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High Temperature Oxidation
Resistant Coatings for
Tantalum Base Alloys

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FOREWORD

This First Quarterly Progress Report was prepared by the Metallurgy Department of Sylvania-Corning Nuclear Corporation in accordance with the provisions of USAF Contract AF 33 (616)-7462 under the direction of the Materials Laboratory, Wright Air Development Division, with Lieutenant N. M. Geyer acting as project engineer.

This quarterly report summarizes the development effort undertaken on this contract from 1 June to 31 August 1960.

L. Sama and D. D. Lawthers cooperated in this experimental work. The aluminum coating phase was performed by G. T. Pepino, Jr., beryllium coating phase by D. Melone, metallography by H. Woods, hardness testing by O. Haines.

ABSTRACT

The effect of various dipping times, temperatures, and diffusion treatments has been determined for tantalum sheet dipped in aluminum alloy baths.

Both aluminide and beryllide coatings have been produced that will withstand oxidation for 10 hours at 2500°F, isothermally and cyclic.

Aluminide coatings have been obtained on a stressed columbium alloy that meet the same test conditions.

Section 1 Introduction

The purpose of this work is to conduct a developmental effort on coatings for tantalum base alloys for protection against high temperature oxidation. The general application is to develop coatings on 0.010" thick sheet for protecting tantalum base alloys from high temperature, 2500°F and above, oxidation as might occur in structural applications in hypersonic or space-flight vehicles.

The program is intended to approach coating development from a fundamental viewpoint. Since tantalum alloys are contemplated for use in a variety of applications, coatings are not to be developed for any specific utilization, but rather to be sufficiently characterized to permit evaluation for the several potential applications. The capabilities of all experimental coatings will be determined by subjecting coated tantalum specimens to at least the following evaluation tests:

- A. Continuous isothermal exposure in air at temperatures from 2500° to 2800°F for a minimum of 10 hours.
- B. Withstand at least 10 cycles during 10 hours in an air blast between test temperature (2500° to 2800°F) and ambient.
- C. The coating must withstand 2% deformation at temperature without oxidation failure.

In addition successful coating systems will be tested on columbium alloys under the same testing conditions but effort will be to a much lesser degree.

The specific approach to be employed in developing oxidation resistant coatings will be to base such coating on intermetallic compounds of tantalum, such as aluminides and beryllides.

All coating development will be performed on unannealed 0.010" thick sheet. Three alloys will be evaluated: (1) pure tantalum as obtained from Fansteel, (2) tantalum plus 10% tungsten alloy, and (3) a high strength tantalum base alloy to be introduced later.

Section 2 Aluminum Coated Tantalum

The first step in the program is to investigate the formation of tantalum aluminide by hot dipping in molten aluminum and aluminum base alloys with and without subsequent reaction-diffusion treatment at elevated temperatures in inert atmospheres. Also to be investigated will be pack-calorizing techniques.

To determine the effect of dipping variables on coatings the following program was established:

- A. Dip in molten aluminum and aluminum alloys at 1700° and 2100°F for 1, 2, and 5 minutes followed by
- B. A vacuum diffusion, or
- C. A pack calorizing heat treatment in Al or Al alloy plus Al_2O_3 powders at 1900° and 2200°F for 1 and 4 hours.

All results on pure tantalum were obtained using annealed sheet. The Ta-7 o/o W sheet was obtained by arc-melting, cold working, recrystallization anneal, and cold rolling to 0.010" thick sheet, all at Sylcor. The test coupons were 0.5" x 0.75" x 0.010".

Samples were dipped in four baths: G3 pure aluminum, G4 Al-11 o/o Si, G5 Al-10 o/o Si-10 o/o Cr, G6 Al-25 o/o Si. The effect of test variables on coating thickness and solution of the tantalum core is given in Table I. All measurements were made on metallographic samples. The original thickness of the tantalum sheet is 9.7 mils (0.0097"). The following statements can be made based on these results:

- D. Using an aluminum bath, varying dipping time and temperature has no effect on the thickness of coating, which is one mil per side, but the tantalum core is dissolved away rapidly with increasing time and temperature.
- E. A vacuum heat treatment cannot be used on dipped tantalum specimens because of the rapid vaporization of free aluminum before the intermetallic tantalum aluminide is formed.

F. Therefore, a pack calorizing heat treatment in argon, for example, must be used in order to form tantalum aluminide ($TaAl_3$) on the surface. Pack calorizing is placing the dipped sample in a powder mixture of 10% Al balance Al_2O_3 , heating to some temperature, say $1900^{\circ}F$, for 1 hour in an argon atmosphere to react and diffuse free aluminum to form $TaAl_3$ type compounds.

