# ENTRY ENVIRONMENTAL SIMULATION TESTING OF REI-MULLITE TPS

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#### INTRODUCTION

(Figure 1)

During the development of REI-Mullite for use in a multimission thermal protection system (TPS) with a 1644° K (2500° F) temperature capability, the General Electric Company's Re-entry and Environmental Systems Division (GE-RESD) has developed and used several specialized tests. These tests were used to obtain material response characteristics and to confirm the applicability of thermophysical properties obtained in laboratory experiments for design purposes. Specifically these studies have included: (1) determination of the melting temperature, noncatalytic nature and variation of total hemispherical emittance with temperature for the SR-2 family of coating materials; (2) evaluation of the adequacy of an analytical modeling approach for predicting thermal responses that uses laboratory measured values of thermal conductivity; and (3) derivation of an analytical modeling approach for routine shuttle orbiter TPS analyses that includes in-depth radiation transport on the insulative composites. The results of these studies are summarized in this paper.

High temperature surface emittance was obtained for coated Mod 1A REI-Mullite by exposing models in the GE-RESD and NASA-Ames plasma arcs until the surface temperatures stabilized, and measuring the incident heat fluxes and the resultant surface temperatures. The heat flux level to the model was increased in steps until there was evidence of coating melting and flowing. The coating emittance was then determined by a surface heat balance. The data obtained in the GE-RESD plasma arc compared favorably with laboratory measurements. However, SR-2 coated Mod IA REI-Mullite tiles displayed totally noncatalytic surface characteristics in the lower pressure tests reported by NASA-Ames.

Evaluation of the adequacy of the model for predicting the thermal response of REI-Mullite revealed some discrepancies in the accuracy of the prediction for the transient response, although the maximum structure temperatures were reasonably well predicted. Evaluation of spectral transmittance data indicated another heat transfer mechanism operative in the model, namely, a radiation shine in effect. The impact of the shine in effect on the bond-structure temperature response was found to be both surface temperature and REI thickness dependent. Shine-in coefficients have been derived from a steady state thermal soak test in a radiantly heated entry simulator. These coefficients, when used in conjunction with the guarded hotplate measured Mod IA REI-Mullite thermal conductivity data, produce excellent agreement between measured and predicted temperature response for both transient and steady state conditions.

The need for consideration of the real backside boundary conditions rather than the assumption of adiabatic conditions was shown to be important as a result of the analyses conducted relative to the NASA-MSC prototype panel test results.

#### INTRODUCTION

- OVERTEMPERATURE AND HIGH TEMPERATURE EMITTANCE EVALUATION
  - MELT TEMPERATURE
  - NONCATALYTIC SURFACE EFFECTS
  - SURFACE EMITTANCE
- THERMAL MODEL VERIFICATION
  - USE OF LABORATORY MEASURED THERMAL CONDUCTIVITY
  - INCLUSION OF RADIATION TRANSPORT
  - SENSITIVITY TO BACKSIDE BOUNDARY CONDITIONS

# HIGH TEMPERATURE EVALUATION OF COATED REI-MULLITE (Figure 2)

For a man-rated vehicle the coated REI-TPS must exhibit capability to survive off-nominal entry heating conditions, such as might occur in an aborted mission. It is necessary to know the surface temperature at which the coated REI starts to melt, and more important, the heat flux necessary to produce surface melting. Thus, the primary objective of this specific test program was to determine the heat flux and surface temperature required to melt the coating materials. Melting in this case is defined as the time at which material is visually flowing from the model. Secondary objectives were to determine the high temperature emissivity values and the catalytic nature of the coating.

These tests were conducted in the GE-RESD Hyperthermal Arc Facility using the tunnel mode of operation. In this mode, flow is delivered to the model at a low supersonic Mach number with a stagnation pressure of about 4137  $N/m^2$  (0.6 psia). Total enthalpy for these runs was nominally 1.64 x 10<sup>7</sup> J/Kg (7100 Btu/lb).

Heat flux was varied by moving the model closer to the nozzle during the run. Sting positioning was remotely controlled from the control room. All models were instrumented with Pt/Pt-10% Rh thermocouples located in the coating for recording the approximate surface temperatures. The test procedure was to incrementally increase the heat transfer rate in gradual steps up to the melt temperature. Prior to each increase the model was retracted for two to three seconds while a calorimeter was inserted to measure the heat flux.

A calorimeter coated with 1 mil of teflon gave a heat flux reading only 7 to 13 percent lower than an uncoated calorimeter. This indicated that, in the GE-RESD facility, there would be no difference observed between the temperature response of a fully noncatalytic or fully catalytic coating.

