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**GRAPHITE FLUORIDE LUBRICATION: THE EFFECT OF FLUORINE
CONTENT, ATMOSPHERE, AND BURNISHING TECHNIQUE**

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ABSTRACT

Eight different graphite fluoride $(CF_x)_n$ compounds with fluorine to carbon ratios varying from $x = 0.25$ to 1.1 were evaluated as burnished films in order to determine the effect of fluorine content on the solid lubricant properties of graphite fluoride. For comparison, similar experiments were conducted on graphite burnished films. It was found that even a small amount of fluorine in graphite fluoride $(CF_{0.25})_n$ improved the lubricating properties of graphite. However, such factors as burnishing atmosphere, burnishing technique, test atmosphere, and specimen temperature affected the results as much as varying the fluorine to carbon ratio of the compound. Best life was found for films that were machine-burnished in moist air and tested in moist air.

E-7492

INTRODUCTION

Graphite fluoride has been shown to be an effective solid lubricant under various conditions and types of applications (1-5). References 1 and 2 have shown that burnished films of graphite fluoride perform better than or equivalent to burnished films of MoS_2 or graphite. These burnished film results were further improved by bonding graphite fluoride to a surface using polyimide (3). Graphite fluoride has also been used as an additive in greases, mechanical carbons, and carbon-fiber-reinforced polytetrafluoroethylene (PTFE) (4). Graphite fluoride has also been combined with silicate or epoxy-phenolic binders with good results (5).

The discovery of graphite fluoride can be attributed to Ruff and Bretschneider, who found in 1934 that graphite and fluorine would combine in a combustionless reaction at about 420°C to form a grey colored solid (6). Since 1934, other experimenters have worked with the compound and have found that, by varying the reaction temperature, pressure, and time, different fluorine to carbon ratios could be obtained (7-14). As the fluorine content of the compound increased, the properties of the compound were changed. For example, with increasing fluorine content, the color of the compound becomes lighter and the electrical conductivity decreases.

In reference 1 it was shown that, in the range of $(\text{CF}_{0.7})_n$ to $(\text{CF}_{1.12})_n$, changing the fluorine to carbon ratio did not appreciably affect the friction and wear life characteristics of burnished graphite fluoride films tested in a dry argon atmosphere. The purpose of the present investigation was to expand this previous study by extending the fluorine to carbon ratio to 0.25 and by studying the effect of atmosphere and burnishing technique. Graphite was included in the study for comparison. This

work is useful not only for lubrication but also from an economic viewpoint in that more highly fluorinated graphite fluoride is more expensive.

FRICTION APPARATUS

A hemisphere-on-flat type of sliding friction apparatus (fig. 1) was used to evaluate the graphite fluoride burnished films. Basically, the device consisted of a flat, 6.3-centimeter-diameter disk in sliding contact with a stationary, 0.476-centimeter-radius, hemispherically tipped rider. A 1-kilogram load was applied to the rider as the disk rotated at 1000 rpm. The rider slid on a 5-centimeter-diameter track on the disk, which gave it a linear sliding speed of 2.6 meters per second.

Induction heating was used to heat the disk. The temperature was monitored by an infrared pyrometer focused on the wear track of the disk. A strain gage sensed the frictional force, which was continuously recorded on a strip-chart recorder.

BURNISHING APPARATUS

The apparatus used for burnishing the 440-C stainless steel disks is shown in figure 2. The disk was attached to the vertical shaft of a small electric motor by use of a cup-shaped holder. Setscrews on the rim of the holder kept the disk from slipping as the holder was rotated. Two vertical rods were used to restrain a floating metal plate to which was attached the solid lubricant applicator. In these experiments the back of polishing cloths were used as the applicators. The load was applied by placing weights on top of the metal plate. A tray positioned under the disk holder was used to catch the solid lubricant spillover.

The burnishing apparatus was designed to fit under the bell jar of a vacuum system. The atmosphere in which burnishing took place could thus be controlled. This was done simply by pulling a vacuum in the bell jar and then backfilling with the desired atmosphere.

PROCEDURE

Surface Preparation and Cleaning Procedure

The hardness range of the 440-C stainless-steel specimens used in this investigation was Rockwell C 58 to 60. The disk surfaces were roughened by sandblasting to an rms of 0.9 to 1.3 micrometers (35×10^{-6} to 50×10^{-6} in.). The cleaning procedure after sandblasting the disks was -

- (1) Scrub surface under running water with a brush to remove abrasive particles.

- (2) Clean surface with pure ethyl alcohol.

- (3) Scrub surface with a water paste of levigated alumina. Clean until water wets the surface readily.

- (4) Rinse under running water to remove levigated alumina (use a brush to facilitate removal).

- (5) Rinse in distilled water.

- (6) Dry surfaces using dry compressed air. (Surfaces not dried quickly have a tendency to oxidize.)

The riders were also cleaned by this procedure; but, since the riders were not sandblasted, step (1) was not necessary.

Disks Burnished by Hand

Hand burnishing was done in room air with no control of relative humidity. The humidity range was about 25 to 60 percent relative humidity. The back of a polishing cloth was used as the applicator. It was made of an open weave fabric (twilled) and served as a good applicator. The following steps were used to burnish the cleaned roughened 440-C stainless steel disks:

(1) Apply graphite fluoride powder to the surface and distribute it evenly using the cloth.

(2) Apply pressure and rub until the surface has a gloss.

Note: it may be necessary to apply more graphite fluoride powder and repeat steps (1) and (2) in order to get a gloss.

Disks Burnished on Apparatus

The procedure using the burnishing apparatus is as follows:

(1) Apply the graphite fluoride powder to the cleaned, roughened disk surface and spread it evenly over the surface with the cloth.

(2) Apply about 1/2 gram of $(CF_x)_n$ to the contact zone of the applicator and distribute it evenly using another cloth.

(3) Assemble the apparatus as shown in figure 2, using two 1-kilogram weights for the applied load.

(4) Evacuate the bell jar and backfill it with the desired atmosphere. The atmospheres used in this program were either dry air (20 ppm H_2O) or moist air (10 000 ppm H_2O).

(5) Set the disk into rotation by gradually increasing the speed to 15 rpm and burnish for 1 hour.

Experimental Technique

The procedure for conducting the friction and wear tests was as follows: a rider and a burnished disk (lubricant was not applied to the riders) were inserted into the friction apparatus (fig. 1). The test chamber was sealed and purged with either dry argon (10 ppm H_2O), dry air (20 ppm H_2O), or moist air (10 000 ppm H_2O) for 15 minutes before starting the test. The flow rate was 1500 cubic centimeters per minute. This flow rate maintained a slight positive pressure in a chamber whose volume was 2000 cubic centimeters.

