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Periodic Technical Report

Extend CoCrAlY and Pt Sputter-Deposition Technology to Provide Coatings on FT4 Turbine Vanes for At-Sea Evaluation

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December 16, 1976

Report Period: January 16, 1976 to Decmber 31, 1976

Prepared under
Contract Number N00024-75-C-4332,
for The Naval Sea Systems Command

Battelle
Pacific Northwest Laboratories

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EXTEND CoCrAlY AND Pt SPUTTER-DEPOSITION TECHNOLOGY TO PROVIDE COATINGS ON FT4 TURBINE VANES FOR AT-SEA EVALUATION

REPORT PERIOD: JANUARY 16 TO DECEMBER 31, 1976

Principal Investigator	J.W. Patten
Contract Number	N00024-75-C-4332
Contract Sponsor	Naval Sea Systems Command
Effective Date of Contract	February 27, 1975
Contract Expiration Date	July 28, 1976
Amount of Contract	$168,000

December 16, 1976

Work Performed by
PACIFIC NORTHWEST LABORATORIES
a division of
Battelle Memorial Institute
P.O. Box 999
Richland, Washington 99352

UNCLASSIFIED ABSTRACT

The application of high-rate dc triode sputtering technology to the deposition of CoCrAlY alloy and Pt coatings on first-stage FT4 and LM-2500 blades and FT4 vanes for sulfidation protection in marine gas turbines is described. The CoCrAlY coatings were approximately 0.005 inch thick and were applied directly to superalloy blades and vanes and to burner rig test pins. In addition, a ~ 0.0002 inch thick layer of Pt was sputter deposited both as an overlayer on the CoCrAlY and as an interlayer between the superalloy airfoil and/or test pin and the CoCrAlY. The Pt coatings were applied on both sputter-deposited CoCrAlY and electron beam evaporated CoCrAlY. Features of the sputter-deposition technology applied are discussed. Microstructural, chemical composition, and thickness distribution results are presented. FT4 vanes coated with CoCrAlY of two Al contents and Pt were delivered to the Navy for at-sea testing.

DISTRIBUTION

Organization	Number of Copies
Naval Ship Engineering Center	40
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TABLE OF CONTENTS

FIGURES

TABLES

INTRODUCTION

This report describes progress on the application of BNW high-rate sputtering technology to the formation of CoCrAlY and CoCrAlY plus Pt coatings on FT4 hot-stage turbine vanes.

The contract objective was to coat a total of 16 vanes for at-sea evaluation by NAVSEC. Details on coating requirements are given in the following:

- Four vanes will have a nominal 0.005 inch coating of sputter-deposited CoCrAlY -- two with 6-9 wt% Al and two with 10-12 wt% Al.
- Six vanes will have a nominal 0.005 inch coating of sputter-deposited CoCrAlY followed by a sputter-deposited Pt layer with a nominal thickness of 0.0002 inch. Three vanes will have CoCrAlY with 6-9 wt% Al and three will have CoCrAlY with 10-12 wt% Al.
- Six vanes will have a nominal 0.0002 inch layer of sputter-deposited Pt followed by a nominal 0.005 inch coating of sputter-deposited CoCrAlY -- three will have CoCrAlY with 6-9 wt% Al and three will have CoCrAlY with 10-12 wt% Al.

SUMMARY

Two techniques were developed by Battelle-Northwest aimed at increasing the life of CoCrAlY coatings on first-stage turbine components during marine use.

The first technique was the sputter deposition of adherent, defect-free, relatively uniform thickness CoCrAlY coatings. A total of sixteen FT4 hot-stage turbine vanes were coated with sputter-deposited CoCrAlY of two Al compositions, 6-9% Al and 10-12% Al, for at-sea testing by NAVSEC. An important part of this work was the development of fabrication procedures for a 9-inch diameter CoCrAlY target.

The second technique involves providing a Pt coating. This was accomplished by the sputter deposition of Pt as an over or inner layer on either PVD or sputter-deposited CoCrAlY. The Pt is intended to provide additional corrosion resistance by acting as an Al reservoir.

The results of the at-sea tests will add to the data regarding the effect of Pt additions on the CoCrAlY coating corrosion resistance and provide the first indication of the influence of high integrity CoCrAlY coating structured on corrosion resistance.

BACKGROUND

Current state-of-art for production coatings to protect gas turbine hot stages is a CoCrAlY alloy coating ~ 0.005 in. thick applied by physical vapor deposition (PVD). These coatings, as deposited, typically exhibit relatively large grain size and columnar growth defects (leaders) perpendicular to the plane of the coating. The defects are actually interfaces between adjacent growth columns, each column originating at a separate nucleation site on the substrate (vane or blade) surface and resulting in coating discontinuities through at least a large fraction of the coating thickness. Several post-coating techniques are used in production to close up these leaders, but the techniques are only partially sucessful and add to the cost of the complete production coating process. Nevertheless, such PVD deposited and modified CoCrAlY coatings greatly enhance the hot corrosion resistance of gas turbine hot stages and are invaluable to the performance of gas turbines for certain types of service.

