ARC JET TESTS OF RSI MATERIALS - SCREENING AND COMPARATIVE EVALUATION[†]

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CONTRACT TEST PROGRAM OBJECTIVES (NAS2-6445)

(Figure 1)

An extensive screening test program including a comparative evaluation of candidate RSI materials was performed under cyclic convective heating conditions (ref. 1).* The RSI materials evaluated were LI-1500, HCF, REI, and silicon carbide foam. The test samples were nominally exposed to 30 half hour cycles at conditions that covered the spectrum of surface temperature and heat flux appropriate to the application of RSI materials to the shuttle orbiter vehicle.

The test configurations and procedures maximized efficiency and minimized cost by utilizing multiple (up to six) test samples in each test model, and by employing continuous testing of two models, which were alternately exposed to the test stream. The test model and test sample designs allowed a quick change of test samples, which employed common instrumentation, to minimize turnaround time between each series of cyclic tests.

Measurements were made during each cycle and nominally after every six cycles to define thermal response in terms of surface and in-depth temperatures, mass loss and surface recession, surface properties in terms of emissivity and catalycity, failure modes, and operating limits. A calibration test series was also performed to measure the distribution of properties across the test stream and across the model face, and to measure surface catalycity effects.

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* Metallics, carbon-carbon composites, and ablators were also evaluated under the program.

CONTRACT TEST PROGRAM OBJECTIVES (NAS2-6445)

- PROVIDE A TEST STREAM WHICH SIMULATES THE EXPECTED SHUTTLE ORBITER REENTRY ENVIRONMENT (HEATING, ENTHALPY, PRESSURE)

- DESIGN AND FABRICATE A TEST SAMPLE AND MODEL CONFIGURATION WHICH MAXIMIZES DATA AND MINIMIZES ASSOCIATED TEST COSTS

- CONDUCT CYCLIC SCREENING TESTS OF ALL TPS CANDIDATES WITH SUFFICIEN INSTRUMENTATION TO PROVIDE THE FOLLOWING COMPARATIVE INFORMATION:

  SURFACE AND IN-DEPTH TEMPERATURE RESPONSE CHARACTERISTICS

  SURFACE PROPERTIES (EMITTANCE AND SURFACE CATALYCITY)

  MASS LOSS AND SURFACE RECESSION

  RESPONSE CHARACTERISTICS AND FAILURE MODES

Figure 1

SCOPE AND COST OF TESTS

(Figure 2)

The total facility test time for all material types - metallics, RSI, carbon-carbon composites, and ablators - was 271-1/3 hours and the total sample exposure time was 870 hours. For the RSI tests, the facility time was 131-1/2 hours and the sample exposure time was 288 hours.

At maximum testing efficiency the demonstrated test cost was as low as $70 per hour of exposure, which, with a six sample model configuration, resulted in a cost of roughly $12 per sample hour.

SCOPE AND COST OF TESTS

	FACILITY HOURS	SAMPLE HOURS
• METALLICS	82-1/4	487-1/2
• SURFACE INSULATORS (RSI)	131-1/2	288
• CARBON-CARBON COMPOSITES	54-1/4	91
• ABLATORS	3-1/3	3-1/3

- COST PER FACILITY HOUR = $70 MINIMUM
- COST PER SAMPLE HOUR = $12 MINIMUM

Figure 2

DESCRIPTION OF RSI MATERIALS TEST

(Figure 3)

The types of RSI materials tested were LI-1500, with five different coating versions; HCF, with three different coating versions; REI; and silicon carbide foam. The most extensive testing was performed on the LI-1500 and HCF versions. Direct comparative performance results were obtained for LI-1500, HCF, and REI (two sets of three 120° samples) for a total of 30 sample hours on each material.

DESCRIPTION OF RSI MATERIALS TEST

MATERIAL	FABRICATOR	NUMBER OF DIFFERENT COATINGS	BASIC MATERIAL SYSTEM	SAMPLE HOURS
LI-1500	LMSC	5	SILICA	104
HCF	MDAC	3	MULLITE	102
REI	GE	1	MULLITE	30
SiC FOAM	LMSC	-	SILICON CARBIDE	51