# OF COATED REI-MULLITE

PRIMARY OBJECTIVE: DETERMINE HEAT FLUX AND SURFACE TEMPERATURE TO MELT COATING

SECONDARY OBJECTIVES: DETERMINE HIGH TEMPERATURE EMISSIVITY VALUES OF COATING

DETERMINE CATALYTIC NATURE OF COATING

VARIABLES: HIGH TEMPERATURE EMITTANCE OF COATING

CATALYTIC RECOMBINATION RATE COEFFICIENT OF COATING MELTING AND/OR SUBLIMATION THRESHOLD OF COATED REI

TEST CONFIGURATION AT GE HYPERTHERMAL ARC:

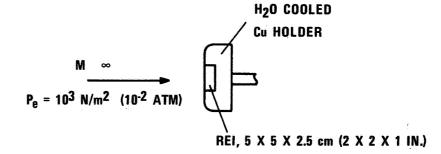


Figure 2

#### PLASMA ARC TEST RESULTS

(Figure 3)

The SR-2 coating system remained shape stable until a temperature of  $1967^{\circ}$  K ( $3080^{\circ}$  F) was reached. At that time the change in flow field appearance indicated products were being removed or vaporized from the coating. A heat flux of about  $44 \text{ w/cm}^2$  ( $39 \text{ BTU/ft}^2 \text{ sec}$ ) was required to reach this surface temperature.

Data comparing the variation of surface temperatures with hot wall heat fluxes in the GE-RESD and NASA-Ames\* test facility are presented in this figure. It is seen that the SR-2 coating exhibits totally noncatalytic surface characteristics for the combinations of test conditions used at NASA-Ames and fully catalytic characteristics for the GE-RESD test conditions.

<sup>\*</sup>Reported in the April 1972 Monthly Status Report for NASA Space Shuttle Structures and Technology Working Group

### PLASMA ARC TEST RESULTS

		HEATING RATE		PRESSURE		RECOVERY Enthalpy	
SYMBOL	FACILITY	BTU/FT <sup>2</sup> SEC	W/cm <sup>2</sup>	ATM	N/m <sup>2</sup>	BTU/LB	MJ/Kg
Δ	GE 5MW	ALL	ALL	4 X 10 <sup>-2</sup>	4 X 10 <sup>3</sup>	7100	1.65
0	NASA-AMES	20	18	7.5 X 10 <sup>-3</sup>	7.5 X 10 <sup>2</sup>	3510	0.82
0	NASA-AMES	30	27	7.5 X 10 <sup>-3</sup>	7.5 X 10 <sup>2</sup>	4189	0.97
. 0	NASA-AMES	40	35	7.5 X 10 <sup>-3</sup>	7.5 X 10 <sup>2</sup>	5800	1.35

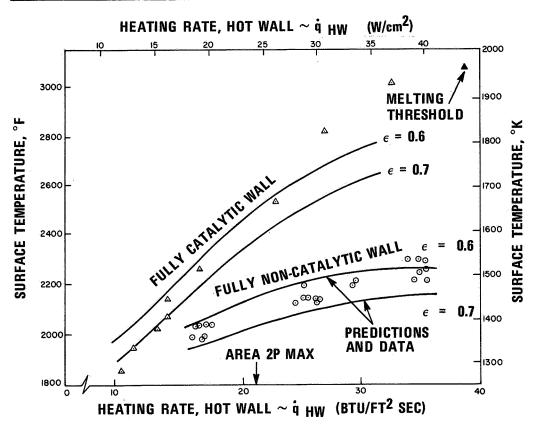


Figure 3

### SR-2 COATING SURFACE EMITTANCE

(Figure 4)

The emittance data deduced from the overtemperature assessment tests are compared with data measured calorimetrically on the SR-2 coating. It can be seen that the two independent sets of results compare favorably. The dimensional stability of the SR-2 coating up to 1967° K (3080° F) is excellent.

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### **SR-2 COATING SURFACE EMITTANCE**

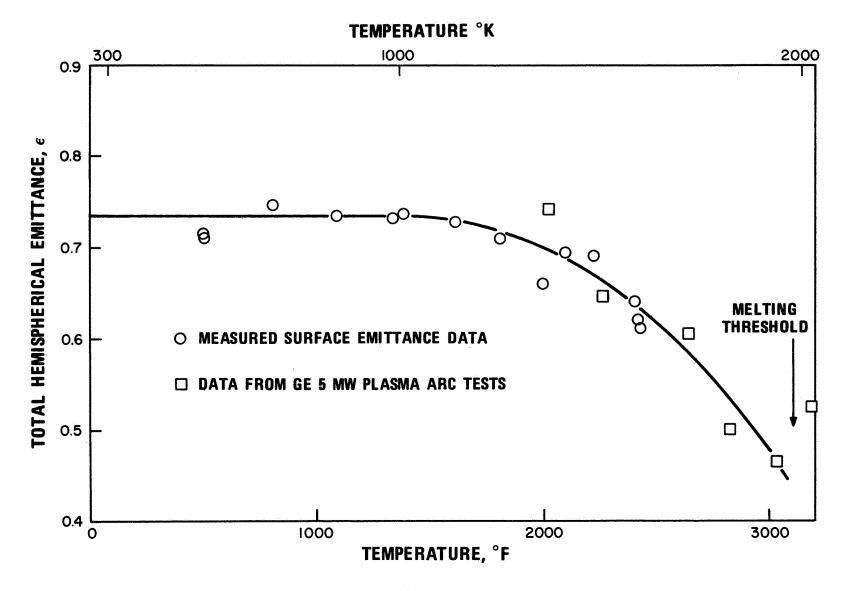


Figure 4

# SUMMARY OF HIGH TEMPERATURE CHARACTERISTICS OF COATED MOD IA REI-MULLITE

(Figure 5)

The melting temperature of the SR-2 coated Mod IA REI-Mullite has been established as about 1967° K (3080° F). This results in a large overtemperature capability for the material in relation to surviving off-nominal entry heating conditions.

