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**AN EXPERIMENTAL, LOW-COST,
SILICON-ALUMINIDE HIGH-TEMPERATURE
COATING FOR SUPERALLOYS**

**(NASA-TM-81455) AN EXPERIMENTAL, LOW-COST,
SILICON-ALUMINIDE HIGH-TEMPERATURE COATING
FOR SUPERALLOYS (NASA) 16 p HC A02/NF A01
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AN EXPERIMENTAL, LOW-COST, SILICON-ALUMINIDE
HIGH TEMPERATURE COATING FOR SUPERALLOYS*

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INTRODUCTION

E-385 The efficiency of gas-turbine engines increases as operating temperatures increase. However, the temperature is restricted by design limitations, material strength, and environmental capabilities. For metal temperatures above 900° C, nickel-and-cobalt-base superalloy components usually must be coated to resist oxidation and hot corrosion. At the Lewis Research Center there is a continuing effort to improve conventional aluminide coatings and to develop new, alternative coatings (refs. 1 to 5). These systems form an adherent Al₂O₃ film which protects the substrate from further oxidation.

Refractory metals and alloys developed for much higher temperatures than superalloys can be protected by fused-silicide coatings (refs. 6 and 7). These alloys are generally protected by M₅Si₃ and MSi₂ compounds, which primarily form SiO₂ upon oxidation (ref. 8). Little is known, however, about the oxidation of NiSi alloys as coatings compared with the MAI and MCrAlY coating alloys. Even less is known about the complex combinations of oxides, intermetallic compounds, and eutectics that could result from silicide/aluminide

* Based on NASA Technical Memorandum 79178, An Experimental, Low-Cost, Silicon SLURRY/Aluminide High-Temperature Coating for Superalloys by Stanley H. Young and Daniel L. Deadmore.

and $\text{SiO}_2/\text{Al}_2\text{O}_3$ combined with the nine or more other elements contained in many superalloys.

Most available information indicates that small amounts of silicon have a favorable effect on the oxidation resistance of iron and nickel-base alloys (refs. 9 to 12). But different results are obtained with different materials (refs. 8, 13). It appears that silicon in a coating must be tailored for use with the particular substrate to be beneficial. Unpublished cyclic furnace oxidation data (by D. Deadmore of Lewis) showed that a high-purity silicon-slurry spray followed by a pack aluminizing treatment greatly improved the high temperature oxidation resistance of NASA VIA alloy.

The purpose of this investigation was to evaluate the Si-Al coating system in severe environments that more closely simulate gas-turbine engines and to compare it with previously tested or in-service coating systems. The Si-Al coating system, if successful, provides a large potential for cost reduction.

EXPERIMENTAL PROCEDURES

The nominal compositions of the substrate alloys (in wt%) are for VIA, 6 Cr, 7 Co, 5 Al, 2 Mo, 1 Ti, 6 W, 9 Ta, 0.5 Hf, 0.5 Nb, 0.4 Re, balance Ni; and for B-1900, 8 Cr, 10 Co, 6 Al, 6 Mo, 1 Ti, 0.1 W, 4 Ta, 0.1 C, 0.1 Zr, 0.015 B, balance Ni.

The two specimen configurations used in this investigation are shown in figure 1. Specimens were grit-blasted and cleaned before coating. Silicon powder was sprayed onto the superalloy specimens, they were air dried, and

then pack aluminized at 1100°C for 16 hours under argon. A more complete description of the process is given in reference 15.

Oxidation and thermal fatigue specimens were run in a natural-gas burner with a gas velocity of Mach 1. A detailed description of the burner facility is given in references 16 and 17. In each test cycle the specimens were rotated in the hot gas for 1 hour at a metal temperature of 1093°C , and cooled to room temperature by lowering into a high-velocity air blast for 3 minutes.

The hot-corrosion specimens were run in a smaller facility operating on a Jet A fuel with a gas velocity of Mach 0.3. Five ppm salt was added to the combustion gases. The specimens were rotated in the gas flame for 1 hour at 900°C and then cooled to near room temperature in 3 minutes. At intervals of 20 or 50 hours of testing the specimens were weighed, photographed, and inspected for cracks. Each test was run with duplicate specimens.

