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Summary

To minimize the segregation of the powder mixture during the plasma spraying of NASA LUBE PS106 (35 wt % NiCr–35 wt % Ag–30 wt % CaF_2), monoaluminum phosphate was added to form agglomerate particles. Three concentrations of $AlPO_4$ were added: 1.25 (coating 1), 2.5 (coating 2), and 6.25 wt % (coating 3).

Specimens of coating surfaces were prepared in standard metallographic mounts. They were diamond polished and lightly etched before analysis. The Image Analyzer Quantitative Metallurgical System (IA) provided automatic assessment and computation of the area and the percentage of area of the components in each specimen. Measurements were made of the lightest area (Ag) and of all other light areas (Ag plus NiCr). The dark area (CaF_2 plus impurities and voids) was obtained by difference. The X-ray intensity of each element was measured with the Electron Microprobe X-ray Analyzer (EMXA), compared to a 100-percent-pure standard, and computer corrected by the U.S. Bureau of Standards COR 2 program. Photomicrographs of coating cross sections and surfaces, backscatter electron photomicrographs, and X-ray maps are presented.

The results show that using 2.5 wt % $AlPO_4$ kept the coating composition closest to specification. With 1.25 wt % $AlPO_4$, the coatings were deficient in CaF_2 (7 percent low by IA and 13 percent low by EMXA). With 6.25 percent $AlPO_4$ the coatings contained excess CaF_2 (3 percent high by IA and 10 percent high by EMXA). Also, impurities and voids increased and there was a deficiency in silver.

The methods of IA and EMXA complement each other, and the reasonable agreement in the results increases confidence in determining the coating composition. IA appears to be adequate for coating characterization and is recommended because it is simpler, less expensive, and more readily treated by statistical analysis.

Introduction

Higher efficiency gas-turbine engines require higher operating temperatures, which demand improved high-temperature materials throughout the engine. Because most lubricating oils oxidize above 250° C, composites are needed that are self-lubricating not only at high temperatures, but also at lower startup temperatures. Another demand for these composites is in airframe bearings for the space shuttle. They must be self-lubricating in the very cold vacuum of space as well as in air at reentry temperatures.

At the NASA Lewis Research Center self-lubricating, multicomponent coatings have recently been developed that lubricate over a wide range of operating conditions (refs. 1 to 4). These coatings have been successfully applied by plasma spraying mixed powders onto superalloy substrates. They have been evaluated in friction and wear experiments and used in some journal bearings. The coatings consist of mixtures of silver, NiCr, glass, and CaF_2. They are self-lubricating from cryogenic temperatures to about 900° C, fresh lubricant being replenished at the rubbing surface by the wear process itself (ref. 4).

Plasma-spray processing technology is very complex. A number of variables make it difficult to prepare reproducible coatings with uniformity of chemical composition, microstructure, and mechanical properties. Under a NASA contract to determine the effects of some of these variables (ref. 5), monoaluminum phosphate was added to agglomerate the powder particles of the various components, thereby minimizing powder segregation during handling and plasma spraying.

However, it is difficult to quantitatively characterize uniformity of structure. Subjectivity is involved in most data interpretations. Statistical analysis can be helpful in certain situations, if it can be assumed that most variables are controlled.

This study was conducted to determine, by two widely different methods, the coating composition of three plasma-sprayed, self-lubricating composites and to compare the results with each other and with the nominal composition of the original powder. One method makes use of an image analyzer, which distinguishes components by their different reflectivities in a cut, polished, and etched section of the sprayed coating. The other method makes use of an electron microprobe X-ray analyzer in an area scan mode. The X-ray counts are computer corrected to give an elemental chemical analysis of the scanned surface.

This study was made on a three-component (35 wt % NiCr–35 wt % Ag–30 wt % CaF_2) plasma-sprayed coating, NASA LUBE PS106, with three concentrations of monoaluminum phosphate—1.25 (coating 1), 2.5 (coating 2), and 6.25 wt % (coating 3). All other controllable variables of the plasma spray process were held constant. The microstructural data and details of the analytical process are presented in this report.

Materials and Procedures

Coating Compositions and Preparation

The coatings used in this study were prepared by the Illinois Institute of Technology Research Institute (IITRI) under NASA contract to optimize the powder processing and plasma spray parameters for NASA LUBE PS106. The three coatings analyzed for this report were prepared by using constant spraying parameters. The only variable is the $AlPO_4$ concentration in the powder mixture. The compositions, the powder preparation, and the spraying parameters are described in detail in reference 5. The compositions of the powder batches prepared for plasma spraying of NASA LUBE PS106 are shown in table I.

Constituent powders were weighed and mixed to make a nominal PS106 composition. Then an aqueous solution of $AlPO_4$ was added to the mixed powders to give the desired concentration of $AlPO_4$ by weight. The mixtures were heated to drive off the water and then ground and sieved to give a $-70, +325$ mesh powder. The CaF_2 was added in two steps: half with the solution, and the other half after sieving. Two types of CaF_2 were used: fine particles (nominally 5 μm) and coarser particles ($-150, +325$ mesh, nominally 80 μm). Scanning electron microscope (SEM) photomicrographs of the powder batches for the three coatings are shown in figure 1 (from ref. 5): Batch 1 used the coarse CaF_2 for both steps. Figure 1(a) shows that agglomerates did not form as desired. Batches 2 and 3 both used fine CaF_2 initially, with coarse CaF_2 added later. Batch 2, with 2.5 wt % $AlPO_4$, showed good agglomeration (fig. 1(b)). Batch 3, with 6.25 wt % $AlPO_4$, appeared to have formed more agglomerates composed mainly of CaF_2 rather than the desired CaF_2-NiCr-Ag composite particles (fig. 1(c)). The powders were plasma sprayed onto René 41 substrates by IITRI in a square array of eight bars, each 5 mm by 50 mm, as shown in figure 2. A bar from each set was cut in half for metallographic mounting at Lewis. One piece was mounted so that a cross section of the coating and substrate could be viewed; the other was mounted so that the plasma-sprayed coating was parallel to the plane of view. Specimens were diamond polished, photographed as polished, and then lightly etched with a solution of $HCl + H_2O_2$ and rephotographed.

Photomicrographs of the three plasma-sprayed coatings are shown in figures 3 to 5. These photomicrographs were taken after polishing but before etching. Photomicrographs of the same specimens after etching are shown in figures 6 to 8. In each set of photomicrographs the upper left one shows a view of the coating cross section at a magnification of 100; and the upper right one shows a view of the coating surface, also at a magnification of 100. The bottom photomicrographs show the same views at a magnification of 500.

TABLE I. – NOMINAL COMPOSITION OF STARTING POWDER

Powder batch	NiCr	Ag	CaF_2	$AlPO_4$
	Batch composition, wt %			
1	34.57	34.57	29.62	1.25
2	34.13	34.13	29.24	2.50
3	32.82	32.82	28.12	6.25

As previously identified by scanning electron microscope and energy dispersive qualitative analysis, the silver component appears lightest in the photomicrographs and the NiCr component is intermediate in brightness. The CaF_2 component plus the $AlPO_4$, voids, and other impurities all appear dark. After etching, the silver remains bright and the CaF_2 dark, but the NiCr has a mottled appearance and is more clearly separated from the silver. However, the CaF_2 cannot be distinguished from the voids, etc.

Surface Analyses

All analyses were made on specimens mounted with the plasma-sprayed coating parallel to the viewing surface and lightly etched. The IA method provided automatic assessment and computation of the area and the percentage of area of the components in the specimens. Two measurements were made: The first was at a brightness level that included only the silver area. The second measurement was at a brightness level that included both the NiCr area and the silver area. Then two calculations were made: The first measurement was subtracted from the second to obtain the NiCr area. Then the second measurement was subtracted from 100 percent to obtain the dark area (CaF_2 + other). Measurements were made at 10 random locations on each specimen and averaged.

