FOREWORD

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ABSTRACT

A number of substances have been examined for suitability under sliding conditions in the range from room temperature to 2000 F. Low melting metals applied as surface coatings show a peak in their friction at their melting points, unless they form an oxide with good lubricating ability. Pyrolytic boron nitride and graphite have the same friction and wear properties as do ordinary forms of these materials. A discussion is presented of how information may be obtained systematically from friction-temperature runs, and examples of the various techniques are given.

This technical documentary report has been reviewed and is approved.

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I INTRODUCTION

This project is a continuation of an investigation carried out during the past few years under USAF Contract No. AF 33(616) - 5963. The results obtained have been summarized in WADC Technical Reports 59-603, Part I being issued in January 1960, and Part II in May 1961. As these reports show, tests were carried out mainly on three types of materials, namely ceramics, carbon, and metals lubricated by glasses. No really consistent pattern could be found in the results obtained with the ceramics, while in the case of the carbons the friction was generally about .10 at room temperature, then decreased to .05 at about 800°F, and finally rose to above .20 at 1000°F and above.

In the case of the glasses, it was found that, without exception, the friction was moderate at room temperature (about .4), then rose to very high values, often above 2.0, at an intermediate temperature corresponding to the softening point of the glass, and then fell to a low value of .2 or less at high temperatures. These results could be explained in terms of the adhesion and shearing of the glass film. Wear tests showed that at high temperatures, some of the glasses were able to act as boundary lubricants.

These data suggested that it might be profitable to investigate a number of substances which exist in liquid form at elevated temperatures as possible high temperature boundary lubricants. However, since most materials exist in the liquid form over only a limited temperature range, it appears likely that a substance which is to be used as a boundary lubricant at elevated temperatures will exist as a solid at room temperatures. Hence, if this substance is present in a mechanism which is started cold and whose temperature is then gradually raised to elevated temperatures while sliding occurs, it is necessary to surmount the temperature region in which the substance is softening and melting. Since experience with the glasses showed that very high friction could be encountered over this temperature range, it was decided to investigate this friction-during-melting phenomenon with other materials.

The choice of materials was, to a large extent, dictated by the fact that we were interested in substances which melted at temperatures in the range of room temperatures to 2000°F, preferably about half way, without chemical change. This requirement for chemical stability eliminates most organic compounds, and some inorganic ones. Of the remaining inorganic compounds, substances such as sodium chloride and sodium hydroxide combine ready availability with suitable melting point. A large number of low melting metals also come up for consideration. However, they are not in air, as stable chemically as are the inorganic compounds since they are subject to oxidation.

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Hence, it was necessary to study the oxides of the low melting metals as well as the metals themselves, to see whether the effects observed were attributable to the metals or to their oxides.

In previous studies, we had confined ourselves mainly to one metal on which the lubricants were to be tested, namely 303 grade stainless steel. In order to see if the properties of this material influenced our results, it became necessary to extend our experiments to other metals, such as titanium.

Besides these studies on the behavior of high temperature lubricants, we have continued our investigation of materials which might be considered for use at elevated temperatures. The study of molybdenum alloys shows the important influence of the low melting oxide MoO_3, as well as the annoying effects produced when this substance evaporates from the sliding surfaces, condenses elsewhere in the friction apparatus, and re-appears during subsequent tests.

Another series of tests involves the pyrolytic materials, pyrolytic graphite and pyrolytic boron nitride, and a comparison of these properties with those of ordinary graphite and ordinary boron nitride.

Lastly, we have attempted to generalize our experience, and that of other workers in the high temperature friction field, by drawing up in a systematic fashion a summary of testing methods suitable for deducing the significance of features in the friction-temperature plots obtained during sliding. This material is given here in the form of an appendix.

II APPARATUS

The friction apparatus used in this study uses the geometry of three pins of one material contacting a rotating plate of the other material, the region of contact being inside a furnace. The plate is mounted on a shaft which extends outside the furnace and is connected through a pulley system to a variable speed electric motor. The pins are mounted in a self-aligning holder which is connected to another shaft, the far end of which is outside the furnace and is restrained from rotation by means of strain gages. The loading is by means of weights applied to this shaft. The furnace is made of welded steel plate and is air tight, except for the front door, which is bolted on and supplied with a cooling system to protect the Neoprene gasket, and the two holes for the shafts, which are covered by rotating seals and also cooled. The chamber is electrically heated, using resistance wires. A schematic drawing of the apparatus is shown in Figure 1.

For the runs described in this report, the temperature was normally raised from room temperature to 2000°F during two hours of steady heating, during which time sliding was continuous and the friction was monitored by an electrical recorder. The pins were of 1/4" diameter and were normally given a slightly rounded end (radius about 2"). The flat specimen was of dimension 2" X 2" X 1/4" and was given its final finish on a polishing wheel.

III RESULTS

A) Frictional Behavior of Low-melting Point Metals in the Vicinity of Melting Point

a) Test Using Steel Surfaces

Friction runs were made with several low melting point metals in the temperature range between 70°F and 1830°F. The metals prepared for the runs were lead, zinc, cadmium and tin, the melting points being 622°F, 786°F, 610°F and 450°F, respectively. When the melting point was approached in the runs, interesting changes in friction were observed.

In the runs reported in the previous report, the metals were applied to the surface of a steel flat as coatings. Since the coatings were thin, it was possible that the metals were oxidized or worn out during the runs. In order to reduce such effects, the metals were applied to the steel surface in the present experiments as powders and in excess quantity. (Figure 2)

Runs were made both in air and in argon. Friction was measured as the temperature was raised continuously from room temperature to 1830°F. Also, the electrical contact resistance during a run was continuously monitored.

The results are shown in Figure 3. It is noted that atmosphere has large effects on the friction of these metals. This indicates that the results were affected greatly by the formation of oxides during the runs. Hence, runs were made with the oxides of the metals in order to study this effect. Results are shown in Figure 4.

The friction-temperature plot of lead and of lead monoxide are quite similar. Also, the variations of the electrical contact resistance were alike in both runs. It would seem that lead was oxidized very rapidly during the runs and the friction curve obtained in the test was actually that of lead monoxide. It is considered likely that even in the test performed in argon, the lead was oxidized by oxygen impurity contained in the argon. The friction of the red oxide (Pb_3O_4) and of the black dioxide (PbO_2) was a little higher than that of the monoxide (PbO), but also decreased to a low value above 900°F. This decrease is probably also due to the formation of lead monoxide, since the latter is the main form stable above 900°F. Monoxide melts at 1630°F, and all curves showed an increase in friction at this temperature.

It is interesting to note that the low friction between 500°F and 1600°F is accompanied by an increase in electrical contact resistance. This indicates that lead monoxide forms a good protecting film on the steel surface in this temperature range, and that this results in the low friction.

Similarly, comparison between the friction curves of zinc and zinc oxide shows that zinc was oxidized in the run in air. However, the friction curve of zinc in argon is quite different from that of the oxide. It gave a very distinct peak at the melting point of zinc.

Cadmium and tin also gave a peak in the friction at their melting point, while the oxides of these metals showed little change at this temperature. It would seem that these two tests were free from the effects of oxidation.

The above results show that metal powders give a peak at their melting point, if the metals have not become oxidized at some lower temperature. However, the peak disappeared or became very dull in the friction temperature plots of cadmium and tin in argon, while the friction plots of these metals in air gave a distinct peak at the melting point. Evidently, the disappearance of the peak in these results is not attributable to the oxidation of the metals, and further study is required to explain these results.

b) Tests Using Titanium Surfaces

To investigate the frictional behavior of cadmium and tin, furthe runs were made using these metal powders in the same way as in the previous runs, but this time not with steel pins and flats, but with friction specimens made of titanium. In Figure 5 the results of the experiments are shown. In this test, the peak appeared at the melting point both in air and in argon.

Molten metals could form a lubricating film and give a low friction under favorable conditions. However, Figure 3 and 5 show rather high friction above the melting point of the metals, indicating that a lubricating film did not develop. Comparing this result to that of molten glasses, which showed a friction of as low as 0.1, it could be said that these metals have poorer ability to develop lubricating film than molten glasses.

Many steel on steel tests gave a rise in friction at approximately 1700°F, and titanium on titanium tests gave a rise at about 1500°F. It is felt that this rise is due to the frictional change of the bulk surface, and runs were made with steel on steel and titanium on titanium. Indeed, the friction, as shown in Figure 6 did show a peak at about 1700°F and 1500°F, respectively.

B) Frictional Behavior of Inorganic Compounds in the Vicinity of the Melting Point

Friction runs were made with several inorganic compounds in the same way as in the previous runs. Results are shown in Figure 7.

It is noted that the friction of sodium chloride and potassium chloride is quite similar to that of the steel substrate itself. However, a disturbance is observed at the melting point of the inorganic compounds. On the other hand, sodium hydroxide (NaOH) and sodium tetraborate ($Na_2B_4O_7$) did not show any change at the melting point. Further work with inorganic materials will be required to explain these results.

C) Friction of Molybdenum Alloys

The frictional behavior of molybdenum alloys is of interest, since these alloys at high temperatures are known to form the low-melting molybdenum oxide (MoO_3), which has a melting point of $1463^{o}F$. Above this temperature, the alloys could be lubricated by the molten oxide, provided that a lubricating film is formed.

Figure 8 shows the temperature-friction plot of two commercial molybdenum alloys (Climelt). They exhibited low friction at moderate temperatures, but the friction increased sharply at about $500^{o}F$. Later, the friction showed a decrease and gave a trough at about $1450^{o}F$. With the decrease of friction formation of molybdenum oxide on the sliding surface was observed, and above the melting point of the molybdenum oxide, friction rose again. It is likely that the decrease in friction is caused by the oxide formation. High friction above the melting points of the oxide indicates that a lubricating film was not formed in these tests.

D) Contamination Effects

During the frictional tests with the two molybdenum alloys, an annoying effect was observed. It was found that the molybdenum oxide formed on the sliding surfaces sublimated from the surfaces, condensed elsewhere in the friction apparatus, and reappeared during subsequent tests.

In order to check these effects, friction runs were made, first with the molybdenum alloys and then with stainless steel on stainless steel, unlubricated. Figure 9 shows the results of these runs. The number shown for each run corresponds to the order of the runs made after the contamination. The average friction value of the runs, No. 1 and No. 2 shows the characteristic friction trough of the molybdenum oxide at $1450^{o}F$. After three or four runs, this trough disappears presumably because by this time the molybdenum oxide has disappeared and the friction shows a peak at $1700^{o}F$ which is characteristic of uncontaminated stainless steel.

It was suspected that similarly contamination of the friction apparatus could occur also during tests involving boric oxide. Hence, studies were carried out with boric oxide to detect such contamination. The results are shown also in Figure 9. The average friction value of the runs No. 1 and No. 2 showed a peak at about 900°F due to boric oxide. However, the peak was removed and the friction temperature plot became that characteristic of uncontaminated stainless steel on stainless steel in runs 3 and 4.

E) Friction of Pyrolytic Graphite and Pyrolytic Boron Nitride

High temperature and room temperature friction tests were made with pyrolytic graphite and pyrolytic boron nitride. The pyrolytic speciments have a lamellar structure parallel to the sliding surface. For comparison, ordinary graphite and boron nitride were also tested.

Figure 10 shows the results of the high temperature tests. Since the friction of graphite is very much influenced by the adsorption of gas and moisture, runs were made both in air and in argon.

As is shown in the figure, little difference was found between the friction of pyrolytic graphite and ordinary graphite. The friction was about .1 -.2 at room temperature and dropped to a very low value at about 500°F. Beyond this friction trough, the friction rose again. However, the rise in friction was moderate even at high temperatures, and it is considered that these results show the promise of graphite for use at high temperatures.

Both ordinary and pyrolytic boron nitride gave fairly low friction at room temperature, but the friction rose to a high value as the temperature was raised (Figure 10). Again, no appreciable difference was found between the pyrolytic boron nitride and ordinary boron nitride.

It is generally agreed that the low friction of graphite and boron nitride is due to the low resistance to shear of planes of large atomic spacing (1). Then, it might follow that pyrolytic specimens which have an oriented lamellar structure may give lower friction than the ordinary specimens in which the orientation of the crystallites is random. However, the present results were contrary to the above idea.

In order to investigate the difference between pyrolytic specimens and ordinary specimens, more detailed friction runs were made for varying loads, speeds and lubrication, but this time at room temperature and using a one-pin on rotating disk friction apparatus.

Table 1 - 3 show the results. Again, no appreciable difference was found between the friction of pyrolytic specimens and ordinary specimens. Wear of both specimens were also measured, but they were the same within the limit of the experimental error.

Thus, it is concluded that there is no difference between pyrolytic and ordinary specimens. Probably because ordinary graphite and boron nitride give an oriented structure on repeated sliding.

IV SUMMARY AND CONCLUSION

During the period covered by this report, friction studies on several high temperature lubricants have been continued. The results of the tests are summarized as follows:

(1) The possibilities of low melting metals as high temperature liquid lubricants have been investigated. Since the metals exist as solids at room temperature and the melting temperature must be surmounted when sliding devices are operated over a wide temperature range, the "friction during melting phenomenon" has been studied intensively. In order to check the effects of oxidation, friction tests have been carried out both in air and in inert gas atmosphere and the results are compared with those obtained with the metal oxides. Some low melting metals gave a friction peak at the melting point, in the same way as do glass-like lubricants. Other metals which did not give the peak have been affected by oxidation. Exceptions to this rule are found with tin and cadmium in argon. Although they were not oxidized, they did not give any friction peak.

(2) Inorganic compounds gave quite a different type of disturbance in friction-temperature plot at the melting point. Further investigation of the frictional behavior of this class of materials may give us a clue to the solution of the mechanism of the "friction during melting" phenomenon.

(3) The friction of molybdenum alloys has been studied at elevated temperatures. The results have shown the marked effects of molybdenum oxide formed on the sliding surface.

(4) It was found that molybdenum oxide and boric oxide vapor can contaminate the friction apparatus and effect subsequent friction tests.

(5) Pyrolytic graphite and pyrolytic boron nitride have been tested both at high temperatures and at room temperature, and the results are compared with those obtained with ordinary graphite and boron nitride. No appreciable difference was found between the pyrolytic specimens and ordinary specimens. Graphite gave low friction at high temperatures and showed substantial promise for the use at high temperatures, while boron nitride gave poor frictional performance at high temperatures.

APPENDIX

The Systematic Analysis of High Temperature Frictional Data

A number of experimenters (2), (3), (4), (5), (6) have published results obtained with high temperature friction machines. The testing procedure usually consists of continued sliding at constant speeds over a circular wear track while the temperature is steadily raised from room temperature to some maximum temperature, and the friction is continuously monitored. In this appendix an examination will be undertaken of the data which may be obtained with machines of this type, and the way that the data may be used to deduce the changes that are taking place at the sliding interface. The test geometries used by the various experimenters are rather similar, in that the specimens of the materials to be tested are small and of simple shape, and they are so arranged that contact is made over a restricted area, rather than over a large interface. The advantage of keeping specimen size small and specimen shape simple is that it reduces the difficulty of procuring and preparing the specimens, while the constricted geometry insures that slight amounts of wear will not drastically change the geometry of contact.

