NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 2460

FORMATION OF SULFIDE FILMS ON STEEL AND EFFECT OF SUCH FILMS ON STATIC FRICTION

By Erva C. Levine and Marshall B. Peterson

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SUMMARY

Experimental studies were conducted to evaluate the formation, under transient temperature conditions, of sulfide films on heated steel specimens immersed in solutions of free sulfur in cetane and to establish the necessary sulfide-film thickness for effective lubrication under static-friction conditions.

With the particular steel specimens used in these experiments, the minimum temperatures at which iron sulfide is formed on the heated specimens in cetane solutions containing 0.1 to 1.0 percent free sulfur by weight were from 700° to 375° F, respectively. By establishing the cooling rates of hot specimens inserted in cool solutions, amounts of reaction product formed at various temperatures with varied concentrations, and time-temperature relations in all experiments, it was possible to calculate the rate of formation of the reaction film. The calculated rate of film formation on steel in a solution of 0.5 percent free sulfur in cetane was approximately 1.8×10^5 Angstrom units per second in the temperature range from 1100° to 1000° F.

Static-friction data were obtained with clean dry steel (coefficient of static friction, 0.79) and for steel coated with sulfide films of various thickness. Dry film thicknesses of 5000 Angstrom units or greater were found necessary to prevent surface welding completely and to produce relatively low friction (coefficient of static friction, 0.32 to 0.39). Welding was appreciably reduced, however, with film thicknesses as low as 3400 Angstrom units, although friction was reduced only slightly. This result indicated that thin sulfide films were somewhat effective even though complete prevention of welding was not obtained.

INTRODUCTION

Experimental studies by numerous investigators (references 1 to 5) have established the concept that the effectiveness of so-called extreme-pressure lubricant additives is dependent on chemical reaction between the additives and the lubricated surfaces to produce a solid surface film having desirable lubricating properties. High surface temperatures are necessary for the proper functioning of extreme-pressure lubricants. Bowden and Ridler (reference 6) have demonstrated that surface temperatures occurring on effectively lubricated sliders may be above 600° C (1112° F) and that, in many cases, localized surface-temperature flashes approach the melting point of one of the slider materials. With continued motion of sliders, a series of localized temperature flashes occurs with the total amount of heat energy (but not necessarily the maximum temperature of the surface) being dependent on the loading. As suggested in reference 5, "extreme temperature" lubricants might be a more accurate name because they are primarily temperature active.

The complete mechanism of action of the temperature-active lubricants is not known because of its complexity. However, the action is dependent on the composition and structure of slider materials, the composition of the atmosphere, and the physical conditions of sliding as well as the composition of the lubricants. A series of possible chemical reactions that might occur with sulfur additives (among others) is outlined in reference 1; the lubricating film that is formed on steel surfaces was found to be iron sulfide.

The solid reaction film formed by the temperature-active lubricants must possess desirable lubricating properties under static and dynamic conditions. An investigation of the lubricating properties of such films under dynamic conditions is reported in reference 7. It is suggested, in reference 7, that at high sliding velocities the time increment during which mating surface areas are in intimate contact is so short that an effective lubricating film cannot be formed; this hypothesis is substantiated in reference 8. Static-friction experiments to establish the minimum film thickness of sulfide on copper surfaces that is effective in providing solid-film lubrication are reported in references 9 and 10. Such information is unavailable for steel surfaces and a basis for quantitatively defining an effective lubricating film of sulfide on steel is needed.

The present investigation was conducted at the NACA Lewis laboratory: (1) to study chemically the formation of sulfide films on steel, and (2) to establish by a friction investigation the sulfide film thickness necessary for effective lubrication under static conditions.

The data on film formation were obtained under transient temperature conditions with heated small steel specimens inserted in solutions of free sulfur dissolved in cetane. The effect of sulfide film thickness on lubrication was determined with a static-friction apparatus.

APPARATUS AND PROCEDURE

The separate phases of this study (chemical and static-friction investigations) will be discussed independently.

Chemical Investigation

Method. - The apparatus used for the formation of iron sulfide films on rectangular specimens is shown in figure 1. This equipment consisted primarily of a small electric furnace and two weighing bottles, which contained the lubricant solutions, all mounted on a turntable within a covered glass chamber. The chamber was provided with an atmosphere of helium, external controls for rotating the turntable, and a means for raising or lowering the steel specimen into the furnace and the solutions.

The specimen and specimen holder (fig. 1(b)) were first lowered into the furnace and heated to a predetermined temperature, which was measured by a contact thermocouple pressed into an indentation at the top of the specimen. With the thermocouple in this position, the bulk temperature of the material rather than the surface temperature was measured. The heated specimen was then raised from the furnace, the turntable was rotated to the proper position, and the hot steel specimen was inserted in the cool (room temperature) solution of free sulfur in cetane. The concentration of sulfur in cetane was varied from 0.1 to 1 percent. After being taken from the furnace and while in the solution, the specimen temperature was observed and recorded using a recording oscillograph, which gave a sensitive record of temperature as a function of time. The temperature sensitivity of ±10° F is dependent on the cyclical frequency of the recording equipment and the maximum cooling rate. The instant of contact and the temperature of the specimen at contact with the solution was established by the abrupt change in the cooling rate of the specimen, as indicated on the time-temperature record. The experiments were repeated a number of times to obtain data at the initial temperatures desired.

With this apparatus, experiments were conducted to establish the minimum temperature at which detectable (by means of chemical spot test) iron sulfide films would be formed in solutions of free sulfur in cetane. This minimum temperature was taken as that temperature below which a negligible amount of sulfide is formed; the total amount of sulfide formed on the specimen at each immersion temperature was obtained by quantitative chemical tests. If the amount of sulfide formed per unit area at various temperatures and the time-temperature relations are known, the amount of sulfide formed per second and consequently the sulfide-film thickness formed per second can be determined.

In the chemical experiments, a transient temperature was generally used because it approaches the condition found in sliding surfaces and because a fairly constant cooling rate in the temperature range of interest allowed a reasonable understanding of the time-temperature phenomena. For time increments which are within the measuring sensitivity of the instrument employed (0.0083 sec), reasonable approximations in rate of film formation and cooling rate could be made. At the beginning of the surface reaction, the rate of film formation may be an exponential function of time; but for the purposes of these experiments it is necessary within small time (or temperature) increments to assume this relation to be linear.

Specimen preparation. - Specimen material with low sulfur content was selected to minimize errors in the spot test used because untreated steel specimens with high sulfur content gave positive tests for sulfide. Untreated pure iron (National Bureau of Standards 99.8-percent iron) and selected low-sulfur steels gave negative results; accordingly, steels containing less than 0.035 percent sulfur were used. Because it was noted that the presence of oxides on the specimen inhibited the formation of sulfide films, a helium atmosphere was used to minimize oxidation of the metal and the solution.

The rectangular specimens (1/4 by 1/4 by 3/32 in.) were cleaned by abrasion with 0 and 3/0 metallographic paper and then washed with acetone and benzene.

Qualitative and quantitative identification of sulfide. - The Feigl spot test (reference 11) was used to establish the presence of sulfur as sulfide on the treated specimens in the experimental work reported herein. The identity of the reaction product iron sulfide was verified in several control tests by means of electron diffraction. The Feigl spot test is sensitive to less than 1 part per million of sulfur as sulfide; therefore, it was possible to establish the presence of a sulfide film of such minute thickness that it could not be detected by increase in weight or change in the appearance of the surface.

Quantitative determinations were obtained by a modification of the Feigl spot test. Standard solutions of p-aminodimethylaniline containing sulfide have easily discernible characteristic colors that vary greatly with concentration of sulfide. By comparing the color of the standard solutions having known concentrations of sulfide with solutions prepared by dissolving the sulfide film from the specimens (in a spot plate containing 2 cc of solution), it was possible to establish the concentration of sulfur as sulfide. The color of the sulfide and p-aminodimethlyaniline solutions is unstable for extended periods of time, consequently, standard water-color solutions were mixed very carefully to match the desired gradation of sulfide colors. The color from the tested sulfide solution was immediately visually compared with the standard water-color solutions to establish concentrations. The sensitivity of this method of quantitative evaluation was sufficient for the detection of a change of 0.1 milligrams per 100 cubic centimeters in the amount of sulfur from the sulfide in the test solutions.

The thicknesses of the films were calculated from simple weight, area, and density values. The following assumptions were made in the calculations:

(1) The increase in weight was entirely due to the combination of sulfur with the iron of the specimen

(2) The density of the FeS film was 4.84 grams per cubic centimeter

(3) The film thickness on the specimens was uniform

(4) The calculated surface area of the specimen was the real surface area

The real surface area is appreciably greater than the apparent area; and because accurate determination of the real surface area is difficult, the apparent area was used in the calculations. The real film thickness was therefore less than that calculated.

In order to verify the results obtained by the method just described, a second method of establishing the minimum reaction temperature was used. Solutions of various concentrations of free sulfur in cetane were heated to specified temperatures. Small steel specimens were each immersed in the heated solutions for a period of 1 minute and then removed. The minimum temperatures at which the sulfide was formed were again determined.

