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**REFRACTORY AIR VANE AND REFRACTORY
MATERIAL RESEARCH AND DEVELOPMENT
Task II -- Refractory Materials for a Thrust Vector
Control Valve (U)**

July 1974

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Final Report -- Contract Number DAAG46-73-C-0051

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This document reports the results of a 12-month contract to evaluate candidate refractory and insulator materials for use as the inlet duct liner, rotor, and nozzle exit, of a hot gas thrust vector control system. The purpose of this investigation was to define materials that would provide a minimum weight valve design. Materials were compared for erosion resistance when subjected to the particle-laden hot gas environment from a solid propellant motor firing approximating that expected in an advanced interceptor control system application. The best materials selected through design, analysis and test were chemically vapor deposited tungsten for the valve rotor and nozzle and rubber modified silica phenolic for the inlet duct liner.

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FOREWORD

(U) This report was prepared by Martin Marietta Aerospace, Orlando Division, for the Army Materials and Mechanics Research Center, Watertown, Mass., under contract DAAG 46-73-C-0051, "Refractory Materials Research and Development." The work is part of the AMMRC program on Development of Hardened ABM Materials. Mr. John F. Dignam, Program Manager. The AMMRC technical supervisor was Mr. Lewis R. Aronin.

(U) The work on this contract consisted of two tasks. Task I was concerned with development of materials for refractory air vanes and is discussed separately in Volume I of this report. Task II which is reported herein (Volume II) was aimed at development of materials for thrust vector control valves.

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SUMMARY

(U) The purpose of this task was to select through design, analysis, and tests the best candidate materials for use as the inlet duct liner, rotor, and nozzle exit cone of a hot gas TVC system for an Advanced Interceptor Missile. The table shown below summarizes the total weight reduction of 49 percent that can be achieved through redesign of the chamber bleed hot gas thrust vector control/jet interaction control (TVC/JIC) system as a result of the development and tests on refractory and insulator materials conducted under this contract. The primary weight reductions are achieved in the rotor and nozzle. The current rotor design uses 0.3-inch thick copper infiltrated tungsten which can be changed to 0.06-inch thick vapor deposited tungsten in the improved design. The nozzles which are currently machined from copper infiltrated bar stock can be made of rubber modified silica phenolic structure coated internally with 0.060-inch layer of vapor-deposited tungsten.

Expected Control System Weight Improvement

	Current Design		Improved Design	
	Material	Weight (lb)	Material	Weight (lb)
Rotor	W-Cu, Ti	12.5	W, Ti	4.28
Structure	Steel	12.3	Titanium	7.75
Nozzles (2)	W-Cu	11.4	W, Rubber Modified Silica Phenolic	4.74
Miscellaneous	-	3.8	-	3.8
		40.0/Valve 160.0/System		20.57/Valve 82.28/System
Total Weight Reduction Expected = 78 Pounds (49 percent)				

(C) This weight reduction results in an increase in burnout velocity as follows: for an interceptor missile such as SPRINT, an advanced interceptor for which the aerodynamic drag is small compared to the thrust, the change in burnout velocity with respect to missile weight may be approximated by the expression:

$$\frac{\partial V_{BO}}{\partial W_{TOT}} = g I_{sp} \left(\frac{1}{W_{BO}} - \frac{1}{W_{TOT}} \right)$$

where

V_{BO} = velocity at burnout

W_{BO} = weight at burnout

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W_{TOT} = total weight at launch

I_{sp} = specific impulse

g = gravitation constant

(C) For the Advanced Terminal Interceptor (ATI) configuration studied, if the propellant weight is assumed to be held constant and the inert weight of the first stage varied, the above expression becomes 1.37 ft/s/lb. Therefore, the expected total weight reduction of 78 lb will result in an increase in ATI burnout velocity of 107 ft/s.

(U) To screen candidate materials, four materials tests were made. In each test, twelve specimens of refractory materials were subjected to supersonic flow of high-temperature, particle-laden gases for durations in excess of that required for Advanced Interceptor applications. In the first two tests, a production propellant (Sprint motor, FAE-7) was used, producing 6100°F gas with 13.6 percent aluminum oxide particles. In the third and fourth tests, an experimental propellant (FVB) was used which produced 6700 °F gas with 29.9 percent aluminum and zirconium oxide particles. The higher temperature and particle content of the FVB propellant produced higher erosion rates on all test specimens, as would be expected.

(U) Testing of the refractory materials revealed that vapor-deposited tungsten provides excellent erosion resistance and protection of lightweight substrate materials (specifically, Speer graphite, titanium, and rubber modified silica phenolic). Subsequent analyses showed that for the hot gas valve application the superior material combination is vapor-deposited tungsten protecting titanium for the valve rotor and protecting rubber modified silica phenolic for the valve nozzles.

(U) In the first test, nine candidate insulator materials were evaluated in the parallel, subsonic flow upstream of the nozzle. The flow was similar to that experienced by the valve inlet duct liner. Test results of the erosion measurements, when combined analytically with thermal conductivity characteristics, showed that rubber modified silica phenolic is the superior material for an insulator material. This material was used in the subsequent tests and erosion data were confirmed.

(U) Refractory materials which proved marginal in the test environment were plasma-sprayed tungsten and tantalum carbide, while those which proved unsuccessful because of excessive erosion or cracking were silicon carbide reinforced pyrolytic graphite; columbium, both with and without silicide coating; tantalum with 0.002-inch coating of tantalum carbide; Poco graphite and tantalum carbide coatings on graphite phenolic, rubber modified silica phenolic, Poco graphite, and titanium.

(U) Insulator materials which were only slightly inferior to the rubber modified silica phenolic were: MXA-150 stranded asbestos and laminated graphite phenolic, both end grain and 45 degree shingle angle. Those which proved unsatisfactory because of either high erosion or high thermal conductivity were: silicon carbide reinforced pyrolytic graphite, Poco graphite, epoxy Kynol, PRD-49 polyimide, and DC93-104 silicone rubber.

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I. INTRODUCTION

(U) Over the past several years, the Orlando Division of Martin Marietta Aerospace has been analyzing system requirements and potential technology improvements which might lead to a reduced-cost, higher-performance, more reliable interceptor system. One of these areas of investigation was the attitude control system of the type required on the first stage of an advanced interceptor or the first and second stages of a three-stage interceptor. These interceptors were configured to have high lateral maneuver requirements during motor burn to provide good intercept capability against maneuvering targets.

(C) In the Hardpoint Interceptor Study (HARPI) accomplished under ABMDA Contract DAHC60-70-C-0093, all three contractors involved in the study concluded that the first stage of the interceptor should be controlled by a chamber bleed hot gas thrust vector control (TVC) system. Such a system involves reliably valving hot, particle laden gases produced by the solid propellant rocket motor at a pressure of 2000 to 3000 psia and a temperature of 6100°F. The exhaust products include 13 percent aluminum oxide particles.

(U) The primary advantages of such a system for an Advanced Interceptor are: (1) minimum space and weight providing maximum overall vehicle performance by allowing, together with use of a buried nozzle, additional motor propellant packaging, and (2) very fast response time. The alternative of providing a movable nozzle was examined extensively in the HARPI study and found to have too long a response time for the Advanced Interceptor requirements.

(U) A survey of the field of the hot-gas TVC effort prior to 1968 showed that development of hardware to turn and valve the hot erosive gases had limited success; moreover, the work to that time had been primarily conducted on low pressure, long burn-time motors with low maneuver acceleration and, accordingly, was not applicable to advanced interceptor requirements. Thus, efforts to develop a chamber bleed hot-gas TVC design suitable for advanced interceptors was undertaken by Martin Marietta in 1968.

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(U) In general, the problem of designing a chamber bleed, hot-gas control system involved developing a flight-weight valving system having fast response and capable of withstanding temperatures to 6100°F and pressures up to 3000 psia. An analysis of the flow rates required to obtain the needed thrust deflection showed that the size of the valves was critical for the packaging space available in the aft missile skirt surrounding the nozzle. The necessity of reducing the valve size produced a design which resulted in combining the thrust vector control force with a complementing external force on the opposite side of the missile produced by the interaction of an external jet with the airstream. The design concept is illustrated in Figure 1. This opposite force, referred to as jet interaction control (JIC), reduced the required size of individual valves. Problems of pressure fluctuations in the motor, combined with the problems of clogging and erosion of a closure-type valve, led to consideration of a continuously flowing valve. It was found that, when duty cycle was high and when some thrust could be recovered during the "off" mode by flowing all valves into the main nozzle, the continuously flowing system could compete with a shut-off or demand type system.

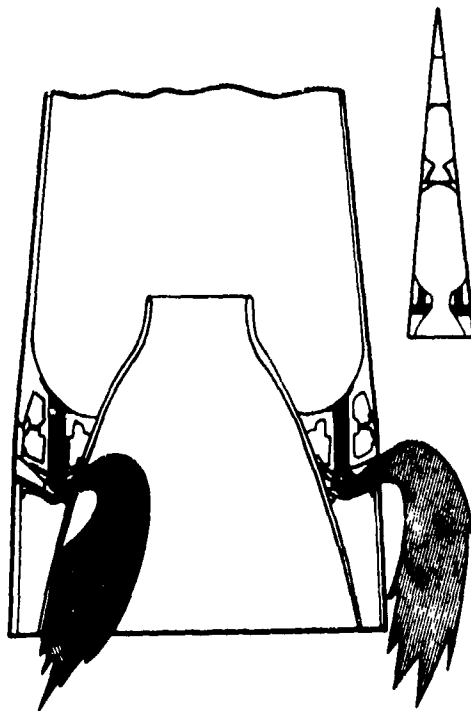


Figure 1 (C). Hot Gas TVC/JIC
Concept (U)

(U) Subsequent to initial development by Martin Marietta, a study contract (DAHC60-69-C-0137) for the U.S. Army Advanced Ballistic Missile Defense Agency (ABMDA) was completed in July 1970. Specific study tasks and their objectives were:

- Task I - Technology Evaluation: Evaluate existing technology, assess against requirements and select the best candidate for tests

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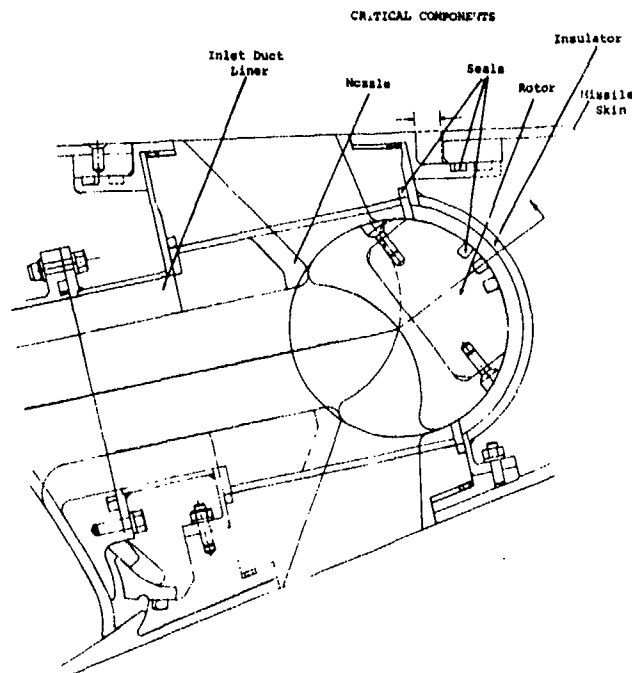
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- Task II - Design Study: Identify and evaluate interaction with other system components
- Task III - Component Testing: Perform exploratory tests to assess feasibility
- Task IV - Program Definition: Identify critical technologies and plan a program leading to complete development.

(U) During the technology evaluation, those requirements and factors which influence the design of a hot-gas TVC system were identified and a set of baseline requirements established.

(U) Subsequently, all conceivable systems were compared on a total system weight basis, including the weight of nonthrust producing propellant. These system comparison studies include seven types of shutoff and continuous flow valves arranged in all combinations of TVC only, TVC and thrust, and TVC and JIC. The results showed that the rotary valve (suitable for high acceleration loads) was superior since, with valves used in complementary pairs, TVC forces from the gas flow into the nozzle could be added to JIC forces produced by the flow through a valve on the opposite side of the missile. This system yielded the lowest total weight system, principally because the requirements could be met with four valves, whereas most of the other systems required eight or more valves because of size and packaging space restrictions. During the design study, thrust and torque were identified as critical parameters affecting other missile components. The position of the injection point was studied and found to be of secondary importance within certain limits. Survivability of the seals and suitability of the materials selected for a lightweight design were identified as critical. A full scale design layout was made to conduct a detailed weight analysis and to study the sensitivity to changes in various requirements. The design is illustrated in Figure 2.

Figure 2 (U). TVC/JIC Valve Installation



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(U) During the component testing task, the critical, lightweight internal components of the valve were subjected to hot-gas tests at various pressures and test times. Design problems were uncovered and corrective action demonstrated in subsequent tests. The following information was obtained from these tests:

- 1 Structural Integrity: the hardware proved capable of turning and valving the 6100°F gas supplied at 3000 psia.
- 2 Erosion: The erosion of the closed nozzle proved more severe than that of the open nozzle and in all cases occurred remote from the valve throat, affecting structural integrity but not performance. Inlet duct erosion was negligible.
- 3 Sealing: The redundant seal design proved effective with static radial seals of metal surfaces more reliable than face seals of non-metal surfaces.
- 4 Temperatures: Internal temperatures agreed with thermal analysis predictions. Dimensional clearances provided for thermal expansion proved to be critical from a frictional torque standpoint.
- 5 Thrust and Torque: The measured thrust and rotor torque values agreed with analytical predictions. The thrust-position relationship is close to linear. The total torque required was found to consist of approximately equal components of frictional and gas flow torque.

The tests produced needed design data on erosion, thrust, and actuator torque requirements.

(U) The program definition was completed with the overall development program outlined in five logical phases, including both tests and analyses; as planned, each phase provides solutions to specific technical problems plus investigations of those problems to be solved in a subsequent phase.

(U) The final report on that study contract, OR 10,913, entitled "Hot Gas Thrust Vector Control Study," dated October 1970, may be obtained through SAFEGUARD System Command, Huntsville, Alabama 35807, Attn: CRDABH-M.

(U) After the study contract, a development contract (DAHC60-71-C-0044) for the U.S. Army Advanced Ballistic Missile Defense Agency (ABMDA) was completed in May 1972. The objective was to develop, by testing a continuous flow TVC/JIC rotary valve.

(C) Through a series of five development tests involving design changes, a valve and inlet duct assembly was developed for the rotary valve TVC/JIC system. The developed design which was successfully demonstrated is shown in cross-section in Figure 3. The final development assembly weighs 40 pounds and, at a chamber pressure of 3100 psia, has a flow rate of 55 lb/s with a 6100°F gas that has an aluminum oxide content of 13.6 percent. The tests included valve operation with gas generator burn times of 1.215 to 2.65 seconds with two valve and duct assemblies (27.5 and 55 lb/s) successfully demonstrating a lack of scaling problems. During the course of the

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development study, the weight of the 55 lb/s valve and duct assembly was successfully reduced from 75 to 40 pounds primarily by decreasing the valve diameter. The weight of the rotary valve less duct would be 37 pounds. Additionally, friction was identified as the primary contributor to rotor torque, which was reduced from a 16,500 in-lb maximum to 9000 in-lb. A summary of the test results is presented in Table I. The tests were numbered 4 through 8 to maintain continuity of development with the previous 3-valve test program under ABMDA Contract DAHC60-69-C-0137 conducted in 1969 and 1970. Throughout the aforementioned development efforts attention was directed mainly toward the solution of sealing problems, the measurement of required actuator torque, the linearity of thrust versus rotor position, the structural adequacy of the design, and the updating of thermal analysis and temperature prediction techniques. Little effort was directed toward materials optimization.

(U) Further studies of Advanced Interceptor performance highlighted the importance of minimizing control system weight to obtain maximum interceptor velocity at burnout. An examination of the design and material selections for the chamber bleed TVC/JIC system showed that important weight savings could probably be achieved through optimization of the hot-gas valve material selections. Referring to Figure 3, a cross-section of the developed 55 lb/s valve assembly various design features will be noted. The 6100°F gas at 3000 psia enters the valve at the bottom of the figure, flowing upward subsonically through the passageway lined with laminated graphite phenolic insulator material. The purpose of this material is to maintain a suitable heat insulation for the steel structure. A material with lower erosion and/or lower thermal conductivity would allow use of a smaller diameter inlet duct reducing structural weight.

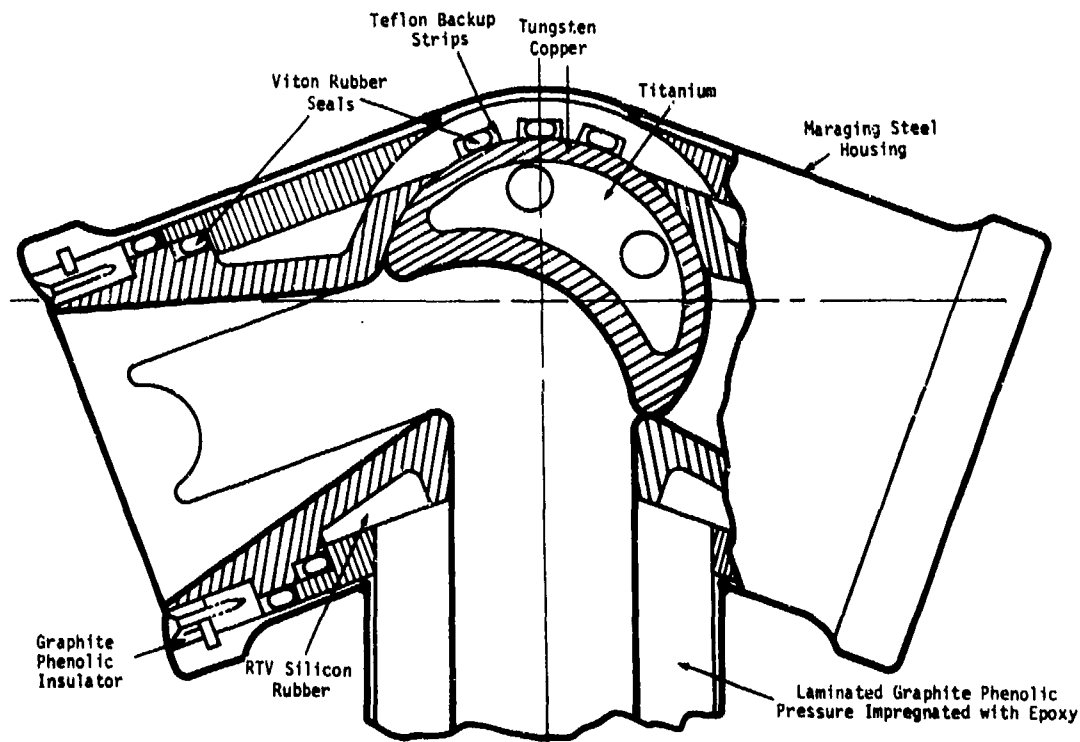


Figure 3 (C). Cross-Section of 55 lb/s Valve Assembly
for ABMDA Test No. 8 (U)

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TABLE I (C)

Summary of Test Results
Obtained from Previous Contract (U)

	Test No. 4 (051471)	Test No. 5 (071571)	Test No. 6 (102071)	Test No. 7 (012572)	Test No. 8 (032872)
Test Environment					
Burn Time (seconds)	2.65	2.40	2.32	2.07	1.21
Pressure (psi)	2270 to 2720	2500 to 3020	2550 to 3000	2600 to 3100	2550 to 3250
Test Results					
Actuator Torque Required (in-lb)	6000	8000	16,000	16,500	9000
Erosion Rate of Duct (in/s)	0.038	0.042	0.040	N/A	0.041
Rotor Centerline Temperature (°F at 2.0 seconds)	2020	2080	1800	1810	-
Insert Side Temperature (°F at 2.0 seconds)	1850	1830	1910	1750	-
Unaugmented Thrust (lb)	5000	4400	11,200	10,500	9500
Valve and Duct Characteristics					
Gas Flow Rate (lb/s at 3100 psi)	27.5	27.5	55.0	55.0	55.0
Weight of Assembly (lb)	44	44	75	72	40
Rotor Diameter (in)	3.125	3.125	4.250	4.250	3.125

(U) It can be seen that the subsonic flowing gas impacts the curved surface of the rotor face which turns the flow. The flow speeds up in the turn reaching sonic velocity at the throat and expands supersonically to the nozzle exit. Test results showed that negligible oxidation of the rotor and nozzle materials occurred since the gas presented a reducing environment, that rotor surface erosion and internal temperatures were both lower than originally predicted probably due to a buildup of a protective surface layer of aluminum oxide in liquid form at these temperatures and pressures. It could be further seen from tests that negligible erosion of the rotor or nozzles occurred when they were made of copper infiltrated tungsten. However, the copper, which melted and exuded from the porous tungsten tended to condense on the rotor backface increasing actuator torque requirements. It was postulated that major weight reductions could be achieved in the rotor and nozzle by reducing the thickness of the tungsten coating or by using a lighter weight refractory coating even at the expense of a small amount of erosion. It was further postulated that a lighter weight servo-actuator could be used if torque requirements were reduced through elimination of the molten copper at the rotor surface.

(U) A refractory materials development program was therefore defined for AMMRC to screen and select optimum refractory and insulator materials and compositions for the purpose of reducing the weight of the hot-gas TVC/JIC valves for Advanced Interceptor control systems. This program was later expanded to include materials screening tests in which the materials were subjected to the environment of the higher temperature and solids content of a high burn rate propellant (FVB) under development for Advanced Interceptor as well as the current available solid propellant (FAE-7).

