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THE EFFECT OF COBALT CONTENT IN U-700 TYPE ALLOYS ON DEGRADATION
OF ALUMINIDE COATINGS

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SUMMARY

The influence of cobalt content in U-700 type alloys on the behavior of aluminide coatings was studied in burner rig cyclic oxidation tests at 1100 °C. It was determined that aluminide coatings on alloys with higher cobalt offered better oxidation protection than the same coatings on alloys containing less cobalt.

INTRODUCTION

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The influence of nickel-based substrate alloys on the behavior of nickel aluminide coatings has been studied by several investigators (refs. 1 to 6). Because an extensive review of the work in this area has been recently published by Tomaszewicz and Wallwork (ref. 7) we will mention only its essence. It has been demonstrated that the mechanism of coating degradation is a function of alloy substrate composition (refs. 1 to 4), test temperature (refs. 3 and 4), and the environmental conditions of attack (refs. 3 and 4). The temperature and time dependence of interdiffusion is closely connected with the solubility of the various phases present in the alloy substrate and the self-diffusion coefficients of various atomic species (within the interdiffusion zone). Also it has been proposed (ref. 2) that substrate alloys, rich in refractory elements (Nb, Ta, W, Mo, Cr), will produce a zone, which will act as a diffusion barrier and thus improve the stability of NiAl coatings. A related work (ref. 1) was limited to determination of relative diffusional stability of NiAl on some binary, ternary, and other nickel-base alloys.

The work described herein is a part of the Conservation of Strategic Aerospace Materials (COSAM) program. Its objective has been to investigate the effect of cobalt content in Udimet 700¹ type alloys on the stability of NiAl coating in burner rig cyclic oxidation tests. Knowledge of this effect will help determine the impact of modifying alloys or selecting alternative alloys when cost and availability of strategic alloying elements are of concern. Udimet 700 is a nickel-based superalloy which contains nominally by weight percent, 17 cobalt, 15 chromium, 5 molybdenum, 4 aluminum, and 3.5 titanium. It is used in cast and wrought forms in turbine blades and turbine disks, and as isostatically hot pressed powder metallurgy products.

¹Trade name of Special Metals Corp.

Five alloys containing different amounts of cobalt were obtained in the wrought form and two alloys were available as powder metallurgical product. The aluminate coating was applied by the RT-21 process.² Cyclic oxidation tests were performed in a Mach 0.3 burner rig for 150 and 310 1-hr cycles with a sample temperature of 1100 °C. Weight change of the specimens was determined by removing them periodically from the burner rig and weighing. After testing, the specimens were subjected to metallographic, x-ray diffraction (XRD), and x-ray fluorescence (XRF) analyses.

MATERIALS AND EXPERIMENTAL PROCEDURES

Preparation of the Specimens

Several alloys, based on U-700 alloy, containing variable amounts of cobalt were used in this investigation. Five alloys containing 0.1, 4.3, 8.6, 12.8, and 17 wt % cobalt were obtained in the form of wrought coupons and two alloys containing 4.3 and 12.6 wt % cobalt were obtained as HIP-ed (Hot Isostatically Pressed) billets. The chemical analysis of the alloys is given in table I. The coupons and billets were machined into cylindrical specimens 10.16 cm (4 in.) long and 0.9525 cm (3/8 in.) in diameter. The aluminate coating was applied to all specimens by the RT-21 procedure.

Burner Rig Testing

The Mach 0.3 burner rig used in these tests has been described in reference 8. Eight specimens were placed in a carousel holder which was rotated at 400 rpm in front of the burner nozzle. The centers of the specimens were evenly spaced on a 4.2 cm (approximately 1-3/8 in.) diameter circle. In this cyclic oxidation test, the specimens were heated for 1 hr and then cooled with a stream of compressed air for 3 min. The burner rig was fired at a fuel-to-air ratio of about 0.05 using Jet A fuel and combustion air preheated to 260 °C (500 °F). The test temperature was 1100±20 °C (2012 °F) as determined with a calibrated (for emissivity and window absorption) disappearing filament optical pyrometer when focusing on the center of the hot zone of the specimens, facing the nozzle of the burner. The back side (not facing the nozzle) of the specimens was hotter than the front face by 20 °C. Only the central part of the specimens was exposed to the flame. About 4 min in the flame were required for the center of the specimens to reach the test temperature. After 1 hr at temperature, the specimens were cooled down to 80 °C in 3 min by a stream of compressed air. Duration of tests was 150 and 310 1-hr cycles. The weight of the specimens was monitored periodically by removing them from the holder and weighing. Thermogravimetric data are presented as total weight change (ΔW) versus the number of cycles. Weight change per unit area ($\Delta W/A$) was not used because the longitudinal distribution of temperature on the specimens was not uniform. The most heavily oxidized region was the central part of the specimens with area of about 4 cm². After testing, all the specimens were plasma sprayed with copper, for edge preservation, and cross-sectioned through the center of the hot zone for metallographic examination to ascertain the extent of oxidation damage and accompanying microstructural changes.

