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December 6, 1948

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JUL 12 1949

The Chief of Ordnance
Department of the Army
Pentagon Building
Washington, D. C.

Re: Contract W-36-034-ORD-7654

Dear Sir:

With this letter, we transmit to you herewith thirty copies of our report on "Surface Treatment Research in Conjunction with the Cold Shaping of Steel", sub-project ORD TB-4-61C, the third volume of four such reports being delivered on this contract.

The findings in this report are significant not only technically but economically.

While the best coating, without which the novel heavy extrusion of steel is impossible, is clearly indicated as conventional zinc phosphate material, which is the cheapest and most practical to apply, the details given of baths, their control and application are also of prime importance economically.

This research has also developed exact information pointing to the use of the cheapest type of lubricant material and in what might seem excessively dilute form. Nevertheless, this simplification of technique and working directions made possible the extrusion of a billet in a single operation to a reduction in cross section area of 85%, the greatest by far ever reported. Conditions of lubricating are cited which produce reduction in friction of high order and may alter existing conceptions of tool design and life.

In conjunction with the metallurgical research, detailed in the fourth volume of these reports, the indicated conclusion is that steel is truly a plastic material -- at room temperature.

Yours faithfully,

HEINTZ MANUFACTURING COMPANY

Manager, Ordnance Division

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SUPPLEMENTAL NOTES

Pertinent to the Sections Noted Below

Introduction (Page 1) For the detailed understanding of the present report, it is recommended that it be read in conjunction with the report of the original investigation, known as "Cold Shaping of Steel -- Summary Report, July 1947, Contract W-36-034-ORD-7603". Therein are set forth details of preparatory calculations, planning of procedures, and processing of steels, together with the general remarks concerning the art of cold shaping of steel by extrusion.

For example:-

Pickling, surface roughness (etching) and inhibition are completely described on pages 35-36 of the Summary Report.

Rusting is completely described on pages 54 and 56 in the appendix of the Summary Report.

Aging of the bath is described on page 51 in the appendix of the Summary Report.

In this present report, that original report will be referred to as "Summary Report".

Complete drawings of dies, punches, and fixtures used in this project were forwarded to Watertown Arsenal for file.

Pickling (Page 8, Par. 3b (2) For the pickling the laboratory samples, hydrochloric acid was used (instead of sulfuric acid which was used in pickling material used for the Ordnance items made on this contract) purely for reasons of convenience. Either acid may be used satisfactorily, if applied in accordance with common practice.

Phosphatizing (Page 8, Par. 3b (3) For satisfactory phosphatizing, it is not necessary that copper be included in the processing bath. However, a small amount of copper is desirable in that it supports the accelerating effect of the nitrate and is introduced by means of the replenishing solution. Proper replenishing, governed by the surface processed, provides sufficiently small amounts of copper which are not detrimental to the process. This copper content is critical and for precautionary reasons, as a result of experience, the make-up solution does not contain copper. The permissible critical copper content is therefore introduced by controlled replenishing which in turn is based on the amount of steel surface processed.

Copper, however, which is present on the steel surface prior to phosphatizing cannot be tolerated since such a contamination will result in poorly adherent coatings.

Newly prepared phosphatizing baths are "aged" by the introduction of clean iron scrap-such as degreased turnings - at the operating temperature. The scrap is removed after five minutes, rinsed, pickled for

a short time to remove the phosphate coating, rinsed both cold and hot and again phosphatized for five minutes. This is repeated until scrap to the extent of approximately four square feet of steel surface to each gallon of solution has been phosphatized. The bath may then be considered as sufficiently "aged".

The iron content of the bath should not exceed the zinc content; the ratio between the two being 1:1 which is an incidental result of a proper ly operated bath. Specific control of the iron content is not necessary.

Sequence of operations (page 10) Surface roughness, an incidental result of the pickling operation, insures a good interlocking of the phosphate coating. Pickling, therefore, serves a dual purpose: (1) de-scaling, and (2) etching to improve the bond of the phosphate coating to the metal surface. The desired surface etch prior to phosphatizing will be obtained, following the recommended pickling procedure provided in the report. When de-scaling can be accomplished within the time limits given in the report, inhibition of the pickling acid is not necessary. Should the pickling time exceed the prescribed limits, then an inhibitor such as Rodine, as supplied by the American Chemical Paint Co., Ambler, Pa., should be used, following the specifications of its manufacturer. Over etching is not desirable.

The rusting operation is performed between the cold and hot water rinses before phosphatizing by allowing the work to come in contact with the steam escaping from the hot water rinse tank for 2-5 minutes. The degree of rust desired may be described as a greenish-brown blush covering the work.

Phosphatizing Solutions (Page 26, Par. 4c) The zinc phosphate solution described does not deviate from the phosphatizing solution as standardized for government work since its nature is specified as a zinc phosphate-zinc nitrate solution. Of the zinc phosphate make-up solution given on page 8, 90 ml. when diluted to one liter, will give a processing bath having the following composition:

Zinc	16-17 gms/l
Phosphorous Pentoxide	18-29 gms/l
Nitrate	20-21 gms/l
Nickel	0.45 gms/l

Five minute immersion at 95°C.

The composition of the Manganese Phosphatizing bath was:

Manganese	8-9 gms/l
Iron (ferrous salt)	1-2 gms/l
Phosphorous Pentoxide	12-13 gms/l
Nitrate	11-12 gms/l

Twenty minute immersion at 90°C.

