PRELIMINARY RESULTS OF A STUDY OF COATED Ta-10W SHEET FOR HEAT-SHIELD APPLICATIONS

By Gregory R. Wichorek and Bland A. Stein

NASA Langley Research Center
Langley Station, Hampton, Va.

Presented at the Tenth Meeting of
The Refractory Composites Working Group

Atlanta, Georgia [redacted]
April 12-14, 1965

PRELIMINARY RESULTS OF A STUDY OF COATED Ta-10W SHEET

FOR HEAT-SHIELD APPLICATIONS

By Gregory R. Wichorek and Bland A. Stein

NASA Langley Research Center

INTRODUCTION

Various programs at the Langley Research Center of NASA have been directed toward the use of refractory metals for thermal protection systems. A previous study explored some of the capabilities and limitations of a thermal protection system utilizing thin-gage molybdenum-alloy and columbium-alloy heat shields (ref. 1). A current program is concerned with the applicability of coated tantalum-alloy sheet for heat-shield applications in high-temperature oxidizing environments. The initial phase of this study was concerned primarily with the fabrication and evaluation of a limited number of small feasibility specimens. The results of static oxidation tests on these feasibility specimens indicated that the coated tantalum alloy was of sufficient promise to warrant the initiation of the second phase of the program. Heat shields have been designed and fabricated from thin-gage tantalum-alloy sheet to evaluate their performance under cyclic high-temperature exposure. Included in the second phase are additional oxidation tests of small specimens and mechanical property tests at room and elevated temperatures.

In this report, the results of continuous and cyclic oxidation tests at temperatures from 2000° F to 2900° F on the feasibility specimens are presented. In addition, the design and fabrication of the tantalum-alloy heat shields is discussed.

[redacted]

SPECIMENS, EQUIPMENT, AND PROCEDURES

Feasibility Specimens

Feasibility specimens used in the first phase of this investigation are shown in figure 1. Coupons 3/4 inch by $1\frac{1}{2}$ inches and leading edges of 1/2-inch radius by $1\frac{1}{2}$ inches wide were fabricated from Ta-10W tantalum-alloy sheet with a nominal thickness of 0.008 inch. Open-face sandwich specimens $1\frac{3}{4}$ inches by $2\frac{1}{8}$ inches were fabricated from 0.005-inch tantalum-alloy sheet by resistance welding. The sandwich consists of a face sheet, a corrugated sheet, and two transverse V-section stiffeners. All specimens were coated after forming with a tin-aluminum-molybdenum coating (Sn-27Al-5.5 Mo) by the Sylcor Division of the Sylvania Corporation under U.S. Air Force contract AF 33(657)-11272. This coating will be referred to hereinafter as aluminide coating.

Oxidation tests on the coated coupons were conducted in a vertical tube furnace in slowly moving air. Coupons were supported in high-purity alumina boats. For continuous oxidation tests the coupons were continuously weighed. In 1-hour cyclic tests, coupons were weighed and visually inspected before each cycle. In the cyclic exposure tests, visual evidence of tantalum oxide constituted coating failure. All coupons were subjected to bend tests and metallurgical studies following oxidation tests.

Coated leading-edge specimens were tested in an arc jet with a 4-inch-diameter nozzle which produces a subsonic airstream with a mass flow of 0.4 lb/ft^2-sec. The leading-edge specimens were exposed to the airstream for 6-minute cycles until coating failure was observed or an accumulated time of 1 hour (10 cycles) was achieved.

Cyclic oxidation tests on the coated open-face sandwiches were performed in a horizontal tube furnace. Sandwich specimens were supported on high-purity aluminum-oxide boats. After 1 hour at temperature, the specimens were removed from the furnace, allowed to air cool, weighed, and visually inspected before the cycle was repeated. Sandwich specimens were cycled until coating failure was observed or an accumulated time of 10 hours was achieved.

