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16. Abstract
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SUMMARY

Studies were conducted to determine the friction and wear properties of experimental oxidation-resistant carbon-graphites. The effects of boron carbide, phosphate treatment, and purification (with chlorine) were investigated. Hemispherically tipped carbon-graphite riders were run in sliding contact with rotating Inconel X-750 disks in air. A surface speed of 1.33 meters per second, a load of 500 grams, and a specimen temperature of 650° C were used. The test duration was 30 or 60 minutes, depending on relative oxidation resistance.

Results of this study indicate that hardness does not have a major effect on friction and wear under the conditions of these studies. Two materials of greatly different hardness, when phosphate treated and when phosphate treated and purified, showed about the same wear rates. Wear rates and friction similar to those of a commonly used carbon-graphite seal material were attained with some of the experimental materials studied. The inclusion of boron carbide (an oxidation inhibitor) had a strong influence on wear rate; whether the effect was beneficial or detrimental depended on boron carbide (B_4C) content and processing. The inclusion of B_4C also reduced the friction coefficient below that obtained for the nonboronated composition. Phosphate treatment reduced the friction coefficient only when B_4C was not present in the base material. Nonboronated materials showed the friction reduction when phosphate treated whether purified or not, but boronated materials did so only when purification had removed B_4C.

INTRODUCTION

Advanced aircraft turbine engines require dynamic shaft seals to operate in air at temperatures up to 650° C. Mechanical carbons have advantages over other seal materials at high temperatures: high strength to mass ratios, high sublimation temperature

(>3500° C), low elastic moduli (conformability to mating surfaces), and ability to run in sliding contact without galling. However, use of these carbons at high temperatures in air is limited by relatively poor oxidation resistance. Previous studies (refs. 1 and 2) have shown that the oxidation resistance of carbon-graphites is improved by inclusion of boron carbide (B_4C), by purification with chlorine, and by treatment with a solution of zinc phosphate and phosphoric acid.

In addition to oxidation resistance, friction and wear behavior must be considered in the use of carbon-graphites for rubbing-contact seals at high temperature. Therefore, the effects of B_4C, purification, and phosphate treatment on friction and wear should be determined. Significant effects may occur because of, for example, altered microstructure (B_4C), changed porosity (purification), or formation of transfer films on mating surfaces (phosphate treatment).

The objectives of this experimental study were to determine (1) the friction and wear properties of experimental carbon-graphites developed for high temperatures and (2) the effects of B_4C, purification, and phosphate treatment on friction and wear.. Experiments were conducted with a hemispherically tipped carbon-graphite rider in sliding contact with a rotating Inconel X-750 disk in clean, dry air (<100 ppm water) at 650° C. The load was 500 grams, and the sliding velocity was 1.33 meters per second. The duration of runs was 30 or 60 minutes.

APPARATUS AND PROCEDURE

The apparatus used is shown in figure 1. The basic elements were a rotating disk (diam, 6.35 cm) and a hemispherically tipped carbon-graphite rider specimen (diam, 0.95 cm) in sliding contact. The disk was rotated by an electric motor operating through a spindle-mounted shaft. The rider specimen was loaded against the rotating disk by a deadweight system, the force of which was transmitted through a shaft vertically mounted in porous-metal gas-lubricated journal bearings. The specimens were mounted in the test chamber, and the atmosphere was supplied by air flowing into the bottom of the chamber as well as through the gas bearings. The specimen was heated by an induction-heated susceptor.

The friction force was measured by a dynamometer ring (with strain gages) attached as shown in figure 1, and the strain-gage output was transmitted to a strip-chart recorder. Rotational speed was measured by a magnetic pickup connected to an event counter. Rider temperature was controlled with an infrared pyrometer which was sighted through a hole in the chamber wall onto the rider specimen just above the rider-disk contact area.

Disk specimens were lapped, ultrasonically cleaned in heptane (ASTM reference fuel), brushed with a stiff bristle brush, polished on 4/0 emery paper, and then scrubbed with levigated alumina paste. The specimens were given a final scrubbing with levigated alumina and rinsed in tap water and distilled water just before use. The disk surface finish was 0.1 micrometer root mean square.

