

WEAR AND FRICTION IN LIQUID NITROGEN AND HYDROGEN OF THE MATERIALS COMBINATIONS CARBON-STAINLESS STEEL AND CARBON-CARBON

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ABSTRACT

Wear and friction of conventional mechanical carbons for seals and bearings are excessive in liquid nitrogen and in liquid hydrogen. Better performance is needed for components of rocket engine turbopumps using liquid hydrogen. The problem was considered to result from the inability of the carbon to form an adherent lubricating graphitic film on the mating metal surface. The sliding of carbon on carbon rather than of carbon on metal was considered to offer a possible solution to this wear and friction problem.

Data were obtained with 3/16-inch-radius rider specimens sliding on the flat surface of a rotating disk submerged in liquid nitrogen (-320° F), liquid hydrogen (-423° F), or dry air (75° F). Surface speed was 2300 feet per minute and load was 1000 grams. The wear of a typical hard mechanical carbon sliding on itself in liquid nitrogen was less than 1 percent of that obtained with the same carbon in sliding contact with type 304 stainless steel. Friction coefficient was reduced from 0.18 to 0.04. In liquid hydrogen the wear reduction was not as great, while the difference in friction coefficient was greater (0.26 and 0.03). These data support the considerations on the importance of graphitic films on the mating surface.

INTRODUCTION

Mechanical carbon is the most commonly used material for nose pieces of sliding contact dynamic seals; the study reported is concerned with the reduction of wear for such carbons. In several areas of application where the carbon is in sliding contact with metal in an environment devoid of oxygen, rapid wear of the carbon occurs. (1) One such problem area involves dynamic shaft seals for mechanical devices such as pumps operating with liquid hydrogen and liquid nitrogen. Many of these devices have a relatively short life requirement as compared with industrial machinery. In cryogenic liquids, carbon wear may not be critical where the operating life of the mechanism is concerned. Carbon wear in these seals is very apt to be a critical factor, however, because it can produce increased seal leakage. Wear particles cause separation of sealing surfaces, thus increasing the cross-sectional area of the leakage path. In turbopump units for rocket propulsion, seal leakage can create a real hazard by resulting in premature contact of fuel and oxidant.

In more conventional applications where air, moisture, or other vapors are present, mechanical carbons usually have very low wear rates. This low wear is considered to result from the self-lubrication characteristic of carbons. Effective lubrication is achieved only when an oriented surface layer of graphitic carbon is established on the mating metal surface (2); under this circumstance the seal sliding materials combination is carbon on carbon rather than carbon on metal.

Factors necessary for the formation of the surface layer of graphitic carbon on metal surfaces have been the subject of intensive investigation for aircraft generator brushes operated in the dry and rarified air at high altitudes. (2) The formation of graphitic surface films with the help of adjuncts or addition agents (of the metal halide variety) to the carbons has resulted in reduced carbon wear in seals for cryogenic fuel as well as in aircraft generator brushes. Without such adjuncts, these films do not form in either liquid nitrogen or liquid hydrogen.

It has been suggested by a series of NASA investigations summarized in Reference 3 that the primary condition necessary for graphite lubrication is an affinity of graphite for the metal surface. This affinity may be obtained with intermediate films of adsorbed moisture, vapors, or oxides but usually is not obtained with nascent metals. A more common thesis for the mechanism of graphite lubrication involves shearing of interlamellar adsorption films (i. e., between adsorption films on graphite platelets); this thesis is much less concerned with the adhesion of graphite to the lubricated surface. Additional data are required to help establish the true mechanism of lubrication with graphite.

The objectives of this investigation were to clarify further the mechanism of graphite lubrication while suggesting a possible means of reducing wear in dynamic seals. Carbons without adjuncts were run in sliding contact against metals or carbons in dry air at 75° F, in liquid nitrogen at -320° F, and in liquid hydrogen at -423° F. Data were obtained with a 3/16-inch-radius rider specimen sliding on the flat surface of a rotating 2-1/2-inch-diameter disk. The sliding velocity was 2300 feet per minute and the load was 1000 grams. In sliding of nonhalide-treated carbons against metals, and also against a similar carbon in the above environments, the role of interlamellar adsorption can be further resolved. If wear of carbon on carbon is low, surface adhesion rather than interlamellar adsorption must be considered the primary variable since the solid body contains the lubricating material (and therefore no special conditions for film formation are required). Conversely, high carbon-carbon wear would indicate that interlamellar adsorption was the more important factor.