Progress in the investigation of two possibilities for improving the performance of these coatings is presented in this report. First, a technique was developed independently by Battelle-Northwest to sputter deposit CoCrAlY coatings without producing any columnar growth defects or coating discontinuities. Second, Pt layers were sputter deposited either as underlayers or overlayers to PVD deposited and sputter deposited CoCrAlY coatings and heat treatments were conducted to produce extensive diffusion between Pt and CoCrAlY layers. The desired effect of the Pt layer was to retain Al near the free surface for corrosion resistance by forming very stable Pt-Al compounds which would then give up Al to form Al oxides. The results of initial burner-rig results indicate that the Pt addition did in fact provide a significant increase in life over current production PVD CoCrAlY.[1]

The sputter deposition techniques developed by Battelle-Northwest were expected to offer several advantages for the application of the CoCrAlY and Pt coatings to complex turbine component geometries. For CoCrAlY coatings, extremely good coating-to-substrate adherence is observed even for low substrate temperatures during deposition. This adherence is expected because of the atomically clean substrate surface produced immediately before deposition by ion bombardment etching and because of the high kinetic energies of the sputtered Co, Cr, Al, and Y adatoms. Because of this good adherence it is possible to simultaneously provide high-rate deposited coatings with very fine uniform grain size of a few microns. It is speculated that this grain structure in CoCrAlY coatings may be an aid to coating ductility and to hot corrosion resistance. Further, Battelle-Northwest sputtering techniques offer the freedom to investigate elemental additions to the CoCrAlY coatings without regard to vapor pressure, melting point or other physical properties that place constraints on PVD techniques. The process also is conducted in a very clean vacuum environment so that concentrations of impurities are quite low. A final advantage is the capability to routinely monitor and adjust 30 separate sputter deposition parameters by computer control. This assures reproducibility of deposit properties and minimizes manpower requirements.

With respect to Pt, sputter deposition procedures in general are simplified because a single element is much easier to work with than a quaternary system such as CoCrAlY. Further, the Pt is deposited either as an underlayer or overlayer to CoCrAlY coatings and is diffused into the CoCrAlY coatings so that microstructure of the Pt coating is relatively unimportant. Perhaps one of the most significant advantages offered by Battelle-Northwest sputter deposition technology to Pt coating is high material efficiency. With the system used to perform the work described in this paper more than 50% of the Pt removed from the sputtering targets was deposited in desired locations on vanes, blades, or burner rig test pins. This could be increased by decreasing target-target gap and/or increasing target length, both of which decrease end losses, or by increasing substrate area, i.e. more or larger substrates coated in each run.

PLATINUM COATING DEVELOPMENT

PROCEDURE

The sputtering chamber used is shown schematically in Figure 1. The two concentric Pt targets used were of the same composition as those used in earlier work.[2] A total of three vanes per run were coated in the annulus between the targets.

The sputter deposition conditions developed in previous Pt coating research[3] were used in all Pt coating work described in this report.

Pt SPUTTER COATING OF FT4 VANES IN COMBINATION WITH CoCrAlY SPUTTERED COATINGS FOR AT-SEA TESTING AND EVALUATION

Table I contains the deposition parameters for sputter coating of ~ 0.0002 inch Pt on both bare and CoCrAlY sputter coated FT4 vanes. The deposition rate was approximately 0.0003 inch/hr. This rate was selected to allow sufficient deposition time for accurate thickness control. Run-to-run reproducibility of coating quality was provided by: monitoring the sputtering system's atmosphere with a quadrapole mass spectrometer, computer control over sputter deposition parameters, monitoring the change in weight of the vanes and the Pt targets, and sectioning and metallographically examining the Ni foil mask from each end of a vane from each deposition. A typical microstructure of the Pt coating on the Ni foil end masks is illustrated in Figure 2.

CoCrAlY COATING DEVELOPMENT

OBJECTIVE

The objective of this work was to sputter coat FT4 first-stage turbine vanes for at-sea testing to provide information demonstrating the advantages of sputter deposited coatings for hot corrosion protection. To accomplish this objective the following combinations of coatings were provided:

	Coating Combination	Vanes to be Delivered
A.	CoCrAlY alloy with approximately 6-9% Al.	2
B.	CoCrAlY alloy with approximately 10-12% Al.	2
C.	Coating A with a 0.0002 inch Pt underlayer.	3
D.	Coating A with a 0.0002 inch Pt overlayer.	3
E.	Coating B with a 0.0002 inch Pt underlayer.	3
F.	Coating B with a 0.0002 inch Pt overlayer.	3

PROCEDURE

Sputtering Chamber

The sputtering chamber used to deposit CoCrAlY is shown schematically in Figure 3. Note the flat plate CoCrAlY target and the provision for use of a second electrically independent flat plate target. This feature was introduced to allow continuous variations in the chemistry of CoCrAlY (or CoCrAlY + X) deposits to be produced using the same CoCrAlY target.

CoCrAlY Sputtering Targets

The previous Periodic Technical Report(3) discussed the difficulties experienced in obtaining suitable CoCrAlY sputtering targets. At the time of that report the only available CoCrAlY targets were 5 inches in diameter which had been purchased on an earlier NAVSEC contract. These targets were not large enough for FT4 vane coating.

During the period from January to March 1976, hot pressing techniques were developed jointly by BNW and Wayne Castledyne of UDIMET Powder Division of Special Metals Corporation, for fabrication of suitable 9-inch diameter CoCrAlY targets. It is believed that improved bonding of the CoCrAlY to the mild steel support and a higher pressing temperature contributed to the successful performance of these targets during sputtering. Analytical and other information on the CoCrAlY targets used in this work are presented in Table II.

Preliminary Experiments

The data for initial experiments to resolve system design problems (electrical shorting between removable shields and targets, peeling of stray deposit material from shields and fixturing), and establish deposition parameters to produce low Al content deposits from a CoCrAlY target are shown in Table III. The data for experiments to refine the design of a second target (Al) and establish deposition parameters for higher Al content deposits are presented in

Table IV. Analysis of aluminum content on portions of the vane airfoil and adjacent Ni mask, Figure 3, resulted in the data presented in Table V. The data in Table V should be considered preliminary as our x-ray fluorescence techniques were not fully developed and not enough examples of coating composition distribution on airfoil shapes were examined to allow reliable prediction of variations in Al content. Also, no determination of Y concentration has been made as that capability is not available at BNW although it is currently being acquired.

