MASTER

**EFFECT OF PRESTRESS AND STRESS ON THE
STRENGTH AND OXIDATION RATE OF
NUCLEAR GRAPHITE**

M. Eto and F.B. Growcock

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**HTGR SAFETY DIVISION
DEPARTMENT OF NUCLEAR ENERGY, BROOKHAVEN NATIONAL LABORATORY
UPTON, NEW YORK 11973**

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**HTGR SAFETY DIVISION, DEPARTMENT OF NUCLEAR ENERGY
BROOKHAVEN NATIONAL LABORATORY, ASSOCIATED UNIVERSITIES, INC.
UPTON, NEW YORK 11973**

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ABSTRACT

Effects of prestress or stress during oxidation on the reactivities and strengths of PGX and H451 graphites were examined at oxidation temperatures of 450-600°C in air and 550 - 750°C in 2% H₂O/He. Little or no effect of compressive prestress at stress levels of up to 75% of the mean fracture strengths, was found. A small effect on the reactivity of H451 graphite was observed in the case of 2% H₂O/He, i.e. the rate was enhanced by no more than 30% for compressive prestress levels of up to 90% of the fracture strength.

Tensile and compressive stresses during oxidation did not affect the reactivities of these graphites at stress levels of up to 28% of the mean compressive strength and 35% of the mean tensile strength for H451 graphite, and 44% and 56% for PGX graphite, respectively.

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1. INTRODUCTION

In high temperature gas-cooled reactors (HTGRs), nuclear graphite is used as structural material, as well as moderator and reflector. Mechanical strength is one of the most important properties by which structural graphite is judged. During normal reactor operation or following an accident that leads to depressurization, impurity gases in the helium coolant may oxidize the graphite and compromise the integrity of the reactor core and core support regions; in addition, the graphite components are subjected to various types of stresses. Evaluation of the effect of stress on the oxidation behavior and strength of nuclear graphite is believed to be important in terms of the design and safety analysis of the reactors.

It is well known that prestress or stress can cause pronounced changes in some physical and mechanical properties of graphite.²⁻⁷ No studies on oxidation of the material have, however, been reported in which both prestress and stress were investigated. Krefeld et al.⁸ first reported that a tensile stress of 20 kg/cm² (ca. 2 MN/m²) enhanced the oxidation rates of several graphites by a factor of ca. 3 and decreased their ultimate tensile strengths by ca. 10% compared with unstressed specimens. Kubaschewski et al.⁹ obtained no enhancement of the oxidation rates of similar graphites exposed to a wide range of tensile stress, and Growcock and Chow¹⁰ likewise saw no effect on the oxidation rates of HTGR graphites over a limited range of tensile stress, flow rate and temperature. Compressive stresses were applied to grades 2020 and H440 graphites by Thrower, who, again, found no effect of stress on oxidation rate.¹¹ Imai and Sasaki, however, found some increase in the rate of oxidation in air of H327 graphite prestressed in compression to 90% of the mean fracture stress; they also obtained data showing that the effect diminishes with increasing reaction temperature.¹² Since the results of these investigators are not entirely consistent, it was judged worthwhile to reinvestigate the effects of stress and prestress on oxidation and strength in more detail. Data concerning the effects of prestress on the oxidation rates and ultimate compressive and tensile strengths of graphite grades H451 and PGX will be presented and discussed in this report. The former is a candidate for the core and reflector materials, while the latter is used for the core support blocks in the Fort St. Vrain HTGR. Additionally, data pertaining to the effect of stress during oxidation on the reaction rates of these graphites will be presented.

2. EXPERIMENTAL

2.1 Materials

The graphites used in this study were grades H451 (Great Lakes Carbon Corporation) and PGX (Union Carbide Corporation). Some properties of these graphites are summarized in Table 1.

Table 1

Some Properties of the Materials Used in the Study

		Density (g/cm ³)	Young's Modulus (GN/m ²)	Tensile Strength (MN/m ²)	Compressive Strength (MN/m ²)	Method of Manufacture
PGX*	WG	1.78	7.0	9.5	41.0	Conventional Molding
	AG	1.74	6.0	10.3	43.0	
H451**	WG	1.75	8.27	16.3	55.3	Extrusion
	AG		7.13	12.9	52.7	

*After Johnson and Engle, GA-A-13752(1976)

**After Engle, GA-A-14328(1977)

Cylindrical compressive specimens 19.05 mm ϕ by 38.10 mm in length were used for the experiments on the effect of prestress on the reaction rates and strengths of PGX and H451 graphites. Specimens of the former were machined from a half-moon shaped block of PGX graphite 48 cm in radius and 9 cm thick which was cut from a mid-length portion of a production grade block ca. 127 cm ϕ by 196 cm length. H451 specimens were machined from a half-moon shaped H451 graphite block (46 cm ϕ by 15 cm thickness) which was cut from a mid-length portion of a production grade block 86 cm in length. The longitudinal axis of the H451 specimens was parallel to the grain, while the axis of the PGX specimens was perpendicular to it.

