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EIGHTH QUARTERLY REPORT
EVALUATION TESTING OF PROTECTIVE
COATINGS ON REFRACTORY METALS

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September 1969

CONTRACT NAS 7-460

Submitted to

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EIGHTH QUARTERLY REPORT
EVALUATION TESTING OF PROTECTIVE
COATINGS ON REFRACTORY METALS

CONTRACT NAS 7-460

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1. INTRODUCTION

This is the Eighth Quarterly Progress Report on Evaluation Testing of Protective Coatings on Refractory Metals under Contract NAS 7-460 for NASA/JPL, Pasadena, California. This report consists of a summary of the work accomplished during this quarter, work to be accomplished during the next reporting period and the current expenditures of dollars and manpower.

Briefly, the evaluation program consists of subjecting a series of coated radiation nozzles and chambers to engine firing to evaluate their protective capabilities in a propulsive environment. Each Task III test specimen is to be test fired 10 seconds then 1000 seconds or to failure at each of the nominal combustion temperatures of 4000°F and 4500°F. Selected Task IV chamber specimens are to be test fired for extended duration to failure at the nominal combustion temperature of 4500°F. The objectives of the engine evaluation are to:

- Determine the time and coating temperature at point of tangible coating degradation and ultimate failure.
- Determine failure mode or condition of coating system metallurgically at completion of the test sequence.
- Correlate the life of the coatings with processing methods and thermal environment.

2. SUMMARY OF EIGHTH QUARTER PROGRESS

During this quarter the following work was accomplished.

2.1 EVALUATION TESTS AND THERMAL ANALYSIS

All evaluation tests planned for this contract have been completed. A summary of the completed experimental program is given in Table I. All tests were conducted at ambient sea level conditions using N_2O_4/N_2H_4 propellants at the nominal mixture ratios (O/F) of 0.85 and 1.2 to obtain combustion temperatures of 4000°F and 4500°F, respectively. Target chamber pressure for all tests was 145 psia. The entered values of operating mixture ratio (O/F) and chamber pressure (Pc) in Table I are preliminary at this time and will be refined for final reporting.

The Phase I - Verification of Operating Environment Tests were completed mid-way into the quarter (see Twentieth Monthly Report). This test phase consisted of a series of heat sink engine, radiation nozzle, and ablative nozzle streak tests to define the thermal environment and ultimately the specimen wall temperature at the point of specimen failure. The heat sink engine and radiation nozzle were instrumented with thermocouple probes to measure wall temperatures and heat flux. Measured values of wall temperature and heat flux were then used to compute gas side film coefficients and gas recovery temperatures at the two nominal operating mixture ratio conditions. The derived values of the film coefficient and gas recovery temperature were in turn inputs into a thermal model of the coated test specimens to predict the two operating wall temperatures. Results of this thermal analysis are presently being finalized and shall be released in a forthcoming report. Also included in this most recent thermal analysis effort is a re-evaluation of the earlier thermal data obtained at the start of the contract.

During the Phase I radiation nozzle tests, a radiation pyrometer was evaluated as a means to measure specimen wall temperature. It was concluded that due to the many varied textures and surface finishes associated with the test specimens and consequently the inability to assign accurate values of emissivity, the radiation pyrometer could not be regarded as a prime measuring device for nozzle wall temperature. Pyrometer data was however taken during all specimen tests in the event realistic emissivities could later be assigned, at which point the pyrometer temperature data could be corrected for the emissivity error. The pyrometer also served as a useful monitoring tool to detect relative changes in temperature during a test firing.

Ablative streak nozzle tests were conducted during Phase I to define the combustion pattern and locate any nozzle hot spots which may be present at the two nominal operating mixture ratios of 0.85 and 1.20. Results of the two streak nozzle tests indicated a uniform erosion pattern with no outstanding hot spots evident at the operating mixture ratio of 0.85, and a pattern characterized by six heat zones nearly equally spaced around the throat diameter with one outstanding hot spot at 140° orientation at the operating mixture ratio of 1.20. Detail tests results and photographs of the streak nozzles were presented in the Twentieth Monthly Report.

A total of sixteen radiation cooled specimens were evaluated during this quarter in completing the Phase II Coating Evaluation Tests. Eight nozzles (S/N 003, 007, 013, 022, 023, 121, 124, and 125) and two chambers (S/N 53-3-3 and 53-8-1) were tested under Task III, and six chambers (S/N A-2, A-3, A-5, A-6, B-2, and B-5) were tested under Task IV. The Task III specimens were tested for 1000 seconds duration or until specimen failure at one or both operating combustion temperatures of 4000°F and 4500°F; whereas, the Task IV specimens were tested to failure at the combustion temperature of 4500°F. Prior to and after each combustion temperature test sequence, a visual examination of the specimen was made and logged and photographs of the specimen inlet and exit were taken. Post-test condition of the sixteen specimens tested during this quarter is shown in the photographs in Figures 1 through 16. The angular orientation noted in the figures is referenced to the specimen index assigned during the pre-test metallurgical examination.

The preliminary specimen test results obtained during this quarter are presented in Table II. Pertinent failure remarks are included under the comments in Table II. As was the case with all previous specimens tested, coating degradation or specimen failure usually occurred at an injector hot spot or at a defect in the original coating. Those specimens and/or operating temperatures not entered in Table II were evaluated early in the program and are discussed in detail in the Sixth Quarterly Report and are briefly summarized in Table I.

It will be noted in Table II that the number of engine starts has been given in achieving the stated test duration. Engine starts exceeding one reflect the number of restarts required as a result of a temperature flip phenomenon (see Twentieth Monthly Report). The only exception to this was in the firing of S/N A-2 in which multiple engine starts was required for propellant retanking. This temperature flip phenomenon came to light early in the Task III nozzle specimen test phase. It was noted that during some tests, a temperature flip to a higher wall temperature level would occur in the engine. This temperature flip is a step function and is characterized by increased heat loading to the injector and chamber water coolant flow, increased brightness of the test specimen, and a change in the appearance of the exhaust plume from a fuel rich yellow/orange well defined Mach diamond

pattern to a clear transparent high performance appearing flame. Combustion performance parameters did not change significantly when the temperature flip occurred; propellant flows remained constant and chamber pressure increased only about one percent. The temperature flip was uncontrollable and unpredictable as to when it would occur in a firing; however, it was never noted during the start-up of the engine. Since the temperature flip is not believed to be the normal operational mode of the engine and it does not reflect the conditions under which all previous heat transfer data was acquired, all specimen tests were terminated at the first signs of a temperature flip. If a test had to be terminated short of the desired test duration, the engine was permitted to cool to ambient and then refired to obtain the remaining required test duration. This procedure was repeated as often as necessary. The exception to this procedure was in the testing of S/N 023 in which the temperature flip was not detected during the firing but was detected upon examination of the test data.

Inspection of Table II reveals that the temperature flip occurs at both operating temperatures or mixture ratios and for practically every nozzle specimen tested. The only circumstances under which the temperature flip did not occur was in the testing of the chamber specimens. It has been postulated that the temperature flip is a result of disturbing the normally cool boundary layer on the engine wall and thereby increasing the heat flux to the walls; however, the mechanism by which this is apparently accomplished only in the case of nozzle specimens is not yet known. The test history would suggest the temperature flip is in some manner related to the geometry differences between the nozzle and chamber configurations.

Referring back to Tables I and II and Figures 1 through 16, it is noted that of the original sixteen nozzles and two chamber specimens evaluated under Task III, eight nozzles and one chamber survived the 1000 seconds duration test at a combustion temperature of 4000°F. The surviving eight nozzles included all three Ha-20Ta clad specimens (S/N 003, 006, and 013), one Ir coated specimen (S/N 007), two of the molybdenum disilicide coated specimens (S/N 120 and 121), and the two Ha-20Ta slurry coated specimens (S/N 124 and 125). The one surviving chamber was a Hf-20Ta clad specimen (S/N 53-8-1). Of these surviving nine specimens, only three survived the additional 1000 seconds duration firing at the combustion

temperature of 4500°F. These consisted of two Hf-20Ta clad nozzles (S/N 003 and 013) and one molybdenum disilicide (S/N 121) coated nozzle. The survival of the molybdenum disilicide coated nozzle at the combustion temperature of 4500°F was somewhat of a surprise due to the previous failures of identical nozzles S/N 122 and 120 at 4000°F and 4500°F combustion temperatures, respectively. Perhaps the post-test metallurgical analysis will shed some light on this inconsistent behavior of the molybdenum disilicide specimens. For the present it is concluded the survival of S/N 121 at a combustion temperature of 4500°F is indicative of a specimen wall temperature which is borderline in being equal to 3200°F; the approximate melting point of molybdenum disilicide.

During the life firing tests of six selected chambers from Task IV, one of the Ha-20Ta clad chambers had notable success. Specimen S/N A-2 accumulated a total of 3937 seconds firing time at the maximum chamber temperature of 4500°F before failure. Included in this time was four engine start-ups from ambient temperature; the multiple engine starts being required for propellant re-tanking. The significance of this firing together with the success of the two Hf-20Ta clad nozzles from Task III is that it has demonstrated the practicality of fabricating a reliable Ha-20Ta clad specimen capable of providing long specimen life at elevated temperatures. The approximate specimen wall temperature during these firings was 3200°F. Although this temperature level may not be regarded as a severe test of the coating system when compared to its theoretical temperature limitation, it is reasonable to believe the Ha-20Ta clad system would also demonstrate long life at even higher wall temperatures. The premise for this conclusion is that prior experience during this program has indicated the fabrication techniques and/or controls used to achieve a uniform, defect-free coating is more crucial to the coatings success than the operating temperature.

