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FUSED SILICON-RICH COATINGS FOR SUPERALLOYS

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16. Abstract
Various compositions of nickel-silicon and aluminum-silicon slurries were sprayed on IN 100 specimens and fusion-sintered to form fully dense coatings. Cyclic furnace oxidation tests in 1 atm air at 1100° C showed all the coatings to be protective for at least 600 hours, and one slurry, Al-60Si, was protective for 1000 hours. This coating also protected NASA TAZ 8A and NASA-TRW VIA for 1000 hours in the same furnace test. Alloys B 1900, TD-NiCr, and Mar-M200 were protected for lesser times, while NX 188 and NASA WAZ 20 were scarcely protected at all. Limited stress-rupture testing on 0.64-cm-diam IN 100 specimens detected no degradation of mechanical properties due to silicon diffusion.

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SUMMARY

Various nickel-silicon (Ni-Si) and aluminum-silicon (Al-Si) slurry coatings were applied to IN 100 and WI-52 oxidation screening specimens. Slurry compositions of Ni-67 atomic percent Si, Al-20 atomic percent Si, Al-40 atomic percent Si, Al-60 atomic percent Si, and Al-80 atomic percent Si were used because they fused to form fully dense structures with a high remelt temperature. Cyclic furnace oxidation tests in 1 atmosphere air at 1100° C showed all the coatings to protect IN 100 adequately for at least 600 hours; one coating, Al-60Si, was protective for at least 1000 hours. Although this coating was moderately thin (10 mg/cm^2), considerable amounts of silicon diffused to a depth of 200 micrometers into the substrate during furnace testing. Since the presence of even a small amount of silicon was felt to be potentially harmful to substrate mechanical properties, stress-rupture tests were run on bare and Al-60Si coated, 0.64-centimeter-diameter specimens of IN 100. Bare and coated specimens were annealed at 1100° C for 1000 hours in argon before stress-rupture testing. The 982° C, 138 meganewtons per square meter (1800° F, 20 ksi) stress-rupture lives of both the bare and coated IN 100 were about one-half that of the as-cast material, indicating that the loss of strength was due to simple aging with no additional degradation due to silicon diffusion. Attempts to reduce the amount of silicon diffusion by depositing thinner coatings resulted in reduced coating lives in the 1100° C cyclic oxidation tests. Specifically Al-60Si coatings deposited at 5.0 and 2.2 milligrams per square centimeter protected IN 100 for about 600 and 300 hours, respectively. All the coatings applied to WI-52 were unprotective. Coating spalling in the first few cycles of testing was the primary failure mode.

Cyclic 1100° C furnace tests were performed on seven additional nickel-base superalloys coated with the best coating composition, Al-60Si. NASA TAZ 8A and NASA-TRW VIA were protected for 1000 hours. Alloys B 1900, TD-NiCr, and Mar-M200 were protected for intermediate times, while NX 188 and NASA WAZ 20 were scarcely protected at all.

INTRODUCTION

Research in oxidation resistant coatings for superalloys has primarily involved the metal- (M) aluminum intermetallic compound and the metal-chromium-aluminum-yttrium alloy compositions. The popularity of these systems is based on their ability to form an adherent Al_2O_3 film. However, refractory metals, such as tantalum, niobium, tungsten, and molybdenum, are generally protected by M_5Si_3 and MSi_2 silicide compounds which form primarily SiO_2.

In general these silicide coatings were developed for higher temperature use than the alumina-forming coatings because refractory metals have useful strengths at much higher temperatures than do superalloys. For example, a fused-silicide coated columbium alloy tested for 50 hours in a cyclic 1200° C oxidation test experienced only a 2.5 milligram per square centimeter (mg/cm^2) weight gain with no coating failures (ref. 1). On the other hand, a commercially pack-aluminized nickel alloy tested in cyclic oxidation at 1150° C lost 12 mg/cm^2 in 50 hours and suffered from overall coating failure (ref. 2).

These results suggest that some benefit might be obtained if suitable silicide coatings could be applied to advanced superalloy materials. However, intermetallic compounds and low-melting eutectics are present in the binary Ni-Si and Co-Si systems (ref. 3). Thus, silicide coatings could be deleterious to the mechanical properties of the superalloy substrates. In addition, little is known about the oxidation of Ni-Si and Co-Si alloys as compared to the MAl and MCrAlY coating alloys.

The information available up to now indicates that silicon usually has a favorable effect on the oxidation resistance of nickel alloys. Silicon additions (~4 wt.%) to a pack aluminide coating were found to improve the 1100° C oxidation behavior of a NiAl coated Ni-base superalloy, ZhS6-K (ref. 4). A survey of commercial coatings has also identified silicon-containing aluminide coatings to be the most oxidation-resistant coatings on IN 100 and B 1900 in 1000° to 1120° C burner rig tests (ref. 5). Chemical vapor deposited silicon coatings on IN 738 provided 600 hours of protection in 850° C hot corrosion tests (ref. 6). Also, 1 weight percent (wt.%) additions of silicon to γ'-Ni_3Al and B 1900 markedly improved their cyclic oxidation behavior at 1200° C (refs. 7 and 8). On the other hand, silicon additions were observed to be detrimental to aluminum "metallide" coatings on IN 100 in 1093° C oxidation and in 900° C sulfidation tests (ref. 9).

The purpose of this investigation was to determine the feasibility of protecting superalloys by silicide coatings. These coatings were to consist of primarily silicide compounds, have silicon as a major constituent (~25 wt.%), and owe their oxidation resistance to the formation of SiO_2 or other silicates. The approach was to produce dense and adherent silicon-rich coatings using a fused slurry process and to evaluate these coatings in cyclic furnace oxidation tests. Various compositions of Ni-Si and Al-Si slurries forming low-melting eutectics were fused on nickel and cobalt superalloys and cyclic furnace tested at 1100° C for times up to 1000 hours. Coating thicknesses ranged from 30 to 260 micrometers (μm). As-coated and as-oxidized coatings were analyzed by standard metallographic, X-ray diffraction, and electron microprobe techniques.

EXPERIMENTAL PROCEDURE

Lacquer slurries of Ni-Si and Al-Si powder mixtures were air brush sprayed onto nickel and cobalt alloys. The powders used were 2.5 μm, 99.9 percent pure nickel; -325 mesh, 99.6 percent pure silicon; and -270 mesh, 99.5 percent pure aluminum. The sprayed slurries were air dried, then sintered in titanium-gettered, 10^{-6} torr vacuum at 1150° C for 5 hours. Various coating compositions were examined with respect to fusion, density, adherence, and remelt temperature on Inconel 600 and WI-52 substrates. These trial coating compositions are listed in table I. This fusion screening guided coating selection for subsequent cyclic oxidation tests.

Coupons for the initial furnace tests were 0.25- by 1.2- by 2.5-centimeter specimens of IN 100 and WI-52. Various coatings of the compositions which passed the fusion screening tests above were sprayed and sintered as before. The furnace oxidation screening tests consisted of 1-hour heating at 1100° C in 1-atmosphere air and 1/3 hour cooling to ~150° C for each cycle. A description of this apparatus is given in reference 7. Coating behavior was evaluated on the basis of gravimetric data obtained every 15 cycles and metallographic observation at the end of testing. The most oxidation resistant coating composition was then deposited on NASA TAZ 8A, NASA-TRW VIA, TD-NiCr, B 1900, Mar-M200, NX 188, and NASA WAZ 20 coupons. Oxidation testing of these coated superalloys consisted of the same 1100° C, 1-hour-cycle furnace test. Nominal substrate alloy compositions are listed in table II.

Limited 982° C, 138-meganewton-per-square-meter (MN/m^2) stress rupture tests were performed on coated and bare 0.64-centimeter-diameter (cm-diam) IN 100 specimens to determine the effects of silicon diffusion on substrate strengths. Duplicate specimens (fig. 1) were first aged at 1100° C for 1000 hours in slowly flowing argon to provide a thermal (diffusion) treatment with a minimum of oxidation. The rupture tests were then conducted in air.

Coating evaluation was based on gravimetric data and metallographic analyses. Limited microprobe line-trace analyses were performed on one coating/substrate system using a 15-kilovolt, 45-nanoampere electron beam. Coating surface phases were identified by in-situ X-ray diffractometer scans before and after oxidation exposure.

RESULTS

Coating Composition Screening

The thicknesses and phases of the Ni-Si and Al-Si coatings applied to IN 100 oxidation coupons are listed in table III. Since the preliminary fusion studies showed that the Al-60Si and Al-80Si coatings were too thick, less slurry of these compositions was deposited. The as-coated surface phases identified by X-ray diffraction consisted of a $Ni_{16}(Ti, Cr)_6Si_7$ G-phase (refs. 10 and 11) in all cases, in addition to various Ni-Al phases. This silicide phase was also found in the Al-Si fused salt metallide coating on IN 100 (ref. 9).