EMXA was used as the alternative method for characterizing the plasma-sprayed coating for elemental content. In this method a beam of electrons strikes the surfaces to be analyzed. X-rays emitted by the elements are directed to a diffraction grating, where they are dispersed according to wavelength. Instrumentation detects each element separately at different wavelengths. For quantitative analysis the microprobe was used in an "area scan" mode; that is, instead of the beam remaining on one spot, the beam was scanned over the entire area to be analyzed. Three features of EMXA were used:

(1) Backscatter electron photographs of the areas to be analyzed

(2) X-ray rastor photographs for each element involved

(3) Area count data for comparison with pure standards to obtain quantitative analysis

Results and Discussion

Three multicomponent, plasma-sprayed coatings were analyzed. The coatings were NASA LUBE PS106 (35NiCr-35Ag-30CaF_2) with three concentrations of $AlPO_4$ added to the spray powder mix: 1.25, 2.5, and 6.25. (All numbers are in weight percent.) Analyses were made on coating surfaces that were diamond polished and lightly etched.

The IA method provided automatic assessment and computation of the area and the percentage of area of the components in the specimens. Ten random locations on each coating surface were analyzed. Table II presents the mean and standard deviations of the area percentage for each component. To compare these IA results with the specified starting powder composition and electron microprobe results, the area percent data given in table II had to be converted into weight percent. Each mean area percent fraction was assumed to be equal to volume percent and multiplied by the component density. The densities of NiCr, silver, and CaF_2 were 8.36, 10.5 and 3.18 g/cm^3, respectively. The average density of each coating was calculated, and the weight percent ratio was determined for each component as shown in table III. Note that coating 2 was closest to specification. Also, it had less scatter, implying a more uniform composition. This indicates that 2.5 wt % $AlPO_4$ was the best concentration for the powder mix.

The EMXA-generated photographs are shown in figures 9 to 11. The backscatter electron photograph is shown in the upper left. Heavier atomic elements show up brightest, so the light phase in these photographs is silver; the gray phase, NiCr; and the black, CaF_2. This is further substantiated by the X-ray photographs: Silver is on the upper right; nickel and chromium, in the middle; and calcium and fluoride, on the bottom. Some aluminum was present from the $AlPO_4$, but the amount was so small that it was not clearly distinguishable from the background.

Quantitative analysis with EMXA was done by scanning these areas. The X-ray intensity of each element was first measured on the high-purity elemental standard presented in table IV. Next the intensity of each element in the coating was measured identically and ratioed to the standard. Count data were computer corrected for background, atomic number, absorption, fluorescence, etc., to obtain final results in weight percent of NiCr, silver, and CaF_2. Weight percent of $AlPO_4$, voids, and impurities were obtained by difference. Details of the U.S. Bureau of Standards COR 2 computer correction program used are reported in reference 6. Measurements, calculations, and the final stoichiometric analyses for the three coatings of NASA LUBE PS106 (with 1.25, 2.5, and 6.25 wt % $AlPO_4$) are presented in table V. Note that the amount of CaF_2 increased with increased $AlPO_4$ concentration.

TABLE III. – COATING COMPOSITION AS DETERMINED BY IMAGE ANALYSIS

NASA LUBE PS106	NiCr	Ag	CaF_2 + other
	Coating composition, wt %		
Coating 1 (with 1.25 wt % $AlPO_4$)	37.8 ± 7.6	37.9 ± 5.6	24.3 ± 2.9
Coating 2 (with 2.5 wt % $AlPO_4$)	33.2 ± 3.3	36.4 ± 5.0	30.4 ± 1.6
Coating 3 (with 6.25 wt % $AlPO_4$)	39.1 ± 6.7	23.5 ± 7.2	37.4 ± 3.3

TABLE II. – AREA PERCENTAGE OF COATING COMPONENTS AS DETERMINED BY IMAGE ANALYSIS

NASA LUBE PS106	Measured by IA		Calculated by difference	
	Ag	Ag + NiCr	NiCr	CaF_2 + other
	Area percentage (mean and standard deviation)			
Coating 1 (with 1.25 wt % $AlPO_4$)	22.9 ± 3.4	51.6 ± 5.2	28.7 ± 5.8	48.4 ± 5.2
Coating 2 (with 2.5 wt % $AlPO_4$)	20.4 ± 2.8	43.8 ± 2.8	23.4 ± 2.3	56.2 ± 2.9
Coating 3 (with 6.25 wt % $AlPO_4$)	12.0 ± 3.7	37.0 ± 5.6	25.0 ± 4.3	63.0 ± 5.6

TABLE IV. – QUANTITATIVE DATA FROM HIGH-PURITY ELEMENTAL STANDARDS BY ELECTRON MICROPROBE X-RAY ANALYSIS (EMXA)

[EMXA conditions: operating voltage, 15 kV; specimen current, 0.05 μA.]

Standard		Diffraction crystal	Spectrometer setting	Standard 10-second counts, I_s	Background counts	Standard minus background counts, I_0
Element	Line					
Ca	$K\alpha$	LiF	3.359	102 250	170	102 080
F	$K\alpha$	RAP	2.824	4 400	20	4 380
Cr	$K\alpha$	LiF	2.290	126 720	320	126 400
Ni	$K\alpha$	LiF	1.659	65 870	540	65 330
Ag	$L\alpha$	KDP	1.572	29 800	280	29 520

TABLE V. – QUANTITATIVE ANALYSIS OF PLASMA-SPRAYED SOLID LUBRICANT COATINGS BY ELECTRON MICROPROBE X-RAY ANALYSIS (EMXA)

[EMXA conditions: operating voltage, 15 kV; specimen current, 0.05 μA.]

Coating element	Coating minus background counts, I_c	I_c/I_0	COR 2 computer-generated analysis, wt %	Final stoichiometric analysis, wt %
NASA LUBE PS106 coating 1 (with 1.25 wt % $AlPO_4$)				
Ca	16 890	0.165	8.79 } CaF_2	CaF_2 = 17.12
F	545	.124	[a](.9482)(8.79) = 8.33 }	NiCr = 36.26
Cr	10 760	.085	8.82 } NiCr	Ag = 38.02
Ni	18 060	.276	27.44 }	Σ = 91.40
Ag	10 030	.340	38.02 Ag	[b]X = 8.60
NASA LUBE PS106 coating 2 (with 2.5 wt % $AlPO_4$)				
Ca	28 470	0.279	14.85 } CaF_2	CaF_2 = 28.93
F	870	.199	[a](.9482)(14.85) = 14.08 }	NiCr = 30.17
Cr	10 670	.084	8.90 } NiCr	Ag = 30.26
Ni	13 670	.209	21.27 }	Σ = 89.36
Ag	7 880	.267	30.26 Ag	[b]X = 10.64
NASA LUBE PS106 coating 3 (with 6.25 wt % $AlPO_4$)				
Ca	37 900	0.371	19.72 } CaF_2	CaF_2 = 38.42
F	1 150	.263	[a](.9482)(19.72) = 18.70 }	NiCr = 25.54
Cr	8 390	.066	7.12 } NiCr	Ag = 21.11
Ni	11 560	.177	18.42 }	Σ = 85.07
Ag	5 410	.183	21.11 Ag	[b]X = 14.93

[a]CaF_2 calculation: at.wt Ca = 40.080; F/Ca = 0.4867/0.5133; ∴ F = 0.9482 Ca.
at.wt F = 2(18.898)/78.076

[b]X = 100 – Σ of analysis. (This includes $AlPO_4$ binder, voids, and impurities.)

Comparisons and Evaluations

The second objective of this study was to compare the results of the two analysis methods (IA and EMXA) with each other and with the nominal compositions of the initial powder batch. This comparison is made in table VI, which summarizes the content of NiCr, silver, and CaF_2 in the three coatings in weight percent, including nominal composition (table I), IA results (table III), and EMXA results (table V). In this table, quantities listed as "Other" include $AlPO_4$, voids, impurities, or any other unaccounted-for constituents. Numbers in parentheses indicate a calculated value.

