NASA CR 108469

11602-6026-R0-00

TORCH TEST MATERIALS EVALUATION

APOLLO LUNAR MODULE DESCENT ENGINE ABLATIVE CHAMBER — INJECTOR COMPATIBILITY IMPROVEMENT STUDY

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

MSC PRIMARY PROPULSION BRANCH

Contract NAS 9-8229

11 APRIL 1969

TRW SYSTEMS GROUP

11602-6026-R0-00

APOLLO LUNAR MODULE DESCENT ENGINE ABLATIVE CHAMBER-INJECTOR COMPATIBILITY IMPROVEMENT STUDY

TORCH TEST MATERIALS EVALUATION

11 April 1969

Prepared for

NASA-MSC PRIMARY PROPULSION BRANCH

PROPULSION AND POWER DIVISION

UNDER CONTRACT NO. NAS 9-8229

Approved: R. E. Parsons

R. E. Parsons
Program Manager

TRW SYSTEMS
ONE SPACE PARK · REDONDO BEACH, CALIFORNIA

CONTENTS

ILLUSTRATIONS

ILLUSTRATIONS (Continued)

ILLUSTRATIONS (Continued)

ILLUSTRATIONS (Continued)

ILLUSTRATIONS (Continued)

1. INTRODUCTION

Although MX-2600 silica fabric phenolic laminate has proved to be highly successful as LMDE chamber material, throat erosion does occur during normal engine duty-cycle firing. Throat erosion produces a drop in chamber pressure which affects engine performance. For this reason, the search for and the development of a better chamber throat material has been the object of this and other studies.

A number of laboratory test methods have been used for the evaluation of chamber throat materials, but none can simulate exactly the conditions of the engine environments. The actual environment consists of at least four major components which combine, affecting materials behavior: thermal, reactive gas, velocity, and pressure. In this investigation, the methane-oxygen-nitric oxide mixture was used to approximate the thermal and reactive gas environment, but not the pressure and velocity effects. The purpose of this study was to evaluate and compare a large number of candidate throat materials for their thermo-chemical behavior under LMDE gas environments. Post torch test analysis criteria included visual examination, weight change and dimensional change (erosion). This report describes and discusses the procedures, results, and conclusions of the torch tests.

2. EXPERIMENTAL PROCEDURES

2.1 TEST EQUIPMENT

The torch testing facility is pictured in Figure 1. The containment vessel is a stainless steel tank 4-feet diameter x 7-feet high. The tank is divided at the center and flanged with an O-ring seal for vacuum-tight operation. The upper chamber half is provided with three view ports for monitoring flame interaction experiments. Provisions are made to heat each view port to prevent fogging. The lower chamber half is equipped with two access ports through which metered gas flows are fed to the torch assembly and all electrical and instrumentation connections are made. The vacuum chamber is connected through a filtration system to a large roughing pump which exhausts through the roof to the atmosphere.

An A-frame and chain hoist are provided to facilitate removal of the upper chamber half which can be moved laterally to clear its lower section.

All torch and sample handling controls and instrumentation are built into the lower half of the vacuum chamber. Test specimens to be exposed to the flame environment are supported on a circular platform. This plate can be positioned up or down to adjust torch-to-sample spacing. The plate pivots about the rear support to move experiments into and out of the established flame environment by means of an externally operated reach rod.

A schematic of the test apparatus is shown in Figure 2, and a close-up view of the torch test in progress is shown in Figure 3. The oxygen and nitric oxide gases are premixed prior to entry to the torch tip manifold where the oxidizers are mixed with methane before burning. The flow rates of the gases to the torch are metered with accurately calibrated flow-meters. A two-color pyrometer is positioned outside of the chamber to monitor the surface temperature of the test specimen during testing.

2.2 TEST PROCEDURE

Test conditions were established to maximize the thermal environments and to approximate an oxygen-rich LMDE chamber gas composition. A computer program based on thermodynamic data of CH_4-O_2-NO was used to guide the selection of the desired gas mixture. After experimenting with a number of fuel/oxidizer ratios using MX-2600 specimens and comparing with the computer program output on equilibrium chemical species produced from these mixtures, a composition of 40 CH_4-50 O_2-10 NO by volume was selected as the best compromise of the flame temperature and combustion products. This flame was used in all torch testing in this program unless otherwise specified. A comparison of equilibrium torch temperature and chemical species with LMDE chamber gas composition and temperature for a number of mixture ratios is shown in Table 1.

The heat flux was determined with a copper calorimeter having the same configuration as the test specimen, Figure 4. The heat flux radially through the wall of the specimen was found to be 120 Btu/sq. ft./sec. and was checked periodically to assure uniformity of test condition from specimen to specimen.

Having standardized the gas mixture and heat flux, the next step was to determine the most appropriate test duration for comparing the various materials under the same test conditions. Experiments were conducted with MX-2600

TABLE 1. CHEMICAL SPECIES IN OXYGEN/FUEL MIXTURES

Mixture	Condition	Temperature °R	Mole Fraction of Chemical Species at Equilibrium					
			CO	CO_2	H_2	H_2O	N_2	O_2
N_2O_4/50-50 N_2H_4-UDMH MR = 1.6 P_c = 100 psi	LMDE Throat	5180	9.4×10^{-2}	3.7×10^{-2}	1.3×10^{-1}	3.6×10^{-1}	3.3×10^{-1}	1.7×10^{-3}
Same as Above	LMDE Chamber	5502	3.5×10^{-2}	3.4×10^{-2}	1.3×10^{-1}	3.5×10^{-1}	3.3×10^{-1}	3.0×10^{-3}
40 CH_4 - 50 O_2- 10 NO by Volume	Torch Test	5407	2.3×10^{-1}	6.9×10^{-2}	1.7×10^{-1}	3.7×10^{-1}	3.6×10^{-2}	1.0×10^{-2}
50 CH_4 - 50 O_2 by Volume	Torch Test	4707	2.9×10^{-1}	3.7×10^{-2}	3.5×10^{-1}	2.8×10^{-1}	-0-	7.8×10^{-5}

for test durations of 150 seconds, 300 seconds, 600 seconds, 1200 seconds and 1800 seconds. Results of weight losses and diametrical changes are tabulated in Table 2 and plotted in Figure 5.

TABLE 2

WEIGHT LOSS AND PERCENT EROSION OF
MX-2600 FROM TORCH TESTING

Test Duration, Seconds	Weight Loss, %	Erosion, %
150	10.8	1.3
300	15.5	3.1
600	19.4	4.8
1200	24.7	7.4
1800	27.3	9.6

On the basis of the MX-2600 test results, 300-second and 600-second test durations were selected as reference points for evaluation and comparison of candidate throat materials. Therefore, all testing was performed using the two time durations in this investigation unless otherwise specified. Test specimen preparation and manufacturing methods for these materials are described in detail in the Appendix. In the main text of this report, the commercial names or brief descriptions of the materials are used rather than the part numbers shown in the Appendix.

The weight and dimensions of each specimen were measured before and after torch testing. In the beginning of the run, the heat flux was measured and the flame and/or specimen positions were adjusted to assure alignment and concentricity of the flame with the center line of the test specimen. After evacuating the chamber and with the test specimen in place on the platform, but out of the test position, the flame was lit using the desired gas mixture. The specimen was then swung into the test position as timing started. Temperature readings were taken at 30-second intervals by focusing the pyrometer on the inside diameter surface at about 1/16-inch below the top edge of the hole. At the conclusion of the test duration, gas flow was immediately shut off and the specimen was allowed to cool naturally while the chamber was still being evacuated.

Post-test analyses included visual examination, weight loss determination, and erosion or dimensional changes. Metallographic and X-ray diffraction analyses were performed on certain specimens as required.

3. EXPERIMENTAL RESULTS

Post-torch test analyses included visual examination, metallographic examination, weight changes, and dimensional changes (erosion). Photographs of torch tested specimens are shown by groups and individually in Figures 6 through 31. Table 3 summarizes the complete test data showing the equilibrium surface temperature, test duration, weight loss and percent erosion. Results are discussed in the following sections.

3.1 VISUAL EXAMINATION

Figures 6 through 9 show appearance of torch tested specimens and their identifications by groups. Group 1, shown in Figure 6, is the "hard throat" materials which show typically very little dimensional change. Figures 10 through 14 are close-up photographs of typical specimens. The large erosion which occurred in the SiC-coated Carbone 2239 graphite specimen after 30 minutes of testing was attributed to probable misalignment of the flame or localized imperfection in the coating. The 300-second and 600-second SiC coated graphite specimens show no sign of deterioration. There was a crust formed on the surface of the JTA specimen which consists probably of the SiO_2 and ZrO_2 reaction products. The white materials that formed around the porous tungsten L13 treated specimens are oxidation products of the infiltrant. The ZrB_2 V and ZrB_2 VIII specimens were discolored but physically unaffected. Both zirconia specimens show transverse cracks and delamination along the lamination plane; otherwise, no visible erosion effect was evident.

Group 2 specimens in Figure 7 are the precharred or pyrolyzed laminate materials. Figures 15 through 17 are close-up photographs of typical specimens in this group. Pyrolyzed silica phenolic composites HR211 and HR212 demonstrated considerably less erosion effect than the Carbitex materials. All Carbitex 100 materials showed disappointingly low erosion resistance. The Carbitex specimens containing SiC additive appeared to erode less than specimens containing ZrB_2, TiB_2, W_2B, or B_4Si additives. The beneficial effect of SiC having higher oxidation resistance is evident.

TABLE 3. TORCH TEST DATA

Specimen	Specimen Surface Temperature (°C)	Time (Seconds)	Weight Loss		Average Percent Erosion*
			Grams	Percent	
MX-2600	1760	300	11.73	16.4	3.1
MX-2600	1835	600	13.75	19.4	4.8
TRW-2A	1750	300	13.16	20.1	2.0
TRW-2A	1760	600	15.05	22.3	4.9
TRW-2A Precharred and Reimpregnated	1710	600	11.31	17.8	4.7
MXQ-190	1850	300	10.32	16.2	2.2
MXQ-190	1770	600	14.07	19.7	6.4
HRX-211	1900	300	3.19	4.7	6.5
HRX-212	1825	300	2.62	3.9	5.9
Carbitex 100 (SiC mod.)	1750	600	9.16	22.8	34.0
Carbitex 100 (ZrB_2 mod.)	1960	600	11.27	22.8	43.0
Carbitex 100 (TiB_2 mod.)	1660	600	11.39	24.6	45.1
Carbitex 100 (W_2B mod.)	1650	600	17.16	30.7	74.9
Carbitex 100 (B_4Si mod.)	1645	600	9.26	19.4	37.3
Pyrocarb 751	1800	300	2.31	2.8	0.1
Pyrocarb 751	1780	600	5.09	6.7	0.1
TRW-5	1765	300	11.41	15.4	2.2
TRW-5	1770	600	14.29	20.2	9.2
TRW-5 Precharred and Reimpregnated	1770	600	9.67	17.3	7.5
TRW-2	1820	300	12.09	18.1	4.2
TRW-2	1810	600	14.60	21.9	9.1
TRW-2 Mold	1790	300	9.87	14.7	1.9
TRW-2 Mold	1790	600	11.51	20.1	9.0
Quartz Polyimide	1750	300	8.40	12.5	6.7
Quartz Polyimide	1740	600	12.10	17.8	8.0
EC-260 Silica (W mod.)	1850	300	8.24	9.7	5.7
EC-260 Silica (W mod.)	1850	600	17.01	25.0	8.9
Ironsides Resin with Astroquartz	1700	300	10.97	14.1	5.2
Ironside Resin with Astroquartz	1750	600	13.31	19.6	2.1
C-100-48 Refrasil Low Resin	1940	300	7.82	11.3	12.9
C-100-48 Refrasil Low Resin	1920	600	10.49	15.2	4.4

TABLE 3. TORCH TEST DATA (Continued)

Specimen	Specimen Surface Temperature (°C)	Time (Seconds)	Weight Loss		Average Percent Erosion*
			Grams	Percent	
C-100-48 Refrasil High Resin	1860	300	6.50	12.5	3.8
C-100-48 Refrasil High Resin	1840	600	10.43	15.4	5.5
C-100-28 Refrasil	1790	300	8.48	13.0	4.9
C-100-28 Refrasil	1850	600	12.30	18.7	7.7
TRW-3	1810	300	10.51	16.6	2.3
TRW-3	1815	600	13.06	20.6	11.5
Carbitex 100 (SiC mod.)	1770	300	3.87	9.5	13.8
Carbitex 100 (ZrB_2 mod.)	1950	300	5.42	10.8	25.0
Carbitex 100 (W_2B mod.)	1720	300	9.40	14.0	27.6
Carbitex 100 (B_4Si mod.)	1645	300	5.02	10.4	21.8
Carbitex 100 (TiB_2 mod.)	1660	300	5.41	11.8	31.4
TRW-11A	1650	300	11.67	18.5	5.4
TRW-11A	1660	600	16.00	25.1	16.3
TRW-2 Precharred	1880	600	7.10	12.1	5.2
TRW-2 Precharred and Reimpregnated	1780	600	10.31	15.8	5.7
Type C W-3Re Reinforced Zirconia	1980	600	10.20	--	--
Type A W-3Re Reinforced	2000	600	--	--	--
Zirconia Bonded Zirconia	2030	300	--	--	--
Zirconia Bonded Zirconia	2020	600	--	--	--
Carbone Graphite (SiC coated)	1610	600	0.46	0.153	--
ZrB_2 V	1760	600	--	--	--
ZrB_2 VIII	1760	600	--	--	--
Porous Tungsten with L13 Treatment	1620	300	--	--	--
Porous Tungsten with L13 Treatment	1720	600	--	--	--
JTA Graphite	1800	600	2.59	0.53	--
Crystar (SiC mod.)	1640	600	--	--	--
Graphite Phenolic (ZrB_2 mod.)	2020	600	13.18	16.0	4.2
SiC Powder 1200 Mesh Graphite Fiber Reinforced SC-1000 Phenolic Precharred	1815	600	5.66	8.4	13.8

TABLE 3. TORCH TEST DATA (Continued)

Specimen	Specimen Surface Temperature (°C)	Time (Seconds)	Weight Loss		Average Percent Erosion*
			Grams	Percent	
TRW-2A Precharred and Reimpregnated**	1600	600	10.15	15.6	5.0
TRW-5 Precharred and Reimpregnated**	1700	600	7.41	10.7	1.7
MX-2600**	1750	300	10.33	14.6	0.82
[illegible]**	1740	600	13.43	19.0	1.17
EC-260 Resin with Astroquartz	1750	300	9.35	13.4	2.7
EC-260 Resin with Astroquartz	1745	600	12.96	19.0	3.7

*Average percent erosion is obtained by taking the average of the absolute percent diametrical changes throughout the length of the hole in the test specimen. Thus, average erosion = $\frac{\Sigma|\Delta x_i|}{N}$ where x_i is percent erosion at location i, and N is the number of measurements.

**These specimens were tested using a gas mixture of 50 CH_4 to 50 O_2 by volume.

--Weight loss or erosion less than 0.1%.

The third group of specimens is the ablative resin matrix type shown in Figure 8. Figures 18 through 23 are photographs of typical specimens in this group. From visual examination it appears that, in general, specimens containing quartz fibers such as MXQ-190 and Ironside/Astroquartz exhibited better erosion-resistant properties. MXQ-190, in particular, showed greater dimensional stability than MX-2600 in the 300-second test, although both formed vitreous silica from torch testing. The silica coat on the MX-2600 has a glass-like appearance, whereas the silica flow over the MXQ-190 looks like white enamel. Although both quartz/polyimide and quartz/Ironside resin 5471 appear to be promising, the former showed gross delamination and the latter considerable longitudinal shrinkage that would make them undesirable as throat material. Visually, none of the Refrasil reinforced resin materials indicated erosion resistance equal to or better than MX-2600. Silica/EC-260 with tungsten powder additive and graphite/phenolic resin with 1200-mesh SiC powder performed very poorly in torch testing. Graphite/SC-1008 phenolic resin containing ZrB_2 powder exhibited little erosion; but numerous cracks throughout the specimen make it a doubtful candidate material.

The fourth group of specimens, Figure 9, represents a number of TRW formulations prepared on the basis of results of charring reaction studies which revealed that a higher carbon-silica ratio favors the formation of silicon carbide. It was postulated that beneficial effects can be derived by the endothermic reaction of $SiO_2 + 3C \rightleftarrows SiC + 2CO$. Description of the TRW formulations and fabrication methods are tabulated in the Appendix. Several specimens were precharred, or precharred and reimpregnated with resin prior to torch testing, to compare with as-fabricated resin-base materials. Table 4 lists this series of torch tested specimens after testing. The resin-base materials in the as-fabricated condition appear to be not much better than MX-2600 with respect to erosion resistance.

All specimens showed a tendency to delaminate, except TRW-5. Three formulations TRW-2, TRW-2A, and TRW-5 performed decidedly better than TRW-3 and TRW-11, and as well as MX-2600. Visual examination of the precharred, or precharred and reimpregnated specimens after torch test indicated an improvement in performance over the corresponding as-fabricated resin-base materials.

TABLE 4. DESCRIPTION OF TRW FORMULATED SPECIMENS

Sample Designation	Sample Type	Composition	Sample Condition Prior to Test
TRW-2	Molded	SC-1008 Phenolic + 27% Graphite + 43% Silica Fibers	As Molded
TRW-2-1	Laminated	SC-1008 Phenolic + 27% Graphite Cloth and 43% Refrasil Fabric	As Laminated
TRW-3	Laminated	SC-1008 Phenolic + 17% Graphite Cloth and 53% Silica Fabric	As Laminated
TRW-5-1	Laminated	SC-1008 Phenolic + 15% Graphite Cloth + 46% Refrasil Cloth + 9% SiC Powder	As Laminated
TRW-11A	Laminated	TRW Type A Polyimide + 47% Astroquartz Cloth + 21% Graphite Cloth	As Laminated
TRW-2A-1	Laminated	SC-1008 Phenolic + 27% Graphite Cloth + 43% MXQ-190 Quartz Phenolic Prepreg	As Laminated
TRW-2-2	Laminated	Same as TRW-2-1	Laminated; Precharred at 800°C for 2 Hours
TRW-2-3	Laminated	Same as TRW-2	Laminated; Precharred at 800°C for 2 Hours; Vacuum impregnation Furfuryl Alcohol
TRW-2A-2	Laminated	Same as TRW-2A	Same as TRW-2-3
TRW-5-2	Laminated	Same as TRW-5	Same as TRW-2-3

3.2 WEIGHT LOSS AND DIMENSIONAL CHANGES (EROSION)

Weight loss and dimensional change measurements were made on torch-tested specimens to provide a more quantitative comparison of the performance of various candidate materials after subjection to torch testing. Table 3 summarizes the test results. Column 1 in the table presents names of brief designations of the test specimens whose formulations, preparation methods, or suppliers are described in the Appendix. Column 2 lists the steady state surface temperature of the specimen during torch testing. Column 3 is the test duration at the steady state temperature. The weight loss is reported in grams and percent of the original weight. The average percent erosion, as defined in the footnote of the table, is the mean average of absolute percent diametrical changes (either increase or decrease) computed from the erosion profiles of the test specimens. In other words, the increase in diameter due to erosion and the decrease in diameter due to silica flow as in the case of MX-2600 are both regarded as "erosion" in this report. Figures 32 through 81 are plots of the erosion profiles; i.e., percent erosion versus location of measurements along the length of the specimen. MX-2600 data are plotted in dash lines in some graphs for comparison. Data of some materials are either not available or are not appropriate for plotting due to nonstandard specimens or test conditions employed.

Figures 82 through 85 are bar graphs showing percent weight loss and percent erosion for each material in the order of increasing weight loss or decreasing erosion resistance. Although some correlation exists between weight loss and erosion for some materials, it is difficult to correlate weight loss and erosion resistance performance of ablative materials because of their variations in resin content.

Results of weight loss and percent erosion are in general agreement with those of visual examination discussed earlier. As expected, hard throat materials such as the zirconium diborides, ZrB_2 V and ZrB_2 VIII, JTA graphite zirconia, and SiC exhibited neither weight change nor erosion. Pyrocarb 751 and SiC-coated Carbone 2239 graphite also demonstrated excellent erosion resistance. All others are ablative materials showing various magnitudes of weight loss and erosion resulting from torch flame testing.

On the basis of the 300-second test results, Table 3 and Figure 84, several resin-base ablatives showed better erosion resistance than MX-2600. These are TRW-2 molded (1.9% erosion), TRW-2A (2.0%), MXQ-190 (2.2%), TRW-5 (2.2%), and TRW-3 (2.3%). However, similar results were not reproduced in the 600-second tests. Except for TRW-2A which showed about equal performance to MX-2600, all others eroded more than the latter in the 600-second test.

The Carbitex 100 materials showed a 13.8 to 31.4% and 34.0 to 74.9% erosion for 300-second and 600-second tests respectively, an order of magnitude higher than the resin-base materials. Therefore, Carbitex materials are considered very poor candidate throat materials.

Inconsistency was found in several sets of test data. Astroquartz/Ironside and C-100-48 refrasil/low resin data showed a lower percent erosion for the 600-second test although the weight losses were consistent with the test duration. There is no good explanation for this discrepancy except possibly that diametrical measurements were inaccurate due to longitudinal dimensional change (shrinkage was observed in quartz/Ironside resin specimens).

If one assumes a deviation of ± 1.0 in the accuracy of percent erosion measurements and compares the averages of the 300-second and 600-second test results, one would consider TRW-A quartz/Ironside resin, MX-2600 and MXQ-190 about equally good resin base LMDE throat candidate materials (see Table 3 and Figures 84 and 85).

Studies were made to improve the erosion resistance of the resin-base materials by precharring and precharring followed by reimpregnation prior to torch testing. Results of visual examination have already been discussed (refer to Table 4 for description and treatment of these specimens).

