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PRELIMINARY INVESTIGATION OF ABLATIVE MATERIAL RESPONSE TO HIGH-INTENSITY CO_2 LASER RADIATION

by William D. Brewer

Langley Research Center
Langley Station, Hampton, Va.

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SUMMARY

Results are presented from an experimental study to obtain preliminary information about the performance of ablative materials subjected to very high radiative-heat fluxes. The radiative environment was produced by the 2-kW CO_2 continuous-wave laser at Redstone Arsenal. Various ablative materials were tested in a controlled atmosphere at average radiative fluxes in the range from 30 to 45 MW/m^2. Graphite models were tested in air at pressures of 0.1, 0.3, and 1.0 atm and in nitrogen and helium at 0.1 and 0.3 atm. Carbon-phenolic, high- and low-density phenolic-nylon, a silicone elastomer, and a polybenzimidazole were tested in air at 0.1 and 0.3 atm and in nitrogen at 0.1 atm. Because the laser beam did not heat the surface of the models uniformly, most of the results are qualitative.

INTRODUCTION

Entry into various planetary atmospheres, as well as hypervelocity entry into the earth's atmosphere, is characterized by very large radiative heat inputs into an entry vehicle. Recently, emphasis has been placed on the study of such entry environments. Analytical routines have been developed to determine convective- and radiative-heat fluxes to a vehicle and the flow-field characteristics about the vehicle (refs. 1 and 2). However, little has been done toward obtaining a better understanding of the interaction of the radiative environment with ablative materials in the thermal-protection systems.

A test program was undertaken to obtain preliminary information about the performance of ablative materials subjected to severe radiative environments. At present, gas lasers appear to be the only facilities capable of producing radiative-heat fluxes comparable to those encountered in hypervelocity entry. Therefore, for this investigation, the 2-kW CO_2 continuous-wave laser at Redstone Arsenal was used to produce the radiative environment. Various ablative materials were placed in a controlled atmosphere and subjected to average radiative flux densities in the range from 30 to 45 MW/m^2. Flat-faced cylindrical models 6.4 mm in diameter were tested in air, nitrogen, and helium atmospheres.

The units used for physical quantities are given in the International System of Units (SI). Reference 3 gives factors for converting SI units to U.S. Customary Units.

TEST APPARATUS

All models were tested in the radiative-heating environment produced by the CO_2 laser. The laser produces continuous radiation at 10.6-μm wavelength and has a maximum power output of about 2 kW. The unfocused beam diameter at the laser-tube exit is about 5 cm. The design and operating characteristics of the laser are described in detail in reference 4. As shown in figure 1, the laser beam emerges from the tube and is directed onto the model surface by a gold-plated flat mirror and a gold-plated focusing mirror. The model is positioned in the test chamber so that the beam is incident normal to the model surface. The distance between the focusing mirror and the model insures that the beam diameter at the model position is equal to the model diameter. The test-gas supply and the vacuum pump are used to control the atmosphere and pressure in the test chamber as well as to remove the gases produced by ablation. The radiometer is used to monitor the beam energy during the tests.

Details of the test chamber are given in figure 2. The chamber is about 30 cm long and 15 cm in diameter and has glass viewing windows located on each side. The laser beam enters through a 5-cm-diameter opening over which an NaCl window is positioned. The front surface of the window is about 10° from the normal to the direction of the laser beam and reflects about 5 percent of the total beam energy into the radiometer used to determine the energy reaching the model surface.

A plenum chamber into which the test gas is injected is located inside the test chamber. The test gas is injected into the chamber through a 3.2-mm-diameter sonic orifice. In this arrangement, the mass-flow rate of the gas through the orifice is independent of the pressure downstream of the orifice and can be calculated if the temperature and pressure of the gas upstream of the orifice are known. A 13-mm-diameter orifice in the end of the plenum chamber allows the test gas to flow past the test model. The radiation from the laser also passes through this orifice onto the model surface.

INSTRUMENTATION AND CALIBRATION

A pressure transducer and a thermocouple were placed upstream of the sonic orifice (see fig. 2) to measure the pressure and temperature of the test gas. A second pressure transducer was used to measure the pressure inside the test chamber. During each test, outputs from the transducers, the thermocouple, and the radiometer were recorded continuously by a four-channel strip chart recorder.

The radiometer used to monitor the portion of the beam reflected from the NaCl window was calibrated with a water-cooled calorimeter in which a nickel cone was used to collect the main laser beam. This calorimeter is described in detail in reference 5. The beam power was calculated from the flow rate and the temperature of the input and output water in the calorimeter. The power calibration setup is shown schematically in figure 3. The small radiometer was positioned to accept the reflected energy, which was about 5 percent of the total beam energy. The calorimeter was located at the rear of the test chamber to accept the transmitted radiation. A graphite stop with a 6.4-mm-diameter hole through the center was placed at the model test position. Since all models were 6.4-mm-diameter flat-faced models, the total energy reaching the calorimeter through the carbon stop was the same as that impinging on the surface of a model during a test.