With respect to Cr content in the CoCrAlY deposit, the data obtained by x-ray fluorescence indicated a nominal value of 20%. This is essentially identical to the 20.0-20.3% Cr values reported for the targets in Table II.

The data in Table V indicate that the parameters used in Experiments 146 and 156 with a single CoCrAlY target result in an Al content on the vane ranging from 6-9%. The parameters of Experiment 157 for the addition of Al from an Al target resulted in values of 8-11% Al on the airfoil. Since it was not possible to obtain Al content data on the vanes to be delivered, the data obtained on the Ni masks provide additional assurance, over and above the run-to-run control exercised on deposition parameters, that the expected Al content (based on deposition parameters) was obtained on the vanes.

In addition to the chemical analysis data obtained on the Ni masks, sections were taken for metallographic evaluation, again primarily for assurance of run-to-run reproducibility. Typical microstructures are illustrated in Figures 4, 5, 6 and 7. They demonstrate the high integrity, leader-free, fine-grained microstructure obtained. Note the lighter shaded band in the coating in Figure 4, center arrow. This was caused by two interruptions in the experiments, one at the beginning and one at the end of the band. The microstructure indicates that a very good bond was achieved in spite of the experiment interruptions. This characteristic might be quite useful in a production application for restarting after equipment shutdown or for recoating blades or vanes to extend service life after consumption of an initial coating.

<u>COATING OF 16 VANES FOR AT-SEA TESTING</u>

Based on the information developed from the work described in the preceding section, the 16 vanes required for at-sea testing were coated using the deposition parameters presented in Table VI. Reproducibility of coating quality and properties was assured by constant monitoring of deposition conditions, periodic data logging by computer, microstructural and compositional evaluation of Ni masks and visual examination and evaluation of coated vanes. In general, it was felt that the Al composition and coating thickness (0.003 - 0.005 inch) for the vanes fell within [illegible] desired ranges. Coating integrity or quality appeared [illegible] be excellent. A typical microstructure after heat treat for 4 hours at 1080°C in vacuum for the 10-12% Al CoCrAlY with Pt underlayer is illustrated in Figure 8. A summary of the 16 delivered vanes and their coatings is presented in Table VII.

ACKNOWLEDGMENT

The authors gratefully acknowledge the technical support of L.K. Fetrow, R.F. Stratton, and E.L. McDonald for the conduct of the sputter deposition experiments, and R.H. Beauchamp for metallography.

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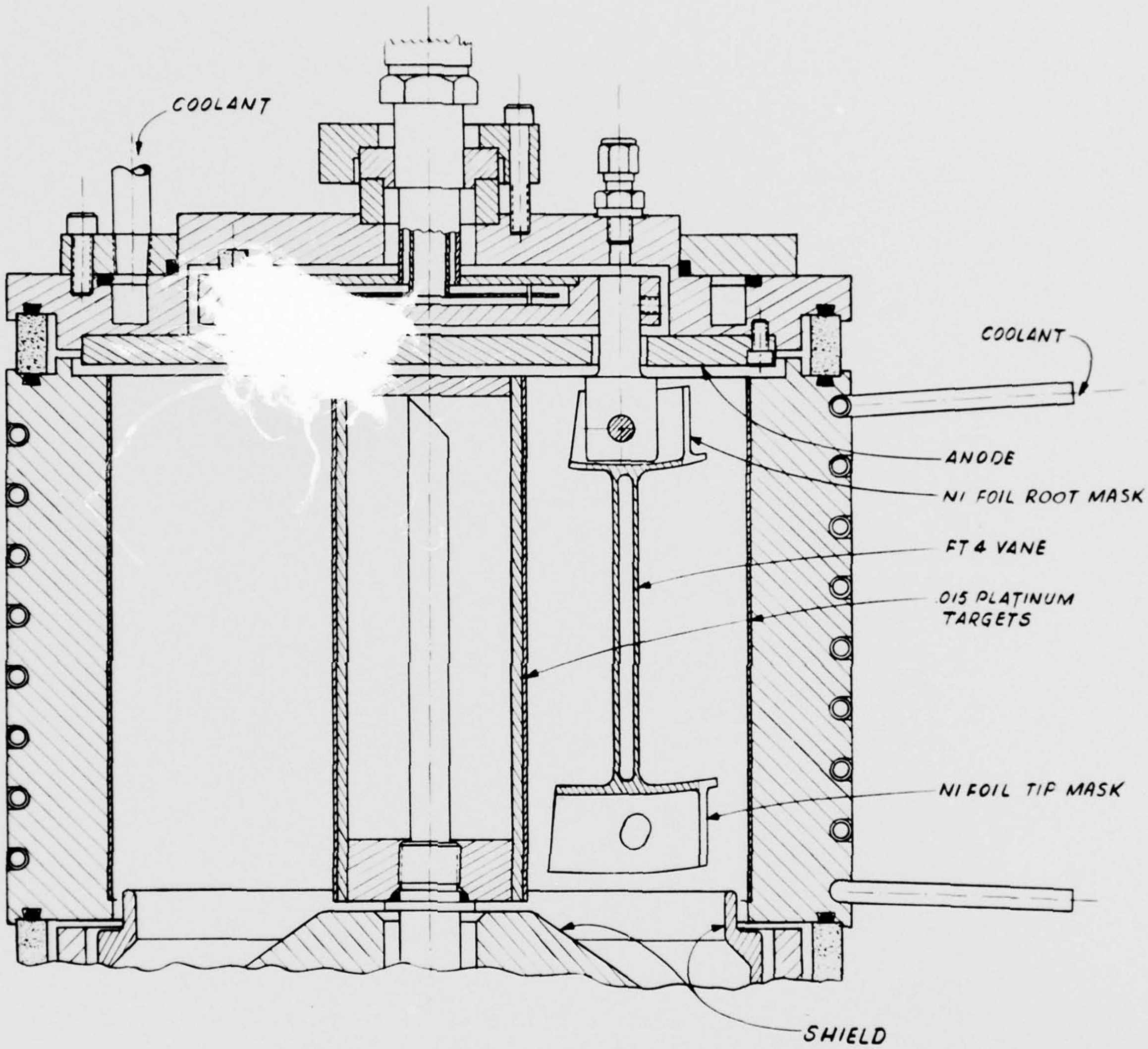