Riders were ultrasonically cleaned in heptane and evacuated to a container pressure of 10 to 20 torr for at least 36 hours before use. The container was repressurized with high-purity nitrogen ($<$100 ppm water).

Specimens were mounted in the test chamber, which was closed and purged with nigrogen during heating to avoid oxidation before the run. The disk was rotated and heated until the specimen temperature stabilized at 600° C. (The 50° C difference between this value and the test temperature allowed for the temperature increase due to frictional heating.) After reaching 600° C, the nitrogen was replaced by air ($<$100 ppm water), and the chamber was purged for 5 minutes. The rider was then loaded against the rotating disk.

MATERIALS

Results for six experimental carbon-graphites are presented in this report. Results for a seal material (a mechanical carbon-graphite) commonly used in present aircraft engines are included for comparison; this material is designated material A. Some properties of the commercial and experimental materials are presented in table I.

The designation E indicates the experimental material contained 30 parts per hundred (pph) particulates (consisting of 80 parts by weight (pbw) CHP graphite, 20 pbw Thermax, and any B_4C added) and 70 pph number 30 medium hard pitch (ref. 1). The designation X indicates the material contained 40 pph particulates (consisting of 80 pbw CHP graphite and 20 pbw Thermax) and 60 pph pitch (ref. 2). Pitch and Bakelite resin impregnated materials are designated P and B, respectively. The amount of B_4C added to the other particulates, 0, 5, or 7.5 pbw, is indicated next. Then the graphitization temperature (2200° or 2800° C) is indicated as 22 or 28. For purified materials, the designation Cl (chlorine) is used. Finally, the designation for the phosphate treatment is Zn (zinc phosphate). The use of these designations is illustrated in the following example:

For the material EP-5-28-Cl-Zn

E	30 pph particulates and 70 pph pitch
P	pitch impregnation of carbonized compact (before graphitization)
5	5 pbw B_4C included in particulates
28	2800° C graphitization temperature
Cl	chlorine purification
Zn	phosphate treatment

Processing of Experimental Materials

The following steps outline the processing of these materials. Except as noted, the steps were identical for all six experimental materials.

(1) The particulates except B_4C were blended.

(2) The particulates and the mixer were heated; pitch (binder) was added and mixed with the particulates.

(3) The mixture was cooled.

(a) The X mixture was cooled and crushed.

(b) The E mixture was pelletized and cooled.

(4) The mixture was micromilled.

(5) The mixture was blended.

(a) For nonboronated materials, the milled mixture was blended.

(b) For boronated materials, equal weights of B_4C and the basic E mixture were preblended; the E mixture required to obtain the desired B_4C content was added and blended; the final mixture was screened and reblended.

(6) The blended material was molded at room temperature (X mixtures) or 150° C (E mixtures).

(7) Green compacts were packed with coke, covered with charcoal, and heated to 1000° C.

(a) The X compacts were heated 5° C per hour to 1000° C and held for 4 hours.

(b) The E compacts were heated quickly to 50° C, heated 2.5° C per hour to 500° C, heated 5° C per hour to 1000° C, and held for 4 hours.

(8) The compacts were impregnated with pitch or Bakelite resin and given a second carbonization (except E-0-28).

(a) The E compacts were impregnated with pitch at 70 newtons per square centimeter and carbonized as in step 7(b).

(b) The X compacts were evacuated, impregnated with a 50-percent-acetone - 50-percent-Bakelite resin at 57 newtons per square centimeter, heated while under a pressure of 69 newtons per square centimeter, and finish-cured at ambient pressure. The subsequent carbonization schedule was as follows: the compacts were heated quickly to 150° C, heated 10° C per hour to 1000° C, and held for 4 hours.

(9) The carbonized compacts were packed with graphite and heated to the graphitization temperature indicated for each material (2200° or 2800° C). The heating schedule was the same for all.

More details on the processing of material XB-0-28 can be found in reference 3 and on the processing of the E materials in reference 4.

Purification

The purification was a commercial process of exposure to chlorine at 2800° C. During this treatment, impurities were converted to chlorides and evaporated.

