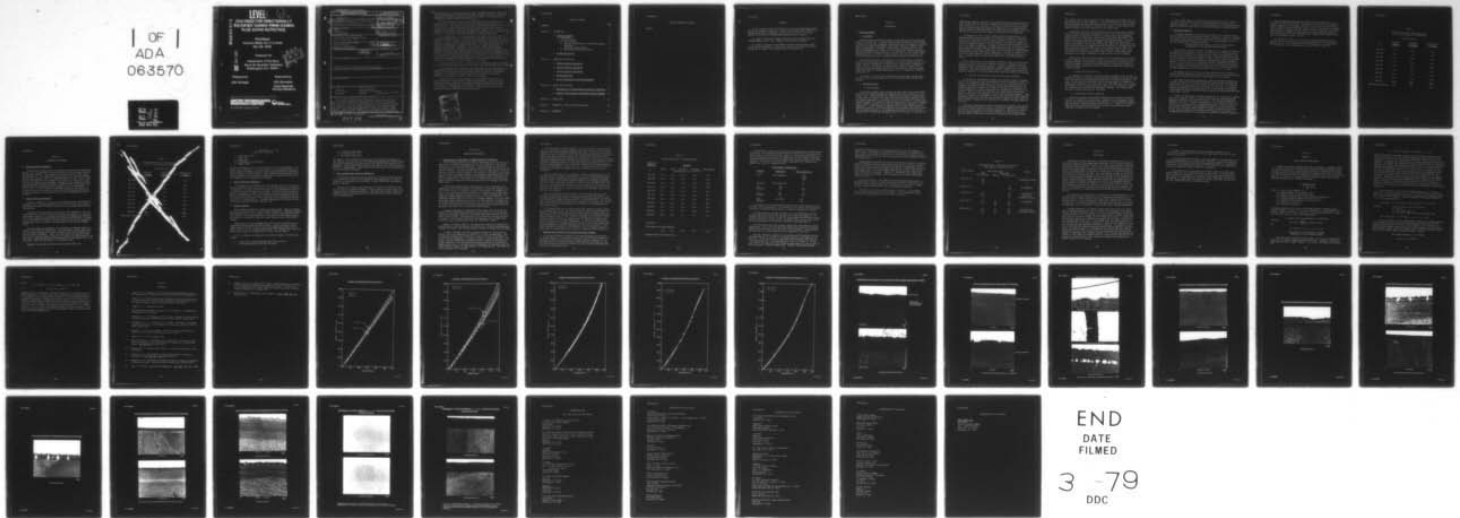


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**COATINGS FOR DIRECTIONALLY
SOLIDIFIED GAMMA PRIME-GAMMA
PLUS ALPHA EUTECTICS**

Final Report

Contract N000 19-77-C-0424

Oct. 22, 1978

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Prepared for

**Department of the Navy
Naval Air Systems Command
Washington, D.C. 20361**

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**Chief Materials
Process Research**

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the latter will not crack under thermal cycling. The approach taken in this study includes ~~a)~~ minimizing the differences in thermal expansion between the protective coating and the eutectic substrate by selective alloying of the coating and ~~b)~~ increasing the strain capacity of the coating.

In this study it was shown that the coefficient of thermal expansion of the beta phase (NiAl) closely matches that of the directionally solidified γ/γ' - α eutectic alloys. The eutectic alloy composition examined here was Ni-33 (wt. % Mo) - 5.5 (wt. %) Al. However, beta-rich coatings, overlay coatings with increased aluminum contents, are not diffusionally stable with respect to the γ/γ' - α eutectic substrate. At elevated temperatures the gamma in the substrate is converted to gamma prime with the formation of porosity resulting in coating delamination.

Chromium additions to the overlay coating sufficiently reduce the coefficient of thermal expansion such that the coating can be applied, but not enough for low temperature (1950°F) cycling. All of the chromium-modified coatings containing more than 18 w/o chromium were successfully tested at 2100°F, even though they, like the beta-rich coatings, were not diffusionally stable. At 1650°F the chromium-rich coatings were sulfidation resistant but invariably cracked during the low temperature cyclic test. The addition of platinum to the NiCoCrAlY composition markedly improved coating adherence, oxidation, and hot corrosion resistance. More important, no tensile cracks were observed indicating that the strain capacity of the coating modified by platinum is sufficient to accommodate cyclic thermal stresses. It is realized that improved performance is most likely the combined result of increased strain capacity and the reduction of tensile stresses since platinum also reduced the coefficient of thermal expansion of the coating.

The platinum modified overlay coatings are not diffusionally stable at 2100°F. At the elevated temperatures the coating markedly alters the substrate microstructure.

An intervening layer consisting of the phases gamma (γ) and gamma prime (γ') containing molybdenum, and to a lesser extent titanium, increased the diffusional stability of the coating without sacrificing mechanical stability. Intervening layers poor in molybdenum or titanium result in the formation of Kirkendall porosity which causes the coating to eventually delaminate.

It was noted in the cyclic oxidation studies at 2100°F that there was a marked tendency for the α -molybdenum fibers to spheroidize. It was also noted that the mechanical properties of coatings applied by the plasma spray process were superior to these applied by sputtering. These differences could be due to the presence of oxides within the coating or inherent pores which in effect reduce the average coefficient of thermal expansion.

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FOREWORD

The work reported herein was performed by the United Technologies Research Center, East Hartford, Connecticut, 06108, under Contract Number N00019-77-C-0424 during the period from August 1, 1977 to July 31, 1978. Mr. Irving Machlin of the Naval Air Systems ~~Command~~ acted as project engineer.

Mr. Norman S. Bornstein, Manager, Materials Process Research, UTRC, was Program Manager, and Dr. John G. Smeggil was Principal Investigator.

The author is pleased to acknowledge the help received from R. Hecht for the sputtered coatings, C. Hulse and B. Jacob for the mechanical property testing and A. Manzione for scanning electron microprobe data.

Section I.

INTRODUCTION

A. General Background1. Requirements

Advanced Naval aircraft gas turbines are required to exhibit improved performance and durability. To achieve higher turbine efficiency, the development of alloys with very high temperature capabilities from the standpoint of melting point, creep strength, fabricability, and surface stability (oxidation and/or corrosion resistance) is required. Directionally solidified eutectic alloys such as gamma/gamma prime - alpha, $\gamma/\gamma'-\alpha$ (Ni-31Mo-6.2Al), exhibit mechanical properties offering the potential of a 56 to 83°C (100 to 150°F) metal temperature advantage or a 50 percent strength increase over the best currently used nickel-base superalloys, such as directionally solidified Mar-M-200 plus Hf.

The oxidation/corrosion characteristics of the directionally solidified eutectics are sufficiently well documented to state that oxidation and hot corrosion resistant coatings will be required over a wide range of temperatures (Refs. 1 and 2). Accordingly, external airfoil surfaces, internal cooling channels, and platform sections will need to be protected. Root sections may or may not require a coating depending upon the $\gamma/\gamma'-\alpha$ composition ultimately chosen (Ref. 3).

This study is directed toward identifying and developing coating systems for $\gamma/\gamma'-\alpha$ alloys so such alloys can be used in advanced Naval aircraft gas turbines.

2. Coating Processesa. Pack Techniques

The earliest coatings used to extend the useful life of gas turbine superalloys were the "simple" aluminides, approximately 0.0015 to 0.003 inch (1.5 to 3 mils) thick. These aluminides are formed by either applying aluminum powder or paste onto the surface to be coated and subsequently heat treating the part at elevated temperatures or by packing the part to be coated in a canister containing aluminum, an accelerator (usually a halide salt), and an inert dispersant (used to prevent sintering of the pack) and likewise heat treating at elevated temperatures. In these processes aluminum reacts with and diffuses into the superalloy substrate. After the appropriate heat treatment, the resultant coating is composed primarily of the intermetallic phase NiAl (or CoAl) but also contains all of the substrate components present in the alloy. In general, the

elements and compounds insoluble in the aluminide phase precipitate during the heat treatment and can subsequently degrade the performance of these coatings under conditions of hot corrosion. It should also be noted that such hardware does not show any significant dimensional change due to the coating process. Because aluminum is diffused inwards, the major dimensional changes result from differences in specific volumes between the substrate and the aluminide.

To minimize the effect of alloy substrate upon coating morphology, the simple aluminide process was modified so coatings were formed by the outward diffusion of nickel rather than the inward diffusion of aluminum. This is accomplished by limiting the activity of aluminum in the pack--for example, by the use of prealloyed powders. Although the coating operation must now be carried out at higher temperatures for longer periods of time, the resultant coating is essentially single phase, saturated with respect to the alloying elements present in the alloy. Because this coating is formed by an outward diffusional process, slight dimensional changes occur. Although an outward, diffusionally-formed aluminide coating is desirable for corrosion problems, this kind of coating cannot be formed on many alloys due to Kirkendall porosity effects.

Recent modifications of the aluminide coatings are based on precious metal additions in which a thin film of platinum, palladium or rhodium is electro-deposited prior to the aluminizing steps. In general, compared to the former aluminides, these coatings have exhibited superior oxidation and hot corrosion resistance. Most recent studies suggest that such precious metal additions increase the adherence of the protective alumina scale which forms on the coating.

b. Physical Vapor Deposition (PVD) and Sputtering Techniques

On one hand for all diffusion coatings, the substrate becomes an integral part of the coating, and hence the composition of such a coating is in part controlled by the substrate composition. On the other hand, because all of the components of overlay coatings are applied by vapor deposition processes, and because diffusion is required only to bond the coating to the substrate, the initial composition of overlay coatings does not depend upon the substrate composition. Overlay coatings are based upon the system MCrAlY where M can be nickel, cobalt, iron or a combination of these transition metal elements. Static laboratory oxidation and sulfidation tests, as well as dynamic burner oxidation/corrosion/erosion tests which have been verified by engine tests, have shown that these coatings are among the most oxidation and sulfidation resistant materials available.

One method of applying the overlay coatings involves physical vapor deposition (PVD) processes in which the part to be coated is exposed to the vapors of a molten pool of the ingot of the desired composition. The process is conducted in a vacuum chamber where the pool is heated by an electron beam source. Some anticipated limitations of this process include (a) line of sight deposi-

tion patterns and (b) the composition of the coating partially depends upon the vapor pressure of the ingot components. In an alternate process used to deposit overlay coatings involving sputtering techniques, the atoms of a selected target are accelerated to the surface of the part to be coated. In this case the deposited coating has the same composition as the target ingot.

Conventional sputtering transfers material from the liquid state (evaporation) or from the solid state (sublimation) source. In the triode sputtering system, however, a tungsten wire, heated to a sufficiently high temperature, thermionically emits electrons. These electrons are weakly accelerated to the opposite anode by a small positive potential difference potential. In transit through the atmosphere between the tungsten wire filament and the positive anode, a percentage of the electrons collide with some of the argon atoms yielding two electrons from the original atom plus one electron. The argon ions near the target (the coating material source) are accelerated toward it by an impressed negative potential. Such argon ions impact the target surface, and a percentage of them eject atoms from the target. These ejected target atoms traverse the open region, condensing onto the substrate to form a coating.