The gravimetric data in figure 2 show that none of the coatings lost more than 2 mg/cm^2 after 600 hours of testing. One slurry composition, Al-60Si, gave very little indication of any spalling after 600 hours and was therefore tested for 1000 hours. No coating failures were visually observed on any of the specimens. The microstructures of the coatings before and after oxidation are shown in figure 3. In all cases the as-deposited coatings were 100 percent dense, fully bonded complex structures. Oxidized coatings were significantly different in structure, although still somewhat complex. Striations were apparent in the dark-etching phases of the oxidized Al-20Si and Al-40Si coatings, suggesting the presence of low-aluminum martensitic NiAl (ref. 11).

The surface phases identified by X-ray diffraction of oxidized specimens are listed in table III. All the coatings formed primarily α-Al_2O_3 - a surprising result, especially for the Ni-67Si coating in which the only aluminum available was the 5 wt.% in the substrate. This implies that silicon significantly promoted Al_2O_3 formation for this coating. Less-protective TiO_2 and spinel oxides were found on all the coatings except Al-60Si. The G-phase was identified on all but the Al-20Si coating, implying that it was both a stable and oxidation resistant phase. X-ray diffraction analyses were also performed after 450 hours of testing with identical results, except that pattern for the G-phase was stronger then.

Similar coatings were also applied to cobalt-base WI-52 substrates. Fusion produced fully dense coatings with some interface porosity. This porosity became more noticeable for slurries having greater than 50 wt.% Si. X-ray diffractometer scans produced patterns that could not be identified. All coatings spalled on subsequent cycling in the furnace oxidation tests. Therefore no further work was done on silicon-rich coatings for cobalt alloys.

Silicon Diffusion

It was apparent from these studies that just a small amount of silicon-rich slurry produced a very thick coating. For example, 9 mg/cm^2 of Al-80Si produced 223 μm of coating - more than double that expected using an equal amount of a pure aluminum slurry. It was also apparent from the microstructures of oxidized coatings that denuded zones extended up to 200 μm beneath the coating into the substrate (fig. 3). The diffusion of silicon well into the substrate is indicated more graphically by the microprobe profiles of as-coated and oxidized IN 100 in figure 4. The silicon content of the surface layer was decreased by the thermal exposure, and a considerable amount of silicon did diffuse to a depth of about 200 μm into the substrate.

Because silicon decreases the melting point of nickel by forming low-melting eutectics (ref. 3), this diffusion of silicon into the substrate was a matter of concern. Consequently, stress-rupture tests were run to indicate the extent of property degradation caused by silicon diffusion. Duplicate 0.64-cm-diam specimens of IN 100 were coated with 11 mg/cm^2 of Al-60Si and annealed at 1100° C for 1000 hours in argon. Identical bare control specimens were aged in the same annealing run. The results of the 982° C, 138-meganewton-per-square-meter (1800° F, 20 ksi) stress-rupture test are listed in table IV along with data for the as-cast material. The stress-rupture life of the uncoated, aged IN 100 was decreased by half the as-cast value due to the aging treatment. No additional degradation was observable as a result of silicon diffusion in the coated specimens. Although more extensive testing is necessary to prove that silicon diffusion from the coating does not seriously degrade bulk mechanical properties, these preliminary indications were favorable.

An attempt was made to reduce the total amount of silicon present by depositing only one-half or one-fourth as much slurry as the original 10 mg/cm^2 of Al-60Si on IN 100 (table V). The gravimetric data in figure 5 shows that the thin coatings were superior to bare IN 100 (unpublished data obtained by C. A. Barrett of Lewis Research Center), but not as protective as the thicker coatings. The 2.2-mg/cm^2 coating was protective for about 300 hours, and the 5.0 mg/cm^2 coating was protective for about 600 hours. (Data from the original 10 mg/cm^2 coating and two new specimens were presented for comparison.) Although considerable weight losses were recorded for both thin coatings, the major surface of the 5.0 mg/cm^2 coating were intact after 1000 hours (figs. 6 and 7). Apparently most of the weight loss was due to coating failure at edges and rapid disintegration of the IN 100 at these areas. Thus even though considerable protection was offered by most areas of the 5.0 mg/cm^2 coating, decreasing the thickness reduced coating effectiveness sufficiently to warrant dropping this approach.

Other Superalloys Coated with Al-60Si

The excellent performance of the Al-60Si coating warranted its testing on alloys other than IN 100. Accordingly, 7 mg/cm^2 of Al-60Si slurry was fused on NASA TAZ 8A, NASA-TRW VIA, TD-NiCr, B 1900, Mar-M200, NX 188, and NASA WAZ 20 coupons to produce an estimated 140 μm of coating. The as-coated surface phases identified from in-situ diffractometer scans are listed in table VI. The Ni or Ni_3Al phases appeared on five alloys, the G-phase appeared on four of the alloys, and unidentified phases were present on five. The gravimetric data in figure 8 show that TAZ 8A and VIA were protected for 1000 hours and that protection for TD-NiCr and B 1900 was less than 1000 hours. Coated Mar-M200, NX 188, and WAZ 20 were even less oxidation resistant, in that order. After 1000 hours of oxidation, X-ray diffraction scans identified Al_2O_3 as the major oxide on TAZ 8A, VIA, and B 1900; and Cr_2O_3 was identified on TD-NiCr (table VI). Metallic γ and/or γ' phase were identified on all four of these alloys, and the G-phase was found only on VIA.

The microstructures after oxidation testing are shown in figure 9. No substrate attack is apparent for TAZ 8A, VIA, B 1900, or Mar-M200, even though the last two alloys lost 4 mg/cm^2 after 1000 and 679 hours, respectively. (No coating failures were observed visually for TAZ 8A, VIA, or B 1900.) The coating on TD-NiCr appeared to be seriously degraded and heavily oxidized, even though only 2.5 mg/cm^2 were lost after 1000 hours. The coatings on NX 188 and WAZ 20 offered little protection, as judged from both metallography and weight loss data.

It was, therefore, clear that various substrates affected the Al-60Si coating behavior very differently. However, inspection of the alloy chemistries in table II failed to identify any elements that could be closely associated with good or bad oxidation performance. Some clue to the role of substrate effects may be taken from the occurrence of the refractory metal tapiolite oxide, $Ni(Nb, Ta)_2O_6$, observed for three of the better systems (table VI). The formation of this oxide has been suggested as the reason why the high refractory metal contents in B 1900 and VIA do not seriously degrade oxidation resistance in the bare condition (ref. 14).

Although TAZ 8A, VIA, and B 1900 contain 2 to 6 wt.% Mo or W, they apparently have enough Ta or Nb to form tapiolite which accommodates oxidized Mo and W in solution. On the other hand, Mar-M200, NX 188, and WAZ 20 have no Nb or Ta and did not form tapiolite scales. These alloys were the least protected by the Al-60Si coating. It is equally important to point out that the coating phases formed on NX 188 and WAZ 20 were different from those for the other alloys and that their ability to form even Al_2O_3 was apparently diminished.

CONCLUDING REMARKS

The excellent oxidation resistance of diverse compositions of silicon-rich coatings on various nickel-base superalloys in cyclic furnace tests at 1100° C has been shown.

Furthermore, the processing of these fused coatings was most simple, involving only slurry-spraying and vacuum sintering. Not fully realized, however, was the original goal of the program, namely, to produce coatings with an average composition of ≥25 atomic percent silicon, whose protection was derived from silicon oxide formation.

While considerable amounts of silicon were used in the slurry mixtures, the resultant coatings still largely consisted of Ni-Al phases. Thus, while the silicon did form the $Ni_{16}(Ti, Cr)_6Si_7$ silicide compound, oxidation resistance appeared to result from Al_2O_3 formation rather than any crystalline silicate oxides. Therefore, the benefit of silicon probably resulted more from promoting Al_2O_3 formation than from any new mechanisms, although the possibility of amorphous SiO_2 formation existed. Nevertheless, the $Ni_{16}(Ti, Cr)_6Si_7$ phase was associated with the better coating systems. This is somewhat surprising because this phase was previously associated with the poor behavior of the Al-Si metallide coating on IN 100 (ref. 9).

It is unfortunate that these silicon-rich coatings were not more protective for the alloys having high Mo and W contents, NX 188 and WAZ 20. It had been expected that some of the benefits derived from siliciding refractory metals could be realized by silicide coating these superalloys. But since effective protection still occurred by Al_2O_3 formation, the anticipated benefits of silicon-rich coatings were not obtained for these two alloys.

The limited stress-rupture tests reported here showed that long-time diffusion of the Al-60Si coating did not shorten the life of 0.64-cm-diam IN 100 specimens any more than did the concomittant coarsening of strengthening phases due to aging. However, the exclusive effects of silicon diffusion will be clearly seen only if the diffusion treatment is performed on alloys or at temperatures at which degradation by aging does not exist. The use of smaller specimen test sections would also accentuate diffusion effects. Since some of the coating systems showed 1000-hour lives at 1100° C, silicon-rich coatings are worthy of further consideration for protecting superalloys, and they may find use in the protection of directionally solidified eutectic superalloys.