Static-Friction Investigation

Apparatus. - The static-friction experiments were performed with the apparatus shown in figure 2. The principal parts of the apparatus (fig. 2(b)) were the rider assembly (containing three ball specimens of hardened steel; Rockwell C-60) and the plate specimen (hardness, Rockwell A-150) which the balls contacted. A ball specimen was securely clamped in the rider at each of the vertices of an equilateral triangle (as shown in figure 2(c)) and the total load was applied at the center of the triangle normal to its plane. It was assumed that the total load was supported equally on the three balls. The plate (SAE 1020) was securely fastened to the base of the apparatus. The static-friction force was measured with a dynamometer-ring, strain-gage assembly similar to that described in reference 12. A photoelectric recording potentiometer was used as a friction-force indicator. The apparatus was contained within a chamber having provisions for maintaining an atmosphere of dry air. Preliminary experiments showed that humidities above 10 percent reduced the static-friction force; therefore, friction runs were made with relative humidity maintained in a range between approximately 7 to 10 percent.

The force necessary to overcome static friction was applied slowly by elastically extending a dynamometer ring on which the force indicating strain gages were mounted. The dynamometer ring in the strain-gage assembly was connected to the rider assembly by a fine wire. The displacement rate of 0.0156 inch per minute in this system was produced by a 1 rpm motor rotating a fine pitch (64 threads/in.) screw. The initial motion of the rider assembly was observed through a telescope fitted with cross-hairs. When motion was observed, an indicating mark was made on the time-force record in the potentiometer.

Misalinement resulted in decreased and erratic friction values as well as skewing of the rider assembly. This difficulty was minimized by a jig placed over the plate (fig. 2(b)). The jig caused the rider assembly to be positioned directly in the line of force application and strain measurement.

Specimen preparation. - The plate surfaces were cleaned by the procedure described in reference 12. The steel balls (SAE 52100) were cleaned by rigorous washing in 100-percent alcohol, then in a solution of 50 percent acetene and 50 percent benzene, followed by two washings in 100-percent alcohol. This procedure removed all grease from the surface; the absence of contamination was confirmed by electron diffraction.

For the friction investigation, iron sulfide films were formed on the ball specimens by heating and then cooling, both in an atmosphere of hydrogen sulfide, for predetermined periods of time. Thickness of the film was calculated from weight gain and was checked by means of the modified spot test described earlier. In most cases, the results agreed within 10 percent. All weight gain was assumed to be caused by the addition of sulfur which was stoichiometrically combined with iron to form iron sulfide.

RESULTS AND DISCUSSION

The separate phases of this study (chemical and static-friction investigations) will be discussed independently.

Chemical Investigation

Minimum reaction temperatures. - The minimum reaction temperature obtained in this investigation is defined as that immersion temperature below which no iron sulfide could be detected by the Feigl spot test on the specimens. A plot of temperature against weight concentration of sulfur in cetane showing the minimum reaction temperatures obtained by two methods is shown in figure 3. The first method used in most of the work reported herein consisted in immersing heated specimens in solutions of sulfur in cetane. In the second method, solutions of various concentrations of free sulfur in cetane were heated to specified temperatures below the boiling point of cetane. Small steel specimens were each immersed in the heated solutions for a period of 1 minute and then removed. The data obtained after a 1-minute immersion of unheated steel specimens in heated solutions checks the data obtained by immersing heated specimens in room-temperature solutions. The minimum reaction temperatures indicated for the first method are those that were observed at the instant of contact of the specimens with the solutions. As expected, these data show that as concentration increases, minimum reaction temperature decreases; the greatest decrease in minimum reaction temperature occurs at concentrations up to 0.4 weight percent. Very little decrease in minimum reaction temperature occurs with increased concentration above 0.4 weight percent. As an example, when the sulfur concentration was doubled (0.8 weight percent), the minimum reaction temperature was decreased approximately 35° F. The minimum reaction temperatures obtained with pure iron wire and tool steel were essentially the same as the data for mild steel (low sulfur content) presented in figure 3.

Film formation characteristics. - The data reported in figure 4 show that the rate of sulfide film formation was the greatest with the initial specimen temperatures from 700° to 1100° F for all concentrations of sulfur solutions. As previously mentioned, reference 6 indicates that temperatures in this range may be characteristic of boundary lubricated surfaces. It is therefore apparent that the greatest film formation occurs at temperatures of importance in lubrication.

Study of the oscillograph time-temperature records from the experiments that provided the data presented in figures 4 and 5 indicated that the rate of cooling of the specimens was essentially constant within the temperature range of interest. The typical cooling-rate curve presented in figure 5 indicates that the average time required for the rectangular specimens to cool 100° F, in a range of temperatures (from 1100° to 500° F) in which chemical reaction would occur, was approximately 0.04 second.

For purposes of evaluation, it is assumed that it took the specimen 0.04 second to cool from 1100° to 1000° F, a 100° F increment, in a 0.5 percent solution. The amount of sulfide formed in that increment can be determined by reference to figure 4. The difference between the quantity formed at 1100° F and the quantity formed at 1000° F is divided by the cooling time for the 100° F increment (0.04 sec). This calculation indicates that the rate of film formation during that increment of time and temperature was approximately 1.8×10^5 Angstrom units per second.

For comparison purposes, experiments were made with a commercial extreme-pressure additive, dibenzyl disulfide (reference 3), in cetane.

The results were approximately the same as those obtained for similar sulfur weight concentrations of free sulfur.

According to reference 13, the reaction between sulfur bearing compounds and ferrous metals can be accelerated by the presence of sodium sulfide Na_2S. When Na_2S is used as a catalyst, the minimum reaction temperature is reduced and the rate of film formation is increased. Sodium sulfide (0.5 weight percent) and free sulfur (0.5 weight percent) dissolved in cetane reduced the minimum reaction temperature to below 300° F and caused an appreciable increase in rate of film formation at temperatures above 700° F. The use of such catalysts requires more extensive investigation because there are many obvious complications involved.

Static-Friction Investigation

Untreated steel. - The effect of load on the coefficient of static friction for clean dry steel balls on dry steel plates is shown in figure 6, which is a plot of the data from one representative experiment. These data along with substantiating unreported data establish the mean value of 0.79 for coefficient of static friction. In general, these results agree with Amonton's law showing that the coefficient of static friction is independent of load. The multiple data points shown in figure 6 for the 300-gram load were obtained at intervals throughout the experiment after runs with the heavier loads. Surface welding occurred under all conditions of loading with clean dry steel.

As indicated in figure 7 and in visual studies of surfaces, sulfide film thicknesses of 4500 Angstrom units or less were only partly effective in reducing friction and preventing welding of the steel specimens. The friction values with film thicknesses of 3400 and 4500 Angstrom units were generally somewhat less than for untreated steel and showed a slight increase in friction with increased loading. The general increase in friction with greater loads for films having thicknesses of 4500 Angstrom units or less is a deviation from Amonton's law that may indicate further progressive surface failure. For film thicknesses greater than 4500 Angstrom units, welding was effectively prevented. Static-friction-coefficient values for these effective films (thicknesses greater than 4500 A) were from 0.32 to 0.39. In general, friction-coefficient values were independent of load for all film thicknesses greater than 4500 Angstrom units.

A faired curve of cross-plotted data from figure 7 is presented in figure 8, which shows directly the effect of thickness on coefficient of static friction. A critical film thickness was established at approximately 5000 A, below which friction increased markedly and welding occurred. (A similar critical sulfide film thickness for copper was found by Campbell, reference 9, and by Greenhill, reference 10.) Little change in friction was evident with film thicknesses greater than 5000 Angstrom units and a marked decrease in the scatter of the data obtained with various loads was noted as thickness of the film was increased.

Kinetic-friction data were obtained (reference 13) for a steel sphere against a steel flat coated with a film of FeS. The FeS film reported in reference 14 was believed to be approximately 1000 Angstrom units thick. Comparison of the results of reference 14 and figure 8 show that kinetic-friction data at low sliding velocities are somewhat lower (as would be expected) than the static-friction data for the same thickness of film. The surface damage (welding) in the kinetic-friction experiments showed good correlation with that occurring in the static-friction experiments; in the kinetic-friction experiments, welding occurred with the film of 1000 Angstrom units thickness. Similarly, in the static-friction experiments, welding occurred with film thicknesses of less than 5000 Angstrom units.

Three photomicrographs (X100) of surface conditions on the flat specimens after continued motion of the ball rider specimen across the plate are presented in figure 9. The surface disturbance in figure 9(a) is the result of metallic adhesion. This adhesion or welding may cause progressive increase in track width. The appearance of this surface damage is very similar to that in figure 9(a) of reference 12 where severe welding and surface damage took place between experiments of clean steel under conditions of high sliding velocities. The ball specimen coated with an iron sulfide film 14,400 Angstrom units thick caused simple surface abrasion and smearing of a film as shown in figure 9(b); no surface disturbance comparable with that of figure 9(a) is observed. The smeared film is believed to be a transferred iron sulfide film. There is no evidence of harmful surface disturbance at the initial point of contact. Figure 9(c) shows a track made under conditions similar to those of figure 9(b) except for an increase in total load from 2400 to 4500 grams. In this case, considerably more smearing can be observed; but, as before, no harmful surface disturbance can be discerned.

