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**EFFECT OF THE CHEMICAL STATE OF
PYROLYSIS GASES ON HEAT-SHIELD MASS**

by William D. Brewer, C. W. Stroud, and Ronald K. Clark.

Langley Research Center

Langley Station, Hampton, Va.



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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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EFFECT OF THE CHEMICAL STATE OF PYROLYSIS GASES ON HEAT-SHIELD MASS

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SUMMARY

Results are presented from an analytical investigation to determine the effect of the chemical state of the pyrolysis gases on the heat-shield mass required for a reentry vehicle in a given environment. Mass per unit area of ablative material required is calculated for a number of constant-heating-rate environments and the total mass is calculated for a lifting-body vehicle in a typical reentry trajectory. The required mass of ablative material is calculated for three different assumptions: (1) The pyrolysis gases are in chemical equilibrium at all times, (2) the gases are chemically frozen at 500° K, and (3) the gases change from the frozen state to equilibrium over some temperature range.

The results show that in all calculations considerably more material is required if the gases remain chemically frozen as initially formed than if the gases are in equilibrium at all times. For the lifting-body vehicle, 28 percent more material is required if the gases are chemically frozen than if the gases are in chemical equilibrium. Some experimental data are given which indicate that the transition from the frozen state to equilibrium may take place above 1400° K and over a temperature range of about 200° K.

INTRODUCTION

Charring ablators have been found to be efficient heat-shield materials and are commonly used to protect spacecraft reentering the earth's atmosphere. Therefore, this type of ablative material has been subjected to considerable investigation.

The behavior of a charring ablator subjected to a heating environment can be briefly described, qualitatively, as follows: As the material is heated, it decomposes giving off a quantity of gases (referred to as pyrolysis gases) and leaving a carbonaceous residue commonly known as the char. The gases are heated as they travel through the char layer and various chemical reactions occur. The gases are eventually injected into the boundary layer.

The actual thermochemical processes which take place as the ablative material decomposes are numerous and very complex and at present are not well understood.

Considerable work has been done in an attempt to determine the composition and chemical state of the pyrolysis gases as well as to characterize completely the specific reactions which occur within the gases themselves and which take place between the gases and the char. (See refs. 1 and 2, for example.) However, little has been done toward determining whether any of these factors is important in determining the overall response of a material to a given reentry environment. Some information concerning the effect of the specific heat of the gases on material performance in a ground test environment is reported in reference 3.

In analytical studies of the charring process, the practice has been to assume that the gases are either in chemical equilibrium or are chemically frozen at all times. It is conceivable that significantly different results may be obtained when different assumptions are made concerning the state of the gases. This paper represents the results of an analytical investigation to determine the effect of the chemical state of the pyrolysis gases on the mass of ablative material required for a reentry vehicle in various environments. Mass per unit area of ablative material required is calculated for constant-heating-rate environments with the heating rates ranging from 0.1 to 5.7 MW/m². In addition, the total mass of the heat shield required for a lifting-body vehicle in a typical reentry trajectory is calculated. Required mass of ablative material is calculated for three different assumptions: (1) Pyrolysis gases are in chemical equilibrium at all times, (2) the composition of the gases is frozen at 500^o K and remains frozen at all higher temperatures, and (3) the gases are allowed to change from the frozen state to equilibrium at some transition temperature. The transition temperature is defined as that temperature at which the reactions begin which eventually lead to the equilibrium state. The reactions are assumed to take place over a temperature range of 200^o K. Calculations are made for transition temperatures ranging from 800^o K to 1700^o K.

Some experimental data are presented to give an indication as to when transition might occur. The data are limited to results from tests with methane, one of the principal gases of pyrolysis.

SYMBOLS

The units used for the physical quantities defined in this paper are given in the International System of Units (SI). Factors relating the International System with other systems of units are given in reference 4.

\bar{c}_p	specific heat of gases of pyrolysis, Joules/kilogram-degree Kelvin (J/kg- ^o K)
m	unit mass of ablative material required to limit back-surface temperature to 530 ^o K, kilograms/meter ² (kg/m ²)

M total mass of ablative material, kilogram (kg)

q heating rate, watts/meter² (W/m²)

T temperature, degrees Kelvin (°K)

X_{H₂} mole fraction of hydrogen

Subscripts:

s stagnation-point value

tr transition

ANALYTICAL PROGRAMS

Equilibrium Program

An analytical program has been developed with which the equilibrium composition of the pyrolysis gases resulting from the decomposition of a charring ablator can be determined. This analysis can be used to calculate the equilibrium composition and enthalpy of the pyrolysis gases as functions of temperature for a given initial atomic composition. The method used to calculate the equilibrium composition is based on the minimization of the free energy. At present, the program is capable of handling 100 species, 90 of which are gaseous and 10 of which are condensed. Each species may be composed of as many as 10 elements. This program is referred to herein as the equilibrium program.

Charring-Ablator Program

The equations governing the transient response of ablative materials to a heating environment are, in general, nonlinear partial-differential equations with variable coefficients. The appropriate equations are developed in reference 5 and have been programmed at the Langley Research Center for numerical solutions on a high-speed digital computer. The computer program yields temperature distributions within a material as well as dimensional changes of the material as functions of time for a material subjected to a heating environment. For the present investigation, a program which utilizes an implicit method of solution of the equations in reference 5 was used. The analysis in reference 5 is one-dimensional and the assumptions are that the thermal properties of a given layer of material are functions of temperature only, that the pyrolysis gases traveling through the char are at the same temperature as the char, and that pyrolysis occurs

in an infinitely thin plane (i.e., reaction-front analysis). Previous investigations have shown that the reaction-front analysis gives results which are in good agreement with the results from a reaction-zone analysis. For the present investigation, the only method of char removal is oxidation.

ANALYTICAL PROCEDURES

Determination of Composition and Enthalpy of Pyrolysis Gases

The analysis in reference 5 (i.e., the charring-ablator program) does not consider, directly, the chemical kinetics of the pyrolysis gases as the gases travel through the char. That is, the particular gases, reactions, reaction constants, and so forth, which may be involved in the ablative process are not specified in the program. The chemical kinetics are, however, taken into account indirectly by specifying the specific heat of the gaseous mixture (designated \bar{c}_p). The specific heats are determined with the aid of the equilibrium program discussed previously.

The equilibrium program was used to determine the equilibrium composition as well as the enthalpy of the gases as functions of temperature. Figure 1 shows the equilibrium composition of the pyrolysis gases of phenolic-nylon at a pressure of 1 atmosphere. Mass fraction of the species present is plotted as a function of temperature. A mass fraction of 1.0 corresponds to the total mass of ablation products – solid and gaseous. Only the gas-phase composition is given since the solid material produced does not affect the present results (i.e., does not affect \bar{c}_p). A total of 38 molecular species was included in the equilibrium calculations. The species which appear in trace amounts are not shown since they have negligible effect on the results.

From the known composition of the gases, the enthalpy is determined and plotted as a function of temperature as shown in figure 2. Figure 2 shows the enthalpy of the pyrolysis gases for the three different cases considered: the equilibrium and frozen cases, and a typical transition case. The value for the equilibrium specific heat is determined from the slope of the enthalpy curve at various temperatures. The value for the frozen specific heat is determined from the composition of the gas mixture at 500° K and is constant for all temperatures.

For the transition cases, the composition of the pyrolysis gases is assumed to be frozen at 500° K and to remain frozen up to some transition temperature.

The transition reactions are assumed to take place over a temperature interval of 200° K. Therefore, at a temperature equal to the transition temperature plus 200° K, the gases are in complete equilibrium. To describe this situation in the charring-ablator program, combinations of the frozen \bar{c}_p and equilibrium \bar{c}_p are used. The frozen \bar{c}_p

is used up to the transition temperature. After transition, the equilibrium \bar{c}_p is used. In the transition range, the value for \bar{c}_p is determined from the slope of the transition enthalpy curve (fig. 2).

Figure 3 shows the specific-heat curves as obtained from the data given in figure 2. These curves are typical of those used as inputs in the charring-ablator program. Specific-heat curves for transition temperatures other than 1100° K are similar to the one shown in the figure. Calculations are made for transition temperatures of 800°, 1100°, 1400°, and 1700° K. With this procedure, it can be determined whether the temperature at which the equilibrium reactions occur has any effect on the required heat-shield mass.

