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FRICTION STUDIES OF GRAPHITE AND MIXTURES OF GRAPHITE WITH SEVERAL METALLIC OXIDES AND SALTS AT TEMPERATURES TO 1000° F

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SUMMARY

An experimental friction study was made using graphite and mixtures of graphite with lead oxide, cadmium oxide, sodium sulfate, or cadmium sulfate as solid lubricants. Runs were made at temperatures up to 1000° F with various steel and Inconel combinations.

Graphite powder lubricated metal surfaces at temperatures sufficiently high to promote oxidation of the metal surfaces. At intermediate temperatures graphite powder alone failed to function as a lubricant. Graphite would not lubricate cast Inconel on Inconel X at temperatures between approximately 150° and 800° F. Similarly, with steel, which oxidized more readily than Inconel, the temperature below which graphite would not lubricate was 475° F as compared with 800° F for Inconel.

Mixtures of metallic compounds and graphite were effective lubricants from room temperature to 1000° F provided no reaction occurred between the metallic compounds and the graphite. A metallic salt mixed with graphite showed friction coefficients similar to those of a resin-bonded film of graphite.

INTRODUCTION

Using solids as lubricants offers a method of reducing friction and wear between moving metal surfaces at high temperatures (e.g., 1000° F) where liquid lubricants are unsuitable (ref. 1). Graphite is generally used at these temperatures. For example, experience with rolling contact bearings (ref. 2) showed dry graphite powder to be an effective lubricant at temperatures of 1000° F. However, many of the factors which affect its lubricating characteristics are not completely understood and further investigations are desirable.

For graphite brushes, adsorbed water vapor and oxygen are shown to be essential in order to ensure low friction and wear (refs. 3 to 5).

Incorporation of organic liquids and inorganic solids had a similar effect (ref. 6).

Studying various inorganic solids as lubricants (ref. 7) showed their effectiveness to be associated with the formation of adherent films on the lubricating surfaces. This would imply that a method of increasing the adherence of a solid, such as graphite, to a metal might enhance its lubricating ability. One method of increasing the adherence of graphite might be to mix it with a soft metallic oxide or salt. The soft compound would be mechanically held in the recesses on the metal surface or, possibly more important, either react chemically or alloy at its interface with the metal surface. In other words, such compounds would serve as bonding media for the graphite.

The research reported herein was conducted to determine some of the characteristics of graphite and graphite mixed with several metallic salts and oxides as high-temperature solid lubricants, and also to study several of the factors that affect graphite lubrication at high temperatures.

Friction studies were made at temperatures up to 1000° F, at a low sliding velocity (5.7 ft/min), and with a load of 40 pounds supported on three 3/16-inch-radius hemispherical contacts. Various metals, including Inconel and tool steel, were lubricated by synthetic graphite and by mixtures of graphite with oxides of lead and cadmium as well as sulfates of sodium and cadmium.

MATERIALS, APPARATUS, AND PROCEDURE

Lubricants

The solid lubricants used in these experiments were graphite, lead oxide, sodium sulfate, cadmium sulfate, and cadmium oxide. They were all used as fine powders with particle sizes generally finer than 200 mesh. The graphite was a high-purity electric-furnace synthetic graphite. For several runs the graphite was dried by heating to 1500° F in a vacuum (<1 μ of Hg.) for 4 hours in order to determine whether this affected the results. The lead oxide was predominantly PbO although X-ray diffraction analysis indicated contamination with Pb_3O_4. Sodium sulfate and cadmium sulfate were "chemically pure" materials; cadmium oxide was a "technical grade" material.

Test Specimens

The solid lubricants were used to lubricate metal slider specimens. For most experiments the specimens consisted of cast Inconel rider specimens sliding on an Inconel X disk specimen (2.5-in. diam., 0.5-in. thickness). The rider specimens were cylindrical (3/8-in. diam. and

3/4-in. length) and had a hemispherical tip (3/16-in. rad.) ground on one end. The test surface of the disk was ground flat and then vapor-blasted to give a nondirectional surface finish. The materials of the test specimens were varied in some of these experiments, and where applicable they will be specifically described.

Apparatus

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The apparatus is shown diagrammatically in figure 1(a). In this investigation, three hemispheres were slid against a flat disk in a circumferential path. The hemispheres were mounted in a holder on which vanes had been added in such a way that the graphite would be continually returned into the path of the sliders (fig. 1(b)). The slider holder was clamped in the jaws of a spindle. The spindle could be raised, lowered, or locked in any position. The disk was placed on a support table and kept from rotating by means of a pin in the support table that meshed with a hole in the edge of the disk. A heater was mounted under the support table. A ball bearing in the base of the apparatus allowed free movement and had a negligible effect on the total friction force. The friction force was measured by restraining the lower assembly through a dynamometer ring on which strain gages were mounted. Temperatures were measured by a thermocouple mounted on the bottom of the disk.