After the purge was completed, the desired temperature was obtained by induction heating. The disk was then set into rotation at 1000 rpm and a 1-kilogram load was applied.

The criterion for failure in these tests was a friction coefficient of 0.3. An automatic cutoff switch shut down the apparatus when the friction coefficient reached 0.3.

The wear scar diameter on the hemispherically tipped rider was measured after each test and wear volume calculated. Rider wear volume per meter of sliding was then determined.

RESULTS

Wear Life of Hand-Burnished Films

The first series of tests on the effect of fluorine was conducted using hand-burnished graphite fluoride films. These films were applied in room air with no control of relative humidity (RH varied from 25 to 60 percent). Eight different graphite fluoride powders with fluorine to carbon

ratios of 0.25, 0.33, 0.5, 0.6, 0.7, 0.9, 1.0, and 1.1 were examined. Burnished films of natural (Madagascar) graphite was also tested for comparison purposes. To determine atmospheric effect, each burnished film was tested in atmospheres of dry argon (10 ppm, H_2O), dry air (20 ppm, H_2O), and moist air (10 000 ppm, H_2O).

Figure 3 presents the results of wear life tests on these burnished films. The failure criterion was arbitrarily set at a friction coefficient of 0.3. Figure 3 shows that the wear life for the tests conducted in moist air or dry air increased with increasing fluorine to carbon ratio (x). In dry argon, however, the fluorine to carbon ratio had a somewhat different effect on wear life. Wear life increased up to a value of $x = 0.6$ and then leveled off. Above $x = 0.6$ the wear life was about double that at $x = 0.25$. Figure 3 also indicates that moisture in air was beneficial in providing longer wear lifes. It is important to note that, although moisture in air is beneficial to lubrication with $(CF_x)_n$, it is not essential as it is with graphite.

Wear Life of Machine-Burnished Disks

Hand-burnishing is a convenient method for applying a solid lubricant film, but, it is not a very precise method of application. Such variables as amount of lubricant, application pressure, burnishing atmosphere, burnishing time, etc., are not easy to control. In order to control burnishing conditions, a burnishing apparatus was designed (fig. 2). The following burnishing conditions were adhered to: lubricant amount, approximately 1/2 gram; burnishing load, 2 kilograms; burnishing speed, 15 rpm; and burnishing atmosphere, moist air (10 000 ppm H_2O ; approximately 50 percent

relative humidity).

Figure 4 compares the results of tests on the machine-burnished disks with those on hand-burnished disks. As can be seen from the figure, burnishing technique is extremely important in determining the wear life of the films. Burnishing technique, in fact, seems more critical in determining the wear life of the various graphite fluoride films than does the fluorine to carbon ratio. For example, in all three test atmospheres (dry argon, dry air, and moist air) the wear life of machine-burnished $(CF_{0.5})_n$ is greater than hand-burnished $(CF_{1.1})_n$. Some possible explanations for this will be discussed later.

Several additional observations can be made from the data of figure 4. First, it is evident that any amount of fluorine in graphite fluoride improved the wear life results of graphite. Even a fluorine to carbon ratio as small as $x = 0.25$ doubled the wear life. Taking into consideration data from all three test atmospheres, however, it seems advisable, for best wear life results, to use a graphite fluoride powder with a fluorine to carbon ratio of $x = 0.5$ or higher. As with the hand-burnished films, the longest wear lives for the machine-burnished films were obtained in the moist air test atmosphere. The dry air test atmosphere gave the next longest wear lives with the dry argon test atmosphere giving the shortest lives.

Friction Coefficient

Friction coefficient as a function of fluorine content is presented in figure 5. Friction coefficient for graphite fluoride films is time dependent. Usually, at the start of each test, there is a run-in period

where the friction coefficient can attain a value as high as 0.2. The length of this run-in time varies but is usually less than 5 minutes, after which the friction coefficient falls to some minimum value. The friction coefficient stays at this minimum value for a period of time and then gradually increases with time until the cutoff friction coefficient of 0.3 is reached. The friction coefficient values presented in figure 5 are the minimum values obtained for each test.

The method used to burnish the graphite fluoride films did not affect the minimum friction coefficients obtained. Thus the data points in figure 5 represent results from both hand-burnished and machine-burnished films. The type of atmosphere seemed to affect the friction coefficient more than the fluorine to carbon ratio. The friction coefficient in a moist air test atmosphere was 0.07 for all graphite fluoride samples. The friction coefficient in dry air or dry argon was about 0.03. By comparison, the friction coefficient for graphite in moist air was 0.10 and in dry air and dry argon it was 0.15.

It is of interest to know how the friction coefficient varied during the life of the films. Tables I and II give the numbers of revolutions (in kilocycles) that elapsed before the friction coefficient reached values of 0.1, 0.2, and 0.3 for the hand-burnished and machine-burnished films, respectively. It is evident that burnishing technique and test atmosphere play important roles in influencing the friction coefficient over the life of the films.

Rider Wear Rate

After each test the wear scar diameter on the hemispherically tipped

rider was measured. From this, wear volume per meter of sliding was calculated. Figure 6(a) presents results from the hand-burnished films, and figure 6(b) from the machine-burnished films. Figure 6 indicates that the wear rate for riders sliding on burnished graphite fluoride films was not strongly dependent on the fluorine to carbon ratio (x) of $(CF_x)_n$. The rider wear rate with burnished $(CF_x)_n$ films, however, was considerably less than the rider wear rate with burnished graphite films. In all instances but one, the machine-burnished films provided lower wear rates than the hand-burnished films. Burnishing technique was thus influential in determining rider wear rate as well as film wear life.

Test atmosphere also influenced rider wear rate. The lowest wear rates were obtained in dry argon, and in most cases a higher wear rate was obtained in moist air than in dry air.

Effect of Burnishing Atmosphere

To determine the effect of moisture in the burnishing atmosphere, graphite fluoride films were burnished onto disks in dry air (20 ppm H_2O) instead of moist air (10 000 ppm H_2O). Three different $(CF_x)_n$ films with fluorine to carbon ratios (x) of 0.6, 0.9, and 1.1 were used for these experiments.