Photomicrographs taken at higher magnification (X270; fig. 10) show surfaces of both the wear track on the flat specimen and the mating contact surface on the ball. The surface shown in figure 10(a) is the same one shown in figure 9(c). The appearance of the specimens at high magnification indicates that the transferred iron sulfide has smeared over surface recesses such as finishing marks.

The mating rider (ball) surface shown in figure 10(b) has a thick film of iron sulfide. The wear area can be matched with that shown in figure 10(a). The well defined edges of the contact surface in figure 10(b) and the stratification visible on the principal accumulations of iron sulfide in figure 10(a) would tend to indicate that fragmentation of the film material occurred. The light area in figure 10(b) indicates where the high load condition occurred on the stable film. The two well defined spots within the light area are believed to be load carrying "build-up's" of iron sulfide and not areas of harmful surface disturbance.

Significance of Chemical and Friction Investigations

The minimum reaction temperatures, for the particular specimens used in the chemical study, are sufficiently low so that there exists a considerable temperature range (1100° to 700° or 375° F) on sliding surfaces where a reaction film can be formed by an additive such as free sulfur. The rate of film formation calculated from the curves for both the 0.5 and 1.0 weight percent solutions of free sulfur in cetane at the maximum temperatures was approximately 1.8×10^5 Angstrom units per second.

The friction results presented in figure 8 indicate that a film of iron sulfide approximately 5000 Angstrom units thick is necessary to obtain minimum friction during unlubricated sliding. Also, with this thickness of 5000 Angstrom units welding was completely prevented. With sulfide film thicknesses as low as 3400 Angstrom units, however, visual observation indicated that welding was appreciably reduced in spite of the fact that friction was reduced only slightly. This result indicates that thin iron sulfide films can be somewhat effective even through complete prevention of welding is not obtained. These results correlated generally with those of reference 14 (for kinetic friction) where a film of iron sulfide approximately 1000 Angstrom units thick reduced friction only slightly while appreciably reducing welding. This reduction in welding for the iron sulfide film as compared with that for dry steel may be observed by comparison, resepctively, of figure 8 of reference 14 with figure 9(a) of reference 12.

In analyses of these data, it should be appreciated that many factors characteristic of dynamic sliding conditions can change the rates of reaction. For example, the rate of film formation is appreciably affected by rate of cooling and any change in the cooling rate from that obtained with the specimens of this report will thus change the rate of film formation. There are other factors of considerable importance which, because of experimental limitations, have not been included in the research reported herein. One factor is that of the effect of material stresses on rate of reaction. There is no doubt that the high contact stresses encountered in practical lubrication problems will cause a marked increase in rate of reaction in a manner similar to the mechanical activation of chemical reactions described in reference 15. Another factor is that under conditions requiring extreme-pressure lubrication, surface temperatures may be considerably above the value of 1100° F indicated in reference 6. The data of reference 6 are for effectively boundary-lubricated

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surfaces. It is obvious that additives of the type being considered will not be necessary until conventional boundary-lubricating fluids are no longer adequate. In such cases, surface temperatures would be higher, possibly approaching the conditions for dry sliding reported in reference 6 where maximum temperatures were almost at the melting point of one of the materials in contact. Higher surface temperatures would very markedly increase the reaction rate and consequently the rate of film formation. Data have also been obtained which indicate that minimum reaction temperature could be reduced and rate of reaction increased by the use of a catalyst.

SUMMARY OF RESULTS

Formation, under transient temperature conditions, of sulfide films on heated steel specimens immersed in cetane containing dissolved free sulfur were studied and the static-friction properties of such films were investigated. The following results were obtained:

1. Minimum temperatures below which iron sulfide is not formed on heated steel specimens in cetane solutions containing 0.1 to 1.0 percent free sulfur by weight were from 700° to 375° F, respectively. The catalyst sodium sulfide Na_2S effectively decreased the minimum reaction temperature.

2. The calculated mean rate of sulfide film formation on steel in a solution of 0.5 percent by weight free sulfur in cetane was approximately 1.8×10^5 Angstrom units per second during an initial time increment of 0.04 second at temperatures from 1100 to 1000° F.

3. Static-friction data obtained for clean dry steel (coefficient of static friction, 0.79) and for steel coated with sulfide films of various thicknesses showed that dry film thicknesses of 5000 Angstrom units or greater were necessary to prevent surface welding completely and to produce low friction (coefficient of static friction, 0.32 to 0.39). Welding was appreciably reduced, however, with film thicknesses as low as 3400 Angstrom units although friction was reduced only slightly. This result indicated that thin sulfide films were somewhat effective even though complete prevention of welding was not obtained.

Lewis Flight Propulsion Laboratory,
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Cleveland, Ohio, June 8, 1951.

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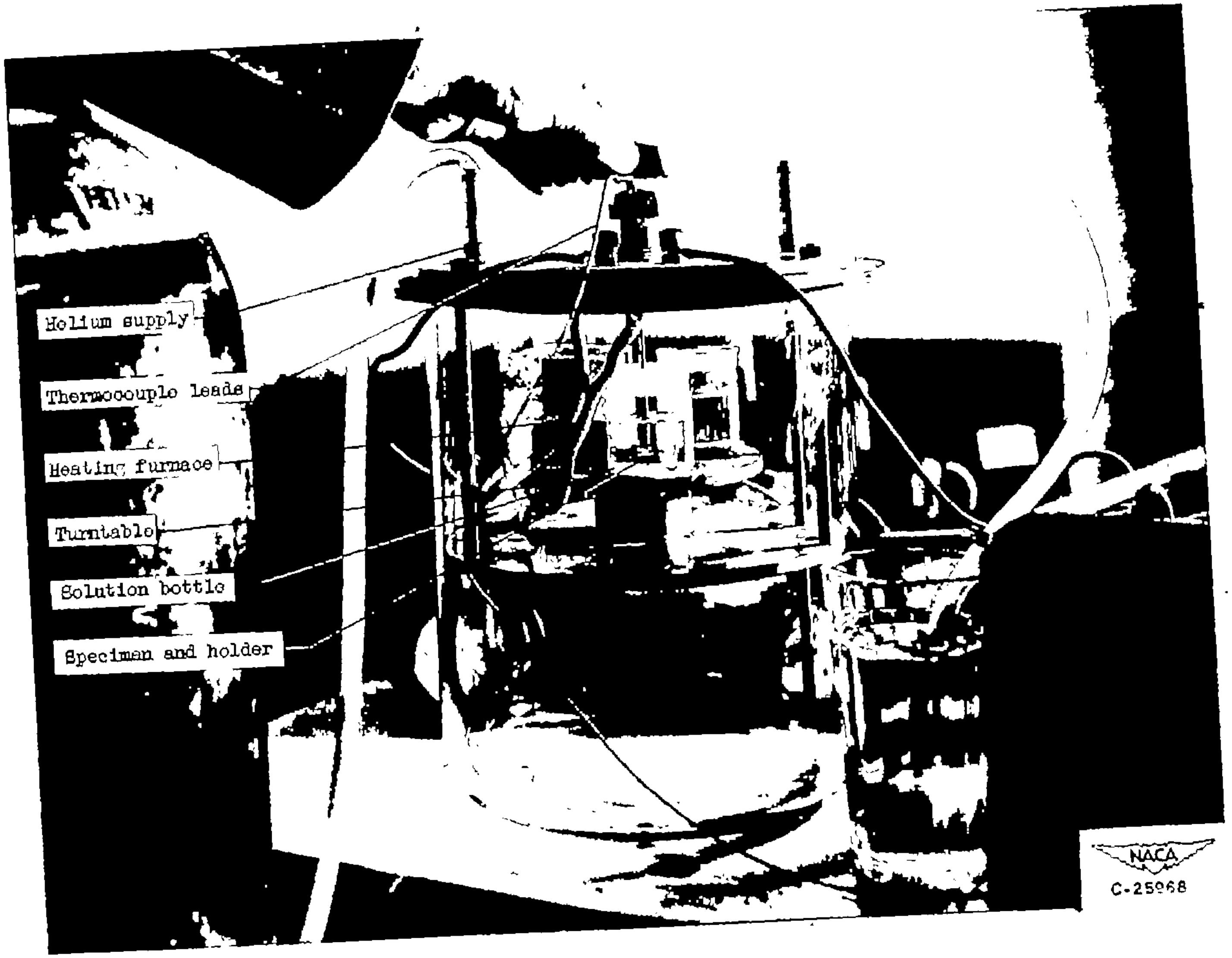

(a) Over-all view.

Figure 1. - Apparatus used for formation of iron sulfide films.

