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OXIDATION RESISTANCE OF SELECTED MECHANICAL CARBONS AT 650° C IN DRY FLOWING AIR

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SUMMARY

Experiments were conducted with experimental carbon-graphites to determine the effects of material variables on oxidation resistance. Specimens were heated to 650° C in dry (100 ppm H_2O) air flowing at 28 cubic centimeters per second standard temperature and pressure (STP).

The four material variables studied were: (1) oxidation inhibiting additive (B_4C), (2) pitch impregnation, (3) graphitization temperature, and (4) chemical treatment.

Oxidation resistance was definitely improved by addition of boron carbide to the 80 parts by weight (pbw) graphite-20 pbw carbon composition. Pitch impregnation had some beneficial effect on oxidation resistance and markedly improved flexure strength and hardness. Increasing the graphitization temperature from 2200° to 2800° C did not affect oxidation resistance except when B_4C and the higher graphitization temperature caused recrystallization of the graphite. This recrystallization improved oxidation resistance but greatly reduced hardness. Treatment with a phosphate solution increased the oxidation resistance of all the carbon-graphites.

INTRODUCTION

Advanced aircraft turbine engines will require dynamic shaft seals to operate in high temperature (e.g., 650° C) air. Some current engines use carbon element contact seals that operate at temperatures up to 480° C. Above this temperature, oxidation prevents the use of conventional mechanical carbons, and labyrinth seals, which can use materials of satisfactory oxidation resistance, must be used instead of carbon element contact seals. But labyrinth seals have the disadvantage of high leakage (about ten times the rate of conventional contact seals) and associated efficiency loss. Also the high air leakage flow of labyrinth seals at the engine bearing sump promotes high lubricant loss through the breather and increases sump fire potential. Thus there is a need for low leakage seals, and for these seals mechanical carbons of improved oxidation resistance will be required.

Mechanical carbons are attractive materials for contentional rubbing contact face seals because they have high strength-to-mass ratios, very high sublimation temperature, low elastic moduli (conformability to mating surfaces), and high strength at high temperatures. However, the high effective surface area and the permeability of carbon, as well as the volatility of its oxides (CO, CO_2), contribute to its poor oxidation resistance above 500° C.

For carbon-base materials to be effective at high temperature for extended periods of time, it is necessary to protect them from oxidation. The various treatments used to improve the oxidation resistance do so, at least partly, by reducing porosity and permeability. In other words, they effectively reduce the area of surface exposed to oxygen.

Another approach is to reduce the specific oxidation rate (oxidation loss per unit area of surface). The catalytic effect on carbon oxidation of many elements are described in reference 1. The elements are grouped according to relative strength of acceleration or inhibition of oxidation. Many of the impurities found in carbon accelerate oxidation (e.g., V, Fe, Pb, No). A few inhibit oxidation (e.g., B, P). This evaluation indicates that incorporation of certain additives into the basic material should be beneficial.

References 2 and 3 show the effects of boron compounds, particularly B_4C, on graphite oxidation. Over the range of boron percentages studied, B_4C showed increasing inhibition with increasing amounts added. B_4C appears to be a desirable additive to increase oxidation resistance of carbons at 650° C.

Most of the investigations of carbon oxidation reported are primarily concerned with the effects of additives. The development of an oxidation resistant mechanical carbon for seals also requires attention to hardness and strength. Also, as reference 1 indicates, factors such as operating temperature, types of carbon used, and method of preparation have a complex effect on carbon oxidation.

A 5 percent weight loss is usually considered the maximum loss compatible with unimpaired strength (ref. 4). Therefore a 5 percent loss is the basic criterion used in this study. However, the specimens used vary slightly in size so the results were normalized as explained in appendix A.

The objectives of this investigation were (1) to determine the oxidation lives (time to lose weight equivalent to 0.05 cu cm from 6.45 cm^2 of surface) of some experimental carbon-graphites and compare them with those of two commercial materials and (2) to determine the effect of an oxidation inhibiting additive (B_4C), pitch impregnation, graphitization temperature and chemical treatment (zinc phosphate) on the oxidation resistance of the experimental materials.