Figure 7 compares the wear life results of the films burnished in dry air to the films burnished in moist air. Each film was tested in atmospheres of moist air, dry air, and dry argon. Compared with the films burnished in moist air, the wear lives of films burnished in dry air were considerably shortened. The effect is especially marked in a moist air test atmosphere, where wear lives of the dry air burnished films were only one-fourth as long

as those of moist air burnished films. In a dry air test atmosphere, wear life was reduced in half and in a dry argon test atmosphere the reduction was about 30 percent. Thus the atmosphere in which films are burnished has a significant influence on wear life of graphite fluoride films. This will be discussed in more detail in a later section.

Effect of Temperature

To determine the effect of fluorine to carbon ratio on the lubricating properties of graphite fluoride at high temperatures, a series of experiments were conducted at 300° C. Figures 8 and 9 give the results of those tests.

Figure 8(a) presents the wear lives of graphite fluoride films machine-burnished in moist air and tested in a dry air atmosphere (20 ppm H₂O) at 300° C. As fluorine content is increased, wear life is increased. Thus there is an advantage in using the more highly fluorinated forms of graphite fluoride at high temperatures. Useful life, however, was obtained with fluorine to carbon ratios as low as 0.33. In general, wear life at 300° C was reduced to one-seventh its value at 25° C.

The minimum value for the friction coefficient did not seem to be affected noticeably by fluorine content or by the increased temperature. The friction coefficient for the various graphite fluoride films was about 0.04 (fig. 8(b)).

Figure 9 presents rider wear rates at 300° C in dry air as a function of fluorine content. Fluorine content did not noticeably affect rider wear rate at 300° C in dry air; however, compared with similar tests at room temperature (25° C), rider wear rate has increased about an order of magnitude.

Table III gives the number of revolutions (in kilocycles) that elapsed before the friction coefficient reached values 0.1, 0.2, and 0.3 for the tests conducted at 300° C in dry air. The general trend is that as fluorine content is increased, the number of kilocycles to reach 0.1, 0.2, or 0.3 is also increased.

Thus it appears that wear life of burnished graphite fluoride films is influenced by the following factors: (1) the burnishing technique used, (2) the atmosphere in which the film was burnished, (3) the atmosphere in which the test was run, and (4) the temperature at which the test was run.

Film Appearance

Since wide variations in wear lives were obtained simply by varying the burnishing technique, a few burnished films were examined by taking photomicrographs and surface profiles. Figure 10 presents these data for graphite fluoride films $(CF_x)_n$ which were machine-burnished in moist air (10 000 ppm H_2O). A film of Madagascar graphite and a sandblasted 440-C stainless steel disk are shown for comparison.

Figure 10 illustrates how the fluorine to carbon ratio influenced film formation. Not much graphite has adhered to the roughened 440-C stainless steel. However, a considerable amount of graphite fluoride is present, even for a 0.25 carbon to fluorine ratio.

It is not immediately obvious from the photographs of figure 10 but the graphite fluoride powder appears to be "flowing." Perhaps machine-burnishing the films causes some ordering on the surface. The higher the fluorine to carbon ratio, the smoother the films appeared. The surface roughness for

a disk burnished with $(\text{CF}_{0.25})_n$ was 1.27 micrometers rms and decreased with increasing fluorine content, until for $(\text{CF}_{1.1})_n$ the surface roughness was 0.53 micrometer rms.

A few general statements can be made about film forming characteristics of graphite fluoride. In the most highly fluorinated state $[\text{CF}_{1.1})_n]$, graphite fluoride is not very cohesive. The powder flows (almost like a liquid) when it is contained, and does not compact very well even when using extreme pressure. The material does seem to have good adhesiveness, adhering very strongly to metal surfaces. In an attempt to make powder compacts, the graphite fluoride adhered to the metal plunger of the press and, when the compact was removed, the compact broke internally instead of at the metal interface.

Photomicrographs and surface profiles (fig. 11) were taken for films hand-burnished in room air with fluorine to carbon ratios of 1.1, 0.7, 0.5 and 0.33. The hand-burnished film (fig. 11) and machine-burnished film (fig. 10) for $(\text{CF}_{1.1})_n$ look very similar, except that the machine-burnished film is smoother (rms 0.53 compared with rms 0.83 μm). The other three hand-burnished films ($(\text{CF}_{0.7})_n$, $(\text{CF}_{0.5})_n$, and $(\text{CF}_{0.33})_n$) look much different from the machine burnished films, however. The "flowing" nature and the buildup that was seen with machine-burnished films is absent. Thus, the most likely reason for the increased wear lives obtained with the machine-burnished films is that thicker, more dense films were obtained.

Figure 12 gives photomicrographs and surface profiles for three graphite fluoride films ($(\text{CF}_{0.6})_n$, $(\text{CF}_{0.9})_n$, and $(\text{CF}_{1.1})_n$), which were burnished in a dry air atmosphere (20 ppm H_2O). There is not much difference in the

appearance of the $(CF_{1.1})_n$ films machine-burnished in dry air or moist air. For $(CF_{0.6})_n$ and $(CF_{0.9})_n$, however, there is much more of a difference. The surfaces of these two dry air burnished disks have a streaky appearance, lacking the "flowing" nature of the moist air burnished films, and are not as dense or as thick. The presence of water vapor in air apparently improves the film forming properties of $(CF_x)_n$.

The formation of thicker, more dense solid lubricant films in a moist air burnishing atmosphere is not unique to graphite fluoride; Johnston and Moore (15) made the same observation with MoS_2 films. Using X-ray fluorescence techniques, they found that, by increasing the relative humidity from 6 to 85 percent, the film density of burnished MoS_2 films could be increased by a factor of 7 to 8.

SUMMARY OF RESULTS

The effect of fluorine to carbon ratio on the friction, wear and wear life properties of burnished graphite fluoride $(CF_x)_n$ films was studied using various experimental and burnishing conditions. The results indicate that:

1. The wear life of burnished graphite fluoride films is influenced by (a) the burnishing technique, (b) the atmosphere in which the film was burnished, (c) the atmosphere in which the test was conducted, and (d) the temperature at which the test was conducted.

2. In air (moist or dry), wear life of burnished graphite fluoride films increased with increasing ratio of fluorine to carbon. In dry argon, wear life increased up to a value of $(CF_{0.6})_n$, then leveled off.

3. Minimum friction coefficient and rider wear rate were not strongly influenced by the degree of fluorination.

4. Longer wear lives were obtained for films tested in moist air than in dry air or dry argon; however minimum friction coefficients and rider wear rates were higher.

