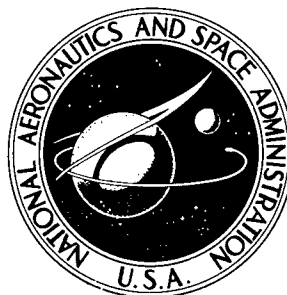


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AN EVALUATION OF COATED
MOLYBDENUM AND COLUMBIUM
ALLOYS IN HIGH-TEMPERATURE
SUPERSONIC AIRSTREAMS

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AN EVALUATION OF COATED MOLYBDENUM AND COLUMBIUM ALLOYS
IN HIGH-TEMPERATURE SUPERSONIC AIRSTREAMS

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Langley Research Center

SUMMARY

6/10/8
An experimental study was made to investigate the oxidation behavior of four commercially coated refractory metal systems in supersonic airstreams: Durak B^{Mc} and Vought IX^{Mc} coated TZM^{MS} and Cr-Ti-Si^{MS} and Cr-MoSi₂^{MS} coated Cb-752^{MS}. Cyclic coating life and temperature data were obtained on 0.375-inch-diameter (0.953 cm) blunt-nose models at surface temperatures from 2600° to 3000° F (1700° to 1920° K) and at air stagnation pressures behind the shock wave of 0.03, 0.10, and 5.5 atmospheres. The oxidation behavior of uncoated molybdenum- and columbium-alloy models of the same size and shape was also studied in the same test environments.

Coating life of the TZM/Durak B, Cb-752/Cr-Ti-Si, and Cb-752/Cr-MoSi₂ systems was significantly lower at air stagnation pressures of 0.10 and 0.03 atmosphere than at 5.5 atmospheres. At these low-pressure conditions, the coating life of these three coating systems did not exceed 32 minutes. The uncoated columbium alloys oxidized catastrophically at air stagnation pressures of 0.10 and 0.03 atmosphere in less than 20 seconds at temperatures well below the melting points of the alloys.

[500 - 1000 F]

INTRODUCTION

Alloys of the refractory metals, such as molybdenum and columbium, are among the materials being considered for radiatively cooled heat shields in lifting reentry vehicles. These heat shields are expected to have maximum surface temperatures from 2040° to 3540° F (1390° to 2220° K) at less than normal atmospheric pressure. (See ref. 1.) Heat shields using the refractory metals are capable of multiple reentries, depending on the time-temperature environment. However, these alloys begin to oxidize below 2000° F (1370° K) and form oxides which do not protect the base metal. Molybdenum alloys oxidize to a gaseous product with a consequent loss of material. Columbium alloys oxidize to porous oxide scales which permit further alloy oxidation. Numerous attempts have been made to increase the oxidation resistance of these alloys to an acceptable level without degrading alloy properties, but none have been successful. (See refs. 2, 3, and 4.) Therefore, refractory inorganic coatings were developed to protect these alloys from oxidation.

To evaluate the oxidation resistance of coated refractory alloys, many oxidation studies have been made in static or low flow-rate environments. (See ref. 5.) These studies were usually carried out at an air pressure of 1 atmosphere (0.1013 MN/m²) and for 1-hour exposure cycles. Some studies were made in static environments at reduced pressures. (See ref. 6.) Very few studies have been made to determine the oxidation response of these coated alloy systems in dynamic environments.

A dynamic environment has two possible effects on a coated surface which are not present in a static environment: one is a shearing effect of a liquid or loose solid at the surface; the other is a more rapid transport of oxygen to the surface and a more rapid diffusion from the surface of any gas produced at the surface. Very limited dynamic test data exist for a pressure range from 0.01 to 1 atmosphere; therefore, it was difficult to evaluate the effects of a dynamic environment on coating performance. (For example, see refs. 7, 8, and 9.)

The primary purpose of the present experimental study was to investigate the oxidation behavior of selected coating-alloy systems in a range of dynamic environments. Another purpose was to compare coating performance in dynamic environments with static test data in the literature. Since a coating failure could expose the substrate material to oxidation, a final purpose was to determine the oxidation behavior of uncoated alloys in the same dynamic environments used for the coated alloys.

The experimental program was carried out with four commercially coated refractory metal systems: Durak B and Vought IX on molybdenum alloy TZM and Cr-Ti-Si and Cr-MoSi₂ on columbium alloy Cb-752. These two alloys and columbium alloy B-66 were used in uncoated-alloy oxidation studies.

Two test parameters considered important in the response of coated refractory metals to earth reentry flight environments are surface pressure and surface temperature. In the tests reported herein, the stream conditions produced stagnation pressures behind the shock wave $p_{t,2}$ of 0.03, 0.10, and 5.5 atmospheres and surface temperatures from 2545° to 3180° F (1670° to 2020° K) on the coated models. The same stream conditions were used for the uncoated alloy models and produced the same stagnation pressures, but surface temperatures ranged from 2770° to 4400° F (1795° to 2700° K).

METHODS AND TESTS

Models and Techniques

The refractory metal models used in the present study were machined from solid stock to a 0.375-inch-diameter (0.953 cm) blunt-nose shape as shown in figure 1. This shape was selected in an attempt to obtain a more uniform heating rate and surface temperature on the face of the model than could be obtained on a hemispherical model (see

ref. 10). Maximum heating rate limitations in two of the test facilities made the small model size necessary to obtain surface temperatures in the range of interest. Early tests produced surface temperatures lower than expected, and this was corrected by installing behind the model an alumina insulator which minimized heat conduction losses from the back of the model to the water-cooled holder as shown in figure 2.

The two alloys and four coatings used in the tests are listed in table 1, along with the one additional alloy which was tested in the uncoated condition. Alloy compositions and coating compositions, processes, and thicknesses are given in the table. The coatings are all commercially available silicide-base coatings. They were selected on the basis of superior oxidation protection in static oxidation tests made by the University of Dayton Research Institute. (See ref. 11.) Nominal coating thicknesses listed in table 1 are contract specifications consistent with values reported in the literature (see ref. 12).

Both uncoated and coated models were tested at three stagnation pressure levels – 5.5, 0.10, and 0.03 atm – with each pressure level being obtained in a different facility, as shown in table 2. The observed surface-temperature ranges are listed for both the coated and uncoated models. The use of different facilities prevented the exact duplication of surface temperatures at all three pressure conditions. Surface temperatures on the coated models were approximately the same in two of the facilities (for pressures of 5.5 and 0.10 atm) but were approximately 200° to 400° F (111° to 167° K) higher in the third facility (for a pressure of 0.03 atm) due to a minimum heating rate limitation at this pressure condition. All uncoated models were tested in the same supersonic airstreams used for the coated models, but surface temperatures were higher on the uncoated models due to the exothermic heat of reaction between the metal alloy and the heated airstream.

The three facilities used in this study are listed in table 2. The Langley 11-inch ceramic-heated tunnel is described in reference 13. It was fitted with a Mach 2 nozzle having an exit diameter of 1.332 in. (3.38 cm) and exhausting to the atmosphere. Figure 3 shows a coated model in test position in this facility. Each coated model was tested in 8-min cycles. The Langley 20-inch hypersonic arc-heated tunnel is described in references 14 and 15. It was fitted with a Mach 6 nozzle having an exit diameter of 6.6 in. (16.76 cm). Figure 4 shows four coated models in test position in this facility. These models were tested in 5-min cycles. The high-enthalpy arc tunnel at the Langley Research Center is described in the appendix. Figures 5 and 6 show models in test position in this facility. These models were also tested in 5-min cycles.

The models were held in the test streams by water-cooled stings. A model holder used in the ceramic-heated tunnel is shown in figure 2. A model holder used to test 4 models simultaneously in the two arc-heated facilities is shown in figure 7. The models

in this holder are located on a diameter concentric with the diameter of the test stream. The area blocked by the model holder is about 23 percent of the cross-sectional area of the test stream. Results of unpublished surveys of pressure and heating rate in the high-enthalpy arc tunnel have indicated that the four models were in a region of uniform flow and were exposed to the same test conditions. A similar result has been obtained for the 20-inch hypersonic arc-heated tunnel with regard to pressure. No detailed surveys of the heating rate in this tunnel have been made, but temperature surveys made with models of like composition indicated that the four models were probably exposed to the same test conditions in the tunnel.