MATERIALS

Rider and disk materials of this investigation included mechanical carbons (Table 1) of the types used in nose pieces of face-type dynamic seals. The harder carbon grade included approximately 70 to 80 percent amorphous carbon and 30 to 20 percent graphitic carbon. The softer carbon grade was predominantly graphitic carbon. In these studies two commercial impregnants (phenolic and metal fluoride) had been introduced into the pores of the hard carbon.

TABLE 1. TYPICAL MECHANICAL PROPERTIES OF CARBONS USED

Manufacturer's Data

<u>Material Designation</u>	<u>Hardness Scleroscope</u>	<u>Traverse Strength psi</u>	<u>Compressive Strength psi</u>
Nonimpregnated dense graphitic carbon	85	12,000	25,000
Nonimpregnated hard carbon	85	9,000	24,000
Phenolic impregnated hard carbon.	90-100	10,000-13,000	28,000-37,000
Metal fluoride impregnated hard carbon	90	10,000	34,000

The metal disks used in these experiments were of type 304 austenitic stainless steel. This alloy is commonly used in cryogenic equipment.

APPARATUS AND PROCEDURE

The apparatus used in this investigation is shown in Figure 1. The basic elements consist of a rotating-disk specimen (2-1/2-in. diam., 1/2-inch thick) and a hemisphere-tipped (3/16-in. rad.) rider specimen. The rider specimen slides in a circumferential path on the lower flat surface of the rotating disk. The specimens were run submerged in the experimental fluid. Surface speeds of 2300 ft/min were used for the data reported herein.

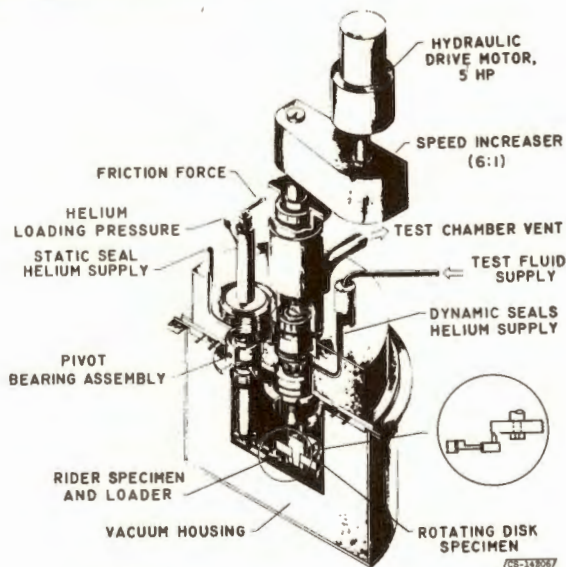


FIGURE 1. CRYOGENIC FRICTION APPARATUS

The rider specimen was supported by an arm assembly that allowed the measurement of friction force outside the test chamber. The vertical shaft of this assembly was pivoted through a bearing assembly mounted in the top housing. Friction force was measured by a strain-gage dynamometer ring connected to the top of the shaft.

The rider specimen was loaded (1000 g) with a helium-pressurized piston assembly. The internal pressure of the test chamber was held at a gage pressure of 1-1/2 lb/sq in. by a pressure-relief valve in the vent line.

The disks were all finish ground on the test surface and the metal disks had surface roughness from 4 to 8 microin. centerline average as measured with a Talysurf. The radius (3/16 in.) of each rider specimen was checked with a radius gage.

The metal disks were cleaned by the following procedure: (a) washed with acetone, (b) repeatedly scrubbed with moist levigated alumina, (c) washed in tap water, (d) washed in distilled water, (e) washed in 95 percent ethyl alcohol or ACS certified acetone, and (f) dried in clean warm air and stored in a desiccator.