For the experiments on stress-during-oxidation, two kinds of cylindrical specimens were used. Compressive specimens of PGX and H451 measured 12.7 mm ϕ by 25.4 mm length. Tensile specimens measured 12.7 mm ϕ by 168.3 mm length; machine threads were put on both ends of the tensile specimens used in the stress experiments, according to the dimensions in the drawing in Figure 1. The longitudinal axis of all the H451 and PGX graphite specimens was parallel to the radial direction of their parent blocks; these half-moon shaped blocks measured 43 cm ϕ by 17 cm length (H451) and 54 cm ϕ by 9 cm length (PGX).

Prior to use, all the specimens were cleaned ultrasonically in absolute methanol and dried in air at 75 to 80°C for about 10 hr.

2.2 Experiments on Tensile and Compressive Stresses before Oxidation

Prestressing and Strength Measurements:

In the prestress experiments, compressive prestresses were applied along the longitudinal axis of the specimens to a stress level between 25 and 90% of the mean compressive strength of each graphite. The strain rate was $1.1 \times 10^{-3} \text{ sec}^{-1}$. The residual strain caused by prestressing ranged from 0 to 0.4% (depending on prestress level and kind of graphite), a part of which was recovered during oxidation in air or water vapor/helium mixture. Prestressing and strength measurements of the larger compressive specimens were

Figure 1. Dimensions (in inches) of the Specimens Used in the Present Experiments: (A) and (B), Compressive Specimen; (C) Tensile Specimen for Stress Rig.

carried out using a Tinius Olsen Universal Test Machine with maximum allowable load of ca. 5.45 tons. These strength tests were also done at a strain rate of $1.1 \times 10^{-3} \text{ sec}^{-1}$.

Oxidation:

Oxidation of specimens was carried out in a quartz tube through which various atmospheres such as air and 2% H₂O/He with or without H₂ were passed, generally at a flow rate of 300 sccm. Higher flow rates were necessary in a few cases (high oxidation rates) to limit conversion of the oxidant to <5%. Gas metering was performed with Aalborg flow controllers which had been calibrated against Tylan electronic flow controllers. The water vapor mixtures were made by passing helium with or without hydrogen either over a 9:1 mixture of oxalic acid dihydrate (H₂C₂O₄·2H₂O): oxalic acid anhydrate (H₂C₂O₄) maintained at 50°C or through a bubbler containing doubly-distilled H₂O which was maintained at ca. 18°C. Both blended mixtures provided water vapor at a concentration of ca. 2%.

Reaction temperatures ranged from 400 to 650°C for oxidation in air and 550 to ca. 900°C for oxidation in 2% H₂O/He with or without H₂.

2.3 Experiments on Tensile and Compressive Stresses during Oxidation

A stress rig was set up to examine the effect of tensile and compressive stresses during oxidation on the reaction rates of PGX and H451 graphites in 2% H₂O/He. Figure 2 shows the main part of the rig. A quartz tube 100 mm O.D. by 96 mm I.D. enveloped the rig and served as a containment vessel for the gaseous reactants. Three tensile and three compressive specimens were stressed during oxidation by applying a load to the lower pull-rod using an air piston. The stress level ranged from 0 to 55% of the mean tensile or compressive strength. The experiments were carried out at 650 or 700°C with a reactant flow rate of 300 sccm. A view of the stress rig is shown in Figure 3.

2.4 Measurements of Reaction Rate

Reaction rates for oxidation in different atmospheres were calculated from the concentrations of the carbon-containing product gases, which were monitored with a Carle Analytical Gas Chromatograph Model 311. Methanation of these gases followed by analysis with an FID gave rise to minimum detectable limits of 20 and 50 ppb for CO₂ and CO, respectively. All other species were measured with a thermistor TCD. The detectors were calibrated with mixtures of gases of similar composition as that of the product gases. The oxidation rate is given by the weight loss expression

$$-\frac{dw}{dt} = \frac{12q}{RT} \sum_i \frac{P_i}{v_i} ,$$

STRESS APPARATUS

Figure 2. Schematic of the Main Part of the Stress Rig

Figure 3. Overview of the Stress Experiment

where W_C is the weight of carbon, q is the flow rate of the gas mixture, P_j is the partial pressure of carbon-containing product species, and ν_j is a stoichiometric coefficient (e.g., $\nu_{CO} = 2$ for $C + CO_2 \rightarrow 2CO$).

The mean reaction rate for each oxidation experiment was estimated from the weight loss of a specimen and the reaction time.