The support table rested on three springs, which compensated for minor misalinement of the sliders and disk. The load was applied by lowering the spindle and compressing the base springs. The magnitude of the load was indicated by a dial gage, measuring spring compression, which had previously been calibrated using dead weights.

Cleaning Procedure

Prior to each run, both the disk and the slider specimens were cleaned according to the following procedure:

(1) Washing in a solution of 50 percent acetone and 50 percent benzene

(2) Scrubbing with repeated applications of moist levigated alumina

(3) Washing in tap water

(4) Washing in distilled water

(5) Washing in 95-percent ethyl alcohol

(6) Drying in a stream of warm air and storing in a dessicator

Test Procedure

The procedure used in these experiments was varied in different series of runs. Experience indicated that control of humidity was not critical in runs at high temperatures. Most of the data reported herein were obtained at temperatures from 400° to 1000° F. All runs were made with a sliding velocity of 5.7 feet per minute and at a total load of 40 pounds. The temperatures reported are those of the disk specimen. 3922

Unless otherwise specified, approximately 20 grams of the powdered solid lubricant was placed on the disk specimen prior to operation. The apparatus was heated to the desired operating temperature before running, which took approximately 2 hours. The load was applied when the run was started. In runs made at stated temperature levels the duration of test was usually 30 minutes. Friction force was measured continually and was recorded every 5 minutes, or more often when friction force was changing. Several of the runs included temperature cycling. A number of the runs were started at the maximum temperature (as high as 1000° F) and the apparatus was allowed to cool. Sliding was continued unless lubrication failure was experienced. After cooling to some minimum temperature the temperature was increased during sliding until the original maximum temperature level was attained. Usually this cooling and heating cycle was repeated several times with the same specimen and lubricant. In some other cases initial operation was at room temperature and thereafter the temperature level was increased. Because of the varied methods of experimentation, the specific procedure employed will be described in each case as the results are presented.

In preliminary testing, when the graphite was not returned to the sliding track using the vanes the amount of graphite initially placed on the disk was a critical factor. When large amounts of graphite (approx. 3/8 in. thick) were placed on the disk, the friction coefficient values were the same as when the graphite was returned to the track. With small amounts of graphite (1/16 in. thick) the friction-time curves were erratic and not reproducible. This erratic behavior was caused by variations in the amount of metal contact that took place as the graphite film was worn from the surface. Thus, in the experiments described herein the graphite was returned to the track using the vanes.

RESULTS AND DISCUSSION

Graphite

Tests were run at 1000° F with cast Inconel sliders and an Inconel X disk. The friction-time curves for graphite are shown in figure 2. The band represents the variation in friction coefficient obtained from five tests with commercially pure graphite. The friction coefficient was initially low (0.05 to 0.1) and then increased to a rather steady value (0.12 to 0.16). There was a film of graphite on both the sliders and the disk, which, however, was not continuous. The metal contact that resulted probably caused the increase in friction after several minutes of sliding. Figure 2 also shows data for graphite that had been dried by vacuum heating. Because the vacuum heating had no effect on friction, commercial graphite was used without special treatment in subsequent runs.

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Tests were also made at 650° F with cast Inconel sliders and Inconel X disks. However, at this temperature, graphite would not form a lubricating film; high friction and severe galling took place. The obvious difference between the 650° and 1000° F runs was in the amount of oxide present on the metal surfaces in the 1000° F run. Therefore, the hypothesis was drawn that this increase in the amount of oxide present was aiding the graphite lubrication.

In order to locate the critical temperature between 650° and 1000° F, the following procedure was followed: after 30 minutes running with graphite at 1000° F, the temperature was lowered while sliding continued until failure occurred; then the specimens were heated until the friction coefficient returned to its original value. These data for cast Inconel sliding against Inconel X are shown in figure 3(a). The friction coefficient was nearly constant (0.15) until failure occurred. The failure temperature was not readily reproducible but varied with different tests from 450° to 600° F. When the specimens were heated the friction coefficient decreased to its original value at approximately 800° to 850° F. It was possible to repeat this cycle with the same set of test specimens; however, the friction values became more erratic after each cycle. These data show that graphite would not lubricate cast Inconel against Inconel X below 800° F without prerunning at a higher temperature.