SUMMARY OF RESULTS

Sintering studies have shown that slurries of Ni-67Si, Al-20Si, Al-40Si, Al-60Si, and Al-80Si form dense fused coatings on nickel alloy IN 100 and cobalt alloy WI-52. Cyclic 1100° C furnace tests have shown that all of these coatings protected IN 100 for at least 600 hours and that one coating, 10 mg/cm^2 of Al-60Si, was protective for 1000 hours. None of these coatings protected WI-52.

Limited 982° C, 138 MN/m^2 stress-rupture tests of bare and Al-60Si coated 0.64-cm-diam IN 100 annealed at 1100° C for 1000 hours in argon detected no degradation over and above that due to thermal aging of the substrate. Decreasing the coating thickness from 10 mg/cm^2 to 5.0 and 2.2 mg/cm^2 resulted in reduced furnace oxidation lives of about 600 and 300 hours, respectively.

An Al-60Si coating of 7 mg/cm^2 protected TAZ 8A and NASA-TRW VIA for 1000 hours in 1100° C cyclic furnace tests. Alloys B 1900, TD-NiCr, and Mar-M200 were protected for less time, and NX 188 and WAZ 20 were scarcely protected at all. The poor behavior of this coating on some of the alloys was apparently related to a high molybdenum or tungsten content in the substrate and the absence of the G-phase in the coating. The effect of other refractory metals, Cb and Ta, was diminished by their formation of an innocuous $Ni(Nb,Ta)_2O_6$ tapiolite oxide.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, November 14, 1973,
501-01.

REFERENCES

1. Priceman, Seymour; and Kubick, Richard: Development of Protective Coatings for Columbium Alloy Gas Turbine Blades. Sylvania Electric Products, Inc. (AFML-TR-71-172, AD-748837), June 1972, p. 33.
2. Levine, Stanley R.: Cyclic Furnace and High-Velocity Oxidation of an Aluminide-Coated High-Strength Nickel Alloy (B-1900). NASA TM X-2370, 1971, p. 26.
3. Hansen, Max: Constitution of Binary Alloys. Second ed., McGraw-Hill Book Co., Inc., 1958.
4. Zemskov, G. V.; Kogan, R. L.; Kostenko, A. V.; and Luk'yanchenko, E. M.: Effect of Silicon Additions on the Oxidation Resistance of Aluminized Layers on a Nickel-Chromium Alloy. Protective Coatings on Metals. Vol. 4. G. V. Samsonov, ed., Consultants Bureau, 1972.
5. Moore, V. S.; Brentnall, W. D.; and Stetson, A. R.: Evaluation of Coatings for Cobalt- and Nickel-Base Superalloys. Vol. 2. Rep. RDR-1474-3, Solar (NASA CR-72714), July 1970.
6. Felix, P.; and Eroos, E.: CVD-Silicon Coatings for the Corrosion Protection of Stationary Gas Turbine Blades. Werkstoffe und Korrosion, vol. 8, 1972, pp. 627-636.

7. Lowell, Carl E.; and Santoro, Gilbert J.: The 1200° C Cyclic Oxidation Behavior of Two Nickel-Aluminum Alloys (Ni_3Al and NiAl) with Additions of Chromium, Silicon, and Titanium. NASA TN D-6838, 1972.

8. Lowell, Carl E.; and Miner, R. V.: Improvement in Cyclic Oxidation of the Nickel-Base Superalloy B-1900 by Addition of One Percent Silicon. NASA TM X-68191, 1973.

9. Fiedler, H. C.; and Sieraski, R. J.: Aluminide Coatings for Nickel-Base Alloys. Rep. SRD-72-028, General Electric Co. (NASA CR-120871), Nov. 12, 1971.

10. Spiegel, F. X.; Bardos, D.; and Beck, Paul A.: Ternary G and E Silicides and Germanides of Transition Elements. Trans. AIME, vol. 227, no. 3, June 1963, pp. 575-579.

11. Gladyshevskii, E. I.; and Borusevich, L. K.: The Cr-Ni-Si System. Russian J. Inorg. Chem., vol. 8, no. 8, Aug. 1963, pp. 997-1000.

12. Smialek, James L.: Martensite in NiAl Oxidation-Resistant Coatings. Met. Trans., vol. 2, no. 3, Mar. 1971, pp. 913-915.

13. Dreshfield, Robert L.; and Ashbrook, Richard L.: Sigma Phase Formation and its Effect on Stress-Rupture Properties of IN-100. NASA TN D-5185, 1969.

14. Garlick, Ralph G.; and Lowell, Carl E.: Alloy Composition Effects on Oxidation Products of VIA, B-1900, 713C, and 738X: A High Temperature Diffractometer Study. NASA TM X-2796, 1973.

TABLE I. - PRELIMINARY SCREENING OF SILICIDE COATINGS

Substrate	Slurry composition, at. %	Weight, mg/cm^2	Comments
Inconel 600	Al-11.3Si	25	OK
	Al-20Si	25	OK
	Al-30Si	25	OK
	Al-40Si	25	OK
	Al-50Si	25	OK
	Al-80Si	25	Low remelt temperature
	Ni-50Si	35	Porous, spalled
	Ni-67Si	37	OK
	100% Si	25	Low remelt temperature
	Ni-33Al-33Si	35	Porous
WI-52	Al-11.3Si	20	OK
	Al-40Si	20	OK
	Al-70Si	20	Spalled
	Ni-50Si	30	Porous
	Ni-67Si	30	OK
	Ni-80Si	25	OK
	100% Si	20	Spalled

TABLE II. - NOMINAL COMPOSITION OF SUPERALLOYS

Alloy	Content, wt.%												
	Ni	Cr	Co	Al	Ti	Zr	B	C	W	Mo	Nb	Ta	Other
TAZ 8A	Bal.	6.0	----	6.0	---	1.0	-----	0.125	4.0	4.0	2.5	8.0	----------------------
VIA	Bal.	6.0	7.5	5.4	1.0	.10	0.015	.13	5.8	2.0	.5	9.0	0.40 Re, 0.50 Hf
IN 100	Bal.	10.0	15.0	5.5	4.7	.06	.014	.18	----	3.0	---	---	----------------------
TD-NiCr	Bal.	20.0	----	---	---	----	-----	-----	----	----	---	---	2.34 ThO_2
B 1900	Bal.	8.0	10.0	6.0	1.0	.10	.015	.10	----	6.0	---	4.0	----------------------
Mar-M200	Bal.	9.0	10.0	5.0	2.0	.05	.015	.15	12.5	----	1.0	---	----------------------
NX 188	Bal.	----	----	8.0	---	----	-----	.04	----	18.0	---	---	----------------------
WAZ 20	Bal.	----	----	6.5	---	----	1.5	.2	20.0	----	---	---	----------------------
WI-52	----	21.0	Bal.	---	---	----	-----	.45	11.0	----	2.0	---	0.25 Mn, 0.25 Si, 2.0 Fe

TABLE III. - FUSED SILICIDE COATING THICKNESSES AND PHASES ON IN 100

Coating composition, at.%	Coating thickness		Coating phases[a]	
	mg/cm^2	μm	As-coated	Oxidized for 600 hr
Ni-67Si	28	260	γ', G	A, T, S, γ/γ', G
Al-20Si	18	213	NiAl, G, u	A, T, S, γ/γ'
Al-40Si	19	226	NiAl, G, u	A, T, S, γ/γ', G
Al-60Si	10	160	γ/γ', G	A, γ/γ', G
Al-80Si	9	223	γ', G	A, T, S, γ/γ', G

[a] A denotes α-Al_2O_3; T, TiO_2 (rutile); S, spinel (a_0 = 8.10×10^{-10} m); γ/γ', γ-Ni or γ'-Ni_3Al; G, $Ni_{16}(Ti, Cr)_6Si_7$ (G-phase); and u denotes unidentified peaks.

TABLE IV. - STRESS RUPTURE DATA FOR BARE AND Al-60Si COATED IN 100

Specimen condition		982° C, 138 MN/m^2 stress-rupture life, hr
Bare	As-cast[a]	$\left.\begin{matrix}242\\211\end{matrix}\right\}$ 226 av
	Annealed 1100° C, 1000 hr	$\left.\begin{matrix}116\\82\end{matrix}\right\}$ 96 av
Coated[b]	Annealed 1100° C, 1000 hr	$\left.\begin{matrix}138\\99\end{matrix}\right\}$ 118 av

[a] Ref. 13

[b] 11 mg/cm^2 thick.

TABLE V. - THIN Al-60Si COATINGS ON IN 100

THICKNESSES AND PHASES

As-coated thickness		Surface phases[a]		
mg/cm^2	μm	As-coated	Oxidized	
5.0	120	γ/γ', G	1000 hr	Al_2O_3, spinel, TiO_2, γ/γ'
2.2	33	γ/γ', G	755 hr	Spinel, TiO_2, Al_2O_3, γ/γ'

[a] γ/γ' denotes γ-Ni or γ'-Ni_3Al; G denotes $Ni_{16}(Ti, Cr)_6Si_7$ (G-phase).

