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TANTALUM ALLOY STRUCTURAL FASTENERS

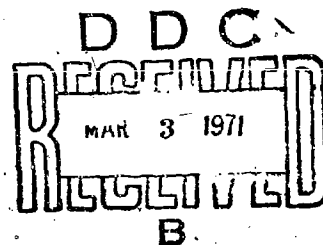
T. A. Roach

Standard Pressed Steel Co.

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Technical Report AFFDL-TR-70-168

December, 1970



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TANTALUM ALLOY
STRUCTURAL FASTENERS

T. A. Roach

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FOREWORD

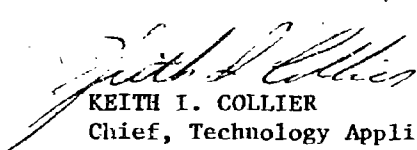
This report covers work performed under Contract AF33615-67-C-1494 from March 1967 to December 1969. The contract was initiated under Project 1368, "Structural Design Concepts for Military Vehicles," Task 136807, "Airframe Structural Fastening and Joining Techniques." The work was administered by the Air Force Flight Dynamics Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, Ohio, Mr. James F. Nicholson (FBS) Project Engineer.

All of the items compared in this report were commercial items that were not developed or manufactured to meet Government specifications, to withstand the tests to which they were subjected, or to operate as applied during this study. Any failure to meet the objectives of this study is no reflection on any of the commercial items discussed herein or on any manufacturer.

This report was prepared by Thomas A. Roach of Standard Pressed Steel Laboratories.

This report was submitted by the Author in October 1970.

This technical report has been reviewed and is approved.


KEITH I. COLLIER
Chief, Technology Applications Br
Structures Division

ABSTRACT

A survey of available tantalum coatings was conducted to determine which might be capable of protecting threaded fasteners from oxidation at up to 3500°F. Sufficient tests were run on each selected coating to demonstrate that no coating is available to provide consistent protection at 3500°F, or in fact for 3000°F. Problems associated with thread forms and stress-relaxation were studied, but only in a general way due to the limitations imposed by the lack of satisfactory coatings.

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SECTION I

INTRODUCTION

This program was initiated in March 1967 with the following objectives:

1. Determine the structural utilization potential of Ta alloy mechanical fasteners for component assembly and attachment wherein the environmental exposure may range from -320°F to 3500°F .
2. Develop preliminary design allowables which will assist designers in making optimum selections of configurations and construction concepts.

These objectives were to be accomplished by means of the following activities:

1. Conduct a brief survey of the state-of-the-art of tantalum coatings and tantalum alloys to permit selection of those coatings and alloys which possess sufficient potential to warrant their consideration for inclusion in the program.
2. Perform sufficient oxidation tests on selected coating - substrate combination specimens to determine which possesses the best combination of properties for use at up to 3500°F .
3. Fabricate, coat, and test sufficient fasteners and fastener assemblies to determine preliminary design properties of the selected coating - substrate combination in typical fastener configurations.

SECTION II

SURVEY

The objectives of the survey were to maintain an awareness of the state-of-the-art in advanced tantalum alloys and tantalum alloy coatings and to consider and study the possible fastener designs and joint configurations which would be incorporated into the later portions of the program.

Since this program, in many ways, paralleled the early portions of the McDonnell program, ("Tantalum System Evaluation" under AF33(615)-3935) much of the early coating screening performed under that program formed a basis for our early coating considerations. (1) This is also true, to some extent, in the case of the tantalum alloys.

A discussion of each of the individual aspects of the survey follows:

1. Tantalum Alloy Coatings

The coating performance objective of some useful life at 3500°F, both in slowly moving air at 1 atmosphere and in air at 1 Torr, seemed to limit the possible coatings to those utilizing some form of tungsten disilicide either by itself, or in combination with barrier layers of tungsten or tungsten combined with other metals. The coating sources which were considered were therefore:

a. Solar Division of International Harvester

The solar coating is a two step process involving the application of a sprayed slurry tungsten + 5% titanium coating which is vacuum sintered at 2400-2700°F followed by pack siliciding of the tungsten-titanium layer at 2100°F.

The thickness of coating suggested by Solar for this program was .007 ± .001".

b. Sylvania High Temperature Composites Lab of Sylvania Electric Products Co.

The Sylvania W Si₂ coating (R516) consists of a chemical vapor deposited tungsten layer which is subsequently covered by a tungsten disilicide layer by a slurry process.

The thickness of the coating suggested by Sylvania was .0055" minimum with the maximum indeterminable at this time. During the course of the program, the Sylvania R512C was tested. (Si-20Ti-10Mo)

c. Thompson-Ramo-Wooldridge

The TRW coating consisted of a tungsten (or modified tungsten) layer which is vacuum pack silicided. The tungsten layer can be deposited by chemical vapor deposition, electrophoresis or other applicable process.

