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THE EFFECT OF OXIDATION ON THE COMPRESSION
STRENGTH OF CS-GBF GRAPHITE.

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April 11, 1956

AEC RESEARCH AND DEVELOPMENT REPORT

THE EFFECT OF OXIDATION ON THE COMPRESSION STRENGTH

OF CS-GBF GRAPHITE

by

Bruce Griggs

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INTRODUCTION

Limitations on the maximum temperature of the pile moderator have been imposed to prevent or control the gasification reaction of the graphite stack with the pile gas atmosphere.^(1,2) An uncontrolled reaction would cause a loss of structural strength, density, and thermal conductivity and would thus result in an unnecessary shortening of the effective operating life of the HAPO piles. Numerous studies^(3,4,5) have been made to define the rates of reaction of the pile stack with various components of the pile gas and the influence on these rates of various pile operating conditions. Based on these studies, maximum graphite temperature has been so restricted that after 10 years of pile operation the graphite stack has sustained less than 1% oxidation (based on recent experimental data).⁽⁶⁾

As pile power levels increased, the maximum graphite temperature has also increased and will increase still more as present and future power level programs are completed.⁽⁷⁾ In order to evaluate the effect on the graphite stack of the present programs, and to some extent determine the feasibility of future programs, a more realistic approach to the question of structural integrity and oxidation versus pile life is necessary.

It is the purpose of this report to summarize the data obtained in a preliminary study of the effect of oxidation on the compressive strength of graphite.

SUMMARY AND CONCLUSIONS

An experimental procedure was developed which gave relative results on the compressive strength of both irradiated and non-irradiated graphite samples as a function of oxidation. Sufficient difficulty was encountered in the experimental procedure to warrant further refinement. The results obtained indicate the following:

- 1) For non-irradiated graphite there is a rapid decrease of compressive strength during the initial oxidation. The rate of decrease in compressive strength gradually lessens as the sample is further oxidized.
- 2) For non-irradiated graphite, the compressive strength is decreased by a factor of ~ 2 for 10% oxidation and a factor of ~ 6 for 25% oxidation.
- 3) Irradiated samples exhibited greater compressive strength than the non-irradiated. However, the same relationship between compressive strength and density was observed for both irradiated and non-irradiated samples.
- 4) The compressive strength observed for non-irradiated graphite was ~ 900 psi at 23% oxidation and 2600 psi at 10% oxidation; for irradiated graphite the compressive strength was 3100 psi at 10% oxidation. Based on these data, it would appear that the 10% permissible oxidation which is used as a basis for setting the maximum permissible graphite burnout rate and thereby the pile moderator temperature, is quite conservative.

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DISCUSSION

A. Experimental Procedure

The test procedure consisted of preparing the samples, oxidizing the samples in a quartz tube furnace, and subsequently performing compression testing on a 30 ton Baldwin testing machine utilizing a Baldwin sub-press.

In these initial experiments, the type of graphite was restricted to CS-GBF. The samples were 1" diameter cylinders cut perpendicular to the axis of extrusion of a bar 4 3/16" square. Only the central 2" of the 4 3/16" length was used. As a means of assuring uniformity in the oxidation of the test samples, each sample was machined to 1" x 2" right circular cylinder with a 3/4" axial hole. Initially, density profiles were obtained on test samples to determine the structural uniformity of the test bar. Subsequently these density profiles were used to monitor the uniformity of the oxidation of the test samples. These profiles were obtained by carefully measuring the samples as to weight and dimensions, mounting on a lathe and reducing the diameter in 0.02" increments, and carefully remeasuring. From these measurements, the density of the incremental shell and the residual cylinder could be calculated.

Two methods of achieving a uniform oxidation of the sample were suggested in a report by Petersen.⁽⁸⁾ One of these methods was to reduce the thickness of graphite which the reactant gas had to penetrate. The other method was to reduce the rate of the reaction. Because of the time consideration, the first method was adopted. This led to the choice of hollow cylinders as a test sample. Initial compression testing indicated that the data obtained from such samples would be reproducible and could be correlated with solid test samples.

The actual oxidation of the samples was accomplished by passing a stream of dehydrated, deoxygenated carbon dioxide over the heated samples at a rate of about 500 cc/min. S.T.P. The samples were held in a quartz carrier in groups of three. A ring of graphite containing a chromel-alumel thermocouple was placed at the end of each sample. This assembly was heated in a 1.7" inside diameter quartz tube contained in a 2" x 16" electric furnace. The assembly was located in the tube to give a minimum temperature gradient along the three samples (usually $\pm 20^\circ\text{C}$). The oxidation rate varied between 0.5%/day for the solid samples to as high as 6%/day for the hollow samples. The temperature of oxidation varied between 900°C and 1000°C with the majority falling around 950°C .