5. In a dry air test atmosphere, similar lubrication trends were found at 25° C and at 300° C; however wear life was 4 to 8 times shorter at 300° C and rider wear rate was about 10 times greater.

6. In general, better lubrication results were obtained when graphite fluoride films were applied by machine-burnishing than by hand-burnishing.

7. Longer wear lives were obtained when the films were applied in moist air as compared to dry air.

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TABLE I. - HAND-BURNISHED FILM LIFE AT 25° C

[Disks burnished in room air (relative humidity range 25 to 60 percent); 440-C stainless steel riders and disks; load, 1 kg; sliding speed, 2.6 m/sec.]

Fluorine to carbon ratio	Number of revolutions (kilocycles of sliding) for friction coefficient to reach a value of -								
	0.1	0.2	0.3	0.1	0.2	0.3	0.1	0.2	0.3
	Test atmosphere								
	Moist air (10 000 ppm H ₂ O)			Dry air (20 ppm H ₂ O)			Dry argon (10 ppm H ₂ O)		
1.1	37	387	473	9	142	301	10	152	230
1.0	12	462	525	23	175	320	30	110	198
.9	13	305	408	32	170	200	10	53	232
.7	12	330	336	32	113	193	40	91	222
.6	6	230	250	12	80	143	75	136	203
.5	15	200	228	2	36	100	20	59	180
.33	2	75	100	6	33	96	11	49	115
.25	0	85	102	0	0	49	1	20	71
Graphite	0	16	44	--	---	---	--	---	---

TABLE II. - MACHINE-BURNISHED FILM LIFE AT 25° C

[Disks burnished in moist air (10 000 ppm H₂O); 440-C stainless-steel riders and disks; load, 1 kg; sliding speed, 2.6 m/sec.]

Fluorine to carbon ratio	Number of revolutions (kilocycles of sliding) for friction coefficient to reach a value of -								
	0.1	0.2	0.3	0.1	0.2	0.3	0.1	0.2	0.3
	Test atmosphere								
	Moist air (10 000 ppm H ₂ O)			Dry air (20 ppm H ₂ O)			Dry argon (10 ppm H ₂ O)		
1.1	100	750	1140	75	240	340	40	130	200
1.0	110	890	930	55	168	590	20	125	190
.9	64	1127	1130	60	220	570	56	140	275
.7	30	630	690	75	160	505	42	248	286
.6	23	910	947	140	255	400	65	128	304
.5	40	582	690	95	220	410	85	190	300
.33	25	505	624	40	210	260	28	52	132
.25	17	38	108	13	32	88	7	45	93
Graphite	0	15	55	0	1	11	0	1	2

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TABLE III. - MACHINED-BURNISHED FILM LIFE AT

300° C IN DRY AIR TEST ATMOSPHERE (20 ppm H₂O)

[Disks burnished in moist air (10 000 ppm H₂O);
440-C stainless-steel riders and disks; load,
1 kg; speed, 2.6 m/sec.]

Fluorine to carbon ratio	Number of revolutions (kilocycles of sliding) for friction coefficient to reach a value of -		
	0.1	0.2	0.3
Graphite	1	2	9
.25	5	19	20
.33	8	38	47
.5	12	38	54
.6	12	40	60
.7	8	40	65
.9	12	57	80
1.0	22	45	77
1.1	38	60	86

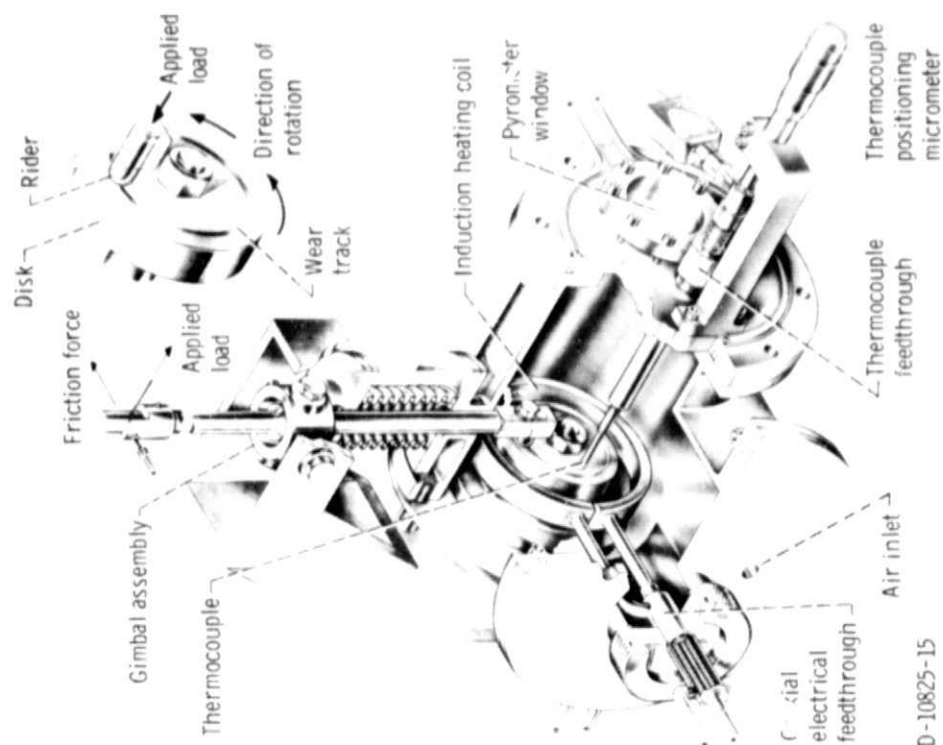


Figure 1. - Friction and wear apparatus.

