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Composition Optimization of Self-Lubricating Chromium Carbide-Based Composite Coatings for Use to 760 °C

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Work performed for
U.S. DEPARTMENT OF ENERGY
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Prepared for
American Society of Lubrication Engineers Annual Meeting
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COMPOSITION OPTIMIZATION OF SELF-LUBRICATING CHROMIUM
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Abstract

This paper describes new compositions of self-lubricating coatings that contain chromium carbide. A bonded chromium carbide was used as the "base stock" because of the known excellent wear resistance and the chemical stability of chromium carbide. "Additives" were silver and barium fluoride/calcium fluoride eutectic. The coating constituents were treated as a ternary system consisting of: (1) the bonded carbide base material, (2) silver, and (3) the eutectic. A study to determine the optimum amounts of each constituent was performed. The various compositions were prepared by powder blending. The blended powders were then plasma sprayed onto superalloy substrates and diamond ground to the desired coating thickness. Friction and wear studies were performed at temperatures from 25 to 760 °C in helium and hydrogen. A variety of counterface materials were evaluated with the objective of discovering a satisfactory metal/coating sliding combination for potential applications such as piston ring/cylinder liner couples for Stirling engines.

In general, silver and fluoride additions to chromium carbide reduced the friction coefficient and increased the wear resistance relative to the unmodified carbide coating. Silver and the fluoride eutectic acted synergistically in reducing friction and counterface wear. Counterface

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materials included cobalt, nickel, and ferrous alloys. Several coating compositions gave good results in hydrogen and in helium to 760 °C.

INTRODUCTION

The difficulty of lubricating sliding contacts (e.g., piston/cylinder walls, foil bearing journals) at high temperatures is a major obstacle to the development of heat engines (1). It would be advantageous if certain tribological components in advanced engines, such as the adiabatic diesel and the Stirling engine, could operate at temperatures well above the failure point of traditional solid lubricants such as molybdenum disulphide and graphite. Clearly, the need exists for durable and chemically-stable lubricants capable of withstanding these high temperature environments. Plasma-sprayed, ceramic based coating compositions have been developed at NASA Lewis, to satisfy these requirements. One composite coating system that shows promise is based upon chromium carbide with solid lubricant additives (2-4).

Chromium carbide has excellent wear resistance and thermal stability, but exhibits high friction coefficients when used in sliding contacts. By blending solid lubricants with chromium carbide friction characteristics can be improved. Silver metal and barium fluoride/calcium fluoride eutectic were chosen as the solid lubricants. Silver, because of its low shear strength, provides low friction at low temperatures and the eutectic has been shown to effectively lubricate above 500 °C (3). Like chromium carbide, silver and the fluoride eutectic are thermally and chemically stable to at least 900 °C. Since silver lubricates from low to moderately high temperatures, and the eutectic lubricates at high temperatures, the resulting composite lubricant should not show any sharp discontinuities in its friction and wear behavior over a wide temperature range. The coating system was designed to successfully lubricate in applications where low temperature starts and high operating temperatures are encountered.

Preliminary friction and wear data in air for a coating composition designated PS200, which contains 10 wt % of silver and 10 wt % barium fluoride/calcium fluoride eutectic in, a metal-bonded chromium carbide matrix have been reported (2,3). Additional friction and wear data for PS200 in helium, hydrogen and air are given in reference 4. These references describe the performance of only one coating, PS200. This paper describes an optimization program to determine the proportions of the three components (metal-bonded chromium carbide, silver, and fluoride eutectic) which give the best friction and wear properties. The scope of this paper includes optimization experiments in helium and in hydrogen atmospheres, at temperatures to 760 °C, to simulate the environment in the Stirling engine.

EXPERIMENTAL MATERIALS

Wear Pin Materials

A number of counterface materials were chosen for evaluation of their sliding behavior against the metal-bonded chromium carbide coatings. The pin materials were chosen for thermal and chemical stability and strength at elevated temperatures. They also had to be practical materials for use as engine components. The materials chosen were: a precipitation-hardened nickel-chromium alloy A; two stainless steels, alloy B and alloy D; a hardened cobalt alloy C; and PS200 coated on alloy B. Table 1 gives the hardness and the compositions of these materials. The wear pins were hemispherically-tipped with a radius of 0.476 cm and a length of 2 cm.

Coating Composition Matrix

Coating identification numbers and compositions are given in Tables 2 and 3. No coating compositions were chosen that contained less than 60 wt % of the metal-bonded chromium carbide because it was expected that the wear resistance of coatings with less of the hard constituent would be inadequate. Previous work had indicated that the composition 80 wt % metal-bonded

chromium carbide, 10 wt % silver and 10 wt % fluoride eutectic, (PS200), would be a good starting point upon which to base other compositions (2-4). Two compositions which also have a 1:1 ratio of silver to eutectic were selected. They were PS212 with 15 wt % of each additive and PS213 with 20 wt % of each. To determine the effect of each constituent, PS203 with 5 wt % silver and 10 wt % eutectic, PS204 with 10 wt % silver and 5 wt % eutectic, PS215 with 30 wt % silver and 0 wt % eutectic, and PS216 with 0 wt % silver and 30 wt % eutectic were evaluated. Finally, as a control, unmodified, metal-bonded chromium carbide, PS218 was also evaluated.

Coating and Finishing Procedure

The test disks are 6.35 cm in diameter, 1.27 cm thick, and made of high temperature alloys. The disks are first sandblasted, then a thin bond coat (0.0076 cm) of nichrome (NiCr) powder is plasma sprayed onto the roughened surface. The blended powder mixture is plasma sprayed onto the bond coat to a thickness of about 0.038 cm. (The spraying parameters are given in Table 4.) The coating is then diamond ground to give a total coating thickness (bond coat plus lubricant coat) of 0.025 cm. Appendix 1 describes the grinding procedure. During grinding, particular care must be taken to avoid smearing of the coating by excessive grinding pressure. Selective removal of the soft phases is prevented by avoiding lapping processes or the use of too soft a grinding wheel for the efficient removal of chromium carbide.

Apparatus and Test Procedure

A pin and disk type of apparatus (fig. 1) was used in this study. A hemispherically-tipped pin is loaded against the disk by means of dead weights. Friction force is continuously measured by means of a temperature compensated strain gauge bridge. Generally, only the disk is coated, but the pin or both specimens may be coated. The pin generates a 51 mm diameter wear track on the disk. Sliding is unidirectional and the velocity is 2.7 m/sec. The specimens

are heated by a low frequency induction coil. The surface temperature of the disk is monitored with an infrared pyrometer capable of measuring temperatures from 100 to 1400 °C \pm 5 percent. Disk surface temperature is measured on the disk wear track 90° ahead of the sliding contact.

Prior to each test, the disks are heated in a vacuum oven at 150 °C for 3 hr to remove any volatile residue remaining from the finishing operation and subsequent handling. Both the pin and the disk are then cleaned with ethyl alcohol, scrubbed with levigated alumina and then rinsed with distilled water and dried.

Usually, the test duration is 1 hr at each of three temperatures, 25, 350, and 760 °C. Rider wear is measured every 20 min by removing the pin and measuring the wear scar diameter on the hemispherical surface from which the wear volume can be calculated. Locating dowels allowed accurate relocation of the pin.

Disk wear is measured after each hour by recording a surface profile of the wear track, computing the area of removed/displaced coating, and multiplying by the circumference of the wear track to obtain the wear volume.

