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COMBUSTION ZONE DURABILITY PROGRAM-B
TASK VIII - SPUTTER DEPOSITED CERAMIC AND METALLIC COATINGS
EXECUTIVE SUMMARY: October 1, 1979 - September 30, 1980

J. W. Patten
R. W. Moss
D. D. Hays

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DOE Richland, WA

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INTRODUCTION

This Task is described in three parts as follows:

Graded Metal Coatings

The coatings will be of the CoCrAlY type modified by including high Cr surface compositions, gradients in Cr and Al composition, underlayers and graded Pt additions, and Hf substitutions for Y.

Metal/Ceramic Layered Coatings

Coatings will consist of alternate metal (Ni, Ni-Cr, CoCrAlY or Pt) and ceramic (Al_2O_3 or ZrO_2 + Y) layers. The ceramic layers will provide most of the corrosion resistance and thermal insulation. The metal layers will provide the required mechanical properties adherence, thermal expansion compatability, resistance to impact damage, etc.)

Dense Surface Ceramic Coatings

Investigations are directed towards methods for obtaining adherent impermeable ceramic protective coatings for gas turbine hot section components. Increased coating adherence is being sought through two coating designs intended to accommodate expansion and modulus mismatches at the coating-substrate interface.

GRADED METAL COATINGS

Objective

Attempt to control early formation and regeneration of protective oxide scales on metallic coatings thru design of coating surface compositions and composition gradients thru the thickness of coatings (diffusion of oxide scale forming elements to the coating surface). Thru this control, it is intended to extend the durability provided by current state-of-the art metallic coatings for metallic components in combustion zone hot sections of heat engines operated on advanced alternate fuels.

Approach

Produce defect-free, fine-grained, uniform composition (free of segregation) coatings on superalloy substrates by high-rate triode sputter deposition. Thru burner rig testing (in coal-derived fuel at the Cranfield Institute and in petroleum distillate at Annapolis) of coatings produced in this way, examine the influence on durability of Cr content and Cr/Al proportion at the coating surface, Cr gradient to the coating surface, Al gradient to the coating surface, Al content at the coating-substrate surface, Pt content, and Hf content.

Progress (September 30, 1980)

A large number of coating compositions and composition gradients were sputter deposited on Ni-base and Co-base superalloy pins for burner rig testing. Cr concentrations up to 35% were investigated on coating outer surfaces.

Duplicate coated pins were sent to the Cranfield Institute (Table I) for testing with coal derived liquid fuel and to Annapolis (Table II) for testing with doped #2 diesel fuel.

Representative metallography of coatings prior to burner rig testing is included here in Figures 1-13.

All burner rig testing at Annapolis has been completed. The first test at Cranfield has been completed. Preliminary evaluation of results has been completed at Cranfield and Annapolis began evaluation of results the first week of June, 1980.

Tip sections of each tested pin from Annapolis have been received at PNL. Extensive evaluation of these specimens has been initiated. No specimens have yet been received from Cranfield.

Specimens will also be forwarded to LBL for evaluation of oxide scale formation mechanisms after examinations at PNL are completed.

Burner Rig Testing - Cranfield Institute:

This was a very aggressive test environment, more so than would be expected in actual service. Characteristics were as follows:

- 750°C (1382°F), 1 atmosphere pressure, 100 hours, 8.1 U.S. gal/hr fuel consumption.
- 2.9 to 1 blend of middle to heavy distillate SRCII. (At present, use of heavy distillate in internally fired heat engines is not anticipated.)
- Vanadium content was 30 ppm, so that vanadium attack dominated other coating degradation processes.
- Inspite of concentrated efforts to prevent it, carbon deposition from spalling of combustion zone surfaces was very severe. As a result, carbon particles on the order of 1 cm in diameter were constantly impinging on the coated specimens. However, all coatings tested provided protection from this environment.
- It was concluded that testing with (vanadium free) middle distillate SRCII would be much more simple and would probably produce entirely different hot corrosion behavior.
- Graded Coatings were 2-3 times better than uniform composition coatings with the same microstructure and identical outer surface compositions.
- The PVD coating (proprietary) was slightly better than the graded sputtered coatings. However, chemical compositions were not identical.
- Coatings without Pt underlayers were, in every instance, slightly better than the identical coatings with Pt underlayers.
- There was a significant influence of metal substrate on the corrosion resistance of the graded coatings. Coatings on IN972 were more resistant than coatings on MAR M 509. Coatings on X40 were least resistant.

Problems Encountered

Work loads at Annapolis and technical problems in obtaining satisfactory combustion of the coal-derived fuel at Cranfield delayed availability of tested specimens for evaluation.

METAL/CERAMIC LAYERED COATINGS

Objective

Combine the low thermal conductivity and high corrosion/erosion resistance of ceramics with the mechanical compatability (with metal substrates) and toughness of metals in a coatings system. Test the potential of such a coating system for increasing durability of metallic components in combustion zone hot sections of heat engines operated on advanced alternate fuels.

Approach

Sputter deposit coating systems comprised of many alternate layers of metal and ceramic. Each ceramic layer is intended to be segmented, with each segment extending thru the coating thickness and attached to adjacent metal layers. Each metal layer is intended to be continuous and contain the segmented ceramic layers.

Progress (September 30, 1980)

Parameters and equipment were developed on this program to allow coating hemispherical burner rig tests specimens for Westinghouse with alternate layers of NiCrAlY and ZrO_2 (+Y_2O_3). A paper describing early results will be published in Thin Solid Films [1].

These coatings included graded transitions from metal to ceramic layers but not from ceramic to metal layers. An evaluation of the burner rig tested specimens indicated:

- All coating layers remained continuous after testing.
- Delamination frequently occurred at the ceramic-metal interfaces (not graded) while metal-ceramic interfaces (graded transitions) remained intact.
- In some instances, fractures parallel to the layer planes were observed in ceramic layers.

Previous results on this program and recent results on the Dense Surface Ceramic Coatings program suggest that:

- Graded transition zones should be more durable as their thickness increases.
- Ceramic layers thicker than approximately 0.0002 inches (0.0005 cm) can be expected to fracture parallel to the layer plane.

Based on these results, the test results could be improved by reducing ceramic layer thickness and by incorporating graded ceramic to metal transition zones.

A sputtering chamber with shutters for both metal and ceramic targets is required to produce this type of coating, as was pointed out at Castine in August of 1979. Such a chamber has been designed and is near completion. The first coatings made in this chamber are expected to be available for test in November of 1980. A schematic of the dual-shutter sputtering chamber is included here.

Problems Encountered

Fabrication of the two-shutter system has required much more time than anticipated. No technical problems have been encountered, however.

DENSE SURFACE CERAMIC COATINGS

Objective

Develop ceramic coatings that, thru innovative microstructural design and composition modification, can accommodate differences in thermal expansion and modulus between ceramic coatings and metal substrates. Additionally, these coatings must be impervious at their outer surfaces to penetration by combustion byproducts and must be smooth to provide high heat transfer coefficients. It is anticipated that such coatings will permit use of the corrosion/erosion resistance of ceramic materials to increase durability of metallic components in combustion zone hot sections or heat engines operated on advanced alternate fuels.

Approach

Early PNL (Pacific Northwest Laboratory) research directed towards producing high integrity sputter-deposited CoCrAlY coatings on marine gas turbine first-stage airfoils indicated that most line-of-sight deposition techniques resulted in columnar growth structures in the coatings, with voids between adjacent growth columns that often extend entirely through the coating thickness. These voids are commonly referred to as leaders, and have recently been associated with a geometrical shadowing mechanism. A shuttered dual target combined mode (rf and dc) sputtering system capable of coating seven substrates simultaneously was used to deposit metal bond layers, layers graded from metal to ceramic, and ceramic layers, Figure 14.

Progress (September 30, 1980)

Metal/ceramic hybrid coatings as described below have been successfully sputter deposited with a segmented columnar (fibrous) microstructure oriented perpendicular to the coating surface [2].

It was demonstrated that entirely ceramic (ZrO_2 + Y_2O_3) coatings or metallic (NiCr or CoCrAlY) coatings graded thru the coating thickness to ceramic can be deposited. Fibrous growth is densely packed, approximately 0.5µm in diameter, and extends through the entire coating thickness. Fracture occurs along the columnar fiber boundaries.

A thick, graded metal-to-ceramic transition zone was found to improve coating adherence. The graded transition from metal to ceramic may be accomplished over as wide or as narrow a portion of the coating thickness as desired.

Any selected portion of the coating thickness may be deposited with either continuous or segmented columnar microstructure. A sealing or close-out layer is required on the outer surface of segmented ceramic coatings before testing in heavily-contaminated diesel fuel or coal-derived liquid fuel is undertaken.

Early results on this program indicate that a continuous NiCrAlY sealing layer can be expected to separate from the segmented oxide coatings due to difference in thermal expansion.

Techniques developed on this program have allowed application of continuous sealing layers of several compositions (less than 0.003 cm = 0.001 in. thick) to plasma sprayed ZrO_2 coatings for burner rig evaluation at Westinghouse:

- ZrO_2 (20% Y_2O_3)
- Pt
- ZrO_2 (+20% Y_2O_3) graded to Pt
- ZrO_2 (+20% Y_2O_3) graded to Pt_3Al

Photographs of typical close-out layer structures are included in Figures 15 - 23.

Experiments have been conducted to apply similar ZrO_2 - based sealing layers to sputter-deposited segmented ZrO_2 coatings. Evaluation of structures obtained is in progress.

An advanced version of this coating type is being investigated. Composition grading extends thru the entire coating thickness. Individual columns are metal at their inner ends and ceramic at their outer ends, with the last material deposited (mostly ZrO_2) being continuous to provide a sealing function. These coatings have been produced and are being evaluated.

Problems Encountered

None

REFERENCES

1. Patten, J.W., J.T. Prater, D.D. Hays, R.W. Moss, and J.W. Fairbanks, "Mechanical Behavior of Segmented Oxide Protective Coatings," presented at the International Conference on Metallurgical Coatings, San Diego, California, 1980. To be published in Thin Solid Films.

2. Patten, J.W., M.A. Bayne, D.D. Hays, R.W. Moss, E.D. McClanahan, J.W. Fairbanks, Thin Solid Films, 64 (1979) 337-343.

ACKNOWLEDGMENT

The Authors gratefully acknowledge R.H. Beauchamp for optical metallography.