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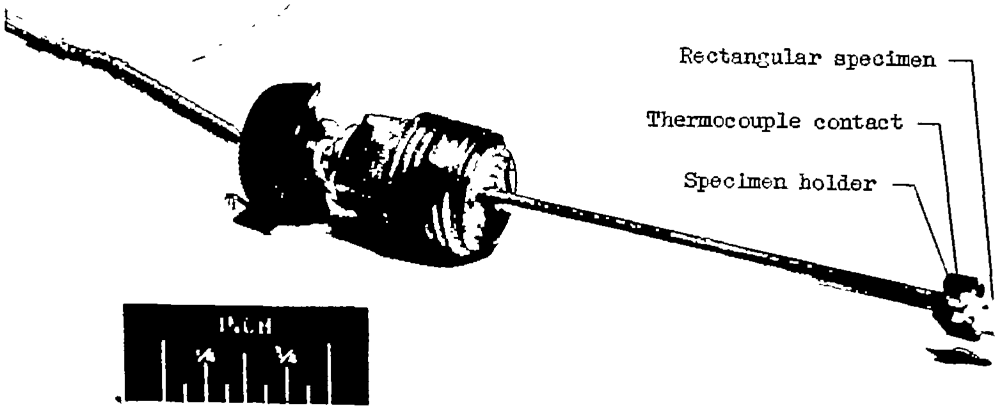

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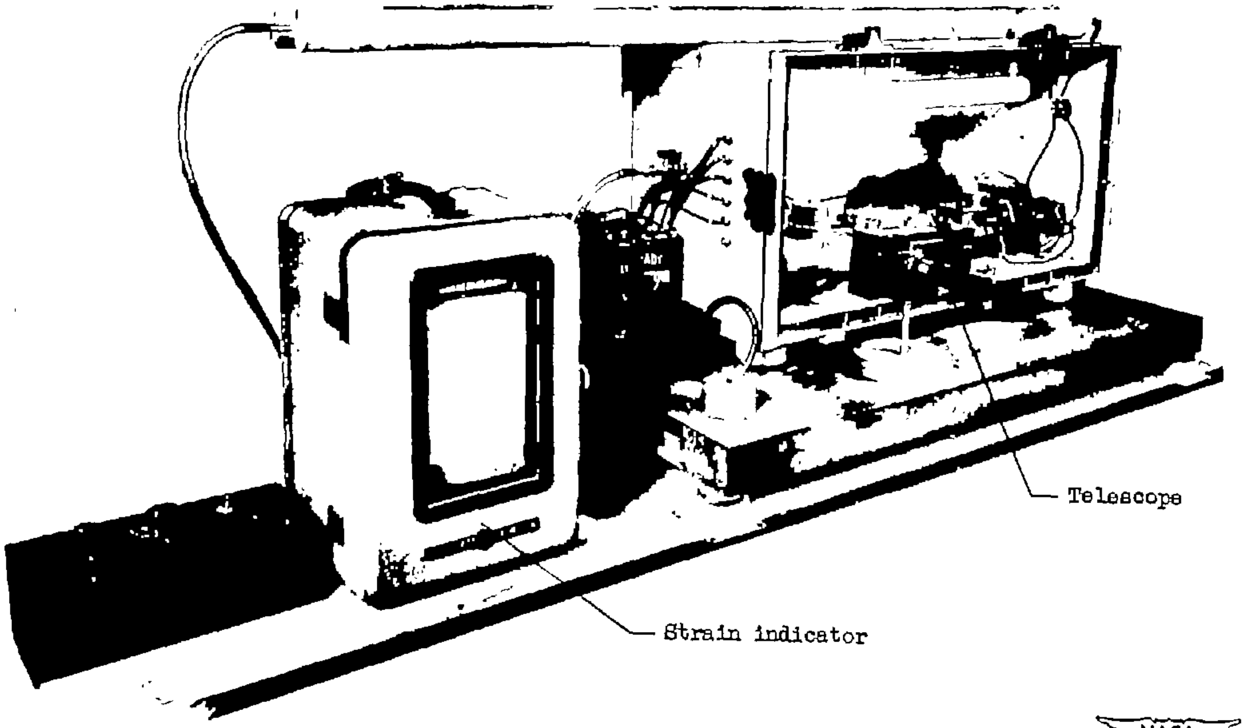
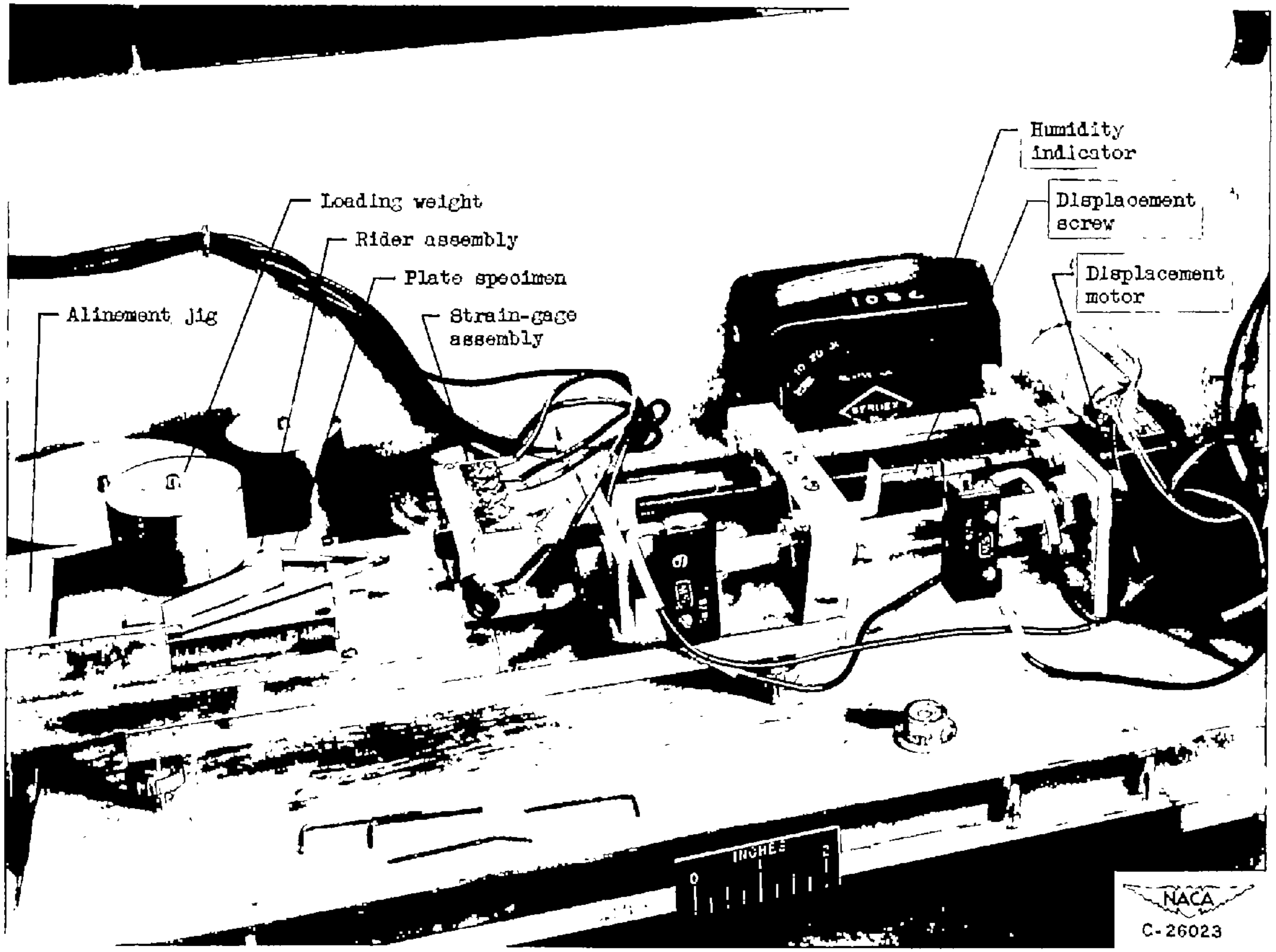
(b) Specimen and specimen holder with thermocouple and leads passing through the hollow shaft.

Figure 1. - Concluded. Apparatus used for formation of iron sulfide films.