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II TEST APPROACH

1. Test Fixture

(U) A test fixture was designed to simulate the environment experienced by the inlet duct, rotor and nozzle discussed in Section I, "Introduction". A cutaway view of the test fixture is shown in Figure 4. It consists of a subsonic inlet duct, in which insulator specimens can be subjected to the low velocity parallel flow exactly duplicating that of the actual valve inlet, and provisions for mounting cylindrical test specimens in the supersonic flow downstream of the sonic nozzle. The cylindrical specimens are in the gas stream in a region that simulates the environment experienced by both the rotor and nozzles in the actual valve. The gas and particles, in their expansion from the sonic throat of the test fixture, impinge on the cylindrical specimens as shown in Figures 5 through 7. Because of erosion of the test fixture throat and differences in the chamber pressure of the gas generator, different gas and particle impingement angles were experienced by the cylindrical specimens. In all cases, calculations showed that the velocity component normal to the specimen exceeded by at least two times the impingement velocity experienced by the rotor in the actual valve while the particle impact angle of approximately 20 degrees at supersonic velocity simulates the actual impact angle experienced by the most vulnerable area of the nozzle, that is the upper area of the nozzle exit cone where the centrifugal force on the particles has thrown them to the outside of the turned stream (See Figure 3).

2. Specimen Installation

(U) The insulator specimens in the first test, consisting of 1/2 inch thick rings, were installed upstream of the nozzle where they were subjected to subsonic parallel flow. Washers made of the baseline insulator material (laminated graphite phenolic end grain) were installed between the specimens to prevent fratricide. In addition, a graphite phenolic sleeve was installed between the specimens and the tube to assure a margin of safety for the test allowing for complete loss of a specimen. The refractory specimens, consisting of 3/4 inch diameter rods 2 inches long, were installed vertically around the nozzle exit where they were subjected to the supersonic flow and particle impingement of the expanding gases. Instrumentation consisted of chamber pressure, three calorimeters (on one of the refractory specimens), and five high speed motion picture cameras.

(U) In the second, third and fourth tests, the inlet duct liner and nozzle throat were made of laminated rubber modified silica phenolic, the insulator material which proved superior to the other candidates in the

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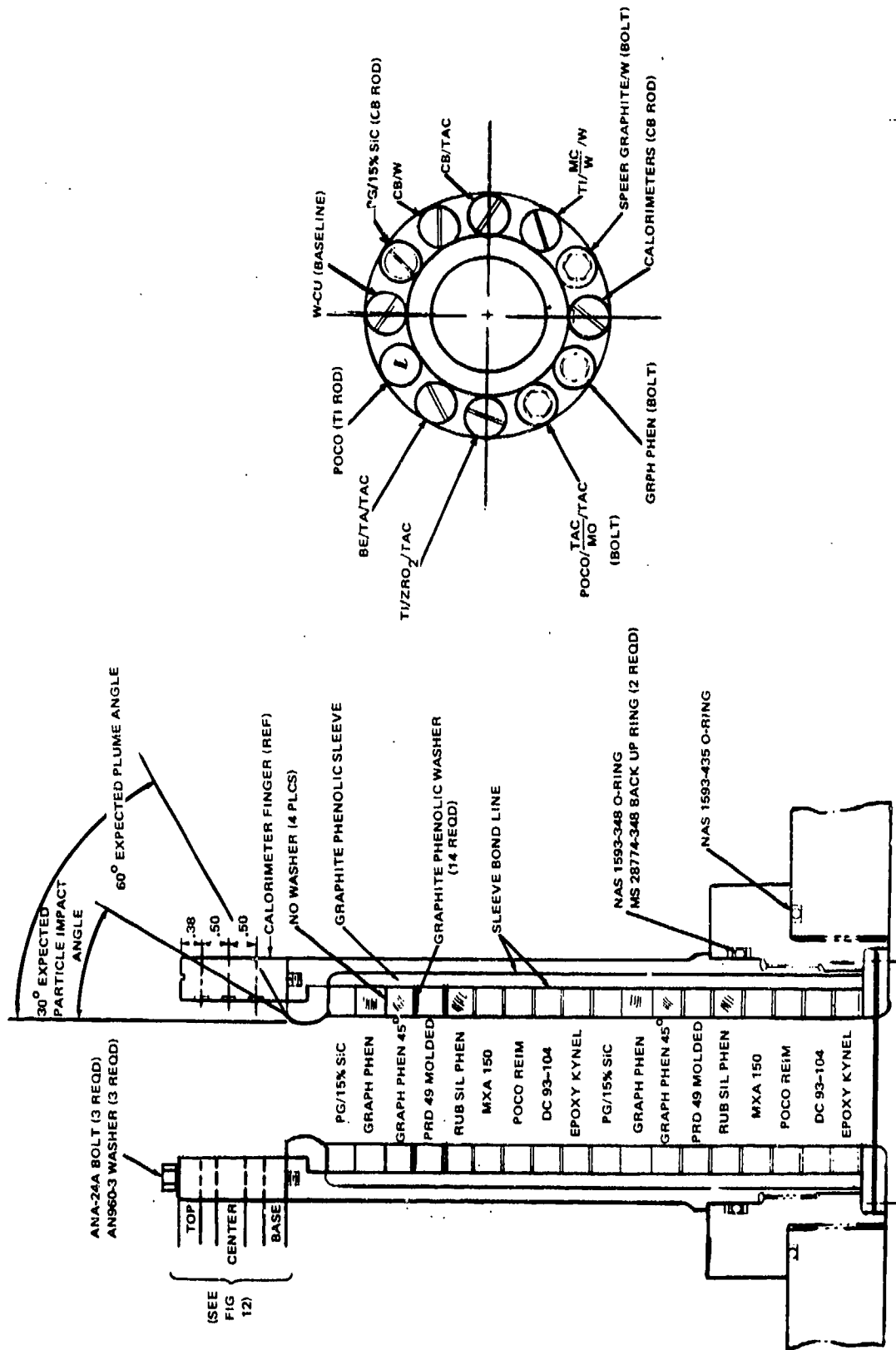
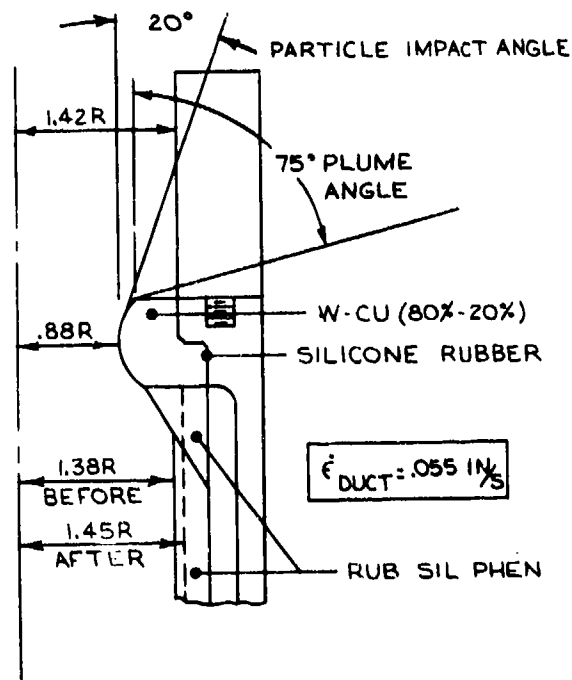
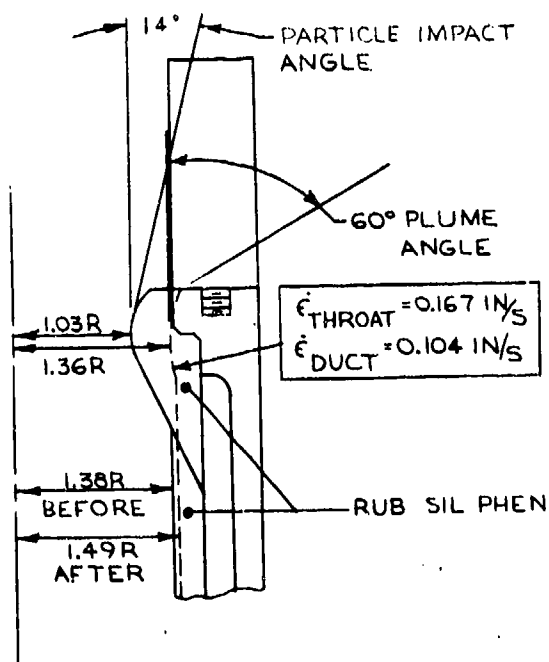
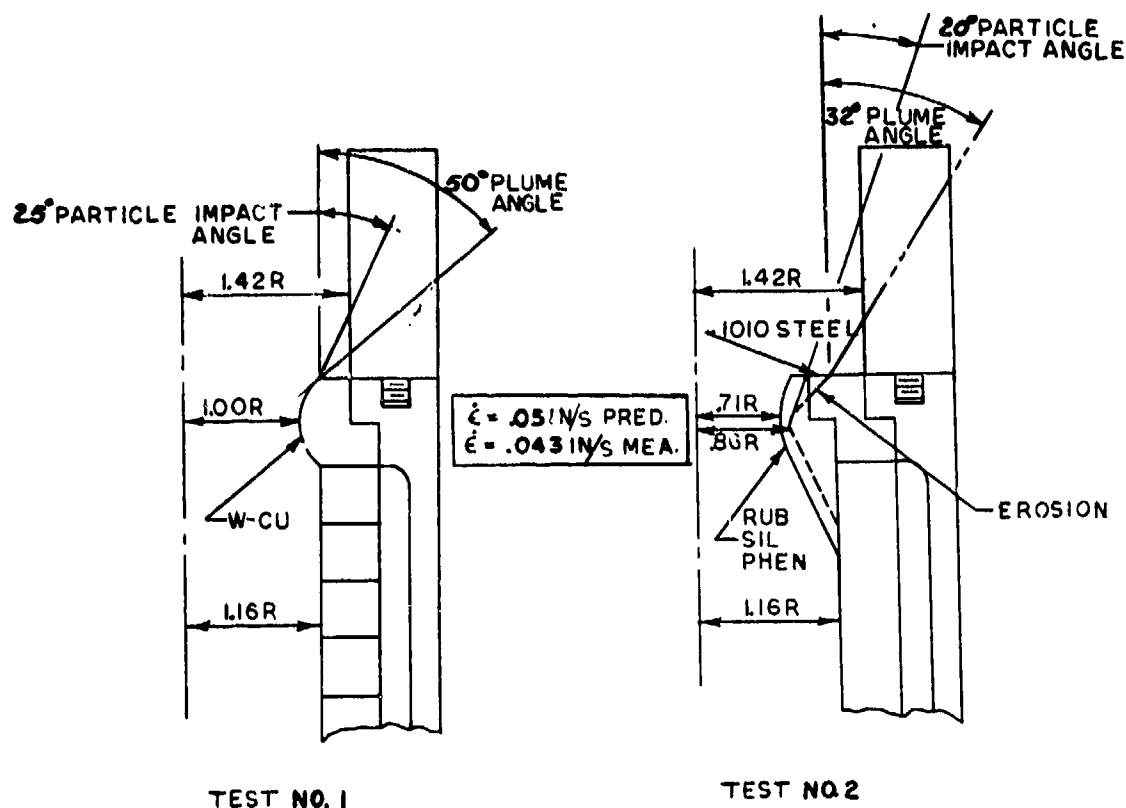


Figure 4 (U) - Test Fixture

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initial test. Figure 5 illustrates a cross-sectional view of the test nozzle showing the configuration for test numbers 1 and 2. The throat material was changed from tungsten-copper to rubber modified silica phenolic to avoid copper particles eroding the specimens and obscuring test results, although erosion of the throat results in a regressive pressure trace. Figure 6 illustrates the cross-sectional view of the test nozzle for test No. 3. It can be seen that the propellant produced a severe erosion of the rubber modified silica phenolic in the throat area. Figure 7 illustrates before and after views of the nozzle throat area for test no. 4. The throat material was tungsten-copper which did not erode, producing a relatively constant pressure over the test time.

(U) Figure 8 shows a view of the overall test setup. The duct with the twelve refractory specimens mounted at the top is installed on top of a gas generator so that all the products of combustion pass through the duct. The gas generator case is adapted from a SPRINT launch eject gas generator and is capable of withstanding pressure up to 10,000 psia. The view shows the igniter wires on the left leading down through the duct to the pyrotechnic igniter installed on top of the end-burning propellant grain. The wires on the right lead to the three calorimeters installed on one of the refractory test specimens.

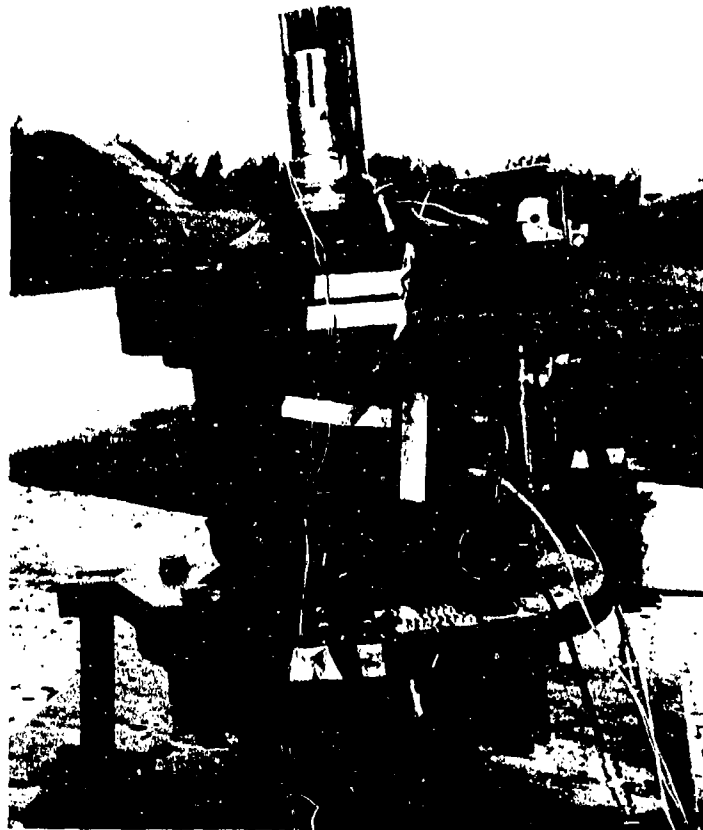


Figure 8 (U). Test Setup

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III. ENVIRONMENT IMPOSED

(U) The heating imposed on the refractory specimens between 0.5 and 1.0 inch from the base was equivalent to that experienced by the valve nozzle at the throat and approximately 40 percent of that experienced by the valve rotor. This information was obtained by a comparison study in which the depth of depletion of copper in the tungsten-copper test specimen was compared with the depth of copper depletion experienced by the rotor and nozzle used in the previous ABMDA-sponsored valve development test. Figure 9 shows a cross-sectional view of the last valve built and tested under the ABMDA sponsored program with the areas indicated from which samples were cut. These two areas were known to be those of highest heating from earlier analysis and tests. Figures 10 and 11 are photographs (enlarged 4 times) of the samples cut from the rotor and nozzle respectively showing the heat affected zones along the surface. The copper was depleted to a depth of 0.075 inch on the rotor sample and 0.020 inch on the nozzle sample.

(U) Figure 12 shows a tungsten-copper refractory specimen from Test Firing No. 2 of this contract which was similarly sectioned. Three areas were examined, one near the mounting base, one about half-way up the specimen, and one near the top of the specimen. The maximum depth of copper depletion found was 0.030 inch, which occurred in the area sectioned from about half-way up the specimen and was found to be 0.860 inch up from the mounting base. (Note - The notches shown in the parts illustrated in Figure 12 were for convenience of identification only).

(U) Figure 13 shows a plot of the measured pressure-time curves of the gas generator for the four tests. For tests number 1 and 2, the propellant was FAE-7 (SPRINT) which produces 6100°F gas with 13.6 percent aluminum oxide with the grains sized for 1.5 and 3.5 seconds duration, respectively while for tests 3 and 4, the propellant was FVB (an experimental propellant under development by Hercules, Inc., and supplied to this program by MICOM) which produces 6750°F gas with 29.7 percent solids consisting of aluminum oxide and zirconium oxide. The regressive traces of tests 2 and 3 were due to enlargement of the throat through erosion of the rubber modified silica phenolic (RMSP).

(U) Figure 14 shows a set of sequential views of the test in progress.

(U) While the gas generator pressure affects the impact angle of the particles and the edge of the plume against the test specimens, the primary item of interest on these curves is the time that the specimens were subjected to the hot, erosive environment. It can be seen that in all cases the design time of 1.3 seconds was met or exceeded.

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(C) Table II shows the theoretical characteristics of the two motor propellants used in the tests. The purpose of developing the new FVB propellant is to obtain a higher burn rate which will allow a higher loading efficiency for the motor. The higher burn rate of FVB (12 inches per second instead of 3.6 inches per second for FAE-7) is achieved by increasing the aluminum staple, adding zirconium staple, and by using ultra-fine ammonium perchlorate (2 micron particle size instead of 9 to 10 micron size used in FAE-7). Because the addition of zirconium tends to decrease the specific impulse, compensation is achieved by using greater amounts of aluminum powder so that approximately the same delivered specific impulse is achieved. For the two FVB test grains, which are still in the experimental development phase, the actual burn rate was 10.07 inches per second at 2000 psia instead of the anticipated 12 inches per second. This variation was due to processing problems associated with a low screen loading density.

(C) The disadvantages of FVB propellant are: (a) more vulnerability to some types of nuclear effects particularly exo-atmospheric where there is no X-ray attenuation (but not necessarily the Advanced Terminal Interceptor environment) and (b) higher RF attenuation and phase shift shown in laboratory tests. In addition, the zirconium is a more expensive raw material and has higher processing costs due to the necessity of keeping the zirconium immersed in a solvent during some of the processing steps for safety reasons.

(C) The advantages of FVB propellant are: (a) it is the only propellant formulation approaching an off-the-shelf status that has the required ballistic and physical properties for Advanced Interceptor and (b) it does not rely on ammonium perchlorate of less than one micron and a catalyst to achieve the required burning rates.

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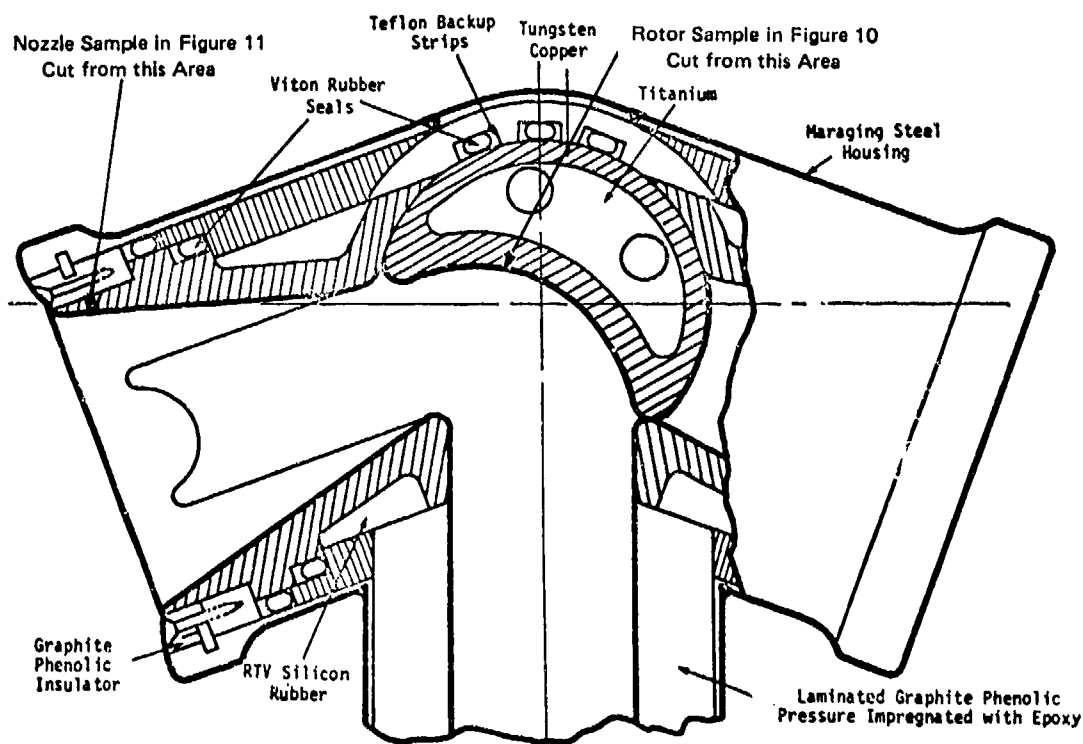


Figure 9 (C). Location of Rotor and Nozzle Samples Taken From
55 lb/s Valve Assembly for ABMDA Test No. 8 (U)

TABLE II (C)

Theoretical Characteristics of Motor Propellant (U)

	Percent by Weight	
	FAB-7 (SPRINT)	FVB (Advanced Interceptor Candidate)
Constituent		
Nitro-cellulose	18	16.6
Nitro-glycerine	30	34.05
Ammonium perchlorate	36	27.1
Aluminum	7.2	11.9
Triacetin	6.7	2.8
Resorcinol	1.1	1.08
2-nitro-diphenylamine	1.0	0.72
Zirconium	-	5.75
	100.0	100.0
Exhaust Products		
Al ₂ O ₃ (liquid)	13.4	22
ZrO ₂ (liquid)	0	7.7
Combustion Chamber Properties		
Temperature	6037°F	6745°F
C*	5191	5160
Vacuum specific impulse	305.3	307.1
Ratio of specific heats	1.197	1.170
Effective gas constant	52.63	46.29
Effective molecular weight	29.356	33.377
Propellant density	0.0618 lb/in ³	0.0661 lb/in ³