The dc triode sputtering system, employed to coat specimens for this study, is used primarily to effect advanced oxidation-hot corrosion resistant and wear resistant coatings. Multi-element coatings are deposited at controlled rates from alloy targets onto heated rotating substrates. Complex alloy systems, including 10 element compositions, have been successfully deposited by such sputtering techniques.

c. Plasma Spraying Techniques

The deposition of coatings by plasma spraying techniques is not new (Ref. 4). However, the porosity inherent in such as-prepared coatings has hindered the large scale application of this potentially flexible and cost-effective procedure. Recently, it has been demonstrated that MCrAlY coatings applied by conventional plasma spray techniques, then treated such that a thin improvous skin is formed, and subsequently hot isostatically pressed (HIP'd) are as resistant to oxidation and corrosion as the sputtered and PVD applied coatings. This technique was also used to produce specimens for this study.

d. Miscellaneous Coating Techniques

Protective coatings for gas turbine applications can also be conceptually effected by a number of other techniques. Examples of such procedures include electro- and electro-less plating, electrophoretic, slurry dipping, and chemical vapor deposition (excluding the pack process) (Ref. 4). However, the successes shown by the pack and PVD techniques have largely eclipsed the use of these techniques to apply protective coatings.

Thus a large variety of coating procedures exist with which to deposit a coating onto directionally solidified gamma prime/gamma plus alpha ($\gamma/\gamma'-\alpha$) eutectic substrates. Accordingly, the problem addressed in coating gamma prime/gamma plus alpha eutectics does not involve developing new coating procedures.

B. Problem and Approach

The gamma prime/gamma plus alpha eutectic alloy ($\gamma/\gamma'-\alpha$) like all super-alloys currently used in gas turbines require a protective coating for increased hot corrosion and elevated temperature oxidation resistance (Refs. 5, 6, and 7). The overall coating problem as it concerns $\gamma/\gamma'-\alpha$ can be separated into two broadly disparate yet interconnected areas:

- coating chemistry and coating - substrate interdiffusion
- thermomechanical coating-substrate interactions.

The specific problem with respect to coating the $\gamma/\gamma'-\alpha$ family of alloys is not simply coating chemistry. Rather, the major obstacle involves mating the thermomechanical properties of the coating with those of the substrate so that the former will not crack under thermal cycling. Cracks developed in the protective coating are expected to result in severe substrate attack under the simultaneous exposure to the high temperature oxidizing/sulfidizing environment. Moreover, to be successful, the coating must exhibit diffusional stability and not, as a result of extensive interdiffusion, alter the mechanical properties of the substrate since the mechanical properties of $\gamma/\gamma'-\alpha$ eutectic alloys are strongly dependent upon eutectic alloy composition (Refs. 1, 2, and 8).

The approach taken in this study includes minimizing the differences in thermal expansion between the protective coating and the eutectic substrate and, by selective alloying, increasing the strain capacity of the coating.

Based upon the extensive oxidation-hot corrosion experience derived from in-house activities at the United Technologies Corporation and the thermal expansion data in Table I, the coating family of NiCoCrAlY based on the nominal composition Ni-23Co-18Cr-12Al-0.5Y was selected for this study. A coating of this general composition was identified for the $\gamma/\gamma'-\delta$ system, where moderate thermal expansion mismatch problems between substrate and coating have been identified (Refs. 5 and 6).

To alter the coefficient of thermal expansion of the coatings, the chromium level of the deposited NiCoAlY coating can be raised in an intervening layer immediately adjacent to the eutectic substrate. Then additional unmodified NiCoCrAlY ($15 \times 10^{-6} \text{ C}^{-1}$) would be deposited onto the chromium rich layer ($13 \times 10^{-6} \text{ C}^{-1}$) to form the protective environmental coating. The high Cr level in the NiCoCrAlY composition in the immediate vicinity of the $\gamma/\gamma'-\alpha$ substrate should lower the thermal expansion mismatch between the substrate and coating so that coating cracking due to thermal expansion mismatch is minimized. A thermomechanical analysis of this approach is discussed in detail in Appendix A.

The thermomechanical matching of the protective coating with the γ/γ' - α substrate can also be improved by increasing the strain to fracture properties (strain capacity) of the coating. This effect in the case of γ/γ' - δ has been shown to depend on coating chemistry (Refs. 5 and 6). The addition of platinum to the coating can modify the strain capacity of the coating since the precious metal reacts with aluminum from the NiCoCrAlY coating, creating a relatively ductile NiCoCrAlY zone at the coating-substrate interface which appears to retard crack propagation (Refs. 5 and 6).

Alternatively, either a γ' or a γ'/γ layer can be inserted between the eutectic and the outer NiCoCrAlY coating to "grade" differences in thermal expansion behavior. The coefficient of thermal expansion of γ' (Ni_3Al) with 8 wt. % Mo and 12.9 wt.% Cr is reported to be $14.4 \times 10^{-6} \text{ C}^{-1}$ (Ref. 9).

Table I
Average Coefficient of Thermal Expansion
for γ/γ' - α as a Function of Temperature

(Ni-31.5Mo-6.2Al)

<u>°C</u>	Transverse <u>Average</u> 10 ⁻⁶ °C ⁻¹	Longitudinal <u>Average</u> 10 ⁻⁶ °C ⁻¹	Diagonal (45°) <u>Average</u>
100- 200	11.2	8.2	9.6
200- 300	11.5	9.6	10.6
300- 400	12.0	10.5	11.2
400- 500	12.5	11.3	12.0
500- 600	12.0	11.4	12.0
600- 700	13.7	11.6	12.6
700- 800	16.0	12.3	14.8
800- 900	16.5	13.0	15.8
900-1000	<u>19.3</u>	<u>14.5</u>	<u>16.3</u>
Mean Value-(100-1000°C)	13.9	11.4	12.8

Section II.

EXPERIMENTAL PROCEDURES

A. Thermal Expansion Experiments

The rod-shaped specimens used to determine the coefficients of thermal expansion of the coating materials were fabricated by the hot isostatic pressing of appropriate powders. These powders were CoCrAlY, NiCoCrAlY, NiAl, Ni₃Al, and NiCoCrAlY + Pt. The same conditions were used for hot isostatic pressing all the samples--namely, 4 hours at 1975°F (1080°C) and 15 KSI. Prior to compaction, the powders were hot outgassed 930°F (500°C) to a vacuum of 1×10^{-6} torr and then sealed in pyrex ampoules. Subsequent to hot isostatic pressing, the specimens were annealed in hydrogen at 2200°F (1200°C) for 48 hours. The annealed specimens were machined into rectangular bars with dimensions 1" x 0.25" x 0.25". A THETA II Research dilatometer was used in all studies. An NBS platinum reference standard was used to provide thermal expansion data in an argon atmosphere in the temperature range from room temperature to 2000°F (1093°C).

B. Strain-to-Failure Experiments

The strain capacity of the alloys was determined from four point bend tests performed in air. Test bars measuring 1.25" long x 0.50" wide x 0.10" thick were machined from HIP'd cylindrical specimens. The crosshead speed was 0.020 inches/minute.

Two different testing machines were used for these experiments. For elevated temperature tests UTRC used its own ceramic test apparatus. In this facility loads are applied to the samples using 3/16 inch tungsten pins connected to 1-3/4 inch diameter molybdenum rams and molybdenum bend fixtures. The upper part of the fixture which applies the load to the test specimen is supported by a single pivot point to equalize the loads applied at the two upper loading points.

The room temperature experiments were performed on a modified 60,000 lb. capacity Tinius Olsen testing machine. Loads were applied through Inconel rams. The test fixture was made of 400 series stainless steel, and the loading pins were 3/16 inches in diameter. Again, the upper fixture was supported by and the loads were applied through a single pin to equalize the loads applied at the upper span. Cross-head deflections were measured by a deflectometer with a sensitivity of 0.005" of travel equal to 1" of machine chart.

Values for strain were calculated from the equation (Ref. 15):

Table I

Average Coefficient of Thermal Expansion
for γ/γ' - α as a Function of Temperature

(Ni-31.5Mo-6.2Al)

	<u>Transverse Average</u> $10^{-6} \text{ } ^\circ\text{C}^{-1}$	<u>Longitudinal Average</u> $10^{-6} \text{ } ^\circ\text{C}^{-1}$	<u>Diagonal (45°) Average</u>
100- 200	11.2		9.6
200- 300	11.5	9.6	10.6
300- 400	12.0	10.5	11.2
400- 500		11.3	12.0
500- 600	12.0	11.4	12.0
600- 700	13.0	11.6	12.6
700- 800		12.3	14.8
800- 900	16.5	13.0	15.8
900-1000	<u>19.3</u>	15.5	<u>16.3</u>
Mean Value-(100-1000)	13.9	11.5	12.8

$$\epsilon = \frac{2h (n + 2)}{(L - A) L + A (n + 1)} (\gamma_L)$$

- ϵ = outer fiber strain
 L = lower span
 A = upper span
 γ_L = deflection at loading points
 h = sample height

For this calculation a value of n was assumed to be one, corresponding to completely viscous behavior. A continuous record of the crosshead motion was obtained during the deformation using a linear voltage displacement transformer mounted externally from the furnace. Prior calibration runs were made at different temperatures to determine the corrections to be applied to the data for the compliance of the loading rams and fixtures.

C. Eutectic Substrate Preparation

All specimens were fabricated from 1-1/4" diameter bars of γ/γ' - α with the nominal composition Ni-33 (wt.%) Mo-5.5 (wt.%) Al, directionally solidified at 1.5 cm/hour. No cellular microstructural features were subsequently observed in a longitudinal stripe polished into the ingot. The ingot was then centerless ground to a diameter of 1-1/8". This step was included to remove from the surface of the ingot ceramic inclusions introduced by the solidification crucible. The presence of gross amounts of such inclusions would be expected to deleteriously affect coating adherence. Specimens approximately 0.2 cm thick were then cut from the ingot and polished to 600 grit SiC.

D. Coating Deposition

The coatings were applied in triode sputtering systems. Based on standardization runs, time was used to control deposit thicknesses. Supplemental targets were cosputtered with the main target to add the elements platinum, molybdenum, and titanium to the coating composition. The amount of these elements added to the deposit was controlled through the voltage impressed upon the appropriate targets.

Gamma (with 5 wt.% Al) and gamma prime (with 14 wt.% Al) layers were deposited from hollow cathode sputtering targets. Chemical analysis of these ingots indicate 4.9 and 5.0 wt.% Al for the two gamma nickel targets and 13.3 and 13.4 wt.% Al for the two gamma prime targets. The specimens were slowly rotated (2 RPM) during the coating process to insure an even deposit.

Prior to coating, the substrates were cleaned according to the following scheme:

1. a light blast of trichloroethane vapor (~80 psi at 6"),
2. a chlorinated abrasive scrub with hot water,

3. a cold water wash/rinse,
4. an ethanol rinse, and
5. dry with an argon blast.

For support a 30 mil diameter platinum wire was attached to the substrate by tack welding. The power used for this welding was approximately 95 watt-seconds. The vacuum systems were all pumped down into the low 10^{-7} torr range prior to deposition. After deposition all specimens were heated for four hours at 1975°F (1079°C) in flowing argon. Compositions of sputtered deposits were determined by electron microprobe techniques.

E. Cyclic Oxidation/Hot Corrosion Experiments

The oxidation test involved cyclic exposure to 2100°F (1150°C) for 55 minutes in still air followed by five minutes at room temperature. The time required for the specimens to go from 2100°F to about 210°F (100°C) was approximately 15 seconds.