TABLE VI. – COMPARISON OF COATING COMPOSITION AS DETERMINED BY IMAGE ANALYSIS (IA) AND ELECTRON MICROPROBE X-RAY ANALYSIS (EMXA) TO ORIGINAL POWDER MIX (BATCH)

NASA LUBE PS106		NiCr	Ag	CaF_2	Other	CaF_2 + other
Coating composition,[a] wt %						
Coating 1 (with 1.25 wt % $AlPO_4$)	Batch	34.6	34.6	29.6	1.25	(30.9)
	IA	37.8	37.9	(23.0)	(1.25)	24.26
	EMXA	36.3	38.0	17.1	8.6	(25.7)
Coating 2 (with 2.5 wt % $AlPO_4$)	Batch	34.1	34.1	29.2	2.5	(31.7)
	IA	33.2	36.4	(27.9)	(2.5)	30.4
	EMXA	30.2	30.3	28.9	10.6	(39.6)
Coating 3 (with 6.25 wt % $AlPO_4$)	Batch	32.8	32.8	28.1	6.25	(34.4)
	IA	39.1	23.5	(31.1)	(6.25)	37.4
	EMXA	25.5	21.1	38.4	14.9	(53.3)

[a]Parentheses indicate a calculated value.

Coating 1, with 1.25 wt % $AlPO_4$ in the starting powder, gave compositions that were higher in NiCr (3 percent by IA and 2 percent by EMXA) and silver (3 percent by both) and lower in CaF_2 (7 percent by IA and 13 percent by EMXA) than that indicated for the starting powder. In the EMXA analysis, when the value for "Other" is added to the CaF_2 determination, the total is close to the IA value for "CaF_2 plus other." Therefore, IA and EMXA are in agreement that there is a loss of CaF_2 powder during the coating process.

For coating 2, with 2.5 wt % $AlPO_4$ in the starting powder, NiCr and CaF_2 were both slightly lower than specified. Results with silver were mixed: IA indicating high by 2 percent; and EMXA, low by 4 percent. Both IA and EMXA showed that this coating was closest to the specified composition of NASA LUBE PS106.

For coating 3, with 6.25 wt % $AlPO_4$ in the starting powder, IA indicated 6 percent more NiCr and EMXA 7 percent less NiCr than for the powder batch. Both showed lower silver content, 9 percent by IA and 12 percent by EMXA, and higher CaF_2 content, 3 percent by IA and 10 percent by EMXA, than specified. Thus there appears to be a real deficiency in silver and an excess in CaF_2.

Confidence in determining the coating composition was increased by the reasonable agreement of results from IA and EMXA, which to a degree compensated for the lack of an absolute standard PS106 coating. However, if only one method must be selected, image analysis is recommended because it is simpler, less expensive, and more readily treated by statistical analysis.

Summary of Results

A characterization study was made of three variations of a plasma-sprayed coating, NASA LUBE PS106, specified by weight percent as 35 Ag, 35 NiCr, and 30 CaF_2. Three concentrations of monoaluminum phosphate (1.25, 2.5, and 6.25 wt %) were added to the powder mix to minimize powder segregation during spraying onto René 41 substrates. Image analysis (IA) and electron microprobe X-ray analysis (EMXA) were used to determine coating composition. The following results were obtained:

1. Both IA and EMXA showed that when 2.5 wt % $AlPO_4$ was used to agglomerate the powders before spraying, the coating was closest to specification.

2. With 1.25 wt % $AlPO_4$, CaF_2 was lost during the spraying process. With 6.25 wt % $AlPO_4$ there was an excess of CaF_2, more impurities and voids, and a deficiency in silver.

3. Both IA and EMXA provided satisfactory analysis. The methods complemented each other, and the reasonable agreement in the results increased confidence in determining the coating composition. However, image analysis is recommended because it is simpler, less expensive, and more readily treated by statistical analysis.

Lewis Research Center
National Aeronautics and Space Administration
Cleveland, Ohio, August 20, 1981

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1. Sliney, Harold E: Plasma-Sprayed Metal-Glass and Metal-Glass Fluoride Coatings for Lubrication to 900° C. NASA TM X-71432, 1974.
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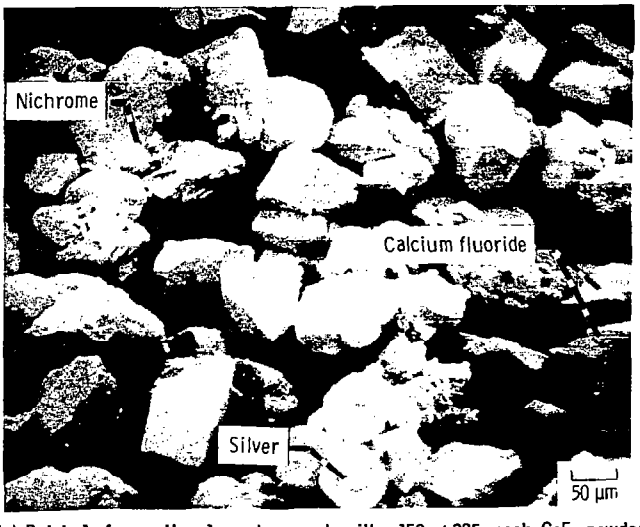
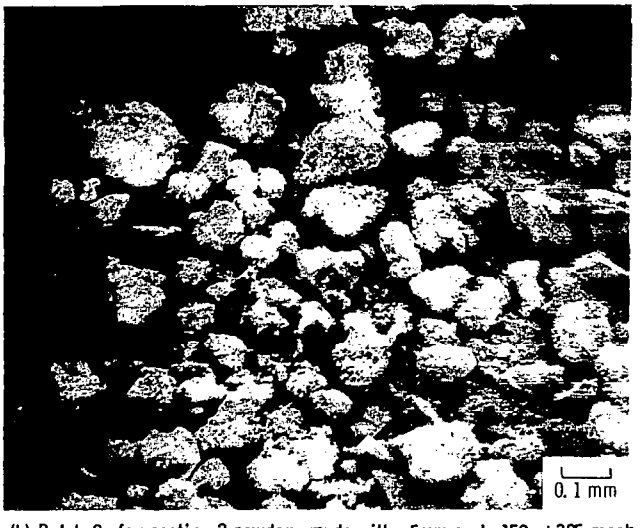
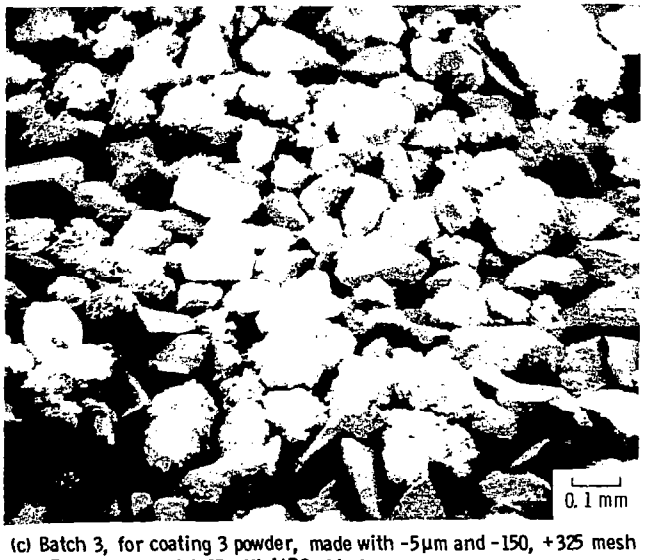
6. Henoc, Jean; Heinrich, Kurt F. J.; and Myklebust, Robert L.: A Rigorous Correction Procedure for Quantitative Electron Probe Microanalysis (COR 2). NBS TN-769, National Bureau of Standards, August 1973.



(a) Batch 1, for coating 1 powder, made with -150, +325 mesh CaF_2 powder and 1.25 wt% $AlPO_4$ binder.



(b) Batch 2, for coating 2 powder, made with -5μm and -150, +325 mesh CaF_2 powders and 2.5 wt% $AlPO_4$ binder.



(c) Batch 3, for coating 3 powder, made with -5μm and -150, +325 mesh CaF_2 powders and 6.25 wt% $AlPO_4$ binder.