In all three test facilities the test procedure was generally the same. After flow stabilization of the airstream was obtained, motion-picture cameras and a photographic pyrometer were turned on and the model, or models, was inserted into the test stream. Surface temperature on the coated model stabilized in about 20 seconds after insertion. An optical pyrometer was used to observe the model under test. The model was removed from the test stream at the specified test time, or earlier if it appeared to oxidize rapidly. In tests of coated models, coating failure (i.e., exposure of the substrate) was detected either during the test cycle by observation of a melting or flame phenomenon or after the test cycle by eye or with the aid of a low-power microscope. The life of a coating is defined as the cumulative test time to failure.

Instrumentation and Data Reduction

The surface temperature of the model was measured by a photographic pyrometer, which is described in reference 16. Since the model could only be photographed from the side, the area selected for temperature measurement and resolved by the readout system was located on the curved shoulder of the model adjacent to the stagnation zone, as shown in figure 8. Temperature traverses across the curved shoulder of several models indicated a linear temperature decrease with axial distance from the front face. A linear extrapolation to the visible edge of the front face or stagnation zone indicated that the temperature measured on the shoulder was about 2 percent lower than the extrapolated value at the edge of the stagnation zone. The surface temperature at the edge of the stagnation zone is believed to be representative of the temperature in the stagnation zone, based on the assumption of a constant heating rate in this zone (see ref. 10). Therefore, measured surface temperatures are believed to be about 2 percent below the stagnation-zone surface temperatures. Surface brightness temperatures were corrected for spectral emittance according to the assumed values listed in table 3 and cited in references 17 and 18. The photographic pyrometer has a measurement imprecision of ± 2 percent. A ± 20 -percent error in the assumed emittance values would result in a measurement error of about ∓ 2 percent in the temperature range from 2500° to 3000° F (1645° to 1920° K). (See ref. 16.) Temperature data were recorded every 1 or 2 sec for

all test runs. The temperature reported for each coated model under test is the average of the temperatures measured in two or more test cycles. A 2-percent temperature variation was observed between test cycles.

RESULTS AND DISCUSSION

Uncoated-Model Tests

If a coating failure occurs on a coated alloy and exposes that alloy to the oxidizing atmosphere, the exposed alloy could oxidize as an uncoated alloy. Therefore, it was important to characterize the oxidation behavior of uncoated alloys in the same dynamic environments used for the coated alloys. The responses of the three uncoated metals, which were exposed to the same dynamic oxidizing environments as the coated metals, are listed in table 4 and are shown in figure 9. In figure 9, "noncatastrophic" oxidation describes a test in which no liquid phase formed; "catastrophic" oxidation describes a test in which a liquid phase formed with a consequent rapid removal of material.

Molybdenum alloy TZM.- At 5.5 atm, the surface temperatures of the two uncoated models of molybdenum alloy TZM were well above the surface temperatures of 2655° to 2680° F (1730° to 1745° K) for the coated molybdenum-alloy models in the same stream (see tables 2 and 4). A liquid phase was observed to form in less than 30 seconds at temperatures well below the melting point of the alloy (see table 4 and fig. 9). A similar response was reported for pure molybdenum models exposed to a 10.5-atm airstream in reference 19. At 0.1 atm, temperatures of 3220° to 3315° F (2045° to 2095° K) were observed, but no liquid phase formed. At 0.03 atm, temperatures of 3600° to 3845° F (2255° to 2390° K) were observed. A liquid phase formed on one of the four models at a temperature of 3835° F (2385° K), which is well below the melting point of the alloy. This liquescent phenomenon was similar to that observed for this alloy at 5.5 atm except that the model did not recede rapidly at 0.03 atm. The surface temperatures at 0.03 atm were higher than those at 0.10 atm, which was probably due to the higher heating rate at 0.03 atm (see table 2).

Columbium alloys Cb-752 and B-66.- The uncoated columbium alloys (Cb-752 and B-66) are discussed together because of similarities in oxidation response at all three pressure conditions. (See table 4 and fig. 9.) A liquid phase was observed to form on both alloys at all three pressure levels and at surface temperatures well below the melting point of the metal. This liquescent phenomenon occurred in less than 25 sec at 5.5 atm and in less than 20 sec at 0.03 and 0.10 atm. This phenomenon has been observed on columbium metal in a static oxidation test at 1 atm in oxygen and at 2550° F (1670° K) (see ref. 20). The observed temperatures at which liquid formed are near the melting points of the base-metal oxides (see ref. 21); this result suggests that one or more of these oxides may have formed on these alloys.

From the foregoing discussion on the oxidation response of the uncoated metals, it is concluded that at the low pressures of interest for reentry flight missions, the exposure of any of the columbium alloys of this study by a coating failure could result in the catastrophic oxidation of the base metal in 20 sec or less.

Coated-Model Tests

All four coating-alloy systems are represented in figures 10 and 11 which show magnified front-face views of some of the models tested in the three dynamic environments. In general, the response shown by each of the models in these figures illustrates failure mode. Average surface temperature and coating life are listed for each model shown. The coating life of the coated Cb-752 models tested at 5.5 atm is shown as >72 min, meaning that testing was discontinued after 72 min of total test time without visual evidence of coating failure. Table 5 lists the test results for the coated TZM and Cb-752 models and figures 12 and 13 show these data plotted for each coating system.

Molybdenum alloy TZM.- Figure 12 shows that the coating life of the TZM/Durak B system was significantly lower at 0.10 and 0.03 atm than at 5.5 atm. At the lower pressures, coating life did not exceed 32 min. The lower lifetimes at reduced pressure may be due to a low-pressure decomposition of the protective silica layer that forms on the surface of silicide-base coatings. (See ref. 9.)

A comparison was made between the coating life of the TZM/Durak B system in the dynamic oxidation environment of this study and the coating life of this system in a static oxidation environment (see table 6(a)). At approximately the same pressure of 0.03 atm and average coating life, surface temperatures for the dynamic tests were about 200° to 300° F (111° to 167° K) lower than they were for the continuous static tests. This behavior is equivalent to lower coating life at a given temperature and is not surprising in view of the dynamic environment and short test cycle time (see ref. 7).

Lifetimes for the TZM/Vought IX system varied considerably at both high and low pressures, probably an indication of coating nonuniformity among models (see fig. 12). Because of this large variation, no trend with pressure could be defined.

Columbium alloy Cb-752.- Since the behavior of the Cr-Ti-Si and Cr-MoSi₂ coatings on columbium alloy Cb-752 was similar at all three test conditions, the response of these coatings will be discussed jointly.

At 5.5 atm, tests were stopped on both coatings when no failure occurred after a total test time of 72 min (see fig. 13). At 0.10 atm, both coatings failed in less than 20 min. At 0.03 atm, both coatings failed in 32 min or less. These results indicate that a sharp drop in coating life occurred for the Cb-752/Cr-Ti-Si and Cb-752/Cr-MoSi₂ systems when air stagnation pressure was reduced from 5.5 to 0.10 atm. At the lower pressures of 0.10 and 0.03 atm, coating life did not exceed 32 min.

The response of the Cb-752/Cr-Ti-Si system in the dynamic environment of this study is compared with the response of this system in a static environment at approximately the same pressure conditions (see table 6(b)). The results indicate that the static or dynamic state of the environment was apparently not critical to coating performance at an air pressure of 0.03 atm and at surface temperatures of 2880° to 3180° F (1855° to 2020° K) as evidenced by the low coating life and the melting of the models.

CONCLUDING REMARKS

An experimental study was made to investigate the oxidation behavior of uncoated and commercially coated molybdenum alloy TZM and columbium alloy Cb-752 in supersonic airstreams.

Coating life for the TZM/Durak B system was lower at air stagnation pressures of 0.10 and 0.03 atmosphere than at 5.5 atmospheres. At the lower pressures, coating life did not exceed 32 minutes. At approximately the same pressure of 0.03 atmosphere and average coating life, surface temperatures for the dynamic tests were 200° to 300° F (111° to 167° K) lower than that reported for continuous static tests.