Calculations of Mass of Ablative Material


The heat-shield system consists of the ablative material backed by a 0.25-cm-thick aluminum sheet simulating the skin over the vehicle substructure. The charring-ablator program, with the appropriate \bar{c}_p input, was used to determine the thickness of ablative material required to limit the maximum back-surface temperature of the aluminum sheet to 530° K when subjected to the various heating environments. From the results of these calculations and the known density of the ablative material, the unit mass (mass per unit area) required to limit the back-surface temperature to 530° K was determined as a function of heating rate. For the lifting-body case, the thickness of the material required was calculated for various locations on the surface of the reentry vehicle. The unit mass required was then determined as a function of q/q_s . By using these results in conjunction with the known heating-rate distribution over the surface of the vehicle, the unit mass of ablative material required was determined as a function of the percent vehicle surface area requiring this unit mass. The total mass was then determined by integrating the mass-distribution curve over the entire surface of the vehicle. Calculations of the mass were made for each of the three different pyrolysis-gas assumptions.

HEATING ENVIRONMENTS AND THERMAL-PROTECTION MATERIAL

Heating Environments

For each of the constant-heating-rate environments, the heating time and the stream enthalpy are 1500 seconds and 232 MJ/kg, respectively. Calculations are made for heating rates ranging from 0.1 to 5.7 MW/m². These values are in the range typical of manned reentry.

The vehicle used for the lifting-body calculations is characterized by a lift-drag ratio of about 1.5 and has a total surface area of 62 m². The assumed laminar heating-rate distribution over the vehicle is shown in figure 4.



A nominal, constant-altitude, maximum-lift-drag-ratio reentry is used for the lifting body. The stagnation-point heating history is given in figure 5. The maximum stagnation-point heating rate encountered is 1.08 MW/m^2 . The total heat load at the stagnation point is 1.2 GJ/m^2 .

Thermal-Protection Material

The thermal-protection material used is a low-density charring ablator commonly known as low-density phenolic-nylon. This material is composed of a nylon powder (50 percent by mass), a phenolic bonding material (25 percent), and cured phenolic resin in the form of minute hollow spheres (25 percent). The material property values used as inputs in the charring-ablator program are given in table I. With the exception of the thermal conductivity of the char, the values given are measured values obtained from references 6 and 7. The char conductivity given in table I has been used previously with the measured property values to obtain good agreement between calculated and experimental results.

RESULTS AND DISCUSSION

Analytical

The analytical results from this investigation are given in figures 6 to 9. Figure 6 shows the unit mass of ablative material required to limit the back-surface temperature to 530°K as a function of heating rate for the constant-heating-rate environments. Considerable differences exist between the results for the various pyrolysis-gas assumptions. As would be expected, for a given value of the heating rate, the equilibrium \bar{c}_p case requires the smallest amount of material and the frozen \bar{c}_p case requires the largest amount of material.

For a wide range of heating rates, the unit mass required increases with increasing transition temperature. As the transition temperature is increased, the temperatures throughout the material reach a higher value before the heat absorption associated with the transition process becomes available. The result is that the additional heat which must be accommodated to keep the back surface at the prescribed temperature is accommodated by increased pyrolysis at the interface. Therefore, in general, the higher transition temperatures require more material to limit the back-surface temperature to the given value. Equivalently, it is noted that for the lower transition temperatures, the heat absorption due to the transition reactions takes place deeper within the material than that for the higher transition temperatures (i.e., near the char—uncharred-material interface). As the transition temperature is increased, the location of the heat absorption moves nearer the front surface of the material with resulting loss in thermal-protection effectiveness.

As the heating rate is decreased, the unit mass required in all cases approaches that required for the frozen \bar{c}_p case. At very low heating rates, the temperatures within the material do not reach the prescribed transition temperature. Therefore, the material responds as if the pyrolysis gases were chemically frozen throughout the char layer.

At the higher heating rates, the results for the transition cases approach those for the equilibrium case. As the heating rate is increased, the temperature gradients through the char layer become greater and the temperatures near the interface are increased. Therefore, the point within the char layer at which the transition reactions take place moves nearer the interface. Since the temperature gradients are large, a relatively large change in transition temperature corresponds to only a very small change in location of the transition point with respect to the interface. Hence, the temperature at which the transition reactions take place becomes unimportant.

Results given in figure 6 indicate that there is little difference between the mass requirement for the 800° K transition case and that for the equilibrium case for the constant-heating-rate environments. However, it will be shown that for the lifting-body reentry vehicle considered in this paper, this small difference in unit mass at low heating rates can result in a significant difference in the total mass of ablative material required.

Figure 7 shows the unit mass of material required for the lifting-body vehicle in the environment given in figure 5. The data indicate the same behavior as in the low-heating-rate portion of figure 6. Again, significantly different results are obtained with the different pyrolysis-gas assumptions.

Figure 8 shows the unit-mass distribution over the surface of the lifting vehicle. The unit mass required to limit the back-surface temperature to 530° K is plotted against percent vehicle area for each of the pyrolysis-gas assumptions. The total mass of ablative material required is determined by integrating the appropriate mass-distribution curve over the entire surface of the vehicle and is shown in figure 9.

From figure 8 it is apparent that there would be little difference between the total mass required for the 1400° K and 1700° K transition cases and the frozen case even though the data in figure 7 showed rather large differences between these three cases for heating rates greater than $0.4q_s$. The reason for these results is that less than 3 percent of the vehicle surface area is exposed to heating rates greater than $0.4q_s$. (See fig. 4.) Therefore, a relatively large increase in the unit mass at points with heating rates greater than $0.4q_s$ results in a small increase in the total mass since only a small area of the surface is affected. The opposite is true for the low-heating-rate area. For example, in figure 7, there appears to be little difference between the results for the 800° K transition case and those for the equilibrium case. However, from figure 8, there is a significant difference in the results for these two cases. This behavior results from the fact that the

differences in unit mass required for the two cases occur at heating rates less than about $0.3q_S$; and, from figure 4, it can be seen that more than 90 percent of the vehicle surface area is subjected to heating rates less than $0.3q_S$. Therefore, a relatively small increase in the unit mass at points with heating rates less than $0.3q_S$ results in a large increase in total mass since a large area of the surface is affected.

In figure 9, total mass of ablative material required is plotted as a function of transition temperature for the lifting-body vehicle. The 500°K transition temperature corresponds to the equilibrium \bar{c}_p case. Pyrolysis of the ablative material begins at about 500°K , and since the gases are in equilibrium at all times, this temperature can be considered an effective transition temperature. The point at 1900°K corresponds to the frozen \bar{c}_p case. This temperature is the maximum temperature reached by the gases in any of the calculations for the lifting body. Transition temperatures above 1900°K would produce the same results as the frozen \bar{c}_p would.

It can be seen in figure 9 that the total amount of ablative material required is less for lower transition temperatures. The frozen \bar{c}_p requires about 28 percent more mass than does the equilibrium \bar{c}_p . Above 1400°K , changes in transition temperature have little effect on the total mass of ablative material required. However, below 1400°K , significant reductions in the mass required are obtained with lower transition temperatures.

Experimental

An experimental investigation has been undertaken at the Langley Research Center to obtain information concerning the behavior of pyrolysis gases as they move through the char layer. The experiment consists of passing methane gas through a hot porous-carbon slab at a known temperature, sampling the gas as it exits from the hot slab, and then determining the composition of the gas sample. The experimental procedures and apparatus are described in more detail in the appendix. From equilibrium theory, it has been determined that at temperatures above 1000°K , the equilibrium molecular composition of the gas-phase mixture resulting from the decomposition of methane is essentially hydrogen. The carbon in the methane gas goes to a solid state. Therefore, the mole fraction of hydrogen in a mixture of gas resulting from the decomposition of methane is an indication of the extent to which the decomposition reactions have been completed. If the mixture is almost all hydrogen, the reactions are nearly complete and the mixture is essentially in chemical equilibrium.