Figure 1. - Photomicrographs of powder batches prepared for plasma spraying of NASA LUBE PS106. (From ref. 5.)

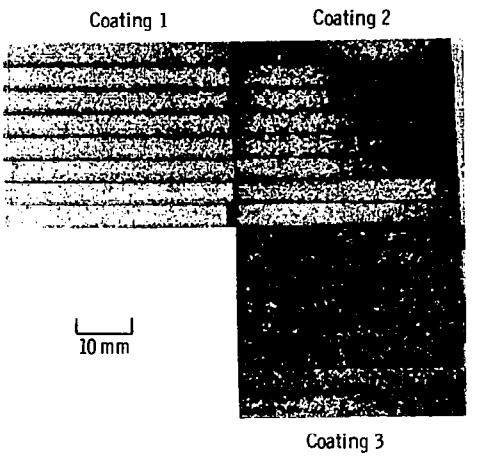
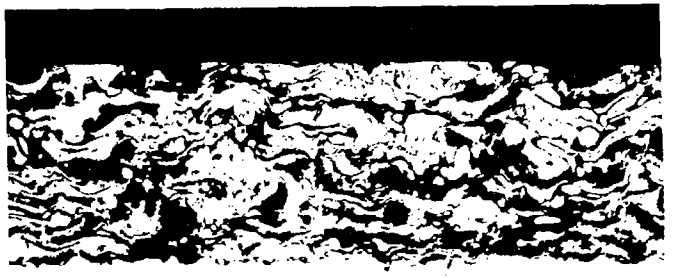
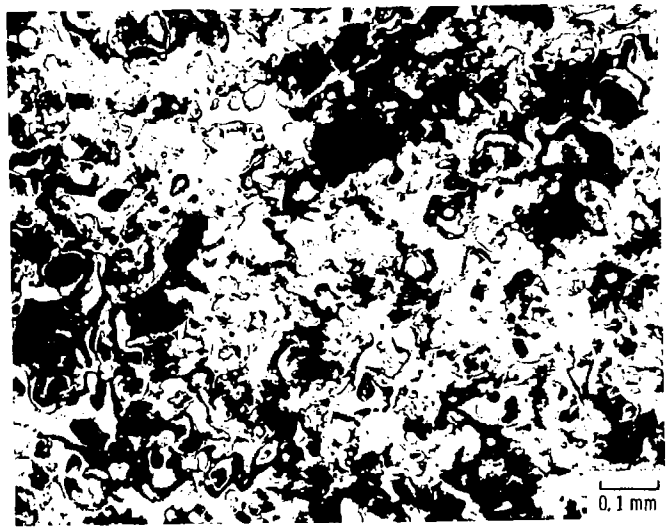


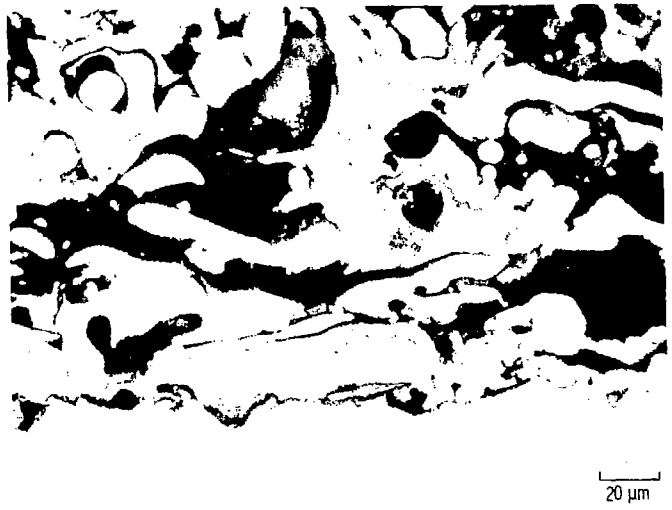
Figure 2. - NASA LUBE PS 106 coatings as sprayed on sets of eight bars (5 mm by 50 mm). (From ref. 5.)



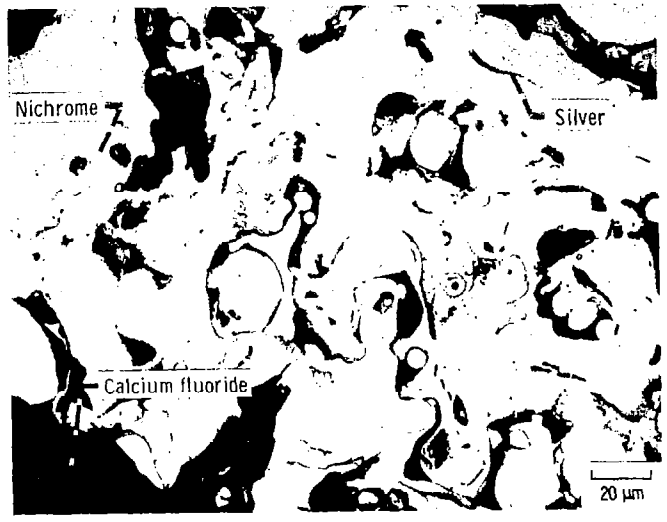
(a) Cross section at magnification of 100.



(b) Surface at magnification of 100.

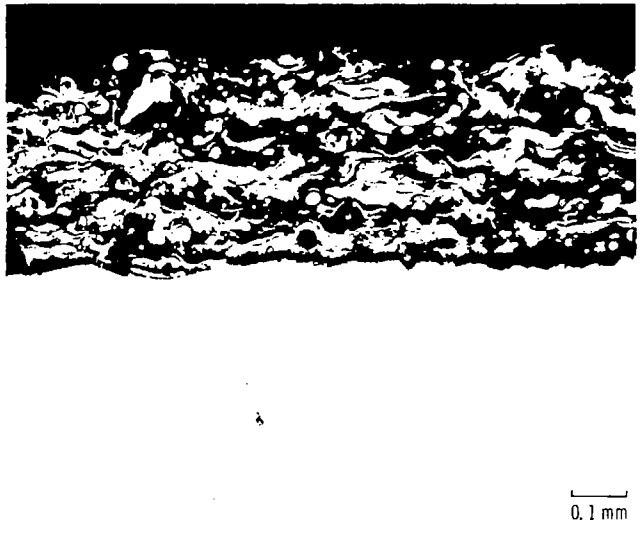


(c) Cross section at magnification of 500.

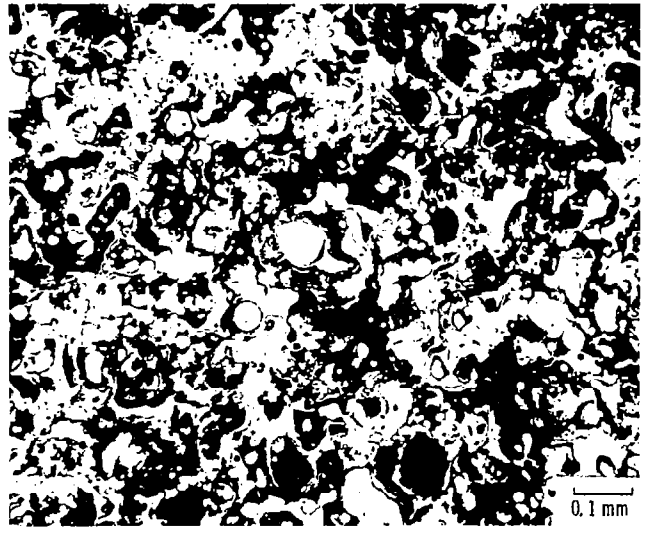


(d) Surface at magnification of 500.

Figure 3. - Photomicrographs of NASA LUBE PS106 coating 1 (with 1.25 wt % $AlPO_4$).

