GA-A16054

FRICTION AND WEAR STUDIES OF GRAPHITE AND A CARBON-CARBON COMPOSITE IN AIR AND IN HELIUM

by

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This is a preprint of a paper to be presented at the International Conference on Wear of Materials – 1981, March 30 - April 1, 1981, San Francisco, California, and to be published in the Proceedings.

Work supported by the
Department of Energy
under Contract DE-AT03-76ET35300

GENERAL ATOMIC PROJECT 6400

OCTOBER 1980

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ABSTRACT

Sliding friction and wear tests were conducted on a commercial isotropic graphite and a carbon-carbon composite in air, purified helium, and a helium environment containing controlled amounts of impurities simulating the primary coolant chemistry of a high-temperature gas-cooled reactor (HTGR). The friction and wear characteristics of the materials investigated were stable and were found to be very sensitive to the testing temperature. In general, friction and wear decreased with increasing temperature in the range from ambient to 950 C. This temperature dependence is concluded to be due to chemisorption of impurities to form lubricating films and oxidation at higher temperatures, which reduce friction and wear. Graphite and carbon-carbon composites are concluded to be favorable candidate materials for high-temperature sliding service in helium-cooled reactors.

INTRODUCTION

Premium quality graphite and carbon fiber/carbon matrix composite materials (carbon-carbon composites) have been proposed for use as high-temperature out-of-core structures in HTGR systems. Specific applications include thermal barrier cover plates and attachment fixtures and heat exchanger components. In many instances relative movement must be allowed to accommodate temperature transients while maintaining structural support. This creates the necessity for motion between contacting surfaces under load and a concern for friction and wear behavior.

Low wear is important for maintaining support and integrity and low friction is needed to prevent overstressing. The purpose of the work presented here was to investigate the friction and wear behavior of a premium grade graphite and a carbon-carbon composite under conditions pertinent to use in advanced helium-cooled reactors.

EXPERIMENTAL

Materials

Materials employed in this study were POCO AXF-5Q graphite and a three-dimensional reinforced carbon-carbon composite supplied by McDonnell-Douglas Corporation. The AXF-5Q is a fine-grain premium quality isotropic graphite with an average bulk density of 1.84 Mg/m^3. The carbon-carbon composite specimens were cut from segments of a large cylinder containing 40 to 50 vol % rayon precursor graphite fibers in the form of yarn bundles. About 22% of the fibers were in the radial direction, with the remaining 78% divided equally between the axial and circumferential directions. The matrix was formed by pyrolysis of about equal portions of resin and pitch at 2700 C. According to this, about half the matrix was expected to be graphitic. The bulk density was in the range 1.77 to 1.80 Mg/m^3. Figure 1 is a polished axial section of the carbon-carbon cylinder showing the orthogonal weave of the fiber (due to the large cylinder radius) and pockets of matrix between the fiber bundles.

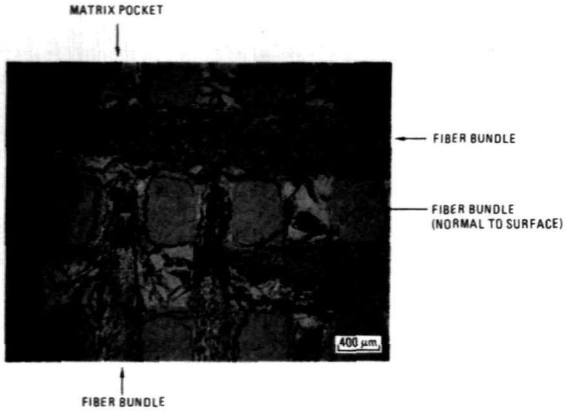


Fig. 1 Polished section of carbon-carbon composite

Apparatus and Test Environment

The major part of the test rig was a metal vacuum chamber that was surrounded by a resistance-heated furnace. A detailed description of this test rig was given previously (1). The test couple, which consisted of a 25.4 mm x 12.7 mm x 9.5 mm plate and two 6.4 mm diameter x 3.2 mm high buttons, was positioned at the center of the chamber where linear-reciprocating rubbing was carried out. The button-on-plate configuration and test mode are illustrated in Fig. 2. The rubbing distance was 6.4 mm for each stroke. The plate specimen was held in a horizontal plane by a cantilever shaft that was driven by an electric motor through reduction gears. A load cell was connected to the shaft, acting as an integral part, and gave a voltage output proportional to the force necessary to slide the plate between opposite buttons. The sliding force was recorded by a fast response (1/4 s full scale) strip-chart recorder. The buttons were held stationary under a vertical load. A Chromel-Alumel thermocouple was inserted into the holder of the upper button to monitor the specimen temperature as shown in Fig. 1. Throughout the test, the temperature was maintained within ±2 C of the testing temperature.