The composition of the Cadmium Phosphatizing bath was:

Cadmium
Phosphorous Pentoxide
Nitrate
Sodium

Seven minute immersion at 90°C.

April 19, 1949

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Introduction - Directive and Program

This report is submitted in fulfillment of the requirement described in Article 1, Paragraph 2, Sub-paragraph C, of Contract W-36-034-ORD-7654 as follows:

> "Surface treatments, including both boundary lubrication and semi-liquid lubrications. This to refer to chemicals as well as applied research."

The detailed program for this work was outlined in writing, reviewed and revised by the Director of the Watertown Arsenal Laboratory as provided in the contract and the following approved outline of the program was transmitted by the Arsenal to the contractor for action on 17 March 1948:

"C. Determination of Effect of Surface Treatments

1. Effect of Type of Coating - Low and High Pressure Range

Determine the apparent coefficient of friction within the pressure range of 2500 to 200,000 psi using emulsified sulphonated tallow as lubricant on steel coated with

a. Zinc Phosphate
b. Manganese Phosphate
c. Cadmium Phosphate
d. Calcium Phosphate
e. Barium Phosphate

2. Effect of Lubricants on Standard Zinc Phosphate Coated Steel - Low Pressure Range

Determine apparent coefficient of friction within a pressure range of 2500 to 12,500 psi (tests at 2500, 5000, 7500, and 12,500 are suggested) using different types of lubricants on zinc phosphate coated steel (coating as standardized for government program).

a. Dilute emulsion type lubricants (emulsified in soap)

(1) Lard oil (low acidity)
(2) Lard oil (high acidity)
(3) Lard oil containing aluminum stearate
(4) Lard oil containing oxidized fatty oil
(5) Lard oil containing surface active agent
(6) Lard oil containing chlorinated paraffin
(7) Sulphonated tallow
(8) Tall oil ester

b. Commercial lubricants

(1) Animal oil or fat base
(2) Animal oil or fat base partly sulphonized
(3) Pigmented type

c. Solid lubricants

(1) Sodium stearate
(2) Calcium "
(3) Aluminum "
(4) Zinc "

3. Effect of Lubricants on Standard Zinc Phosphate Coated Steel - High Pressure Range

Determine apparent coefficient of friction within a pressure range of 50,000 to 200,000 psi (tests at 50,000, 100,000, and 200,000 psi are suggested) using different types of lubricants on zinc phosphate coated steel (coating as standardized for government program).

a. Dilute emulsion type lubricants

Use the 6 lubricants listed under C2a

b. Commercial lubricants

Use lubricants listed under C2b only if they were satisfactory at the low pressure range indicated in C2.

c. Solid lubricants

(1) Sodium stearate
(2) Aluminum stearate
(3) Calcium stearate
(4) Zinc stearate

4. Effect of Lubricants on Uncoated Steel - Low and High Pressure Ranges

Determine the apparent coefficient of friction within a pressure range of 2500 to 200,000 psi using uncoated steel (not phosphate treated).

a. Lubricant

(1) Emulsified sulphonated tallow (this is the most satisfactory lubricant from past experience), or
(2) The most satisfactory lubricant from C2a.

5. Effect of Coating and Lubricant on Cold Reduction under Tension (Drawing)

Determine effect of reduction (35-38%) under tension on apparent coefficient of friction and total load including effect on coating and lubricant using the microscope to aid in this determination.

a. Dilute emulsion type lubricants as selected from tests under C3a.

(1) The most satisfactory lubricant
(2) The least satisfactory lubricant

b. Commercial lubricants

Test commercial lubricants only if they were satisfactory under C2b

c. Solid lubricants

(1) Sodium Stearate
(2) Calcium Stearate
(3) Aluminum Stearate
(4) Zinc Stearate

6. Effect of Coating and Lubricant on Cold Reduction under Compression.

Determine apparent coefficient of friction and total load of WD 1010 Steel extruded under compression to each reduction stated in B4 and lubricated in accordance with the most satisfactory method of C3a with surface

a. Uncoated

b. Zinc Phosphate coated (thin)

c. Zinc Phosphate coated as standardized for government program."

- - - - - - - -

While the entire program above detailed was carried out with only such deviation as was dictated by the progress and development of the work, the order in which the work was done and also presented in the following report is in a somewhat different and more logical sequence as indicated in the Table of Contents.

REPORT

Contract W-36-034-ORD-7654
Subject Project ORD-TB-4-61C, Part C, Article 2
Surface Treatment Research
in Conjunction With Cold Shaping of Steel

1. Summary

To select suitable lubricants and lubrication conditions in combination with a phosphate coating for particular use in cold extrusion and severe drawing operations, friction tests were made using various type lubricants of known chemical composition. Unit pressures were applied covering a very wide range from those of the stamping operation to those of the severe drawing and cold extrusion operation.

The lubricants used may be classified:

Dilute emulsion type - highly diluted soap emulsion in concentrations not exceeding 5% of soap plus animal or vegetable oils or fats.

Commercial type - more concentrated soap emulsion containing animal or vegetable oils or fats, pigmented or non-pigmented. In this case the lubricants were purchased and applied in the "as-received" condition.

Dry type - solid form, such as the dry metal stearates.

a. Effect of Lubricants on Standard Zinc Phosphate Coated Steel at all Pressures

(1) While zinc phosphate coated steel alone, that is, without the addition of any other lubricant, is known to have a lower coefficient of friction than uncoated steel without lubricant, addition of a suitable lubricant to the phosphate coating lowers the coefficient of friction still further.

