ATOMIC ENERGY RESEARCH DEPARTMENT

THE EROSION OF GRAPHITE BY HIGH TEMPERATURE HELIUM JETS

Written by:

L. Green

Work done by:

D. Collins
L. Green
R. Jewell
C. Malmstrom
H. Strahl

APPROVED BY

C. STARR
TECHNICAL DIRECTOR

ISSUE DATE

May 24, 1950

NORTH AMERICAN AVIATION, INC.

P. O. BOX 309

DOWNEY, CALIFORNIA

CONTRACT AT-11-1-GEN-8

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Category: METALLURGY AND CERAMICS

Standard Distribution

	No. of Copies
Argonne National Laboratory	8
Atomic Energy Commission, Washington	4
Battelle Memorial Institute	1
Brookhaven National Laboratory	4
Brush Beryllium Company	1
Bureau of Ships	1
Carbide and Carbon Chemicals Division (K-25 Plant)	2
Carbide and Carbon Chemicals Division (Y-12 Plant)	4
General Electric Company, Richland	4
Hanford Operations Office	1
Idaho Operations Office	2
Iowa State College	1
Knolls Atomic Power Laboratory	4
Los Alamos	3
Mallinckrodt Chemical Works	1
Massachusetts Institute of Technology (Kaufmann)	1
Mound Laboratory	3
National Advisory Committee for Aeronautics	1
National Bureau of Standards	1
NEPA Project	3
New York Operations Office	5
Oak Ridge National Laboratory	6
Patent Branch, Washington	1
Sylvania Electric Products, Inc.	1
Technical Information Division, ORE	15
University of California Radiation Laboratory	3
Westinghouse Electric Corporation	4
File	25

110

ABSTRACT

The action of a heated helium jet upon a machined graphite surface was investigated. Tests with the gas and the surface heated to 2000° C revealed that once the loosely bound particles smeared into the surface during the machining operation were blown free, no further erosion was apparent provided the helium was free of oxygen and entrained particles.

This report is based upon studies conducted for the Atomic Energy Commission under Contract AT-11-1-GEN-8.

I. INTRODUCTION

In order to evaluate the suitability of graphite for structural applications in which it would be subjected to gas streams of high temperature and high velocity, it is necessary to consider the resistance of graphite to erosion by a fluid stream. Despite the known susceptibility of graphite to abrasion by action of the "sand blasting" type, there was available no information that it would be eroded due to the mechanical action of a fluid free of entrained particles. An experimental investigation of the erosion problem was therefore necessary.

II. EXPERIMENTAL REQUIREMENTS

It was desired to carry out the investigation under conditions which were at least as severe as those expected to prevail in the contemplated application. Since a maximum temperature of the order of 2000° C was considered, an inert gas was required as a working fluid. Helium was used for this purpose. Since the degree of erosion was presumed to be a function of the fluid velocity, high velocities were required. Helium also possessed advantages in this respect because of the high speed at which it transmits sound (a Mach number of 0.5 in helium at 2000° C corresponds roughly to a linear velocity of 4500 ft/sec).

An investigation of erosion behavior ideally should require a quantitative evaluation of the amount of erosion obtained and exact knowledge of the flow conditions under which the erosion took place. The second of these requirements was sacrificed in the present investigation in exchange for a highly severe test condition. For this purpose, the specimens used in the experiments were made in the form of a flat plate upon which a jet of the heated gas impinged at an angle. The flow conditions prevailing at the surface of the specimen under this arrangement appeared highly conducive to erosion but not to

analysis. Originally it was planned to evaluate the degree of erosion by measuring the loss in weight of the specimen. When later it was found that the weight changes due to erosion could be reduced in magnitude until they were comparable to the changes incurred during handling, photomicrographs of the specimen surface provided a highly sensitive (if qualitative) method of detecting erosion.

III. EQUIPMENT, PROCEDURE, AND RESULTS

A. Closed Circuit System I

A schematic diagram of the first system designed for erosion testing is shown in Fig. 1. Circulation was provided by a 10 hp Ingersoll-Rand 6 x 5 inch double acting compressor fitted with graphite rings in order to eliminate any lubricating oil from the system. After being cooled in a heat exchanger following the compressor, the gas passed into the receiver through a filter consisting of a sheet of No. 4 filter paper supported by a cylindrical screen. The rate of flow entering the furnace was measured by a Fischer-Porter Flowrator. After being heated in the furnace (described below) and passing over the erosion specimen, the gas was again cooled and filtered before re-entering the compressor. The flow rate through the furnace could be controlled either by the throttle valve ahead of the Flowrator or by diverting part of the flow directly back to the compressor through the bypass. A photograph of the assembly is presented in Fig. 2.

The general design of the furnace can be seen from the cutaway drawing in Fig. 3. The cold gas, entering from the right, was heated in passing through a hollow graphite tube carrying an electric current. The heater tube, with an outside diameter of 0.75 inch, an inside diameter of 0.25 inch, and a length of 18 inches was supplied with direct current by a 75 kw motor-generator set. In the original design, the space between the furnace wall and the graphite cylinder enclosing the heater tube was filled with carbon black in order to provide thermal insulation. Three windows permitted optical pyrometer measurements of the specimen and heater tube temperature.