The liquid was transferred to the test chamber through a closed system. The storage Dewar was pressurized from 2 to 6 lb/sq in. to transfer the liquid. It was possible to maintain the proper liquid level during an experiment (about 3 in. above the test specimen) by controlling the Dewar pressure.

The carbon specimens were cleaned by using the same procedure as for metals but without using levigated alumina. The specimens were handled with tongs and rubber-gloves to minimize contamination and dried in a vacuum prior to being stored in a desiccator. The test chamber was cleaned with acetone just prior to each run.

After stabilization of the liquid level, the drive motor was adjusted to the proper speed. The load was then applied to begin the experiment. Frictional force was measured by using a recording

potentiometer as a strain indicator. Most runs were 1-hr duration. The wear of the rider specimens was determined by measuring the diameter of the wear scar and calculating wear volume. Disk wear was determined by calculating volume based on four equally spaced wear track cross-section areas obtained with a Talysurf surface profile indicator and measured with a planimeter.

RESULTS AND DISCUSSION

Reference Data

The data of Figure 2 show typical wear values for a series of carbon rider specimens discussed in Reference 1. These data are for the basic grade of hard carbon (Table 1) with (1) no impregnation, (2) phenolic impregnation, and (3) impregnation by a metal fluoride, all sliding against austen-

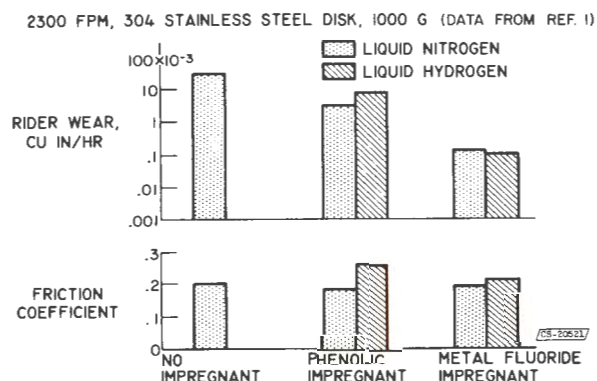


FIGURE 2. WEAR AND FRICTION OF MECHANICAL CARBON IN LIQUID NITROGEN AND HYDROGEN

itic stainless steel. Phenolic impregnated materials are most commonly used as mechanical carbons in bearing and seal applications. Figure 2 shows that the phenolic impregnant reduced wear during operation in liquid nitrogen. The wear of this material was higher in liquid hydrogen than in liquid nitrogen. The metal fluoride impregnant caused substantial reduction in wear for both liquid nitrogen and liquid hydrogen; this impregnant is the same type material that has been used to solve excessive wear (dusting) problems in high-altitude brushes for aircraft generators. (2) Such additives as the metal fluorides serve as adjuncts to establish a film of graphitic carbon on the metal used in sliding contact with mechanical carbon.

The metal-fluoride impregnated carbon is one of the better seal materials now used in experimental cryogenic applications. Data reported herein for this material can therefore be used for reference purposes.

The friction data of Figure 2 consistently show friction coefficients around 0.2 (0.18 to 0.26) for the three carbons sliding against type 304 stainless steel in either liquid nitrogen or liquid hydrogen. These values can represent substantial energy dissipation in seals; this is particularly true when seals are not pressure balanced. Most commercially available seals have substantial pressure loading and therefore friction forces can be important.

Mechanism of Lubrication by Graphite

A mechanism for lubrication by graphite has been postulated⁽³⁾ wherein the adhesion of graphitic carbon particles to the mating metal surface is the primary requirement for low wear with a mechanical carbon sliding on a metal surface. Other investigators have previously suggested that the action of air, moisture, or other vapors adsorbed on the surface of graphite lamellae is the basic requirement for lubrication. The question as to the relative roles of these contributions of the operating environment on slip between particles, or adherence of these particles to the mating surface, must be resolved.