The cyclic hot corrosion test consisted of 55 minutes at 900°C and 5 minutes at 200°C in a stagnant atmosphere. About 1 minute is required to move the specimen across the temperature gradient. Specimens were initially coated with 1 mg/cm² of Na₂SO₄, then removed every 24 hours and recoated with a fresh deposit of sodium sulfate.

Section III.

RESULTS AND DISCUSSION

A. Modification of Coefficient of Thermal Expansion of NiCoCrAlY

The base coating, NiCoCrAlY, of nominal composition Ni-23Co-18Cr-12Al-0.5Y, could not be successfully sputtered onto the γ/γ' - α substrates. The coating spalled as it cooled to room temperature. Coating spallation is attributed to the differences in coefficients of thermal expansion between the overlay and the eutectic substrate. The only exception was a series of experiments in which the coating of nominal NiCoCrAlY composition was applied by the plasma spray process onto the face of pie shaped specimens. Metallographic examination revealed the presence of extraneous phases, which were metallographically identified as oxides or inherent pores. The presence of such oxides within the coating would lower the average coefficient of thermal expansion of the coating, a desirable effect here.

To successfully apply overlay coatings onto the γ/γ' - α substrates, the composition of the base coating had to be modified. The thermal expansion data for NiAl (β), Ni₃Al (γ'), CoCrAlY, NiCoCrAlY, and NiCoCrAlY + Pt measured as part of this study are presented in Figures 1-5. Moreover, the average coefficients of thermal expansion (referenced to 0°C) in 100°C increments over the temperature ranges examined are listed in Table II in units of 10^{-6}C^{-1} . It is immediately apparent that the coefficient of thermal expansion of the beta phase (NiAl) most closely matches that of the γ/γ' - α alloy. Therefore, coatings rich in the beta phase are expected to perform well. Moreover, it is observed that the addition of platinum to the NiCoCrAlY composition substantially lowered the expansion coefficient over the temperature range examined.

A coating consisting of essentially the beta phase (Ni-4.1Cr-20.4Al) was applied onto the directionally solidified eutectic alloy, and the coated substrate was successfully exposed for one hundred eighty-nine thermal cycles from 2100°F without failure. The microstructure of the coating after test is shown in Figure 6. The successful application and completion of the thermal shock tests was in part anticipated based upon the dilatometric studies.

However, as shown in Figure 6, the coating has significantly modified the microstructure of the directionally solidified alloy. Based upon metallographic analyses, the phases at the coating-substrate interface are gamma prime and alpha.

In order to minimize the activity gradients between the coating and the eutectic alloy, the composition was modified to include the gamma phase. Thus coatings were prepared which consisted of a mixture of beta (β) and gamma (γ). In two series of experiments, the volume fraction of the beta phase was, as compared to the base composition, increased in order to reduce the coefficient of thermal expansion of the coating while maintaining chromium at the nominal 18 weight percent level. In a third series of experiments the chromium content was reduced from the nominal 18 weight percent to 14 weight percent. The results of these experiments are as follows:

With the aluminum content increased to 15 or 16.6 percent, the volume fraction of the beta phase was also increased, and it was observed that the coating remained intact and successfully completed 189 hours of cyclic tests at 2100°F. However, as shown in Figures 7A and 7B, the coatings were again not diffusively stable with aluminum readily diffusing into and modifying the substrate alloy. When the chromium content was reduced from the nominal 18 w/o to 14 w/o, the coating readily delaminated when cyclically tested at 2100°F. The delaminated coating is shown in Figures 8A and 8B. In this case failure is believed to be due to both the stresses imposed by the differences in coefficient of thermal expansion as well as the formation of voids, Figure 8C. Voids are formed as a result of preferential diffusion of aluminum and vacancy coalescence at the prior β - γ boundaries.

When the weight fraction of chromium (at constant aluminum content) is increased, adherent coatings are again formed. In fact, the coatings containing more than 18 w/o chromium were successfully applied and completed 189 thermal cycles at 2100°F. Representative photomicrographs of the coatings containing 13.6Al-27Cr and 14.3Al-26.5Cr are shown in Figures 9A and 9B respectively. However, it is again immediately apparent that, as a result of the large activity gradients, the composition of the coating and the substrate alloy are modified. Note that void formation as a result of coalescence of vacancies at the β - γ interface is not observed.

Rapid interdiffusion between candidate coating systems and the eutectic substrate simultaneously effects both deleterious and beneficial results. On one hand, interdiffusion between the coating and substrate would tend to smooth out differences in thermal expansion behavior, a beneficial effect. On the other hand, such interdiffusion would also effect changes in the eutectic alloy microstructure and cause coating delamination resulting from void formation, both undesirable effects.

All coatings based upon the modified NiCoCrAlY composition cracked during the sulfidation test cycle. The least sulfidation resistant coatings were the family of coatings containing less than 14 weight percent chromium, Figure 10, and in general, the hot corrosion resistance increased with increasing chromium content, Figures 11 and 12. The reason that cracking occurred only in the hot corrosion cycle and not in the oxidation cycle is related to the more rapid rates of diffusion at the higher temperatures. In the hotter oxidation cycle, the phase formed at the coating substrate interface is the ductile gamma prime which accommodates the tensile loads applied by the cyclic stresses. At the lower temperatures employed in the sulfidation cycle, the diffusion rates are slower, and the coating exhibits the typical β - γ structure shown in Figure 12.

B. Improved Strain Capacity of the Modified NiCoCrAlY Coatings

As previously noted, the addition of platinum decreases the coefficient of thermal expansion of the overlay coatings, thereby reducing the magnitude of the stresses arising from differences in thermal expansion behavior. But as discussed earlier, the phase which most closely matches the substrate is beta (β), and even the beta-rich or essentially all beta coatings cracked when subjected to the sulfidation cycle.

Table II

Average Coefficient of Thermal Expansion

<u>Temperature Interval</u> °C	<u>Substrate</u>				
	Ni ₃ Al*	NiAl*	CoCrAlY*	NiCoCrAlY	NiCoCrAlY+Pt
	(expressed in units of 10 ⁻⁶ °C ⁻¹)				
100- 200	11.3	11.7	10.7	10.5	9.3
200- 300	12.2	12.8	12.0	12.4	11.2
300- 400	12.9	13.2	12.6	13.1	11.8
400- 500	13.3	13.6	13.2	14.0	12.4
500- 600	13.7	13.9	13.5	14.5	13.0
600- 700	14.0	14.0	14.2	15.4	13.6
700- 800	14.3	14.0	14.8	16.3	14.4
800- 900	14.5	14.1	15.5	17.5	15.5
900-1000	14.8	14.5	16.1	18.1	16.7
1000-1100	15.3	14.4	17.4	18.6	17.5
1100-1200	15.8	14.4	---	---	---
Mean Value					
Over Temperature Range Measured:					
	13.8	13.7	14.0	15.1	13.5

* Averaged values for multiple data.

The addition of platinum to the NiCoCrAlY coating markedly affected coating performance. The platinum-containing NiCoCrAlY coatings did not crack when exposed in the cyclic sulfidation test, Figure 13. Based upon metallographic studies, the platinum interacted with the constituents of the coating to form what was metallographically identified as gamma prime, the phase noted for its strain capacity. For example, the strain capacity measurements for NiCoCrAlY and gamma prime, Ni₃Al, are summarized below.

Strain Capacity Measurements

<u>Material</u>	<u>Temperature</u>	<u>Strain Capacity, %</u>
Ni ₃ Al	Room Temperature	2.06
		1.96
		1.88
NiCoCrAlY	Room Temperature	0.6
Ni ₃ Al	700°C	1.4
NiCoCrAlY	750	2.06
Ni ₃ Al	800	1.6
NiCoCrAlY	800	5.9
Ni ₃ Al	1000-1100	>10
NiCoCrAlY		>10

Even though the platinum-containing specimen could not be tested due to the difficulty in the processing of a bulk specimen, it is evident from the thermal tests that the objective--the formation of crack-free coating--was realized.

The high temperature thermal stability of the platinum-modified overlay coating, like all of the previous coatings was at best, poor, Figure 14. At elevated temperatures during the oxidation cycle, marked interdiffusion occurred between the substrate and the coating. To minimize these effects, the role of the intervening layer was modified to perform as a diffusion barrier.

The intervening layers were formed by sequentially sputtering the desired compositions. In all cases the last layer applied was the protective platinum modified NiCoCrAlY (31Ni-7.1Co-6.8Cr-8.1Al-0.16Y and 46.6Pt). The composition and thickness of the intervening layers are summarized in Table III.

The high temperature stability of the platinum modified NiCoCrAlY coating separated from the γ/γ' - α matrix by intervening layers is also summarized in Table III. In the first series of experiments the intervening layer consisted of the gamma phase which is thermodynamically stable with respect to the substrate and is the phase which forms during prolonged exposure of the uncoated eutectic substrate at elevated temperatures. However, the platinum modified

NiCoCrAlY-coating applied over γ layers spalled during the high temperature cyclic tests. The failure is associated with the formation of Kirkendall porosity, which is the result of differences in the diffusion of elements between the coating and the substrate, with the resulting vacancies coalescing within the intervening layer.

Although marked interdiffusion between the modified NiCoCrAlYPt coating and eutectic substrate occurred, no extended porosity was noted; hence no spallation. In the case of the intervening layers composed of gamma/gamma prime compositions, the observation of Kirkendall porosity was related to the thickness of the gamma layer. The thinner gamma layers quickly saturated limiting the deleterious effect of porosity. Complex γ/γ' intervening layer compositions were successfully tested under cyclic oxidation and hot corrosion conditions, Figures 15 and 16.

Titanium partitions to the gamma prime whereas molybdenum partitions to the gamma phase. In fact, as shown in the last two series of experiments, the addition of molybdenum alleviated the Kirkendall effect independent of gamma thickness whereas titanium was at best only marginally effective.

Additionally, in cyclic oxidation studies at 2100°F, the α -molybdenum fibers showed a marked tendency to spheroidize. No spheroidization effects were detected in specimens tested under 1650°F conditions.

Table III

Intervening Layers - Description and Results
(High Temperature Oxidation Test)

Outer Coating	Intervening Layers			Results
	Gamma	Modifier	Gamma Prime	
	Thickness mils		Thickness mils	
NiCoCrAlY + Pt	0.3			Kirkendall porosity
	0.4			"
	0.6			"
NiCoCrAlY + Pt			0.25	No spallation
			0.5	"
NiCoCrAlY + Pt	0.3		0.5	No spallation
	0.4		0.5	Spallation
	0.6		0.5	Kirkendall porosity
NiCoCrAlY + Pt	0.4	Mo	0.5	No spallation
	0.6	Mo	0.5	"
		Mo	0.5	"
NiCoCrAlY + Pt	0.3	Ti	0.5	No spallation
	0.4	Ti	0.5	Kirkendall porosity
	0.6	Ti	0.5	"

Section IV.

CONCLUSIONS

The objective of this study was to determine the composition of a coating for directionally solidified gamma-gamma prime-alpha ($\gamma/\gamma'-\alpha$) eutectic alloys of the nominal composition Ni-35 w/o Mo-5.5 w/o Al which would protect the substrate from oxidation and sulfidation. The specific problem addressed here involves the anisotropic thermal expansion behavior of the directionally solidified eutectic alloy. Specifically many of the overlay coatings such as Ni-23Co-18Cr-12Al-0.5Y would afford protection to the alloy. But, upon thermal cycling, the differences in coefficients of thermal expansion along the longitudinal direction would result in tensile stresses sufficient to crack the coating. In the transverse direction, the differences in thermal expansion coefficients are minimal and cracking as a result of thermal cycling is not a problem. The approaches employed here consisted of modifying the composition of the overlay coating to (a) minimize thermal stresses and (b) increase the strain capacity of the coating. The use of an intervening layer between the overlay coating and the substrate could grade the differences in thermal expansion behavior and perform as a diffusion barrier.