A) Interpretation of Friction Data

The first step in interpreting friction data consists in drawing a plot of the friction as a function of temperature, which hereafter will be referred to as an f-T plot. The f-T plot has two principal uses, the first being the obvious one of showing the friction of the combination tested at various temperatures, while secondly, it allows a means for deducing the changes that are taking place during sliding. In some cases there are no changes (Figure 11). In this case, it can be assumed that the materials and the nature of their interaction with each other remain essentially unaffected by the temperature to which they are exposed. In most cases, however, there are features of interest to be seen on the f-T plot; perhaps a sharp dip, or a peak, or a number of such phenomena. Unless considerable information about the sliding materials is available, it is not possible to interpret these features without resorting to auxiliary testing techniques. The following techniques are those which are used most frequently and most successfully.

B) Testing in Environments of Different Oxygen Concentrations

When this approach is used, the materials are first tested in an air environment and then the test is repeated in an atmosphere of nitrogen, argon, helium, or in a vacuum. While it is difficult to exclude oxygen completely from a high temperature friction apparatus, it is usually easy to diminish its partial pressure drastically. In consequence, peaks and valleys due to oxidation are shifted in the direction of higher temperature. An estimate of the order of magnitude of this effect may be made by noting that chemical reactions are usually speeded up by a factor of about 2 for every 10°C rise in temperature. Hence, assuming that the reaction rate of the oxidation process is proportional to the oxygen concentration, and doubles for every 10°C rise in temperature, it may be calculated that each drop in partial pressure of oxygen by a factor of 50 shifts the position of features on an f-T plot by 100°F.

These points are illustrated by Figures 12, 13, and 14. In Figure 12 are shown the f-T traces of a carbide system in air and in argon, and the shift in the peaks and troughs by about 200°F is very evident. Clearly, some constituents in the carbide mixture are undergoing oxidation, and the argon atmosphere has reduced the oxygen partial pressure by a factor of about 2500. Figure 13 shows experiments using a steel sliding on boron carbide, and the peak due to boric oxide formation is seen to be shifted in the inert environment. Figure 14, in contract, shows friction data obtained for steel surfaces, with boric oxide as a lubricant. Here no oxidation changes are involved, hence the peaks obtained in air and in the inert gas coincide.

C) Temperature Cycling

A form of testing which is often very revealing consists of carrying out a standard f-T test and of continuing testing while the apparatus is cooled. Thus, two f-T curves are obtained, one for T rising and the other for T falling. If the cooling curve does not match the heating curve, then there is strong evidence that an irreversible change, perhaps a chemical reaction has occurred. Figure 15 summarizes data obtained by Peterson, Florek and Lee (3) with various metals, and the effect of oxidation of the metals is clearly evident. It is possible to know something about the tenacity of the oxide by noting how long it takes for the friction to reach its initial value again. Figure 16 shows some data the author has obtained with a silicate glass as lubricant. In this case good reversibility is observed.

D) Changes of Load and Speed

With most types of high temperature friction apparatus, it is possible to carry out sliding tests at widely varying speeds and at moderately varying loads. However, most investigators keep the load and speed constant because little of interest is generally observed when these parameters are varied. As regards load variations, it is only in the case of metals which form oxide films that any effects are likely to be produced, and these are due to the fact that at light loads sliding is of oxide on oxide, while at heavy loads, it is metal on metal (7).

As regards speed changes, variations in the friction are likely to become pronounced only at very high speeds, when frictional heating becomes significant. A very simple formula gives the temperature rise under typical sliding conditions in the form

$$\Delta T = v/4 \pm \text{ a factor of } 3$$

where v is the velocity in cm/sec, and ΔT the temperature rise in ^{o}F. To get a temperature rise of $100^{o}F$, which might in favorable cases produce an observable change in the friction, we would need sliding speeds of the order of 400 cm/sec. At speeds which are one or two order of magnitude lower than this, friction phenomena are generally rather speed independent.

Two notable effects of speed have, however, been observed even at low speeds. The first is obtained in systems in which a viscous substance is present (e.g. a glass), in which case the system may be operating in a viscous lubrication regime and the friction will vary very greatly as the speed is changed (Figure 17). The second case is that of metallic systems in which the rate of formation of oxide films is of the same order of magnitude as the rate of their removal during sliding. (Figure 18).

E) Wear Measurements

The wear rate is an important quantity to be determined during sliding tests. Unfortunately, it is very difficult to get a continuous reading of the amount of wear, analagous to the continuous reading of the friction. Instead, a sliding test of some duration, perhaps 1 hour, must be carried out while the temperature is kept constant, and then the test must be stopped, and the wear determined by weight or geometry changes. Subsequently, wear measurements are carried out at other temperatures.

A typical use of wear measurements as an aid in diagnosis is shown in Figure 19, and it will be seen that the peak in the friction of boric oxide lubricated surfaces corresponds to a minimum in the wear rate. This shows that the peak is due to viscous effects in the boric oxide, rather than to excessive metal-to-metal contact because of lubricant failures.

Since loss in weight of sliding specimens at high temperatures is due to two causes, wear and corrosion (oxidation), it is helpful to be able to separate them. This is best done by placing in the high temperature friction apparatus, dummy specimens identical with the wear specimens. The weight change of the dummy specimens then gives the effect due to oxidation.

A word of caution is in order, namely that wear measurements may give misleading results in the case of brittle materials. Under these circumstances, a high wear rate may indicate poor mechanical properties rather than a severe surface interaction. However, the friction will be almost unaffected by the brittleness and will indicate whether the surface interaction is, indeed, severe. This effect is illustrated in Figure 20, which shows friction and wear data obtained on three propriatory sintered powder composites of essentially the same composition but of widely different mechanical properties. The friction was very nearly the same for all three materials, but the total loss in weight during the three tests varied by a factor of 22. Clearly, the friction tests more reliably indicates the surface interaction of the two materials.

F) Electric Contact Measurements

In some circumstances valuable information may be obtained by measuring the electrical resistance across the sliding interface. Oxide formation and break-up, also the formation of hydrodynamic lubricant layers, can often be detected (Figure 21). Even though in some systems the measurement of electrical resistance is impractical, because the interfaces are electrical insulators, while in other cases nothing of interest is observed, yet it is felt that this technique has great promise because it can often be very revealing, and it can usually be added very readily as another signal to be monitored by the friction recording device.

G) Contamination of Friction Apparatus

A feature of friction testing at elevated temperatures which has received little attention is the production of artifacts in the f-T curve by volatile material derived from previous tests. Such features have been observed in friction experiments immediately subsequent to tests in which volatile oxides such as boric oxide are formed. (Figure 9). These evidently deposited on the walls of the friction apparatus and then were re-deposited on to the surfaces of the clean friction specimens used in a subsequent test.

H) Statistics of Friction-Temperature Plots

Having discussed methods of discovering the meaning of features of an f-T plot, consider a more fundamental question; namely, how can a feature be recognized when one appears. In order to decide this point, it is helpful to take a large number of f-T curves and study them statistically to see which trends are general, and which are special.

Fifty representative friction-temperature curves have been analyzed out of the several hundred obtained over the past 6 years of experimentation. The curves used in our analysis came from a wide variety of materials, were taken on two different high temperature friction machines, but the testing conditions were similar (load 1 kg ± a factor of 2), (sliding speed 1.5 cm/sec ± a factor of 3), (heating rate 1000°F per hour ± a factor of 2). The friction coefficient was measured every 100°F.

Figure 22 shows the average friction coefficient for all the 50 runs as a function of temperature. The average friction coefficients were almost independent of temperature, varying from .37 to .52. There is no indication that the friction coefficient varies systematically with sliding temperature (a cynic might re-phrase this and state that there is no indication that the sensitivity of our measuring system is temperature dependent).

If the friction coefficient is independent of temperature, the individual friction-temperature traces can be regarded as so-called time-stationary series, from which autocorrelation coefficients can be computed. For the 50 friction traces, it is possible to compute the simple autocorrelation coefficient γt defined in an earlier paper (8).

$$\gamma_t = \frac{n}{n-50t} \frac{\Sigma |f_j - f_{j+t}|}{\sqrt{2}\,|f_j - \bar{f}\,|}$$

where f_j is the friction at some temperature j, $f_j + t$ is the friction at a temperature t hundreds of degrees higher, and n is the total number of friction values. Figure 23 plots the average autocorrelation coefficient for all the 50 traces used in devising Figure 22 as a function of temperature separation T. This autocorrelation plot gives a definition of what constitutes a rise or drop in an f-T curve. If, for example, in the course of a 100°F change of temperature, the friction has changed by considerably more than .08, or if during a 200°F change in temperature the friction has changed by considerably more than .14, a feature is indicated.

The f-T plot has other uses. Thus, it reveals something about the validity of extrapolation of friction data to higher temperatures. It will be seen that by the time the temperature separation is about 500°F, the correlation is close to zero and the two situations are, as far as friction is concerned, completely independent of each other.

A third factor that can be evaluated is the distribution of friction values for the 50 runs, which is shown in Figure 24. It should be noted that fully 80% of all the friction values lie between the limits .2 to .7, and that friction coefficients below .1 or above 1 are quite rare. In view of the rareness of these extreme friction values, it could be said that friction coefficients above 1.0 or below .1 are features which are worthy of further examination.

A fourth aspect of high temperature friction testing which may be evaluated statistically is the reproducibility of friction tests. From the data of Figure 24, a mean friction value of .44 and a standard deviation of .18 are obtained.

From this it is calculated that two friction values picked at random are likely to differ by .25 (mean deviation). However, a comparison carried out on 20 pairs of repeat friction tests shows that the average variation between friction values under apparently identical situations is only .09 (mean variation); hence, it is deduced that friction-temperature traces are real, i.e. they are not random squiggles drawn by the friction recording device. However, it is clear that considerable variation in friction coefficients can occur without having any real significance, e.g. if of two similar materials, one gives a friction coefficient of .40, and the other a friction of .50, then there is no really strong reason for believing that there is a significant variation between the materials. A friction coefficient difference of .23 would, however, be significant at the 95% confidence level.

I) Discussion

This appendix has considered a number of ways of elucidating the features of an f-T curve, and has indicated in some cases to what these features are attributable. In due course, as further progress in this field is made, it should be possible to explain all the features of the f-T plots in terms of the surface and bulk properties of the sliding materials. At this stage, it will be possible to examine an f-T curve, and use the information it contains to deduce modifications to be made in the sliding materials either to eliminate a harmful feature or else to extend a helpful one. Alternatively, it will be possible to use the f-T curve to deduce what is happening to the materials as they are heated. In regard to features such as oxide formation, the f-T curve can already in favorable cases provide such information more quickly and inexpensively than can other tests. It is expected that, in the future, this area of usefulness of f-T tests will be enlarged.

REFERENCES

1. R. H. Savage, "The Vapor-Lubrication of Graphite in Relation to Carbon Brush Wear", Mechanical Wear, A.S.M., 1950, p. 95.

2. Kingsbury E. P. and Rabinowicz E., "Friction and Wear of Metals to 1000 C" ASME Trans. D, <u>81</u> No. 2, 1959, p. 118

3. Peterson M. B. Florek J. J. and Lee R. E., "Sliding Characteristics of Metals at High Temperatures" ASLE Trans., <u>3</u>, No. 1, 1960, p. 101

4. Mordike B. L. "Lubrication of Solids at High Temperatures" ASLE, Trans. <u>3</u>, No. 1, 1960, p. 110

5. Zeman K. P. and Coffin L. F. "Friction and Wear of Refractory Compounds", ASLE Trans. <u>3</u>, No. 2, 1960, p. 191

6. Klemgard E. N. "Fundamental Processes in Lubricating Metal Surfaces at 100°F to 1700°F", Lubr. Eng., <u>16</u>, No. 10, 1960, p. 468

7. Whitehead, J. R. "Surface Deformation and Friction of Metals at Light Loads", Proc. Roy. Soc. A, <u>201A</u>, 1950, p. 109