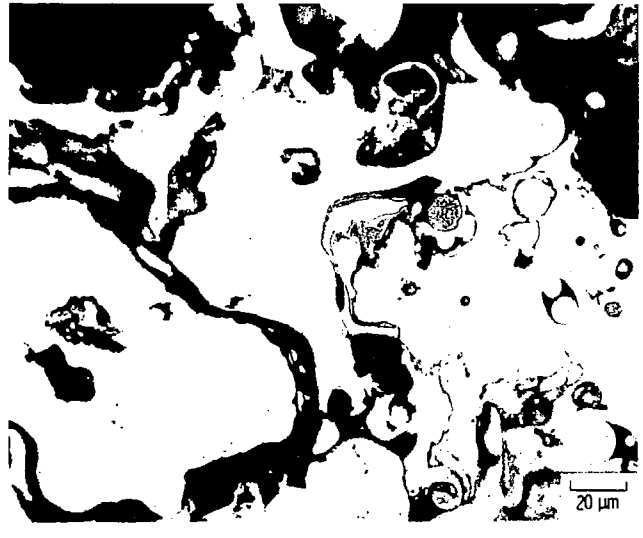


(a) Cross section at magnification of 100.



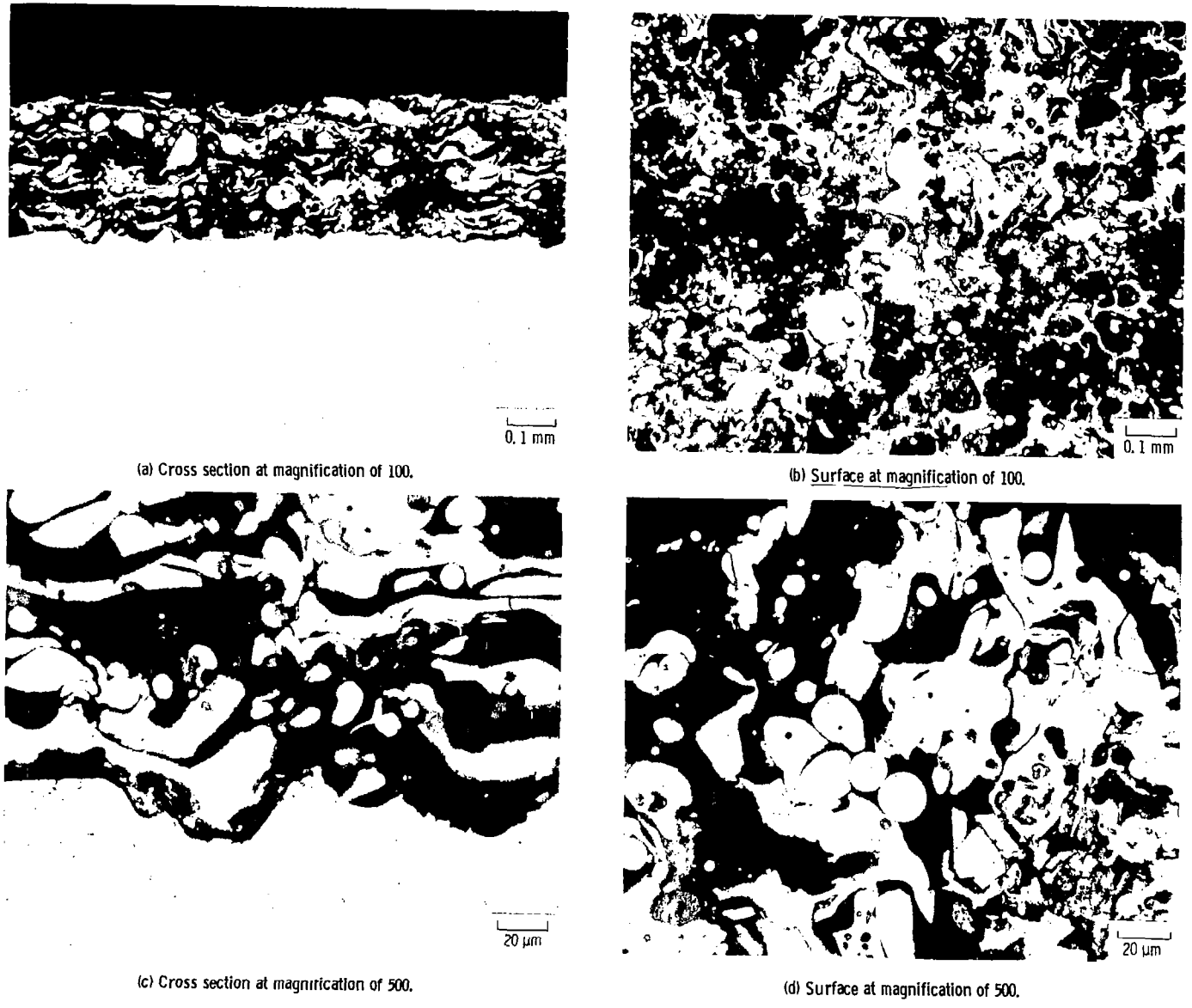
(b) Surface at magnification of 100.

(c) Cross section at magnification of 500.



(d) Surface at magnification of 500.

Figure 4. - Photomicrographs of NASA LUBE PS106 coating 2 (with 2.5 wt % $AlPO_4$).



(a) Cross section at magnification of 100.

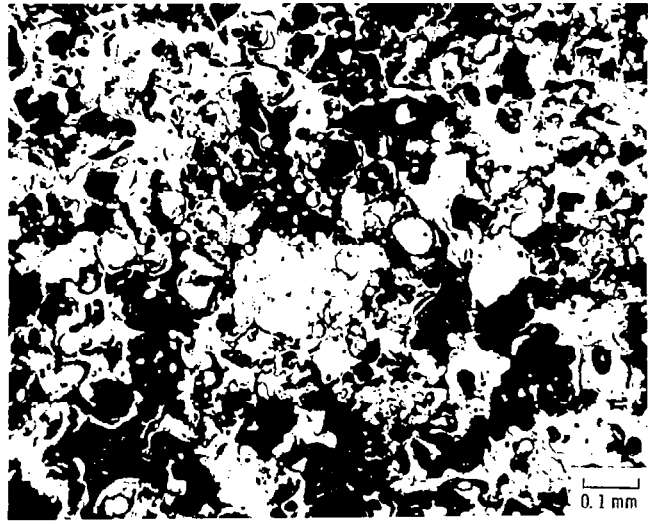
(b) Surface at magnification of 100.

(c) Cross section at magnification of 500.

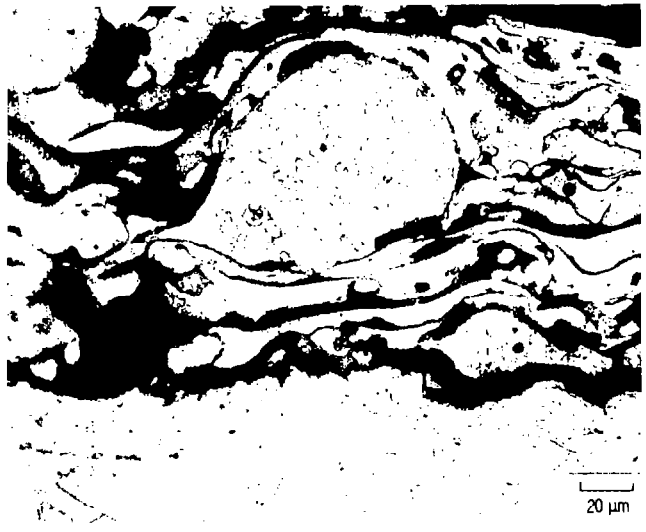
(d) Surface at magnification of 500.

Figure 5. - Photomicrographs of NASA LUBE PS106 coating 3 (with 6.25 wt % $AlPO_4$).

(a) Cross section at magnification of 100.