In this study it was shown that the coefficient of thermal expansion of the beta phase (NiAl) closely matches that of the directionally solidified $\gamma/\gamma'-\alpha$ eutectic alloys. However, beta-rich coatings, overlay coatings with increased aluminum contents, are not diffusionally stable with respect to the $\gamma/\gamma'-\alpha$ eutectic substrate. At elevated temperatures the gamma in the substrate is converted to gamma prime with the formation of porosity resulting in coating delamination.

Chromium additions to the overlay coating sufficiently reduce the coefficient of thermal expansion such that the coating can be applied, but not enough for low temperature (1950°F) cycling. All of the chromium-modified coatings containing more than 18 w/o chromium were successfully tested at 2100°F, even though they, like the beta-rich coatings, were not diffusionally stable. At 1650°F the chromium-rich coatings were sulfidation resistant, but invariably cracked during the low temperature cyclic test. It is concluded that the chromium content of the coating cannot be sufficiently increased to obtain both low temperature crack free coatings and high temperature diffusional stability. The addition of platinum to the NiCoCrAlY composition markedly improved coating adherence, oxidation, and hot corrosion resistance. More important, no tensile cracks were observed indicating that the strain capacity of the coating modified by platinum is sufficient to accommodate cyclic thermal stresses. It is realized that improved performance is most likely the combined result of increased strain capacity and the reduction of tensile stresses since platinum also reduced the coefficient of thermal expansion of the coating.

Although the platinum modified overlay coatings are among the most diffusionally stable coatings at 2100°F, long time exposure did alter the substrate microstructure.

An intervening layer consisting of the phases gamma (γ) and gamma prime (γ') containing molybdenum, and to a lesser extent titanium, increased the diffusional stability of the coating without sacrificing mechanical stability. Intervening layers poor in molybdenum or titanium result in the formation of Kirkendall porosity which causes the coating to eventually delaminate.

It was noted in the cyclic oxidation studies at 2100°F that there was a marked tendency for the α -molybdenum fibers to spheroidize. It was also noted that the mechanical properties of coatings applied by the plasma spray process were superior to these applied by sputtering. These differences could be due to the presence of oxides within the coating or inherent pores which in effect reduce the average coefficient of thermal expansion.

Section V.

APPENDIX A

NORMAL STRESS CONSIDERATIONS

Although no rigorous treatment exists for the problem of cracking of protective coatings resulting from coating-substrate thermal expansion mismatch, there are several treatments all yielding similar results for calculating the stresses in growing oxides resulting from oxide-substrate mismatch (Refs. 10, 11, 12, and 13). For the purpose at hand the treatment by Douglass (Ref. 12) and Oxx (Ref. 13) modified for the case of a coating-substrate interaction rather than an oxide-substrate interaction will suffice, accordingly:

$$\sigma_c = \frac{E_c \Delta T (\alpha_c - \alpha_s)}{1 + \frac{2E_c}{E_s} (t_c/t_s)}$$

where σ_c = stress in the coating, N/m^2

E_c = elastic modulus of the coating, N/m^2

E_s = elastic modulus of the substrate, N/m^2

ΔT = difference between high and low temperature expansion, $^{\circ}C$

α_c = coating thermal expansion coefficient over ΔT

α_s = substrate thermal expansion coefficient over ΔT

t_c = thickness of coating, cm

t_s = thickness of metal, cm

The primary assumption here is that only thermal expansion mismatches effect coating cracking. Thus for a Ni-Co-Cr-Al-Y composition t_c is approximately 126 microns, a substrate thickness t_s such that t_s is much greater than t_c , (except for thin walls) and, to a first order approximation

E_s equals E_c and is approximately $21 \times 10^{10} N/m^2$;

therefore,

the term $1 + \frac{2E_c}{E_s} (\frac{t_c}{t_s})$ is equal to unity

and

$$\sigma_c = E_c \Delta T (\alpha_c - \alpha_s) \dots \dots \dots 1a$$

Calculation of the Coefficient of Thermal
Expansion for the NiCoCrAlY Coatings

The directionally solidified eutectic alloy, $\gamma/\gamma'-\delta$, has been successfully coated with the overlay composition Ni-18Cr-12Al-0.3Y. Assuming a temperature differential of $1065^{\circ}C$, $1090^{\circ}C$ ($2000^{\circ}F$) - $25^{\circ}C$ ($77^{\circ}F$), the stress in the coating is:

$$\sigma_c = -21 \times 10^{10} (1065)(\alpha_c - \alpha_s) \text{ N/m}^2 \dots\dots\dots \text{ lb}$$

The mean coefficient of thermal expansion for the transverse direction of the D.S. eutectic alloy $\gamma/\gamma'-\delta$ over the above temperature range is 14×10^{-6} and since $\gamma/\gamma'-\delta$ has been coated successfully with NiCrAlY (Ni-18Cr-12Al-0.3Y), the resistance to cracking of a similar NiCoCrAlY coating composition should be acceptable. Accordingly, the differential thermal stresses between the coating and substrate will involve the longitudinal eutectic direction. The mean thermal coefficients of expansion between 25°C and 1090°C for the $\gamma'/\gamma-\alpha$ alloy are 12×10^{-6} for the longitudinal direction and 15×10^{-6} for the transverse direction. The value of the thermal coefficient of expansion for the longitudinal surface of (12×10^{-6}) is below similar values for all the segregate phases in the NiCoCrAlY system, (Ref. 14), and is due to the large fraction of α -Mo fibers in the alloy. Therefore, NiCoCrAlY coatings would be in tension for all compositions on the $\gamma/\gamma'-\alpha$ eutectic upon cooling from 1090°C (2000°F) to 25°C (77°F). However, although an ideal situation would be one in which the coating is in mechanical compression upon such thermal cycling, the real world may tolerate a certain tensile loading of the coating without its cracking under thermal cycling conditions.

The key here lies in the successful NiCrAlY coating of $\gamma/\gamma'-\delta$ with a coefficient of thermal expansion of $14 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ (Refs. 5 and 6) and the resistance of this coating-substrate composite to cracking over the temperature range 25°C - 1090°C (77°F - 2000°F). The stress applied to the NiCrAlY coating on $\gamma/\gamma'-\delta$ can be approximated by:

$$\sigma(\text{NiCrAlY}) = -21 \times 10^{10} \text{ N/m}^2 (1065^\circ\text{C})(14 \times 10^{-6} - 18 \times 10^{-6})^\circ\text{C}^{-1}$$

assuming $E(\text{NiCrAlY})$ equal to $21 \times 10^{10} \text{ N/m}^2$ and
 $\Delta T = 1065^\circ\text{C}$
 $\alpha_s = 14 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ (Ref. 6)
 $\alpha_c = 18 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ (Ref. 6)(the worst possible case)

$$\text{then } \sigma(\text{NiCrAlY}) = -9 \times 10^8 \text{ N/m}^2 \dots\dots\dots (2)$$

If this is assumed to be the maximum tensile stress that a NiCrAlY coating can withstand without cracking under thermal cycling between 25°C and 1090°C (77°F and 2000°F), then for the case of the NiCoCrAlY - $\gamma/\gamma'-\alpha$ eutectic, the maximum thermal expansion coefficient for the coating without the tensile stresses in thermal cycling cracking the coating can be calculated assuming NiCoCrAlY and NiCrAlY to exhibit similar mechanical behavior. Thusly

$$\text{with } \sigma(\text{NiCoCrAlY})_{\text{max.}} = -9 \times 10^8 \text{ N/m}^2$$

$$\text{and } \alpha_s = 12 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$$

we find

$$-9 \times 10^8 \text{ N/m}^2 = 21 \times 10^{10} (-1065)(\alpha_c - 12 \times 10^{-6}) \text{ N/m}^2$$

or

$$(\alpha_c) \text{ max.} = 16 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$$

Thus because of the coefficient of thermal expansion of Cr is below that of $16 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ —namely, $13 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ (Ref. 14), then NiCoCrAlY coatings enriched in α -Cr primarily at the coating-substrate interface could lower thermal expansion coefficient mismatches. Although such coating would still be in tension upon thermal cycling, critical tensile stress levels leading to coating cracking would not develop in the coating. The α -Cr content could be enriched in the coating near the eutectic interface by depositing an intervening layer of α -Cr enriched NiCoCrAlY. The α -Cr content away from the coating could then be graded to lower levels near the coating-gas interface where presumably higher Al levels would be found for oxidation resistance.

Section IV.