In order to determine whether the change in the friction coefficient at high temperatures was due to a change in the graphite itself or a function of the surfaces, identical tests were run with different slider and disk combinations. The data for graphite lubricating Inconel X sliding against Inconel X, SAE 1095 steel sliding against SAE

4340 steel, M-1 tool steel sliding against M-1 tool steel, and cast Inconel sliding against Inconel X are shown in figure 3. The SAE 1095 - SAE 4340 combination was run at 550° F rather than 1000° F in order to avoid annealing the test specimens.

The friction coefficients for the Inconel X combinations were higher than for cast Inconel - Inconel X combination (fig. 3(b)). Failure took place at about 650° F in the decreasing temperature portion of the cycles; recovery during heating took place between 800° and 850° F. This is the same temperature of recovery that was found for the cast Inconel - Inconel X combination. 3922

When SAE 1095 steel sliders were used against an SAE 4340 steel disk the friction coefficient was approximately 0.07 at 550° F (fig. 3(c)). As the temperature was lowered, friction increased slightly until complete failure occurred at 450° F. Failure was accompanied by severe galling and tearing of the surfaces. Friction began to decrease almost immediately as the temperature was increased again.

For the M-1 tool steel combinations (fig. 3(d)) the friction coefficients at 900° F were lower than for the Inconel combinations. Upon cooling, friction began to increase at 550° F. Failure did not take place rapidly with further cooling but the friction coefficient increased with decreasing temperature. As the temperature was increased again the friction coefficients decreased along the same curve obtained during cooling. Severe galling of the surfaces did not take place with this combination as was characteristic of the failure of the other combinations.

The results included in figure 3 show that graphite lubricates steel at a lower temperature than it does Inconel. Since steel oxidizes more readily than Inconel, this result indicates the importance of a sufficient amount of oxide for graphite to lubricate above room temperature. The presence of an oxide did not always ensure adequate lubrication with graphite; for example, when cast Inconel was slid on a wrought Inconel disk, which is softer than Inconel X, (data not shown) the friction coefficient was 0.41 after 5 minutes sliding at 1000° F. Severe galling of both sliders and disk had taken place. The reason for this anomaly may be explained by considering the relative physical properties of the oxide and substrate material (refs. 8 to 10) as well as the strength of the oxide attachment to the surface. A brittle oxide on a soft metal (e.g., Inconel), or one that is loosely attached, may be easily removed from the surface. Graphite attached to such an oxide would not provide adequate protection. If a soft oxide is present on a hard surface, extremely heavy loads may be necessary to deform the surface enough to rupture the graphite and oxide film.

Reaction films on the base metal in conjunction with a lubricant have been shown to be beneficial; for example, molybdenum disulfide on phosphated surfaces (ref. 11), graphite on phosphated surfaces (ref. 12), and lubricating oils on oxidized surfaces (ref. 13). The role of the oxide film in the present experiment may be essentially the same as that reported in references 11 to 13. However, one important difference is apparent: during sliding at high temperatures the oxide film could be reformed to replace the film that was worn away.

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The precise role of the oxide film in lubrication with graphite could not be determined from these experiments. However, to be an effective lubricant, a solid must be firmly attached to the surface. It is possible that the oxide acts in this capacity; that is, the graphite may adhere to the oxide better than to the unoxidized metal.

Mixtures of Graphite and Metallic Compounds

In order to provide a binder for the graphite, some soft metallic oxides and sulfates ($66\frac{2}{3}$ percent by weight) were added to graphite; tests were then run to determine the lubricating effectiveness of the mixture for the cast Inconel - Inconel X combination. Two types of test were run. In one test the mixture was "run-in" for 30 minutes at 1000° F, then the temperature was decreased to a lower level and the friction noted for 5 minutes of sliding. This procedure was followed until room temperature was reached. In other tests the same procedure was followed except that the combination was run-in at room temperature and the temperature was increased. The material was returned to the sliding track in all tests.

The data for graphite, lead oxide, and a mixture of lead oxide and graphite are shown in figure 4. The curves for the mixture can be compared with graphite and lead oxide alone. For graphite (fig. 4(a)), low-temperature data was obtained. Friction values increased considerably between 100° and 150° F. For lead oxide the friction coefficient is higher than 0.42 at room temperature and decreases to a value of 0.15 at 1000° F. Upon cooling, the friction coefficients were higher but galling failure did not take place. These data indicate that lead oxide alone may be an effective lubricant at 1000° F. The friction coefficients for the mixture of lead oxide and graphite (fig. 4(c)) are low throughout the entire temperature range with both increasing and decreasing temperature. However, at 850° F a noticeable reaction occurred between the lead oxide and the graphite. In order to avoid reaction problems other mixtures of metallic compounds and graphite were tried. These data are shown in figure 5 along with the data for the metallic compounds at 1000° F. The addition of the metallic salts and oxides to graphite lowers the friction coefficient at all temperatures except room temperature. The mixtures are particularly effective from 150° to 800° F where graphite alone would not lubricate. The very low friction-coefficient values

which are shown in figures 4 and 5 for the mixtures at temperatures from 400° to 600° F were not maintained when the temperature was cycled several times. During cycling the friction coefficient increased to approximately 0.08.