After burner tests were complete, as determined from the appearance of the specimen or weight loss rate, the specimens were inspected metallographically. Samples of surface oxide were scraped from selected specimens for X-ray diffraction analyses. Microprobe analyses were conducted on the coated VIA specimens before and after exposure to determine the extent of elemental diffusion and to indicate elements present in the metallic and oxide forms.

OXIDATION AND THERMAL FATIGUE RESULTS

The results of the Mach 1 burner test on coated and uncoated specimens are shown in figure 2. The curves drawn through actual points are for the

Si-Al coating data of this paper. The other curves are data from previous tests in the same facility, under the same conditions. Specimens were removed from testing after a weight change of -50 mg or if a thermal-fatigue crack had propagated approximately 1/3 of the way through the specimen.

From figure 2(a) the Si-Al coated specimens have lives (determined as the zero crossover point on the x-axis) of from 675 to 960 hours, nearly 4 times the life of commercial pack-aluminide specimens tested under the same conditions. The uncoated specimen shows rapid weight loss. The Pt-Al specimen had an extrapolated life approximately twice that of the Si-Al coated VIA specimen.

When the Si-Al coating was applied to B-1900 a burner life of one of the specimens surpassed the extrapolated life of the Pt-Al on VIA system by 100 hours. The Si-Al coated B-1900 specimen ran >2100 hours before removal. No thermal fatigue cracks were observed until after 800 hours (200 hours longer than for the Pt-Al specimen). These tests clearly demonstrated the potential of the Si-Al coating applied to alloys that form favorable combinations with this protective coating.

For the B-1900 substrate the specimen which ran the longest (2129 hr) looked remarkably good even though it was cracked and had lost >200 mg. A fine fissure was observed on the trailing edge after 800 hours which lengthened throughout the test. Other cracks appeared later, in both the leading and trailing edges. For the other B-1900 specimen the coating appeared to erode over a larger area of the leading edge before cracks appeared.

Photomicrographs of the as-coated specimens and cross sections of the as-tested specimens of the Si-Al on VIA alloy are shown in figure 3, and electron microprobe analyses were made on these specimens.

Two distinct layers were observed above the as-coated VIA substrate. The inner layer is what remains of the original silicon layer after the 16-hour pack treatment. This layer is a multiphase region of small elongated grains, high in Ni, and Si, with varying amounts of the other elements. Silicon diffusion into the base metal was less than expected, but Si diffusion outward was significant. The top layer was mostly NiAl, with nodules of the Si-rich phase. The surface texture consisted of rounded grains with an estimated average diameter of 100 μm .

After exposure, the surface became smoother and the microstructure of the coating appeared entirely different. Layers were not as distinct, and larger grains of discontinuous phases were present. Silicon diffused about 200 μm into the substrate. Two distinct phases were observed in the originally Si-rich zones close to the substrate. The continuous phase is rich in Si, Ti, W, Cr, Mo, and Ta. Above this region a large-grained phase, rich in Cr, Si, Ni, Al, and Co was observed. This phase may be a form of low-Al martensitic B-NiAl as described in reference 8. At the surface a metallic phase rich in Ni, Al, Ta, W, and Ti (light region) was present. This is believed to be either Ni solid solution or Ni_3Al . In the oxides, Mo, Ta, Si, and Al were detected. X-ray analysis of the surface indicated Al_2O_3 , Ni solid solution, spinel, and other unidentified phases.

It appears that Si, combined with Ta and Mo from the substrate, diffused into the outer Ni and Al-rich phases and possibly formed protective oxides in addition to Al_2O_3 . The protective ability of this coating seems to be highly sensitive to the elements in the substrate. Small amounts of Si in the alloy have been shown to increase the oxidation resistance of B-1900 (ref. 18) and Ni_3Al (ref. 19). Further analysis is needed to more clearly identify specific phases, but the complexity and high number of elements makes this very difficult.

HOT-CORROSION RESULTS

The results of the hot-corrosion tests on bare and Si-Al coated B-1900 are shown in figure 4. Specimens were subjected to exposure to Mach 0.3 combustion gases containing 5 ppm sea salt and were cycled between $900^{\circ}C$ and room temperature. The coating protected B-1900 for more than 500 hours compared to less than 50 hours for the uncoated specimens.

In the coated specimen blisters formed after 345 hours. Hot corrosion occurred at these spots and made deep holes in the specimens by 779 hours.