In calibrating the radiometer, the laser was turned on; and by using the setup described, a relationship was obtained between the energy reaching the surface of the model and that reflected to the radiometer. Therefore, only the energy reflected to the radiometer needed to be monitored during a test to determine the energy reaching the model. After calibration, the graphite stop and the calorimeter were removed, and the vacuum system was attached to the test chamber.

TEST MODELS

To obtain the required heat flux, the diameter of the laser beam was reduced to about 6.4 mm. Therefore, the test models were made with a 6.4-mm diameter so that the entire front surface could be exposed to the beam. The models were flat-faced cylinders 7.6 cm in length which were positioned in a carbon model holder (fig. 2) so that only 4 cm of the cylinder extended from the model holder.

The six test materials were commercial grade ATJ graphite (density = 1800 kg/m^3), a carbon-phenolic composite, high- and low-density phenolic-nylon, a silicone elastomer, and a polybenzimidazole (PBI). The carbon-phenolic material is approximately 50 percent phenolic resin and 50 percent carbon fibers and has a density of about 1390 kg/m^3. The high-density phenolic-nylon, with a density of 1200 kg/m^3, is 50 percent phenolic resin and 50 percent nylon powder. The low-density phenolic-nylon consists of 25 percent phenolic resin, 25 percent phenolic Microballoons, and 50 percent nylon powder and has a density of 550 kg/m^3. The silicone elastomer is approximately 75 percent silicone resin, 15 percent silicone dioxide in the form of tiny hollow spheres (11 percent) and fibers (4 percent), and 10 percent phenolic Microballoons. The density of the elastomer is about 640 kg/m^3. These materials, except graphite and PBI, are discussed in more detail in reference 6. The PBI (material 5, ref. 7) has a density of 500 kg/m^3 and

consists of 69 percent PBI prepolymer, 13 percent carbon fibers and 18 percent phenolic Microballoons. All percentages are based on mass.

TEST CONDITIONS AND PROCEDURES

Graphite models were tested in air at pressures of 0.1, 0.3, and 1.0 atm and in nitrogen and helium at 0.1 and 0.3 atm. The other materials were tested in air at 0.1 and 0.3 atm and in nitrogen at 0.1 atm. Average heat fluxes varied from 30 to 45 MW/m^2. The gas mass-flow rates were very small ($\approx$1.0 g/s), but observations during the tests indicated that the rates were sufficient to remove the gases produced by ablation.

The test models were placed in the test chamber, the appropriate test gas was injected into the chamber, and the pressure was regulated by use of the vacuum system. After the desired conditions were established, the laser was turned on. While the models were subjected to the high-intensity radiation for the specified length of time, the chamber pressure, the test-gas flow rate, and the average heat flux were held constant. After the test durations indicated in table I, the laser was turned off. Each model was weighed before and after testing to determine the mass lost.

RESULTS AND DISCUSSION

Nonuniform heating of the model surface proved to be an insurmountable problem in the present experiment. Consequently, most of the test results are qualitative. Because the Redstone Arsenal laser produces a beam which is more intense near the center than at the edge, the heating rate at the center of the model surface was greater. As a result, deep holes were burned in the center of the models while little or no recession occurred at the edges.

The severity of the problem of nonuniform heating is apparent in figure 4(a). This figure shows three graphite models which were tested for 30 seconds in air, nitrogen, and helium at a pressure of 0.1 atm. The laser beam was incident from the top. The test models have been sectioned to show the variation of heating over the surface. The dark, cone-shaped areas at the top of the model indicate the burned-out portions. The lighter areas are a result of the sectioning process. The average flux density over the surface of each model was about 42 MW/m^2; however, the flux density was obviously much greater near the center of the model surfaces.

Although evaluation of material performance was complicated by the nonuniform energy distribution, some significant observations can be made concerning the behavior of the graphite models in the different environments. Since no material spallation was observed, the mass loss in the helium test was due to sublimation only. The model tested

in nitrogen lost about twice as much mass as the model tested in helium. This behavior indicates that, in addition to mass loss by sublimation, significant mass loss was caused by carbon-nitrogen reactions. The additional effects of oxidation caused the model tested in air to lose about twice as much mass as the one tested in nitrogen.

Figure 4(b) shows three graphite models which were tested for 30 seconds in air, nitrogen, and helium at a pressure of 0.3 atm. The same general type of behavior is seen here as in the tests at 0.1 atm. However, for the air tests, about 50 percent more material was lost at 0.3 atm than at 0.1 atm. For the nitrogen and helium tests, the mass lost was independent of pressure. The loss of material along the side of the model tested in air is apparent. Although the side of the model was not heated directly by the laser beam or by the test gas, a considerable amount of heat was conducted backward through the model from the heated surface as well as radially outward from the hole in the center. Therefore, the temperature of the sides of the models was high enough for appreciable oxidation to occur but was most likely below the sublimation temperature.