Studies were made at 650° C in air flowing at 28 cubic centimeters per second standard temperature and pressure (STP).

BACKGROUND

The manufacture of mechanical carbons is an involved process. The properties of the final product are affected by the ingredients used, the particle sizes of the solids (or particulates), processing steps and post processing steps (refs. 5 and 6).

In brief, mechanical carbon-graphites are produced by blending carbon and graphite powders with a binder (e.g., pitch, bakelite). This mixture is pressed (e.g., 3450 N/cm^2) to obtain a "green" compact sufficiently strong to endure subsequent handling. The compact is then baked to 1000° C to remove volatiles and carbonize the binder, forming a pyrolyzed compact. At this point the compact can be impregnated to decrease porosity and increase strength. Impregnation is accomplished by evacuating the compact, adding a molten material such as pitch and pressurizing. After impregnation, the compact is pyrolyzed again. If a second impregnation is desired, the aforementioned process is repeated. After pyrolyzation, the compact may be graphitized at a temperature up to 3000° C, vaporizing large amounts of impurities. The degree of graphitization is determined by time, temperature, and composition. Also, additives to improve oxidation resistance or other properties may be included.

Graphite is a completely crystallized form of carbon consisting of parallel layers of hexagonally arranged atoms. Nongraphitic carbons have a highly disordered structure. They have the basic layers of hexagonally arranged atoms but the layers are of relatively small dimensions and randomly oriented. The difference is primarily one of degree. Graphitization involves increasing the amount of ordered structure in the carbon body. The most commonly used nongraphitic carbon types are petroleum coke, gas black, and furnace black. Gas black and furnace black do not graphitize to any significant degree (ref. 6) unless catalyzed (e.g., by Al, Ti, B_4C).

Both graphite and nongraphitic carbon have beneficial as well as detrimental effects on the carbon-graphite body. Graphite is more oxidation resistant than nongraphitic carbon. However, nongraphitic carbon is stronger and much harder than graphite. For oxidation resistant mechanical carbons, this means a compromise must be made. The degree of graphitization should be as great as possible without resulting in a body that is too soft and low in strength for the application.

APPARATUS AND PROCEDURE

The specimens used were hemispherically tipped cylinders (fig. 1). The nominal hemisphere and cylinder radii were the same, 0.475 centimeter, and the nominal length was 1.9 centimeters. A hole was drilled near the base of the hemisphere to admit a Chromel wire hook.

The volume and apparent (or geometrical) surface area were calculated. The probable maximum error was less than 1.6 percent of volume and less than 0.25 percent of surface area. Before use, the specimens were submerged in n-heptane (ASTM reference fuel) and subjected to ultrasonic cleaning to remove loose material and contaminants. They were evacuated (20 to 30 torr) for at least 36 hours. High-purity nitrogen was used to repressurize the vacuum chamber. The specimens were weighed just before using them.

A schematic of the oxidation apparatus is shown in figure 1. Specimen weight was continuously measured with a recording microbalance. The specimen was heated by an induction heated susceptor located in a 5-centimeter-diameter pyrex tube. Specimen temperature was monitored with an infrared pyrometer. The pyrometer emissivity setting was 0.85, established by thermocouple measurement on a carbon-graphite specimen. This emissivity is in the range expected for a rough graphite surface (ref. 7) which was the condition of the cylindrical surfaces of the specimens. Varying the emissivity setting by 0.05 changes the indicated temperature by 7° C at 650° C. The pyrometer output was electrically coupled to a temperature controller which maintained the specimen temperature within an indicated maximum deviation of 10° C.

Specimens were suspended from one arm of the microbalance by a preoxidized Chromel wire which appeared to have no catalytic effect on the specimen. The test envelope was purged with high-purity nitrogen gas until the test temperature was reached or until a steady-state weight was indicated on the recorder. In a few cases a slight constant weight loss was accepted as inherent in that specimen. The nitrogen flow was then replaced by clean, dry (to about 100 ppm water) air flowing at 28 cubic centimeters per second (STP). The dry (room temperature) air was directed at the heated specimen. Preliminary observations indicated the major oxidation loss was occurring within the pores and, therefore, at approximately the measured specimen surface temperature. Those specimens for which the mean cylinder radius was remeasured after testing showed an apparent loss from the geometrical surface less than the uncertainty in determining the radius. Oxidation was continued until about 5 percent of the specimen weight was lost.