TABLE VI. - X-RAY DIFFRACTION DATA FOR Al-60Si (7 mg/cm^2)

COATED Ni-BASE SUPERALLOYS

Substrates	Surface phases[a]			Substrate elements, wt.%	
	As-coated	Oxidized at 1100° C		Mo, W	Cb, Ta
TAZ 8A	G, γ/γ', u	1000 hr	A, S, TP, γ/γ', u	4, 4	2.5, 8
NASA-TRW VIA	G, γ/γ', u	1000 hr	A, TP, S, G, γ/γ'	2, 6	.5, 9
TD-NiCr	γ, γ', u	1000 hr	C, γ/γ', u	0, 0	0, 0
B 1900	G, γ/γ'	1000 hr	A, S, TP, γ/γ', u	6, 0	0, 4
Mar-M200	G, γ/γ'	649 hr	u, γ/γ'	0, 12.5	1, 0
NX 188	u	126 hr	A, S, NiO, γ/γ', u	18, 0	0, 0
WAZ 20	u	108 hr	NiO, $NiWO_4$	0, 20	0, 0

[a] A, Al_2O_3; S, spinel, $a_o = 8.10\times10^{-10}$ m; TP, tapiolite, $Ni(Cb, Ta)_2O_6$; G, G-phase, $Ni_{16}(Ti, Cr)_6Si_7$; γ/γ', γ-Ni or γ'-Ni_3Al; u, unidentified peaks; C, Cr_2O_3.

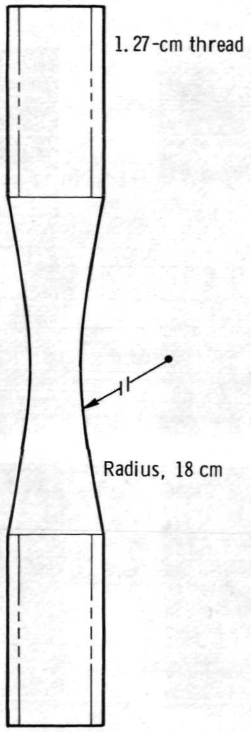


Figure 1. - Stress-rupture specimen configuration.

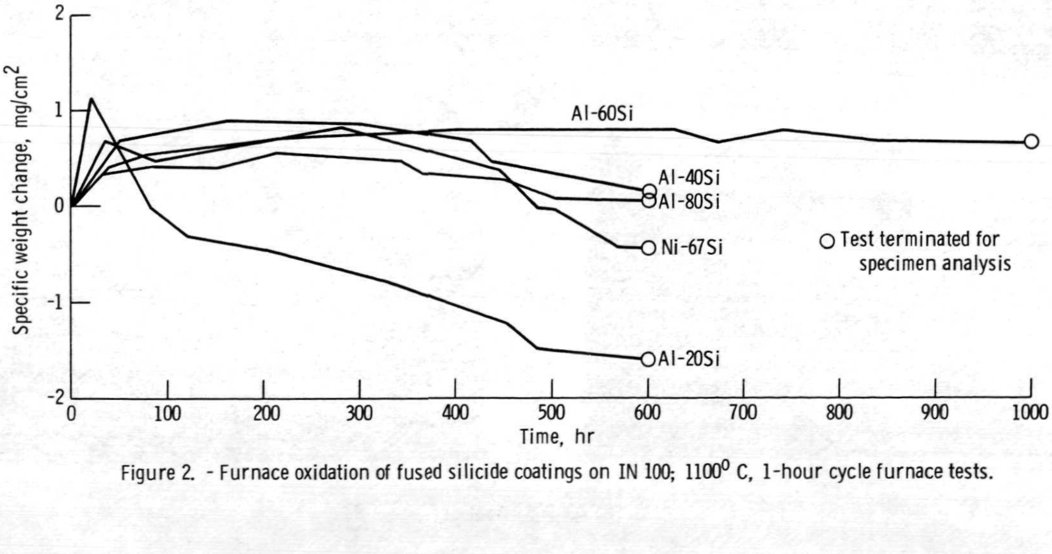
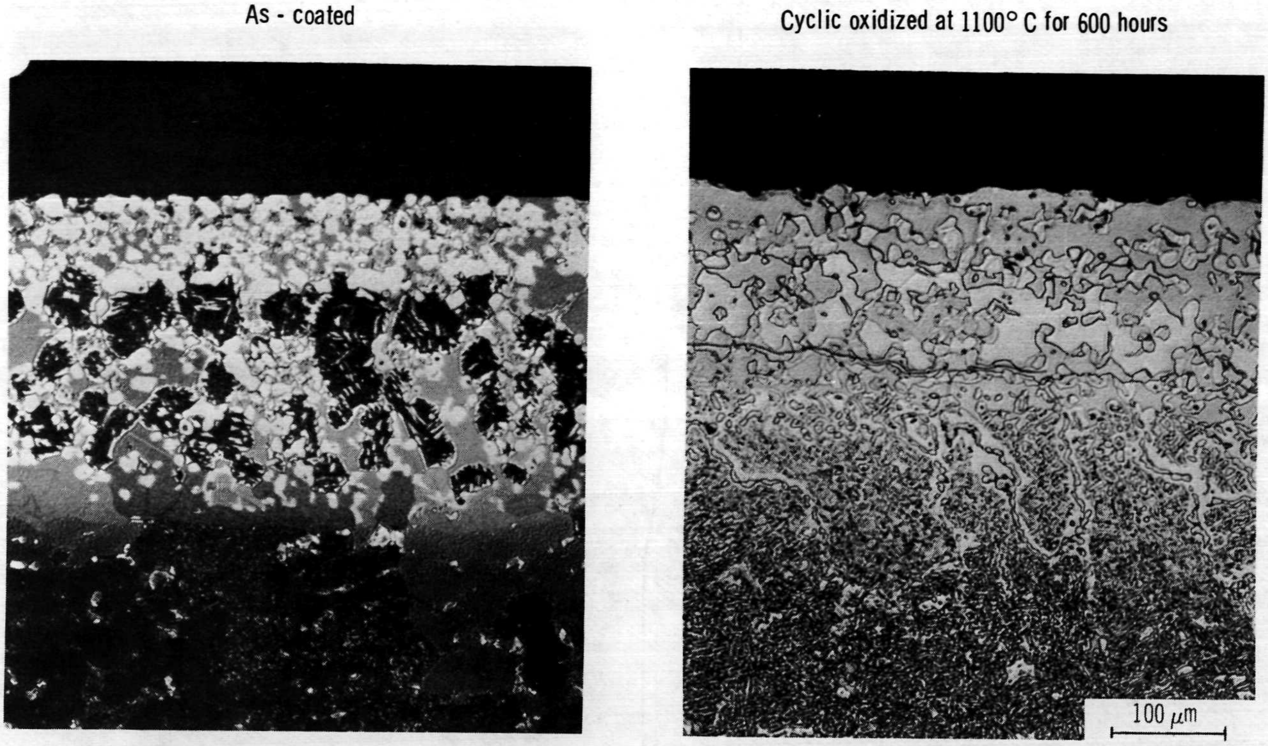
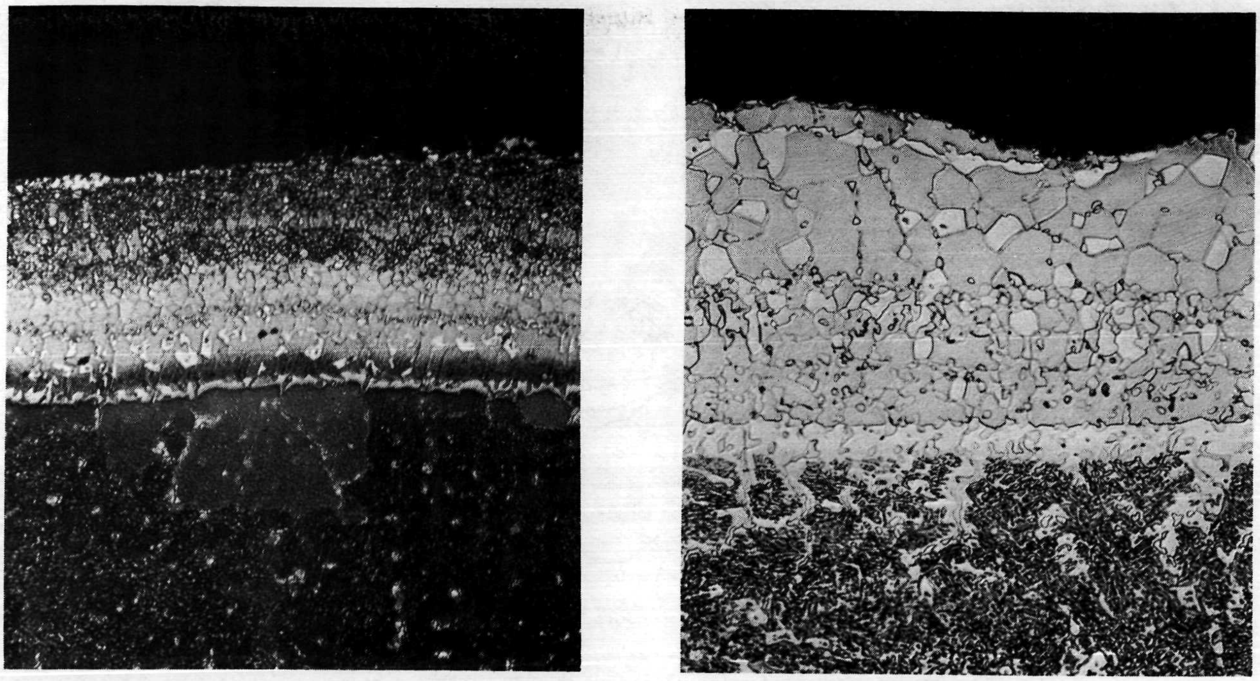


Figure 2. - Furnace oxidation of fused silicide coatings on IN 100; 1100° C, 1-hour cycle furnace tests.