A thickness of coating was not specifically recommended, but was on the order of .005 with the attainable tolerance greatly dependent on the method of depositing the tungsten layer.

d. Vitro Laboratories

This coating consisted of an electrophoretically deposited layer of tungsten disilicide which is isostatically pressed and then silicided by a vacuum pack process in a non-activated pack. (2)

A coating thickness of .0025 $\pm$.0004 was proposed in accordance with our request to keep the coating as thin as possible commensurate with the oxidation protection requirements.

During the course of the program, consideration was given to the electrophoretic application of a tungsten + 5% titanium barrier followed by vacuum siliconization.

2. Tantalum Alloys

It was intended to utilize the most advanced tantalum alloy in this program, the selection to be based on tensile and creep properties at up to 3500°F and compatibility with the candidate coatings.

The selection of candidate alloys was made and included the three following alloys:

- a. T-222 (Ta - 9.6W - 2.4 Hf - .01C)
- b. GE473 (Ta - 7W - 3Rc)
- c. Type 161 (Ta - 6.6W - 3Rc - 1.6Hf - .03 Zr - .005Y)

Limited mechanical property data was available from the "Tantalum Systems Evaluation" program at McDonnell-Douglas under AF33(615)-3935. (1) This data is shown in Table I.

TABLE I

MECHANICAL PROPERTIES OF COATED TANTALUM ALLOYS
FROM AF33(615)-3935 McDONNELL-DOUGLAS

All data in ksi

Property	Material and Coating			
	Ta-9.6W-2.Hf-.01C		Ta-7W-3Re *	
	Solar	Sylvania	Solar	Sylvania
2600°F UTS	— 31.7	— 38.2	— 29.7	— 32.0
3100°F UTS	2 — 20.6	— 22.4	— 18.6	— 21.4
2600°F Yield Str.	— 26.9	— 31.2	— 24.1	— 29.3
3100°F Yield Str.	— 19.8	— 20.3	— 16.0	— 19.1
2600°F Elong.	— 52	— 15.3	— 59	— 11.3
3100°F Elong.	— 66	— 52	— 88	— 26
2600°F Shear Ult.	— 37.4	— 31.3	— 29.2	— 26.2
3100°F Shear Ult.	— 17.3	— 17.2	— 19.0	— 19.3

3. Configurations and Thread Forms

It became apparent early in the program that one common element to most of the candidate coatings was thickness greatly in excess of those previously applied to threaded fasteners. By way of reference, all the fastener coatings applied for AF33(657)-11684, "Structural Fasteners for Extreme Elevated Temperatures" (2) were less than .003" thick, whereas three of the four candidate coatings for this program were .004" thick or greater. This presented several serious problems relating to the maintaining of an acceptable thread form and the relaxation of preload during high temperature exposure. These are discussed further in subsequent sections.

SECTION III

THREAD FORM

All fastener coating studies have previously indicated that the service integrity of a coated fastener is greatly enhanced by the elimination of sharp contours such as at the thread crests, hex corners, etc. Previous work at SPS resulted in the development of a thread form which has generously rounded thread crests and thread roots. This form is shown in Figure 1. The basic form can be adjusted to provide for coating thickness allowance within reasonable limits, but not to the levels apparently required by this program.

To fully understand the problems created by the thick coating, it is necessary first to consider the inter-relationship between coating thickness and thread geometry. The underlying factor is that involving the relationship between pitch diameter and coating thickness.