Heat Shields

The tantalum-alloy heat-shield design used in the second phase of this investigation is shown in figure 2. The corrugated skin, approximately 10 by 18 inches, and the plugs, approximately 3/4 by 3/4 inch, were formed from 0.008-inch Ta-10W tantalum-alloy sheet. Hat-section stiffeners and support clips were fabricated from 0.025-inch-thick sheet. The skin is corrugated in the longitudinal direction and resistance spotwelded to two transverse hat-section stiffeners to provide the necessary stiffness to transmit aerodynamic loads and resist flutter. Four support clips are resistance spotwelded to the hat sections and oriented to accommodate thermal expansion. Access holes in the corrugated skin permit attachment of the shield to a substructure. Plugs cover the access holes after the shield is attached to the substructure. Photographs of a fabricated heat shield are shown in figure 3.

The heat shields will be coated with the aluminide coating and subjected to cyclic oxidation tests in still air under a bank of quartz lamps. The shields will be attached to a substructure with fibrous insulation between the shield and substructure.

RESULTS OF FEASIBILITY SPECIMEN TESTS

The results of the oxidation tests on the coated feasibility specimens are given in table I. For heat-shield applications, cyclic temperature exposure of coupon, sandwich, and leading-edge specimens was considered to be a more realistic assessment of the oxidation protection provided by the coating than continuous exposure. Embrittlement and substrate losses after high-temperature exposure were determined from coupon specimens.

Oxidation

The effect of 1-hour temperature cycles in still air on the coating life of coupons is indicated in figure 4. Coating life of sandwich specimens is also presented in this figure to evaluate the effect of the more complex configuration on the capability of the coating to provide oxidation protection. The limited test data suggest that these two specimen configurations have comparable lifetimes at 2300° F and 2600° F. Above 2600° F, the shorter coating life of the sandwich specimens suggests that specimen configuration may be a controlling factor in coating life.

The coating life of leading-edge specimens subjected to 0.1-hour cycles in the arc jet is indicated in figure 5. Under these airflow conditions, ignition (a sudden increase in oxidation rate that could result in total disintegration of the specimen in a minute or less) occurred shortly after initial evidence of coating failure at 2600° F and 2900° F. Ignition was not observed in the static oxidation tests. The results of these arc-jet oxidation tests indicate that aluminide coated tantalum-alloy components may have limited applicability above 2600° F in dynamic oxidation conditions similiar to the test environment.

Embrittlement and Diffusion

Bend tests and microhardness traverses on the coupons as-coated and after oxidation tests indicated that although some embrittlement occurred during coating and subsequent exposure, sufficient ductility for heat-shield applications was apparently retained in the tantalum alloy until coating failure. Metallurgical examinations of the specimens indicated that solid-state diffusion significantly reduced substrate thickness, as illustrated in figure 6. In the as-coated condition, the Ta-10W substrate was 0.0074 inch thick. After 10 hours at 2600° F, the substrate thickness had been substantially reduced and after 190 hours at 2600° F, the substrate was 0.0039 inch thick.

During high-temperature exposure in air the coating can be considered to consist of three zones: (1) an outer zone of aluminum oxide, (2) a liquid aluminum-tin zone, and (3) a zone of one or more tantalum aluminides adjacent to the substrate. The results of substrate loss measurements (table I) indicated that, for comparable exposure times, approximately the same degree of substrate thickness loss occurred at 2300° F and 2900° F as that shown by the curve in figure 6 for 2600° F. A possible explanation for this behavior may be a more rapid increase in the rate of formation of the aluminum oxide zone compared to that of the aluminide zone with increasing temperature causing the depletion of aluminum in the aluminum-tin zone in the temperature range from 2300° F to 2900° F. Similar results were observed in reference 2 for aluminide coated Ta-10W.

CURRENT STATUS

Additional coupon and leading-edge specimens have been coated and testing is planned to expand the present data. The effects of flowing air at different mass flows will be investigated. Also tensile specimens have been coated for testing at room and elevated temperatures. Two heat shields have been fabricated and are presently being coated. The design and construction of the quartz-lamp heating apparatus is currently in progress.

REFERENCES

1. Wichorek, Gregory R., and Stein, Bland A.: Experimental Investigation of Insulating Refractory-Metal Heat-Shield Panels. NASA TN D-1861, 1964.
2. Sama, L.: High-Temperature Oxidation-Resistant Coatings for Tantalum Base Alloys. ASD-TDR-63-160, U.S. Air Force, February 1963.