8. Rabinowicz E. "Autocorrelation Analysis of the Sliding Process", J. Appl. Phys., <u>27</u>, 1956, p. 131.

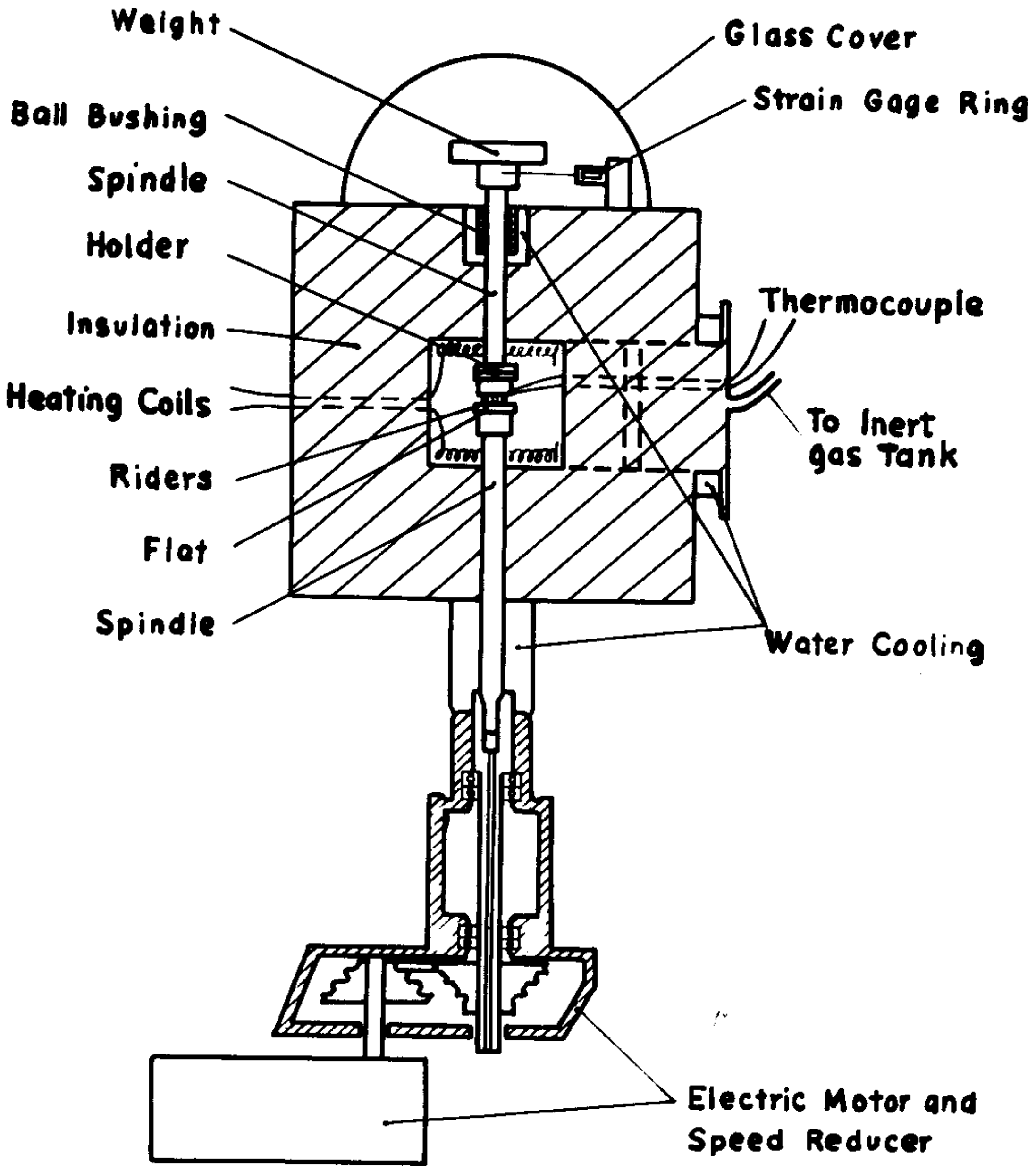


FIGURE 1

Schematic Illustration of the High-Temperature Friction Apparatus

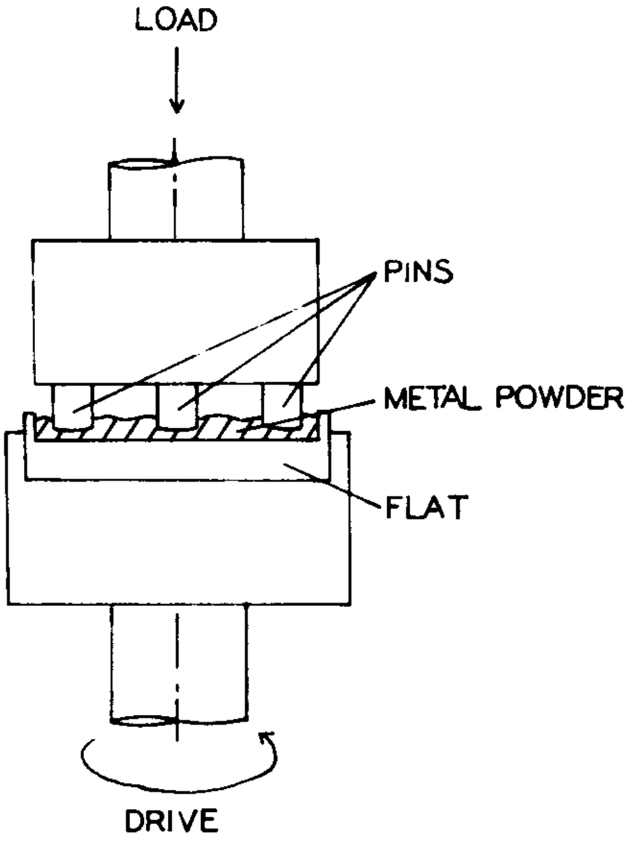


FIGURE 2

Schematic Illustration of the Test Specimens for metal powder runs

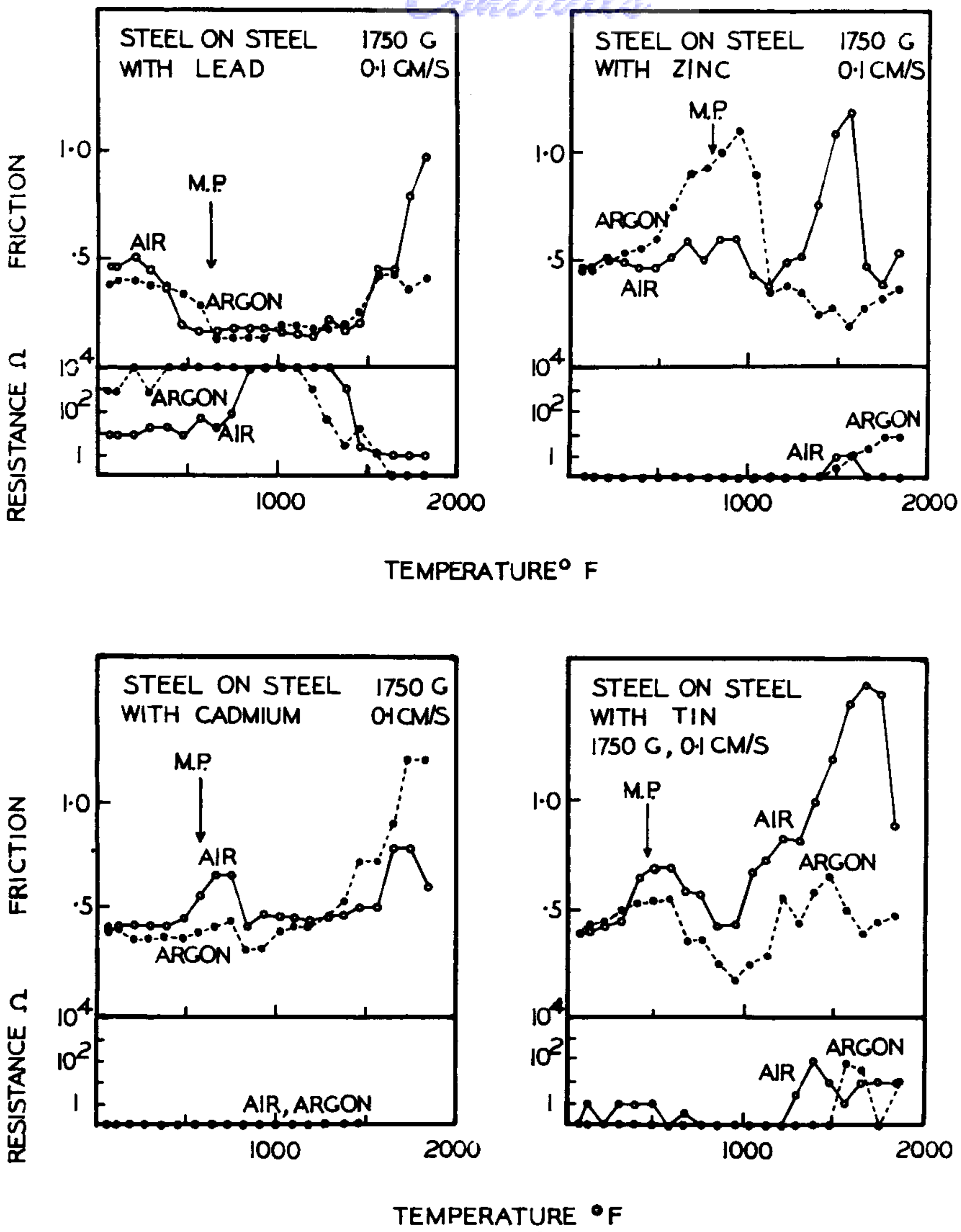


FIGURE 3

Friction-temperature Plot for Steel on Steel with Metal Powders

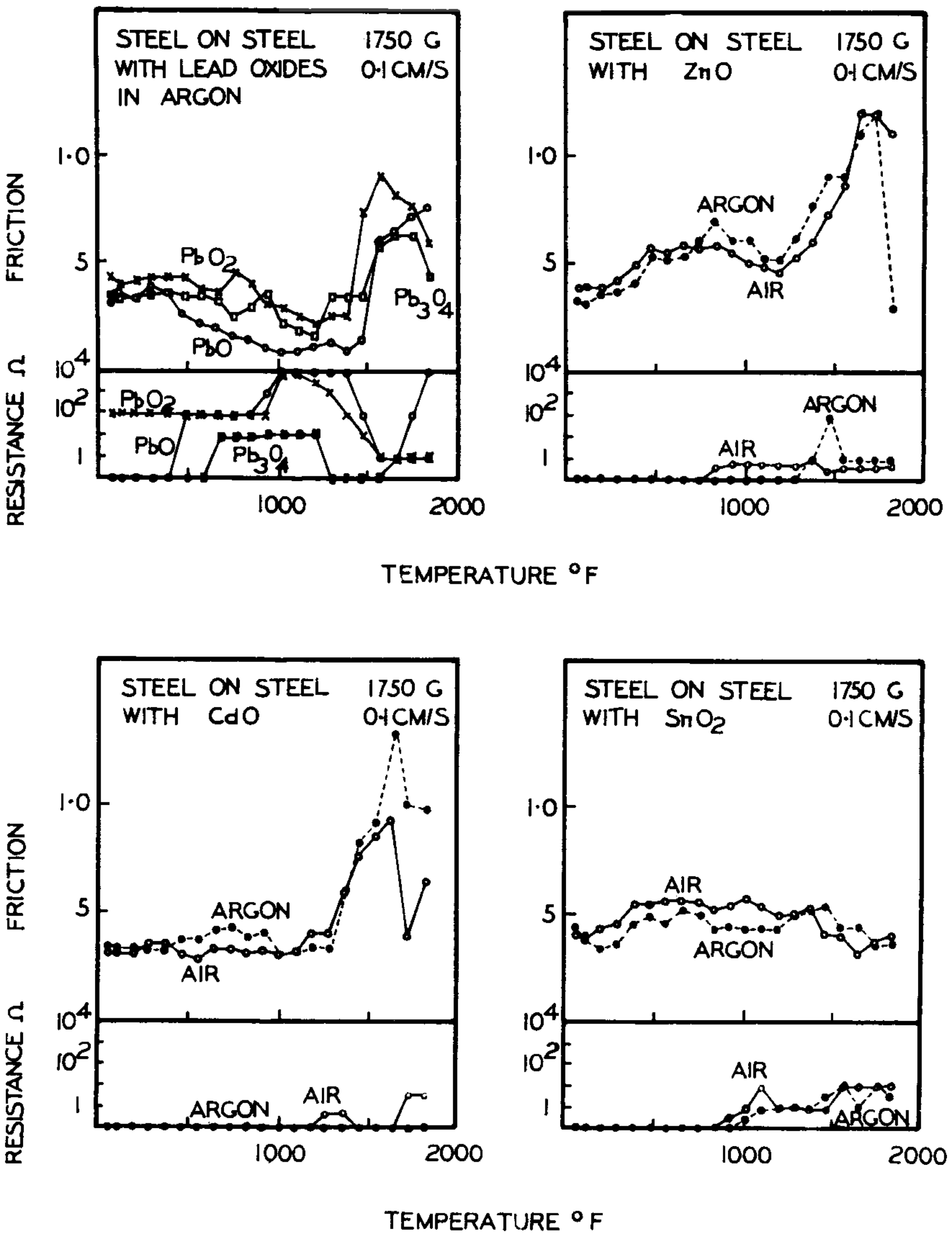
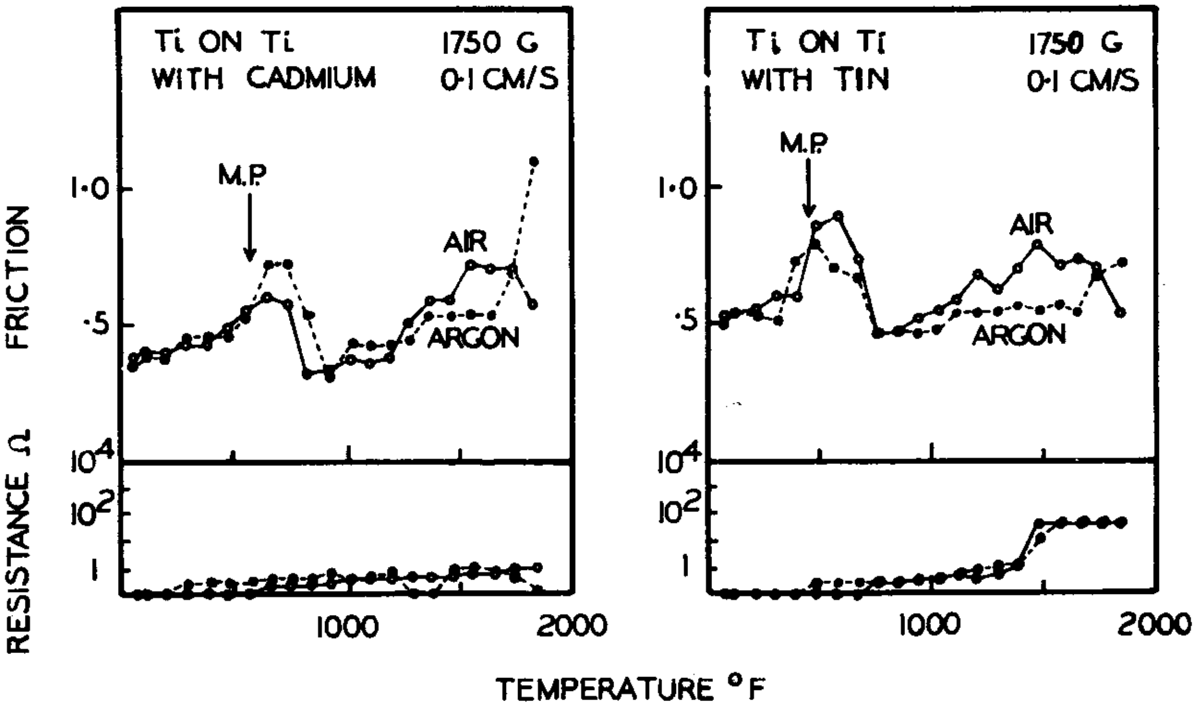


