THE EFFECT OF OXIDATION ON THE COMPRESSIVE STRENGTH OF GRAPHITE*

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Introduction

The use of graphite for load bearing members in a nuclear reactor with a slightly corrosive environment calls for an understanding of those factors which influence the degradation of mechanical properties by gasification processes. The work reported here is solely concerned with the way in which oxidation in air influences the compressive strength and strain-to-failure of two different graphites, viz., Stackpole 2020 and Great Lakes H440 grades. It is expected to have data on gasification by CO_2, and possibly H_2, available for presentation at the conference.

Experimental

Samples of both graphites in the form of right cylinders 0.25" diameter (ϕ) x 0.50" long (ℓ) and 0.75"ϕ x 1.50"ℓ were compressed to fracture in an Instron testing machine. It was found that thin pieces of Teflon tape at the ends of the sample minimized frictional effects. The compressive strengths and strains-to-failure were found to have the same mean values for both sample sizes in each case.

Strain rate was varied between 0.1"/min and 0.002"/min and again no effect was found on either the fracture stress or strain-to-failure. It was therefore decided to use a strain rate of 0.01"/min and samples of 0.25"ϕ x 0.50"ℓ.

The data presented here were obtained by oxidizing samples in air in an open ended tube furnace at temperatures between 500°C and 800°C. The graphite cylinders were held in Pyrex tongs during the oxidation which protected the end faces from reaction and thus allowed the samples to be used in subsequent compression tests without their failing due to these faces crumbling.

Results

A comparison of compressive strength and strain-to-failure as measured on unoxidized samples and the corresponding values given by the manufacturers is given in Table 1. The measured strains-to-failure are approximately double those anticipated.

The effect of oxidation on the compressive strength (σ_c) of the two graphites is shown in Figure 1. It can be seen that even though oxidation temperatures varied between 500°C and 800°C, there is apparently no effect of temperature on the relationship between strength and burn-off. For both graphites, a burn-off of 8-9% is sufficient to produce a 50% loss in strength.

Table 1

Graphite	Fracture Stress (psi)		Strain-to-Failure (%)	
	Given	Expt.	Given	Expt.
H440	7,800	8,100	~2	4.4
2020	13,000	11,000	1.9	4.3

While it was impossible to accurately measure the diameter of oxidized specimens due to their friable surface, it was somewhat surprising that even up to 50% burn-off there was no appreciable change in external sample dimensions.

Examination of sections of oxidized samples using the scanning electron microscope reveals no boundary between oxidized and unoxidized areas and oxidation appears to be reasonably uniform throughout the sample. Figure 2 shows regions of a sample of H440 graphite oxidized to 15% burn-off. While there is no significant difference between photographs from different regions of the sample, it is obvious that the binder is oxidized much more rapidly than the filler. Whereas oxidation of filler particles occurs mainly at their edges, oxidation of binder occurs at sites throughout its volume. The larger number of active sites in the binder is responsible for its rapidly being transformed into a spongy material while surrounding filler particles are relatively untouched. A higher magnification view of binder material (Fig. 2c) clearly shows that the binder has a rather crude layer structure and that oxidation has occurred at points within layers as well as at layer edges.

Discussion

The production of a 50% loss in compressive strength by a 8-9% burn-off irrespective of oxidation temperature is in agreement with the results of Board and Squires [1] on the effect of CO_2 oxidation on British PGA graphite. It was assumed that such a large strength reduction was due to loss of binder which holds filler particles together. Fig. 2 does in fact show greater reactivity of binder over filler and hence the hypothesis is probably valid.

A plot of $\log_{10} \sigma_c$ against burn-off is shown in Figure 3. For the 2020 graphite, the relationship appears to fall on two parallel lines with a jog at about 28% burn-off. On the other hand, all data for H440 seems to fall on one straight line. The importance of the apparent discontinuity in the 2020 data is not yet clear but there is a discontinuity in the strain-to-failure vs burn-off plot for the same graphite (Figure 4). Both Board and Squires[1] and Rounthwaite et al.,[2] indicate that strain-to-failure for <u>tensile</u> fracture is approximately constant with respect to burn-off and this is true here for H440 but only up to ~28% burn-off for 2020 after which it increases rapidly.