The coating emittance determined from arc tests agree with that measured calorimetrically and has a value of ·0.73 to 1100° K (1600° F) where it tends to decrease to a small extent with increasing temperature.

The SR-2 coating system exhibited totally noncatalytic surface characteristics in the tests conducted at NASA Ames where flow conditions were conducive to measuring differences in net convective heat flux to catalytic and non-catalytic surfaces. Thus for the shuttle orbiter TPS, if flow conditions are such that during any portion of the entry, a significant portion of the total heat transfer is due to atomic recombination at the surfaces, the SR-2 coated Mod IA REI-Mullite will exhibit a fully noncatalytic response and run at significantly cooler surface temperatures.

# SUMMARY OF HIGH TEMPERATURE CHARACTERISTICS OF COATED MOD IA REI-MULLITE

- MELTING TEMPERATURE  $\sim 1967^{\circ}$ K (3080°F)
- COATING EMITTANCE  $\sim$  0.73 TO 1100°K (1600°F)
- SR-2 COATING IS NONCATALYTIC

### THERMAL DESIGN MODEL VERIFICATION

(Figure 6)

To verify the validity of the thermal design model used for Space Shuttle TPS design, high temperature thermal response tests were conducted on a 15 x 20 cm (6 in. x 8 in.) sample of coated REI-Mullite bonded to an aluminum backplate. The data have been used to determine the validity of the current thermal design model, both for the transient and steady state conditions of the tests. The tests were run at ambient pressure and at  $10^3 \text{ N/m}^2$  ( $10^{-2}$  ATM) pressure. Steady state conditions were achieved on the 5 cm (2 in.) thick model by heating the surface at constant temperatures of  $1370^{\circ}$  K ( $2000^{\circ}$  F) and  $1644^{\circ}$  K ( $2500^{\circ}$  F) and by providing cooling water at the aluminum backplate to maintain its temperature at about  $311^{\circ}$  K ( $100^{\circ}$  F). Steady state conditions were obtained after 40-55 minutes for the two tests.

The test facility consists of eight silicon carbide heaters spaced on 2.5 cm (1 in.) centers, enclosed in a foam quartz holder with water cooled terminal ends. The test samples are held with their backplates in contact with a water cooled copper plate. The test facility is operated within a vacuum chamber, with water cooled walls wherein the vacuum is maintained during the run. Tests were also run at ambient pressure.

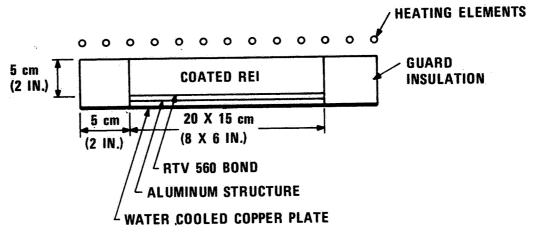
Control instrumentation in the coating consisted of platinum 10 percent rhodium/platinum (Type "S") thermocouples. Also recorded were in-depth REI, bond-line and backplate temperatures, using chromel-alumel thermocouples. The in-depth instrumentation was installed using a thermocouple plug. Exact locations of thermocouples were determined from x-ray photographs. Test surface temperature was controlled by one surface thermocouple and recorded with the other. Gaps between the model and the foamed quartz were stuffed with mullite fibers.

## THERMAL DESIGN MODEL VERIFICATION

### • PRIMARY OBJECTIVE:

VERIFY VALIDITY OF THERMAL DESIGN MODEL EMPLOYED FOR MOD IA REI-MULLITE

**■** CONFIGURATION



THERMOCOUPLES IN THE COATING AND THROUGH THE THICKNESS

### • TEST CONDITIONS:

- SURFACE TEMPERATURE ~ 1370°K (2000°F) AND 1644°K (2500°F)
- LOCAL PRESSURE  $\sim 10^3 \text{ N/m}^2 (10^{-2} \text{ ATM}) \text{ AND } 10^5 \text{ N/m}^2 (1 \text{ ATM})$

#### REI-MULLITE THERMAL RESPONSE AT 1 ATM TEST PRESSURE

(Figure 7)

The measured transient temperature responses for the ambient pressure test are shown. The surface temperature was driven to 1370° K (2000° F) in about 10 minutes and was increased to 1644° K (2500° F) after a time period of 25 minutes at 1370° K (2000° F). Analytical predictions were made using the surface and backplate temperature histories as boundary conditions in the one-dimensional thermal analysis using the Reaction Kinetics Ablation Program (REKAP). The predictions were made using the  $10^5$  N/m<sup>2</sup> (1 ATM) thermal conductivity design curve shown on Figure 9. As can be seen, there is reasonable agreement between the test data and the analytical predictions.