G. Dipping in Al-11 o/o Si results in a better coating than Al. The reaction rate is much faster than with Al and the growth rate of the aluminide coating is time and temperature dependent.

H. A 2 mil coating seems to be needed for oxidation resistance under the test conditions. More about this later.

Photomicrographs and hardness values for aluminum coated tantalum samples dipped for 1 minute at $1700^{\circ}F$ with various diffusion treatments are given in Figures 1 to 3. Ideally under equilibrium conditions there should be three intermetallic compounds in the Ta-Al system, $TaAl_3$ on the outer surface, then Ta_2Al and Ta_3Al adjacent to the tantalum core. The latter two compounds cannot be separated in the microstructure. A photomicrograph is shown taken under polarized light to show how optically active $TaAl_3$ is. It is known from other work that this optically active type compound must be present to confer elevated temperature oxidation resistance.¹ Microstructures using other dipping baths of aluminum alloys are very similar.

¹ "An Investigation of Intermetallic Compounds for Very High Temperature Applications", WADC TR 59-29, Part 1, January, 1960.

Section 3 Oxidation Results

Samples were oxidized at 2500°F in a furnace with slowly flowing air until failure or until they had withstood 10 hours at temperature. The results are listed in Table II. This preliminary work indicates that:

- A. A 1700°F dip is too low to confer oxidation resistance to tantalum when dipped in (a) pure Al, (b) Al-11 o/o Si, (c) bath composition A*, and (d) Al-25 o/o Si.
- B. Samples as dipped in pure aluminum at 2100°F without a diffusion heat treatment will withstand 4 hours at 2500°F isothermally.
- C. Using bath composition A, dipped at 2000°F, followed by pack calorizing for 1 hour at 1900°F greatly improves oxidation resistance at 2500°F, but just one cycle to room temperature is enough to cause complete failure.

It was concluded that direct resistance heating of coated tantalum samples would serve as a more flexible means of oxidation testing in that the high temperatures (2500° to 2800°F) required could be easily obtained, more rapid thermal cycling was possible, and the effect on the oxidation resistance of the coating over the entire temperature range from ambient to test temperature could be observed. Therefore, standard tensile type specimens, 1.25" x 0.100" x 0.010", were made. Oxidation test results at 2500°F, unless otherwise noted, are tabulated in Table III. All samples had a stress of at least 600 psi calculated on overall thickness and width. One problem with this type of oxidation testing is that the emissivity of the coating varies with composition and temperature. A correction was made to the reading obtained by the optical pyrometer but in all cases the reported temperature is a minimum and in fact is usually higher.

Although direct resistance heated oxidation samples failed after longer times at test conditions than coupons

* For patent and security reasons the composition of three aluminum dipping baths cannot be disclosed at this time. They are identified as bath compositions A, B, and C.

tested in a furnace, the reason is that coating coverage is poor around the hole in which wire is placed to enable the coupons to be dipped. Oxidation failure almost always starts at this point or at edges, but with the tensile type specimens the hole area is at room temperature since it is in the current carrying grip. The same conclusion, stated before, that dipping at 1700°F and vacuum diffusion heat treatments are unsatisfactory still holds true. No explanation can be given now for the one good test using those coating variables, G4172VD191-4. (See Appendix for code identification.)

Limited test results have been obtained on the effect of lower temperature oxidation characteristics on coated tantalum samples. These results are listed in Table IV. Running oxidation tests at 1100°, 1400°, and 1800°F, it is clearly indicated that the oxidation rate at 1800°F is much higher than at 2500°F. Even the 1100° and 1400°F results show that the coatings have a finite life. The lower temperature phenomenon must be investigated and solved by all workers striving for elevated temperature oxidation resistance with coated metal systems.

Before the fundamental phase of the program was started some preliminary work was done using tantalum, on bath compositions known to coat columbium successfully, which is reported herein.

Four samples of tantalum, 0.010" x 0.5" x 1.0" were dipped in a molten bath for two minutes at 1975°F containing bath composition B. These samples were pack calorized in a 10% Al, balance Al_2O_3 powder mix for one hour at 1900°F in an argon atmosphere to give a diffusion coating. Two other samples were dipped in bath composition C for two minutes at 1975°F. These were vacuum diffused for one hour at 2000°F. All six samples were oxidized in a Marshall platinum furnace with the results listed in Table V. None of the samples failed.

The test results showed that a coating of the $TaAl_3$ type on tantalum would protect the metallic core from oxidation for at least 10 hours at 2500° and 2600°F and could withstand thermal shock. Additional samples tested by resistance heating are listed in Table VI.