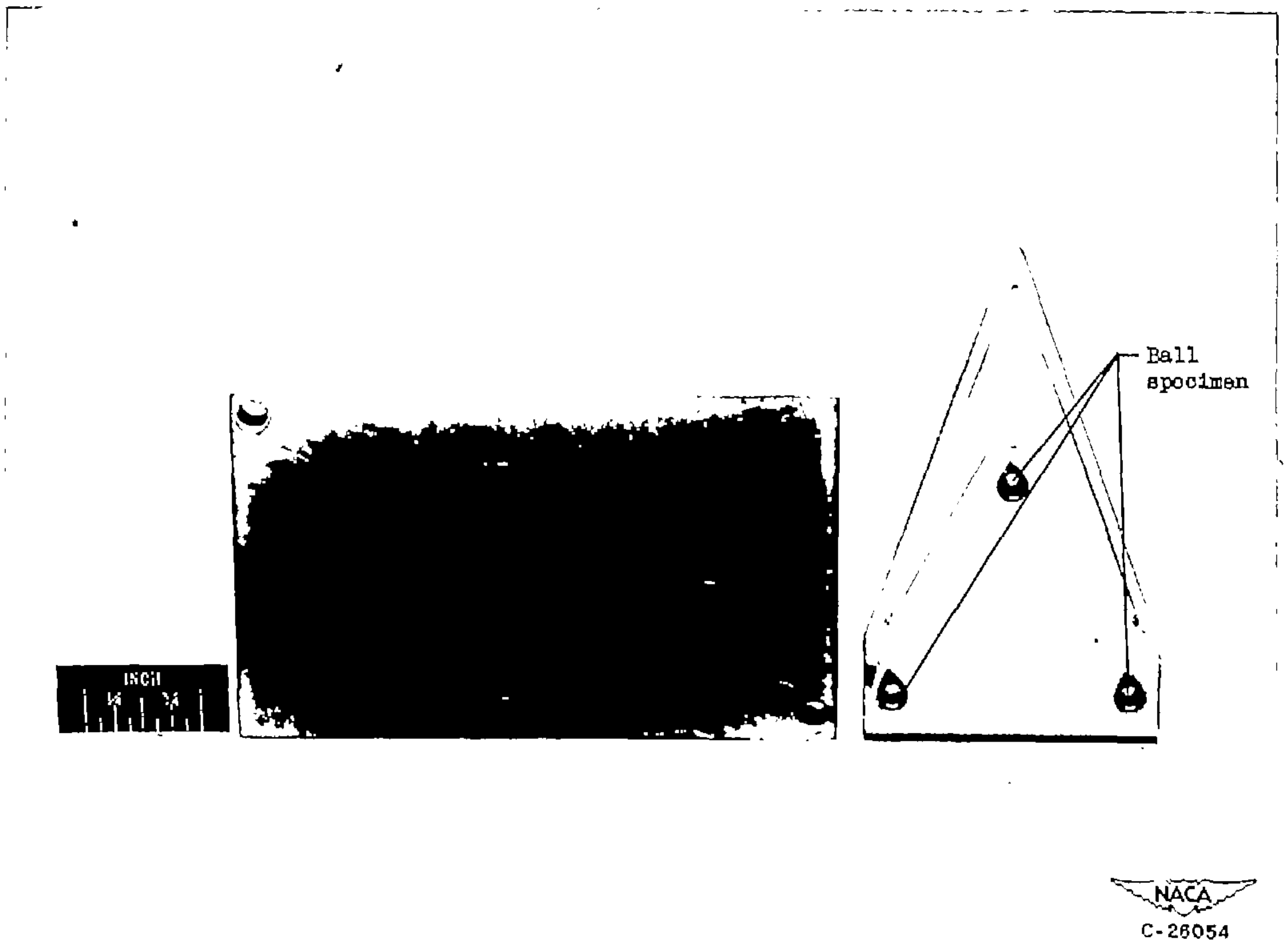
(a) Over-all view.

Figure 2. - Static-friction apparatus.



(b) Basic apparatus.

Figure 2. - Continued. Static-friction apparatus.



(c) Plate specimen and rider specimens in rider assembly.

Figure 2. - Concluded. Static-friction apparatus.

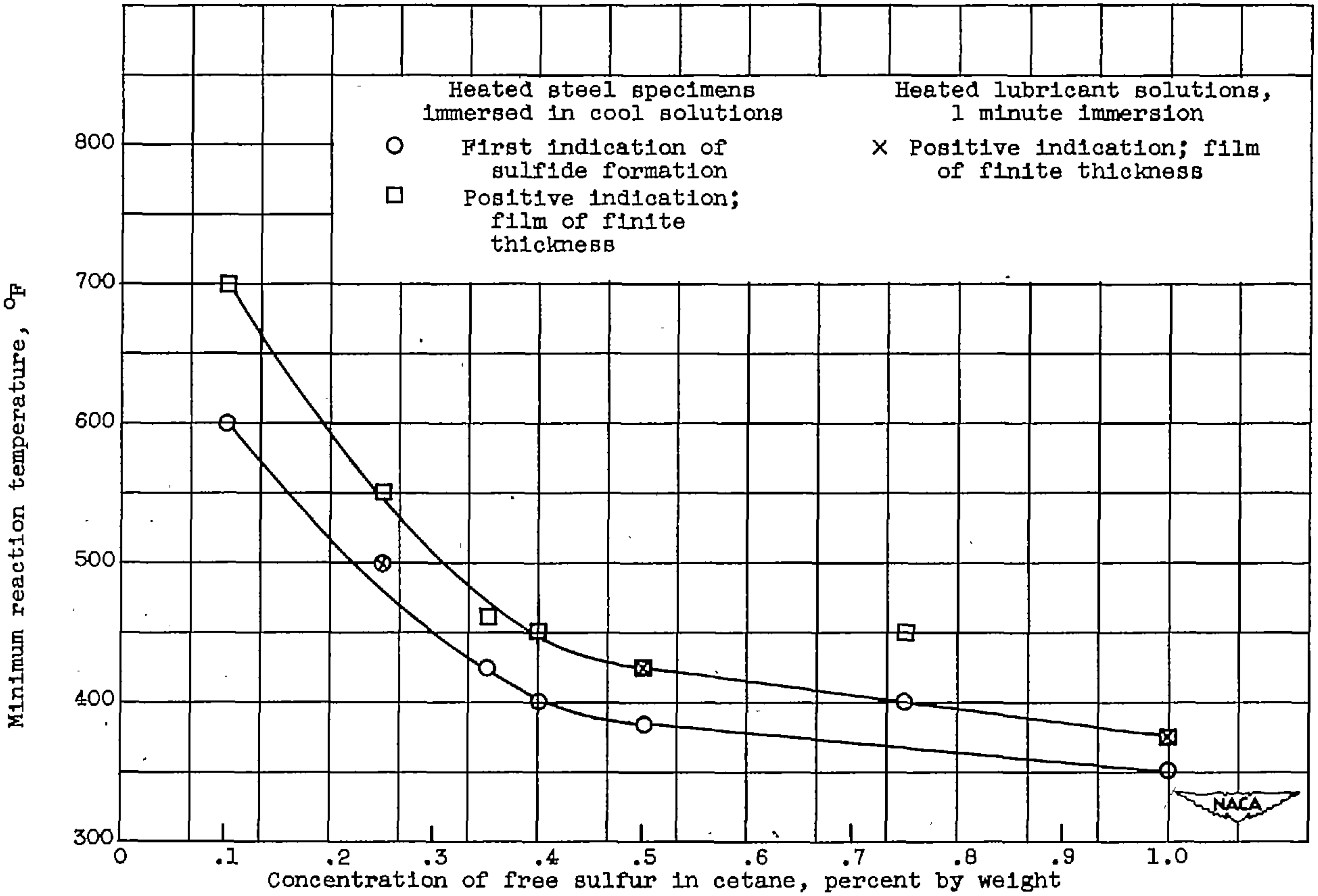


Figure 3. - Effect of concentration on temperature below which no reaction takes place to form iron sulfide on steel. Atmosphere, helium.