TABLE I

1/8" Diameter Pins for Burner Rig Testing at the Cranfield Institute of Technology Cranfield, Bedford, England

Run	Sample	Pin Alloy	Voltage on Auxiliary Target[1]	Pt Underlayer Thickness (mils)	CoCrAlY Thickness (mils)	Thermal Treatment of Pins	Metallography Sample[2]	Deposit Composition %[3]
5	2002	IN 792	50' @ 500, 600, 700 V, 5' @ 800 V	0.2	~4.6	Vac HT 4 hrs @ 1080°C	None	Cr: 25-30 (est);
	2019	"	" "	0	"	" "	"	Al: 10- 8 (est)
6	2004	IN 792	55' @ 500, 600, 700, 800 V	0.2	~5.5	Vac HT 4 hrs @ 1080°C	None	Cr: 25-30 (est);
	2021	"	" "	0	"	" "	"	Al: 10- 8 (est)
7	2006	IN 792	60' @ 600, 700, 800, 10' @ 900 V	0.2	4.2	Vac HT 4 hrs @ 1080°C	2005B	Cr: 24.1-32.6
	2022	"	" "	0	"	" "	"	Al: 9.8-8.4
8	2007	IN 792	60' @ 600, 700, 800, 900, 1000 V	0.2	~7.0	Vac HT 4 hrs @ 1080°C	2025B	Cr: 25-35 (est);
	2008	"	" "	"	"	" "	"	Al: 10- 8 (est)
	2024	"	" "	0	"	Vac HT 4 hrs @ 1080°C + 1 hr in air @ 1080°C	2025C	
9	1038	X-40	60' @ 600, 700, 800, 900, 1000 V	0	~7.0	None (as deposited)	None	Cr: 25-35 (est);
	5038	MAR-M-509	" "	"	"	" "	"	Al: 10- 8 (est)
13	2028	IN 792	1000 V	0	6.5	Vac HT 4 hrs @ 1080°C	2026B	Cr: 32.6 [7]
	2029	"						Al: 9.4 [7]
14	2037	IN 792	800 V	0	6	Vac HT 4 hrs @ 1080°C	2030B	Cr: 30 [7]
	2053	"						Al: 9.5 [7]
17	2052	IN 792	60' @ 600 V, 5' @ 700 V	0.2	1.4	Sputter etched after nitriding @ 900°C during HT	2043NE	Cr: 22.5 (est); Al: 10 (est)
	2036	"	" "	0	"	" "	"	
18	2056	IN 792	60' @ 600, 700, 800, 900, 1000 V	0.2	7	Sputter etched after nitriding @ 900°C during HT	2055NE	Cr: 32-33.5 Al: 9.3-9.2
24	1002	X-40	60' @ 700, 600, 500, 400, 300 V;	0.2	5.3	Vac HT 4 hrs @ 1080°C	1041B	Cr: 19.4-26.3
	1020	"	20' @ 200 V	0.5				Al: 18.4-9.1
	1042	"		0				

TABLE I (continued)

1/8" Diameter Pins for Burner Rig Testing at the Cranfield Institute of Technology Cranfield, Bedford, England

Run	Sample	Pin Alloy	Voltage on Auxiliary Target[1]	Pt Underlayer Thickness (mils)	CoCrAlY Thickness (mils)	Thermal Treatment of Pins	Metallography Sample[2]	Deposit Composition %[3]
25	5004	MAR-M-509	60' @ 700, 600, 500, 400, 300 V;[4]	0.2	5.0	Vac HT 4 hrs @ 1080°C	2044B	Cr: 23.8-30.2
	2011	IN 792	33' @ 200 V	0.5				Al 19.6-2.6
	2043	IN 792		0				
27	5006	MAR-M-509	60' @ 500, 450, 400, 350, 200 V;	0.2	4.8	Vac HT 4 hrs @ 1080°C	5032B	Cr: 27-29
	1022	X-40	23' @ 0V	0.5				Al: 6-4
	5033	MAR-M-509		0				
28	1006	X-40	60' @ 500, 450, 400, 350, 200 V;	0.2	4.1	Vac HT 4 hrs @ 1080°C	2045B	Cr: 33.8[7]
	5013	MAR-M-509	15' @ 0V	0.5				Al: 2.7[7]
	2046	IN 792		0				
29	5008	MAR-M-509	240' @ 500 V	0.2	5.4	Vac HT 4 hrs @ 1080°C	1046B	Cr: 11.7-3.1-21.3[8]
	2015	IN 792	60' @ 400, 300	0.5				Pt: 67.5-86.9-17.1[8]
	1047	X-40	0V[5]	0				
30	1008	X-40	0V[6]	0.2	4.2	Vac HT 4 hrs @ 1080°C	5035B	Cr: 35.4[7]
	5015	MAR-M-509		0.5				Al: 1.1[7]
	5036	MAR-M-509		0				
	2039[9]	IN 792		0	3.5	Vac HT 2 hrs @ 1052°C	2040B	Cr: 23.0 Al: 9.1 Y: 0.3
	2059[9]	IN 792		0.2	3.5	Vac HT 2 hrs @ 1052°C	2040B	Cr: 23.0 Al: 9.1 Y: 0.3

(1) Auxiliary target is Cr unless otherwise indicated.

(2) Metallography sample has same thermal history as pins.

(3) CoCrAlY composition from substrate to outer surface.

(4) Al Auxiliary target.

(5) Pt auxiliary target. CoCrAlY target voltage ramp; 60' @ 400, 900, 1500, 2400 V during 240' @ 500 V on Pt target. The CoCrAlY target was held at 2400 V while Pt target voltage was lowered.

(6) No auxiliary target used.

(7) Average composition. No gradient measured.

(8) Max Pt (Min Cr) at ~1 mil from interface.

(9) PVD coated pins.

TABLE II

1/8" Diameter Pins for Burner Rig Testing at the Naval Ship R&D Center, Annapolis, MD

Run	Sample	Pin Alloy	Voltage on Auxiliary Target[1]	Pt Underlayer Thickness (mils)	CoCrAlY Thickness (mils)	Thermal Treatment of Pins	Metallography Sample[2]	Deposit Composition %[3]
5	2001	IN 792	50' @ 500, 600, 700 V, 5' @ 800 V	0.2	~4.6	Vac HT 4 hrs @ 1080°C	None	Cr: 25-30 (est);
	2018	"	" "	0	"	" "	"	Al: 10- 8 (est)
6	2003	IN 792	55' @ 500, 600, 700, 800 V	0.2	~5.5	Vac HT 4 hrs @ 1080°C	None	Cr: 25-30 (est);
	2020	"	" "	0	"	" "	"	Al: 10- 8 (est)
9	1037	X-40	60' @ 600, 700, 800, 900, 1000 V	0	~7.0	None (as deposited)	None	Cr: 25-35 (est);
	5027	MAR-M-509	" "	"	"	" "	"	Al: 10- 8 (est)
13	2027	IN 792	1000 V	0	6.5	Vac HT 4 hrs @ 1080°C	2026B	Cr: 32.6 [7] Al: 9.4 [7]
14	2031	IN 792	800 V	0	6	Vac HT 4 hrs @ 1080°C	2030B	Cr: 30 [7] Al: 9.5 [7]
17	2051	IN 792	60' @ 600 V, 5' @ 700 V	0.2	1.4	Sputter etched after nitriding @ 900°C during HT	2043NE	Cr: 22.5 (est); Al: 10 (est)
	2035	"	" "	0	"	" "	"	
18	2054	IN 792	60' @ 600, 700, 800, 900, 1000 V	0.2	7	Sputter etched after nitriding @ 900°C during HT	2055NE	Cr: 32-33.5 Al: 9.3-9.2
24	1001	X-40	60' @ 700, 600, 500, 400, 300 V;	0.2	5.3	Vac HT 4 hrs @ 1080°C	1041B	Cr: 19.4-26.3
	1019	"	20' @ 200 V	0.5				Al: 18.4-9.1
	1040			0				

TABLE II (continued)

1/8" Diameter Pins for Burner Rig Testing at the Naval Ship R&D Center, Annapolis, MD

Run	Sample	Pin Alloy	Voltage on Auxiliary Target[1]	Pt Underlayer Thickness (mils)	CoCrAlY Thickness (mils)	Thermal Treatment of Pins	Metallography Sample[2]	Deposit Composition %[3]
25	5003	MAR-M-509	60' @ 700, 600, 500, 400, 300 V;[4]	0.2	5.0	Vac HT 4 hrs @ 1080°C	2044B	Cr: 23.8-30.2
	2010	IN 792	33' @ 200 V	0.5				Al 19.6-2.6
	2042	IN 792		0				
27	5005	MAR-M-509	60' @ 500, 450, 400, 350, 200 V;	0.2	4.8	Vac HT 4 hrs @ 1080°C	5032B	Cr: 27-29
	1021	X-40	23' @ 0V	0.5				Al: 6-4
	5034	MAR-M-509		0				
28	1005	X-40	60' @ 500, 450, 400, 350, 200 V;	0.2	4.1	Vac HT 4 hrs @ 1080°C	2045B	Cr: 33.8[7]
	5012	MAR-M-509	15' @ 0V	0.5				Al: 2.7[7]
	2047	IN 792		0				
29	5007	MAR-M-509	240' @ 500 V	0.2	5.4	Vac HT 4 hrs @ 1080°C	1046B	Cr: 11.7-3.1-21.3[8]
	2014	IN 792	60' @ 400, 300	0.5				Pt: 67.5-86.9-17.1[8]
	1048	X-40	0V[5]	0				
30	1007	X-40	0V[6]	0.2	4.2	Vac HT 4 hrs @ 1080°C	5035B	Cr: 35.4[7]
	5014	MAR-M-509		0.5				Al: 1.1[7]
	5037	MAR-M-509		0				
	2038[9]	IN 792		0	3.5	Vac HT 2 hrs @ 1052°C	2040B	Cr: 23.0 Al: 9.1 Y: 0.3
	2058[9]	IN 792		0.2	3.5	Vac HT 2 hrs @ 1052°C	2040B	Cr: 23.0 Al: 9.1 Y: 0.3

(1) Auxiliary target is Cr unless otherwise indicated.

(2) Metallography sample has same thermal history as pins.

(3) CoCrAlY composition from substrate to outer surface.

(4) Al Auxiliary target.

(5) Pt auxiliary target. CoCrAlY target voltage ramp; 60' @ 400, 900, 1500, 2400 V during 240' @ 500 V on Pt target. The CoCrAlY target was held at 2400 V while Pt target voltage was lowered.

(6) No auxiliary target used.

(7) Average composition. No gradient measured.

(8) Max Pt (Min Cr) at ~1 mil from interface.

(9) PVD coated pins.

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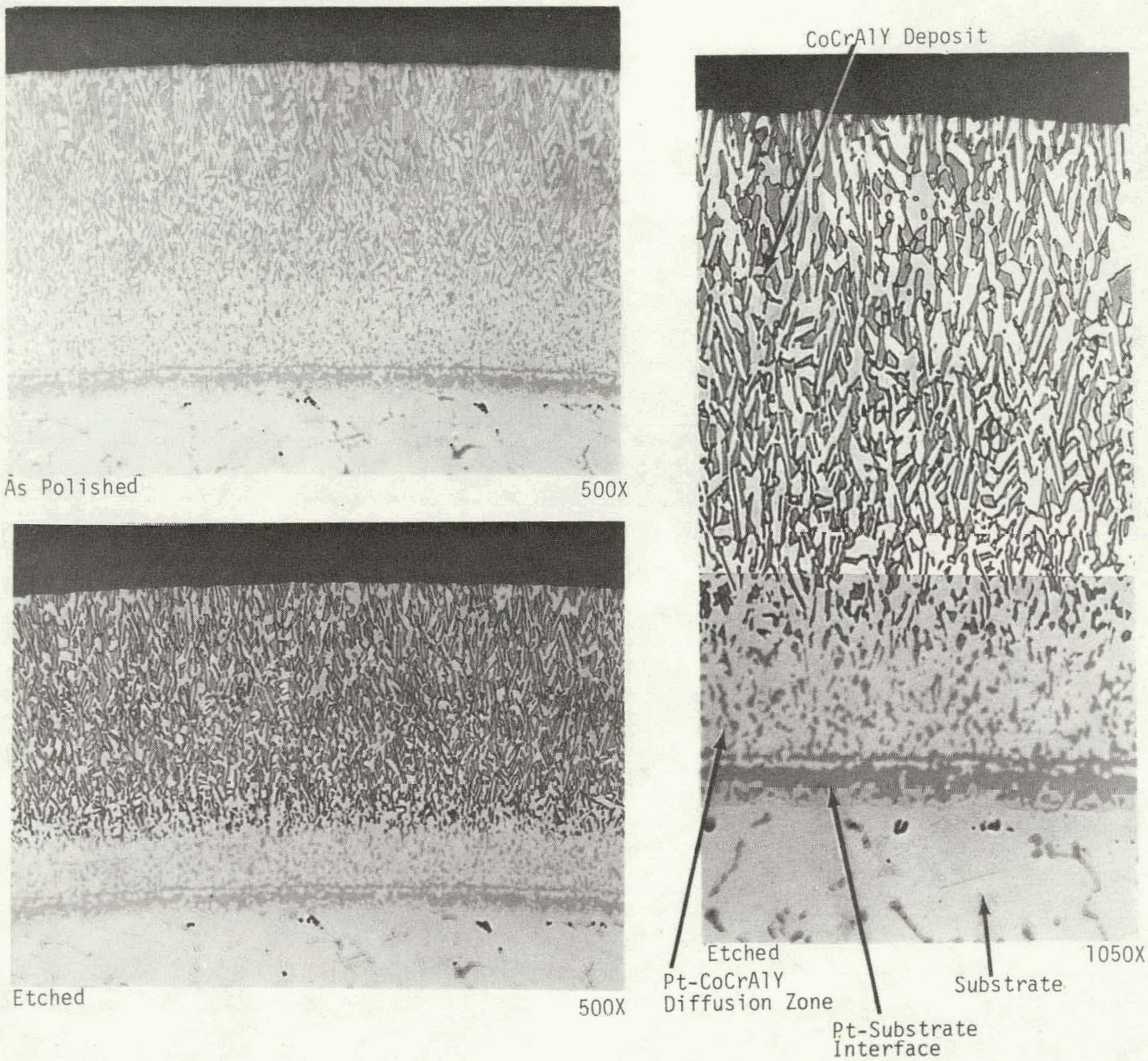

Figure 1 Run #7 Sample # 2005B
CoCrAlY sputter deposited on a Pt sputter deposited underlayer. Substrate is IN-792 1/8" dia. pin. Cr content of CoCrAlY graded from interface to outer surface. Vacuum heat treated 4 hrs at 1080°C.