RESULTS AND DISCUSSION

The oxidation rate of seal ring carbon-graphite materials is dependent on many material and processing variables. This oxidation study concerns four material variables: (1) oxidation inhibiting additive (B_4C), (2) pitch impregnation, (3) graphitization temperature, and (4) chemical treatment (zinc phosphate). The experimental materials used in this study were produced under the following known and well-controlled conditions:

(1) Blend filler raw materials for 1 hour.

(2) Preheat filler and mixer to 150° C, add pitch, and mix filler and pitch binder in sigma blade mixer for 1 hour.

(3) Cool the mix and crush in hammer pulverizer.

(4) Micromill the crushed mix.

(5) Blend milled material for 1 hour.

(6) Mold the milled material at 100° C using a hydraulic press, forming the green compact.

(7) Pack green compact with coke in a sagger, cover with charcoal, and bake. Temperature is programmed to increase 5° C per hour up to 1000° C. Compact is baked at 1000° C for 4 hours.

(8) Compact is packed in graphite particles and graphitized. Temperature program is: room temperature to 900° C at 400° C per hour, 900° to 1600° C at 200° C per hour and 1600° C to either 2200° or 2800° C at 300° C per hour then hold at final temperature for 1 hour.

The basic composition selected contained 80 percent artificial graphite and 20 percent nongraphitic carbon, was graphitized at 2800° C and was designated E-0-28. Designations are explained in the footnote of table I. Compositions and some properties are listed in table I.

For a basis of oxidation life comparison, a widely used commercial carbon-graphite seal material and a high-temperature carbon-base material were included in the study. Some properties of the two are given in table I; however, no details of manufacture are available.

The values presented are based on the results from at least two specimens of each material.

Effect of Adding Boron Carbide

Boron carbide (B_4C) powder was added to the composition of the base material as an inhibitor. The oxidation lives for various amounts of B_4C (0, 5, 7.5, 10 parts by weight (pbw) added to 100 pbw of other particulates) are given in figure 2. The materials containing B_4C showed a varying weight gain effect (see appendix B) - probably a result of oxidation of B_4C to boric oxide (B_2O_3).

Material E-5-22 showed four times the oxidation life of the basic E-0-28 and material E-7.5-22 showed about seven times the oxidation life of E-0-28. However, E-10-22 showed no improvement over E-7.5-22. This indicates that with the basic material E-0-28, addition of B_4C ceases to improve oxidation life somewhere around 7.5 pbw. Material E-10-22 had lower strength and hardness and therefore no further work was done on 10 pbw B_4C materials.

Effect of Pitch Impregnation

Oxidation rate is a function of the porosity of carbon-graphite material because porosity determines the surface area exposed to oxidizing media. Typically the experimental materials under study had pore surface areas in the range of 2 square meters per gram. Reducing this pore surface area should increase the oxidation resistance and improve strength. With this in mind the experimental material with 5 and 7.5 pbw boron carbide (E-5-22 and E-7.5-22) were subjected to a pitch impregnation process. In this process a coal tar pitch (Barrett no. 30 medium hard) was forced under pressure (70 N/cm^2) into the pores of compacts of these materials that had received only a 1000° C bake. This impregnated material was then rebaked at 1000° C followed by graphitization at 2200° C.

The effect of this impregnation can be seen in figure 3 which shows a comparison of the E-5-22 and E-7.5-22 (nonimpregnated materials) with EP-5-22 and EP-7.5-22 (impregnated materials). In the case of the 5 pbw B_4C, the impregnated material (EP-5-22) had twice the oxidation life of the unimpregnated material (E-5-22) - a definite improvement. For the 7.5 pbw materials, there is just over 50 percent increase in life. In addition, the flexure strengths of EP-5-22 and EP-7.5-22 were significantly better than those of the unimpregnated materials (table I).