Graphite models which were tested for 30 seconds in air at pressures of 1.0, 0.3, and 0.1 atm are shown in figure 5. As indicated in table I, twice as much mass was lost in the 1.0-atm test as in the 0.1-atm test. The increase in mass loss with increasing pressure is to be expected. However, in the present tests, material was probably removed by reaction-rate-controlled oxidation on the cooler parts of the sides of the models, by diffusion-controlled oxidation on the hotter parts of the sides and some parts of the front surface, as well as by sublimation at the center of the front surface and by carbon-nitrogen reactions. Each of these processes is affected to some extent by the pressure; therefore, it is difficult to attach any significance to the manner in which the mass loss varied with pressure. Again, the loss of material along the sides of the models is apparent, especially for the 1.0 atm test.

Figure 6 shows photographs of the other materials which were tested at a pressure of 0.1 atm in air (fig. 6(a)) and in nitrogen (fig. 6(b)). The carbon-phenolic models were tested for 10 seconds. All other models were tested for 5 seconds. The results of the rather violent reactions of the materials to the two environments are apparent. The laser beam burned completely through about 3 cm of the PBI material in about 2 seconds and emerged from the hole in the side of the model. (See fig. 6(a).) Likewise, in about 2 seconds, the beam burned through the entire exposed length of both elastomer models. (See figs. 6(a) and 6(b).) These models were burned somewhat irregularly along the sides rather than through the center because the laser beam struck the front surfaces slightly off center; thus, the models did not expand uniformly on all sides and tended to curl away from the beam. The other materials were severely burned, but none of them was burned completely through. The physical appearance of the materials after testing in air was essentially the same as the appearance after testing in nitrogen; however, the

mass loss was different in the two environments. (See table I.) The behavior of the materials was not significantly affected by chamber pressure.

Although the results of these tests are not definitive, they are qualitatively of some significance. It is apparent, however, that much more investigation is required to evaluate thermal-protection materials for use in severe radiative environments as well as to evaluate the use of laser facilities for materials testing.

CONCLUDING REMARKS

A test program was undertaken to obtain preliminary information about the performance of various ablative materials subjected to very high radiative-heat fluxes. A commercial grade ATJ graphite, a carbon-phenolic composite, high- and low-density phenolic-nylon, a silicone elastomer, and a polybenzimidazole were tested in controlled atmospheres at average heat fluxes in the range from 30 to 45 MW/m^2. The radiative heating was produced by the 2-kW CO_2 continuous-wave laser at Redstone Arsenal. Because the laser beam did not produce uniform heating over the surface of the models, deep holes were burned into the models near the center of the heated surface. Consequently, the test results are primarily qualitative. The results of the graphite tests indicate that considerable mass is lost due to carbon-nitrogen reactions. As expected, models tested in air showed greater mass loss than those tested in nitrogen or helium due to oxidation effects, in addition to sublimation and carbon-nitrogen reactions. The graphite models tested in air also showed a sizable increase in mass loss when the chamber pressure was increased from 0.1 atm to 1.0 atm. The materials other than graphite were severely damaged and were somewhat distorted by the test environments. Little or no effect of chamber pressure on the behavior of these materials was observed. However, because of the undesirable characteristics of the laser beam, the results are not definitive. Much more investigation is required to evaluate thermal-protection materials for use in severe radiative environments.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., November 5, 1969.

REFERENCES

1. Chin, Jin H.: Radiation Transport for Stagnation Flows Including the Effect of Lines and Ablation Layer. AIAA Paper No. 68-664, June 1968.

2. Hoshizaki, H.; and Wilson, K. H.: Convective and Radiative Heat Transfer During Superorbital Entry. NASA CR-584, 1966.

3. Comm. on Metric Pract.: ASTM Metric Practice Guide. NBS Handbook 102, U.S. Dep. Com., Mar. 10, 1967.

4. Roberts, T. G.; Hutcheson, G. J.; Ehrlich, J. J.; Hales, W. L.; and Barr, T. A., Jr.: High-Power N_2-CO_2-He Laser Development. IEEE J. Quantum Electron., vol. QE-3, no. 11, Nov. 1967, pp. 605-609.

5. Roberts, T. G.; Hutcheson, G. J.; Ehrlich, J. J.; and Hales, W. L.: Experimental Development of N_2-CO_2 Lasers. Rep. No. RR-TR-67-11, U.S. Army Missile Command, July 1967.

6. Wilson, R. Gale, compiler: Thermophysical Properties of Six Charring Ablators From 140° to 700° K and Two Chars From 800° to 3000° K. NASA TN D-2991, 1965.

7. Dickey, Robert R.; Lundell, John H.; and Parker, John A.: The Development of polybenzimidazole Composites as Ablative Heat Shields. J. Macromol. Sci.-Chem., vol. A3, no. 4, July 1969, pp. 573-584.

