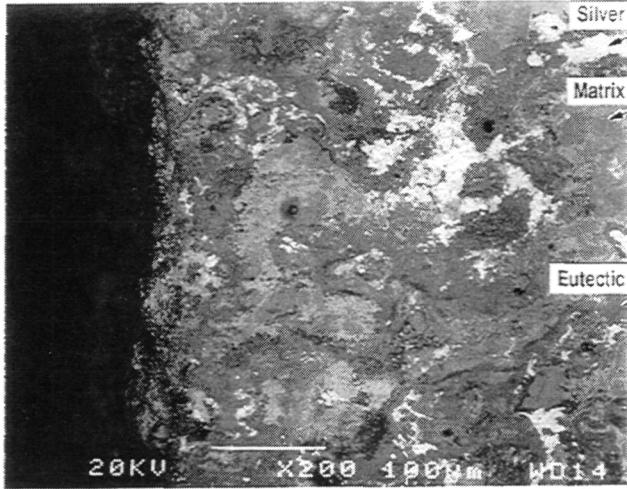


(c) 750°C test temperature.

Figure 5.—Block wear variation with time at the different test temperatures.

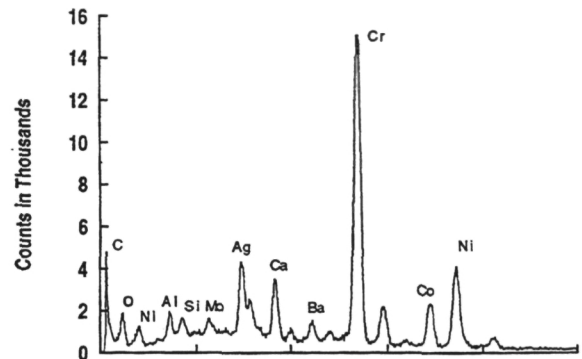


(a) Unexposed.

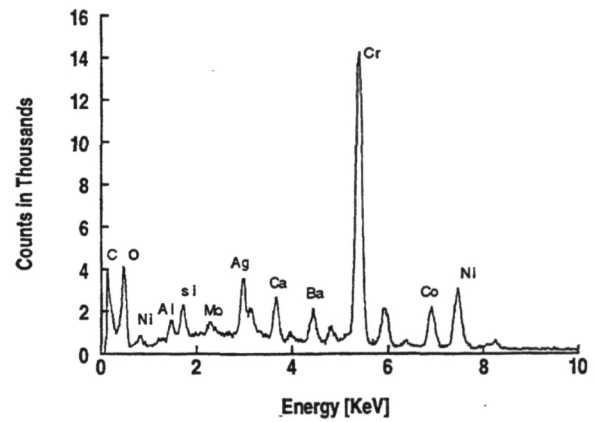


(b) Exposed for 500 hours.

Figure 6.—Backscattered SEM micrographs of cross-sections for unexposed and exposed rub blocks showing microstructural differences near surface.

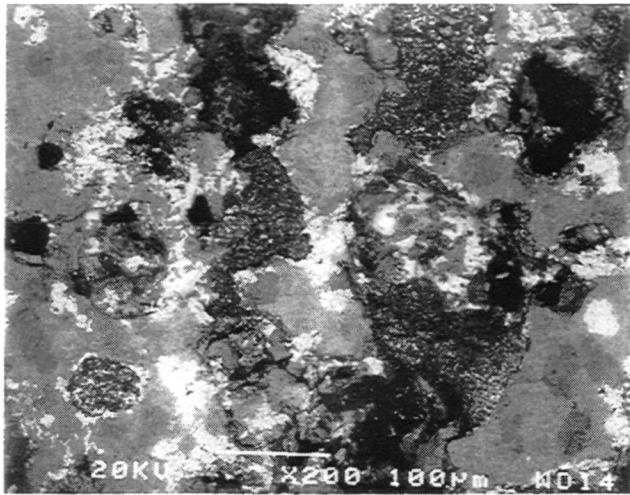


(a) Unexposed.