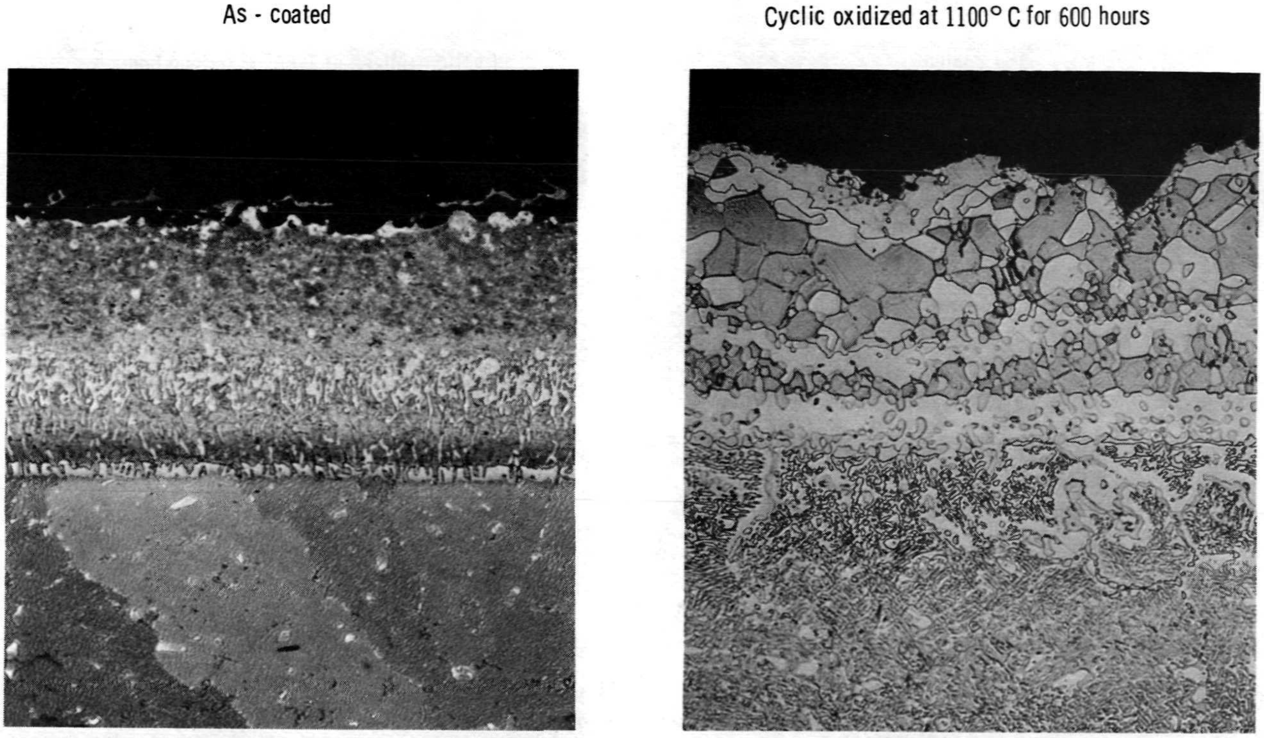


(a) Ni-67Si coating.



(b) Al-20Si coating.

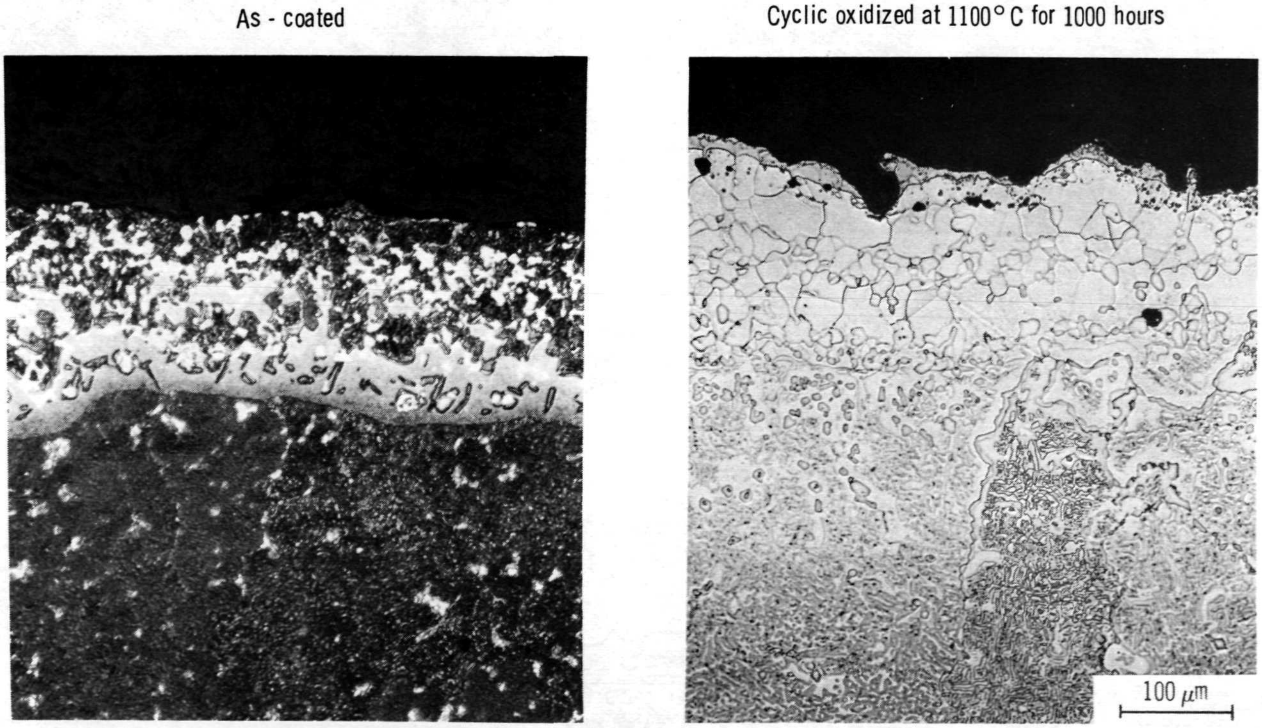
Figure 3. - Effect of oxidation on the structure of coated IN 100; etched. X150.



As - coated

Cyclic oxidized at 1100° C for 600 hours

(c) Al-40Si.

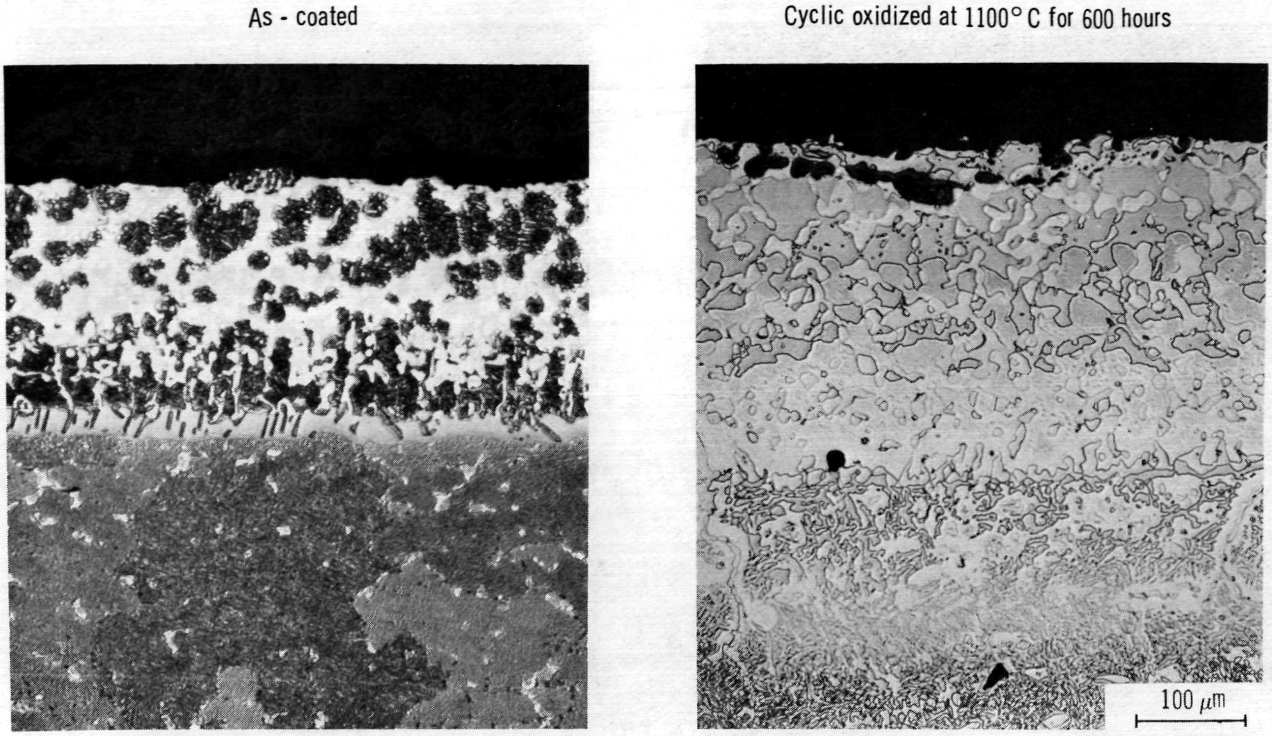


As - coated

Cyclic oxidized at 1100° C for 1000 hours

(d) Al-60Si.