Trailing the success of the S/N A-2 Hf-20Ta clad radiation cooled chamber was the Hf-Ta slurry coated chamber (S/N B-5) with a demonstrated life of 634 seconds; the second Hf-20Ta clad chamber (S/N A-3) with a demonstrated life of 587 seconds; and the Ir-Re (S/N B-2), Hf-Ta clad (S/N A-5), and Hf-Ta-Mo clad (S/N A-6) chambers all with a demonstrated life of less than 70 seconds. The rapid failure of the Ir-Re coated chamber (S/N B-2) is in part due to the presence of gas leaks through the chamber

barrel section. This leakage was detected during the pressure check made prior to the firing. Similar but more extensive leakage was detected on the unfired identical specimen (S/N B-4). The leakage in these two chambers may have been through cracks in the substrate which were not detectable during the pre-test metallurgical examination. The differences noted in the firing durations for the various Hf-Ta clad specimens again points out the importance of having proper fabrication techniques and/or controls in providing a reproducible, uniform, and defect free coating; and consequently, long specimen life.

During the Task IV chamber tests an attempt was made to test one of the Ir-Re coated tungsten chambers (S/N C-1 and C-11); however, both chambers cracked at the junction of the flange to the chamber barrel as they were being installed. Cracking occurred at a bolt torque value of approximately 25 in.-lb. Photographs of the cracked areas are shown in Figures 17 and 18.

As a point of information, the S/N 005 injector has survived the test program in reasonably good shape. Some slight surface erosion has occurred in the center of the injector face. The erosion is centered between the four central propellant doublets; the region of minimum injector cooling. The erosion is a strip about 0.5 inch long by 0.1 inch wide. Depth of the eroded area is perceptible but is only a few thousandths of an inch. There appears to have been very little change in this eroded area since its first appearance early in the test program. Numerous repairs have been made to the injector throughout the test program; primarily at the braze joints of the oxidizer distribution tubes to the inlet supply fitting and at the braze joints of the oxidizer orifice tubes to the injector top plate. The injector presently has oxidizer leakage at four of the aforementioned braze joints but these leaks could be sealed with LOCTITE for future injector usage.

2.2 METALLURGICAL ANALYSIS

Pre-test metallurgical examinations of all tests specimens were completed during this quarter. The pre-test examinations have consisted of specimen identification, visual and photographic examinations, and a record of all observations. Suspect areas in the coatings were noted by detailed sketches and photographs which are referenced to an index line on each specimen.

Post-test metallurgical examinations of the failed specimens have begun. The post-test analysis will duplicate the original visual examination and also include a metallographic examination on selected specimens of a full insert longitudinal cross section and other insert areas as required. Particular attention shall be given to the Ha-Ta class of coatings since they have demonstrated superior performance throughout the test program.

3. WORK TO BE ACCOMPLISHED DURING NEXT REPORTING PERIOD

During the next monthly reporting period, the following work is planned to be accomplished:

- Start finalizing and documenting the test data obtained during the test program.
- Proceed with the post-test metallurgical examinations.
- Finalize the heat transfer analysis predicting the operating specimen wall temperatures.

4. CONTRACT FINANCIAL STATUS

The contract expenditure as of 31 August 1969 was \$281,990. The projected expenditure to complete the contract and the actual dollar and man-power expenditures to date are summarized in Figure 19. Also included in Figure 19 are the major milestones of the program. The reason for the actual dollars falling below the estimate is the delays in getting the test program started.

TABLE I
EVALUATION TESTING OF PROTECTIVE COATINGS ON REFRACTORY METALS
EXPERIMENTAL SUMMARY - TASK III AND IV

| TASK | MATERIAL TYPE OR PURPOSE OF TESTS | TEST DESCRIPTION | | | | SPECIMEN FAILURE COMMENTS |
|--|---|---|-----|---|-----|---|
| | | Target Combustion Temperature of 4000°F | | Target Combustion Temperature of 4500°F | | |
| | | O/F | Pc | O/F | Pc | |
| Phase I - Verification of Operating Environment | | | | | | |
| A. Heat Sink Engine Tests (5 tests) | 1. Calibrate facility and engine at operating mixture ratios | .85 | 149 | 1.20 | 150 | DNA |
| | 2. Obtain temperature data for input in thermal analysis program to determine recovery temperatures | | | | | |
| B. Streak Tests S/N 005 | Determine injector combustion pattern at operating mixture ratios | .86 | -- | DNA | DNA | DNA |
| S/N 006 | | DNA | DNA | 1.24 | -- | DNA |
| C. Radiation Nozzle Tests (3 tests) | 1. Obtain temperature data for input in thermal analysis program to determine recovery temperatures | .86 | 143 | 1.22 | 142 | DNA |
| | 2. Check out optical pyrometer method of measuring wall temperature | | | | | |
| Phase II - Coating Evaluation Tests - Radiation Cooled | | | | | | |
| A. Task III Nozzle Tests | 1. Determine time and temperature at the point of coating failure | | | | | |
| | 2. Determine failure mode or condition of coating at completion of tests | | | | | |
| S/N003 | HF-20Ta Clad, Ta Barrier | .854 | 145 | 1.20 | 144 | Nozzle in servicable condition after 1000 sec. duration at 4500°F. Gross clad separation at exit. Craters are starting to develop on convergent section inside & outside. |
| S/N 006 | HF-20Ta Clad, Ta Barrier | 1.02 | 137 | 1.26 | 149 | Clad material separated from substrate at nozzle exit. The test was aborted due to burnout of the injector seal ring at 78 sec duration at 4500°F. The test specimen is heavily contaminated with iron oxides and will not be further tested. |

Notes: (1) DNA - Does not apply
(2) Entered values of mixture ratio and chamber pressure are preliminary test conditions.

TABLE I
EVALUATION TESTING OF PROTECTIVE COATINGS ON REFRACTORY METALS
EXPERIMENTAL SUMMARY - TASK III AND IV (CONTINUED)

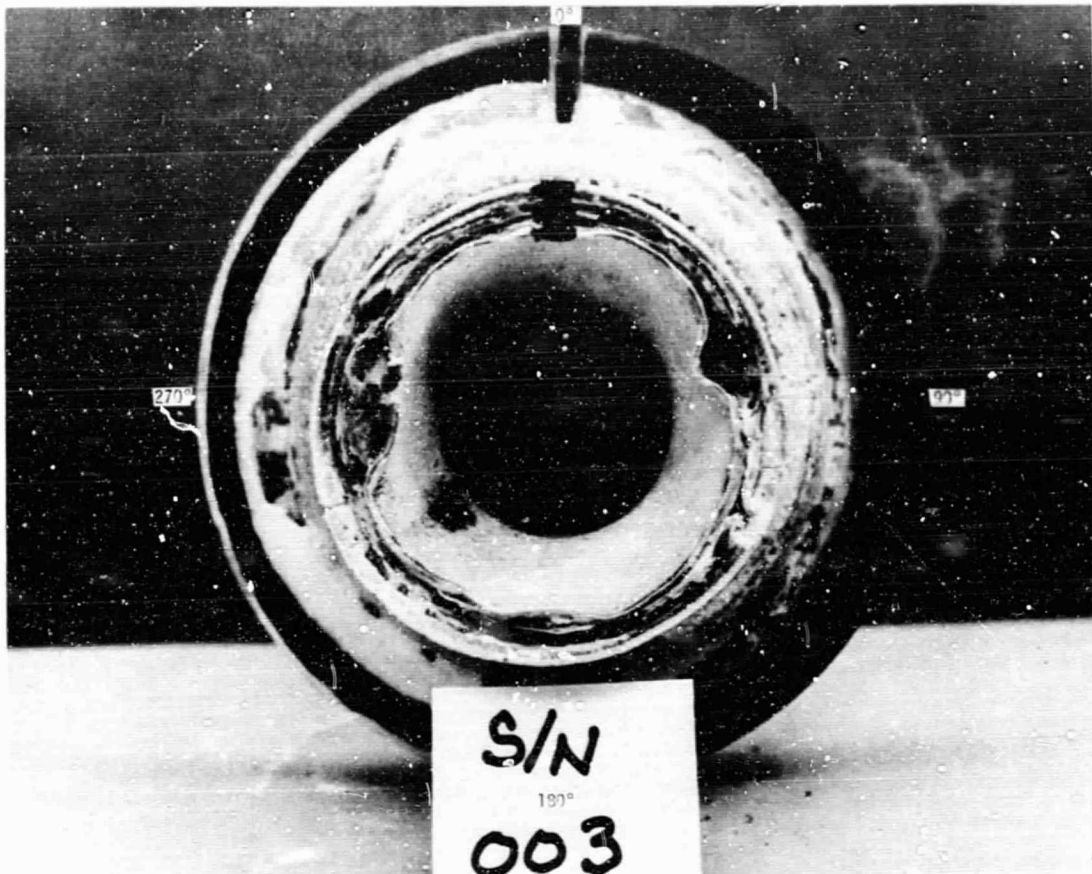
| TASK | MATERIAL TYPE OR PURPOSE OF TESTS | Target Combustion Temperature of 4000°F | | Target Combustion temperature of 4500°F | | SPECIMEN FAILURE COMMENTS |
|---|--------------------------------------|--|-----|--|-----|--|
| | | O/F | Pc | O/F | Pc | |
| A. Task III Nozzle Tests (continued) | | | | | | |
| S/N 013 | Hf-20Ta-clad, Cb barrier | 1.55 | 135 | 1.19 | 144 | Nozzle in good condition after 1000 sec. duration at 4500°F. Some clad separation at exit but otherwise cladding is intact. |
| S/N 007 | Ir | .886 | 144 | 1.20 | 145 | Nozzle burnout at throat at 120° orientation after 63 sec. duration at 4500°F. Extensive substrate oxidation on inside and outside. Much of exterior coating has spalled off and remaining coating is badly cracked and blistered. |
| S/N 012 | Ir | .862 | 137 | DNA | DNA | Nozzle burnout occurred at 180° orientation at 960 sec. duration at 4000°F. |
| S/N 014 | Ir/Re | .867 | 151 | DNA | DNA | Nozzle burnout occurred at 180° orientation at 557 sec. duration at 4000°F. |
| S/N 022 | Ir/Re | .85 | 144 | DNA | DNA | Extensive spalling of coating on exterior and subsequent oxidation of substrate after 480 sec. duration at 4000°F. Internal spalling of coating at throat. |
| S/N 023 | Ir/Re | .89 | 151 | DNA | DNA | Nozzle burnout at throat at 200° orientation after 670 sec. duration at 4000°F. Gross coating spalling and oxidation of substrate on exterior. |
| S/N 015 | Ir/Re | .879 | 143 | DNA | DNA | Nozzle shattered after 15 sec. duration at 4000°F. Primary failure occurred at an existing crack in nozzle throat. |
| S/N 020 | Hf-20Ta, Nitride Composite | .917 | 157 | DNA | DNA | Spalling erosion failure after 36 sec. duration at 4000°F. Primarily at 280° orientation. Realized a chamber pressure decay of 15 percent. |
| S/N 021 | Hf-20Ta, Nitride Composite | .881 | 159 | DNA | DNA | Nozzle burnout occurred at 180° orientation at 563 sec. duration at 4000°F. |
| S/N 120 | Molybdenum Disilicide | .876 | 143 | 1.18 | 133 | Coating melted followed by burnout of the nozzle throat at 180° orientation at 542 sec. duration at 4500°F. |
| S/N 121 | Molybdenum Disilicide | .85 | 143 | 1.20 | 145 | Nozzle in excellent condition after 1000 sec. duration at 4500°F. A nick in the coating at 225° orientation on the exit rim has been enlarged due to substrate oxidation. |
| S/N 122 | Molybdenum Disilicide | .867 | 141 | DNA | DNA | Burnout at 270° orientation after 592 sec. duration at 4000°F. Coating apparently spalled off exposing the substrate which subsequently oxidized rapidly. |