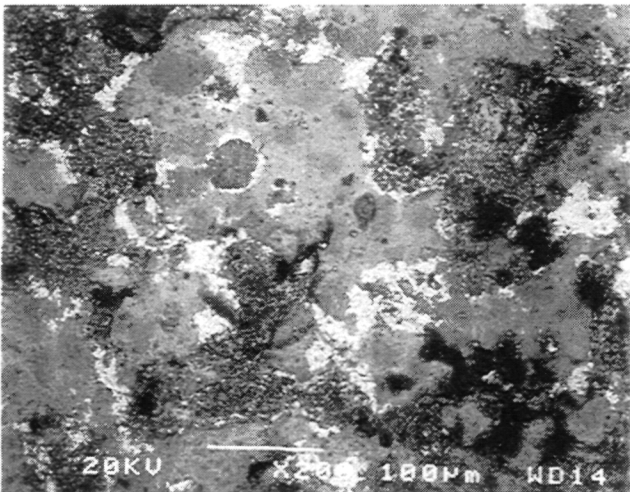


(b) Exposed for 500 hours.

Figure 7.—EDS spectra of rub block cross-sections near surface showing increased oxygen for the exposed block.

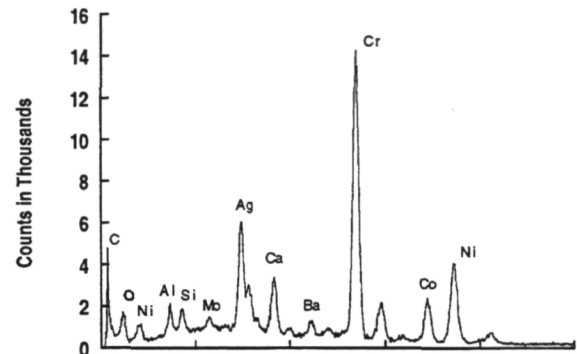


(a) Unexposed.

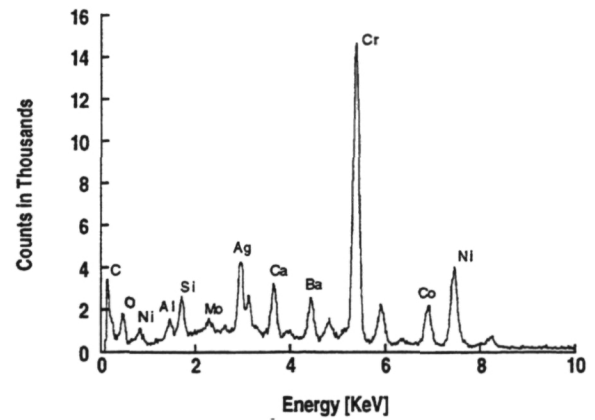


(b) Exposed for 500 hours.

Figure 8.—Backscattered SEM micrographs of cross-sections for unexposed and exposed rub blocks at the core, which appears unaffected and microstructurally similar.



(a) Unexposed.



(b) Exposed for 500 hours.

Figure 9.—EDS spectra of rub block cross-sections at core showing similar presence of oxygen.