Table 5 shows the sequential weight changes of these specimens resulting from charring, resin impregnation, and torch testing. The weight loss after charring ranges between 13.1 to 15.6% which is equivalent to a char yield of 55% to 50%, typical of phenolic resin materials. Vacuum reimpregnation with furfuryl alcohol produced varying degrees of weight increase,

TABLE 5. SEQUENTIAL WEIGHT CHANGES OF TRW FORMULATED SAMPLES
(all weights are in grams; percent changes in parentheses)

Specimen	Sample Type	Weight Prior to Charring	Weight After Charring	Weight After Reimpregnation	*Weight After Torch Test	Average Percent Erosion
TRW-2-1	Laminated	66.793	--	--	52.187 (-21.9%)	9.1
TRW-2-2	Laminated	66.508	57.785 (-13.2%)	--	50.684 (-12.1%)	5.2
TRW-2-3	Laminated	66.995	57.685 (-13.5%	65.300 (+13.4%)	55.015 (-15.8%)	5.7
TRW-2A-1	Laminated	65.233	--	--	52.066 (-22.3%)	4.9
TRW-2A-2	Laminated	65.789	55.906 (-15.1%)	63.35157 (+13.2%)	52.040 (-17.8%)	4.7
TRW-2A-3	Laminated	64.743	54.607 (-15.6%)	64.642 (+18.4%)	54.491 (-15.7%)**	5.0
TRW-5-1	Laminated	69.818	--	--	55.524 (-20.2%)	9.2
TRW-5-2	Laminated	71.344	61.603 (-13.6%)	67.982 (+9.8%)	58.310 (-14.3%)	7.5
TRW-5-3	Laminated	69.951	60.824 (-13.1%)	67.504 (+14.3%)	62.084 (-10.7%)**	1.7
MX-2600		70.540	--	--	57.100 (-19%)**	1 2
MX-2600		70.981	--	--	57.230 (-19.4%)	4.8

*All torch tests were run for 600 seconds using a gas mixture of 40 CH_4-50 O_2 -10 NO by volume except those marked

**A fuel rich mixture of 50 CH_4-50 O_2 was used. Total test duration was 600 seconds for all specimens.

probably due to variations in porosity level in the as-charred materials. Torch test results indicated that weight loss was considerably less in the precharred and precharred-resin impregnated specimens. The weight loss for all TRW specimens in the laminated condition, including MX-2600, is about equal. There appears to be an improvement in erosion resistance from precharring and resin reimpregnating as in the case of TRW-2 and TRW-5. The percent erosion of TRW-2 was reduced to 5.7% from 9.1% in the as-laminated condition after the precharring treatment. For TRW-5, the erosion reduction was from 9.2% to 7.5%. However, TRW-2A exhibited little improvement.

All torch testing had been conducted with an oxidizer-rich flame simulating the most severe LMDE chamber environment. An apparent anomaly was the fact that there was no trace of silicon carbide in any of the specimens exposed to the torch, even those formulations that did form silicon carbide in laboratory experiments. In addition, silicon carbide was also found in the surface char of LMDE chamber liners that had been exposed to a duty-cycle firing. It was postulated, therefore, that at full thrust operation, the boundary layer of the LMDE chamber was fuel-rich and that fact promoted the in situ reaction between carbon and silica to form silicon carbide. Therefore, two of the most promising materials, TRW-2A and TRW-5 (see Tables 3 and 5), were retested, along with the baseline MX-2600 material, with a fuel-rich flame.

The effect of gas atmosphere on weight loss and erosion from torch testing appears to be significant for certain composite formulations. A lower weight loss was obtained in TRW-2A and TRW-5, but not in MX-2600 when a fuel-rich mixture was used. However, lower erosion rate was also evident by comparing the dimensional changes in all specimens except TRW-2A tested at fuel-rich gas mixtures.

X-ray diffraction analyses were performed on several specimens after torch testing to determine if silicon carbide had formed. The following are results of these analyses:

Specimen	Test Condition	Silicon Carbide Detection
MX-2600	LMDE Throat (duty cycle)	Yes
MX-2600	Torch Test (40 CH_4 - 50 O_2 - 10 NO)	No
MX-2600	Torch Test (50 CH_4 - 50 O_2)	Yes
TRW-2A	Torch Test (50 CH_4 - 50 O_2)	Yes

It is interesting to note that a less oxidizing atmosphere is favorable for the formation of silicon carbide which formed preferentially on the surface in contact with the flame.

4. CONCLUSIONS AND RECOMMENDATIONS

On the basis of erosion or dimensional change measurements of torch tested specimens, it is concluded that only hard throat materials such as JTA, SiC, W-reinforced zirconia, zirconia, and coated carbon/graphite materials such as SiC-coated Carbone graphite and Pyrocarb 751 can withstand the simulated LMDE throat environments with little or no erosion. However, poor thermal shock resistance in some of these materials and insufficient development in others may preclude the selection of these materials for full-scale evaluation.

Of the ablative type of resin-base materials, TRW-2A, quartz/Ironside resin, MXQ-190, and MX-2600 showed about equal erosion resistance in torch tests. On the basis of 300-second testing, several formulations such as TRW-2A and MXQ-190 appear to have an edge over MX-2600. Full-scale engine tests are recommended to verify these conclusions.

Some indications were shown in test results that precharring or precharring followed by resin reimpregnation is effective in improving the erosion performance of the precursor resin-base material. Encouraging results shown by precharred TRW-2 and TRW-5 formulations warrant verification by engine testing.