The atmosphere of the test chamber is hydrogen or helium; with purities of 99 and 99.997 percent respectively. The gases are routed through a flow meter at a rate of 0.014 m³/min. The volume of the test chamber is 0.002 m³.

Initially, the chamber is purged with nitrogen for 10 min before introducing the test gas. The chamber is then thoroughly purged with the test gas before beginning the friction and wear experiments.

EXPERIMENTAL RESULTS

Table 5 gives the results obtained in the friction and wear experiments with various pin materials sliding on PS200. This coating had previously (3) shown promise as a back-up lubricant for foil bearings. Therefore, it was used

to select a single counterface material for the optimization study. Based upon the results shown, alloy C, a hardenable cobalt-chromium alloy was chosen.

Table 6 summarizes the results of the coating optimization study. The metal-bonded Cr_3C_2 formulation with no added lubricants (PS218) provided adequate wear resistance for the counterface material, but the coating wear factors were higher than with the formulated coatings. (Appendix 2 gives an explanation of wear factors (k) and how they relate to coating performance.) Friction coefficients with PS218 were very high and erratic and there was a strong tendency to transfer coating material to the surface of the counterface material. Eventually, this resulted in PS218 sliding against PS218, a situation where abrasive wear of the parent coating by hard carbide particles lodged in the softer pin material occurs. Although very useful for wear control, in many applications chromium carbide coatings are not truly self-lubricating without solid lubricant additives.

The lowest solid lubricant content was in PS203 and PS204 which contained a total of 15 wt % silver and fluorides. Friction coefficients were very high, about 0.5, for both compositions. Friction coefficients for PS200 (80 wt % metal-bonded Cr_3C_2 -10 wt % Ag-10 wt % eutectic) were typically 0.25 to 0.35 in helium and somewhat lower in hydrogen. Wear factors were also lower than those of the previous coatings. Two other compositions with 1:1 ratios of the additives, but in higher percentages were formulated and evaluated. PS212 contains 15 percent each of the additives and PS213 contains 20 percent of each. PS212 exhibited lower friction and coating wear than PS200. Friction coefficients with PS213 were about the same as PS212, 0.20 to 0.28, but coating wear was higher. See Table 6. Therefore, for a 1:1 ratio of the two solid lubricant additives, a total additive content of about 30 percent appears to be near optimum for this coating system.

Two additional compositions were evaluated to determine whether both silver and the fluoride eutectic were really required in the coating composition to achieve satisfactory friction and wear performance over the desired temperature range. PS215 contains 30 percent silver and no fluoride eutectic, while PS216 contains 30 percent fluoride eutectic and no silver. PS215 exhibited a very erratic stick-slip behavior at 760 °C, and coating wear was high. Excessive transfer of silver to the pin occurred. At 350 °C, the same behavior occurred but in a less severe manner. At room temperature, sliding was smooth and a steady friction coefficient of 0.23 was observed. This behavior indicates that silver is a good low temperature lubricant, but (except when used as a thin film lubricant) transfers excessively at elevated temperatures.

For PS216 (the composition containing eutectic but no silver) the friction coefficients are about 0.4. This is approximately one-third lower than the unformulated Cr_3C_2 . However the coating experienced heavy wear and transfer to the pin tip. The results indicate that the addition of eutectic alone in the composite lowers the friction compared to the unmodified metal bonded chromium carbide at all temperatures but increases the coating wear especially at room temperature.

Discussion of Experimental Results

The Cr_3C_2 coating system lubricates best when combined with both silver and the eutectic. In fact, the friction and wear properties of the coating are substantially improved with the addition of silver and eutectic up to an additive content of 30 percent. The mechanism for this behavior appears to be the following: when a counterface material is slid against the unformulated coating, some carbide particles adhere to the counterface surface. When carbide transfer occurs, the sliding condition becomes one of transferred chromium carbide particles sliding against the parent coating. The pin then abrasively wears the coating. On the other hand, when silver and the eutectic are added

to the coating composition, the soft lubricants tend to form a lubricating film that has the effect of preventing carbide transfer. The friction and wear characteristics of the composite coating are continuously improved with additional solid lubricant until the concentration of the metal-bonded carbide is insufficient to form a continuous network, thus weakening the coating. This weakening leads to ploughing of the lubricants by the counterface material tending to increase friction and coating wear. The data indicates that the matrix breakdown begins at a metal-bonded chromium carbide composition of about 60 percent.

Interestingly, the compositions that were lacking in one lubricant or another, PS215 and PS216, behaved very much like the unmodified Cr_3C_2 . They both exhibited excessive coating to pin transfer and coating wear was generally high. It is only when both lubricants are present that an effective lubricating film forms on the surface.

EDS X-Ray analyses of the wear specimens verify that selective transfer of the coating components to the rider does occur. For the unmodified coating (PS218), the carbide transfers and imbeds into the rider more predominantly than the Ni-Al binder metal. Figure 2 shows the relative concentrations of chromium, nickel, and aluminum outside and on the wear scar. The coating transfer to the pin results in a chromium carbide enriched surface film on the pin which further wears the disk coating. A buildup of the soft phase materials, namely the nickel and the aluminum, occurs at the sliding inlet. This indicates that only the harder material is retained on the pin wear surface while the softer binder material characteristically accumulates at the inlet to the sliding contact.

On the other hand, in a coating containing silver and the eutectic the opposite occurs; the soft phase materials transfer to the pin while the carbide transfer is actually blocked. This effect is illustrated with PS212 (15 wt %

silver and eutectic) in Fig. 3. Clearly, this situation is beneficial to lubrication as the soft lubricants provide a lubricative rather than an abrasive film between the sliding materials.

Both the silver and the eutectic act synergistically to improve the lubrication properties of the coating. Having only one of the two in the coating does not prevent abrasive wear of the coating and the counterface material. Also, each lubricant alone can only lubricate over a relatively narrow temperature range. When both lubricants are present in the coating, the temperature range in which the system can lubricate widens to include the temperature ranges of both the silver and the eutectic.

CONCLUSIONS

In a composition optimization study of plasma sprayed, chromium carbide-base coating, it is demonstrated that tribological performance can be greatly improved by the addition of silver and of $\text{BaF}_2/\text{CaF}_2$ eutectic to the coating composition. Friction and wear experiments were conducted in helium and hydrogen atmospheres at 25, 350, and 760 °C and the following general results were obtained:

(1) The mechanism describing the effect of the lubricant additives on the coating system is: in the absence of the solid lubricant additives, chromium carbide particles transfer from the coating and imbed into the surface of the softer counterface pin alloy. This leads to abrasive wear of the remaining parent coating. The presence of silver and $\text{BaF}_2/\text{CaF}_2$ additions in 1:1 ratio amounts were effective in reducing friction and wear. These additions formed lubricative films on the sliding surfaces which effectively inhibited transfer of carbide particles to the counterface material and prevented self-abrasion of the coating.

(2) The best of the counterface alloys evaluated for these coatings was a hardenable cobalt alloy C. Favorable results were also obtained with a formulated coating sliding against itself.

(3) The optimum results were obtained with alloy C sliding on a coating consisting of 70 wt % bonded chromium carbide and 15 wt % each of silver and $\text{BaF}_2/\text{CaF}_2$ eutectic (PS212). Friction coefficients were typically 0.2 ± 0.05 and very low wear was observed from 25 to 760 °C.

(4) The results of this study suggest that some of the compositions described may be valuable for coating sliding contact components in advanced heat engines. The results in hydrogen are especially applicable to the Stirling engine.