TABLE I
EVALUATION TESTING OF PROTECTIVE COATINGS ON REFRACTORY METALS
EXPERIMENTAL SUMMARY - TASK III AND IV (CONTINUED)

| TASK | MATERIAL TYPE OR PURPOSE OF TESTS | TEST DESCRIPTION | | | | SPECIMEN FAILURE COMMENTS |
|-------------------------------------|--|---|-----|---|-----|---|
| | | Target Combustion Temperature of 4000°F | | Target Combustion Temperature of 4500°F | | |
| | | O/F | Pc | O/F | Pc | |
| A. Task III Nozzle Test (continued) | | | | | | |
| S/N 124 | Hf-20Ta-0.25 Si | .86 | 144 | 1.20 | 145 | Substrate oxidation initiated at 320° orientation at throat after 216 sec. duration at 4500°F. Flow of yellowish material from the oxidized area. Some thin surface spalling and cracks on remaining coating. |
| S/N 125 | Hf-20Ta-0.25 Si | .85 | 145 | 1.21 | 149 | Substrate oxidation initiated at 105° orientation at throat after 845 sec. duration at 4500°F. Flow of yellowish material from the oxidized area. Remaining coating has some surface spalling and cracks. |
| B. Task III Chamber Tests | 1. Determine time and temperature at the point of coating failure. 2. Determine failure mode or condition of coating at completion of tests. | | | | | |
| S/N 53-3-3 | Hf-20Ta Clad | .86 | 147 | DNA | DNA | Chamber burnout in barrel section at 245° orientation after 478 sec. duration at 4000°F. Heavily cratered and oxidized on interior for first 1.5 inch into the chamber barrel. |
| S/N 53-8-1 | Hf-20Ta Clad | .85 | 147 | 1.21 | 145 | Chamber burnout in barrel sections at 30° orientation after 88 sec. duration at 4500°F. Cratering on interior for first 1.5 inch into chamber barrel. Extensive spalling of exterior coating during cooldown after the test. |
| C. Task IV - Chamber Tests | 1. Extended duration firings at highest temperature indicated in Task III to determine life capability. 2. Determine time at coating failure and condition at completion of test. | | | | | |
| S/N A-2 | Hf-Ta-Clad | DNA | DNA | 1.19 | 144 | Burn through at throat and tearing to the nozzle exit at 350° orientation after 3937 sec duration at 4500°F. Extensive clad separation and loss of substrate at exit. Crater formations on interior and exterior. Internal chamber barrel in reasonably good condition. |

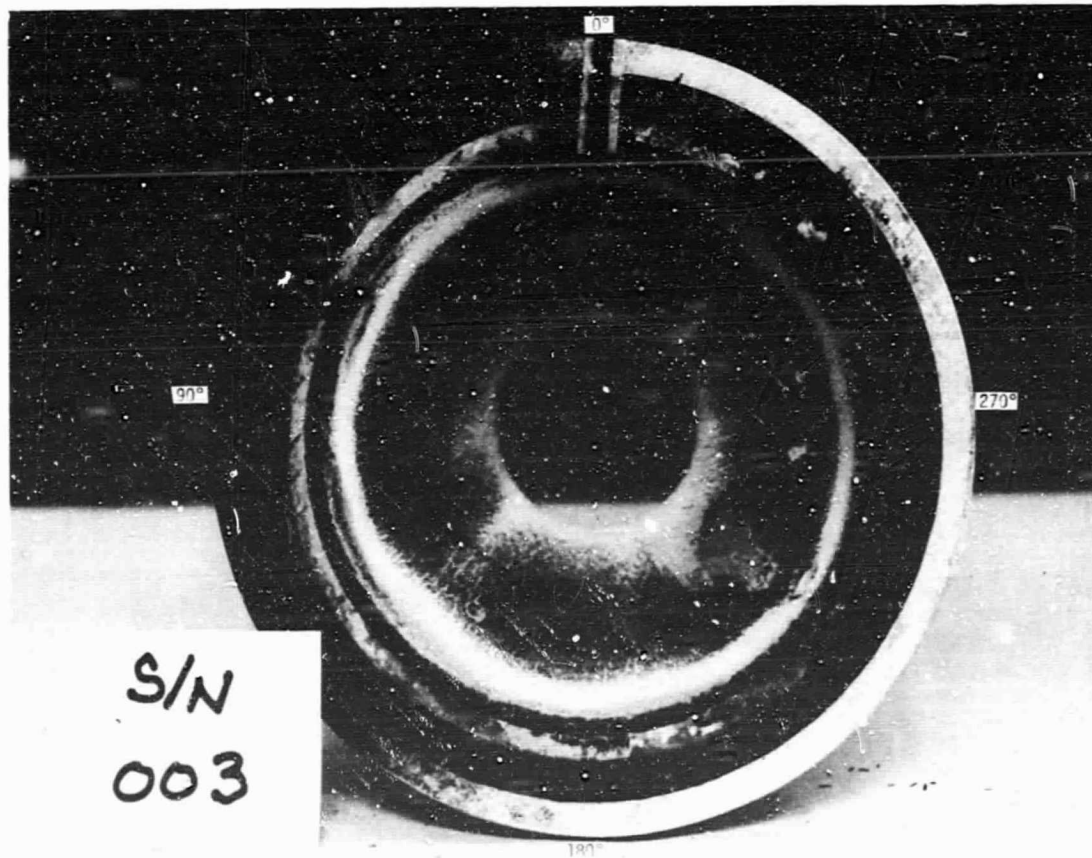
TABLE I
EVALUATION TESTING OF PROTECTIVE COATINGS ON REFRACTORY METALS
EXPERIMENTAL SUMMARY - TASK III AND IV (CONTINUED)

| TASK | MATERIAL TYPE OR PURPOSE OF TESTS | TEST DESCRIPTION | | | | SPECIMEN FAILURE COMMENTS |
|----------|-----------------------------------|---|-----|---|-----|--|
| | | Target Combustion Temperature of 4000°F | | Target Combustion Temperature of 4500°F | | |
| | | Q/F | Pc | Q/F | Pc | |
| S/N A-3 | Hf-Ta-Clad | DNA | DNA | 1.19 | 148 | Burn through at throat at 300° orientation after 587 sec. duration at 4500°F. Clad separation at exit. Some crater formation developing on outside and inside at throat. Internal chamber barrel in good condition. |
| S/N A-5 | Hf-Ta-W Clad | DNA | DNA | 1.20 | 145 | Chamber burnout in barrel at 280° orientation after 50sec. duration at 4500°F. Extensive cratering exposing oxidized substrate on interior of chamber barrel. Considerable flow of yellow/brown material from oxidized regions. Nozzle section and exterior in good condition. |
| S/N A-6 | Hf-Ta-Mo Clad | DNA | DNA | 1.20 | 145 | Chamber burnout in barrel at 230° orientation after 63 sec duration at 4500°F. Extensive substrate oxidation on interior of chamber barrel. Considerable flow of grey material from oxidized regions. Nozzle section and exterior in good condition. |
| S/N B-5 | Hf-Ta-Slurry | DNA | DNA | 1.21 | 150 | Substrate oxidation in throat region at 220° orientation after 634 sec. duration at 4500°F. Large blister formation downstream of throat on outside at 230°. Remaining coating in good condition. |
| S/N B-2 | Ir-Re-90Ta-10W | DNA | DNA | 1.20 | 145 | Burn through at throat at 220° orientation after 57 sec duration at 4500°F. Many deep longitudinal cracks in chamber barrel. Some cracks completely through the specimen. Prior to the test the specimen had gas leakage through the chamber walls. Heavy blister formations on exterior barrel section. |
| S/N B-4 | Ir-Re-90Ta-10W | DNA | DNA | DNA | DNA | No test. Specimen leaked excessively through chamber barrel during pressure check. |
| S/N C-1 | Re-Ir-W | DNA | DNA | DNA | DNA | Specimen could not be tested due to cracks developing at the flange during installation. |
| S/N C-11 | Re-Ir-W | DNA | DNA | DNA | DNA | Specimen could not be tested due to cracks developing at the flange during installation. |