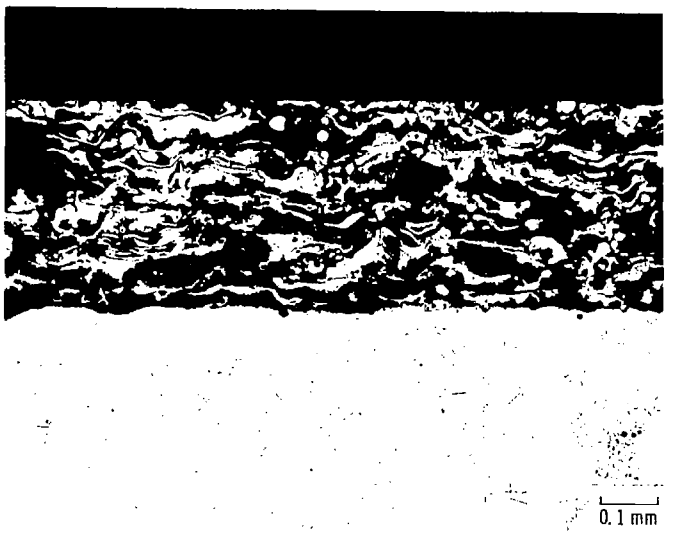
(b) Surface at magnification of 100.



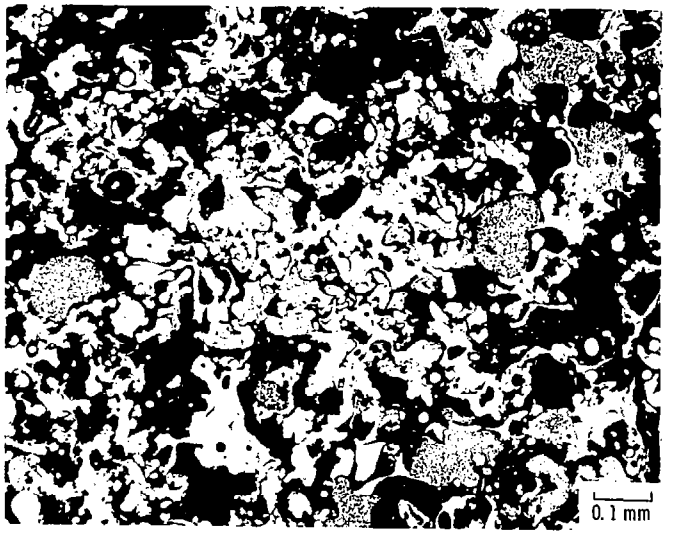
(c) Cross section at magnification of 500.

(d) Surface at magnification of 500.

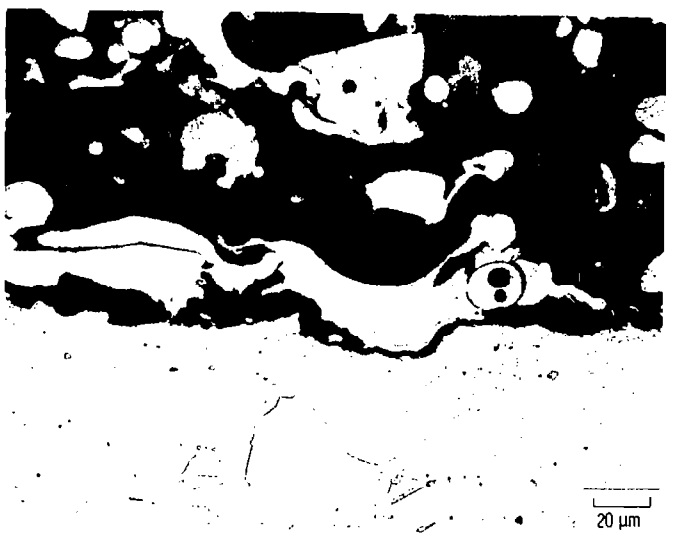
Figure 6. - Photomicrographs after etching of NASA LUBE PS106 coating 1 (with 1.25 wt % $AlPO_4$).



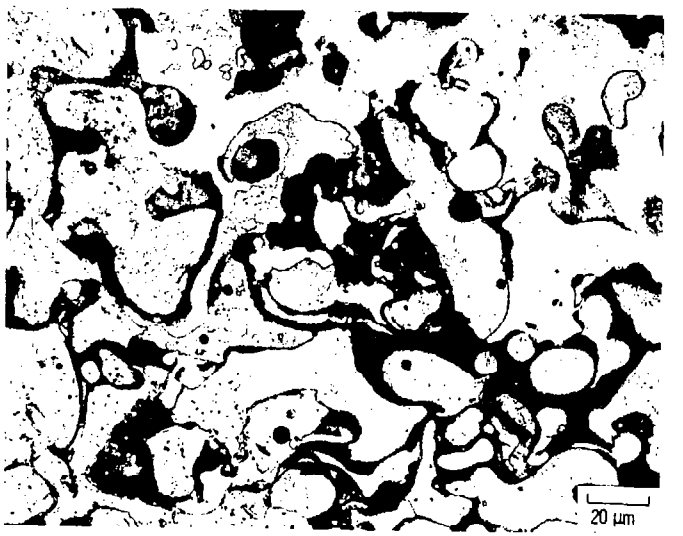
(a) Cross section at magnification of 100.



(b) Surface at magnification of 100.

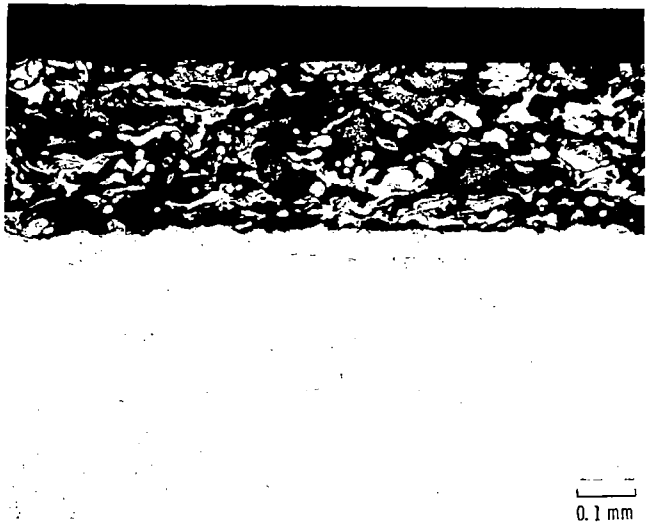


(c) Cross section at magnification of 500.

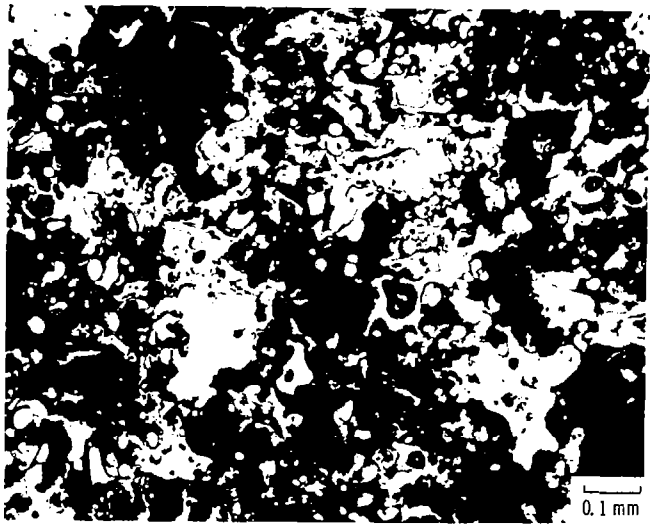


(d) Surface at magnification of 500.

Figure 7. - Photomicrographs after etching of NASA LUBE PS106 coating 2 (with 2.5 wt % $AlPO_4$).

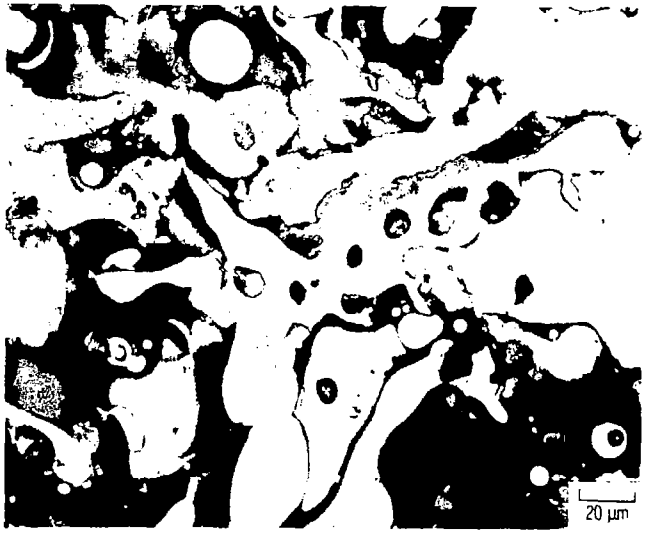


(a) Cross section at magnification of 100.