APPENDIX 1

RECOMMENDED GRINDING PROCEDURE

1. Use a diamond grinding wheel only.
2. Use water as lubricant - use no oil.
3. Initial grinding depth should be 0.0025 cm.
4. Final cuts should be 0.001 to 0.0015 cm.

Taking too deep a cut, i.e., 0.01 cm, will pluck softer phases (Ag and $\text{BaF}_2/\text{CaF}_2$) from surface.

Taking too light a cut, i.e., less than 0.001 cm, will smear the metal-bonded chromium carbide. This will result in an "Orange Peel" type finish.

5. Ground surface should be matte not glossy and have a speckled appearance representing the three separate phases.

APPENDIX 2

EXPLANATION OF WEAR FACTORS

The wear factor (K) used in this paper is a coefficient which relates the volume of material worn from a surface to the distance slid and the normal load at the contact. Mathematically, K is defined as:

$$K = \frac{V}{(S \times W)}$$

where:

W the normal load at the sliding contact in kilograms

S the total distance slid in centimeters

V the volume of material worn away in cubic centimeters

The physical interpretation of the numeric value of the K factor is as follows:

$K = 10^{-8} \text{ cm}^3/\text{cm-kg}$ high wear rate

$K = 10^{-9} \text{ to } 10^{-10} \text{ cm}^3/\text{cm-kg}$ moderate to low wear rate

$K = 10^{-11} \text{ cm}^3/\text{cm-kg}$ very low wear rate

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TABLE 1. - NOMINAL COMPOSITION AND ROCKWELL HARDNESS OF CANDIDATE PIN MATERIALS

[Compositions taken from manufacturer's data Hardness values taken at room temperature.]

Pin material	Element, wt %														Rockwell hardness
	Ni	Cr	Co	C	Fe	Al	Si	Ti	Mo	Mn	B	W	N	Cb	
Alloy A	70	16	1	0.1	7.5	1	----	2.5	----	1	---	-	----	---	R31C
Alloy B	18	18	--	.2	54.6	-	0.3	---	7.5	.15	0.7	-	0.12	0.4	R18C
Alloy C	2	30	59	1	1	-	.75	---	.75	1.25	---	4	----	---	R40C
Alloy D	8	18	--	1	61.8	-	4.0	---	----	8	---	-	.12	---	R28C

TABLE 2. - COMPOSITION OF THE THREE MAJOR COATING COMPONENTS

Component	Composition, wt %	Particle size
Bonded Chromium Carbide		
Ni	28	-200 + 400 Mesh
Al	2	
Cr ₃ C ₂	58	
Co	12	
Silver Metal		
Ag	100	-100 + 325
Eutectic		
BaF ₂	62	-200 + 325
CaF ₂	38	

TABLE 3. - COMPOSITION AND IDENTIFICATION NUMBER OF COATINGS EVALUATED

Composition	Wt %		
	Bonded Cr ₃ C ₂	Silver	Eutectic
PS200	80	10	10
PS203	85	5	10
PS204	85	10	5
PS212	70	15	15
PS213	60	20	20
PS215	70	30	0
PS216	70	0	30
PS218	100	0	0

TABLE 4. - TYPICAL PLASMA SPRAY PARAMETERS

Parameter	Material, value
Arc gas	Argon 1.4 m ³ /hr
Powder carrier gas	Argon 0.4 m ³ /hr
Coating powder flow rate	1 kg/hr
Amperage	450 to 475 A
Voltage	32 V
Gun to specimen distance	≈15 cm

TABLE 6. - FRICTION AND WEAR SUMMARY FOR VARIOUS COATINGS AGAINST ALLOY C
IN HELIUM AND HYDROGEN

[Test conditions: 0.5 kg, load; 2.7 m/s, sliding velocity; and
38.97 kPa, chamber pressure]

Coating, PS number	Atmosphere	Temperature, °C	Average, μ	Pin wear factor, K cm ³ /cm-kg	Coating wear factor, K cm ³ /cm-kg
PS200 Cr ₃ C ₂ -Ag-Eut (80-10-10)	He ↓	760	0.35	7x10 ⁻¹¹	9.2x10 ⁻¹⁰
		350	.25	2x10 ⁻¹¹	8.2x10 ⁻¹¹
		25	.38	5x10 ⁻¹¹	1.7x10 ⁻⁹
PS203 (85-5-10)		760	.50	1.5x10 ⁻¹⁰	Average value
		350	.45	Not measurable	3.4x10 ⁻⁹
		25	.47	7.5x10 ⁻¹⁰	1.3x10 ⁻⁹
PS204 (85-10-5)		760	.50	4.3x10 ⁻¹⁰	1.0x10 ⁻⁹
		350	.50	7x10 ⁻¹¹	1.1x10 ⁻¹⁰
		25	.47	1x10 ⁻¹¹	1.1x10 ⁻¹⁰
PS212 (70-15-15)		760	.26	4x10 ⁻¹⁰	5.0x10 ⁻¹⁰
	350	.20	6x10 ⁻¹⁰	2.4x10 ⁻¹⁰	
	25	.21	5x10 ⁻¹¹	8x10 ⁻¹²	
PS213 (60-20-20)	760	.28	3x10 ⁻¹⁰	6x10 ⁻¹⁰	
	350	.20	3x10 ⁻¹¹	4x10 ⁻¹⁰	
	25	.21	2x10 ⁻¹²	2x10 ⁻¹⁰	
PS215 (70-0-30)	760	.35	Not measurable	1x10 ⁻⁸	
	350	.34	Not measurable	Erratic	
	25	.23	4x10 ⁻¹¹	Erratic	
PS216 (70-0-30)	760	.40	Not measurable	8.x10 ⁻⁹	
	350	.42	Not measurable	2.4x10 ⁻⁹	
	25	.42	1x10 ⁻¹¹	1.2x10 ⁻⁸	
PS218 (100-0-0)	760	.60	7x10 ⁻¹¹	2x10 ⁻⁹	
	350	.50	2x10 ⁻¹²	4x10 ⁻¹⁰	
	25	.55	1x10 ⁻¹²	1.4x10 ⁻⁹	
PS218 (100-0-0)	H ₂	760	.50	Not measurable	2.8x10 ⁻⁹
		350	.48	coating trans-	1.2x10 ⁻⁹
		25	.35	fer to rider surface	1.7x10 ⁻⁹

TABLE 5. - SCREENING OF PIN MATERIALS AGAINST PS200 COATING

[Test conditions: 0.5 kg, load; 2.70 m/s, sliding velocity, 38.97 kPa, chamber pressure.]