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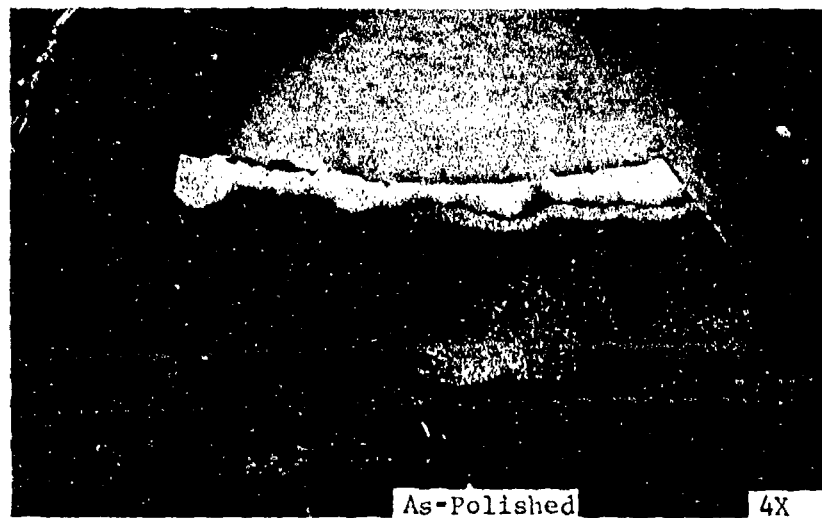


Figure 10 (U). Heat Affected Zones Along the Surface of the Hot Area of the Rotor

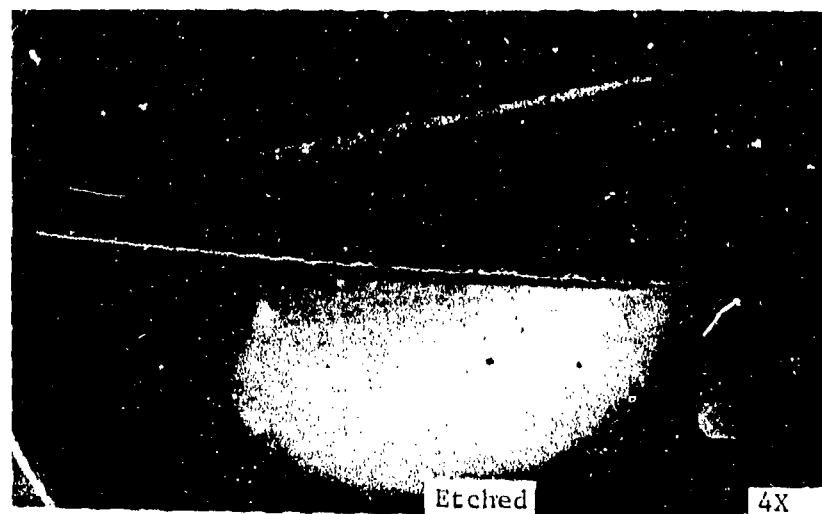


Figure 11 (U). The Heat Affected Zones Along the Surface of the Hot Area of the Nozzle

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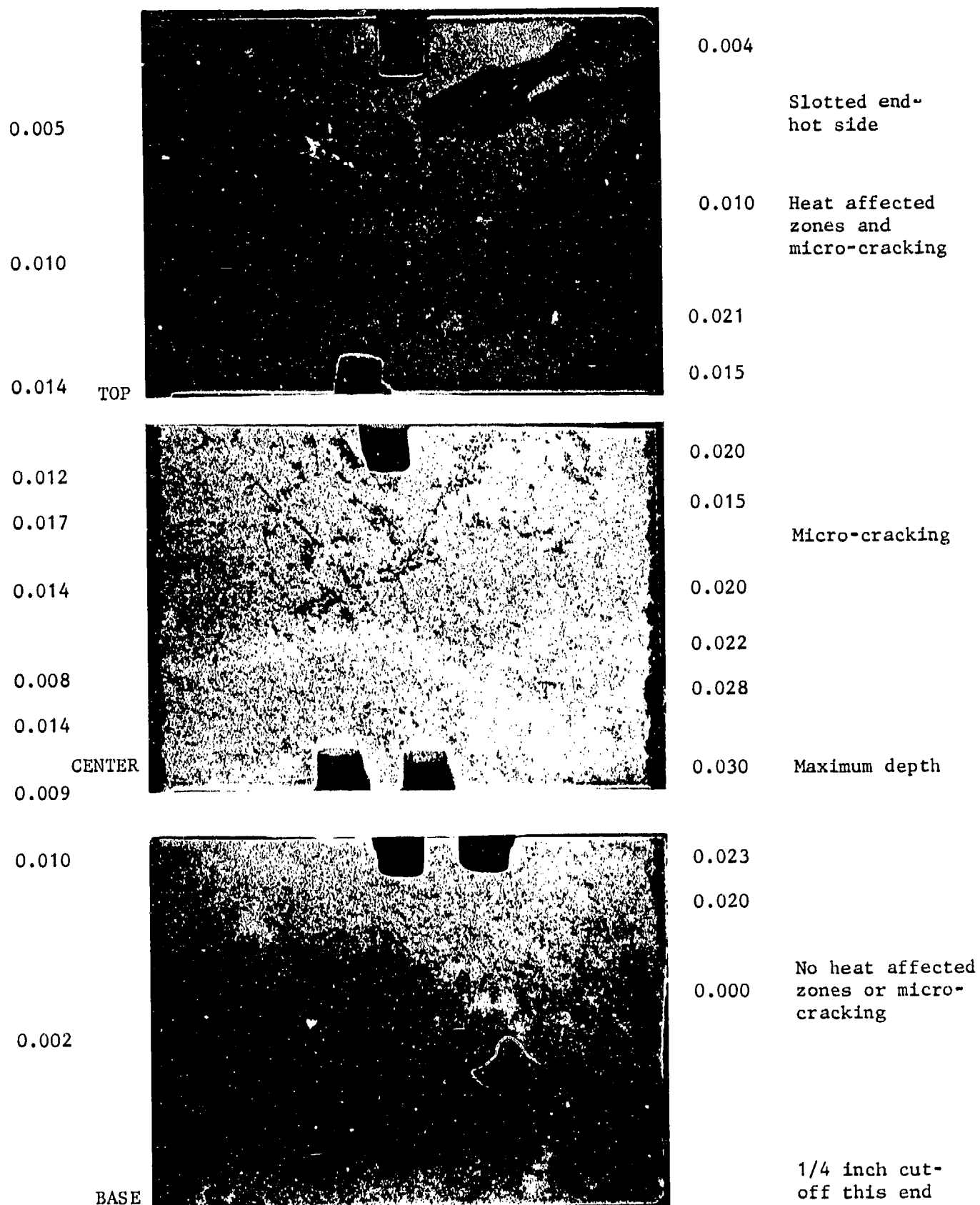


Figure 12 (U). Sectional Tungsten-Copper Test Specimen Showing Surface Depths of Copper Depletion - Mag 5x (See Figure 4)

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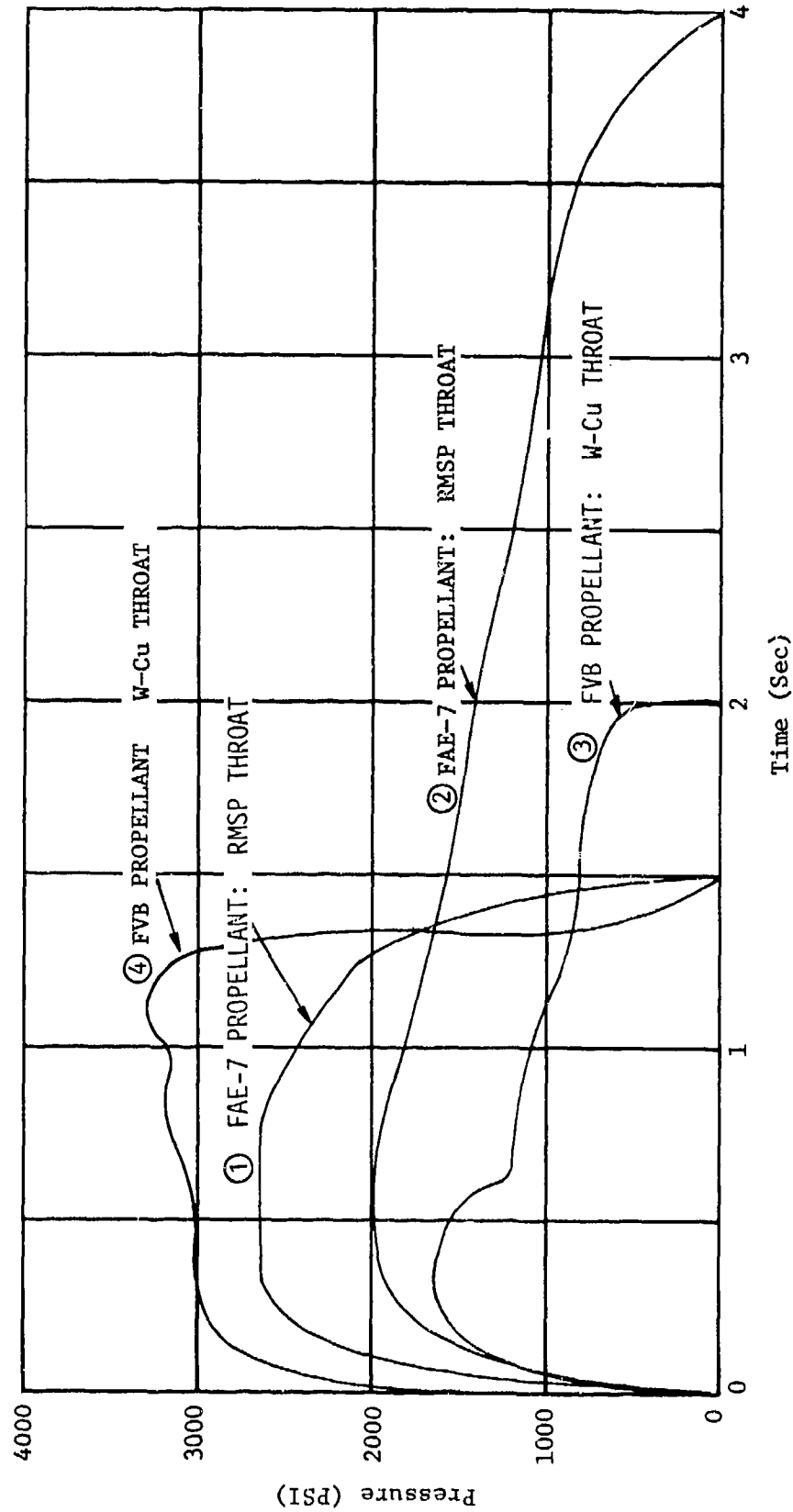


Figure 13 (U). Variation of Chamber Pressure with Time

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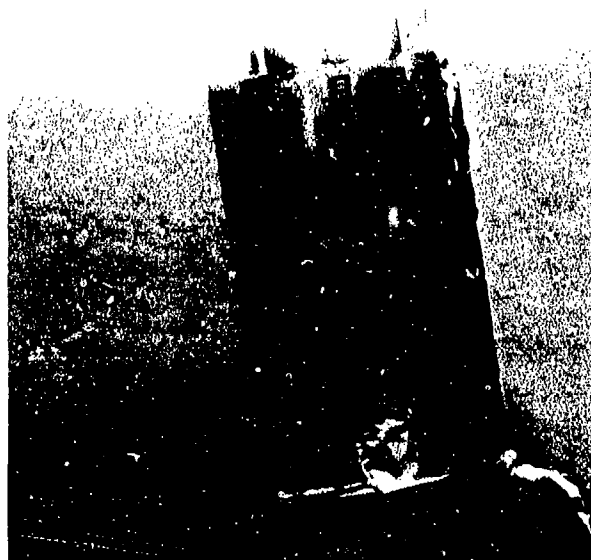
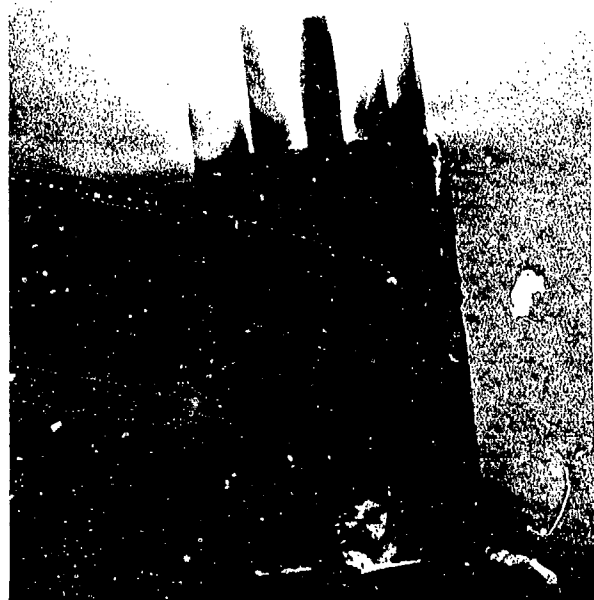


Figure 14 (U). Sequential Views of Test in Progress

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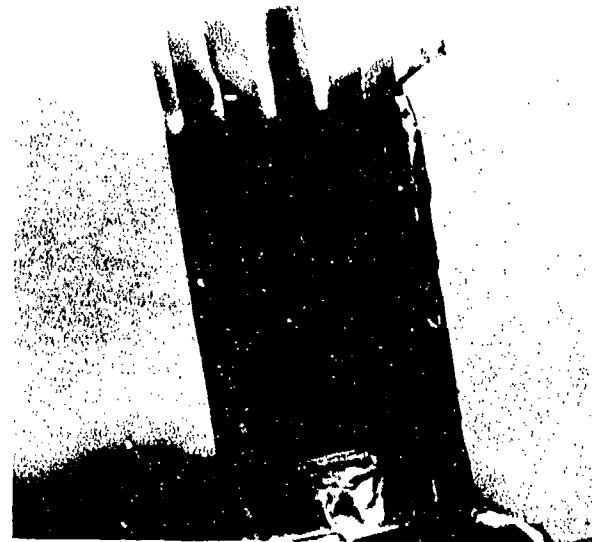
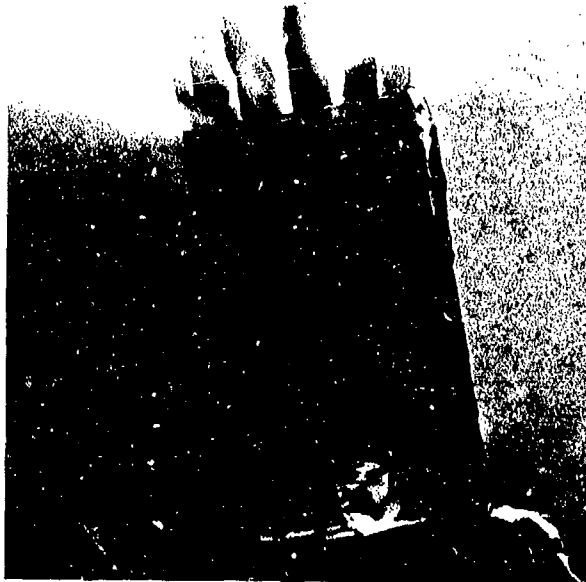


Figure 14 (U). Sequential Views of Test in Progress (cont)

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IV. MATERIALS SELECTION

A. REQUIREMENTS

(U) An analysis was made of the requirements for rotor and nozzle materials. The rotor was found to be critical in tension, with the operating pressure tending to push the two ends of the rotor apart due to the U-shaped configuration of the rotor cavity, the force being reacted in tension by the local rotor cross-section. Considering an operating pressure of 3000 psia and using the applicable areas of the current rotor design, it was found that a minimum tensile strength of 4260 psi was required. The nozzle was found to be critical in shear, with the operating pressure tending to blow out the nozzle, the force being reacted in shear by a ring of the nozzle material. Considering the operating pressure, the area of the nozzle and the shear area of the ring in the current nozzle design, a shear strength of 2600 psi results. The requirements for structure and coatings are summarized below.

(U) A minimum rotor substrate tensile strength of 4260 psi is required while the coating must withstand subsonic particle impact at gas temperatures of 6100 to 6700°F.

(U) For the nozzle, a minimum substrate shear strength of 2600 psi is required while the coating must withstand supersonic turbulent flow of 6100-6700°F gases with solid particle content of 13 to 30 percent.

(U) In the inlet duct liner, a minimum erosion rate must be coupled with a low thermal conductivity when exposed to the parallel subsonic flow of the above described gases at 3000 psia pressure.

(U) Shown in Table III is a strength comparison of the three primary candidate substrate materials which shows that the titanium and the rubber modified silica phenolic are better choices than the speer graphite.

(U) Table IV shows the important physical characteristics of the various candidate materials tested. Fabrication techniques used for each material are discussed below.

B. REFRACTORY MATERIALS

(U) Shown in Table V is a listing of the refractory test specimens used in the four tests. Below is the detailed explanation of the reasoning for selecting these specimens:

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TABLE III (U)
Substrate Strength Comparisons

Nozzle shear strength requirement assuming no design modification	
2600 psi	
Material	Shear Strength
Speer Graphite	100 psi (ultimate)
Rubber Modified Silica Phenolic	1762 (Portashear) 7636 (Punch shear)
Titanium	79,000 ultimate
Rotor tensile strength requirement assuming no design modification	
4260 psi	
Material	Tensile Strength
Titanium	130,000 ultimate at 70°F, 70,000 ultimate at 1000°F
Speer Graphite	1825 with grain 1625 against grain
Rubber Modified Silica Phenolic	11,900 parallel 3600 normal

TABLE V (U)
Refractory Test Specimens

Specimen	Test No.	Remarks
W-Cu	1 2 3 4	Bar Stock
W (0.040) - Speer Graphite	1 2 3 4	Vapor Deposited
W (0.060) - Cb	1 2 3 4	Plasma Sprayed
TaC (0.090) - ZrO ₂ (0.005) - Ti	1 2 3	Plasma Sprayed
Cb	1 2 3	Bar Stock
TaC (0.060) - Cb	1 2	Plasma Sprayed
W (0.060) - Sil Rub - RMSP	2 3 4	Vapor Deposited
W (0.060) - Sil Rub - Ti	2 3 4	Vapor Deposited
TaC - TaC/Mo - POCO	1 3 4	Plasma Sprayed
W-Cu with Calorimeters	3 4	Bar Stock
W (0.020) - RMSP	3 4	Vapor Deposited
W (0.040) - RMSP	3 4	Vapor Deposited
W (0.060) - RMSP	3	Vapor Deposited
POCO Graphite	1	Bar Stock
SiC - Pyrolitic Graphite	1	Sintered Coating
Graphite Phenolic	1	Laminated
W - W/Mo - Ti	1	Plasma Sprayed
TaC (0.002) - Ta (0.040) Be	1 2	Vapor Deposited
TaC (0.002) - Ta (0.040) - RMSP	2	Vapor Deposited
TaC (0.002) - Ta (0.040) - Ti	2	Vapor Deposited
SiC - Cb	2	Sintered Coating
TaC (0.002) - Ta (0.040) Graphite Phenolic	4	Vapor Deposited
Thoriated Tungsten	4	Bar Stock
Tungsten - Hafnium Carbide	4	Bar Stock

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TABLE IV (U)

Candidate Materials Characteristics

Material	Melting Point/°F	Density lb/in ³	Thermal Conductivity Btu/hr/ft ² /ft/°F	Thermal Expansion 10 ⁻⁶ /°F	Specific Heat Btu/lb/°F	Ultimate Tensile Strength (ksi)
Ti-6Al-4V	2920	0.16	RT: 3.9 1000°F: 8.4	RT: 4.8 1000°F: 5.6	RT: 0.135 1000°F: 0.17	RT: 130 1000°F: 70
Ba	2345	0.055	RT: 104 1000°F: 65 2000°F: 32	RT: 6 1000°F: 8.6	RT: 45 1000°F: 0.67 2000°F: 82	RT: 45 1000°F: 28
W-20 percent/Cu	W6170 Cu: 1980	0.62	RT: 150 3000°F: 40	2.55	W: 0.033 Cu: 0.092	RT: 78 3000°F: 14
W-2 percent/ThO ₂	6170	0.69	RT: 80	2.55	0.033	~20 at 3000°F
W	6170	0.69	RT: 80	2.55	0.033	
TaC	7080	0.52	0.053 cal/s/°C/ cm ² /cm (~11)	70-4000°F: 3.7	RT: 0.045 2000°F: 0.080 4000°F: 0.085	E=52x10 ⁶ psi bend RT to 1830: 40-45 3200°F: ~55
POCO Graphite ACF-10Q	Vaporizes	0.064-0.068	50	70-1830°F 4.6		10
Pyrolytic Graphite in plane: plane:	Vaporizes >6600°F	0.079	70 0.6 at 2000°F	0.4 12 at 1000°F	RT: 0.23 2000°F: 0.5	10
SiC-PG Co-deposited 	Vaporizes	(1.40 gm/cc) 0.0506	18.0 50		(0.004 at 200°) (0.013 at 4000°F) (0.012 at 4000°F) (0.004 at 2000	28
Graphite Phenolic 11181058 Bias Tape Ply Ply	No-Melt-Degrades Start ~ 800°F	0.050	0.44		0.2 at 500 0.3 at 200 of	14, (~19)
Silicone DC 93-104	No melt-Degrades Start ~ 700°F	0.052	0.27			300
POCO (POCO)C across	Vaporizes	0.064-0.068	50	4.6	10	10
PG/SiC (ARC)C across	Vaporizes	0.051	18 50	00.4		28
P.G. (U.C.)C across	Vaporizes	0.079	70 0.06	0.4 12	0.23	10
PRD-49-Laminate Epoxy Polyimide Phenolic		0.05				70
POCO-Reimpregn. Rubberized Phen. Silica 1185068	Vaporizes Forms melt					

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1. W-Cb (rotor candidate)

(U) The columbium is used as a structural base to test the erosive resistance of plasma sprayed tungsten. All materials that have a melt phase and can be reduced to a fine powder and sprayed through a nozzle can be plasma sprayed. A high temperature (20,000°F) electric arc melts the powder as it is sprayed under nitrogen pressure out of a gun so that the molten droplets impinge on the work surface forming a porous coating. The process is quite similar to paint spraying; a hand held gun can be used and the part turned to aid in applying a uniform coating. The plasma sprayed coatings used in these tests were all made by Plasmadyne Corporation, 3839 S. Main Street, Santa Ana, California. The W was plasma sprayed to a coating of 0.060 which approximates that required to prevent the interface from exceeding the melting point of columbium (see predicted temperature gradient shown in Figure 21).