The data of figure 6 show that the results with the mixtures of sodium sulfate and graphite are similar to the results with a resin-bonded graphite film at 400° F. Interpretation of these results would imply that the metallic salt may be acting as a bonding agent for the graphite. The fact that the preheated mixture gave identical results shows that residual moisture present on the salts does not substantially influence the results. 3922

These friction tests show that the mixtures are effective lubricants to 1000° F in spite of the oxidation of graphite. However, no endurance data were obtained on these mixtures, and the comparison (fig. 6) between the mixture and the resin-bonded film does not imply that they are similar in this regard.

SUMMARY OF RESULTS

Under conditions of the experiments reported herein, the following lubricating properties of graphite and mixtures of graphite with metallic compounds as high temperature solid lubricants were observed:

1. Graphite powder lubricated metal surfaces at temperatures sufficiently high to promote oxidation of the metal surfaces. At intermediate temperatures, graphite failed to function as a lubricant.

2. Mixtures of graphite with lead oxide or cadmium oxide effectively lubricated from room temperature to 800° and 1000° F, respectively. Sodium sulfate and cadmium sulfate were also effective additives.

3. Above room temperature, the friction coefficients for the graphite mixtures were lower than for either the graphite or the metallic compounds alone. At 400° F a metallic salt mixed with graphite showed friction coefficients similar to results with a resin-bonded film.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 14, 1955

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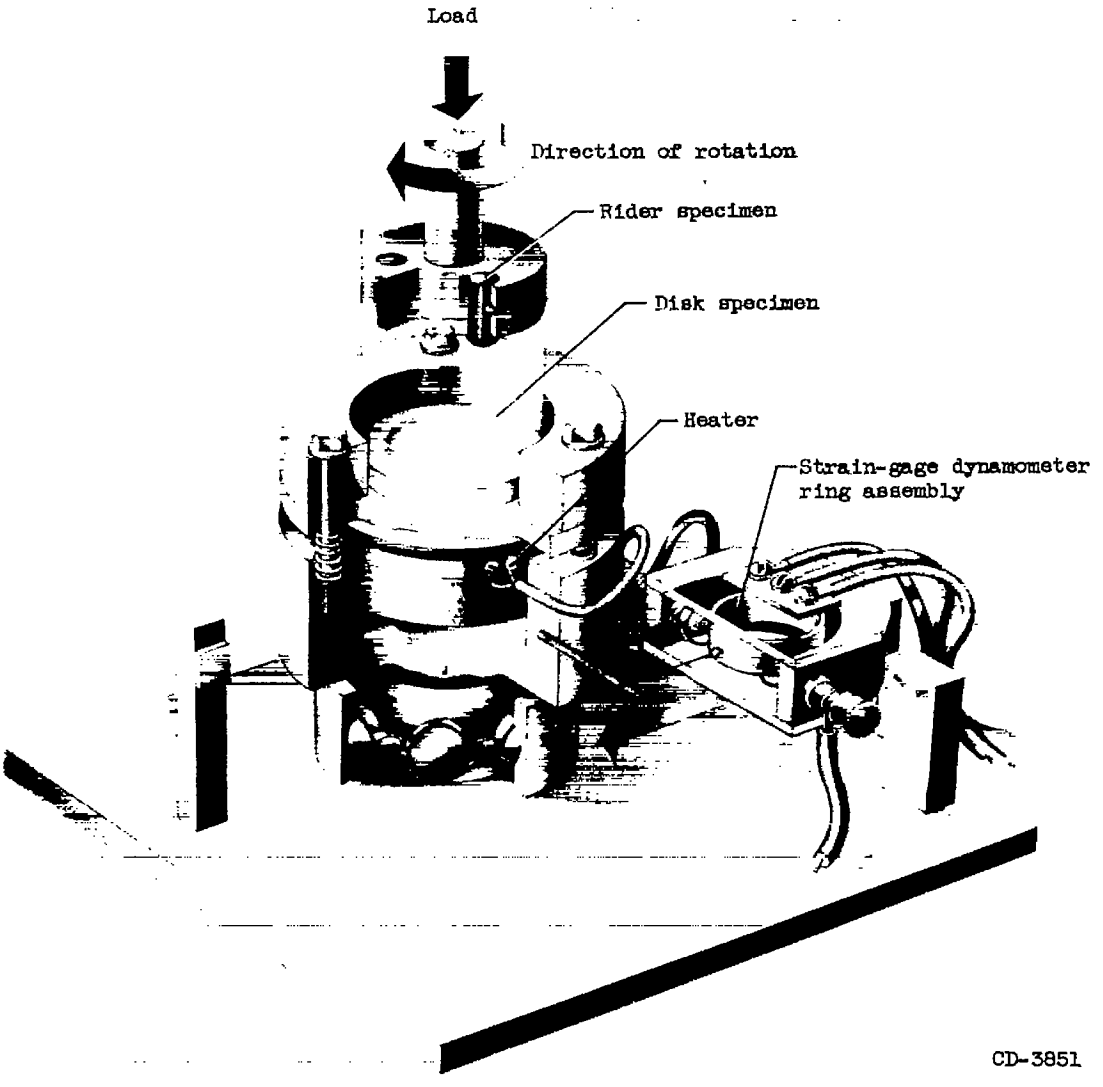