Pin material	Temperature, °C	Friction coefficient, μ		Wear		Wear	
		He	H2	Pin wear factor, K, $\text{cm}^3/\text{cm-kg}$		Coating wear factor, K, $\text{cm}^3/\text{cm-kg}$	
				He	H2	He	H2
Alloy A	760	0.41	----	4×10^{-10}	-----	-----	-----
	350	.41	----	2×10^{-9}	-----	Average value	-----
	25	.45	----	2×10^{-9}	-----	1.7×10^{-9}	-----
Alloy D	760	.40	----	4×10^{-10}	-----	Average value	-----
	350	.46	----	3×10^{-10}	-----	value	-----
	25	.45	----	2×10^{-10}	-----	1.6×10^{-9}	-----
Alloy B	760	.40	----	6×10^{-10}	-----	Average value	-----
	350	.30	----	3×10^{-10}	-----	value	-----
	25	.30	----	2×10^{-10}	-----	8.6×10^{-9}	-----
PS200	760	.29	----	2×10^{-10}	-----	Too small	-----
	350	.28	----	4×10^{-10}	-----	to mea-	-----
	25	.31	----	6×10^{-10}	-----	sure	-----
Alloy C	760	.35	0.20	7×10^{-11}	5×10^{-12}	9.2×10^{-10}	9×10^{-10}
	350	.25	.25	2×10^{-11}	1×10^{-11}	8.2×10^{-11}	6×10^{-10}
	25	.38	.24	5×10^{-11}	2×10^{-11}	1.7×10^{-9}	2×10^{-10}

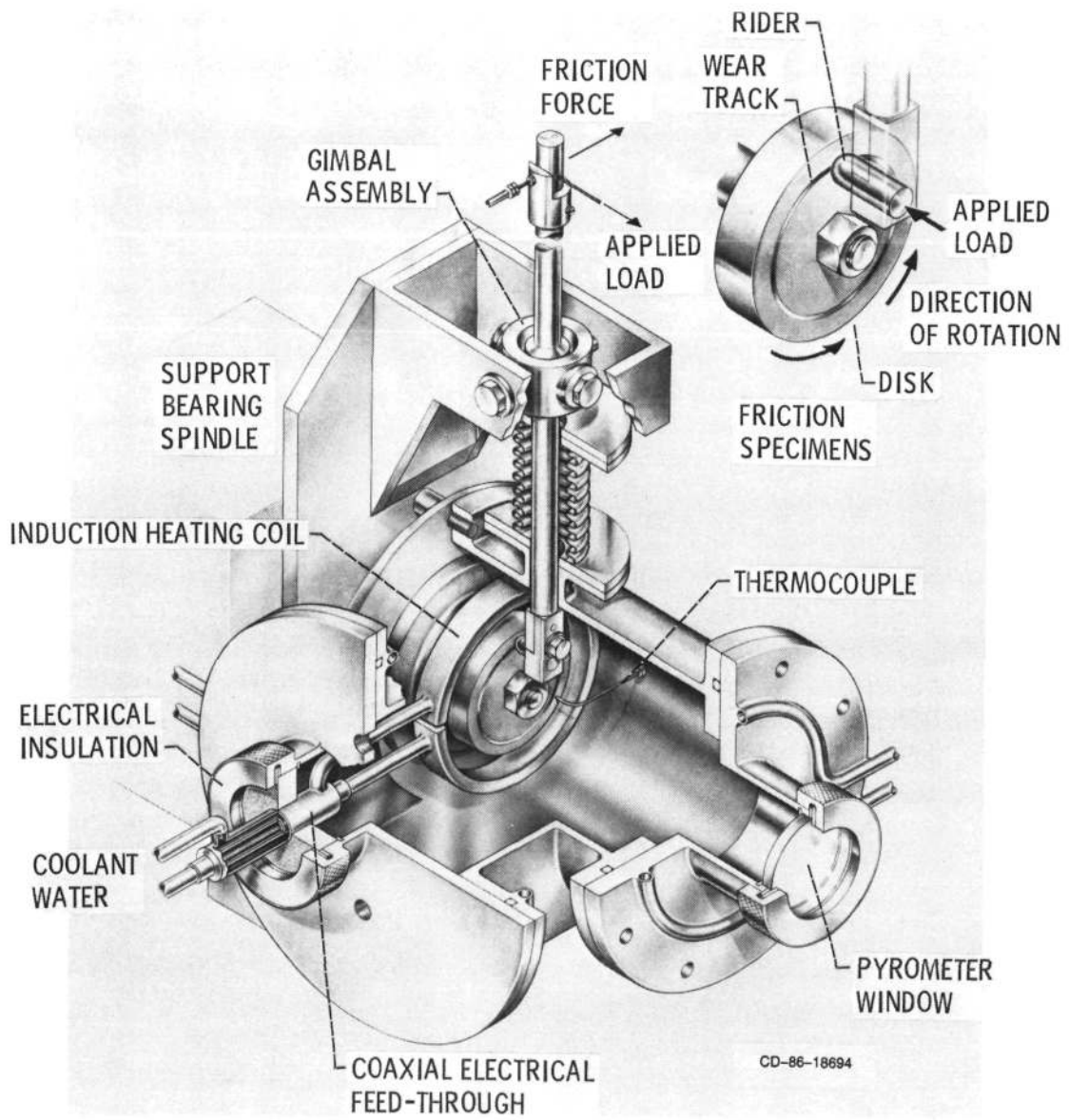
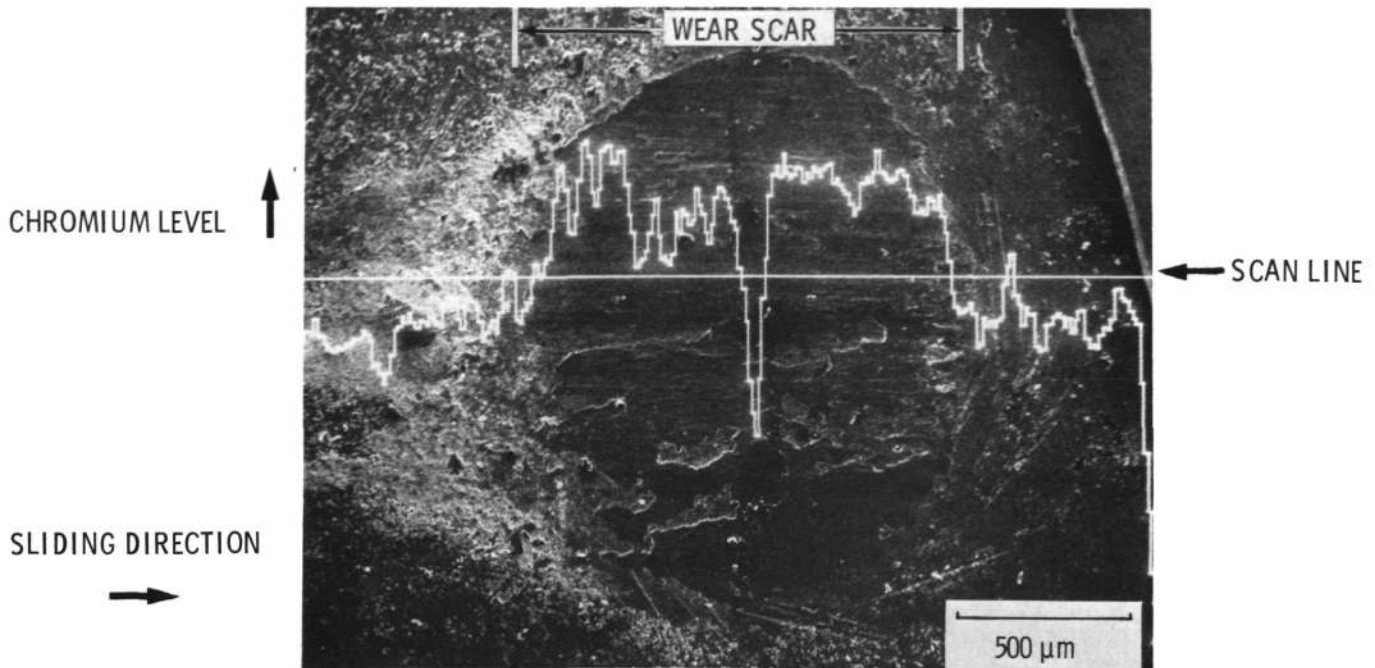
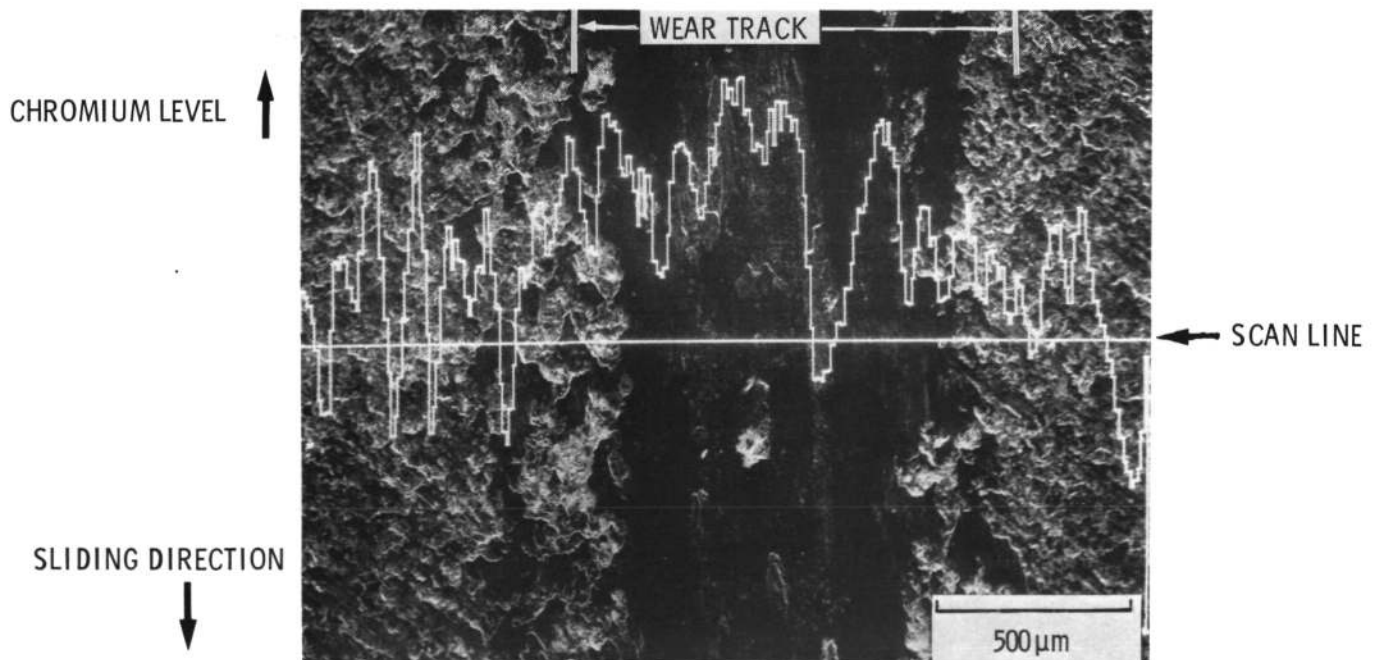