Dry Air Experiments

Figure 3 presents wear and friction data for a typical phenolic impregnated mechanical carbon operating in dry air (dried by passage through a liquid nitrogen cold trap). Data are reported for the carbon sliding on type 304 stainless steel and sliding on carbon. Wear differences in dry air were small compared with data shown later for nitrogen and hydrogen. In these experiments, the graphitic carbon from the specimens was aided in its lubricating function by oxygen from the air. It should also be noted that these runs were made at 75° F rather than the cryogenic temperatures of the experiments that will be described later. Some reduction in friction was obtained with carbon on carbon over that of carbon on metal.

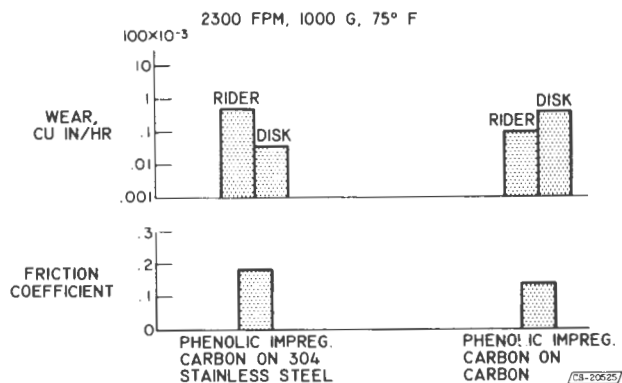


FIGURE 3. WEAR AND FRICTION OF PHENOLIC IMPREGNATED CARBON IN DRY AIR WITH DIFFERENT DISKS

Liquid Nitrogen Experiments.

Figure 4 presents wear and friction data for two mechanical carbons in sliding contact with austenitic stainless steel and with like carbon in liquid nitrogen. With both of these carbons the wear of the rider specimens was lower by at least one order of magnitude when they were in sliding contact with like carbons rather than with the metal surface.

Liquid nitrogen can be considered free of the adsorbate materials (moisture, oxygen, etc.) that are usually considered essential for effective lubrication with graphite. The lower wear and lower friction experienced with carbon on carbon where the film formation problem is avoided indicates that this is an important variable. Sliding causes the desired orientation of graphitic particles that are constituents of the base materials and therefore need not be deposited during the sliding process. It may be presumed that the residual oxide layer on the metal surface can provide a basis for surface adhesion of a graphite layer and, therefore, somewhat different performance would be obtained with nascent surfaces than with those reported herein.

The surface appearances of both the rider and disk experiments for carbon riders sliding on stainless steel and carbon disks showed substantial differences in film forming properties. Little film formation was evident on the stainless steel surface and a highly polished film was evident on the carbon surface (Fig. 5). The highly polished smooth film present on the carbon disk is indicative of desirable filming tendencies and suggests orientation of the graphitic particles. It is apparent that there was no problem in obtaining and maintaining a graphitic lubricating film on the surface of mechanical carbons. It should be noted that the phenolic impregnated hard mechanical carbon, which showed the least wear, contained approximately 20 to 30 percent graphitic carbon; the softer dense graphitic carbon body, which was not impregnated, was primarily graphitic carbon. Experience with self-lubricating materials has indicated that concentrations of the lubricating media, as low as 5 percent can provide effective lubrication. Also, the functioning of a hard matrix in obtaining low friction and wear with duplex systems is widely appreciated. These factors contribute to the more successful experience with the phenolic impregnated, hard carbon. The role of hardness of the bulk materials is of particular importance in the disk wear, as shown in Figure 4. It is of interest that, with carbon disks, the wear was of the same magnitude as for the rider; whereas, the wear of the metal disks was substantially less.