3. RESULTS AND DISCUSSION

3.1 Effect of Prestress on Strength and Oxidation Rate

The compressive strength of PGX graphite oxidized in a 2% H₂O/He mixture (with or without H₂) and in air is plotted in Figure 4 as a function of burnoff. In the case of 2% H₂O/H₂/He, the flow rate of H₂ was set at 120 sccm and that of He at 480 sccm, giving [H₂] = 20%.

The data obtained for H451 graphite are shown in Figure 5, where the specimens were oxidized either in a 2% H₂O/He mixture or in air, with or without prestress up to 75% of the mean strength.

Effect of prestress on strength:

The results displayed in Figures 4 and 5 indicate that prestressing these graphites up to 90% of the mean strength does not affect their compressive strengths. In the temperature ranges examined, the strength loss does not depend on the reaction temperature either, which is believed to indicate that burnoff of all these specimens occurred in a similar manner. This is discussed elsewhere.¹³

It has been claimed that compressive prestress has no effect on the ultimate compressive strength of unoxidized graphites⁵, though many physical properties such as coefficient of thermal expansion, electrical resistivity and Young's modulus are drastically influenced by prestress.^{2-4,6,7} It is also believed that the compressive strength of both unoxidized and oxidized graphites is not very sensitive to microstructural changes.

Effect of prestress on reaction rate:

Figure 6 illustrates the time dependence of the reaction rate of PGX graphite specimens prestressed to various levels and oxidized at 500°C in air. The rates of prestressed specimens lie within the scatter band. The oxidation rate in a 2% H₂O/He mixture at 100°C is plotted as a function of exposure time in Figure 7. The open circles represent the oxidation rate before stressing and filled circles represent the rate after stressing.

The figure indicates that no enhancement of the rate of PGX graphite results from the application of prestress at room temperature. This result does not agree with that obtained for H327 graphite by other investigators.¹²

Figure 4. Compressive Strength of Prestressed or Non-Stressed PGX Graphite Oxidized in Air or in 2% H₂O/(20% H₂)/He at Different Temperatures

Figure 5. Compressive Strength of Prestressed or Non-Stressed H451 Graphite Oxidized in Air or in 2% H₂O/He at Various Temperatures

Figure 6. Reaction Rate of PGX Graphite Specimens Prestressed to Various Levels and Oxidized in Air at 500°C as a Function of Exposure Time

Figure 7. Reaction Rate of PGX Graphite Oxidized in 2% H₂O/He at 700°C as a Function of Exposure Time

Similar experiments were done for H451 graphite oxidized in air and in 2% H₂O/He. An H451 graphite specimen was first oxidized at 500°C or 550°C in air at 300 sccm or at 750°C in 2% H₂O/He at 200 sccm until a stabilized rate was obtained. The specimen was then stressed to 75% of its mean compressive strength, which was estimated from the results given in Figure 5. The results for air oxidation are shown in Figures 8 and 9; the observed rate increase of less than 15% is too small to be considered significant at this time. Figure 10 shows the result obtained for H451 graphite oxidized in a 2% H₂O/He mixture at 750°C. The data indicate that prestress caused ca. 50% increase in the reaction rate. The data plotted in Figure 11 were obtained after oxidation had been permitted to continue for 40 hr. To confirm that the enhancement observed above was not an artifact, an H451 control specimen, which was prestressed to 75% of the mean compressive strength before the first run, was oxidized in a similar manner. This time no rate enhancement was observed upon reexposing the specimen to the oxidizing conditions. This can be seen in Figure 12. In these experiments maximum burnoff of the specimens was less than 1%.

The oxidation rate was also measured for an H451 graphite specimen which was prestressed in stepwise fashion, ranging from 25% to 85% of the mean compressive strength. The specimen was first oxidized in a 2% H₂O/He mixture at 750°C at a flow rate of 300 sccm until a stabilized rate of reaction was attained. Then the specimen was taken out of the reactor, given a prestress of 25% of the mean strength and oxidized in the same manner as before, until a stabilized rate was attained again. The same procedures were followed for prestressing to 50% and 85% of the mean compressive strength. The result is shown in Figure 13, where the maximum burnoff was ca. 0.4%. This result suggests that a prestress level larger than 50% of mean compressive strength causes some enhancement of the reaction rate of H451 graphite. Figure 14 shows the stabilized rates in units of g/g/min versus prestress levels normalized to the mean compressive strength. It has been shown previously that substantial changes in various properties and microstructure are observed for different nuclear graphites if the applied stress level is higher than 50% of the ultimate compressive strength.²⁻⁴ The results obtained here suggest that reactivity may be enhanced by compressive prestresses greater than 50% of the mean strength, but the effect, if real, is not very large. This conclusion is consistent also with the results of Imai et al.,¹² who obtained, for H327 graphite prestressed to 90% of the mean compressive strength and oxidized in air at 430°C, a reaction rate twice as high as that obtained for unstressed specimens.