Section 4 Aluminum Coated Columbium

A total of 5 oxidation results at 2500°F were obtained on a columbium alloy that had been dipped in an aluminum alloy bath followed by a diffusion heat treatment. The results are as follows:

Direct Resistance Heated

1. 2 hour test	Sample good, no oxidation failure
2. 38 minute test	1 cycle, oxidation failure
3. 1-1/2 hour test	4 cycles, oxidation failure
4. 9 hour test	14 cycles, 1000 psi stress, oxidation failure
5. 10 hour test	Minimum 500 psi stress, 4% elongation minimum, no oxidation failure

The results of tests 4 and 5 can be considered quite promising for a columbium alloy-coating combination operating at 2500°F. It should be noted that these two samples were processed differently than the others during coating, which is felt to be responsible for their superior performance. They were dipped at a higher temperature.

Section 5 Beryllium Coated Tantalum

It is known that the tantalum beryllides are oxidation resistant at and over 2500°F.¹ A second objective of this program is to vapor deposit beryllium onto tantalum sheet in order to form a beryllide coating. The method used was to place tantalum samples between powder rolled sheets of beryllium and vacuum heat treat. Five coating trials were performed as follows:

- A. Be 184, 4 hours at 1800°F, 0.7 mil coating
- B. Be 201, 1 hour at 2000°F, 1.5 mil coating
- C. Be 204, 4 hours at 2000°F, 2 mil coating
- D. Be 221, 1 hour at 2200°F
- E. BeS 221, 1 hour at 2200°F, using previously sintered Be sheets

Furnace oxidation runs were made at 2500°F with the results given in Table VII. A photomicrograph is shown in Figure 4 with the Knoop hardness values for three of the four beryllides present. The beryllides are: TaBe₁₂, Ta₂Be₁₇, TaBe₃, and TaBe₂. It is apparent that beryllium coatings will confer the required oxidation resistance at 2500°F but that the difference in thermal expansion between the coating and the core must be matched more closely. Therefore, attempts will be made to produce a lower beryllium content beryllide at the surface before oxidation testing. It is assumed that lower beryllides will have lower coefficients of expansion more compatible with that of tantalum.

Section 6 Discussion

In summary the following statements may be made:

- A. There are similarities in dipping behavior between Ta and Cb.²
 - 1. Varying dipping conditions with a pure aluminum bath gives no control over coating thickness.
 - 2. Low temperature accelerated oxidation failure.
 - 3. Aluminum alloy dips are more promising.
- B. Both the aluminide and beryllide coatings show some promise since the minimum goals are in reach or attained already. More rigorous tests are required to check and prove these statements.

² Classified work at Sylcor for AEC.

Section 7 Appendix

The code used to identify and give prior history of the sample for aluminum dipped samples is as follows: the letter G followed by a number gives the code for the aluminum bath composition, dipping temperature is given in hundred °F, dipping time in minutes, VD for vacuum diffused using no calorizing mix and PC for pack calorized in powder mixture of 10% Al balance Al_2O_3 . In argon, diffusion time in hundred °F, time in hours, then a dash followed by a sample number. For example: G4171PC194-5 means dipped in Al-11 o/o Si bath (G4) at 1700°F for 1 minute followed by pack calorizing at 1900°F for 4 hours, sample number 5.

- G3 pure aluminum dipping bath
- G4 Al-11 o/o Si dipping bath
- G5 Al-10 o/o Si-10 Cr dipping bath
- G6 Al-25 o/o Si dipping bath
- G7 Bath composition A

TABLE I

THICKNESS OF ALUMINUM COATINGS ON TANTALUM SHEET

<u>Sample No.</u>	<u>Dipping</u>		<u>Thickness in mils</u>			<u>Total</u>
	<u>Temp.,</u> <u>°F</u>	<u>Time,</u> <u>min.</u>	<u>Coating</u>	<u>Core</u>	<u>Coating</u>	
<u>Pure Aluminum Dip, As Dipped</u>						
G3171	1700	1	1.3	9.3		11.9
G3172	1700	2	1.3	9.1		11.7
G3175	1700	5	1.2	8.9		11.3
G3211	2100	1	1.7	8.6		12.0
G3212	2100	2	1.2	6.3		8.7
<u>Vacuum Diffused 1900°F 1 hour</u>						
G3171VD191	1700	1	1.4	8.4	1.7	11.5
G3172VD191	1700	2	1.4	8.4	1.6	11.4
G3175VD191	1700	5	1.2	7.6	1.2	10.7
G3211VD191	2100	1	1.1	7.6	1.2	9.9
G3212VD191	2100	2	1.3	7.3	1.3	9.9
<u>Vacuum Diffused 1900°F 4 hours</u>						
G3171VD194	1700	1	1.2	8.3	1.2	10.7
G3172VD194	1700	2	1.2	8.3	1.2	10.7
G3175VD194	1700	5	1.2	8.3	1.2	10.7
G3211VD194	2100	1	1.0	7.8	1.2	9.8
G3212VD194	2100	2	0.62	7.1	1.2	8.3
<u>Vacuum Diffused 2200°F 1 hour</u>						
G3171VD221	1700	1	1.1	8.4		10.6
G3172VD221	1700	2	1.1	8.4		10.6
G3175VD221	1700	5	1.1	8.4		10.6
G3211VD221	2100	1	0.99	7.7		9.7
G3212VD221	2100	2	0.76	6.4		7.6
<u>Vacuum Diffused 2200°F 4 hours</u>						
G3171VD224	1700	1	1.0	8.2	1.0	10.3
G3172VD224	1700	2	1.0	8.4	0.74	10.1
G3175VD224	1700	5	0.74	8.2	0.49	9.4
G3211VD224	2100	1	0.49	7.9	0.44	8.8
G3212VD224	2100	2	0.45	6.9	0.45	7.8