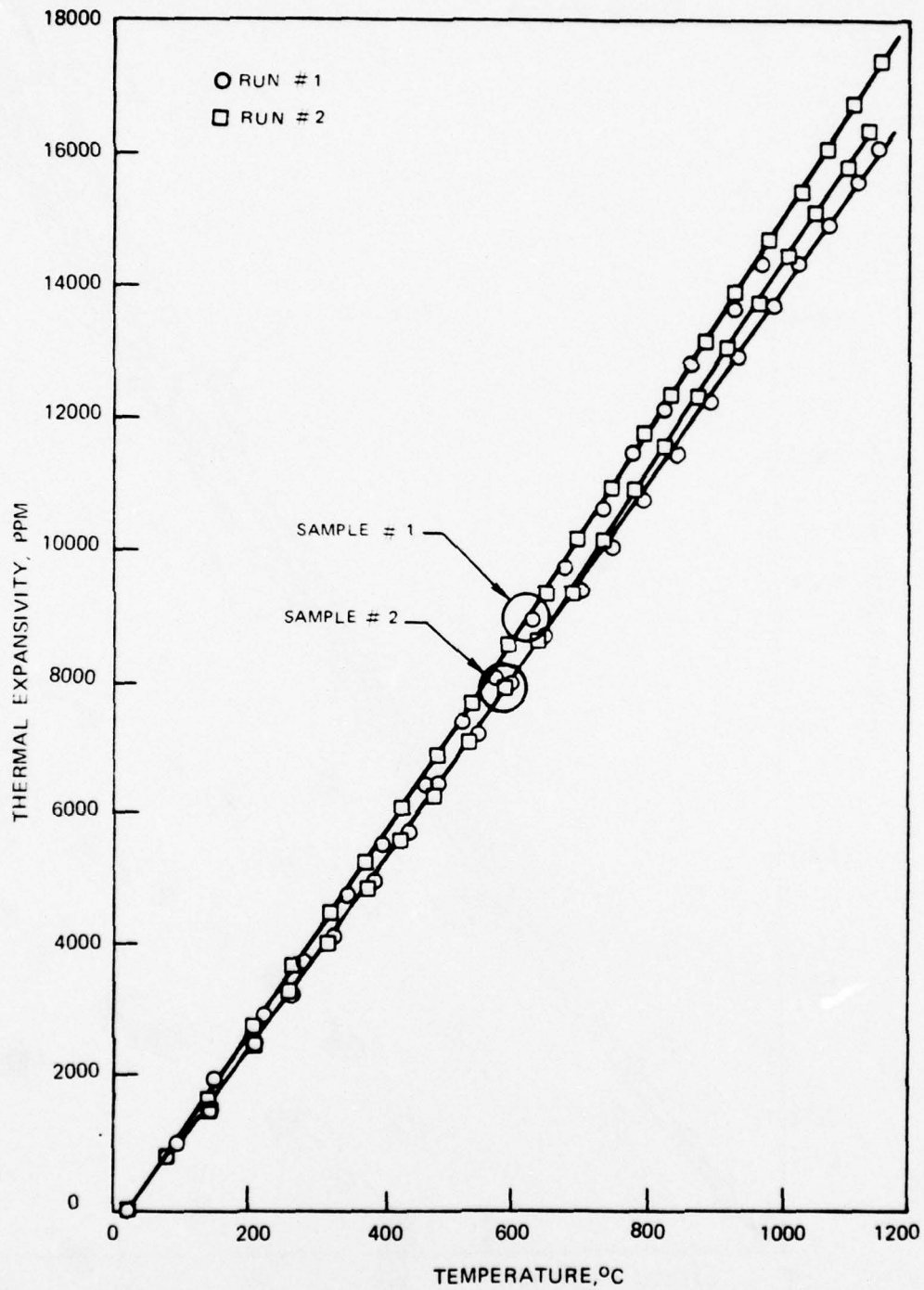
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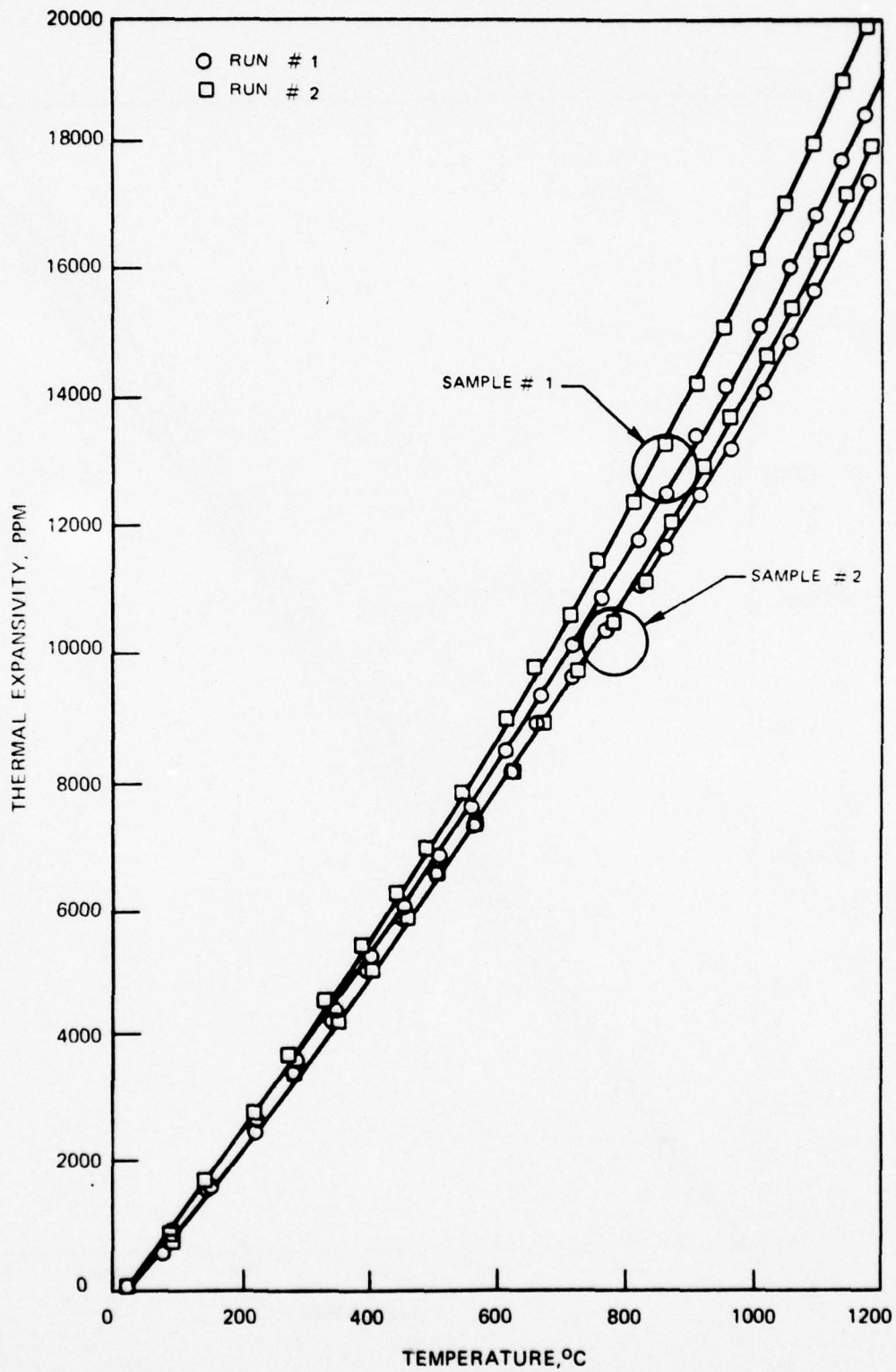
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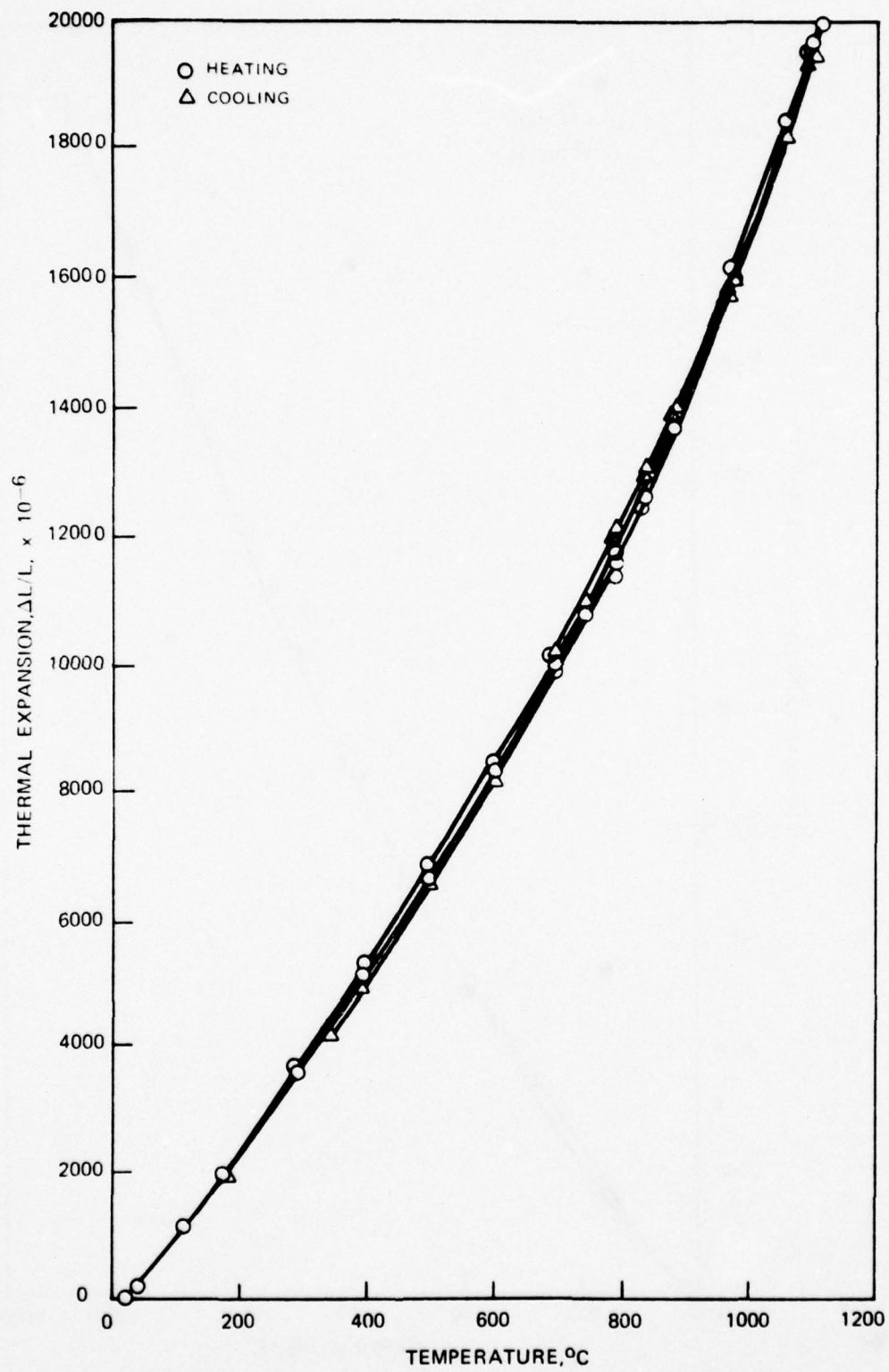
THERMAL EXPANSION BEHAVIOR OF NiAl(β)



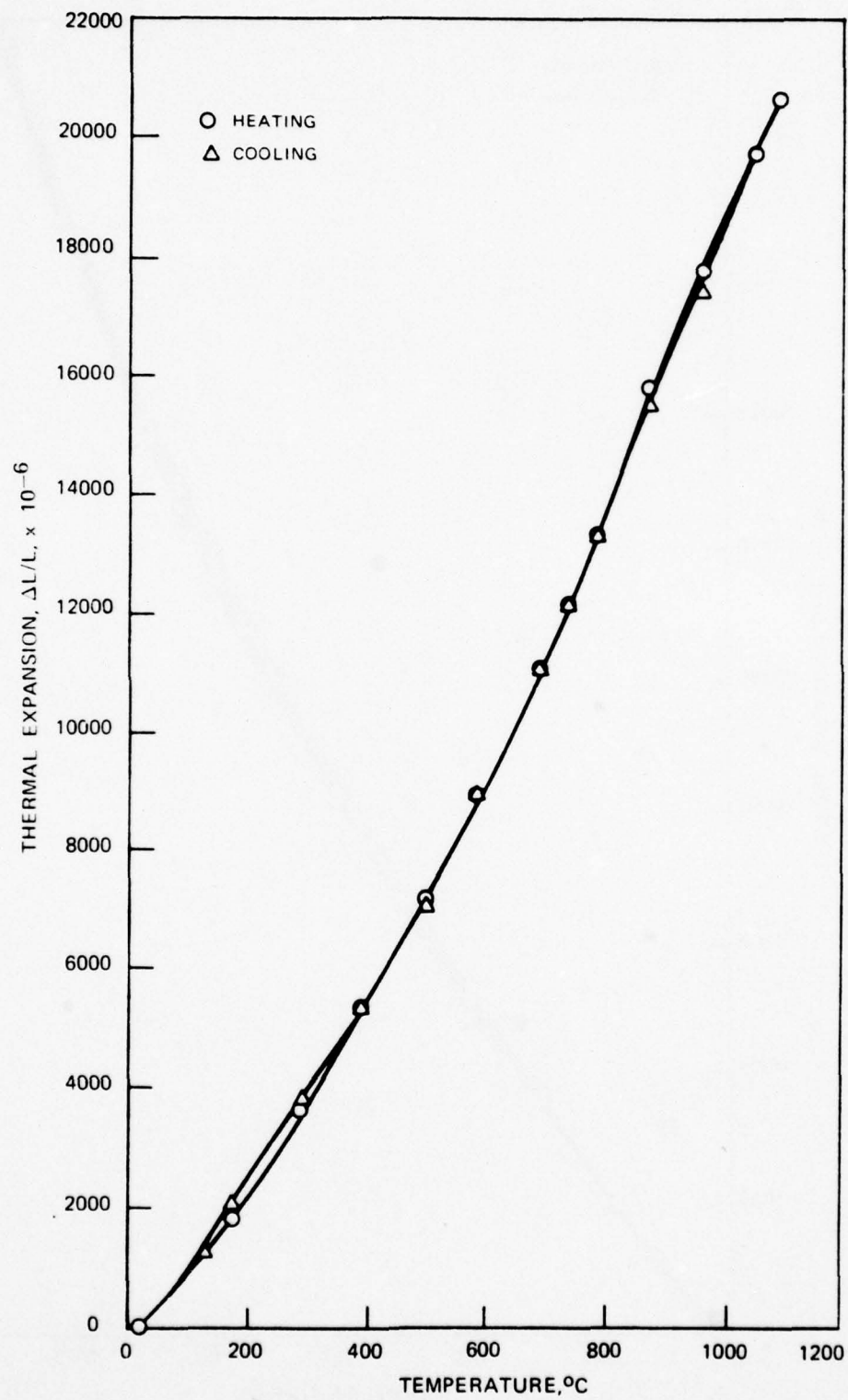
THERMAL EXPANSION BEHAVIOR OF Ni₃Al(γ')