Exit View

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Inlet View

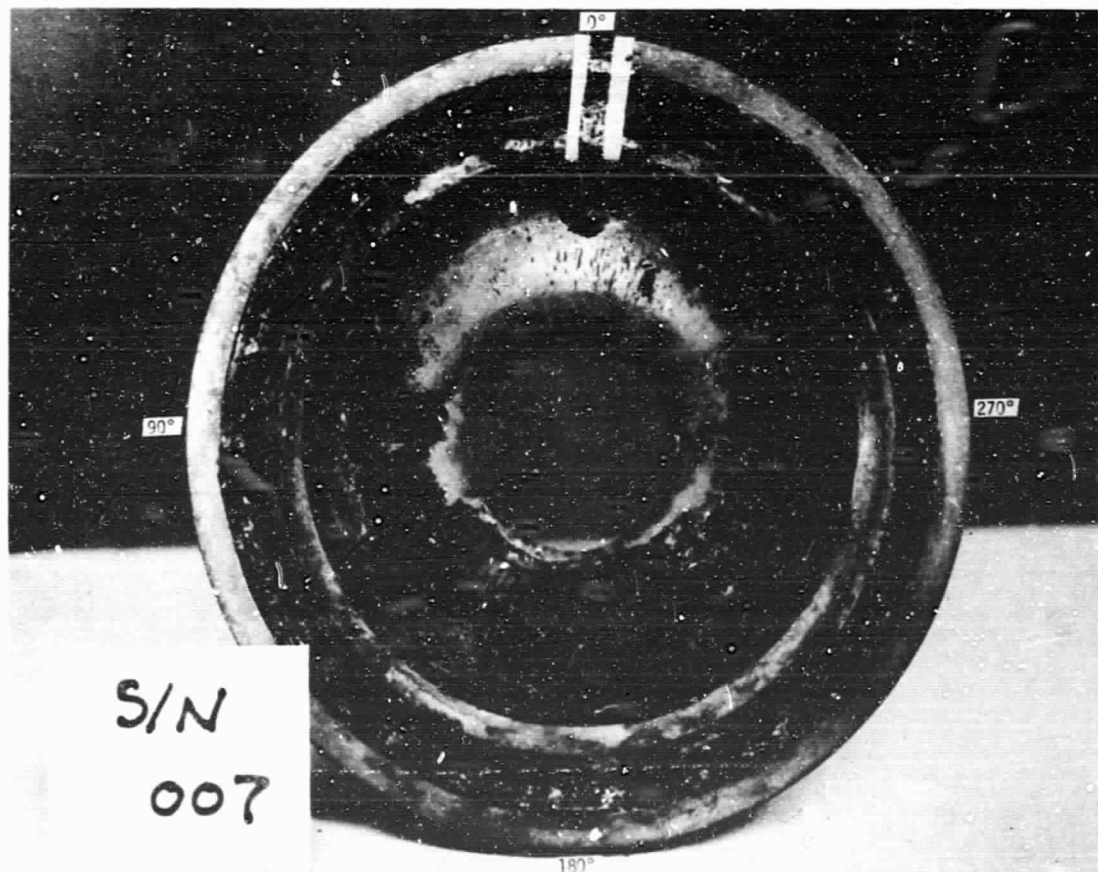
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Figure 1. Post-Test Condition of S/N 003 - 1000 sec at 4500°F



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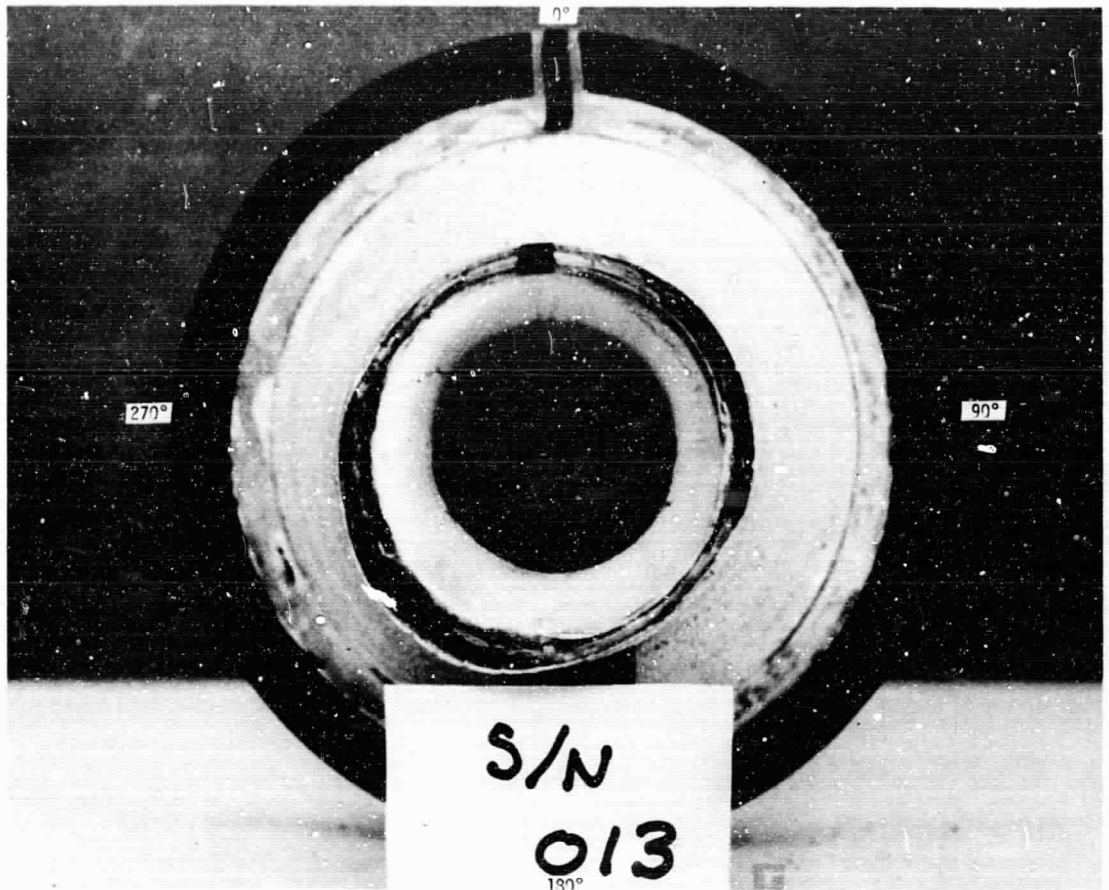
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Inlet View

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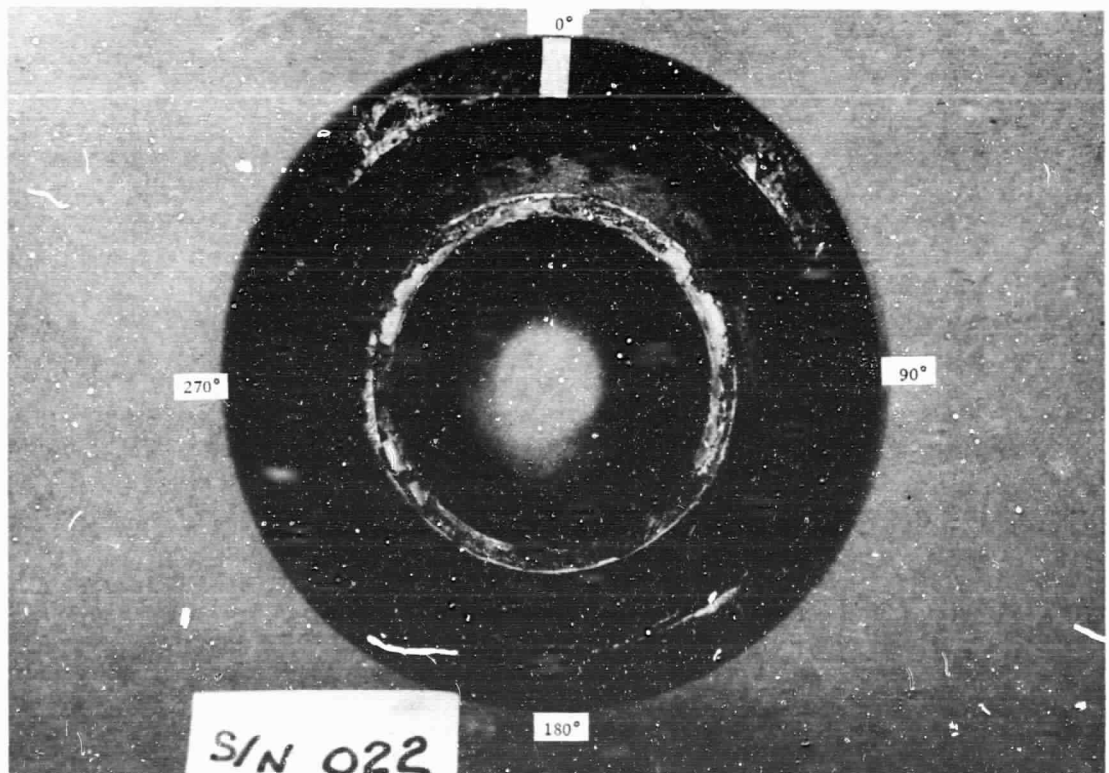
Figure 2. Post-Test Condition of S/N 007 - 63 sec at 4500°F