FIGURE 1. Sputtering chamber for Pt deposition.

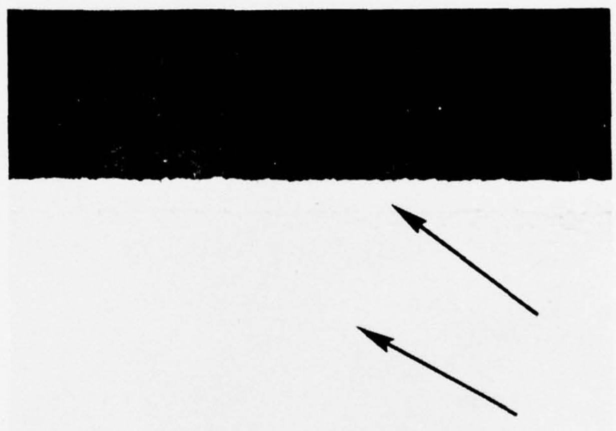


FIGURE 2. Pt coating (top arrow) on Ni tip mask (bottom arrow). Note extensive inter-diffusion resulting from elevated temperature of the Ni mask during deposition. As-polished. 500X

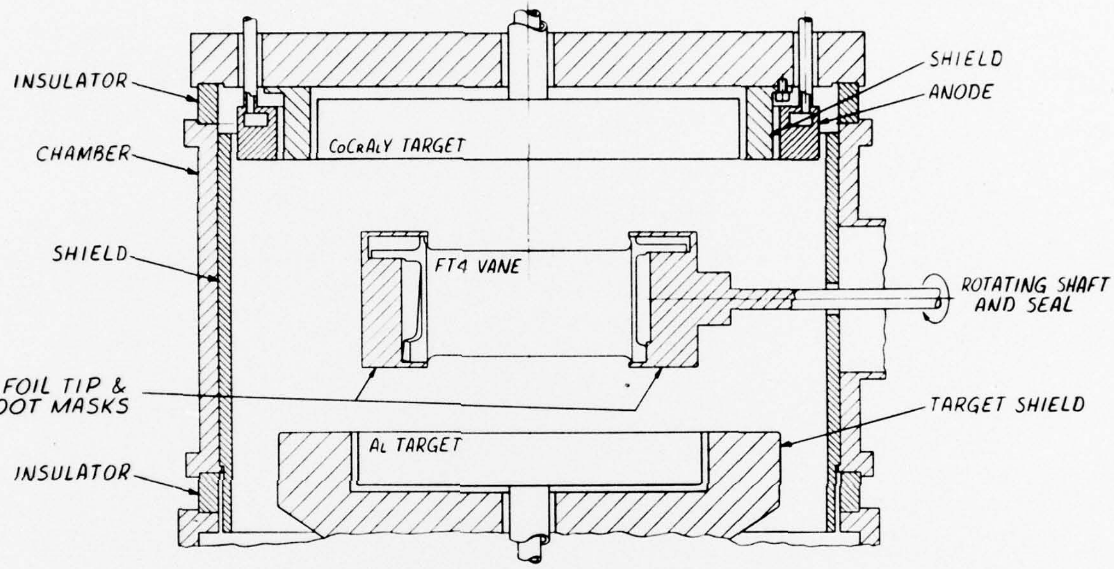
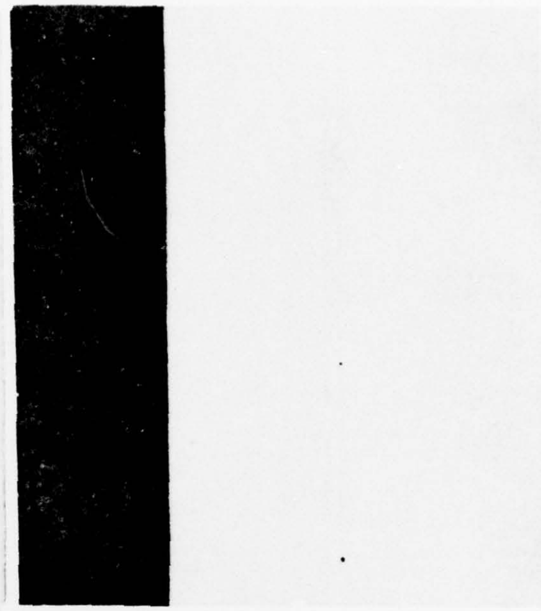


FIGURE 3. BNW-Developed CoCrAlY Sputtering Chamber.

