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COATING DEVELOPMENT FOR COLUMBIUM AND TANTALUM ALLOYS

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Oxidation protection of columbium and tantalum alloys by Cr-Ti-Si coatings was the subject of a four-phase program at IRW. (1) Phase I was a study of basic factors associated with the diffusion formation and oxidation protection of vacuum pack Cr-Ti and Cr-Ti-Si coatings on unalloyed columbium, D-43, and B-66 alloys. As-coated specimens representing various stages in diffusion formation were analyzed. Coatings were studied after various oxidation exposures at pressures of 0.01 torr to 1 atm and temperatures up to 2700 F. An evaluation of the specimens showed that, during the chromium-titanium coating cycle, the diffusion zone and scale formed at equivalent rates. The latter was a laves phase of the form (Cb,Ti)Cr<sub>2</sub> with a face-centered cubic MgCu<sub>2</sub> type structure, containing some beta titanium, especially in the case of the D-43 alloy. The diffusion zone contained chromium and titanium in solid solution. Siliconizing the coating resulted in the formation of a graded disilicide of the form (Cb,Cr,Ti...)Si<sub>2</sub>. Beneath this layer were subsilicide bands of the form (Cb,Cr,Ti...)Si<sub>2</sub> and (Cb,Cr,Ti...)Si<sub>3</sub>. Some silicon was also present in the diffusion zone. During oxidation, chromium and titanium migrated rapidly to the coating surface. During initial exposure, the major surface oxide was Cr<sub>2</sub>O<sub>3</sub>. Chromium subsequently vaporized to leave an oxide rich in titanium and silicon, and ultimately columbium. At reduced pressure, silicon vaporized as SiO. The study showed that three factors contributed to eventual coating failure: (1) loss of chromium, (2) enrichment of the protective oxide with columbium, and (3) depletion of chromium and titanium from the diffusion zone.

The Phase II portion of the program resulted in a laboratory-scale development of a spray-diffusion process capable of producing Cr-Ti-Si coatings comparable both in microstructure and in protective properties to vacuum-pack coatings. The process consists of two cycles and uses the spray deposition of a halide-activated metal slurry on the substrate. Each cycle was followed by diffusion anneals under 150 torr argon pressure.

In Phase III of the program, two concepts were explored for protection of tantalum, D-43, B-66, and Ta-10W alloys at 2700 to 3500 F. These were (1) alloy modification of existing Cr-Ti-Si coating and (2) the barrier layer approach. Alloying the Cr-Ti-Si coating with molybdenum and tungsten was generally unsuccessful. The potential of the barrier layer coating system was well demonstrated as tungsten/tungsten-silicon gave protective lives of 2 to 3 hours at 3400 to 3500 F.

Metallographic, oxidation, tensile, and creep studies of the various coating-substrate systems comprised the final phase. Mechanical-property evaluations indicated little difference between the vacuum-pack and slurry-coated columbium alloys.

Sylcor is continuing studies of fused-slurry silicide coatings for protecting D-43 alloy sheet. On the basis of 1-hour cyclic oxidation tests (between 800 and 2500 F) in air, coating compositions were optimized from 25 compositions chosen from the Cr-Ti-Fe-V-Si system. Optimum compositions found were Si-20Cr-20Fe-10VSi<sub>2</sub> and Si-20Cr-20Fe, which respectively survived about 30 and 50 cycles at 99 percent reliability. Several compositions were characterized using x-ray diffraction and electron microprobe analysis and by testing for wettability, faying surface penetration, and coating repair. The feasibility of using fused silicides as a one-step coating braze was demonstrated. Use of Si-20Cr-5Ti resulted in shear strength of about 5000 psi at temperatures up to 2500 F and about 2000 psi at 3000 F. Preliminary evaluations were made of the utility of fused silicide coatings for protecting mechanical fasteners, gas-turbine components, and T-222 alloy sheet.

Iridium-base alloys have been explored by Solar for use on tantalum alloys at temperatures above 3500 F. (4) In coating-alloy development, Ir-30Rh had the lowest surface recession rate, 1 to 4 mils/hour in plasma arc tests at 3850 F. Addition of 15 Os, 5ThO<sub>2</sub>-5Ta<sub>2</sub>O<sub>5</sub>, 10HfO<sub>2</sub>, 10SiO<sub>2</sub>, 10Al<sub>2</sub>O<sub>3</sub>, 10ThO<sub>2</sub>, 10MgO\* to iridium by arc melting or by powder metallurgy resulted in no improvement. In other work, iridium coatings were electrodeposited on 125-mil-diameter tungsten rod, and an Ir-30Rh coating was prepared by alternate deposition of iridium from fused salt and rhodium from an aqueous bath. Pin holes that developed in both the iridium and Ir-30Rh coatings caused premature failure during self-resistance heating in air above 3500 F. In other studies, cursory attempts to use gold as a fugitive solvent or HfO<sub>2</sub> for emittance enhancement were not successful.