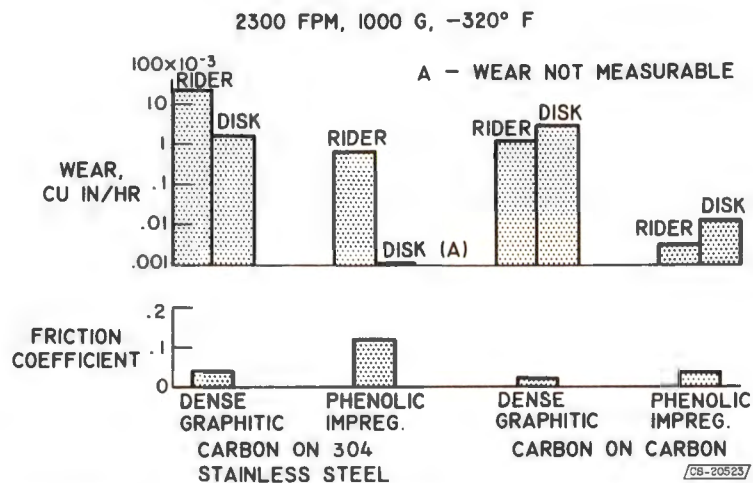


FIGURE 4. WEAR AND FRICTION OF TWO MECHANICAL CARBONS IN LIQUID NITROGEN

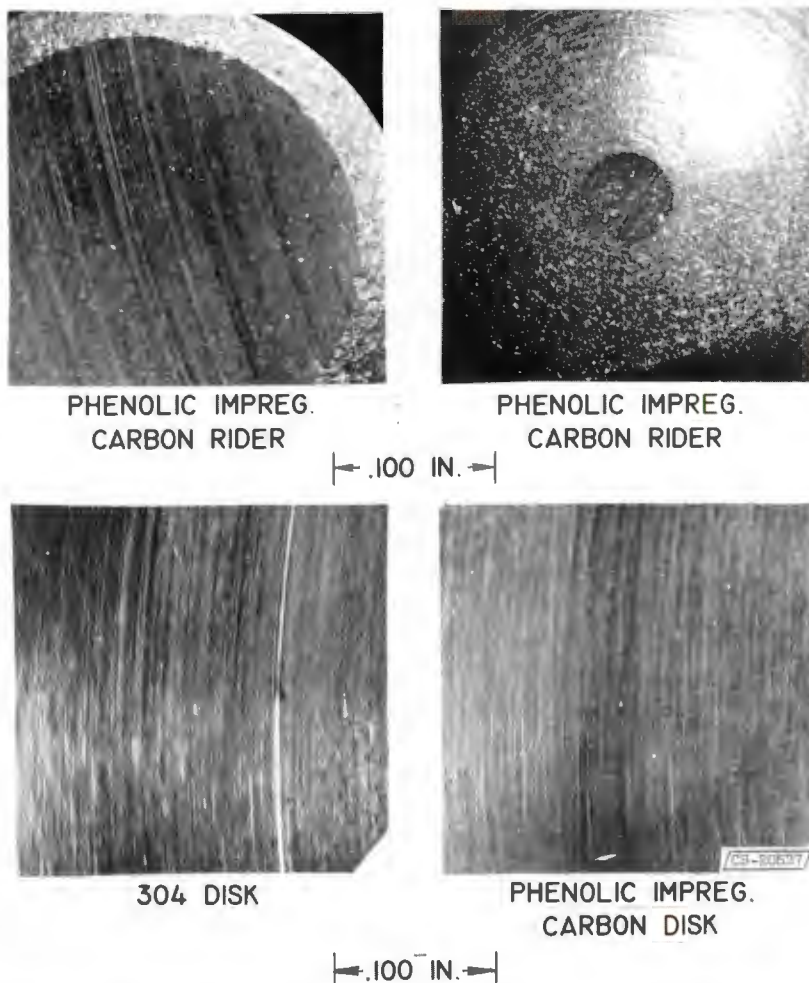


FIGURE 5. SURFACES OF RIDERS AND DISKS AFTER SLIDING IN LIQUID NITROGEN

Liquid Hydrogen Experiments.

Wear and friction data for the phenolic impregnated hard carbon and the dense graphitic carbon previously described were obtained in liquid hydrogen and are presented in Figure 6. The phenolic impregnated hard carbon provided the most desirable combination of low wear and low friction in liquid hydrogen. The wear of rider specimens of this carbon sliding against austenitic stainless steel was 60 times greater than when the carbon was sliding against itself. This advantage was not as great as the wear ratio would indicate, however, because disk wear was greater with the carbon material. The total reduction in wear by obtaining a carbon-carbon system was however, sufficiently great that this type of application should be attractive for operation in liquid hydrogen. Apparatus seals using carbon on carbon have shown much greater life than seals using carbon on metal in the same application.