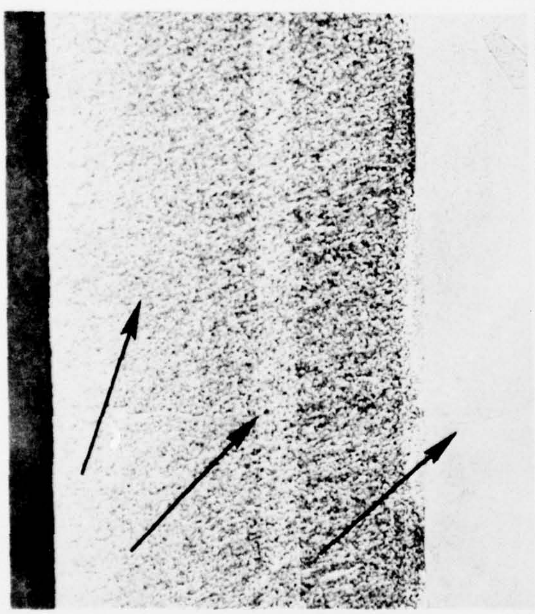
Tip Mask. As-Polished, 250X

Figure 4a



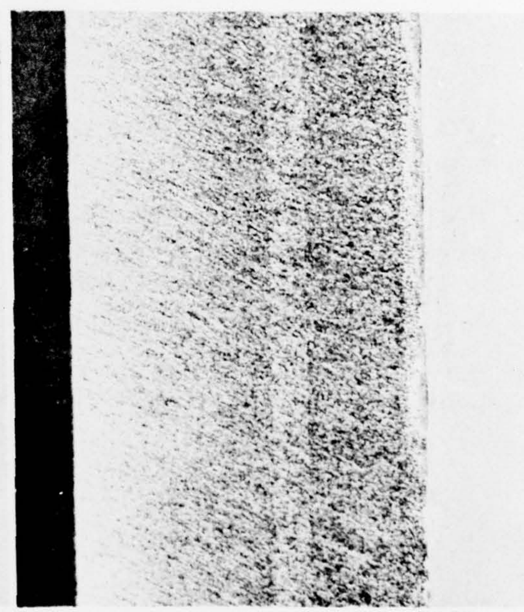
Root Mask. As-Polished, 250X

Figure 4b



Tip Mask. Etched, 500X

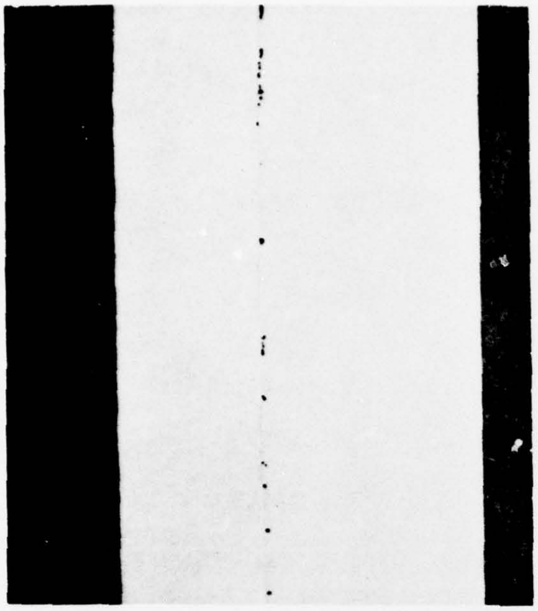
Figure 4c



Root Mask. Etched, 500X

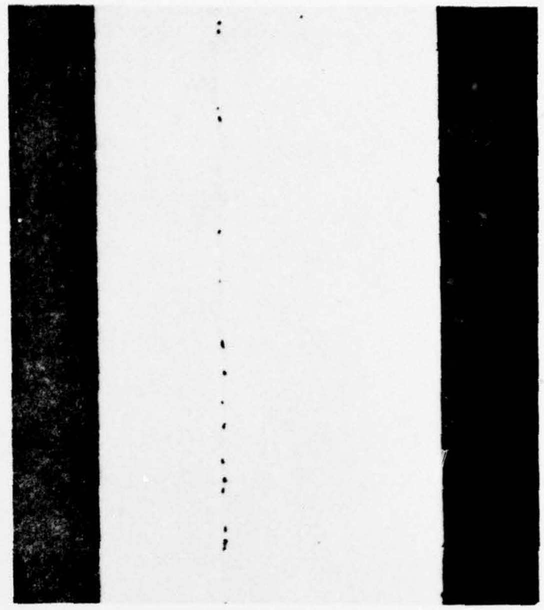
Figure 4d

FIGURE 4. As-sputtered CoCrAlY coating on Ni tip and root masks, experiment 146. In Figure 4c, the left arrow indicates the CoCrAlY coating, the center arrow indicates material deposited between two sputtering interruptions, and the right arrow indicates the Ni mask material.