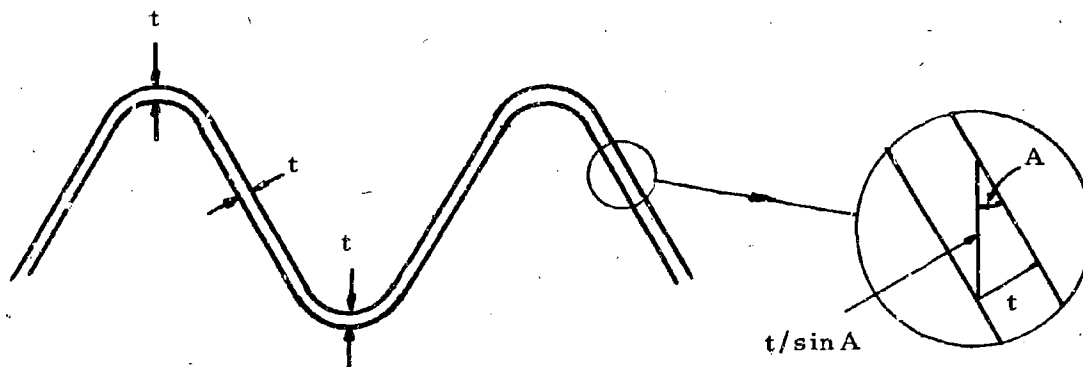
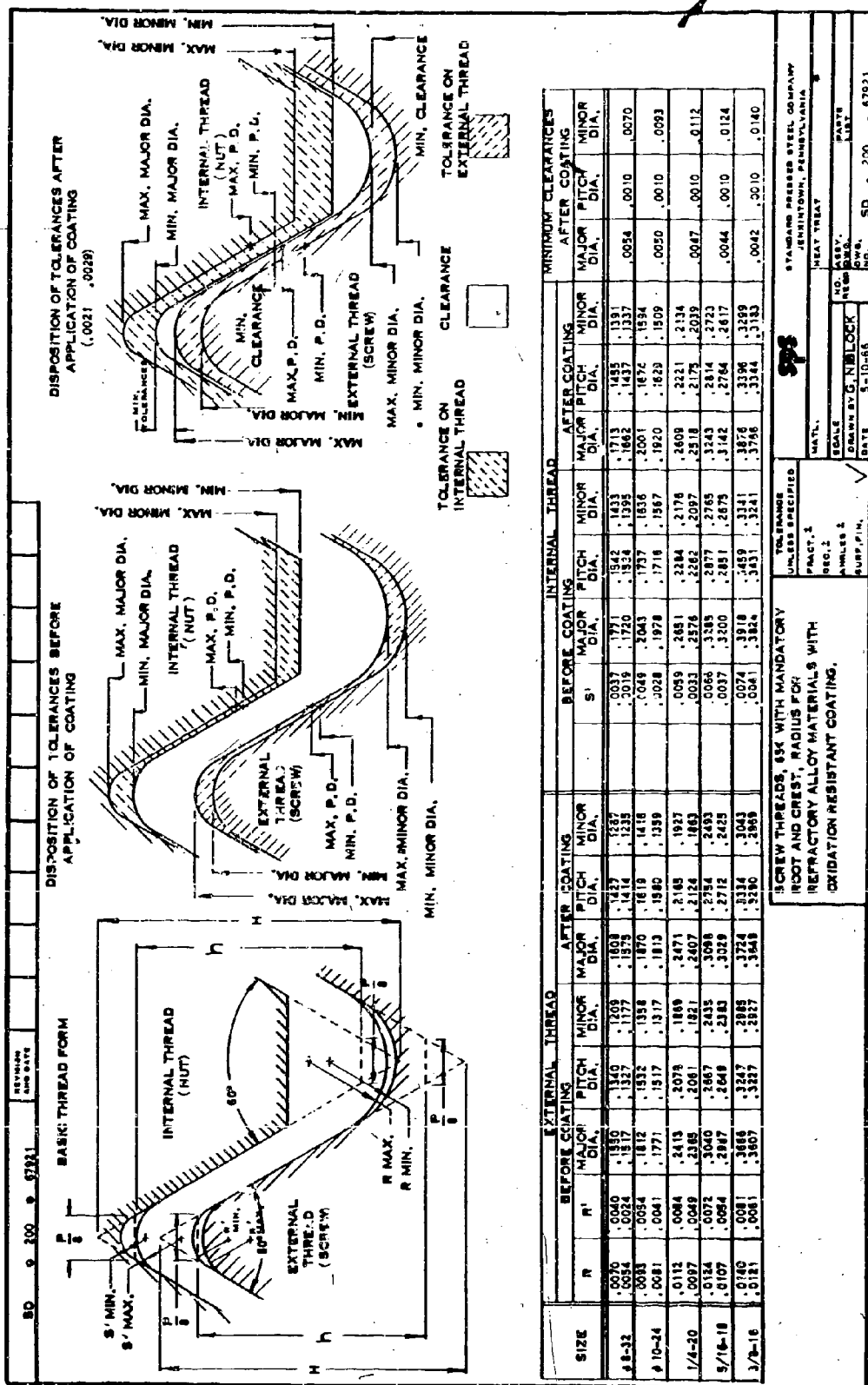


Figure 2. Factors Involved in Coating Thickness Allowance Determinations

Figure 1 illustrates the factors involved. Coating thickness (t) builds up such that its dimension measured normal to the surface is essentially constant at any point. Thus, as shown, the coating thickness measured normal to the bolt centerline on the flank of the thread would indicate a measurement of $t/\sin A$ and the pitch diameter of the bolt would experience an increase of $2 t/\sin A$. In the case of a conventional thread form (or the refractory thread form) angle A would be 30° thus resulting in a pitch diameter change of $2 t/.5 = 4t$. This relationship is accepted and widely used to calculate dimensional allowances for plating on conventional fasteners for lubrication, corrosion prevention, etc. It has also been used to calculate coating allowances for refractory metal fasteners with up to .003" thick coating. Beyond this thickness a factor which has little influence on the thinner coatings becomes effective. This is shown in Figure 3. In adjusting the pitch diameter for coating allowance, the bolt minor and major diameters change by the same amount, as the pitch diameter.