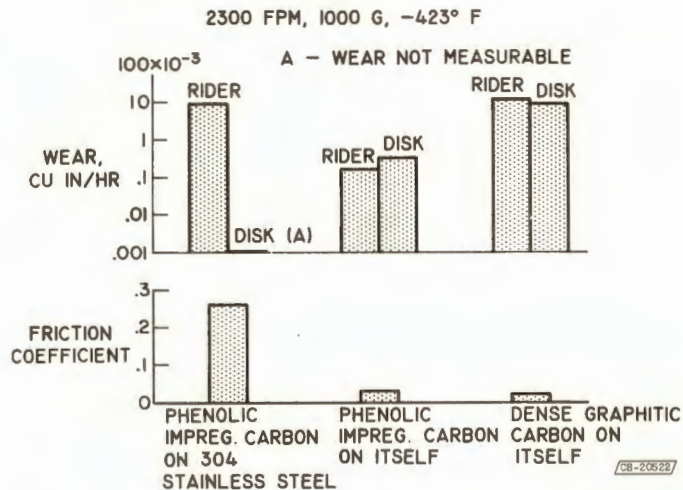


FIGURE 6. WEAR AND FRICTION OF TWO MECHANICAL CARBONS IN LIQUID HYDROGEN

The appearance of the surface of the carbon rider and disk after sliding contact in liquid hydrogen indicated film forming properties that were less effective than in liquid nitrogen. Such surfaces are shown in the photographs of Figure 7. The primary difference in these surfaces from those obtained in liquid nitrogen is the degree of surface polishing. The lower temperature of liquid hydrogen may have contributed to this appearance. Bodies composed only of carbon materials should not be affected by this change in temperature; however, the phenolic impregnant is substantially more brittle at the lower temperature of liquid hydrogen. Subsurface brittle fracture of the phenolic component could cause localized removal of surface material sufficient to give the surface appearance indicated by Figure 7.

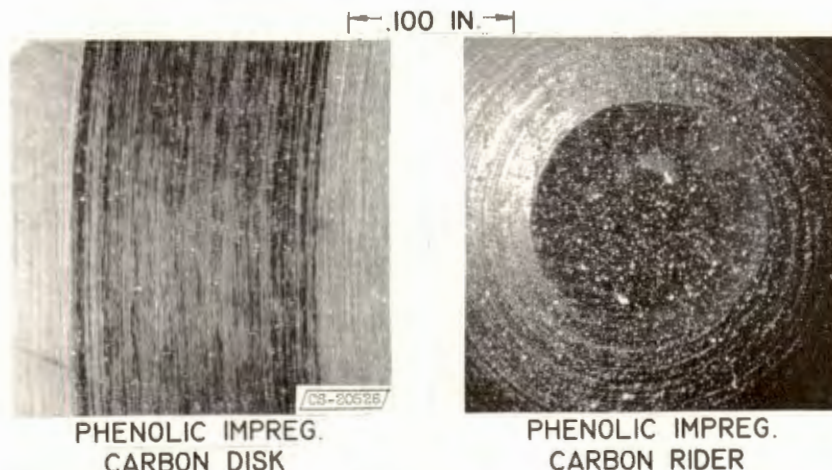


FIGURE 7. SURFACE OF RIDER AND DISK AFTER SLIDING IN LIQUID HYDROGEN

As indicated previously, the phenolic impregnated hard carbon sliding against itself gave better wear and friction properties in dry air, liquid nitrogen, and liquid hydrogen than the nonimpregnated dense graphitic carbon. The data for this combination are again presented in Figure 8. This comparison shows directly that the wear of this combination is lowest in liquid nitrogen and that friction was lowest in liquid hydrogen. If the wear of the metal fluoride impregnated carbon against steel presented in Figure 2 (0.14×10^{-3} cu in./hr) can be considered acceptable, we should also consider that the wear of the phenolic impregnated carbon against itself (0.16×10^{-3} cu in./hr) is acceptable. The wear rates for these two materials in liquid hydrogen are similar. A substantial reduction in friction can be obtained by using the carbon-carbon combination ($f = 0.03$) rather than the metal fluoride impregnated carbon in sliding contact with austenitic stainless steel ($f = 0.21$).