Effect of Graphitization Temperature

Increasing the graphitization temperature of a carbon-graphite can improve oxidation life by removing higher boiling point impurities and by increasing the graphite content. Some additives, such as B_4C, can further increase the degree of graphitization by catalysis and simultaneously cause recrystallization of the graphite already present.

It can be seen in figure 4 that the higher graphitization temperature of EP-5-28 did not change the oxidation life from that of EP-5-22. In comparison, EP-7.5-28 had three times the oxidation life of EP-7.5-22. Examination of EP-7.5-28 revealed considerable recrystallization.

It is important to point out that although the oxidation life of EP-7.5-28 is greater than that of EP-7.5-22, the mechanical properties were degraded (table I). Flexure strength was reduced by 29 percent and hardness by 79 percent.

Effect of an Oxidation Inhibitor

The effect of treatment with an oxidation inhibitor on oxidation resistance of the experimental materials was investigated. Zinc phosphates have been shown to inhibit oxidation of carbon-graphites (ref. 7). Details of the treatment are as follows:

(1) Carbon specimens are placed in a closed stainless-steel container, evacuated to 15 to 20 torr, then heated to 800° C and held at temperature for 60 minutes.

(2) The container, still evacuated, was allowed to cool to room temperature.

(3) The container was backfilled (by suction) with a 25 percent aqueous solution of zinc phosphate. The solution included enough phosphoric acid to give a zinc oxide to phosphorous pentoxide ratio of 1 to 3.

(4) The container was pressurized to 35 Newtons per square centimeter with nitrogen and held for 60 minutes.

(5) After depressurization, the specimens were removed and oven dried for about 24 hours at 175° C.

The effect of the treatment on oxidation lives is presented in figure 5. The basic experimental material (E-0-28) and the four impregnated materials (EP-5-22, EP-7.5-22, EP-5-28 and EP-7.5-28) were treated. The effect of treatment on specimens of the commercial seal carbon (untreated) was observed for comparison.

The four pitch impregnated materials can be grouped together as showing about a doubling of oxidation life due to treatment. The differences in the relative increases are probably not significant. In contrast, the treated basic E-0-28 showed 17 times the oxidation life of the untreated material. This is probably a result of the greater porosity of E-0-28 which had more than twice the pickup (i.e., the increased weight due to, in this case, the phosphate treatment) of the other four experimental materials.

Increased inhibitor pickup can increase life in one or both of two ways. First, a greater amount of phosphate would be expected to last longer. The effectiveness of phosphate decreases with time (ref. 9) probably due to evaporation of P_2O_5 or H_3PO_4. Second, the treatment solution will only penetrate pores larger than a critical diameter. This diameter depends on the surface energies of the carbon and the solution. Since the pitch impregnation reduces pore diameter, the impregnated materials can be expected to have a larger proportion of surface not covered by zinc phosphate.

Comparison of Experimental and Commercial Carbon-Graphites

The two carbon-graphite materials found to have the longest oxidation lives in this study (i.e., EP-5-28-Zn and EP-7.5-28-Zn) are compared with two commercial mechanical carbons in figure 6. Both experimental materials outlasted the seal carbon but only EP-7.5-28-Zn outlasted the commercial high-temperature mechanical carbon. However, the commercial mechanical carbon had at least twice the coefficient of friction of any of the other three materials (at 650° C, 1.33-m/sec sliding speed, 500-gram load against Inconel-X). This may be detrimental in some seal applications.

These two commercial carbons were selected from an unpublished oxidation study of fifteen commercial mechanical carbons recommended by the manufacturers for high-temperature applications. The two selected were (1) the one with the longest oxidation life and (2) the one most commonly used.

SUMMARY OF RESULTS

Studies of the oxidation lives of several carbon-graphite materials at 650° C in flowing dry air reveal the following:

1. Addition of boron carbide (B_4C) definitely increased the oxidation resistance of the basic carbon-graphite composition. Adding 5 pbw B_4C to 100 pbw of other particulates increased resistance four times.