Imai et al.¹² also found that the enhancement of reaction rate was annealed out at higher temperatures; this suggests that the smaller effect observed in the present work, in the case of oxidation in air, may result from the higher oxidizing temperatures employed. In the case of H451 oxidized in 2% H₂O/He at 750°C (Figures 10 and 13), the temperature is believed to be too high for the same mechanism to apply, which leads us to think that some unannealable defects, such as cracks initiated above ca. 50% of the mean strength,³ are related to the enhancement of the rate.

The lack of effect observed in the case of PGX graphite oxidized in 2% H₂O/He may be attributed to the high concentration of impurities in this

Figure 8. Rate of Oxidation of H451 Graphite in Air at 500°C as a Function of Exposure Time

Figure 9. Rate of Oxidation of H451 Graphite in Air at 550°C as a Function of Exposure Time

Figure 10. Rate of Oxidation of H451 Graphite in 2% H₂O/He at 750°C as a Function of Exposure Time

Figure 11. Reaction Rate as a Function of Exposure Time for the H451 Graphite Specimen Shown in Figure 10 After 40 Hr. of Exposure

Figure 12. Reaction Rate of an H451 Control Specimen, Which Was Prestressed to 75% of the Mean Compressive Strength Before the First Run, as a Function of Exposure Time

Figure 13. Reaction Rate of an H451 Graphite Specimen Which Was Prestressed in Stepwise Fashion and Oxidized in 2% H₂O/He at 750°C

Figure 14. Stabilized Reaction Rate of H451 Graphite Specimen Which Was Prestressed in Stepwise Fashion as a Function of Normalized Prestress Level

material; impurities such as iron may act as catalysts for the oxidation and mask the effect of structural changes produced by prestress. This point will be discussed elsewhere in a report dealing with the strength loss mechanisms in nuclear graphites.¹³

3.2 Effect of Stress during Oxidation on Reaction Rate

The effect of stress applied during oxidation on the reaction rates of PGX and H451 graphites was studied using compressive loads of up to 44% and 28%, respectively, of the graphites' ultimate compressive strength and tensile loads of 56% and 35%, respectively, of the graphites' ultimate tensile strength.

The results for PGX graphite oxidized at 440° and 650°C in 2% H₂O/He are plotted in Figures 15 to 17 in the form reaction rate vs. time. Application of stress during the oxidation, even in alternating fashion -- stress, no stress, stress -- did not produce any measurable difference in the reaction rates. Results for H451 graphite, likewise oxidized in 2% H₂O/He but at a temperature of 700°C, are shown in Figure 18. Here again no effect of stress on reaction rate was observed.

These results are consistent with all known data on the effect of stress on reactivity, except that of Krefeld et al.⁸ It is possible that the experimental parameters were not in the proper range to see such an effect: the stress loads may have been too small, stress may have been applied for too short a time, the temperature may have been too low, or a combination of these may have been at work. However, data have been gathered by other workers over a wide range of conditions, even using flow rates, temperatures, and materials similar to those of the Petten workers; no effect whatsoever of stress on reactivity has been observed in any of these cases. One cannot exclude the possibility, however, that at very high stress levels some enhancement of the reaction may occur, such as appears to be the case in prestress experiments.

4. CONCLUSIONS

The effects of prestress on the reactivities and ultimate compressive strengths of PGX and H451 graphites have been examined. Little or no effect of prestressing these graphites, generally up to 75% of the mean fracture strength, was found when these graphites were oxidized in air or 2% H₂O/He. There may be a small effect on the reactivity of H451 graphite oxidized in 2% H₂O/He, but even in this case the rate is enhanced by no more than 30% for prestress levels less than 90% of the ultimate compressive strength.

The effect of stress during oxidation on the reactivities of nuclear graphites in 2% H₂O/He was also investigated. The results, which are consistent with those of most investigators, indicate that stress does not affect the reactivities of H451 and PGX graphites. Maximum stress levels used for H451 graphite were 28% of the mean compressive strength and 35% of the mean tensile strength. Maximum stress levels used for PGX graphite were 44% of the mean compressive strength and 56% of the mean tensile strength.

Figure 15. Reaction Rate vs. Exposure Time of PGX Graphite Subjected to Various Levels of Stress While Being Oxidized at 400°C in 2% H₂O/He

Figure 16. Reaction Rate vs. Exposure Time of PGX Graphite Oxidized Under Stress at 650°C in 2% H₂O/He

Figure 17. Reaction Rate vs. Exposure Time of PGX Graphite Subjected to Various Stress Levels While Being Oxidized at 650°C in 2% H₂O/He

Figure 18. Reaction Rate vs. Exposure Time of H451 Graphite Subjected to Various Levels of Stress While Being Oxidized at 700°C in 2% H₂O/He

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