Antioxidant Treatment

The antioxidant (phosphate) treatment was as follows:

(1) Carbon specimens were placed in a closed stainless-steel container, which was evacuated to 15 to 20 torr, and 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.

RESULTS AND DISCUSSION

The experimental materials studied were carbon-graphites developed to have good mechanical properties (flexure strength and hardness) for contact seals and improved oxidation resistance. A commonly used high-temperature seal carbon was included for comparison.

Figures 2 to 6 show the highest and lowest wear rates (total wear volume/time) observed for each basic (as manufactured) or modified (purified and/or phosphate treated) material (two or three specimens of each). Duration of runs was either 30 or 60 minutes.

The effects of B_4C additions on friction and wear are shown in figure 2 for five basic materials. Figure 2(a) indicates that the amount of B_4C added to the E-0-28 composition affected the wear rate. (See the section MATERIALS for designations.) For the 2200° C (graphitization temperature) materials, 7.5 pbw B_4C resulted in a slightly greater wear rate than did 5 pbw B_4C. However, for the 2800° C materials, 7.5 pbw B_4C (EP-7.5-28) resulted in a much greater wear rate than 5 pbw B_4C. The high wear rate of EP-7.5-28 can be attributed to the low hardness (Rockwell S-23) that is related to its highly graphitic nature. This highly graphitic nature is a result of extensive recrystallization (ref. 4) catalyzed by B_4C. (The degree of recrystallization by the B_4C is also influenced by the time and temperature of graphitization.) It should be noted that, with 5 pbw B_4C, EP-5-28 did not undergo such extensive recrystallization (ref. 4), although it showed more recrystallization than the other three materials (E-0-28, EP-5-22, and EP-7.5-22). Of the three 2800° C materials, EP-5-28 had the lowest wear rate.

The results in figure 2(a) suggest that there is an optimum combination of graphitization temperature and B_4C content that will give the lowest wear rate. The 2450° C melting point of B_4C (ref. 5) is thought to be a practical lower limit for this optimum. This conclusion is based on the results for EP-5-22, in which there was no melting of B_4C during graphitization; EP-5-22 had a less graphitic nature and a higher wear rate than EP-5-28. Therefore, it seems that graphitization above 2450° C results in molten B_4C that can spread and come into more extensive contact with the carbon.

Figure 2(b) shows that the presence of B_4C in the basic materials decreases the friction coefficient from the unacceptable levels of E-0-28 to levels that can be accepted (e.g., for aircraft seals). The lower friction coefficient was likely due to the presence of molten boric oxide (B_20_3) (mp, 460° C; ref. 5) resulting from B_4C oxidation, as noted in reference 1. The two 5-pbw-B_4C materials showed a desirable reduction in the friction coefficient, which indicates that optimizing the degree of graphitization could improve friction as well as reduce wear.

Figure 3(a) presents the effects of purification (with chlorine) or treatment with a solution of zinc phosphate and phosphoric acid or both on the wear of a nonboronated (E-0-28) and a boronated (EP-7.5-22) composition. For E-0-28 there was no appreciable effect on wear rate due to either of the single treatments or to the combination treatment. All three treatments reduce the wear rate of EP-7.5-22.

Figure 3(b) shows that the friction coefficient of E-0-28 is significantly reduced by phosphate treatment. However, EP-7.5-22 (and the other three boronated materials in fig. 2) showed a reduction of friction only when purified and then phosphate treated. Reference 2 indicates that purification removes B_4C. These results indicate that the friction coefficient is higher without the purification because the presence of B_4C and the consequent loss of the B_4C (on its surface of the specimen) by chlorination result in a material with friction properties similar to those of the unboronated E-0-28. (This is to be expected since the boronated materials differ from E-0-28 only by the addition of B_4C.) Loss of B_4C would also increase the surface porosity and therefore increase the takeup of the phosphate solution during treatment. Therefore, the conclusion must be that phosphate treatment reduces friction in the absence of B_4C. Reduction of friction is a desirable goal as heat generation in a contact seal is a function of the friction level.