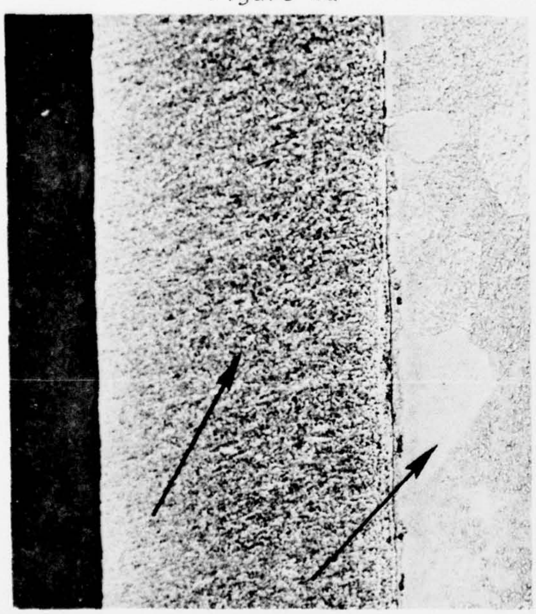
Tip Mask. As-Polished, 250X

Figure 5a



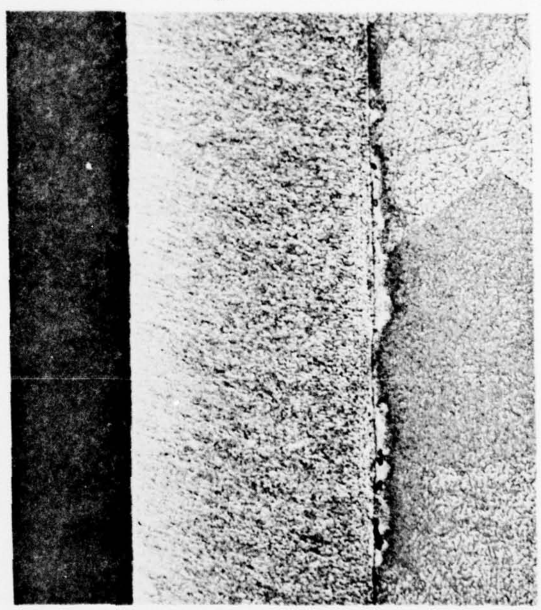
Root Mask. As-Polished, 250X

Figure 5b



Tip Mask. Etched, 500X

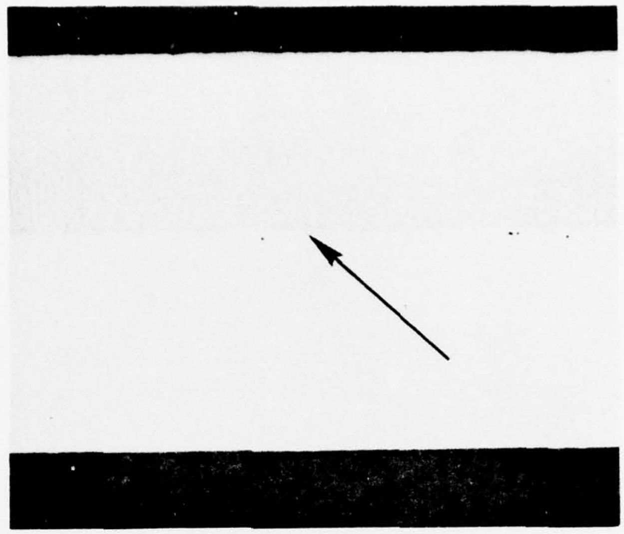
Figure 5c



Root Mask. Etched, 500X

Figure 5d

FIGURE 5. As-sputtered CoCrAlY coating on Ni tip and root masks, experiment 147. In Figure 5c, the left arrow indicates the CoCrAlY coating, the right arrow indicates the Ni mask material. The voids at the coating-mask interface resulted from the grooved as-rolled surface of the Ni foil. The absence of growth defects at these grooves illustrate the potential of this coating method for high integrity coatings.



As-Polished, 250X

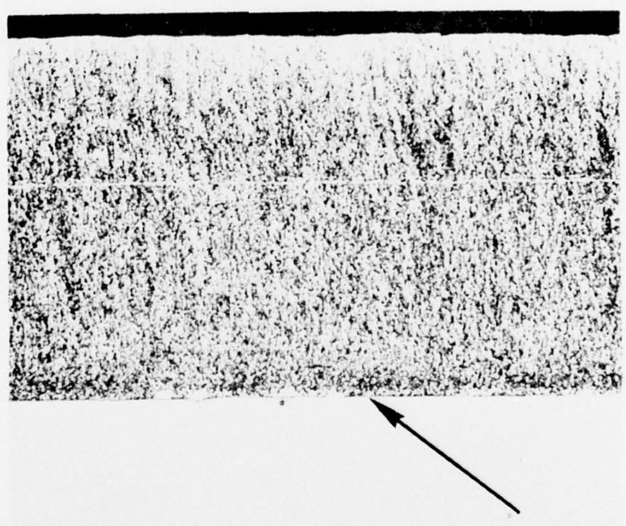


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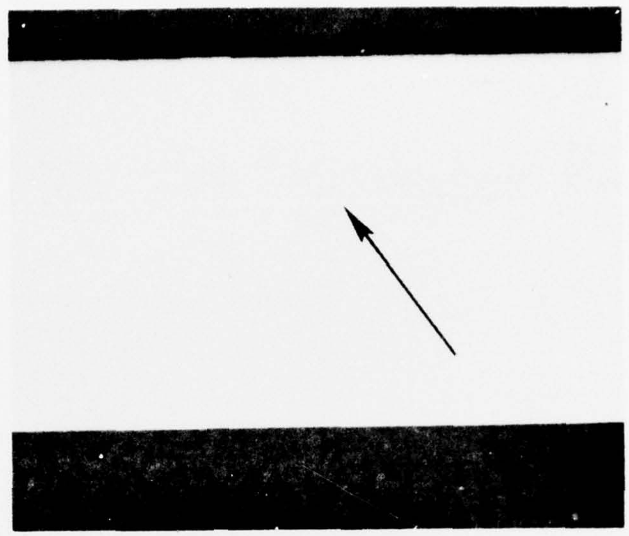
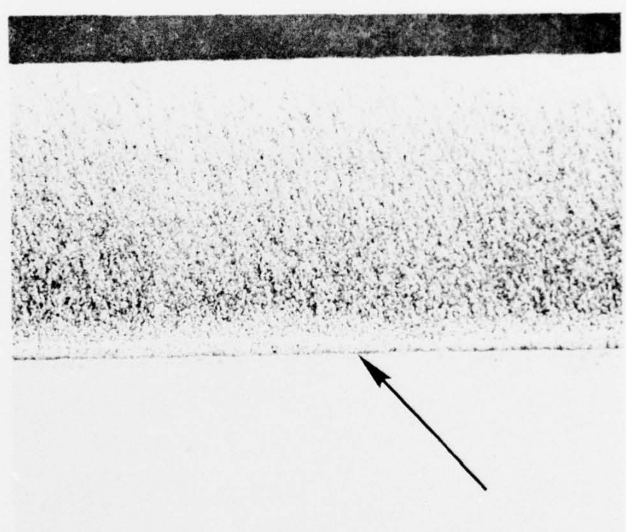
FIGURE 6. CoCrAlY sputtered deposit on Ni mask from experiment 157 (10-12% Al), vane L-2. Arrows indicate CoCrAlY coating.