No coating-life trend with pressure was found with the TZM/Vought IX system due to the large variation of coating life at air stagnation pressures of 5.5, 0.10, and 0.03 atmospheres.

A sharp drop in coating life occurred for the Cb-752/Cr-Ti-Si and Cb-752/Cr-MoSi₂ systems when air stagnation pressure was reduced from 5.5 atmospheres to 0.10 atmosphere. At air stagnation pressures of 0.10 and 0.03 atmosphere, coating life did not exceed 32 minutes. The static or dynamic state of the environment was not found to be critical to the coating performance of the Cb-752/Cr-Ti-Si system at an air pressure of 0.03 atmosphere and at surface temperatures of 2880° to 3180° F (1855° to 2020° K).

The uncoated columbium alloys (Cb-752 and B-66) tested in the same reduced-pressure dynamic test streams used for the coated alloys (0.03 and 0.10 atmosphere) were found to oxidize catastrophically by forming liquid phases in less than 20 seconds. Liquescent temperatures were below the melting points of the uncoated alloys.

The uncoated molybdenum alloy (TZM) and columbium alloys (Cb-752 and B-66) tested in the same high-pressure dynamic test streams used for the coated alloys (5.5 atmospheres) were found to oxidize catastrophically in less than 30 seconds. Liquescent temperatures were below the melting points of the uncoated alloys.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., August 19, 1968,
129-03-12-05-23.

APPENDIX

THE HIGH-ENTHALPY ARC TUNNEL AT THE LANGLEY RESEARCH CENTER

The high-enthalpy arc tunnel at the Langley Research Center is a materials test facility capable of producing high-temperature supersonic gas streams having total enthalpies in the range from 10 000 to 12 000 Btu/lbm (23 to 28 MJ/kg). The high-temperature supersonic gas stream, consisting of air or nitrogen, is produced by passing the gas through an arc heater and then expanding it through a conical nozzle to an evacuated test section.

To provide a wide range of test conditions, three nozzle sizes and a range of arc-chamber pressure are available. All three nozzles have throat diameters of 0.75 in. (1.9 cm), and are provided with exit diameters of 2 in. (5.1 cm), 4.6 in. (12 cm), and 7.6 in. (19 cm). Arc-chamber pressure may be varied between 1.0 and 3.3 atm. The general arrangement of this facility is shown in figure 6.

REFERENCES

1. Czarnecki, E. G.; and Braun, M. T.: Development and Evaluation of Coatings for Earth Re-Entry Systems. The Science and Technology of Tungsten, Tantalum, Molybdenum, Niobium and Their Alloys, N.E. Promisel, ed., AGARDograph 82, Pergamon Press, c.1964, pp. 357-368.
2. Rengstorff, G. W. P.: Search for Oxidation-Resistant Alloys of Molybdenum. Trans. Amer. Inst. Mining Met. Petrol. Eng., vol. 206, 1956, pp. 171-176.
3. Klopp, W. D.: Oxidation Behavior and Protective Coatings for Columbium and Columbium-Base Alloys. DMIC Rep. 123, Battelle Mem. Inst., Jan. 15, 1960.
4. Levinstein, M. A.; and Wlodek, S. T.: Effects of Alloying and Coating on the Elevated Temperature Corrosion Resistance of the Refractory Metals. The Science and Technology of Tungsten, Tantalum, Molybdenum, Niobium and Their Alloys, N. E. Promisel, ed., AGARDograph 82, Pergamon Press, c.1964, pp. 331-356.
5. Gibeaut, W. A.; and Bartlett, E. S.: Properties of Coated Refractory Metals. DMIC Rep. 195, Battelle Mem. Inst., Jan. 10, 1964.
6. Price, W. L.; and Perkins, R. A.: Behavior of Silicide Coatings in High Temperature-Low Pressure Environment. Summary of the Seventh Refractory Composites Working Group Meeting, Vol. I, RTD-TDR-63-4131, U.S. Air Force, Nov. 1963, pp. 266-275.
7. Stein, Bland A.; and Lisagor, W. Barry: Oxidation and Embrittlement in Silicide-Coated Columbium-Alloy and Aluminide-Coated Tantalum-Alloy Sheet. Paper presented at the Fourth Symposium on Refractory Metals, Institute of Metals Division, The Metallurgical Society of AIME (French Lick, Ind.), Oct. 3-5, 1965.
8. Wichorek, Gregory R.; and Stein, Bland A.: Preliminary Results of a Study of Coated Ta-10W Sheet for Heat-Shield Applications. Summary of the Tenth Refractory Composites Working Group Meeting, AFML-TR-65-207, U.S. Air Force, Aug. 1965, pp. 202-215.
9. Perkins, R. A.; and Packer, C. M.: Coatings for Refractory Metals in Aerospace Environments. AFML-TR-65-351, U.S. Air Force, Sept. 1965. (Available from DDC as AD 480862.)
10. McLain, Allen G.; Sutton, Kenneth; and Walberg, Gerald D.: Experimental and Theoretical Investigation of the Ablative Performance of Five Phenolic-Nylon-Based Materials. NASA TN D-4374, 1968.
11. Wurst, J. C.; and Cherry, J. A.: The Evaluation of High Temperature Materials. ML-TDR-64-62, Vol. II, U.S. Air Force, Sept. 1964.

12. Moore, V. S.; and Stetson, A. R.: Evaluation of Coated Refractory Metal Foils. RTD-TDR-63-4006, Pt. I, U.S. Air Force, Sept. 1963.
13. Trout, Otto F., Jr.: Design, Operation, and Testing Capabilities of the Langley 11-Inch Ceramic-Heated Tunnel. NASA TN D-1598, 1963.
14. Schaefer, William T., Jr.: Characteristics of Major Active Wind Tunnels at the Langley Research Center. NASA TM X-1130, 1965.
15. Miller, Irvin M.; and Sutton, Kenneth: An Experimental Study of the Oxidation of Graphite in High-Temperature Supersonic and Hypersonic Environments. NASA TN D-3444, 1966.
16. Exton, Reginald J.: Theory and Operation of a Variable Exposure Photographic Pyrometer Over the Temperature Range 1800° to 3600° F (1255° to 2255° K). NASA TN D-2660, 1965.
17. Hodgman, Charles D.; Weast, Robert C.; and Wallace, Clarence W., eds.: Handbook of Chemistry and Physics. Thirty-fifth ed., Chem. Rubber Pub. Co., c.1953, p. 2678.
18. Refractory Metals Sheet Rolling Panel, Coating Subpanel: Coated Refractory Metal Technology - 1965. Oxidation-Resistant Coatings for Refractory Metals. Publ. MAB-210-M (Contract No. SD-118), Nat. Acad. Sci.-Nat. Res. Council, Nov. 1965. (Available from DDC as AD 475754.)
19. Schryer, David R.; and Walberg, Gerald D.: A Theoretical and Experimental Investigation of the Oxidation of Molybdenum at Temperatures at Which Its Trioxide is Volatile. NASA TR R-232, 1966.
20. Klopp, William D.; Sims, Chester T.; and Jaffee, Robert I.: High-Temperature Oxidation and Contamination of Columbium. Trans. Amer. Soc. Metals, vol. LI, 1959, pp. 282-298.
21. Hague, J. R.; Lynch, J. F.; Rudnick, A.; Holden, F. C.; and Duckworth, W. H., eds.: Refractory Ceramics for Aerospace. Amer. Ceram. Soc., Inc., 1964.