(2) Under the simplest or "normal" conditions of applying a dilute emulsion type of lubricant, i.e., short immersion at room temperature, emulsified sulfonated tallow was found to be the best of the eight emulsified lubricants tested (see figures 3,4,5, and 6).

(3) Included in the fatty emulsion group is lard oil which seemed to have competing qualities by reason of its availability and low cost.

Under the same conditions of application, lard oil of the type containing free fatty acids up to 15% was found to give better results than such oil with lower fatty acid content, such as down to 2.7%. (see Figure 3).

(4) Additives which would be readily adsorbed on a metal surface improved the lubricant quality of high fatty acid lard oil (see Figures 4 and 5) to an effectiveness approaching that of sulfonated tallow.

(5) An important development demonstrated in this experimentation is that all lubricants tested were greatly increased in effectiveness by their application at elevated temperature and for a longer immersion time.

(6) In the lower pressure range under these conditions of time and temperature, the coefficient of friction was only a fraction of that experienced with lubricants applied under "normal" conditions. At higher pressures the differences, although not so great, were nevertheless noteworthy.

(7) The lubricant value of all types tested was so greatly improved by their use at higher temperature and for longer immersion time that this method of application merits serious consideration.

For the maximum reduction of friction in cold working steel, emulsified tallow applied to a zinc phosphate coated surface at 70°C (158°F) by immersion for one hour was found best.

(8) Commercial and solid lubricants did not approach these results. (See Figures 8 and 9).

b. Effect of Lubricants on Uncoated Steel

(1) Using the three groups of lubricants on phosphate coated and uncoated surfaces, it was found that the phosphate coated surface in every case had a lower coefficient of friction.

c. Effect of Type of Phosphate Coating on Working of Steel at all Pressures

(1) It is demonstrated that of the metal phosphate coatings tried, a zinc phosphate coating gives the lowest coefficient of friction. (See Figure 11). This is of special interest since phosphate as a coating is the cheapest and most practical to apply.

(2) The advantage of a zinc phosphate coating over the other types of phosphate coatings is relatively less in the higher pressure range but of a useful magnitude.

(3) At lower pressure ranges the reduction of friction by the use of phosphate coatings and lubricant is relatively a less important factor.

d. Effect of Coating and Lubricant in Drawing Operation (Cold Working under Tension)

(1) Testing of the three groups of lubricants in a conventional drawing operation has demonstrated that the dilute emulsion type and the solid dry type were superior to the more costly commercial types. While little difference in results was noted between the dilute emulsion type and the solid dry type, the former is the cheaper and much the easier to apply.

(2) A comparison of the loads experienced in an operation with a reduction (40% to 50%) in cross section (with resultant increase in surface area) and an operation with no reduction in cross section — applying in both cases the same unit pressure (i.e., 200,000 psi) — showed that in the latter case a considerable reduction in friction was experienced. (See Tables 10 & 11).

e. Effect of Coating and Lubricant in Cold Extrusion Operation (Reduction Performed under Compression)

(1) This part of the program refers to the main aspects of the entire research. The positive results of the foregoing steps are integrated and the comparison is made between the results obtained in the extreme cold working in an extrusion operation employing the lubricant found to have the best results, on a coated and on an uncoated surface.

(2) It is shown that uncoated material could not be used for such an operation; that phosphate coating of a thickness of at least one gram per square foot in combination with a dilute emulsion type lubricant produced the best frictional conditions as well as eliminated "pick-up" on the work.

2. Conclusions

For the cold extrusion of steel, it is necessary to apply a coating which is resistant to high temperatures, that is in the vicinity of 400-600° Centigrade, and in any event in excess of temperatures thus for experienced in cold extruding steel, and of sufficient thickness to insure complete separation of the sliding metal surfaces, thereby avoiding galling and pickup. Such a suitable coating is a zinc phosphate coating which may be considered a tightly interlocked or fixed inorganic pigment having desirable anti-welding properties and the ability to reduce the friction occurring between the sliding metal surfaces.

High fatty acid lubricants, containing polar groups, which enhance their adsorptability, are very effective in combination with phosphate coatings. Such lubricants may be applied in the solid form, as in the case of dry stearates. They may be applied also in liquid form, as either a highly diluted soap emulsion or a more concentrated soap emulsion. Such liquid lubricants, due to their chemical composition, are able to react chemically with a phosphate coating to form a water-insoluble lubricant film, which is well interlocked with the coating. The combination of the so formed lubricant film with the phosphate coating decisively determines the degree of friction.

The dilute emulsion type lubricants should be applied for cold extrusion for the following reasons:

Existence of conditions favorable to hydrolysis, thereby insuring the formation of the insoluble lubricant film.

High cooling effect.

Economy and ease of application.

3. Procedure

This section describes the chemical and mechanical processes and the types of steel used in this investigation.

a. Test Specimens

(1) For the Determination of the Apparent Coefficient of Friction.

(a) Unit pressure range 1250 to 12,500 psi.
Auto Body stock 1" x 6" x .062".
(deep drawing quality)

(b) Unit pressure range 50,000 to 200,000 psi.
SAE 1060 - 1" x 6" x .186".

(2) For the Determination of the Load During Drawing.

(a) SAE 1010 - 1" x 6" x .062" (annealed at 1300°F for 15 minutes before surface treatment).

(3) For the Determination of the Load During Extrusion.