FIGURE 4

Friction-temperature Plot for Steel on Steel with Metal Oxides

FIGURE 5

Friction-temperature Plot for Titanium on Titanium with Metal Powders



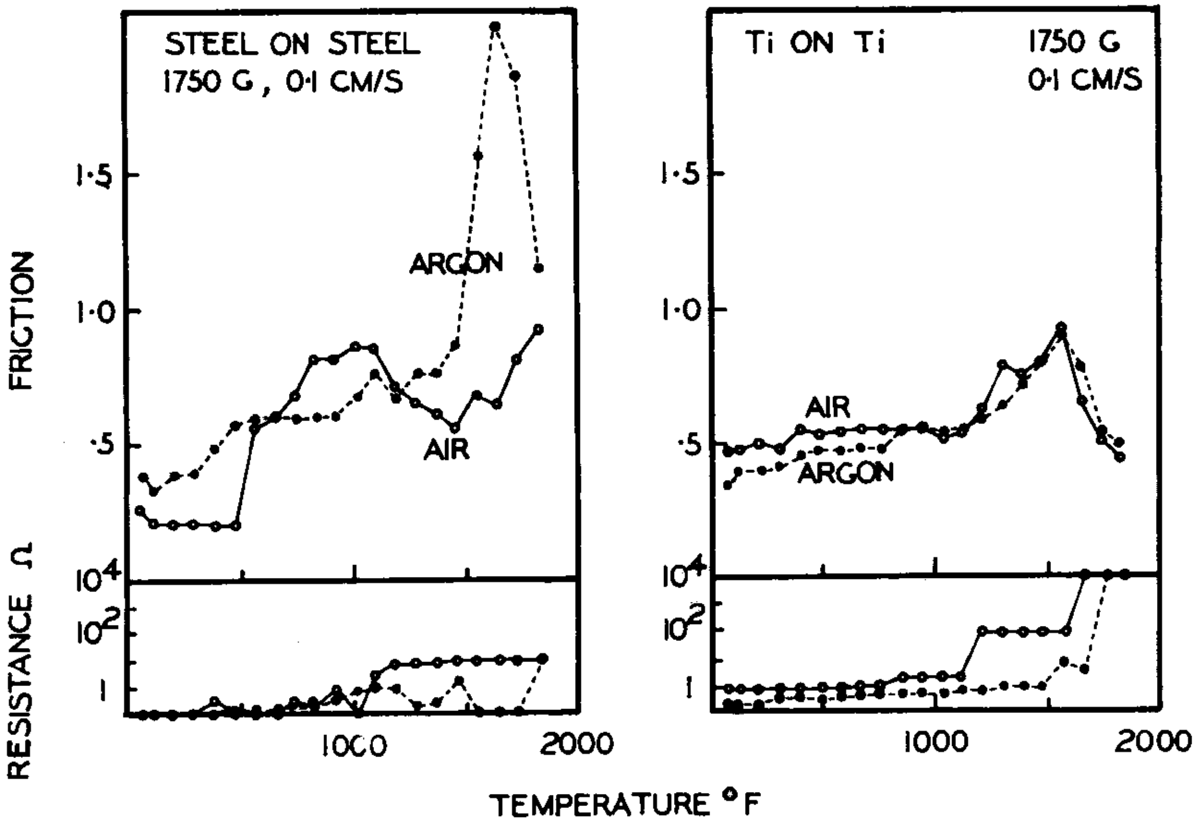


FIGURE 6

Friction-temperature Plot for Steel on Steel and Titanium on Titanium

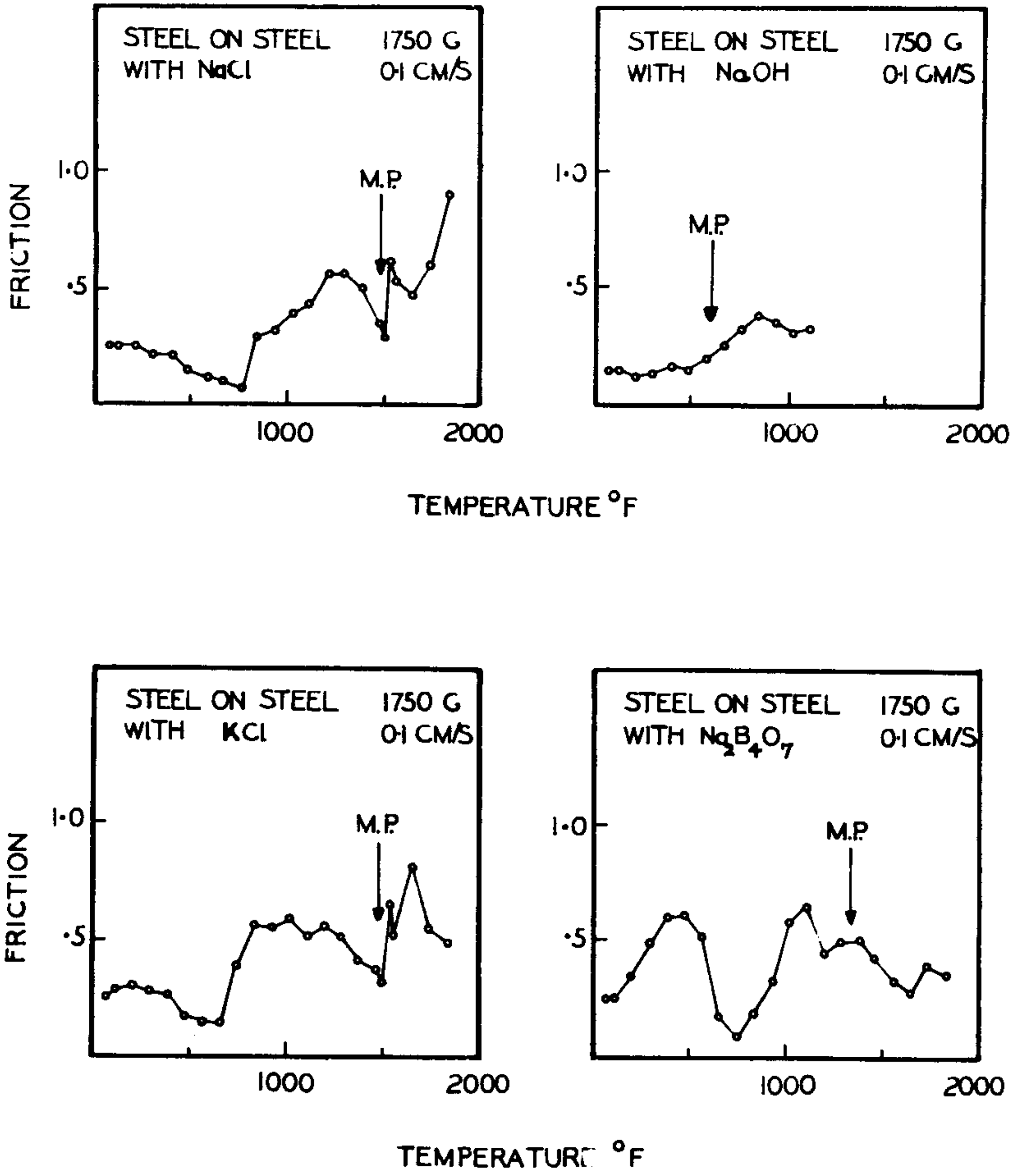


FIGURE 7

Friction-temperature Plot for Steel on Steel with Inorganic Compounds

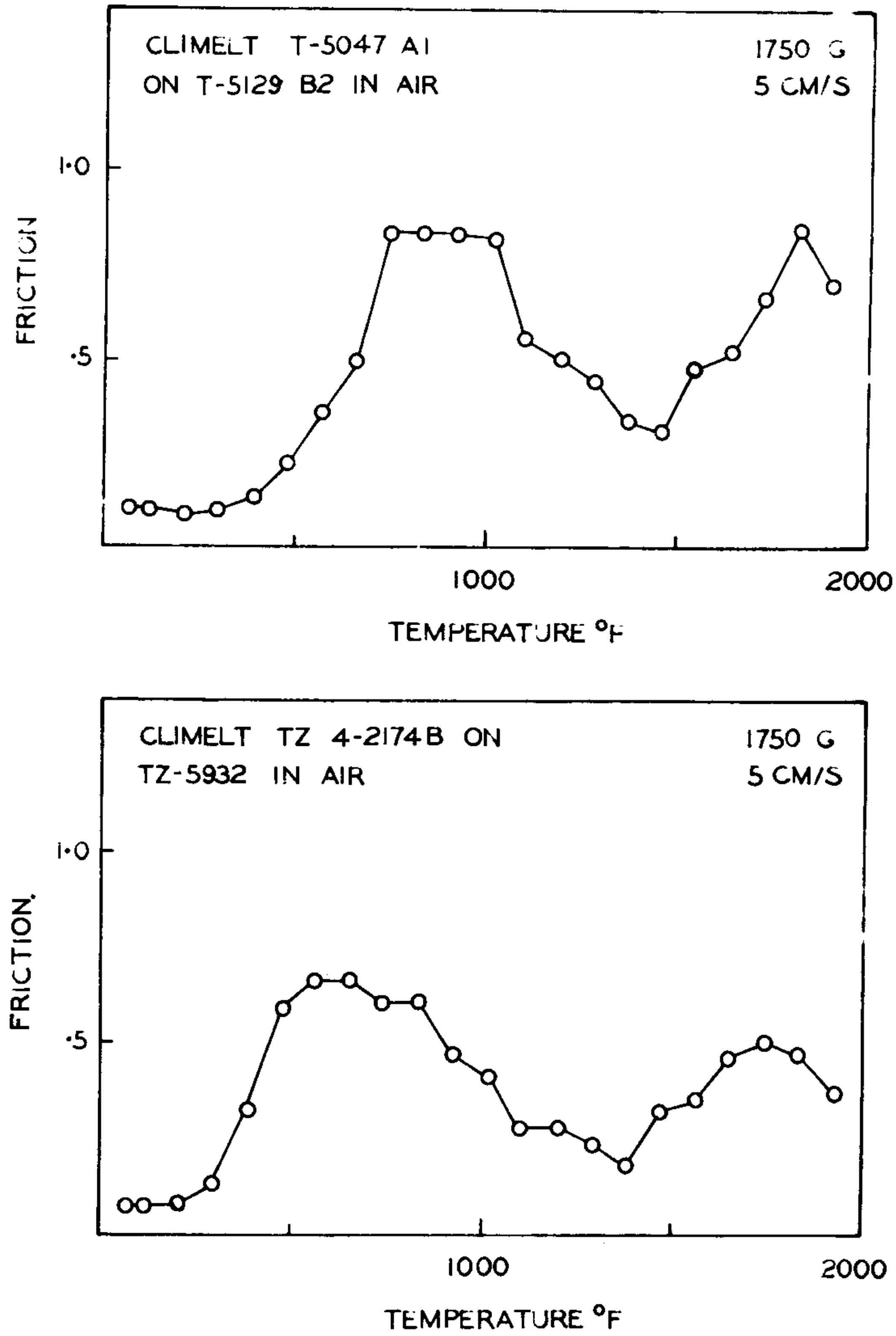
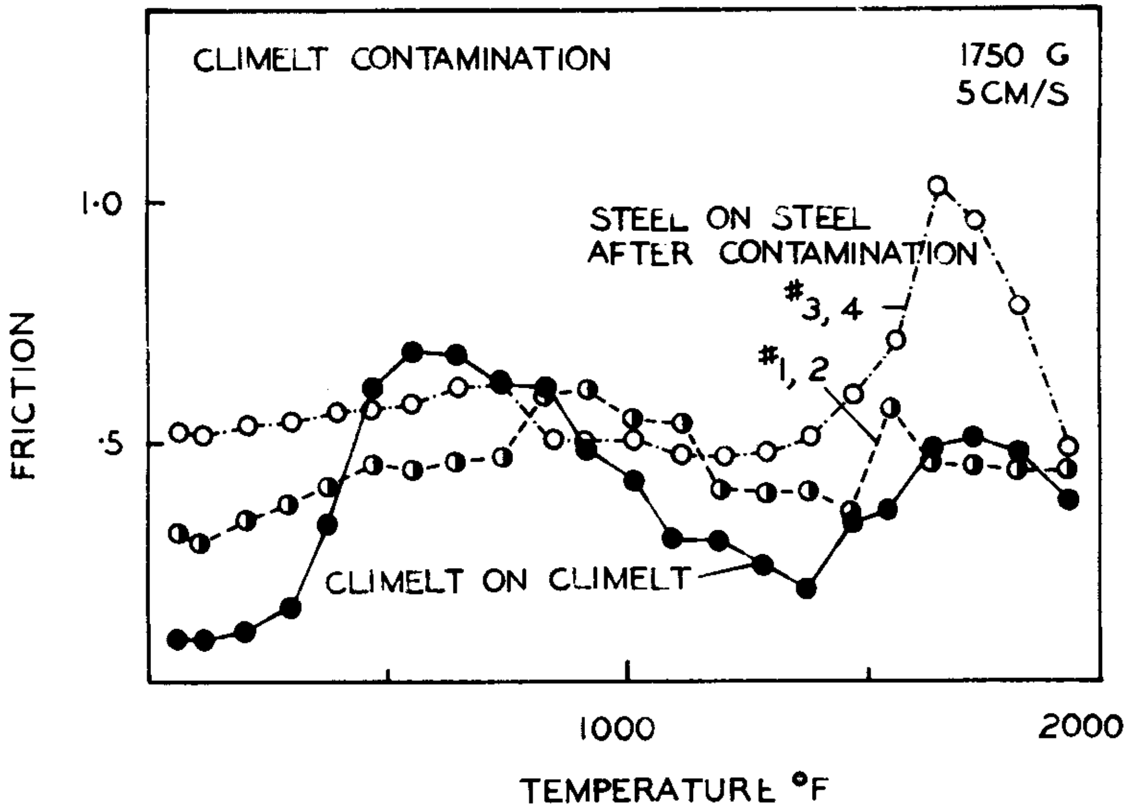