A tentative model of the observed behavior might therefore include the following features

a) more rapid gasification of binder than filler
b) load-bearing properties mainly due to filler
c) total binder removal in 2020 at ~28% burn-off (n.b., this does not correspond to a 28% binder content)
d) loss of specimen ridgidity and a rapid strength decrease on removal of binder
d) a higher binder concentration in H440 (the weaker material) than in 2020.

References
1. J. A. Board and R. L. Squires, Proc. 2nd Conf. on Ind. Carbon and Graphite, Soc. for Chem. Ind., London, 1966, p. 289.
2. C. Rounthwaite, G. A. Lyons and R. A. Snowdon, ibid., p. 299.

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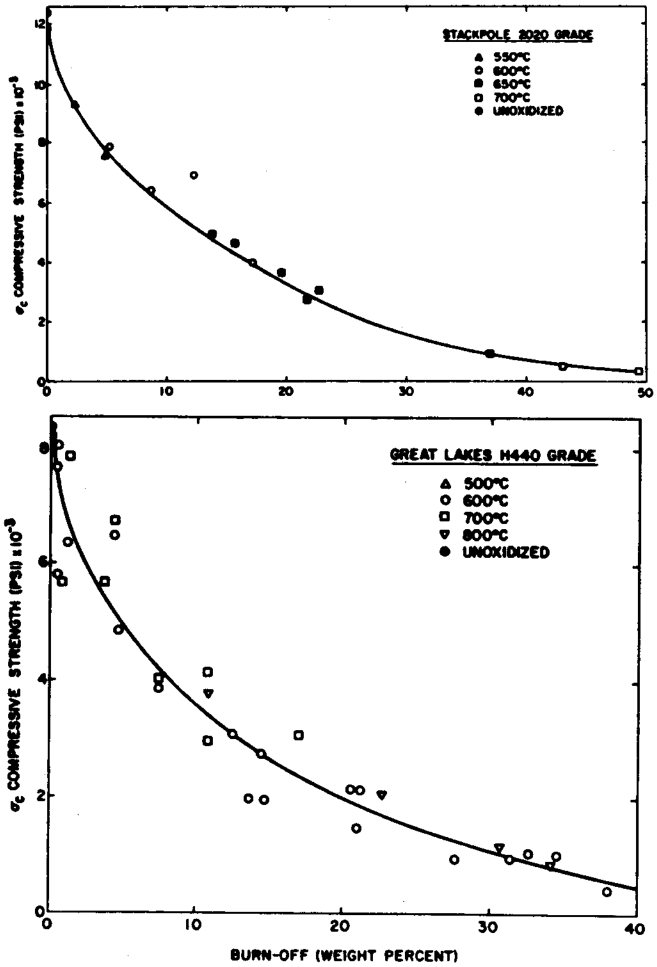
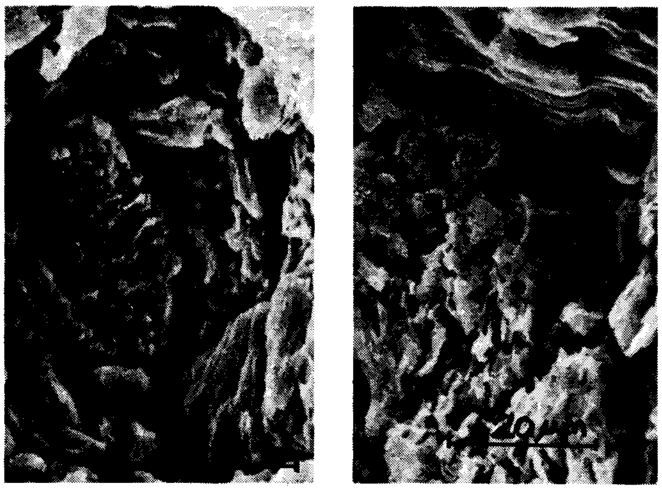