# REI-MULLITE THERMAL RESPONSE AT 1 ATM TEST PRESSURE

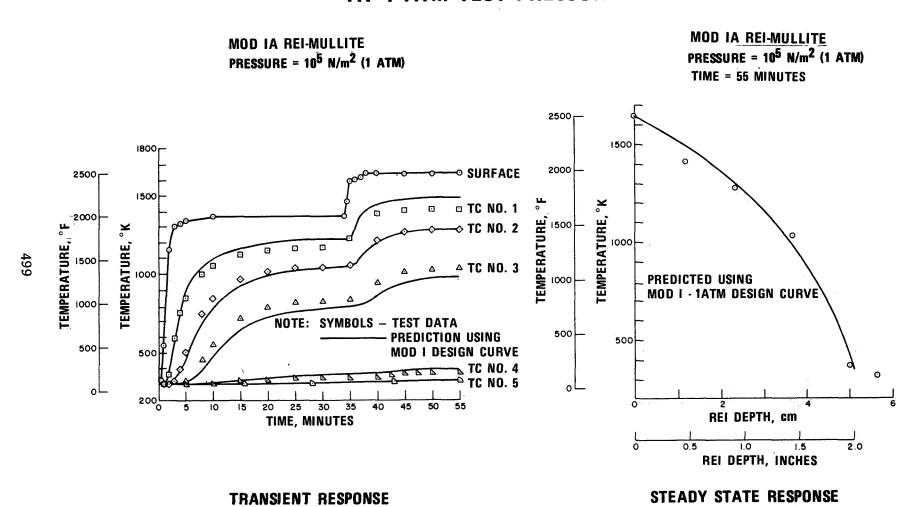


Figure 7

## REI-MULLITE THERMAL RESPONSE AT 10-2 ATM TEST PRESSURE

(Figure 8)

The measured transient temperature response from the  $10^3$  N/m $^2$  ( $10^{-2}$  ATM) test is shown compared with predictions using REKAP and the design thermal conductivity curve shown on Figure 9 for  $10^3$  N/m $^2$  ( $10^{-2}$  ATM) pressure. Again the overall comparison is reasonably good, but the in-depth predicted transient response is lagging behind the data.

It is interesting to note that the steady state in-depth temperatures for the  $10^5 \, \text{N/m}^2$  (1 ATM) pressure tests are lower than for the  $10^3 \, \text{N/m}^2$  ( $10^{-2} \, \text{ATM}$ ) pressure test. This is also predicted with REKAP and the reason is that for the  $10^5 \, \text{N/m}^2$  (1 ATM) pressure test more heat is flowing through the sample and being removed through the backplate into the cooling water than for the reduced pressure test.

### REI-MULLITE THERMAL RESPONSE AT 10-2 ATM TEST PRESSURE

#### TRANSIENT RESPONSE

#### STEADY-STATE RESPONSE

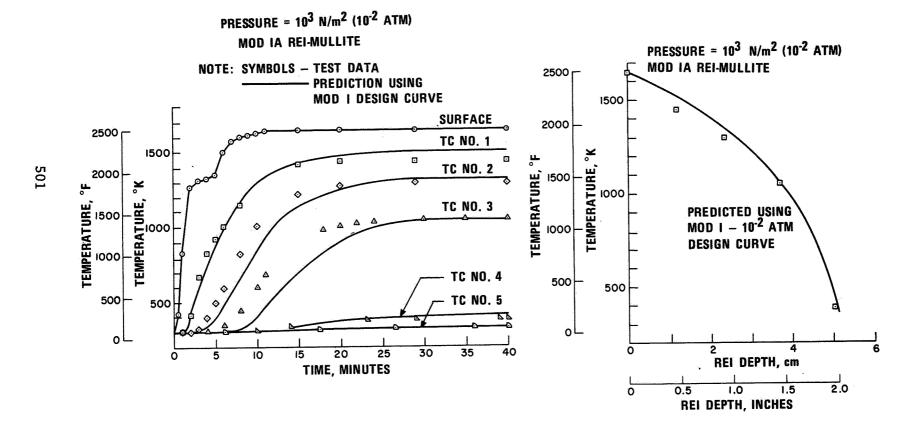


Figure 8

#### ESTABLISHMENT OF REI-MULLITE THERMAL DESIGN MODEL

(Figure 9)

Additional predictions are shown based on the thermal conductivity reported by Reference 1, and a fairing through the guarded hotplate Mod 1A REI-Mullite measured data. The prediction based on the Battelle Memorial Institute data shows extremely high temperatures compared to the data. Also shown is a fairing through the Mod IA data, which underpredicts the in-depth data. A comparison of all thermal conductivity values used in the analysis is shown.