2. Pitch impregnation of carbon-graphite containing B_4C slightly ($1\frac{1}{2}$ or more times) improved the oxidation resistance but significantly improved flexure strength and hardness.

3. Oxidation resistance was improved by increased graphitization temperatures (2200° to 2800° C) when the boron carbide caused recrystallization of the graphite. However, the hardness was greatly decreased (low hardness is undesirable for seal applications).

4. The oxidation resistances of the carbon-graphites were improved by treatment with a zinc phosphate solution (about 2 times for B_4C-containing materials).

Lewis Research Center,
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501-24.

APPENDIX A

NORMALIZATION OF RESULTS

A 5 percent weight loss is generally considered to be the maximum that will not significantly impair the bulk strength of mechanical carbons. However, the time required to lose 5 weight percent is dependent on the size and effective surface area of the specimen. The effective surface area is the area which will give the observed rate of loss when multiplied by the rate per unit area at the geometric surface. It is a function of the porosity and permeability and is ideally proportional to the apparent (geometric) surface for a homogeneous material. The carbon-graphite specimens tested in these experiments varied slightly in size. Therefore they were normalized to a selected volume and apparent surface area in order to compare oxidation rates. The selected volume (1.00 cu cm) and area (6.45 cm^2) were within the size range of the specimens used. For a given specimen, the time required to lose weight equivalent to 0.05 cubic centimeter was determined. This time was multiplied by the ratio

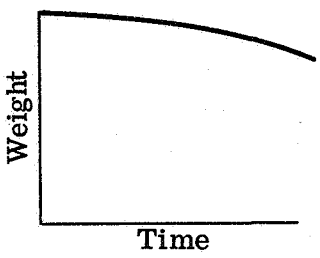
$$\frac{\text{apparent surface area}}{6.45}$$

APPENDIX B

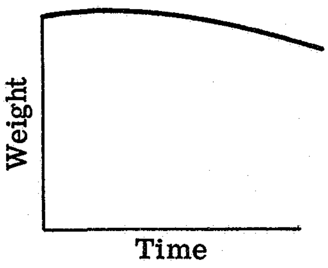
WEIGHT GAIN BEHAVIOR

Examination of the weight against time recordings indicated that the usual behavior (to beyond 5 percent loss) is that illustrated in sketch (a). Most materials (including a number of unreported commercial materials) showed an increasing rate of loss after the first few minutes. This includes materials with and without treatments (e.g., anti-oxidant).

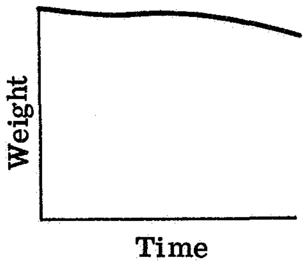
However the boron carbide containing materials often showed weight gains as illustrated in sketches (b), (c), and (d). These gains are probably due to oxidation of boron carbide (to boric oxide). They may occur immediately (sketch (b)) or be delayed (sketch (c)). Often the gain was noticeable only by deviation from the usual curve (sketch (d)).



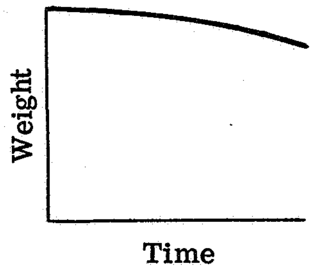
(a)



(b)



(c)



(d)

This weight gain behavior indicates nonuniform distribution of boron carbide throughout the compacts (see the section BACKGROUND) out of which the specimens are machined. Evidently some specimens have a fairly uniform distribution and show no noticeable weight gain (i.e., the rate of gain is less than the rate of loss due to oxidation of carbon). When concentrations of boron carbide exist, the time before gain is noticeable is an indication of distance from the surface and the amount of gain is an indication of the degree of concentration.

There appears to be no correlation between amount of boron carbide and degree of inhomogeneity. Four specimens of E-5-22 (5 pbw B_4C) showed the usual type of behavior as did two out of four specimens of E-10-22 (10 pbw B_4C). However the intermediate (7.5 pbw B_4C) material, E-7.5-22, showed gains for five specimens.