Figure 1. - High-temperature friction apparatus.

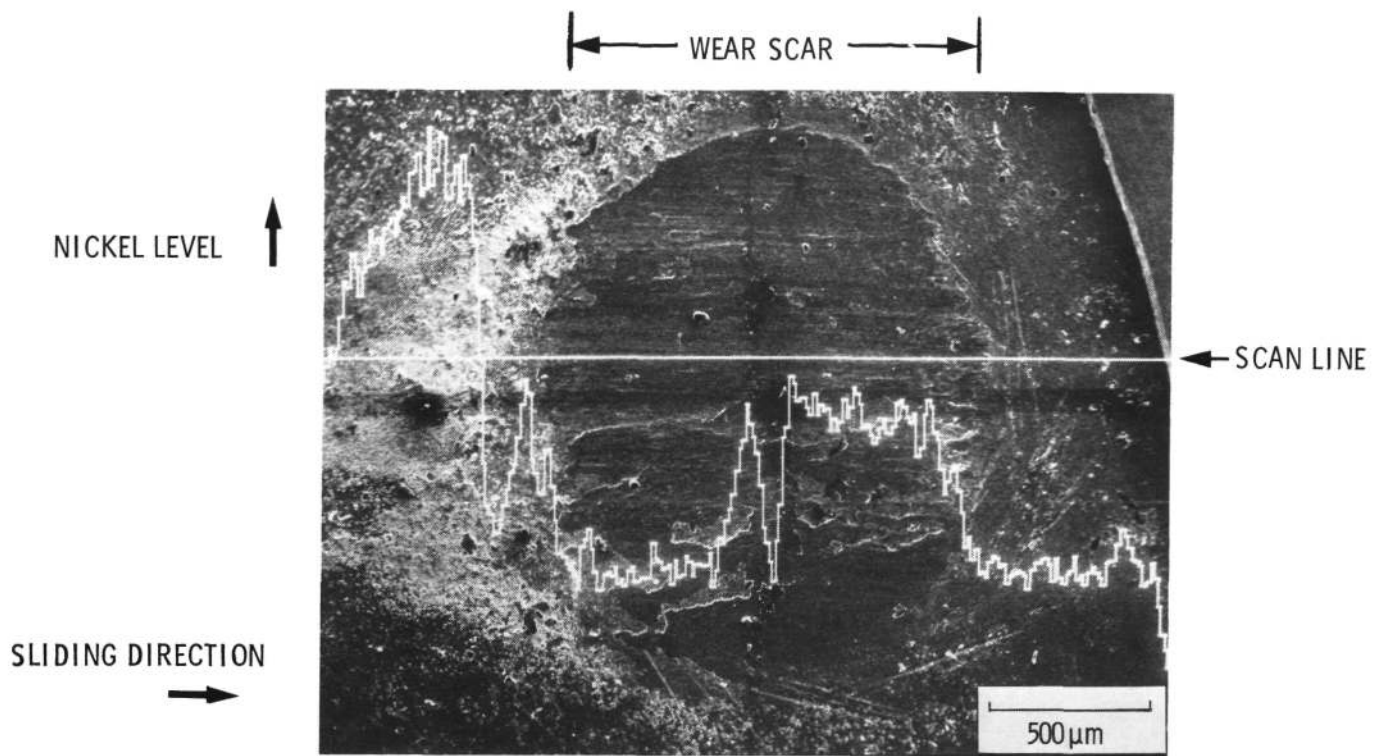


(a) Chromium distribution in vicinity of wear scar.