2. W-Speer Graphite (nozzle candidate)

(U) The speer graphite has the same coefficient of thermal expansion as W and therefore is well suited to coating by the chemical vapor deposition method. A coating of 0.040 W was selected as adequate to withstand the erosive effect of particle impact while keeping the W temperature in the 5000-5500°F temperature range. (See predicted temperature gradient, Figure 23).

3. W (0.060)-Sil Rub-Ti (rotor candidate)

(U) This combination appeared to be an excellent choice for the rotor using the same logic as above plus the known adequacy of the titanium to meet the rotor strength requirements. It was found that a thickness of 0.01 silicone rubber would provide adequate insulation for the titanium (See Figure 25). The analysis was somewhat conservative indicating the tungsten would slightly exceed its melting point. The vapor deposited tungsten tubes became available in time for the second test and showed good results (See Figure 24).

4. W (0.060)-Sil Rub-RMSP (rotor and nozzle candidate)

(U) This combination appeared to be an excellent choice for the nozzles. Since the rubber modified silica phenolic has very low erosion when the flow is parallel to the surface, if some protection can be provided against localized areas of particle impact, this would prove an excellent low weight combination. The tungsten is fabricated by the vapor deposition method as a separate tube.

(U) The mandrel is induction heated to 1000°C; Ta combines with Cl₂ resulting in a volatile TaCl₅ which is reacted with CH₄ (methane) producing TaC which deposits on the hot specimen and HCl which is drawn off. Deposition can be made successfully directly onto materials which have the same coefficient of thermal expansion such as W onto speer graphite. Shell structure up to 1/8 inch thick can be made by using a stainless

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steel or molybdenum mandrel. The vapor deposited specimens used in these tests were all made by Chemetal Corporation, Los Angeles, California. (W is deposited using a similar process.) For test purposes the tungsten tube was attached to the RMSP base material by using a third coating of silicone rubber because its high coefficient of thermal expansion should provide retention of the tube on the rod during the test. This combination of materials became available in time for the second test. Other thicknesses of W were tested in tests 3 and 4. (See Table V and Figure 26).

5. W-Cu (baseline)

(U) The W is sintered with 20 percent voids and impregnated with copper to make it easily machinable. This is the baseline material used in previous rotor and nozzle designs and was included in all four tests as a baseline comparison of erosion.

6. TaC (0.090)-ZrO₂-Ti (rotor candidate)

(U) Titanium represents a lightweight candidate rotor material with a reasonably high melting point. To provide a protective refractory coating, TaC (melting point 7000°F) was selected as the ideal material. Thermal analysis (see Figure 30) showed that a coating of 0.005 ZrO₂ would be adequate to maintain the surface temperature of the titanium below its melting point. Progressively severe problems with the tantalum carbide cracking (See Figure 29) caused it to be deleted for the fourth test.

7. TaC (0.060)-Cb (rotor candidate)

(U) The columbium is used as a structural base to test the erosive resistance of plasma sprayed tantalum carbide. The TaC was plasma sprayed to a coating of 0.060 to provide a direct comparison with plasma sprayed tungsten. Since the erosion of the TaC proved to be greater than W (compare the results shown in Figures 20 and 31) the TaC coated Cb was dropped after the second test.

8. TaC-TaC/Mo-POCO (nozzle candidate)

(U) The TaC can be plasma sprayed directly onto the POCO graphite but because of the mismatch in thermal expansion coefficient, this is accomplished by a graduated coating with Mo gradually changing from molybdenum against the POCO surface to tantalum carbide. In all cases the TaC coating was eroded or cracked through resulting in erosion to the POCO graphite (See Figures 32, 34, and 35).

9. POCO Graphite

(U) The POCO graphite is a very fine grain graphite developed primarily to obtain isotropic properties. It is made by a proprietary process by Dow Graphite Inc., a subsidiary of Union Oil Company of California, P.O. Box 2121, Decatur, Texas 76234. Because of its performance (See Figure 32) it was used only in the first test.

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10. SiC-PG (candidate for both duct liner and nozzle structure)

(U) The SiC co-deposited with PG forms needle-like crystals which reinforces the PG increasing the 'C' direction strength. The specimens were fabricated by a proprietary process by Atlantic Research Corporation, Alexandria, Virginia. Because of its performance (See Figure 32) it was used only on the first test.

11. Graphite Phenolic (nozzle candidate)

(U) This material is very light weight, easily fabricated, and known to be successful in a parallel flow environment from previous tests. It was reasoned that if the particle impact effects were less severe than anticipated this would be a good candidate for nozzle material. The first test demonstrated that the particle impact environment was excessively severe on this material (See Figure 32) and it was dropped from further testing.

12. Cb (instrumentation)

(U) A solid bar of columbium was used to mount three calorimeters. It was reasoned that the extent of melting and erosion would serve as a data point should the calorimeter measurements fail. This assumption proved to be true and the erosion of the columbium (see Figures 32 and 33) was used for input data points to the analysis shown in section VI.

13. W-W/Mo-Ti (rotor candidate)

(U) This part was made by plasma spraying the W directly onto the Ti using the graduated coating of W/Mo to accommodate the thermal expansion mismatch. It was reasoned that if the thermal analysis was over conservative this combination which should produce a melt in the titanium would identify errors and may prove adequate for the environment. Figure 32 shows that assumption was not valid and because of excessively high erosion this material combination was dropped after the first test.

14. TaC (0.002)-Ta (0.040)-Ti (rotor candidate)

(U) One method of producing a TaC coating is to vapor deposit tantalum and subsequently carburize the surface. TaC/Ta tubes were made by this method and attached to the substructure by the method previously discussed using a third layer of silicone rubber. The combination was dropped after the second test because of the excessive erosion illustrated in Figure 33.

15. TaC (0.002)-Ta (0.040)-RMSP (rotor candidate)

(U) This combination of materials is similar to that discussed above. Again the TaC/Ta combination proved inadequate as shown in Figure 33 and was dropped after only one test.

16. TaC (0.002)-Ta (0.040)-Be (rotor candidate)

(U) The information discussed in the two specimens above applies. (See Figure 33).

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17. SiC-Cb (rotor candidate)

(U) This was a protective coating of silicide applied to columbium which afforded very little protection in this severe erosive environment as illustrated in Figure 33. Only the one test was performed.

18. W-Cu with Calorimeters (for instrumentation)

(U) Because of the lack of success in obtaining good calorimeter data when mounted on Cb (See Figure 33), tests 3 and 4 included calorimeters mounted on W-Cu. In both cases the specimens were blown off the fixture before useable electrically transmitted data could be obtained.

19. TaC (0.002)-Ta (0.040) Graphite Phenolic (nozzle candidate)

(U) This refractory material was discussed in 14. In test No. 4 it was included as a protective sleeve over graphite phenolic for completeness of data. It afforded some protection although the sleeve was removed before the test was over as shown in Figure 35.

20. Thoriated Tungsten and Tungsten - Hafnium Carbide (comparison studies)

(U) These two materials were included in test No. 4 for the purpose of comparing their erosion resistance. No differences were noted between the two materials (See Figure 36).

C. INSULATOR MATERIALS

(U) Insulator materials were evaluated in the first test only. The results of this test showed the rubber modified silica phenolic to be superior and this material was used throughout the entrance duct in tests 2, 3, and 4. See Table VII and Figure 37.

1. Graphite Phenolic (Baseline Duct Liner)

(U) The base material consists of graphite fabric impregnated with a "B" staged phenolic resin containing carbon filler. The resin conforms to MIL-R-9299 Type II Class 2 phenolic resin. The carbon filler concentration can vary from 5 to 14 percent by weight.

(U) This material conforms to U.S. Army Material Command, Redstone Arsenal Material Drawing 11181058 and was cured per Redstone Arsenal Process Drawing 11181023. The impregnated fabrics were cured at 325°F minimum for one hour per 1/4 inch laminate thickness under 1000 psi. The reinforcing fibers were perpendicular or oriented 45 degrees to the center of the molded block.

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2. Rubber Modified Silica Phenolic (Insulator for Duct Liner and Structure for Nozzles)

(U) The base material consists of a high silica woven fabric impregnated with a "B" staged latent catalized novalac phenolic resin copolymerized with an acrylonitrile butadiene rubber. This material conforms to the U.S. Army Materiel Command, Redstone Arsenal Process Drawing 11188301.

(U) The impregnated fabric was molded under 100 psi pressure at 325°F and cured one hour per each 1/4 inch laminate thickness.

3. PRD-49 Molded (Insulator - Duct Liner Candidate)

(U) PRD-49 fabric supplied by DuPont was dried at elevated temperatures. A polyimide resin Kerimid 601, supplied by Rhodia, Inc., was dissolved in N-Methylpyrrolidone (NMP). An approximate 45 to 55 percent solution was made. The dried PRD-49 fabric was coated with the solution and then dried at 300°F for 15 minutes. Laminates were made by stacking the impregnated fabric between aluminum foil and placing the stack in a press at 250°F. A 210 psi pressure was maintained as the temperature was increased to 360°F. The temperature rise rate was one hour per 3/8 inch laminate thickness. The part was cured for one hour at 360°F once final temperature was attained. The laminate was cooled under pressure.

4. Epoxy Kynol (Insulator - Duct Liner Candidate)

(U) Kynol is a trademark of the Carborundum Company for a new generic type of manufactured fiber, for which the Federal Trade Commission has established the generic designation CA-0001. These chemically inert fibers are used as carbon precursor in ablative applications. It has the ability to char with minimal shrinkage. The properties reported by the vendor indicate better insulation properties than those of silica or carbon fiber composites.

(U) These fibers were impregnated with a high temperature resistant epoxy resin formulation. The base resin formulation was Epon 828, Shell Chemical Company, 100 pbw (parts by weight); nadic methyl anhydride, Shell Chemical Company, 90 pbw; and diethylene triamine, Magnolia Plastics, 2 pbw.

(U) The Kynol fibers were thoroughly impregnated with the epoxy resin solution. Parts were made by compression molding under 25 psi pressure, cured one hour at 250°F and four hours at 390°F. The parts were post cured 16 hours at 500°F before final machining.

5. MXA-150 (Insulator - Duct Liner Candidate - Used on Pershing)

(U) The base material is a chopped asbestos roving impregnated with phenolic resin (Specification MPD 10936). The material is described in U.S. Army Redstone Arsenal Material Specification MPD 10936. The process for this material is reviewed in Redstone Arsenal Process ABMA-TD-N-1102.

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(U) The test samples were compression molded under 2000 psi pressure at 210°F for one hour with venting, then the temperature increased to 340°F for four hours. Before machining to the final shape the parts were post cured stepwise from 300°F to 400°F.

6. DC93-104 (Insulator - Duct Liner Candidate - Very Low Cost)

(U) Dow Corning 93-104 ablative material is a two component silicone resin highly filled with inert fibers such as carbon particles. When blended, 109 parts by weight (pbw) base to 10 pbw catalyst the materials remain workable for 8 hours but will fully vulcanize in 24 hours. Parts were cast and cured at room temperature.

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V. EROSION TEST RESULTS

A. SUMMARY OF RESULTS FOR REFRACTORY MATERIALS (FOR ROTOR AND NOZZLES)

(U) Table VI summarizes the results of the refractory materials tests. The various candidate materials are listed with both percent weight loss and erosion rate data shown. It can be seen from the tabular data that the vapor deposited tungsten was a markedly superior coating, particularly with regard to low erosion rates. Figure 15 shows the pre-test condition of the specimens and setup for test No. 1. Although the material combinations were changed for each test, Figure 15 depicts a typical pre-test arrangement. Figures 16 through 19 show the post-test views of tests 1 through 4 in order.

1. Tungsten Plasma Sprayed onto Columbium (for Rotor) (See Section IV, B, 1.)

(U) Figure 20 shows the post test views of the specimens from tests 1 through 4 of the 0.060 inch coating of tungsten (W) plasma sprayed onto columbium (Cb). It will be noted that all the specimens are in excellent condition; the crater in the No. 4 specimen being sharp-edged indicating a part of the coating broke off after the test (when cooled by CO₂ from the fire extinguisher). The columbium provides an excellent heat sink for the tungsten coating as can be seen from the predicted temperature gradient of Figure 21.

2. Tungsten Vapor Deposited onto Speer Graphite (for Nozzles) (See Section IV, B, 2.)

(U) Figure 22 shows the post test views of the specimens from tests 1 through 4 of tungsten (W) chemically vapor deposited onto speer graphite. The speer graphite was selected because of the match in thermal expansion with tungsten. It will be noted that all the specimens are in excellent condition. Surface blisters indicating the start of melting on the surface are apparent on the specimen subjected to the high temperature environment for 3.5 seconds. The graphite provides an excellent heat sink for the tungsten coating as can be seen from the predicted temperature gradient of Figure 23.

3. Tungsten Tube over Titanium (for Rotor) (See Section IV, B, 3.)

(U) Tungsten tubes (3/4 inch outer diameter and 0.060 thick) were made by the chemical vapor deposition method and placed around a titanium rod using silicone rubber (0.010 thick) as an insulator and to provide attachment. Figure 24 shows the post test views of specimens of tests 3 and 4.

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The specimens all appear in good condition although the gases reached the titanium at the base and melted it. The surface blisters indicating the start of melt on the surface of the tungsten is apparent on all three specimens which results from insulating the tungsten. The melt condition is less severe than that predicted by the temperature gradient analysis shown in Figure 25.

4. Tungsten Tube over Rubber Modified Silica Phenolic (for Nozzles) (See Section IV, B, 4.)

(U) Tungsten tubes of various thickness were made by the chemical vapor deposition method and placed around rods of RMSP using silicone rubber for attachment. Figure 26 depicts the post test views of the specimens from tests 2, 3 and 4. Surface melting of the tungsten started as evidenced by the surface blisters shown and as predicted but less severe than indicated by the thermal gradient shown in Figure 27. It will be noted that even in those cases where the tungsten tube was removed, the erosive damage to the RMSP was gradual and not catastrophic.

5. Tungsten-Copper Baseline Material (for Rotor and Nozzles) (See Section IV, B, 5.)

(U) Figure 28 shows post test views of the copper infiltrated tungsten (80 percent W, 20 percent Cu) from tests 1 and 2. Similar specimens were included in tests 3 and 4 but blown off during the high pressure shock of propellant ignition. During the shorter time of test No. 1 the tungsten-copper remained sufficiently cool for the aluminum oxide particles from the propellant gases to condense on the specimen and form a white coating.

6. Tantalum Carbide Coating on Titanium (for Rotor) (See Section IV, B, 6.)

(U) Tantalum carbide, which has a melting point of 7100°F, was plasma sprayed onto titanium using a zirconia coating of 0.005 inch as an insulator. Under these environmental conditions, the thermal shock is too severe for the relatively brittle tantalum carbide as evidenced by the severe cracking shown in the post test views of Figure 29. Figure 30 shows the predicted temperature gradient and show, in particular, the large gradient that exists in the tantalum carbide (TaC).

7. Tantalum Carbide Coating on Cb (for Rotor) (See Section IV, B, 7.)

(U) Tantalum carbide was plasma sprayed directly onto columbium. Post-test views of the specimens are shown in Figure 31. The TaC eroded to the surface of the columbium in test No. 1. The cracks that are apparent were sharp-edged indicating they occurred after the test during cool-down. The more severe erosion of test No. 2 is attributed to the longer test time.

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8. Unsuccessful Materials of Test No. 1 (See Section IV, B, 8 through 13 and 23 and 24).

(U) Figure 32 depicts a number of test specimens which sustained sufficient damage during test No. 1 to be regarded as unsuccessful. The silicon carbide coating for pyrolytic graphite, although withstanding the erosive environment, generated a number of cracks during the test due to thermal shock. The other specimens were severely eroded.

9. Unsuccessful Materials of Test No. 2 (See Section IV, B, 12 and 14 through 17.)

(U) Figure 33 depicts a number of test specimens which sustained sufficient damage during test No. 2 to be regarded as unsuccessful.

10. Unsuccessful Materials of Test No. 3 (See Section IV, B, 4, 6, and 8.)

(U) Figure 34 depicts a number of test specimens which sustained sufficient damage during test No. 3 to be regarded as unsuccessful. It will be noted that the vapor deposited tungsten tubes of various thicknesses protecting the rubber modified silica phenolic were removed. Analysis of the films indicates that these came off at 0.45 to 1.1 seconds of test.

11. Unsuccessful Materials of Test No. 4 (See Section IV, B, 4, 8, and 19.)

(U) Figure 35 depicts three test specimens which sustained sufficient damage during test No. 4 to be regarded as unsuccessful. The eroded area near the lower end of the W-RMSP specimen is attributed to impact of copper particles from the test nozzle throat which was made of copper infiltrated tungsten.

12. Tungsten Alloy Specimens (See Section IV, B, 20.)

(U) Under task I of this contract, reported in Vol. I of OR 12,857, the feasibility of fabricating airvane leading edges of various tungsten alloys is being investigated. As an adjunct to these investigations it was decided to compare the performance of thoriated tungsten with tungsten-hafnium carbide by machining two test samples and installing them on test No. 4. Neither in the case of machining nor in the erosion resistance could any marked difference in these two materials be found as evidenced by Figure 36.

B. SUMMARY OF RESULTS FOR INSULATOR SPECIMENS

(U) Table VII shows a ranking of the insulator specimens with respect to required thickness. A comparison of erosion and insulator qualities clearly indicates the rubber modified silica phenolic to be superior to the other materials tested. (See Figures 4 through 7 and 37).

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TABLE VI (U)

Summary of Test Results for Refractory Materials

Specimen	Density of Substrate Material (lbs/in ³)	Percent Weight Loss*				Max. Erosion Rate (in/sec)				Remarks
		Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 4	
W(.060) - Cb	0.310	0.44	1.17	2.5	0.28	0.004	0.004	0.004	0.017	Plasma Sprayed
W(.040) - Speer Graphite	0.062	1.4	2.0	2.7	6.5	0	0.001	0.003	0.061	Vapor Deposited
W(.060) - Sil. Rub. - Ti	0.160	-	6.1	14.3	11.8	-	0.002	0.001	-	Vapor Deposited Tube
W(.060) - RMSP	0.049	-	5.1	83.0	-	-	0.001	0.165	-	Tube Removed on Test 3
W(.040) - RMSP	0.049	-	-	56.2	13.4	-	-	0.122	0.050	Tube Removed on Test 3
W(.020) - RMSP	0.049	-	-	27.5	46.8	-	-	0.118	0.161	Tube Removed
W - Cu	0.600	0.07	0.90	-	-	0	0.001	-	-	No Test on 3 and 4
TaC(.090) - ZrO ₂ - Ti	0.160	4.7	8.4	71.7	-	0.031	0.025	Frag	-	Cracked
TaC(.060) - Cb	0.310	4.5	9.0	-	-	0.046	0.031	-	-	Cracked
TaC - POCO	0.066	15.0	-	-	-	0.168	-	-	-	Plasma Sprayed
POCO	0.066	29.0	-	-	-	0.192	-	-	-	Broke
Silicide - Pyro Graphite	0.051	70.0	-	-	-	0.077	-	-	-	Broke
Columbium	0.310	10.7	27.6	-	-	0.145	0.086	-	-	Extensive Melting
TaC/Ta - Sil. Rub - RMSP	0.049	-	39.1	-	-	-	0.059	-	-	Excessive Erosion
TaC/Ta - Sil. Rub - Be	0.055	-	45.4	-	-	-	0.125	-	-	Excessive Erosion
TaC/Ta - Sil. Rub - Ti	0.160	-	49.5	-	-	-	0.106	-	-	Excessive Erosion
Silicide - Cb	0.310	-	18.0	-	-	-	0.056	-	-	Coating Lost
TaC - RMSP	0.049	-	-	75.5	-	-	-	0.118	-	Coating Eroded Through
TaC - POCO	0.066	-	-	82.3	83.5	-	-	0.106	0.196	Coating Eroded Through
TaC - ZrO ₂ - Ti	0.160	-	-	71.7	-	-	-	Frag	-	Broken
TaC - Graph. Phen.	0.049	-	-	-	87.6	-	-	-	0.216	Tube Removed

*Measured by weighing each specimen before and after test.