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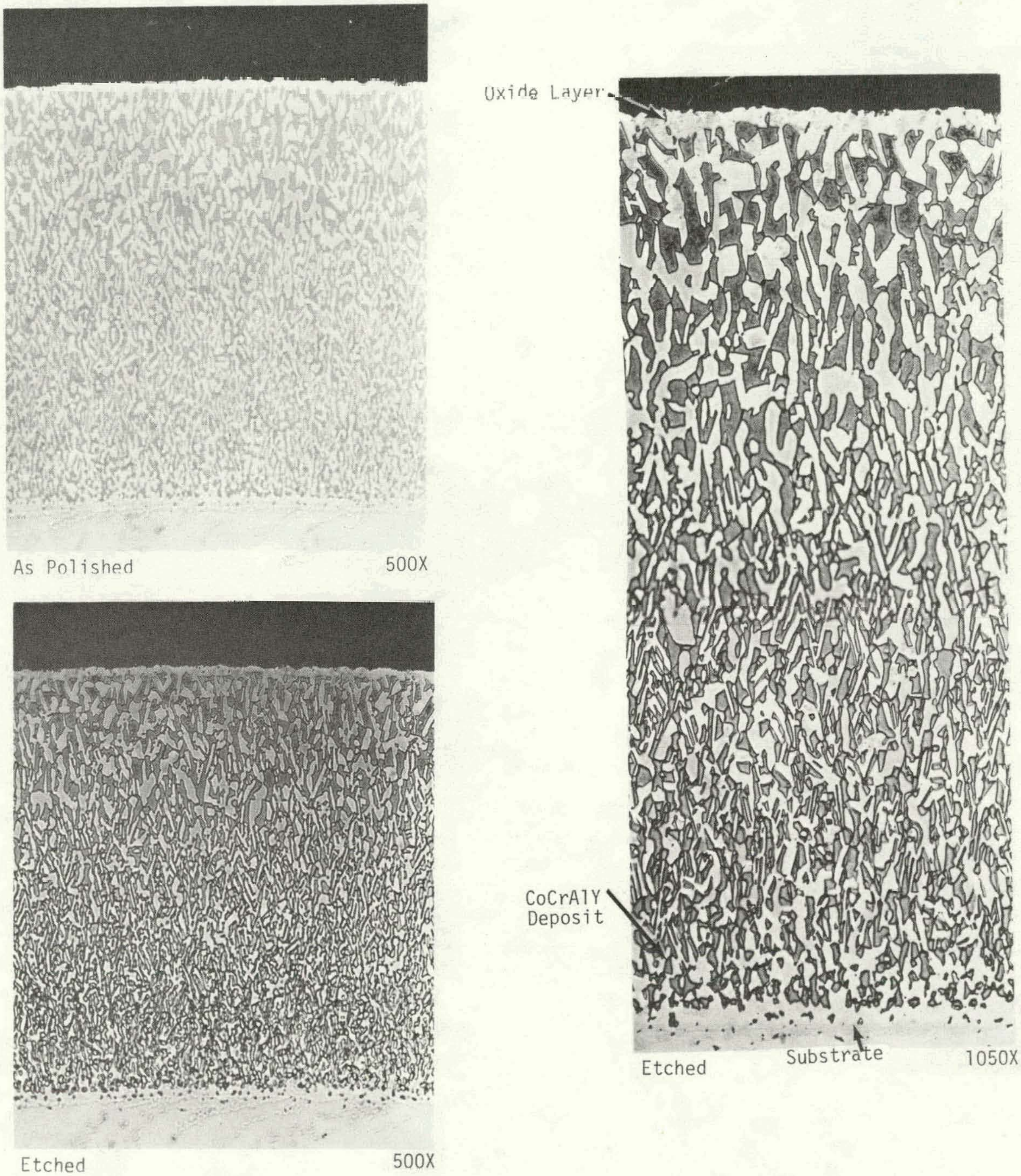

Figure 2 Run #8 Sample # 2025C
CoCrAlY sputter deposited on IN-792 1/8" dia. pin. Cr content of CoCrAlY graded from interface to outer surface. Heat **treated in vacuum 4 hrs** at 1080^{o}C and 1 hr at 1080^{o}C in air.

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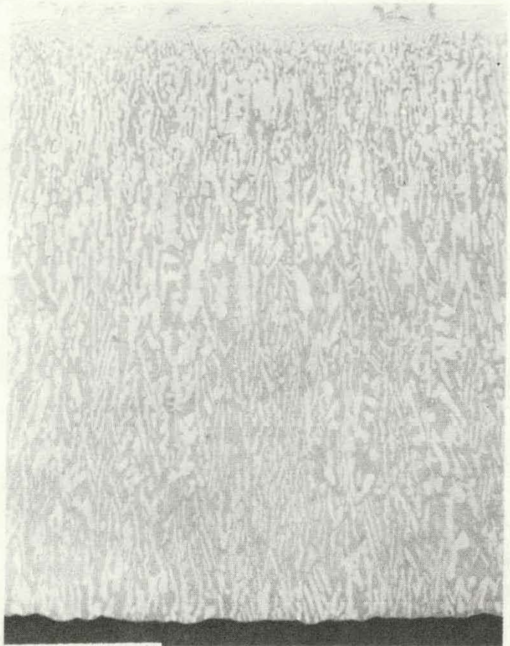

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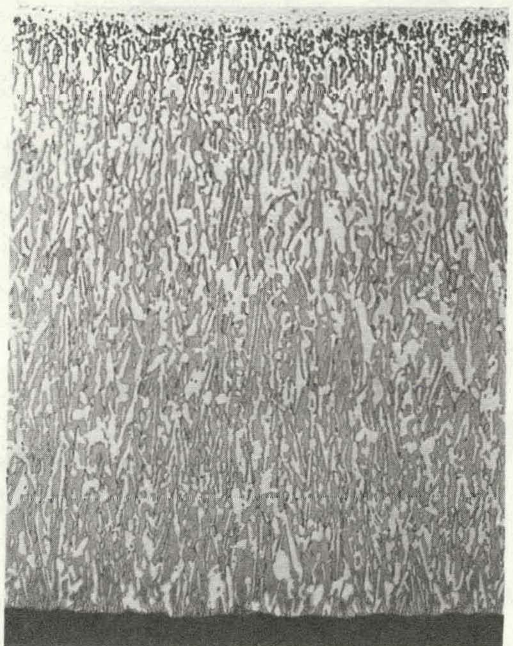

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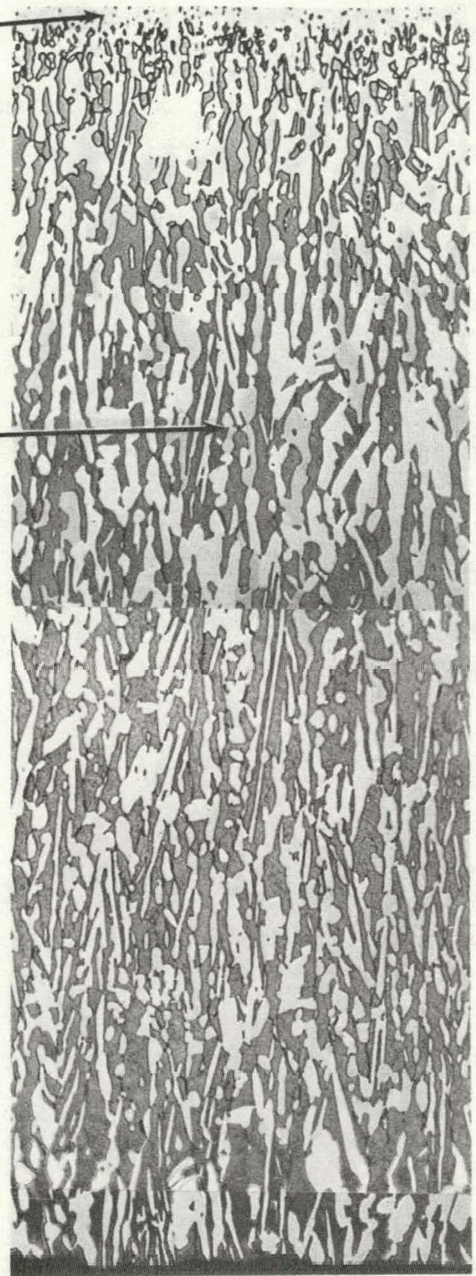

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Figure 3 Run #13 Sample # 2026B
CoCrAlY sputter deposited on a 1/8" dia. IN-792 pin. Deposit contains 33% Cr and 9% Al. Vacuum heat treated 4 hrs at 1080°C.

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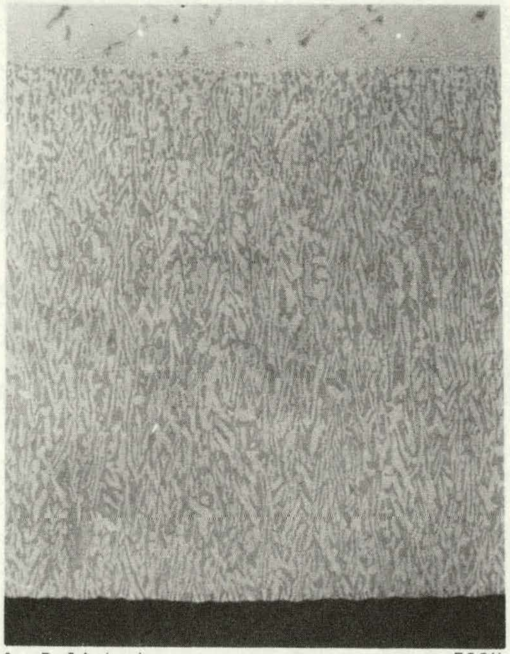

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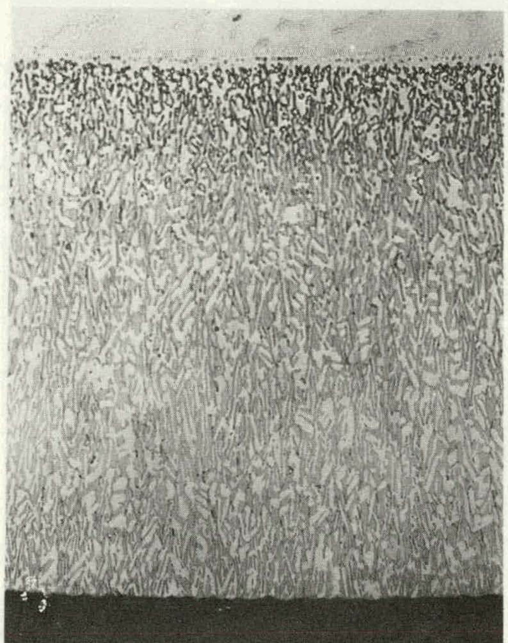