Some experimental data on the mole fraction of hydrogen present in the gas emerging from a hot carbon slab are shown in figure 10, where the mole fraction of hydrogen is plotted as a function of temperature; a mole fraction of 1.0 corresponds to the chemical-equilibrium state of the gas. It can be seen that most of the decomposition

reactions take place in the temperature range from 1500° K to 1700° K. The experimental data represent the results of tests with one particular type of commercially available carbon and only one component of the pyrolysis gases. If an actual char and pyrolysis gases were used in the experiment, the results might be altered somewhat (i.e., transition temperature might be different). Also, the mass flow rate of methane through the carbon slab was from 5 to 10 times greater than the pyrolysis-gas flow rates through a char. The experiment might have indicated a lower transition temperature if the methane had had a longer time in which to react. The results do indicate that there is some relatively small temperature range over which the chemical state of the methane changes from frozen to equilibrium and it is reasonable to assume that the actual pyrolysis gases behave similarly. However, considerably more experimental work is required to obtain a complete understanding and description of what takes place within the char.

If the transition temperature is greater than about 1400° K, as indicated by the experiment, the pyrolysis gases can be assumed to be chemically frozen at all times for lifting-body reentry environments of the type considered herein. From figure 9, it can be seen that the mass of ablative material could be reduced considerably if the equilibrium reactions of the pyrolysis gases could be catalyzed so that the transition from the frozen state to equilibrium occurred at some lower temperature. Catalyzing these reactions must, of course, not adversely affect the other ablative characteristics of the material.

CONCLUDING REMARKS

Results are presented from an analytical investigation to determine the effect of the chemical state of the pyrolysis gases on the mass of ablative material required for various heating conditions. Calculations are made for a number of constant-heating-rate environments as well as for a lifting-body vehicle in a typical reentry trajectory. The results show that in all calculations considerably more material is required if the gases are chemically frozen at 500° K than if the gases are in equilibrium at all times. For the lifting-body vehicle, 28 percent more ablative material is required if the gases are chemically frozen than if the gases are in chemical equilibrium.

Calculations were made in which the gases went from the frozen state to equilibrium at various transition temperatures ranging from 800° K to 1700° K. For the constant-heating-rate trajectories, the amount of material required increases with increasing transition temperatures over a wide range of heating rates. At the lower heating rates, the results for the transition cases approach those for the frozen case. At the higher heating rates, the transition results approach the equilibrium results.

For the lifting-body vehicle, the results for transition temperatures above 1400° K are essentially the same as those for the frozen case. For transition temperatures below 1400° K, significant reductions in the mass of ablative material required are obtained with lower transition temperatures.

Limited experimental results indicate that transition from the frozen state to equilibrium may take place above 1400° K and over a temperature range of about 200° K. Therefore, for lifting-body reentry environments of the type considered in this investigation, it would be desirable to introduce some catalyst into the ablative material to cause the equilibrium reactions of the pyrolysis gases to occur at temperatures below 1400° K, if the addition of the catalyst does not adversely affect the other ablative characteristics of the material.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., September 23, 1968,
124-08-03-21-23.

APPENDIX

EXPERIMENTAL APPARATUS AND PROCEDURES

A schematic diagram of the basic experimental apparatus is shown in figure 11. The overall system has, in addition to the basic apparatus, a gas supply system which includes the necessary flowmeters for measuring the mass flow rate of the gases, an electrical system for heating the test material, and a gas sampling system. As shown in figure 11, the test material is held in place by electrical insulators bonded to it and to metal support plates. The insulators also serve to direct the gases through the material. The test apparatus has a window in each side to allow viewing of the test material during an experiment. The entire apparatus is placed in a bell jar to provide a controlled atmosphere. The temperature of the test material is determined with the use of optical pyrometers located on each side of the apparatus. The system is capable of operating at temperatures up to about 2700^o K.

In all tests, the gas used was methane and the test material was a porous carbon slab about 1 cm thick. Methane was used because it is one of the principal components of the gases produced by the decomposition of phenolic-nylon and its behavior is most likely to be representative of that of the pyrolysis gases. The mass flow rate of the methane was about 0.2 kg/m²-sec.

A typical experiment is conducted in the following manner. Helium is introduced into the system as the carbon slab is heated. The carbon is heated by passing an electric current through it. After the system has stabilized, the helium is replaced by the methane test gas which is forced into the apparatus and through the hot carbon slab. The power input to the carbon slab, the temperature of the slab as indicated by the optical pyrometers, and the gas flow rate are allowed to stabilize. These quantities, along with the pressure inside the test apparatus, are then recorded. The gas emerging from the carbon is sampled and the sample is analyzed with the use of a mass spectrometer to determine its composition.

In these tests, the test gas is assumed to be at the same temperature as the hot carbon slab through which it passes. By making a number of tests, each with a different carbon temperature, the composition of the gas mixture emerging from the carbon is determined as a function of temperature. The usual method of presenting the data from these tests is to plot the mole fraction of hydrogen in the gas sample as a function of temperature. From equilibrium theory, it is found that above 1000^o K, the mole fraction of hydrogen in a mixture of gas resulting from the decomposition of methane is an indication of the extent to which the mixture has approached the equilibrium state. If the mixture is almost all hydrogen, the equilibrium reactions are nearly complete and the mixture is essentially in chemical equilibrium. If there is no hydrogen present, no reactions have

APPENDIX

taken place and the gas is still in its initial state; that is, the gas is still all methane. Therefore, from a plot of mole fraction of hydrogen in the gas sample as a function of temperature, a transition temperature at which the methane goes to the equilibrium state is determined.

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TABLE I.- MATERIAL PROPERTY VALUES USED

Temperature, °K	Thermal conductivity, W/m-°K	Specific heat, kJ/kg-°K
Phenolic-nylon char: Density, 260 kg/m ³ ; emittance, 0.8		
300	---	1.6
810	---	1.6
835	0.15	--
1110	.49	--
1365	---	2.2
1390	1.19	--
1670	1.84	--
1920	---	2.2
1940	2.60	--
2220	3.67	--
2500	4.65	--
2780	6.12	--
3060	7.71	--
Phenolic-nylon: Density, 553 kg/m ³ ; heat of pyrolysis, 1.3 MJ/kg		
300	0.079	1.51
366	----	1.77
389	.083	---
422	----	2.02
477	----	2.27
500	.087	---
533	----	2.51
588	----	2.76
610	.090	---
644	----	2.97
671	----	3.10
710	.092	---