The chamber was connected to a gas-mixing system where the testing environments were provided and monitored. The testing chamber could be evacuated by a mechanical pump and then backfilled and purged with purified helium or helium mixed with controlled amounts of impurities simulating the HTGR primary coolant environment. In the case of pure helium testing, the commercial atomic-grade helium (99.995% purity) was purified by passing through an activated charcoal liquid-nitrogen cold trap with a flow rate of

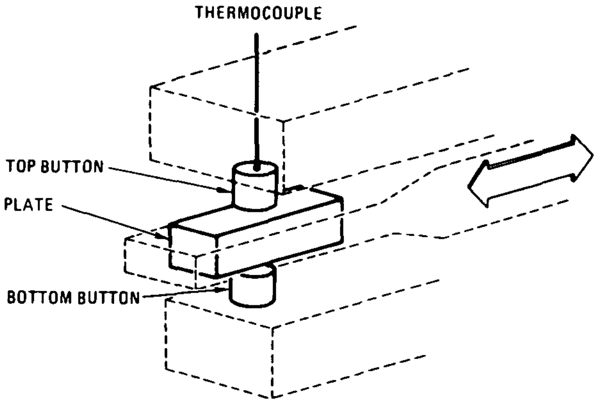


Fig. 2 "Button-on-plate" specimen configuration used in friction and wear tests. Top and bottom buttons remain stationary, while plate oscillates between them. Arrow indicates direction of plate oscillation.

500 cm^3/min. This gave O_2 levels of 4 to 5 ppm and 1 to 2 ppm H_2O as measured by an oxygen probe and moisture monitor. The gas environment was kept at about 1.68 atm pressure throughout the test.

In the case of the controlled-impurity helium (HTGR helium) testing, the cold-trapped helium was injected with the desired amounts of impurity constituents. The nominal impurity level employed in this study was 200 µatm (120 ppm) H_2, 20 µatm (12 ppm) CH_4, and 10 µatm (6 ppm) CO. The gaseous impurity concentrations were measured by a gas chromatograph and the variations of H_2, CH_4, and CO were within ±20%. At low temperatures the O_2 and H_2O levels may have been somewhat higher than for the purified helium due to the injection of the other impurities. At the highest temperatures the oxygen probe indicated values of about 10^{-22} atm O_2 due to interaction with H_2. This correlates well with H_2/H_2O equilibrium. Variations between inlet and outlet concentrations of the other impurities will be discussed in relation to the test results. A detailed description of the once-through helium mixing system used in this work is given elsewhere (2).

Procedures

Specimens of the carbon-carbon composite were all cut so that radial fiber bundles of the cylinder were essentially perpendicular to the wear surfaces. Wear surfaces of the plates were parallel to the cylinder axis so that rubbing was perpendicular to the tangential bundles and parallel to the axial bundles. The button specimens were oriented randomly with respect to rotation.

Prior to installation in the test rig, specimens were exposed to HTGR helium at 850 C for 48 h and furnace cooled to room temperature. The specimens were removed from the furnace retort and exposed to air at ambient temperatures for another 24 h before weight measurement was carried out. The specimen weight was measured using a precision electronic balance with a sensitivity of $\pm 10^{-5}$ g. The surface roughness was measured by a profilometer.

The friction and wear tests comprised rubbing the specimens against themselves in air, helium, or HTGR helium environments. The nominal contact pressure between the rubbing surfaces was 3.45 MPa, and the sliding velocity employed was 7.1 x 10^{-3} mm/s. The testing temperatures ranged from ambient up to 950 C.

After testing, the specimens were furnace-cooled to room temperature under load while the test gas was flowing and then removed from the test rig. The wear debris, if any, were collected. The tested specimens were also exposed to air at room temperature for 24 h and then weighed. Subsequent to weight measurement, the specimens were subjected to surface roughness measurement and metallographic and scanning electron microscopic examinations.

RESULTS

Friction Characteristics

Frictional behavior of the carbon-carbon composite in HTGR helium as a function of sliding distance and temperature is presented in Fig. 3. Each test was conducted at a constant temperature so that the friction and wear characteristics could be established over relatively long time periods. At room temperature the friction coefficient increased from 0.23 to about 0.45 in the first 1.25 m of sliding and then remained fairly constant at about 0.48. At elevated temperatures, the coefficient of friction decreased slightly during the break-in period and then increased