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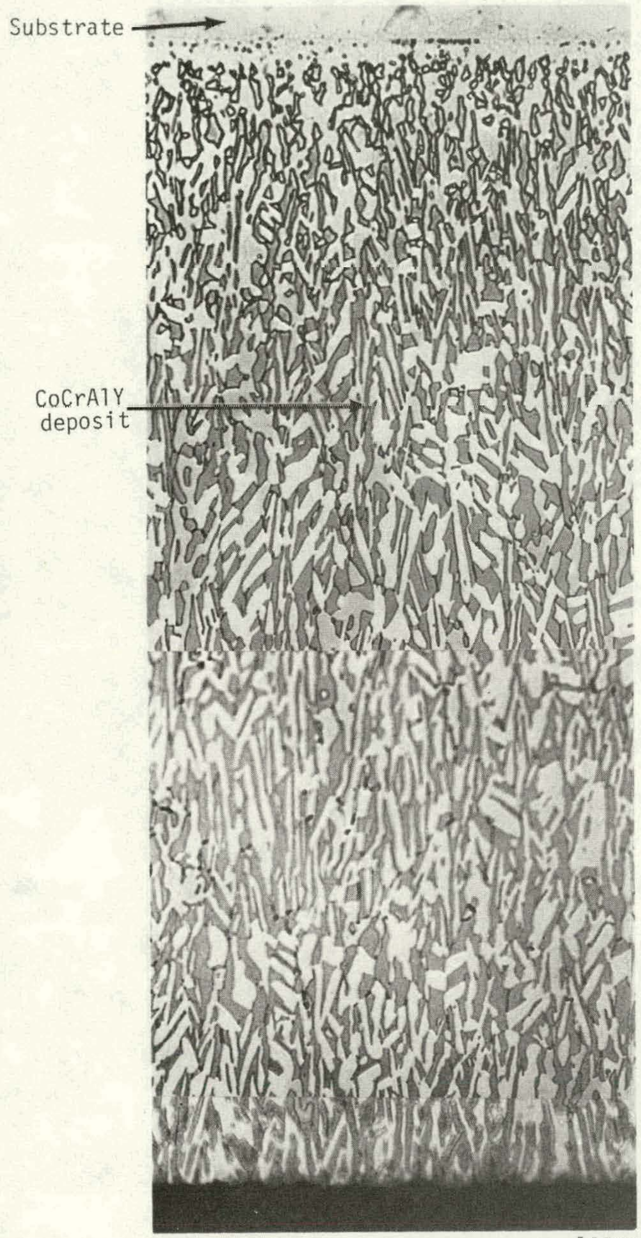

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Figure 4 **Run #14** **Sample # 2030B**
CoCrAlY sputter deposited on a 1/8" dia. IN-792 pin. Deposit contains 30% Cr and 9.5% Al. Vacuum heat treated 4 hrs at 1080°C.

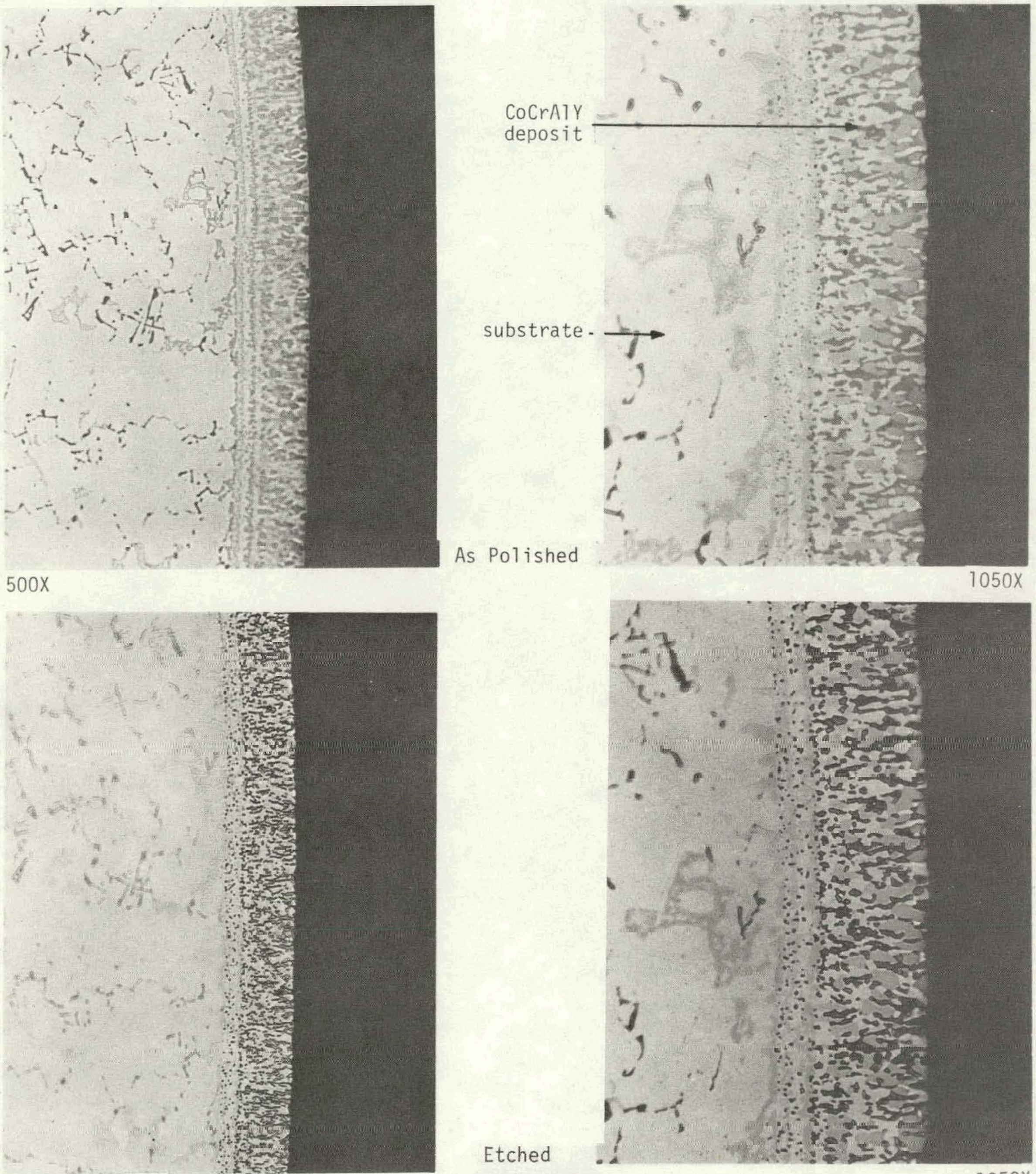

Figure 5 Run #17 Sample # 2034NE
CoCrAlY sputter deposited on a 1/8" dia. IN-792 pin. Sample has been sputter etched after exposure to nitrogen at 900°C during cooling after a 4 hr, 1080°C vacuum heat treatment.

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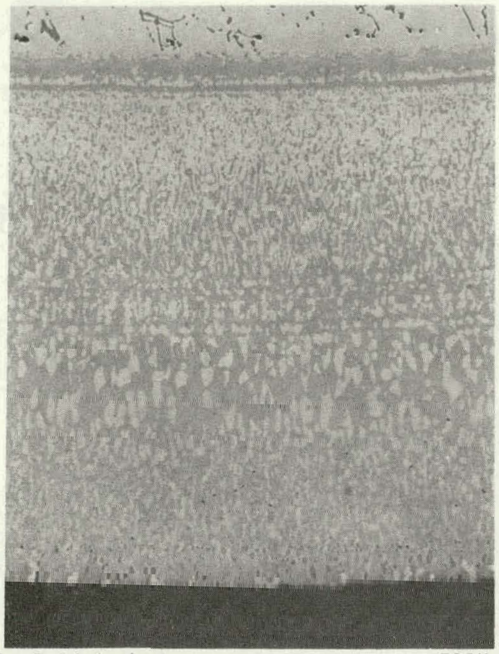

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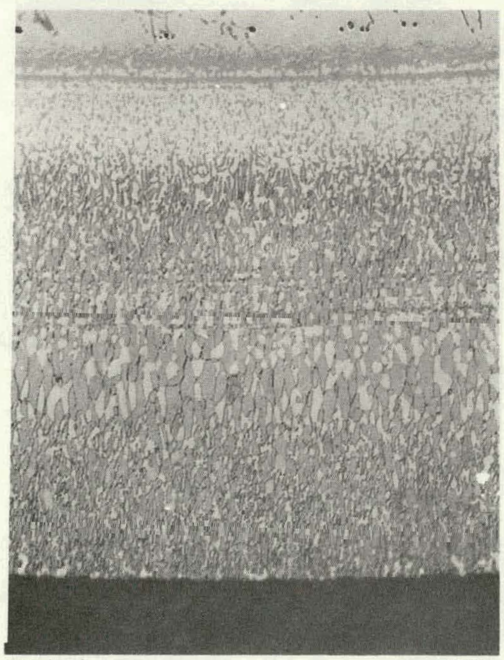

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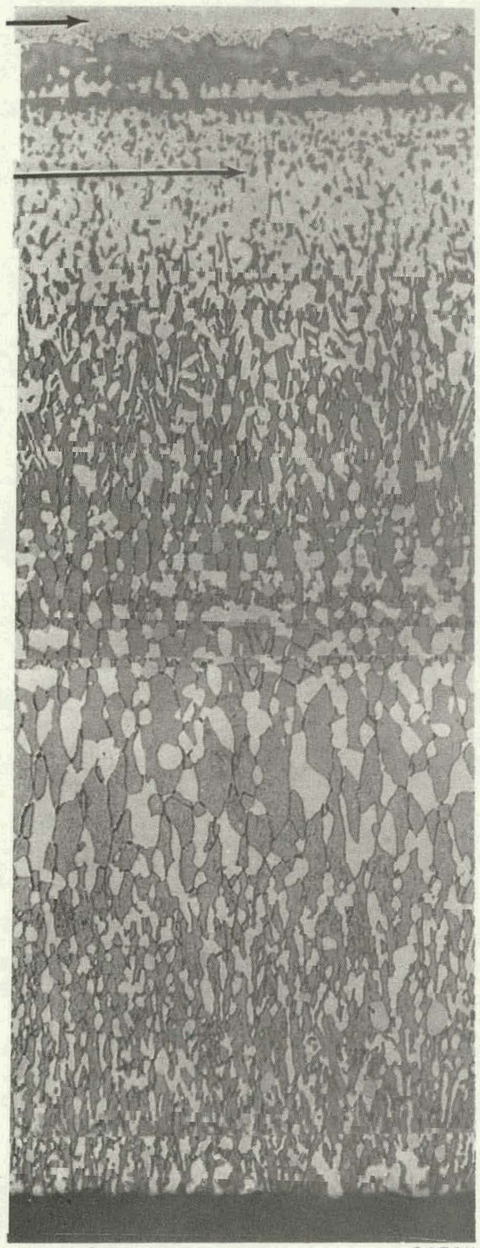

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Figure 6 **Run #18** **Sample # 2055NE**
CoCrAlY sputter deposited on a Pt sputter deposited underlayer. Substrate is 1/8" dia. IN-792 pin. Sample has been sputter etched after exposure to nitrogen during cooling after a 4 hr, 1080°C vacuum heat treatment.

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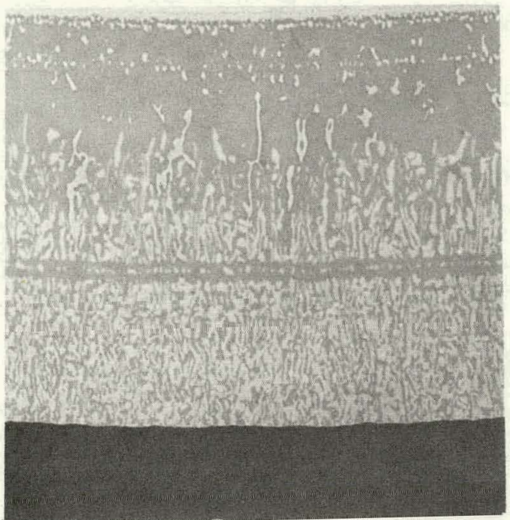
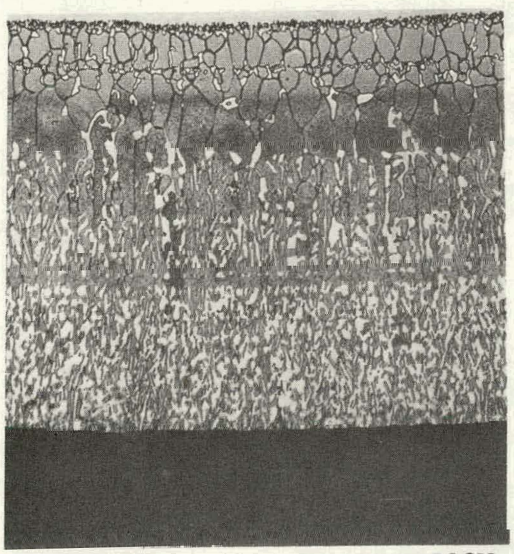


As-Polished 500X



Etched 500X

Etched 1050X

Figure 7 Run #24 Sample # 1041B

CoCrAlY sputter deposited on a 1/8 dia. X-40 pin. Vacuum heat treated 4 hrs at 1080°C.