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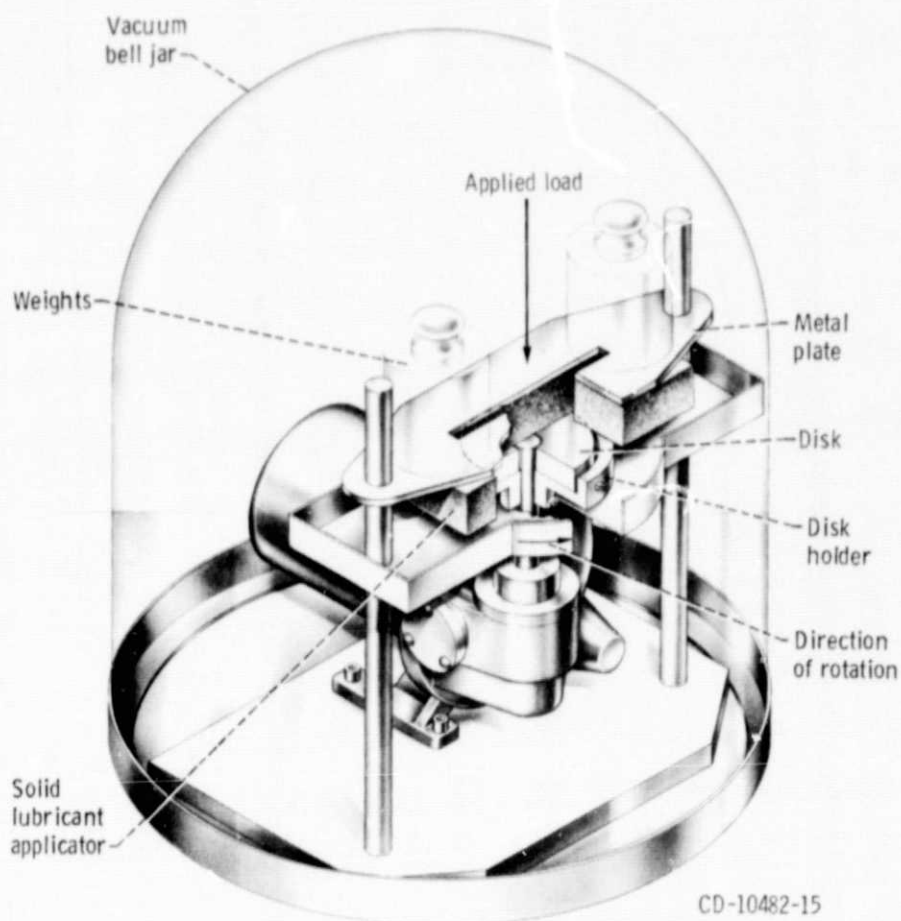


Figure 2. - Apparatus used for machine-burnishing.

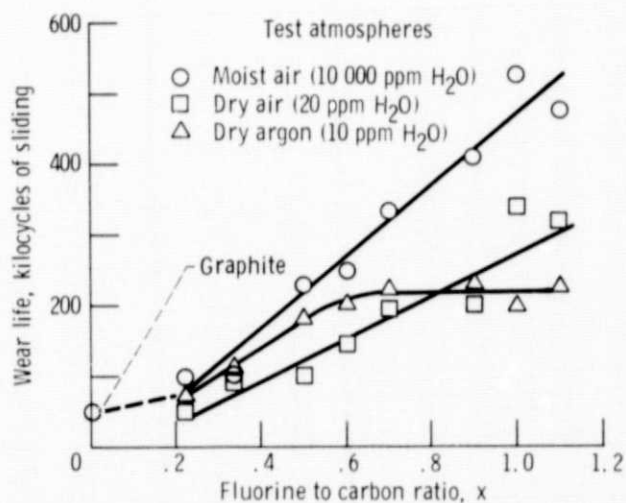


Figure 3. - Effect of fluorine content and test atmosphere (at $25^{\circ}C$) on the wear life of hand-burnished graphite fluoride $(CF_x)_n$ films.

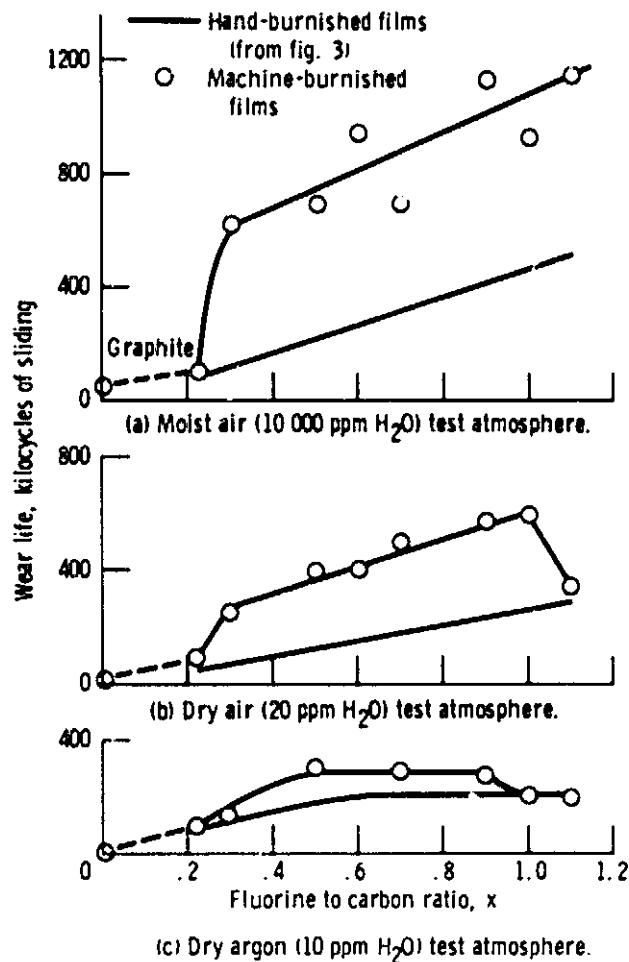


Figure 4. - Effect of burnishing technique, fluorine content, and test atmosphere (at 25° C) on the wear life of burnished graphite fluoride (CF_x)_n films.

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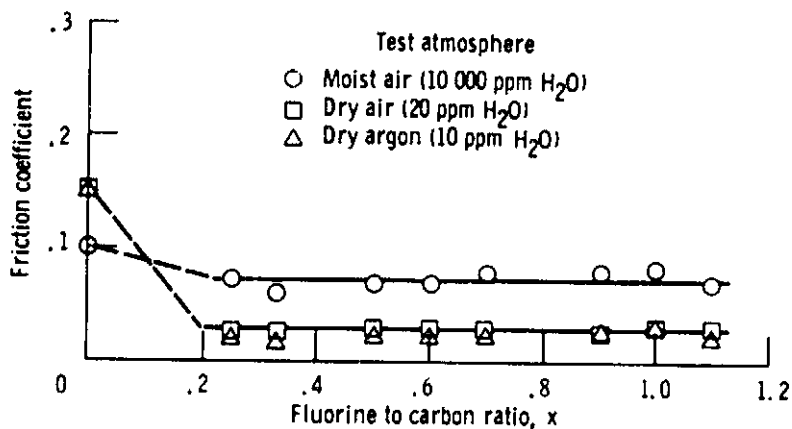


Figure 5. - Minimum friction coefficient (at 25° C) for hand- and machine-burnished graphite fluoride (CF_x)_n films as a function of fluorine content.