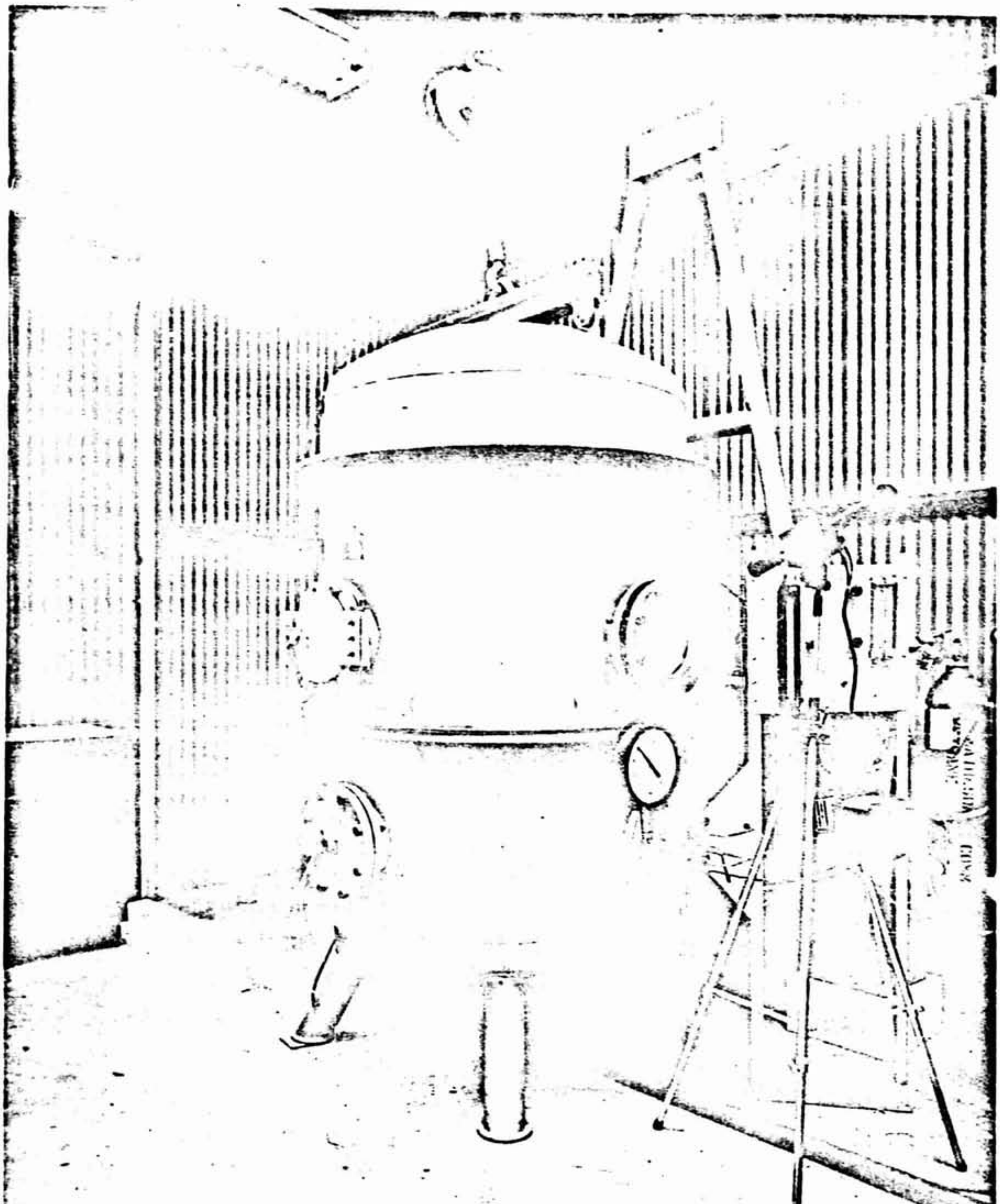

Figure 1. Torch Test Facility

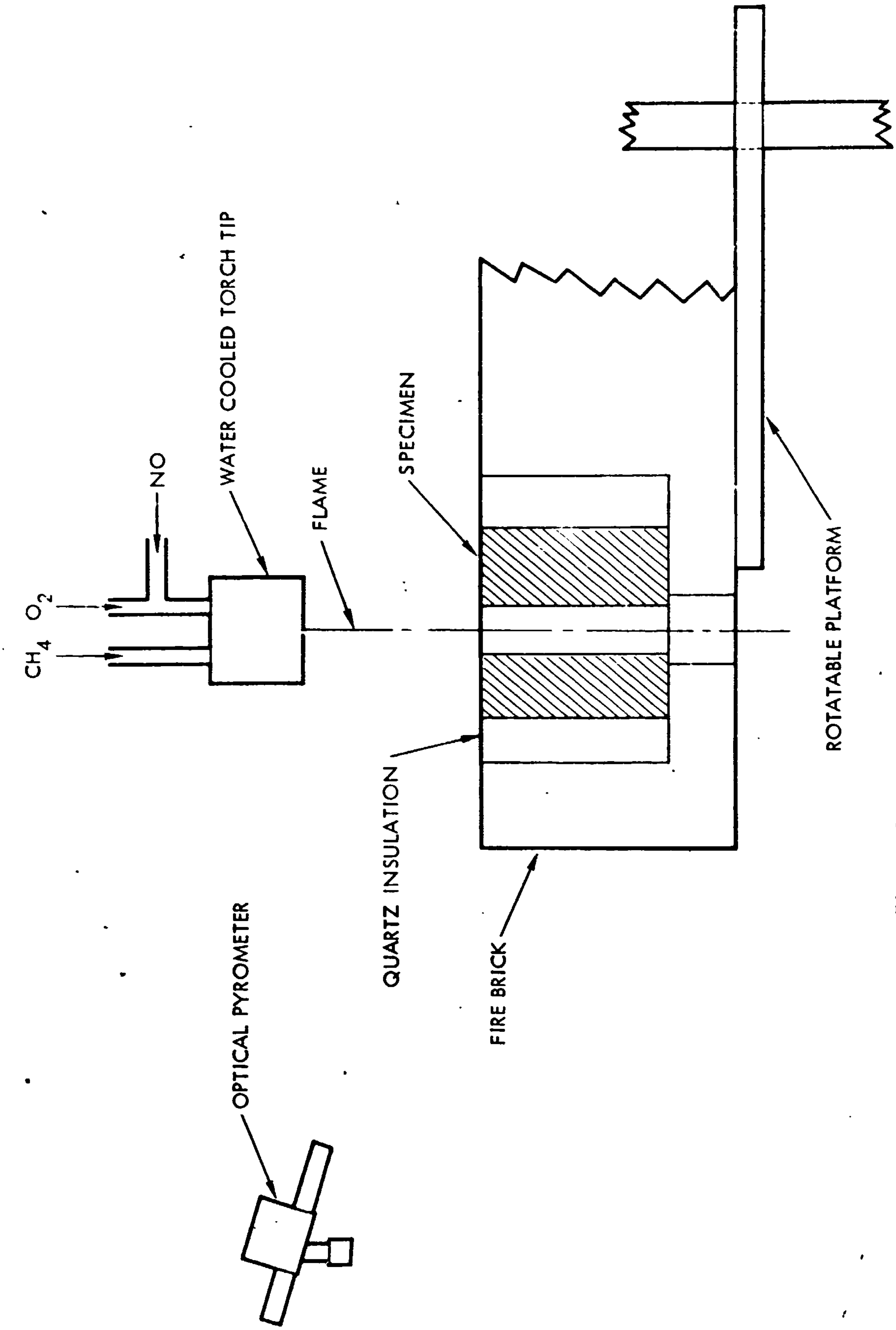

Figure 2. Schematic of Test Apparatus

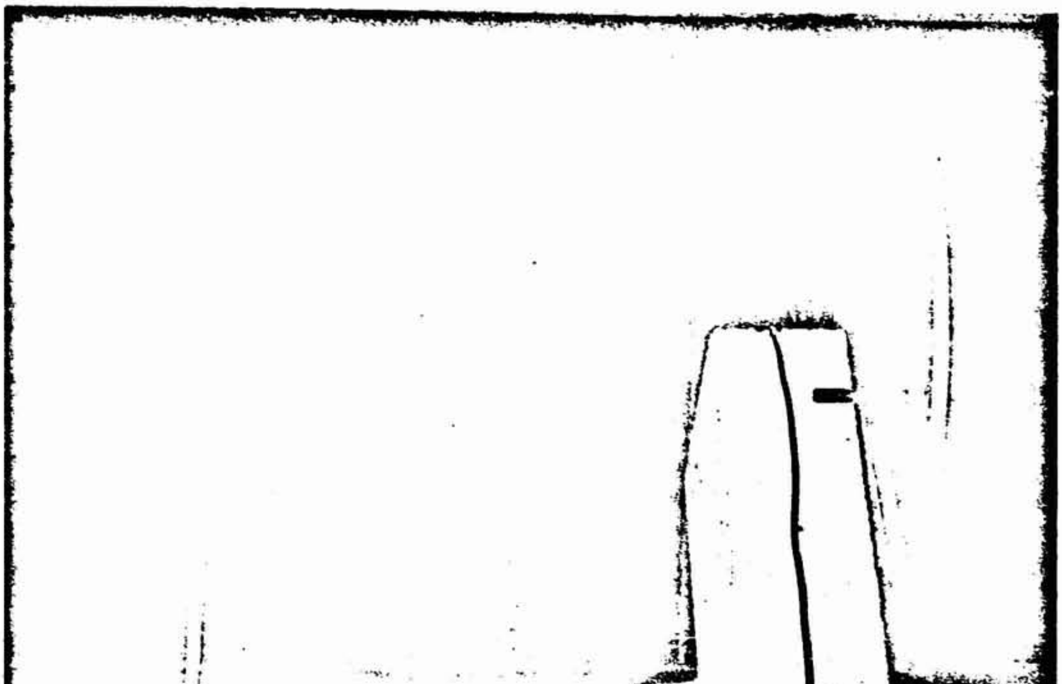

Figure 3. Torch Test in Progress

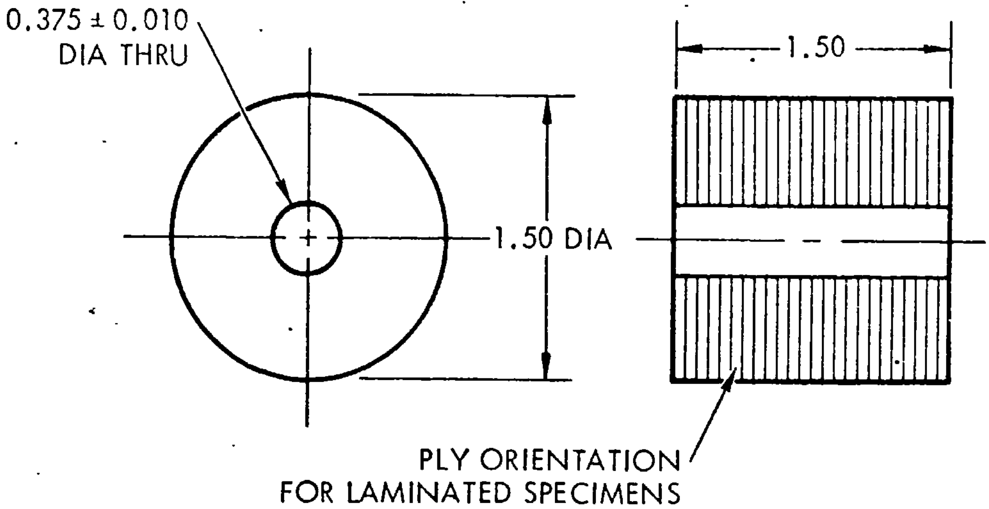

Figure 4. Torch Test Specimen Configuration

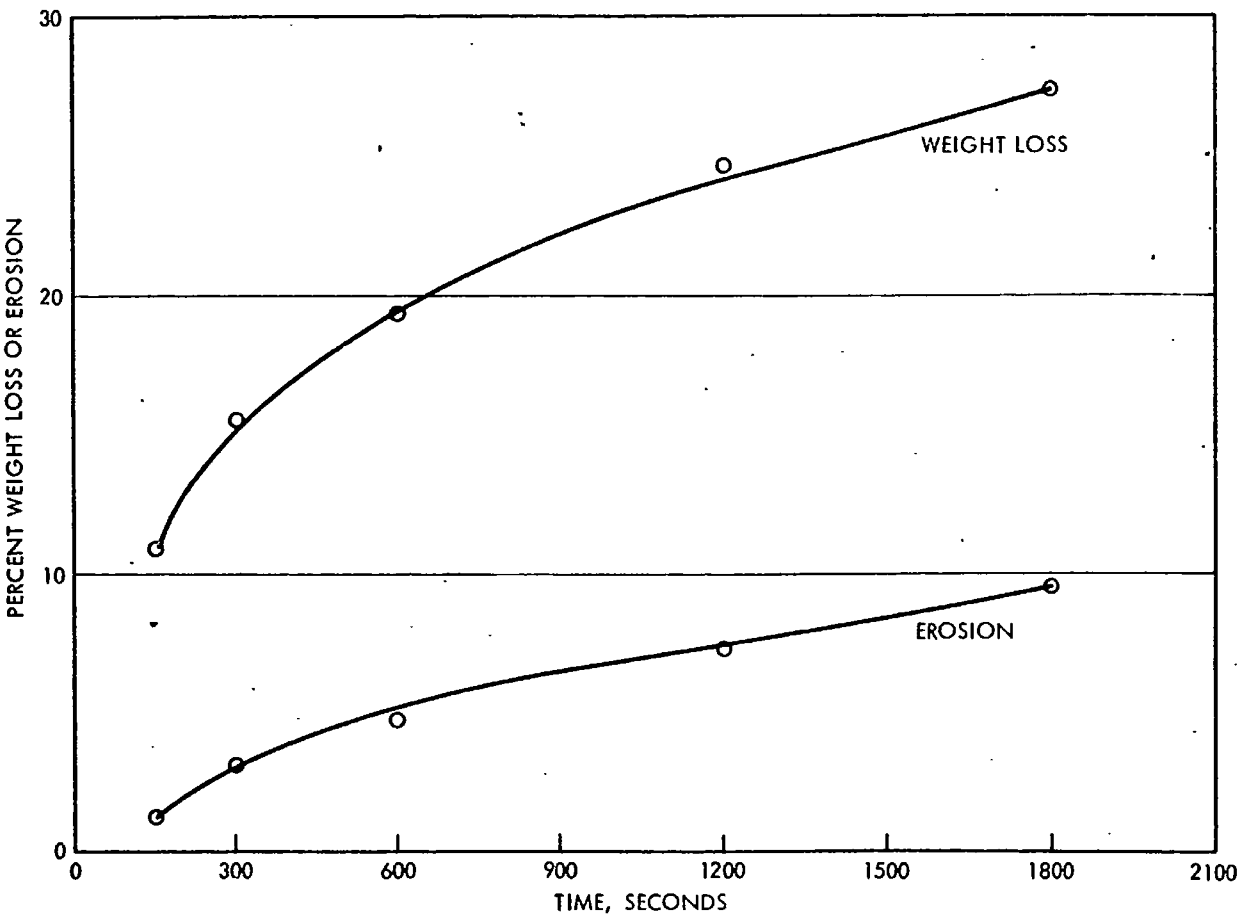

Figure 5. Rates of Weight Loss and Erosion of MX-2600

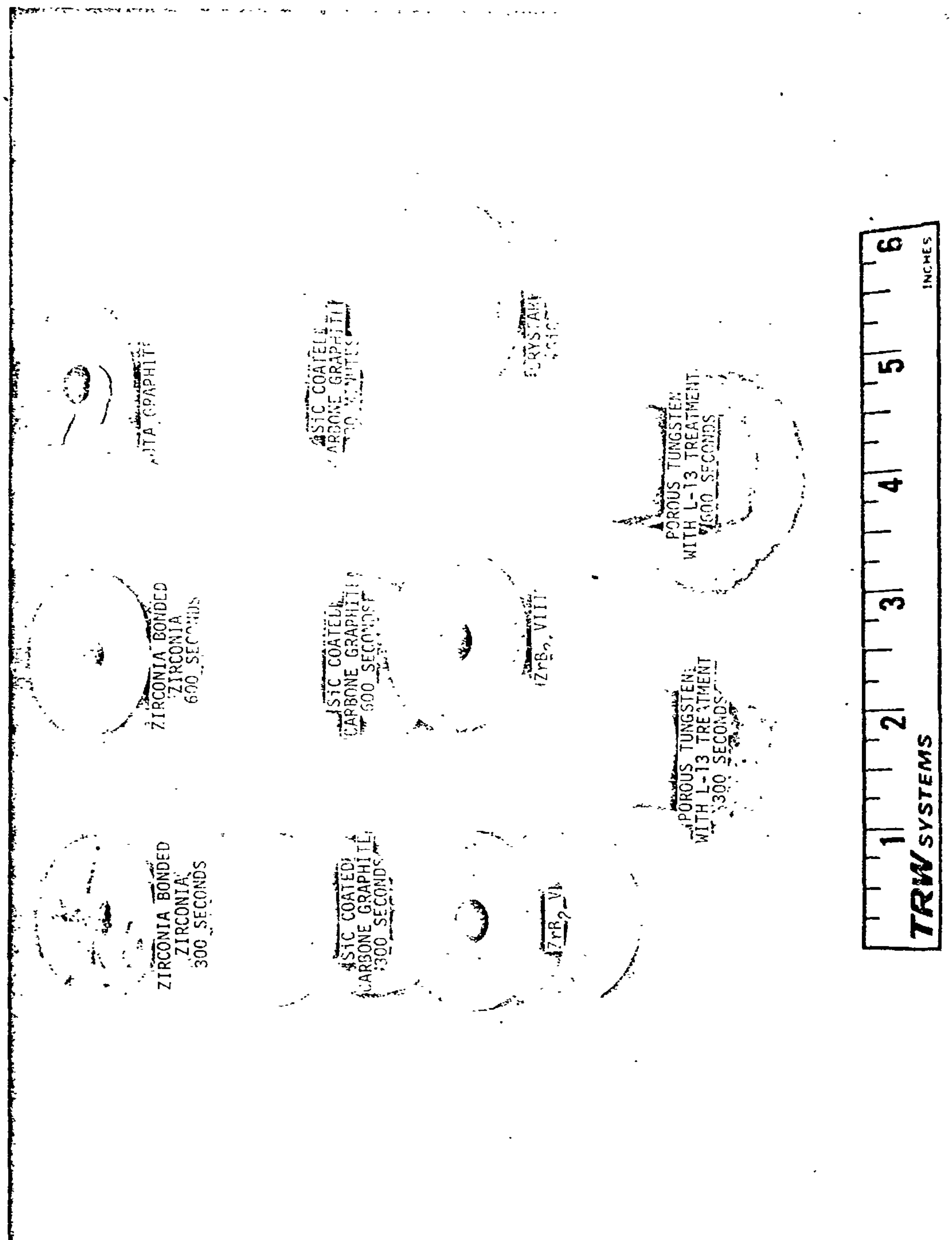

Figure 6. "Hard Throat" Materials after Torch Test

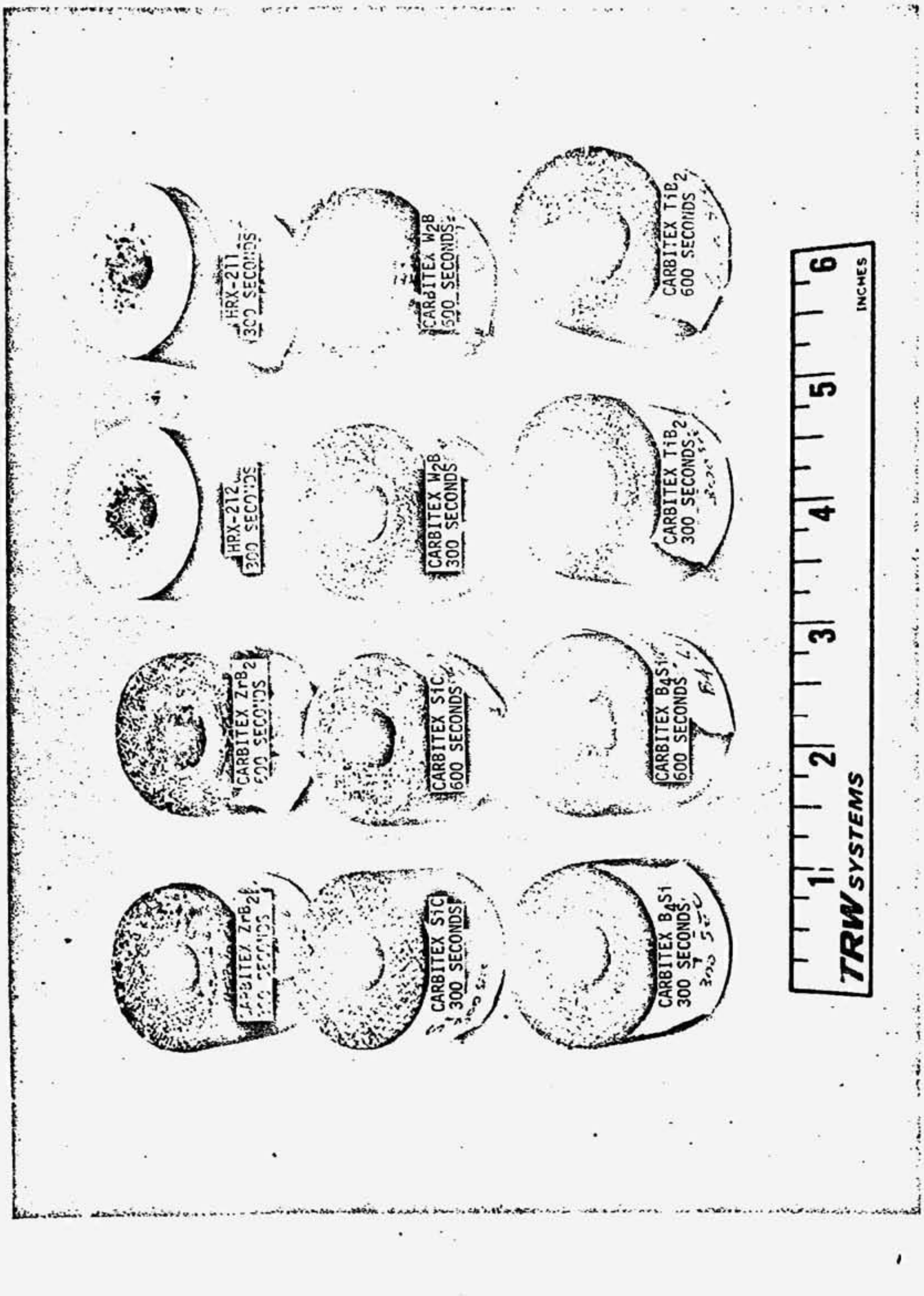

Figure 7. Pyrolyzed Laminate Materials after Torch Test

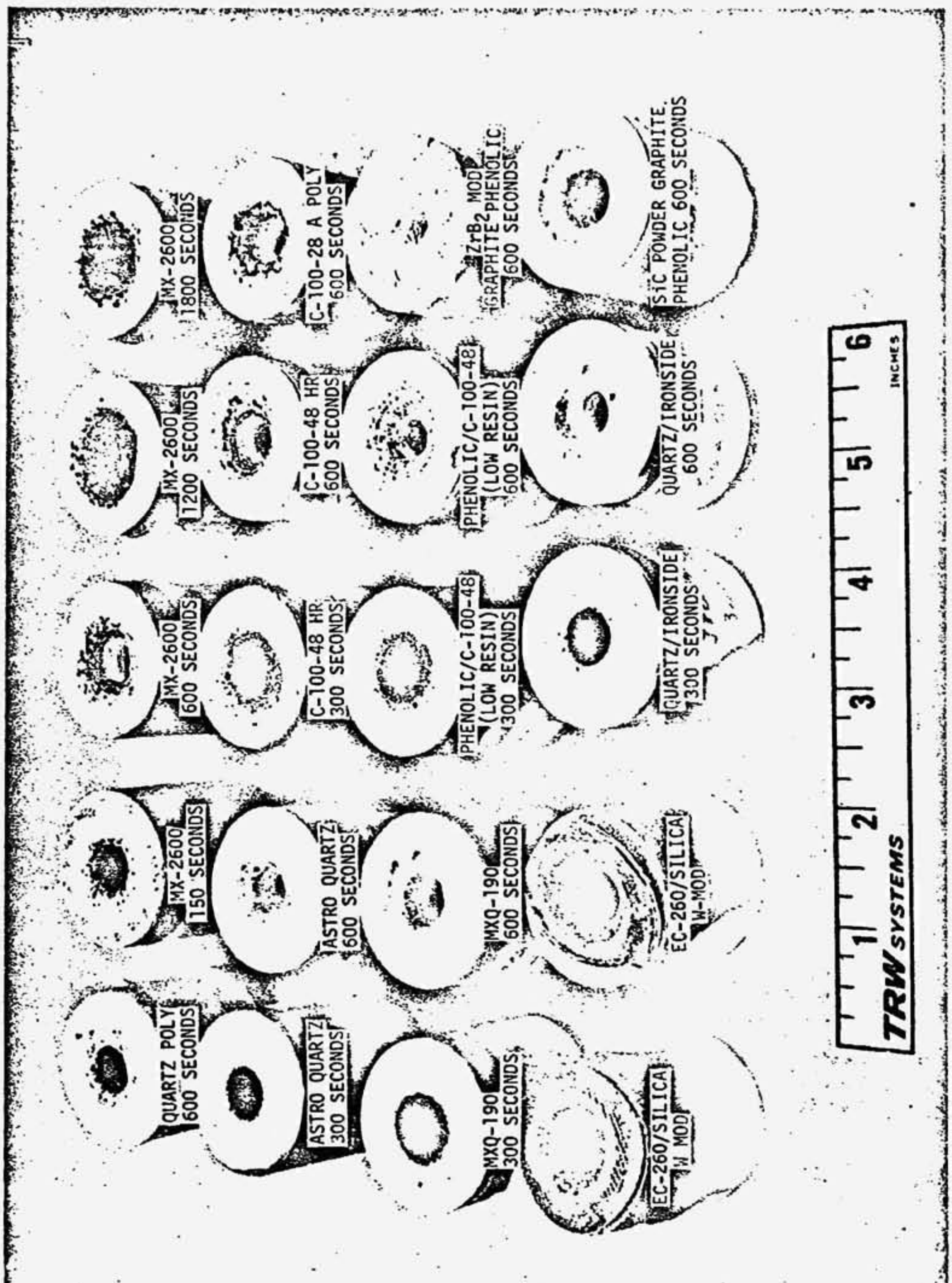

Figure 8. Resin-Base Laminate Materials after Torch Test

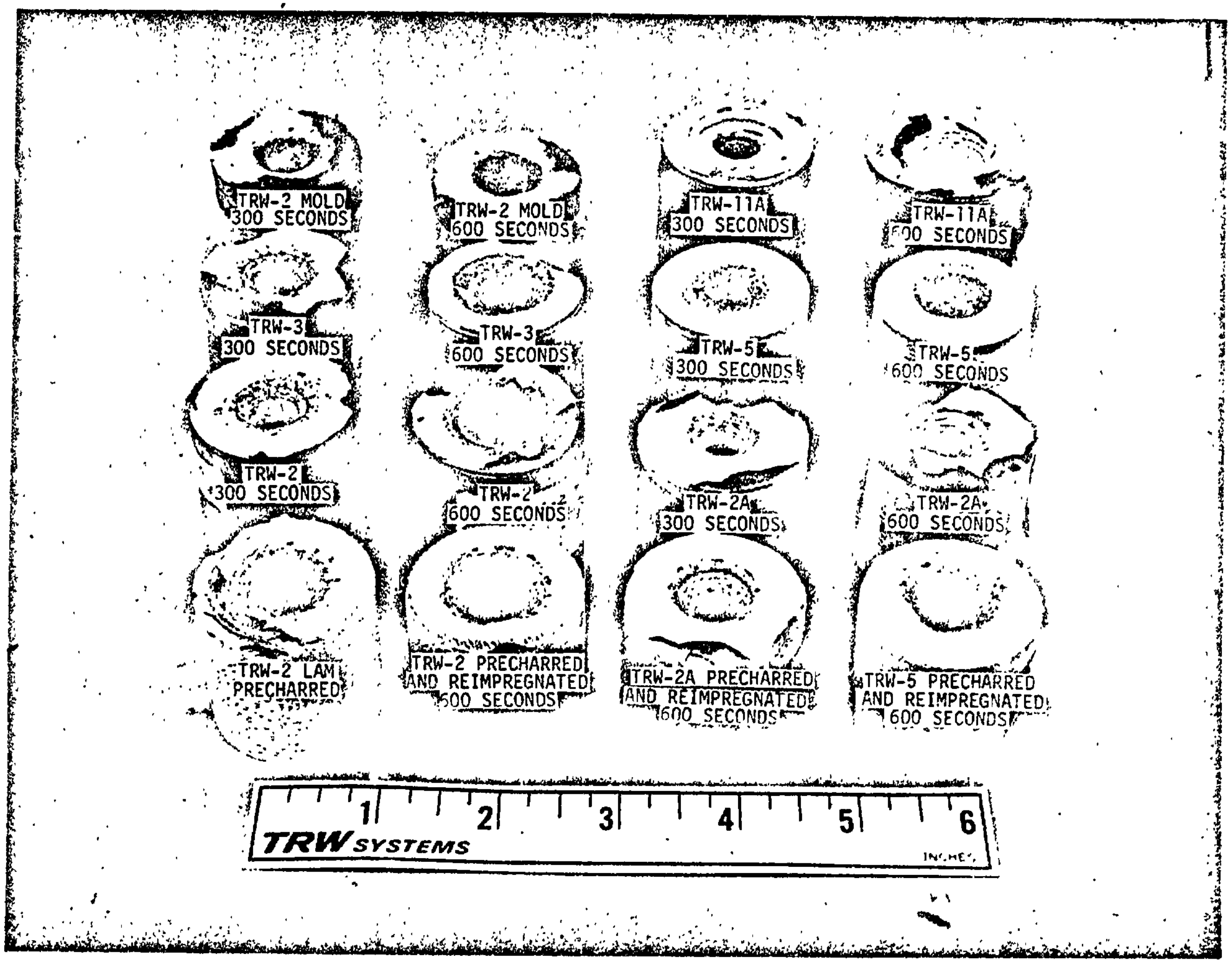

Figure 9. TRW Laminate Formulations after Torch Test

Figure 10. ZrB_2 V (Manlabs) 1500 Second Test (2X)

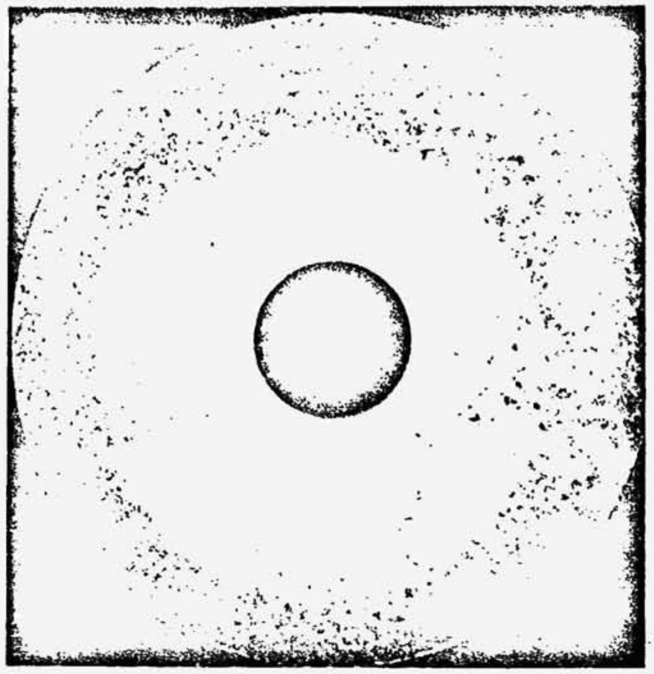

Figure 11. ZrB_2 VIII (Manlabs) 1500 Second Test (2X)

Figure 12. Zirconia Bonded Zirconia 600 Second Test (2X)

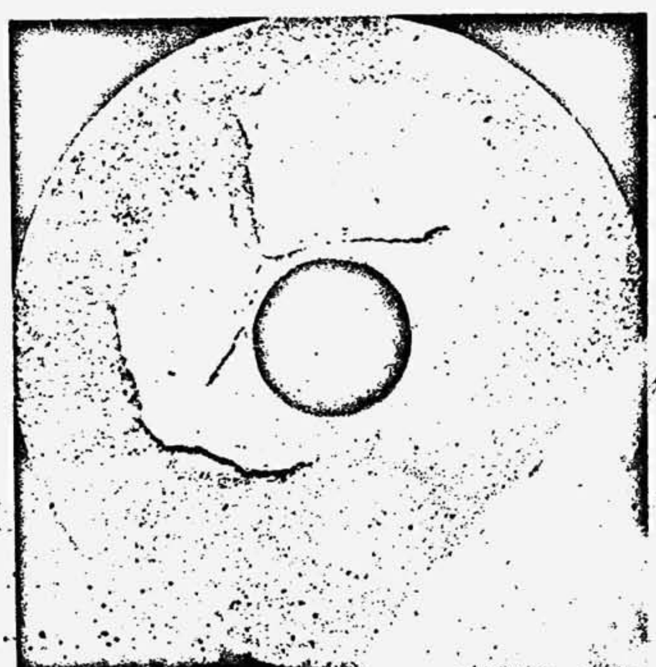

Figure 13. [illegible] 600 [illegible]

Figure 14. SiC Coated Carbone Graphite 600 Second Test (2X)

Figure 15. HRX-211 300 Second Test (2X)

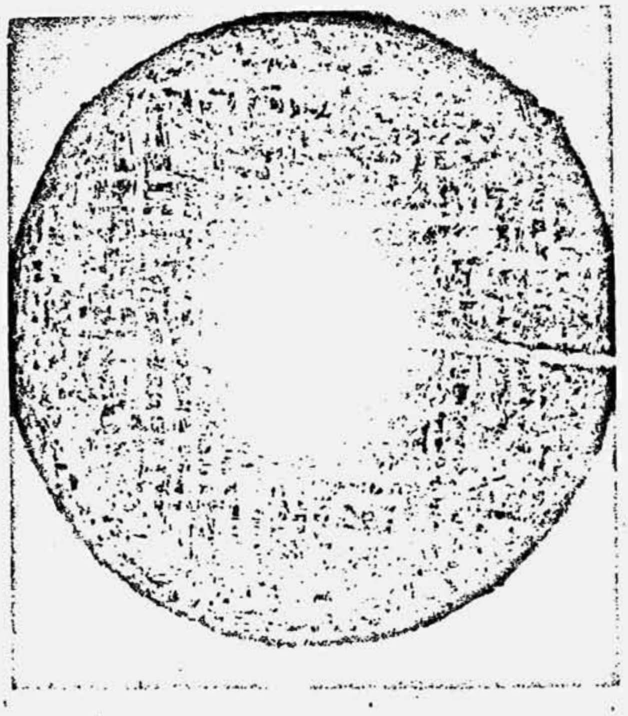

Figure 16. Carbitex 100, SiC Added 600 Second Test (2X)

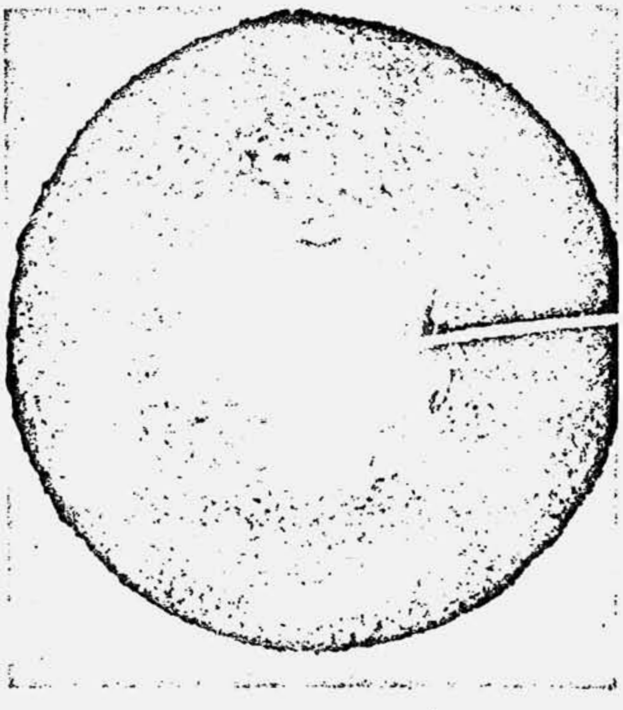

Figure 17. Carbitex 100, B_4Si Added 600 Second Test (2X)

Figure 18. MX-2600 300 Second Test (2X)

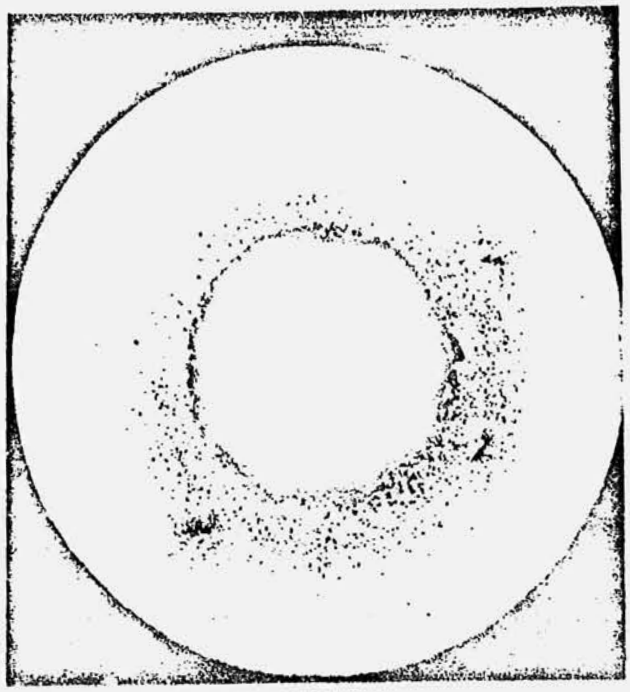

Figure 19. MXQ-190 300 Second Test (2X)