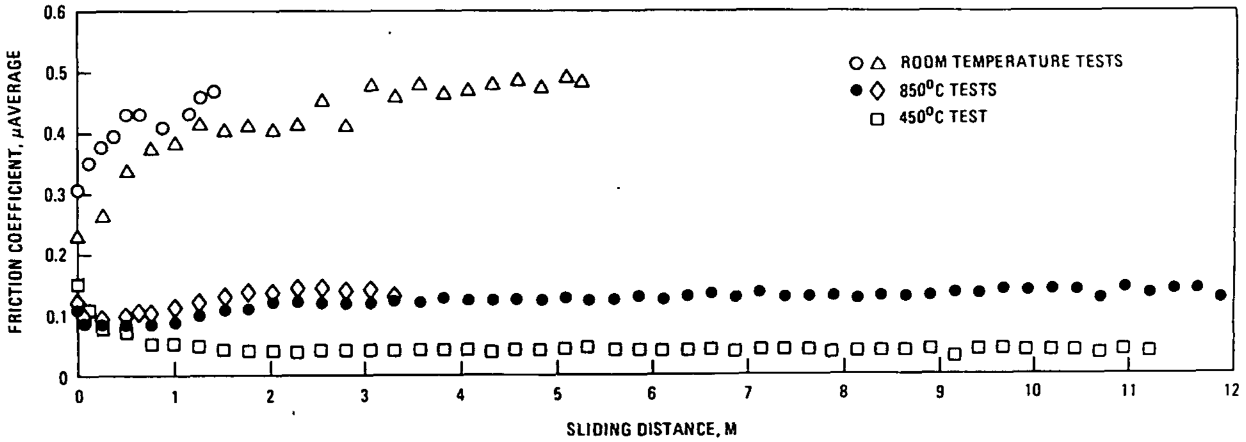
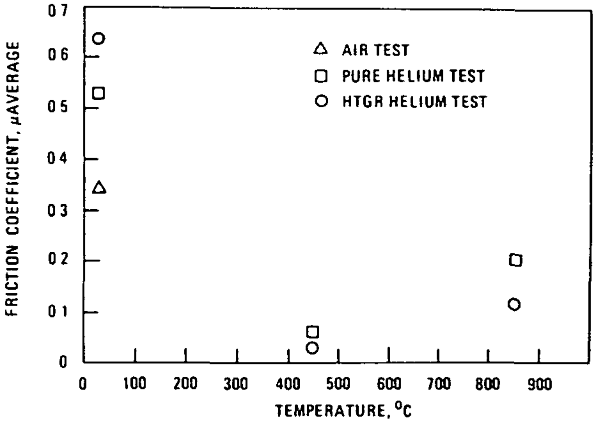


Fig. 3 Friction coefficients of the carbon-carbon composite as a function of sliding distance at various temperatures; test conducted in HTGR helium

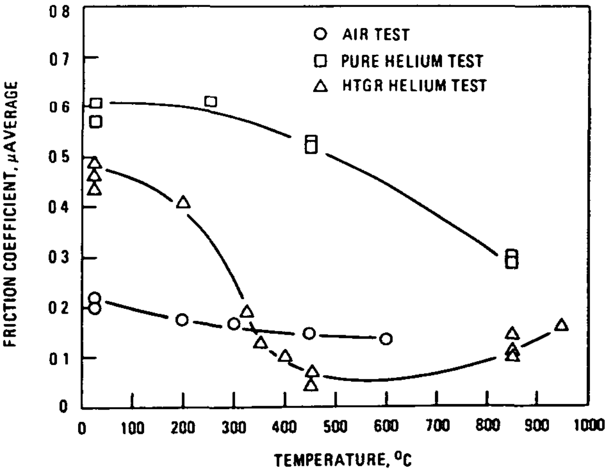
to 0.04 and 0.15 for the 450 and 850 C tests, respectively, throughout the tests. The steady-state friction was very stable. Only the room-temperature tests exhibited a little scattering.

Figures 4(a) and (b) show the results of transient tests designed to define the temperature dependence of the friction coefficients of the graphite and carbon-carbon composite, respectively. Included in the figures are tests conducted in air, pure helium, and HTGR helium environments. The data reported are steady-state coefficients, which were taken after the break-in period. The break-in periods in all cases were about 5 to 25 cycles of sliding. In general, the coefficient of friction decreased with increasing test temperature. In the air test the friction coefficient of the carbon-carbon composite decreased slightly and almost linearly from 0.2 at room temperature to 0.13 at 600 C. At 600 C the specimens were oxidized so severely that after a sliding distance of 0.6 m, only the plate specimen was left while the button specimens were burned away.

In the HTGR helium environment, both the graphite and carbon-carbon composite exhibited a very sharp decrease in friction coefficient in going from room temperature to 450 C. The friction coefficient then increased slightly as temperature increased up to 950 C [Fig. 3(b)]. The friction behavior of the carbon-carbon material tested in pure helium, however, showed a significant difference. Here the friction coefficient remained relatively high (about 0.52) at temperatures up to 450 C and then decreased to about 0.3 at 850 C. Graphite showed a friction behavior very similar to that observed in the HTGR helium tests; namely, the friction coefficient exhibited a minimum of about 0.03 at 450 C.



(a) GRAPHITE



(b) CARBON-CARBON COMPOSITE

Fig. 4 Temperature dependence of the friction coefficient of (a) graphite and (b) carbon-carbon composite in air, pure helium, and HTGR helium environments

The effect of temperature on the frictional behavior of the carbon-carbon composite was consistent and quite reproducible from one specimen combination to another. Buttons were run so that fiber bundles were oriented at various angles to the rubbing direction. Several perpendicular-parallel orientations were included. This work showed no large or obvious influence of orientation on friction characteristics.

Wear

Weight measurements were carried out on specimens tested at constant temperature in HTGR helium, the results are presented in Table 1. Weight loss rates, as determined by weight loss per unit distance of sliding, are also included. This assumes that the wear rates were constant since the steady-state friction of the materials under the present testing conditions was very stable. For the carbon-carbon composite at room temperature, the weight loss rate was about an order of magnitude higher than observed in the elevated-temperature tests. Correspondingly, significant amounts of wear debris were observed in the room-temperature tests. The weight loss rate of the specimens tested at 950 C was also higher than that of the 450 and 850 C specimens. This was due to excessive oxidation of the specimens during testing. At room temperature, the graphite showed a much lower rate than the carbon-carbon composite. In fact, the graphite wear rate under the severe room-temperature condition was comparable to the carbon-carbon composite at higher temperatures where friction was reduced.