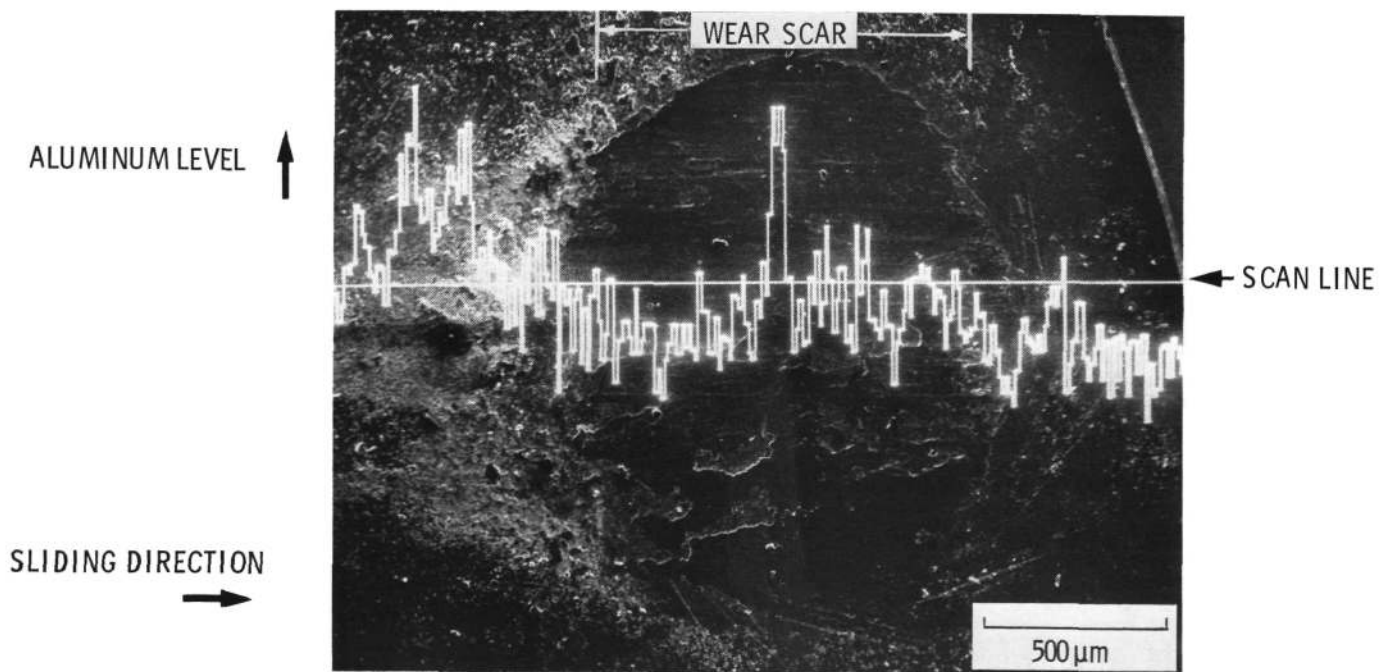


(b) Chromium distribution in vicinity of wear track.

Figure 2. - EDS x-ray distributions on wear specimens: PS218C (without solid lubricant additives) after sliding against hardened cobalt alloy C. Test conditions: helium atmosphere, 0.5 kg load, 2.7 m/s, slid for 3 hrs.

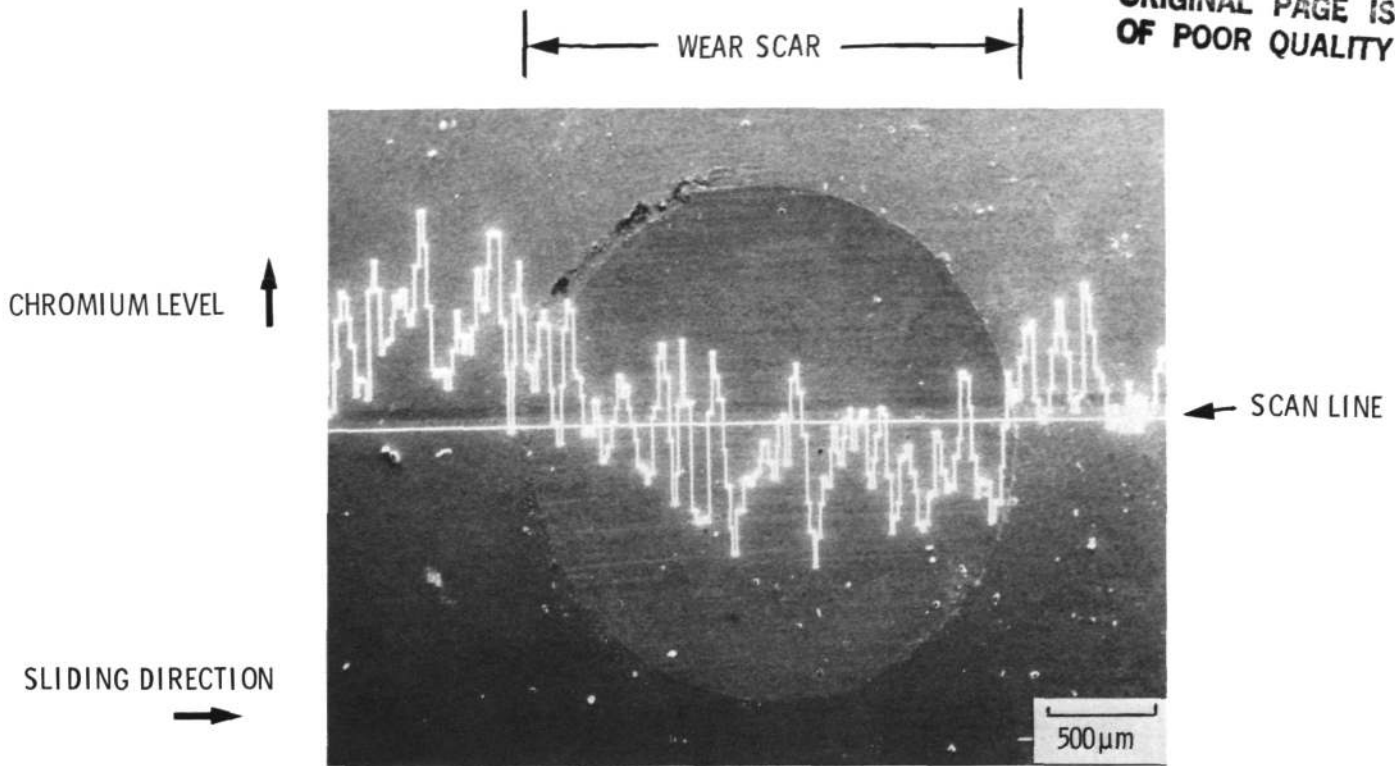


(c) Nickel distribution in vicinity of wear scar.