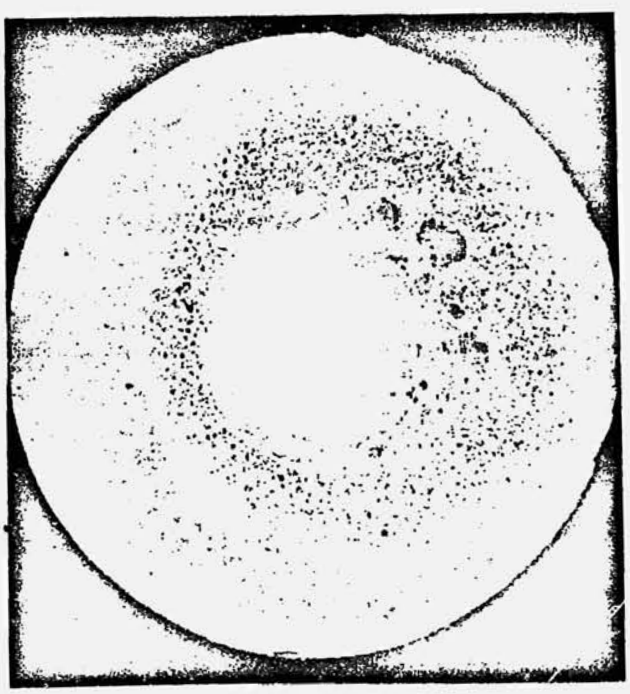

Figure 20. MXQ-190 600 Second Test (2X)

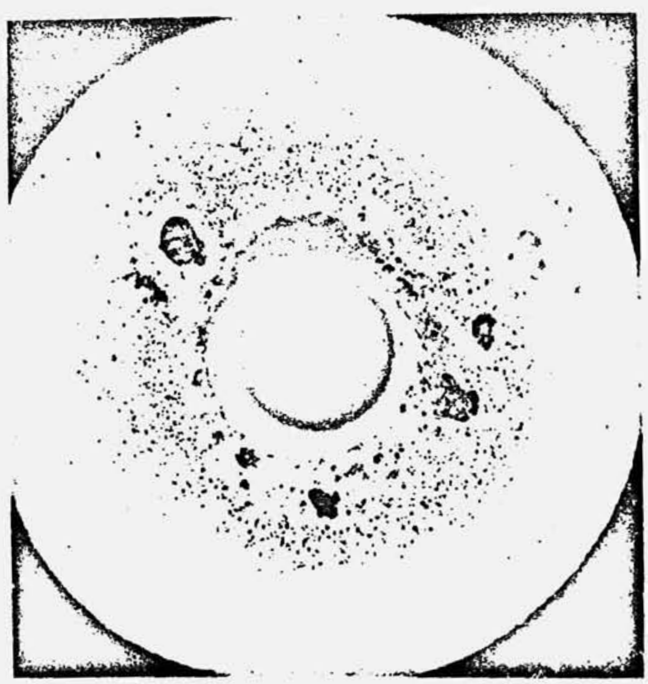

Figure 21. Astro Quartz/Ironside Resin 600 Second Test (2X)

Figure 22. Astro Quartz/EC-260 600 Second Test (2X)

Figure 23. Graphite/SC-1008 Phenolic Resin ZrB_2 Modified, 6[illegible] Second Test (2X)

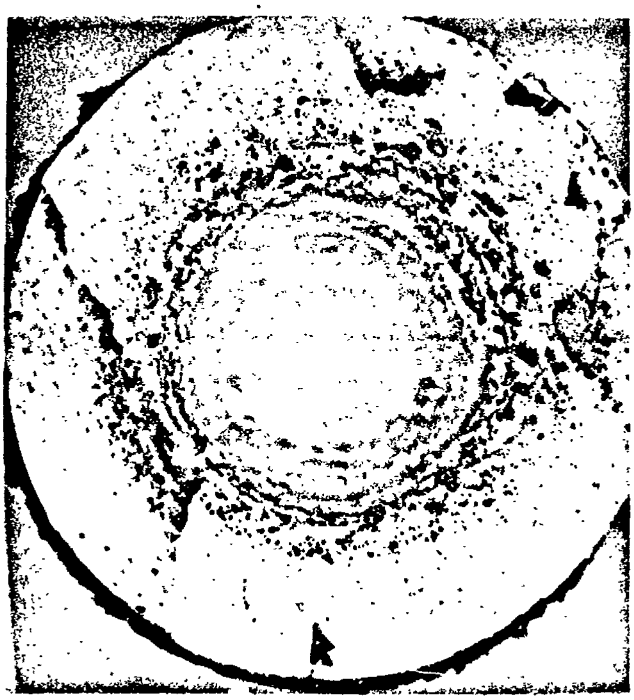

Figure 24. TRW-2 Laminate 600 Second Test (2X)

Figure 25. TRW-2 Mold 600 Second Test (2X)

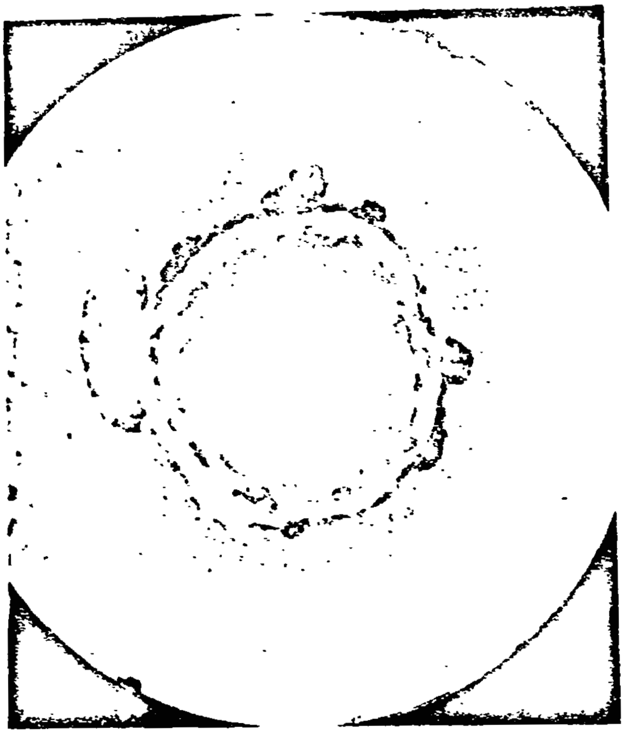

Figure 26. TRW-2A Laminate 600
Second Test (2X)

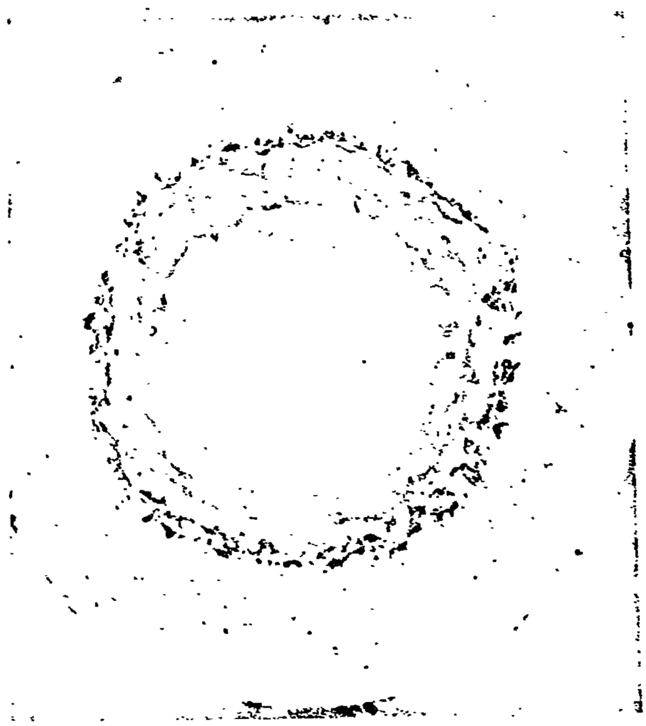

Figure 27. TRW-3 Laminate 600
Second Test (2X)

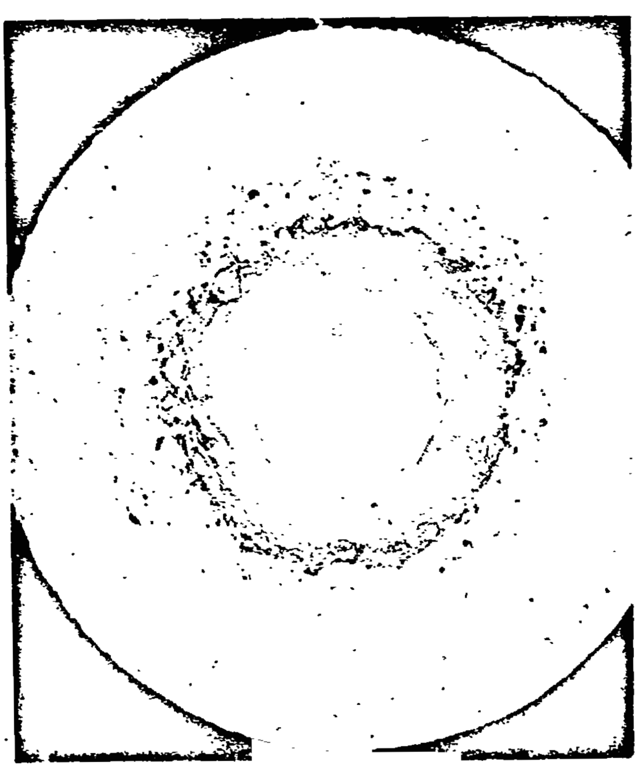

Figure 28. TRW-5 Laminate 600
Second Test (2X)

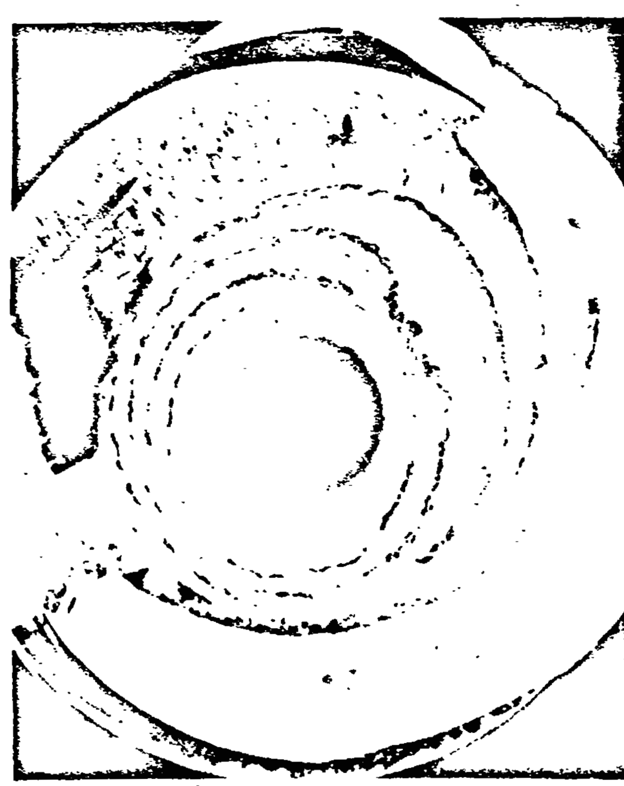

Figure 29. TRW-11A Laminate 600
Second Test (2X)

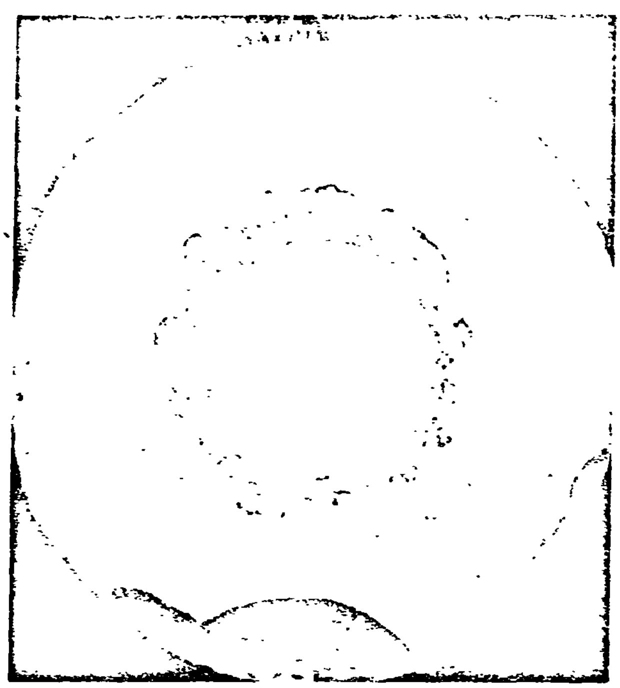

Figure 30. TRW-2A Precharred and Reimpregnated 600 Second Test (2X)

Figure 31. TRW-5 Precharred and Reimpregnated 600 Second Test (2X)