TABLE I.- TEST CONDITIONS AND RESULTS

Material	Test gas	Chamber pressure, atm	Power input to specimen, kW	Test time, s	Mass loss, g
Graphite	Air	0.1	1.33	30	0.20
		.3	1.25		.30
		.3	1.25		.30
		1.0	1.33		.40
Graphite	Nitrogen	0.1	1.28	30	0.10
		.1	1.33		.10
		.3	1.25		.10
		.3	1.25		.10
Graphite	Helium	0.1	1.33	30	0.05
		.3	1.33		.05
Carbon-phenolic	Air	0.1	1.38	10	0.50
		.3	1.34		.45
		.3	1.36		.55
Carbon-phenolic	Nitrogen	0.1	1.38	10	0.40
High-density phenolic-nylon	Air	0.1	1.40	5	0.50
		.3	1.30		.55
		.3	1.00		.30
High-density phenolic-nylon	Nitrogen	0.1	1.40	5	0.40
Low-density phenolic-nylon	Air	0.1	1.40	5	0.45
		.3	1.25		.40
		.3	1.00		.30
Low-density phenolic-nylon	Nitrogen	0.1	1.40	5	0.30
PBI	Air	0.1	1.40	5	0.25
		.3	1.25		.20
PBI	Nitrogen	0.1	1.40	5	0.20
Elastomer	Air	0.1	1.40	5	0.25
		.3	.60		.20
Elastomer	Nitrogen	0.1	1.40	5	0.25

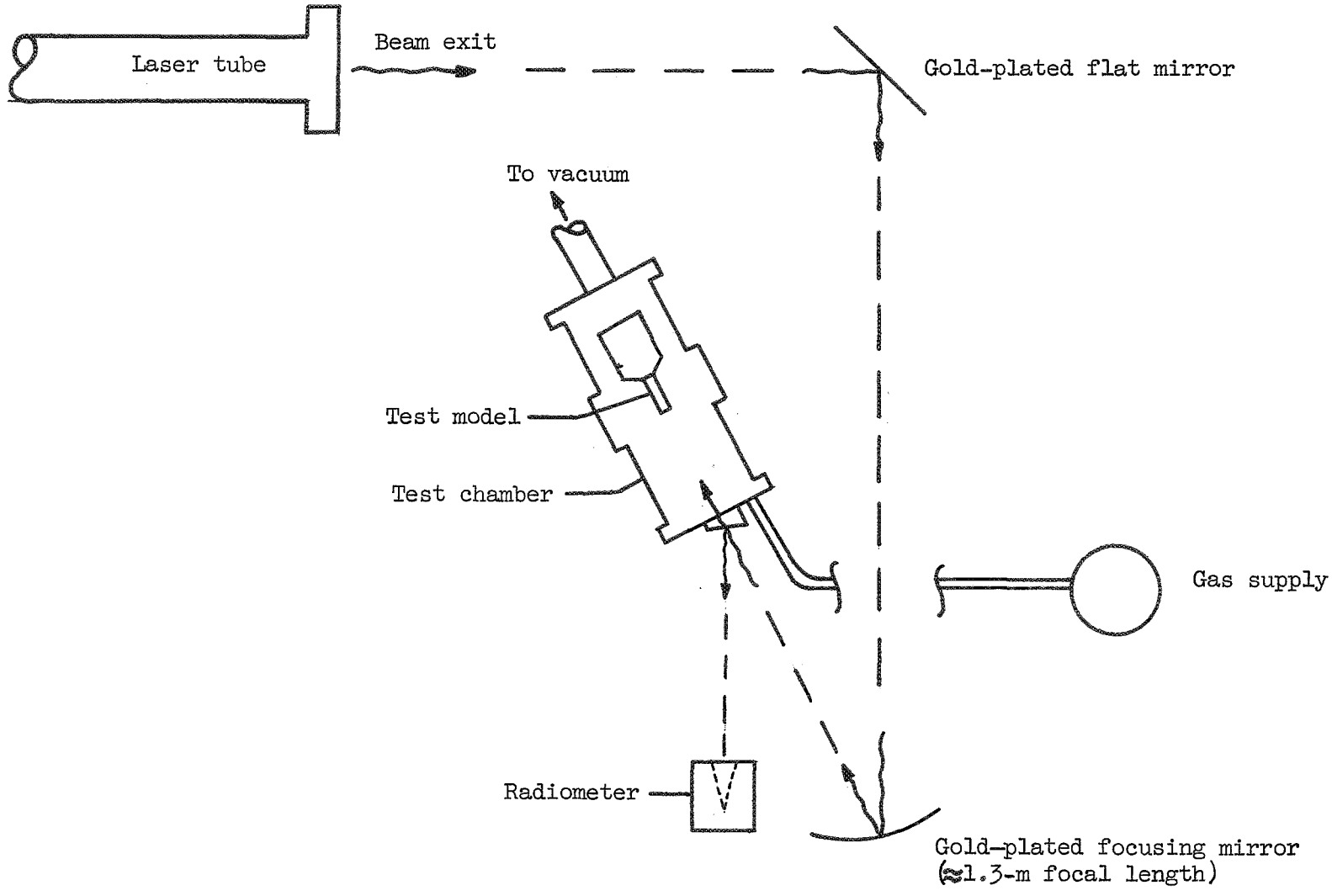


Figure 1.- Diagram of test setup.