²Trade name of Chromalloy American Corporation.

Additional experiments were performed on that part of the specimens which was not affected by oxidation. About 1.5 cm long pieces were cut from the lower end of each specimen and subjected to cyclic oxidation at 1100 °C in a muffle furnace. Specimens were removed from the furnace after 20, 40, 100, and 150 1-hr cycles and XRD patterns were taken from the cylindrical surface in order to identify the phases present in the oxide scale.

RESULTS AND DISCUSSION

Before discussing the results obtained in this investigation it is necessary to emphasize again that a limited amount of each alloy was available for the preparation of the test specimens. Only one specimen per alloy was used in each test. Metallographic examination of the specimens in the as-coated condition (fig. 1) revealed that the coating consisted of two layers. The outer layer is a large grained, single phase β -NiAl with Cr, Mo, Co in solid solution. XRF determination of the elements in the selected alloys (substrates) and the corresponding aluminide coatings indicated (table II) that the outer coatings contained significantly less Cr, Mo, Co, and Ti than the substrate. The content of these elements in NiAl, except for cobalt, was found to be independent of the cobalt content in the alloys. The inner layer next to the substrate, contained columnar NiAl, carbides, and sigma phases (ref. 3). The thickness of the coating was approximately the same for all the alloys, namely 0.003 cm. XRD patterns taken from the coatings confirmed the presence of NiAl ($a_0 = 2.876 \text{ \AA}$). Figures 2 and 3 represent thermogravimetric data for wrought alloys and figures 4 and 5 for HIP-PM type alloys.

All the alloys showed an initial weight gain due to oxidation of NiAl. After about 20 or more cycles the specimens began to lose weight. Also one can see that the weight loss was greater for the specimens with lower cobalt content. The curves representing data obtained during 150 cycles runs (figs. 2 and 4) are in reasonable agreement with the initial portions of the curves representing data obtained during 310 cycles runs and give some idea of the reproducibility of results (figs. 3 and 5). The behavior of the specimen containing 17 wt % Co departed from the general trend. After about 150 cycles it started to lose weight rapidly and after 310 cycles its weight loss was more than twice that of the specimen containing 0.1 wt % Co. Metallographic examination of the polished and etched cross sections of the specimens (figs. 6 to 8) revealed that in all cases the NiAl has disappeared through oxidation and interdiffusion with the substrate (ref. 6). The γ' phase (Ni_3Al) precipitate in the substrate became coarser. It is also apparent that the alloys with higher cobalt content retained more γ' phase near the surface. The micrograph of the specimen, containing 17 wt % Co (fig. 8) shows that after 310 cycles the substrate near the surface is void of γ' phase, which means that the surface region is depleted of aluminum. This observation is consistent with the significant and unexpected weight loss exhibited by this alloy.

No significant difference was apparent between the oxidation behavior of the hot worked alloys and alloys prepared by HIP-PM technique. The thermogravimetric curves for both types of alloys show the same general parabolic shape (figs. 2 and 4 or 3 and 5) and the microstructures resulting from the cyclic oxidation tests (figs. 6 and 7) are very similar.

XRD analysis of the surface of specimens subjected to cyclic oxidation at 1100 °C in static air (table III) revealed the absence of NiAl coating after 20 1-hr cycles. In general, the phases detected were alumina, NiSS, spinel, and rutile. The alloys with low cobalt content developed oxide scale composed of alumina or alumina plus spinel, whereas alloys with higher cobalt content produced oxide scale composed of alumina, rutile, and spinel. The amount of spinel and rutile in the oxide scale increased with the number of oxidation cycles.