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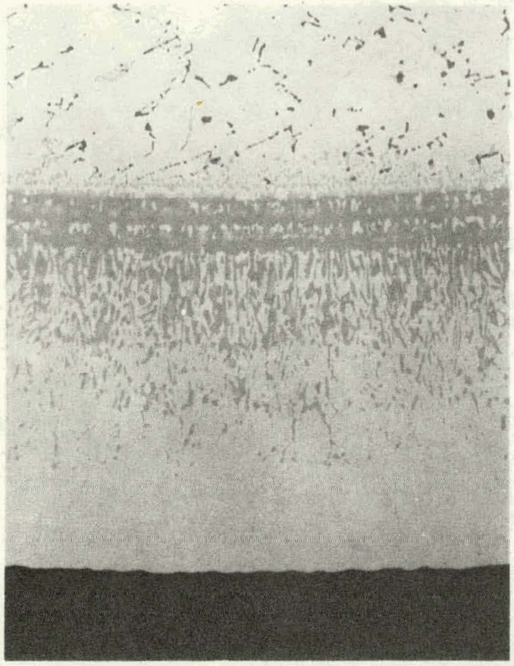
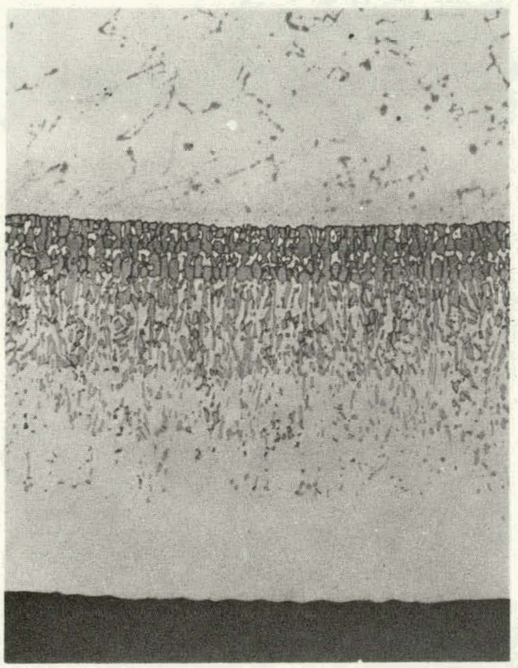


As-Polished 500X



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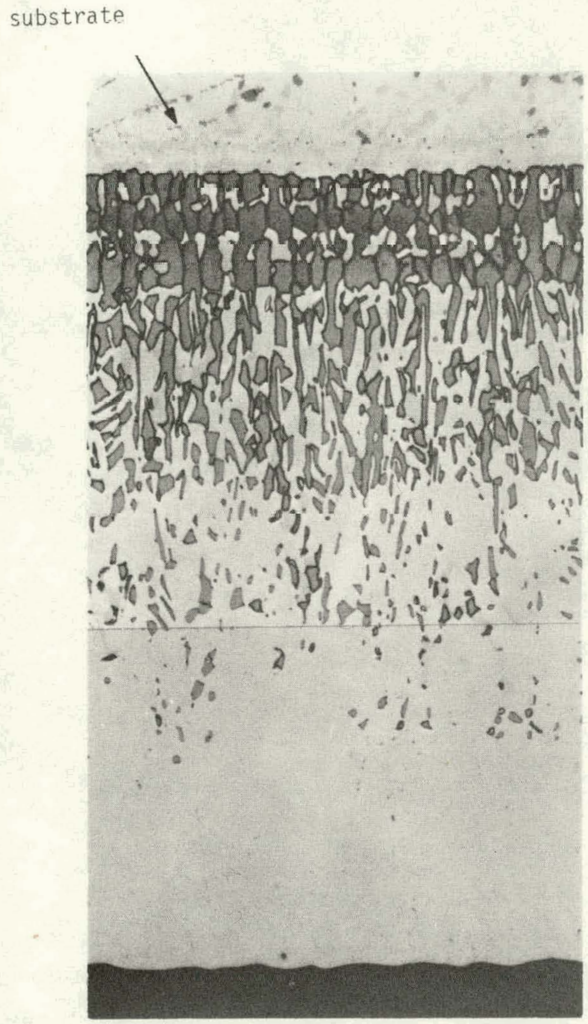

Etched 1050X

Figure 8 Run #25 Sample # 2044B
CoCrAlY sputter deposited on a 1/8" dia. IN 792 pin. Vacuum heat treated 4 hrs at 1080°C.

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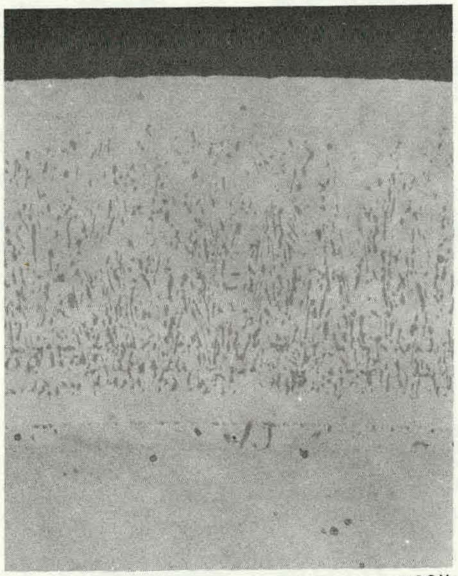
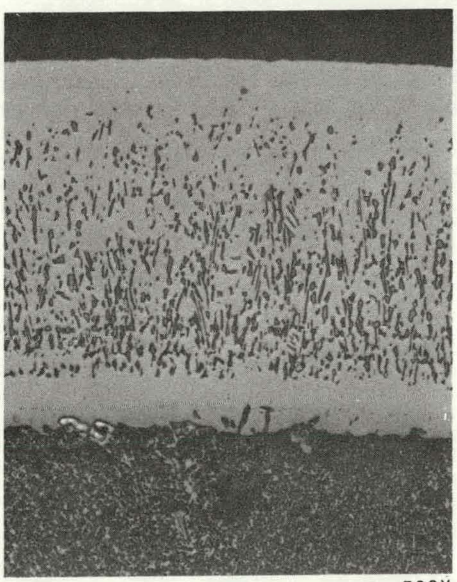
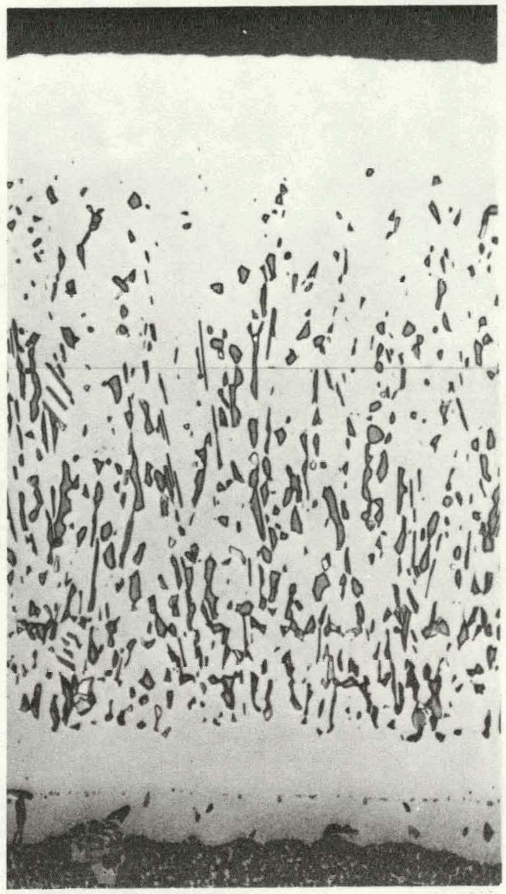


As-Polished 500X



Etched 500X



Etched 1050X

substrate

Figure 9 Run #27 Sample # 5032B
CoCrAlY sputter deposited on a 1/8" dia. MAR-M-509 pin. Vacuum heat treated 4 hrs at 1080°C.

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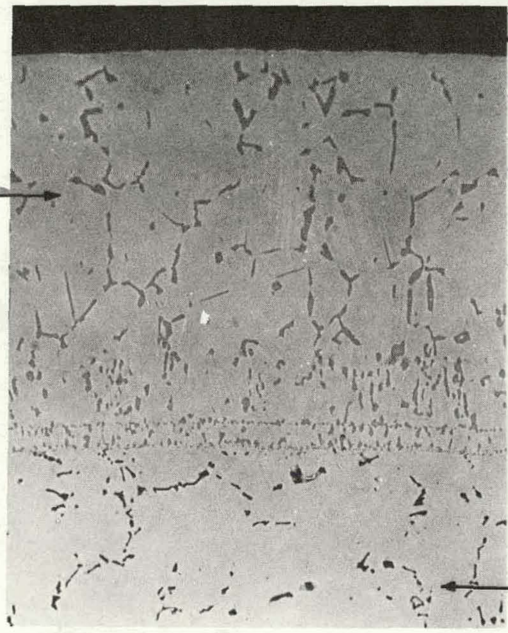

As Polished 500X

Figure 10 Run #28 Sample # 2045B
CoCrAlY sputter deposited on a 1/8" dia. IN 792 pin. Vacuum heat treated 4 hrs at 1080°C.

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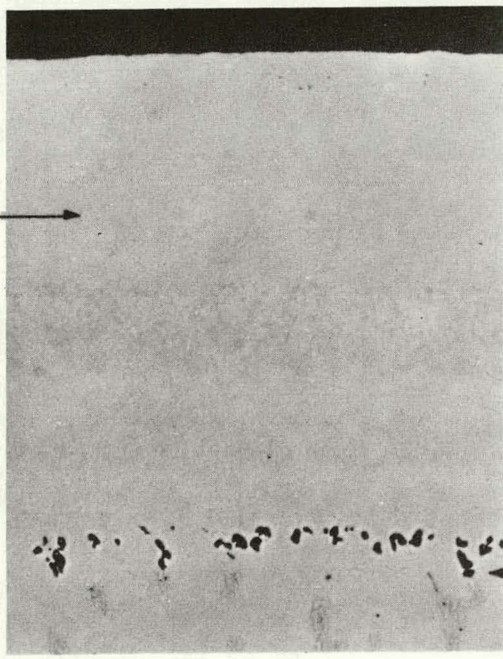

As Polished 500X

Figure 11 Run #29 Sample # 1046B
CoCrAlY sputter deposited on a 1/8" dia. X-40 pin. Vacuum heat treated 4 hrs at 1080°C.