(a) Diagrammatic sketch.

Figure 1. - Friction apparatus.

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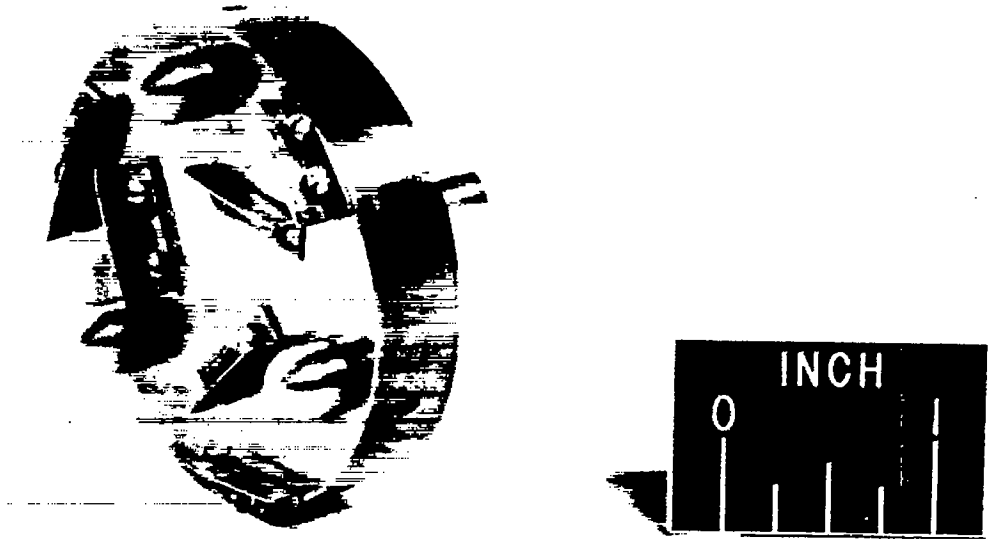

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(b) Rider specimen holder.

Figure 1. - Concluded. Friction apparatus.