(a) 20% Reduction
C1010 AISI rimmed, round bars .635" dia.
(b) 40% Reduction
C1010 AISI rimmed, round bars .7355" dia.
(c) 60% Reduction
C1010 AISI rimmed, round bars .896" dia.
(d) 85% Reduction
C1010 AISI rimmed, round bars .896" dia.

b. Chemicals and Chemical Treatment

(1) Cleaning
Vapor degreasing using trichlorethylene

(2) Pickling
Hydrochloric acid 1:2 at 100°F for 20 minutes

(3) Phosphatizing
Composition of the Concentrated Solutions:

	Makeup - in grams per liter	Replenisher - in grams per liter
Zinc (Zn)	183 ±5	138 ±5
Phosphorous pentoxide (P_2O_5)	207 ±5	268 ±5
Nitrate (NO_3)	230 ±5	160 ±5
Nickel (Ni)	5 ±1	5 ±1
Copper (Cu)	--	1.5 ±0.3
SP. gr. at 20°C	1.600	1.600

To form a normal coating, the following operating bath was used:

90 ml of the above makeup solution was diluted to one liter and operated at a temperature of 200°F with an immersion time of 5 minutes. To obtain ideal working conditions, the bath was broken in by the immersion of steel turnings to form soluble iron.

Total concentration: 70 points.*

Ratio between free and total acid: 1:4-5.

(This is based on a 10ml sample of the bath using N/10 NaOH against phenolphtalein (total acid) and against methyl-orange (free acid).

Average thickness or weight of a normal coating obtained under these conditions is:

SAE 1010 and Auto Body Stock = 2.3 g/sq. ft.

SAE 1060 = 2.0 g/sq. ft.

AISI C1010 = 1.4 g/sq. ft.

To form a thin coating, the following operating bath was used:

20 ml of the makeup solution was diluted to one liter and operated at a temperature of 180°F with an immersion time of 2 minutes. It is necessary that this bath also contain soluble iron.

Total concentration: 15 points.*

Ratio of free to total acid: 1:6-7

Average thickness or weight of a thin coating obtained under these conditions is:

C1010 AISI = 0.75 g/sq. ft.

*The definition of the term "points" as commonly employed in connection with Phosphatizing solutions is, "The number of cubic centimeters of a one-tenth normal solution of sodium hydroxide necessary to titrate a ten cubic centimeter sample of the solution using phenolphthalein.

Below is listed the sequence of operations:

Uncoated	Normal Coating	Thin Coating
Degreasing	Degreasing	Degreasing
Pickling	Pickling	Pickling
Cold Rinse	Cold Rinse	Cold Rinse
Hot Rinse (200°F, 1 minute)	Hot Rinse (200°F, 1 minute)	Hot Rinse (180°F, 1 minute)
	Phosphatizing	Phosphatizing
	Cold Rinse	Cold Rinse
	Hot Rinse (.5 gm CrO_3/1 200°F, 1 minute)	Hot Rinse (.5 gm CrO_3/1 200°F, 1 minute)
Drying (20 min.@ 250°F)	Drying (20 min.@ 250°F)	Drying (20 min.@ 250°F)

The test specimens were then either lubricated and used immediately, or stored in a dessicator.

c. Lubricants and Lubrication

The REILLY-WHITEMAN-WALTON CO., of Conshohocken, Pennsylvania, provided all lubricants used in this investigation and all pertinent data such as composition, characteristics, etc. The lubricants are all identified by a formula number which may be used for exact duplication.

(1) Dilute Emulsion Type Lubricants

To prepare the dilute emulsion type lubricants, the following concentrates were used:

Concentrate	Formula
Lard Oil, Free Fatty Acid (FFA) 2.7% (oleic)	MP-970
Lard Oil, FFA (oleic) 15%	MP-971
98% Lard Oil, FFA (oleic) 15% plus 2% aluminum stearate	MP-973
80% Lard Oil, FFA (oleic) 15% plus 20% oxidized fatty oil	MP-975
95% Lard Oil, FFA (oleic) 15% plus 5% non-ionic surface active agent	MP-976
Tall Oil Ester D	MP-977
Sulphonated Tallow, FFA (oleic) 10%	MP-978

Average Fatty Acid Content of Lard Oil:

Saturated Acids:	Myristic Acid	1%
	Palmitic Acid	21%
	Stearic Acid	8%
Mono-unsaturated Acids:	Palmitoleic Acid	3%
	Oleic Acid	58%
Poly-unsaturated Acids:	Linoleic Acid	9%

Saponification Value - 192-200

Iodine Value - 61-79

Average Acid Content of Tall Oil:

Abietic Acid	40%
Oleic Acid	40%
Unsaponifiable	20%

Average Fatty Acid Content of Tallow:

Saturated Acids:	Myristic Acid	2%
	Palmitic Acid	32%
	Stearic Acid	15%
Mono-unsaturated Acids:	Oleic Acid	48%
Poly-unsaturated Acids:	Linoleic Acid	3%

Saponification Value - 190-199

Iodine Value - 40-48

The sulfonation of tallow results in the sulfate group being bound at an ethylenic linkage.

The dilute emulsion type lubricants, in all cases, contained 2% by weight of one of the above concentrates emulsified in a solution containing 1 1/2% by weight of sodium stearate.