Protective coatings for tantalum alloys in hydrocarbon and halogen combustion environments is the subject of an IIT Research Institute program. (5) Emphasis was placed on hafnium-tantalum and composites based on HfO<sub>2</sub>. Hf-20Ta clad Ta-10W nozzles and throat inserts for rocket engines were prepared by hot-spinning preclad plate. Results with the use of tantalum or columbium diffusion barriers appeared inconclusive. Investigation of spinning variables demonstrated that the system was promising for use near 4000 F. Some of the hardware was converted to an in situ composite of Ta-HfO<sub>2</sub> by controlled oxidation. \*Oxide compositions given in volume percent.

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oxidation of the hafnium-tantalum alloy. The influence of temperature and effective oxygen pressure on the size and distribution of microconstituents was found.

Texas Instruments is investigating the chemical vapor deposition process for coating refractory metals and has demonstrated the feasibility of coating B-66 alloy with Cr-Ti-Si of controlled composition.<sup>(6)</sup> One process utilized a two-stage deposition in which the metal alloy chromium-titanium coating was followed by silicon deposition. A second method involved simultaneous deposition of all three elements in 10 minutes. The effects of process variables were evaluated. On the basis of air oxidation tests at 2300 and 2500 F, the performance of the coatings appeared comparable to the Cr-Ti-Si types produced by other methods. Scale-up to larger and more complex shapes were recommended.

Electrophoretic coatings for Ta-10W alloy have been investigated by Vitro.<sup>(7)</sup> Coatings of  $ZrB_2$ ,  $ZrB_2-10MoSi_2$ , and  $TaAl_3$  were unsatisfactory because of interdiffusions and substrate embrittlement on sintering at 3100 F. Satisfactory adherence and minimum contamination were obtained with  $TaSi_2$ ,  $MoSi_2$ , and  $WSi_2$  when the average particle size was less than  $10\mu$ . Then, a dense graded binary oxide overlayer was applied. A typical oxidation life of 15 minutes at 3500 F in air was obtained with  $WSi_2/Al_2O_3-4MgO$ ,  $WSi_2/ThO_2$ ,  $WSi_2/ZrO_2-21O_2$ , and  $WSi_2/MgO$  coatings. Failure was due to edge and corner defects, or to interaction with the setter material.

In a companion study, Vitro<sup>(8)</sup> investigated other coatings for protecting T-222 alloy. On the basis of cyclic oxidation tests from room temperature to 1500 or 2400 F, Hf-27Ta,  $TaAl_3$ ,  $ZrB_2$ , and  $ReSi_2$  coatings were eliminated. Also, tungsten and rhenium barrier layers were eliminated because of silicide formation during sintering. Effort was concentrated on single-phase and binary silicides. When post-silicided and preoxidized, the coatings  $WSi_2$ ,  $MoSi_2$ ,  $MoSi_2-3VSi_2$ , and  $MoSi_2-3TiSi_2$  behaved erratically and were susceptible to pest failure at 1500 F. The concentration of second phases,  $VSi_2$ ,  $CrSi_2$ , and  $TiSi_2$ , was increased to 10 to 30 percent.  $MoSi_2-30VSi_2$  gave the best performance. Two specimens survived 600 hours (including 30 cycles to room temperature) at 1500 F and at 2500 F without failure, while others failed at 12 to 100 hours.  $WSi_2-30VSi_2$  behaved well at 2400 F, but failed at 1500 F.

Protection offered by a series of coatings on Cb-1Zr alloy was evaluated in 4 cubic feet per hour (STP) argon (containing 1 to 3 ppm oxygen and  $<10$  ppm  $H_2O$  for 24 hours, then 1 to 3 ppm) at 2000 F for 500 hours.<sup>(9)</sup> The coatings consisted of Sylcor Sn-Al, Chromizing Corp. Durak KA, Pfaudler single- and double-cycle silicides, Vitro Cr-Mo-Si, and MoSi with a 5-mil-thick molybdenum barrier layer. Evaluation was based on metallography, hardness, bend tests, and electron-microprobe analysis. The most protective coating appeared to be  $MoSi_2/Mo$ . Tin-aluminum offered adequate oxidation resistance despite spalling. The other coatings were less satisfactory since Cr-Mo-Si allowed chromium diffusion into the substrate, and the Pfaudler and chromizing coatings permitted some substrate oxidation.