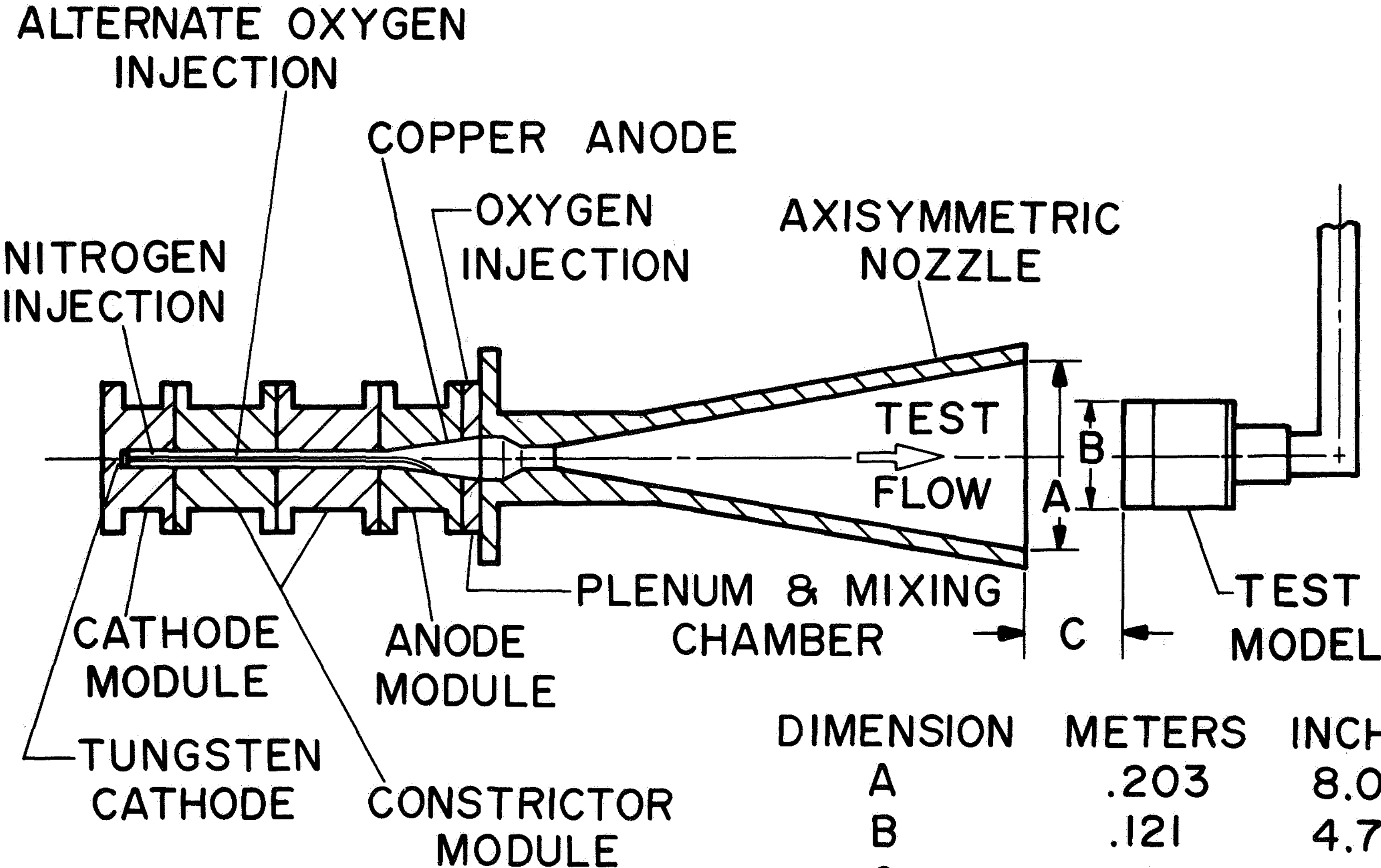
Figure 3

TEST SETUP

(Figure 4)

The test program was performed in the Aerotherm 1.5 MW arc plasma facility, and the hyperthermal test stream was generated by the Aerotherm 300 kW constrictor arc heater. The test gases were nitrogen and oxygen in proper proportion to yield the composition of air. A conventional convergent/divergent nozzle with a 0.025-meter (one-inch) throat diameter and 0.203-meter (eight-inch) exit diameter was employed. The test model configuration was a flat-face stagnation point model which was 0.121-meters (4-3/4 inches) in diameter.

TEST SETUP



DIMENSION	METERS	INCHES
A	.203	8.00
B	.121	4.75
C	.102	4.00

Figure 4

MODEL CONFIGURATION

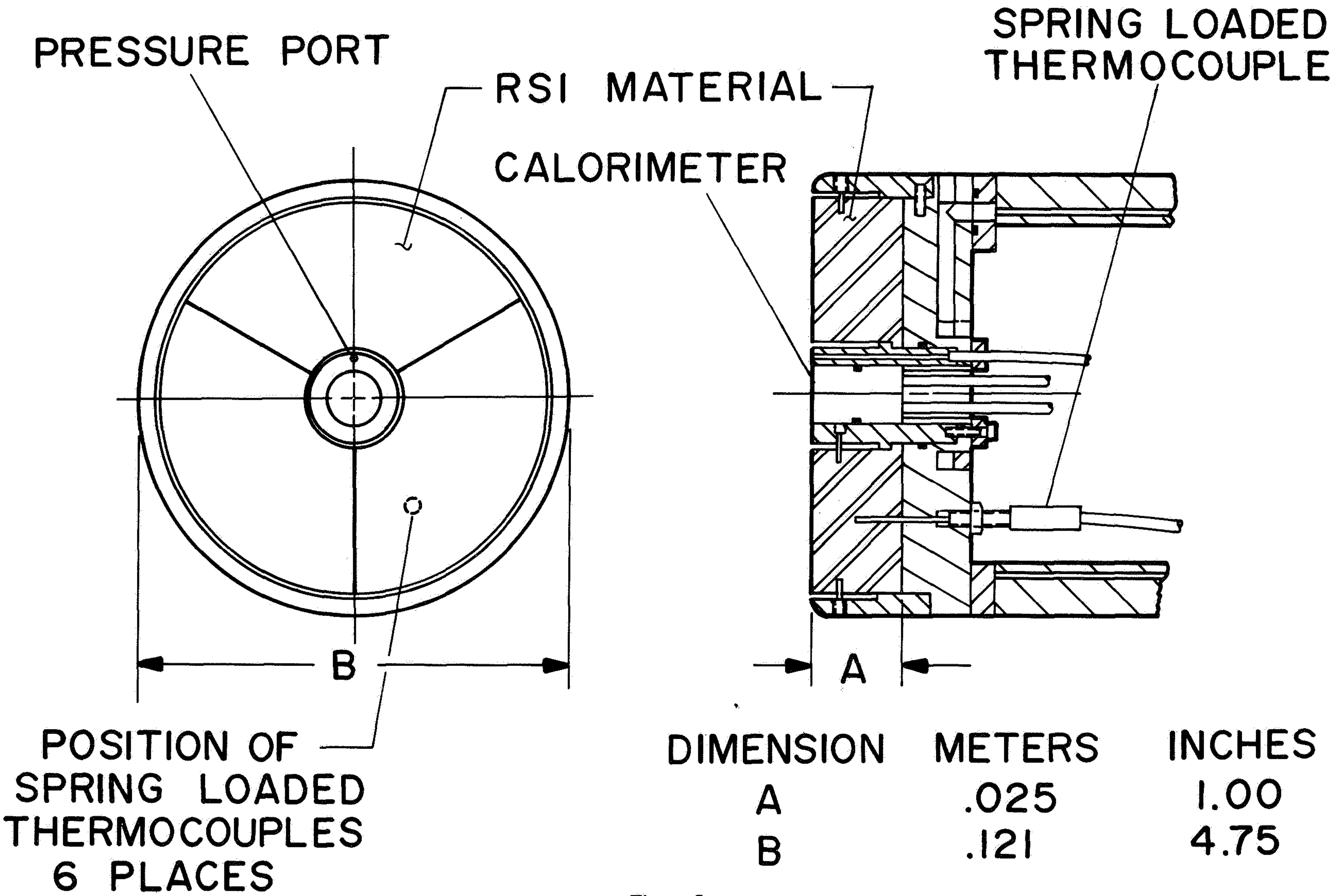
(Figure 5)

The flat-face model configuration was chosen for convenience in test sample fabrication, and was 0.121 meters (4-3/4 inches) in diameter with a 3.2 mm (1/8-inch) corner radius. This model body diameter allowed the maximum practical test sample size consistent with uniform property distributions across the test samples for the 0.203 m (8 in) dia. test stream and the projected test conditions. The test models were made of copper and were water cooled. They included a centerpost containing a calorimeter and pressure tap for continuously monitoring conditions throughout each test. A peripheral copper ring was employed to ensure that the test samples were not exposed to any unusual thermal or aerodynamic edge effects.

The RSI test samples were 120° (as illustrated) or 180° pie-shaped configurations which were 0.025 meters (one-inch) thick. The test samples and models incorporated the necessary quick-change capability for optimum testing efficiency. The test samples were removed simply by removing the retention pins in the peripheral ring. Spring-loaded thermocouples were used throughout to eliminate the requirement for disconnecting instrumentation leads. These thermocouples were located at 60° intervals in a circular pattern through the central region of the test samples.