TABLE 1.- METALS AND COATINGS

Metal alloy		Coating				Nominal thickness	
Name	Composition	Manufacturer	Name	Composition	Process	mils	μm
Molybdenum alloy TZM	Mo-0.5Ti-0.08Zr	Chromizing Corporation	Durak B	MoSi ₂ + proprietary addition	Two-cycle pack cementation	2.0 ± 0.5	0.51 ± 0.13
		Ling-Temco-Vought, Inc.	Vought IX	MoSi ₂ + B and Cr	One-cycle pack cementation	2.0 ± 0.5	0.51 ± 0.13
Columbium alloy Cb-752	Cb-10W-2.5Zr	Thompson Ramo Wooldridge	Cr-Ti-Si	1st layer, Cr and Ti; 2d layer, Si	Two-cycle reduced-pressure pack cementation	2.5 to 3.0	0.64 to 0.76
		Vitro Laboratories	Cr-MoSi ₂	1st layer, Cr; 2d layer, MoSi ₂	Two-cycle electrophoresis	2.5 to 3.0	0.64 to 0.76
Columbium alloy B-66	Cb-5Mo-5V-1Zr	None					

TABLE 2.- TEST CONDITIONS

Test facility		Langley 11-inch ceramic-heated tunnel	Langley 20-inch hypersonic arc-heated tunnel	High-enthalpy arc tunnel at the Langley Research Center
Air stagnation pressure, $P_{t,2}$, atm		5.5	0.10	0.03
Surface temperatures	Molybdenum alloy TZM	Coated	2650° to 2725° F (1730° to 1770° K)	2790° to 2905° F (1805° to 1870° K)
		Uncoated	3220° to 3315° F (2045° to 2095° K)	3600° to 3845° F (2255° to 2390° K)
	Columbium alloy Cb-752	Coated	2630° to 2735° F (1715° to 1775° K)	2545° to 2750° F (1670° to 1785° K)
		Uncoated	2860° to 2920° F (1845° to 1880° K)	2770° F (1795° K)
Test cycle, min		8	5	5
Hot wall heating rate	Coated models	300 to 320 Btu/ft ² -sec (3.40 to 3.63 MW/m ²)	238 to 254 Btu/ft ² -sec (2.70 to 2.88 MW/m ²)	600 to 670 Btu/ft ² -sec (6.82 to 7.61 MW/m ²)
	Uncoated models	-16.0 to 277 Btu/ft ² -sec (-0.181 to 3.14 MW/m ²)	261 to 294 Btu/ft ² -sec (2.96 to 3.34 MW/m ²)	605 to 620 Btu/ft ² -sec (6.87 to 7.04 MW/m ²)

TABLE 3.- SPECTRAL EMITTANCE OF UNCOATED AND COATED
REFRACTORY METAL ALLOYS

Metal alloy			Coating		
Name	Spectral emittance (0.65 μm)	Reference	Name	Spectral emittance (0.65 μm)	Reference
Molybdenum alloy TZM	^a 0.50	17	Durak B	^b 0.80	
			Vought IX	^b 0.80	
Columbium alloy Cb-752	^c 0.70	17	Cr-Ti-Si	0.70	18
			Cr-MoSi ₂	^b 0.80	
Columbium alloy B-66	^c 0.70	17	None		

^aEmittance of titanium oxide used for oxidized form of alloy.

^bEmittance of this coating taken to be same as that for PFR-6 silicide coating in reference 5.

^cEmittance of columbium oxide used for oxidized form of alloy.

TABLE 4.- OXIDATION RESPONSE OF UNCOATED ALLOY MODELS - Concluded

(b) SI Units

Metal alloy	Melting point, °K	Test time, sec	Air stagnation pressure $P_{t,2}$, atm																							
			5.5				0.10				0.03															
			Liquid formation		No liquid; max. surface temp., °K	Liquid formation		No liquid; max. surface temp., °K	Liquid formation		No liquid; max. surface temp., °K	Liquid formation		No liquid; max. surface temp., °K												
Time, sec	Surface temp., °K	Time, sec	Surface temp., °K	Time, sec		Surface temp., °K	Time, sec		Surface temp., °K																	
Molybdenum alloy TZM	2885	24	23	2375																						
		29	28	2700																						
		300																								
		300																								
Columbium alloy Cb-752	2700	133																								
		133																								
		133																								
		133																								
Columbium alloy B-66	2645	120																								
		480																								
		20	20	1845																						
		20	16	1880																						
Columbium alloy B-66	2645	20																								
		24																								
		24	24	1905																						
		19																								
Columbium alloy B-66	2645	24																								
		19																								
		24																								
		24																								

TABLE 5.- RESPONSE TO DYNAMIC TEST ENVIRONMENTS

(a) Coated TZM models

Coating	Air stagnation pressure, $P_{t,2}$, atm	Surface temperature		Coating life, min	
		$^{\circ}\text{F}$	$^{\circ}\text{K}$		
Durak B	5.5	2660	1735	48	
		2680	1745	53	
	0.10	2675	1740	21	
		2725	1770	21	
	0.03	2830	1830	32	
		2890	1860	22	
		2905	1870	28	
	Vought IX	5.5	2655	1730	16
			2665	1735	44
0.10		2650	1730	16	
		2650	1730	42	
		2655	1730	11	
0.03		2790	1805	62	
		2815	1820	25	

(b) Coated Cb-752 models

Coating	Air stagnation pressure, $P_{t,2}$, atm	Surface temperature		Coating life, min
		$^{\circ}\text{F}$	$^{\circ}\text{K}$	
Cr-Ti-Si	5.5	2675	1740	>72
		2735	1775	>72
	0.10	2630	1715	19
		2750	1785	10
	0.03	2880	1855	5
		3000	1920	11
3180		2020	10	
Cr-MoSi ₂	5.5	2630	1715	>72
		2660	1735	>72
	0.10	2545	1670	5
		2565	1680	10
		2595	1700	15
	0.03	2830	1830	12
		2850	1840	22
		2950	1895	32

TABLE 6.- COMPARISON OF COATING LIFE IN DYNAMIC
AND STATIC TEST CONDITIONS

(a) TZM/Durak B system

Type of test	Air stagnation pressure, $p_{t,2}$, atm	Surface temperature		Coating life, min
		$^{\circ}\text{F}$	$^{\circ}\text{K}$	
Dynamic oxidation	0.03	2830	1830	32
		2890	1860	22
		2905	1870	28
		Average		27
Static oxidation (see ref. 9)	0.026	3100	1980	42
		3150	2005	24
		Average		33

(b) Cb-752/Cr-Ti-Si system

Type of test	Air stagnation pressure, $p_{t,2}$, atm	Surface temperature		Coating life, min	Type of failure
		$^{\circ}\text{F}$	$^{\circ}\text{K}$		
Dynamic oxidation	0.03	2880	1855	5	Incipient melting
		3000	1920	10	Melted
		3180	2020	11	Melted
Static oxidation (see ref. 9)	0.026	2900	1865	5	Melted
		2900	1865	5	Melted

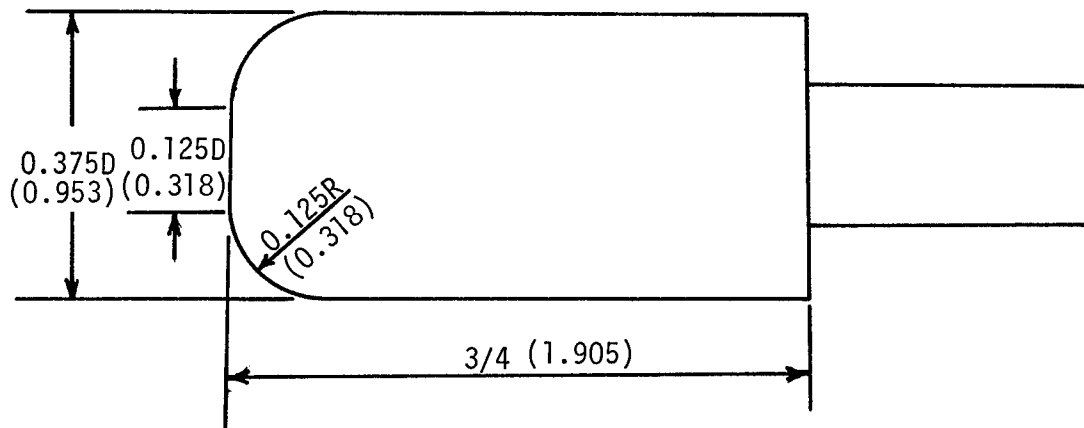


Figure 1.- Construction details of blunt-nose refractory-metal-alloy models. All dimensions are in inches (centimeters).