SUMMARY OF RESULTS

(1) In Mach 1, $1093^{\circ}C$ cyclic oxidation the Si-Al coatings protected the VIA alloy at least four times longer than conventional aluminide coatings and provided about the same thermal fatigue protection.

(2) One Si-Al coating on B-1900 exceeded the oxidation and thermal fatigue performance of more expensive Pt-Al coatings on VIA and significantly

surpassed PVD CoCrAlY on VIA in oxidation and thermal fatigue.

(3) The Si-Al greatly improved B-1900's resistance to hot corrosion.

(4) The Si-Al coating failure in the oxidation tests started at a small pit in the coating followed by a thermal fatigue crack at that spot, which propagated to allow an expanded area of base metal to be exposed to oxidation.

(5) The Si-Al failure in hot-corrosion started with a blister which formed in the coating exposing the base metal. Rapid corrosion and oxidation of the base metal masked any other effects.

(6) Preliminary microstructural, X-ray, and electron microprobe studies after oxidation tests indicated that the high Si phase, which also contained high amounts of Ta and Mo from the substrate, supplied a combination of these elements to the original NiAl and/or Ni solid solution at the surface to allow combinations of oxides of Si, Ta, and Mo, to form in addition to Al_2O_3 . The protective ability of this coating appears to be highly sensitive to the elements contained in the substrate.

REFERENCES

1. Deadmore, Daniel L.: High Velocity-Oxidation Performance of Metal-Chromium-Aluminum (MCrAl), Cermet, and Modified Aluminide Coatings on IN-100 and Type VIA Alloys at 1093^o C. NASA TN D-7530, 1974.
2. Talboom, F. P.; Elam, R. C.; and Wilson, L. W.: Evaluation of Advanced Superalloy Protection Systems. (PW/ -4055, Pratt & Whitney Aircraft, NASA Contract NAS3-12415.) NASA CR-72813, 1970.

3. Levine, Stanley R.: Cyclic Furnace and High-Velocity Oxidation of an Aluminide-Coated High Strength Nickel Alloy (B-1900). NASA TM X-2370, 1971.
4. Gedwill, M. A.; and Grisaffe, S. J.: Oxidation Resistant Claddings for Superalloys. Met. Eng. Q., vol. 12, May 1972, pp. 55-61.
5. Freche, John C.; and Ault, G. Mervin: Progress in Advanced High Temperature Materials and Coating Technology. NASA TM X-73628, 1977.
6. Priceman, Seymour; and Kubick, Richard: Development of Protective Coatings for Columbium Alloy Gas Turbine Blades. Final Tech. Rep. 1 July 1969-30 July 1971, Sylvania Electric Products, Inc., June 1972, p. 33, (AFML-TR-71-172, AD-748837.)
7. Packer, C. M.; and Perkins, R. A.: Modified Fused Silicide Coatings for Tantalum (Ta-10W) Reentry Heat Shields. (D316824, Lockheed Missiles and Space Co.; NASA Contract NAS3-14316.) NASA CR-120877, 1973.
8. Smialek, James L.: Fused Silicon-Rich Coatings for Superalloys. NASA TM X-3001, 1974.
9. Zemskov, G. V.; et al.: Effect of Silicon Additions on the Oxidation Resistance of Aluminided Layers on a Nickel-Chromium Alloy. Zashchitnye Pokrytiia na Metallakh, No. 4, 1971, pp. 151-158. Protective Coatings on Metals. Vol. 4, G. V. Samsonov, ed. (Z. S. Michalewicz, transl.) Consultant Bureau, 1972, pp. 110-113.
10. Moore, V. S.; Bretnall, W. D.; and Stetson, A. R.: Evaluation of Coatings for Cobalt- and Nickel-Base Superalloys, Vol. 2, Aluminide Protective Coatings for Nickel and Cobalt Alloys. (RDR-1474-3, Solar; NASA Contract NAS3-9401.) NASA CR-72714, 1970.