Irradiated samples which were not oxidized were heated for one day in argon at the same temperature as the oxidized samples to assure the radiation damage was annealed to the same extent.

Compression testing was performed on a Baldwin 30 ton hydraulic testing machine utilizing a small Baldwin sub-press. The rate of head movement was between 0.002"/min. and 0.005"/min. Stress-strain curves were obtained for all samples using a standard microformer attachment for the machine. Stress-strain curves which showed a significant fracture below the ultimate crushing strength were discarded. Less than 10% of the samples showed this behavior.

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B. Results and Discussion

The results from these experiments are presented in Figures 1 to 6. Figures 1, 2, and 3 show the loss of ultimate strength (psi) as a function of sample density. Sample density⁽⁹⁾ is plotted in preference to percent oxidation to minimize the scatter and also because it more truly represents the quantity of material resisting the applied stress. Representative stress-strain curves for samples oxidized to various extents are presented in Figures 4, 5, and 6.

From Figure 1, the scatter of the data is self-evident. In view of the nature of graphite and its method of manufacture, such scatter is not unreasonable nor unexpected. Compression test results are sensitive to many variables such as grain size, preferred orientation, and internal flaws. These variables are not carefully controlled, uniformly distributed, or accurately known in pile graphite which accounts for a considerable scatter in data. This scatter of results makes fundamental interpretations difficult and uncertain without large numbers of samples of various types of graphite. The scatter emphasizes the need for multiple sampling and testing to assure that the data are representative and the need for caution in the application of the absolute numbers on ultimate strength.

The validity of the hollow test pieces in showing the relative amount of damage by oxidation is certainly open to question on a quantitative basis. Figures 1 and 2 show that the hollow test pieces displayed about 12% less strength than the solid pieces of the same density. The rate of decrease of strength with density for solid and hollow pieces seem in good agreement in the higher density range. The solid samples seem to decrease in strength more rapidly than the hollow samples in the lower densities. However, because of the time involved the highest oxidation achieved for a solid sample was only 6%. With data covering such a small density range, the scatter of data might lead one to an erroneous slope for the density vs. crushing strength line. In the absence of data in the region of 20% oxidation for solid test pieces, one cannot establish the relation between solid and hollow test pieces with certainty. Nevertheless, the results for the hollow test pieces should be qualitatively correct. A comparison of Figures 4 and 5 shows that the initial portion of the curves for solid and hollow test pieces of equal densities are essentially the same.

RECOMMENDATIONS

The scatter of the data (see Figure 1) and the lack of agreement in the ultimate compression strength of hollow cylinders and solid cylinder test pieces indicates that more appropriate test pieces and procedures should be developed.

Additional testing is necessary to determine the amount of scatter in the data which is exclusive of the test procedure but inherent in the material being tested. This work should be expanded to include other types of pile grade graphite and to include tensile and shear strength.

A study should be made on the application of these and other graphite strength data to methods of pile operation in terms of increased production, decreased unit cost, and effect on pile life.

ACKNOWLEDGMENT

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Fig. 1

Ultimate Crushing Strength of Oxidized and Unoxidized CS-GBF Graphite

Perpendicular cut, 1" x 2" right circular cylinders with 3/4" hole.

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Fig. 2

Ultimate Crushing Strength of Oxidized and Unoxidized CS-GBF Graphite
Perpendicular cut, 1" x 2" right circular cylinders.

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Fig. 3

Ultimate Crushing Strength of Oxidized and Unoxidized CS-GBF Graphite.
 Perpendicular cut. 1" x 2" right circular cylinders with a 3/4" axial hole.

X = Irradiated 3574 MWD/AT. Annealed at 970 C.
 O = Unirradiated from the same bar.

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Fig. 4

Stress-Strain Curves for Oxidized and Unoxidized CS-GBF Graphite.
 Perpendicular cut, 1" x 2" right circular cylinders with 3/4" hole.
 X indicates rupture. Numbers on curves indicate density for that line.

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Fig. 6

Stress-Strain Curves for Oxidized and Unoxidized CS-GBF Graphite.
 Perpendicular cut, 1" x 2" right circular cylinders with an axial 3/4" hole.
 X indicates rupture. Numbers on lines indicate the density in g/cc.

Heavy lines are Irradiated 3574 MWD/AT and annealed at 970°C.
 Light lines are virgin graphite from the same bar.

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