Figure 4 shows the results obtained with another nonboronated experimental material (XB-0-28). This carbon-graphite showed reduction in wear rate and a large decrease in friction when phosphate treated. A comparison with the results for E-0-28 in figures 3(a) and (b) shows that, although both materials had essentially identical wear rate and friction coefficient when untreated, they were significantly different in wear when phosphate-treated.

In figure 5 the wear rates of the basic and modified EP-7.5-28 materials are presented. A comparison with figure 3(a) shows that the wear rates for all the modified materials fell in the range between 1 and 4×10^{-7} cubic centimeter per meter. Hardnesses of the E-0-28 and EP-7.5-22 materials are essentially the same, while the EP-7.5-28 material was much softer (table I). Apparently phosphate treatment negated the effect of softness on EP-7.5-28. Also, figure 2(a) shows that EP-5-28, which was marginally softer than E-0-28, EP-5-22, and EP-7.5-22, had only a fraction of the wear of E-0-28. Evidently, hardness did not have a major influence on friction coefficient and wear under the conditions of this experiment. However, it must be remembered that in this study the only source of abrasion was the small amounts of wear debris from the carbon rider specimens.

Figure 6 shows that several of the experimental materials had friction and wear comparable to those of the commercial seal carbon used as a reference. The nonboronated materials had friction coefficients that were nearly the same as that of the commercial material. The boronated materials had higher friction coefficients (0.4 or more) than the commercial material but would be superior where the combination of low wear rate and high oxidation resistance outweighs the higher friction.

SUMMARY OF RESULTS

The following results were obtained from a study of wear and friction of oxidation-resistant mechanical carbon-graphites at 650° C in air:

1. The inclusion of boron carbide (as an oxidation inhibitor) decreased the friction coefficient and strongly influenced wear rate. The influence on wear rate was a result of graphitization temperature and boron carbide content. An optimum combination of graphitization temperature and boron carbide content might produce even lower wear than observed in these studies.

2. Treatment with zinc phosphate (an oxidation inhibitor) had, in general, little effect on wear rate of the carbon graphites. However, it reduced the friction coefficient of purified and unpurified nonboronated materials and of purified boronated materials.

3. Friction and wear as low as or lower than those of a good commercial seal material were shown by four experimental materials.

4. Hardness did not have a major influence on friction and wear under the conditions of these studies.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 23, 1974,
501-24.

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5. Hodgman, Charles D., ed.: Handbook of Chemistry and Physics. Chemical Rubber Publishing Co., 1963.

TABLE I. - PROPERTIES OF MATERIALS USED IN THIS INVESTIGATION

Material (a)	Average flexure strength (b)		Average Young's modulus (b)		Average hardness, Rockwell-		Nominal density, g/cm^3 (b)	Average oxidation life of basic compositions, hr (c)
	N/m^2	psi	N/m^2	psi	S- (b)	15T-		
A	8.3×10^7	12×10^3	2.1×10^{10}	3.0×10^6	---	87	1.80	----
XB-0-28	3.09	4.48	1.01	1.47	85	--	1.73	1.2
E-0-28	2.63	3.81	1.20	1.74	106	--	1.83	.9
EP-5-22	3.83	5.56	1.68	2.44	112	--	1.86	7.5
EP-7.5-22	4.13	5.99	1.77	2.5	109	50	1.85	9.5
EP-5-28	4.33	6.43	1.78	2.58	96	15	1.84	7.7
EP-7.5-28	2.94	4.27	1.72	2.49	23	--	1.80	30.3

[a] Designations: A, commercial seal material; E or X, 30 or 40 pph, respectively, of particulates (80 pbw CHP graphite, 20 pbw Thermax, and any B_4C added) plus 70 or 60 pph, respectively, of number 30 medium hard pitch as binder; P, pitch impregnation; B, Bakelite resin impregnation; 0, 5, or 7.5, amount of B_4C (in pbw) added to particulates; 22 or 28, graphitization temperature of 2200° or 2800° C.

[b] Value for XB-0-28 from ref. 3; values for other materials from ref. 4.

[c] Value for XB-0-28 from ref. 2; values for other materials from ref. 1.