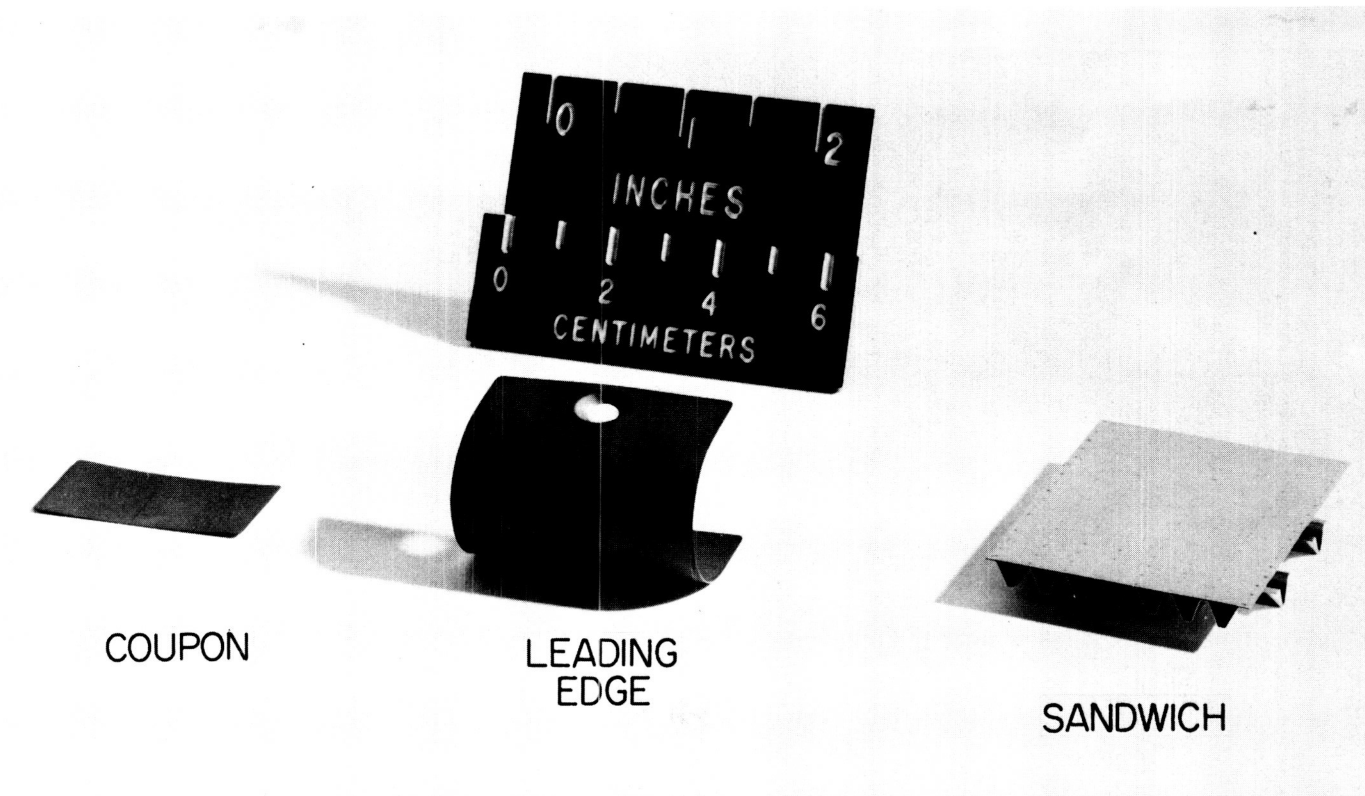
TABLE I.- RESULTS OF OXIDATION TESTS ON Ta-10W FEASIBILITY SPECIMENS

COATED WITH Sn-27Al-5.5 Mo COATING

[Tested in air at 760 mm Hg except as noted]