The emulsions were used under the following conditions:

Temperature	Immersion Time
30°C (Room Temperature)	Few Seconds (5 - 60 sec.)
30°C " "	one hour
70°C	few seconds (5 - 60 sec.)
70°C	one hour

All specimens immersed in the lubricant at room temperature (30°C) were immediately used for the physical tests. All specimens immersed in the lubricant at 70°C were allowed to cool and then were immersed in the same lubricant at room temperature for a few seconds.

pH Value and Hydrolysis (in %) of the Dilute Emulsions:

	pH	% Hydrolysis
Lard Oil, FFA 2.7%	9.45	58.5
Lard Oil, FFA 15%	9.5	51.5
Lard Oil plus Aluminum Stearate	9.4	65.6
Lard Oil plus oxidized fatty oil	9.6	53.0
Lard Oil plus active agent	9.5	45.3
Tall Oil Ester D	9.15	40.0
Sulphonated Tallow	9.82	34.2

(2) Solid, Dry Lubricants

The following pure metal stearates were applied:

Sodium Stearate
Calcium Stearate
Magnesium Stearate
Aluminum Stearate
Zinc Stearate

These lubricants were applied by smearing over the surface of the test specimen.

(3). Commercial Lubricants

	Die Gard 24		Die Gard 80	
Soap:	9. %	Potassium Oleate	15.8%	Potassium Oleate and Stearate
Fatty Esters:	21. %	Glycerides of Palmitic Oleic and Stearic Acids	38.3%	Glycerides of Palmitic, Oleic and Stearic Acids
Free Fatty Acid:	2.4%		2.5%	
Pigment:	33. %	Chalk	---	None
Free Sulphur:	2.3%		---	None
Water:	28. %		39. %	

These lubricants were used as delivered and applied by smearing over the test specimen.

d. Physical Tests

(1) Determination of the Apparent Coefficients of Friction

The total load applied in the cold working of steel is the sum of:

The force necessary to overcome the flow resistance of the steel, which is dependent upon the physical and chemical properties of the steel, and

the force necessary to overcome the frictional resistance occurring between two sliding surfaces.

Therefore, any measure taken which reduces friction will facilitate the cold working process. Hence, the knowledge of the degree of friction is invaluable in cold working.

The coefficient of friction is determined by

$$\mu = \frac{F}{W}$$

where μ is the coefficient of friction,
F the force necessary to cause movement in a direction parallel to the surfaces, and
W the total load to which the surfaces are subjected perpendicular to the surfaces.

The coefficient is a constant of the material or materials (dissimilar). This constant is not of decisive importance in lubrication and surface

treatment. Instead, it is the change in this constant which results by the introduction of specific surface and lubrication conditions which is of prime importance. Hence, it is felt justified that the resulting coefficient of friction as determined under specific surface or lubrication conditions be termed the apparent coefficient of friction, for the purpose of comparison.

The schematic drawing (No.1) shows the device used to determine the apparent coefficient of friction. The test specimen is placed between two highly polished jaws (which have a surface hardness of about 60, Rockwell C). By means of a screw and a torque wrench (previously calibrated), the desired unit pressures are produced. A tensile testing machine was used to supply the necessary moving force. The surface area of the jaws for tests in the unit pressure range 1250 to 12,500 psi was 4 sq. in.; for 50,000 psi, 0.5 sq. in.; and for 100,000 to 200,000 psi, 0.25 sq. in.

The calculation of the apparent coefficient of friction is based on the constant Force F given by the tensile testing machine.

(2) Determination of the Load During Drawing. (i.e., increase of total area under tension).

For this test, the same device was applied with the exception that a drawing jaw was used having a land area of 0.25 sq. in., and a drawing angle of about 12°. By applying a unit pressure of approximately 200,000 psi, the reduction obtainable for SAE 1010, in conjunction with specific lubrication conditions was determined.

Theoretically, it is possible to determine the apparent coefficient of friction during reduction in cross section from the load obtained and the average flow resistance of the material during reduction. The average flow resistance may be calculated from the flow stresses determined before and after cold-working. However, the coefficient of friction was not determined in this case, because the determination of the flow stresses is especially inaccurate, due to the physical shape of the test specimens used. Since only the lubrication conditions are varied, a much better indication of the value,

although relative, of the surface or lubrication conditions is given by both the percentage reduction obtained and the load required. This load, during reduction, was constant for a specific lubrication condition.

(3) Determination of the Load During Extrusion (i.e., increase of total area under compression).

The schematic drawing (No. 2) shows the device used to extrude the test specimens.

The loads necessary for the various reduction were obtained by the use of a tensile testing machine. The end load obtained during extrusion was recorded and, here again, indicated the value of surface and lubrication conditions during reduction.

4. Effectiveness of Surface Treatments and Lubrication Conditions for the Cold Working of Steel

a. Apparent Coefficients of Friction Found for Various Type Lubricants and Lubrication Conditions in Combination with a Phosphate Coated Surface.

(1) Dilute Emulsion Type Lubricants.

To determine the most effective method of application for this type of lubricant, the immersion time and the temperature were varied.

Table I shows the apparent coefficients of friction found using sulphonated tallow emulsified in sodium stearate. For comparison, both uncoated and phosphatized surfaces were used.

TABLE I.