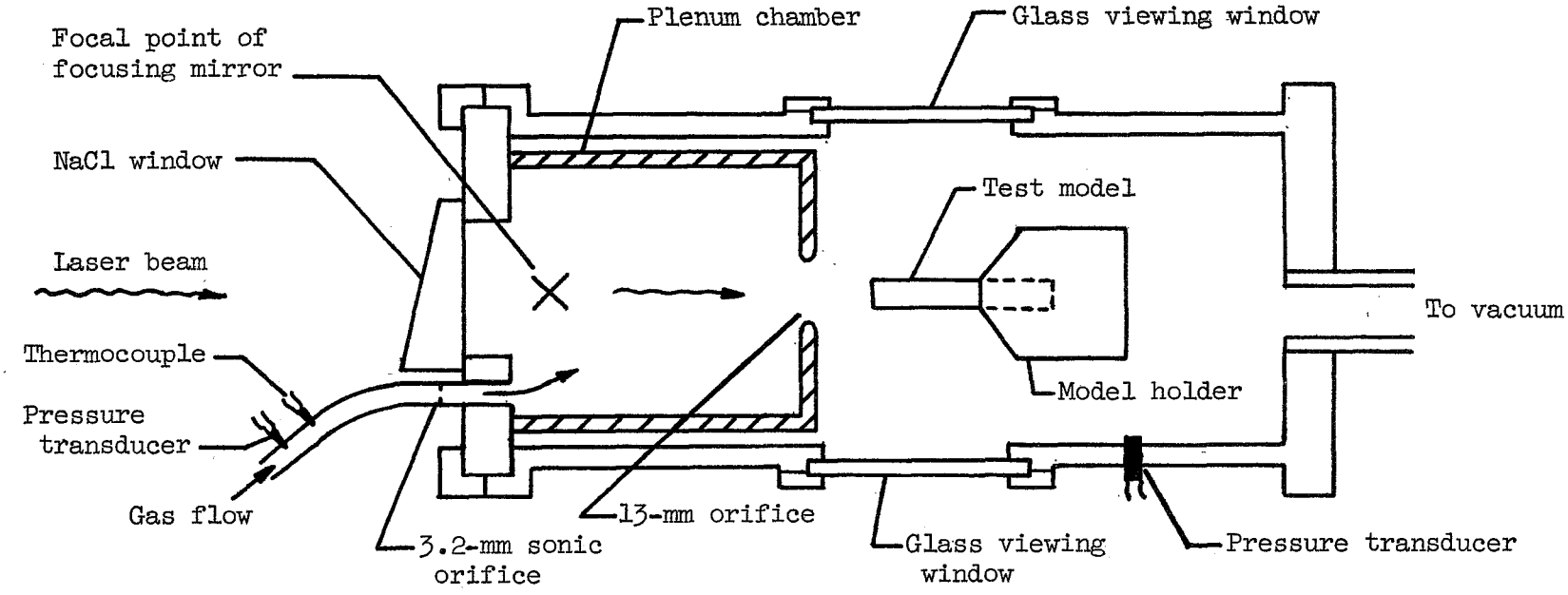


Figure 2.- Diagram of test chamber. Diameter, 15 cm; length, 30 cm.