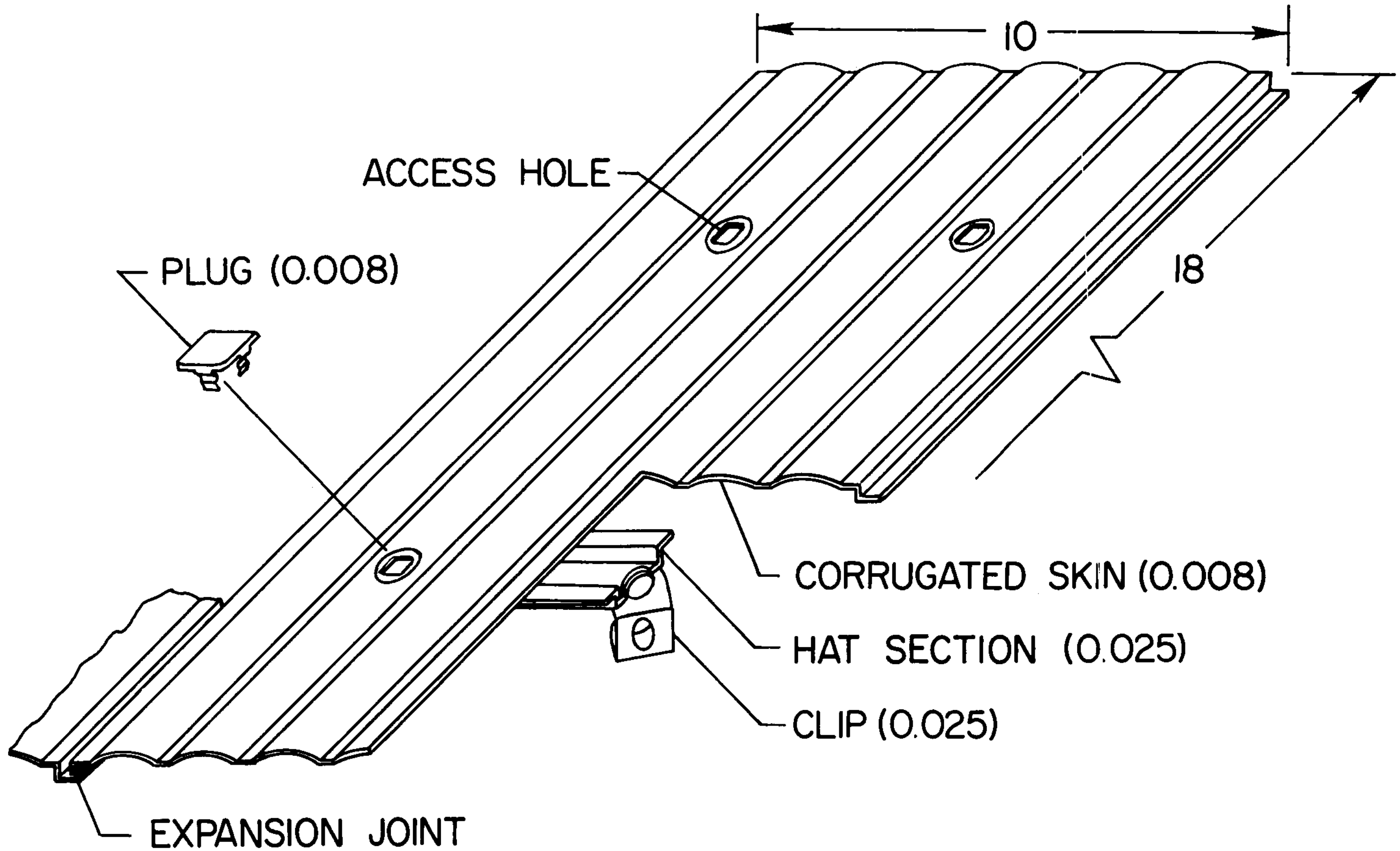
Specimen	Test apparatus	Test conditions	Test temperature, °F	Time to failure, hours	Substrate thickness loss, inch (e)
Coupon	Furnace	Continuous	2300	[a]>230	0.0041
			2600	[a]>190	.0035
			2600	[a]>5	.0017
			2600	[a]>20	.0019
			2900	[a]>75	.0031
		1.0-hour cyclic	2000	45	0.0009
			2300	18	.0022
			2600	10	.0026
			2900	10	.0025
			2900	6	.0019
		1.0-hour cyclic[b]	2400	[a]>20	0.0026
Sandwich	Furnace	1.0-hour cyclic	2300	[a]>10	
			2600	[a]>10	
			2750	3	
			2900	1.2	
Leading edge	Arc jet	0.1-hour cyclic	2300	[a]>1	
			2600	[c]0.50, [d]0.58	
			2900	[c]0.20, [d]0.25	