Surface temperature was measured with an infrared optical pyrometer (TD-9 or TD-7). The pyrometer was mounted on an oscillating mechanism, which indexed the pyrometer in 60° increments in the same circular pattern defined by the spring-loaded thermocouples. Surface recession was measured with a microscope micrometer (no surface contact), mass loss was measured with an analytic balance, and qualitative documentation of the sample response was performed through post-test 35 mm color still photography.

MODEL CONFIGURATION



DIMENSION	METERS	INCHES
A	.025	1.00
B	.121	4.75

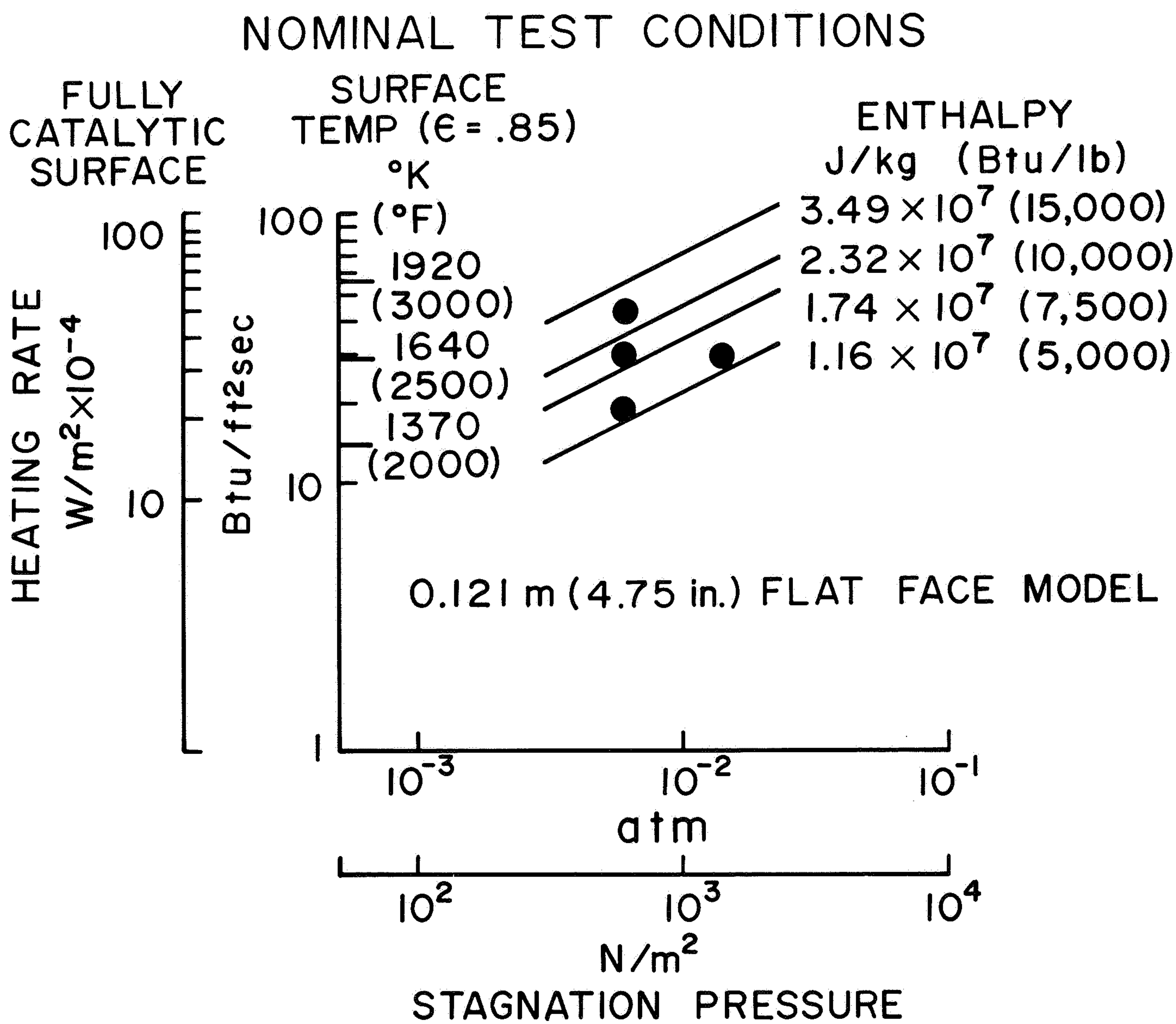
Figure 5

NOMINAL TEST CONDITIONS

(Figure 6)

Tests were run over a range of heat flux primarily at a single stagnation pressure, 6.08×10^2 N/m^2 (0.006 atm), and therefore single heat transfer coefficient, 0.019 kg/m^2sec (0.0038 lb/ft^2sec). One test series was run at the nominal baseline heat flux of 3.4×10^5 W/m^2 (30 Btu/ft^2sec) but at a higher stagnation pressure, 14.2×10^2 N/m^2 (0.014 atm), and heat transfer coefficient, 0.028 kg/m^2sec (0.0058 lb/ft^2sec), to check the effect of these variables.

Testing was nominally performed in blocks of six 30-minute cycles on each of two test models. The models were alternately exposed to the test stream for the required 30 minute intervals. Between each model change, a calibration model of the same configuration as the test sample models and containing six calorimeters and six pressure taps was also exposed to the test stream for a test condition check and reference. The facility therefore was run for approximately 6-1/2 hours continuously--3 hours (6 cycles) on each of the two test sample models and approximately 1/2 hour on the calibration model. In an 8-hour shift, the remaining 1-1/2 hours were occupied by test sample changes and measurements. Much of the program was performed on a two-shift basis, resulting in facility operation of over 65 hours a week.



NOMINAL TEST CONDITIONS
FULLY CATALYTIC SURFACE
SURFACE TEMP (ϵ = .85)
°K
(°F)
1920
(3000)
1640
(2500)
1370
(2000)
ENTHALPY
J/kg (Btu/lb)
3.49 × 10^7 (15,000)
2.32 × 10^7 (10,000)
1.74 × 10^7 (7,500)
1.16 × 10^7 (5,000)
HEATING RATE
W/m^2 × 10^-4
Btu/ft2sec
100
10
1
0.121 m (4.75 in.) FLAT FACE MODEL
10^-3
10^-2
10^-1
atm
10^2
10^3
10^4
N/m^2
STAGNATION PRESSURE

Figure 6

MODEL AND TEST STREAM CALIBRATION RESULTS

(Figure 7)

A detailed calibration test series was performed prior to the start of testing to define:

- Distribution of properties across the test stream (heat flux, stagnation pressure, enthalpy)
- Distribution of properties across the model face (heat flux, pressure)
- Surface catalycity potential as defined by a catalytic wall calorimeter (polished copper) and a noncatalytic wall calorimeter (coated teflon)

Typical model distribution results exhibit a uniform variation in pressure but some scatter in heat flux.* This scatter in the heat flux measurements is felt to be due to scatter in the calorimeter performance and not an indication of the actual distribution on the model. For all RSI conditions, the distributions across the test stream in the model region and the distributions across the model face were relatively flat.