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TABLE I. - PROPERTIES OF MATERIALS USED IN THIS INVESTIGATION

[Experimental materials contain 30 pph of particulates (consisting of 80 pbw CHP graphite, 20 pbw Thermax and any B_4C added) plus 70 pph no. 30 medium hard pitch as binder molded at 3.45×10^6 N/m^2 (5000 psi) and 100° C.]

Material (a)	Average flexure strength[b]		Average Young's modulus[b]		Average hardness,[b] Rockwell S	Range of approximate density,[c] g/cu cm
	N/m^2	psi	N/m^2	psi		
E-0-28	2.63×10^6	3810	1.20×10^9	1.74×10^6	106	1.82 to 1.84
E-5-22	2.46	3570	1.34	1.95	98	1.74 to 1.77
E-7.5-22	2.18	3160	1.24	1.80	83	1.71 to 1.73+
E-10-22	1.83	2660	1.13	1.64	65	1.68
EP-5-22	3.83	5560	1.68	2.44	112	1.85 to 1.86
EP-7.5-22	4.13	5990	1.77	2.5	109	1.85
EP-5-28	4.33	6430	1.78	2.58	96	1.84
EP-7.5-28	2.94	4270	1.72	2.49	23	1.80
Commercial seal carbon	6.1	8800	2.6	3.7	---	1.75
High-temperature mechanical carbon	3.8	5500	1.2	1.8	---	1.87

[a] E denotes experimental material; P denotes pitch impregnated; 0, 5, 7.5, or 10 denote parts by weight of B_4C added to particulates; and 22 or 28 denote 2200° or 2800° C graphitization temperature.

[b] Properties of experimental materials from Ref. 8; those of commercial materials from manufacturer's literature.

[c] Densities are those of specimens used in this study (obtained in process of normalizing, see appendix A).