The worn surfaces were measured by using a surface profilometer; the average wear depths are included in Table 1. Because of the heterogeneous nature of the carbon-carbon composite, surface profile measurements were difficult and the results were often quite varied. Nevertheless, the measurements indicate that at room temperature the wear depth was greater than at elevated temperatures, even though the total sliding distance was less. Graphite exhibited a very small wear depth even at room temperature.

Optical photographs of several worn surfaces typical of those observed in the present study are shown in Fig. 5. In general, under all the testing conditions, the specimens did not exhibit severe wear damage. The fiber bundles of the carbon-carbon composite specimens in the worn surface were still observable after testing. At room temperature the contacting surface area increased substantially so that the edge contour of the button specimens disappeared. Chipping of material at the edge of the buttons was also observed, as shown in Fig. 5(b). The graphite specimens exhibited only a polishing effect and small amounts of fine debris, even after testing at room temperature in helium where friction was high.

Details of the worn carbon-carbon composite surfaces and debris were examined by using a scanning electron microscope. Two types of surface damage were observed. The first kind of wear was associated with the wear tracks and involved smearing of the rubbing

TABLE 1

WEIGHT MEASUREMENTS OF FRICTION AND WEAR SPECIMENS OF GRAPHITE AND CARBON-CARBON COMPOSITES(a)

Material	Test Temp.	Sliding Distance (cm)	Pre-Test Specimen Weight (g)	Weight Loss (g)	Weight Loss Rate (g/cm)	Average Wear Depth (10^{-6} m)	Wear Debris
Carbon-carbon composite	Room temp.	525.8	5.80735	0.03286	6.3×10^{-5}	114.3	Large amount and coarse
Carbon-carbon composite	Room temp.	129.5	5.73783	0.00836	6.5×10^{-5}	63.5	Large amount and coarse
Carbon-carbon composite	450 C	1117.6	5.79740	0.00249	2.2×10^{-6}	30.5	Small amount and very fine
Carbon-carbon composite	850 C	1193.8	5.75401	0.00315	2.6×10^{-6}	55.9	Small amount and very fine
Carbon-carbon composite	950 C	406.4	5.74300	0.00742	1.8×10^{-5}	17.8	Small amount and very fine
Graphite	Room temp.	339.1	5.962310	0.00117	3.5×10^{-6}	7.62	Small amount and very fine

(a)Tests conducted in HTGR helium environment.

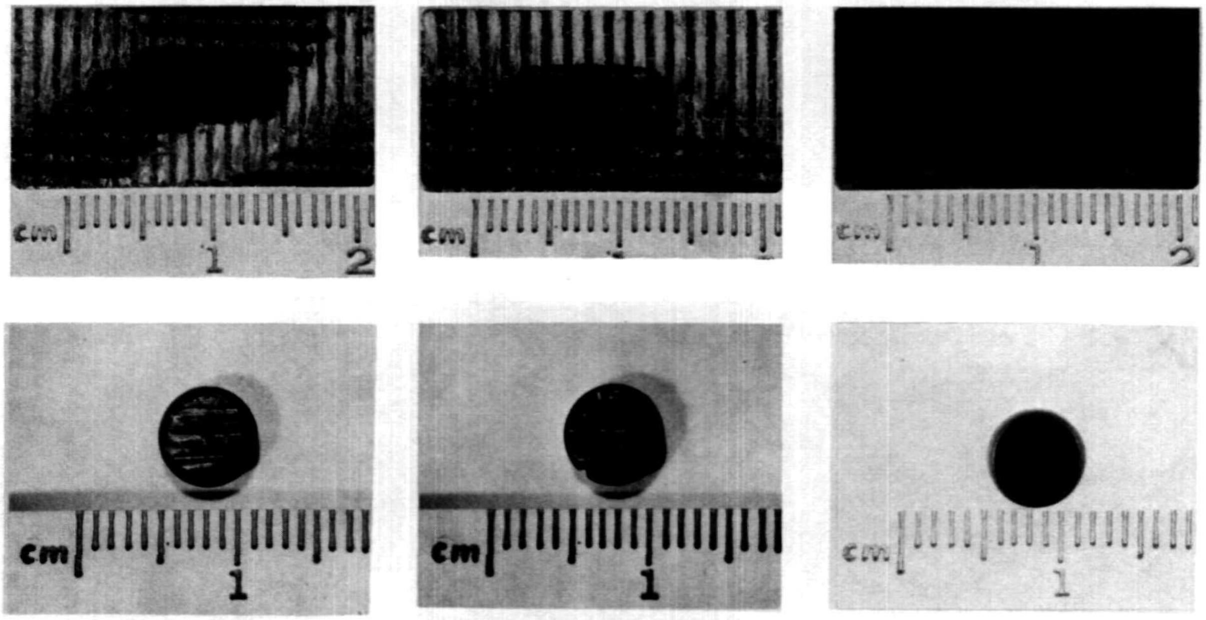


Fig. 5 Photographs of friction and wear tested specimens: (a) carbon-carbon composite, 850 C in htgr helium for 1193.8 cm of sliding, (b) carbon-carbon composite, room temperature in HTGR helium for 525.8 cm of sliding, and (c) graphite, temperature varied from ambient to 850 C in HTGR helium for a total sliding distance of 448.3 cm

surface parallel to the sliding direction. This eventually created surface cracks, which produced the debris shown in Figs. 6(a) and (b). The wear debris generated at 850 C in the HTGR helium test were very fine and flake-like, while the debris generated at room temperature in the HTGR helium test were about 10 times larger and the shapes were more irregular. Both chunky and needle-like debris were observed. The needle-like debris are believed to be the broken fibers.