## COATING DEVELOPMENT FOR TUNGSTEN

Iridium-base coatings for tungsten have been studied by the IIT Research Institute.<sup>(5)</sup> In consideration of phase relations, rhenium was chosen as the most suitable barrier to raise the minimum melting point in the system. Interdiffusion studies indicated that in the absence of surface diffusion effects, about 3 mils rhenium effectively limited interdiffusion for 1 hour at 4000 F. Satisfactory iridium coatings were deposited from a slurry using copper as a fugitive vehicle.

A model was developed for the determination of temperature-dependent stresses in tungsten nozzles that were plasma sprayed with  $HfO_2$ -tungsten. Tensile properties, elastic moduli, and thermal expansion were determined for the sprayed materials. Residual stress measurements on as-fabricated multi-layer nozzles were in reasonable agreement with predicted values.

## COATING DEVELOPMENT FOR CHROMIUM ALLOYS

Silicide,<sup>(10)</sup> aluminide,<sup>(11)</sup> and nickel-chromium<sup>(12)</sup> coatings for chromium alloys for use up to 2400 F in air are being studied under NASA sponsorship. In each of these programs, a Cr-5W-0.1Y alloy is used as the substrate.

Solar<sup>(10)</sup> investigated silicides as well as coatings based on V-(Ti,Mo)Si. The latter intended to serve as an interstitial sink to control nitrogen ingress into the substrate. Vanadium was best applied by low-pressure pack cementation, titanium was deposited from nonelectrolytic fused-salt bath, molybdenum by chemical vapor depositions, and silicon from an argon high-pressure pack.

Cyclic oxidation tests on vanadium-silicon, vanadium-molybdenum-silicon, vanadium-titanium-silicon, and vanadium-titanium-molybdenum-silicon coatings showed the presence of vanadium and/or molybdenum beneath the silicide layer caused catastrophic failure at 2400 F not encountered in the uncoated alloy. In contrast, simple silicide coatings produced a chromium-rich disilicide which was protective for 500 hours at 2100 F and permitted no oxygen or nitrogen ingress to the substrate. It appeared that silicon acted as both a barrier layer and interstitial sink. Spalling reduced protection to about 100 hours at 2400 F.

Chromalloy<sup>(11)</sup> found that chromium-aluminum solid solution or duplex  $Cr_3Al_8$ -aluminum coatings applied by pack cementation offered static-oxidation protection for over 600 hours in air at 2100 F. The duplex coating was resistant for over 300 hours at 2400 F. However, as a result of a 100-hour exposure at either temperature, the bend transition of both systems was raised from 700 to over 1600 F. Modification of the aluminide with a spinel former such as iron gave promising oxidation resistance at 2100 F.

Battelle is investigating the use of thin films, applied by gas-pressure bonding, for the protection of the Cr-5W-0.1Y substrate.<sup>(12)</sup> The coatings primarily consisted of Ni-20Cr and Ni-20Cr-20W, but were modified with 3 to 5 percent aluminum in some cases. Intermediate platinum and/or tungsten layers were utilized to minimize harmful interdiffusion. Cyclic oxidation resistance of

these clads was over 200 hours at 2100 F in air, but at 2300 F, 200-hour oxidation protection was marginal. At 2400 F, rapid oxidation occurred. Cladding caused the bend transition of the coating-substrate system to rise from 700 to 1200 F. In most cases, the oxidation exposure further raised the transition to 1400 to 1600 F. Reasons for this degradation in system ductility are now being sought.

The use of platinum, palladium, and rhenium as nitrogen diffusion barriers for the Cr-5W-0.1Y substrate is being studied at NASA-Lewis.<sup>(13)</sup> On the basis of metallography, electrodeposits of palladium or platinum offered some protection from nitrogen at 2000 F. However, outward diffusion of chromium appeared high. Rhenium increased the sensitivity of the substrate to nitrification.

#### COATING DEVELOPMENT FOR ELECTRICAL INSULATORS

Westinghouse has started a program on the synthesis and study of coatings for electrically insulating refractory conductors at elevated temperatures.<sup>(14)</sup> Aluminum nitride has been deposited on molybdenum, tungsten, and graphite from the  $AlCl_3 \cdot NH_3$  vapor phase. The coating protected molybdenum from oxidation to at least 1300 F. Dielectric tests indicate that AlN on molybdenum had a d-c conductivity on the order of  $10^{-13}$  per ohm-cm at 77 F and increased to  $10^{-6}$  at 1470 F. The a-c properties depended on the method of preparation. The apparent dielectric constant increased markedly with increasing temperature indicating a high level of internal polarization which is probably due to limited mobility of charge carriers.