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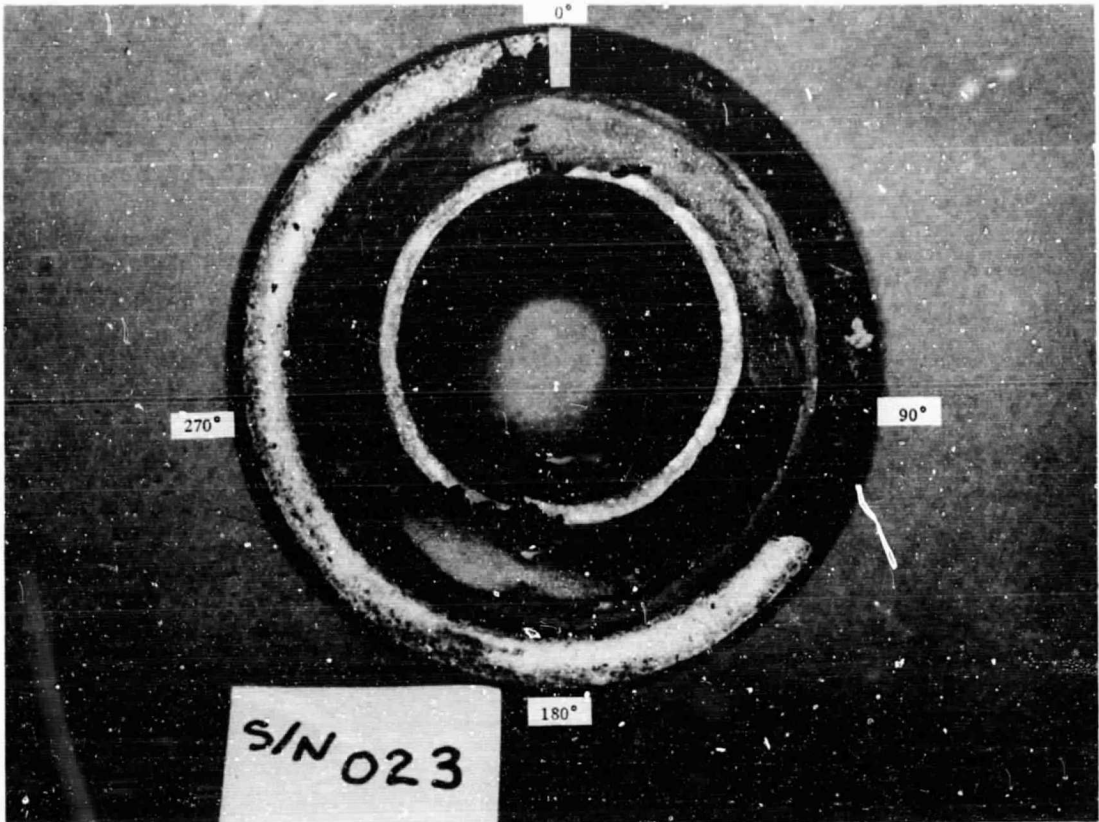
Figure 3. Post-Test Condition of S/N 013 - 1000 sec at 4500°F



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Figure 4. Post-Test Condition of S/N 022 - 480 sec at 4000°F



Exit View

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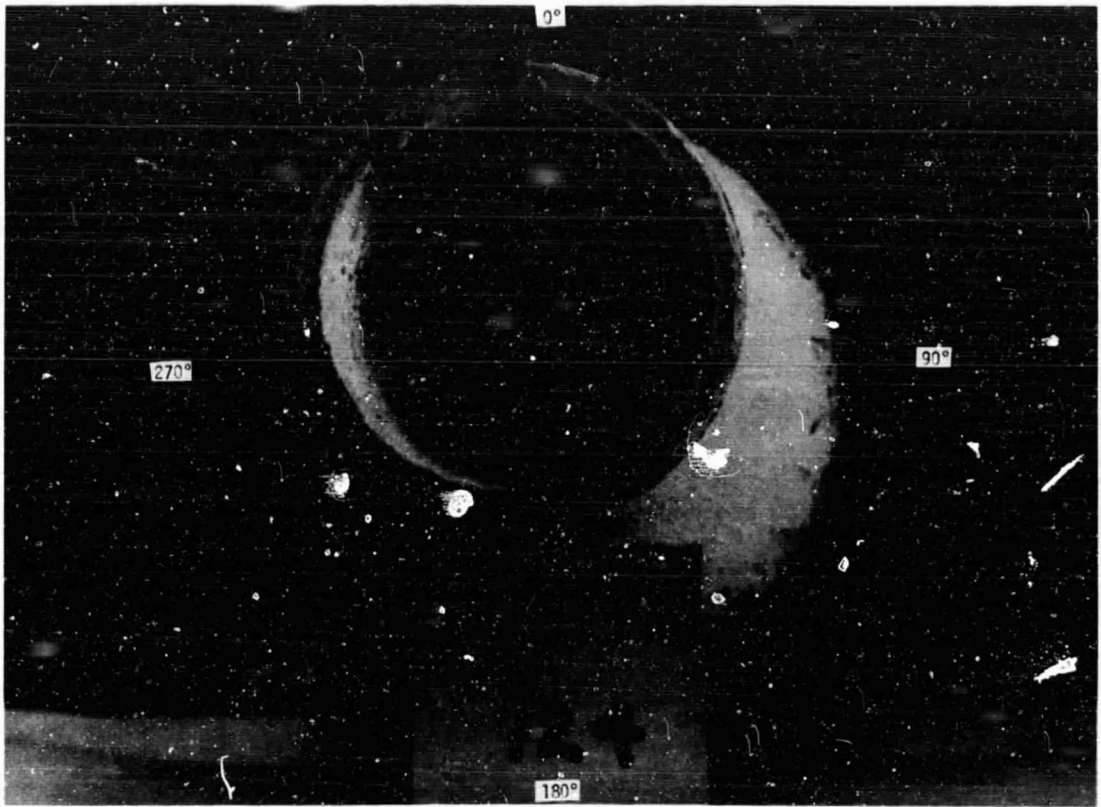
Figure 5. Post-Test Condition of S/N 023 - 670 sec at 4000°F



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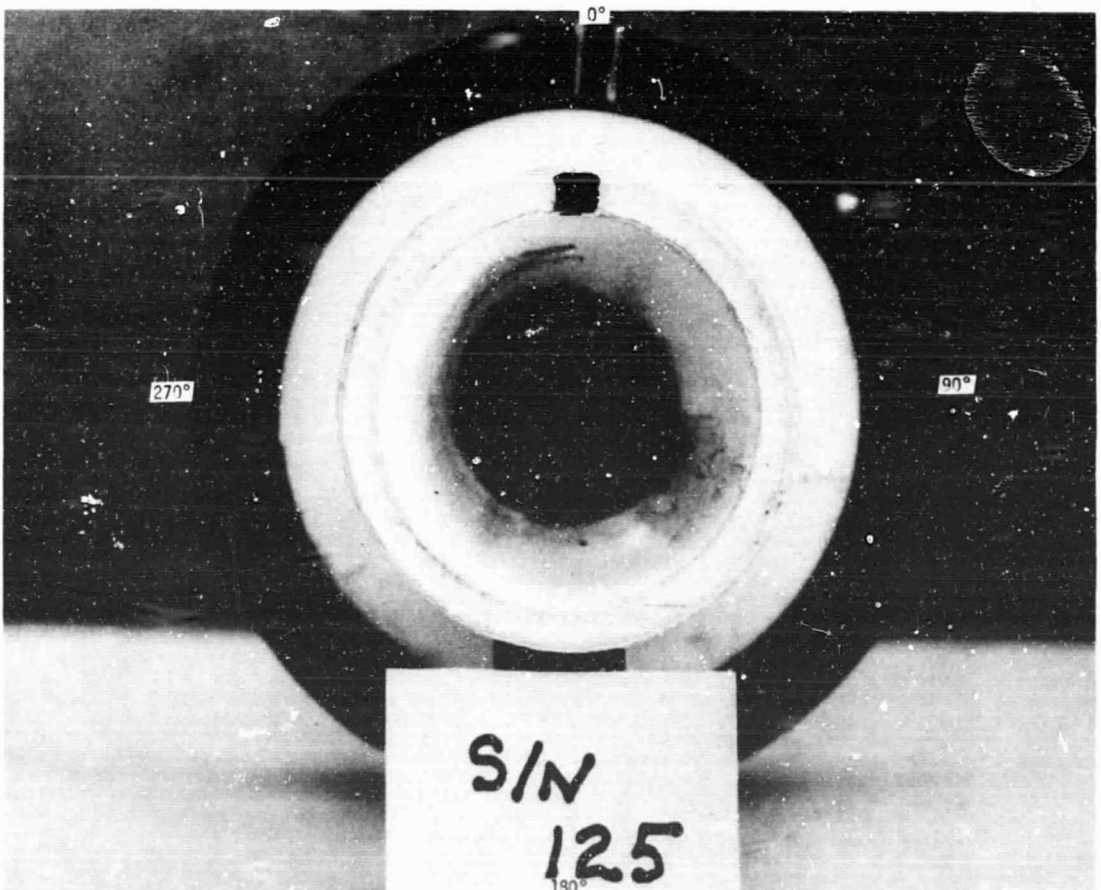
Figure 6. Post-Test Condition of S/N 121 - 1000 sec at 4500°F



Exit View

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Figure 7. Post-Test Condition of S/N 124 - 216 sec at 4500°F