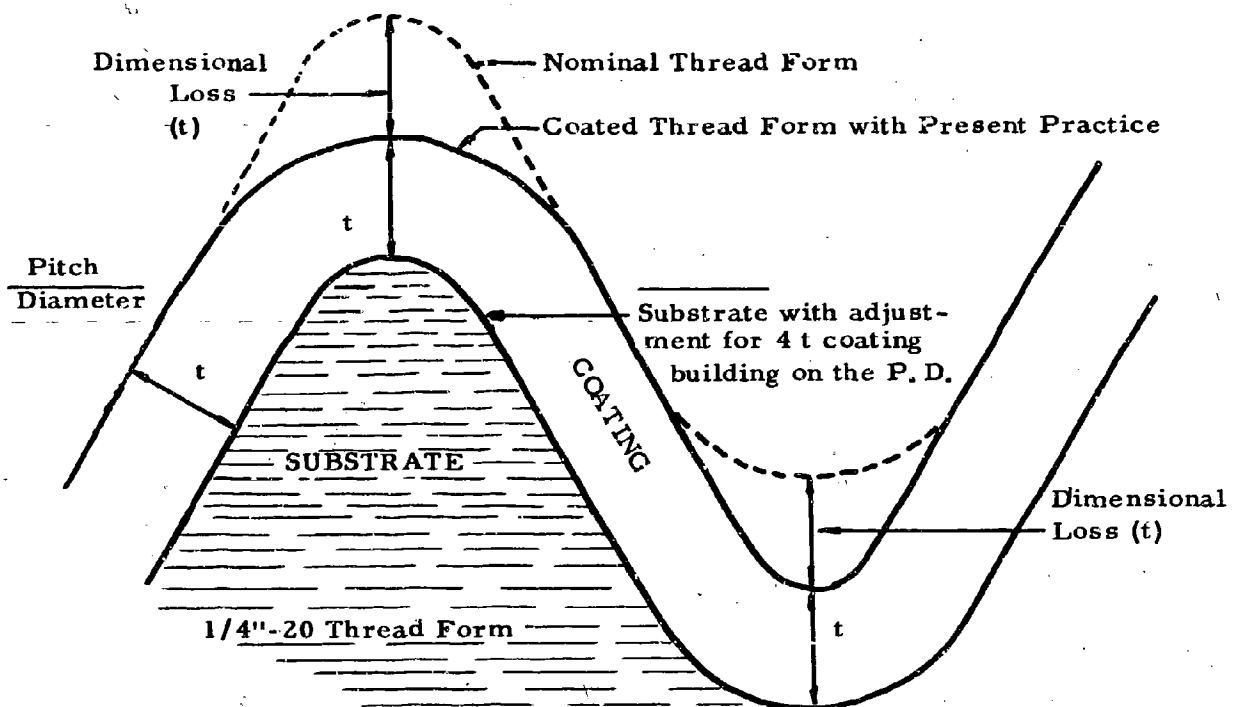


Figure 3. Influence of Coating on Thread Form.

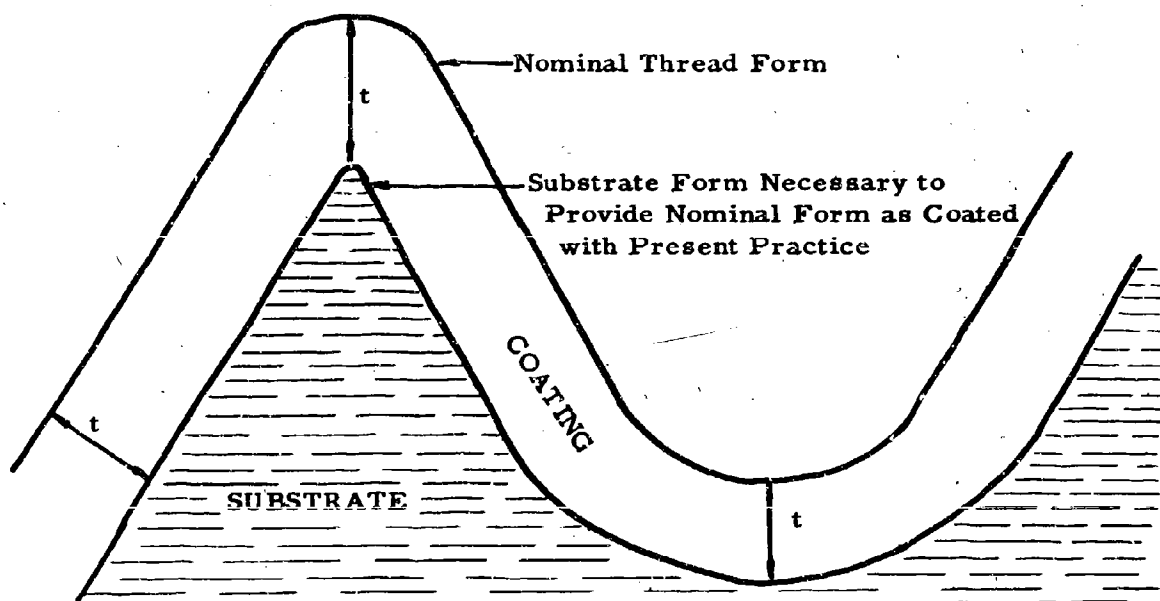


Figure 4. Substrate Form Required to Produce the Refractory Thread Form on 1/4 inch Fasteners with .007 inch Thick Coating.