FIGURE 8

Friction-temperature Plot for Climelt



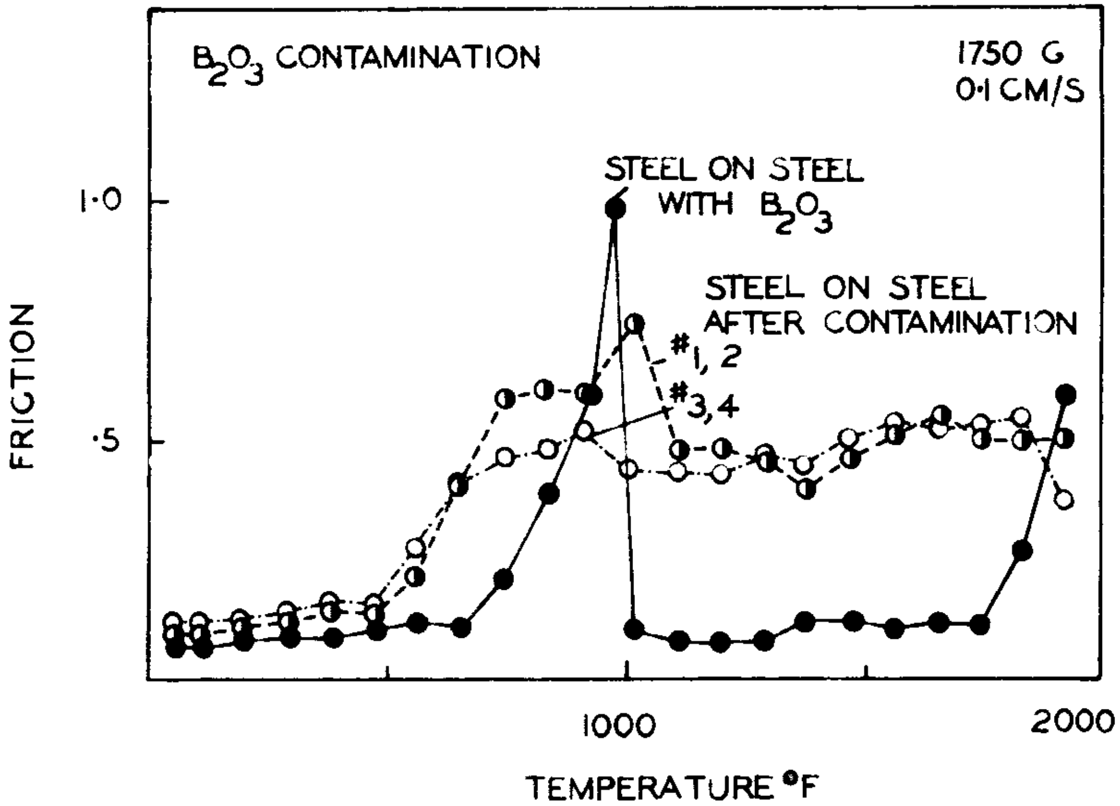


FIGURE 9

Contamination Effects on Friction Data

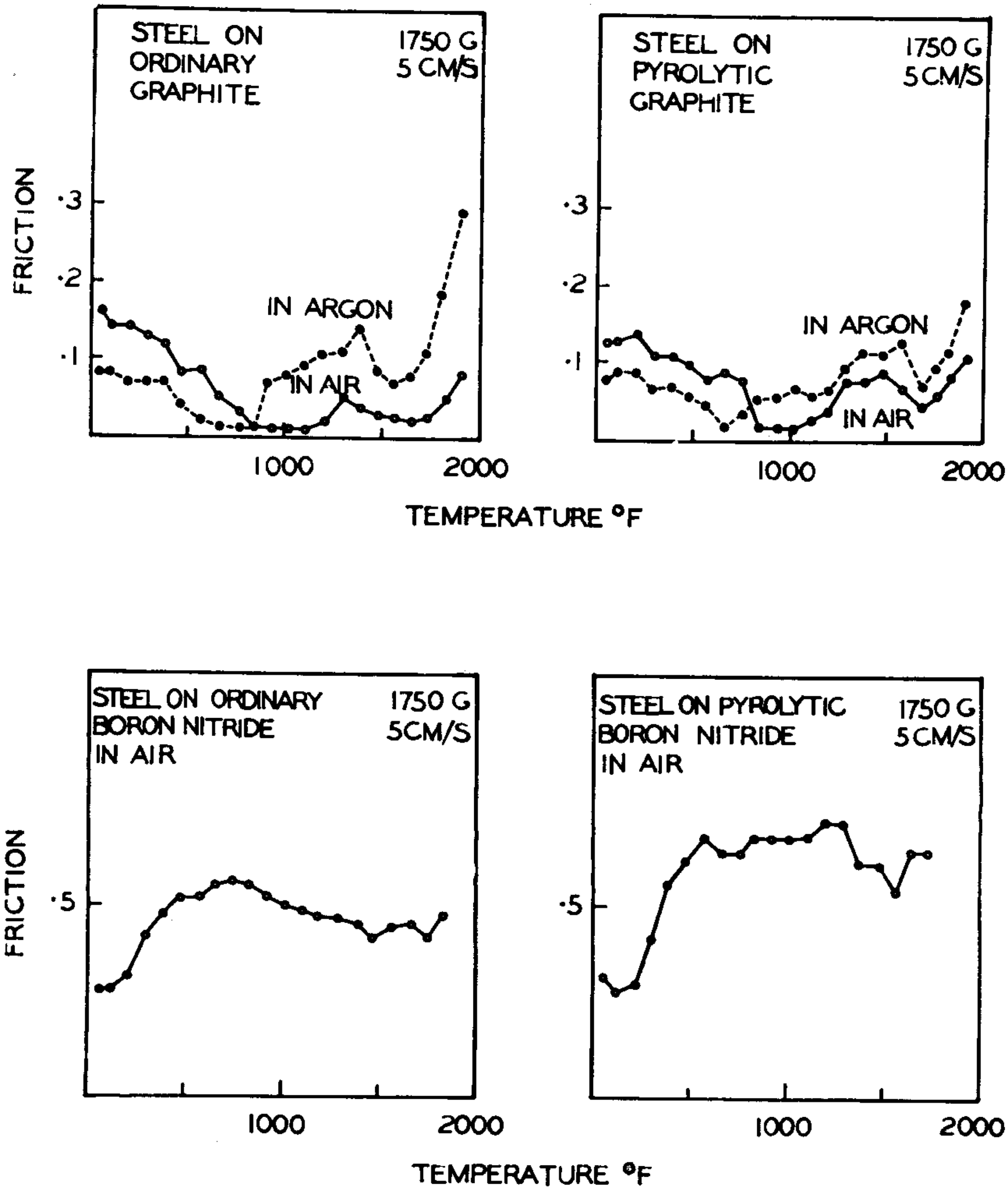


FIGURE 10

Friction-temperature Plot for Ordinary Graphite, Pyrolytic Graphite, Ordinary Boron Nitride and Pyrolytic Boron Nitride

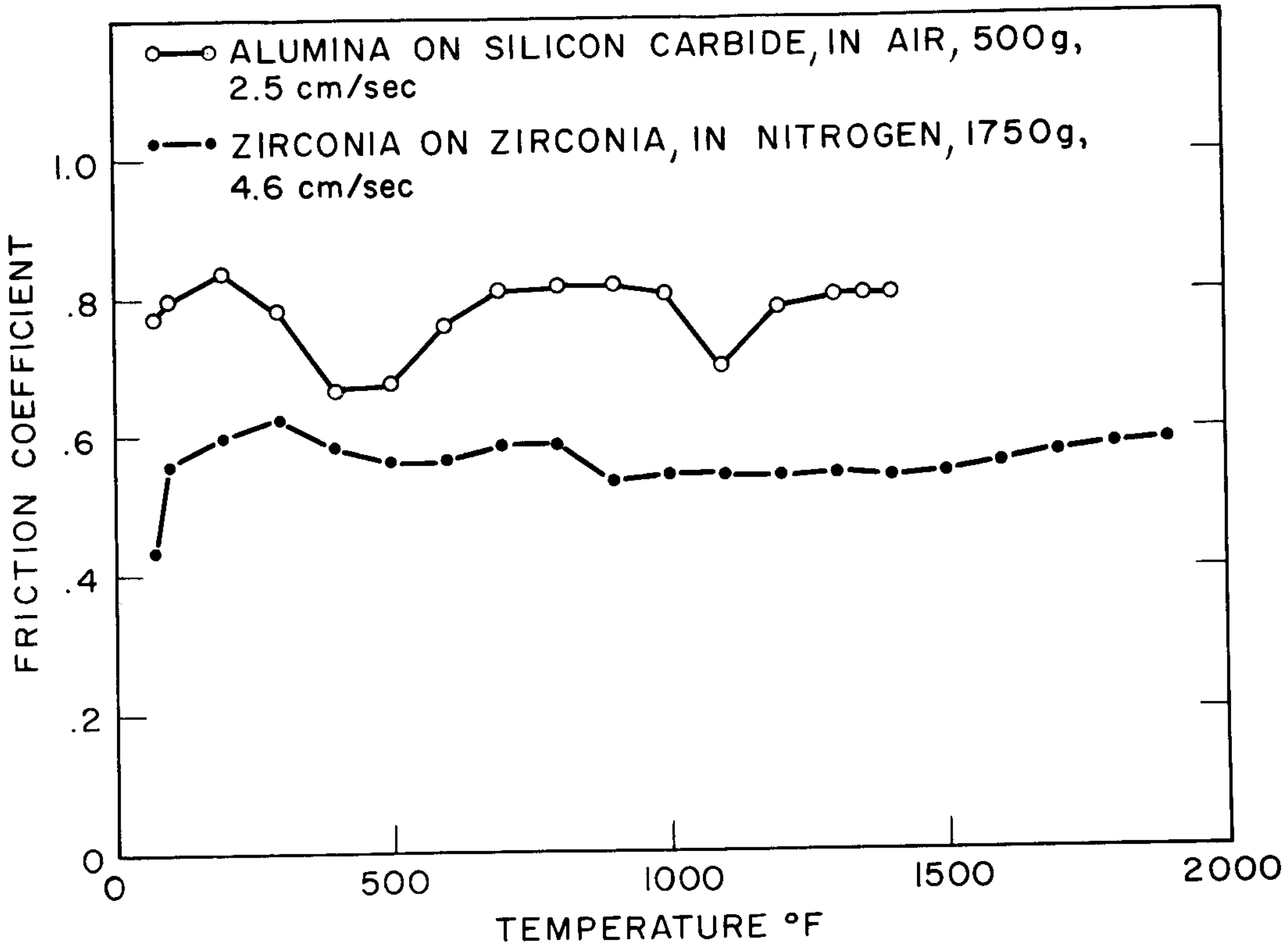


Figure 11. Two Friction-temperature Plots Which Show No Features of Interest Other than Minor Friction Fluctuations

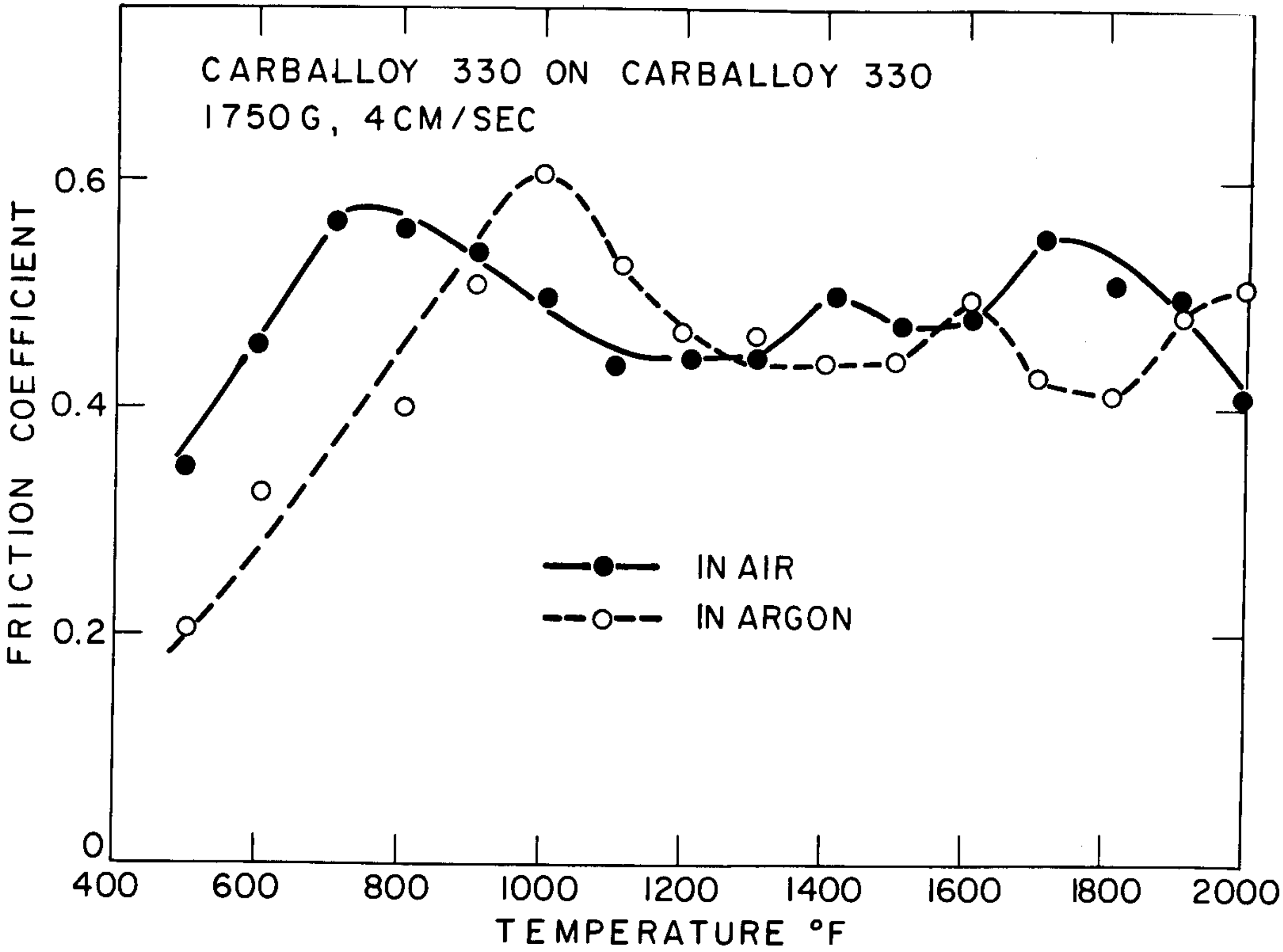


Figure 12. Friction-temperature Plots of a Commercial Tungsten-Titanium Carbide Sliding on Itself in Air and in Argon. The Data Obtained in Argon are Displaced about 200 F from the Data Obtained in Air

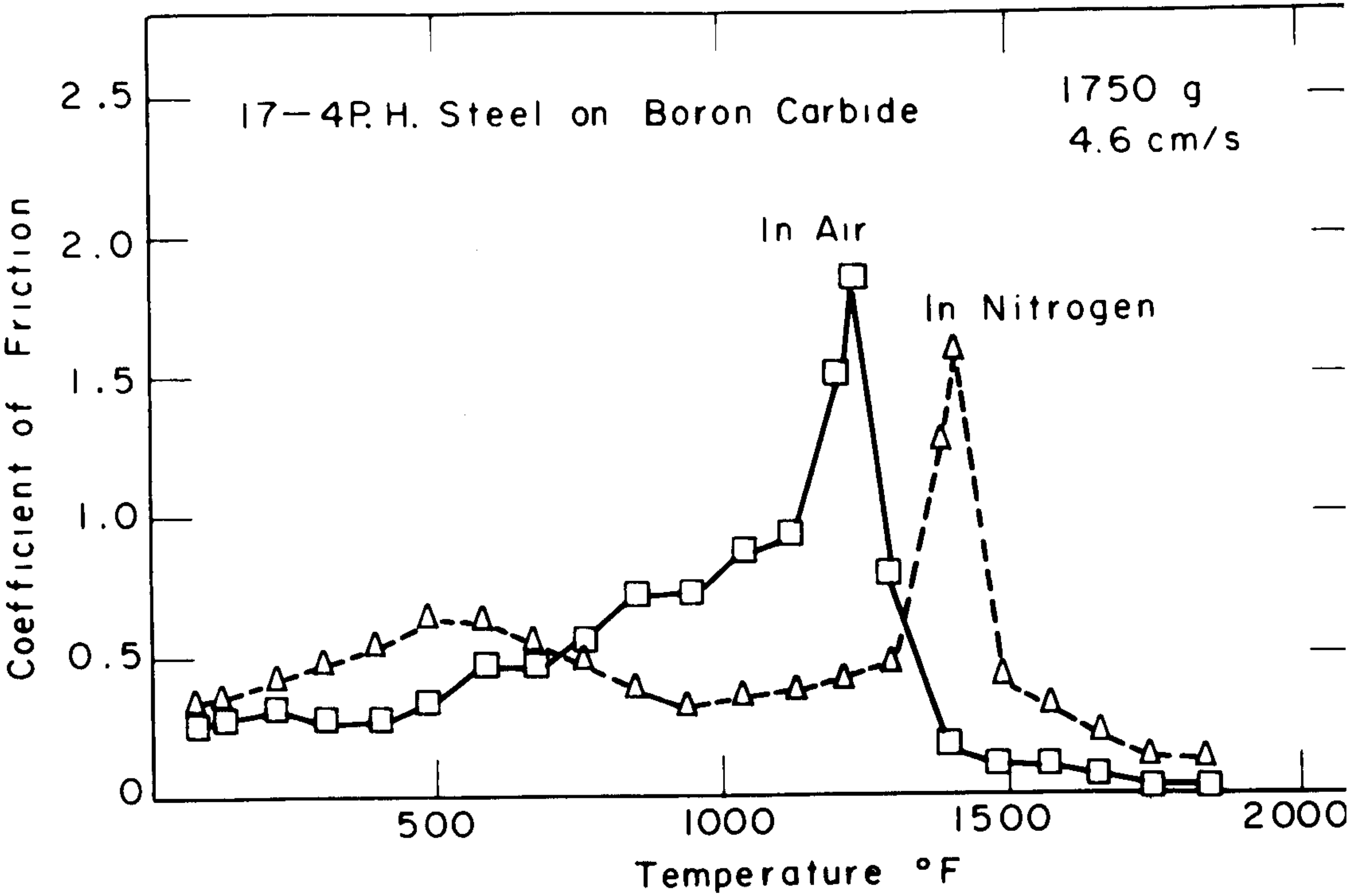


Figure 13. Friction Temperature Plots for a Steel on Boron Carbide in Air and in Nitrogen. The Peak Obtained in Nitrogen is Displaced about 200 F from that Obtained in Air.