TABLE I (Cont'd.)

THICKNESS OF ALUMINUM COATINGS ON TANTALUM SHEET

<u>Sample No.</u>	<u>Dipping</u>		<u>Thickness in mils</u>			<u>Total</u>
	<u>Temp.,</u> <u>°F</u>	<u>Time,</u> <u>min.</u>	<u>Coating</u>	<u>Core</u>	<u>Coating</u>	
<u>Pack Calorized 1900°F 1 hour</u>						
G3171PC191	1700	1	1.9	8.1	1.9	11.9
G3172PC191	1700	2	2.1	7.9	1.7	11.7
G3175PC191	1700	5	2.1	7.8	1.7	11.6
G3211PC191	2100	1	1.9	7.4	2.0	11.3
G3212PC191	2100	2	2.5	6.2	2.2	10.9
<u>Pack Calorized 1900°F 4 hours</u>						
G3171PC194	1700	1	2.1	7.7	2.0	11.8
G3172PC194	1700	2	1.4	7.8	1.7	10.9
G3175PC194	1700	5	1.2	7.9	1.2	10.3
G3211PC194	2100	1	1.2	7.4	1.6	10.2
G3212PC194	2100	2	1.3	6.8	1.3	9.4
<u>Al-11 o/o Si Dip Vacuum Diffused 1900°F 1 hour</u>						
G4171VD191	1700	1	1.7	8.1	1.4	11.2
G4172VD191	1700	2	0.74	8.3	0.74	9.7
G4175VD191	1700	5	0.37	8.3	0.37	9.0
G4201/2VD191	2000	0.5	1.4	8.1	1.2	10.7
G4201VD191	2000	1	2.1	7.2	2.1	11.4
<u>Vacuum Diffused 1900°F 4 hours</u>						
G4171VD194	1700	1	1.3	8.3	1.0	10.6
G4172VD194	1700	2	0.49	8.2	0.49	10.1
G4175VD194	1700	5	0.4	8.7	0.4	9.5
G4201/2VD194	2000	0.5	1.6	7.7	1.4	10.1
G4201VD194	2000	1	0.3	6.9	2.7	12.6
<u>Vacuum Diffused 2200°F 1 hour</u>						
G4171VD221	1700	1	1.0	8.1	1.0	10.1
G4172VD221	1700	2	1.0	8.2	1.0	10.2
G4175VD221	1700	5	1.1	8.4	1.0	10.5
G4201/2VD221	2000	0.5	1.4	8.2	1.2	10.8
G4201VD221	2000	1	2.6	7.1	2.5	12.2

TABLE I (Cont'd.)