[a]Test discontinued for metallurgical examination.
[b]Tested at 0.5 mm Hg.
[c]First visible failure.
[d]Ignition.
[e]Based on as-coated substrate thickness of 0.0074.



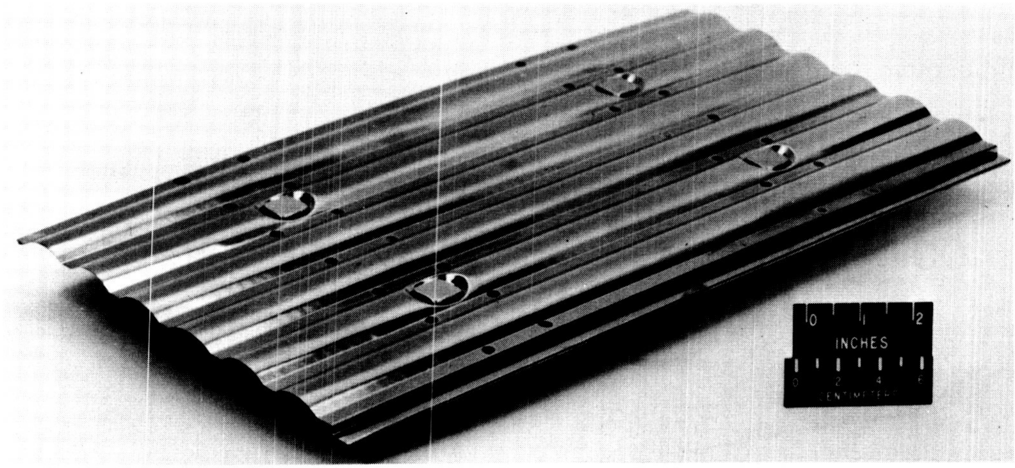
NASA

Figure 1.- Tantalum-alloy feasibility specimens for continuous and cyclic oxidation tests of aluminide coating.

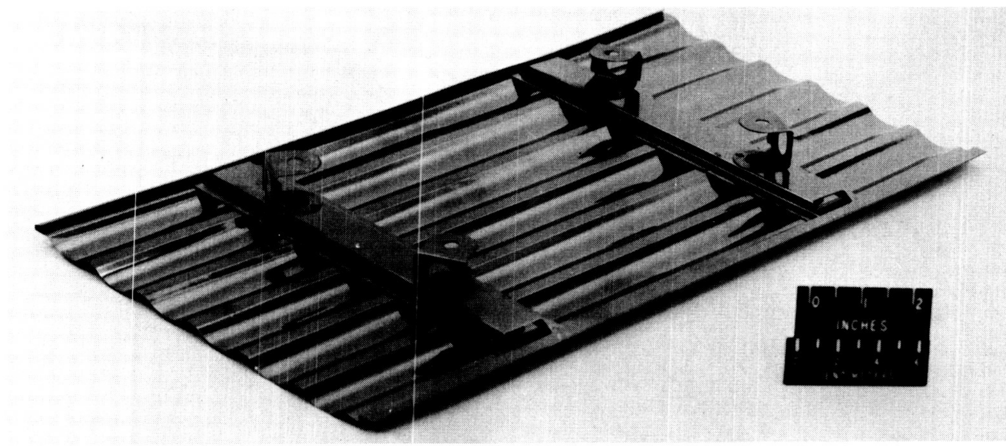


NASA

Figure 2.- Tantalum-alloy heat-shield design. Dimensions in inches.