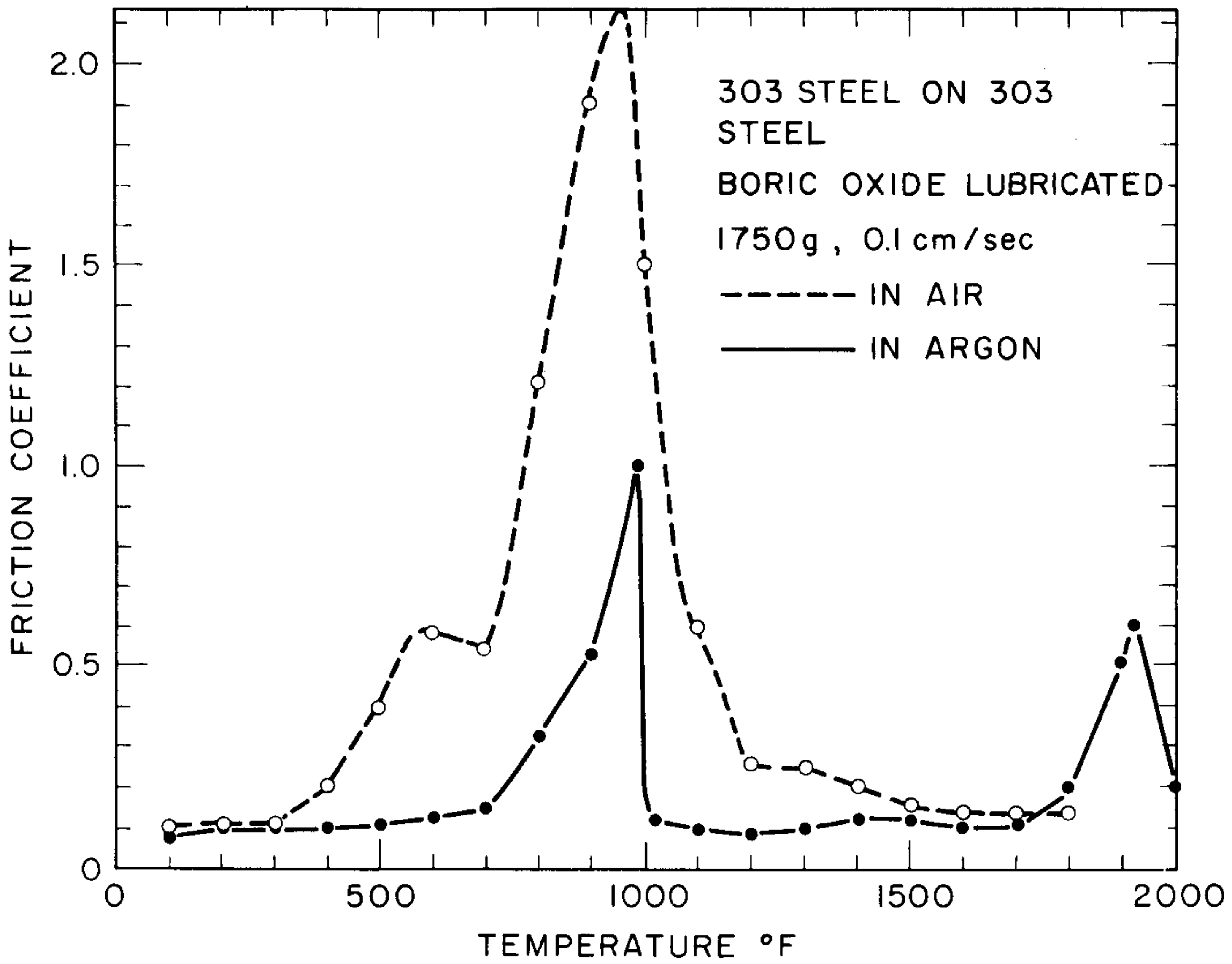


Figure 14. Friction-temperature Plots Obtained in Air and in Argon with a Steel Sliding on Itself while Lubricated by Boric Oxide. The height of the Peaks is Rather Different, but Their Position is the Same

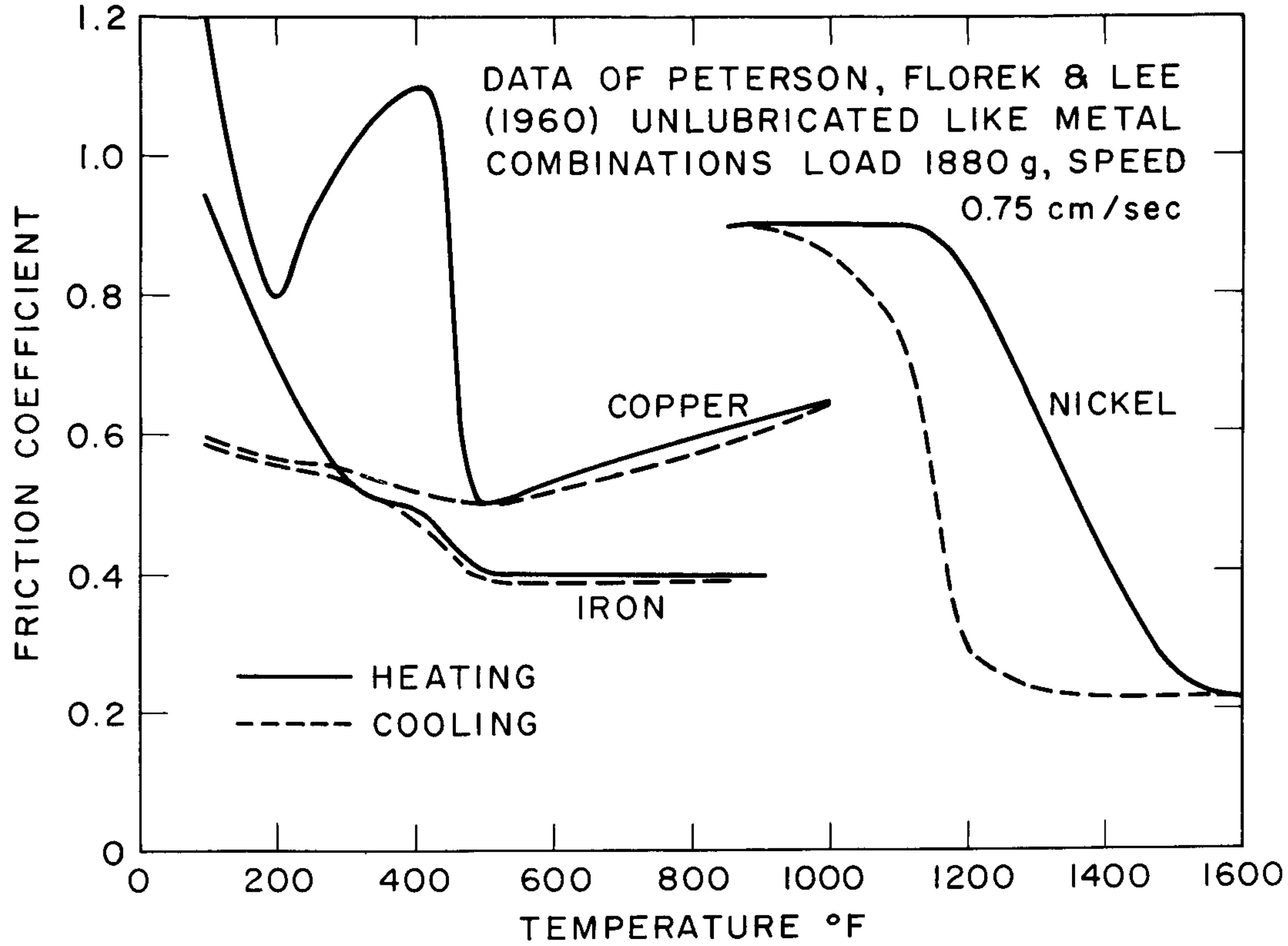


Figure 15. Friction-temperature Plots for Unlubricated Like Metal Combinations. Nickel Forms a Low Friction Oxide Above 1300 F. On Cooling, This Persists to 1100 F. The Oxides Formed with Copper at 400 F and with Iron at 200 F Persist, on Cooling, to Room Temperatures

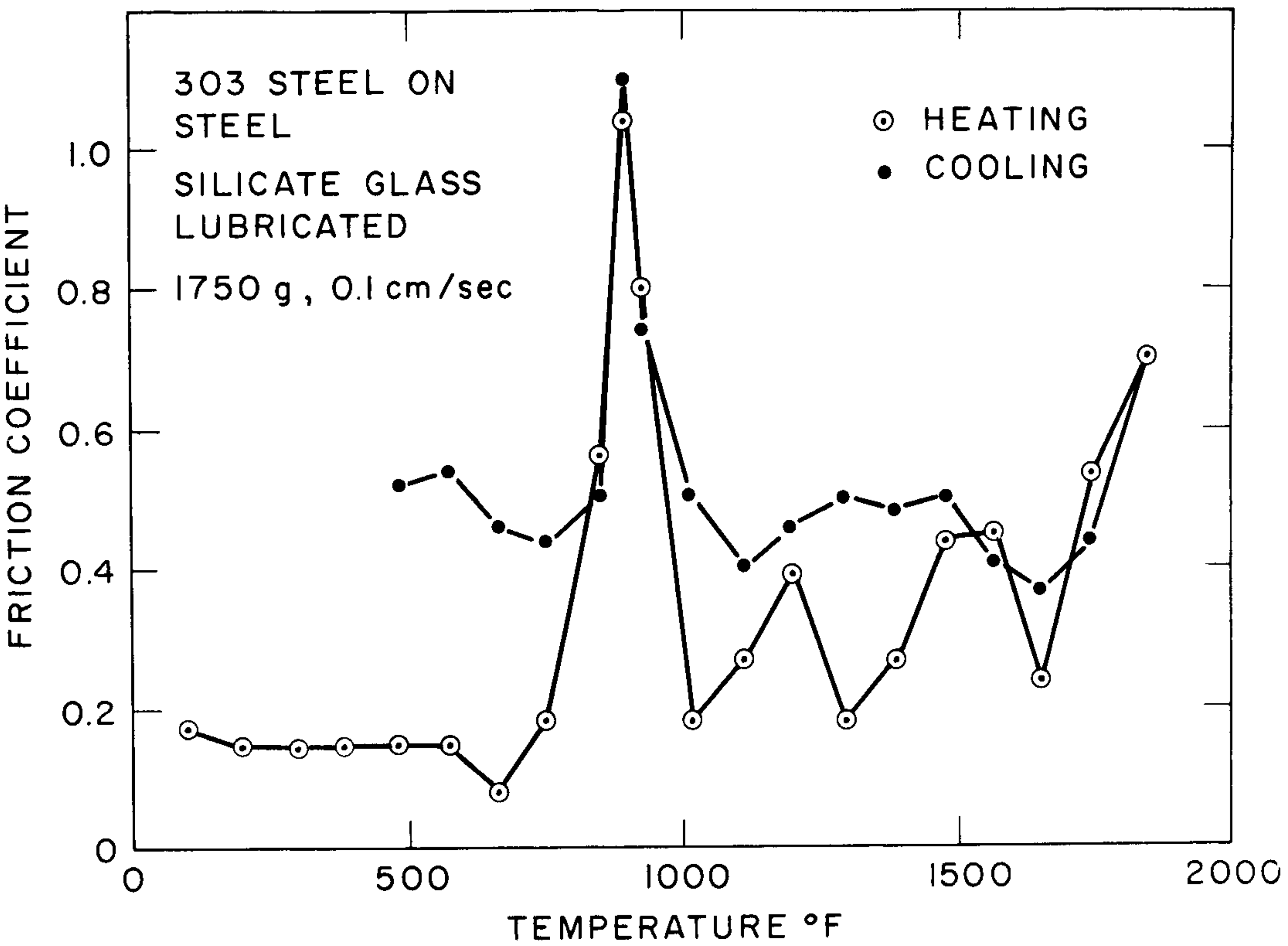


Figure 16. Friction-temperature Plots for a Steel Lubricated by a Silicate Glass. On Cooling the Peak Near 850 F Occurs at the Same Temperature, Suggesting That It Is Not Due to Oxidation of the Steel, But to Viscous Effect of the Glass.

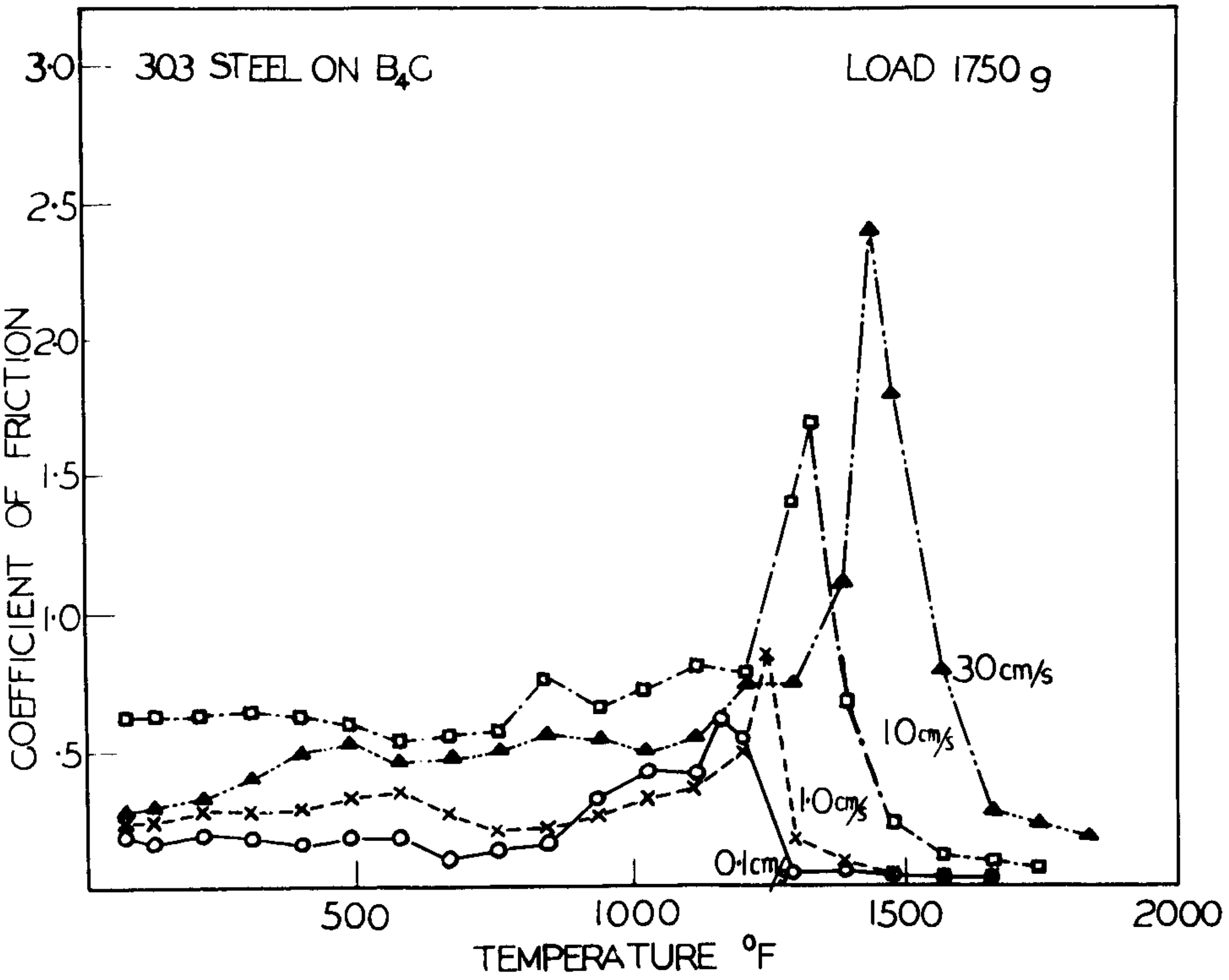


Figure 17. Friction-temperature Plots for a Steel Sliding on Boron Carbide. Since Viscosity of the Boric Oxide, Formed by Oxidation is Responsible for the Peak, the Peak is Very Velocity-dependent.