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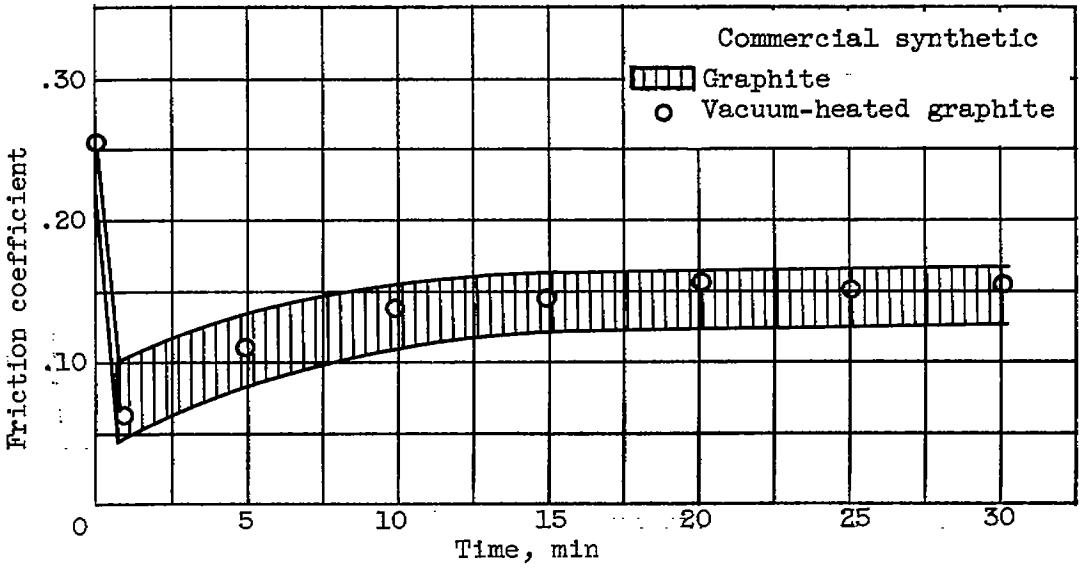
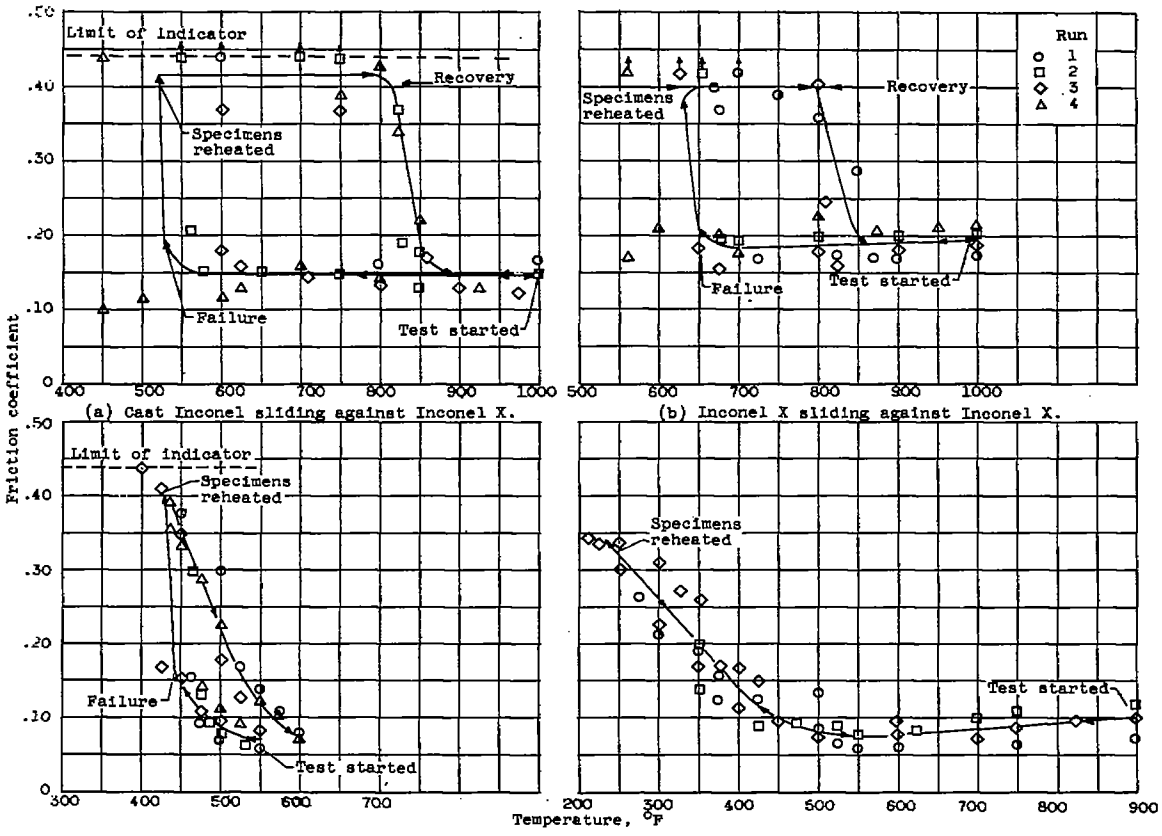


Figure 2. - Effect of sliding time on friction coefficient of cast Inconel sliding against Inconel X using graphite as lubricant. Load, 40 pounds; sliding velocity, 5.7 feet per minute; temperature, 1000° F.