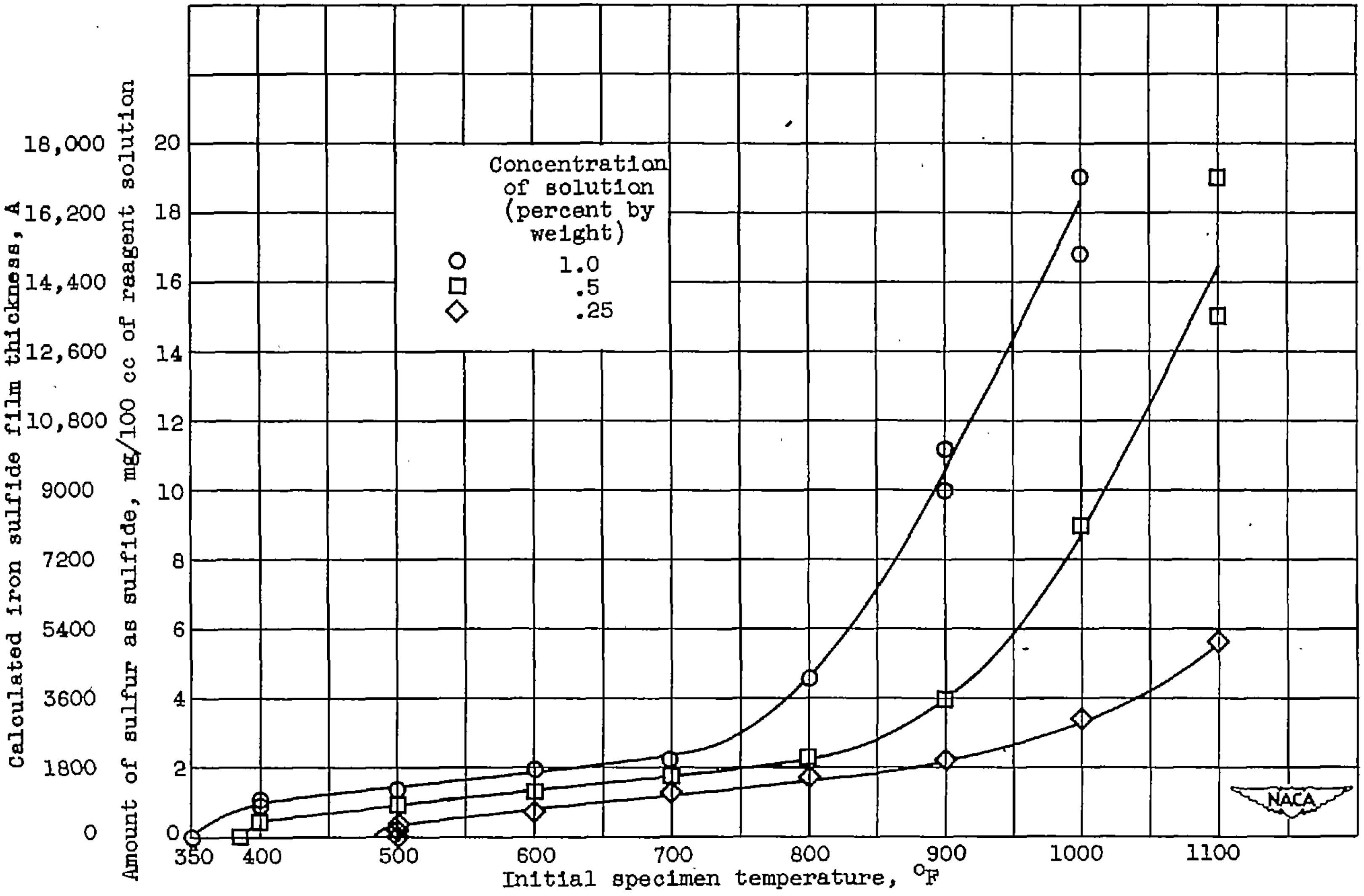


Figure 4. - Effect of initial specimen temperature on total iron sulfide formed on heated steel specimens after immersion in solutions of free sulfur and cetane. Atmosphere, helium.

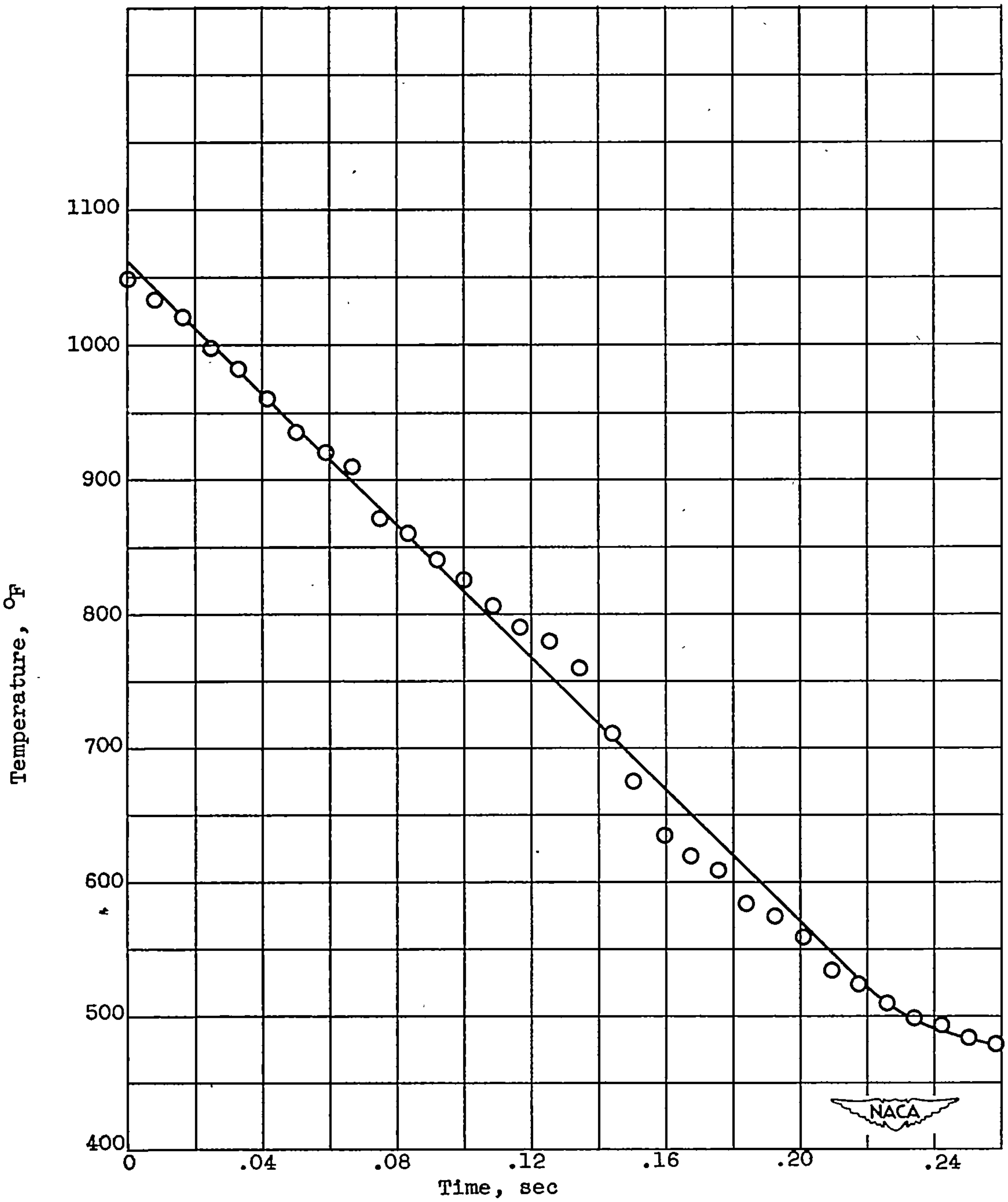


Figure 5. - Typical cooling curve for heated steel specimen (0.72 g) after being submerged in room-temperature solution (25 g) of free sulfur (1 percent by weight) in cetane. Atmosphere, helium.