Surface      Core  
 ↓            →

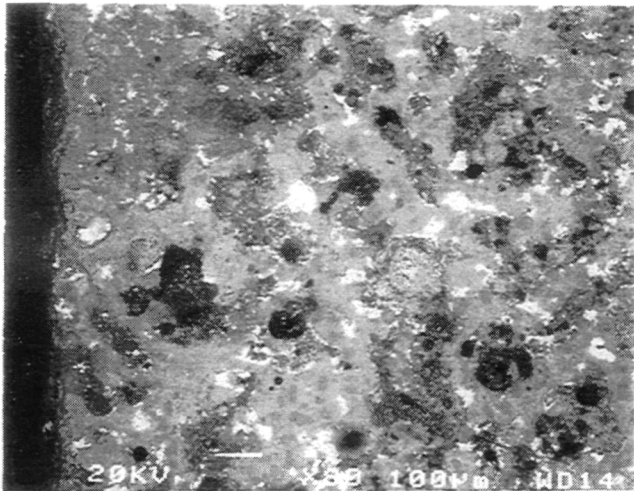
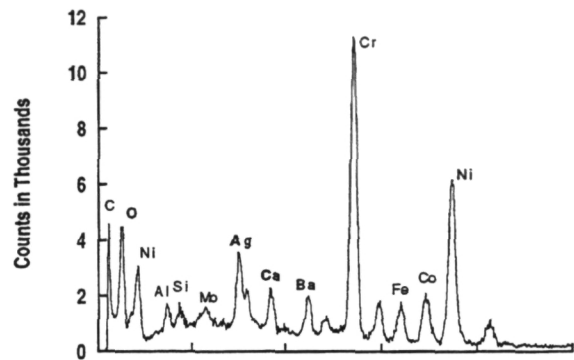
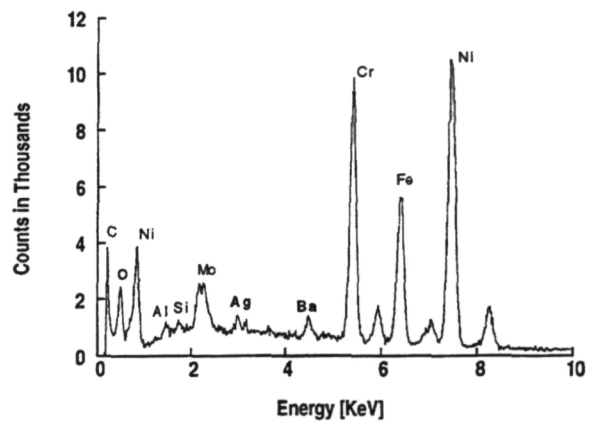


Figure 10.—Lower magnification, backscattered SEM micrograph of cross-sectioned rub block exposed for 500 hours. Note the gradient in the microstructure from the core towards the surface.

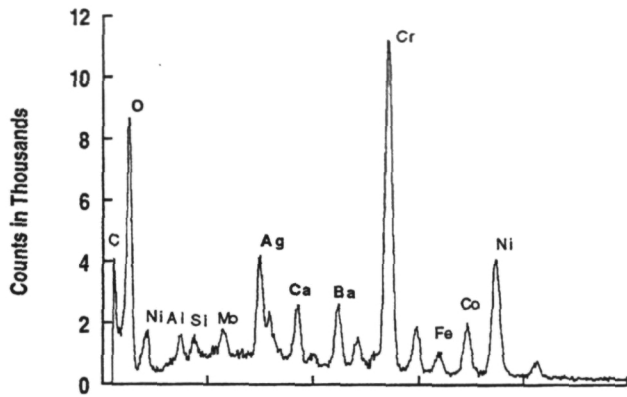


(a) Rub block exposed for 500 hours.

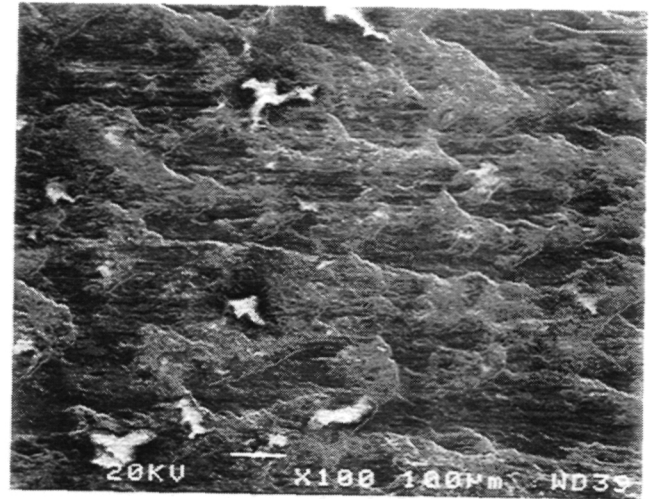


(b) Disk.