The data show the Mod I REI-Mullite design curve provides reasonable agreement with test results, but requires modification above about 1255°K (1800°F).

**ESTABLISHMENT OF REI-MULLITE** 

THERMAL CONDUCTIVITY DESIGN CURVES

COMPARISON OF STEADY STATE THERMAL PREDICTIONS

Figure 9

## SUMMARY OF THERMAL DESIGN MODEL VERIFICATION ANALYSES (Figure 10)

The following conclusions can be drawn from the thermal design verification studies and analyses. They are:

- 1. Use of Mod I REI-Mullite thermal conductivity design curves provides reasonable agreement with test results but requires modification above about 1255° K (1800° F).
- 2. Use of GE-RESD measured guarded hot plate thermal conductivity data for Mod IA REI-Mullite does not improve the modeling accuracy.
- 3. Use of Battelle Memorial Institute measured thermal conductivity data provides poor agreement with test results.

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# SUMMARY OF THERMAL DESIGN MODEL VERIFICATION ANALYSES

- MOD I REI-MULLITE THERMAL CONDUCTIVITY DATA DESIGN CURVES PROVIDE REASONABLE AGREEMENT WITH TEST RESULTS BUT REQUIRE MODIFICATION AT HIGH TEMPERATURES
- MOD IA REI-MULLITE THERMAL CONDUCTIVITY DATA DOES NOT IMPROVE MODELLING ACCURACY
- BATTELLE MEASURED THERMAL CONDUCTIVITY DATA FOR MOD IA REI-MULLITE PROVIDE POOR AGREEMENT WITH TEST RESULTS

#### REI-MULLITE TRANSMITTANCE

(Figure 11)

An evaluation was performed to determine if other modes of heat transfer might be present that, if properly accounted for, could improve the accuracy of the predictions. As a result, transmittance measurements for three different thicknesses of 181 Kg/m<sup>3</sup> (11.3 pcf) density Mod IA REI-Mullite were performed utilizing the Gier-Dunkle intergrating sphere reflectometer. The measurements are a normal incidence/hemispherical exit transmittance type that collects all the energy transmitted by the specimen. The results are compared to those reported in Reference 2 for the MOD III HCF.

### **REI-MULLITE TRANSMITTANCE**

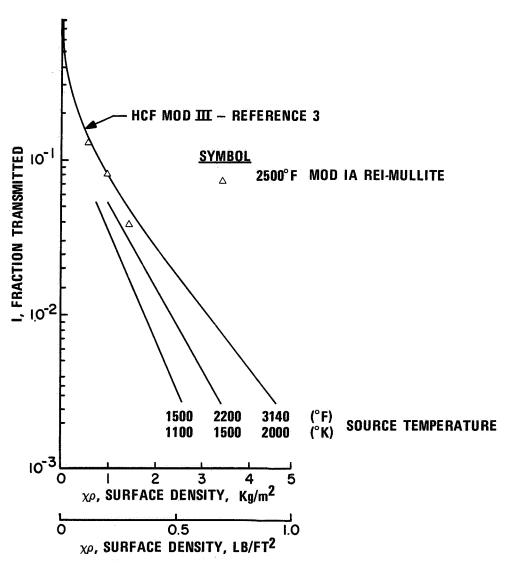


Figure 11

# SHINE-IN PENETRATION DEPTH FOR AREA 2P PROTOTYPE PANEL TEST\*

The quantity of heat conducted into the REI-Mullite is small during the major portion of the heating period. The transmittance data indicate an additional heat input is incurred in the outer 1.25 cm (1/2 in.) of the REI due to shine in from the backside of the coating. These shine in depths were computed as the depth where the heat input due to shine in had decreased to 10% of the heat conducted into the REI surface. It is seen that the shine-in effect is dominant only in the outer 1.25 cm (1/2 in.) of the REI-Mullite.

<sup>\*</sup>Details of design of NASA-MSC Area 2P prototype panels are discussed in GE-RESD final report on Contract No. NAS 9-12084 dated May 1972.

# SHINE-IN PENETRATION DEPTH FOR AREA 2P PROTOTYPE PANEL TEST

#### **MODULATED PRESSURE**

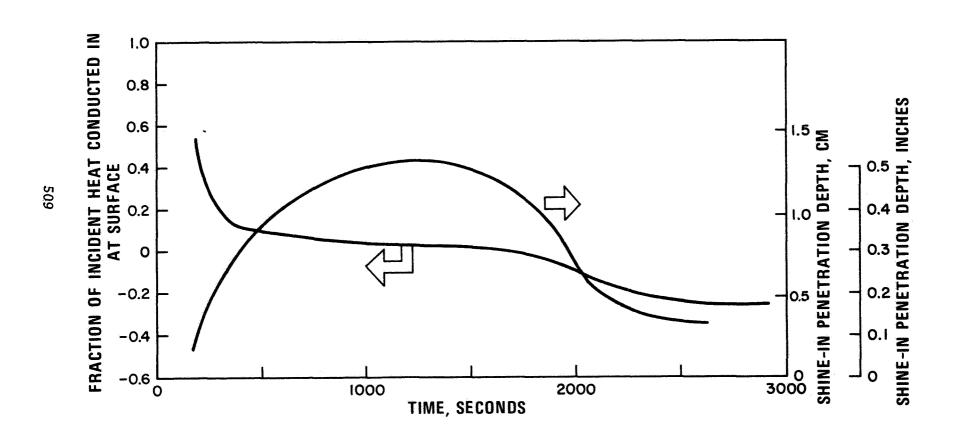


Figure 12

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## SHINE-IN CORRECTION EFFECTS ON THERMAL DESIGN MODEL

(Figure 13)