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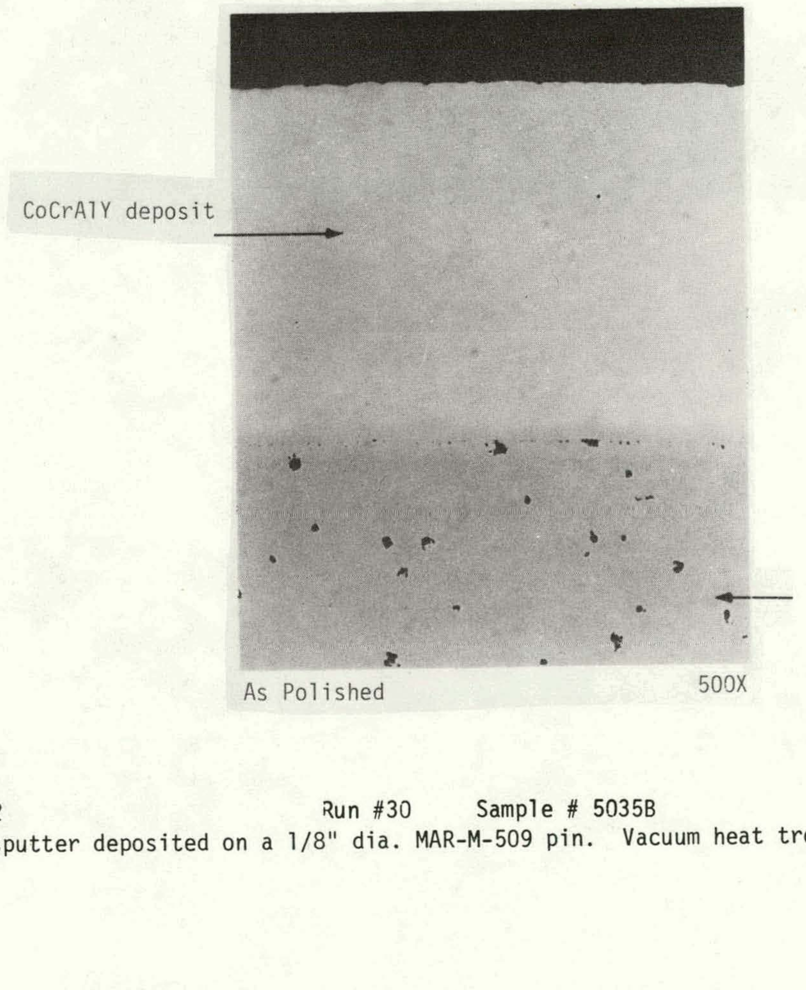

Figure 12 Run #30 Sample # 5035B
CoCrAlY sputter deposited on a 1/8" dia. MAR-M-509 pin. Vacuum heat treated 4 hrs at 1080°C.

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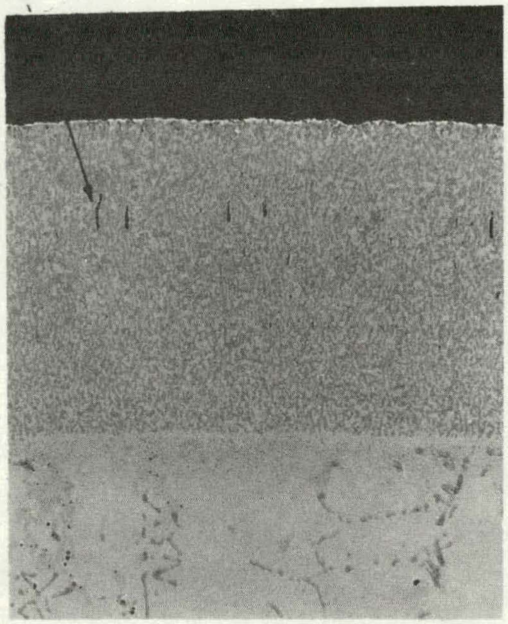
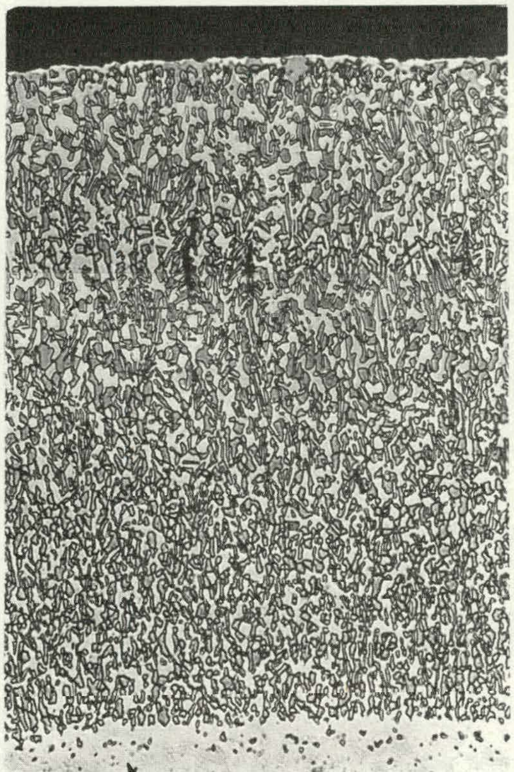
leaders

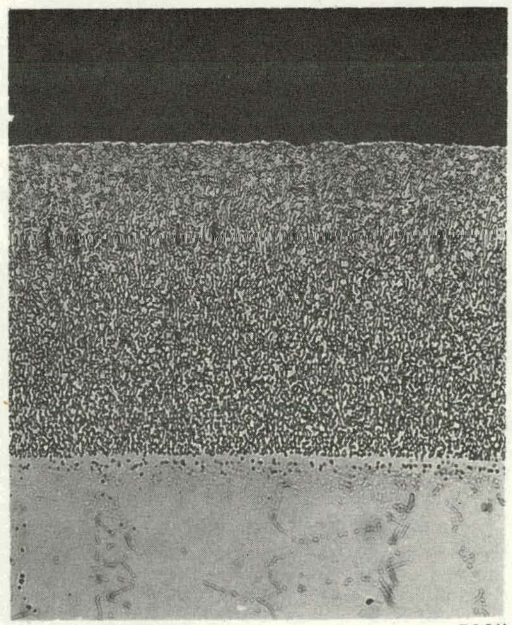


As-Polished 500X



Etched 1050X

substrate



Etched 500X

Figure 13 **PVD Sample # 2040B**

CoCrAlY vapor deposited on a 1/8" dia. IN 792 pin. Vacuum heat treated 2 hrs at 1080°C.

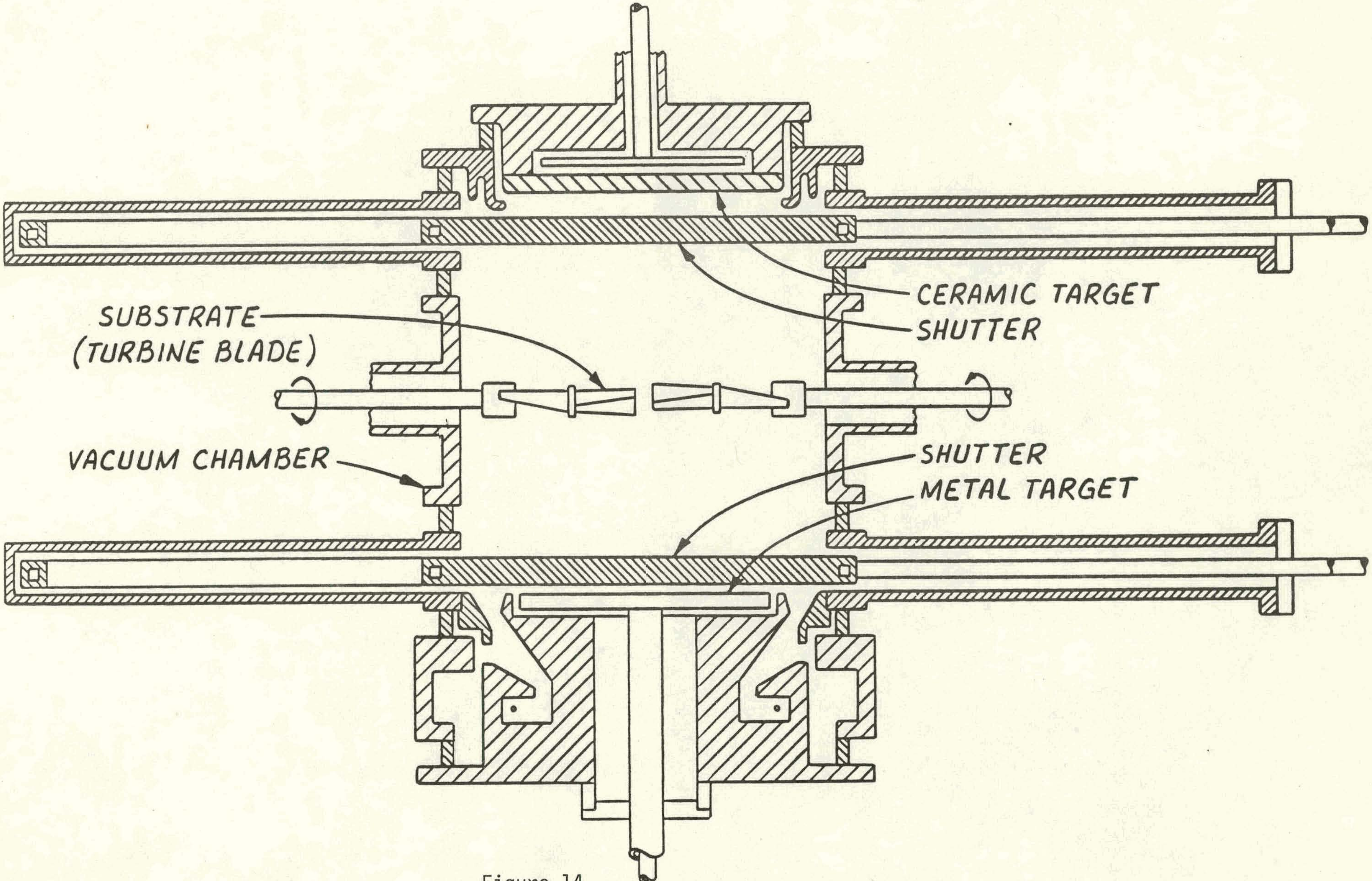


Figure 14

DUAL-SHUTTER SPUTTERING CHAMBER

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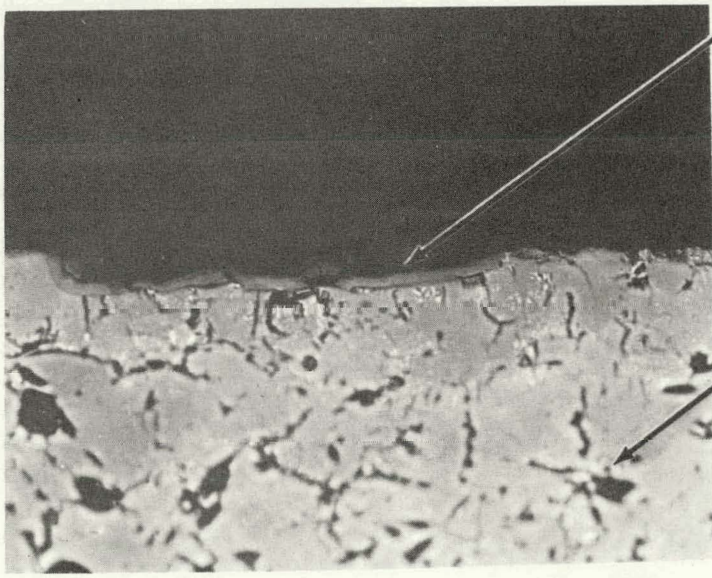

WTB - 006 1000X

The sputtered ZrO_2 ($+Y_2O_3$) was approximately 0.0001 inch thick and was fractured during metallographic specimen preparation.

Figure 15

ZrO_2 ($+Y_2O_3$) coating, 0.0001 in. thick.