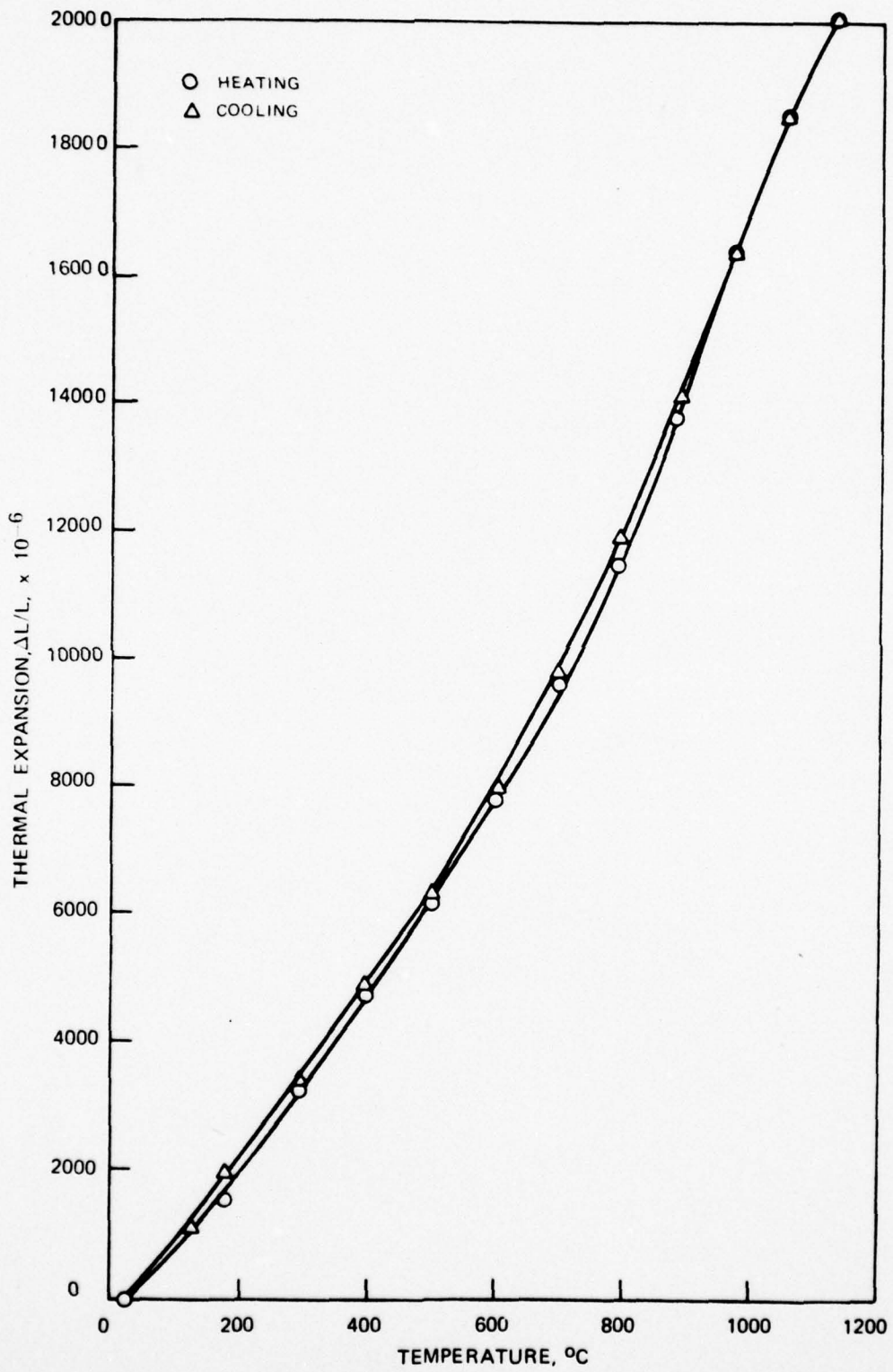
THERMAL EXPANSION BEHAVIOR OF CoCrAlY



THERMAL EXPANSION BEHAVIOR OF NiCoCrAlY



THERMAL EXPANSION BEHAVIOR OF NiCoCrAlY + Pt



MICROSTRUCTURE OF BETA Ni-4.1Cr-20.4Al COATING EXPOSED 189 HOURS AT 2100° F



BETA COATING

GAMMA PRIME +
ALPHA MOLYBDENUM
DIFFUSION ZONE

TRANSVERSE

50μ

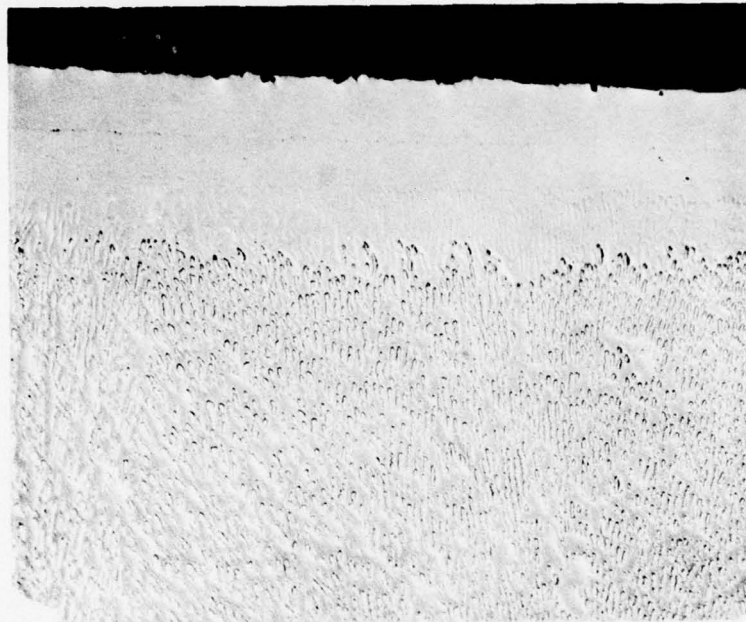


LONGITUDINAL

50μ

NORMANSKI INTERFERENCE CONTRAST

CYCLIC OXIDATION OF BETA-RICH NiCoCrAlY COATINGS



ORIGINAL INTERFACE

A

15 W/O Al

50μ



ORIGINAL INTERFACE

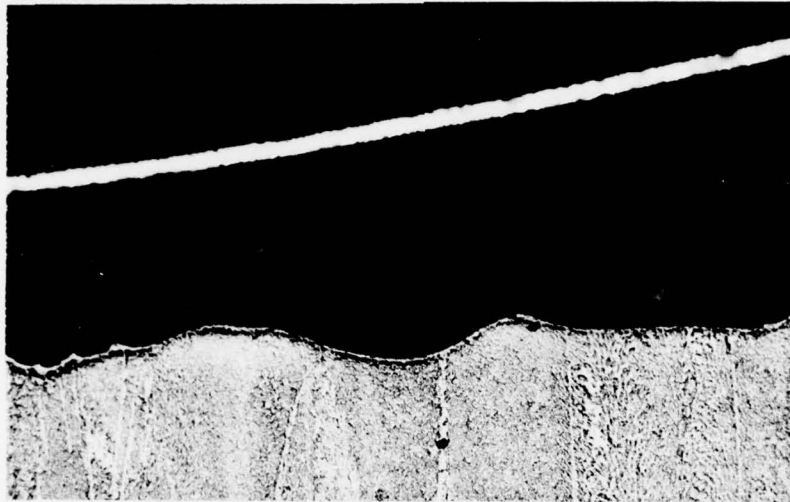
B

16.6 W/O Al

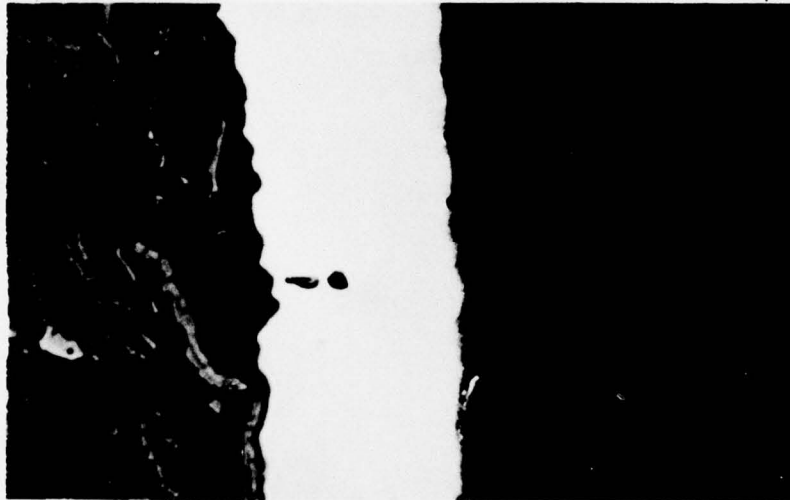
50μ

NOTE EXTENSIVE INTERDIFFUSION OF Al INTO SUBSTRATE

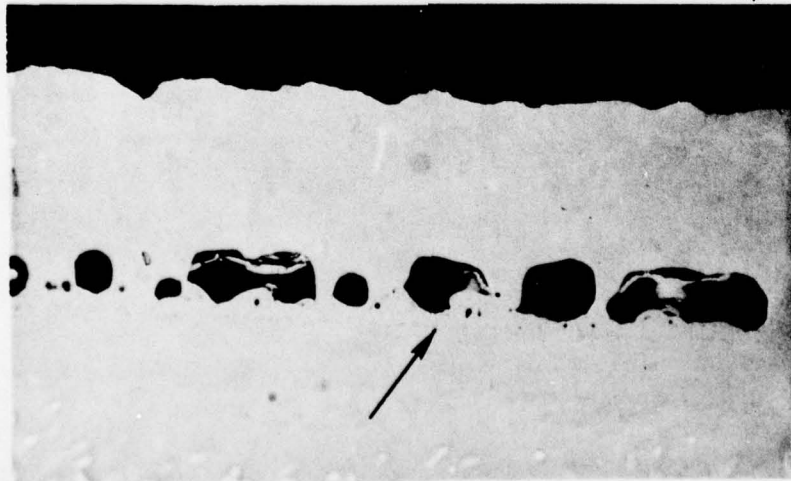
CYCLIC OXIDATION OF BETA-GAMMA COATINGS



A TRANSVERSE SECTION SHOWING COATING DELAMINATION 100μ