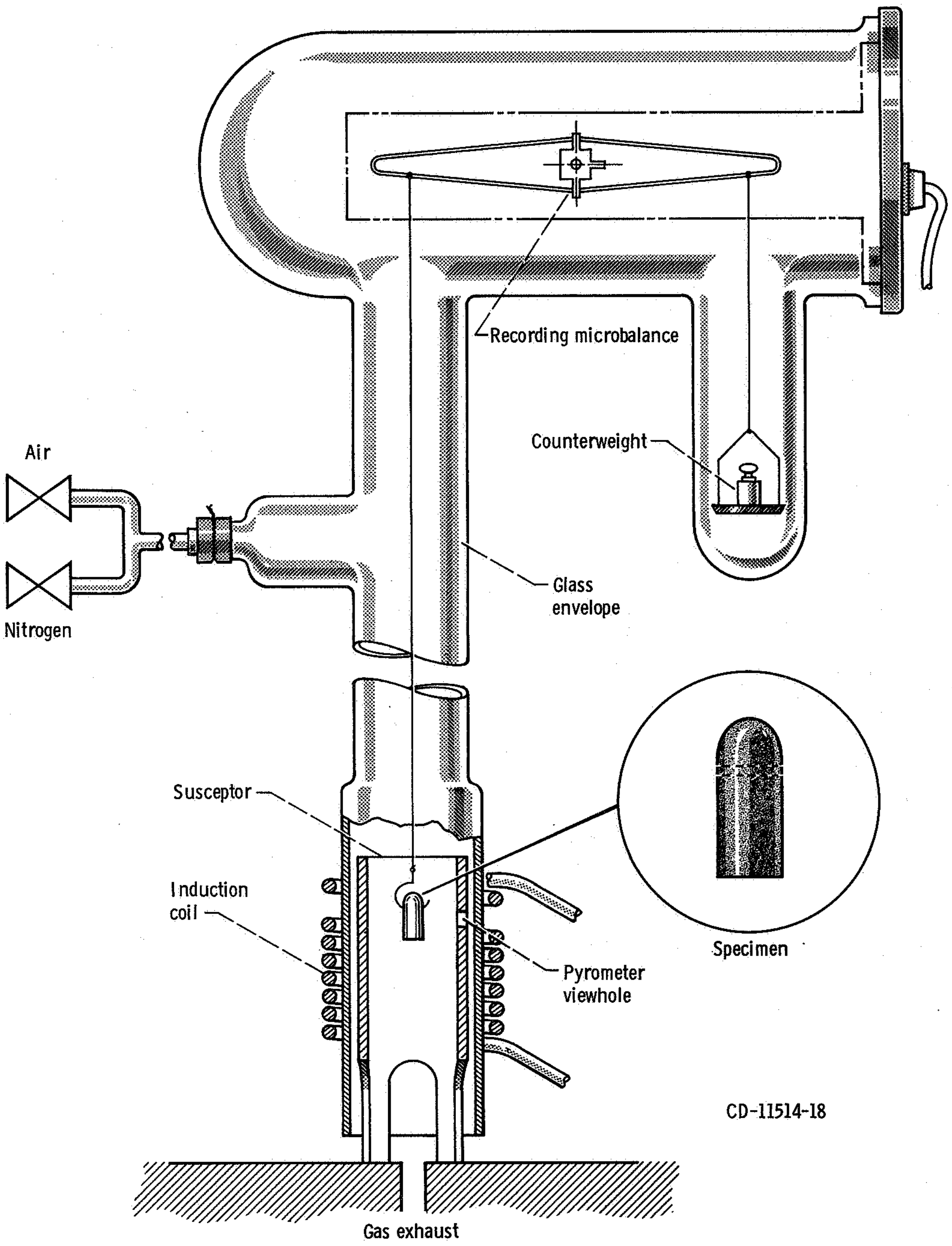

Figure 1. - Schematic of oxidation apparatus.

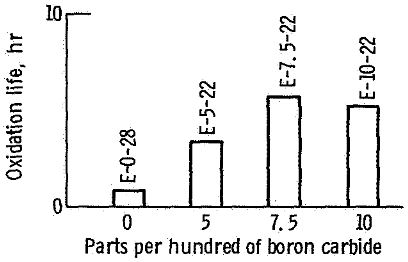

Figure 2. - Effect of addition of boron carbide on oxidation life of experimental carbons at 650° C in dry air flowing at 28 cubic centimeters per minute (STP).

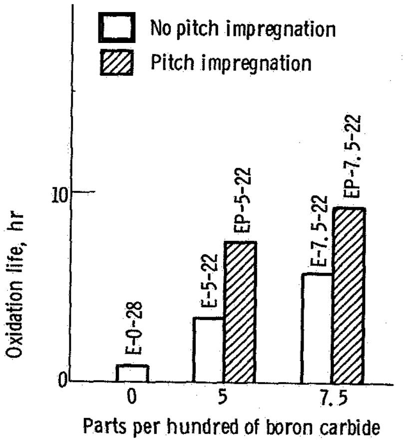

Figure 3. - Effect of pitch impregnation on oxidation life of experimental carbons at 650° C in dry air flowing at 28 cubic centimeters per minute (STP).

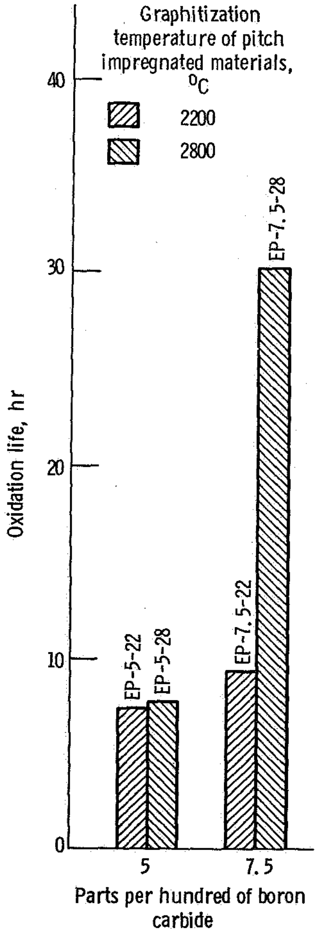

Figure 4. - Effect of graphitization temperature on oxidation life of experimental carbons at 650° C in dry air flowing at 28 cubic centimeters per minute (STP).