THICKNESS OF ALUMINUM COATINGS ON TANTALUM SHEET

<u>Sample No.</u>	<u>Dipping</u>		<u>Thickness in mils</u>			<u>Total</u>
	<u>Temp.,</u> <u>°F</u>	<u>Time,</u> <u>min.</u>	<u>Coating</u>	<u>Core</u>	<u>Coating</u>	
<u>Vacuum Diffused 2200°F 4 hours</u>						
G4171VD224	1700	1	2.0	7.2	2.0	11.2
G4172VD224	1700	2	1.2	8.1	2.1	11.4
G4175VD224	1700	5	0.74	8.3	0.74	9.7
G4201/2VD224	2000	0.5	1.2	7.8	1.2	10.2
G4201VD224	2000	1		9.1		
<u>Pack Calorized 1900°F 1 hour</u>						
G4171PC191	1700	1	1.9	7.6	1.9	11.4
G4172PC191	1700	2	2.2	7.4	2.1	11.7
G4175PC191	1700	5	2.2	7.4	2.0	11.6
G4201/2PC191	2000	0.5	2.0	7.8	1.9	11.7
G4201PC191	2000	1	2.0	7.4	1.9	11.3
<u>Pack Calorized 1900°F 4 hours</u>						
G4171PC194	1700	1	2.5	7.4	2.0	11.9
G4172PC194	1700	2	2.2	7.7	2.0	11.9
G4175PC194	1700	5	1.9	7.7	1.4	11.0
G4201/2PC194	2000	0.5	1.4	7.8	1.7	10.9
G4201PC194	2000	1	2.7	6.8	3.0	12.5
<u>Al-10 o/o Si-10 o/o Cr Dip, Pack Calorized 1900°F 1 hour</u>						
G5171PC191	1700	1	2.0	7.4	1.9	11.3
G5172PC191	1700	2	2.6	7.4	0.49	10.5
G5201/2PC191	2000	0.5	1.7	7.4	1.4	10.5
G5201PC191	2000	1	3.7	5.7	4.0	13.4
<u>Al-25 o/o Si Dip, Pack Calorized 1900°F 1 hour</u>						
G6171PC191	1700	1	2.2	6.8	3.2	12.0
G6172PC191	1700	2	1.6	6.8	2.9	11.3
G6201/2PC191	2000	0.5	2.0	7.4	1.7	11.1
G6201PC191	2000	1	3.3	6.2	3.8	13.3

TABLE II

FURNACE TESTED OXIDATION RESULTS ON COATED
TANTALUM AT 2500°F

<u>Sample No.</u>	<u>Time, Hours</u>	<u>Remarks</u>
<u>Pure Aluminum Dip, As Dipped</u>		
G3171-6	0.5	Oxidized completely
G3172-7	0.5	Oxidized completely
G3175-8	0.5	Oxidized completely
G3211-9	4.5 to 5	Oxidized completely
G3211-10	4 to 5	Oxidized completely
G3211-11	4 to 5	Oxidized completely
<u>Al-11 o/o Si Dip, Pack Calorized 1 hour 1900°F</u>		
G4172PC191-8	1	Oxidized completely
G4172PC191-9	1	Oxidized completely
G4201PC191-10	1	Oxidized completely
G4201PC191-11	1	Oxidized completely
<u>Al-25 o/o Si Dip, Pack Calorized 1 hour 1900°F</u>		
G6172PC191-3	1	Oxidized completely
G6201PC191-2	1	Oxidized completely
<u>Bath Composition A, Pack Calorized 1 hour 1900°F</u>		
G7171PC191-3	1	Oxidized completely
G7171PC191-4	9 to 10	Oxidized completely, failure started at hole
G7201PC191-5	7	Failed at hole
G7201PC191-6	10	No oxidation failure
G7171PC191-18	1	Oxidized completely
G7171PC191-19	8 to 10	These 3 samples were cooled to room temperature after 8 hours. No failure. But on reheating for 2 hours oxidized completely. Will not take cyclic oxidation.
G7201PC191-20	8 to 10	
G7201PC191-21	8 to 10	

TABLE III

OXIDATION TESTING AT 2500°F OF COATED TANTALUM
BY DIRECT RESISTANCE HEATING TECHNIQUES

<u>Sample No.</u>	<u>Time to Failure, Hours</u>	<u>Remarks</u>
<u>Aluminum Dip</u>		
G3212VD201-1	0.02	
G3212VD221-2	0.02	
G3212VD221-3	0.02	
G3212PC191-4	5	
G3215PC191-5	2.3	Failed in low tempera- ture region of sample
<u>Al-11 o/o Si Dip</u>		
G4201PC191-1	8.8	
G4201VD191-2	5	
G4172PC191-3	0.2	Tested at 2800°F, edge failure
G4172VD191-4	10	Test stopped, sample good
G4172PC191-5	1.2	Failed in low tempera- ture region, 2000 psi stress
G4172PC191-6	0.6	Tested at 2600°F
G4201PC191-7	1.2	Failed in low tempera- ture region, 1 cycle
<u>Al-10 o/o Si-10 o/o Cr Dip</u>		
G5201PC191-1	0.6	Tested at 2800°F
G5201PC191-2	0.6	2000 psi stress
G5172PC191-3	8	2000 psi stress
G5172PC191-4	7.2	
<u>Al-25 o/o Si Dip</u>		
G6201PC191-1	2.6	
<u>Bath Composition A</u>		
G7201PC191-1	5.2	Low temperature failure
G7201PC191-2	9 to 10	9 cycles by air blast
G7201PC191-13	10	20 cycles, test stopped, good sample

TABLE III (Cont'd.)