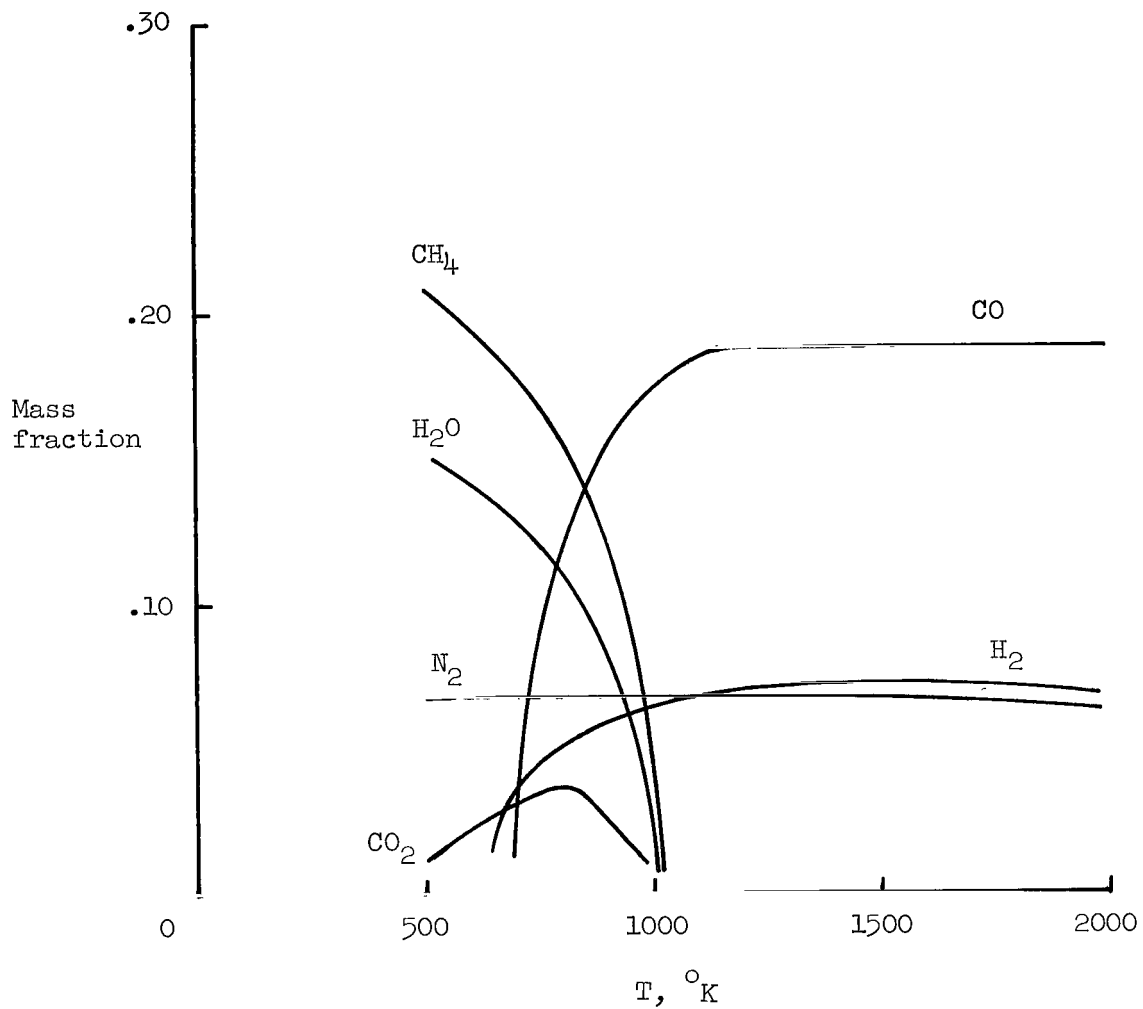


Figure 1.- Equilibrium composition of the gas-phase decomposition products of phenolic nylon at a pressure of 1 atmosphere (101 325 N/m²) calculated from equilibrium program.

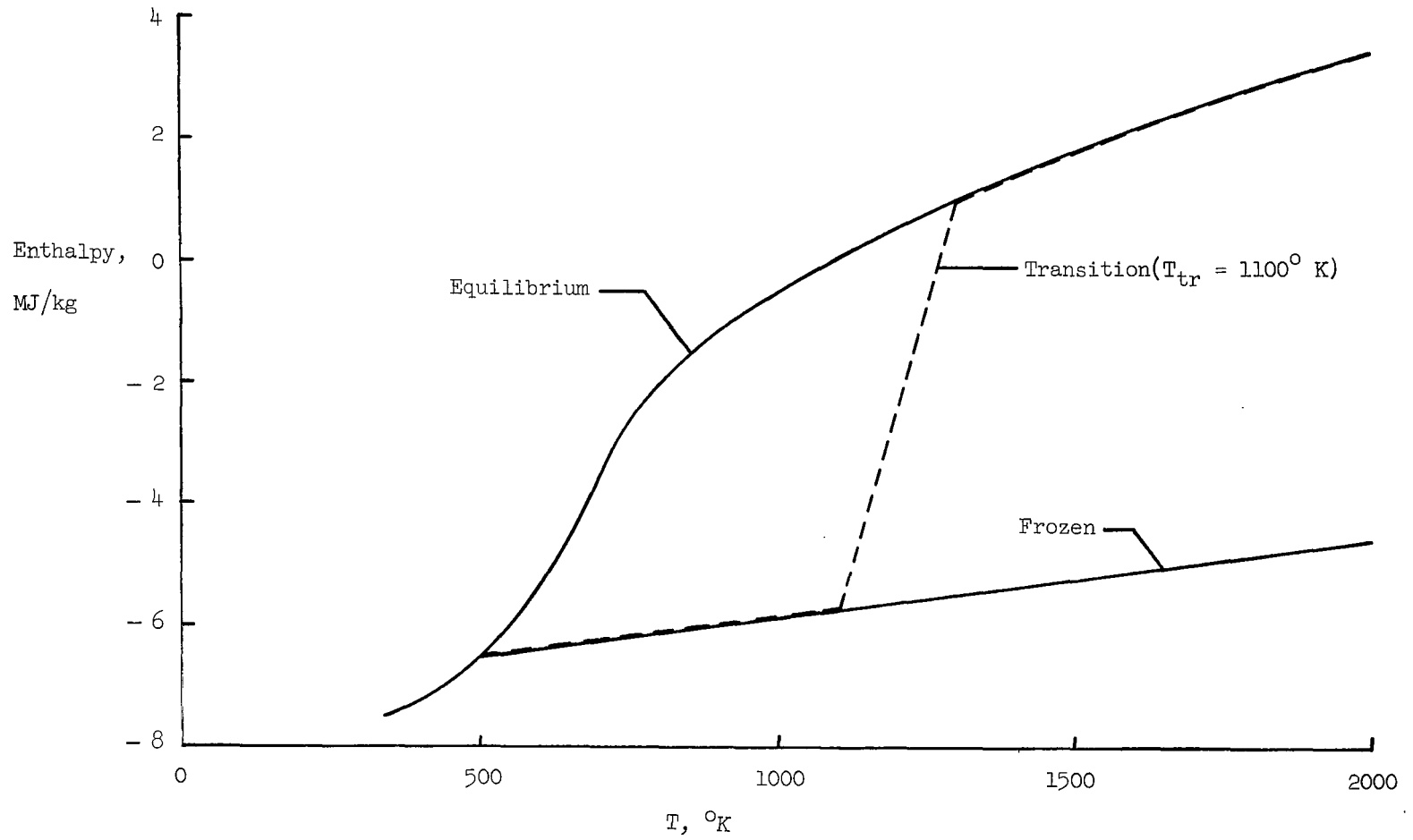


Figure 2.- Enthalpy of pyrolysis gases at a pressure of 1 atmosphere.