The second kind of wear debris was associated with the orientation of fiber bundles rather than the sliding direction. Figures 6(c) and (d) show cracks that outline fiber bundles normal to the wear surface. The cracks run along the weaving direction rather than the wear direction. As sliding proceeded, the cracks propagated and eventually caused material to be chipped away from the edge of the specimens, as shown in Fig. 5(b). Both types of wear were more severe under conditions of high friction coefficient at room temperature.

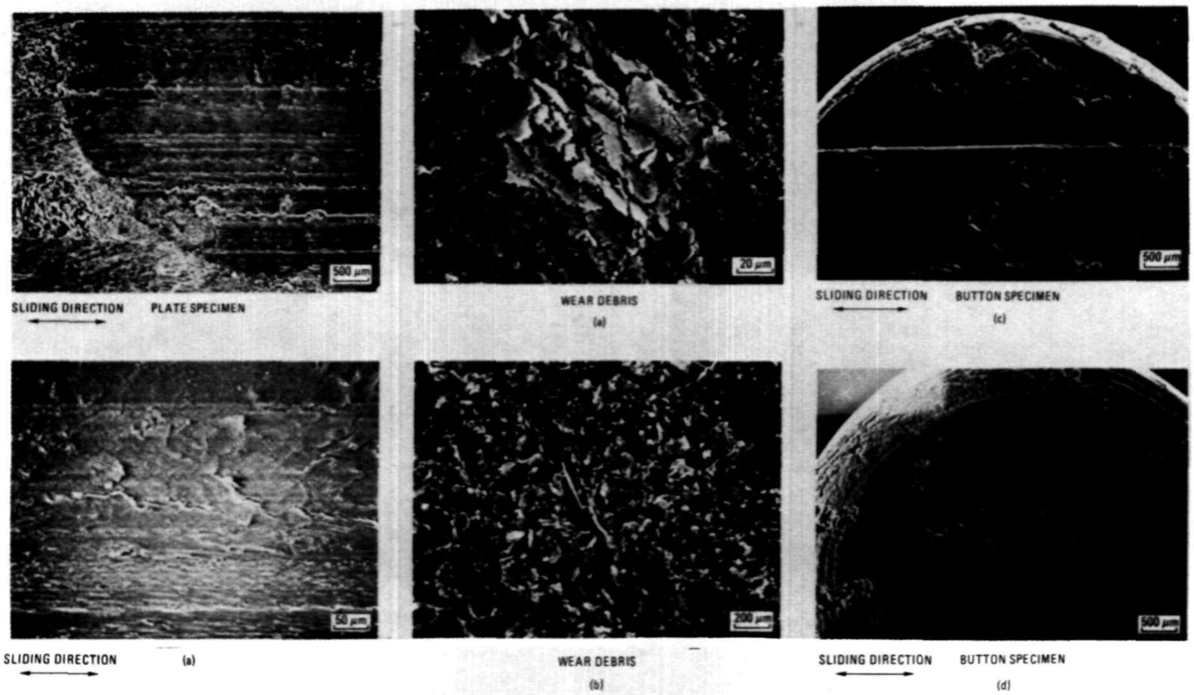


Fig. 6 Scanning electron micrographs showing the worn surface and debris of carbon-carbon composite: (a) 850 C in HTGR helium for 1193.8 cm of sliding, (b) room temperature in HTGR helium, (c) temperature varied from ambient to 850 C in HTGR helium for a total sliding of 403.9 cm, and (d) 450 C in HTGR helium for 1117.6 cm of sliding

DISCUSSION

Background of Tribological Characteristics of Carbons and Graphites

The influence of environment on the tribological behavior of carbon and graphites has been studied quite extensively. Much evidence exists that oxygen and water play important roles in the friction and wear of carbons and graphites (3, 4). Work has shown that oxygen is chemisorbed on graphite surfaces and that the concentration of oxygen complexes increases with increasing temperature up to about 500 C; however, after this, the concentration decreases due to CO formation, so that almost no surface oxygen exists above 950 C (5, 6).

It is generally thought that the basal plane surface of graphite is essentially inactive and that oxygen chemisorption occurs primarily at edge atoms. However, the work of Walker et al. (7) suggested that severe mechanical treatment which introduces basal plane dislocations could provide basal plane sites for oxygen chemisorption. The authors also showed that impurities, such as Fe, could provide additional adsorption sites.

In addition to oxygen chemisorption, cleavage of graphite crystals in controlled environments has demonstrated that oxygen can intrude between basal planes to produce a stress-etching effect that lowers the binding energy of the interlayers. These tests have shown a binding energy in vacuum 10 times that measured in air (8).