OXIDATION TESTING AT 2500°F OF COATED TANTALUM
BY DIRECT RESISTANCE HEATING TECHNIQUES

<u>Sample No.</u>	<u>Time to Failure,</u> <u>Hours</u>	<u>Remarks</u>
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Bath Composition A (Cont'd.)

G7171PC191-14	4	8 cycles
G7201PC191-16	4	8 cycles
G7171PC191-22	4.2	8 cycles

Ta + 7 o/o W Alloy Core

G7T201PC191-7	1.4	2 cycles
G7T201PC191-8	6.1	2000 psi stress
G7T171PC191-15	2	4 cycles
G7T171PC191-17	1.3	

Stress of 600 psi on all samples unless noted.

TABLE IV

LOW TEMPERATURE OXIDATION OF COATED TANTALUM

<u>Sample No.</u>	<u>Test Temperature, °F</u>	<u>Time to Failure, Hours</u>	<u>Remarks</u>
<u>Al-11 o/o Si Dip</u>			
G4172PC191	1100	116	Went to powder
G4201PC191	1100	116	Local failure
G4172PC191	1400	18	Edge failures
G4201PC191	1400	18	Edge failures
G4172PC191	1800	18	Went to powder
G4201PC191	1800	18	Local failure

G5 Al-10 o/o Si-10 o/o Cr Dip, G6 Al-25 o/o Si Dip

G5201PC191	1100	163	O.K. at 18 hours, local failure
G6201PC191	1100	163	O.K. at 18 hours, powder
G5201PC191	1400	19	Spot failures
G6201PC191	1400	19	Gross failure
G5201PC191	1800	3	Spalling oxide
G6201PC191	1800	3	More extreme spalling
G6172PC191-4	2200	0.5	Thick oxide film; edge failure

TABLE V

OXIDATION OF ALUMINUM DIPPED TANTALUM SHEET

<u>Sample No.</u>	<u>Alloy</u>	<u>Time, Hours</u>	<u>Temp., °F</u>	<u>Remarks</u>
<u>Bath Composition B</u>				
1	Ta	5 44	2500 2300	Furnace lowered to 2300°F, sample left in
2	Ta	7.5	2500	Cycled once to room temperature
3	Ta	15	2500	Cycled once
4	Ta	10	2600	Local edge oxidation
<u>Bath Composition C</u>				
5	Ta	10	2500	Cycled eight times
6	Ta + 7 o/o W	10	2500	Cycled five times

No oxidation failure on any of these samples.

TABLE VI

DIRECT RESISTANCE HEATED OXIDATION TESTS ON
ALUMINUM ALLOY COATED TANTALUM SHEET

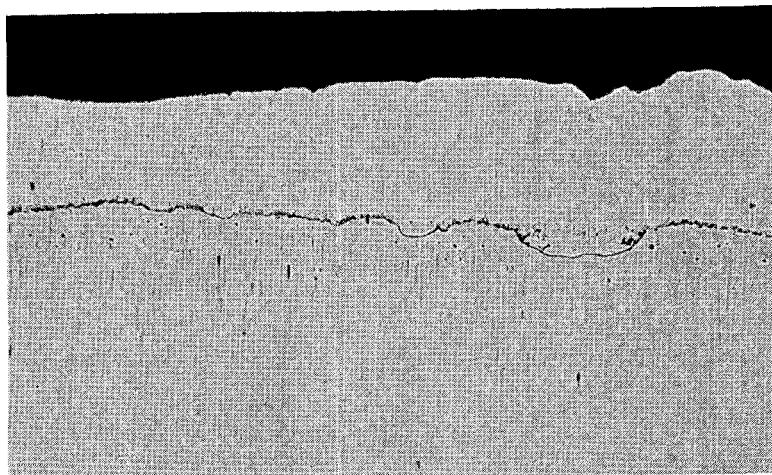
<u>Sample No.</u>	<u>Time, Hours</u>	<u>Temp., °F</u>	<u>Remarks</u>
<u>Bath Composition C</u>			
G1-2	10	2550	
G1-3	>8	2550	Power went off overnight
G1-4			Hot spot, coating melted, test stopped
G2-1	10	2500	Cycled eleven times to room temperature
G2-2	0.1	2370	Failed in gage length
G2-3	10	2550	Cycled five times

No oxidation failure unless noted.