Figure 3. - Continued.



(e) Al-80Si.

Figure 3. - Concluded

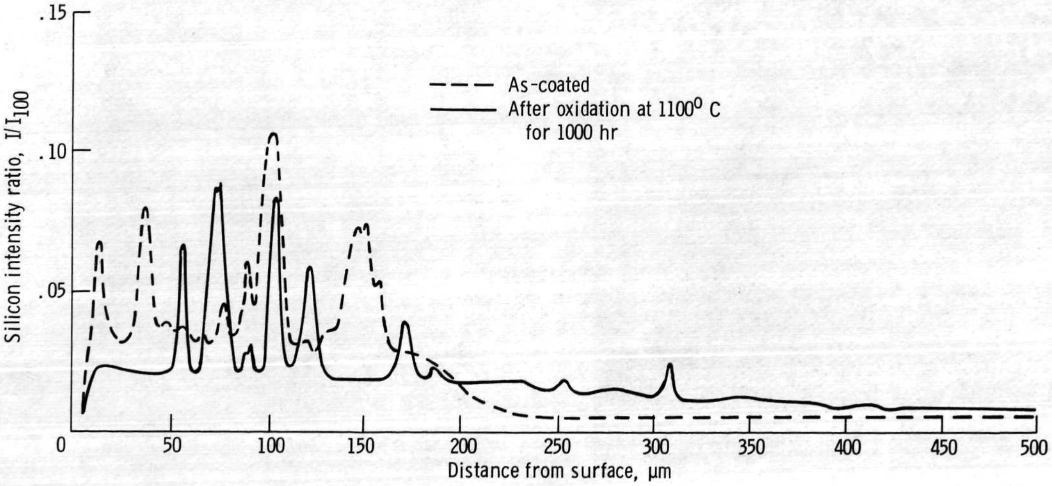


Figure 4. - Silicon microprobe traces for Al-60Si fused coating on IN 100.

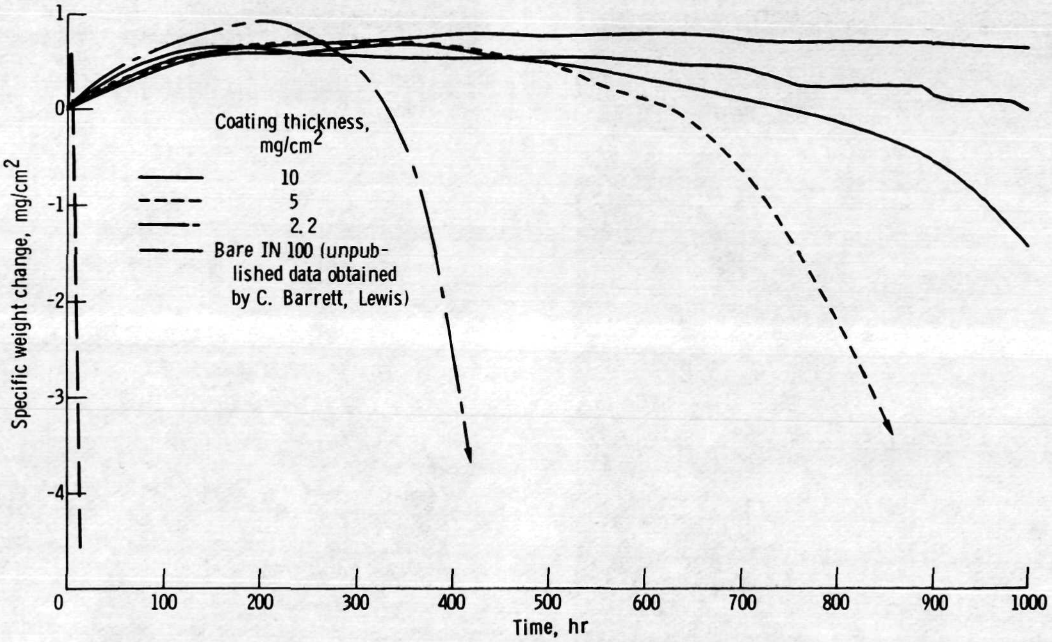
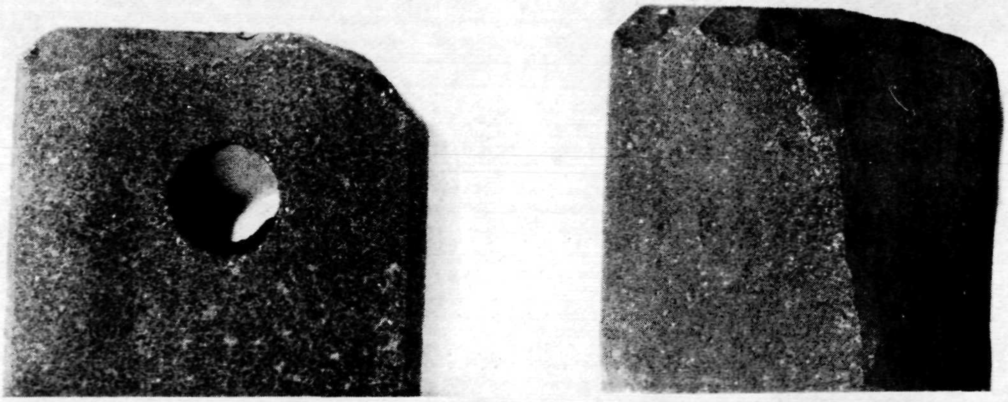
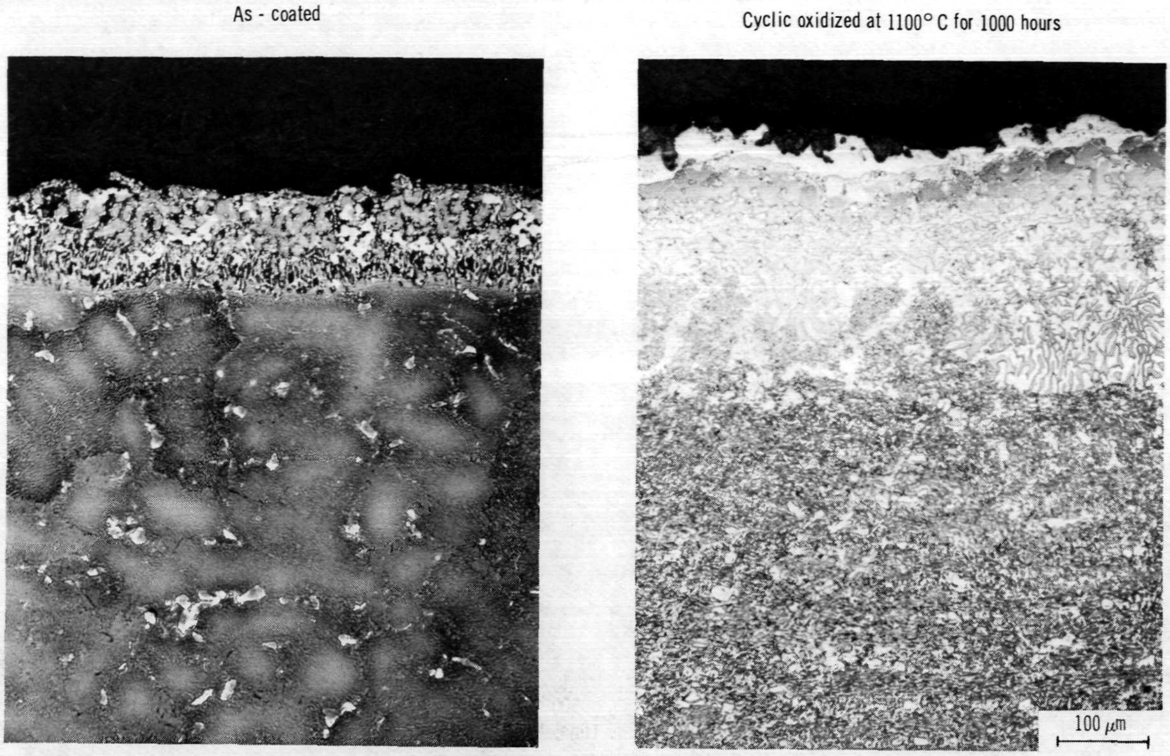


Figure 5. - Effect of coating thickness on oxidation behavior of Al-60Si coated IN 100.