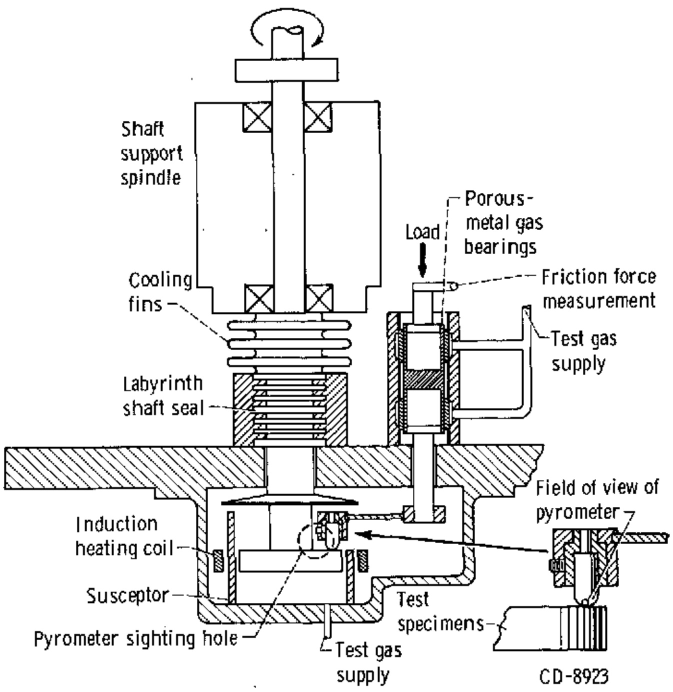
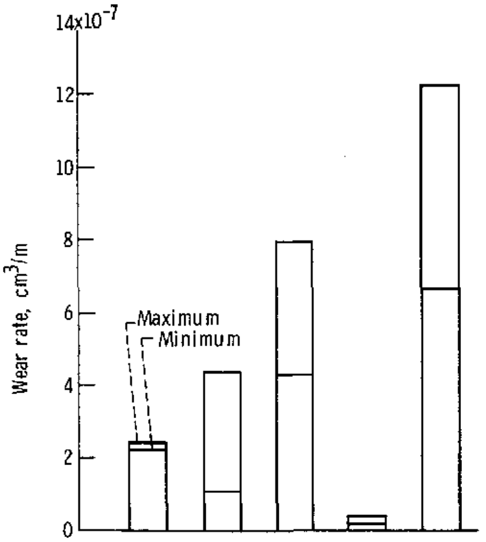
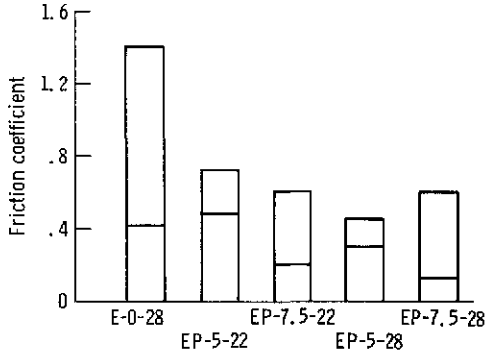


Figure 1. - Friction and wear test apparatus.

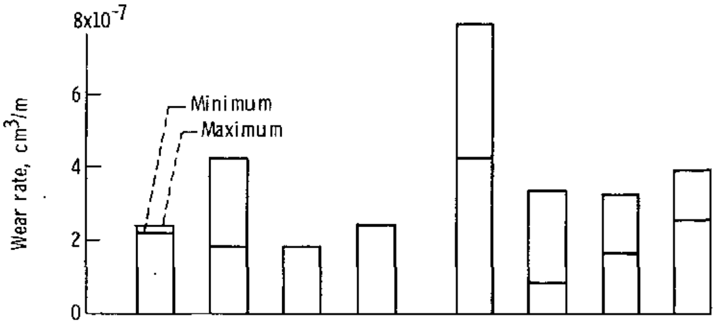


(a) Wear rate.