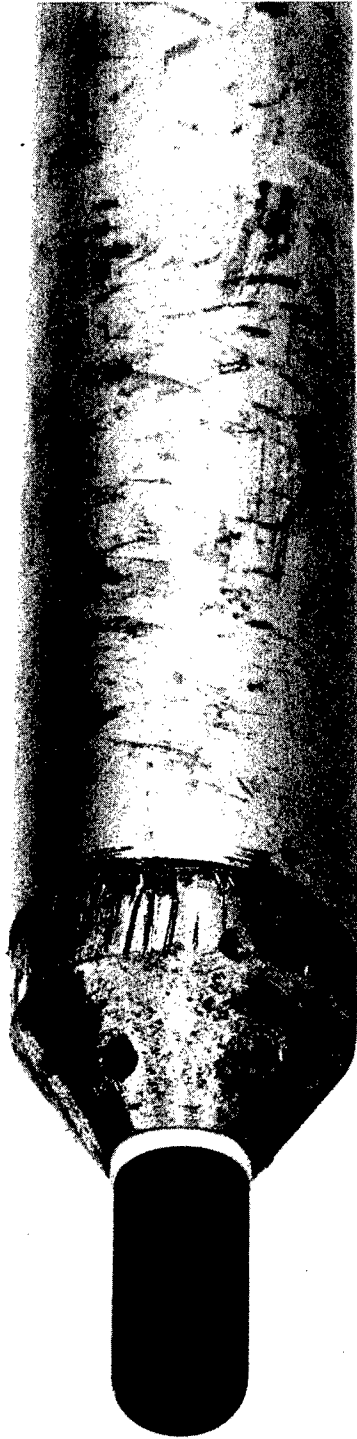


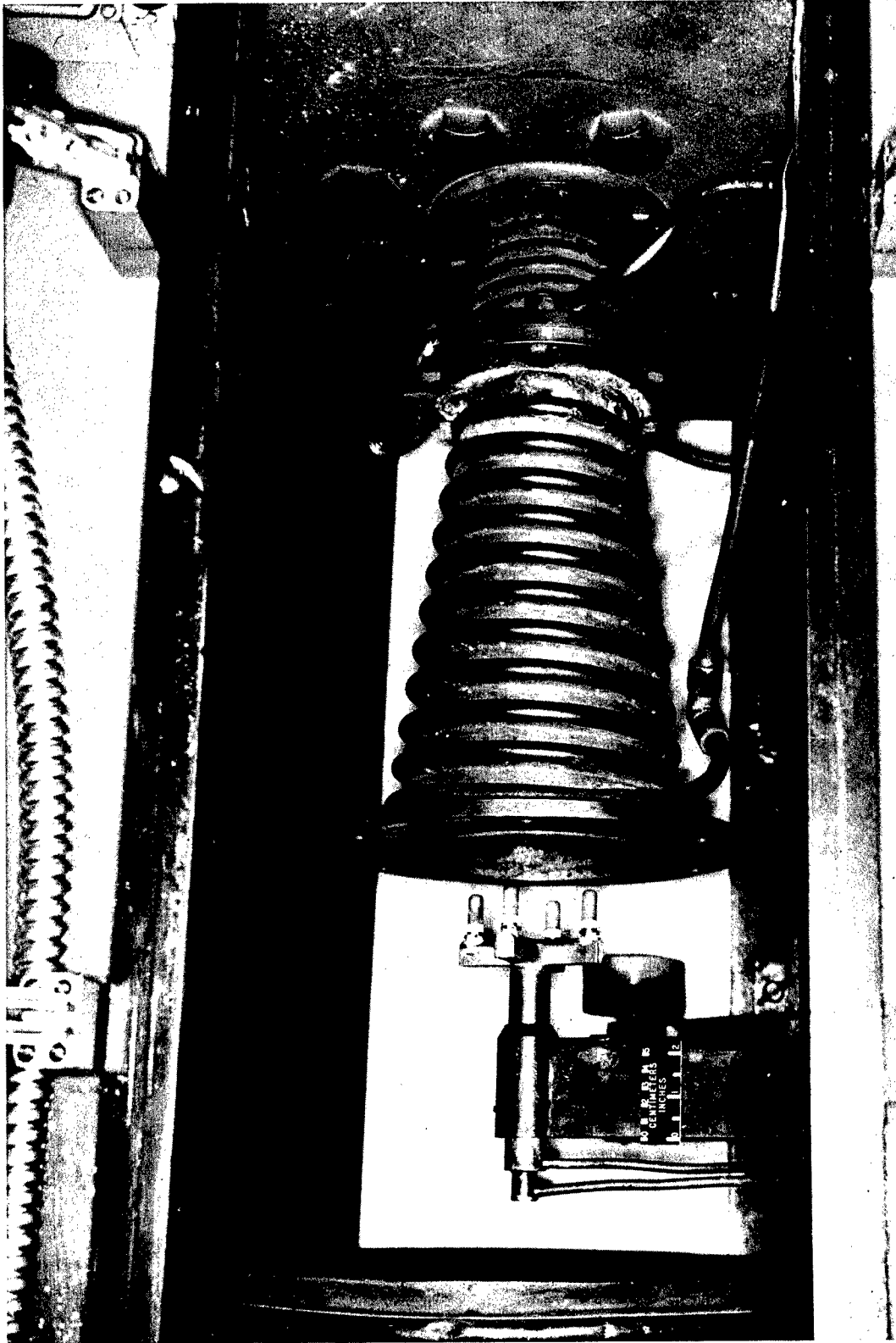
Figure 2.- Model holder used in Langley 11-inch ceramic-heated tunnel. Magnification, X1.87.

L-67-4312



Figure 3.- Coated model in test position in Langley 11-inch ceramic-heated tunnel.

L-67-3945



L-67-3947

Figure 4.- Four coated models in test position in Langley 20-inch hypersonic arc-heated tunnel.