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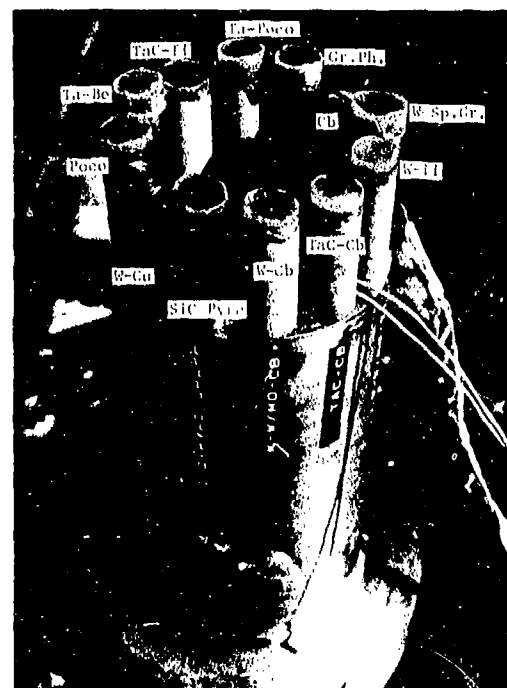


Figure 15 (U). Pre-Test
View of Specimens and Test
Setup-Test No. 1

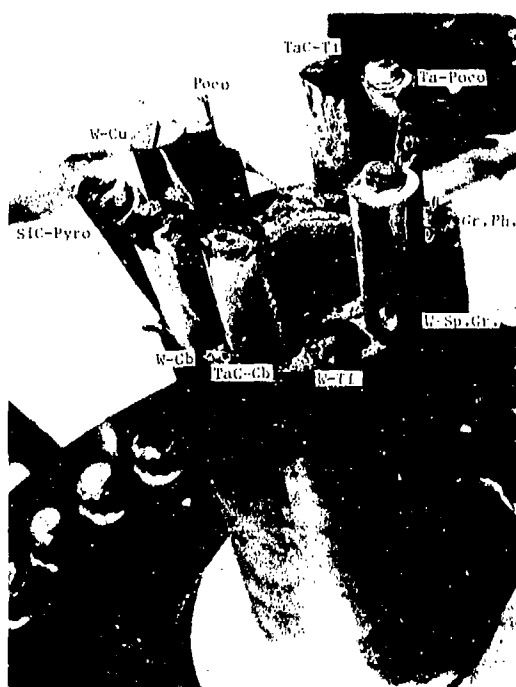


Figure 16 (U). Post-Test
View-Test No. 1

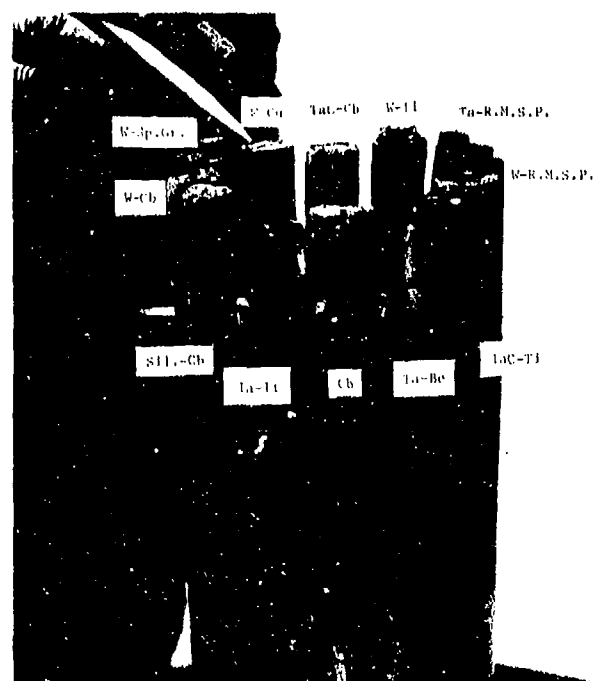


Figure 17 (U). Post-Test
View-Test No. 2

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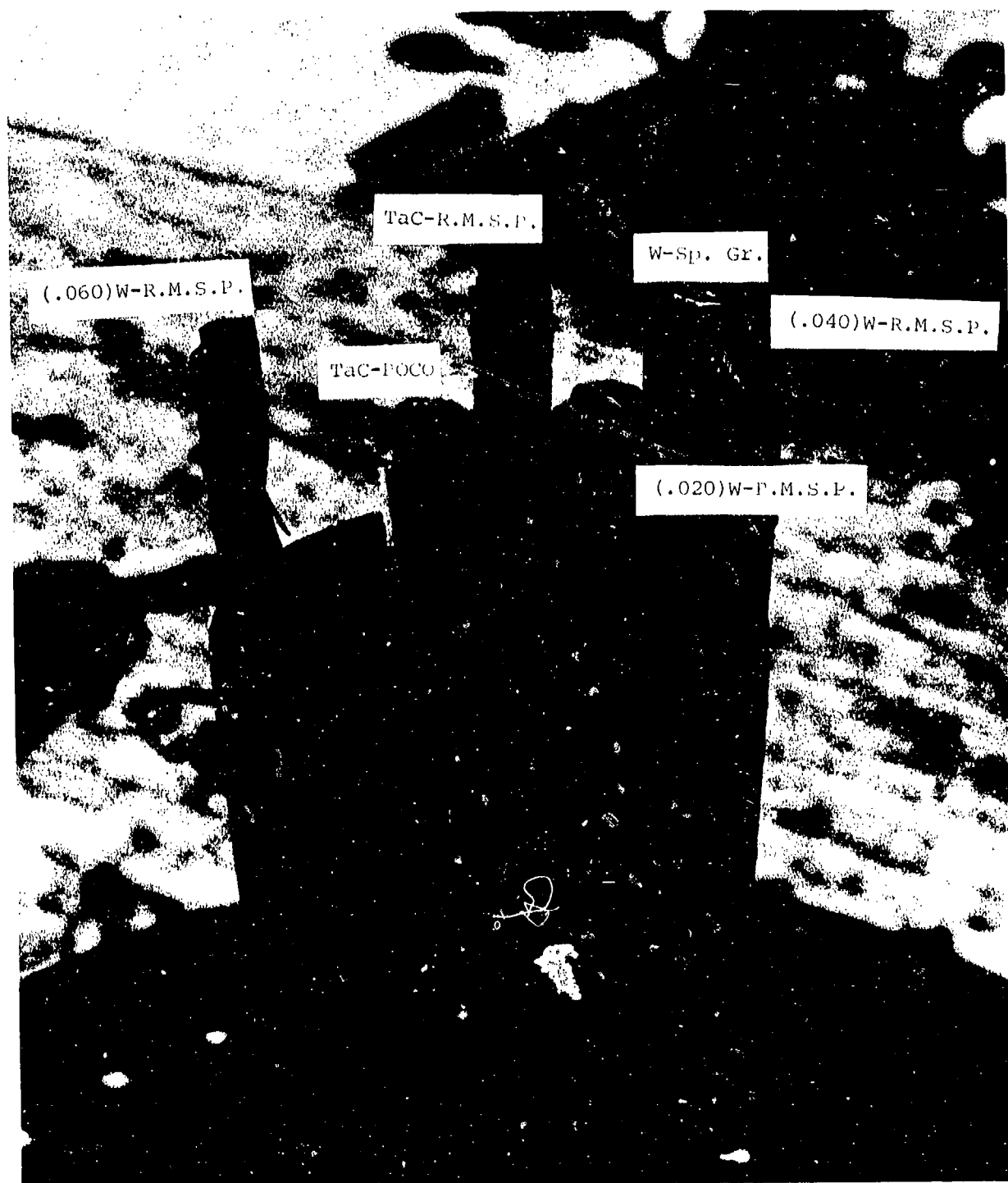


Figure 18 (U). Post-Test - Test No. 3

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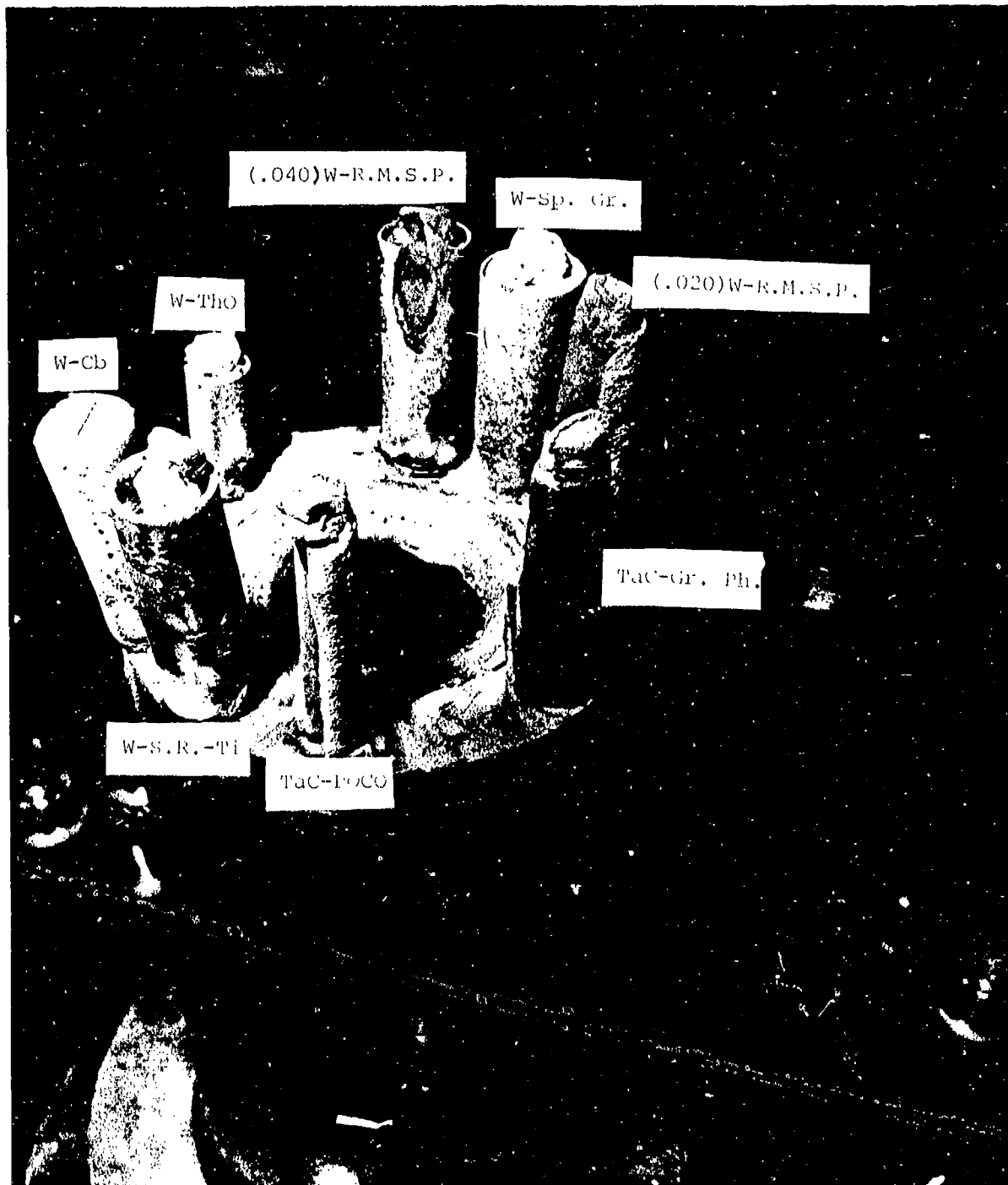
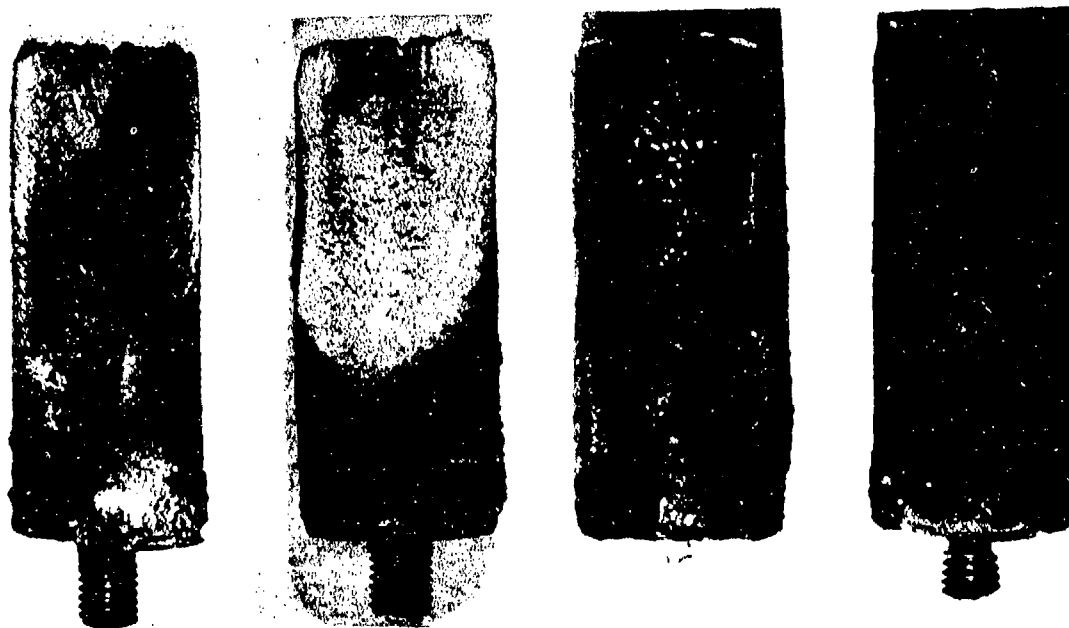


Figure 19 (U). Post-Test View - Test No. 4

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Test No. 1
6100°F; 1.3S

Test No. 2
6100°F; 3.5S

Test No. 3
6700°F; 1.4S

Test No. 4
6700°F; 1.3S

Figure 20 (U). Post-Test Views of W-Cb (0.060 W Plasma Sprayed Onto Columbium)

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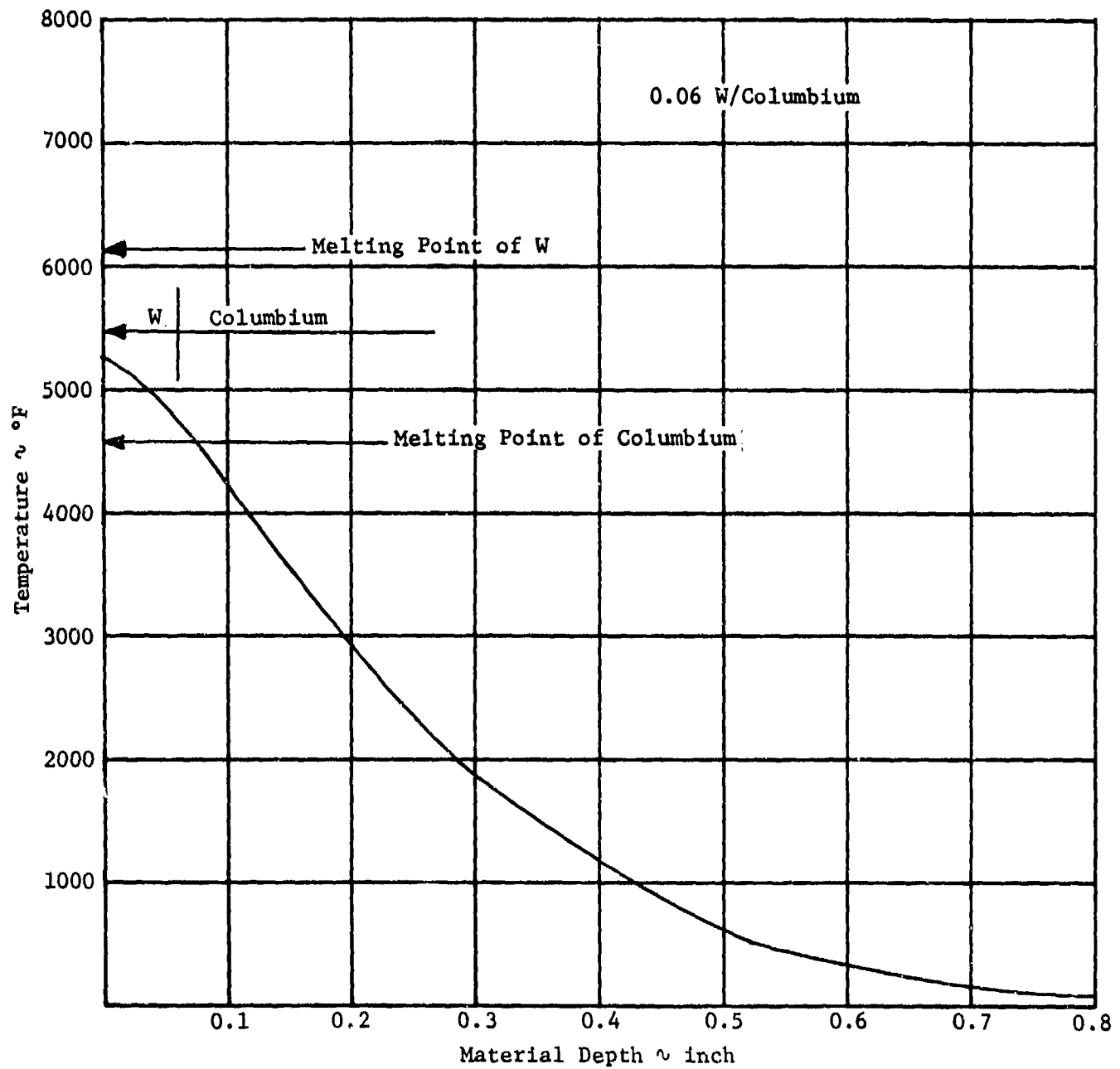
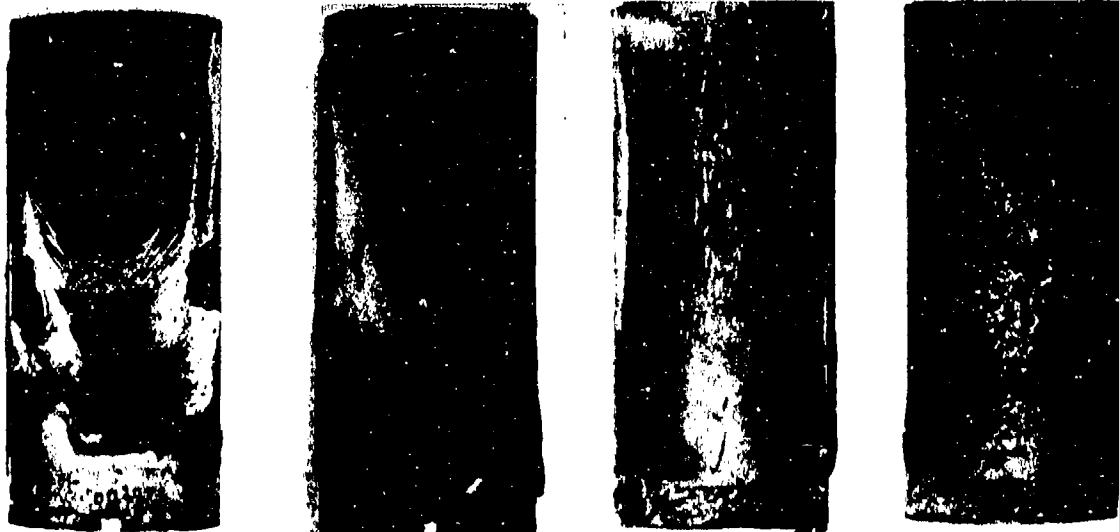


Figure 21 (U). Predicted Temperature Gradient at 1.3 Second Using FVB (6750°F) Propellant (0.06 W/Columbium)

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Test No. 1
6100°F; 1.3S

Test No. 2
6100°F; 3.5S

Test No. 3
6700°F; 1.9S

Test No. 4
6700°F; 1.3S

Figure 22 (U). Post-Test Views of W-Speer Graphite (0.040 W Vapor
Deposited Directly Onto Speer Graphite)