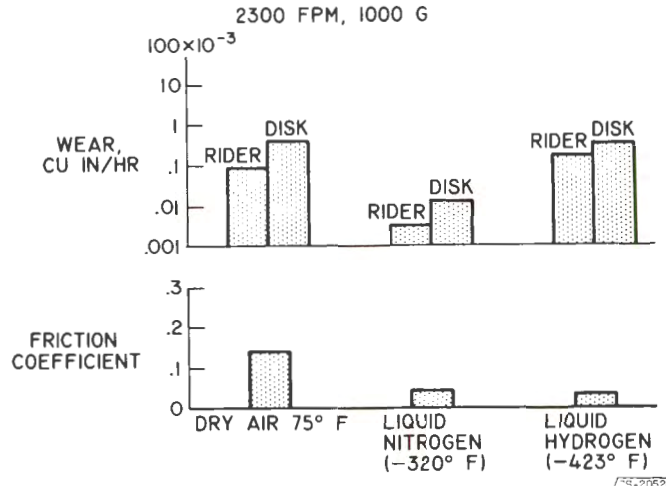


FIGURE 8. WEAR AND FRICTION ON PHENOLIC IMPREGNATED CARBON AGAINST ITSELF IN THREE MEDIA

Wear of the carbon disk specimens in these experiments was greater than for the austenitic stainless steel specimens, and there was substantial difference in the wear of the phenolic impregnated hard carbon and the dense graphitic carbon materials. The differences in wear of disk specimens are illustrated in Figure 9 using typical surface profile traces obtained radially across the wear tracks using a Talysurf surface profile indicator. These greatly magnified wear track profiles suggest that wear of carbon mating surfaces can be a problem. In practical seal application, however, the surface loads are sufficiently low that deterioration of the surface from high surface stresses would be substantially less.

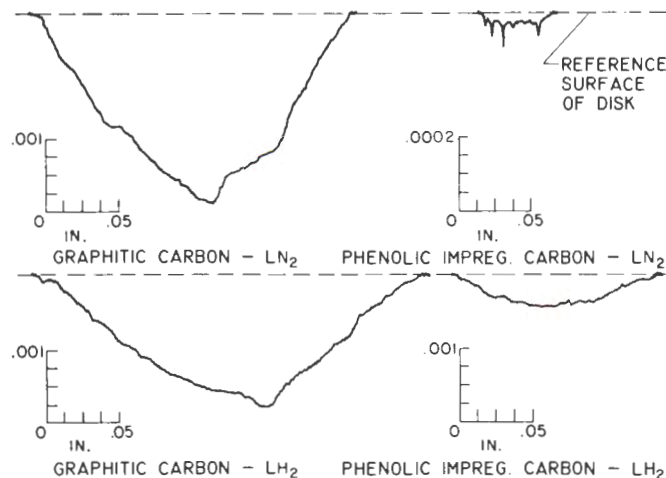


FIGURE 9. DISK SURFACE TRACINGS OF TYPICAL WEAR TRACK CROSS SECTIONS

SUMMARY OF RESULTS

Data obtained in a wear and friction investigation of carbon in liquid nitrogen and liquid hydrogen show that:

- (1) Substantial reductions in carbon rider wear and lower friction can be obtained by using a carbon in sliding contact with carbon rather than with austenitic stainless steel. Carbon disk wear was greater than metal disk wear but experience shows that carbon-carbon seal surfaces have greater life than carbon - stainless steel seal surfaces.
- (2) A harder, phenolic impregnated carbon had lower wear than a nonimpregnated and more highly graphitic carbon. This observation was true for these carbons in contact with like carbons and with stainless steel.
- (3) The data obtained herein support the fundamental consideration that a primary factor in lubrication with graphitic carbon is its ability to form an adherent film on the mating surface. Since lubrication was obtained in the absence of adsorbates that are usually considered essential for graphite lubrication, these data indicate that film formation is more important than interlamellar adsorption. A carbon that showed substantial wear in contact with metal showed improvement in wear and friction in sliding contact with itself.

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