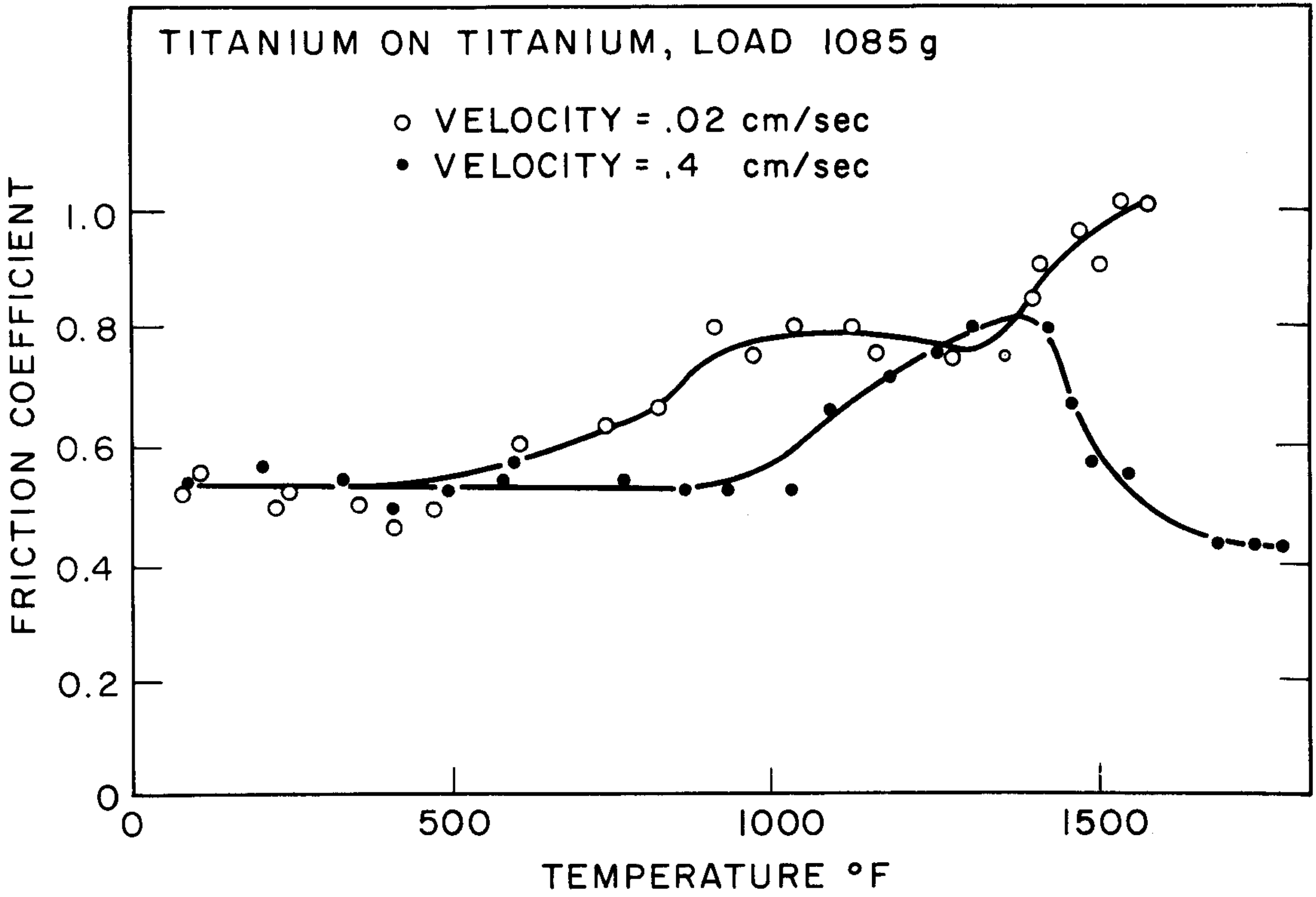


Figure 18. Friction-temperature Plots for Titanium on Titanium at High Temperatures; the Oxide on the Titanium Surface is Apparently Brittle if Allowed to Get too Thick. Hence, at Low Sliding Speeds the Oxide is Removed and Friction is High, While at High Sliding Speeds the Oxide Remains and Friction is Low

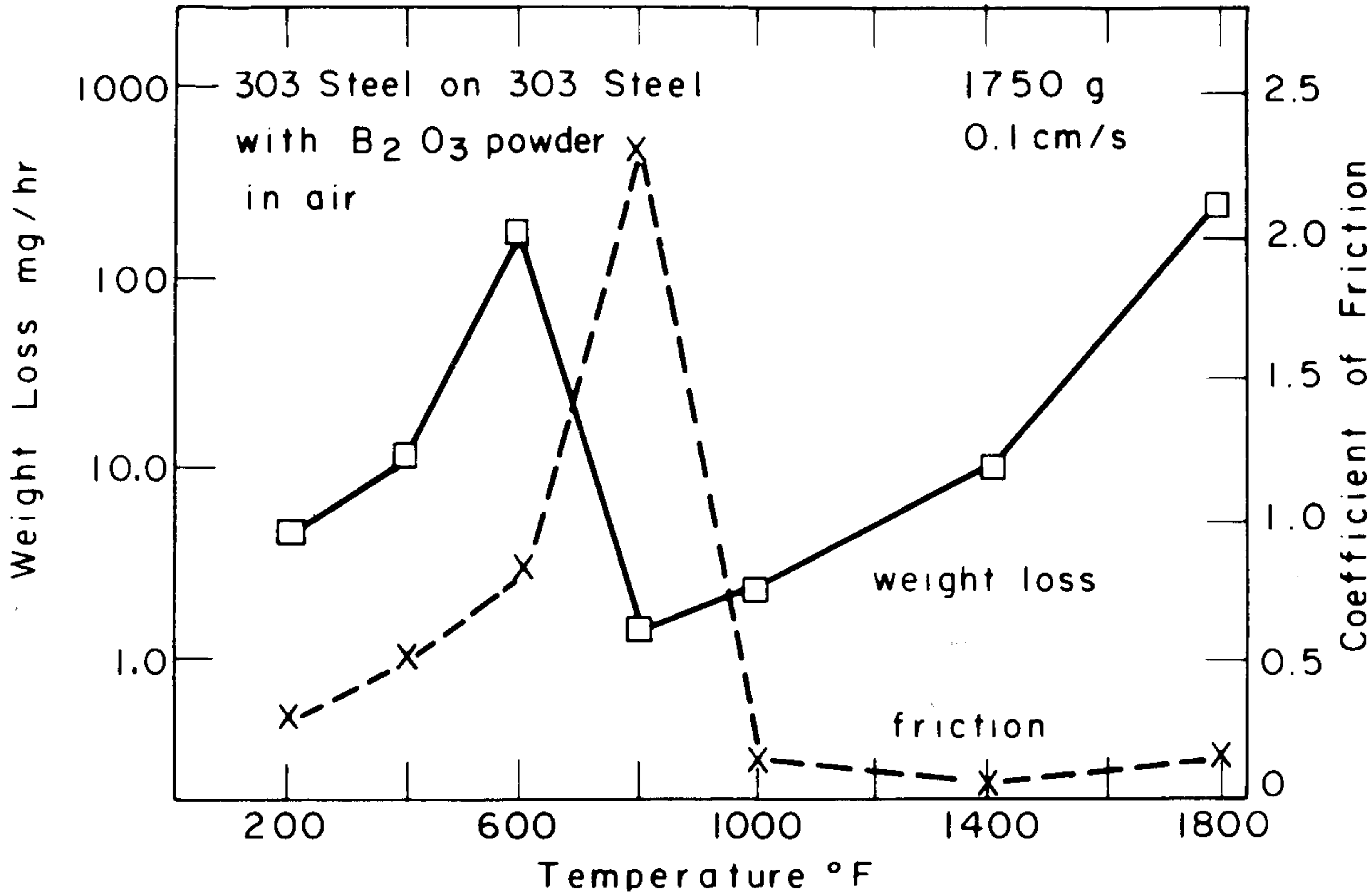


Figure 19. Friction and Loss of Weight as a Function of Temperature on Steel on Steel Lubricated by Boric Oxide. The Weight Loss is a Minimum When the Friction is a Maximum, Suggesting That High Friction is not Caused by High Metallic Interaction

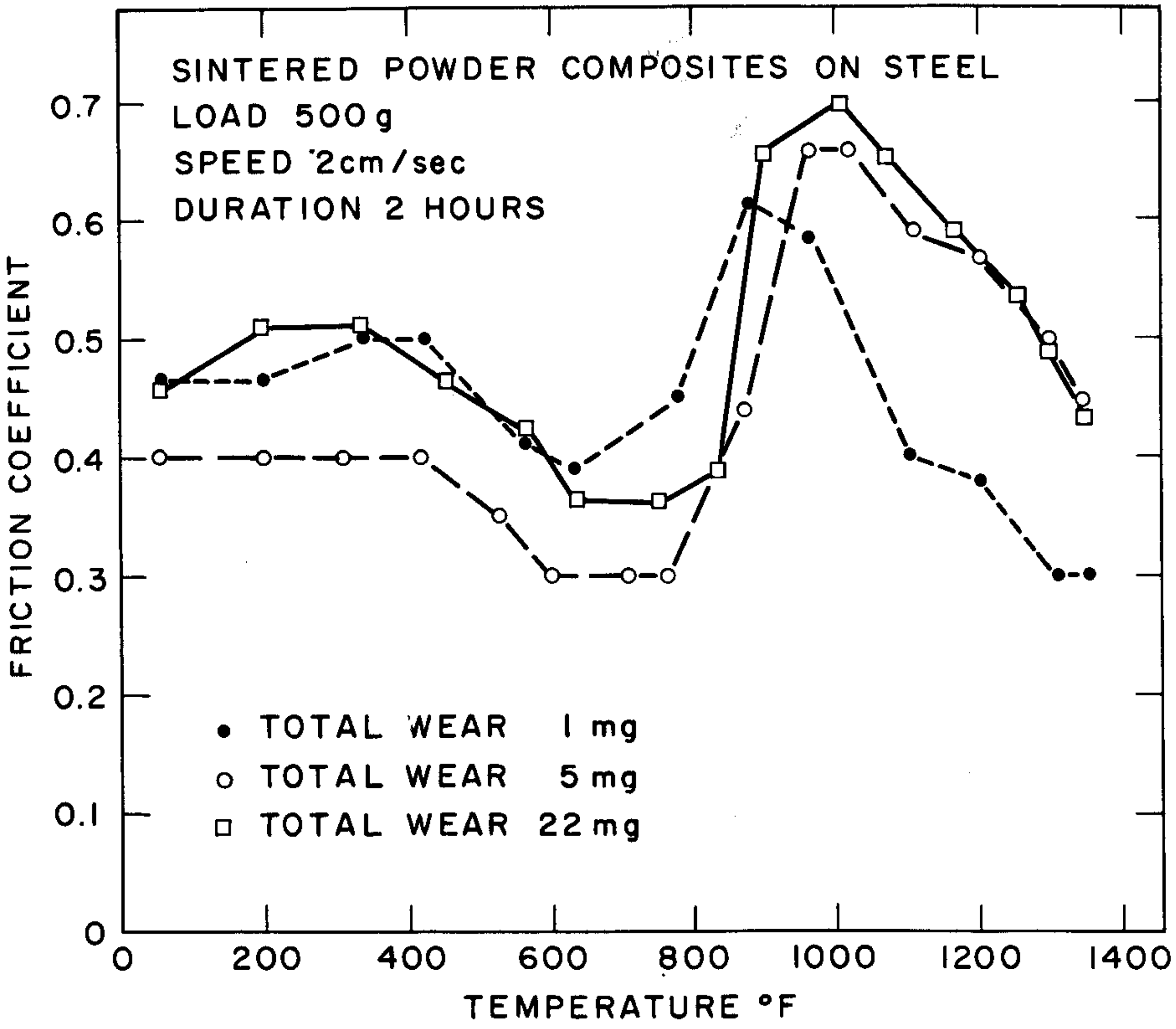


Figure 20. Friction-temperature Plots for Three Sintered Powder Compacts of Almost Identical Composition but Different Mechanical Strength. The Friction Was Very Similar, but the Wear Rates Differed Very Markedly

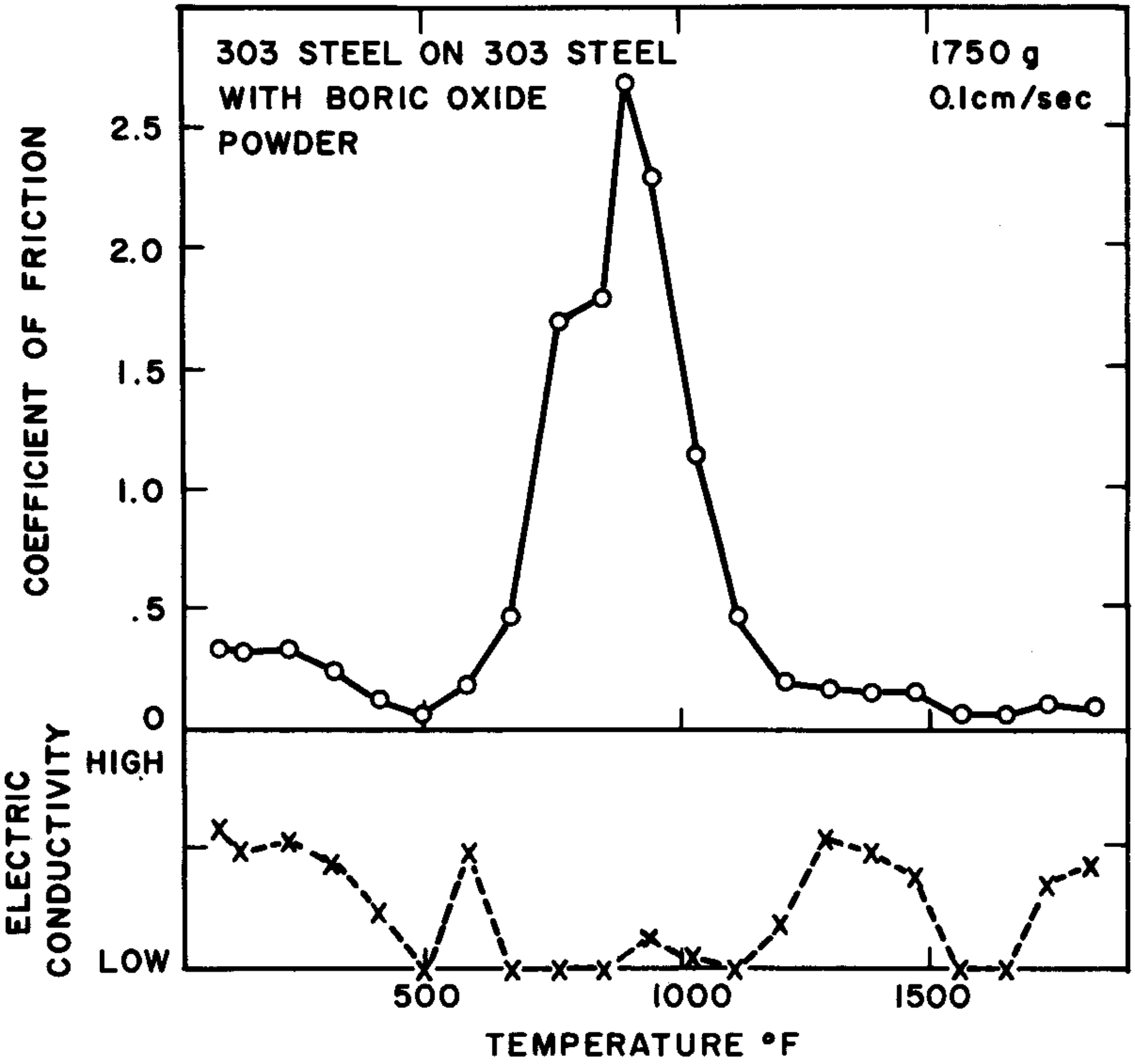


Figure 21

Friction and electrical conductivity data for steel on steel lubricated by boric oxide. The low conductivity at peak friction shows that there is no metallic interaction (i.f. Figure 19)