Pyrolytic  $Si_3N_4$  from  $SiF_2 \cdot 2NH_3$ ,  $SiCl_4 + NH_3$ , or  $SiH_4 + NH_3$  offered some oxidation protection for molybdenum at 1830 F.

#### PROPERTY STUDIES OF COATED REFRACTORY METALS

Microstructure. Identification of refractory metal coating and substrate constituents has been done before and after oxidation in the 0.01 torr to 1 atm pressure range by Sylcor.<sup>(15)</sup> Electron microprobe analyses were supplemented by x-ray diffraction and metallography on the following systems:

<u>Substrate Alloy</u>	<u>Silicide Coating</u>	<u>Temperature, F</u>
Cb, D-43, B-66	Si, Ti-Si, Cr-Si Cr-Ti-Si Ag-Al-Mo-Si	2000, 2500
Mo, TZM	Si, W-Si, Si-B	2500, 2800
Ta, T-222	Si, Ti-W-Si	2500, 2800
W	Si, Si-Al <sub>2</sub> O <sub>3</sub> , W-Si	2800, 3300

In general, it was found that addition of modifiers resulted in increased protection primarily by re-

ducing the concentration of substrate constituents in the coating. During oxidation, heavily alloyed coatings formed areas of  $M_2Si_2$  throughout the coating. Alpha cristobalite was the primary silica phase formed in all systems studied. Oxides tended to form as mixtures rather than discrete layers. No relation was found between oxidation, diffusion rate, disilicide coating phases, and protection. Other factors, such as expansion coefficients, thermal shock resistance, oxide mechanical properties, and self-healing mechanisms, may be controlling. In some cases anomalous intermediate-temperature oxidation behavior was noted and correlated with the presence of the substrate oxide. Exposure to reduced pressure (partial vacuum or impure argon) sometimes changed the oxides formed and their stoichiometry. In the case of tungsten- and molybdenum-alloy substrates where  $SiO_2$  was the primary oxide, oxide volatility was the primary mechanism of degradation and depletion of silicon.

Emissance. In a North American Aviation program, the role of emissance in refractory-metal-coating performance was reviewed and analyzed.<sup>(16)</sup> The purpose was to orient a comprehensive study to provide emissance data for thermal calculations needed in the design of aerospace systems. A number of preliminary emissance experiments were performed on TRW Cr-Ti-Si coated Cb-752, Chromalloy W-3 coated TZM molybdenum, and Sylcor U-n-aluminum coated Ta-10W in air at 2000 F. There was a marked increase in the total normal emissance to about 0.8, which for the silicide coatings was associated with oxide formation. The total normal emissance measured at 2000 F was equal to that calculated by integration of the spectral curve inferred from room temperature reflectance data. This similarity indicated that the shape of the spectral curve at 2000 F was not a strong function of temperature.

Ductility. The factors affecting the tensile ductility minimum behavior of silicide-coated Cb-10W and Cb-752 sheet in air, in the intermediate temperature range of 1300 to 2000 F, has been studied at Battelle.<sup>(17)</sup> In the case of the TRW Cr-Ti-Si coated alloys, contamination and embrittlement by air at the base of coating cracks was found to be the major factor responsible for loss of ductility, e.g., at a strain rate of 0.1 per minute, reductions in area dropped from about 70 percent for the uncoated alloys in vacuum to as low as 1 to 3 percent for the coated alloys in air. Apparent mechanical interaction between coating and substrate played a smaller role, but became more significant with decreasing substrate thickness from 100 to 30 mils. The Sylcor Ag-Al-Mo-Si liquid-phase coating gave somewhat improved ductility above 1380 F by filling cracks in the silicide phase. The presence of zirconium, dynamic strengthening of the substrate, and thickening of the subsilicide layer to 1 mil by annealing had relatively little effect on ductility.