The surface catalycity calibration results are presented in a following plot.

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* The flagged symbols in the plot denote measurement locations 90° either side of the primary locations.

MODEL AND TEST STREAM CALIBRATION RESULTS

TYPICAL MODEL PROPERTIES DISTRIBUTION

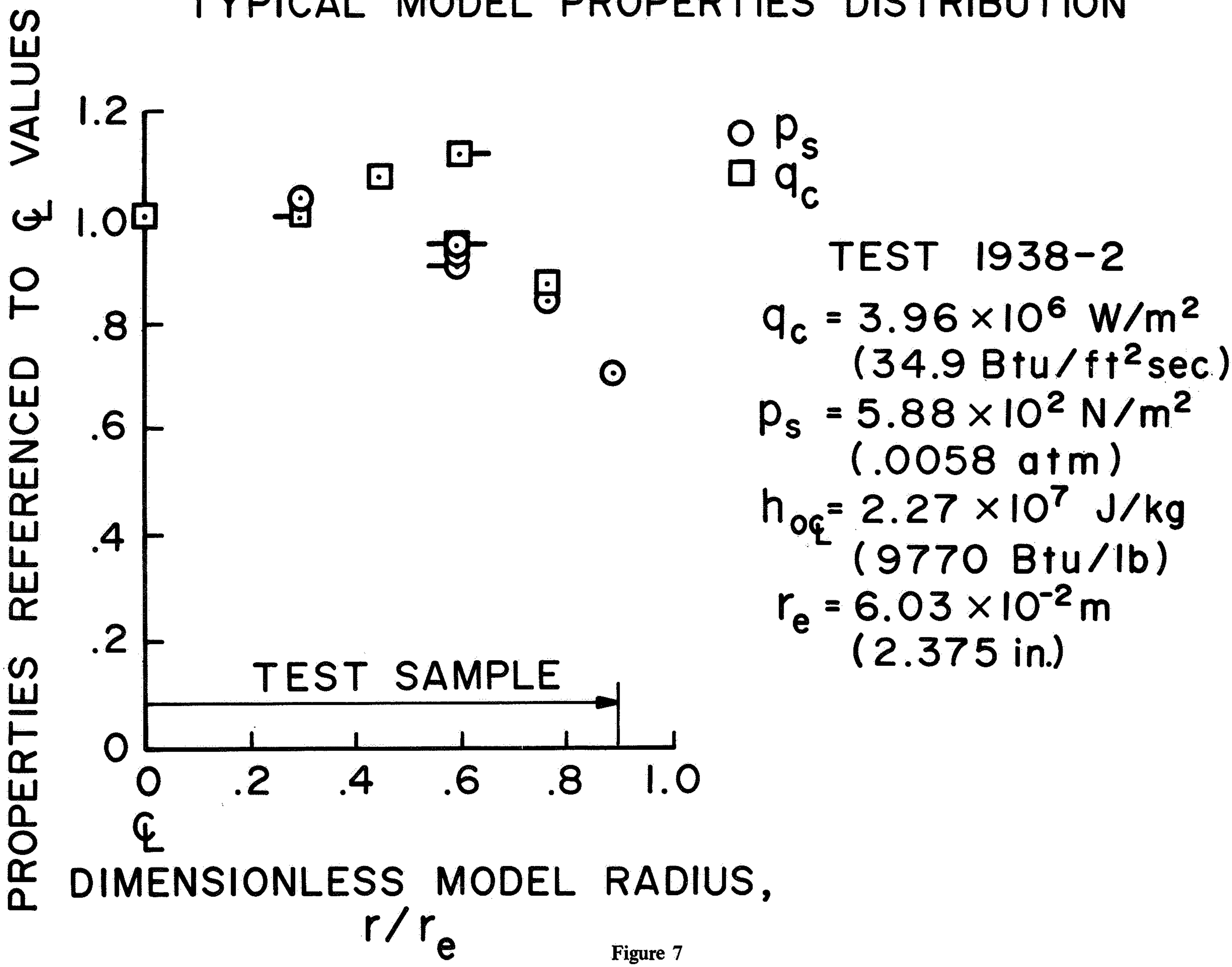


Figure 7

NOMINAL TEST MATRIX AND FAILURE MODES

(Figure 8)