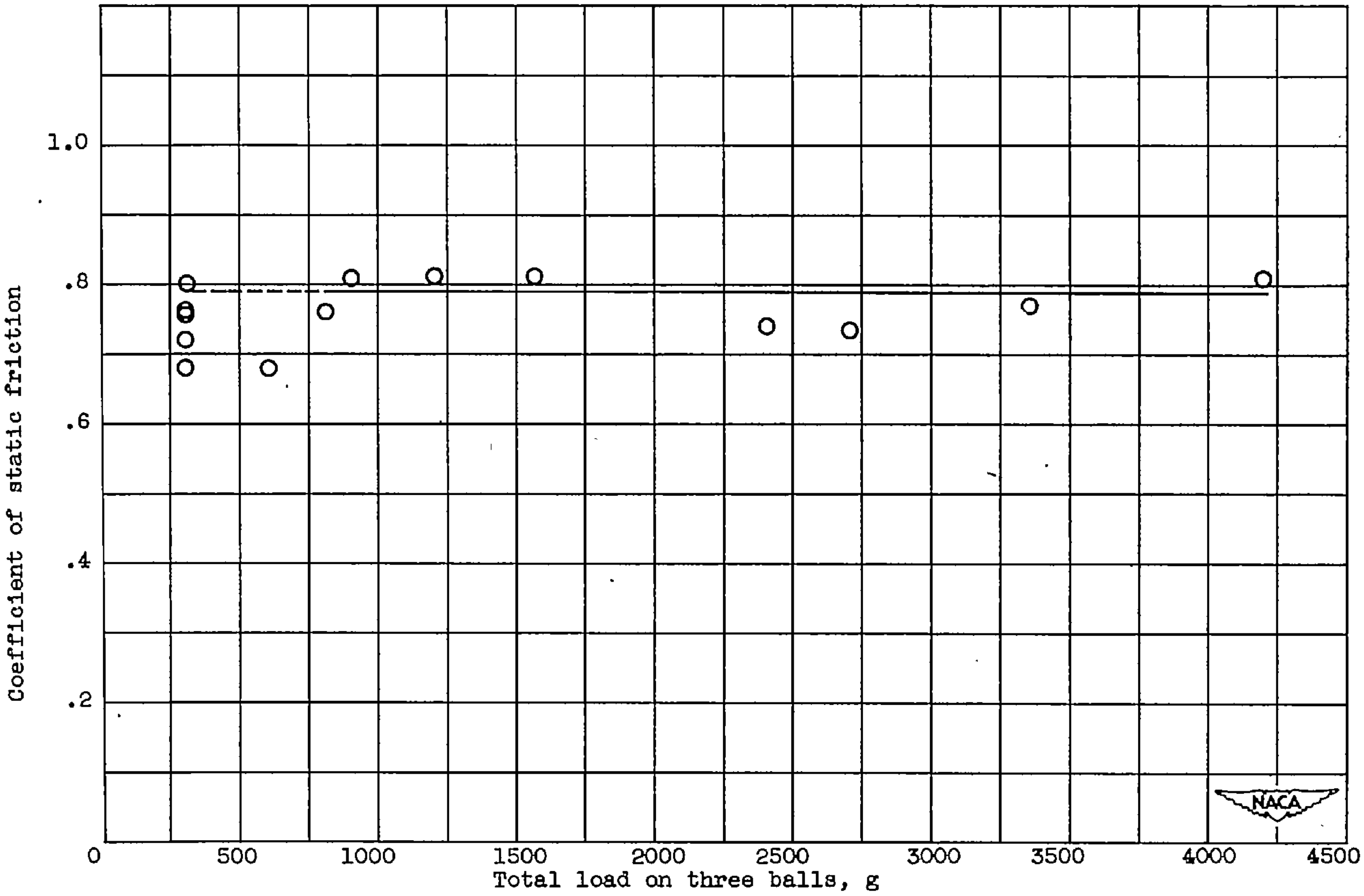


Figure 6. - Effect of load on coefficient of static friction of clean hardened steel balls sliding on dry mild steel plate.

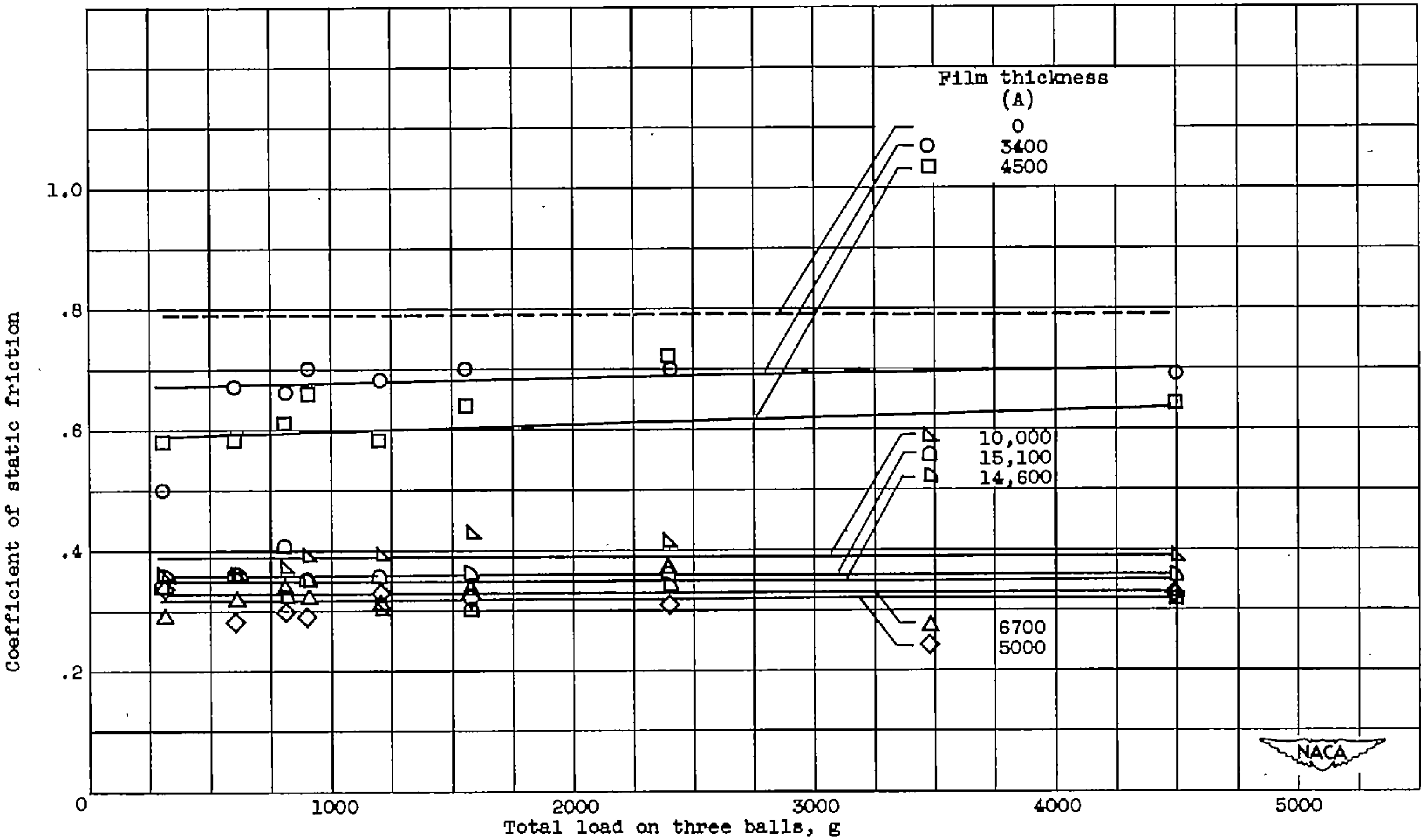


Figure 7. - Effect of load on coefficient of static friction for various thicknesses of iron sulfide films on rider (ball) specimens. Film thickness calculated from weight increase of rider specimens.