(a) Coating thickness, 5.0 mg/cm²; oxidized 1000 hours. (b) Coating thickness, 2.2 mg/cm²; oxidized 755 hours.

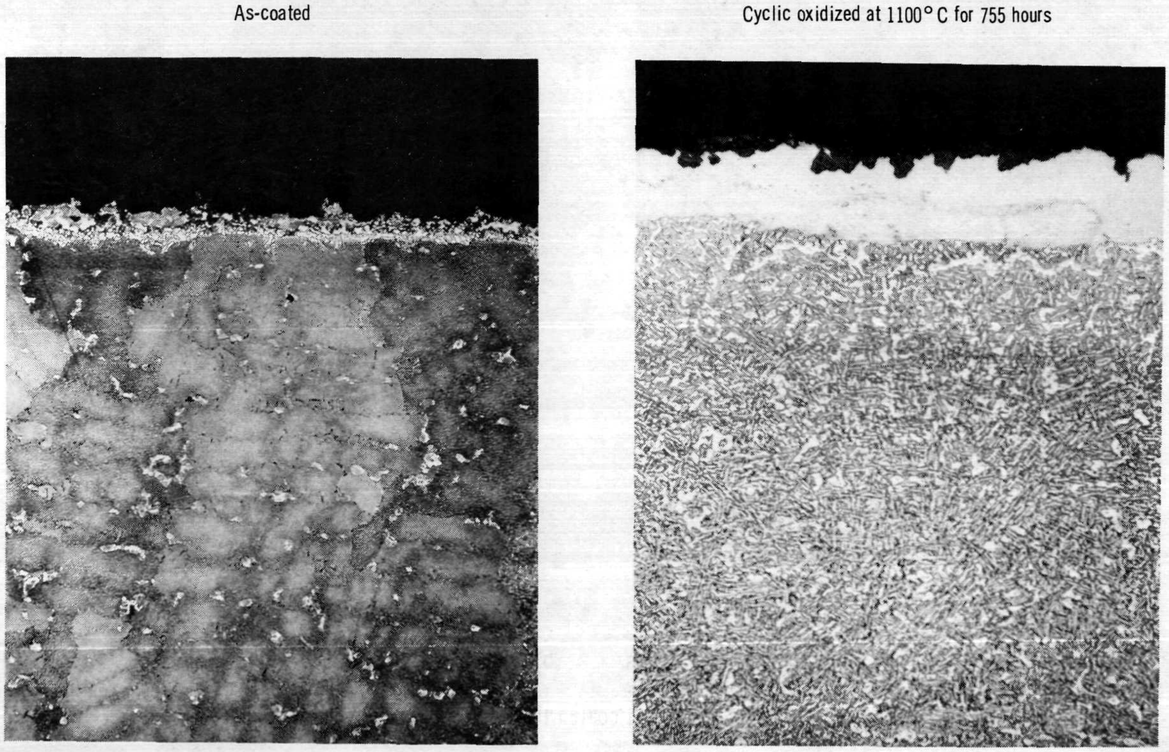
Figure 6. - Appearance of thin Al-60Si coated IN 100 after 1100° C oxidation. X4.5.



As - coated

Cyclic oxidized at 1100° C for 1000 hours

(a) Coating thickness, 5 mg/cm^2 (120 μm).



As-coated

Cyclic oxidized at 1100° C for 755 hours

(b) Coating thickness, 2.2 mg/cm^2 (33 μm).

Figure 7. - Effect of oxidation on thin Al-60Si coatings on IN 100; etched. X150.

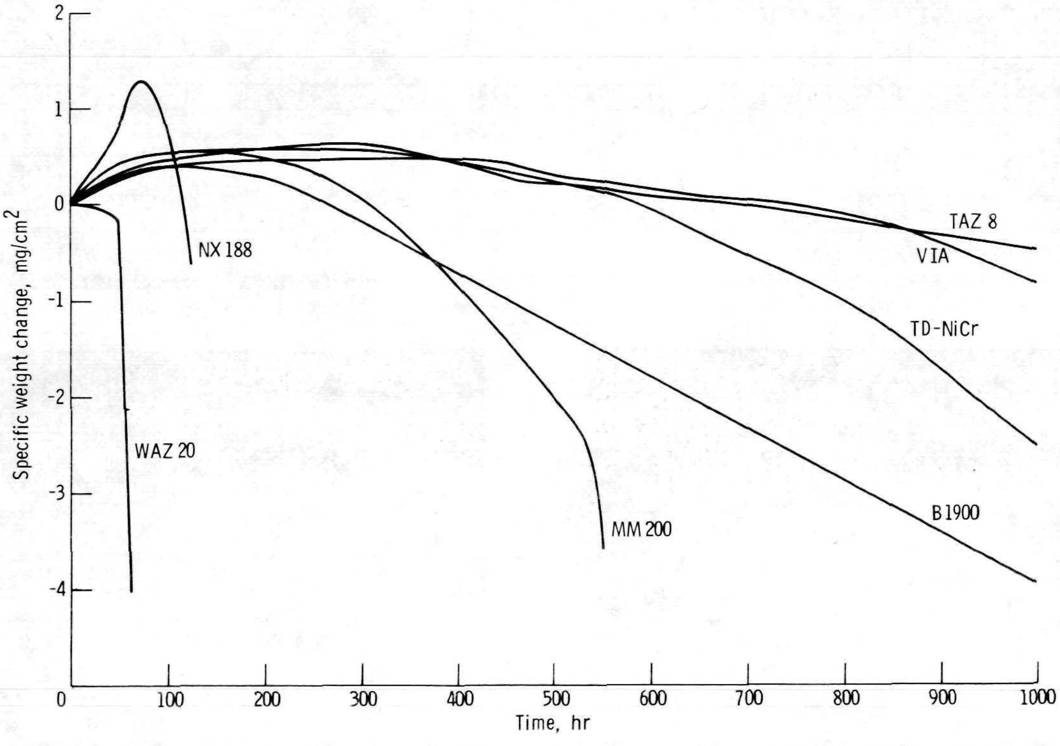
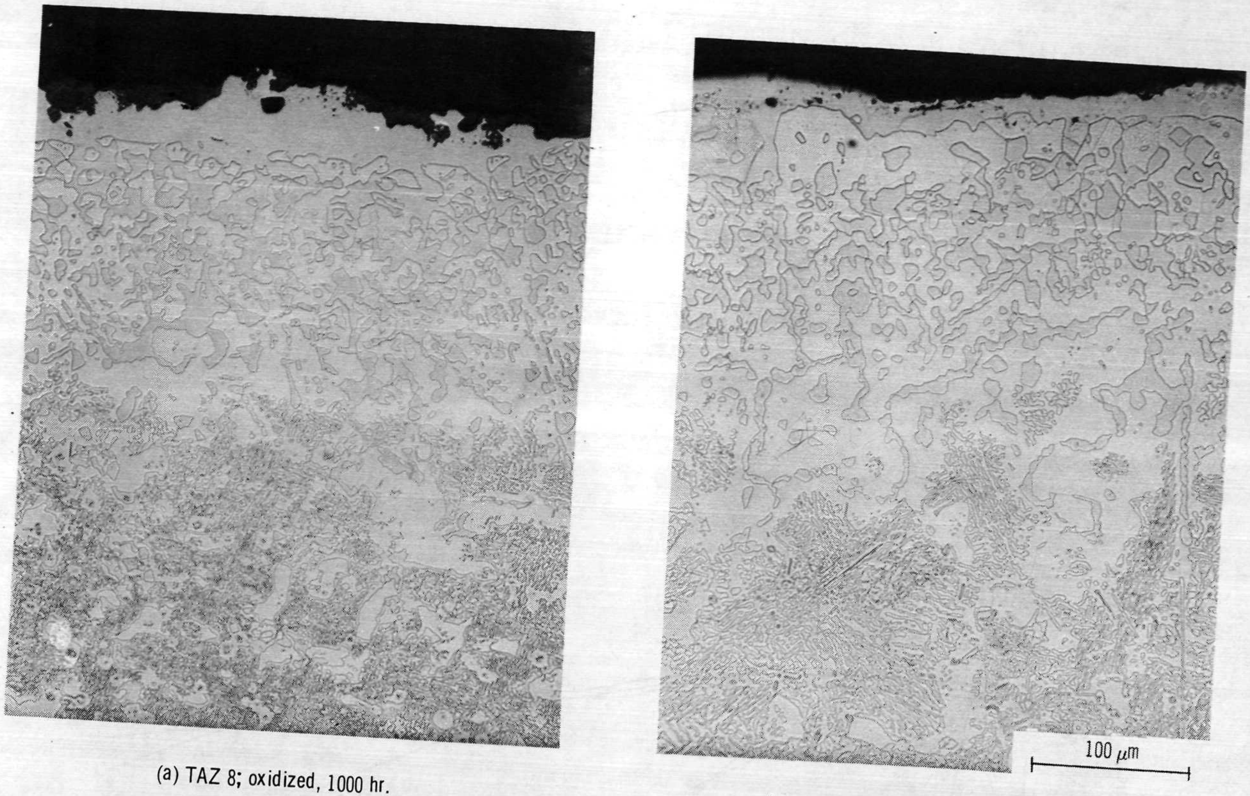
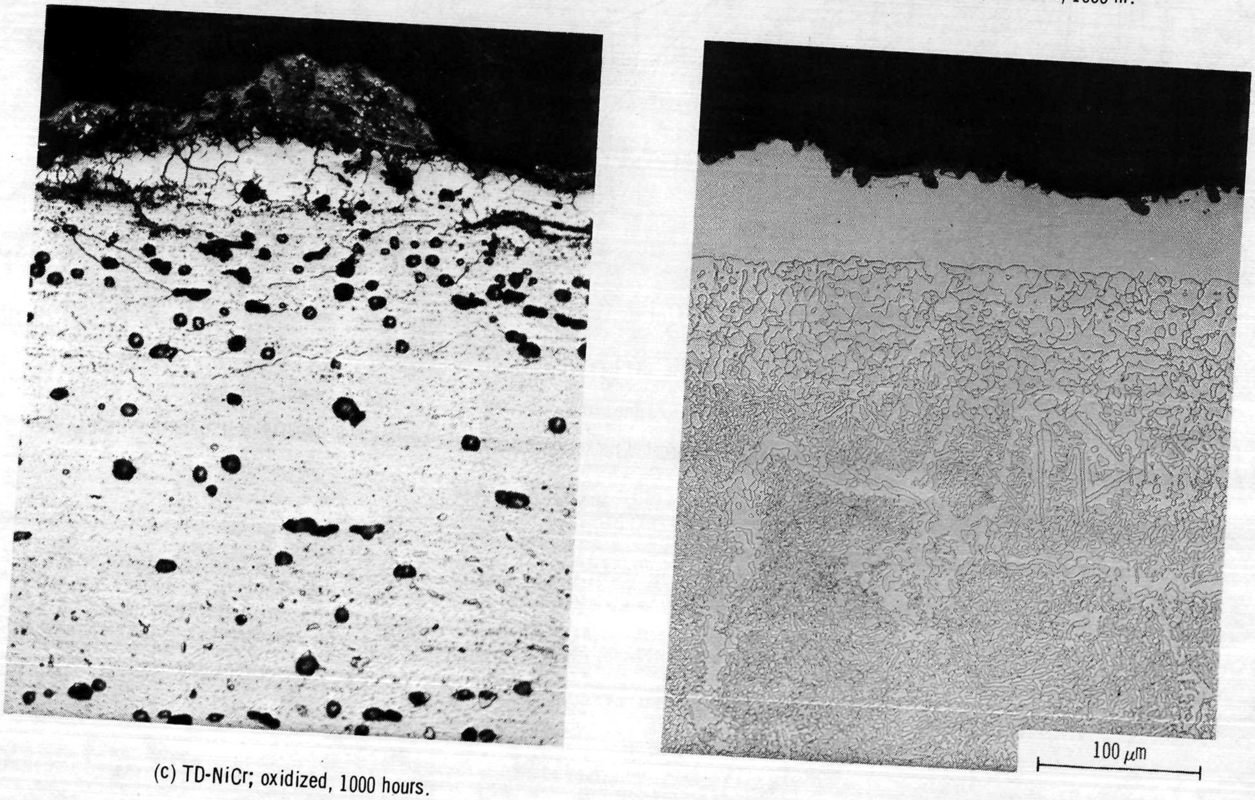