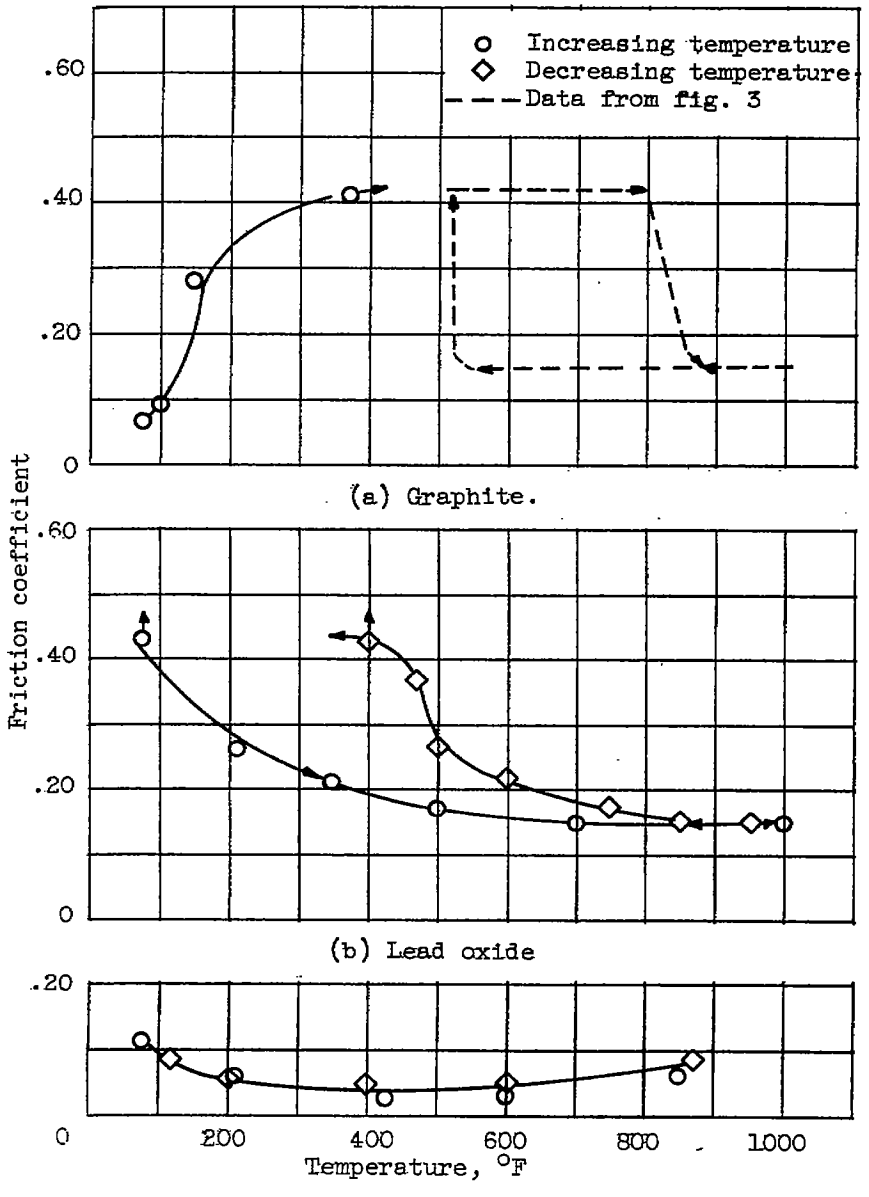
(a) Cast Inconel sliding against Inconel X.

(b) Inconel X sliding against Inconel X.

(c) SAE 1095 steel sliding against SAE 4340 steel.

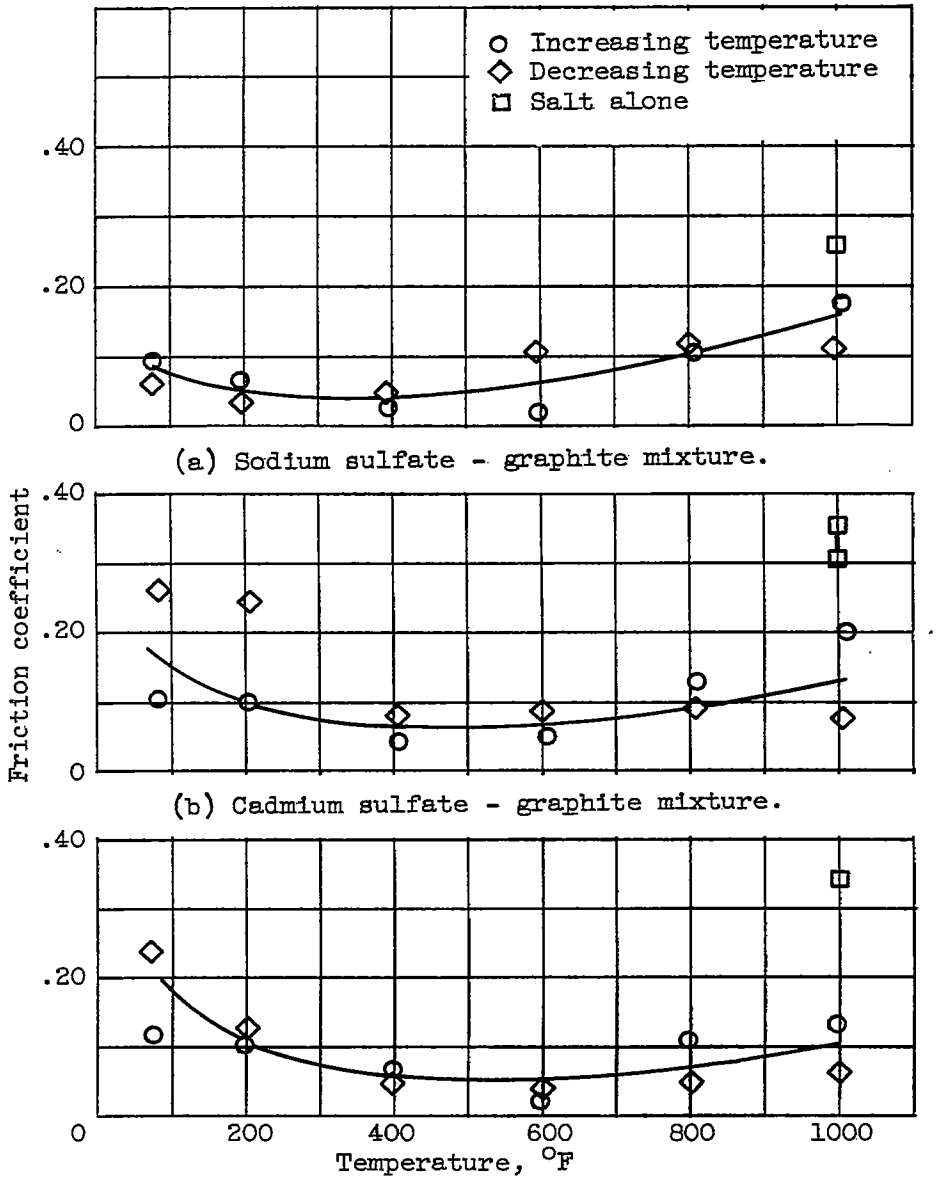
(d) M-1 tool steel sliding against M-1 tool steel.