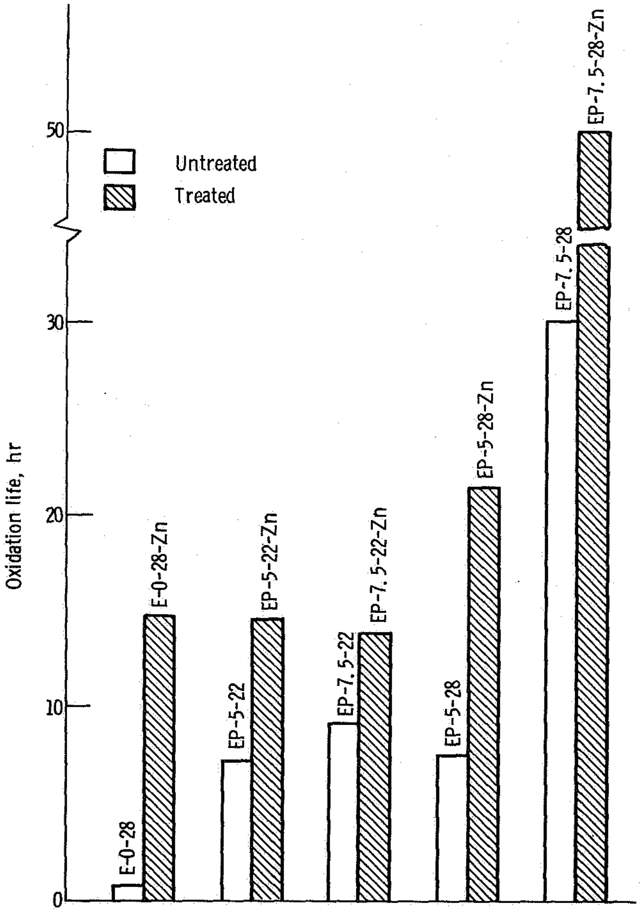

Figure 5. - Effect of zinc phosphate treatment on oxidation life of experimental carbons at 650° C in dry air flowing at 28 cubic centimeters per minute (STP).

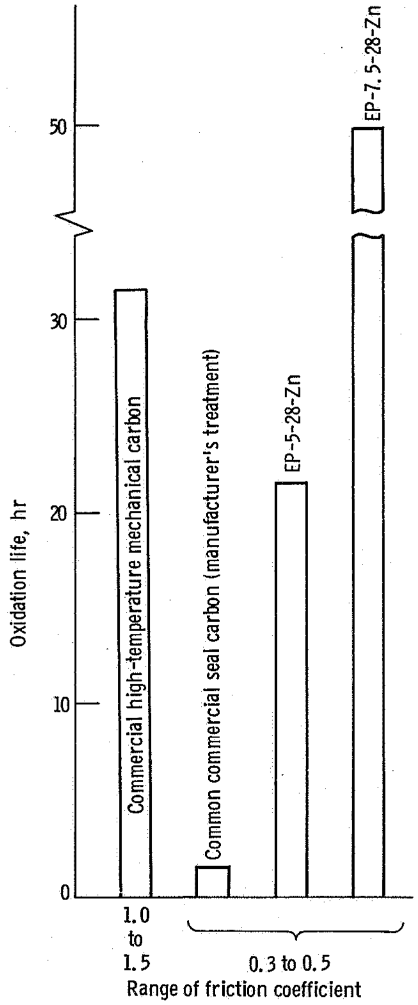

Figure 6. - Comparison of oxidation life of best experimental carbons to two commercial carbons. (Zn indicates treated materials.)

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16. Abstract

Oxidation experiments were conducted with several experimental mechanical carbons at 650° C in air flowing at 28 cu cm/sec (STP). Experiments indicate that boron carbide addition and zinc phosphate treatment definitely improved oxidation resistance. Impregnation with coal tar pitch before final graphitization had some beneficial effect on oxidation resistance and it markedly improved flexure strength and hardness. Graphitization temperature alone did not affect oxidation resistance, but with enough added boron carbide the oxidation resistance was increased although the hardness greatly decreased.

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