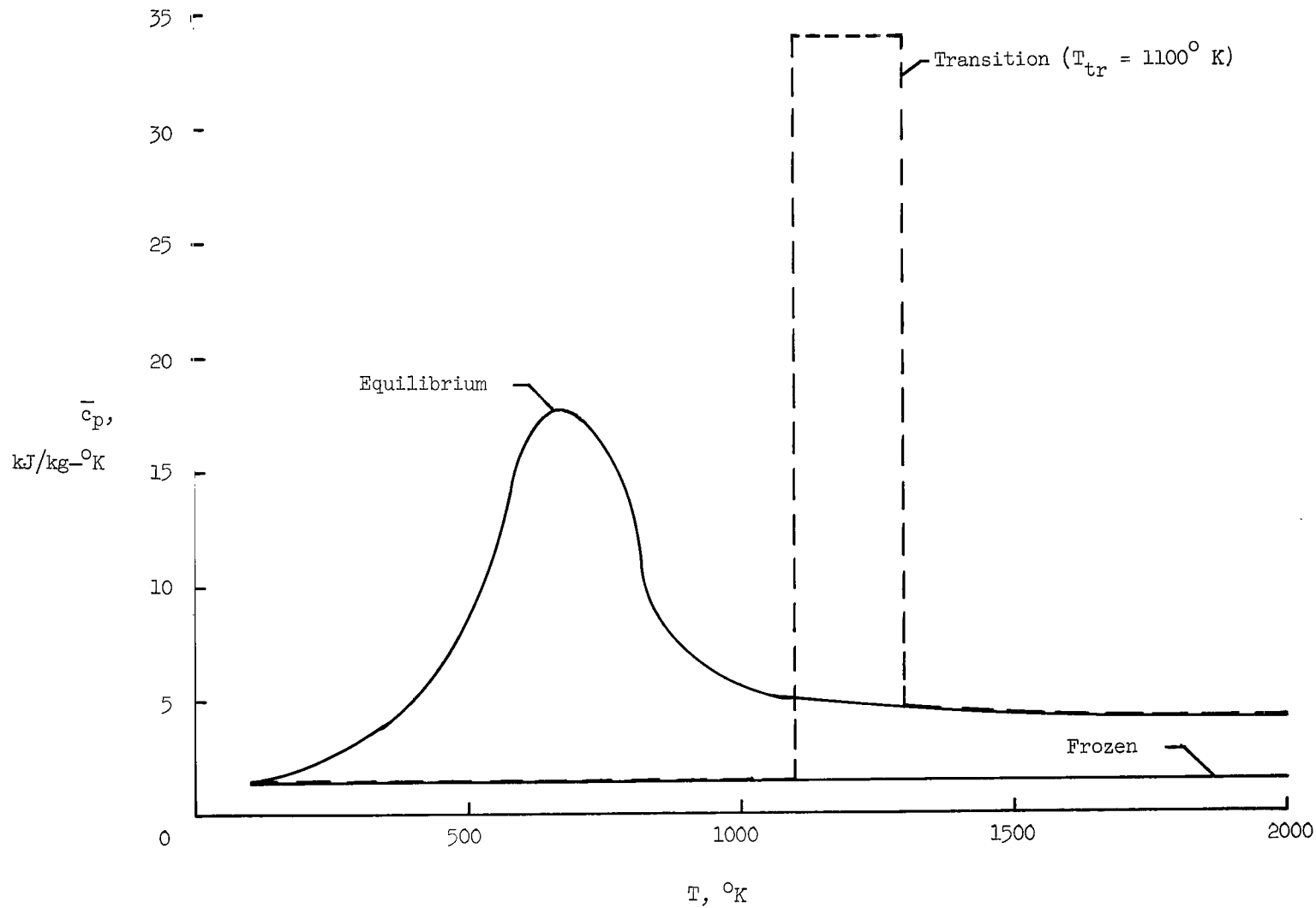


Figure 3.- Specific heat of pyrolysis gases.

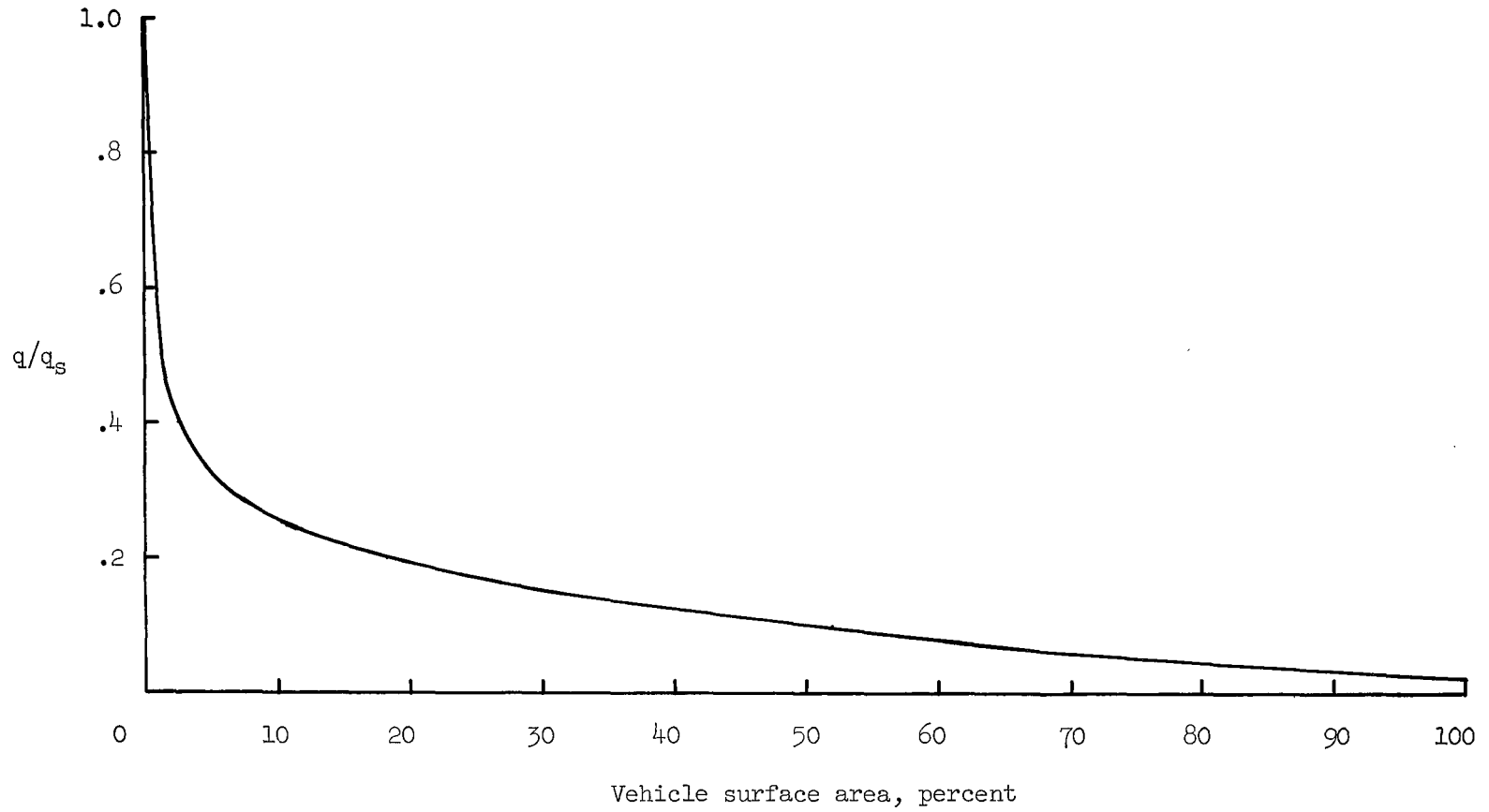


Figure 4.- Heating-rate distribution over surface of lifting-body reentry vehicle.