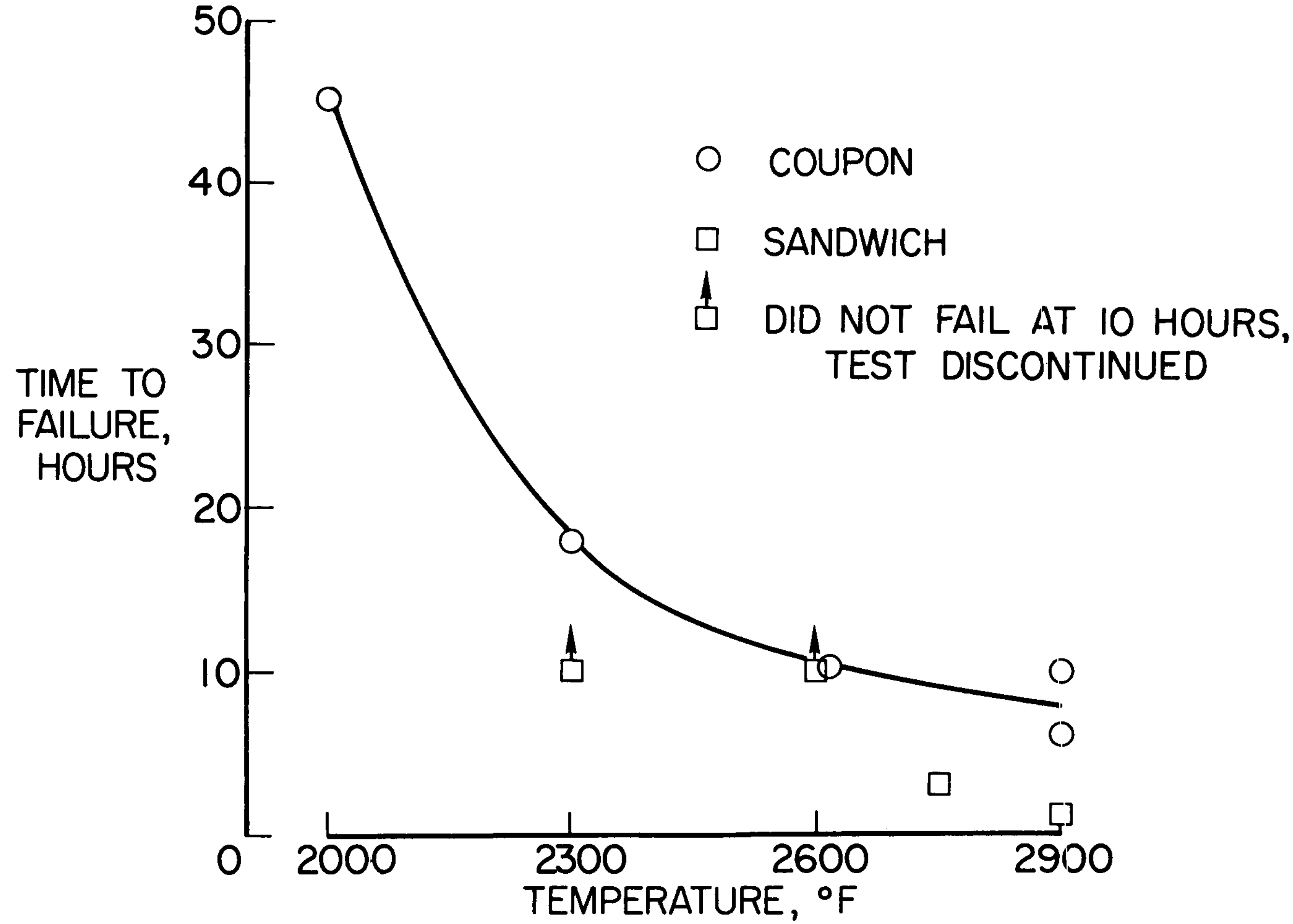
OUTER SURFACE



INNER SURFACE

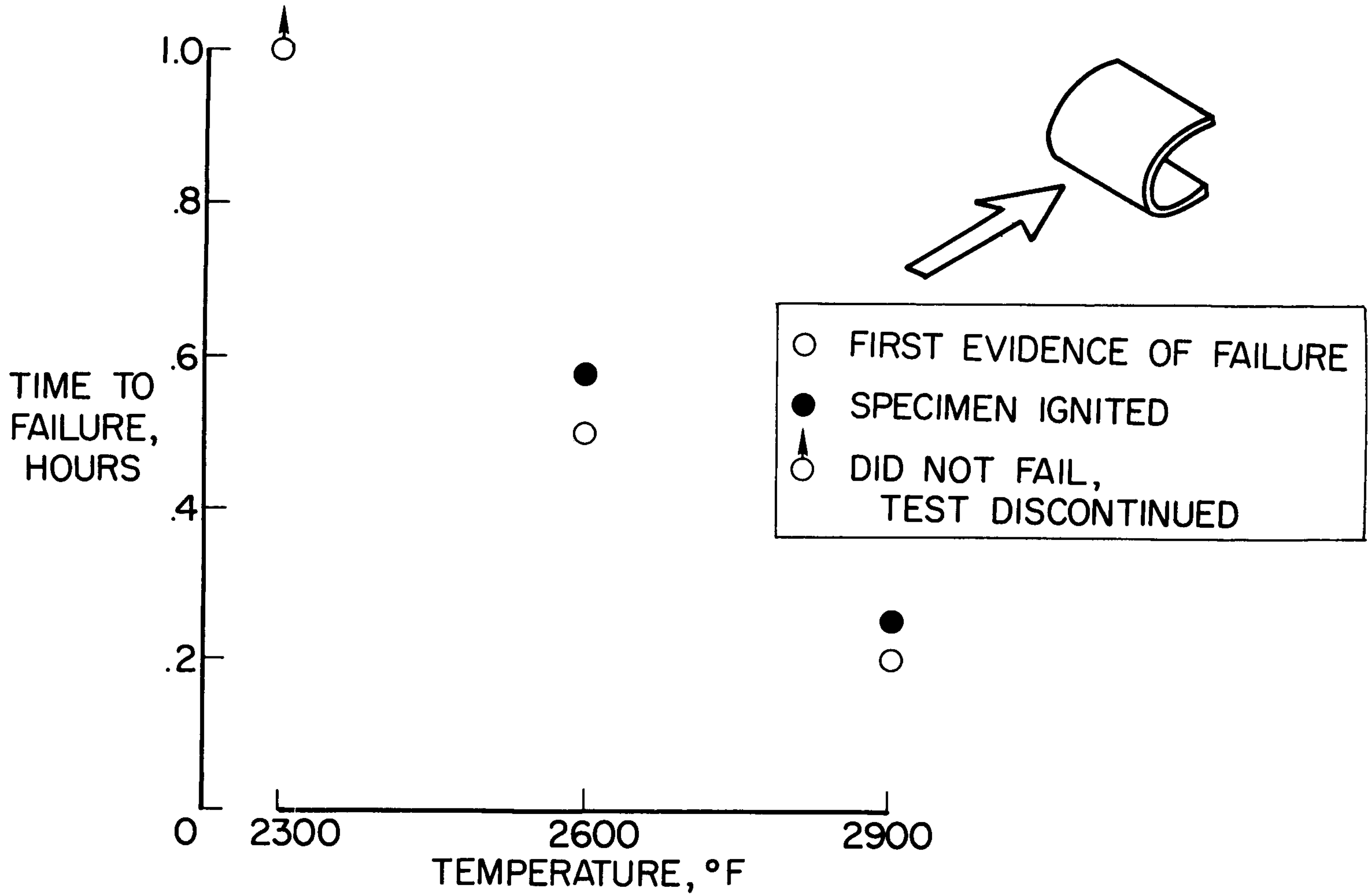
NASA

Figure 3.- Tantalum alloy heat shield for aluminide coating and evaluation under cyclic oxidation.