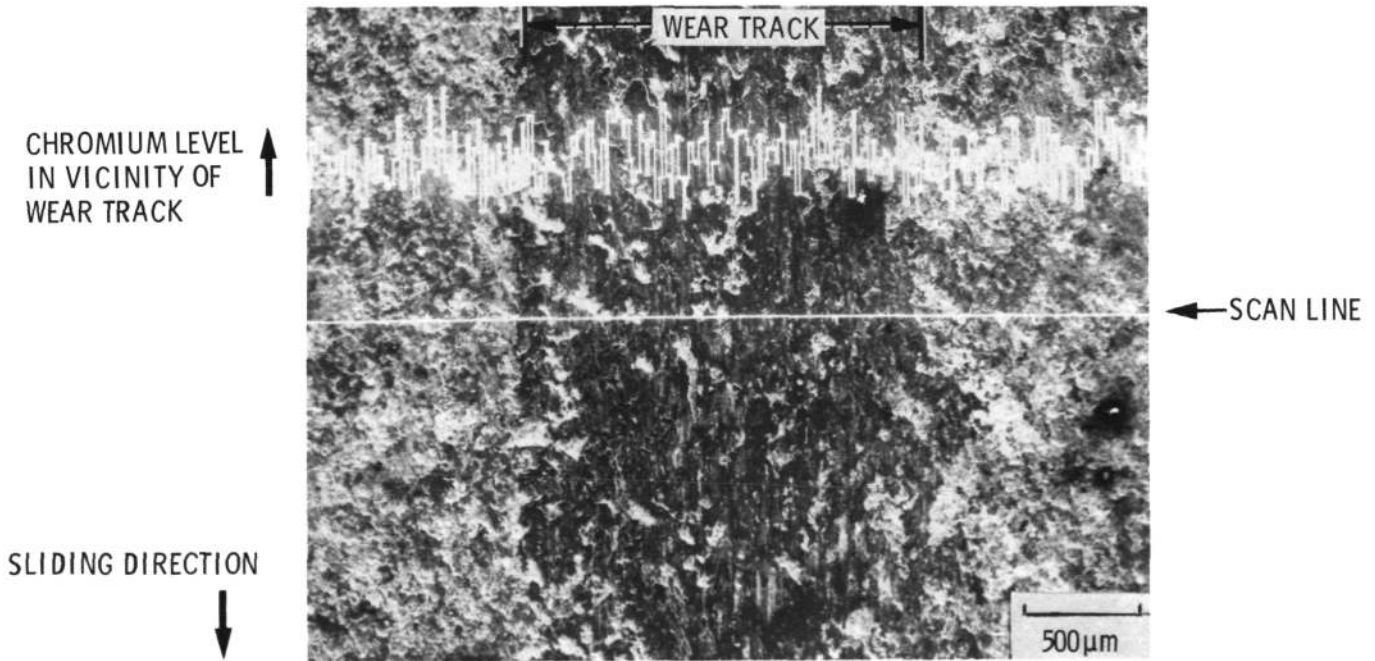


(d) Aluminum distribution in vicinity of wear scar.

Figure 2. - Concluded.

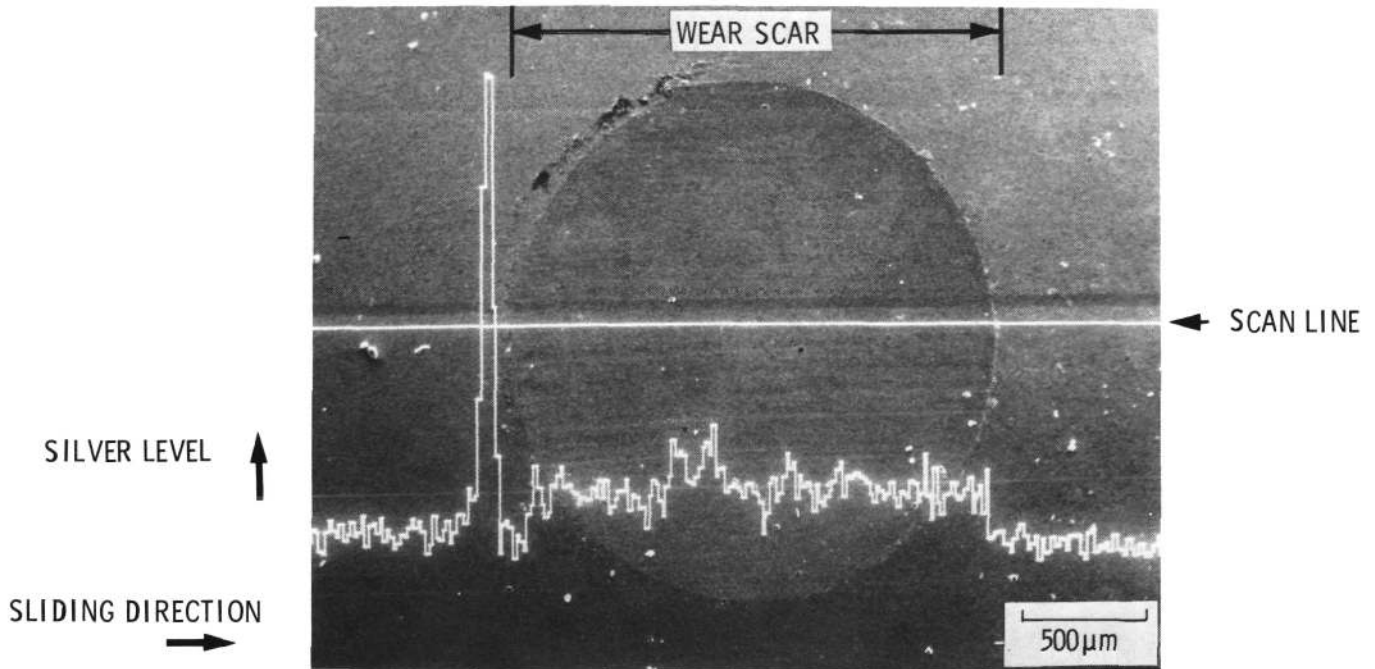


(a) Chromium distribution in vicinity of wear scar.

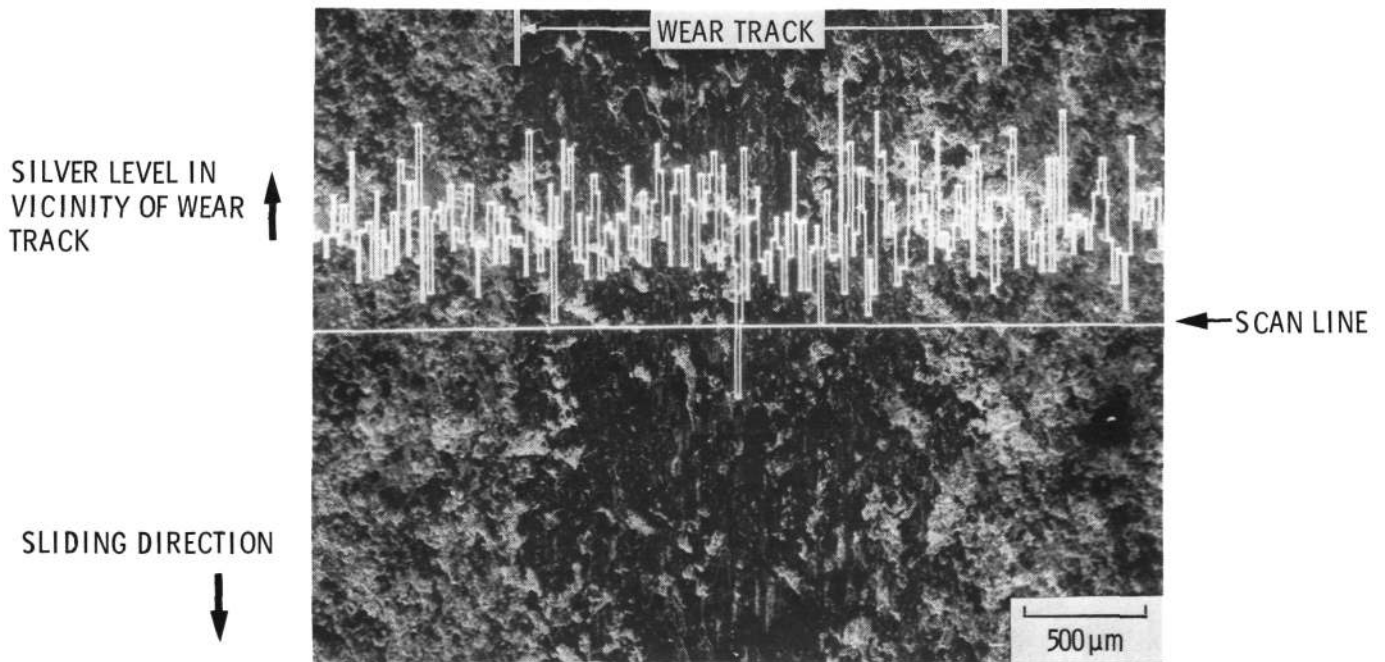


(b) Chromium distribution in vicinity of wear track.