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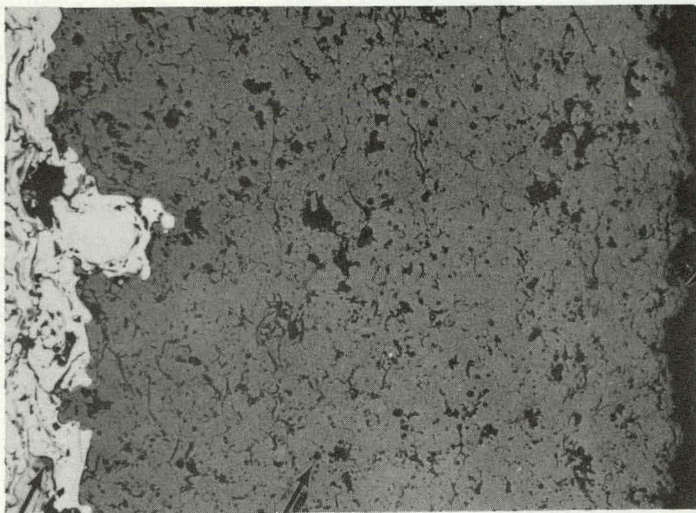

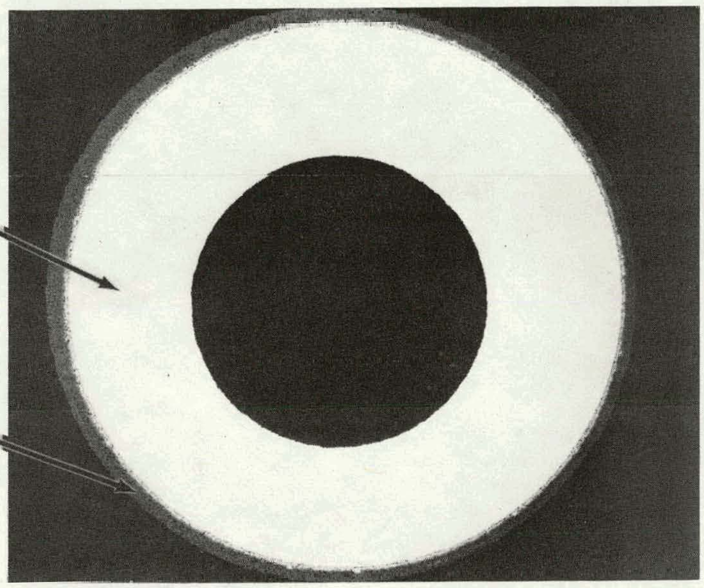

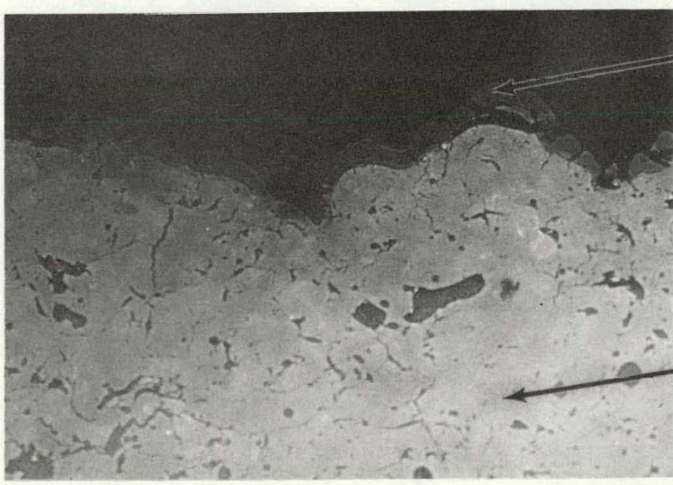

Figure 16

ZrO_2 $(+Y_2O_3)$ coating, 0.00024 in. thick.

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200X

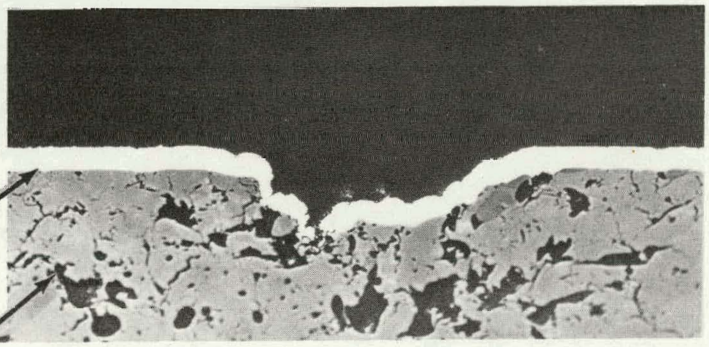
Plasma Sprayed ZrO_2 $(+Y_2O_3)$ surface not polished prior to Pt sputter coating. Surface too irregular for good Pt coverage.

200X

Sputtered Pt

Plasma Sprayed ZrO_2 $(+Y_2O_3)$ polished well enough to allow complete Pt coverage.

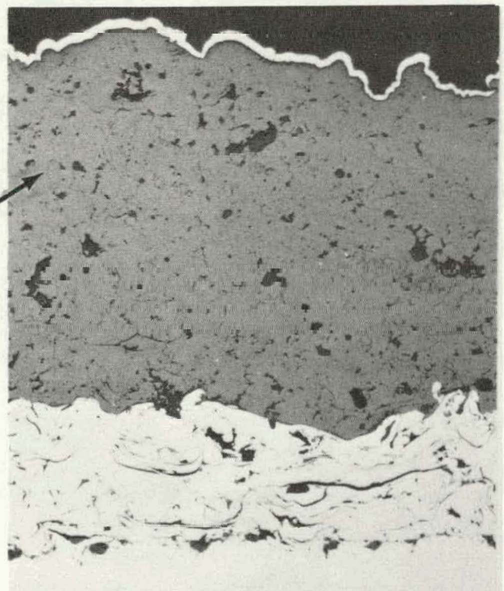
Plasma Sprayed NiCoCrAlY



WTB - 008 500X

Sputtered Pt Overlayer

Plasma Sprayed ZrO_2 $(+Y_2O_3)$ with polished surface. Central area was not polished well enough to allow coverage by Pt.



200X

Plasma Sprayed ZrO_2 $(+Y_2O_3)$ surface not polished but sufficiently regular for good Pt coverage.

Figure 17

ZrO_2 $(+Y_2O_3)$ coating on polished surface.

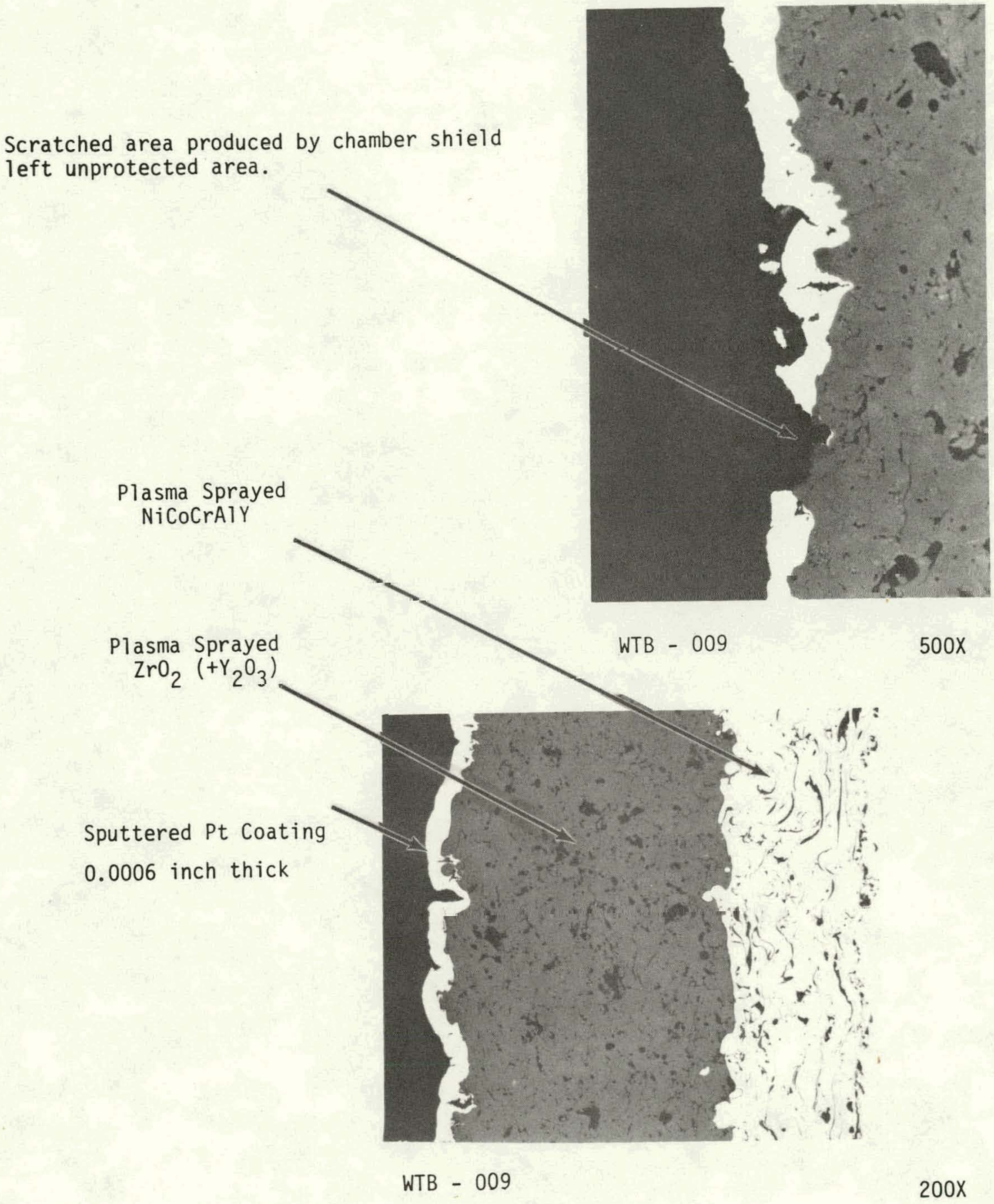


Figure 18

Pt coating 0.0006 in. thick.

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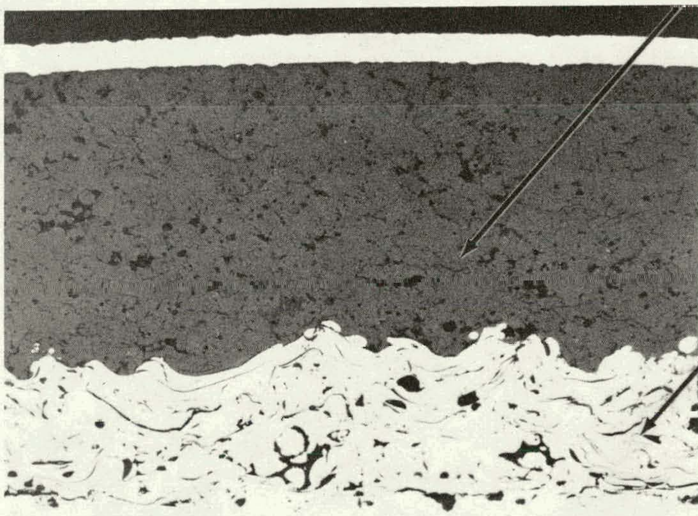

Sputtered coating completely sealed the polished ZrO_2 surface.

Figure 19

ZrO_2 ($+Y_2O_3$) graded to Pt.

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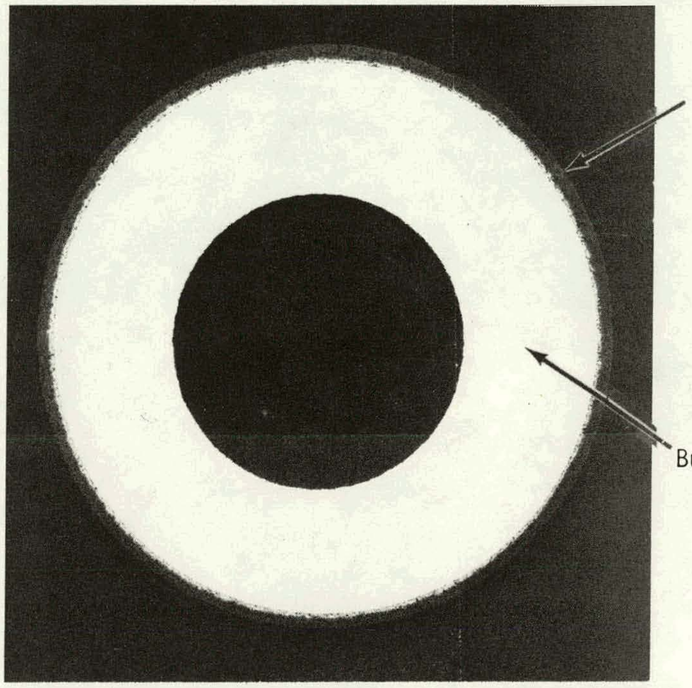

WTB - 011 8.3X

Figure 20
ZrO_2 ($+Y_2O_3$) graded to Pt_3 Al.