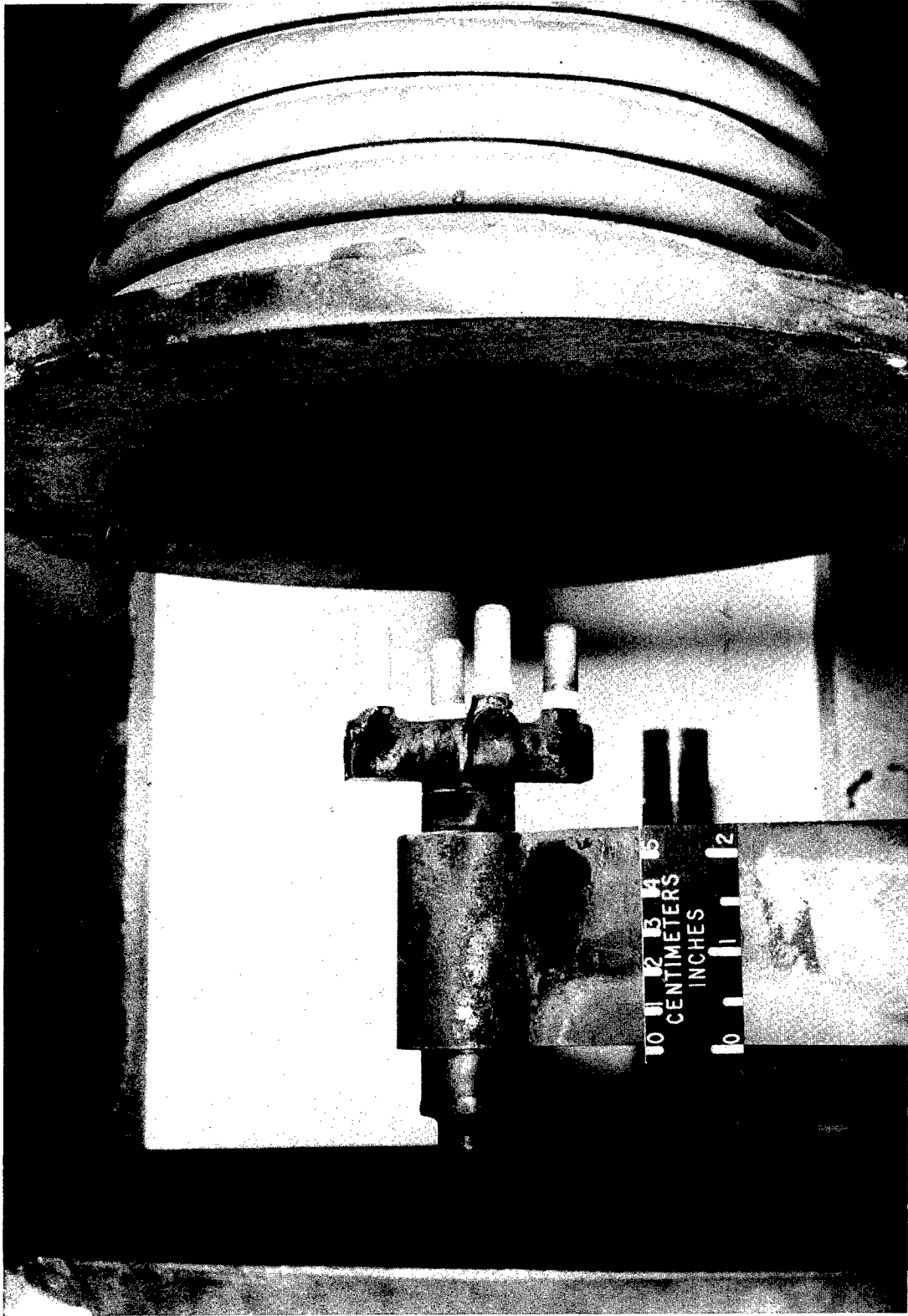


Figure 5.- Four coated models in test position in high-enthalpy arc tunnel at the Langley Research Center. L-67-3944

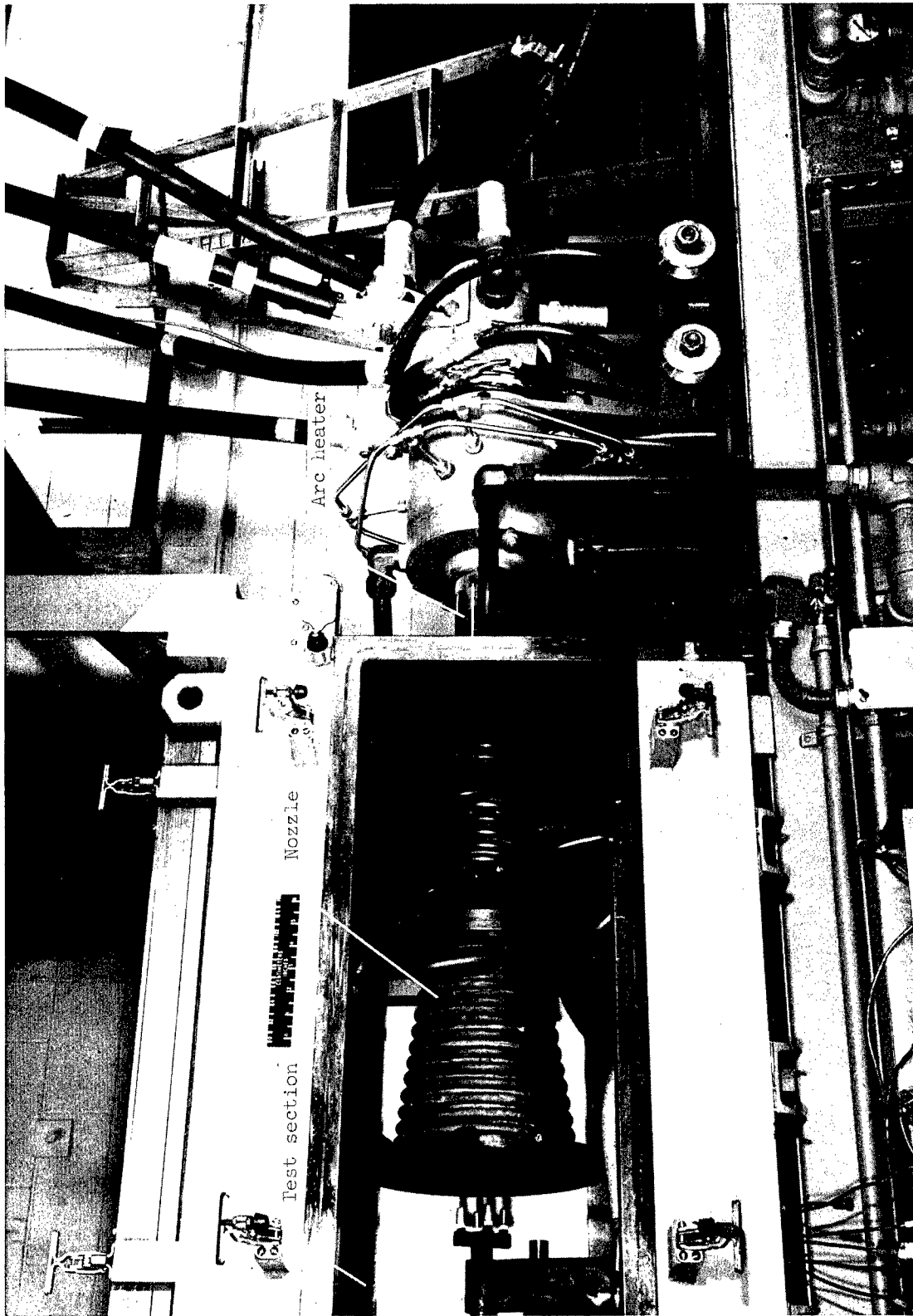
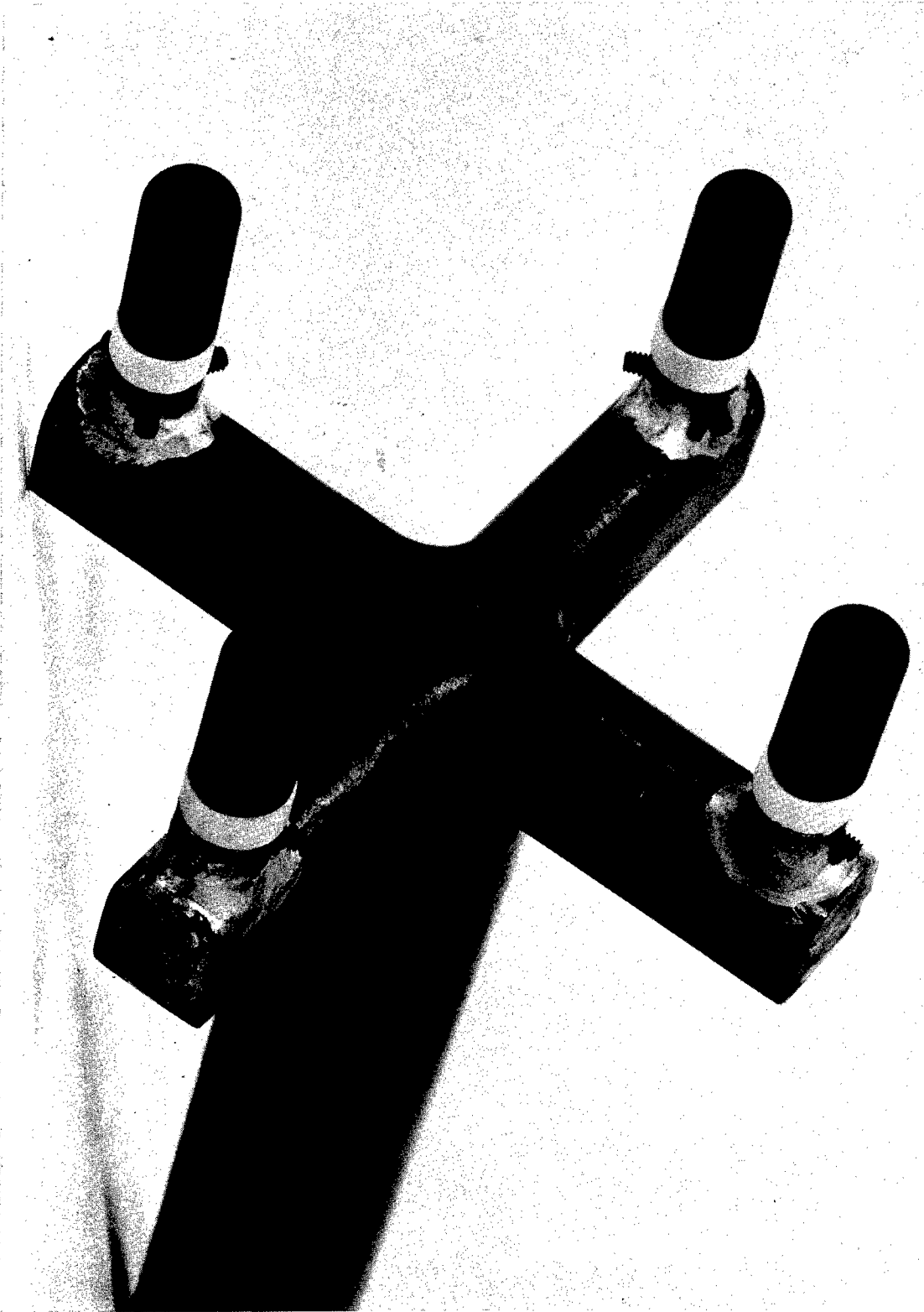


Figure 6.- High-enthalpy arc tunnel at the Langley Research Center.