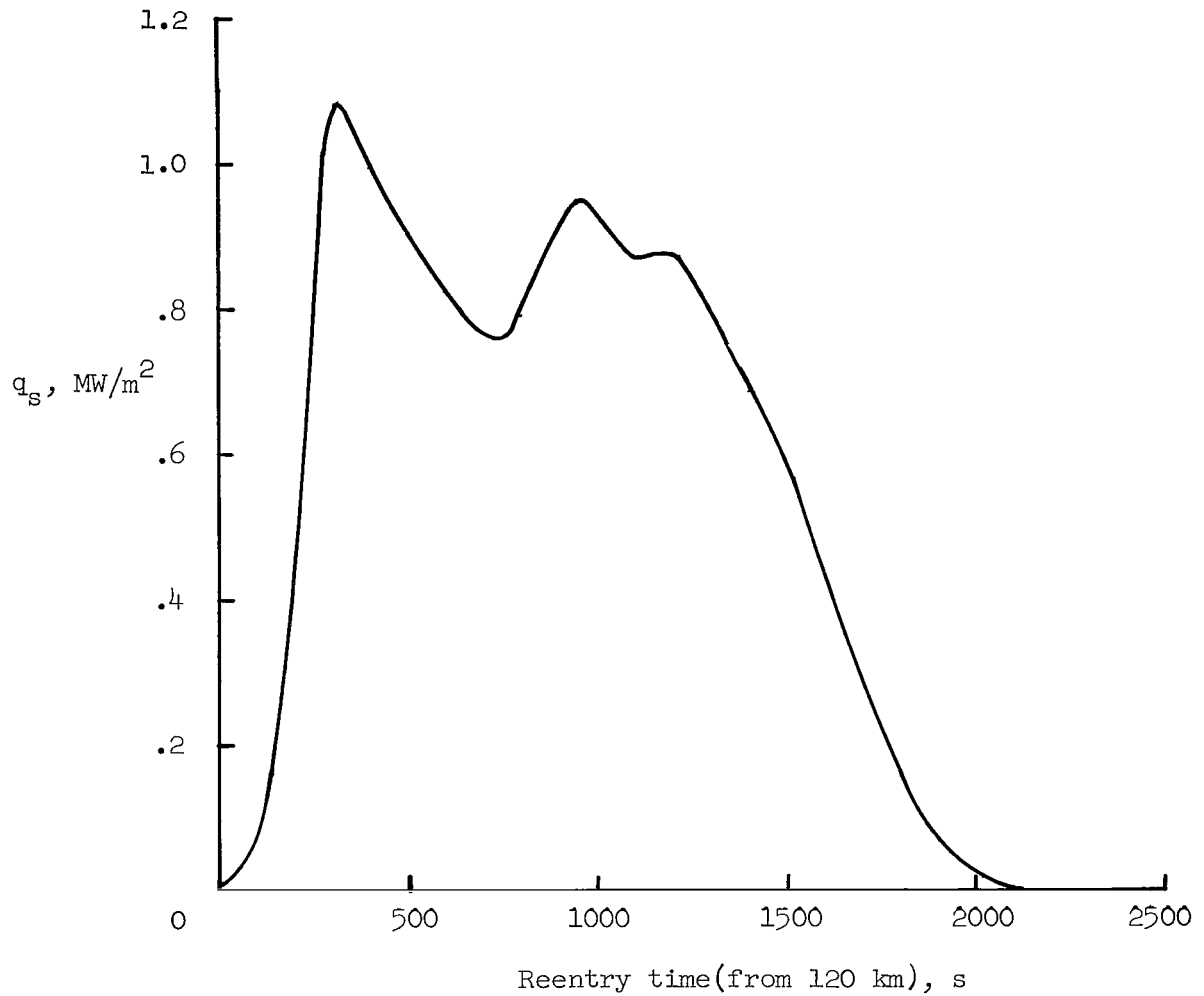


Figure 5.- Typical stagnation-point heating history for lifting-body reentry vehicle.

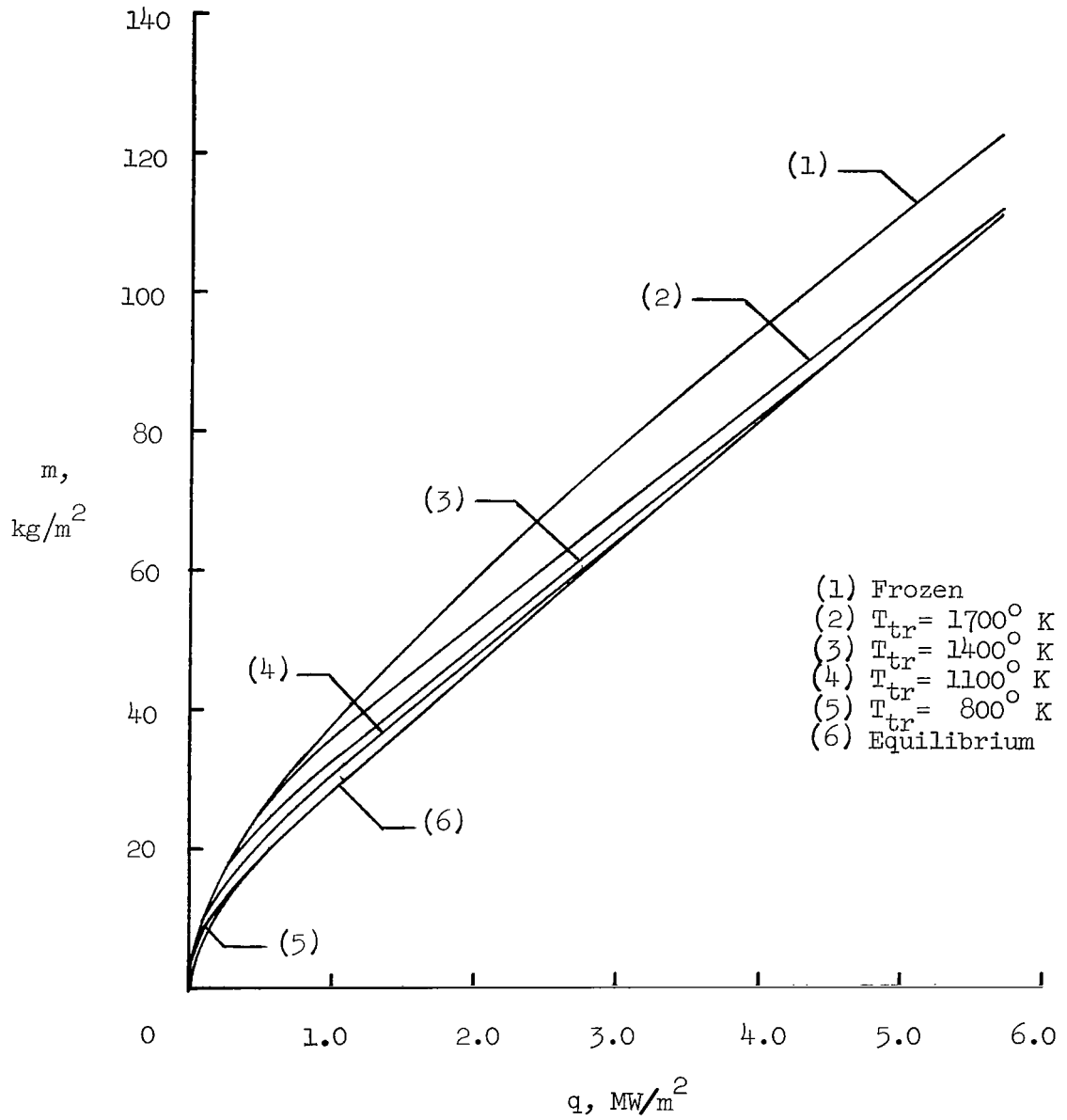


Figure 6.- Effect of the chemical state of the pyrolysis gases on unit mass of ablation material required to limit back-surface temperature to 530° K for the constant-heating-rate environments.

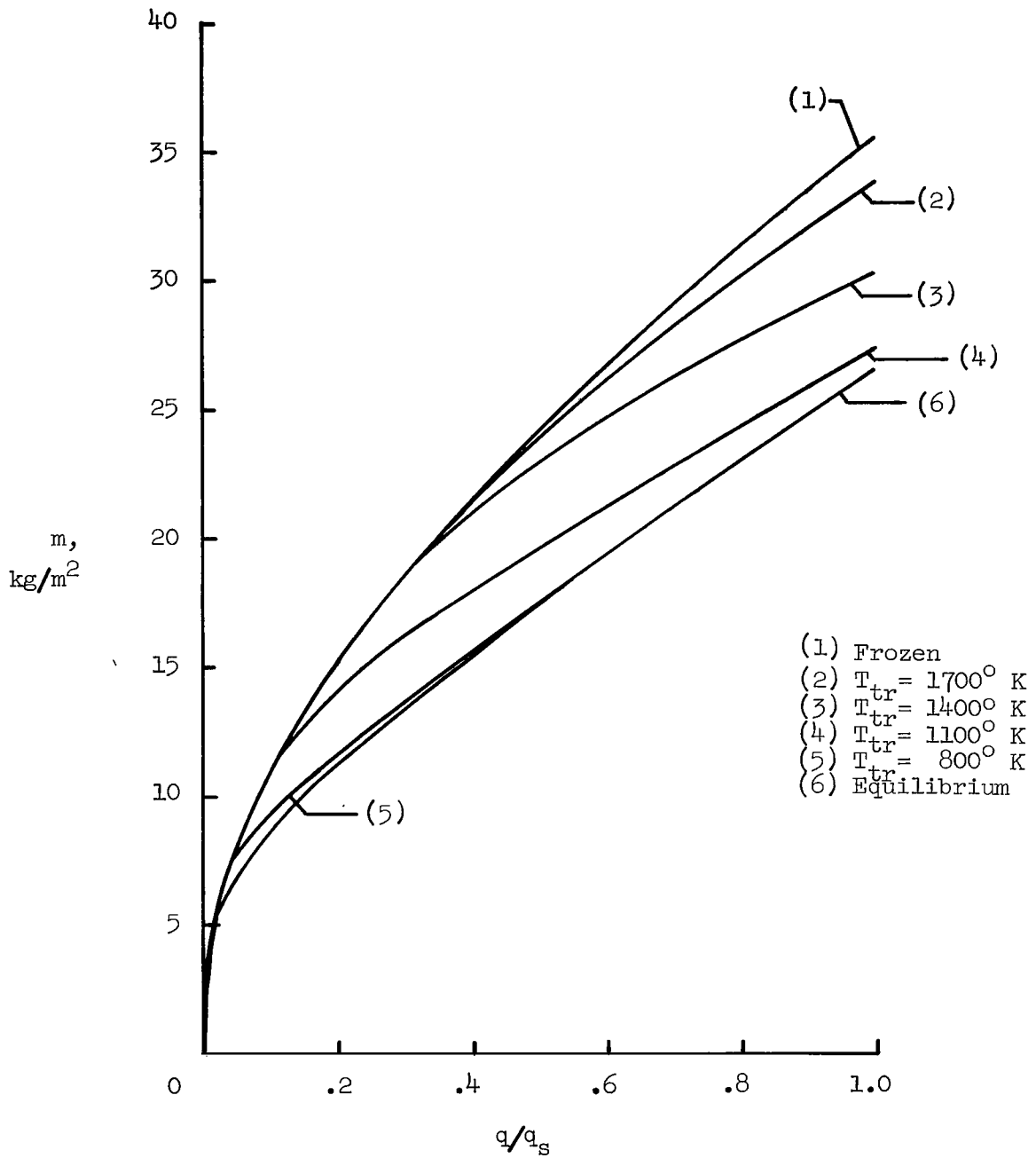


Figure 7.- Effect of the chemical state of the pyrolysis gases on unit mass required to limit back-surface temperature to 530° K for the lifting-body vehicle.