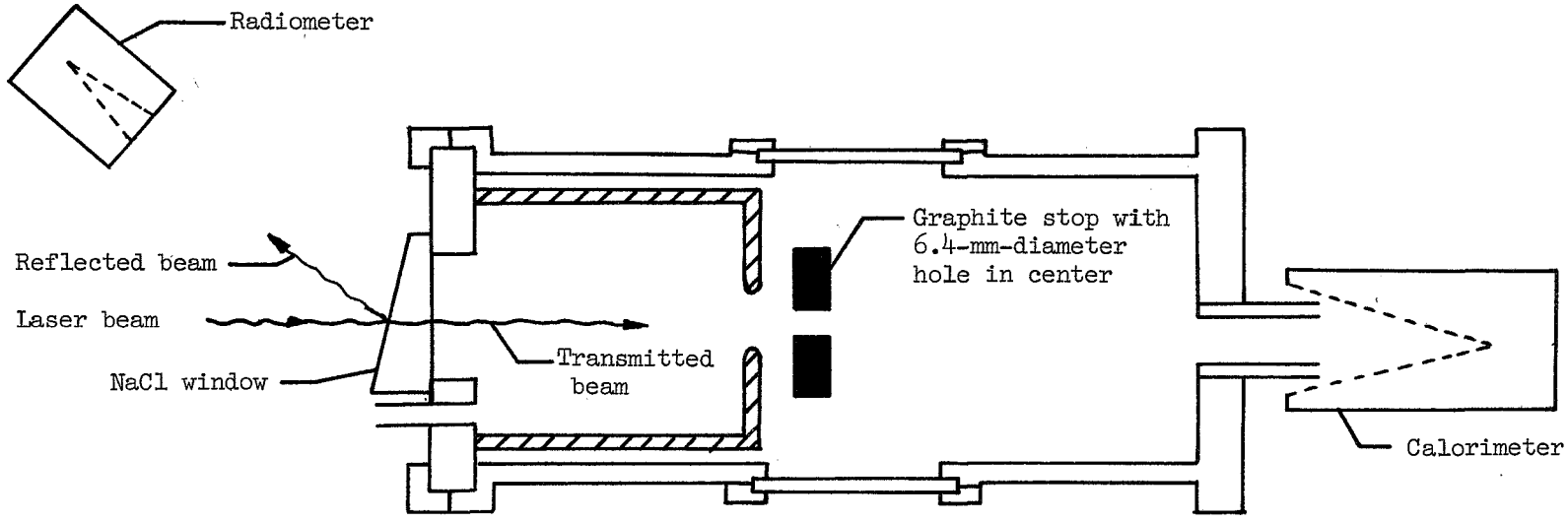
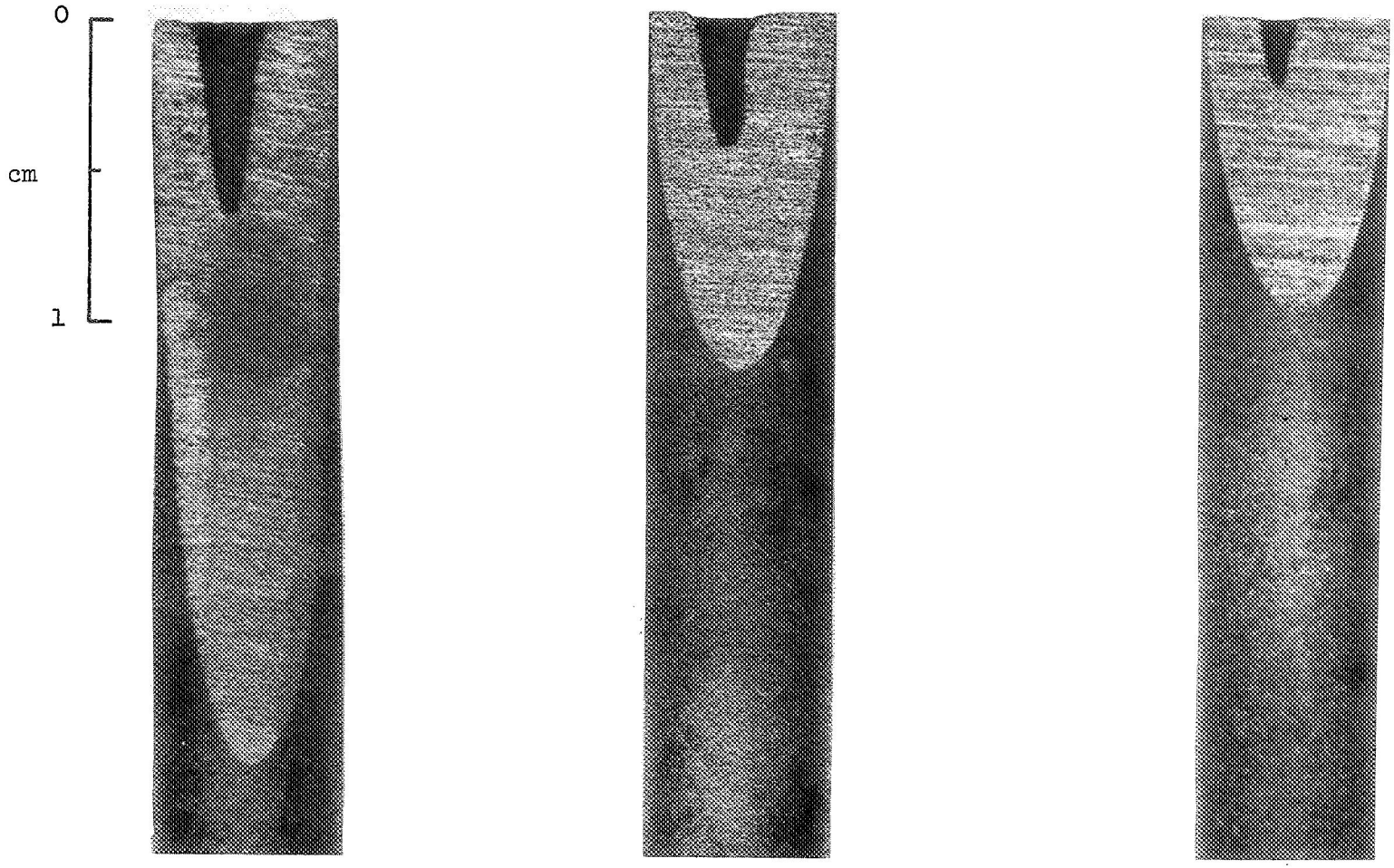


Figure 3.- Diagram of power calibration setup.