The data obtained here for coated U-700 type alloys can be compared to the results reported by Barrett (ref. 9) for the same alloys but in the uncoated condition. Barrett's static air, cyclic oxidation experiments were conducted at temperatures ranging from 1000 to 1150 °C for times to 500 1-hr cycles. He found that alloys with higher cobalt content underwent greater weight loss than those with lower cobalt content. Thus his findings are opposite to those of the present study where the aluminide coated alloys with higher cobalt content yielded reduced weight loss. Barrett found also that the oxide scale on the low cobalt content samples was composed of Ni, NiTiO₃, and Cr₂O₃ while that on the higher cobalt content alloys was NiO, Cr₂O₃, and NiCr₂O₄. The offered explanation was that for Cr/Al ratio > 3.5 the tendency to form Cr₂O₃ is accentuated by higher cobalt levels which leads an increased oxidation rate.

SUMMARY OF RESULTS

1. The aluminide coated cobalt rich alloys exhibited better oxidation resistance than alloys with low cobalt content, the weight loss being the criterion for such evaluation.
2. The aluminide coatings contained significantly less Ti, Cr, Mo, and Co than their substrates. The content of these elements in the NiAl, except for cobalt, was found to be independent of the cobalt content in the alloys.
3. The higher content of cobalt in the aluminide coated U-700 type alloys promoted the formation of spinel and rutile structures in the oxide scales, which could be the reason for improved oxidation resistance.
4. There was no difference between the cyclic oxidation behavior of aluminide coatings on wrought alloys and coatings on alloys of the same composition but prepared by HIP-PM technique.

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TABLE I. - CHEMICAL ANALYSES OF THE ALLOYS

Alloy No.	Co	Cr	Mo	Ti	Al	C	B	Ni
	wt Δ							
U-1 ^a	0.10	15.1	5.00	3.46	4.12	0.06	0.025	Bal.
U-2 ^a	4.30	15.1	4.90	3.55	4.14	.07	.024	↓
U-3 ^a	8.6	15.0	5.05	3.51	4.05	.06	.025	
U-4 ^a	12.8	14.7	5.0	3.61	4.10	.06	.023	
U-5 ^a	17.0	14.9	5.03	3.60	4.08	.06	.028	
U-2-1 ^b	4.3	14.9	4.85	3.53	4.05	.07	.021	
U-4-1 ^b	12.6	14.8	5.10	3.56	4.03	.06	.024	

^aWrought.
^bHIP Alloys.

TABLE II. - XRF DETERMINATION OF ELEMENTS IN ALUMINIDE COATINGS AND THEIR SUBSTRATES

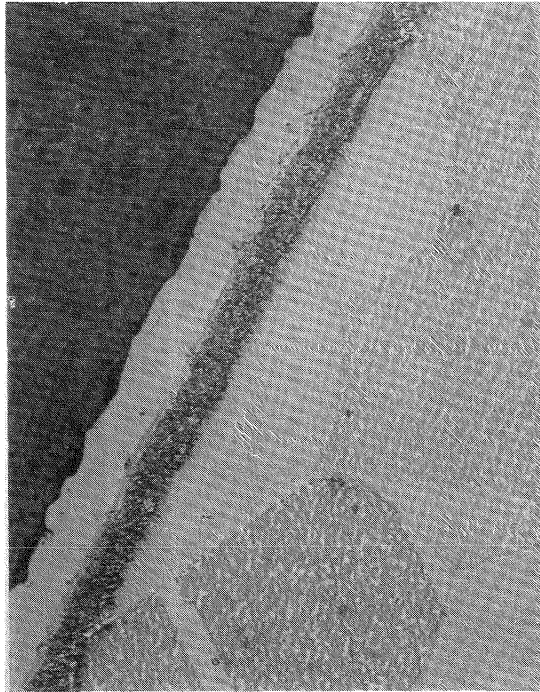
Alloy (cobalt content)	Element	Co	Cr	Mo	Ti	Al	Ni
U-1 (Co = 0.1)	Coating	^a 0.05	2.45	2.86	0.20	30.23	61.23
	Substrate	0.16	15.20	4.41	3.39	6.42	68.90
U-3 (Co = 8.6)	Coating	4.01	2.87	2.80	.19	29.27	58.25
	Substrate	7.80	14.97	4.45	3.25	3.40	64.36
U-5 (Co = 17.0)	Coating	8.64	2.90	2.87	.22	28.97	53.96
	Substrate	14.44	14.74	4.20	3.12	3.43	57.99
U-2-1 (Co = 4.3)	Coating	2.06	2.60	2.70	.19	31.24	58.21
	Substrate	3.73	14.05	4.29	3.19	3.78	69.27