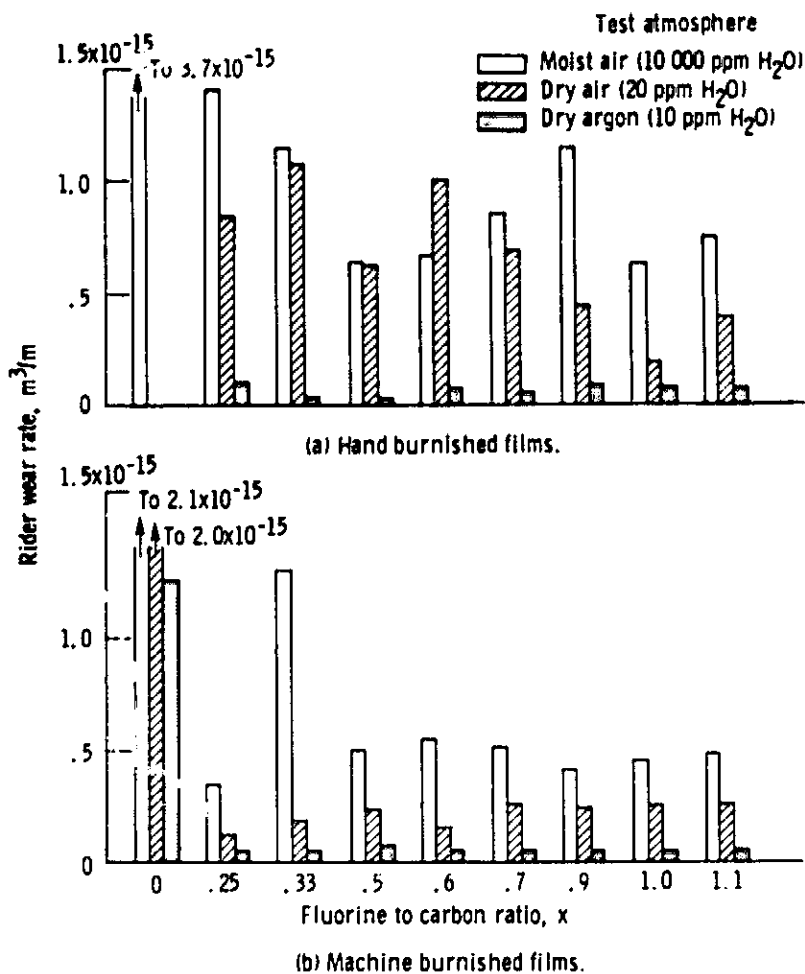


Figure 6. - Effect of fluorine content, test atmosphere, and burnishing technique (at 25° C) on the wear rates of 440C stainless steel riders sliding on burnished graphite fluoride (CF_x)_n films.

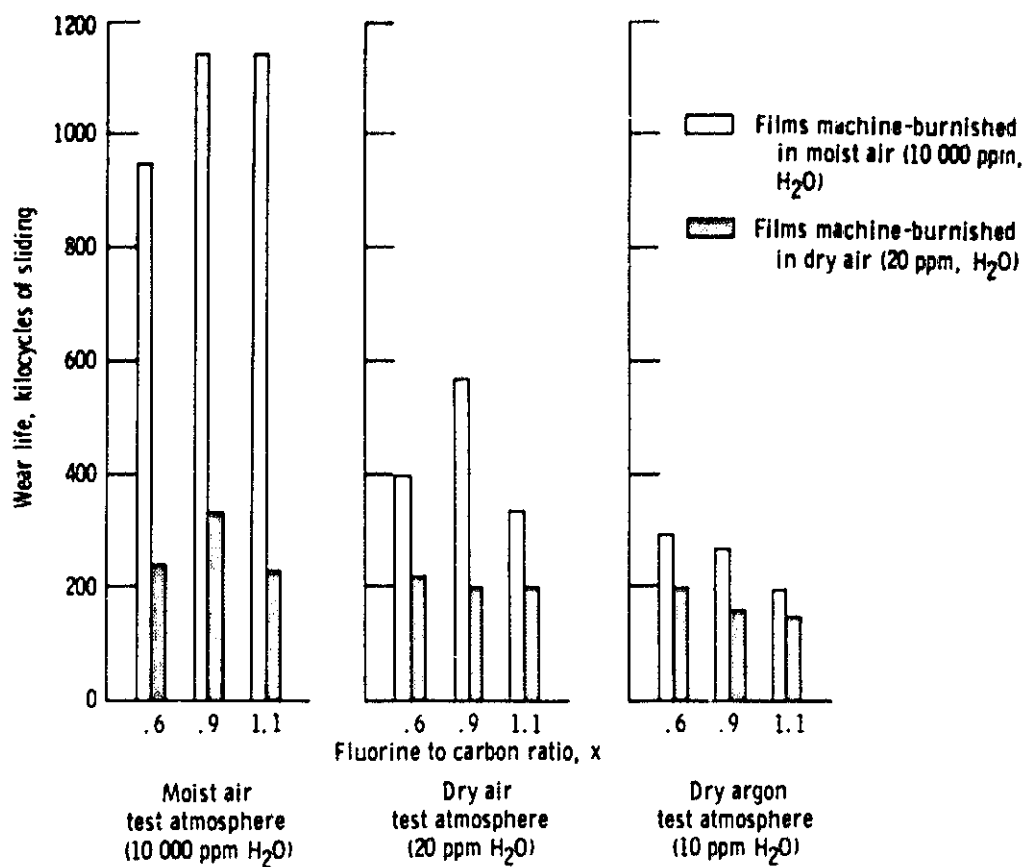
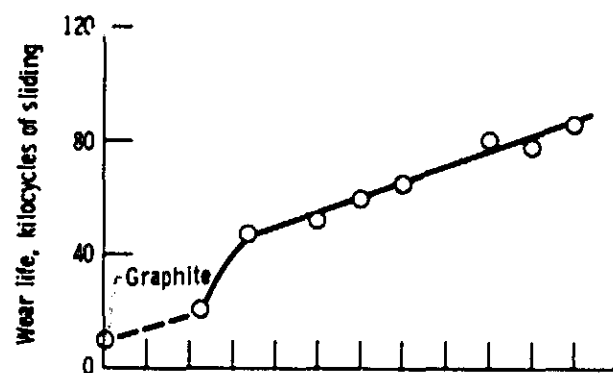
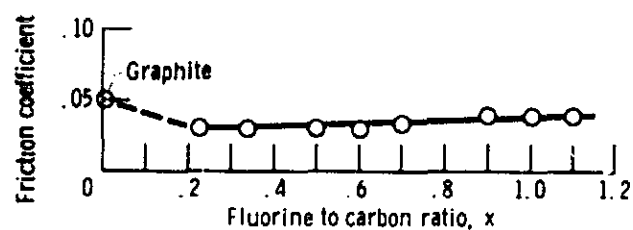


Figure 7. - Effect of burnishing atmosphere on the wear life (at 25° C) of graphite fluoride ($CF_x)_n$ films.