Water adsorption experiments on graphite indicate that undeformed basal planes are inactive (hydrophobic) and that the hydrophilic sites are few in number and are probably edge atoms with associated chemisorbed oxygen (9). Layers of water molecules build up in the active sites rather than absorb on adjacent hydrophobic areas.

Lowering of the friction coefficients and wear rates of graphites and carbons in oxygen- and water-bearing environments is well known (3, 4, 7, 8). The adsorption of oxygen and water to separate and lubricate rubbing surfaces (especially the very active basal plane edges) and, perhaps, lowering binding energy between basal planes to promote shearing has been proposed as the mechanism (3, 8).

Other gases such as H_2 and hydrocarbons have also been shown to lower the friction coefficient of graphite (10). Adsorption of these on graphite has not been studied, but theoretical calculation for hydrogen chemisorption suggests that a minimum activation energy exists for a 3.5 Å spacing between carbon atoms (11). This is very close to the spacing between basal planes in graphite.

The above considerations will be used to discuss the friction and wear observations made in this study for the carbon-carbon composite and graphite.

Present Observations

To better understand the tribological behavior of graphite and carbon-carbon composite materials in this experiment, it is instructive to study the friction curves observed in each test. Figure 7 shows steady-state friction curves for the materials tested at various temperatures and environments. It should be pointed out that friction of the materials was only sensitive to temperature. The distinctive curves shown in Fig. 7 were reproducible regardless of the number of cycles tested or whether the data were taken during cooling or heating. This suggests that the friction characteristics were independent of surface conditions, such as orientation, work hardening, and roughness.

At room temperature, both graphite and the carbon-carbon composite exhibited a higher and less smooth friction curve in pure and HTGR helium than in air. This is consistent with earlier reports (3, 4, 7, 8) that the friction coefficient of carbons and graphites increases with decreasing oxygen and water content in the test environment.

As the test temperature increases, physically adsorbed species are driven out of the material, while chemical interaction of the material surface with the environment becomes more significant. In air tests, the total energy required to slide the specimens (the area below the friction curve) decreased slightly, while the friction curves remained relatively smooth. This suggests that as temperature was increased, recession of edge sites by oxidation may have caused the slight reduction in friction coefficient. When the temperature was increased to 600 C, oxidation of the specimens became so rapid that after 0.6 m of sliding the specimens were burned away.

Maximum oxygen adsorption at about 500 C correlates with the minimum sliding friction coefficients at 450 C shown in Fig. 4 for graphite in the pure and HTGR helium environments and the carbon-carbon composite in HTGR helium (5, 6). As shown in Fig. 7 the friction curves at 450 C were very smooth and the friction coefficient was low except at the beginning of each stroke.

The sliding friction coefficient increased somewhat with increasing temperature above 450 C although it was still quite low, perhaps due to recession of active sites by oxidation. At 850 and 950 C the static friction was significantly lower than at 450 C, suggesting that the high static friction was associated with a dependence on stable adsorbed species, which were briefly absent when the direction of sliding was reversed. Evidence for oxidation at the high temperature was obtained by analyzing the outlet helium. Levels of CO increased from 10 μatm (6 ppm) at room temperature and 450 C to greater than twice this at 950 C in the HTGR helium tests and from <1 μatm (0.6 ppm) at the lower temperatures to 15 μatm (9 ppm) at 850 C in the pure helium tests.

In all cases, friction coefficients in the HTGR helium environment were lower than in pure helium at comparable temperatures. At the lower temperatures this was probably due to the higher impurity content of the HTGR helium. The lower friction and higher CO at 850 C and above suggest greater oxidation in the HTGR environment, perhaps due to a somewhat higher H_2O level. A factor complicating this interpretation was cracking of CH_4, which produced a reduction from 20 μatm (12 ppm) at the lower temperatures to 12 μatm (7 ppm) at 850 C and 3 μatm (1.8 ppm) at 950 C.

The behavior of the carbon-carbon composite in the pure helium environment was much different than that of the graphite. No minimum friction coefficient was observed at 450 C, and the friction coefficient continued to decrease as temperature increased. Adsorption of impurities in the pure helium environment was apparently inadequate. This could have been due to the high number of active sites in the carbon-carbon composite. About 10% of the surface was composed of fibers perpendicular to the surface. These provided a high density of active sites because layer planes are oriented parallel to fiber axes. On the other hand, the graphite was quite isotropic with, perhaps, only about 3% of the total surface being active (12). As the temperature increased above 450 C, oxidation rather than adsorption began to control the friction behavior so that the friction coefficient of the composite continued to decrease and approach the HTGR helium values.

Wear of the carbon-carbon composite material observed in this experiment was consistent with the friction characteristics. Wear was more substantial when higher energy was required to slide the specimens. High friction coefficients and nonsmooth friction curves, as seen in the room-temperature helium tests, produced the greatest wear.

Sliding wear took place through smearing of the rubbing surfaces and creation of wear tracks parallel to the sliding direction. The room-temperature pure and HTGR helium tests produced the greatest wear damage due to a lack of surface lubricity. This high degree of damage resulted from generation of the large and irregular debris shown in Fig. 6(b). When a surface lubrication mechanism was operative, the wear debris were small and flake-like, suggesting that shearing of layer planes was the controlling mechanism [Fig. 6(a)].