However, these dimensions are only built up by one coating thickness per side or 2T on the diameters. Thus for each .001 inch of coating thickness, the minor and major diameters of the coated part decrease .002 inch in relation to the pitch diameter.

This loss results in a reduction of thread engagement. The significance of this loss becomes apparent when considered in the case of a .007 inch thick coating. The bolt major and minor diameters in that case are reduced .014 inch. In the case of a 1/4-20 bolt the difference between the bolt nominal pitch diameter and nominal major diameter is .030 inch. Therefore, with adjustment for a .007 inch thick coating the difference between pitch diameter and major diameter would be reduced to .016.

If the nominal thread form is adjusted for uniform coating thickness over the entire form, the result is shown in Figure 4.

Let us assume for a moment that we can produce a nominal thread form with .007 inch thick coating. A further problem arises in the fact that the strength of the fastener is largely dependent on the shear strength of the coating since little, if any, substrate engagement is present in the smaller size fasteners. For example, in a 1/4-20 fastener system using the full refractory thread form in both nut and bolt, the diametral engagement (nominal) is .033 inches. Thus, with the thread crests of nut and bolt consisting of .007 inch of coating the substrate engagement is .033 - (coating on the nut minor diameter + coating on bolt major diameter) or $.033 - (.014 + .014) = .033 - .028 = .005$. It is easily seen that under these conditions the shear strength of the coating is the overriding factor influencing the strength of the fastener system.

Both the problems described decrease in severity as the bolt dimensions become larger in relation to the coating thickness leading to one partial solution to the problems, that of using larger size fasteners. A logical extension of this is the use of coarse threads.

Several possible courses of action exist, as follows, along with a brief discussion of the limitations each imposes on the use of the fasteners:

1. Restrict coating in the thread areas to .003 inch based on the fact that the thread areas receive protection by virtue of intimate contact between the nut and bolt. This would considerably increase the cost of coating if indeed it is possible to coat two different thicknesses on the same part.
2. Use coating of .003 on the entire fastener and slurry coat after assembly. This may limit the application of mechanical fasteners.

3. Use very coarse threads. This imposes no limitation in itself, but may result in the use of large fasteners to reach the lower numbers of threads per inch.
4. Use large diameter fasteners. This may severely limit applications and will certainly increase cost of material, manufacturing, and coating.
5. Use a square thread form such as an Acme type. This would have a drastic effect on the cost of the fasteners and the coating thereof if indeed they can be made this way.
6. Use extra-height nuts and longer bolt threads to provide sufficient shear area to support tensile loads. This solution would increase cost and limit applications.
7. Restrict use of fasteners to shear applications where reduced thread strength could be tolerated. This solution would eliminate those tensile applications which could not be redesigned to provide primarily shear loading.

SECTION IV
COATING PROCUREMENT

Following the initial survey of coating vendors a review of the coating possibilities revealed the following:

1. The proposed TRW coating utilized a Chemical Vapor Deposited tungsten barrier layer supplied by Sylvania. This coating was therefore not considered significantly different from the Sylvania WSi_2 coating, and since it would involve a logistic problem, the coating was not considered for the screening phase.
2. The Sylvania WSi_2 coating (R516) had little or no history at $3500^\circ F$ but did represent the system which was given the best chance to survive and therefore considered.
3. The Solar coating had been developed for under $3000^\circ F$ service, but, like the Sylvania R516, it seemed to possess some potential for $3500^\circ F$ service and was therefore considered.
4. The Vitro Si/ WSi_2 coating had previously demonstrated short life at $3100^\circ F$ under AF33(657)-11684 (2) and was therefore not initially considered.