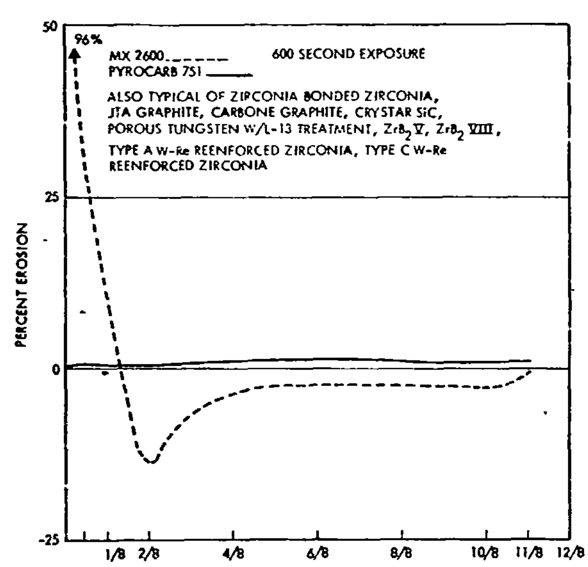

Figure 32. Erosion Profile of "Hard Throat" Materials

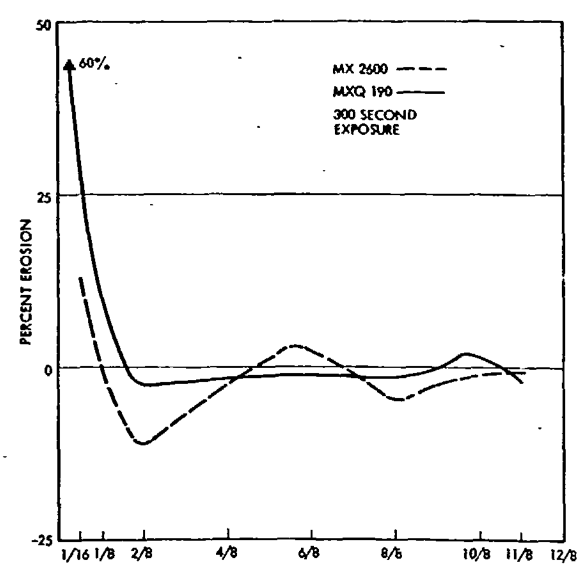

Figure 33. Erosion Profile of MXQ-190 300 Second Test

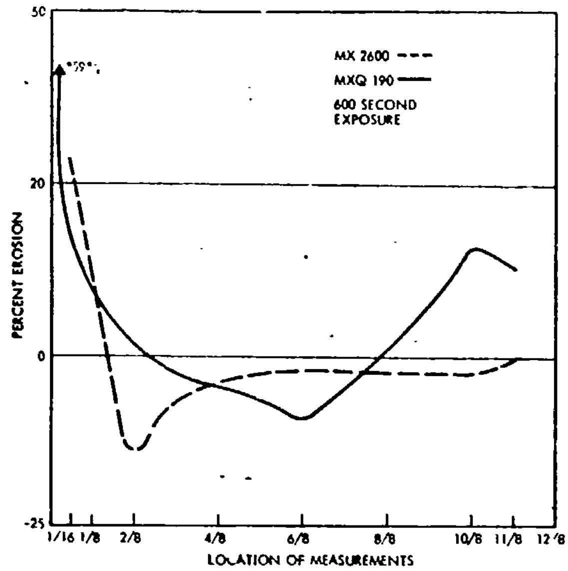

Figure 34. Erosion Profile of MXQ-190 600 Second Test

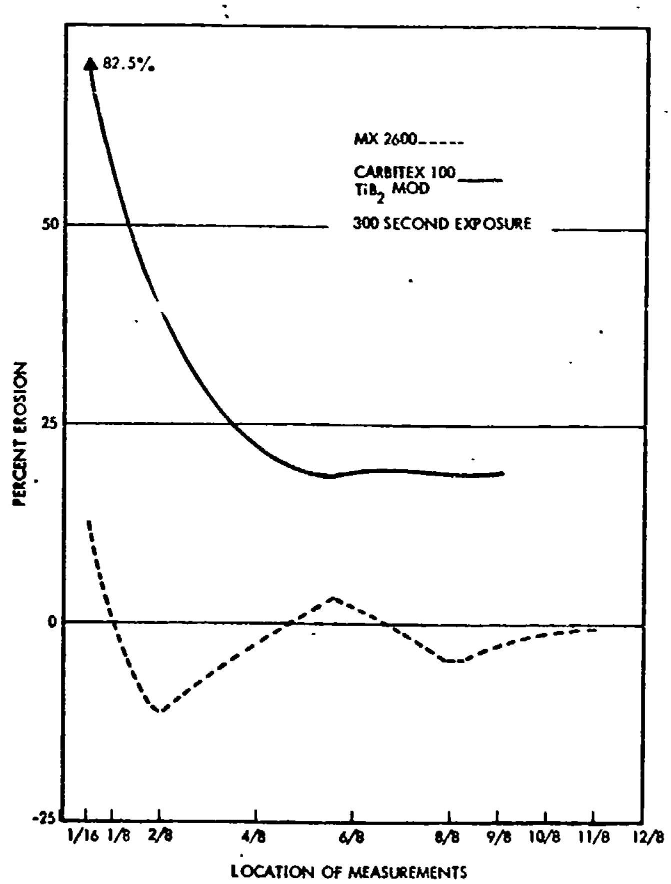

Figure 35. Erosion Profile of Carbitex (TiB_2) 300 Second Test

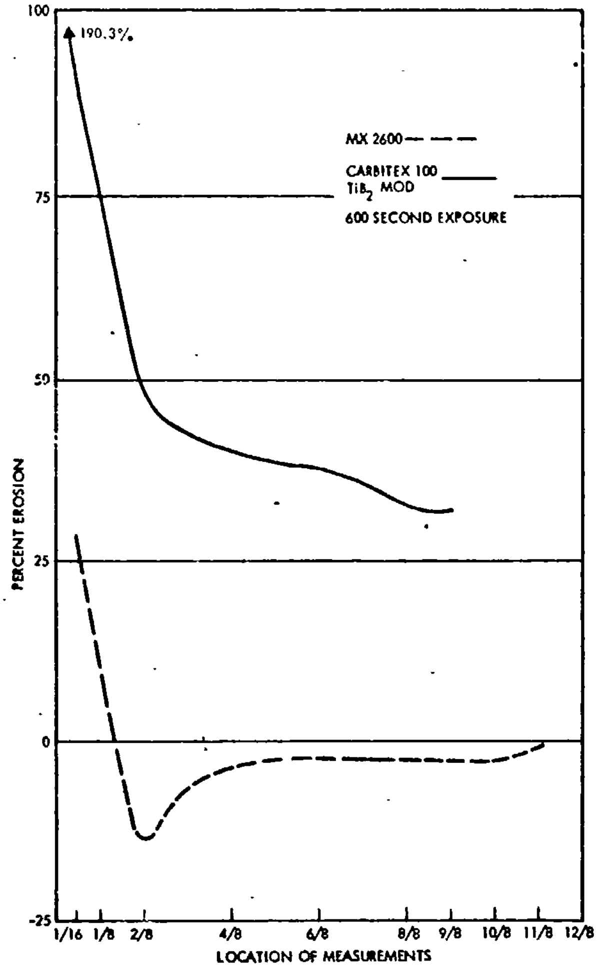

Figure 36. Erosion Profile of Carbitex (TiB_2) 600 Second Test

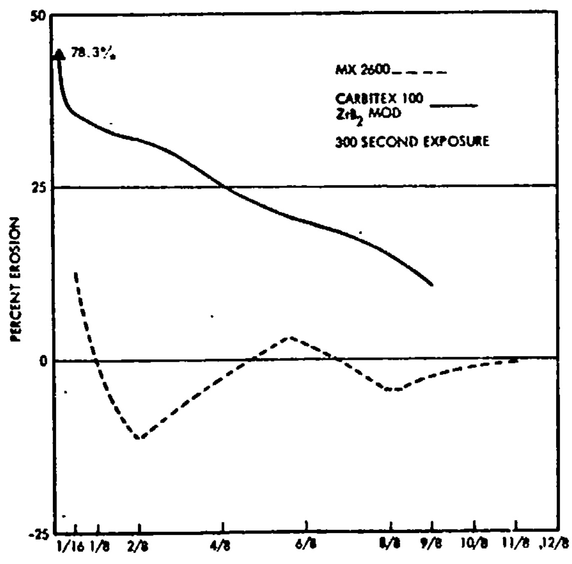

Figure 37. Erosion Profile of Carbitex (ZrB_2) 300 Second Test

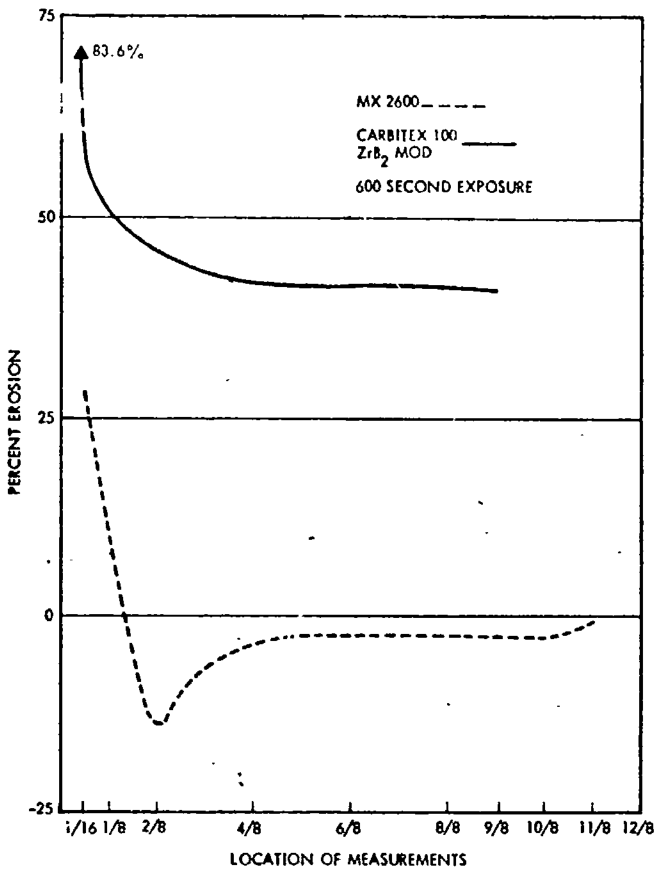

Figure 38. Erosion Profile of Carbitex (ZrB_2) 600 Second Test

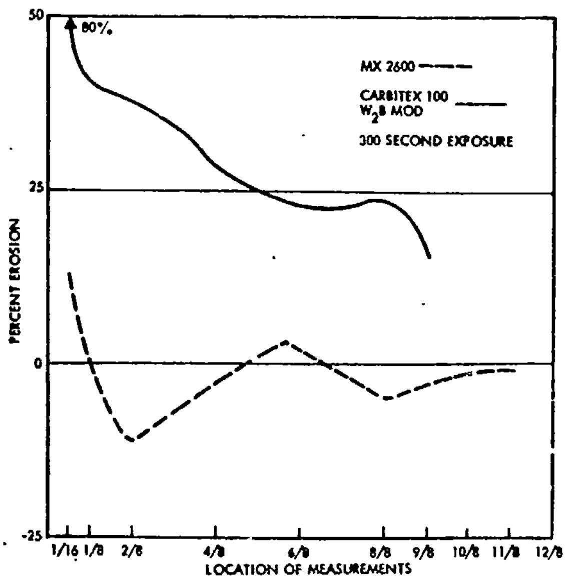

Figure 39. Erosion Profile of Carbitex (W_2B) 300 Second Test

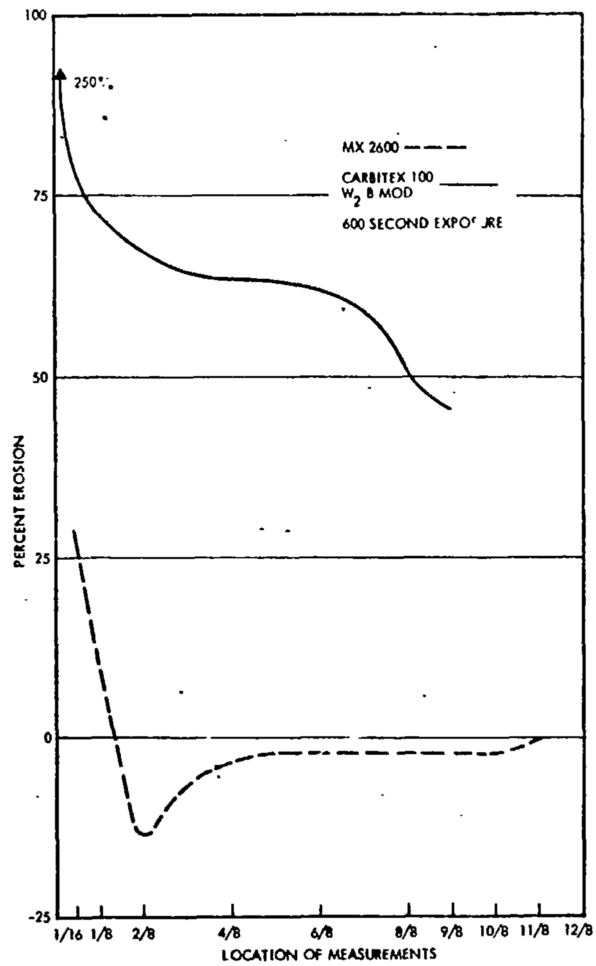

Figure 40. Erosion Profile of Carbitex (W_2B) 600 Second Test

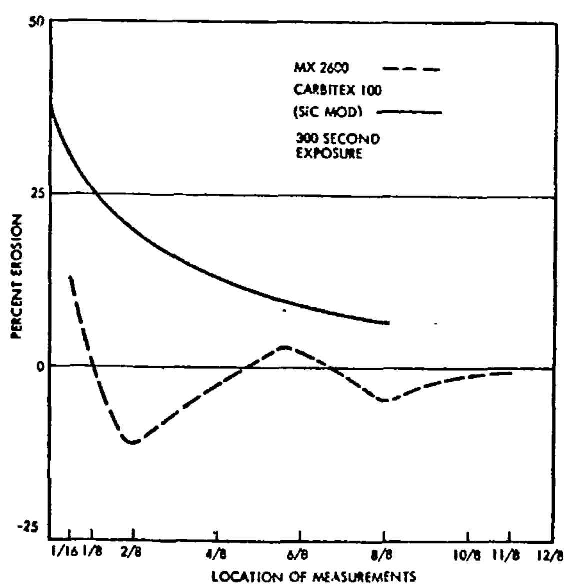

Figure 41. Erosion Profile of Carbitex (SiC) 300 Second Test

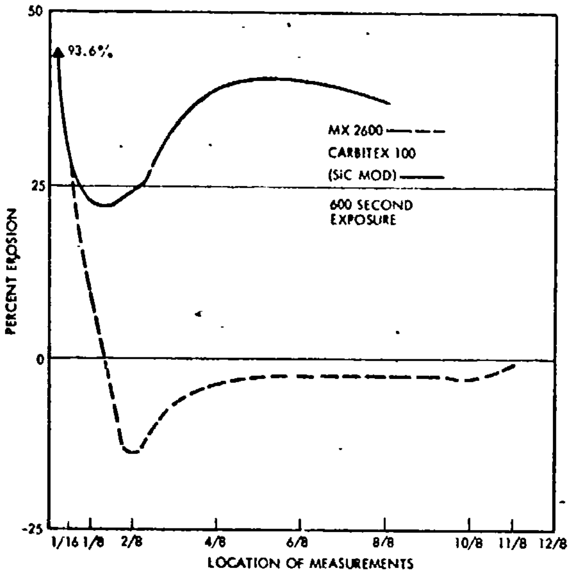

Figure 42. Erosion Profile of Carbitex (SiC) 600 Second Test

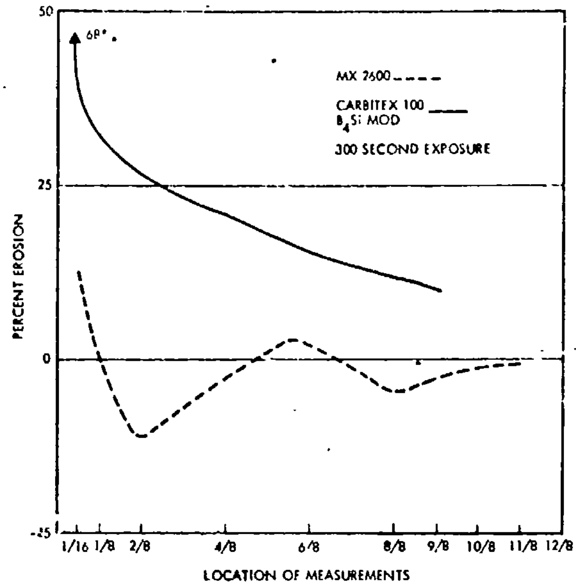

Figure 43. Erosion Profile of Carbitex (B_4Si) 300 Second Test

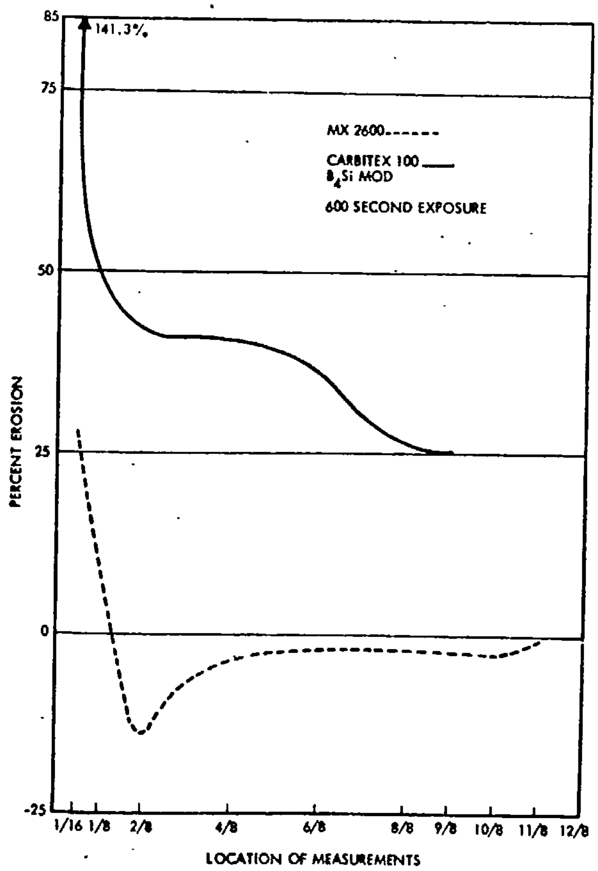

Figure 44. Erosion Profile of Carbitex (B_4Si) 600 Second Test

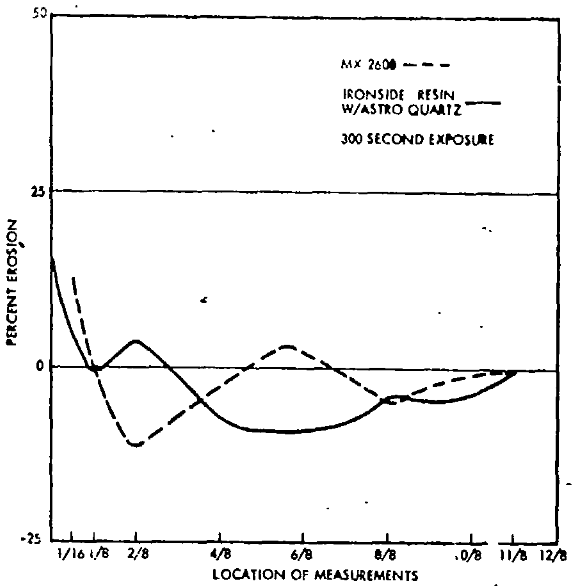

Figure 45. Erosion Profile of Quartz/Ironside Resin 300 Second Test

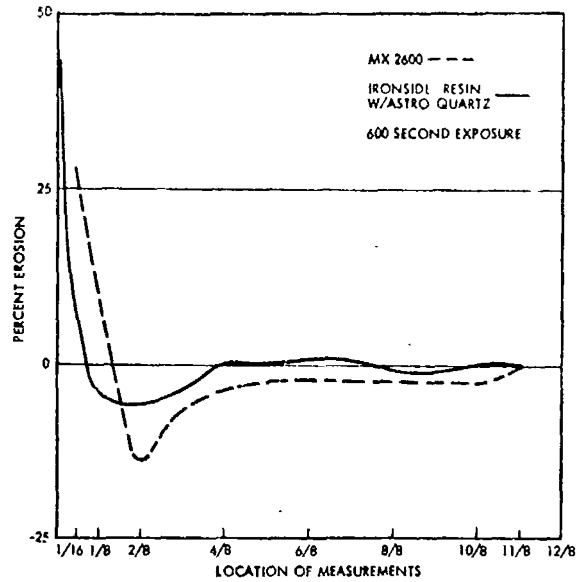

Figure 46. Erosion Profile of Quartz/Ironside Resin 600 Second Test

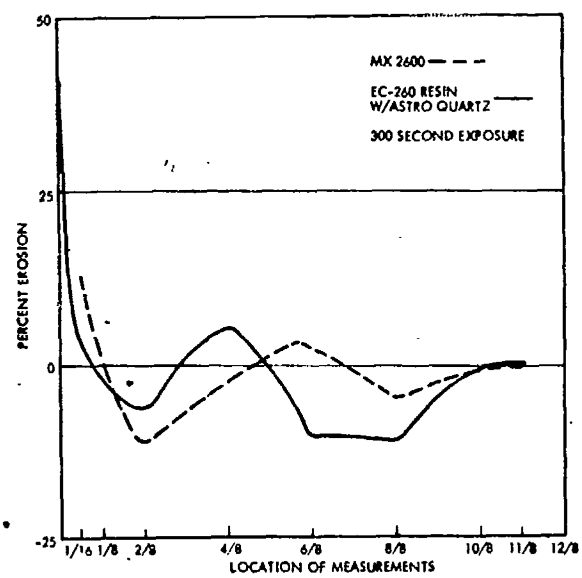

Figure 47. Erosion Profile of Quartz/EC-260 300 Second Test

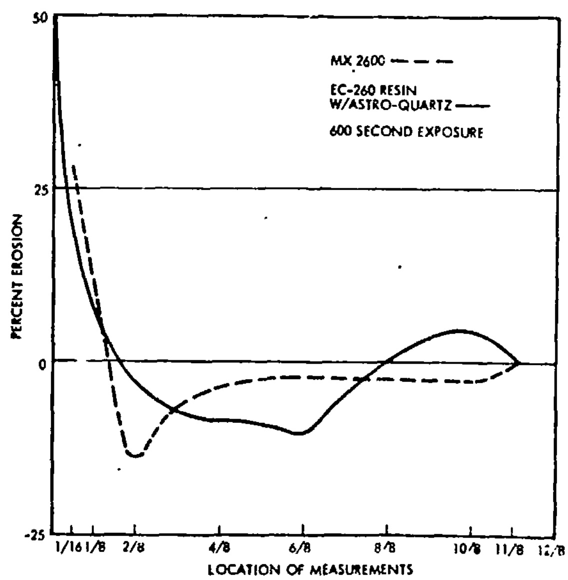

Figure 48. Erosion Profile of Quartz/EC-260 600 Second Test

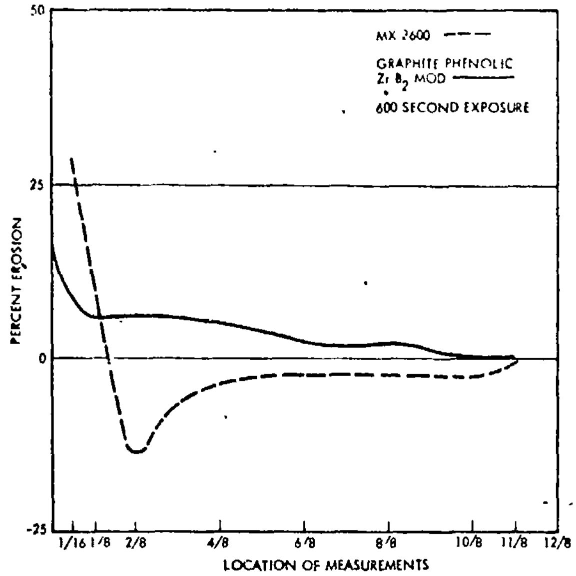

Figure 49. Erosion Profile of Graphite/Phenolic (ZrB_2) 600 Second Test

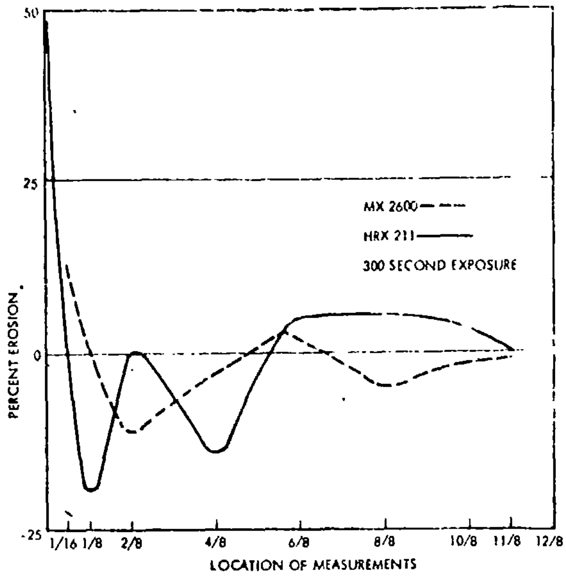

Figure 50. Erosion Profile of HRX-211 300 Second Test

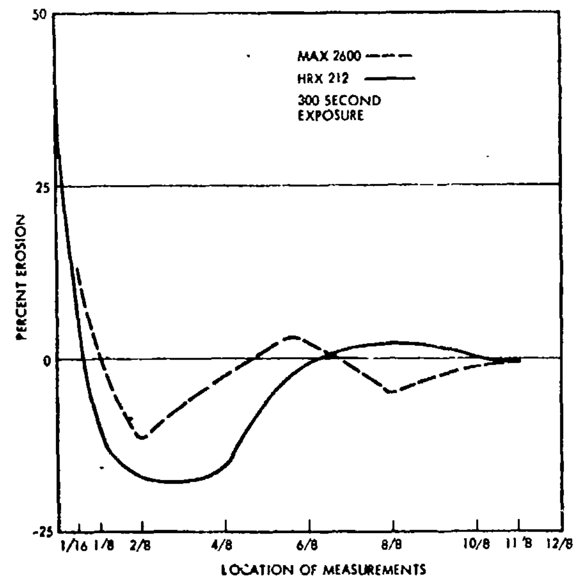

Figure 51. Erosion Profile of HRX-212 300 Second Test

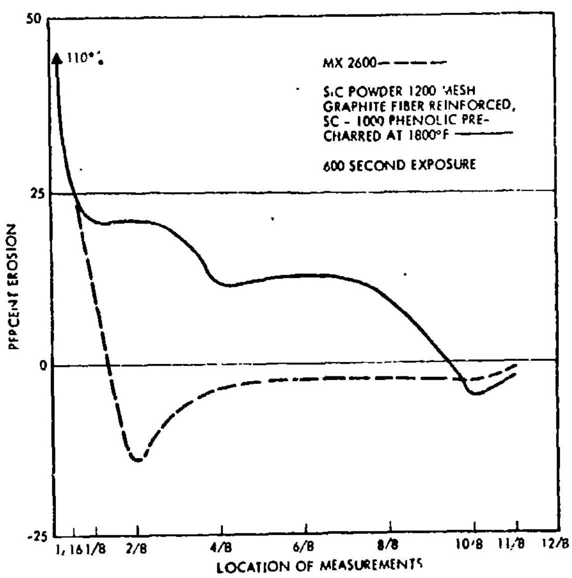

Figure 52. Erosion Profile of Graphite/Phenolic (SiC) Precharred 600 Second Test

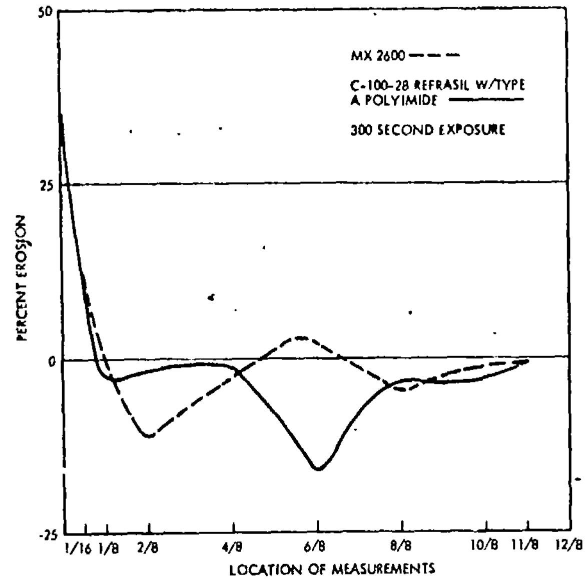

Figure 53. Erosion Profile of C-100-28 Refrasil/Polyimide 300 Second Test

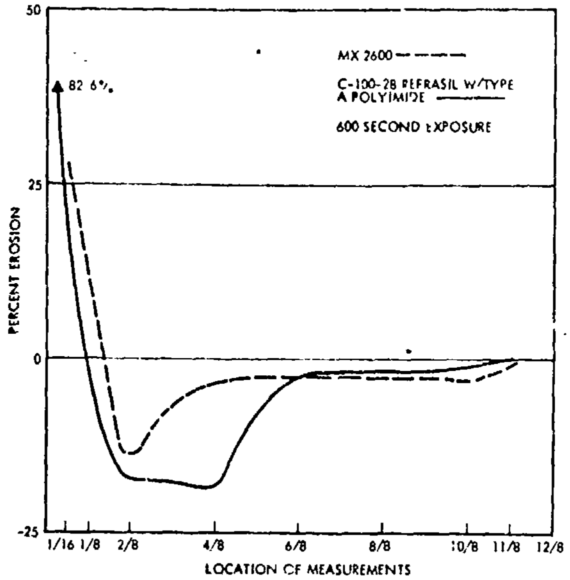

Figure 54. Erosion Profile of C-100-28 Refrasil/Polyimide 600 Second Test

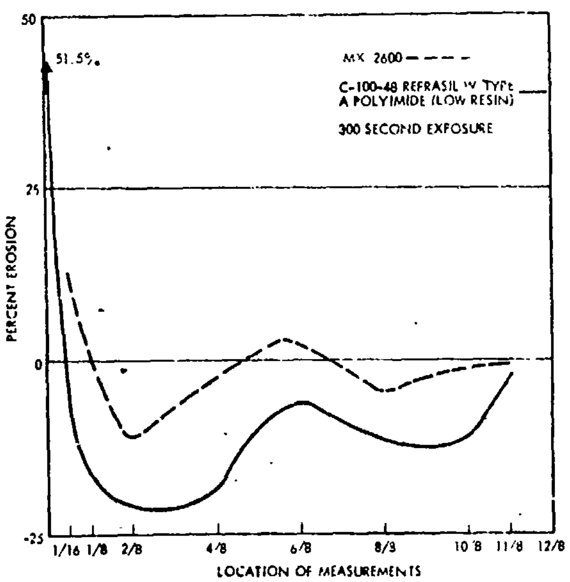

Figure 55. Erosion Profile of C-100-48 Refrasil/Polyimide (low resin) 300 Second Test