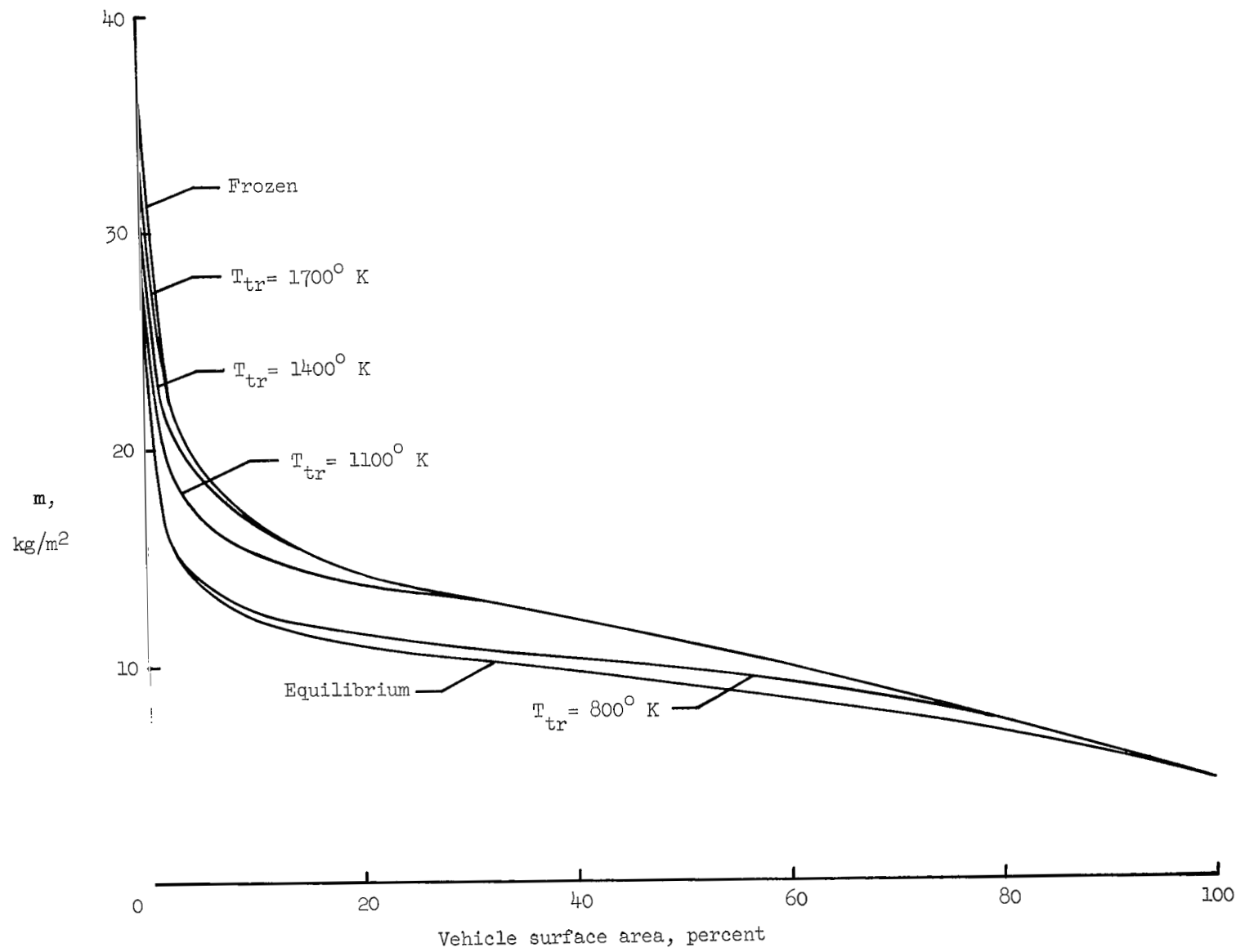


Figure 8.- Unit-mass distribution over reentry vehicle.

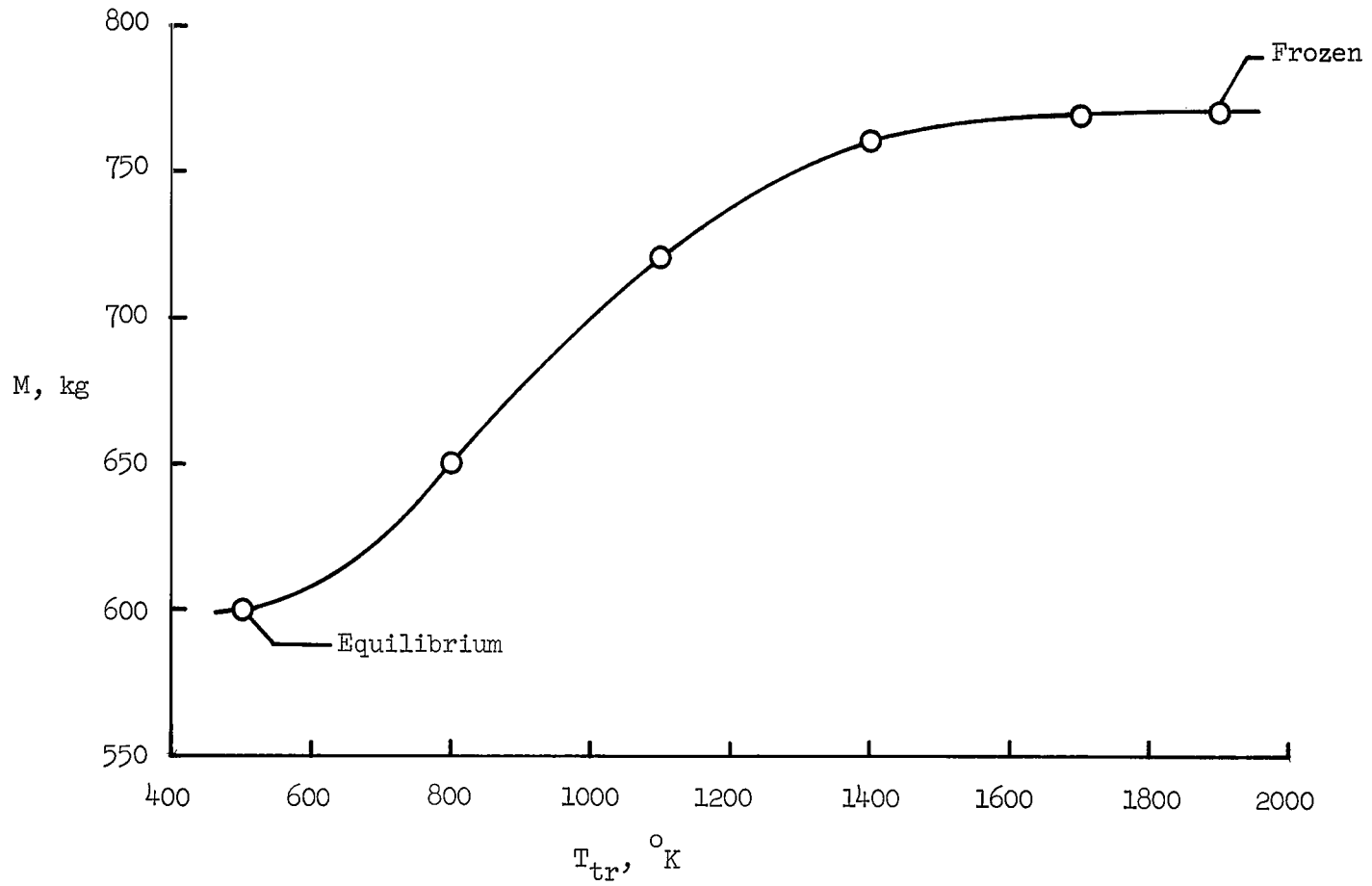


Figure 9.- Effect of transition temperature on total mass of ablation material required for the lifting-body vehicle.