^aThe data are expressed in weight percent. For the aluminide coating the precision is ± 10 percent and accuracy is ± 20 percent and for the substrate they are ± 5.0 percent and ± 15 percent, respectively.

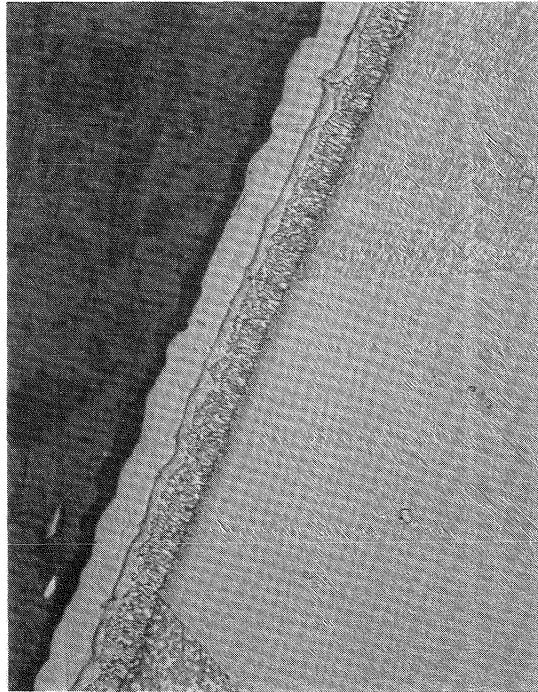
TABLE III. - PHASES DETECTED BY XRD ON THE SURFACE OF THE SPECIMENS AFTER CYCLIC OXIDATION
AT 1100 °C IN A MUFFLE FURNACE

Alloy	Cobalt content, wt Δ	Number of cycles/phases detected			
		20	40	100	150
U-1	0.1	Al ₂ O ₃ (s), ^a NiSS(s)	Al ₂ O ₃ (s), NiSS(s)	Al ₂ O ₃ (s), NiSS(s)	Al ₂ O ₃ (s), NiSS(m) Spinel(s)
U-2	4.3	Al ₂ O ₃ (s), NiSS(s)	Al ₂ O ₃ (s), NiSS(s)	Al ₂ O ₃ (s), NiSS(s) Spinel(w)	Al ₂ O ₃ (s), NiSS(s) Spinel(s)
U-3	8.6	Al ₂ O ₃ (s), NiSS(s)	Al ₂ O ₃ (s), NiSS(s)	Al ₂ O ₃ (s), NiSS(s) Spinel(m)	Al ₂ O ₃ (s), NiSS(m) Spinel(s), Rutile(m)
U-4	12.8	Al ₂ O ₃ (s), NiSS(s) Rutile(w)	Al ₂ O ₃ (s), NiSS(s) Rutile(w)	Al ₂ O ₃ (s), NiSS(m) Rutile(m), Spinel(m)	Al ₂ O ₃ (s), NiSS(m) Spinel(s), Rutile(m)
U-5	17.0	Al ₂ O ₃ (s), NiSS(s) Rutile(w)	Al ₂ O ₃ (s), NiSS(s) Rutile(w)	Al ₂ O ₃ (s), NiSS(s) Rutile(m), Spinel _μ (w)	Al ₂ O ₃ (s), NiSS(m) Spinel(s), Rutile(m)
U-2-1	4.3	Al ₂ O ₃ (s), NiSS(s)	Al ₂ O ₃ (s), NiSS(s)	Al ₂ O ₃ (m), NiSS(m)	Al ₂ O ₃ (s), NiSS(m) Spinel(w), Rutile(m)