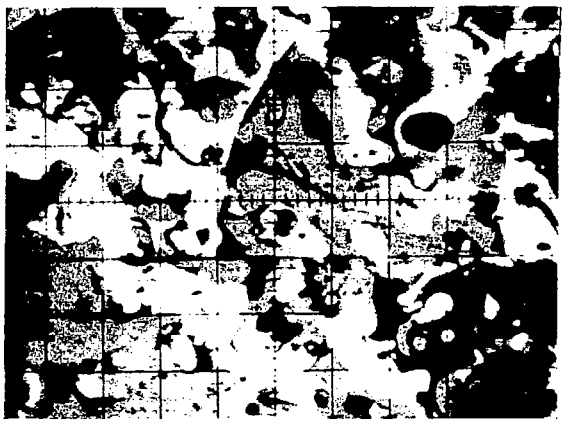
(b) Surface at magnification of 100.

(c) Cross section at magnification of 500.

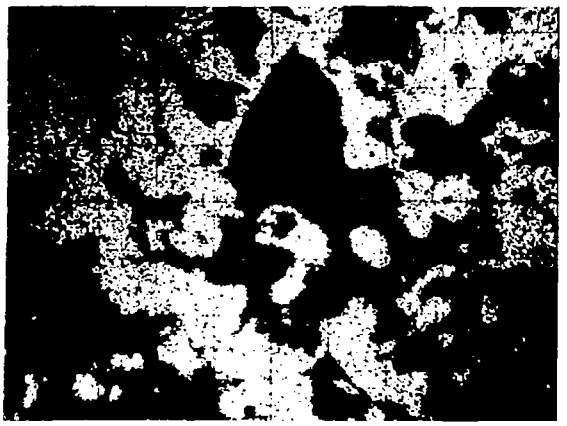


(d) Surface at magnification of 500.

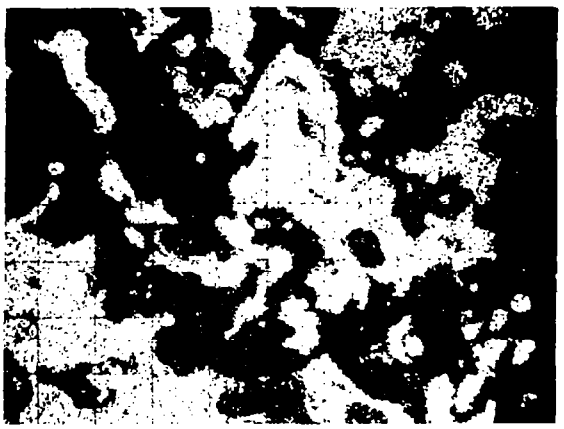
Figure 8. - Photomicrographs after etching of NASA LUBE PS106 coating 3 (with 6.25 wt % $AlPO_4$).



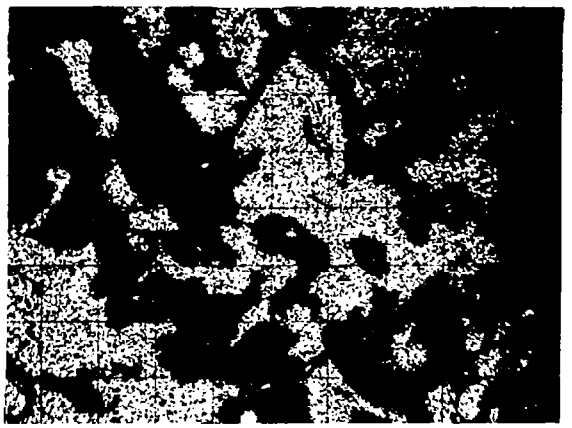
(a) Backscatter electron photograph.



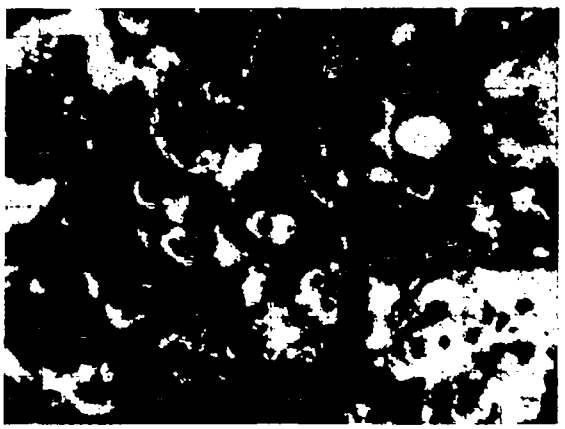
(b) Silver X-ray photograph.



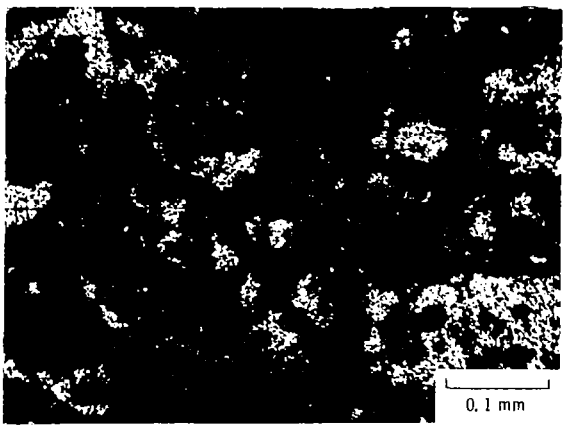
(c) Nickel X-ray photograph.



(d) Chromium X-ray photograph.

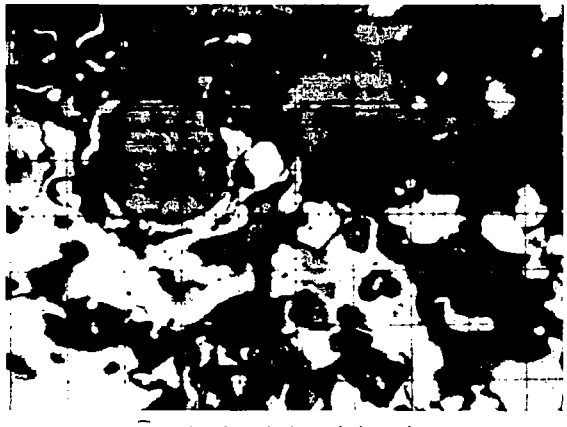


(e) Calcium X-ray photograph.

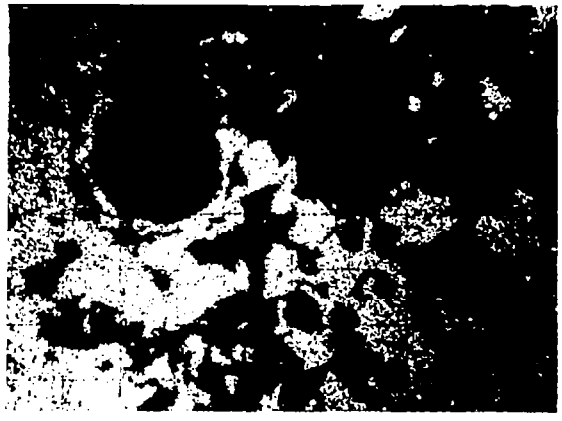


(f) Fluorine X-ray photograph.