L-67-3946.1



L-67-4311

Figure 7.- Four-model holder used in two arc-heated tunnels.

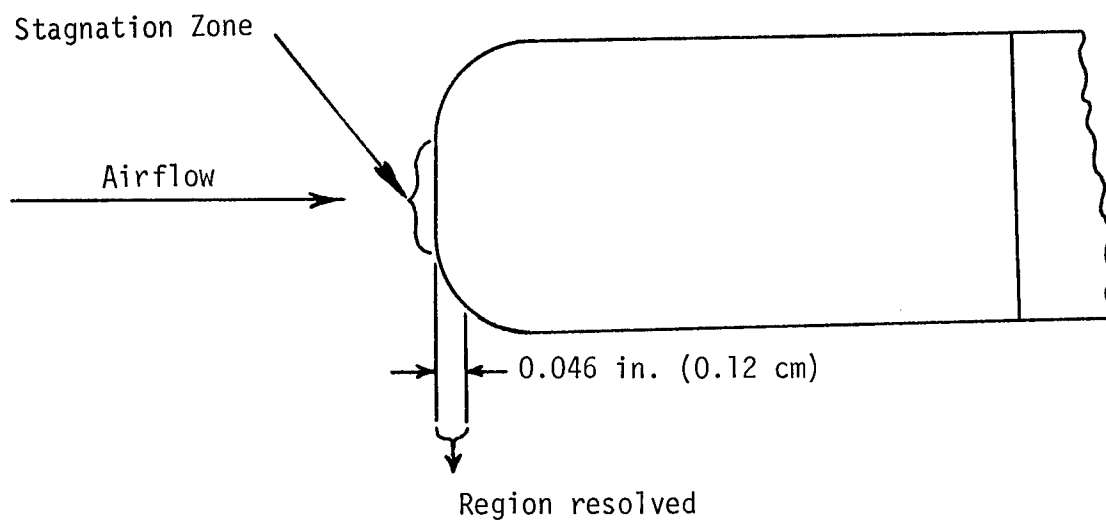


Figure 8.- Region on shoulder of model resolved by photographic-pyrometer readout system.

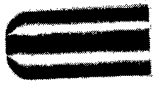


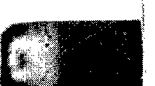








Metal alloy	Untested model	Air stagnation pressure, $p_{t,2}$, atm		
		5.5	0.10	0.03
Molybdenum alloy TZM		 Catastrophic	 Noncatastrophic	 Catastrophic
Columbium alloy Cb-752		 Catastrophic	 Catastrophic	 Catastrophic
Columbium alloy B-66		 Catastrophic	 Catastrophic	 Catastrophic

Figure 9.- Oxidation behavior of uncoated models in three supersonic-airstream environments.

L-68-8503

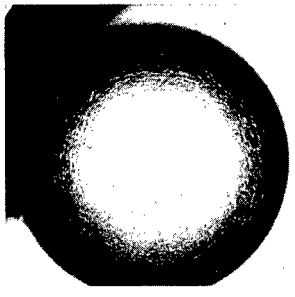
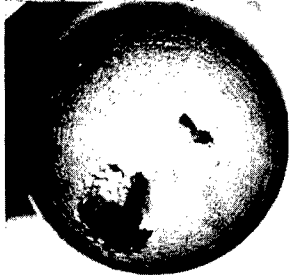
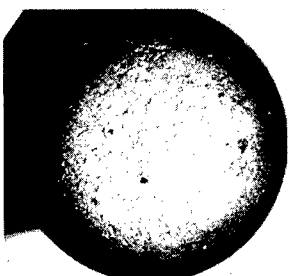
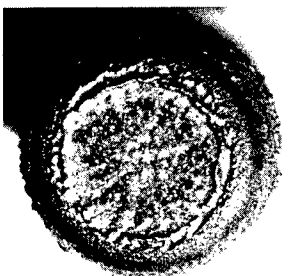
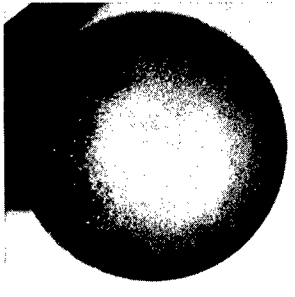

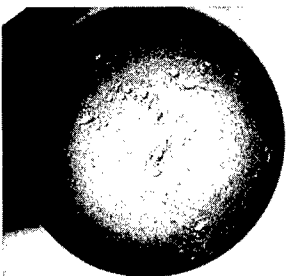
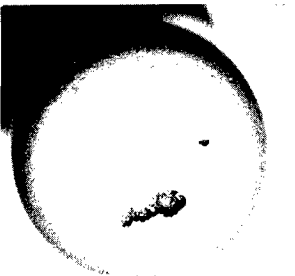
Coating	Untested model	Air stagnation pressure, $p_{t,2}$, atm		
		5.5	0.10	0.03
Durak B				
	Temp., °F (°K)	2680 (1745)	2725 (1770)	2890 (1860)
	Life, min	53	21	22
Vought IX				
	Temp., °F (°K)	2655 (1730)	2655 (1730)	2790 (1805)
	Life, min	16	11	62

Figure 10.- Response of coated TZM models to dynamic test environments. Temperature indicated is average surface temperature; life indicated is coating life. L-68-8504


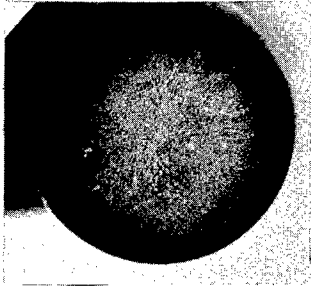
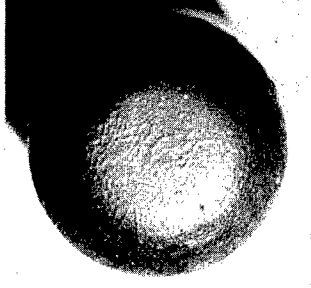
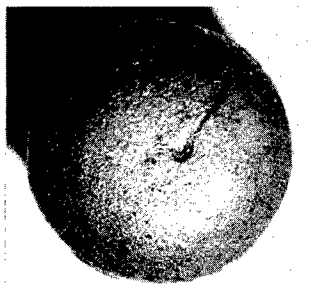
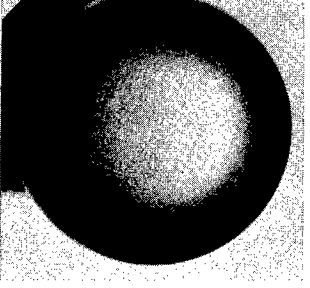

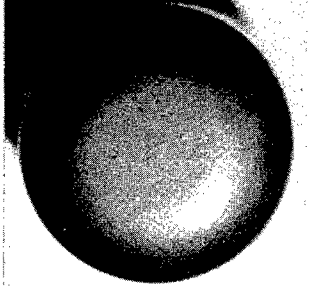
Coating	Untested model	Air stagnation pressure, $p_{t,2}$, atm		
		5.5	0.10	0.03
Cr-Ti-Si				
	Temp., °F (°K) Life, min	2735 (1775) >72	2630 (1715) 19	2880 (1855) 5
	Cr-MoSi ₂			
Temp., °F (°K) Life, min		2670 (1740) >72	2580 (1690) 5	2860 (1845) 12