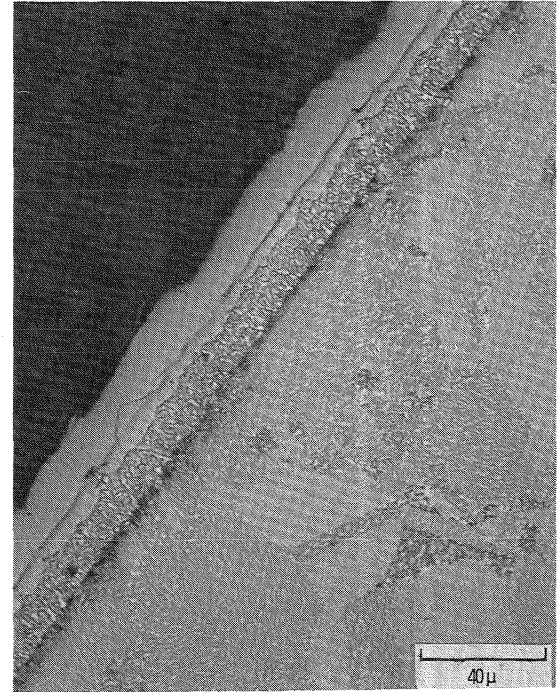
^aRelative intensities of oxides are with respect to the other oxides only.
s - strong; m - medium; w - weak.



(a) Alloy U-1, Co content 0.1%, wrought.



(b) Alloy U-5, Co content 17%, wrought.



(c) Alloy U-4-1, Co content 12.6%, HIP-PM.

Figure 1. - Microstructure of selected alloys after coating with aluminide by RT-21 process.

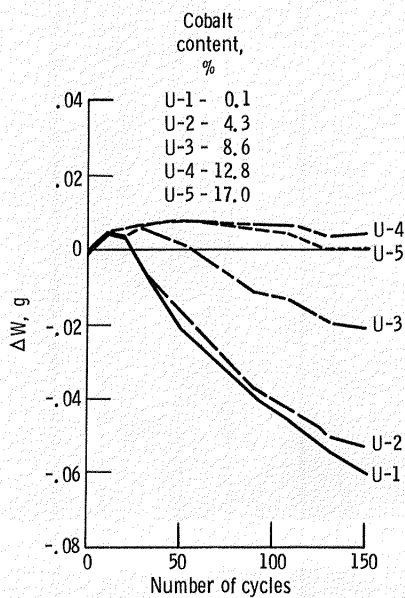


Figure 2. - Oxidation behavior of aluminized U-700 type alloys (wrought) in Mach 0.3 burner rig at 1100 °C.

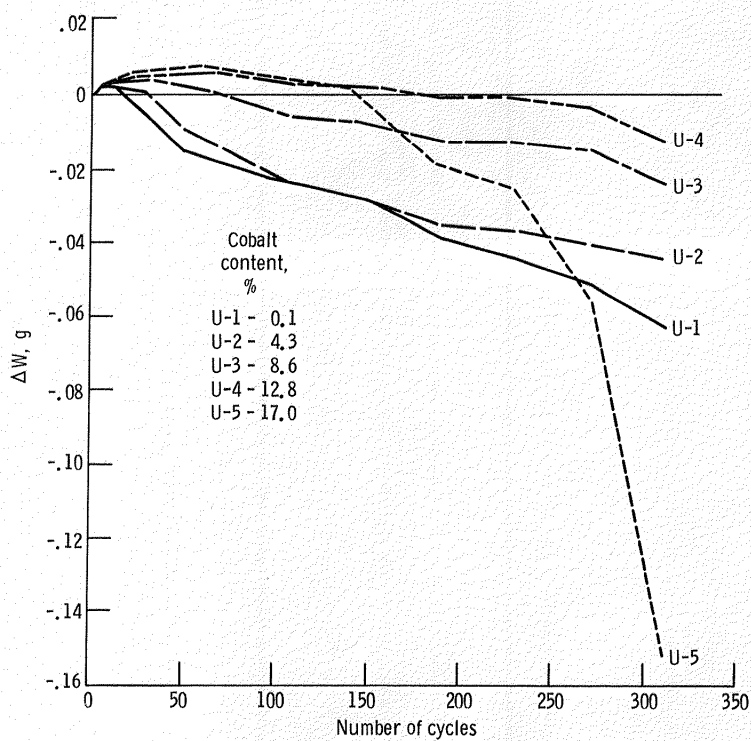


Figure 3. - Oxidation behavior of aluminized U-700 type alloys (wrought) in Mach 0.3 burner rig at 1100 °C.