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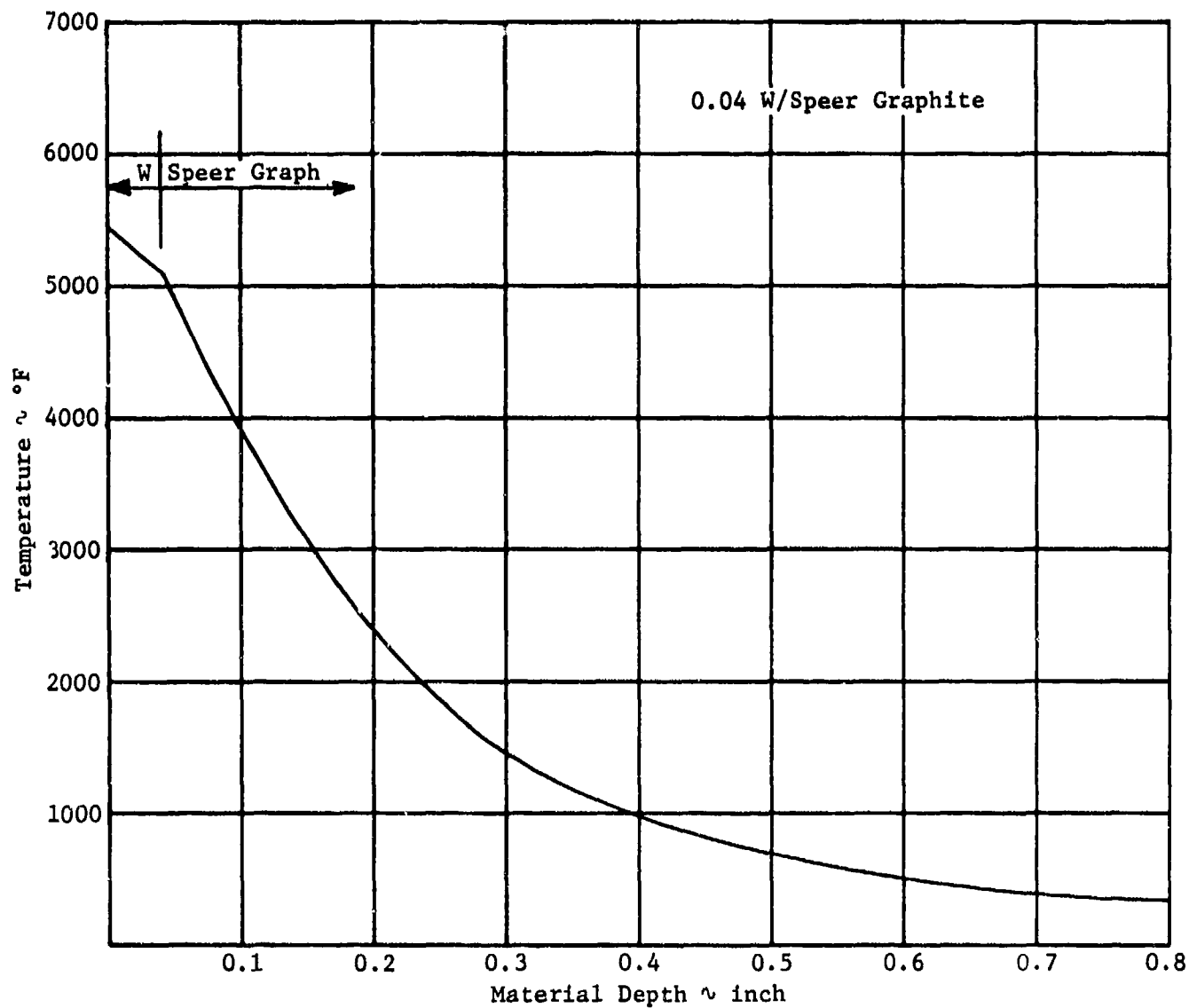
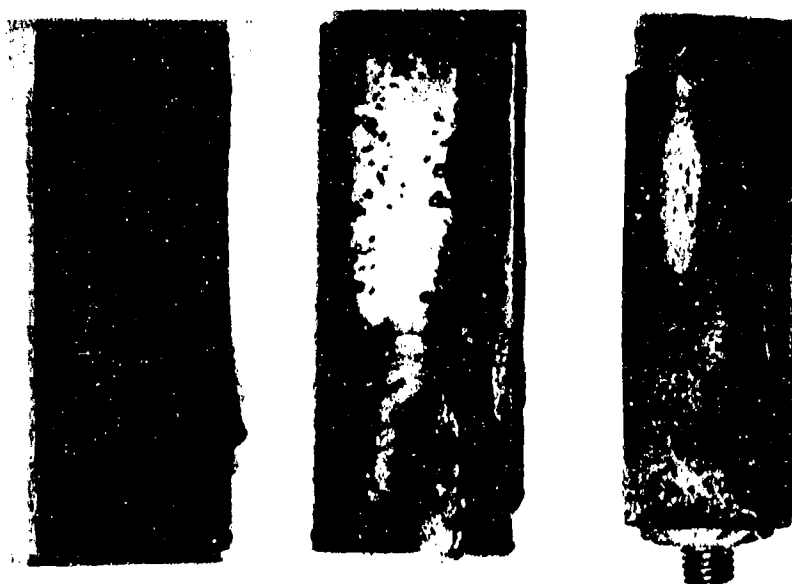


Figure 23 (U). Predicted Temperature Gradient at 1.3 Second Using FVB (6750°F) Propellant (0.04 W/Speer Graphite)

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Test No. 2
6100°F; 3.5S

Test No. 3
6700°F; 1.6S

Test No. 4
6700°F; 1.3S

(0.060 W Vapor Deposited Tube Attached to Titanium Bar
with 0.010 RTV Silicon Rubber)

Figure 24 (U). Post-Test Views of W-Silicone
Rubber-Ti

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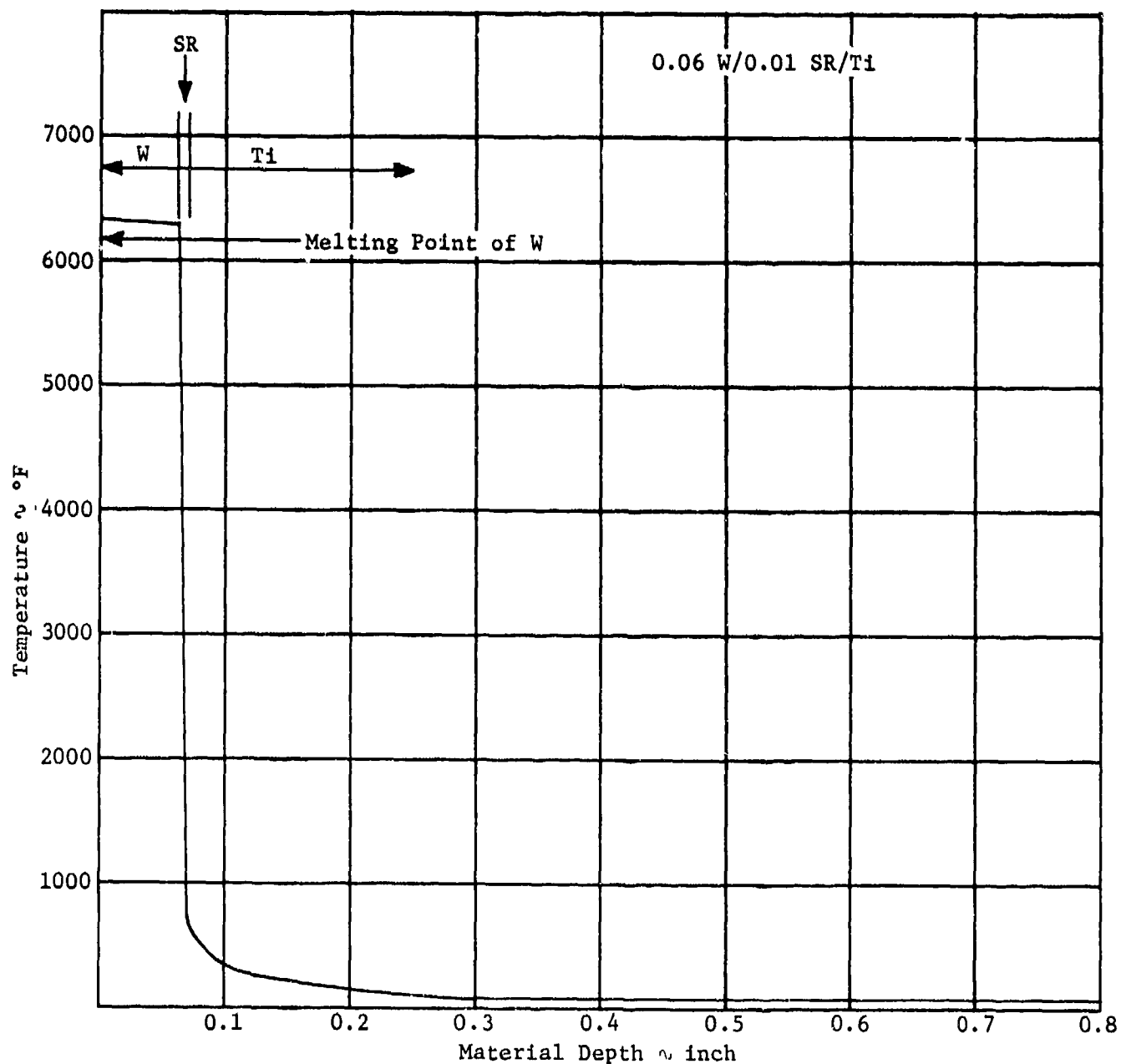
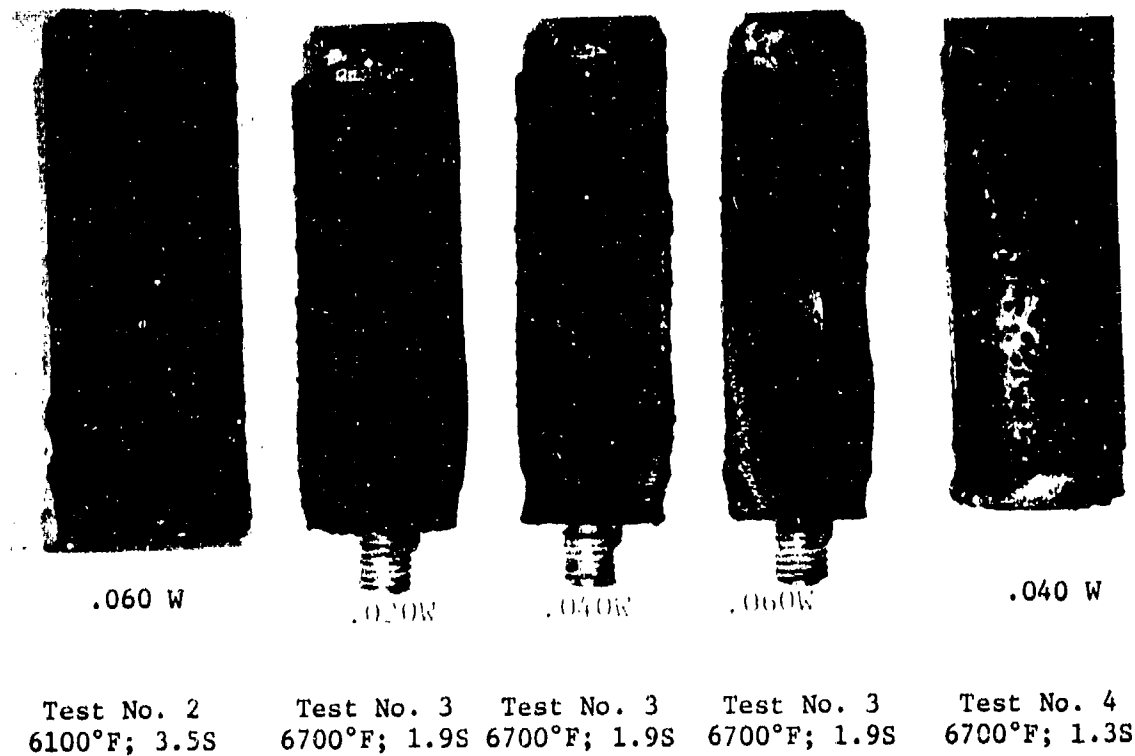


Figure 25 (U). Predicted Temperature Gradient at 1.3 Second Using FVB (6750°F) Propellant (0.06 W/0.01 SR/Ti)

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(W Vapor Deposited Tube Attached to Rubber Modified Silica
Phenolic with 0.010 RTV Silicone Rubber)

Figure 26 (U). Post-Test Views of W-RMSP

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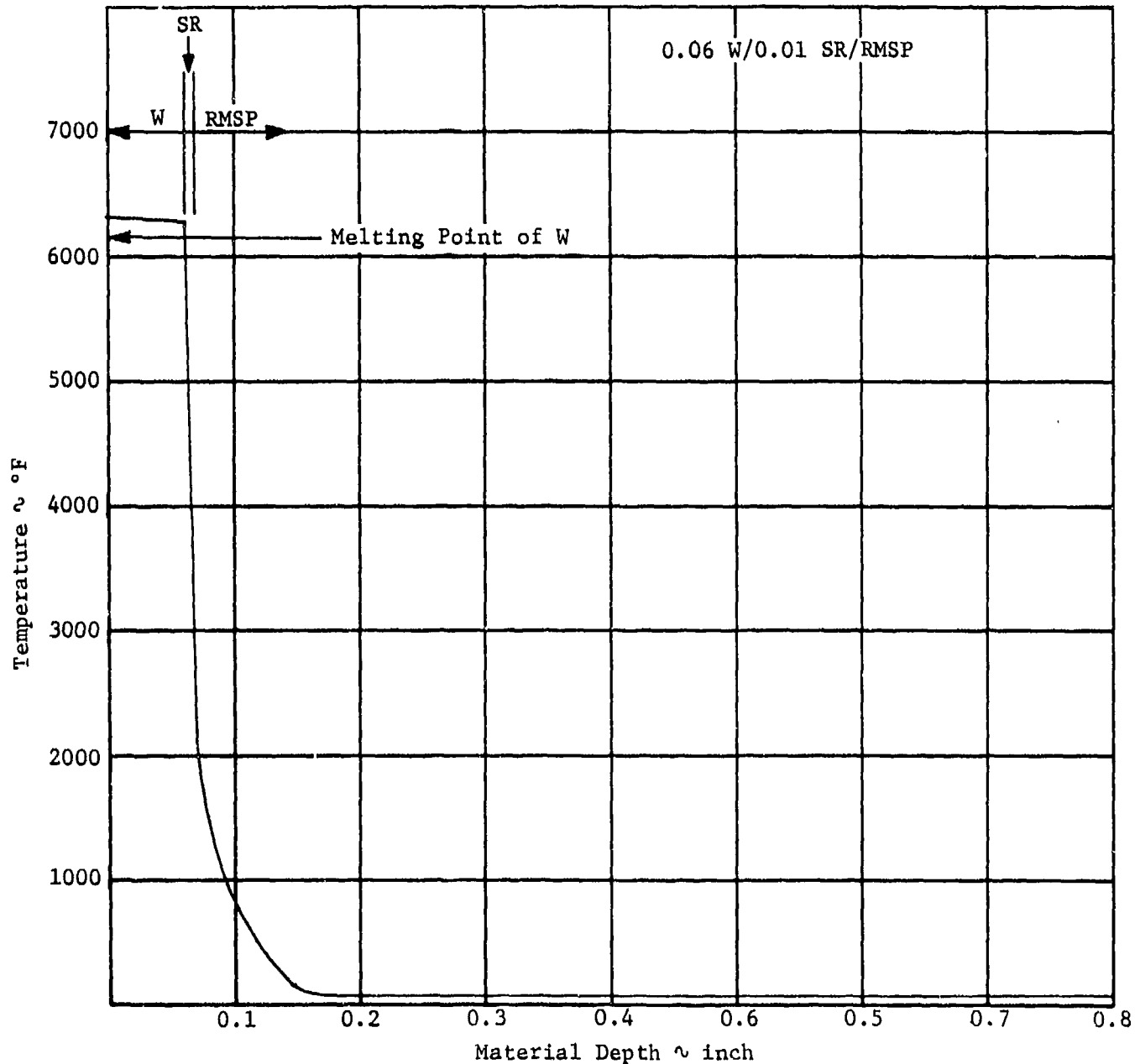


Figure 27 (U). Predicted Temperature Gradient at 1.3 Second Using FVB (6750°F) Propellant (0.06 W/0.01 SR/RMSP)

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Test No. 1
6100°F; 1.3S

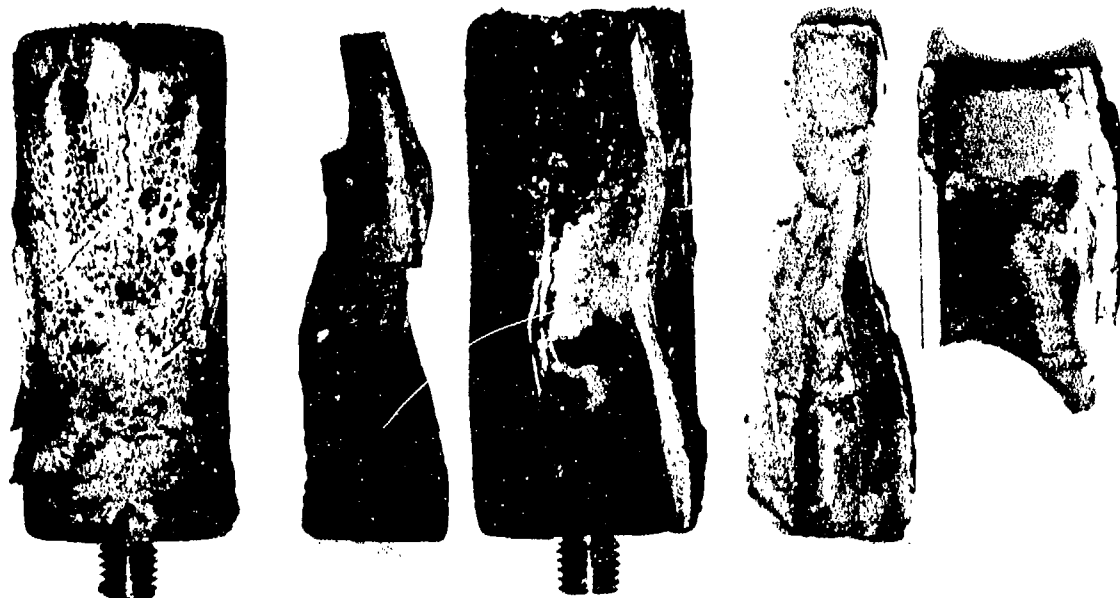


Test No. 2
6100°F; 3.5S

Figure 28 (U). Post-Test Views of
W-Cu (Baseline Refractory Material)

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Test No. 1
6100°F; 1.3S

Test No. 2
6100°F; 3.5S

Test No. 3
6700°F; .75S

(0.070 TaC Plasma Sprayed over 0.005 ZrO₂ on Ti)

Figure 29 (U). Post-Test Views of TaC-ZrO₂-Ti

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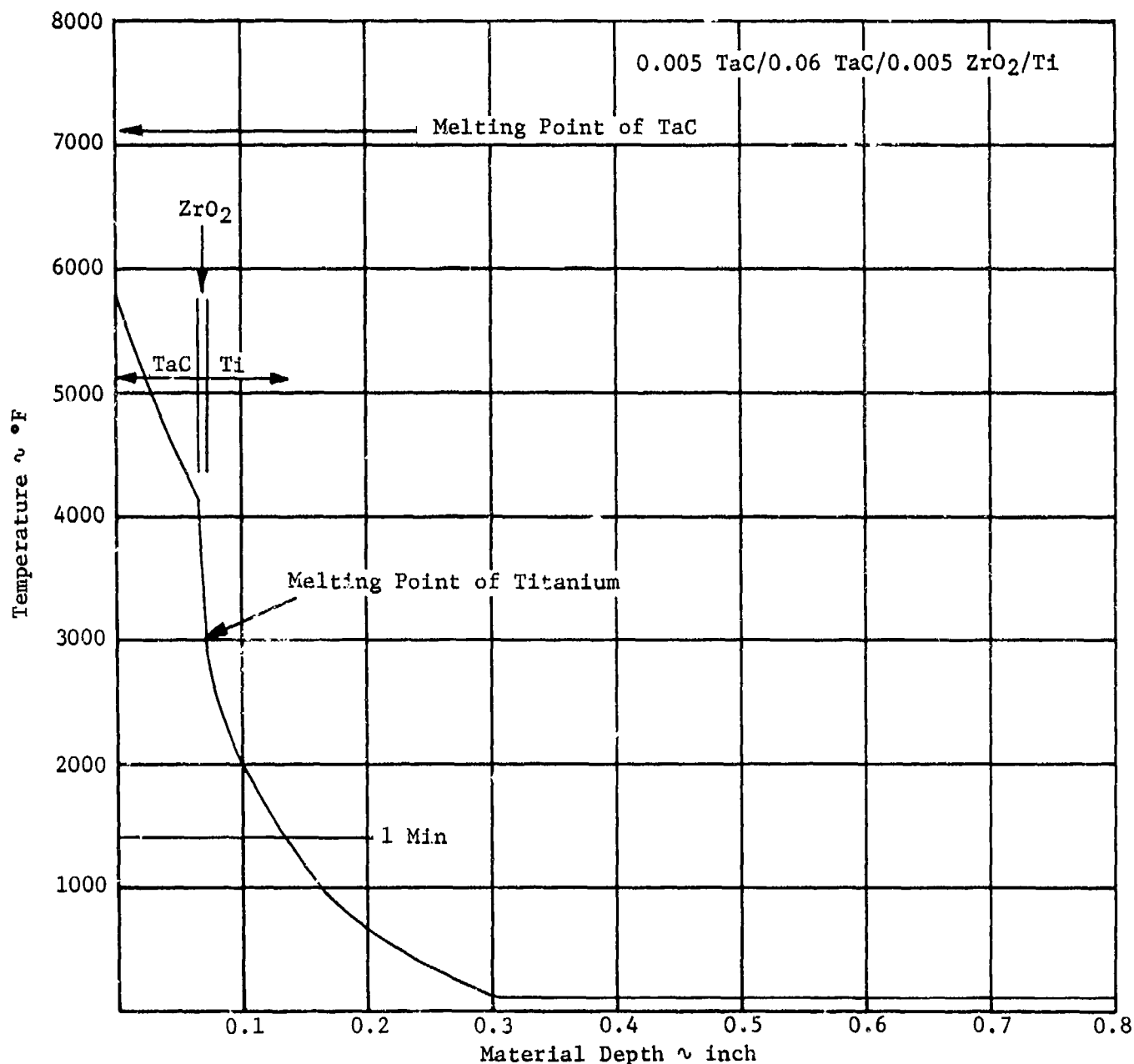
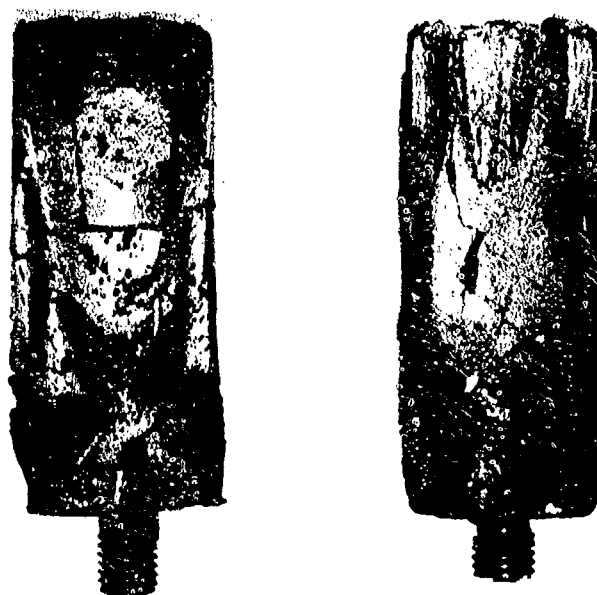


Figure 30 (U). Predicted Temperature Gradient at 1.3 Second Using FVB (6750°F) Propellant (0.005 TaC/0.06 TaC/0.005 ZrO₂/Ti)