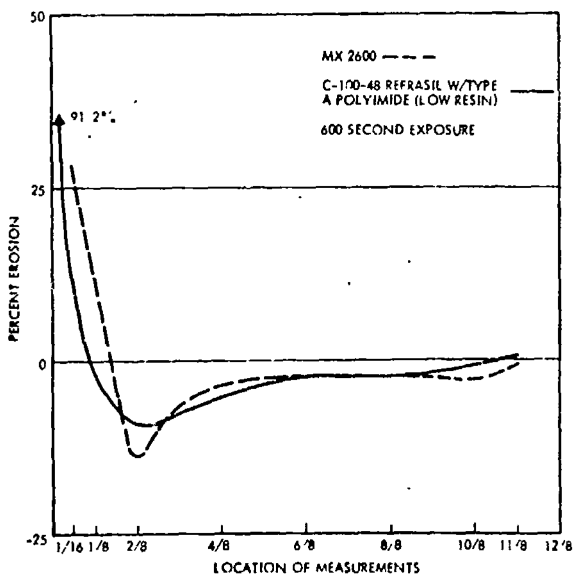

Figure 56. Erosion Profile of C-100-48 Refrasil/Polyimide (low resin) 600 Second Test

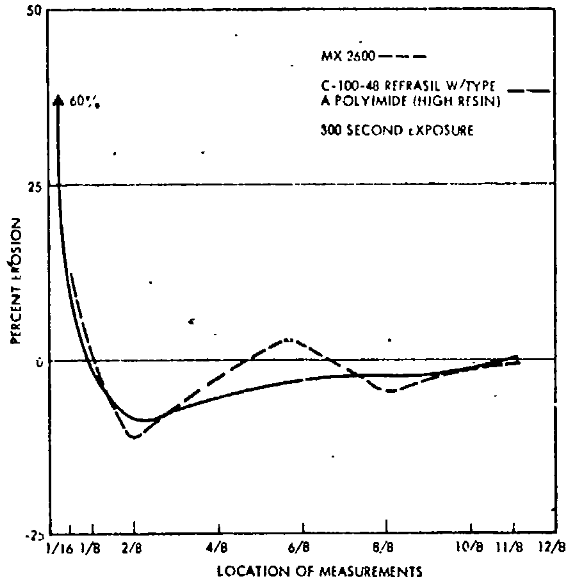

Figure 57. Erosion Profile of C-100-48 Refrasil/Polyimide (high resin) 300 Second Test

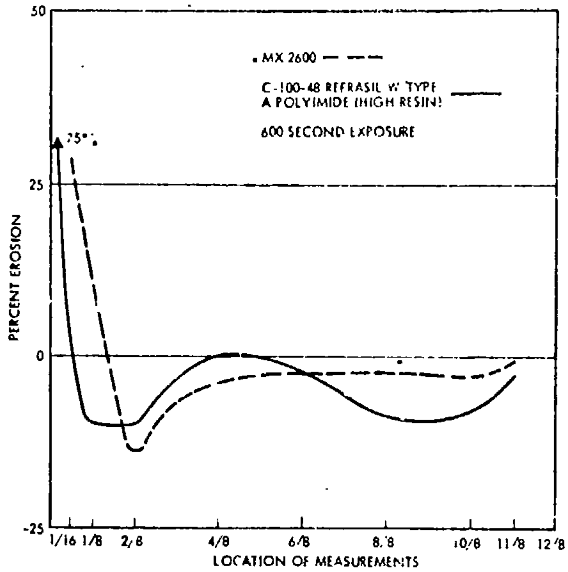

Figure 58. Erosion Profile of C-100-48 Refrasil/Polyimide (high resin) 600 Second Test

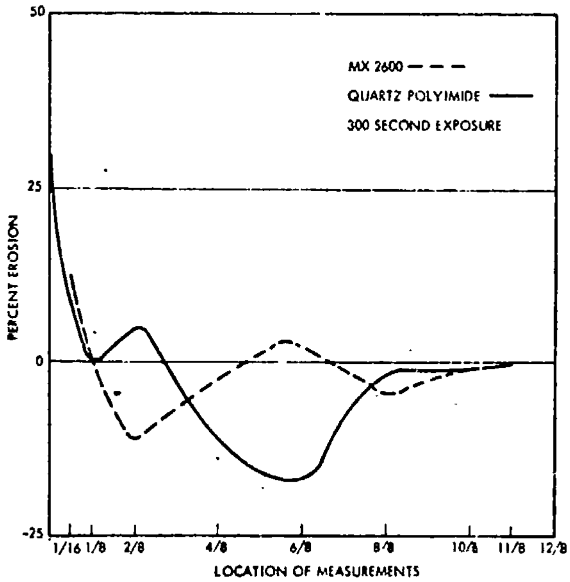

Figure 59. Erosion Profile of Quartz/Polyimide 300 Second Test

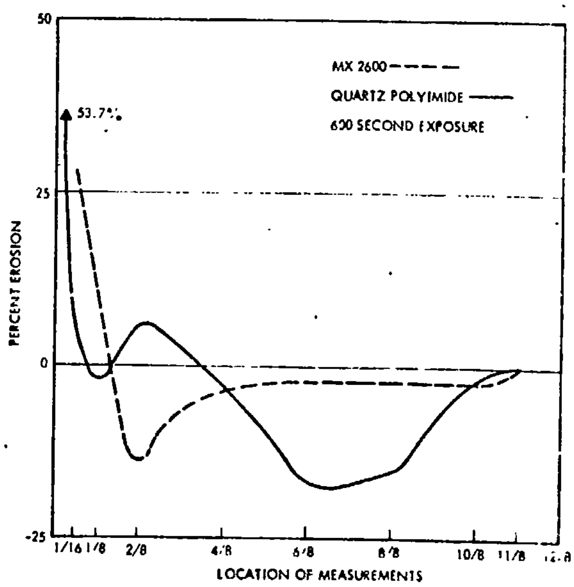

Figure 60. Erosion Profile of Quartz/Polyimide 600 Second Test

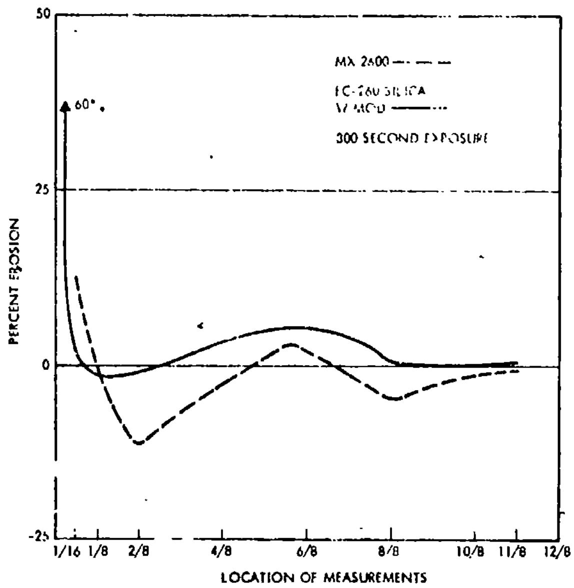

Figure 61. Erosion Profile of Silica/ EC-260 (with modification) 300 Second Test

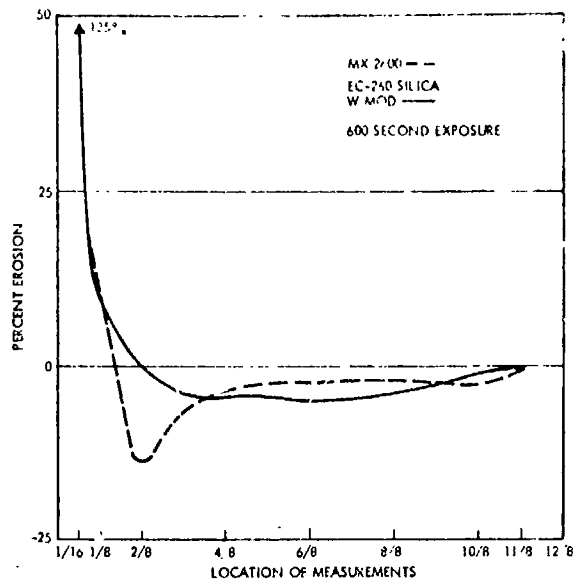

Figure 62. Erosion Profile of Silica/ EC-260 (with modification) 600 Second Test

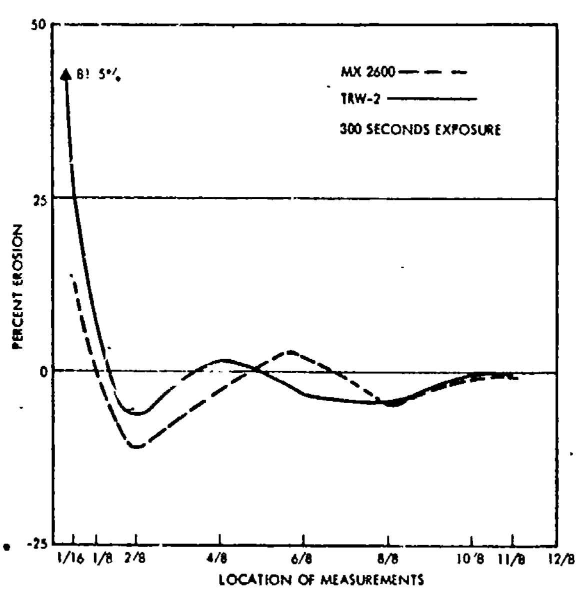

Figure 63. Erosion Profile of TRW-2 300 Second Test

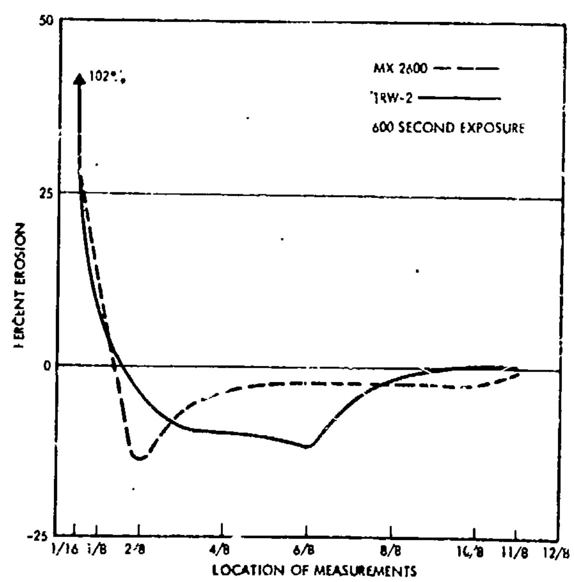

Figure 64. Erosion Profile of TRW-2 600 Second Test

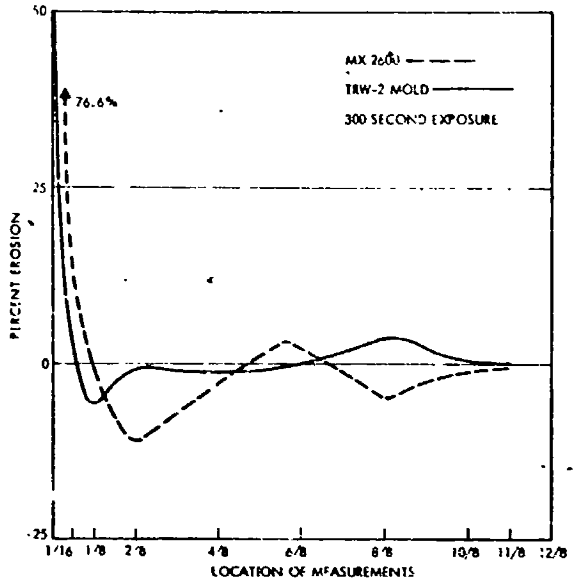

Figure 65. Erosion Profile of TRW-2 Mold 300 Second Test

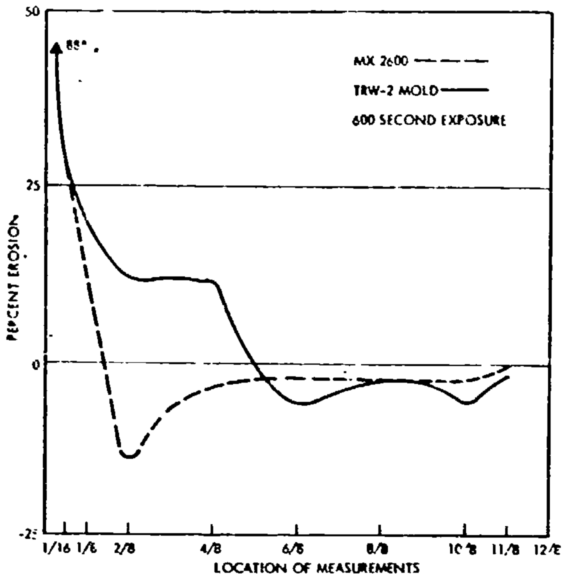

Figure 66. Erosion Profile of TRW-2 Mold 600 Second Test

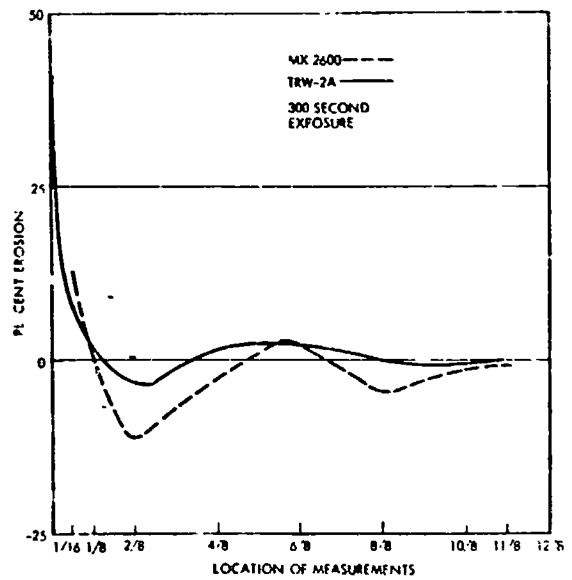

Figure 67. Erosion Profile of TRW-2A 300 Second Test

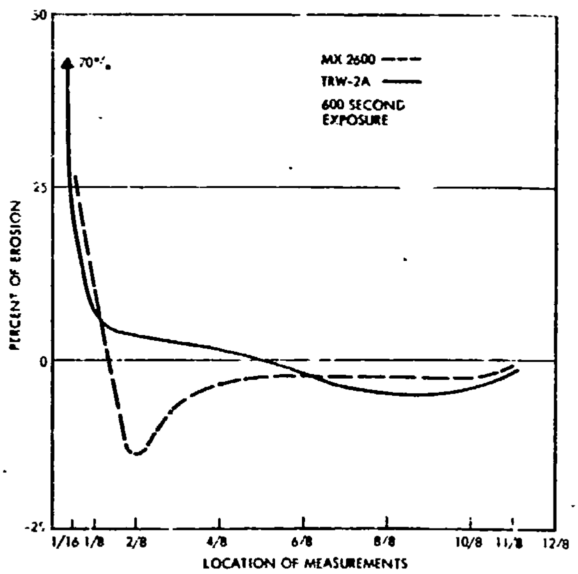

Figure 68. Erosion Profile of TRW-2A 600 Second Test

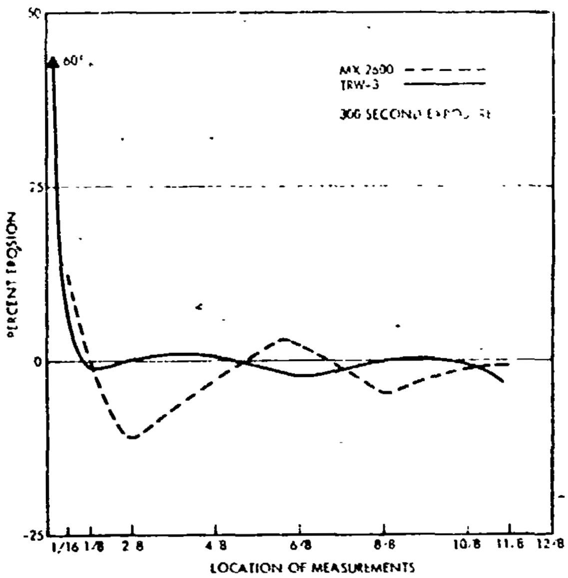

Figure 69. Erosion Profile of TRW-3 300 Second Test

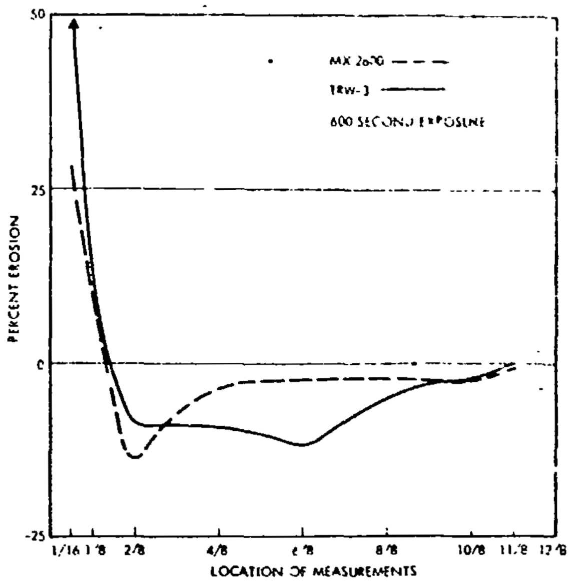

Figure 70. Erosion Profile of TRW-3 600 Second Test

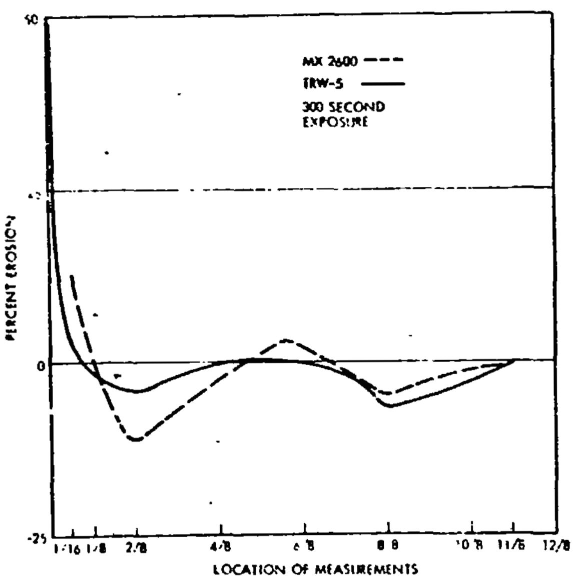

Figure 71. Erosion Profile of TRW-5 300 Second Test

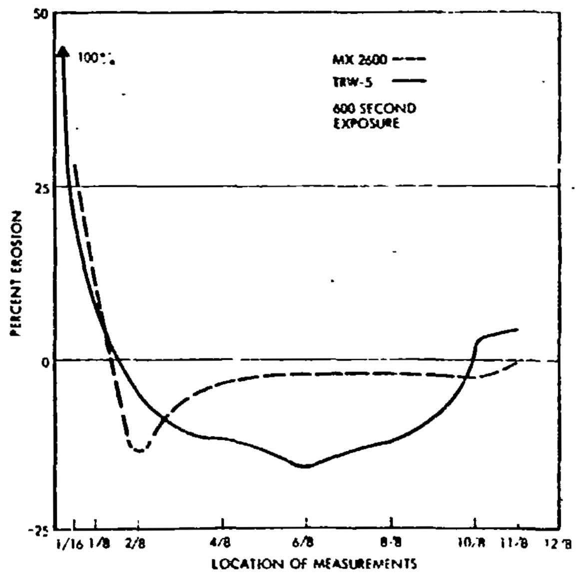

Figure 72. Erosion Profile of TRW-5 600 Second Test

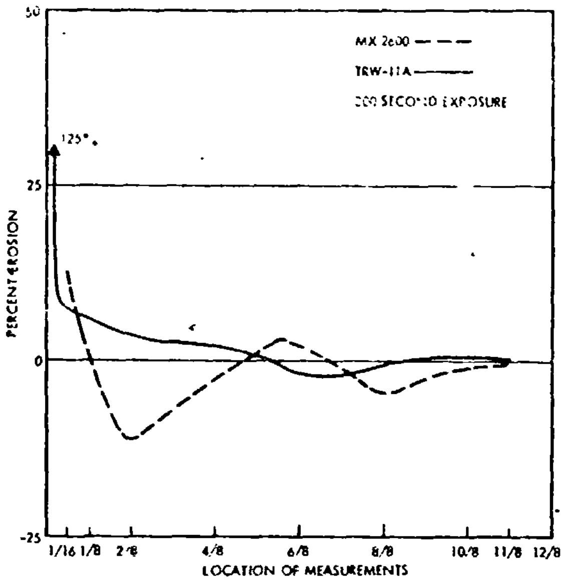

Figure 73. Erosion Profile of TRW-11A 300 Second Test

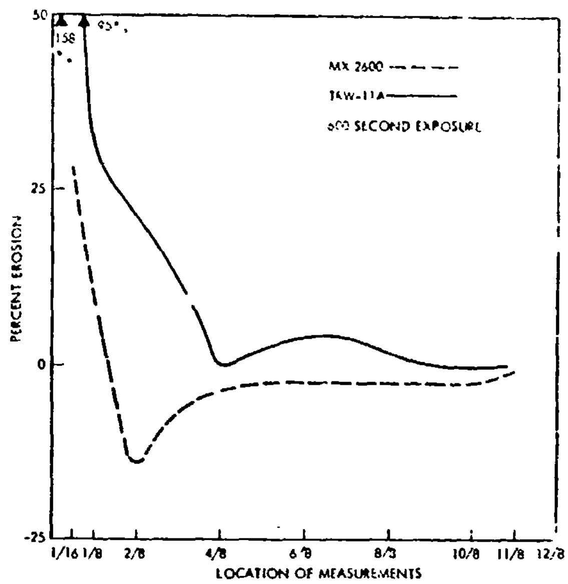

Figure 74. Erosion Profile of TRW-11A 600 Second Test

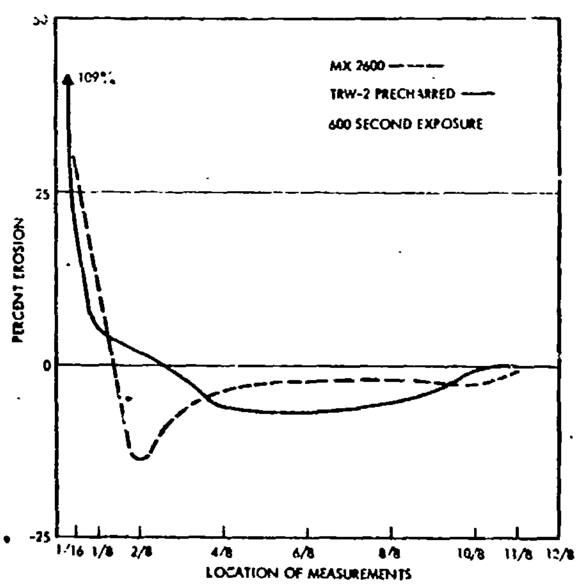

Figure 75. Erosion Profile of TRW-2 Precharred 600 Second Test

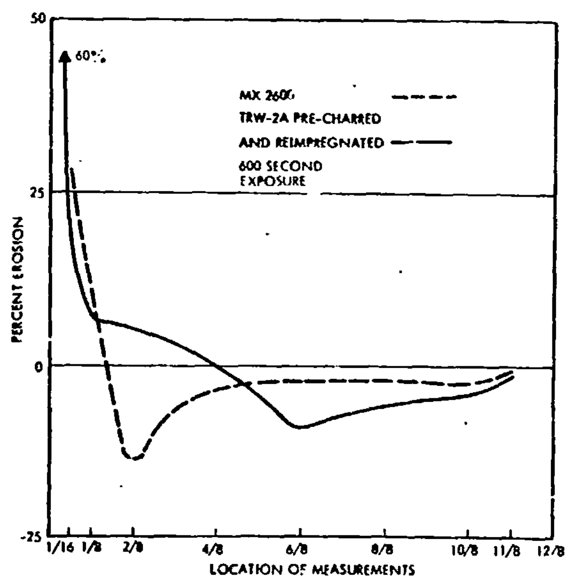

Figure 76. Erosion Profile of TRW-2A Precharred and Reimpregnated 600 Second Test