Fig. 3 does not show an erosion specimen of the type that was used in the actual testing. The location of the specimen relative to the nozzle terminating the heater tube is shown in Fig. 4.

A Kinney mechanical vacuum pump was used to evacuate the closed system as the first step in the original test procedure. Due to an unavoidable leak through the stuffing box of the compressor, the pressure could not be reduced below 200 microns. The system was then filled with grade A helium (nominally of spectroscopic purity) to a pressure of about 50 psig. After being slowly circulated for a short time, this helium was bled out until only a small positive pressure remained, and was replaced with fresh helium in order to dilute the atmospheric gases which had not been removed by the vacuum pump. The rate of flow through the furnace was then increased to its working magnitude and the heater tube was brought up to temperature.

The first test run was 15 minutes in duration. The gas velocity at the nozzle throat was estimated to be about 9000 ft/sec, since the system was operating under choking conditions. Although the heater tube temperature was 2000° C, the specimen was too cold to permit a pyrometer reading, since any radiation from the specimen was obscured by the brilliant light reflected by the specimen from the inside of the heater tube. During the test, incandescent particles were observed emerging from the heater and striking the specimen. The severe erosion produced by this action can be seen from Fig. 5. The heater tube, when removed and sectioned, was found to have been roughened and to have suffered considerable loss of material as if it had been oxidized by heating in air. It was then realized that a large amount of oxygen held by the finely divided carbon insulation during the evacuation process had been released when the furnace was brought up to temperature.

The release of particles from the surface of the heater tube by oxidization was inhibited by two measures. First, the heater was lined with a tantalum tube 0.25 inch in diameter and 0.005 inch in wall thickness. In addition, the finely divided carbon was replaced by more coarsely divided alumina, a material with less affinity for oxygen. When oxidation difficulties persisted, the insulation was removed completely and

three concentric radiation shields were successfully substituted. Additional precautions were also taken to remove oxygen from the working fluid. The traps employed were a coil immersed in liquid nitrogen and a column of copper shavings heated by a muffle furnace.

Each of the measures mentioned above effected a decrease in the amount of erosion experienced by the test specimen, but complete elimination of erosive action was not realized. It was believed that the existence of the large leaks in the compressor and the general lack of cleanliness of the circulating system (a large part of which had been fabricated from commercial galvanized pipe) would preclude any well controlled investigation of gaseous erosion independent of chemical action. Consequently, it was decided to modify the equipment to utilize helium straight from the bottle without recirculation.

B. Open Circuit System

As mentioned above, one defect of the first erosion experiments was that the specimen itself, heated only by the gas impinging upon it, could not be tested at temperatures as high as were desired. This defect was corrected in the second system by heating the specimen with an electric current. The method of supporting the specimen can be seen from Fig. 6. After a few tests were made with cold gas impinging upon the heated specimen, the specimen chamber shown in Fig. 6 was attached to the heater tube assembly of the original system and the tests were continued with heated gas. In this case, the heater tube and the specimen were connected in parallel since only one current source was available. A variable resistor in series with the specimen permitted the specimen temperature to be adjusted as desired relative to the heater temperature.

In starting the operation of this system, the exit valve leading to the atmosphere was closed and the system was evacuated. During evacuation the specimen (and heater, if used) were heated to a few hundred degrees in order to promote outgassing of the graphite parts. Following this process, helium was introduced, the exit valve was opened to establish flow, and the specimen (and heater) were brought up to an operating temperature of 2000° C. Although the open circuit system utilized a nozzle with a throat diameter of only 0.063 inch, the throat velocity was about 9000 ft/sec and the rate of helium consumption was high. A manifold of five bottles would supply only about four hours of opera-

tion. Therefore, the experimental tests, which lasted about eight hours apiece, involved the difficulty of changing bottles.

The results of these short-duration tests, however, were encouraging. It had been noticed in the earlier testing that some erosion of the machined surfaces of the graphite specimens appeared almost immediately upon the beginning of the test, and it was suspected that such early erosion resulted primarily from a "dusting" action in which the loose particles which had been smeared into the surface depressions during the machining operation were blown free (It had also been noted that such "dusted" surfaces freshly revealed by a fracture, lacked the "characteristic greasy feeling" usually associated with graphite, and that the material could not be easily rubbed off). In the early tests, however, continued erosion due to chemical action had obscured the effect. The open circuit system using a fluid of increased chemical purity permitted two separate stages of erosion to be distinguished. It was found that the primary erosion, or "dusting", was completed within a few hours, and that the secondary of progressive erosion might possibly be eliminated by proper purification of the gas.

Although encouraging, the results obtained with the open circuit system were not conclusive because of their short duration. Neither were they consistent, for while one test might show only the primary dusting, another would still exhibit progressive erosion after several consecutive days of operation. Perhaps the bottled helium was not of consistently high enough purity to insure the absence of chemical action. It was, therefore, decided to build another circulating system which could be kept clean, in which there would be no large leaks, and which would incorporate a means of oxygen removal.