As-Polished, 250X



Etched, 500X

FIGURE 7. CoCrAlY sputtered deposit on Ni mask from experiment 159 (6-9% Al), vane C-8. Arrows indicate CoCrAlY coating. The white layer between the CoCrAlY and Ni is the Pt underlayer, as the same Ni mask was used during Pt sputter coating.

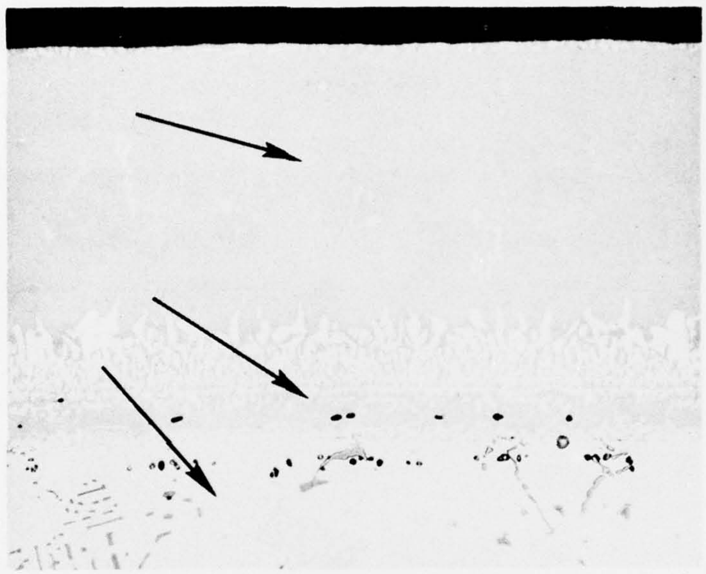


FIGURE 8. Typical heat-treated structure of sputter deposited CoCrAlY (10-12% Al) with Pt underlayer on FT4 turbine vane for at-sea testing. Experiment 169, vane V-2. Bottom arrow indicates vane, middle arrow indicates Pt deposit plus diffusion, top arrow indicates CoCrAlY deposit. 425X

TABLE I. Pt Sputtering Parameters for Coating on FT4 Vanes

Exp. No.	Vane Number	Substrate Material	Target Current[1] (amps)	Substrate Temperature (°C)	Deposition Time (min)	Avg. Substrate Wt. Gain (g)
152	C-6 C-7 C-8	Superalloy plus sputtered CoCrAlY	3.3	750	45	3.21
158 158A	C-2 C-5 L-3	Bare superalloy	3.3	740	45	3.57
162 162A	L-5 L-6	" "	3.6	730	46	4.32
164	L-2 L-4	Superalloy plus sputtered CoCrAlY	3.6	750	46	4.36
168	V-1 V-2 V-3	V-2, V-3 -- bare superalloy. V-1 superalloy plus sputtered CoCrAlY	3.3	750	45	3.9

(1) Target potential: 500 volts.

TABLE II. Composition of CoCrAlY Targets (wt%)

Target No.	Cr (1)	Al (1)	Y (2)	O (3)	N (3)	Density (g/cc)	Sputtering Runs (4)
KB1 40-9 (5)	20.3	12.3	0.58	0.041	0.0029	7.38	147-159
KB1 40-10 (5)	20.0	12.2	0.53	0.046	0.0025	7.24	160-170

(1) Determined by wet chemistry.
(2) " " x-ray techniques.
(3) " " inert gas fusion.
(4) See Table V.
(5) These targets were made from the same batch of powder and were hot isostatic pressed at the same time.

TABLE III. Experiments Conducted to Establish Sputtering Parameters for the Sputter Deposition of CoCrAlY on FT4 Vanes from a Single Target

Exp. No.	Vane Number	Target Potential (volts)	Target Current (amps)	Substrate Temp. (^{o}C)	Deposition Time (hr)	Avg. Substrate Wt. Gain (g)
130	C-3	2000	3.1	730	4.0	6.4
131	Test Section	Run Terminated, target short --		--	--	--
132	CJ-4222	1500	Run Terminated, target short --		--	--
133	CJ-4222	2000	2.8	720	1.5	6.3
134	CJ-4222	2500	3.0	750	5.0	15.5
135	C-3	2400	2.8	750	3.0	8.2
136	Test Section	1500	2.5	650	4.5	5.1
144	B	2400	.45	750	4.0	8.8
145	P-4	2400	2.7	750	4.0	--
146	P-6	2400	2.7	750	8.1	15.3

Note: Substrate etch potential: 100 volts.
Substrate etch time: 8 minutes.