#### SPACECRAFT APPLICATIONS

Pratt & Whitney is investigating the following coated columbium-alloy systems for use in gas turbines:<sup>(18)</sup>

<u>Application</u>	<u>Temperature Range, F</u>	<u>System</u>	<u>Coating</u>	<u>Substrate Alloy</u>
Vane	1800 to 2500	1	TRW Cr-Ti-Si (vacuum pack)	C-129Y
		2	TRW Cr-Ti-Si (vacuum pack)	D-43
		3	Sylcor Cr-Ti-Si (triplex pack)	D-43
		4	Sylcor Cr-Ti-Si (slurry)	D-43
Blade Airfoil	1800 to 2500	5	TRW Cr-Ti-Si (vacuum pack)	Cb-132M
		6	TRW Cr-Ti-Si (2-stage slurry)	Cb-132M
Blade Root	1300 to 1600	7	Sylcor Sn-Al	Cb-132M
		8	Sylcor Ag-Al-Mo-Si	Cb-132M
		9	Zinc	Cb-132M

Based on oxidation-erosion and thermal fatigue at 2200 to 2400 F, ballistic impact at 70 to 2200 F, and previous data, Systems 2 and 5 were selected for advanced evaluation. On the basis of prestrain/oxidation and wear galling tests, System 8 was also chosen. Complete evaluation of System 2, including stress rupture, indicated it as a candidate for engine testing. A partial evaluation of System 8 indicated adequate performance.

Various silicide-coated refractory-metal alloys have been exposed to environmental loading conditions such as an Apollo rocket engine would experience as a result of transient ignition pressure spikes.<sup>(19)</sup> The substrate/coating systems used were molybdenum/Chromizing Durak B, Ta-10W/Sylcor R512, C-103/Chromizing Durak KA, C-103/W+Sylcor Ag-Al-Mo-Si, and C-129Y/Durak KA. Tensile, bend, torch-fatigue, and room-temperature hydrodynamic chamber tests were conducted. Some tensile properties at strain rates of 0.0001 to 0.001 and 100 to 500 per second were determined for the uncoated and coated alloys with and without previous thermal cycling in vacuum or hydrogen. At the temperature of -100 F, room temperature, and 2500 F, the higher strain rate caused a 50 to 100 percent increase in ultimate strength with little change in elongation. Elongation was measured on cylindrical specimens with a 1/8-inch diameter, 1-inch long gage length. The data presented suggest that the coatings did not significantly affect the substrate properties. The columbium-alloy system with Durak KA demonstrated the best properties, even under high-strain-rate conditions. The other systems were less attractive; coated molybdenum was recrystallized and brittle at -100 F and room temperature, coated Ta-10W alloy had inferior bend ductility after fatigue-torch testing in addition to its high density, and the silver-aluminum alloy in the Ag-Al-Mo-Si coating ran off in chamber tests or vaporized in vacuum at 2500 F.

McDonnell has subjected uncoated Cb-752, T-111, and tungsten, in the form of 50-to-60-mil thick sheet, to a laboratory simulation of an ASSET boost-slide, re-entry vehicle, flight environment.<sup>(20)</sup> The metals were degraded by substrate contamination and the formation of gross oxide scales at 2 to 47 torr air pressures for 35 minutes above 1200 F with a maximum temperature of 2850 F. Tungsten was affected the least. Protection of Cb-752 alloy with chromium-titanium was incomplete. Minor defects, simulated by 1-mil-deep slots in Cr-Ti-Si coated Cb-752 alloy, did not affect the coating or the substrate. Major defects, simulated by 4-mil slots extending into the substrate, resulted in localized contamination on the order of that found in uncoated Cb-752 alloy.

Vitro and Standard Pressed Steel collaborated in a development of coated structural fasteners.<sup>(21)</sup> Systems primarily utilized were Ta-10W and T-222 alloys coated with electrophoretic Si-WSi<sub>2</sub>, and Cb-752 and C-129Y alloys coated with electrophoretic Cr-Ti-Si. The latter had properties comparable to the vacuum-pack coating. Tolerance control permitted complete interchangeability of parts with tolerances sufficient for precision aircraft fasteners. Threaded pieces could be removed and reused after air exposures up to 2200 F. Tightening torques were limited to about 50 inch-pounds without injury to the coating. Deformable fasteners could not be produced because of low coating ductility. Oxidation life of the coated columbium alloy fasteners was about 60 to 120 hours in static air and 2 to 4 hours in 200 ft/sec air at 2200 F. Coated C-129Y alloy was unaffected by exposures in air at pressures of 0.01 to 1 torr at 2200 and 2400 F for 5 hours, while Cb-752 alloy was degraded at both temperatures. The tantalum alloys showed considerable degradation of coating at pressures of 0.01 and 1 torr as low as 2200 F. Reduced pressures appeared to remove beneficial effects of preoxidation and result in a 2400 F static oxidation life of 2.8 hours. Dynamic oxidation life was about 2 hours at 2400 F.

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