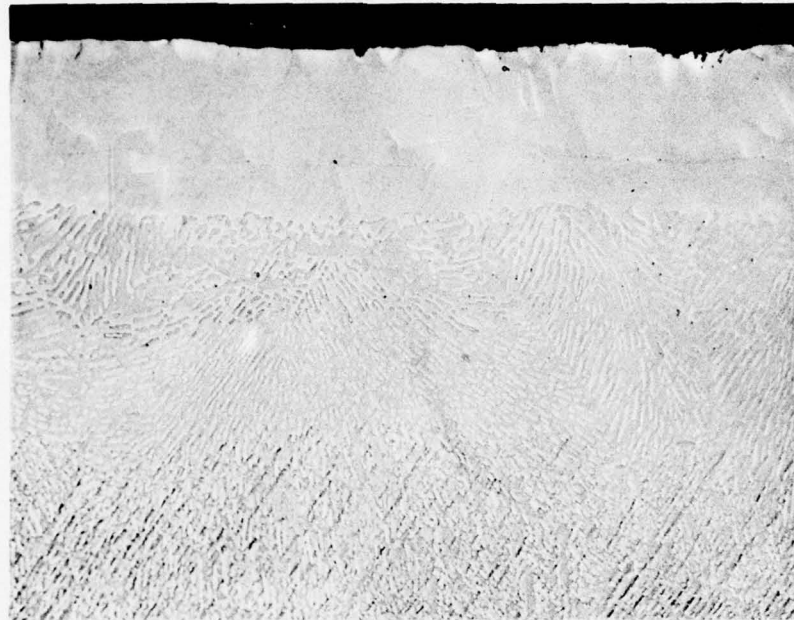


B SEPARATED, OXIDIZED COATING 20μ



C LONGITUDINAL SURFACE
NOTE POROSITY AT ORIGINAL COATING-SURFACE INTERFACE 20μ

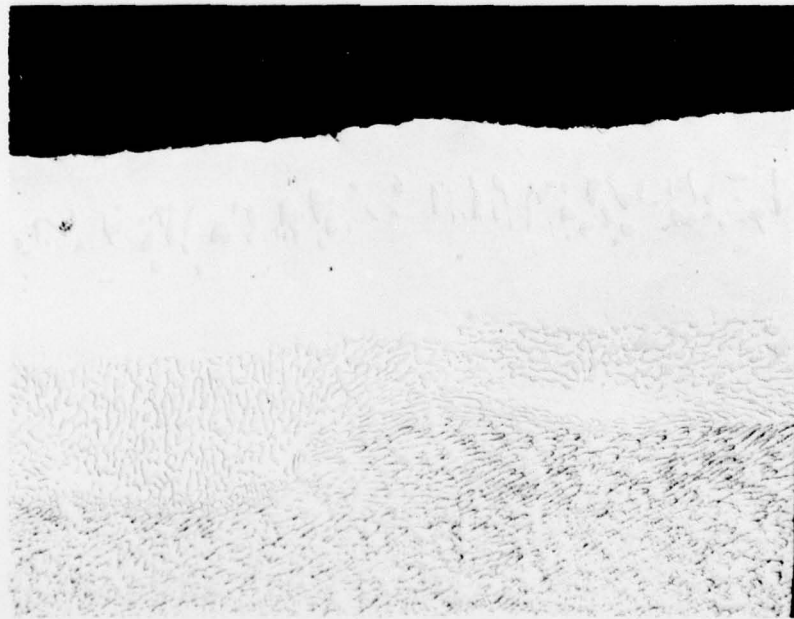
CYCLIC OXIDATION OF CHROMIUM-RICH NiCoCrAlY COATINGS



A

27 W/O Cr-13.6 W/O Al

50μ



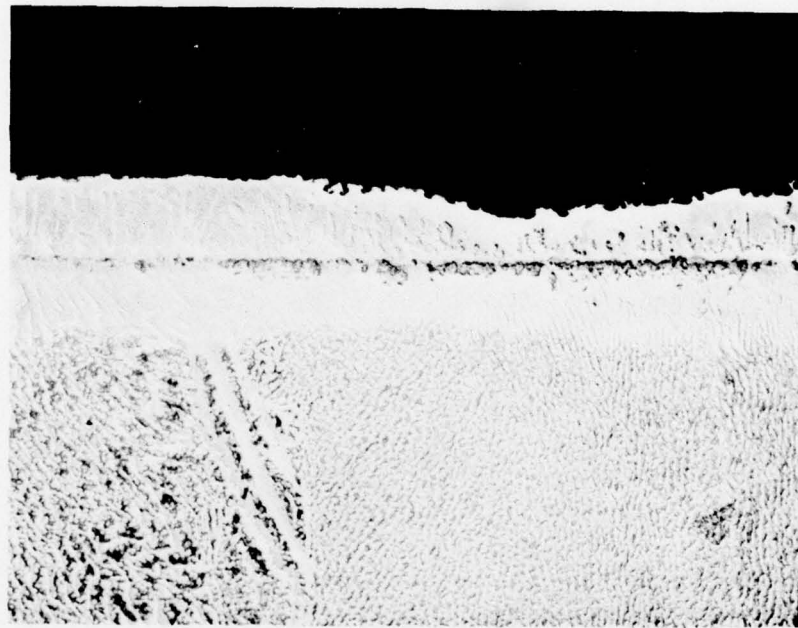
B

26.5 W/O Cr-14.3 W/O Al

50μ

LONGITUDINAL SECTIONS

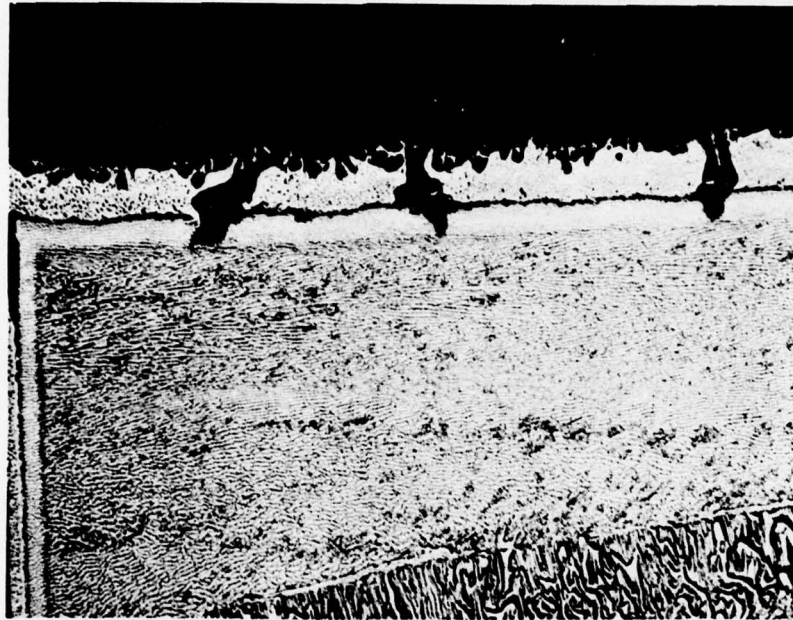
SULFIDATION CORROSION OF BETA-ENRICHED NiCrAlY COATING



50μ

TRANSVERSE SECTION

CYCLIC SULFIDATION OF BETA - RICH NiCoCrAlY COATINGS



A

LONGITUDINAL

100μ



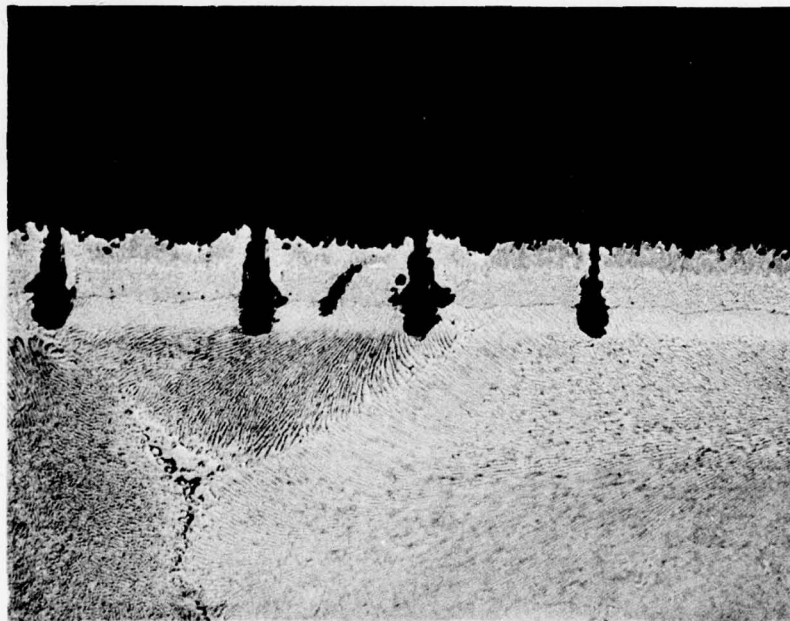
B

TRANSVERSE

100μ

NOTE TENSILE CRACKS IN LONGITUDINAL SECTION

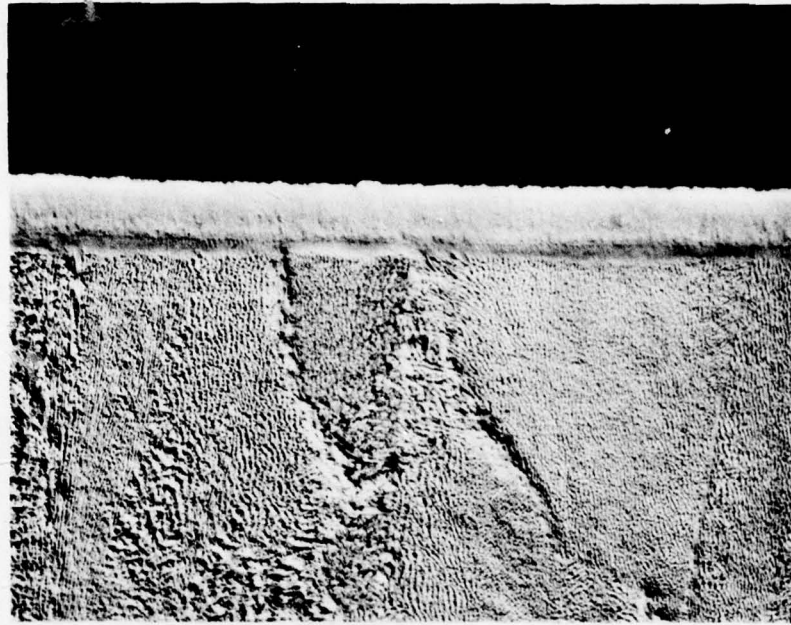
CYCLIC SULFIDATION OF CHROMIUM-RICH NiCoCrAlY COATINGS