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(a) Wear life at 300°C.



(b) Friction coefficient at 300°C.

Figure 8. - Wear life and minimum friction coefficient (at 300°C) in dry air as a function of graphite fluoride $(CF_x)_n$ fluorine content.

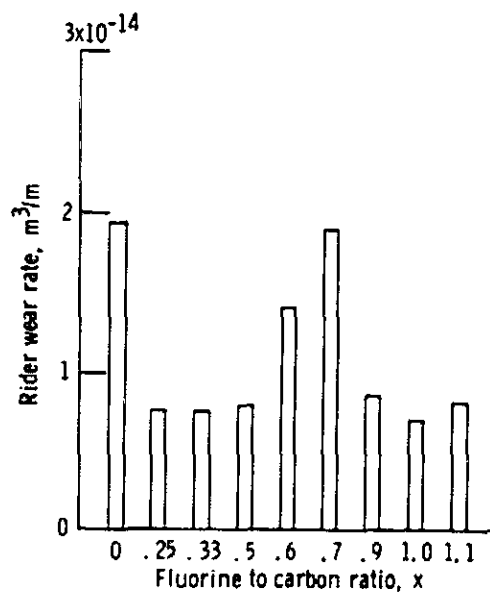


Figure 9. - Rider wear rate at 300°C in a dry air test atmosphere (20 ppm H_2O) as a function of graphite fluoride $(CF_x)_n$ fluorine content.

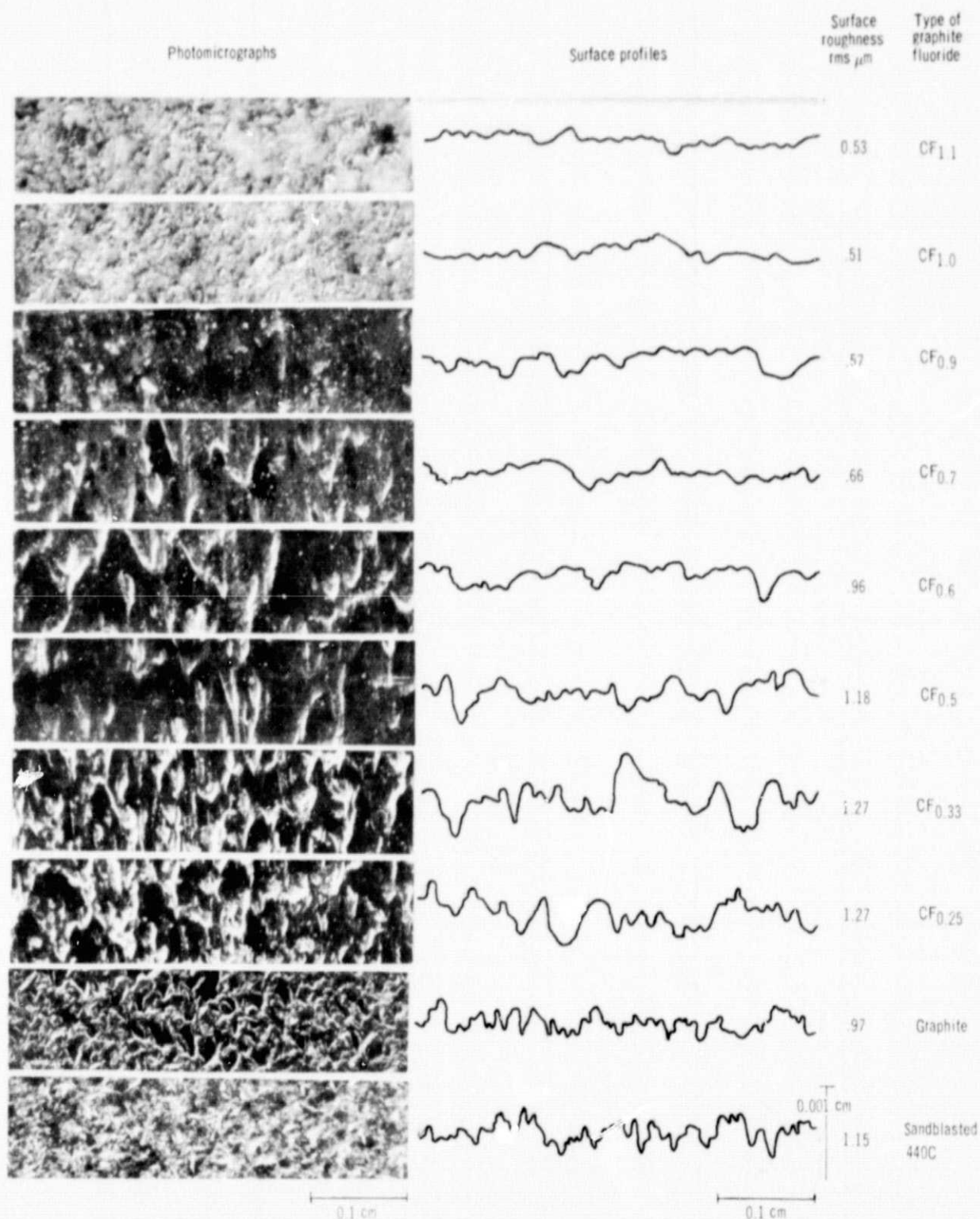


Figure 10 - Effect of fluorine to carbon ratio on film forming properties of machine burnished graphite fluoride (CF_x)_n. Disks burnished in moist air (10 000 ppm H₂O); load, 2 kilograms; sliding speed, 15 rpm; time of burnishing, 60 minutes.