(a) Pressure = 0.1 atm.

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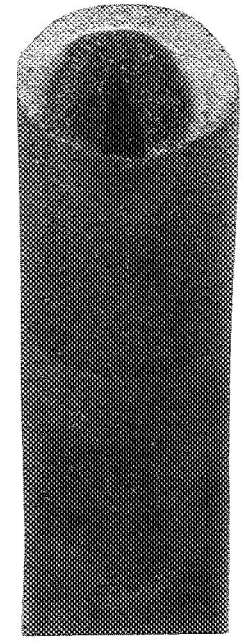
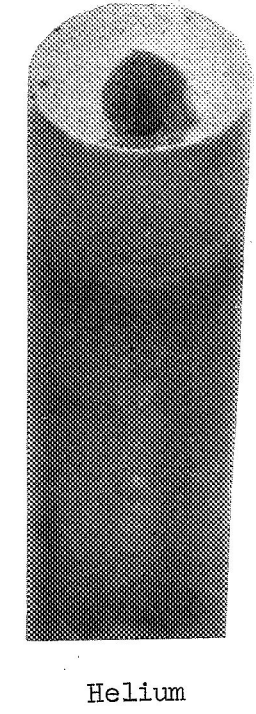
Figure 4.- Graphite models tested for 30 seconds in air, nitrogen, and helium.





(b) Pressure = 0.3 atm.

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Figure 4.- Concluded.