Exit View

65651-59

Figure 8. Post-Test Condition of S/N 125 - 845 sec at 4500°F



Inlet View

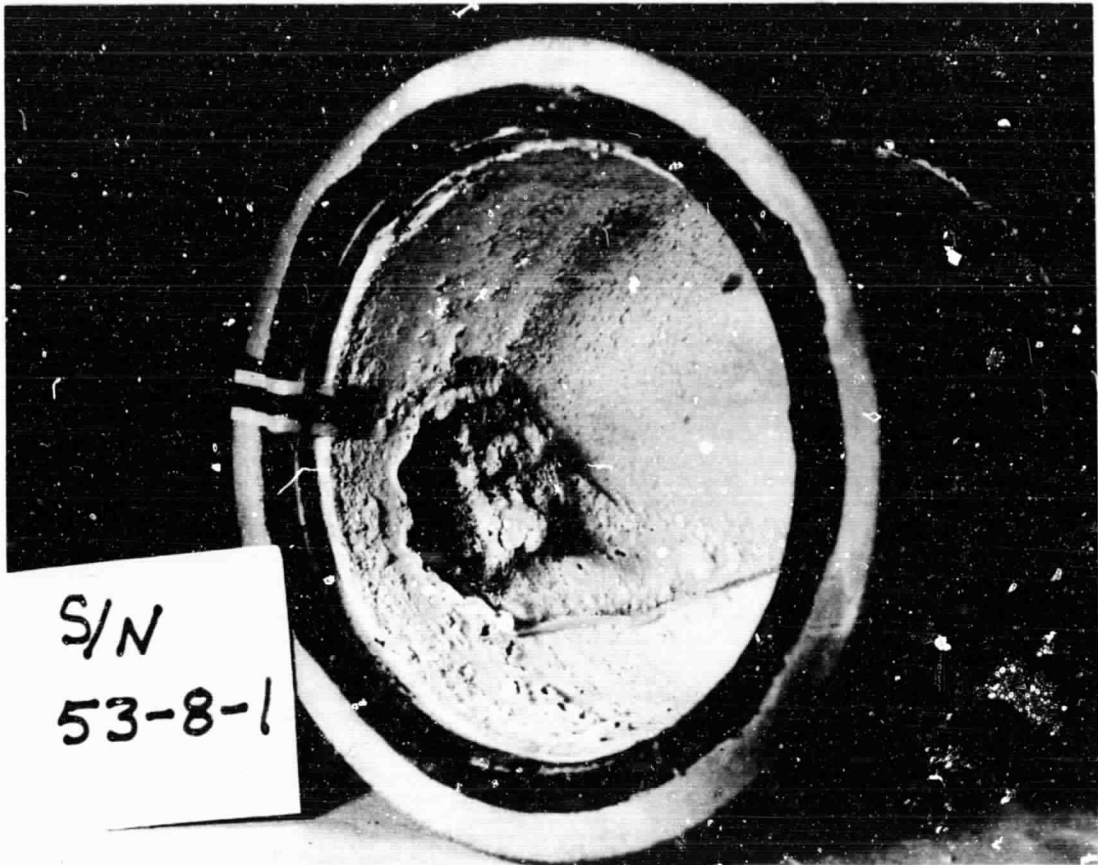
65054-69



Overall View

65056-69

Figure 9. Post-Test Condition of S/N 53-3-3 - 478 sec at 4000°F



S/N
53-8-1

Inlet View

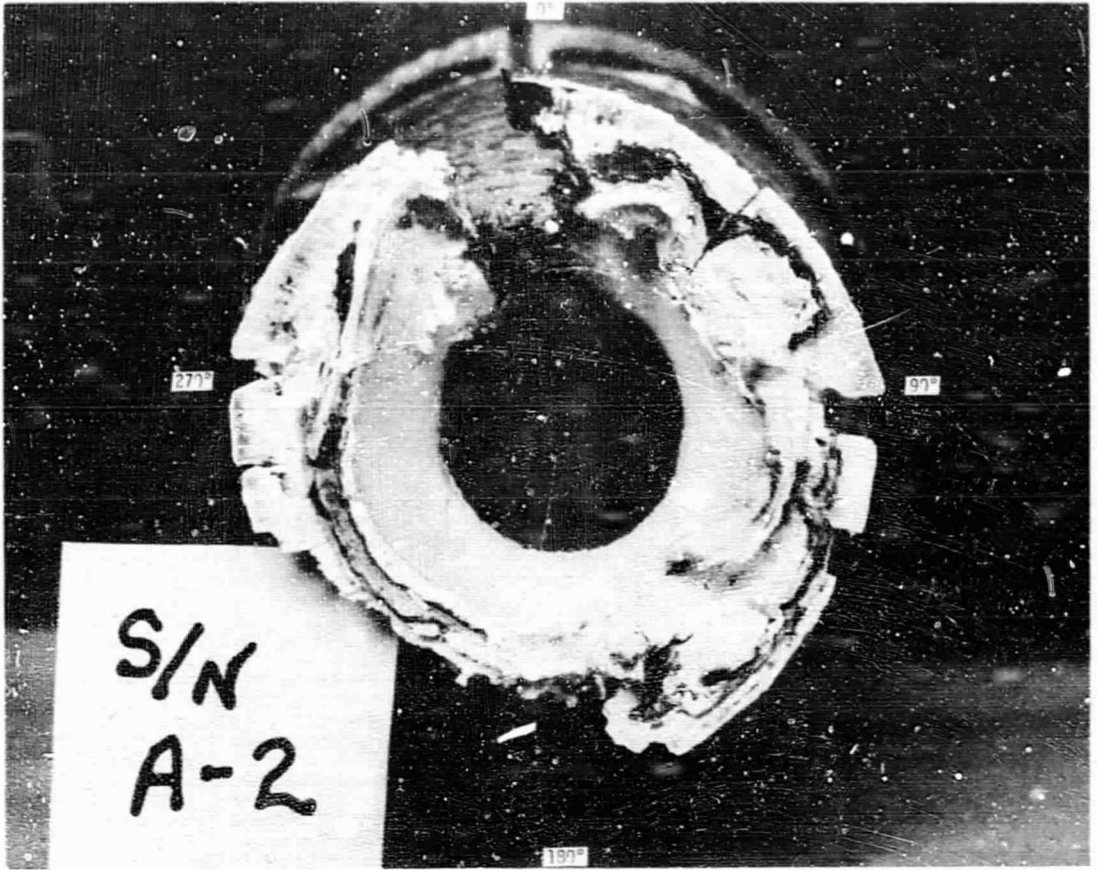
65653-69



Overall View

65654-69

Figure 10. Post-Test Condition of S/N 53-8-1 - 88 sec at 4500°F



Exit View

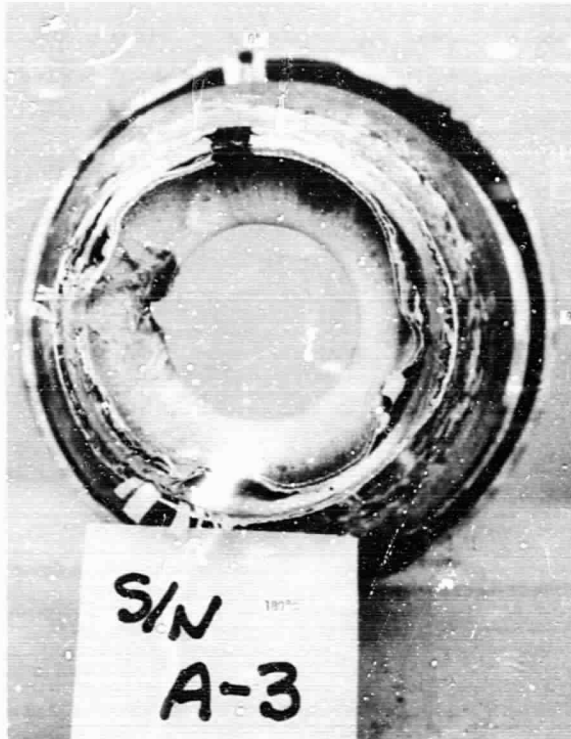
65766-69



Overall View

65777-69

Figure 11. Post-Test Condition of S/N A-2 - 3937 sec at 4500°F



Exit View

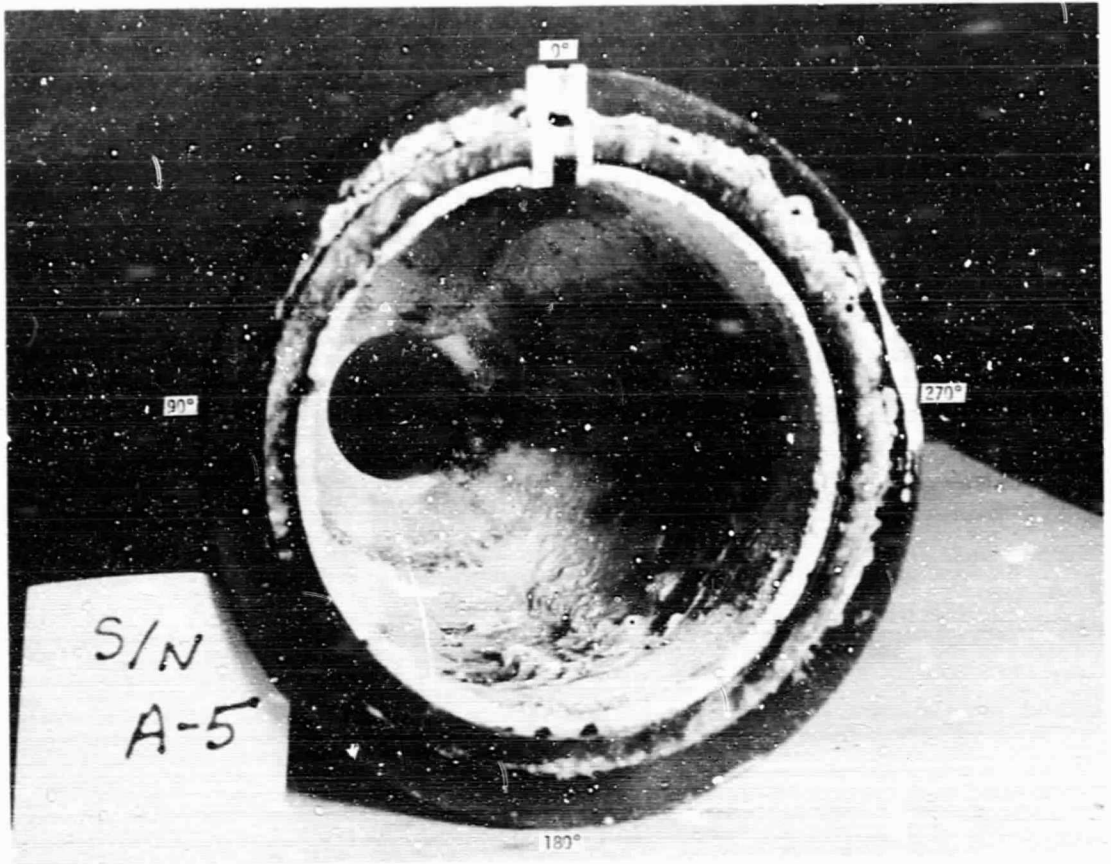
65768-69



Overall View