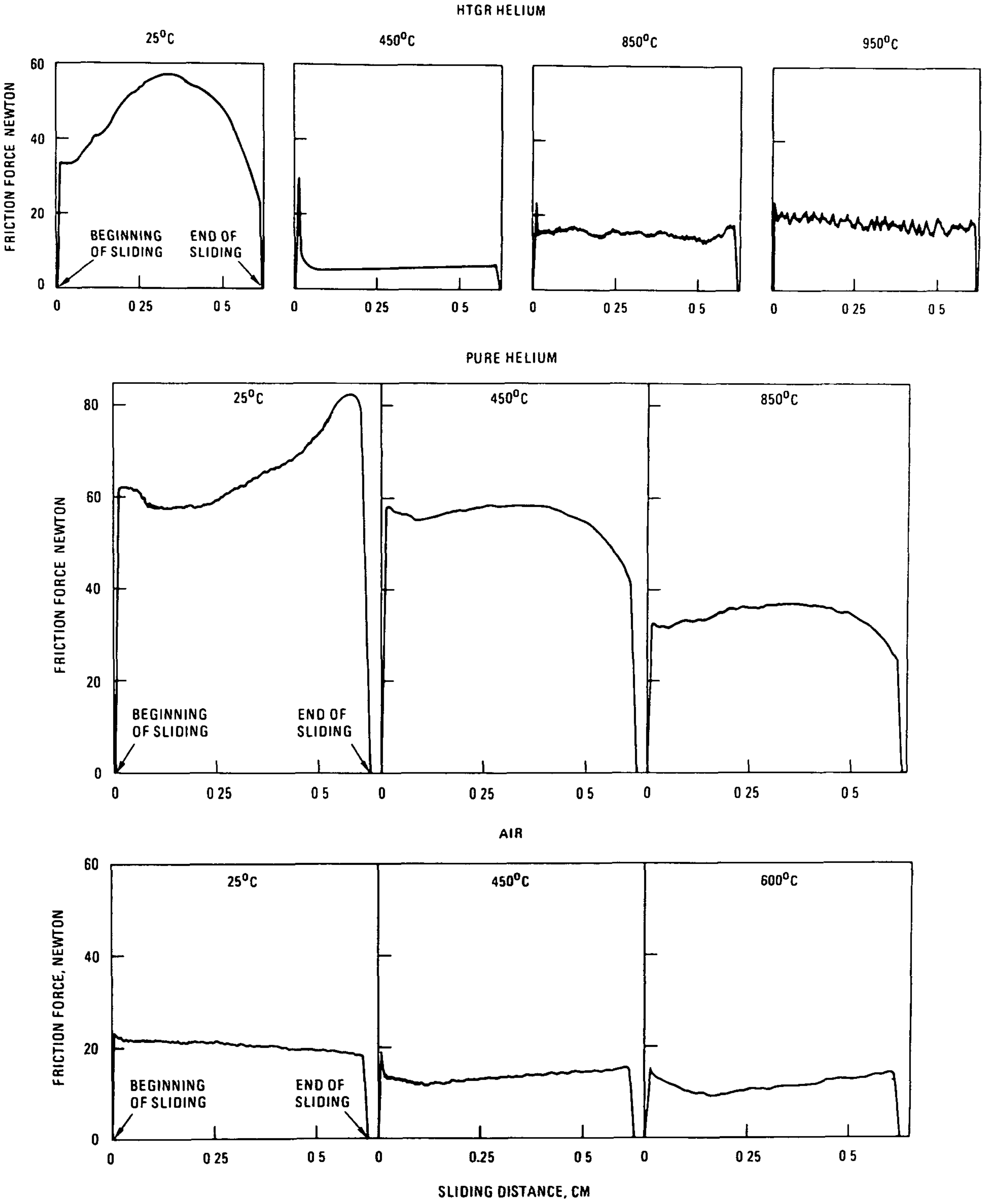
The large cracks observed on the wear surfaces shown in Figs. 5(b), 6(c), and 6(d) were investigated by diamond polishing the rubbed button after testing. The polished surface revealed that the cracks correlated with those shown to exist along the edges of fiber bundles oriented perpendicular to the surface in Fig. 1. It is likely that under severe rubbing conditions, the carbon-carbon composite was susceptible to propagation of these pre-existing cracks, and this resulted in chipping of the material from the specimen edge.

Wear of the graphite was low under all friction conditions. The room-temperature wear rate in helium was an order of magnitude lower than for the carbon-carbon composite under the same condition and about equal to the composite when the composite was tested at higher temperatures. This is believed to be due to the higher integrity, more homogeneous surface of the graphite. One graphite test was conducted at room temperature in air for 4.5 m of sliding. After testing there was virtually no wear.