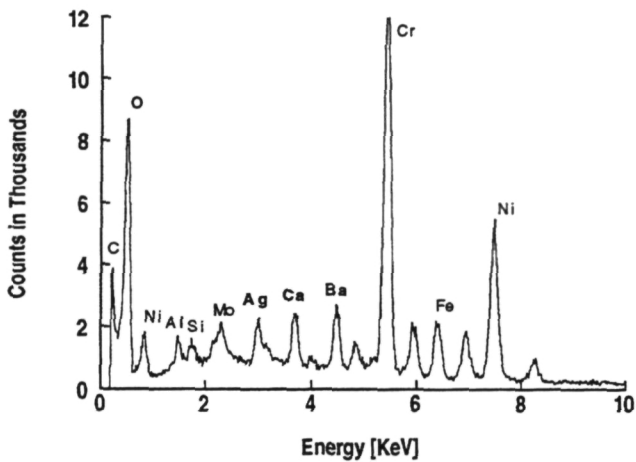
Figure 11.—EDS spectra of specimen wear tracks after room temperature test showing modest amounts of oxygen and lubricant transfer from block to disk.



(a) Rub block exposed for 500 hours.

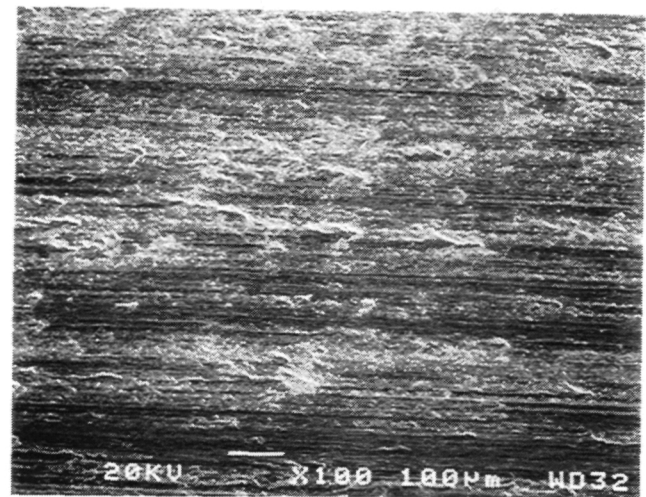


(a) Rub block exposed for 500 hours.



(b) Disk.

Figure 12.—EDS spectra of specimen wear tracks after 750 °C test showing increased oxygen and lubricant transfer from block to disk.



(b) Disk.

Figure 13.—SEM micrographs of specimen wear track surfaces after a room temperature test. Note the plate-like film formation on the rub block and the smooth disk surface.

# REPORT DOCUMENTATION PAGE

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<b>13. ABSTRACT (Maximum 200 words)</b>  The effect of prolonged exposure to 750 °C air on the tribological performance and dimensional stability of PM212, a high temperature, self-lubricating composite, is studied. PM212, by weight, contains 70% metal-bonded Cr <sub>3</sub> C <sub>2</sub> , 15% BaF <sub>2</sub> /CaF <sub>2</sub> eutectic and 15% silver. Rub blocks were fabricated from PM212 by cold isostatic pressing followed by sintering. Prior to tribo-testing, the rub blocks were exposed to 750°C air for periods ranging from 100 to 1000 hours. Then, the rub blocks were slid against nickel-based superalloy disks in a double-rub-block tribometer in air under a 66N load at temperatures from 25 to 750°C with a sliding velocity of 0.36 m/s. Unexposed rub blocks were tested for baseline comparison. Friction coefficients ranged from 0.24 to 0.37 for the unexposed rub blocks and from 0.32 to 0.56 for the exposed ones. Wear for both the composite blocks and superalloy disks was typically in the moderate to low range of 10 <sup>-5</sup> to 10 <sup>-6</sup> mm <sup>3</sup> /N-m. Friction and wear data were similar for the rub blocks exposed for 100, 500, and 1000 hours. Prolonged exposure to 750°C air increased friction and wear of the PM212 rub blocks at room temperature, but their triboperformance remained unaffected at higher temperatures, probably due to the formation of lubricious metal oxides. Dimensional stability of the composite was studied by exposing specimens of varying thicknesses for 500 hours in air at 750°C. Block thicknesses were found to increase with increased exposure time until steady state was reached after 100 hours of exposure, probably due to oxidation.				
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