Figure 8. - Furnace oxidation of several Al-60Si coated nickel-base superalloys. Oxidized at 1100° C using 1-hour cycles.



(a) TAZ 8; oxidized, 1000 hr.

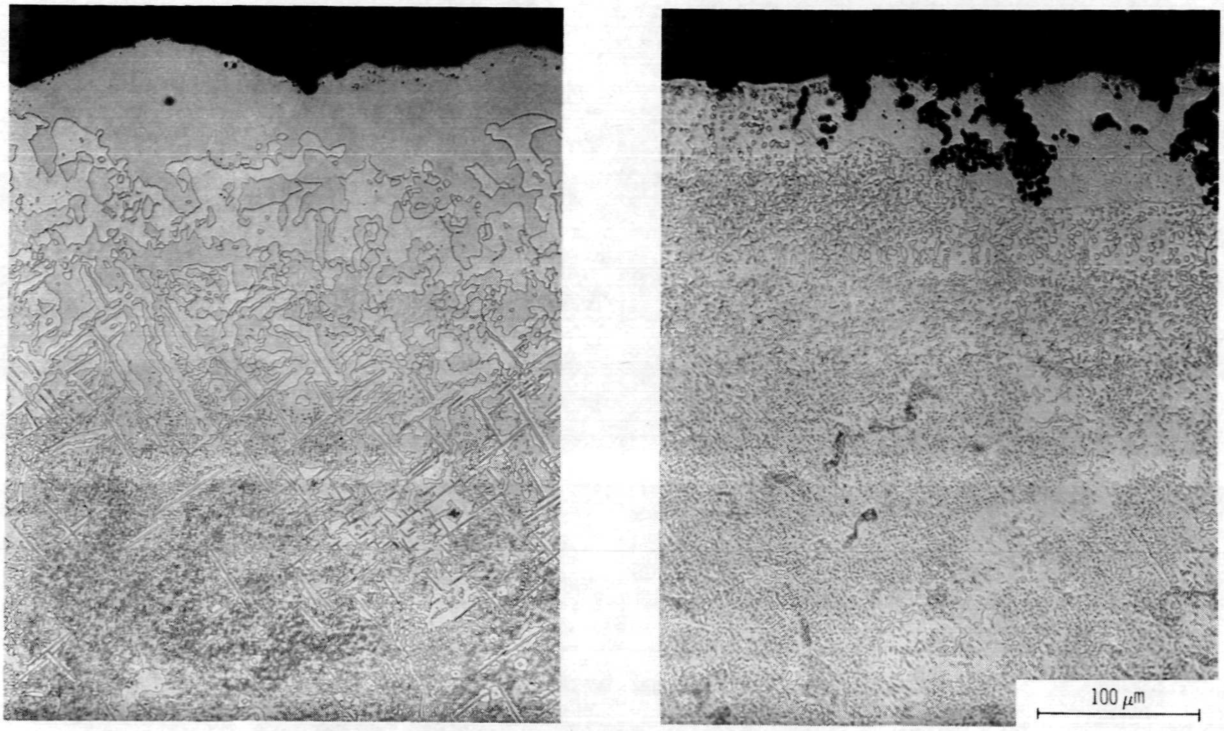
(b) NASA - TRW VI A; oxidized, 1000 hr.



(c) TD-NiCr; oxidized, 1000 hours.

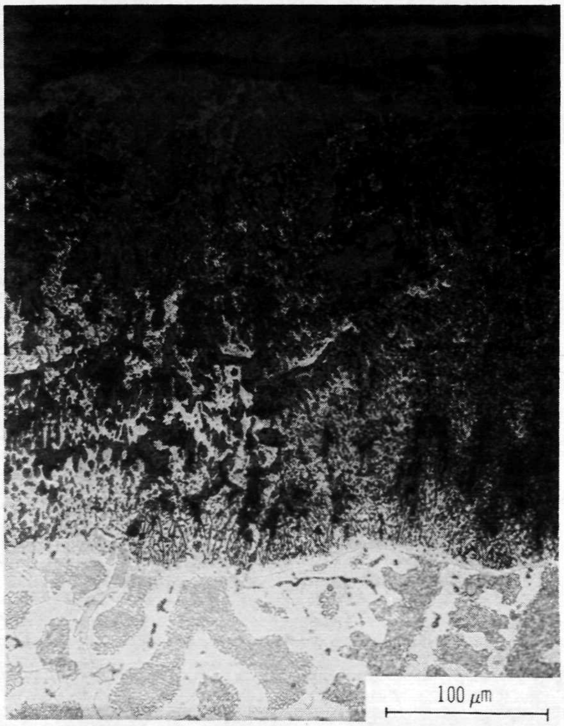
(d) B 1900; oxidized, 1000 hours.

Figure 9. - Structures of fused Al-60Si coatings (7 mg/cm^2) on selected superalloys after cyclic oxidation at 1100° C, etched. X250.



(e) MAR-M200; oxidized, 679 hours.

(f) NX 188; oxidized, 126 hours.



(g) WAZ 20; oxidized, 108 hours.

Figure 9. - Concluded.

> *"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."*
>
> —NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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