11. Crouch, A. G.; and Dooley, R. B.: The Mechanical Integrity and Protective Performance of Silica Coatings. *Corros. Sci.*, vol. 16, no. 6, June 1976, pp. 341-347.
12. Lavendel, H. W.: Corrosion-Resistant Aluminide Coatings for Iron- and Nickel-Base Alloys. *J. Metals*, vol. 27, July 1965, pp. 4-10.
13. Fiedler, H. C.; and Sieraski, R. J.: Aluminide Coatings for Nickel-Base Alloys. (SRD-72-028, General Electric Co.; NASA Contract NAS3-14300.) NASA CR-120871, 1971.
14. Johnston, James R.; and Young, Stanley G.: Oxidation and Thermal Fatigue of Coated and Uncoated NX-188 Nickel-Base Alloy in a High-Velocity Gas Stream. NASA TN D-6795, 1972.
15. Young, Stanley G.; and Deadmore, Daniel L.: An Experimental, Low-Cost, Silicon-Slurry/Aluminide High-Temperature Coating for Superalloys. NASA TM-79178, 1979.
16. Deadmore, Daniel L.: Cyclic Oxidation of Cobalt-Chromium-Aluminum-Yttrium and Aluminide Coatings on IN-100 and VIA Alloys in High Velocity Gases. NASA TN D-6842, 1972.
17. Johnston, James R.; and Ashbrook, Richard L.: Oxidation and Thermal Fatigue Cracking of Nickel- and Cobalt-Base Alloys in a High-Velocity Gas Stream. NASA TN D-5376, 1969.
18. Miner, Robert V., Jr.; and Lowell, Carl E.: Effects of Silicon Additions on Oxidation and Mechanical Behavior of the Nickel-Base Superalloy B-1900. NASA TN D-7989, 1975.

19. Lowell, Carl E.; and Santoro, Gilbert J.: The 1200° C Cyclic Oxidation Behavior of Two Nickel-Aluminum Alloys (Ni₃Al and NiAl) with Additions of Chromium, Silicon, and Titanium. NASA TN D-6836, 1972.

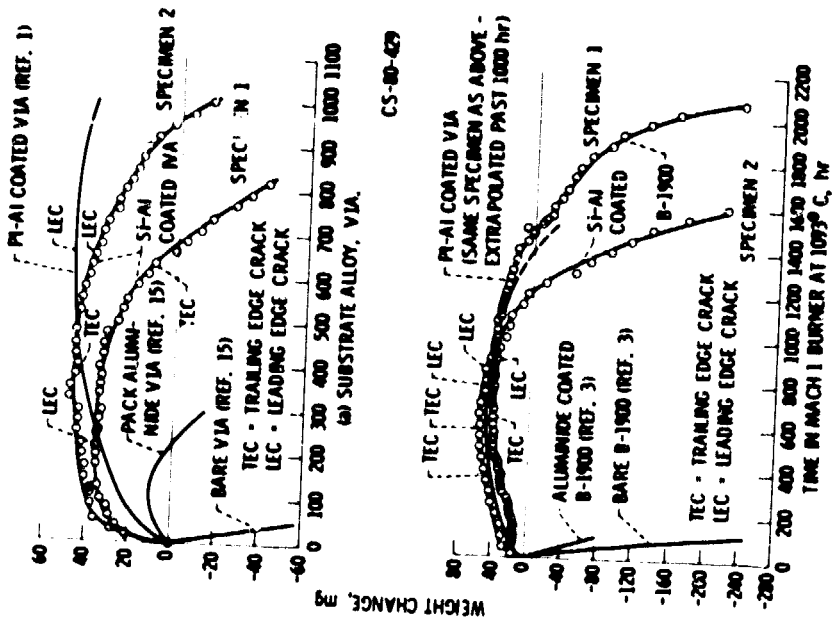


Figure 2 - Comparison of bare and coated alloy Cr-gas turbine high-gas velocity (Mech 1) cyclic oxidation.