The initial coating choice therefore was between the Solar TNV-13 and the Sylvania R516. A decision was made to concentrate our efforts on the TNV-13 in light of some initial success with this coating at McDonnell-Douglas under the "Tantalum System Evaluation" program. (1)

Subsequent testing (see Section VII) indicated that the Solar coating did not possess all the requirements of the program and a series of steps was taken to find an acceptable coating. These steps are best described by a chronological review of the coating lots which were included in the overall program as follows:

1. Coating for Relaxation Tests

Solar TNV-13

- 20-1/4 inch T-222 washers (complete coating)
- 20-1/4 inch T-222 washers (95W-STi barrier only)
- 20-1/4 inch T-222 washers (pre-oxidized $2900^\circ F$)

Vitro Si/ WSi_2

- 20-1/4 inch T-222 washers (pre-oxidized $2900^\circ F$)

2. Oxidation Tests on Threads

Solar TNV-13

80-1/4 inch thread specimens - 5 coating thicknesses

3. Oxidation Tests on Threads

Vitro W/WSi₂ (Silicided Tungsten barrier)

25-1/4 inch thread specimens

4. Oxidation Tests on Threads

Sylvania R512C

40-1/4 inch thread specimens

SECTION V

TANTALUM ALLOY PROCUREMENT

Following the survey, requests for quotation were sent to several tantalum alloy producers for price and delivery information on sufficient quantities of T-222, GE 473 and Type 161 to perform the necessary screening tests to select the best alloy. Subsequently, a purchase order was placed with Union Carbide for the following material:

- a. 10 feet of .270" diameter rod
- b. 150 square inches of .020" thick sheet
- c. 150 square inches of .040" thick sheet
- d. 150 square inches of .060" thick sheet

The above quantities to be produced in each of the three materials:

Ta - 9.6W - 2.4HF - .01C (T-222)

Ta - 7W - 3Re (GE 473)

Ta - 6.6W - 3Re - 1.6Hf - .03Zr - .005Y (Type 161)

Problems were encountered in the production of the three alloys resulting in the no yield of the T-222 and Type 161 and only 65 inches of the GE 473 rod.

Fortunately, when the time arrived for a decision on the purchase of material for the fastener test program, sufficient data had been generated by McDonnell-Douglas in their program to enable us to make a material selection. Our recommendation was to purchase T-222 from Wah Chang.

An order was subsequently placed with Wah Chang for the following lot of T-222:

- 1000 square inches of .060" thick sheet
- 35 feet of .270" diameter rod
- 20 feet of .400" diameter rod
- 6 feet of .625" diameter rod
- 3 feet of 1.000" diameter rod
- 3 feet of 1.000" diameter rod to be reduced as required as further requirements are defined

The order was completed as ordered on schedule.

SECTION VI

TEST PROGRAM AND PROCEDURES

A. Test Program

The testing specified in the original program description was divided into two parts. These were:

1. Screening Tests

These tests were to consist of oxidation tests on threaded specimens and bend tests on coated sheet specimens to determine the optimum tantalum alloy - tantalum coating combination for further evaluation. Relaxation tests were included in the early screening tests as a result of concern over the thick coatings.

2. Fastener Tests

These tests were to consist of mechanical tests of threaded fasteners and mechanically fastened joints made from the optimum combination of tantalum alloy and tantalum coating.

Tensile, shear, stress-relaxation and fatigue tests were to be included at temperatures up to 3500°F.

As the program evolved, it became expedient to devote considerably more attention to the screening phase and as it turned out the fastener test phase was not performed because of the limitations imposed by the coatings.

The procedures described herein are therefore primarily those used to screen the various alloys.

B. Test Procedures

1. Static Oxidation Tests

These tests were performed in an Astro Model 1000 graphite element tube furnace capable of operating with a ZrO_2 muffle to 3500°F. This furnace is shown in Figure 5. The carrier for the test pieces was a block of ZrO_2 foam with holes drilled and lined with a ThO_2 powder slurry.

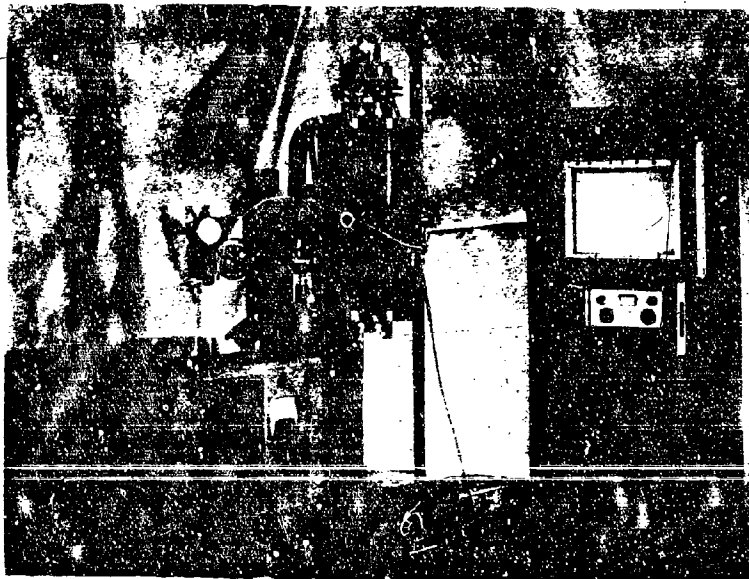


Figure 5. Astro Model 1000 - 4500°F Furnace.