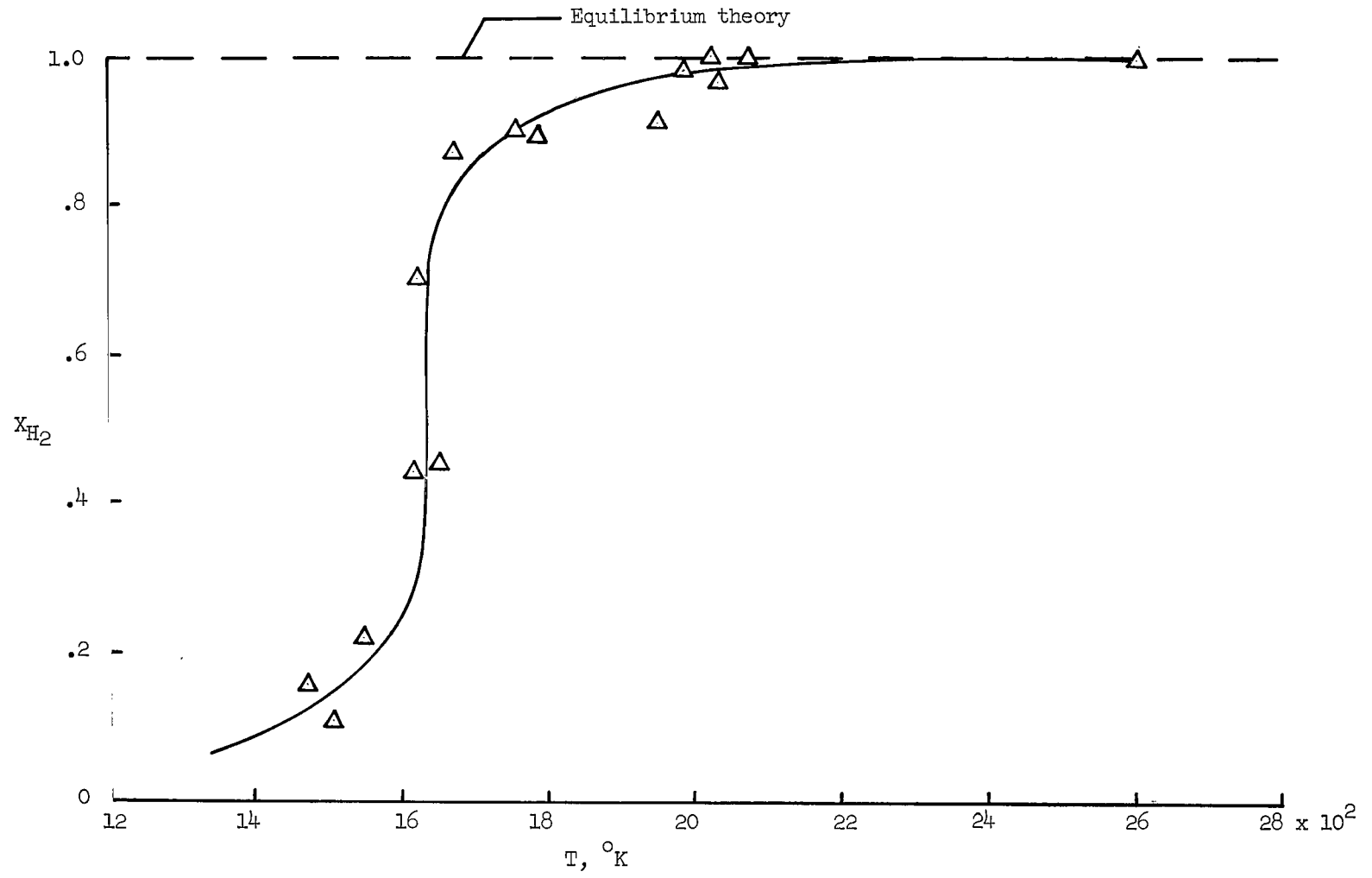


Figure 10.- Mole fraction of hydrogen in gas mixture resulting from decomposition of methane (experimental).

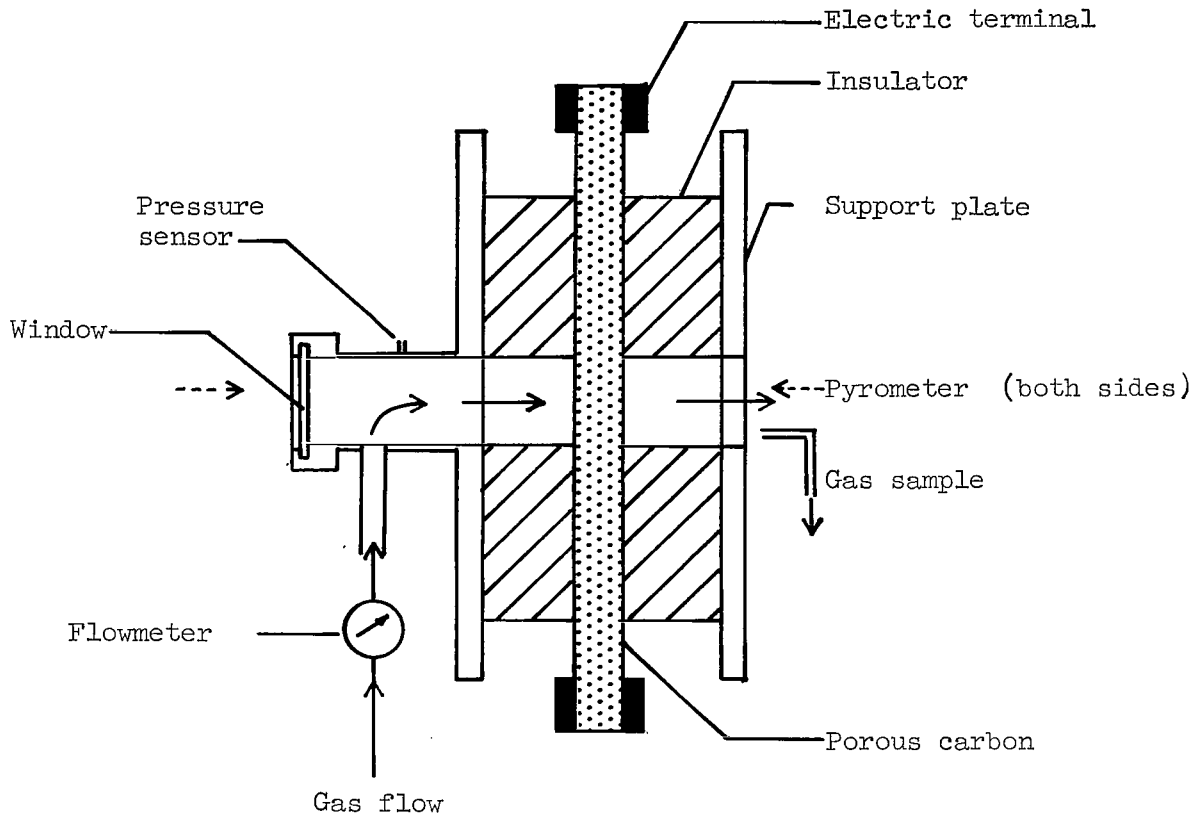


Figure 11.- Schematic diagram of experimental apparatus.

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— NATIONAL AERONAUTICS AND SPACE ACT OF 1958

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