To properly handle this shine in mode of heat transfer analytically, one must resort to a computerized solution of the governing heat transfer differential equations. These equations must include the radiant heat transmitted, absorbed, and backscattered through each element of the material. However, such programs are not now in operation, and it is necessary to use an approximate technique for routine design work.

Consideration of the transmittance characteristics of the Mod IA REI-Mullite indicates that the total effect of shine in on bond and structure temperature response is both surface temperature and REI-Mullite thickness dependent. For the steady-state tests conducted on the 5 cm (2 in.) thick model with surface temperature at 1370 K (2000 F) and 1644 K (2500 F), shine in coefficients have been derived. These coefficients have been employed in combination with the Mod IA REI-Mullite thermal conductivity data to predict the steady state response of the 5 cm (2 in.) thick REI model.

# SHINE-IN CORRECTION EFFECTS ON THERMAL DESIGN MODEL

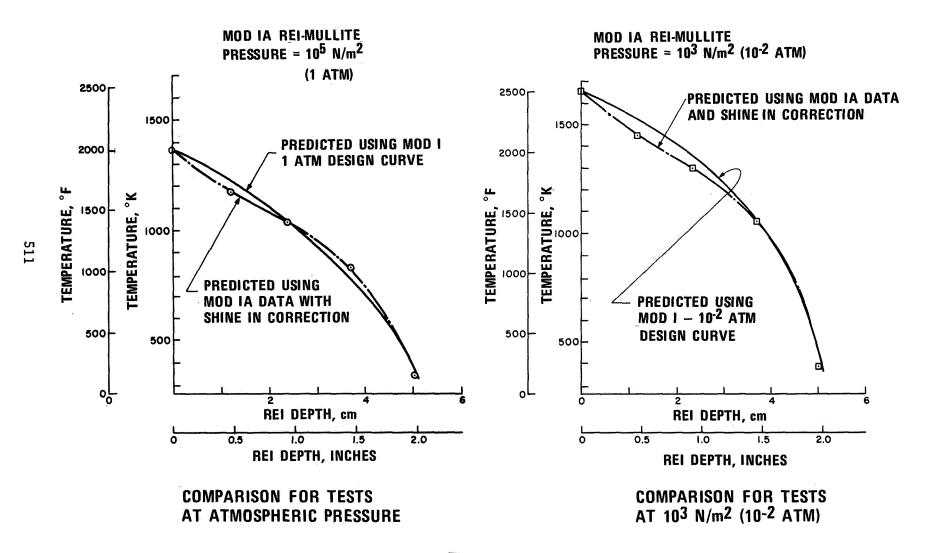


Figure 13

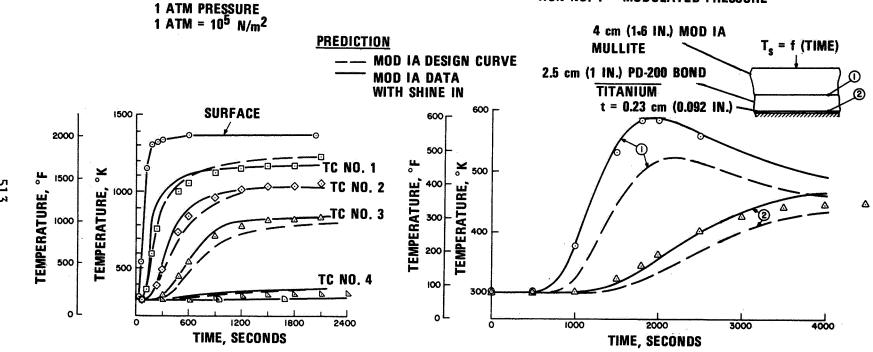
# COMPARISON OF REI-MULLITE TRANSIENT TEMPERATURE RESPONSE WITH PREDICTIONS MADE WITH AND WITHOUT SHINE-IN CORRECTIONS

(Figure 14)

Employing the shine in coefficients derived from the steady state tests and fairings through the Mod IA REI-Mullite thermal conductivity data, predictions of the transient response have been made and compared to predictions made employing the Mod I REI-Mullite thermal conductivity design curves. These comparisons have been made for several different tests run both at GE-RESD and NASA-MSC. It can be seen that consideration of all the heat transfer mechanisms, including both conduction and in-depth shine in from the coating backside, is necessary to accurately predict the transient temperature response.