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Test No. 1
6100°F; 1.3S

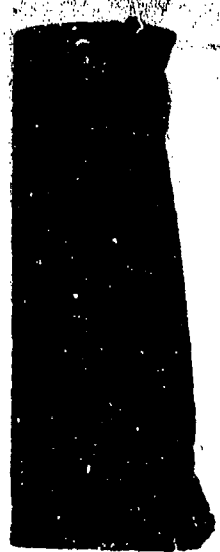
Test No. 2
6100°F; 3.5S

(0.060 TaC Plasma Sprayed Directly onto Cb)

Figure 31 (U). Post-Test Views of TaC

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EAC-TAC/MO-POCO



POCO



SIC-PYRO GRAPH



GRP/THIN



Columbian
CALORIMETERS



W-W/MO-T1

Figure 32 (U). Unsuccessful Specimens of Test No. 1

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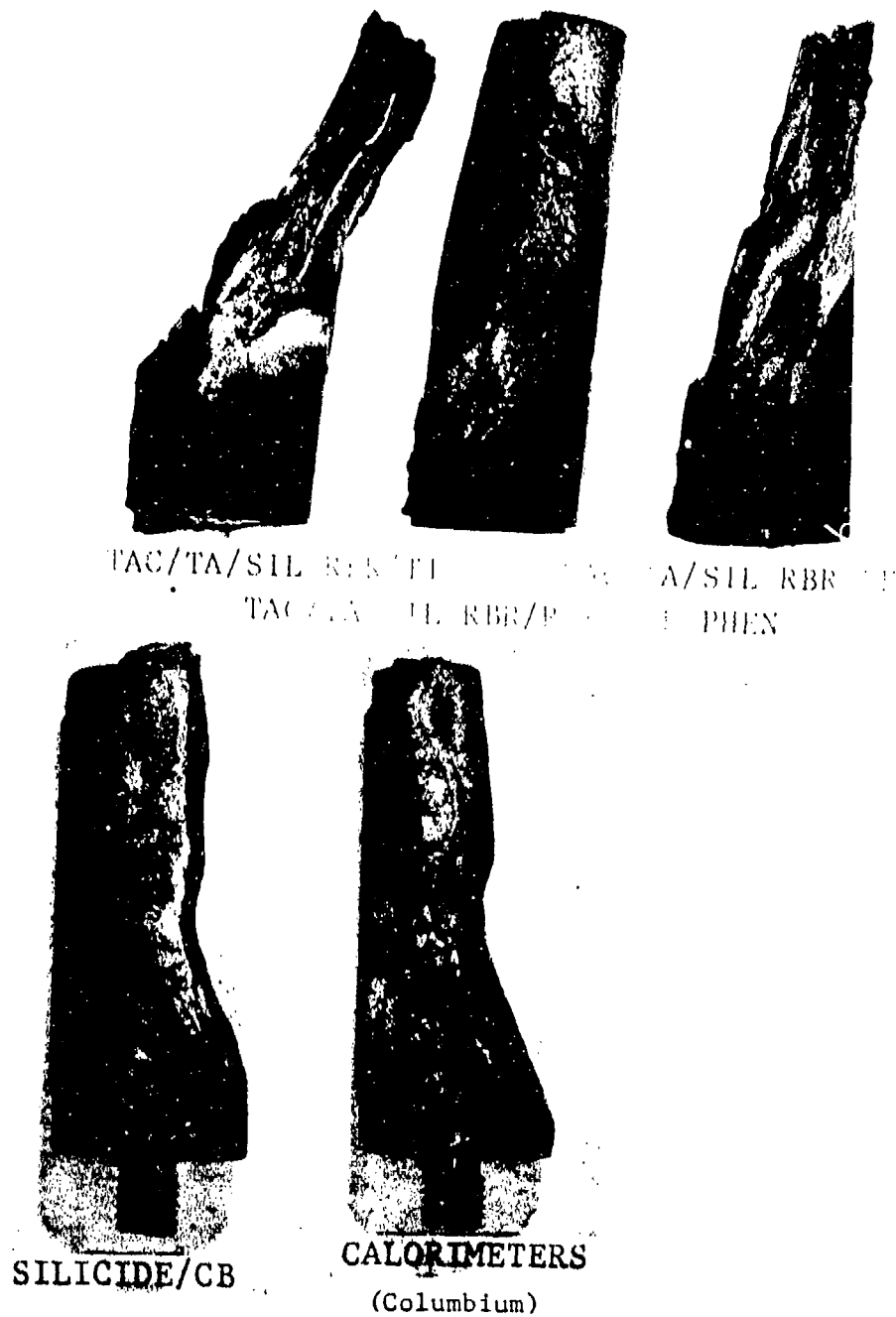


Figure 33 (U). Unsuccessful Specimens of Test No. 2

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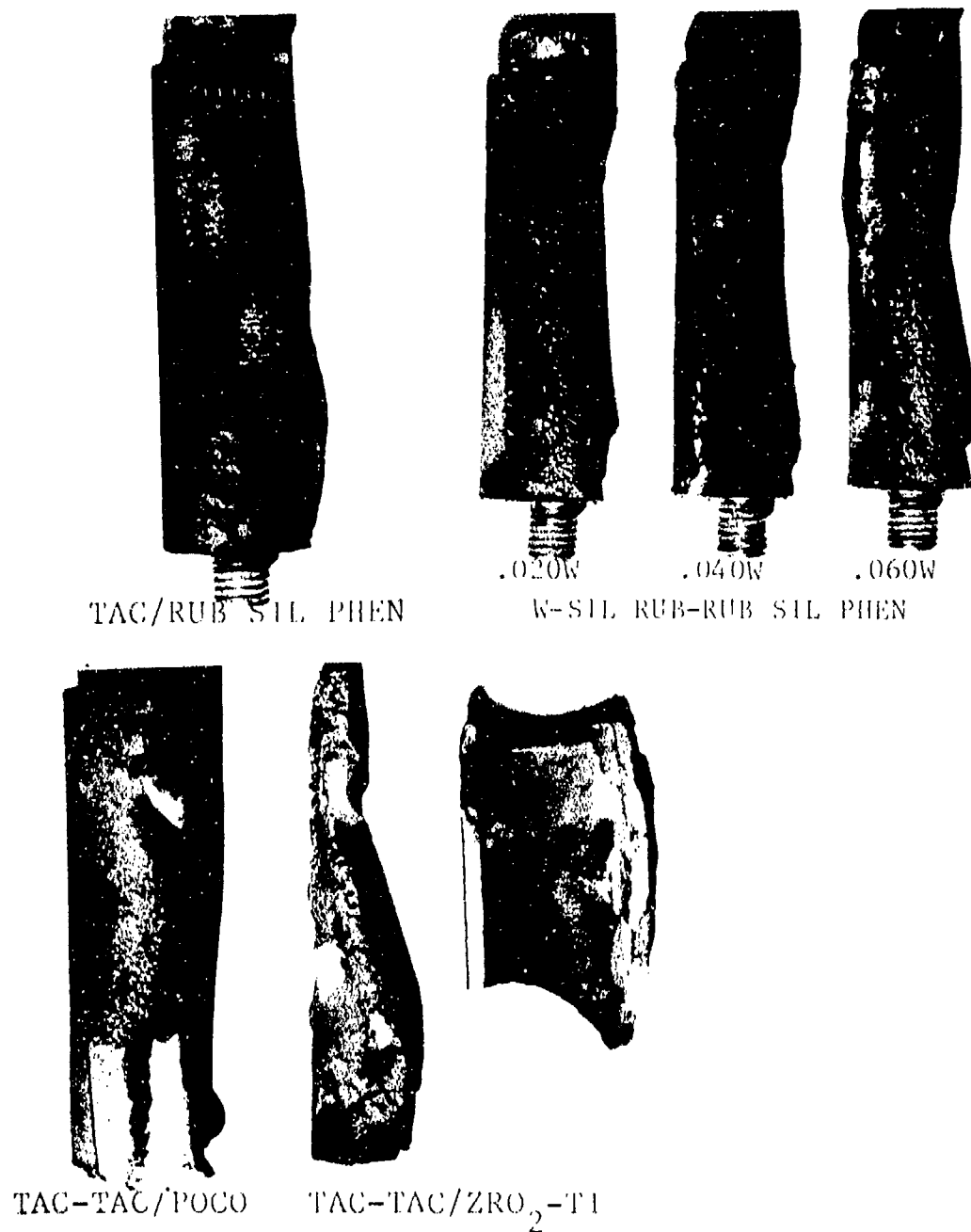


Figure 34 (U). Unsuccessful Specimens of Test No. 3

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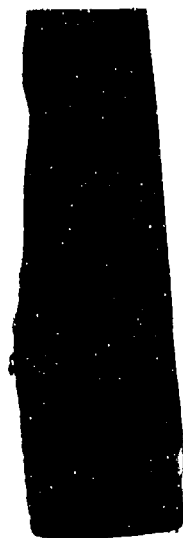
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TaC-TaC/POCO



W(.020)-S11 RUB - RME-P



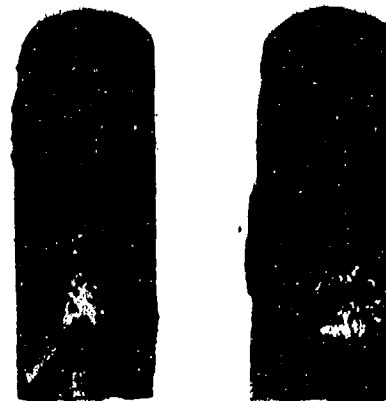
TaC/Ge THEN

Figure 35 (U). Unsuccessful Specimens of Test No. 4

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Figure 36 (U). Tungsten Alloy Specimens



W-ThO₂

W-HfC

TABLE VII (U)

Insulator Materials Test Results

Material	Thermal Conductivity (BTU/Hr/Ft ² /Ft/°F)	Diametral Erosion (Inches)	Required Thickness* (Inches)
Rubber-Modified Silica Phenolic	.135	.025	.073
MXA 150 Stranded Asbestos	.289	.055	.159
Graphite Phenolic (End Grain)	.440	.025	.212
Graphite Phenolic (45°)	.440	.035	.218
Epoxy Kynol (Molded)	.500	.045	.250
PRD-49 - Polyimide	.100	.780	.435
DC 93-104 Silicon Rubber	.270	.10+	.622+
SiC - Pyro Graphite	4.000	0.000	1.820
Poco Graphite	70.000	.005	31.800

* Design required thickness is based on the empirical expression $t = .455 (\text{thermal conductivity}) + .5 (\text{diametral erosion})$ which provided minimal backface temperature rise when the baseline material (graphite phenolic - end grain) was used.

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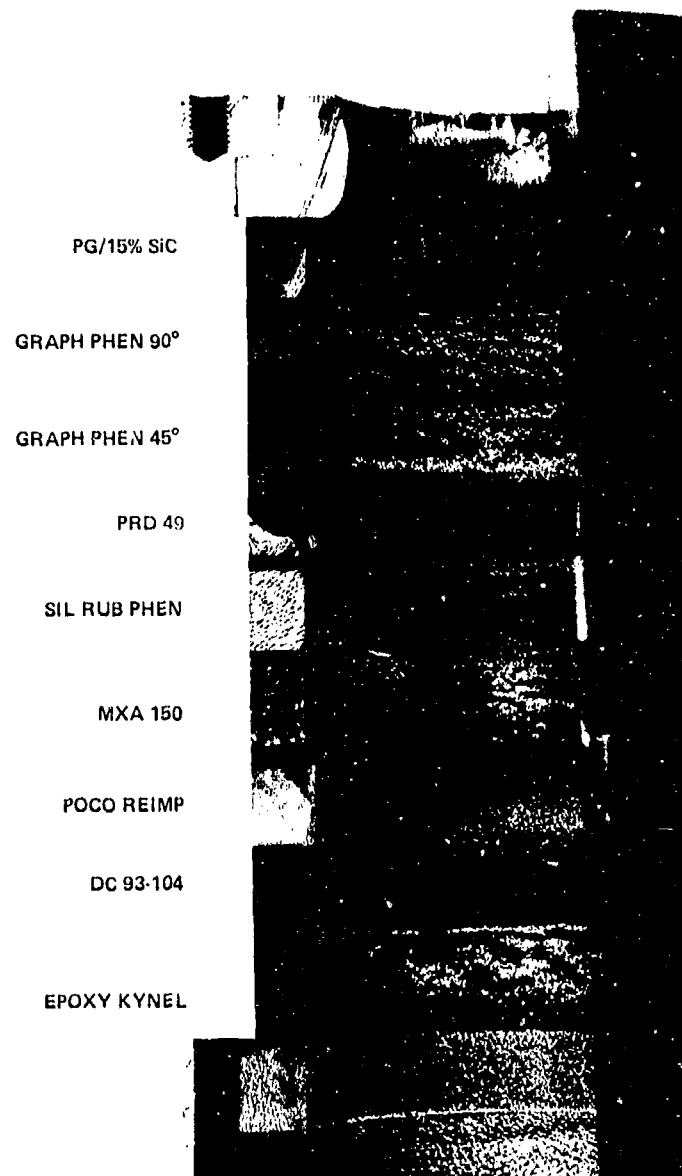


Figure 37 (U). Post-Test View of
Insulator Specimens (Sectioned)

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VI. ANALYSIS

1. General

(U) A thermal analysis was performed in conjunction with the hot gas valve materials selection tests. The purpose of the analysis was to define the thermal environment of the candidate materials for the four hot gas test conditions, and subsequently determine the response of the materials to the imposed thermal environment. Comparisons were made between the thermal environment for the four test conditions and that environment expected in an advanced interceptor control system.

(U) The propellant for tests nos. 1 and 2 was FAE-7 which produces a 6100°F gas with 13.6 percent aluminum oxide content, while the propellant for tests nos. 3 and 4 produces a 6750°F gas with a combined 29.7 percent zirconium oxide and aluminum oxide particle content. The chamber pressure-time history of each test is shown in Figure 13.

2. Analytical Techniques

(U) An analytical investigation prior to testing was necessary in order to size the candidate materials with regards to specified design temperatures for the predicted thermal environments. A semi-empirical analysis consisting of a turbulent swept cylinder convective heating model and a particle impingement heating technique were made for the stagnation line region of the refractory specimens. Analytical relationships for turbulent heating in ducts predicted the thermal environment of the inlet duct materials.

(U) Local flow conditions were calculated using a one-dimensional isentropic flow from the chamber conditions, and expansion angles from experimental data. The propellant mass flow rate was calculated from the equation

$$\dot{m} = \frac{\pi R^2 P_c g}{C^*} \text{ where}$$

- R = nozzle throat radius
- P_c = chamber pressure
- C* = effective exhaust velocity
- g = gravitational constant

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The aluminum oxide particle mass flow rate was calculated as 13.6 percent or 29.7 percent of the total mass flow rate for tests 1 and 2 and tests 3 and 4, respectively. No attempt was made to define a particle size distribution and particle velocity distribution in the exhaust plume since the accuracy of the predictions would not warrant it.

(U) The gas heat transfer coefficient was calculated for the local flow conditions using the Beckwith-Gallagher swept cylinder stagnation line heating model for supersonic flow. The particle heat transfer coefficient was calculated assuming that the heat transferred to the specimen by the particle was proportional to the sum of the particle kinetic energy and internal (heat) energy, the proportionality constant being the accommodation factor. This accommodation factor was empirically determined from existing data and the hot gas tests. From previous TVC valve tests, it was concluded that the maximum rotor heating condition exists only until a layer of molten aluminum oxide particles form on the rotor surface after which the effective heat transfer coefficient drops to approximately 4 percent of the maximum rotor stagnation heat transfer coefficient. This melt layer formed within the first 0.2 seconds of a 2.5 second test. (see Table 6.4-I).

(U) The candidate materials were sized by calculating temperature gradients in the materials for the predicted heating rates in the hot gas tests. The gradients were calculated using a one-dimensional, transient, variable properties heat transfer computer program, Martin Marietta Program FO 086. These sizing data and temperature gradients are shown in Figures 21, 23, 25, 27 and 30.

3. Experimental Techniques

(U) Three copper calorimeters were mounted in a columbium specimen rod and the instrumentation amplifier sensitivity to open loop shielding was reduced to make it insensitive to electrical grounding of the calorimeter. In tests 1 and 2, the calorimeter data became very noisy at ignition. In tests 3 and 4 the calorimeter rod holders were blown out at ignition. Apparently, the dynamic environment of propellant ignition is causing mechanical failures in the calorimeter assembly. The calorimeter data was not useable in any of the tests. However, for tests 1 and 2, the melt recession of the columbium rods was used to calculate the heating environment. For tests 3 and 4, the tungsten/columbium rods were used to calculate the heating environment. It was found in tests 3 and 4 that the tungsten surface did not melt but the columbium had a melt depth of .004 inches. These data provided a \pm time average (for burn time) heat transfer coefficients at the particle impingement and gas impingement points on the specimens.

(U) Motion pictures were analyzed to determine plume expansion angles, specimen failure times and specimen failure modes. These data were used to correlate test heating environments. Motion pictures were made at 1800 frames/s, 500 frames/s, and at real time.

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(U) Visual microscopic investigations of the specimens were made after testing to determine erosion and melting areas and trends, particle impingement angles and copper (from copper/tungsten throats in tests 1 and 4) or aluminum oxide build up on the test specimens.

4. Thermal Analysis Results

(U) Throughout this discussion heat transfer coefficients were used as the primary basis for comparison of thermal environments rather than the usual surface heating rates. This is done because the material surface temperatures involved are relatively near to the environment temperature and, as the surface heating rates are directly proportional to the temperature difference between the surface and the environment, small changes in surface temperature can produce large changes in heating rates. Heat transfer coefficients are much less sensitive to changes in surface temperature and are better suited for purposes of comparison in this analysis.

(U) The thermal environment of the rotor impact surface and nozzle liner surface expected in an advanced interceptor control system have previously been determined in a hot gas test using the FAE-7 propellant (13.6 percent aluminum oxide). The results of the previous rotor impact face tests performed for ABMBA and tests nos. 1 and 2 of this program are compared in Table VIII.

TABLE VIII (U)

Heating Environment Comparison

FAE-7 Propellant	
	Heat Transfer Coefficient (BTU/ft ² -s°F)
Rotor impact face for first 0.2 seconds =	42.0
Rotor impact face for remaining test time =	1.7
Nozzle inner surface =	0.9
Test No. 1 (22 Feb 1973) =	3.3
Test No. 2 (16 Mar 1973) =	1.1

(U) Due to the test specimen configuration of the current tests the plume flow angles were such that the specimens were not covered with an aluminum oxide surface melt layer and thus the results of these tests are considered to be representative of expected performance of the candidate materials under rotor heating conditions.

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(U) Nozzle heat transfer coefficients from previous TVC valve tests were $0.9 \text{ BTU/ft}^2\text{-s-}^\circ\text{F}$. Materials which performed well in this test will be more than adequate for nozzle applications.

(U) The thermal environment of the rotor impact face and the nozzle liner surface expected in an advanced control system using an advanced propellant, which greatly increases the aluminum oxide content, has not been empirically determined. However, this environment can be predicted based upon the results of the FAE-7 propellant tests and an analytical model with certain assumptions.

(U) The rotor impact face maximum heat transfer coefficient results are within 4 percent of the analytical results for particle heating by assuming an accommodation coefficient of one. Therefore, using this model, the maximum heat transfer coefficient on the rotor impact face (before aluminum oxide build-up) for the advanced propellant is calculated to be $75 \text{ BTU/ft}^2\text{-s-}^\circ\text{F}$. This increase in the maximum heat transfer coefficient is due to the increase in the mass flow rate of the particles. After the layer of molten aluminum oxide particles forms on the rotor surface, it is assumed that the heat transfer coefficient does not increase with increasing particle content. The addition of aluminum particles is assumed to only make the molten layer thicker and transfer the additional energy in maintaining the thicker molten layer.