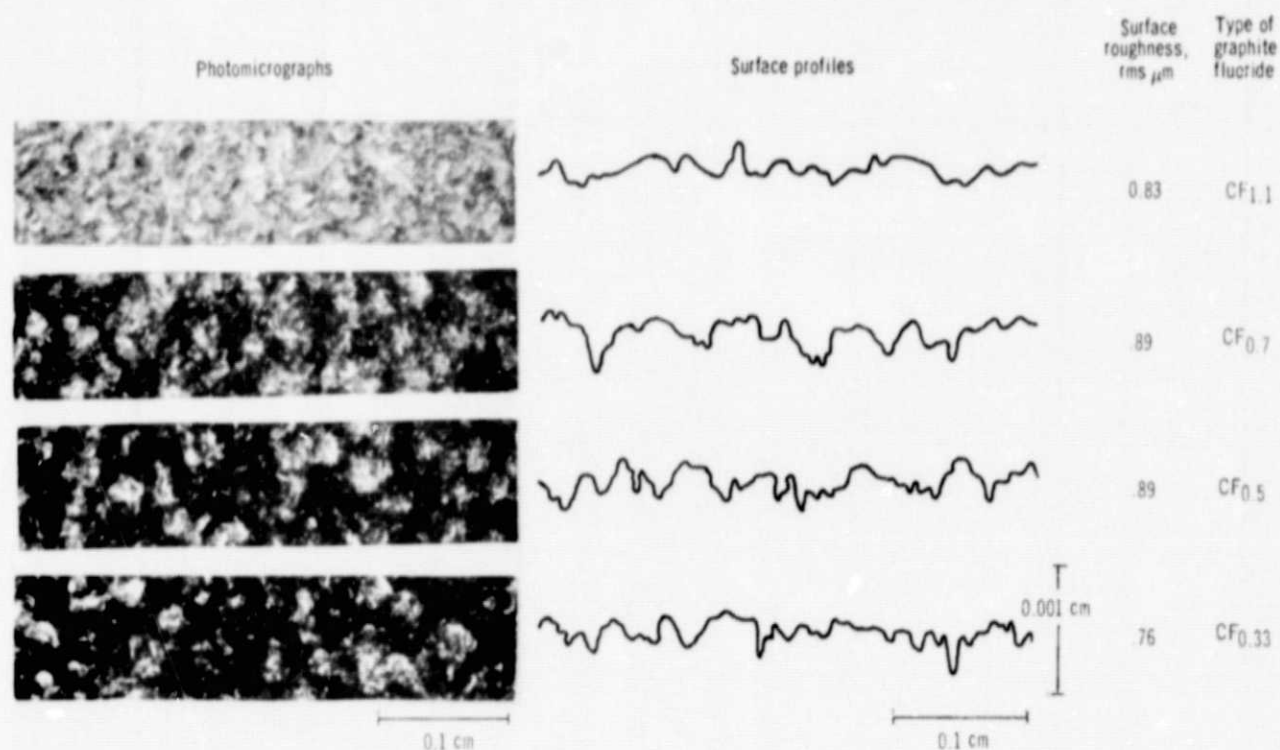


Figure 11. - Effect of fluorine to carbon ratio on film forming properties of hand burnished graphite fluoride (CF_x)_n burnished in room air (relative humidity range, 25 to 60 percent).

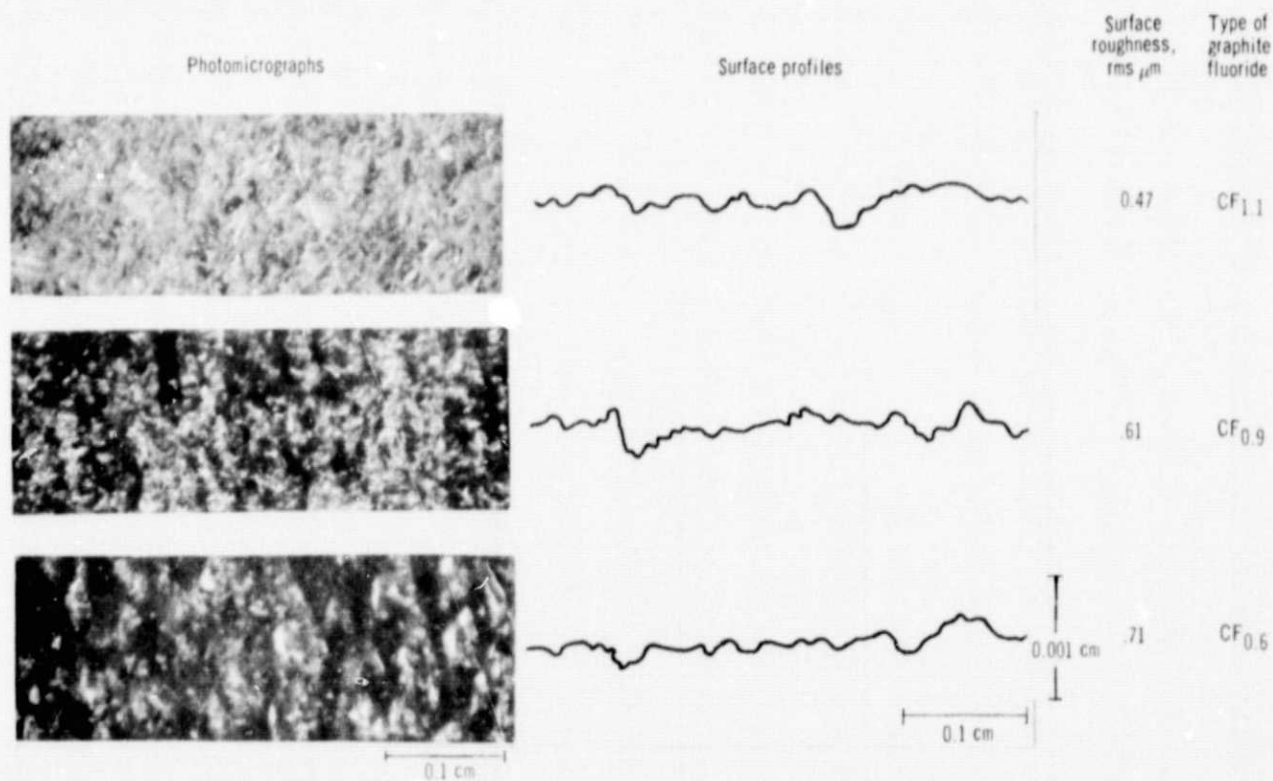


Figure 12. - Effect of a dry air burnishing atmosphere (20 ppm H₂O) on film forming properties of machine burnished graphite fluoride (CF_x)_n. Time of burnishing, 60 minutes; load, 2 kilograms; rotational speed, 15 rpm.