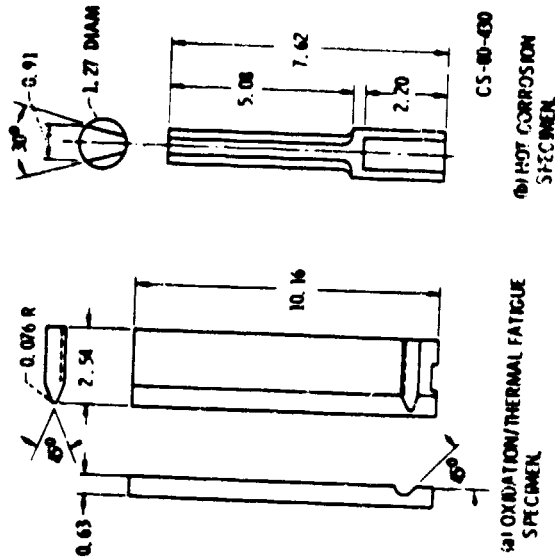
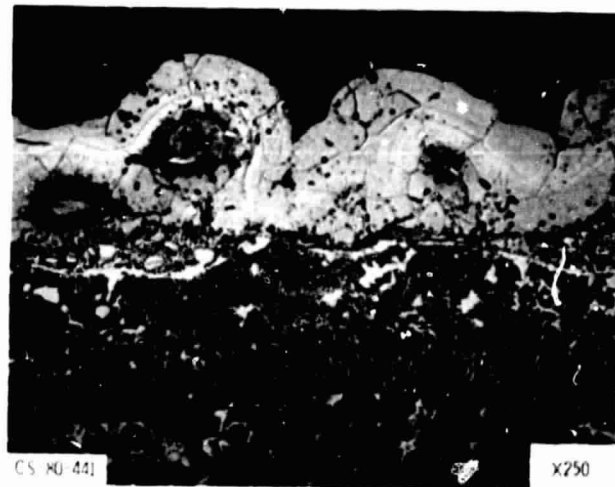
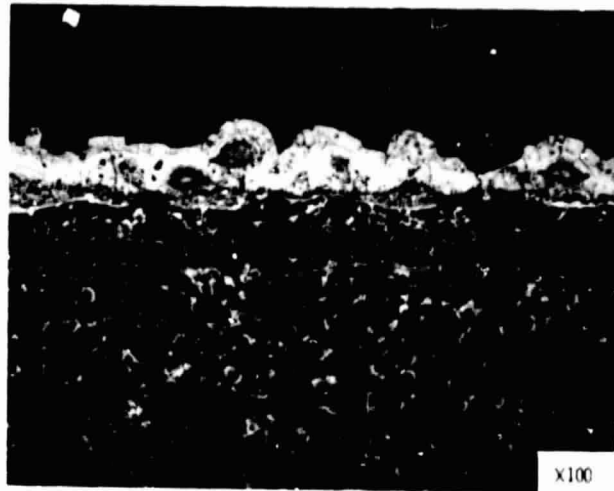


Figure 1 - High-gas-velocity test specimens. (Dimensions are in cm.)

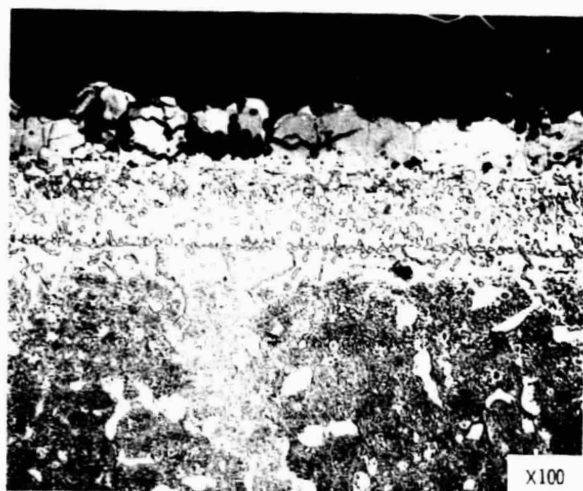
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(a) AS-COATED.

Figure 3. - Microstructure of Si-Al coated V1A alloy before and after testing in Mech 1 burner at 1093° C.

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(b) AFTER 1000, 1 hour CYCLES IN MACH 1 BURNER AT 1093° C.

Figure 3. - Concluded.

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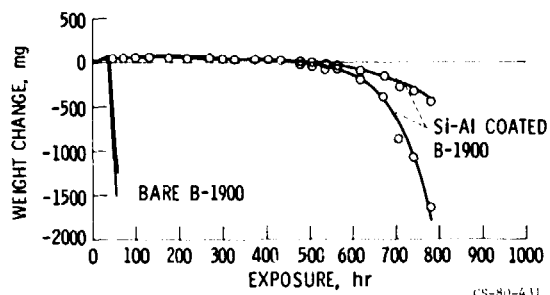


Figure 4. - Bare and coated B-1900 after Mach 0.3 hot-corrosion tests at 900° C for 1 hr in 5 ppm sea salt.

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