Unit Pressure psi	Immersion Time	Temperature °C	Apparent Coefficient of Friction Uncoated	Apparent Coefficient of Friction Phosphatized
10,000	few sec.	30	0.114	0.070
	60 min.	30	0.113	0.032
	few sec.	70	0.112	0.082
	60 min.	70	0.108	0.013
50,000	few sec.	30	0.081	0.076
	60 min.	30	0.077	0.068
	few sec.	70	0.075	0.071
	60 min.	70	0.068	0.032
100,000	few sec.	30	0.086	0.066
	60 min.	30	0.082	0.049
	few sec.	70	0.079	0.056
	60 min.	70	0.057	0.042
200,000	few sec.	30	0.084	0.062
	60 min.	30	0.069	0.063
	few sec.	70	0.071	0.068
	60 min.	70	0.070	0.043

The above results are shown graphically in Figs. 1 and 2. Fig. 1, (Uncoated surfaces), shows that the combined factors of prolonged immersion time and elevated temperature lower the apparent coefficient of friction. Table I indicates that beneficial effects are also obtained by the variation of one or the other of these factors.

Fig. 2 shows the results obtained using a phosphatized surface. As in the case of uncoated surfaces, the combined factors of prolonged immersion time and elevated temperatures give considerably lower coefficients. Beneficial effects are also obtained by the variation of one or the other of these factors. However, in the case of a phosphatized surface, the coefficients of friction obtained are but a fraction of the coefficients obtained for uncoated surfaces, particularly for the lower pressure ranges.

Table II and Fig. 3 show the results obtained using two types of Lard Oil applied by immersing for a few seconds at room temperature (30°C), and for one hour at 70°C with a phosphatized surface.

TABLE II.

Unit Pressure psi	Immersion Time	Temperature °C	Apparent Coefficient of Friction: Emulsified Lard Oil (FFA 2.7%)	Apparent Coefficient of Friction: Emulsified Lard Oil (FFA 15%)
2,500	Few sec.	30°C	0.075	0.080
	one hour	70°C	0.074	0.052
10,000	few sec.	30°C	0.062	0.056
	one hour	70°C	0.043	0.029
12,500	few sec.	30°C	0.062	0.048
	one hour	70°C	0.039	0.024
50,000	few sec.	30°C	0.081	0.082
	one hour	70°C	0.076	0.074
100,000	few sec.	30°C	0.072	0.076
	one hour	70°C	0.066	0.066
200,000	few sec.	30°C	0.062	0.061
	one hour	70°C	0.053	0.058

Some beneficial effect is realized by the use of a lard oil having a higher free fatty acid content (FFA 15%), particularly at the lower pressures.

Table III and Figs. 4 and 5 show the effect on the apparent coefficient of friction of various additives to lard oil in combination with a phosphatized surface. These additives, such as aluminum stearate, oxidized fatty oil, etc., are known to possess the characteristic of being readily adsorbed by a metal surface.

TABLE III.

Unit Pressure psi	Immersion Time	Temp.	Emulsified Lard Oil (FFA 15%)	Emulsified Lard Oil plus Aluminum Stearate	Emulsified Lard Oil plus Oxidized Fatty Oil	Emulsified Lard Oil plus Surface Active Agent
2,500	few sec.	30°C	0.080	0.081	0.077	0.073
	one hour	70°C	0.052	0.041	0.039	0.044
10,000	few sec.	30°C	0.056	0.066	0.065	0.069
	one hour	70°C	0.029	0.016	0.012	0.026
12,500	few sec.	30°C	0.048	0.060	0.059	0.086
	one hour	70°C	0.024	0.011	0.013	0.020
50,000	few sec.	30°C	0.082	0.083	0.079	0.087
	one hour	70°C	0.074	0.073	0.077	0.073
100,000	few sec.	30°C	0.076	0.079	0.079	0.077
	one hour	70°C	0.066	0.066	0.073	0.068
200,000	few sec.	30°C	0.061	0.065	0.065	0.064
	one hour	70°C	0.058	0.058	0.050	0.052

Table III and Figs. 4 and 5 show that for short time immersion at room temperature no beneficial effect is obtained by the inclusion of such additives mentioned above. With increased immersion time and elevated temperature the additives, aluminum stearate and oxidized fatty oil, gave desirable decreases in the coefficient of friction at the lower pressures; whereas, the addition of a surface active agent gave the best result at the higher pressures.

Table IV and Fig. 6 show the results obtained using two other types of dilute emulsified lubricants in combination with phosphatized surfaces.

TABLE IV.

Unit Pressure psi	Immersion Time	Temperature	Apparent Coefficient of Friction Emulsified Sulphonated Tallow	Apparent Coefficient of Friction Emulsified Tall Oil Ester
2,500	few sec.	30°C	0.080	0.120
	one hour	70°C	0.030	0.100
10,000	few sec.	30°C	0.070	0.100
	one hour	70°C	0.013	0.080
12,500	few sec.	30°C	0.051	0.106
	one hour	70°C	0.012	0.095
50,000	few sec.	30°C	0.076	0.084
	one hour	70°C	0.032	0.078
100,000	few sec.	30°C	0.066	0.088
	one hour	70°C	0.042	0.073
200,000	few sec.	30°C	0.068	0.073
	one hour	70°C	0.043	0.064

All tests (see Tables I to IV and Figs. 1 to 6) performed to determine the apparent coefficient of friction, using dilute emulsion type lubricants, show that by the proper application of the lubricant, desirable decreases in the coefficient of friction are obtained. This decrease in the coefficient, for the type lubricants used, is dependent, somewhat, upon their various chemical compositions. However, much greater importance must be attached to the influence of the immersion time and temperature, since much greater differences in the coefficients are obtained with prolonged immersion at elevated temperatures, especially at lower pressures. In the higher pressure range of 50,000 to 200,000 psi, wherein severe drawing and extrusion occur, the differences in the coefficient, although of a lesser degree, are still quite marked. Of the lubricants tested, sulphonated tallow gave the best results.