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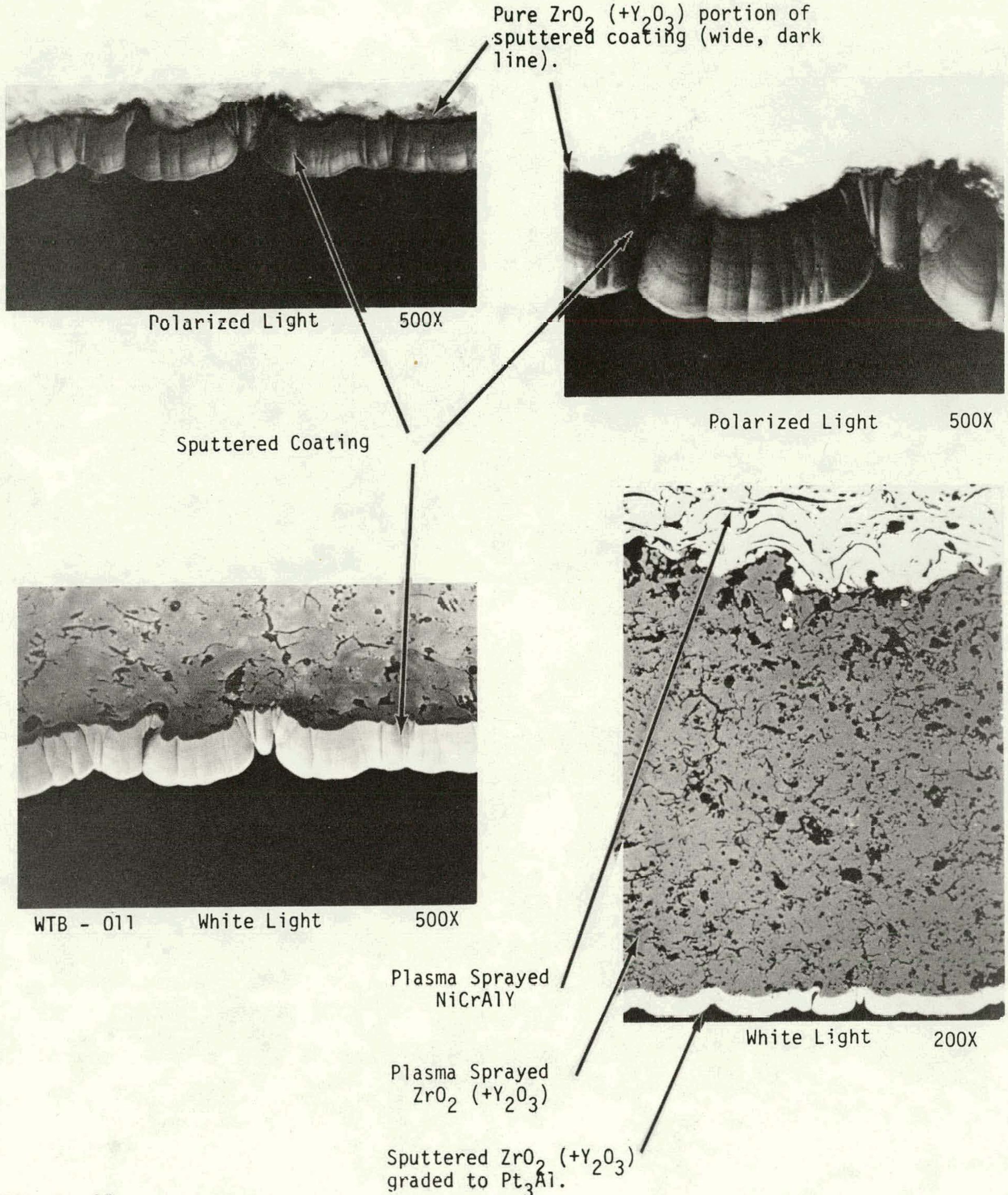

Figure 21

ZrO_2 ($+Y_2O_3$) graded to Pt_3 Al.

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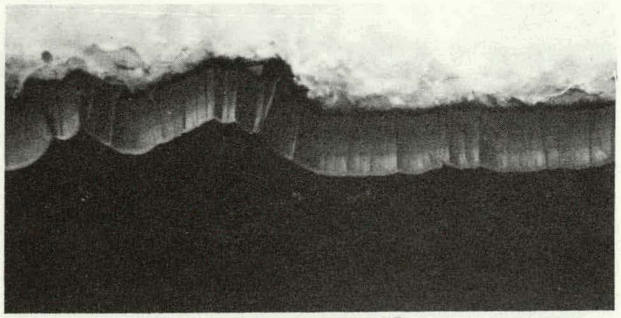
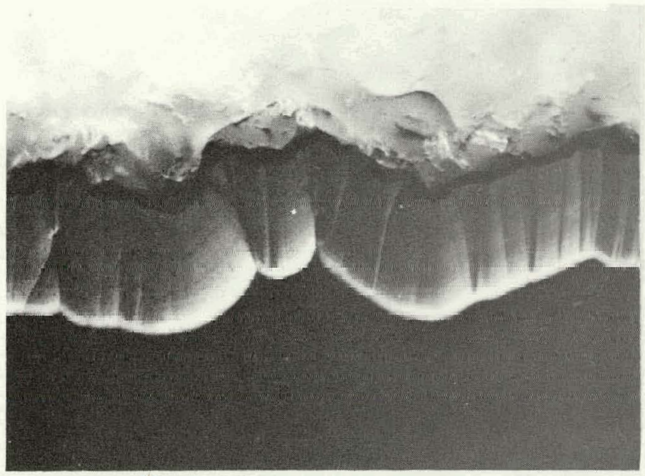
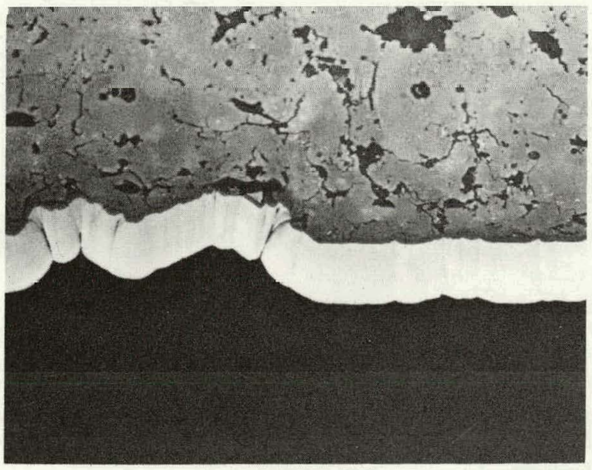


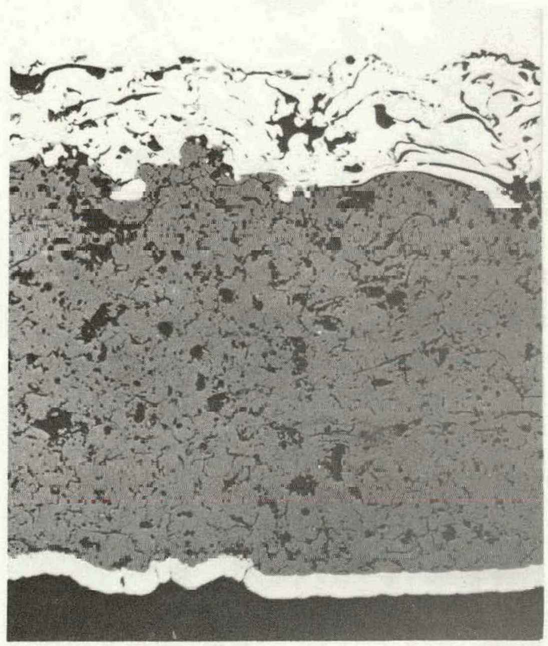
Polarized Light 500X



Polarized Light 1050X



WTB - 011 White Light 500X



White Light 200X

Figure 22
ZrO_2 (+Y_2O_3) graded to Pt_3 Al.

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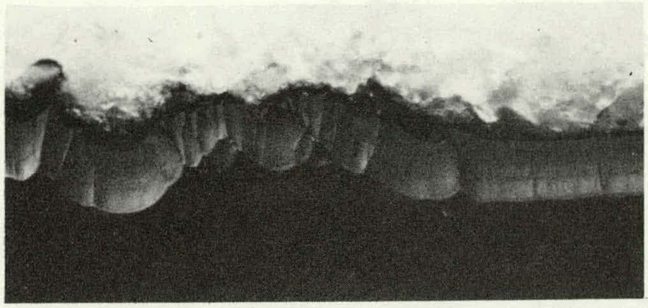
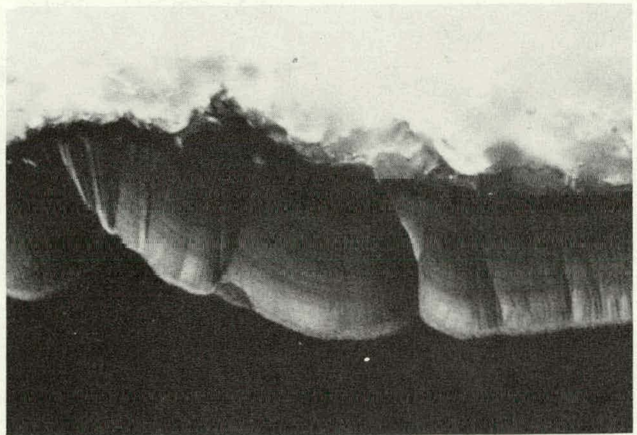
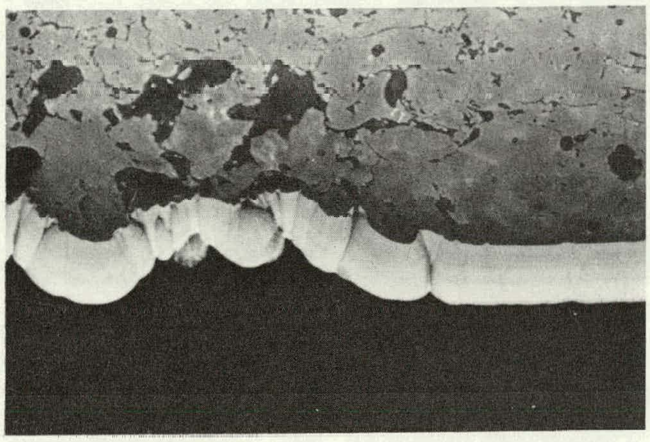


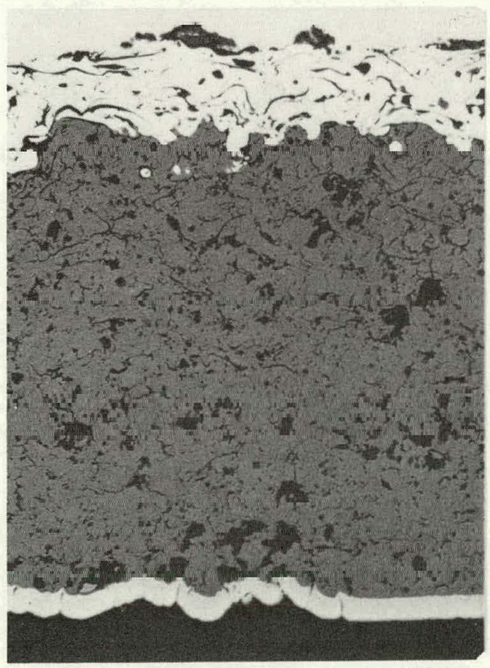
Polarized Light 500X



Polarized Light 1050X



WTB - 011 White Light 500X



White Light 200X

Figure 23

ZrO_2 ($+Y_2O_3$) graded to Pt_3Al.