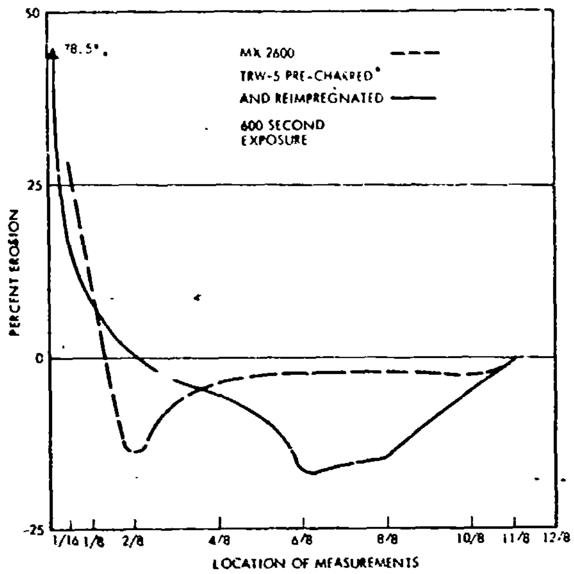

Figure 77. Erosion Profile of TRW-5 Precharred and Reimpregnated 600 Second Test

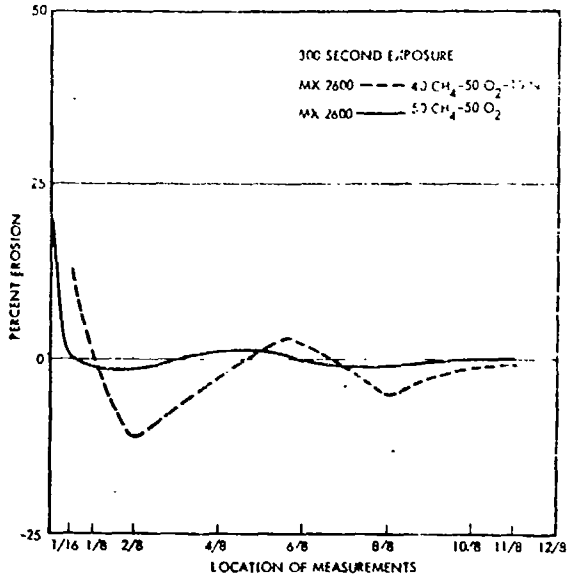

Figure 78. Erosion Profile of MX-2600 300 Second Test at Different Gas Mixtures

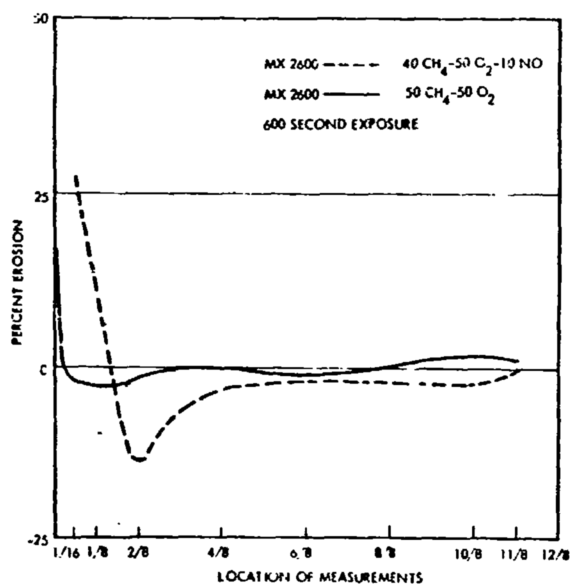

Figure 79. Erosion Profile of MX-2600 600 Second Test at Different Gas Mixtures

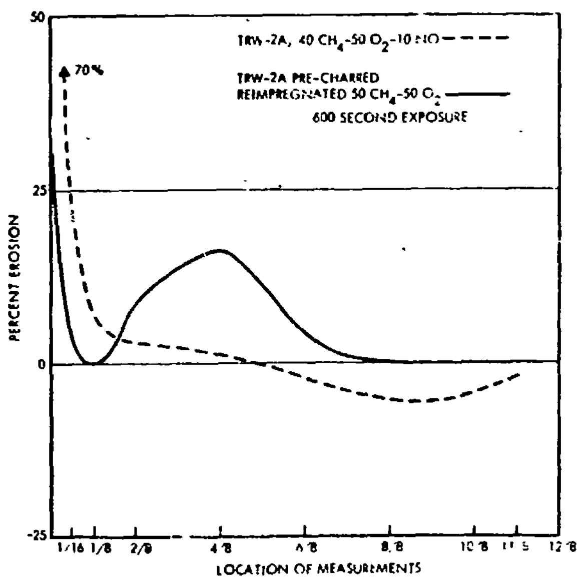

Figure 80. Erosion Profile of TRW-2A Treated, 600 Second Test at Different Gas Mixtures

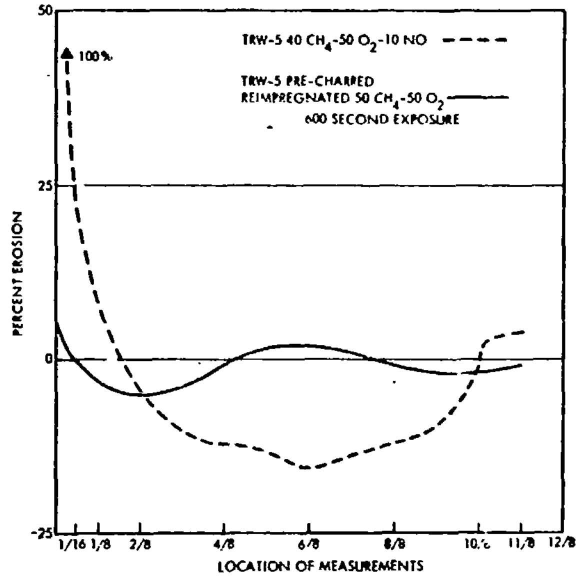

Figure 81. Erosion Profile of TRW-5 Treated, 600 Second Test at Different Gas Mixtures