In determining the effectiveness of dilute emulsion type lubricants, or, more simply, soap solutions, consider first uncoated material. So that no unknown factors may influence the determination, pure sodium stearate was used.

Soap, i.e., sodium stearate, hydrolyzes to a great degree when dissolved in water -- the greater the dilution and the higher the temperature the greater is the degree of hydrolysis with the formation of stearic acid.

$$RCOONa + HOH \rightleftharpoons RCOOH + NaOH$$

$$RCOONa + RCOOH \longrightarrow \underset{\text{(Acid Soap)}}{RCOONa.RCOOH}$$

R represents the stearic acid radical, and may also represent any high fatty acid, or any high molecular weight organic acid radical.

The more free stearic acid or acid soap present, the more effective is the lubrication. Theoretically, the ideal solution would be one in which the sodium stearate is completely hydrolyzed. However, the processing, such as stamping, drawing, extrusion, etc., to which the steel is to be subjected, governs the degree of dilution of the lubricant. The various metal working processes require a unit pressure peculiar to the specific process; therefore, the thickness of the lubricant film must be varied to prevent metal to metal contact.

In Fig. 7 are shown the degree of hydrolysis and pH values of sodium stearate at various concentrations. It is of interest to note the influence of the concentration of sodium stearate on the degree of hydrolysis, in that as the concentration increases, the degree of hydrolysis decreases.

Theoretically, the higher the temperature, the greater the degree of hydrolysis. However, the hydrolysis is influenced by the greater solubility of the stearic acid at higher temperatures; this increased solubility overshadows the hydrolysis of the sodium stearate.

The phenomena occurring between the fatty acid or acid soaps and the metal surface is one of adsorption, the quantity adsorbed depending, to a certain degree, on time. Therefore, longer immersion periods will result in a decrease in the coefficient of friction.

To determine the adsorptive ability of a phosphate coating and to prove that a true adsorption occurs, tests were made as described below, based on the Froundlich Adsorption Isotherm.

$$a = \lambda c^{\frac{1}{n}}$$

where a = quantity adsorbed in moles
c = concentration in moles/liter in solution
λ & $\frac{1}{n}$ are constants

or --

$$\log a = \log \lambda + \frac{1}{n} \log c$$

The plot of log "a" against log "c" formed a straight line, proving that true adsorption exists.

For these tests, solutions of methylene blue were used as adsorbate in the following concentrations:

c_1 = .01 mole, c_2 = .001 mole, c_3 = .0001 mole, etc.

The phosphatized surface, as adsorbent, was always 1 m^2.

The ability of a phosphate coating to adsorb organic compounds is very great. Zinc phosphate coatings adsorb about two to five times as much mineral oil as the same surface uncoated. The quantity of oil adsorbed depends somewhat on the crystal structure and the thickness of the phosphate coating.

If a phosphatized surface (zinc phosphate) is immersed in a sodium stearate solution, an almost completely water-insoluble soap film is formed upon the coated surface. Its appearance also changes, evidencing the existence of an add ,ional reaction between the phosphate coating and the sodium stearate.

An analysis of this lubricant-coating showed that only about 5-10% can be removed by prolonged boiling in water. This soluble portion consists of approximately 95% sodium stearate or acid sodium stearate and 5% zinc stearate. The remaining portion (90-95%) is soluble only in organic solvents and is composed

principally of zinc stearate. Thus, the soluble sodium stearate is converted into the insoluble zinc stearate, which conversion can only take place by a chemical reaction between the phosphate coating and the soap solution.

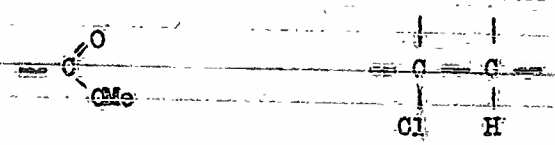
The immersion time and temperature greatly influence the quantity of the lubricant film (zinc stearate) which is formed on the phosphate coating as shown in the following table:

TABLE V.

1% Sodium Stearate Solution

Immersion Time	Temperature	Weight of Lubricant film = gms./sq.ft.
few seconds	20°C	0.070
few seconds	70°C	0.090
one hour	20°C	0.150
one hour	70°C	0.740

Lubricants containing polar groups are readily adsorbed by phosphate coatings. Such polar groups are:



A soap emulsion, which contains a carboxyl group, is a "polar lubricant".

The alkalinity of a solution or emulsion of such polar lubricants in water permits the occurrence of a chemical reaction between the phosphate coating and the lubricant.

The following chemical reactions occur:

$$(6)RCOONa + (6)HOH \rightleftharpoons (6)RCOOH + (6)NaOH$$

$$(6)[RCOO^-] + (6)[H^+] \qquad (6)[Na^+] + (6)[OH^-]$$

$$Zn_3(PO_4)_2 + 6Na^+ + 6OH^- \longrightarrow 3Zn^{++} + 6OH^- + 2Na_3PO_4$$

$$3Zn^{++} + 6RCOO^- + 6H^+ + 6OH^- \longrightarrow 3Zn(RCOO)_2 + 3H_2O$$

$$Zn_3(PO_4)_2 + 6RCOONa + (6HOH) \longrightarrow$$
$$3Zn(RCOO)_2 + 2Na_3PO_4 + (3HOH)$$

By hydrolysis, stearic acid and sodium hydroxide are formed -- the latter dissolving a minute but sufficient amount of the zinc phosphate coating to form zinc ions. The zinc ions then react with the stearate ions to form insoluble zinc stearate which becomes interlocked with the phosphate coating.