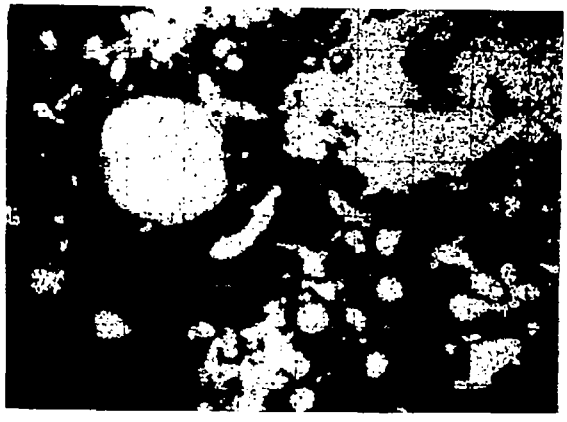
Figure 9. - EMXA photographs showing elemental distribution in NASA LUBE PS106 coating 1 (with 1.25 wt % $AlPO_4$).



(a) Backscatter electron photograph.

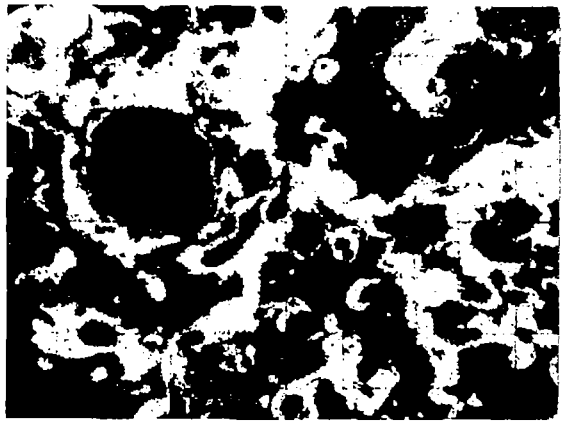


(b) Silver X-ray photograph.

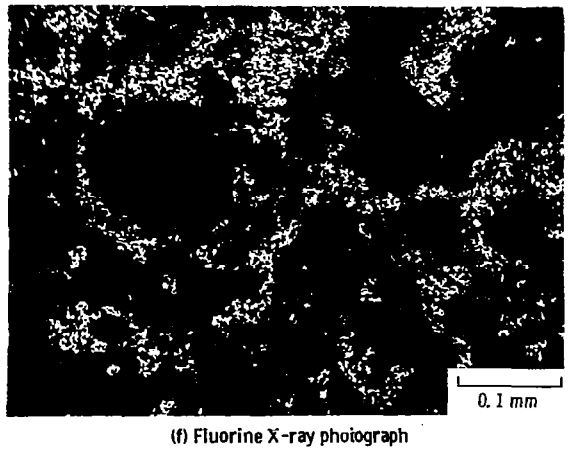


(c) Nickel X-ray photograph.

(d) Chromium X-ray photograph.

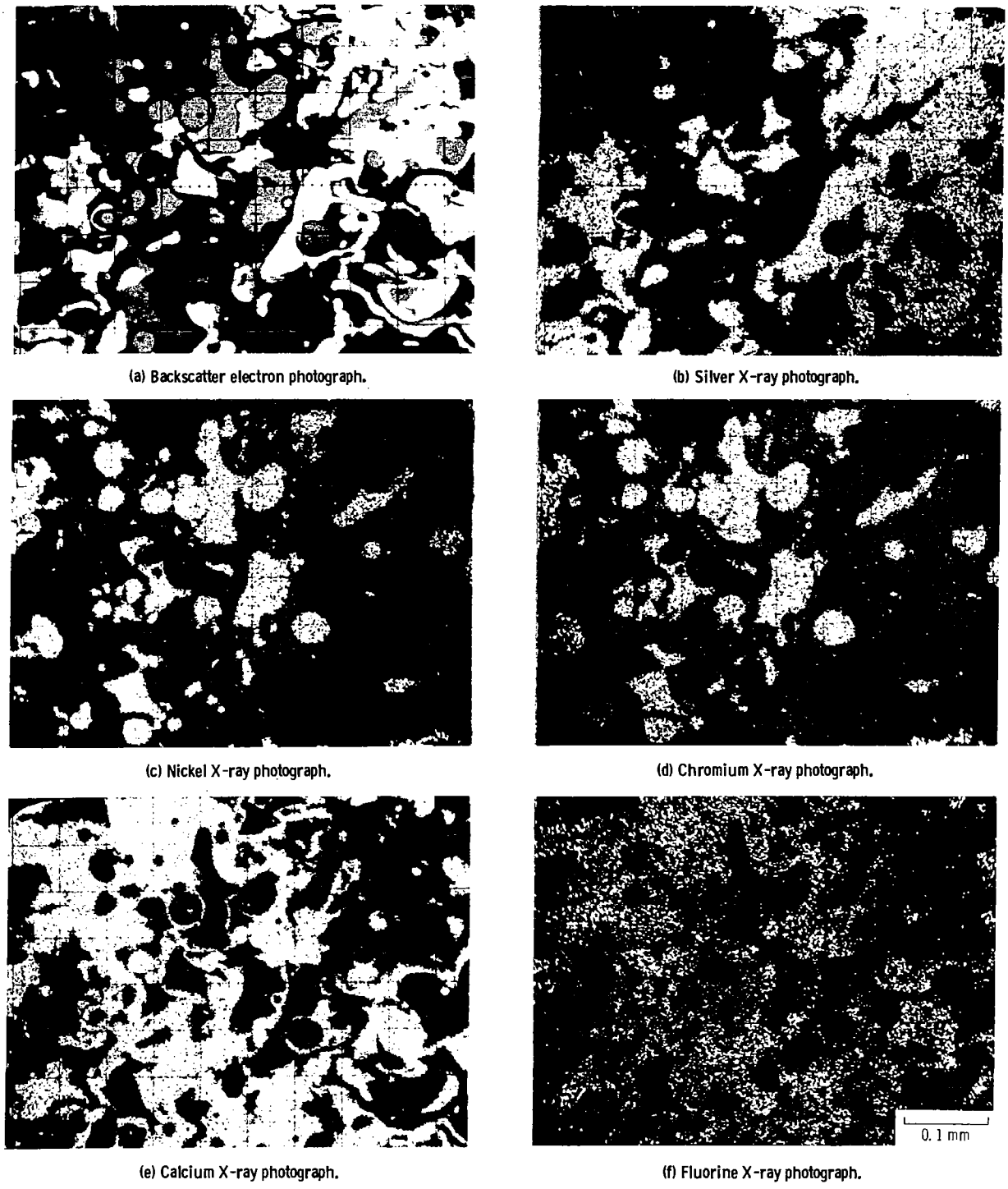


(e) Calcium X-ray photograph.



(f) Fluorine X-ray photograph

Figure 10. - EMXA photographs showing elemental distribution in NASA LUBE PS106 coating 2 (with 2.5 wt % $AlPO_4$).



(a) Backscatter electron photograph.

(b) Silver X-ray photograph.

(c) Nickel X-ray photograph.

(d) Chromium X-ray photograph.

(e) Calcium X-ray photograph.

(f) Fluorine X-ray photograph.

Figure 11. - EMXA photographs showing elemental distribution in NASA LUBE PS-106 coating 3 (with 6.25 wt % $AlPO_4$).

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15. Supplementary Notes

16. Abstract

Image analysis (IA) and electron microprobe X-ray analysis (EMXA) were used to characterize a plasma-sprayed, self-lubricating coating, NASA LUBE PS106, specified by weight percent as 35NiCr-35Ag-30CaF_2. To minimize segregation of the powder mixture during the plasma-spraying procedure, monoaluminum phosphate was added to form agglomerate particles. Three concentrations of $AlPO_4$ were added to the mixtures: 1.25, 2.5, and 6.25 percent by weight. Analysis showed that 1.25 wt% $AlPO_4$ yielded a CaF_2 deficiency, 2.5 wt% kept the coating closest to specification, and 6.25 wt% yielded excess CaF_2 as well as more impurities and voids and a deficiency in silver. Photomicrographs and X-ray maps are presented. The methods of IA and EMXA complement each other, and the reasonable agreement in the results increases the confidence in determining the coating composition.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Solid lubricants; Plasma spray; Material analysis; Characterization; Image analysis; Electron microprobe; Coatings; Aluminum phosphate	Unclassified - unlimited STAR Category 26

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