TABLE VII

OXIDATION RESULTS AT 2500°F ON BERYLLIUM
COATED TANTALUM SHEET

<u>Sample No.</u>	<u>Time to Failure, Hours</u>	<u>Remarks</u>
Be 184-1	0.2	Edge failure
Be 201-1	0.2	Edge failure
Be 201-2	0.1	Edge failure
Be 204-2	4.5	Failed 1/2 hour after cycle
Be 204-3	9.5 to 10	9 cycles, oxidation failure
Be 204-4	15.5	Test stopped, no oxidation
Be 221-1	10	Test stopped, no oxidation failure
Be 221-2	10	Test stopped, 10 cycles, no oxidation failure
Be 221-3	16	Edge failure
BeS 221-11	10	Test stopped, 10 cycles, no oxidation failure
BeS 221-2	16	Went to oxide
BeS 221-3	8	8 cycles, edge failure



Al coating

1.3 mils
38 Knoop

Ta core

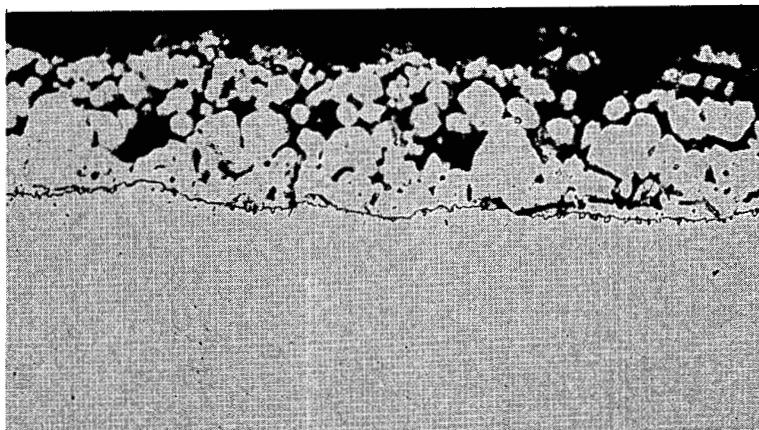
9.3 mils
126 Knoop

Plate No. 23383

G3171

500X

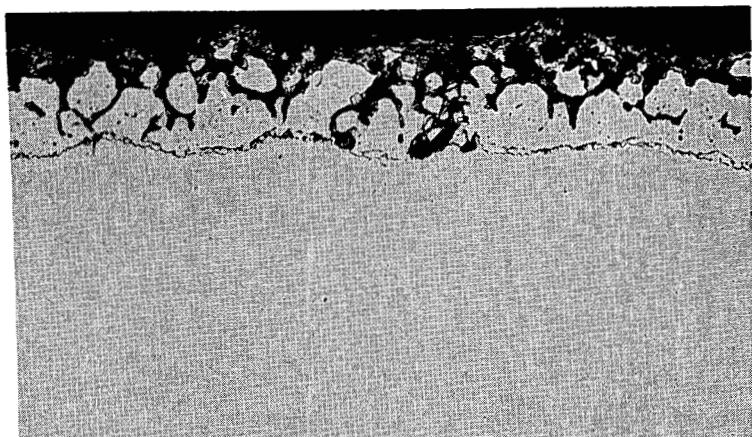
Fig. 1. Aluminum coated tantalum sheet
as dipped 1 minute at 1700°F.



Al coating
1.7 mils
535 Knoop
← 717 Knoop

Ta core
8.4 mils

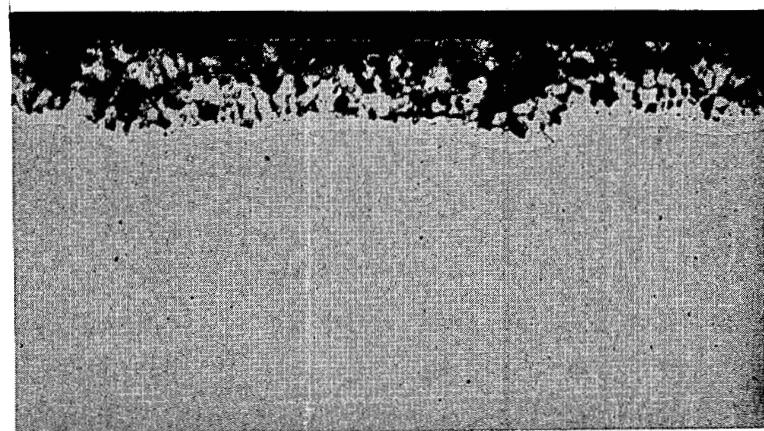
Plate No. 23401 G3171VD191 500X
Vacuum diffused 1900°F 1 hour