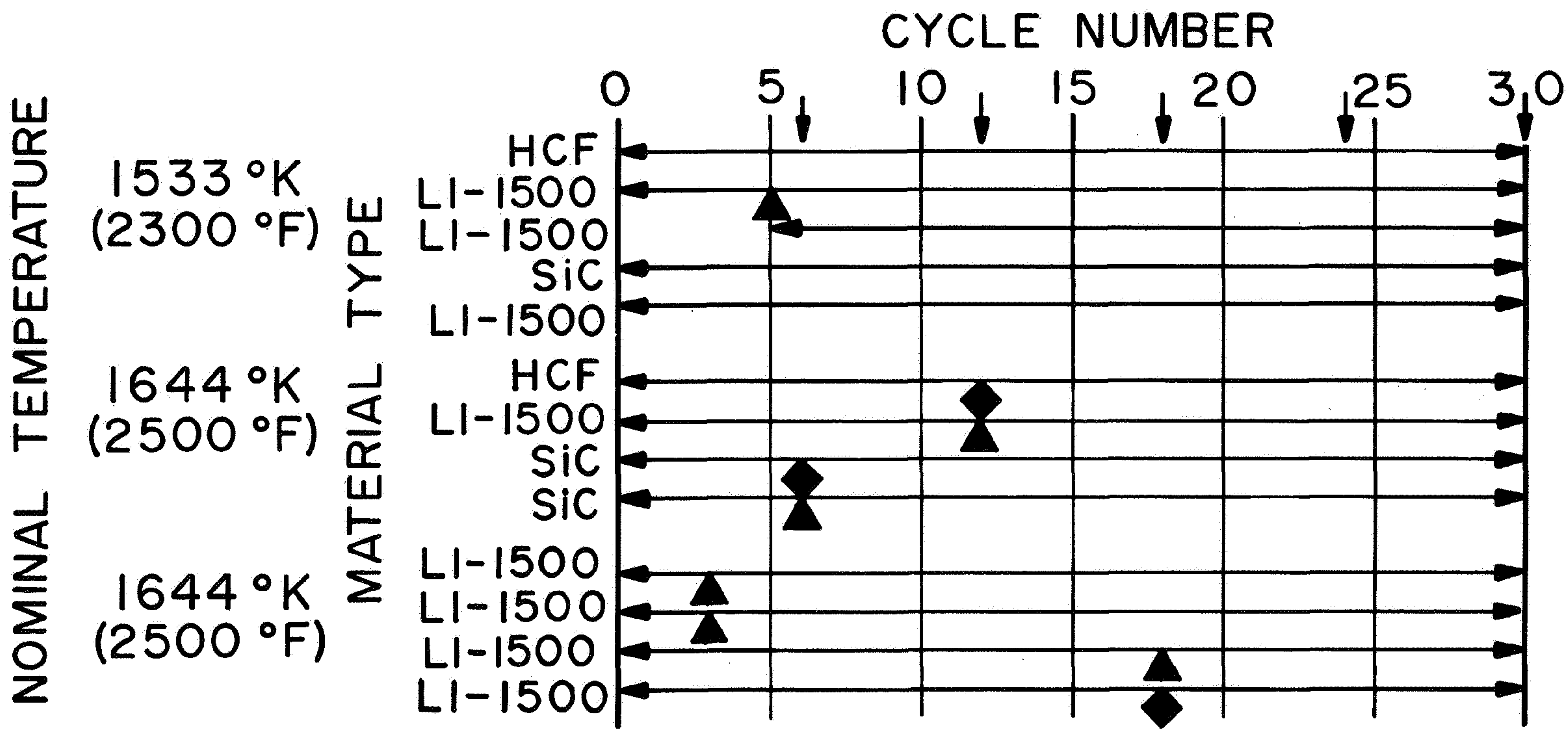
The nominal and actual test matrix for the RSI materials is presented in figure 8. The lines ←——→ indicate the nominal test program, and in the absence of any other symbols the actual test program as well. The symbol ▲ indicates termination of testing on the particular sample due to a sample failure and the symbol ◆ indicates termination of testing on the particular sample due to insufficient companion samples. In the case of a sample failure, testing was continued whenever possible by replacing the failed sample with another to-be-tested sample

The sample failures indicated in the test matrix were not necessarily indicative of the material performance capabilities. The initial emissivity data available and used for LI-1500 was incorrect and resulted in unexpectedly high surface temperatures, the radial split line between samples resulted in a singularity region that promoted failure, and the retention pins could have promoted the SiC foam failure (cracks).

The specific failures in the order presented in the text matrix are defined below:

RSI Type	Reason for Failure
LI-1500	Actual emissivity lower than used for surface temperature control
LI-1500	As above
SiC	Severe cracking
LI-1500	Subsurface removal along radial split line
LI-1500	As above
LI-1500	As above
HCF	Severe coating cracking
HCF	As above
HCF	As above
HCF	As above
HCF	As above

TEST MATRIX AND FAILURE MODES



↓ INDICATES WEIGHT LOSS AND SURFACE RECESSION MEASUREMENTS AND PHOTOGRAPHS

▲ SAMPLE FAILED

◆ INSUFFICIENT SAMPLES TO CONTINUE TESTING

180° OR 120° TEST SAMPLES

Figure 8

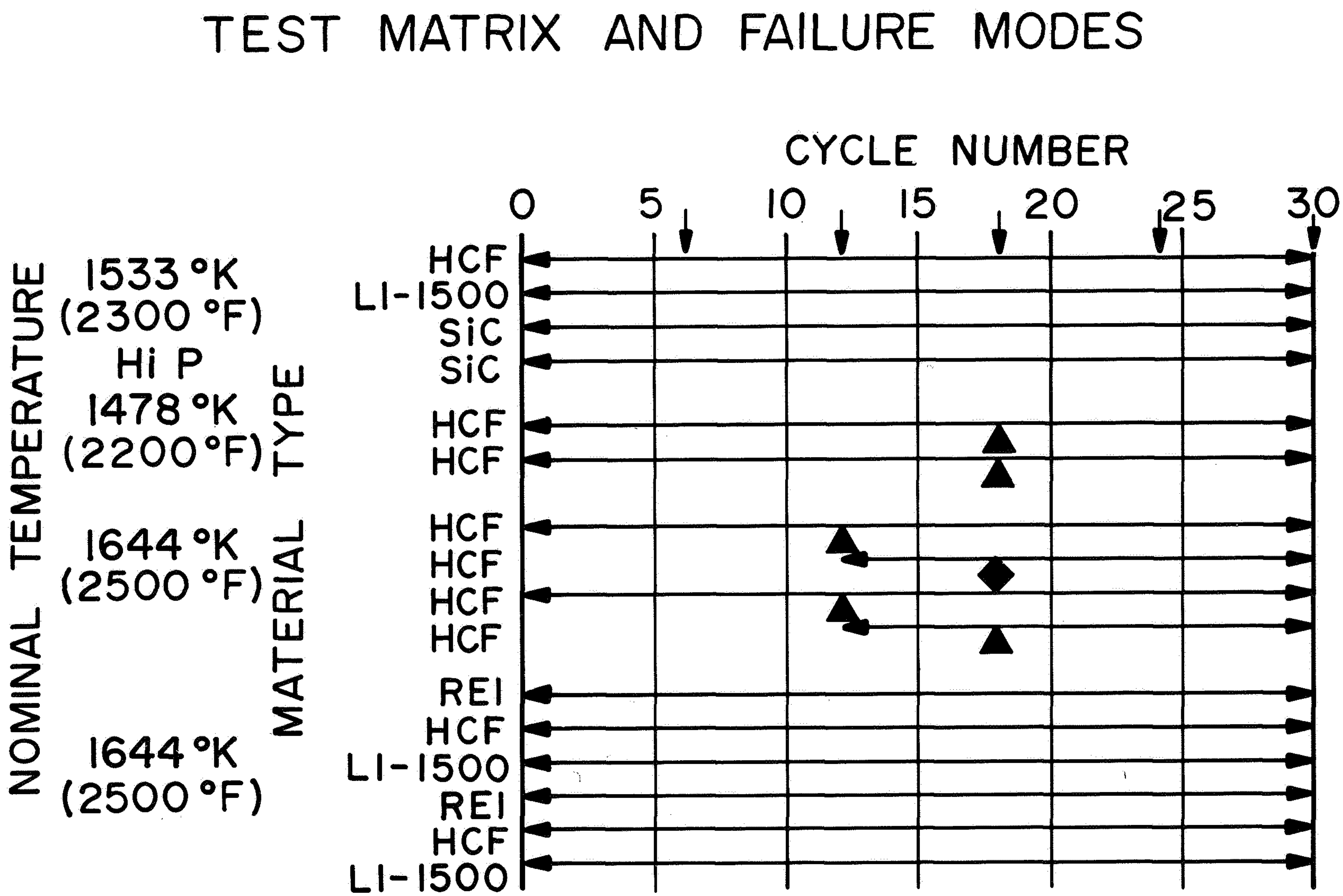


Figure 8 (Concluded)

TYPICAL SURFACE AND IN-DEPTH TEMPERATURE RESPONSE

(Figure 9)

In the presentation of the RSI test results, emphasis is placed on the test series that employed the 120° test samples of REI, HCF, and LI-1500 (the last series in the test matrix) since it provided complete comparative data.