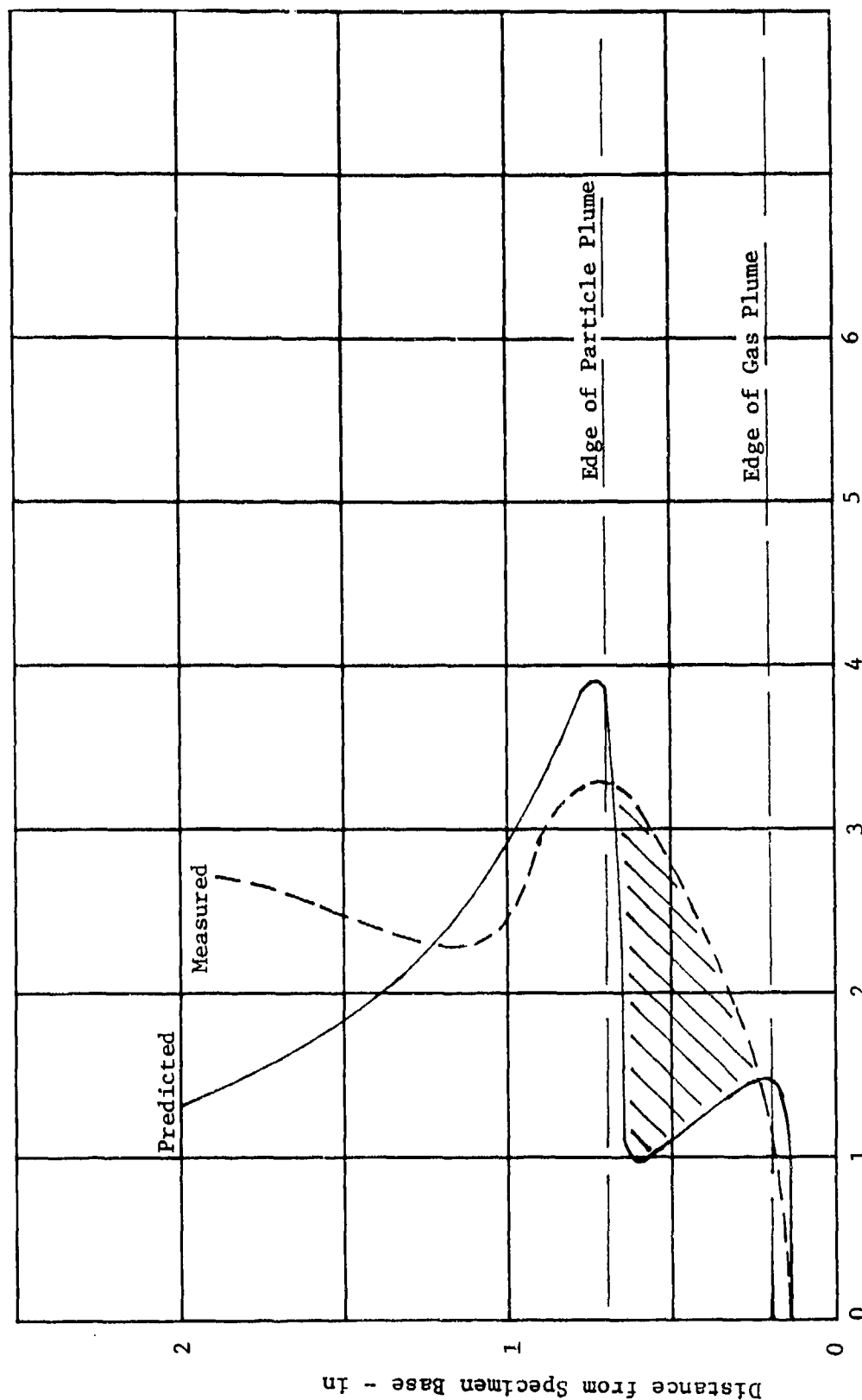
(U) Analytical relationships for turbulent heating in ducts predict an internal heat transfer coefficient of $0.8 \text{ BTU/ft}^2\text{-s-}^\circ\text{F}$. This high heat transfer coefficient is due primarily to the high pressures and the corresponding high gas densities within the tube and not to particle impingement heating. Therefore, it is assumed that the heat transfer coefficient in the nozzle is not appreciably affected by the addition of aluminum oxide particles in the propellant.

(U) The heat transfer coefficients for the rotor impact face and nozzle inner surface as calculated by the above methods for the advanced propellant are compared to the heat transfer coefficients resulting from tests 3 and 4 in Table IX.

(U) The gas and the particle heat transfer coefficients are added together and compared in Figure 38 through 41 to an apparent heat transfer coefficient based on the measured recession of the columbium calorimeter sample, for tests 1 and 2 and the melting depth of columbium in the W/Cb specimens from tests 3 and 4. These measured time average heat transfer coefficients as a function of height on the specimen were calculated only at the gas plume and particle plume impingement points. The curves represent trends of erosion and melt as observed by microscopic examination of the specimens. Considering that the actual flow field phenomena in the test set-up is quite complex, the agreement between the simplified analysis and test data is good.

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Average Heat Transfer Coefficient - BTU/Ft² Sec °R

Figure 38 (U). Comparison of Analytical Model with Test Data - Test No. 1

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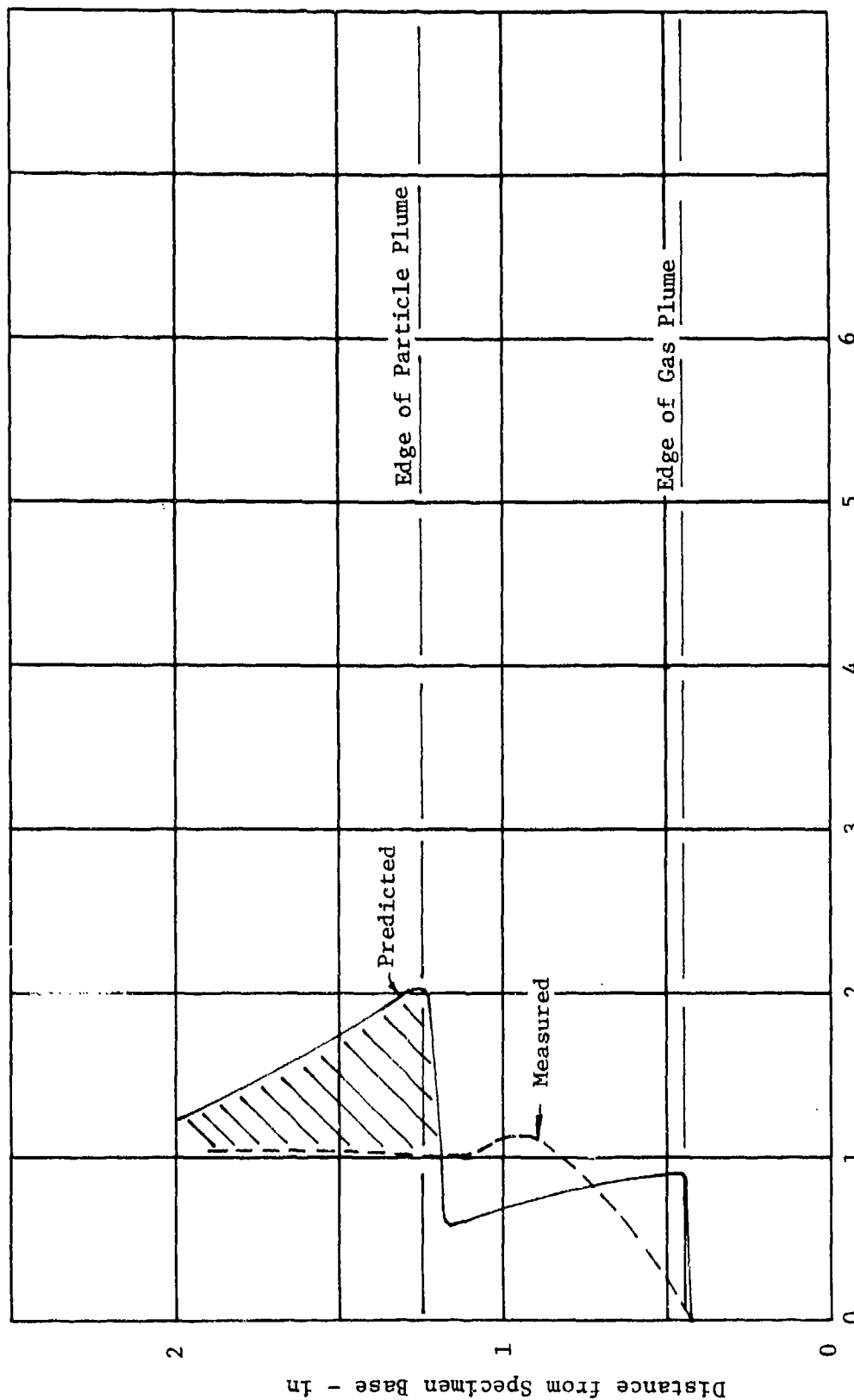


Figure 39 (U). Comparison of Analytical Model with Test Data - Test No. 2

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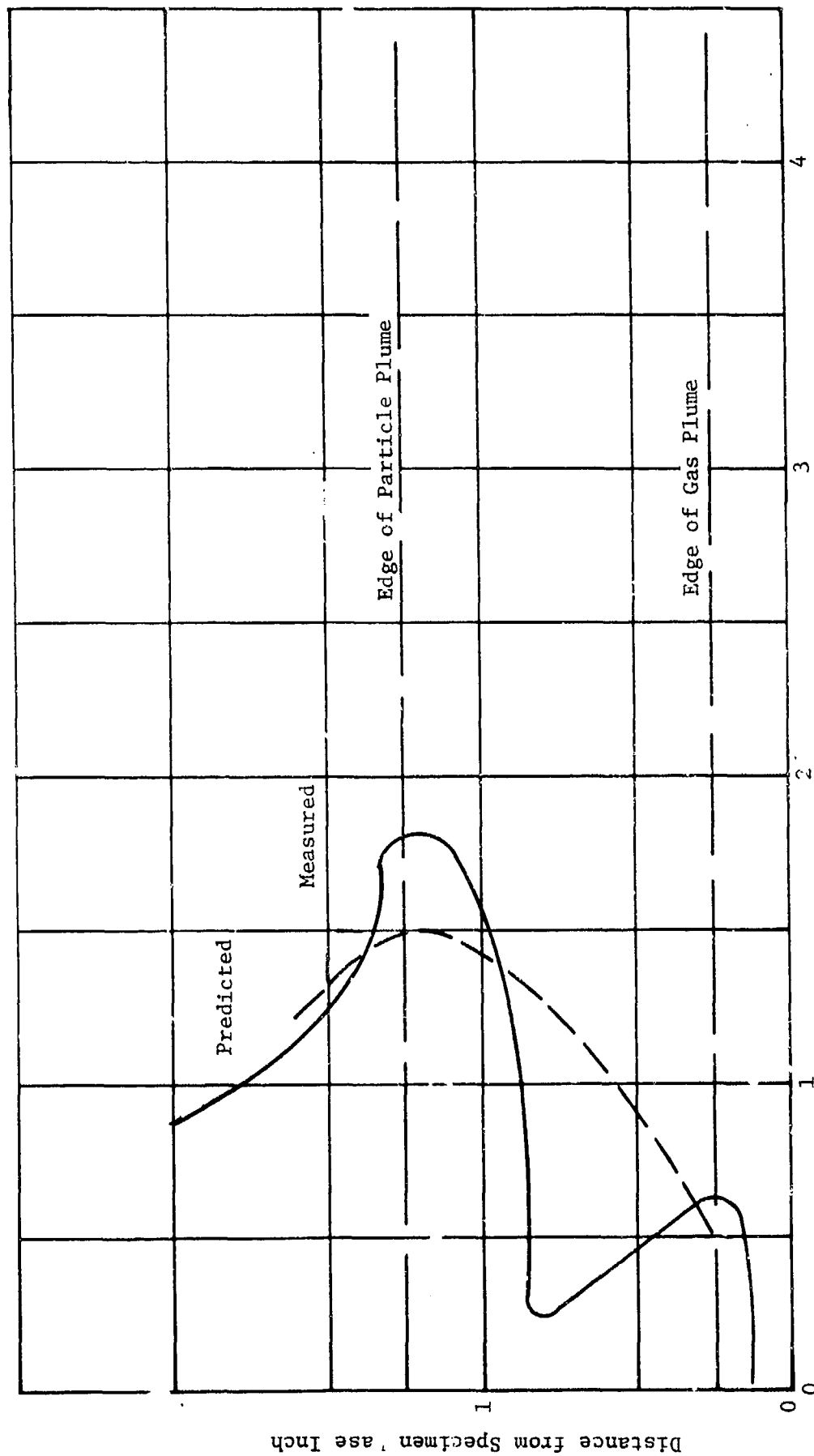


Figure 40 (U). Comparison of Analytical Model with Test Data - Test No. 3

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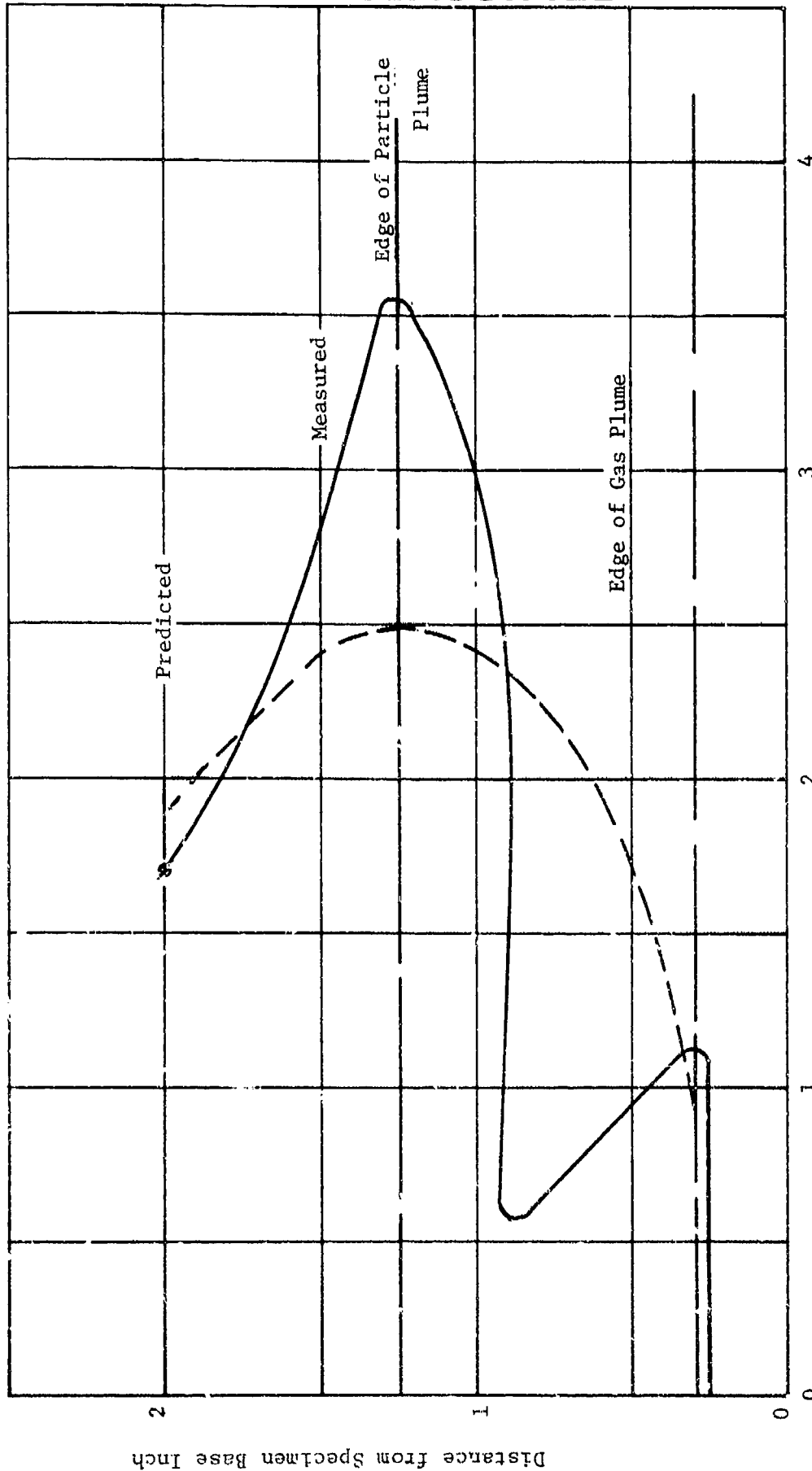


Figure 41 (U). Comparison of Analytical Model with Test Data - Test No. 4

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(U) The heating environments of Table VIII were applied to a tantalum carbide/zirconium dioxide/titanium specimen. The resulting temperature response data are compared in Figure 42. The specimen surface temperature approaches the environmental temperature for all heating conditions, as shown in Figure 42. Thus, the specimen temperature response data is similar for all heating conditions, as shown by the comparison of temperature response at the zirconium dioxide/titanium interface.

(U) Also, the longer duration of Test No. 2 produced melting temperatures in the subsurface titanium and as such was more severe than the higher heating rate, shorter burn time environments.

TABLE IX (U)

Heating Environment Comparison

Advanced Propellant		Heat Transfer Coefficient (BTU/ft ² -s-°F)
Rotor impact face for first 0.2 second	=	75.0
Rotor impact face for remaining test time	=	1.7
Nozzle inner surface	=	0.9
Test no. 3 (16 Aug 1973)	=	1.5
Test no. 4 (13 Sep 1973)	=	2.5

5. Conclusions

(U) The magnitude of the heat transfer coefficients in these tests is sufficient to raise the material surface temperatures relatively close to that of the gas environment. Thus, further increases in heat transfer coefficients over those of these tests will not significantly increase the surface temperature, only the rate at which these temperatures are reached. The results of these tests are considered to be representative of expected performance of the candidate materials under rotor heating conditions expected in an advanced control system.

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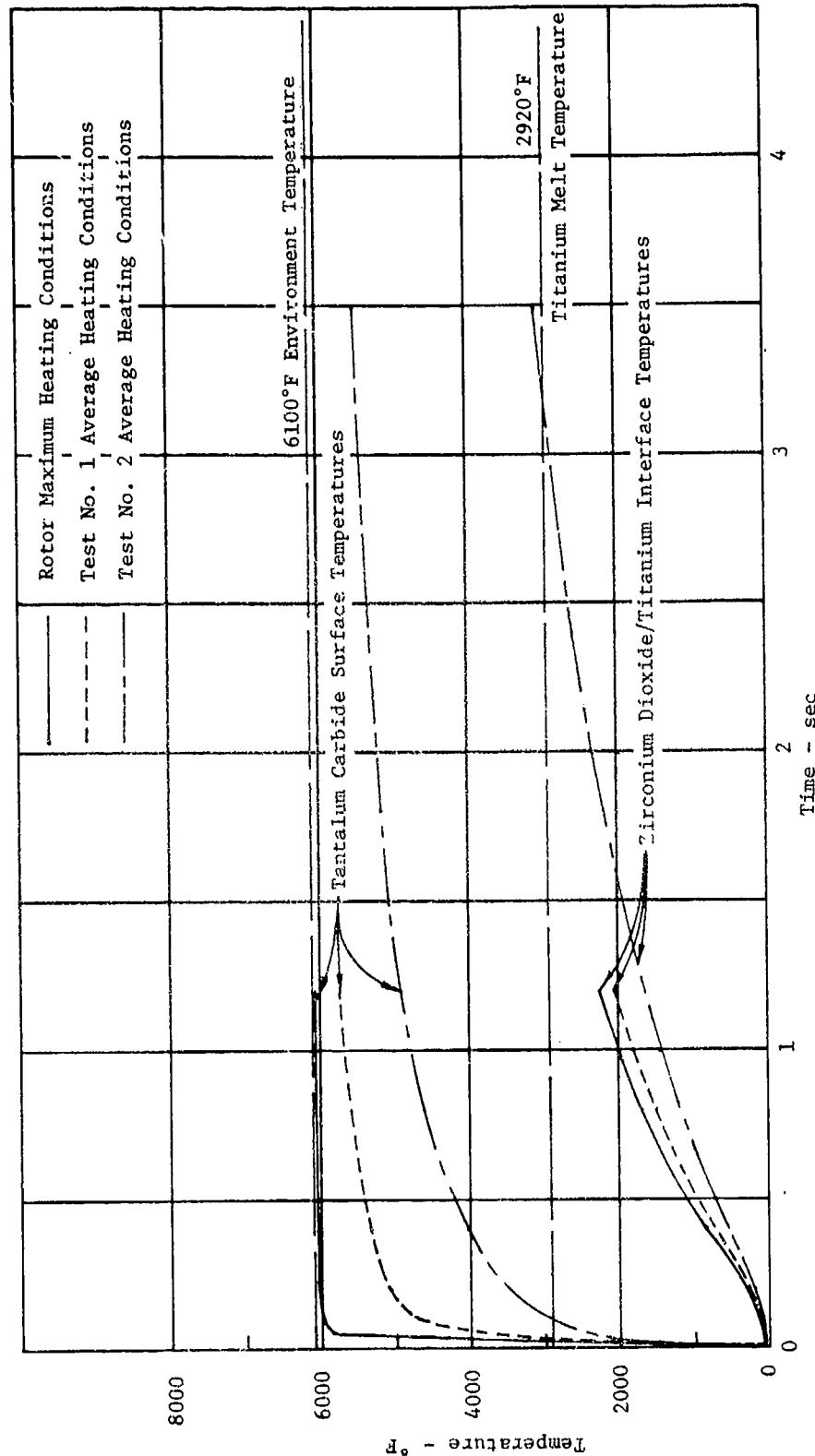


Figure 42 (U). Temperature Response of Test Specimen (0.106 Inch Tantalum Carbide/0.0045 Inch Zirconium Dioxide/0.395 Inch Titanium)

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VII. CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

(U) The following conclusions are the direct result of this refractory and insulator materials development effort together with design analysis:

1. Control System Weight Improvement

(U) It is expected that hot gas TVC/JIC valves in the Advanced Interceptor control system will be approximately 50 percent lighter than the current design as shown in tabular form in the summary.

2. Successful Material Combinations

(U) Vapor deposited tungsten is superior in erosion resistance to other candidates tested and can be used in approximately .060 inch thickness to provide protection for lightweight substrates, specifically titanium (for the rotor), rubber modified silica phenolic (for the nozzles), and Speer graphite. Plasma sprayed tungsten proved to be good protection for columbium but not for titanium primarily because of the porosity characteristic of plasma sprayed materials.

(U) The rubber modified silica phenolic unprotected is suitable for use in subsonic, parallel flow applications such as the inlet duct where a surface recession of 0.043 to 0.055 inch per second can be expected.

B. RECOMMENDATIONS

(U) The following items are recommended for further work:

- (U) 1 Application - Design, fabrication and test of hot gas valves using the newly developed refractory and insulator materials should be accomplished to demonstrate feasibility and work out detailed design and fabrication problems.
- (U) 2 New propellants - MICOM has several new propellants under development for possible use as Advanced Interceptor propulsion. Tests similar to those conducted on this program should be repeated using the same refractory and insulator material combinations and the new propellant to establish and update capabilities.

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- (U) 3 Refractory material improvements - Although tantalum carbide (TaC) has a higher melting point than tungsten, its usefulness is severely limited because of its brittleness. Further work on development of a modification or combination with TaC should be done to obtain a superior refractory coating. Suggested alternatives are: a plasma sprayed coating in which TaC and W are combined in various proportions; and a plasma spray coating of TaC done in an inert atmosphere to preclude oxidation followed by a vapor deposited coating of TaC to seal the pores or a vapor deposited coating of W to aid in heat conduction and reduce internal thermal stress.

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