Al coating
1.2 mils
480 Knoop
←

Ta core
8.3 mils

Plate No. 23382 G3171VD194 500X
Vacuum diffused 1900°F 4 hours

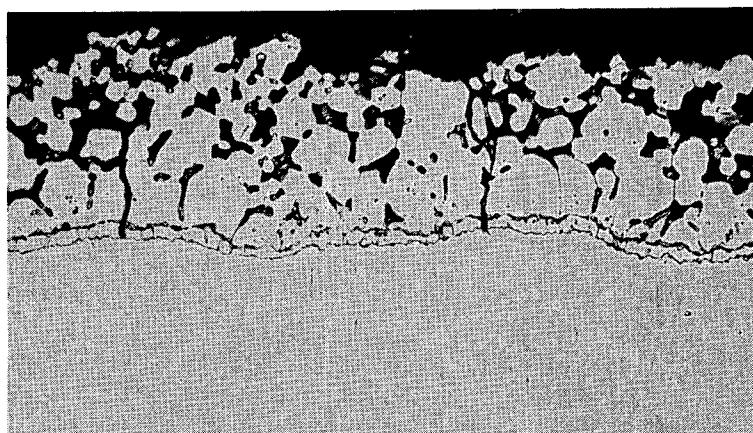


Al coating
1 mil
719 Knoop
←

Ta core
8.2 mils
114 Knoop

Plate No. 23384 G3171VD224 500X
Vacuum diffused 2200°F 4 hours

Fig. 2. Aluminum coated tantalum sheet dipped for
1 minute at 1700°F, vacuum diffused.

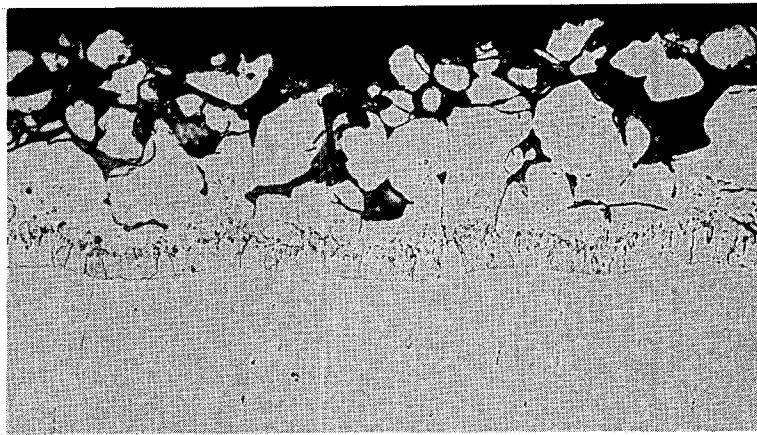


Al coating
1.9 mils
465 Knoop

← 678 Knoop

Ta core
8.1 mils
118 Knoop

Plate No. 23397 G3171PC191 500X
Pack calorized 1900°F 1 hour



Al coating
2 mils
455 Knoop

← 697 Knoop

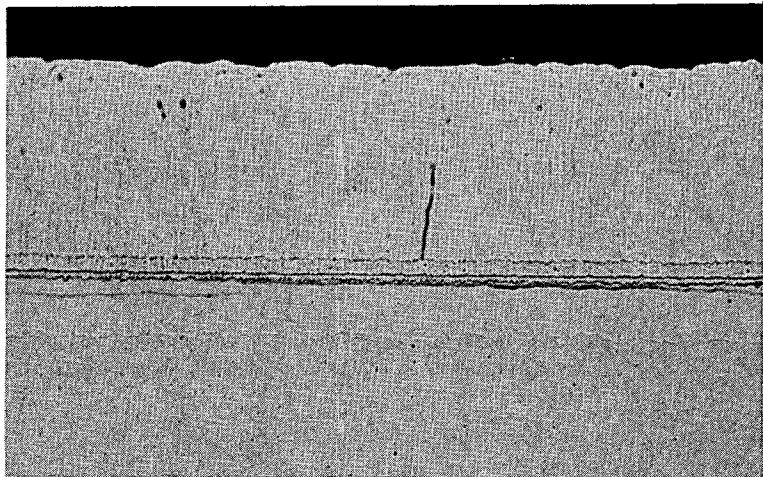
Ta core
7.7 mils

Plate No. 23381 G3171PC194 500X
Pack calorized 1900°F 4 hours



Plate No. 23387 G3171PC194 500X
Pack calorized 1900°F 4 hours polarized light

Fig. 3. Aluminum coated tantalum sheet dipped 1 minute at 1700°F, pack calorized.



Beryllium
Coating
1662 Knoop

← 1630 Knoop
← 1156 Knoop

Ta core
116 Knoop

Plate No. 23385

Be204

500X

Fig. 4. Beryllium coated tantalum sheet,
vacuum deposited 2000°F for 4
hours.

Distribution List - AF 33(616)-7184

1. American Machine and Foundry Co.
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Research and Development Dept.
Attn: W. W. Beaver
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4. Chromalloy Corporation
Attn: Dr. R. Wachtell
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5. Fansteel Metallurgical Corp.
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