C. Closed Circuit System II

The second closed circuit system, outlined schematically in Fig. 7, was constructed on a much smaller scale than the original system. Instead of using large galvanized pipe as done originally, the new system utilized 1/2 inch seamless copper tubing which was carefully cleaned before assembly. Sufficient flow capacity was provided by two 1/2 hp Kellogg diaphragm type compressors connected in parallel, thus eliminating the problem of stuffing box leakage.

The removal of oxygen from the helium stream was accomplished by the use of a column of copper shavings heated by a muffle furnace. A Brown recording

and controlling potentiometer was used to keep the temperature of the shavings (as measured by a chromel-alumel thermocouple) in the vicinity of 350°C , a temperature sufficiently high to insure oxidation of the copper. As mentioned above, this same system of purification had been tried in conjunction with the original large scale system. In that application, however, it had not been completely effective since the mass flow rate of helium through the column of shavings had been so great that the muffle furnace (with a rating of only 3.4 kw) had been unable to keep the column at a sufficiently high temperature.

The rate of flow entering the furnace was measured in the new system by a Schutte-Koerting Rotameter. The furnace itself was redesigned in order to attain higher gas temperatures than had been possible earlier. In the original furnace, the gas flowed down the entire length of the higher tube. Since the tube temperature was lower at the ends than in the middle, the gas would be cooled in flowing out the downstream end. Expansion through the nozzle at that point produced additional undesirable cooling. In order to remedy this defect, the new furnace utilized a heater tube in which the gas passed through only about three-quarters of the tube length and then made its exit normal to the tube at a high temperature. In addition, the inner diameter of the tube was reduced to 0.125 inch, and the constriction at the exit was eliminated. The specimen was heated electrically in the same fashion as in the open circuit system. The essential details of the furnace design are presented in Fig. 8. A view of a typical erosion specimen used in this furnace is presented in Fig. 9.

The results obtained with the system described above justified the belief that the erosion of graphite could be restricted to a primary "dusting" action. Fig. 9 presents photomicrographs taken at a magnification of 17.5X of a surface of C-18 graphite. The photomicrographs were made at intervals of 1, 8.5, 16, and 23.5 hours after the beginning of a test in which the specimen surface at a temperature of 2000°C was subjected to a helium jet with a throat velocity of 9000 ft/sec and a temperature of about 2000°C . The one hour run prior to the first photograph provided enough dusting action to mark the area of impingement of the jet and thus to locate the most suitable region to be photomicrographed.

The noticeable difference in appearance exhibited by the surfaces of Fig. 10-a and 10-b reveals that the initial dusting had not been completed at the end of the first hour of testing. The extreme similarity of Fig. 10-b,

10-c, and 10-d, however, indicates that there was no appreciable change in the surface structure during the last fifteen hours of the test. Fig. 11 presents 50X photomicrographs of the surfaces shown in Fig. 10-c and 10-d. Even at this higher magnification it may be seen that except for a slight apparent shift in the plane of focus, the two surfaces appear identical. The photomicrographs of Fig. 10 and 11 provide a basis for the belief that the erosion of graphite by helium streams can be limited to an initial transient phase in which loose particles are removed from the surface.

IV.. CONCLUSIONS

A machined graphite surface exposed to the action of a high velocity high temperature helium stream is subjected to an initial erosion process in which the loosely held particles smeared into the surface during the machining operation are blown free. Once this process is complete, no further erosion is apparent provided the helium is free of chemically reactive impurities and entrained particles. Difficult problems may be encountered, however, in maintaining the necessary chemical and mechanical purity of the fluid in a practical engineering application.

Figure 1. Closed Circuit System I.

Figure 2. View of Closed Circuit System I.

13

Figure 3. Cutaway Sketch of Erosion Furnace.

Figure 4. Sketch Showing Position of Graphite Erosion Specimen Relative to Nozzle Terminating the Heater Tube.

Figure 5. Graphite Specimen After First Erosion
Test (15 Minute Duration).

Figure 6. Sketch Showing Principal Features of the Specimen Chamber Utilized in the Open Circuit System.

17

Figure 7. Closed Circuit System II.

Figure 8. Sketch Showing Principal Features of the
Furnace Used in Closed Circuit System II.

Figure 9. Type of Specimen Used in Open Circuit System and Closed Circuit System II.

(a) 1 hour

(b) 8.5 hours

(c) 16 hours

(d) 23.5 hours

Figure 10. Photomicrographs Taken at 17.5X Magnification of a Surface of C-18 Graphite Subjected to the Action of a Helium Jet at a Temperature of 2000° C for Stated Intervals of Time.

21

(a) 16 hours

(b) 23.5 hours

Figure 11. Photomicrographs Taken at 50X Magnification of a Surface of C-18 Graphite Subjected to the Action of a Helium Jet at 2000° C for Stated Intervals of Time.