50μ

LONGITUDINAL SECTION

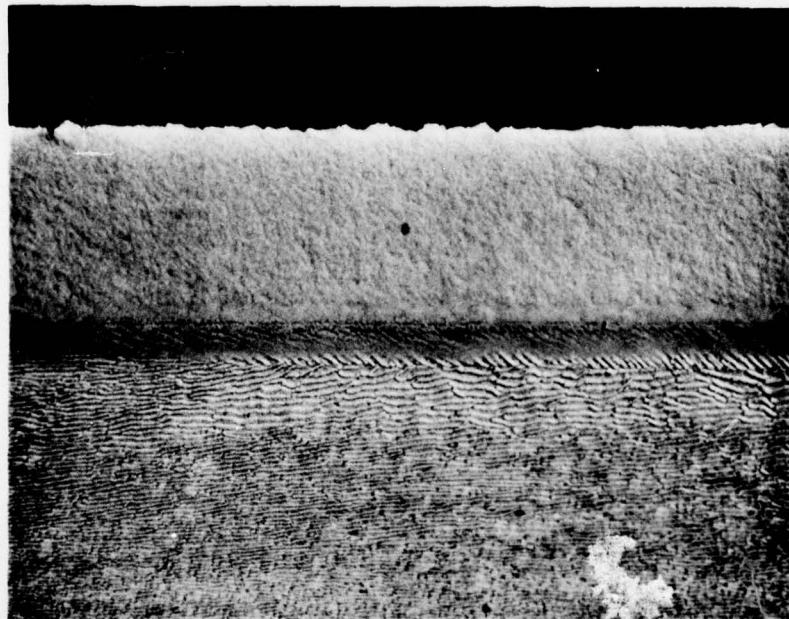
CYCLIC SULFIDATION OF PLATINUM MODIFIED NiCoCrAlY COATINGS



A

TRANSVERSE

100μ



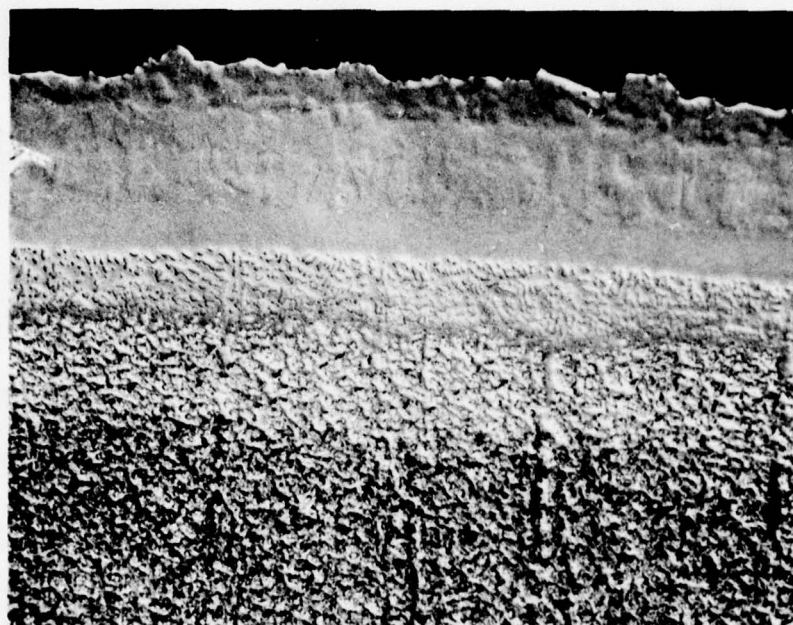
B

LONGITUDINAL

50μ

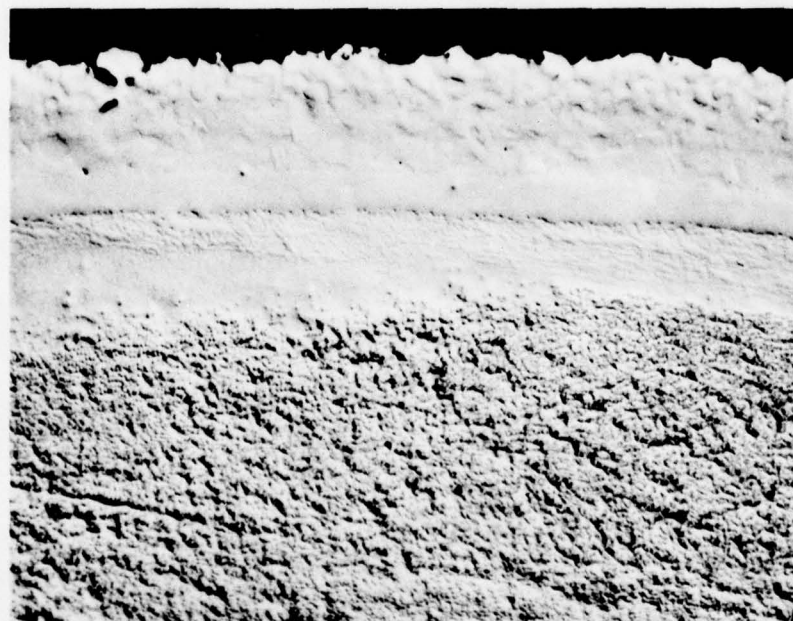
NOTE EXCELLENT SULFIDATION RESISTENCE AND NO TENSILE CRACKS

CYCLIC OXIDATION OF PLATINUM MODIFIED NiCoCrAlY COATINGS



A 15 W/O Pt 320 HRS 100μ

LONGITUDINAL SECTION



B 35 W/O Pt 508 HRS 100μ

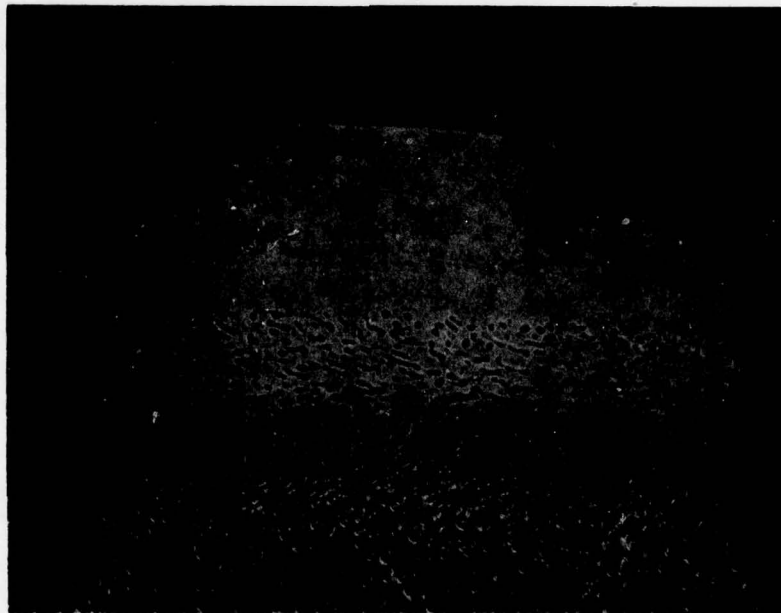
TRANSVERSE SECTION

**SUCCESSFULLY TESTED COMPOSITE γ -(γ' - Ti) - NiCoCrAlYPt COATING
(OXIDATION TEST)**



LONGITUDINAL SURFACE

50 μ

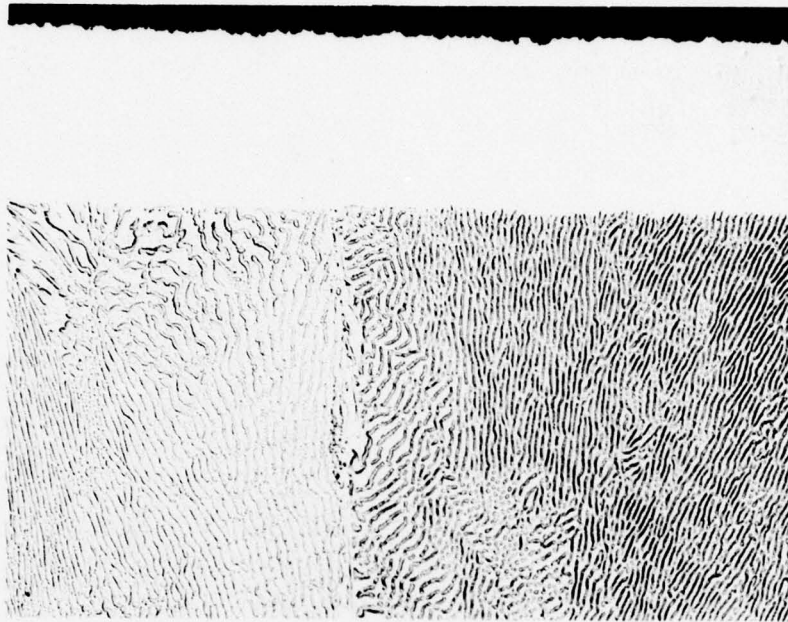


TRANSVERSE SURFACE

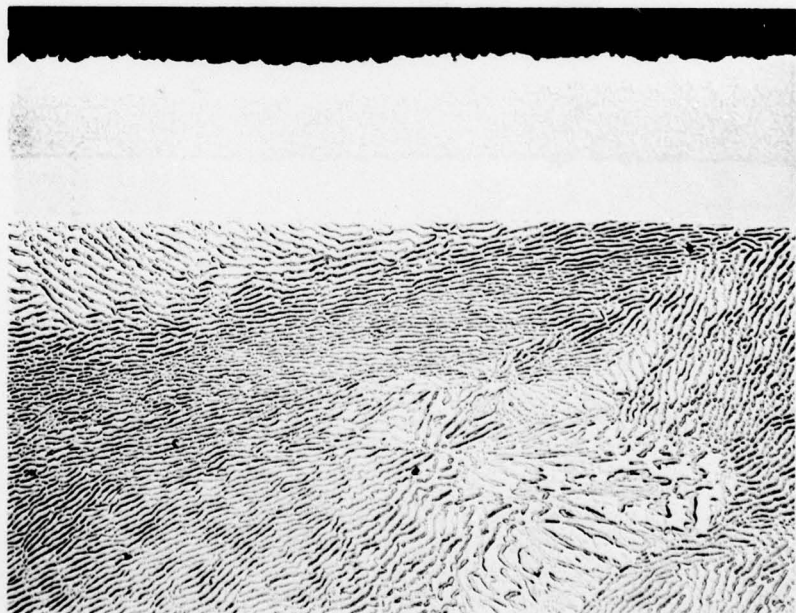
50 μ

**DIRECTIONALLY SOLIDIFIED γ/γ' - α COATED WITH γ (0.3 MILS), γ' + Ti (0.5-0.6 MILS)
AND NiCoCrAlYPt (2.7-3.5 MILS) CYCLICALLY OXIDIZED 2100°F FOR 363 CYCLES (1 CYCLE/HOUR)**

SUCCESSFULLY TESTED COMPOSITE $\gamma - (\gamma' + \text{Ti}) - \text{NiCoCrAlYPt}$ COATING
(SULFIDATION TEST)



TRANSVERSE SURFACE



LONGITUDINAL SURFACE

50 μ

D.S. 'd $\gamma/\gamma' - \alpha$ COATED WITH γ (0.3 MILS), $\gamma' + \text{Ti}$ (0.5-0.6 MILS) AND NiCoCrAlYPt (2.7-3.5 MILS)
AFTER CYCLIC 1650°F SULFIDATION TESTING, IN ADDITION TO THE ABSENCE OF FATIGUE
CRACKS IN THE COATING, NOTE THE ABSENCE OF POROSITY AT THE COATING/SUBSTRATE
INTERFACE.

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