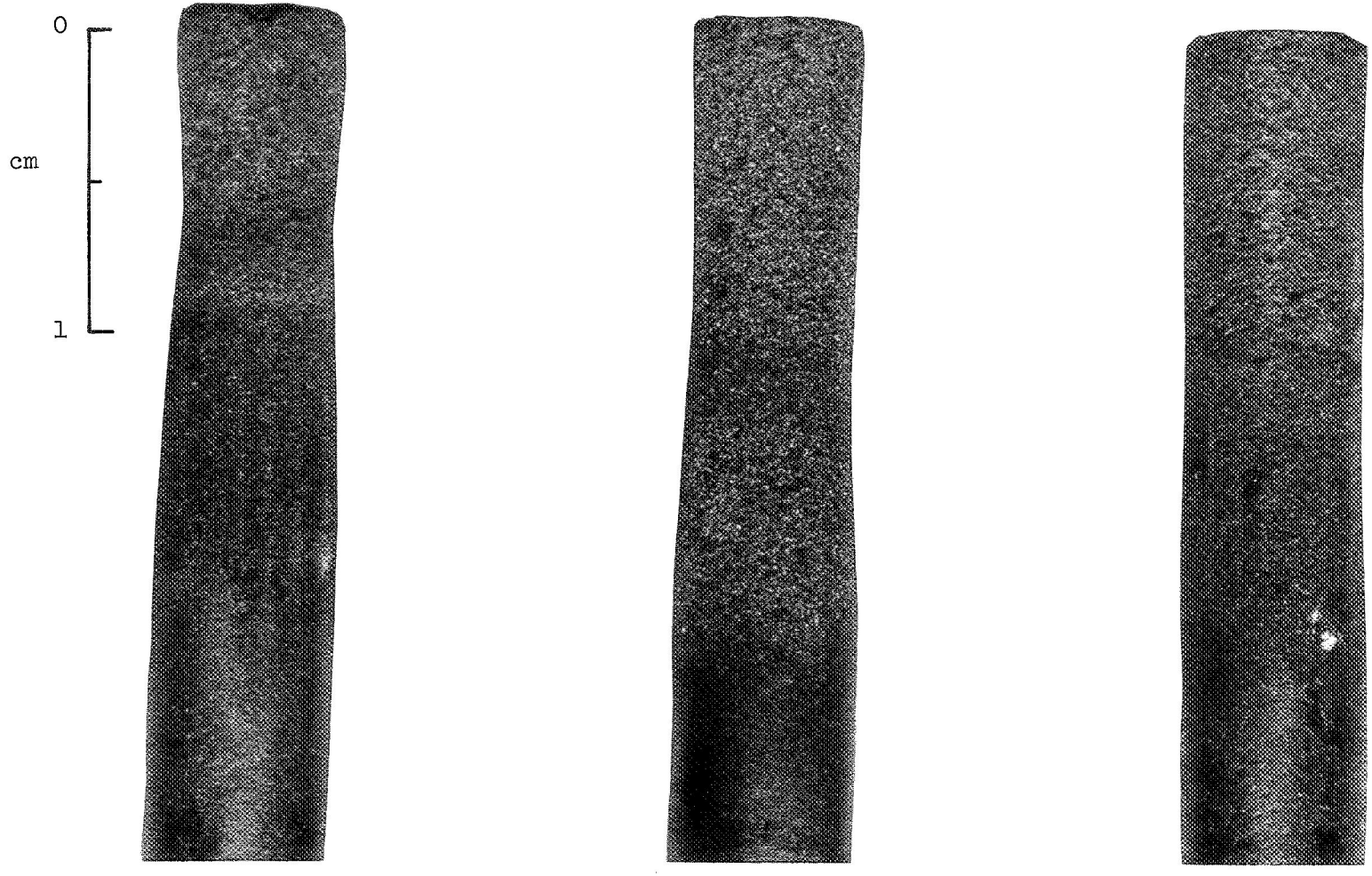
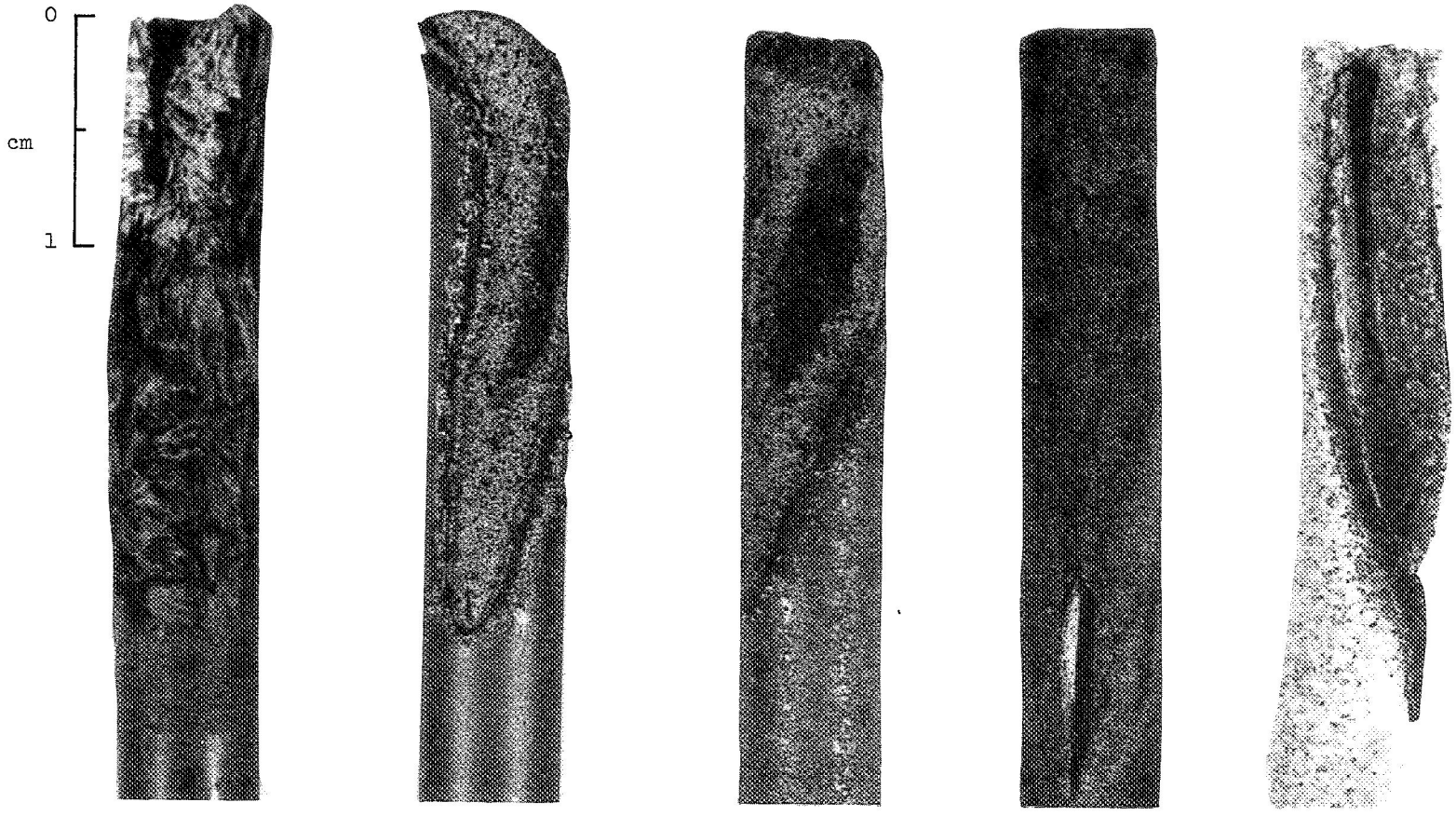


Figure 5.- Graphite models tested for 30 seconds in air at pressures of 1.0, 0.3, and 0.1 atm. L-68-9095.1



(a) Air.

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Figure 6.- Ablative models tested at a pressure of 0.1 atm.

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(b) Nitrogen.

Figure 6.- Concluded.

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