Figure 3. - EDS x-ray distributions on wear specimens: P212(with solid lubricant additives) after sliding against hardened cobalt alloy C. Test conditions: helium atmosphere, 0.5 kg load, 2.7 m/s, slid for 3 hrs.

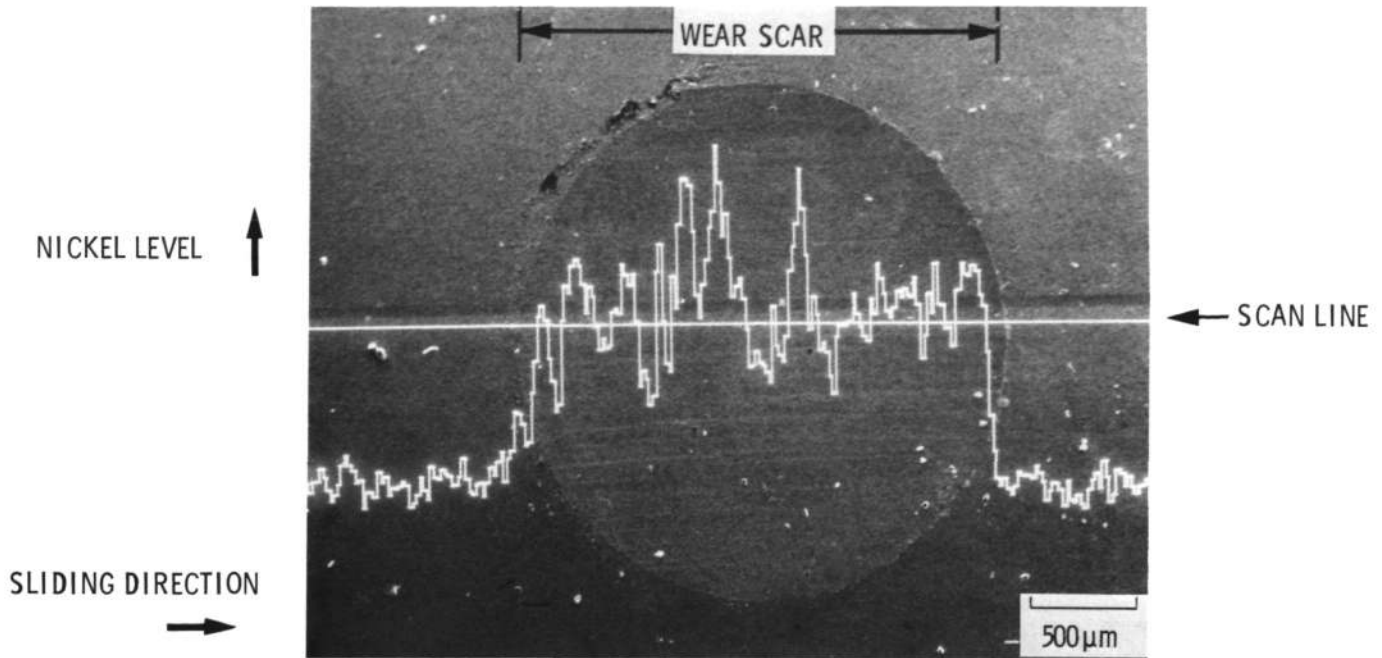


(c) Silver distribution in vicinity of wear scar.

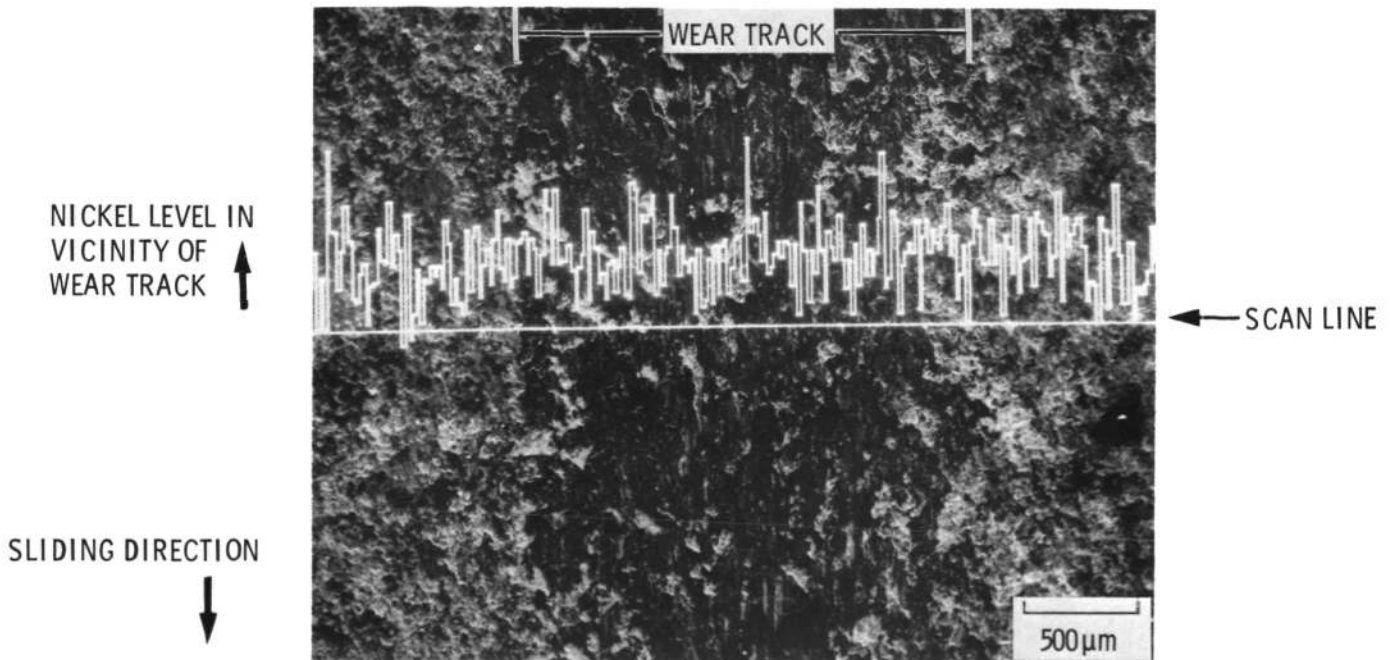


(d) Silver distribution in vicinity of wear track.

Figure 3. - Continued.



(e) Nickel distribution in vicinity of wear scar.



(f) Nickel distribution in vicinity of wear track.

Figure 3. - Concluded.

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16. Abstract This paper describes new compositions of self-lubricating coatings that contain chromium carbide. A bonded chromium carbide was used as the "base stock" because of the known excellent wear resistance and the chemical stability of chromium carbide. "Additives" were silver and barium fluoride/calcium fluoride eutectic. The coating constituents were treated as a ternary system consisting of: (1) the bonded carbide base material, (2) silver, and (3) the eutectic. A study to determine the optimum amounts of each constituent was performed. The various compositions were prepared by powder blending. The blended powders were then plasma sprayed onto superalloy substrates and diamond ground to the desired coating thickness. Friction and wear studies were performed at temperatures from 25 to 760 °C in helium and hydrogen. A variety of counterface materials were evaluated with the objective of discovering a satisfactory metal/coating sliding combination for potential applications such as piston ring/cylinder liner couples for Stirling engines.			
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