65779-69

Figure 12. Post-Test Condition of S/N A-3 - 587 sec at 4500°F



Inlet View

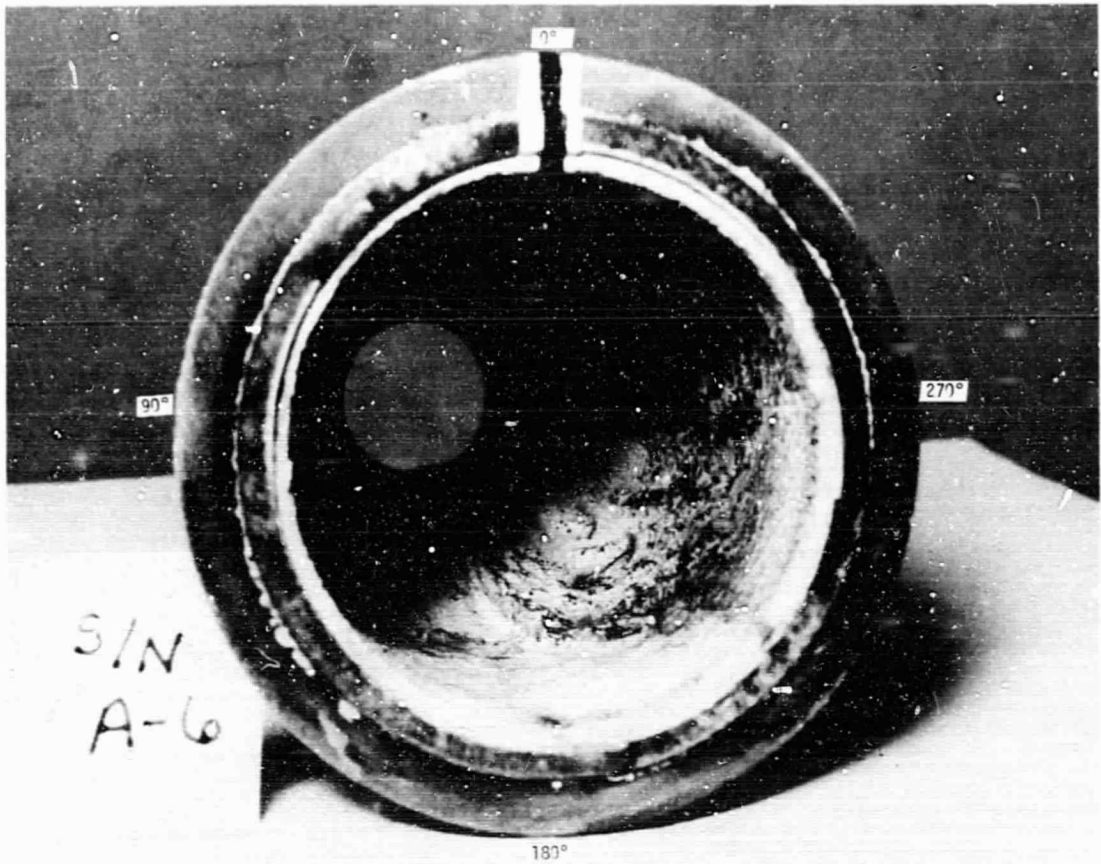
65775-69



Overall View

65782-69

Figure 13. Post-Test Condition of S/N A-5 - 50 sec at 4500°F



Inlet View

65776-69



Overall View

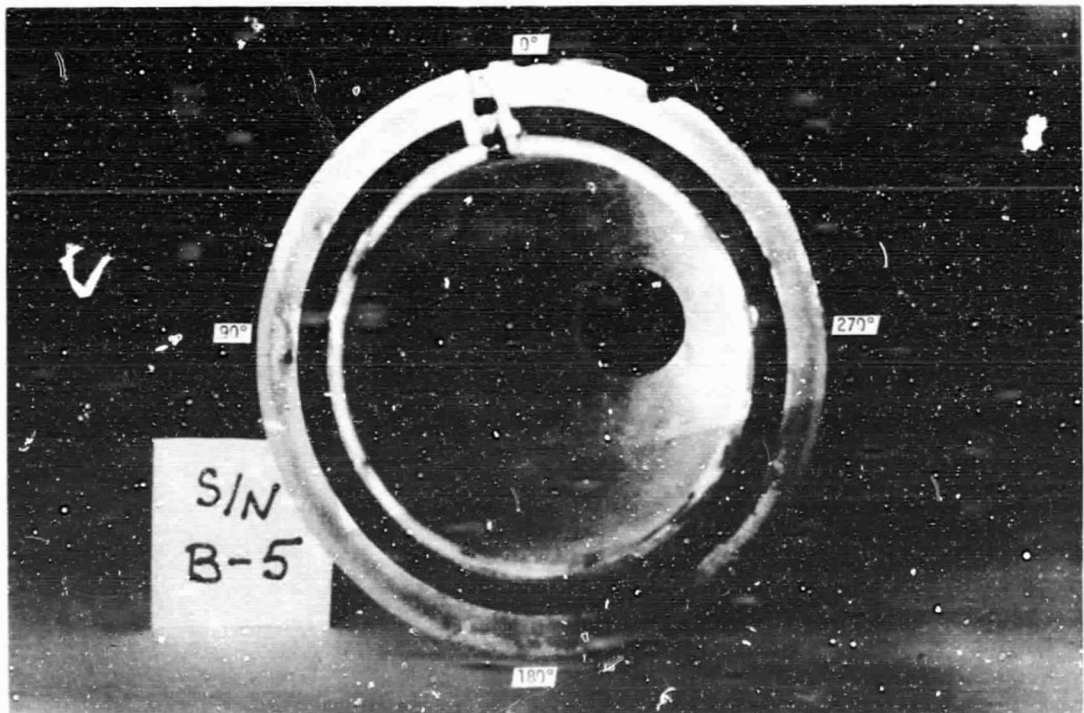
65783-69

Figure 14. Post-Test Condition of S/N A-6 - 63 sec at 4500°F



Exit View

65770-69



Inlet View

65781-69

Figure 15. Post-Test Condition of S/N B-5 - 634 sec at 4500°F



Exit View

65767-69



Overall View

65780-69

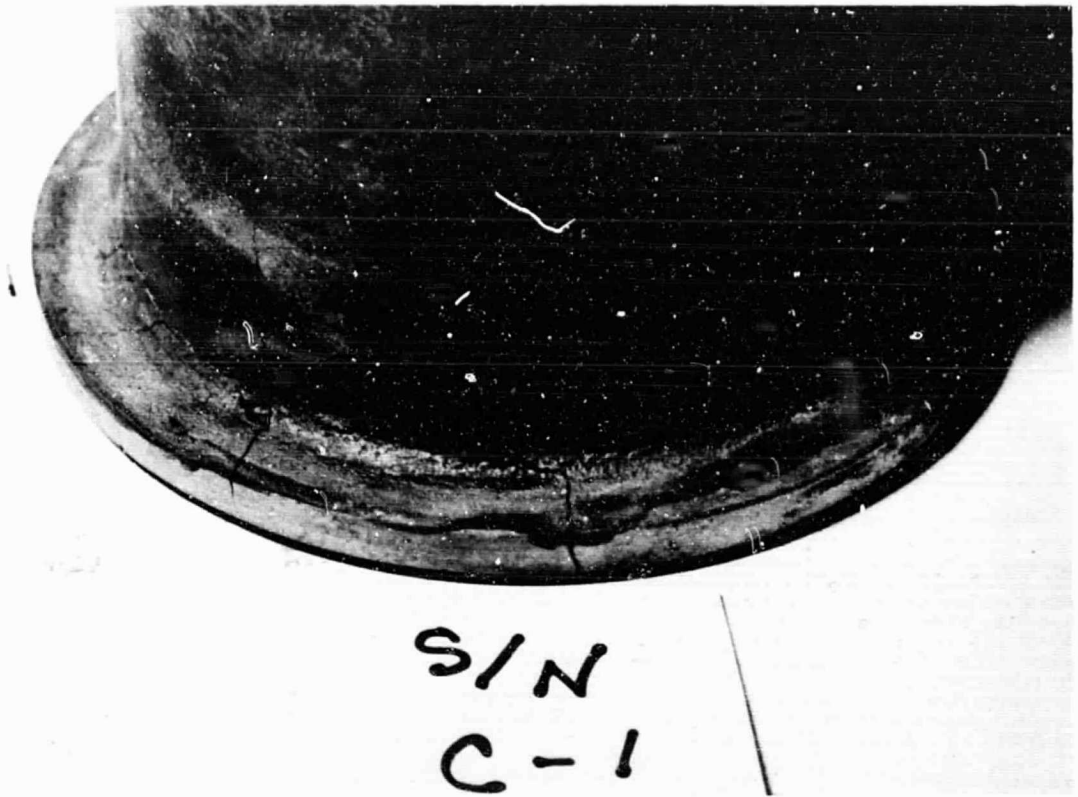
Figure 16. Post-Test Condition of S/N B-2 - 57 sec at 4500°F