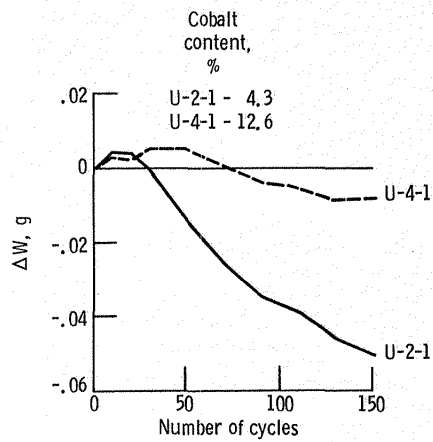


Figure 4. - Oxidation behavior of aluminized U-700 type alloys (HIP-PM) in Mach 0.3 burner rig at 1100 °C.

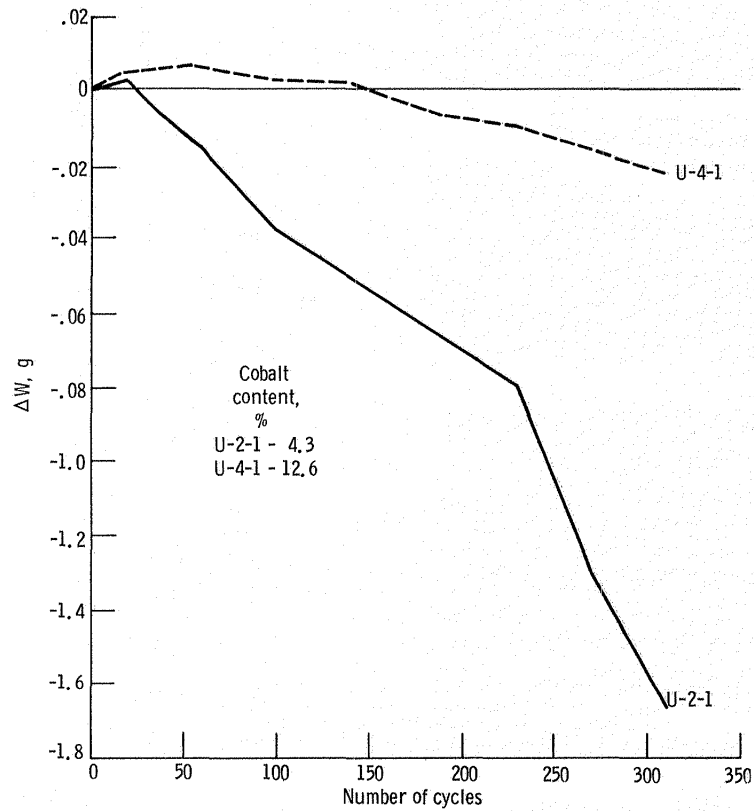
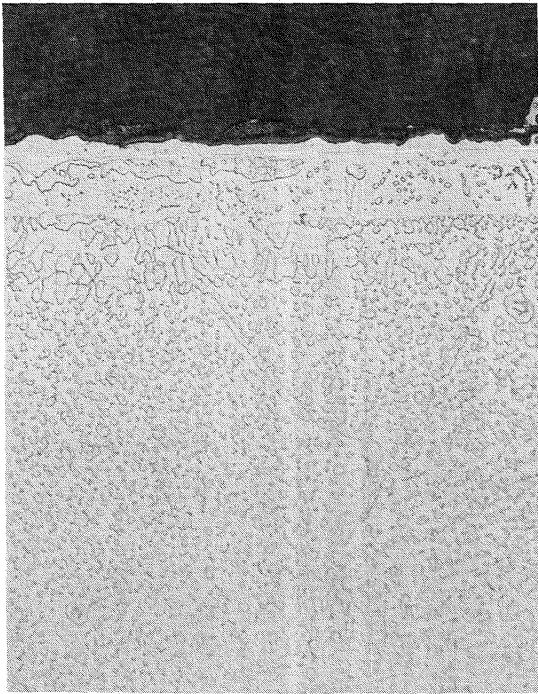
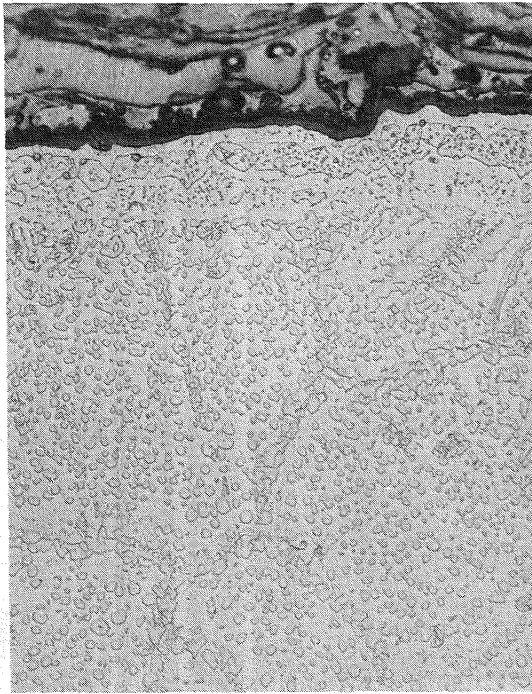