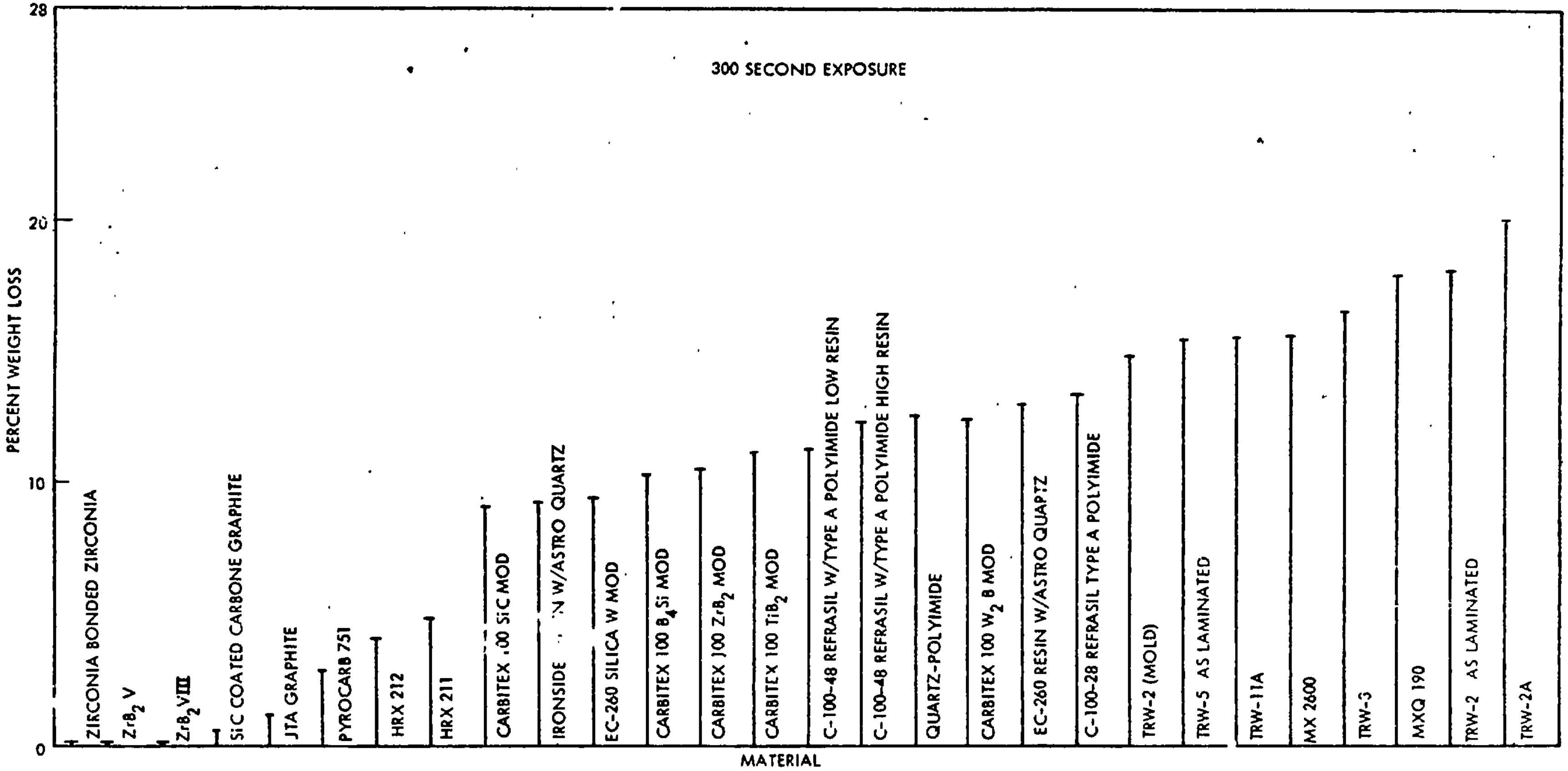

Figure 82. Weight Loss for Various Materials 300 Second Test

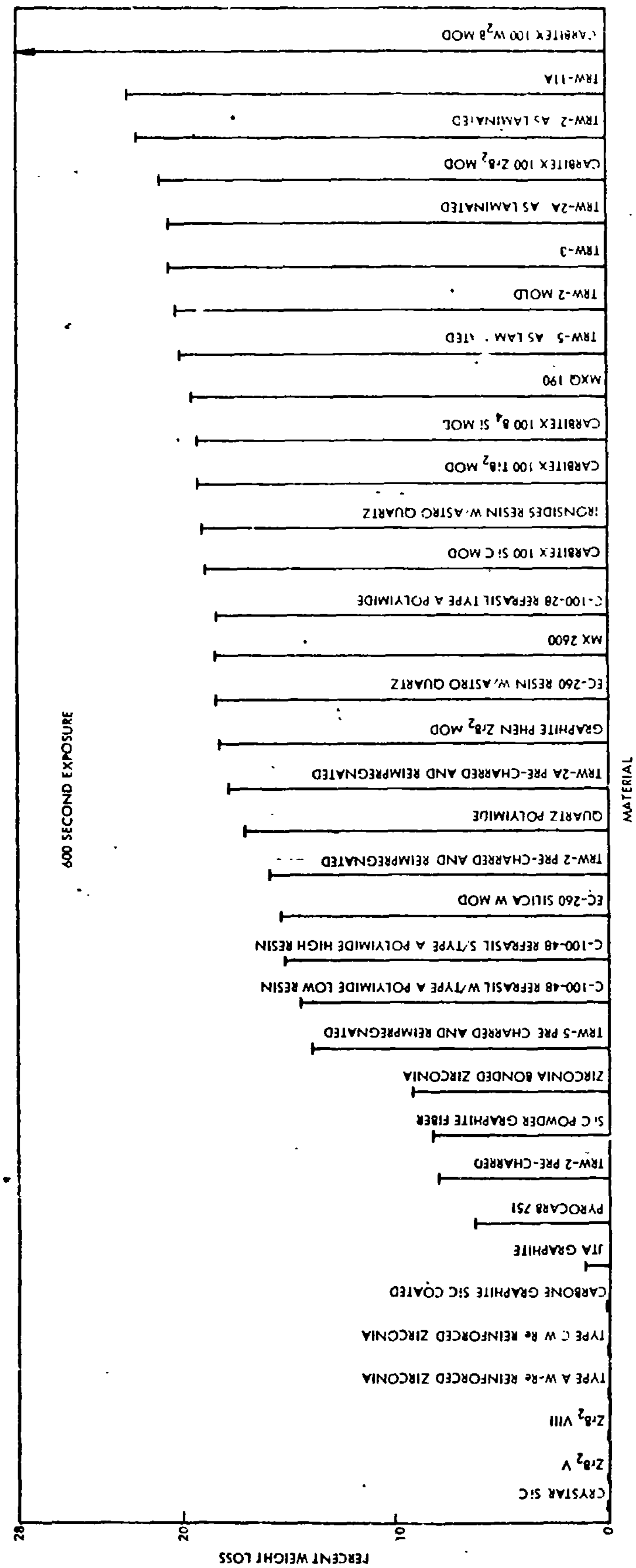

Figure 83. Weight Loss for Various Materials 600 Second Test

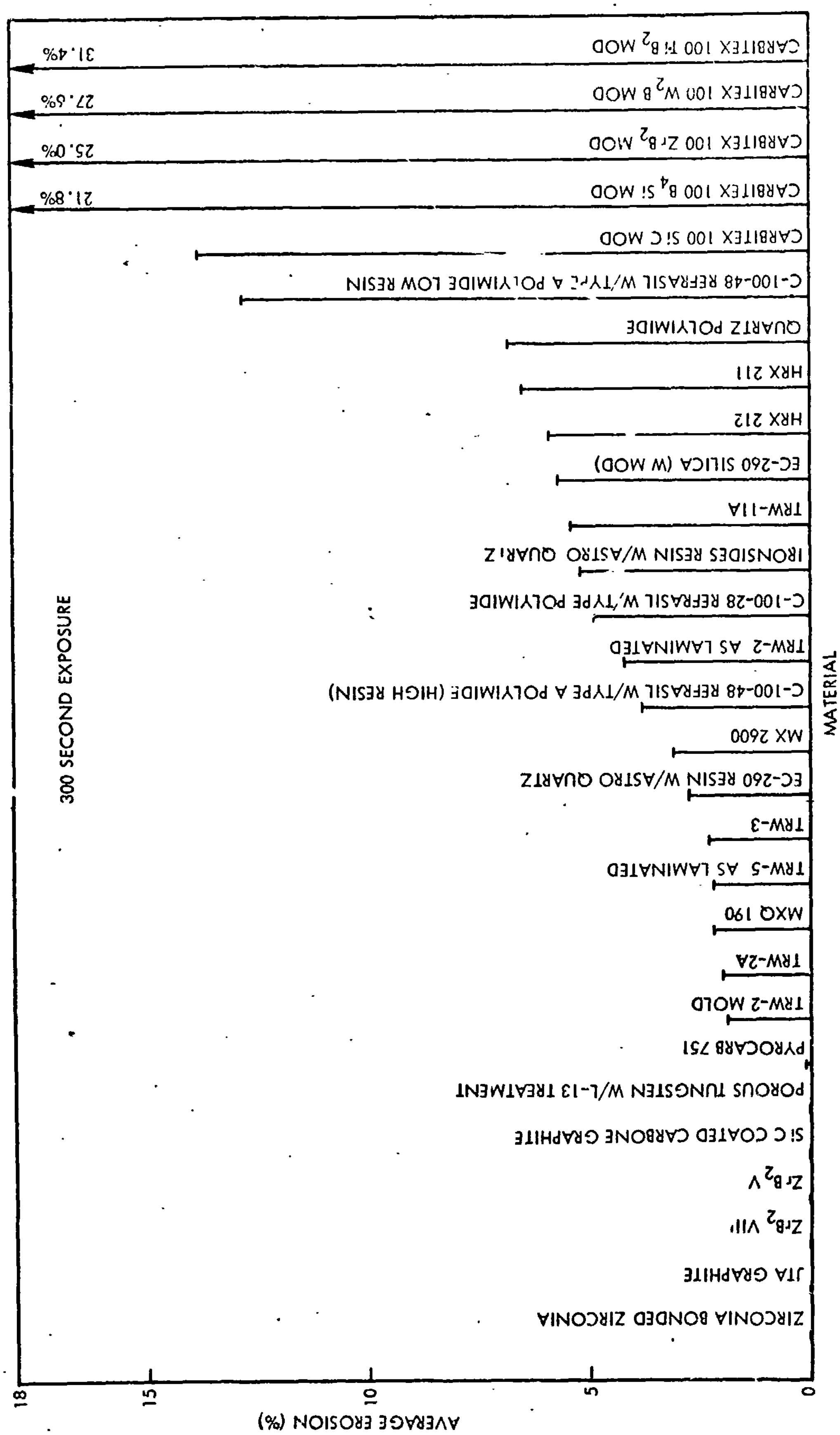

Figure 84. Percent Erosion for Various Materials 300 Second Test

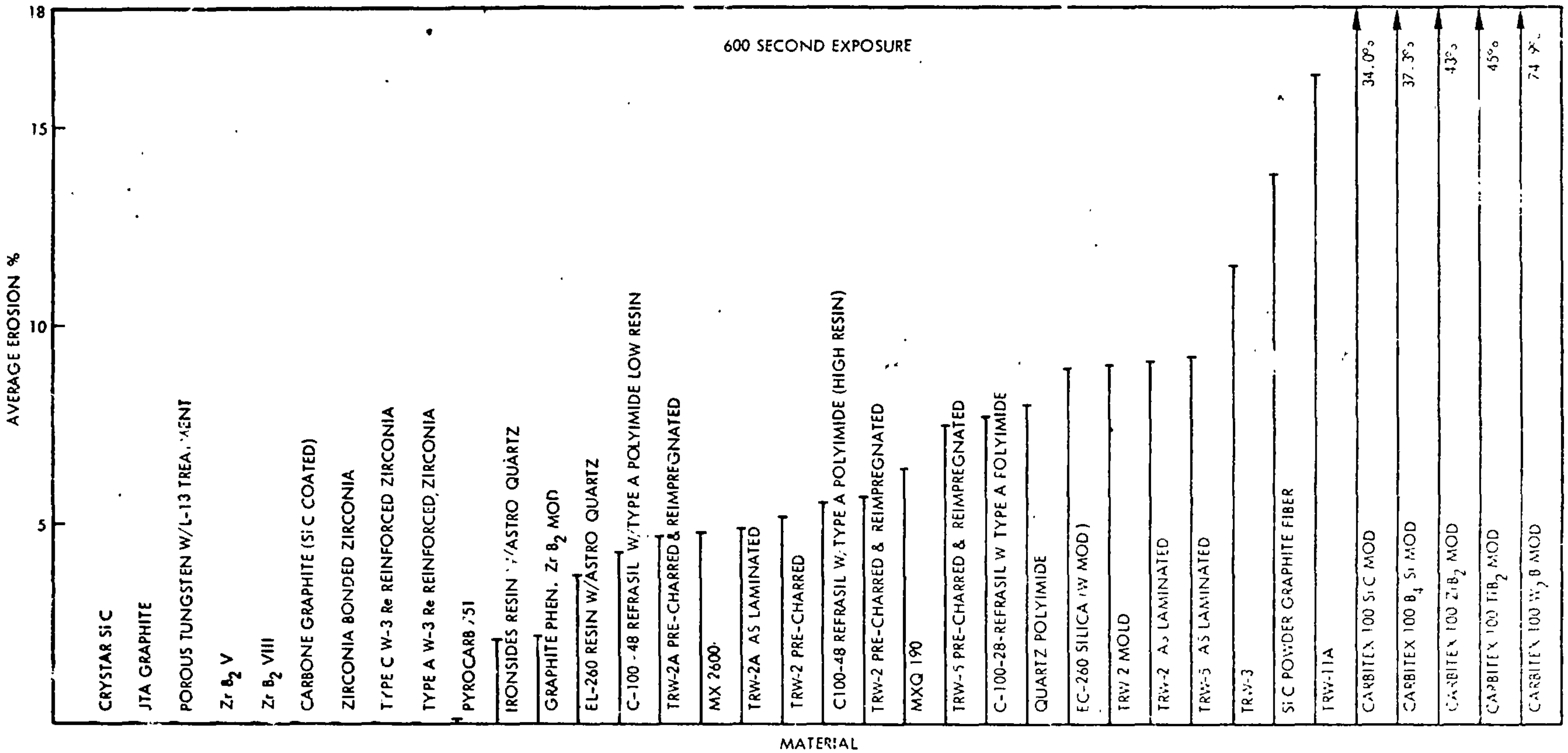

Figure 85. Percent Erosion for Various Materials 600 Second Test

5. APPENDIX: SUMMARY OF FABRICATION PROCEDURES - CIC FLAME TEST SAMPLES

Part Number	Material Description	Fabrication Vendor	Fabrication Procedures	Remarks
SK 404063-1	MX 2600 Silica Phenolic	TRW Cleveland	MX2600 silica phenolic per MT 3-10 form I laminated in accordance with PR 10-10 Class I	Test samples were machined from portion of a scrap LEMDE chamber.
SK 404063-2	Manlabs Zirconium Diboride V	Manlabs, Cambridge, Mass	(1) Metal boride powder (ZrB_2) and alloying ceramic additives, primarily silicon carbide and carbon, are procured as powders conforming to established limits of particle size and chemistry. (2) Powders are thoroughly mixed in proper proportions. (3) Mixed powders are formed using conventional graphite die hot pressing techniques in an inert argon atmosphere. Pressing conditions are 2000 to 2050 C at 4000 PSI pressure.	Detail processing procedures retained by Manlabs.
SK 404063-3	Manlabs Zirconium Diboride VIII			
SK 404063-4	MXQ 190 quartz phenolic	TRW Materials Department	(1) MXQ 190 prepreg meeting requirements of MT 3-29 obtained. (2) Pre-preg staged at 180F for 2-1/2 hours. (3) Pre-preg stacked to 151 plys and molded as follows: pressure - 200 psi cure cycle - 1/2 hour at 180F, 1/2 hour at 230F, 2 hours at 325F.	Material and Processing Data Raw material properties Resin solids-32.2% Volatiles - 4.2% Flow (200 psi)-19.9% Sp.Gr. - 1.75 Ave. Tensile Strength-70.6 KSI Ave. Comp. Strength-63.3 KSI Wt. before molding-701g Wt. after molding-689g Essentially no flow during molding
SK 404063-5	C-100-48 Silica cloth/TRW Type A Polyimide laminate	TRW Chemistry Department	(1) C-100-48 silica cloth impregnated with P13N Resin (Batch 56) (2) Pre-preg staged at 325F (4 min.) and 475F (4min.) (3) Pre-preg cut and stacked to 80 plies. (4) Laminate molded at 580 to 600F at 500 psi for 16 hours.	Material and Processing Data % solids in resin varnish-38% Pre-preg resin content-29.3% Pre-preg volatile content-1.64%
SK 404063-6	C-100-28 Silica cloth/TRW Type A Polyimide Laminate	TRW Chemistry Department	(1) C-100-28 silica cloth impregnated with P 13N Resin (Batch 56). (2) Pre-preg staged at 325F for 4 minutes and 475F for 4 minutes. (3) Pre-preg cut and stacked - 160 plys. (4) Laminate molded at 580 to 600F and s00 psi for 16 hours.	Material and Processing Data % solids in resin varnish-38% Pre-preg resin content-35.8% Pre-preg volatile content-2.05%

APPENDIX (Continued)

Part Number	Material Description	Fabrication Vendor	Fabrication Procedure	Remarks
SK 404063-7	Astroquartz 581-9073 quartz fabric/TRW Type A Polyimide Laminate	TRW Chemistry Department	(1) 581-9073 quartz cloth impregnated with P 13N Resin (Batch 56). (2) Pre-preg staged at 325F for 4 minutes and 475F for 4 minutes. (3) Pre-preg cut and stacked - 180 plys. (4) Laminate molded and cured at 580F and 500 psi for 16 hours.	Materials and Processing Data % solids in resin varnish - 38% Pre-preg resin content - 35.8% Pre-preg volatile content - 1.07%
SK 404063-8	Astroquartz 581-9073 quartz fabric/Ironsides 5471 Resin (formerly DP-25-10) Laminate. Resin is p-phenylphenol phenol formaldehyde copolymer.	Hughes Aircraft	(1) 581-9073 quartz cloth impregnated with Ironsides 5471 resin using spatula to obtain 40% resin pickup. (2) Pre-cured 60 minutes at room temperature, 15 minutes at 160F and 15 minutes at 225F. (3) Pre-preg cut and stacked - 200 plys. (4) Laminate molded at 300F and 2000 psi for 60 min. (5) Post cured as follows: 18 hours at 275F, 72 hours from 275 to 400F, 8 hours at 400F, cool to room temperature (specimens maintained under argon during post-cure).	Materials and Processing Data Resin content as molded - 38.4% Final resin content - 37% Density - 1.87 g/cc
SK 404063-9	Astroquartz 581-9073 quartz fabric/Evercoat EC 260 Resin. a 2, 2' biphenol polymer.	Hughes Aircraft	(1) 581-9073 quartz cloth spatula impregnated with EC 260 resin to obtain 40% resin pickup. (2) Pre-cured at room temperature for 60 minutes, 15 minutes at 160F, and 15 minutes at 225F. (3) Pre-preg cut and stacked to 184 plys. (4) Laminate molded at 300F and 200 psi for 60 minutes. (5) Post cured as follows: 18 hours at 275F, 72 hours from 275 to 400F, 8 hours at 400F, cool to R.T. before removal from oven. Specimens maintained under inert atmosphere (argon) throughout post-cure cycle.	Materials and Processing Data Resin content as molded - 40.7% Final resin content - 39.5% Density - 1.90 g/cc
SK 404063-11	Carbitex 100 made from silicon carbide coated yarn woven into cloth.	Carborundum Niagara Falls, N.Y.	Entire processing procedure is proprietary to Carborundum Co.	-
SK 404063-12	Carbitex 100 with 3-1/2% w/o zirconium diboride	Carborundum Niagara Falls, N.Y.	Entire processing procedure is proprietary to Carborundum Co	-

APPENDIX (Continued)

Part Number	Material Description	Fabrication Vendor	Fabrication Procedure	Remarks
SK 404063-13	Carbitex 100 with 3-1/2% w/o titanium diboride	Carborundum Niagara Falls, N.Y.	Entire processing procedure is proprietary to Carborundum Co.	-
SK 404063-14	Silicon Carbide coated graphite. Depth of coating to be .010"	Marquardt, Van Nuys, CA	(1) Substrate of Carbone 2239 graphite is machined to sample configuration. (2) Silicon carbide is applied to all surfaces of sample by standard chemical vapor deposition process involving decomposition of chlorosilane. (3) Details of processing are proprietary to Marquardt.	Marquardt suspects that coating thickness on specimen I.D. is approximately .003" rather than targeted .010".
SK 404063-16	C-100-48 Refrasil cloth with EC 260 resin plus -325 mesh tungsten powder.	TRW Chemistry Department	(1) C-100-48 Refrasil cloth impregnated with EC 260 resin. (2) Tungsten powder (31.3% by weight) added to pre-preg. (3) Pre-preg staged at room temperature for 60 minutes, 160F for 15 minutes and 225F for 15 minutes. (4) Pre-preg cut and stacked - 100 plys. (5) Laminate molded at 300 psi with the following pressurization cycle, start at 500 psi for 5 minutes, then up 62.5 psi every 5 minutes to 1000 psi, then up 62.5 psi every minute to 2000 psi. Hold at 2000 psi for 2 hours.	<u>Materials and Processing Data</u> (1) Resin content of pre-preg - 50.4%. (2) Resin content of laminate - 55.4% Laminate contained some delaminations and some plys had slid with respect to each other during molding cycle.
SK 404063-17	Vitreous silica with 20% v/o -325 mesh tungsten powder.	TRW Materials Department	(1) -325 mesh powders of tungsten and silica dry blended in 80 silica -20 tungsten volume ratio (1:1 by weight) for 15 minutes in roll mill. (2) Powders then mixed with 15 ml. nitrocellulose lacquer and 75 ml of acetone per 200 gm. of dry powders. (3) Wet mixture pressed in steel die at 15000 psi to 1-1/2 " long billet. (4) Billets air dried at 170F for 1/2 hour. (5) Billets placed on a tantalum sheet and heated at a uniform rate to 3100F in a vacuum furnace (1×10^{-5} torr). (6) Billets held at 3100F for 30 minutes then approximately 1 atmosphere of argon admitted to furnace to collapse internal voids in billet. (7) Billets held at 3100F for additional 15 minutes then cooled rapidly under argon to room temperature.	Billets shrunk and distorted but show approximately 5?% of theoretical density and freedom from voids.

APPENDIX (Continued)

Part Number	Material Description	Fabrication Vendor	Fabrication Procedure	Remarks
SK 404063-18	Pyrocarb 751 infiltrated with silicon	Hitco Gardena, CA	(1) Starting materials are graphite or carbon cloth and phenolic resin. (2) Laminate and cure at approximately 320F and 500 psi. (3) Laminate subjected to proprietary post cure treatment. (4) Carbonize at 750 to 900F for 6-12 days under inert atmosphere - (details proprietary). (5) Pyrolyze at approximately 4000F under inert atmosphere 2 days (details proprietary). (6) Infiltrate with pyrolytic carbon at 1600 to 2500F for 2-10 days (details proprietary). (7) Infiltrate with molten silicon at ~3000F for approximately 1 day (details proprietary).	
SK 404063-19	Porous Tungsten (80% of theoretical density) with TRW L-13 reactive coating	TRW Materials Department	(1) Commercial 80% dense tungsten was procured from Wah Chang and machined to sample shape. (2) Sample immersed in liquid L-13 for 10 minutes at 1800 to 1900F under inert argon atmosphere. (3) Cooled to room temperature under argon atmosphere.	Details of processing are retained by Materials Engineering
SK 404063-21	JTA Graphite	Union Carbide N.Y., N.Y.	Standard product which is a pressed and sintered composite of graphite, zirconium, boron and silicon.	
SK 404063-22	Carbitex 100 with 3-1/2% w/o tungsten diboride	Carborundum Niagara Falls, N.Y.	Entire processing procedure proprietary to Carborundum.	
SK 404063-23	Carbitex 100 with 3-1/2% w/o boron silicide (B_4Si)	Carborundum Niagara Falls, N.Y.	Entire processing procedure proprietary to Carborundum.	
SK 404063-24	Zirconium diboride and graphite reinforced molded phenolic	TRW Materials Department	(1) Constituents mixed in the following proportions: 37% w/o ZrB_2 (-325 mesh), 8.4% w/o silicon (-325 mesh), 17.3% w/o Hitco GFA Graphite fibers, 37% SC1008 Phenolic resin (100% solids). Mixing accomplished in laboratory mixer. (2) Material was staged at 180F for 2-1/2 hours. (3) Molding tool was loaded at 180F and 500 psi and molding was accomplished with the following cure cycle: 190F,2000psi, 30 min. | 230F,6000psi, 30 min. 200F,3000psi, 30 min. | 325F,6000psi, 3 hours Molding was cooled to room temperature under 6000psi	Specific gravity of moldings - 1.98 Finished machined molding exhibited several hair line cracks.

APPENDIX (Continued

Part Number	Material Description	Fabrication Vendor	Fabrication Procedure	Remarks
SK 404063-25	HRX-211 Silica phenolic pre-char	Haveg Santa Fe Springs, California	(1) Silica fabric impregnated with EC-201 phenolic resin. (2) Fabric laminated at approximately 1000 psi and 310F. (3) Laminate pyrolyzed at approximately 800F and carbonized at approxima-ely 1800F under inert atmosphere.	
SK 404063-26	HRX-212 Silica phenolic carbonized re-impregnated and re-carbonized.	Haveg Santa Fe Springs, California	Same as -25 material except that carbonized laminate is vacuum re-impregnated with EC 201 resin and re-pyrolyzed and re-carbonized as above.	
SK 404063-27	Norton Crystar silicon carbide	Norton Worchester, Mass.	Fabricated by Norton using processing procedures retained by them.	
SK 404063-28	TRW-2 Material-Molded composite of SC1008 resin with 27% w/o GFA graphite fiber and 43% w/o silica fibers	TRW Materials Department	(1) Molding composition was: Carbon fiber-54 gm, silica fiber, - 86 gm., SC 1008 resin - 96.8 gm, isopropyl alcohol - 19 gm. (2) B-staged at 180F for 1 hour and 15 minutes. (3) Two specimens die molded as follows: No.1 Tool loaded at 180F, 100 psi Heated to 190F, apply 1000 psi Heated to 210F, apply 2000 psi Heated to 325F maintaining 2000 psi/2 hrs. Cooled under pressure No.3 Tool loaded at 180F, 100 psi Heated to 190F,apply 2000 psi Heated to 200F, apply 4000 psi Heated to 325F, maintaining 4000 psi/2 hrs. Cooled under pressure	Material and Process Data (1) Resin content - 30% (2) Specific gravity - 1.61/1.64
SK 404063-31	Zirconium oxide reinforced with W-3Re wire	TRW Cleveland	Type A Material - (1) Reinforcement of 5% v/o .0035" x 3/16: long W-3Re wires and matrix material of Zircoa F 410 zirconia (stabilized by 3% MgO and 0.5% CaO are mixed and blended dry. (2) Composite is cold compacked in an isostatic press at 30,000 psi. (3) Composite is vacuum sintered at 4000F and slow cooled.	

APPENDIX (Continued)

Part Number	Material Description	Fabrication Vendor	Fabrication Procedure	Remarks
SK 404063-31 (continued from previous page)			Type C Material - (1) Reinforcement of 7% v/o W-Re wire .002" x 1/8" long and zirconia powder (stabilized with 2.85% MgO) are mixed and blended dry. Other processing steps same as for Type A material.	
SK 404063-32	Zirconium oxide fabric with zirconium oxide cement	Union Carbide Research Center, Sterling Forest, N.Y.	(1) ZYW-30 zirconia cloth (yttria sta bilized-8wt.% satin weave) and cement consisting of 100 ml. of liquid binder to 150 g of zirconia powder. Note: Liquid binder contains 35.6% w/o zirconia solids and a yttrium compound which gives tetragonal zirconia bond on firing. (2) Cloth is saturated with cement, excess cement removed and cloth cut to size. (3) Cloth stacked 100 high and placed in mylar lined aluminum die. (4) Laminates are pressed at 150 psi with temperature being raised to 250F and held at that temperature for 3 hours. (5) The laminate is removed from die and cured at 600F in air. (6) Laminate is heated to 3000F in a gas fired Bickley Kiln over a period of 6 hours and held at temperature for 1/2 hour. (7) Specimens machined using diamond grinding medium	(1) Density of laminates 280 lb/ft³. (2) UCC believes that 3000F sintering temperature was excessive and fiber reinforcement was lost accompanied by degradation of thermal shock resistance.
SK 404063-33	TRW-2 (Laminated) SC 1008 phenolic resin with 27% w/o G1736 graphite cloth and 43% w/o C-100-48 Refrasil fabric.	TRW Chemistry Department	(1) Fabrics impregnated with resin and staged at 200F for 60 minutes. (2) Fabrics cut and stacked to form laminate (60 plies of graphite and 60 plie of silica). (3) Laminate molded as follows: 50 psi and 200F for 30 minutes, 300 psi and 350F for 120 minutes, cool under pressure.	Material & Processing Data (1) Resin varnish % solids - 60% (2) Pre preg resin content- graphite - 53.7% c-100-48 - 31.7% (3) Pre preg % volatiles - graphite - 6.4% c-100-48 - 4.4%
SK 404063-34	TRW-3 (Laminated) SC1008 phenolic resin with 17% w/o G1736 graphite cloth and 53% w/o C-100-48 silica fabric	TRW Chemistry Department	(1) Fabric impregnation same as -33. (2) Fabrics cut and stacked to form laminate (40 plies graphite cloth and 80 plies silica cloth). (3) Laminate molded as follows: 50 psi and 180F for 30 minutes, 50 psi and 230F for 30 minutes, 300 psi and 350F for 120 minutes cooled under pressure.	Materials and Processing Data (1) Resin varnish - % solids - 60% (2) Pre preg resin content - graphite - 53.7% c-100-48 - 31.7% (3) Pre preg % volatiles - graphite - 6.4% c-100-48 - 4.4%

APPENDIX (Continued)

Part Number	Material Description	Fabrication Vendor	Fabrication Procedures	Remarks
SK 404063-35	TRW-5 (Laminated) SC1008 phenolic resin, 15% w/o G1736 graphite cloth, 46% w/o c-100-48 refrasil cloth, 9% w/o silicon carbide powder	TRW Chemistry Department	(1) Fabric impregnated with resin and SiC filler and staged at 200F for 60 minutes. (2) Laminates made by stacking plys (graphite-30, silica 60). (3) Laminate molded and cured at 50 psi and 200F for 30 minutes then 300 psi and 350F for 120 minutes, cooled under pressure.	Materials and Processing Data (1) Resin varnish-% solids-71.5% (2) Pre-preg resin content graphite - 61.2% c-100-48 - 57.0% (3) Percentage filler - graphite - 34.1% c-100-48 - 16.9% (4) Pre-preg percentage volatiles graphite - 4.6% silica - 4.8%
SK 404063-36	TRW 2A (Laminated) SC1008 phenolic resin with 27% w/o G1736 graphite cloth and 43% w/o MX Q 190 quartz phenolic pre-preg.	TRW Chemistry Department	(1) Graphite fabric impregnated with resin and staged at 200F for 60 minutes. (2) Laminates made by stacking plys (50 graphite 100 MXQ 190). (3) Laminate molded and cured at 50 psi and 200F, for 30 minutes and 300 psi and 350F for 120 minutes. Cooled under pressure.	Materials and Processing Data (1) Resin varnish - % solids-60% (2) Pre-preg resin content graphite - 49.4% MXQ190 - 34.3% (3) Pre-preg percent volatiles graphite 5.7% MXQ190 - 4.5%
SK 404063-37	TRW 11A (Laminated) TRW Type A polyimide resin with 47% w/o Astroquartz 581/9073 cloth and 21% w/o G1736 graphite cloth.	TRW Chemistry Department	(1) Fabrics impregnated with P13N resin and staged at 325F for 4 minutes and 475F for 4 minutes. (2) Laminate made by stacking plys of pre-preg (50 graphite, 100 Astroquartz). (3) Laminate molded and cured at 550F and 200 psi for 60 minutes then 550F and 500 psi for 180 minutes. Cooled under pressure.	Materials and Processing Data (1) Resin varnish - % solids - 36.6% (2) Pre-preg resin content graphite-38.7% quartz-30.6% (3) Pre-preg percentage volatiles graphite - 0.8% quartz - 0.6%
SK 404063-38	TRW-5 Material except cured laminate charred and re-impregnated with furfuryl alcohol resin	TRW Chemistry Department & Materials Engineering Department	(1) Cured laminate fabricated same as -35. (2) Laminate was charred under an atmosphere at 1475F for 2 hours. Cooled under argon. (3) Charred laminate impregnated with furfuryl alcohol resin (Quaker Oats Fapreg P-5) using following procedure: (a) Laminate placed in dessicator and subjected to vacuum of 1mm Hg for 1-1/2 hours. (b) While under vacuum-sample was covered with resin.	Materials and Processing Data Same as -35 for cured laminate.

APPENDIX (Continued)

Part Number	Material Description	Fabrication Vendor	Fabrication Procedures	Remarks
SK 404063-38			(3) continued (c) Vacuum released and parts soaked in resin overnight. (d) Parts removed from resin and allowed to drain 3 hours. (e) Parts wrapped in nylon film and heated to 200F for 18 hours. (f) Nylon removed and parts placed in 250F oven. (g) Oven heated to 375F with temperature being raised 25F every 30 minutes. (h) Held at 375F for 2 hours and parts cooled in until they reached 200F	
SK 404063-39	TRW-2A Material except cured laminate charred and re-impregnated with furfuryl alcohol resin.	TRW Chemistry and Materials Engineering Departments	(1) Cured laminate fabricated same as -36. (2) Laminate charred under argon atmosphere at 1475F for 2 hours, cooled in argon. (3) Re-impregnated same as -38.	Materials and Processing Data Same as -36 for cured laminate.
SK 404063-40	TRW-2 Material except cured laminate charred	TRW Chemistry Department	(1) Cured laminate fabricated same as -33. (2) Laminate charred under argon atmosphere at 1475F for 2 hours, cooled in argon.	Materials and Processing Data Same as -33 for cured laminate.
SK 404063-41	TRW-2 Material except cured laminate charred and re-impregnated with furfuryl alcohol resin.	TRW Chemistry and Materials Engineering Departments	(1) Cured laminate fabricated same as -33. (2) Laminate charred under argon atmosphere at 1475F for 2 hours, cooled in argon. (3) Re-impregnated same as -38.	Materials and Processing Data Same as -33 for cured laminate.