Fig. 1



a. Near edge

b. Near centre

Fig. 2

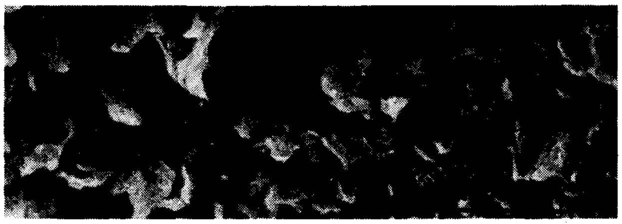


Fig. 2c

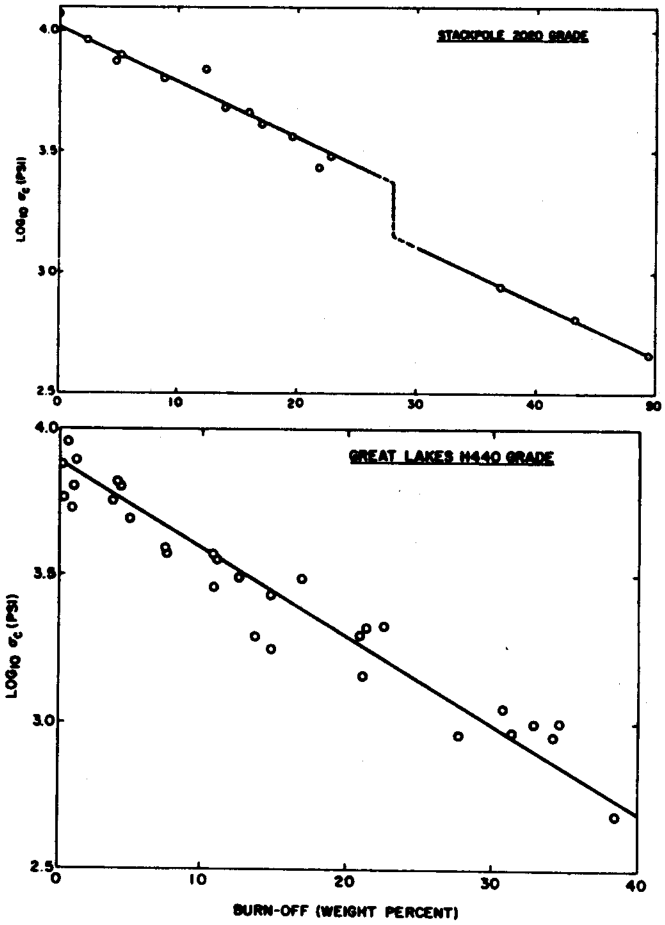


Fig. 3

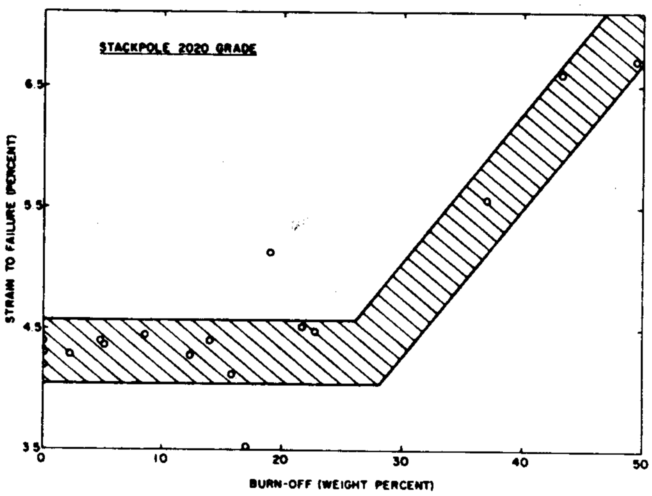


Fig. 4