CONCLUSIONS

The conclusions from the friction and wear tests are:

1. The graphite and carbon-carbon composite materials tested in this study exhibited very stable and reproducible tribological properties in air, pure helium, and HTGR helium environments. The low friction and wear observed at 450 C and above in a simulated reactor environment show that these are promising materials for high-temperature tribological applications in the HTGR.
2. The effect of temperature and environment on



(a)

Fig. 7 Steady-state friction curves for various temperatures and environments (a) carbon-carbon composite

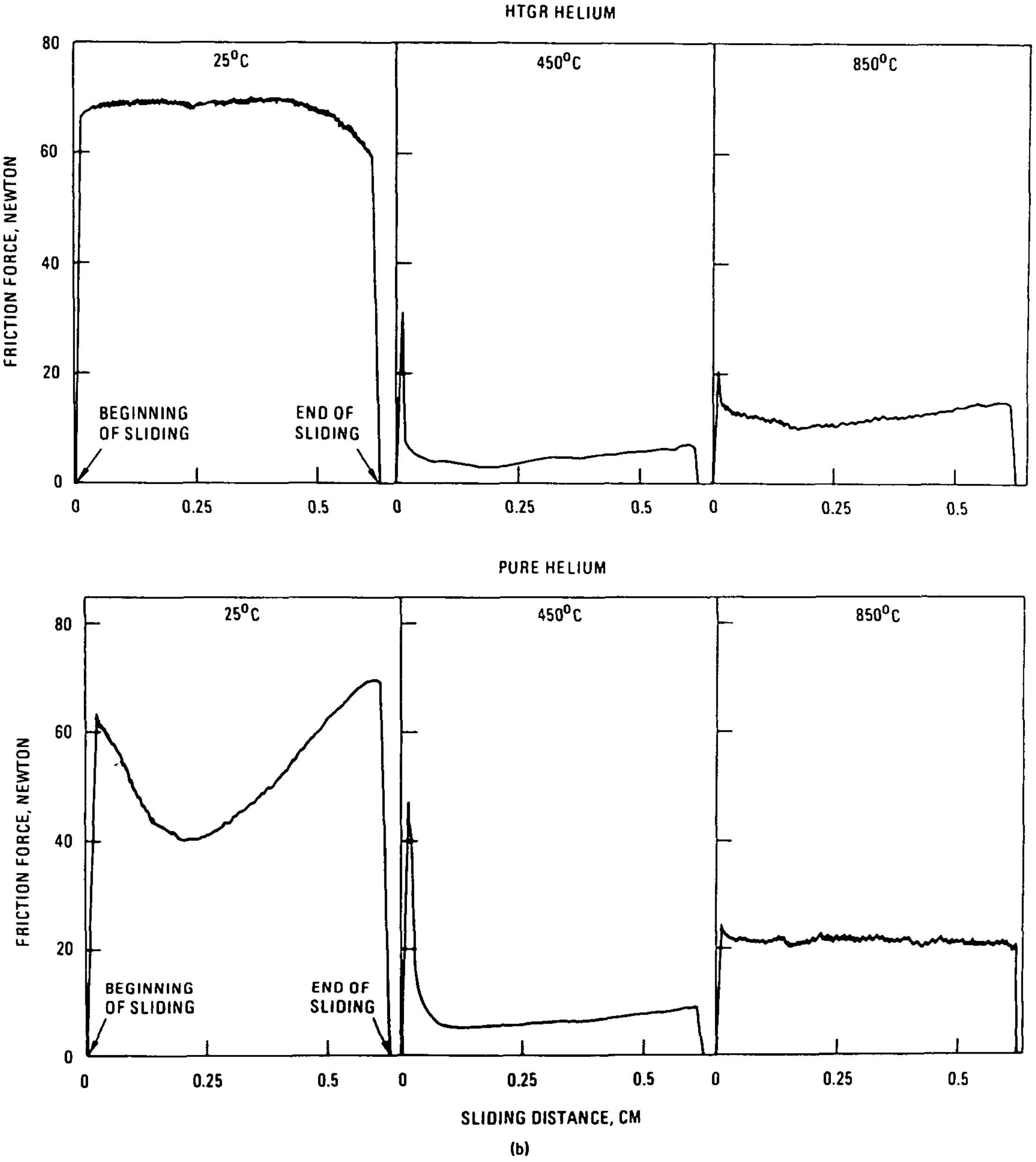


Fig. 7 Steady-state friction curves for various temperatures and environments (b) graphite

the friction behavior of these materials is consistent with demonstrated chemisorption and oxidation phenomena and accepted theories concerning the lubricity of carbons and graphite. Friction curves (force versus displacement) give a detailed picture of the friction behavior and can provide valuable insight for interpreting the effects of temperature, environment, and variations in material characteristics.

3. Wear of the carbon-carbon composite corresponds directly with friction. Wear is abrasive and substantial when friction is high and the friction curve is rough. Under conditions where lubrication mechanisms operate, wear occurred by the generation of small flake-like debris and damage was insignificant. Wear of the isotropic, fine-grained graphite is low even at room temperature in helium where friction is high. The high integrity surface of this material makes it superior to the composite under high friction conditions.

ACKNOWLEDGMENTS

The authors are grateful to R. R. Frazee for assistance in conducting friction and wear tests and D. I. Roberts and J. R. Lindgren for reviewing and commenting on the manuscript. Special thanks are due to Dr. D. H. Buckley, NASA-Lewis Research Center, for valuable discussions. This work was supported by the Department of Energy under Contract DE-AT03-76ET35300.

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