TABLE II
EIGHTH QUARTER PRELIMINARY TEST RESULTS

| Test Specimen S/N | Coating | Target Combustion Temperature | Firing Time sec. | No. of Starts | Comments |
|-------------------|--------------------------|-------------------------------|------------------|---------------|---|
| NOZZLES | | | | | |
| 003 | Hf-20Ta Clad, Ta Barrier | 4500°F | 10 | 1 | Nozzle in serviceable condition. Gross clad separation at exit. Craters are developing on convergent section inside and outside |
| | | 4500°F | 1000 | 3 | |
| 013 | Hf-20Ta Clad, Ta Barrier | 4500°F | 5 | 1 | Nozzle in good condition. Some clad separation at exit but otherwise cladding is intact. |
| | | 4500°F | 10 | 1 | |
| | | 4500°F | 1000 | 5 | |
| 007 | Ir | 4500°F | 10 | 1 | Nozzle burnout at throat at 120° orientation. Extensive substrate oxidation on inside and outside. Much of exterior coating has spalled off and remaining coating is badly cracked and blistered. |
| | | 4500°F | 63 | 1 | |
| 022 | Ir/Re | 4000°F | 10 | 1 | Extensive spalling of coating on exterior and subsequent oxidation of the substrate. Internal spalling of coating at throat region from 170° to 260°. |
| | | 4000°F | 480 | 1 | |
| 023 | Ir/Re | 4000°F | 10 | 1 | Burnout downstream of throat at 200°. Gross coating spalling and oxidation of substrate on exterior. Internal coating surfaces are in good condition. |
| | | 4000°F | 670 | 1 | |
| 121 | Molybdenum Disilicide | 4000°F | 10 | 1 | Nozzle in excellent condition. A nick in the coating at 225° orientation on the exit rim has been enlarged due to substrate oxidation. |
| | | 4000°F | 10 | 1 | |
| | | 4000°F | 1000 | 3 | |
| | | 4500°F | 5 | 1 | |
| | | 4500°F | 1000 | 4 | |
| 124 | Hf-20Ta-0.25 Si | 4000°F | 5 | 1 | Substrate oxidation initiated at 320° orientation at throat. Flow of yellowish material from the oxidized area. Some thin surface spalling and cracks on remaining coating. |
| | | 4000°F | 10 | 1 | |
| | | 4000°F | 1000 | 2 | |
| | | 4500°F | 10 | 1 | |
| | | 4500°F | 216 | 1 | |
| 125 | Hf-20Ta-0.25 Si | 4000°F | 5 | 1 | Substrate oxidation initiated at 105° orientation at throat. Flow of yellowish material from the oxidized area. Remaining coating has some surface spalling and cracks. |
| | | 4000°F | 10 | 1 | |
| | | 4000°F | 1000 | 3 | |
| | | 4500°F | 10 | 1 | |
| | | 4500°F | 845 | 2 | |

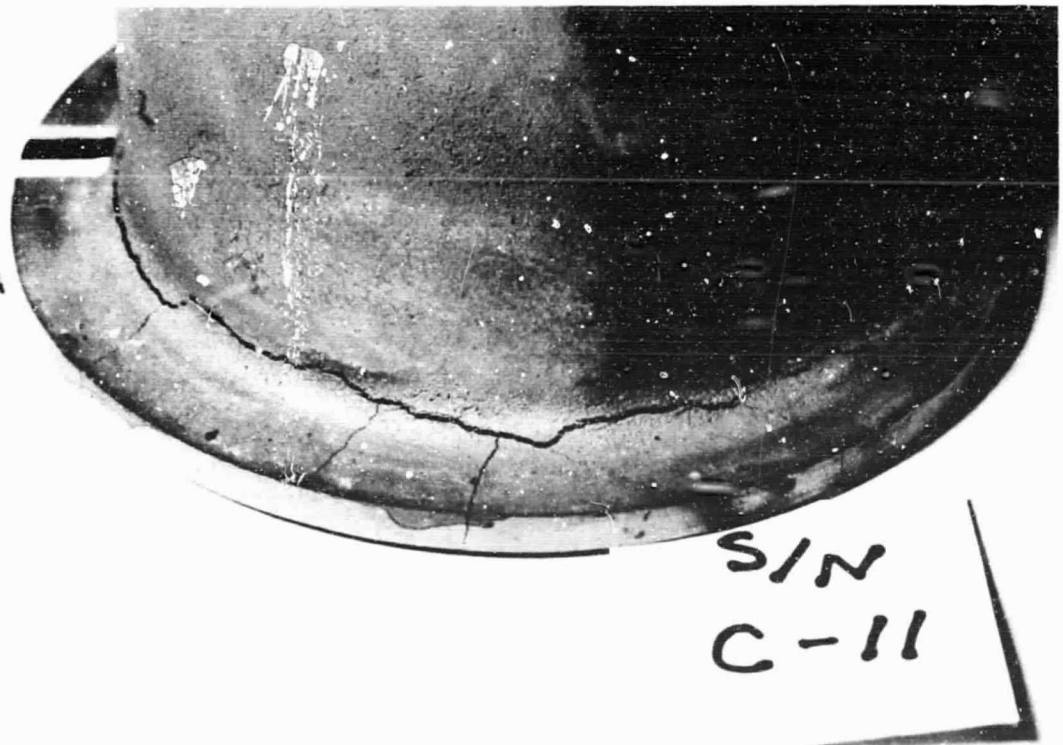
TABLE II
EIGHTH QUARTER PRELIMINARY TEST RESULTS (CONTINUED)

| Test Specimen S/ls | Coating | Target Combustion Temperature | Firing Time sec. | No. of Starts | Comments |
|--------------------|---------------|-------------------------------|------------------|---------------|---|
| CHAMBERS | | | | | |
| 53-3-3 | Hf-20Ta Clad | 4000°F | 5 | 1 | Chamber burnout in barrel at 24° orientation. Heavily cratered and oxidized on interior for 2.5 inch into the chamber barrel. |
| | | 4000°F | 478 | 1 | |
| 53-8-1 | Hf-20Ta Clad | 4000°F | 10 | 1 | Chamber burnout in barrel at 30° orientation. Cratering on interior for 2.5 inch into chamber barrel. Extensive spalling of surface coating during cool down after the test. |
| | | 4000°F | 1000 | 1 | |
| | | 4500°F | 10 | 1 | |
| | | 4500°F | 88 | 1 | |
| A-2 | Hf-Ta Clad | 4500°F | 10 | 1 | Burn through at throat and tearing to the nozzle exit at 350° orientation. Extensive clad separation and loss of substrate at exit. Crater formations on interior and exterior. Internal chamber barrel in reasonably good condition. |
| | | 4500°F | 3937 | 3 | |
| A-3 | Hf-Ta Clad | 4500°F | 10 | 1 | Burn through at throat at 300° orientation. Clad separation at exit. Some crater formation developing on outside and inside at throat. Internal chamber barrel in good condition. |
| | | 4500°F | 587 | 1 | |
| A-5 | Hf-Ta-W Clad | 4500°F | 10 | 1 | Chamber burnout in barrel at 280° orientation. Extensive cratering exposing oxidized substrate on interior of chamber barrel. Considerable flow of yellow/brown material from oxidized regions. Nozzle section and exterior in good condition. |
| | | 4500°F | 50 | 1 | |
| A-6 | Hf-Ta-Mo Clad | 4500°F | 10 | 1 | Chamber burnout in barrel at 230° orientation. Extensive substrate oxidation on interior of chamber barrel. Considerable flow of gray material from oxidized regions. Nozzle section and exterior in good condition. |
| | | 4500°F | 63 | 1 | |
| B-5 | Hf-Ta Slurry | 4500°F | 10 | 1 | Substrate oxidation in throat region at 220° orientation. Large blister formation downstream of throat on outside at 230°. Remaining coating in good condition. |
| | | 4500°F | 634 | 1 | |
| B-2 | Ir-Re | 4500°F | 10 | 1 | Burn through at throat at 220° orientation. Many deep longitudinal cracks in chamber barrel. Some cracks completely through the specimen. Prior to the test, the specimen had gas leakage through the chamber walls. Heavy blister formations on exterior barrel section. |
| | | 4500°F | 57 | 1 | |



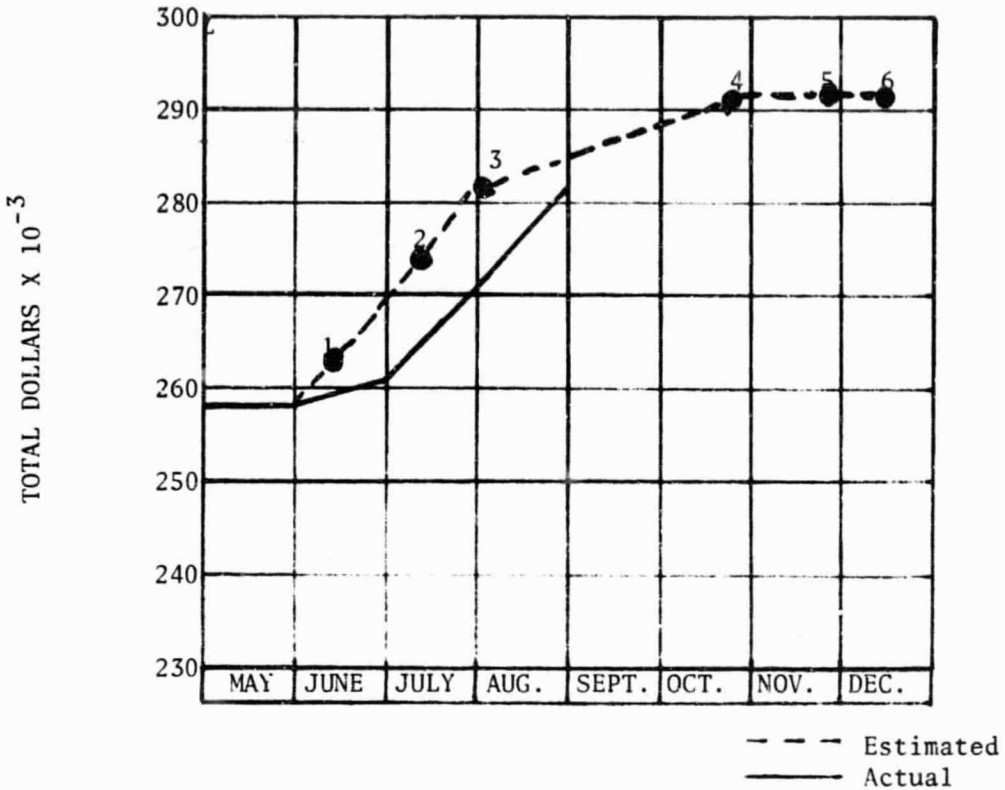
65785-69

Figure 17. Cracked Flange Area on S/N C-1



65784-69

Figure 18. Cracked Flange Area on S/N C-11



Total
Man Hours
x 10⁻³

| | | | | | | | |
|------|------|------|------|--|--|--|--|
| 12.5 | 12.7 | 13.3 | 14.0 | | | | |
|------|------|------|------|--|--|--|--|

MILESTONES:

1. Start Test Program
2. Complete Task III Tests
3. Complete Task IV Tests
4. Complete Task V Analysis
5. Submit Final Report for Approval
6. Distribute Final Report

Figure 19. Dollar and Manpower Expenditure Record