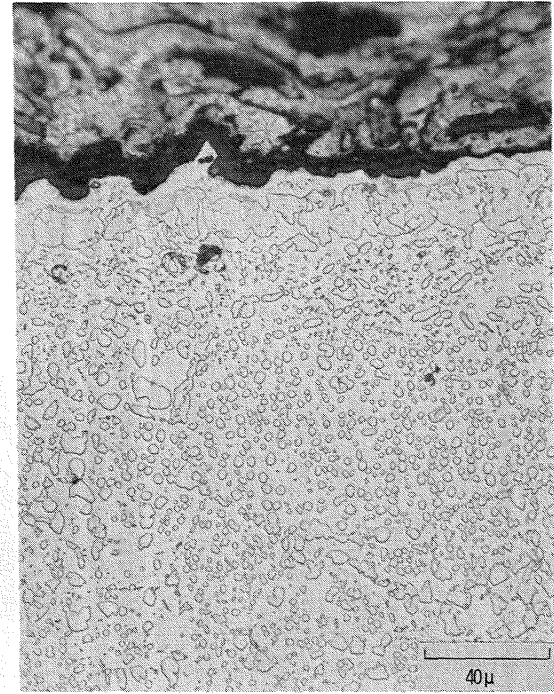
Figure 5. - Oxidation behavior of aluminized U-70 type alloys (HIP-PM) in Mach 0.3 burner rig at 1100 °C.



(a) Alloy U-1, Co content 0.1%, wrought.

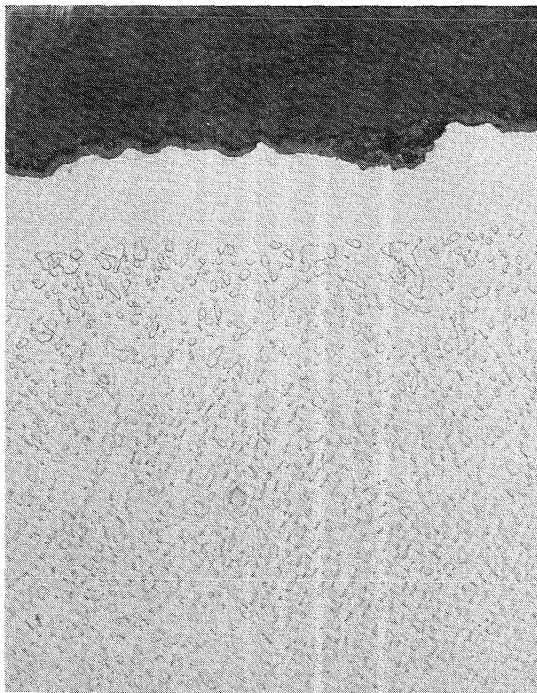


(b) Alloy U-3, Co content 8.6%, wrought.