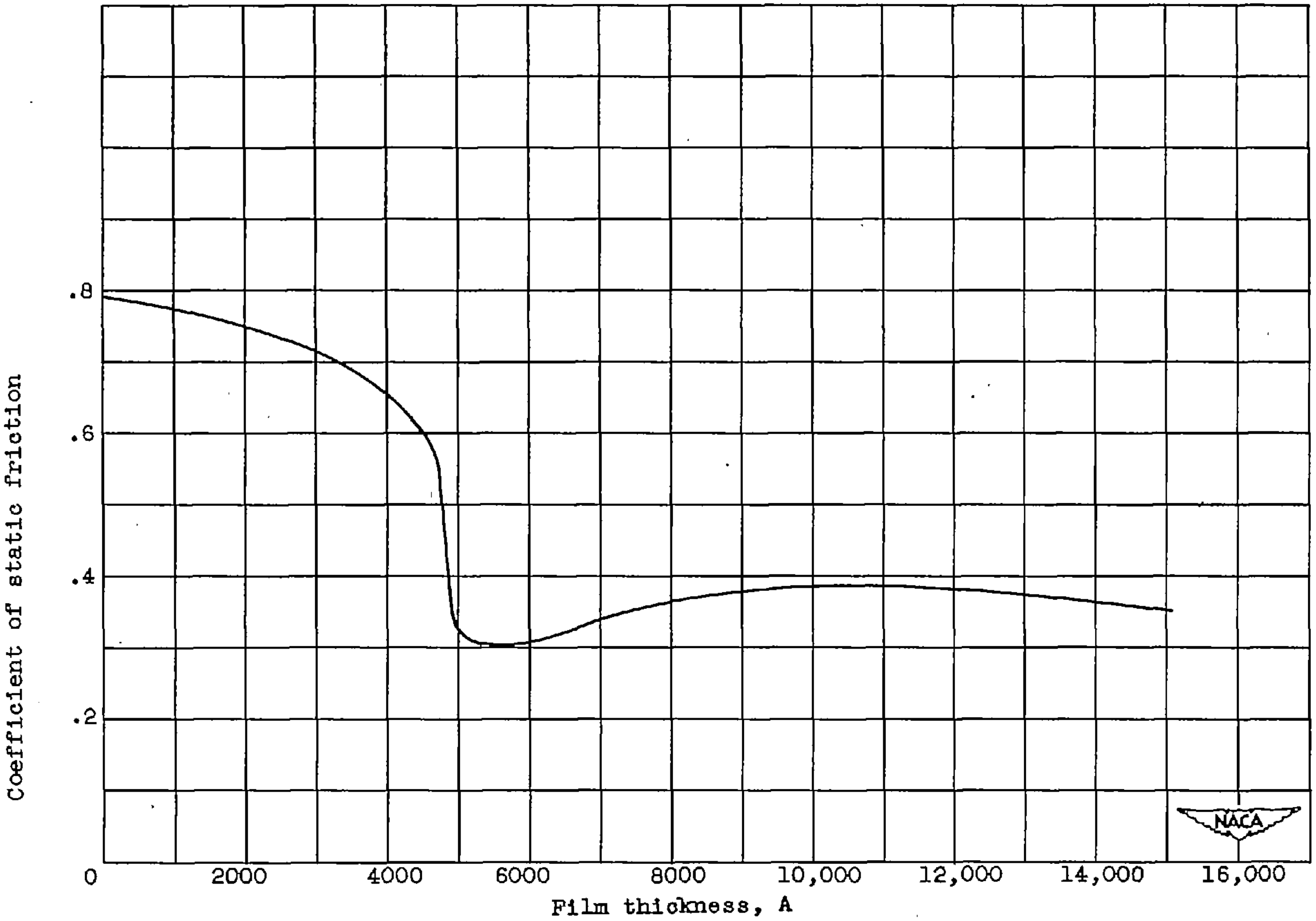
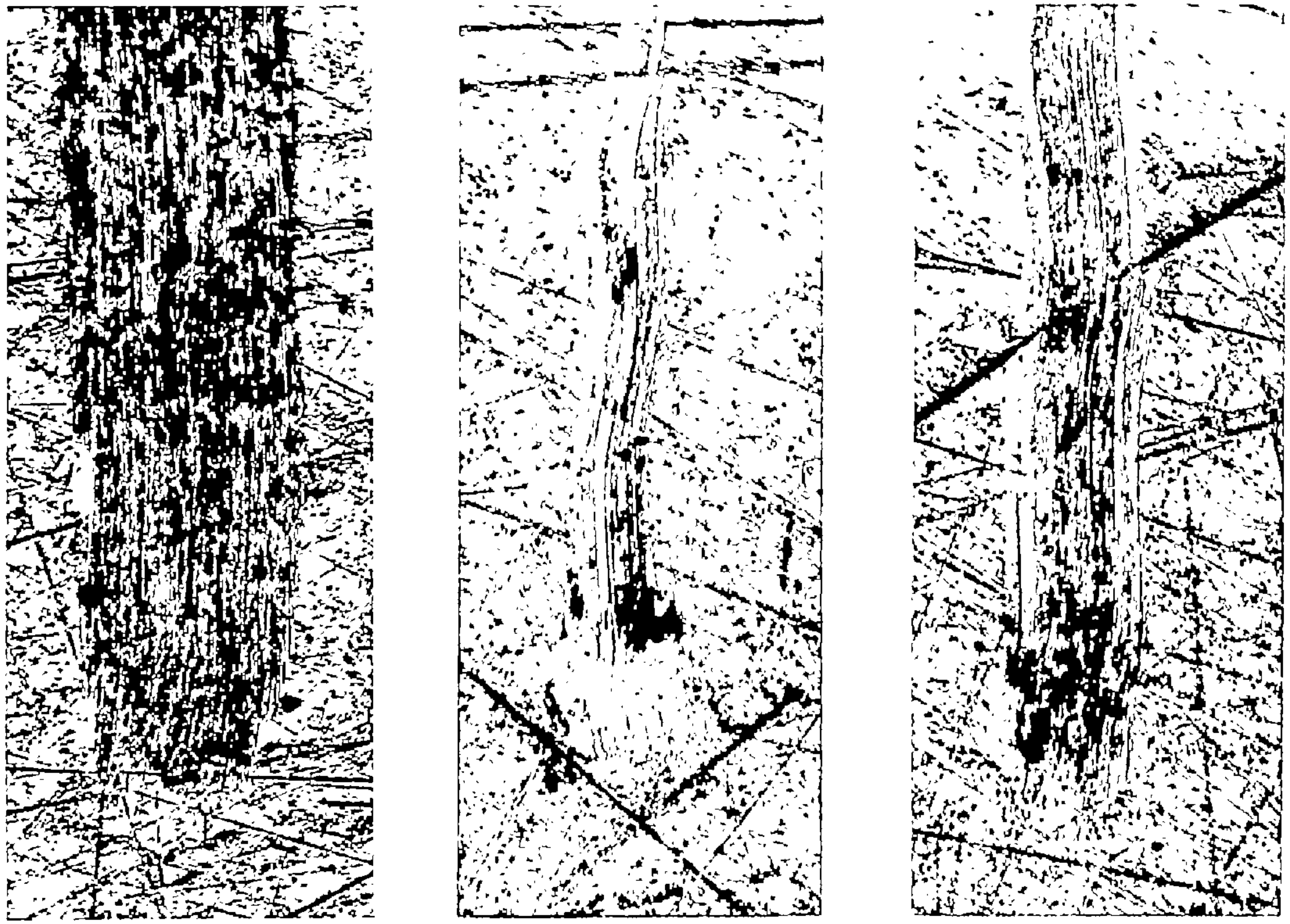


Figure 8. - Effect of iron sulfide film thickness on coefficient of static friction. Total load on three balls, 2400 grams. Cross plot of data from figure 8.



(a) Wear track for untreated steel on steel. Load, 2400 grams.

(b) Wear track caused by ball specimen having iron sulfide film 14,400 Angstrom units thick. Load, 2400 grams.

(c) Wear track caused by ball specimen having iron sulfide film 14,400 Angstrom units thick. Load, 4500 grams.

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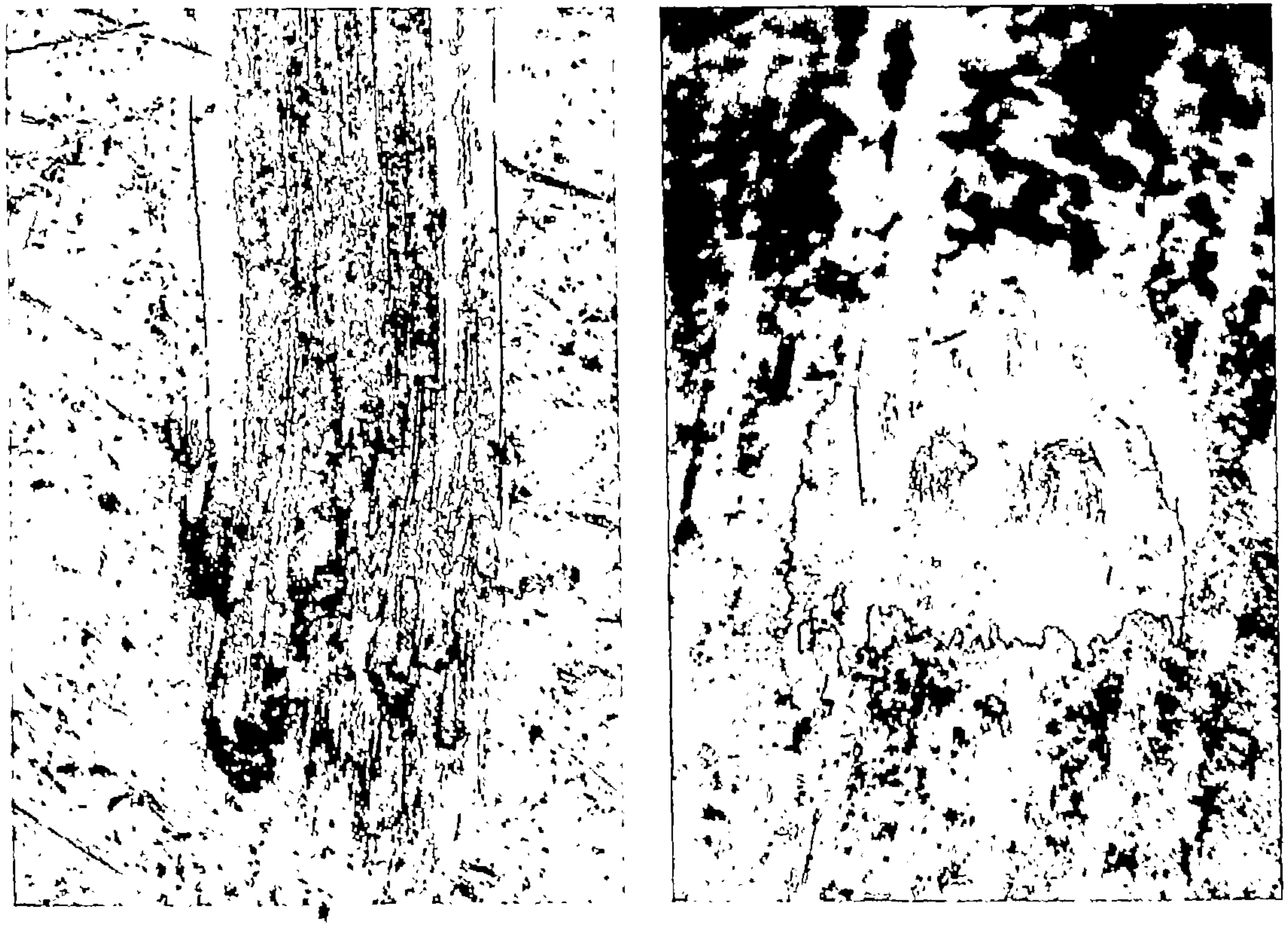
Figure 9. - Photomicrographs of wear tracks on plate specimens after runs of untreated steel at 2400 gram load, and sulfide film on ball specimen of 14,400 Angstrom units thick at 2400- and 4500-gram loads, X100.



(a) Plate specimen showing film transferred from ball specimen.

(b) Ball specimen coated with sulfide film 14,400 Angstrom units thick.

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Figure 10. - Photomicrographs of mating surfaces of plate specimen and ball specimen coated with iron sulfide film 14,400 Angstrom units thick, after static-friction test. Load, 4500 grams; X270.

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