Figure 11.- Response of coated Cb-752 models to dynamic test environments. Temperature indicated is average surface temperature; life indicated is coating life. L-68-8505

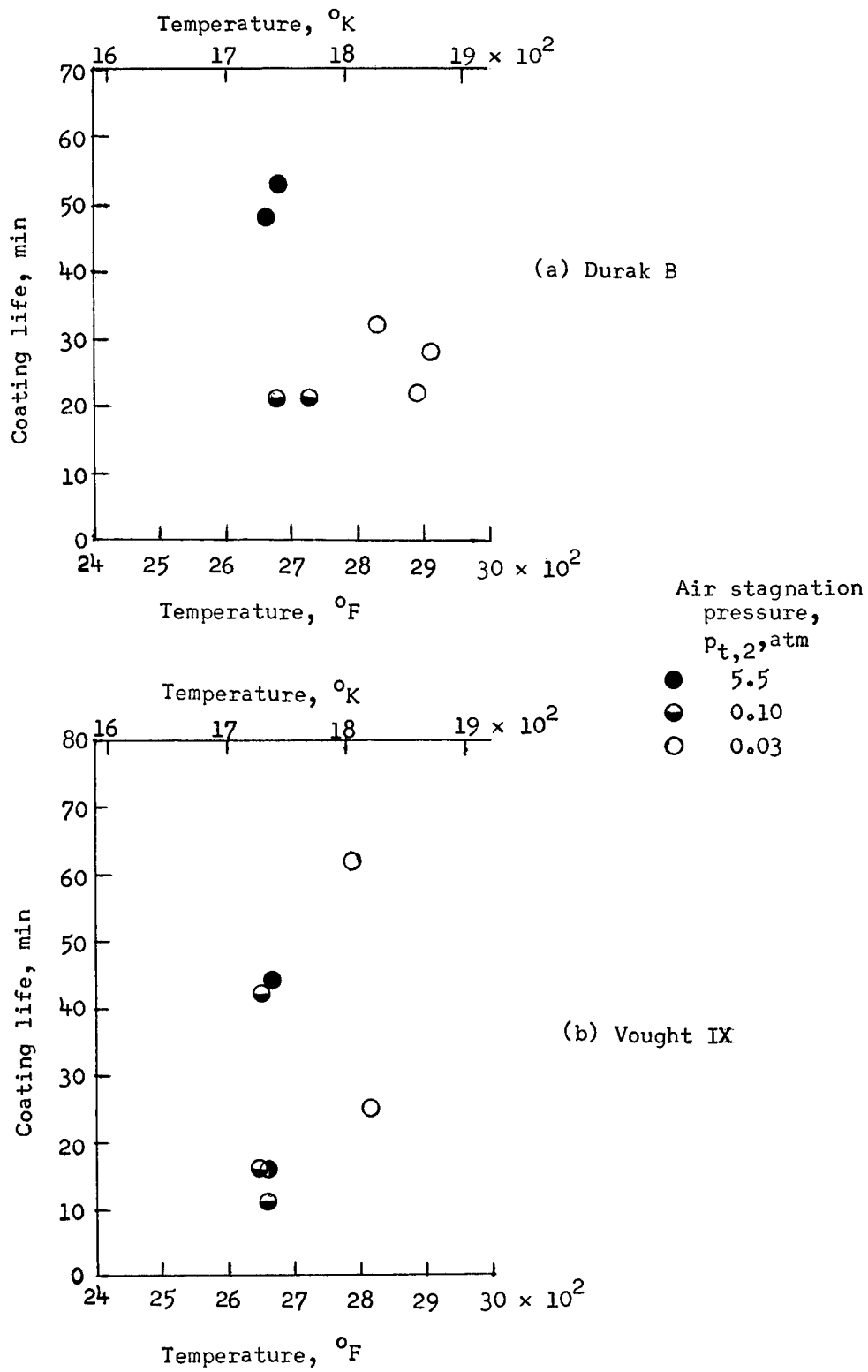


Figure 12.- Response of coated TZM models to dynamic test environments.

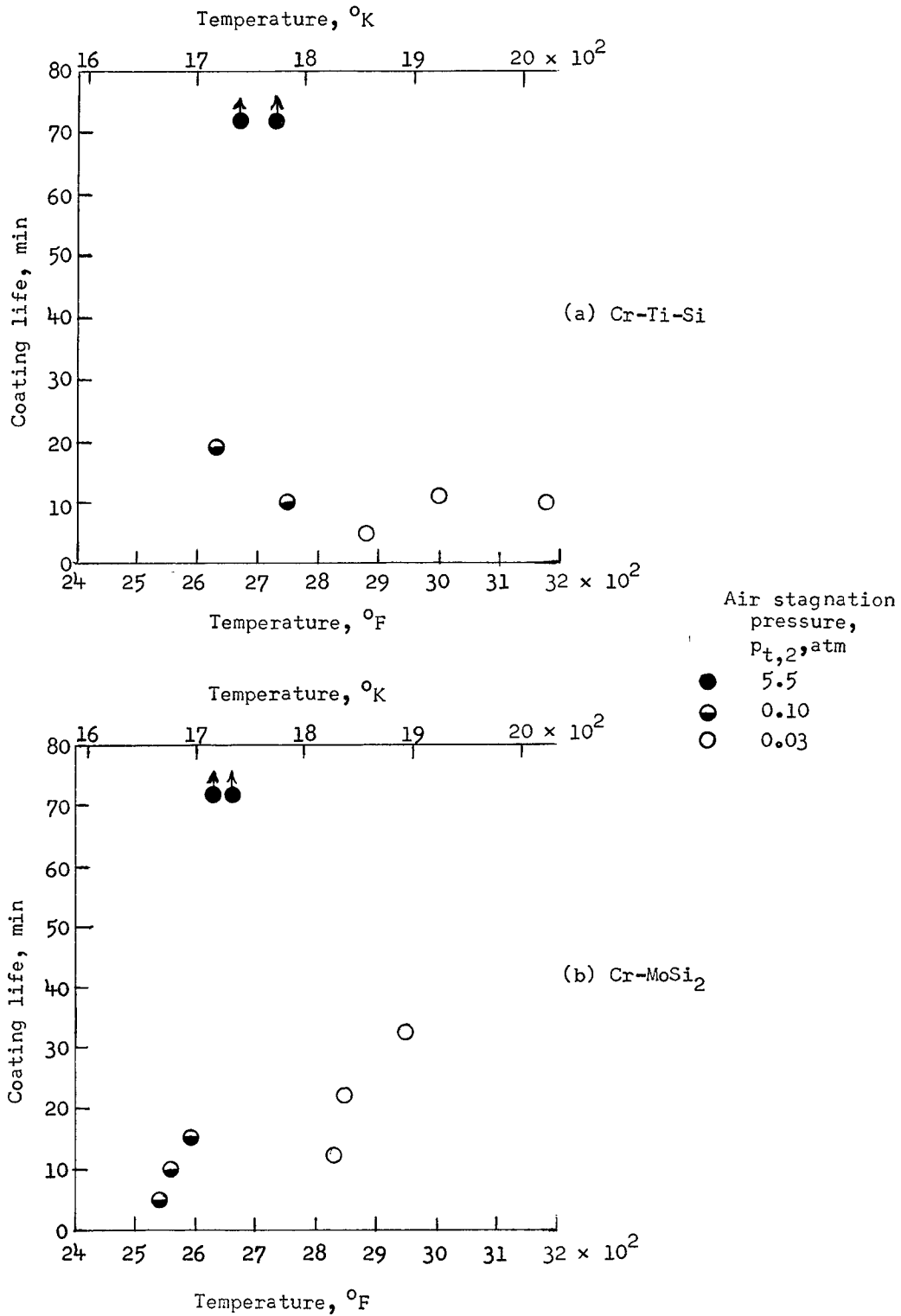


Figure 13.- Response of coated Cb-752 models to dynamic test environments.