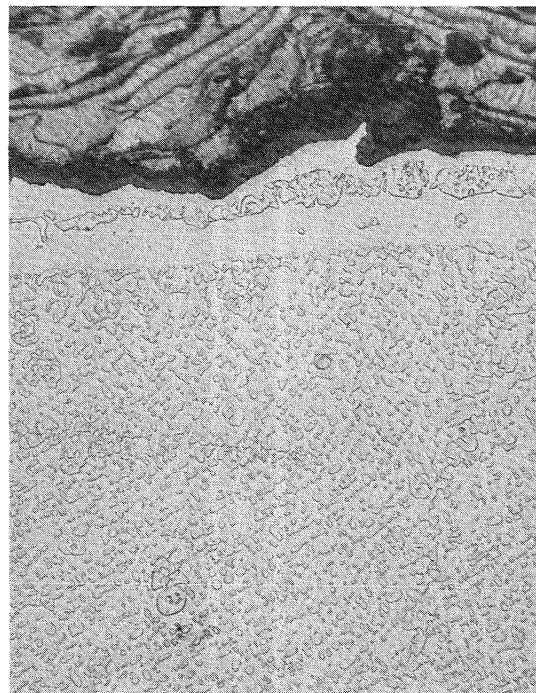


(c) Alloy U-4-1, Co content 12.6%, HIP-PM.

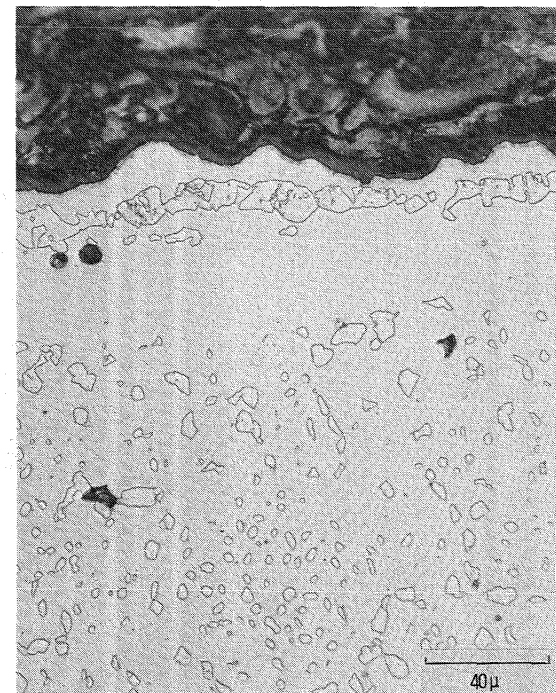
Figure 6. -Typical microstructures of aluminide coated U-700 type alloys after 150 oxidation cycles at 1100 °C in Mach 0.3 burner rig.



(a) Alloy U-1, Co content 0.1%, wrought.



(b) Alloy U-3, Co content 8.6%, wrought.



(c) Alloy U-4-1, Co content 12.6%, HIP-PM.

Figure 7. - Typical microstructures of aluminide coated U-700 type alloys after 310 oxidation cycles at 1100 °C in Mach 0.3 burner rig.

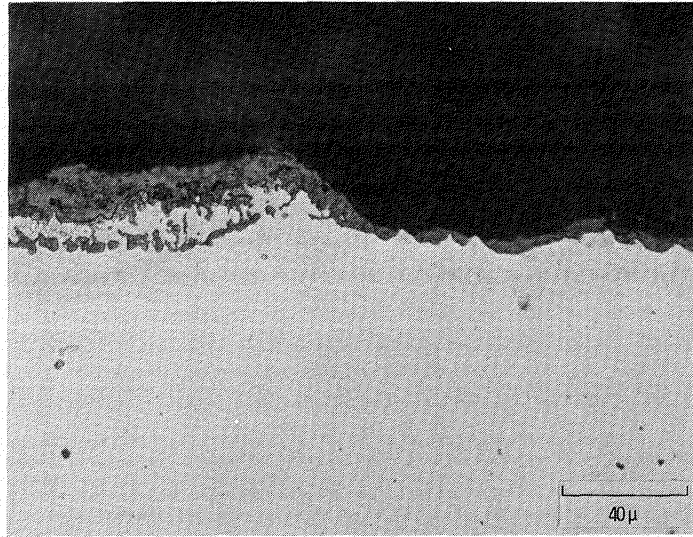


Figure 8. - Microstructure of U-5 alloy (Cobalt content 17 ^W%, wrought) after 310 oxidation cycles at 1100 °C in Mach 0,3 burner rig.

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