# COMPARISON OF REI-MULLITE TRANSIENT TEMPERATURE RESPONSE WITH PREDICTIONS MADE WITH AND WITHOUT SHINE-IN CORRECTIONS

NASA MSC AREA 2P PROTOTYPE PANEL TEST RUN NO. 1 — MODULATED PRESSURE



IN-DEPTH TRANSIENT RESPONSE OF MOD IA REI-MULLITE FROM GE-RESD TESTS

REI/BOND AND BOND/STRUCTURE
TRANSIENT RESPONSE FOR NASA-MSC
AREA 2P PROTOTYPE PANEL

Figure 14

# COMPARISON OF REI-MULLITE TRANSIENT TEMPERATURE RESPONSE WITH PREDICTIONS

(Figure 15)

Employing the thermal model derived from the steady-state tests, predictions of the transient response have been made with and without proper thermal modeling of the backside boundary conditions. These comparisions have been made for a test run at NASA-MSC. It is seen that the impact of proper treatment of this boundary condition on the predicted structure response is about 16° K (30° F).

# COMPARISON OF REI-MULLITE TRANSIENT TEMPERATURE RESPONSE WITH PREDICTIONS

NASA MSC AREA 2P PROTOTYPE PANEL TEST RUN NO. 1

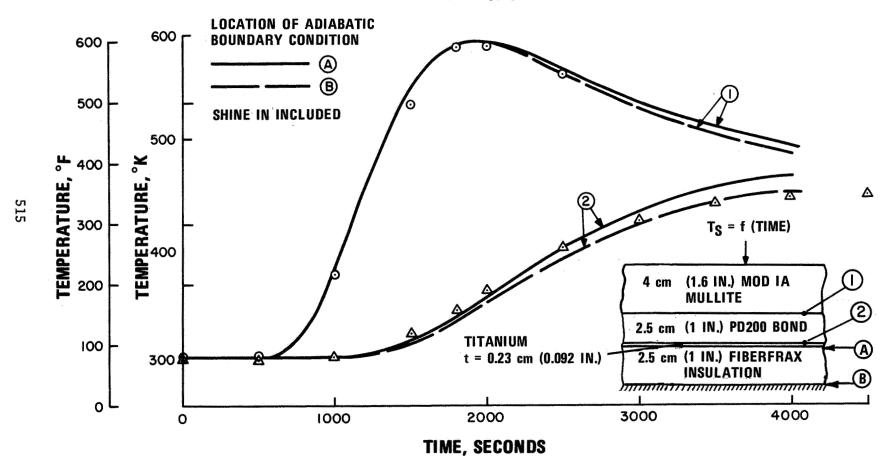


Figure 15

### FUNCTIONAL DEPENDENCE OF SHINE-IN COEFFICIENTS

(Figure 16)

The functional dependence of the shine-in coefficients employed in these analyses is illustrated in this figure. For a constant model thickness, the integrated through-the-model-thickness effect of these shine-in coefficients is seen to diminish rapidly with reduced surface temperatures.

Shine-in effects for REI-Mullite results from the fact that the voids between fibers are relatively large compared to the wavelength of the radiation producing the thermal energy transfer. The shine-in phenomenon will be minimized or eliminated when the smaller diameter (1.7  $\mu$ m) mullite fibers now under development by B&W become available and are utilized in the insulation composites.

# OF SHIRE-IN COEFFICIENTS

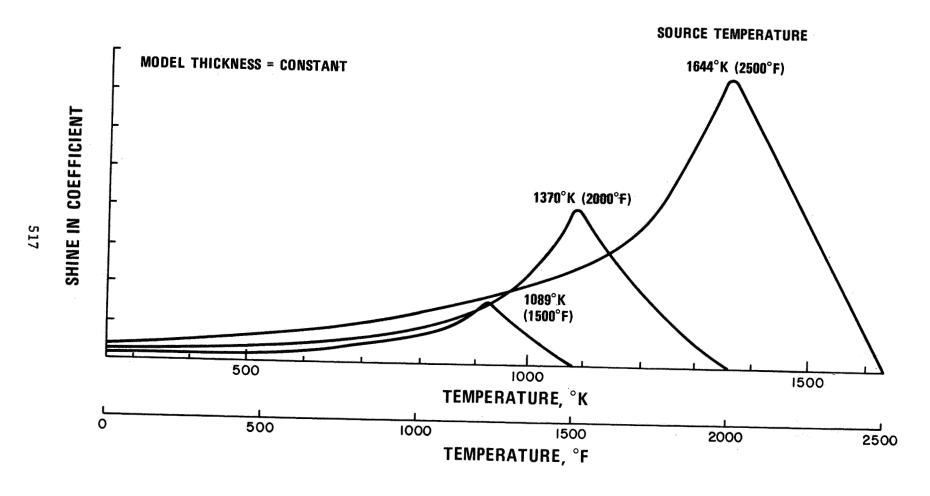


Figure 16

## THERMAL MODEL VERIFICATION SUMMARY

(Figure 17)

Prediction of through the thickness temperature gradients and maximum soak out temperatures is improved through use of an approximate thermal model that includes all of the heat transfer modes and an accurate definition of the structural backside thermal boundary condition. Predicted maximum structure temperature increases 16° K (30° F) for the 1530° K (2300° F) (Area 2P) maximum surface temperature environment, but it is not expected to change for the 980° K (1300° F) (Area 1) maximum surface temperature environment.