TABLE IV. Experiments to Establish Sputtering Parameters for Al Addition from a Second Target

Exp. No.	Vane Number	Target Potential (volts) CoCrAlY	Target Potential (volts) Al	Target Current (amps) CoCrAlY	Target Current (amps) Al	Substrate Temp. (°C)	Deposition Time (hr)	Avg. Substrate Wt. Gain (g)
137	Test Section	2400	1000	2.7	.03	730	3.0	8.8
138	Test Section	2400	200	2.7	2.7	700 - 750	1.5	1.7
139	Test Section	2400	2000	2.7	.4	740 - 580	3.0	3.4
140	P7	Run Terminated, anode short --			--	--	--	--
141	P7	Run Terminated, anode short --			--	--	--	--
142	P7	2400	2000	2.7	.4	750	1.5	3
143*	P7	2400	2000	2.7 1.5	.4 .2	765 700 640	12.55	17.2
157	L-2	2400	1050	2.4	.45	750	6.5	13.5
169	V-2	2400	1100	2.5	.4	750-800	6.25	13.0

*Three stages, each at a different temperature.

Note: Substrate etch potential = 100 volts.
Substrate etch time = 8 minutes.

TABLE V. Measured and Derived Al Concentrations of Sputtered CoCrAlY Coatings on FT4 Vanes Compared to Masks

Experiment Number	Al Concentration on Masks (%) Root	Al Concentration on Masks (%) Tip	Al Concentration on Airfoil (%) Near Root	Al Concentration on Airfoil (%) Near Tip	Desired Al Concentration (%)
146	9.0(2)	11.5(2)	7.0(3)	9.0(3)	6 - 9
147	9.0(2)	11.5(2)	7.0(3)	9.0(3)	"
148	9.4(3)	10.0(3)	8.2(2)	7.8(2)	"
154	9.37(3)	9.4(3)	8.2(2)	7.3(2)	"
155	8.64(3)	8.31(3)	7.6(2)	6.5(2)	"
156(1)	9.28(3)	7.82(3)	8.2(2)	6.1(2)	"
156(1)	9.0(3)	10.0(3)	7.8(2)	7.9(2)	"
157(1)	9.04(3)	13.05(3)	10.2(3)	8.0(3)	10 - 12
157(1)	12.4(3)	14.3(3)	11.2(3)	10.9(3)	"
159	9.66(3)	9.23(3)	8.5(2)	7.2(2)	6 - 9
160	11.93(3)	9.74(3,4)	10.5(2)	7.6(2,4)	10 - 12
161	11.09(3)	13.05(3)	10.2(2)	9.8(2)	"
163	11.98(3)	13.46(3)	10.5(2)	10.5(2)	"
165	12.07(3)	13.52(3)	10.6(2)	10.5(2)	"
166	11.57(3)	13.55(3)	10.6(2)	10.2(2)	"

(1) In some cases samples were examined two times with slightly different analysis techniques as experience was gained with new x-ray fluorescence equipment and modification of this equipment.

(2) These Al concentrations were derived from the relationships root concentration = mask concentration x .88 and tip concentration = mask concentration x .78. The relationships were obtained from Experiment 157 where both mask and airfoil concentrations were measured.

(3) Measured by x-ray fluorescence techniques.

(4) This value is probably low but the analysis has not yet been repeated.

TABLE VI. Deposition History for CoCrAlY Coatings on Sixteen Vanes Delivered for At-Sea Testing

Exp. No.	Vane Number	Target Potential (volts) CoCrAlY	Target Potential (volts) Al	Target Current (amps) CoCrAlY	Target Current (amps) Al	Substrate Temp. (°C)	Deposition Time (hr)	Avg. Substrate Wt. Gain (g)
147	C-2	2400	--	2.7	--	750	6.0	11.1
148	C-4	2400	--	2.7	--	760	5.4	9.5
149	C-4	2400	--	2.7	--	750	Anode short -- run terminated --	
149A	C-4	2400	--	2.7	--	750	3.3	6.3
150	C-5	2400	--	2.7	--	750	Target short -- run terminated --	
151	C-5	2400	--	2.8	--	750	Plasma limiter failed -- run terminated -	
151A/B	C-5	2400	--	2.8	--	750	5.5	13.9
153	L-1	2400	--	2.4	--	725	.9	Run terminated --
153A	L-2	2400	--	2.5	--	730	6.1	13.8
154	L-3	- SYSTEM DOWN DUE TO ANODE SHORT -				--	--	--
154A	L-3	2400	--	2.5	--	750	7.0	13.9
155	C-6	2400	--	2.5	--	750	7.2	14.3
156	C-7	2400	--	2.5	--	750	6.9	13.8
159	C-8	2400	--	2.5	--	750	6.75	13.7
160	L-4	2400	1050	2.4	.45	750-800	Heat dam broke -- run terminated --	
160A	L-4	2400	1100	2.4	.45	785	6.5	14.0
161	L-7	2400	1100	2.4	.45	790	6.55	13.7
163	L-8	2400	1100	2.4	.4	750-800	6.5	13.8
165	L-5	2400	1100	2.5	.4	750-800	6.5	13.4
166	L-6	2400	1100	2.5	.4	750-800	6.25	13.6
167	V-1	2400	1100	2.5	.4	750-800	6.75	--
170	V-3	2400	1100	2.5	.4	750-800	5.75	12.3

TABLE VII. Coatings on Delivered Vanes

Experiment No.	Vane No.	Desired Composition
148, 149, 149A	C-4	CoCrAlY (6-9% Al)
153, 153A	L-1	
155	C-6	CoCrAlY (6-9% Al) + Pt Underlayer
156	C-7	
159	C-8	
147	C-2	CoCrAlY (6-9% Al) + Pt Overlayer
150, 151, 151A/B	C-5	
154, 154A	L-3	
161	L-7	CoCrAlY (10-12% Al)
163	L-8	
165	L-5	CoCrAlY (10-12% Al) + Pt Underlayer
166	L-6	
170	V-3	
157	L-2	CoCrAlY (10-12% Al) + Pt Overlayer
160, 160A	L-4	
167	V-1	