The test specimens consisted of 1" x .250" pieces of T-222 with 1/2 inch of 1/4-20 threads on one end. This specimen configuration was chosen to provide separation of coating characteristics on threads and plain cylindrical surfaces (bolt shanks). In all tests, two specimens were run with one having the threads out of the ZrO_2 block and the other having the shank out of the block. This permitted us to discount the effect of contact with the ThO_2 coated ZrO_2 .

The test sequence was as follows:

- a. Load coated studs into ZrO_2 block.
- b. Load ZrO_2 block on lower furnace hearth and insert hearth into furnace.
- c. Close upper end of furnace leaving plug out to permit air passage through muffle.
- d. Attach air hose at top of furnace to blow across opening and create current of slowly moving air through muffle.
- e. Attach Ray-O-Tube to sight through top of furnace on test specimens.
- f. Purge graphite element chamber with argon.
- g. Turn furnace water and power on.
- h. Set controller to desired temperature.
- i. After parts reach temperature, hold for desired time and shut off power.

2. Stress-Relaxation Tests

These tests were designed to provide an indication of the degree to which the various coatings yield under compressive stress at elevated temperatures and thus cause loss of preload in a tightened joint. In order to do this, simulated joints were made up as in Figure 6.



Figure 6. Typical Stress-Relaxation Test Assembly.

The test sequence was as follows:

- a. Load coated studs into ZrO_2 block.
- b. Load ZrO_2 block on lower furnace hearth and insert hearth into furnace.
- c. Close upper end of furnace leaving plug out to permit air passage through muffle.
- d. Attach air hose at top of furnace to blow across opening and create current of slowly moving air through muffle.
- e. Attach Ray-O-Tube to sight through top of furnace on test specimens.
- f. Purge graphite element chamber with argon.
- g. Turn furnace water and power on.
- h. Set controller to desired temperature.
- i. After parts reach temperature, hold for desired time and shut off power.

The bolts, nuts and cylinders were used bare and the washers were coated as follows:

- a. Vitro Si/ WSi_2 + 2900°F Preoxidation
- b. Solar TNV-13 - Tungsten Barrier Only - No Siliciding
- c. Solar TNV-13 - Complete Coating
- d. Solar TNV-13 - Complete Coating + 2900°F Preoxidation

The bolts and nuts were tightened into the cylinders with two washers under the bolt head to provide a bolt elongation at .004 inch which produces a stress of 50,000 psi in the bolt.

Two washers were used to provide four coated surfaces in compression to more closely approximate the conditions of a coated bolt and nut joining a coated structure.

The assemblies were then exposed at 2400°F for one hour in dry argon.

Dimensions of the washers before and after the thermal cycle provided indications of the coating reaction to the applied stresses.

SECTION VII

TEST RESULTS

A. Oxidation Tests

1. Solar TNV-13

The first tests run were on specimens coated by Solar with their TNV-13 coating to five different coating thicknesses, .003, .004, .005, .006 and .007 inch.

Visual examination of the pieces revealed an uneven coating in the thread area. Metallographic sections of the pieces substantiated the visual examination. Attempts to measure the thread dimensions proved unsuccessful due to the uneven buildup.

Initial oxidation tests were performed on specimens with .003, .005 and .007 inch thick coatings. One cycle at 3000°F for 15 minutes resulted in complete destruction of the pieces.

Subsequently, three specimens were tested at 2600°F for 15 minutes with a Vitro Si/WSi₂ coated bolt included for comparison with the Solar coating. The three Solar coated studs failed in the threads and the Vitro coated piece survived the test. These pieces are shown in Figure 7. The 3500°F test cycle was repeated including a Vitro Si/WSi₂ coated bolt and once again the Solar pieces all failed in the threads while the Vitro specimen survived. No further tests were performed on the Solar TNV-13 coated specimens.

2. Vitro W/WSi₂

The first parts tested were those resulting from a program at Vitro to electrophoretically deposit a tungsten layer and subsequently partially vacuum siliconize to produce a WSi₂ coating over a tungsten diffusion barrier.

The initial parts received looked excellent and tolerance control on the threads was good as has always been the case with the electrophoretically applied coatings. (reference 2)

Oxidation tests at 3100°F for five minutes resulted in failure of the coating in the thread area, as did subsequent tests at 3000°F for five minutes.

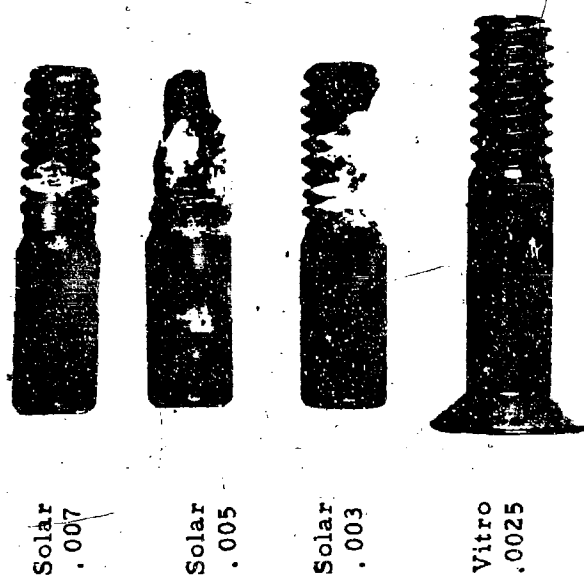


Figure 7. Parts Oxidation Tested Simultaneously at 2600°F for 15 Minutes.