## THERMAL MODEL VERIFICATION SUMMARY

- INCLUSION OF SHINE-IN EFFECTS IMPROVES ACCURACY OF TRANSIENT TEMPERATURE PREDICTIONS.
- INCLUSION OF PROPER STRUCTURE THERMAL BOUNDARY CONDITION IS NECESSARY TO ADEQUATELY PREDICT STRUCTURE TEMPERATURE RESPONSE.

### THERMAL STRESS IMPLICATIONS OF SHINE-IN GRADIENT

(Figure 18)

The effect of shine-in gradients on thermal stress is shown at critical times during heat up for an Area 2P environment. Note that there is negligible difference at this time when the thermal stresses are computed to be maximum. The small increase required in REI-Mullite thickness for this case has no effect on the critical stress/strain parameters.

# THERMAL STRESS IMPLICATIONS OF SHINE-IN GRADIENT

#### NASA-MSC PROTOTYPE PANEL TEST

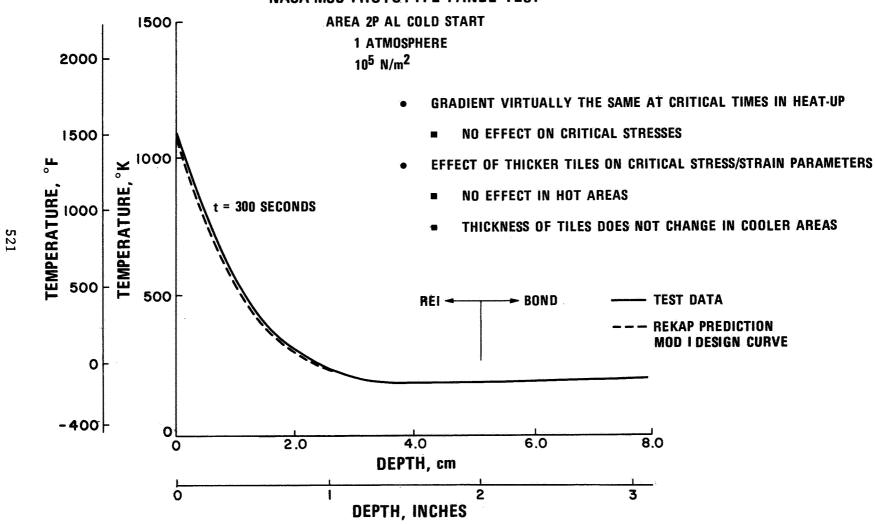


Figure 18

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#### **SUMMARY**

(Figure 19)

The GE-RESD SR-2 coating exhibits totally noncatalytic surface characteristics in those tests where flow conditions are conducive to measuring differences in net convective heat flux to catalytic and noncatalytic surfaces.

The Mod I thermal conductivity design curves and the Mod IA REI-Mullite thermal conductivity data yield much better agreement between predicted and measured in-depth temperature response than do the Battelle Memorial Institute data.

Prediction of through-the-thickness temperature gradients is greatly improved by inclusion of shine-in effects. Inclusion of these shine-in effects has only a limited impact on the severity of predicted thermal stresses, structure temperatures, and TPS system weights (when compared to the weights previously generated employing the Mod I design curves).

#### **SUMMARY**

- SR-2 COATING SYSTEM EXHIBITS NONCATALYTIC SURFACE CHARACTERISTICS WHICH LIMIT SURFACE TEMPERATURE RISE UNDER CERTAIN CONVECTIVE HEAT FLUX CONDITIONS.
- GE-RESD MOD I REI-MULLITE THERMAL CONDUCTIVITY DESIGN CURVES PROVIDE MUCH BETTER AGREEMENT WITH TEST RESULTS THAN DOES BATTELLE MEMORIAL INSTITUTE DATA.
- INCLUSION OF SHINE-IN EFFECTS RESULTS IN IMPROVED ACCURACY OF TRANSIENT TEMPERATURE GRADIENT PREDICTIONS.
- SHINE-IN EFFECTS HAVE LIMITED IMPACT ON SEVERITY OF THERMAL STRESSES, PREDICTED STRUCTURE TEMPERATURE OR TPS SYSTEM WEIGHT.

#### REFERENCES

- 1. Kissler, C. W., et al., "Evaluation of Non-Metallic Thermal Protection Materials for the Manned Space Shuttle," Battelle Columbus Laboratories, Columbus, Ohio, Contract NAS 9-10853 Final Report, Volume V, 1 June 1972.
- 2. Hughes, T. A., "High Temperature Insulation Materials for Radioactive Thermal Protection Systems," Report No. MDC E0666, 19 July 1972.