Surface and in-depth temperature results are presented for the twelfth cycle in the 30 cycle series. The results for REI and LI-1500 are typical for the entire 30 cycles. The HCF sample, however, started out at a surface temperature of about 1672°K (2550°F), and this temperature continuously decreased with increased cycling to a stable temperature of about 1478°K (2200°F) after about the fifteenth cycle. There was a visible change in the surface condition during this period, this change resulting in a change in surface catalycity from nearly fully catalytic to noncatalytic.

The different insulative performance of the three materials is apparent from the spring-loaded thermocouple measurements. As expected the silica system material is a better insulator than the two mullite system materials.

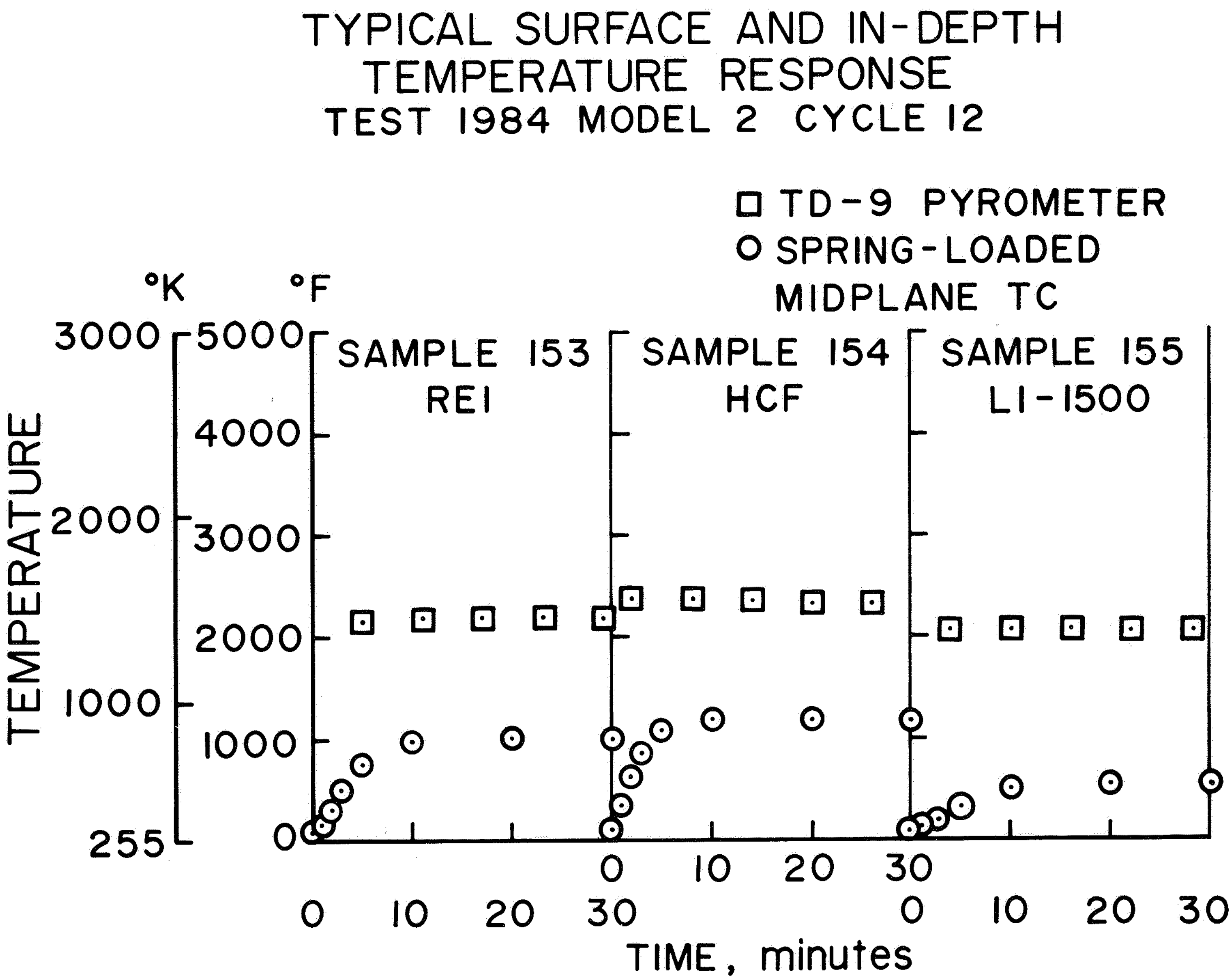

TYPICAL SURFACE AND IN-DEPTH
TEMPERATURE RESPONSE
TEST 1984 MODEL 2 CYCLE 12
TD-9 PYROMETER
SPRING-LOADED
MIDPLANE TC
°K
°F
3000
2000
1000
255
5000
4000
3000
2000
1000
0
TEMPERATURE
SAMPLE 153
REI
SAMPLE 154
HCF
SAMPLE 155
LI-1500
0
10
20
30
TIME, minutes

Figure 9

TYPICAL MASS LOSS RESULTS

(Figure 10)

The same set of three RSI test samples (REI, HCF, and LI-1500) exposed at constant heat flux exhibited average loss rates over 30 cycles as follows:

RSI Type	Average Loss Rate Over 30 Cycles
REI	1.2×10^{-6} kg/m^{2}sec (0.00086 lb/ft^{2}hr)
HCF	3.4×10^{-6} kg/m^{2}sec (0.00250 lb/ft^{2}hr)
LI-1500	0.7×10^{-6} kg/m^{2}sec (0.00050 lb/ft^{2}hr)

Note that the high average rate for HCF is due to the very high rate during the first few cycles (during which the surface temperature was also high due to the catalytic surface condition mentioned earlier).