A second lot of parts coated with a modified process also failed in 5 minutes at 3000°F. No further tests were performed on the Vitro coating

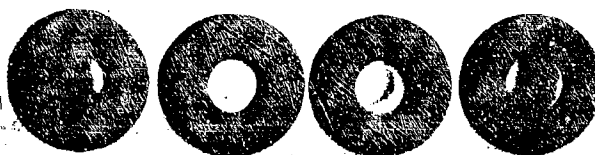
3. Sylvania R512C

The first lot of R512C coated specimens was tested at 3000°F. Thread failures occurred in a 10 minute cycle.

Additional pieces were tested at lower temperatures to define the temperature limit on threaded specimens. Thread failures occurred in the third 10 minute cycle at 2800°F and in the second thirty minute cycle at 2600°F. Both temperatures again produced thread failures.

B. Relaxation Tests

The significant results from the relaxation tests were those relating to the degree of deformation experienced by the various coatings as indicated by thickness changes and visual observations. The thickness readings at various points in the test are shown in Table II and some tested washers are shown in Figure 8.



Vitro Pre-Ox

Solar TNV-13
Pre-Ox

Solar
W Only

Solar TNV-13

Figure 8. Coated T-222 Washers after Relaxation Tests.

TABLE II

Relaxation Test Results
50,000 PSI Bolt Stress, 2400°F, 1 Hr., Argon

Thickness of Two Washers, inches

Coating	Prior to Test A	After Test Area Under Head B	After Test Area Beyond Head C	Thickness Loss Under Head A-B	Thickness Loss Beyond Head A-C	Thickness Difference Across Washers C-B	Coating Thickness Prior to Test $\frac{A-.040^*}{4}$
Solar - W Barrier Only	.0599	.0572	.0599	.0027	.0000	.0027	.0050
Solar TNV-13	.0692	.0659	.0681	.0033	.0011	.0022	.0073
Solar TNV-13							
Pre-oxidized	.0694	.0676	.0683	.0018	.0011	.0007	.0074
Vitro Si/WSi ₂ Pre-oxidized	.0515	.0502	.0489	.0013	.0026	.0013	.0029

* Thickness of two bare washers -.040.

It is important to note the "thickness loss under head" column and relate it to the "coating thickness prior to test" column. The largest loss was experienced by the complete Solar TNV-13 coating (.0033 inch) while the Vitro Si/WSi₂ coating experienced the least loss. This must, of course, be considered in light of the coating thicknesses which are quite different. On this basis it is noted that both coatings lost 45% of their thickness under these conditions.

The Solar TNV-13 coating, when pre-oxidized, loses only 24% of its thickness.

All of these values are significantly high when considered in relationship to the .004 inch elastic strain which was imposed upon the bolt to preload it into the joint. This "imbedding" of the bolt into coating, together with the relaxation of the bolt and nut (conversion of elastic strain to plastic strain), would probably result in nearly complete loss of preload.

SECTION VIII

CONCLUSIONS AND RECOMMENDATIONS

The principle conclusion to be drawn from this program is as follows:

The state-of-the-art in the coating of tantalum alloy fasteners is not sufficiently advanced to permit the use of mechanical fasteners in structures which must operate at 3000°F or above unless the design is such that the fastener threads can be protected by the structure, thus limiting their thermal exposure.

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- (1) R. E. Jackson and A. Lopatin, "Tantalum System Evaluation" AFFDL-TR-70-33, 9 April 1970.
- (2) Roach, Gowen, Ortner, and Klach, "Structural Fasteners for Extreme Elevated Temperatures" Final Report AFFDL TR-66-107, September, 1966.

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10. AVAILABILITY/LIMITATION NOTICES This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Flight Dynamics Laboratory (FBS), Wright-Patterson Air Force Base, Ohio 45433.		
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13. ABSTRACT A survey of available tantalum coatings was conducted to determine which might be capable of protecting threaded fasteners from oxidation at up to 3500°F. Sufficient tests were run on each selected coating to demonstrate that no coating is available to provide consistent protection at 3500°F, or in fact for 3000°F. Problems associated with thread forms and stress-relaxation were studied, but only in a general way due to the limitations imposed by the lack of satisfactory coatings.		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Tantalum Alloys Tantalum Fasteners Refractory Metals Refractory Coatings Structural Fasteners						

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