Figure 3. - Effect of temperature on the friction coefficient of several metals using graphite as a lubricant. Load, 40 pounds; sliding velocity, 5.7 feet per minute.



(c) Lead oxide - graphite mixture.

Figure 4. - Effect of temperature on the friction coefficient of cast Inconel sliding against Inconel X using mixtures of lead oxide and graphite as lubricant. Lead oxide, $66\frac{2}{3}$ percent by weight; load, 40 pounds; sliding velocity, 5.7 feet per minute.



(c) Cadmium oxide - graphite mixture.

Figure 5. - Effect of temperature on the coefficient of cast Inconel sliding against Inconel X using mixtures of various metal compounds and graphite as lubricants. Metal salt, $66\frac{2}{3}$ percent by weight; load, 40 pounds; sliding velocity 5.7 feet per minute.

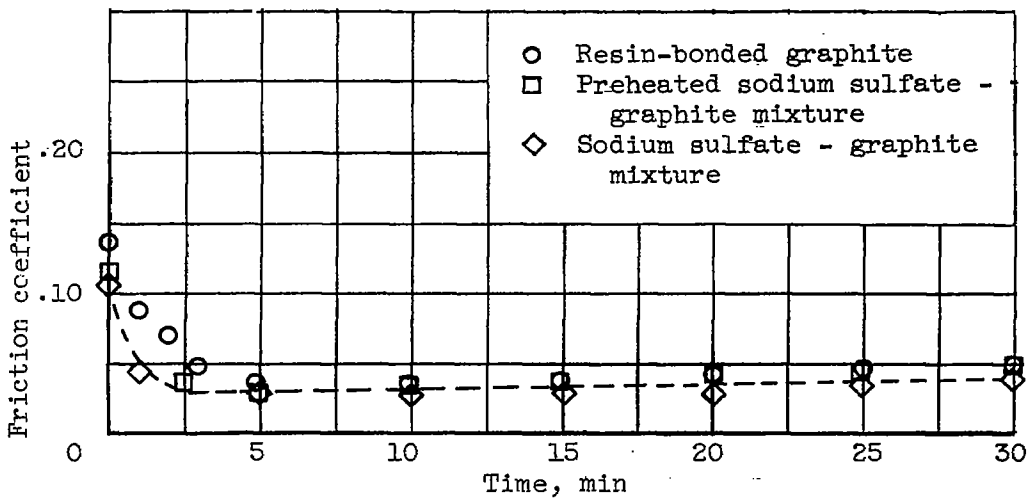


Figure 6. - Effect of sliding time on the friction coefficient of cast Inconel sliding against Inconel X using resin bonded graphite, preheated sodium sulfate - graphite mixture, and sodium sulfate - graphite mixture as lubricants. Lubricant not returned to track; load, 40 pounds; sliding velocity, 5.7 feet per minute; temperature, 400° F.