TYPICAL MASS LOSS RESULTS
TESTS 1983-1987

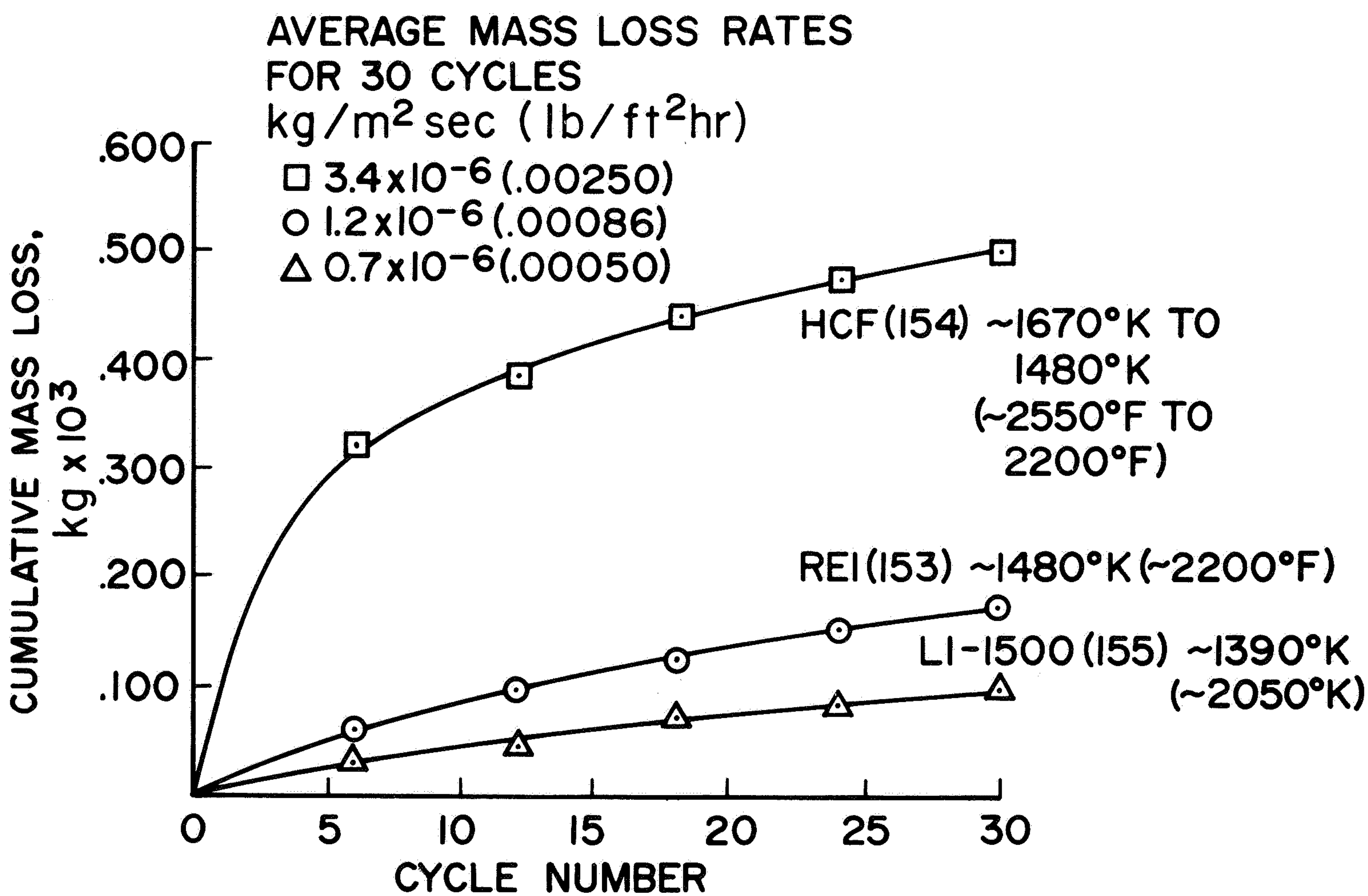


Figure 10

SURFACE CATALYCITY RESULTS

HCF FOR A RANGE OF HEAT FLUX

(Figure 11)

During one cycle on each of two models, the test conditions were varied in steps to define the surface catalycity effect over a flux range from about 28×10^4 to 57×10^4 W/m^2 (25 to 50 Btu/ft^2sec). These results were obtained on one of the HCF versions. The solid line is the calculated fully catalytic wall variation of heat flux with surface temperature. The dotted line is a fit of the catalycity calibration results mentioned previously. This version of HCF is seen to have a noncatalytic surface at approximately the heat flux ratio defined from the calibration tests.