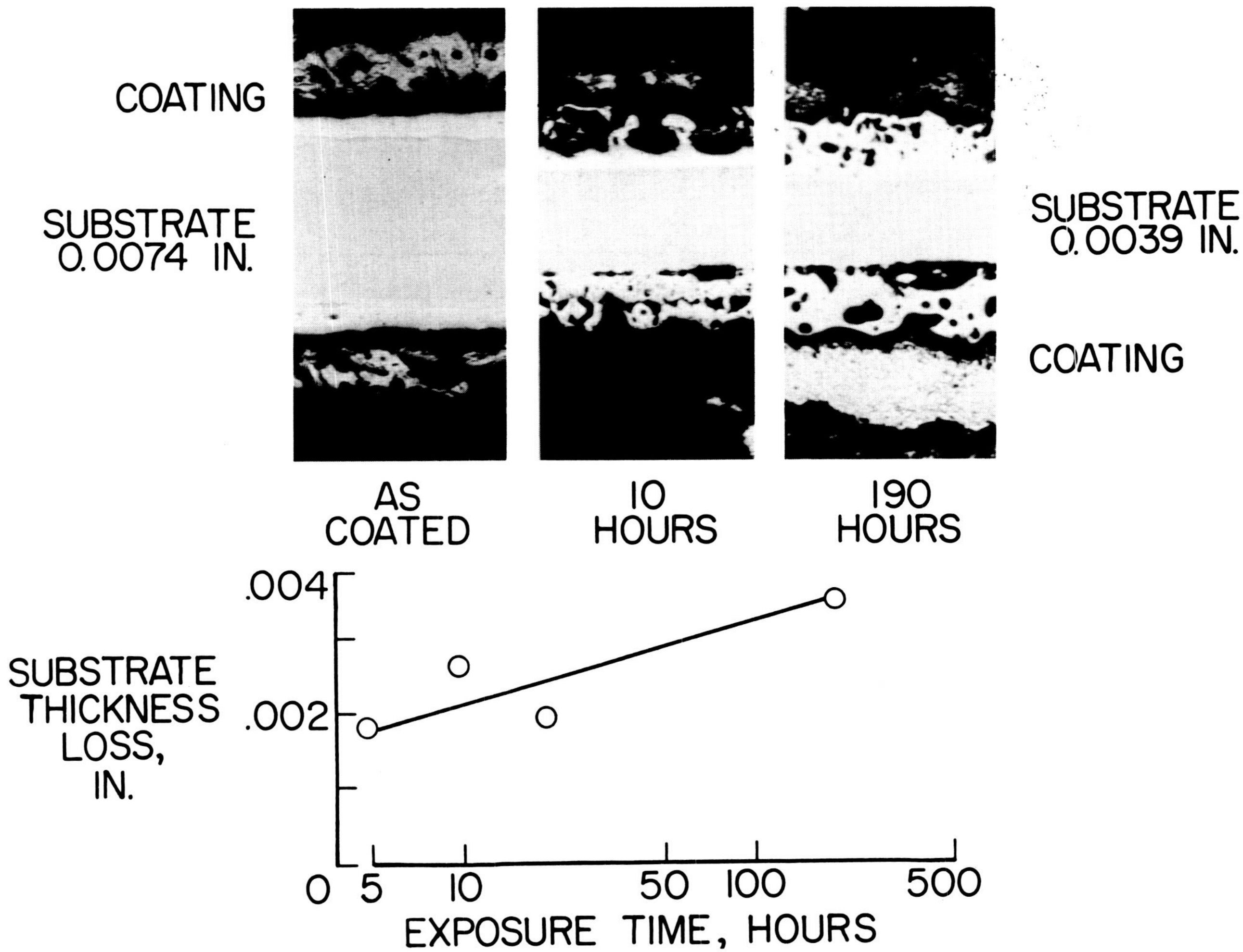
NASA

Figure 4.- Times to coating failure for aluminide coated Ta-10W coupon and sandwich specimens under 1.0-hour cyclic conditions in static air.



NASA

Figure 5.- Times to coating failure for aluminide coated Ta-10W leading-edge specimens under 0.1-hour cyclic conditions in arc jet.



NASA

Figure 6.- Variation of substrate thickness loss with exposure time at 2600° F for aluminide coated Ta-10W coupons. Photomicrographs ×150.

NASA-Langley, 1965