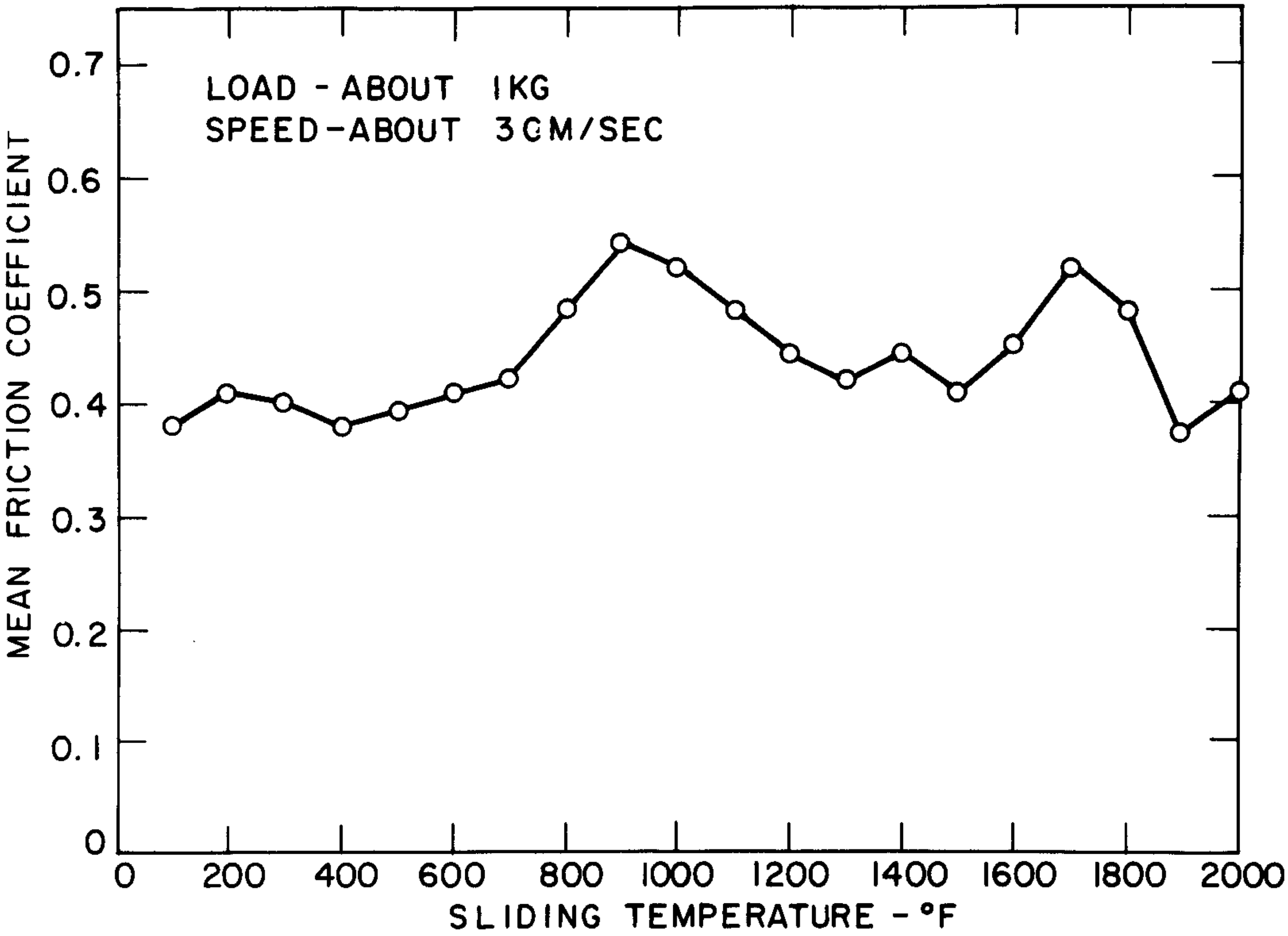


Figure 22. Average Friction for 50 Friction-temperature Runs Plotted as a Function of Temperature. The Peaks and Dips are not Statistically Significant.

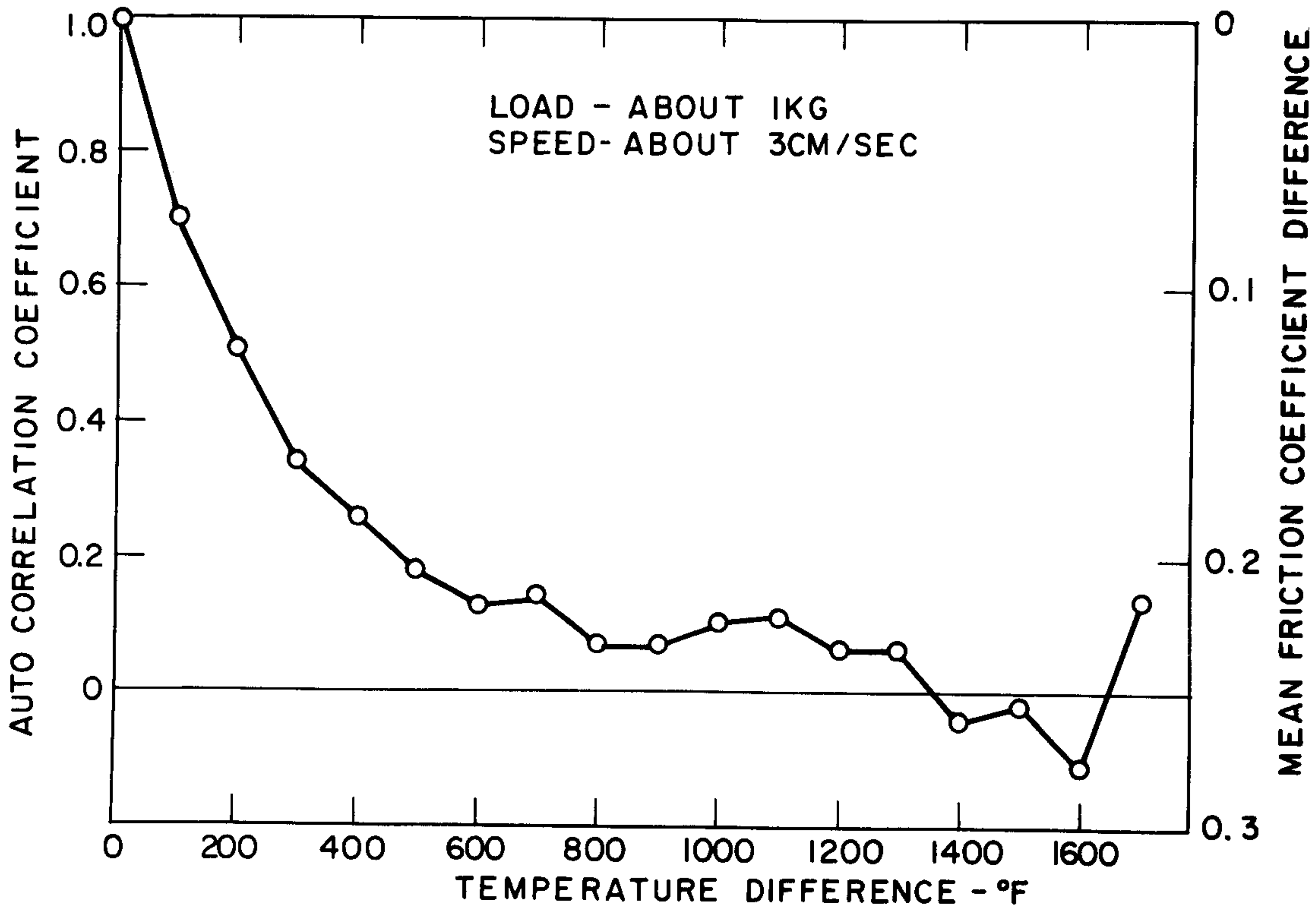


Figure 23. Plot of Autocorrelation Coefficient and Average Friction Coefficient Difference as a Function of Temperature Separation for the Runs Used in Figure 22. At Temperature Difference Exceeding 400 F, the Autocorrelation is Quite Low

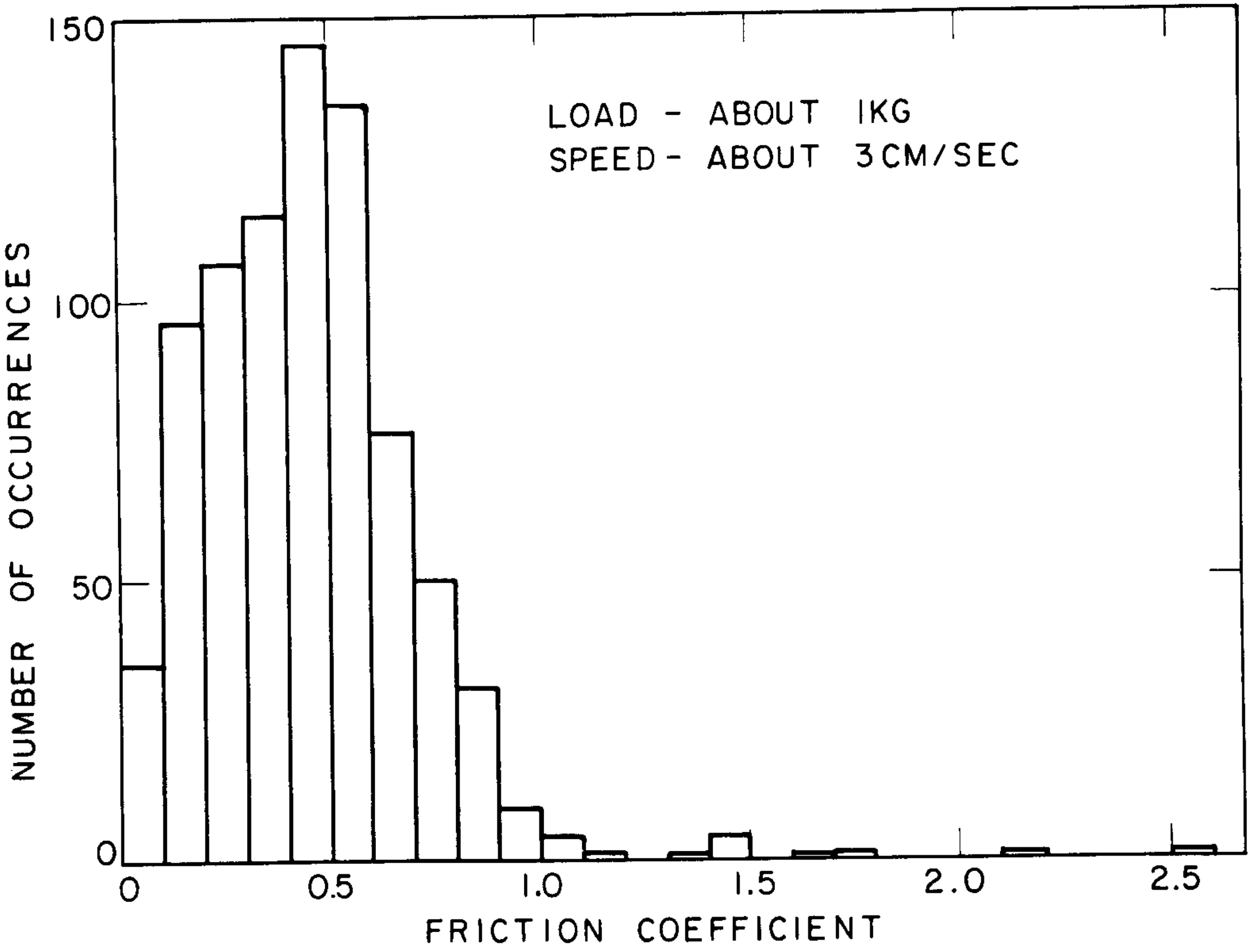


Figure 24. The Distribution of Friction Values for the 50 Runs Used in Devising Figures 22 and 23. Friction Coefficients above 1.0 or Below .1 are Rare

Table 1 Friction of Graphite at Room Temperature

Load Gram	Speed cm/s	Friction		Lubrication
		Steel on Ordinary Graphite	Steel on Pyrolytic Graphite	
500	1.0	.15	.15	Dry
500	.3	.14	.13	"
1000	.3	.15	.13	"
500	1.0	.08	.08	Cetane
500	.3	.08	.09	"
1000	.3	.09	.08	"
500	1.0	.07	.07	Palm. acid
500	.3	.08	.08	"
1000	.3	.08	.08	"

Table 2 Friction of Boron Nitride at Room Temperature

Load Gram	Speed cm/s	Friction			Lubrication
		Ordinary Boron Nitride on Ordinary Boron Nitride	Steel on Ordinary Boron Nitride	Steel on Pyrolytic Boron Nitride	
500	1.0	.18	.18	.20	Dry
500	.3	.16	.17	.22	"
1000	.3	.16	.17	.23	"
500	1.0	.06	.07	.09	Cetane
500	.3	.08	.10	.10	"
1000	.3	.08	.08	.09	"
500	1.0	.06	.06	.08	Palm. Acid
500	.3	.07	.07	.10	"
1000	.3	.07	.07	.07	"

Table 3 Wear of Boron Nitride at Room Temperature

Dry, Load 500 g, Speed 3.0 cm/s

Duration Minute	Wear mg	
	Steel on Ordinary Boron Nitride	Steel on Pyrolytic Boron Nitride
30	2.0	2.3
90	7.2	4.7
120	9.2	7.0