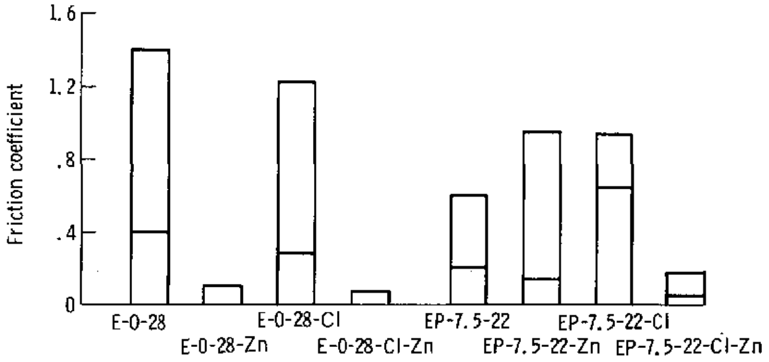


(b) Friction coefficient.

Figure 2. - Effect of boron carbide content and graphitization temperature on experimental carbon-graphites sliding against Inconel X-750. Load, 500 grams; sliding velocity, 1.33 meters per second; rider temperature, 650° C; test duration, 30 or 60 minutes.

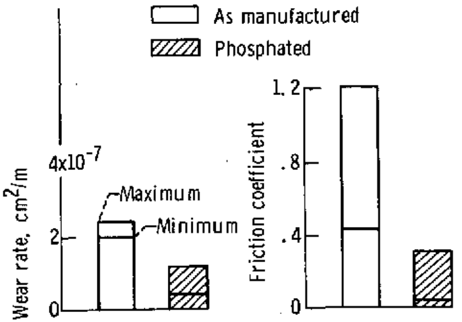


(a) Wear rate.



(b) Friction coefficient.

Figure 3. - Effect of purification and of antioxidant treatments on experimental carbon-graphites E-0-28 and EP-7.5-22 sliding on Inconel X-750. Load, 500 grams; sliding velocity, 1.33 meters per second; rider temperature, 650° C; test duration, 30 or 60 minutes.



(a) Wear rate.

(b) Friction coefficient.

Figure 4. - Effect of antioxidant treatment on experimental carbon-graphite XB-0-28 sliding on Inconel X-750. Load, 500 grams; sliding velocity, 1.33 meters per second; rider temperature, 650° C; test duration, 30 minutes.

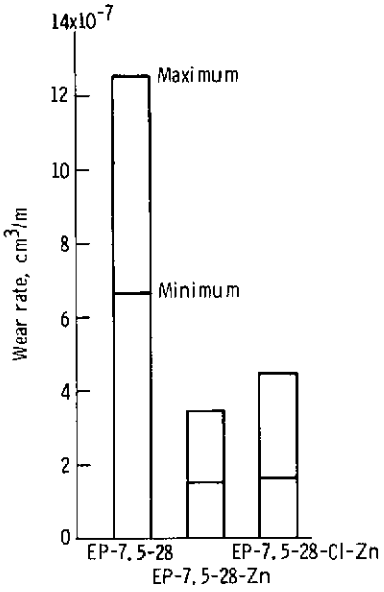
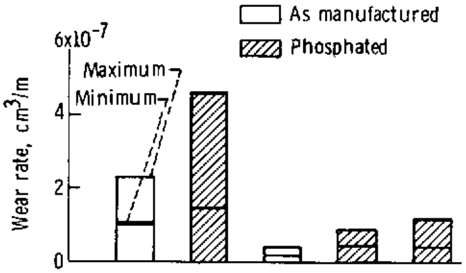
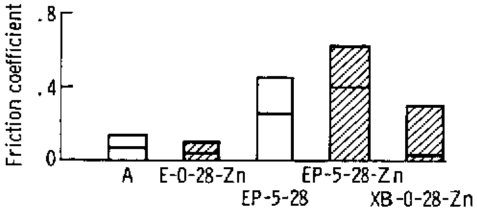


Figure 5. - Effect of purification and antioxidant treatments on experimental carbon-graphite EP-7.5-28 sliding on Inconel X-750. Load, 500 grams; sliding velocity, 1.33 meters per second; rider temperature, 650° C; test duration, 60 minutes.



(a) Wear rate.



(b) Friction coefficient.

Figure 6. - Comparison of various carbon-graphites sliding on Inconel X-750. Load, 500 grams; sliding velocity, 1.33 meters per second; rider temperature, 650° C; test duration, 30 or 60 minutes.

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