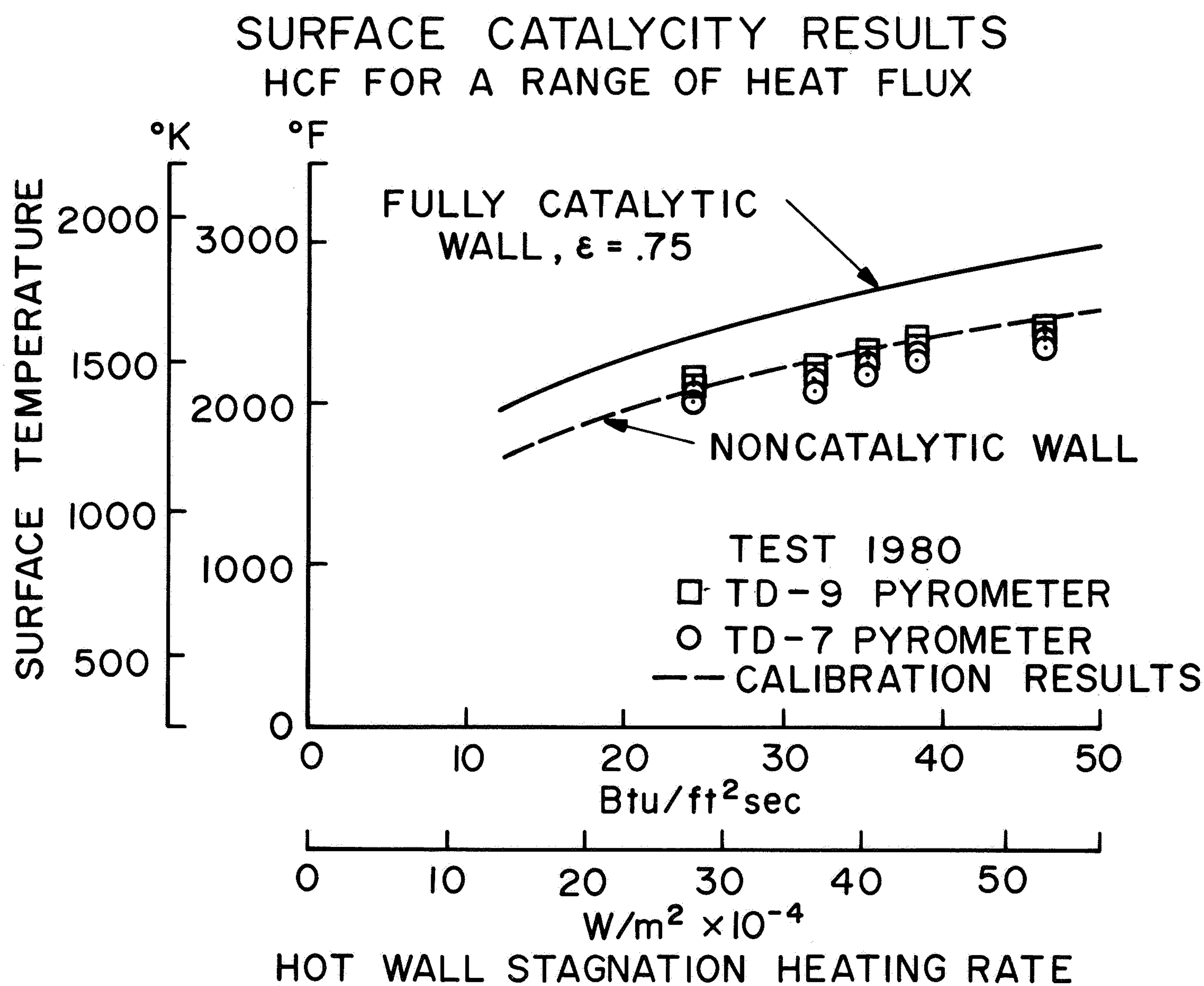

SURFACE CATALYCITY RESULTS
HCF FOR A RANGE OF HEAT FLUX
°K
°F
SURFACE TEMPERATURE
2000
1500
1000
500
3000
2000
1000
0
FULLY CATALYTIC
WALL, ε = .75
NONCATALYTIC WALL
TEST 1980
TD-9 PYROMETER
TD-7 PYROMETER
CALIBRATION RESULTS
0
10
20
30
40
50
Btu/ft²sec
W/m² ×10⁻⁴
HOT WALL STAGNATION HEATING RATE

Figure 11

SURFACE CATALYCITY RESULTS

(Figure 12)

The RSI materials and coating variations tested exhibited a wide range of surface catalycity--from essentially fully catalytic to fully noncatalytic. This catalycity varied with the material and the coating and also varied with exposure time. For example (and as presented previously), at 2.3×10^7 J/kg (10,000 Btu/lb) enthalpy, one version of HCF exhibited an essentially fully catalytic surface for the first several cycles but became noncatalytic such that the ratio of radiation equilibrium heat flux to fully catalytic wall heat flux was about 0.70 after 30 cycles. The same flux ratio at the same enthalpy for the other three materials (LI-1500, REI, and SiC foam) was about 0.60 independent of exposure time. Tests on another version of HCF (as presented previously) over a broad enthalpy and flux range, 28×10^4 to 57×10^4 W/m^2 (25 to 50 Btu/ft^2sec), also exhibited this same 0.60 ratio independent of flux (enthalpy) level.

Surface emissivity exhibited wide variations from about 0.3 to 0.9; the former value corresponded to an early version of LI-1500 in the wavelength band 1.7 to 2.6 microns, and the latter value was typical of SiC foam independent of wavelength.

SURFACE CATALYCITY RESULTS

ALL MATERIALS AT NOMINAL 2.3×10^7 J/KG (10,000 BTU/LB) AND 3.4×10^5 W/M^2 (30 BTU/FT^2 SEC)

MATERIAL	$\frac{Q_{NONCAT}}{Q_{CAT}}$
LI-1500	0.60
HCF	0.95 TO 0.60
REI	0.60
SiC FOAM	0.60

Figure 12

CONCLUSIONS

(Figure 13)

The conclusions shown in this figure are based on the 288 sample hours of testing as exemplified by the results presented in the preceding figures.

CONCLUSIONS

RSI MATERIALS EXHIBIT A WIDE RANGE OF SURFACE EMITTANCE AND CATALYTIC ACTIVITY DEPENDING ON THE SURFACE COATING

RSI COATINGS (OR SURFACES) EXHIBIT A SIGNIFICANT NONCATALYCITY; THE POTENTIAL IMPACT IN TPS DESIGN FOR THE ORBITER VEHICLE IS ALSO SIGNIFICANT

THE INTERPRETATION OF TEST RESULTS RELATIVE TO SURFACE CATALYCITY FOR APPLICATION TO FLIGHT CONDITIONS REQUIRES AN ACCURATE KNOWLEDGE OF THE BOUNDARY LAYER CHARACTERISTICS

COATINGS CAN HAVE A SIGNIFICANT EFFECT ON THE OVERALL RSI RESPONSE

DUE TO DIFFERENCES IN SURFACE CATALYCITY AND EMITTANCE, TESTS AND COMPARISONS OF MATERIALS SHOULD BE MADE AT COMMON LEVELS OF HEATING RATE, NOT SURFACE TEMPERATURE

TEST MODEL SINGULARITY REGIONS WHICH MAY INFLUENCE MATERIAL RESPONSE AND WHICH ARE NOT TYPICAL OF THE FLIGHT APPLICATION SHOULD BE AVOIDED.

Figure 13

PHASE II CONTRACT TEST PROGRAM (NAS2-6600)

(Figure 14)

Material testing in the Phase II contract test program is starting in November 1972 and will emphasize RSI materials and carbon-carbon composites. The list of RSI materials and the test conditions are still subject to additions and revisions at this date.

PHASE II CONTRACT TEST PROGRAM (NAS2-6600)

- MATERIALS
  - RSI
    - LI-1500
    - HCF
    - REI
    - CPI
    - SiC LAMINATE
    - SiC FOAM
    - MAR-SI
  - CARBON-CARBON COMPOSITES
    - LTV
    - CONVAIR
  - METALLICS
    - COATED COLUMBIUM
- MODEL AND SAMPLE CONFIGURATIONS
  - SAME AS PHASE I EXCEPT NO MODEL CENTERPOST IN MOST TESTS
- TEST CONDITIONS
  - HEAT FLUX FROM 4.5×10^5 TO 11.0×10^5 W/M^2 (40 TO 95 $BTU/FT^2 SEC$)
  - STAGNATION PRESSURE OF 1.2×10^3 N/M^2 (0.012 ATM)
  - ENTHALPY FROM 1.7×10^7 TO 4.4×10^7 J/KG (7500 TO 19,000 BTU/LB)
- TEST PROCEDURE
  - SAME AS PHASE I EXCEPT 10 OR 15 MINUTE CYCLES

Figure 14

REFERENCE

1. Schaefer, John W., "Thermal Screening of Shuttle Orbiter Vehicle TPS Materials Under Convective Heating Conditions," Aerotherm Division, Acurex Corporation, Mountain View, California, Aerotherm Report No. 72-56, August 24, 1972, NASA CR (to be published).