Hence, adsorption and chemical reaction determine the formation of a lubricant film well interlocked with the phosphate coating. This combination is irreversible and may be termed chemisorption. Such an irreversible adsorption occurs when a chemical reaction between the adsorbent and the adsorbate is possible.

The kinetics of the foregoing reactions must also be considered. The speed with which the crystals (zinc phosphate) dissolve is determined by the equation:

$$C_x = C_o\left(1 - e^{-\frac{DO}{\delta}}\right)$$

Where C_o = concentration at saturation
C_x = concentration after a certain time
D = coefficient of diffusion
O = area of crystal
δ = coating thickness

The speed of formation and the amount of lubricant film formed vary to a great degree according to the physical and chemical properties of the soap solution, the method of application and, to a certain extent, to the physical properties of the phosphate coating. How these factors greatly affect friction is clearly demonstrated by the determined coefficients of friction, as shown in the foregoing tables.

(2) Solid Lubricants

Table VI and Fig. 8 give the coefficients of friction obtained using solid lubricants in combination with phosphatized surfaces.

TABLE VI

Apparent Coefficients of Friction

Unit Pressure psi	Sodium Stearate	Calcium Stearate	Magnesium Stearate	Zinc Stearate	Aluminum Stearate
2,500	0.059	0.071	0.063	0.075	0.087
10,000	0.048	0.052	0.059	0.066	0.066
12,500	0.045	0.046	0.060	0.050	0.062
50,000	0.079	0.080	0.080	0.082	0.086
100,000	0.077	0.074	0.070	0.078	0.087
200,000	0.064	0.065	0.062	0.066	0.072

The coefficients of friction found using the various stearates show comparatively little differences. Compared with the dilute emulsion type lubricants, the solid stearates gave, generally, higher coefficients for the lower pressure ranges, and somewhat similar coefficients for the higher pressures.

(3) Commercial Lubricants

Table VII and Fig. 9 show the coefficients of friction found using commercial type lubricants in combination with a phosphatized surface.

TABLE VII

Apparent Coefficient of Friction

Unit Pressure psi	Die Gard 24	Die Gard 80
2,500	0.040	0.056
10,000	0.024	0.066
12,500	0.032	0.067
50,000	0.064	0.082
100,000	0.069	0.080
200,000	0.045	0.062

No improvement of frictional conditions is given by the use of commercial lubricants over the proper use of dilute emulsions; rather, the better cooling effect offered by the latter makes them preferable for severe cold-working operations, where great heat is generated.

b. Apparent Coefficients of Friction Found Using Various Type Lubricants on Uncoated and Phosphatized Surfaces.

Table VIII and Fig. 10 show the coefficients of friction found for the best of each of the three type lubricants (dilute emulsion, solid, and commercial) applied on both uncoated and phosphatized surfaces.

TABLE VIII

Apparent Coefficients of Friction

Unit Pressure psi	Sulphonated Tallow		Calcium Stearate		Die Gard 24	
	Uncoated	Phosphatized	Uncoated	Phosphatized	Uncoated	Phosphatized
2,500	0.182	0.030	0.140	0.071	0.073	0.040
10,000	0.108	0.013	0.093	0.052	0.055	0.024
12,500	0.102	0.012	0.092	0.048	0.066	0.032
50,000	0.068	0.032	0.100	0.080	0.086	0.064
100,000	0.057	0.042	0.090	0.074	0.079	0.069
200,000	0.070	0.045	0.078	0.065	0.061	0.045

The results show that appreciably higher coefficients are given by uncoated than by phosphatized surfaces for each type lubricant.

c. Apparent Coefficients of Friction Found Using Various Types of Commercially Applicable Phosphate Coatings.

The coatings applied were produced as follows:

Zinc Phosphate coating produced in a bath containing zinc phosphate and zinc nitrate as accelerator.
Manganese Phosphate coating produced in a bath containing manganese phosphate and sodium nitrate as accelerator.
Cadmium Phosphate coating produced in a bath containing cadmium phosphate and cadmium nitrate as accelerator.

Lubrication: 2% sulphonated tallow emulsified in 1 1/2% sodium stearate.

Immersion Time: 1 hour at 70°C.

In Table IX and Fig. 11 are given the results obtained for these tests.

TABLE IX

Unit Pressure psi	Apparent Coefficient of Friction			
	Uncoated	Zinc Phosphate	Manganese Phosphate	Cadmium Phosphate
10,000	0.108	0.013	0.095	0.054
50,000	0.068	0.032	0.070	0.069
100,000	0.057	0.042	0.059	0.055
200,000	0.070	0.043	0.065	0.055

Zinc phosphate gave the lowest apparent coefficients of friction.

d. Comparison of Various Type Lubricants in Combination with a Phosphate Coating During Reduction (Increase of Total Area) under Tension (Drawing)

For these tests, two of each of the type lubricants were selected as follows:

(1) Dilute Emulsion Type

(a) Saturated and unsaturated carboxylic acids (fatty acids): Emulsified lard oil containing active agent.

(b) Terpene group: Emulsified tall oil ester.

(2) Solid Type

(a) Water soluble: Sodium Stearate

(b) Water insoluble: Calcium Stearate

(3) Commercial Type

(a) Pigmented: Die Gard 24

(b) Unpigmented: Die Gard 80

Table X and Fig. 12 show the loads required and reductions obtained in drawing SAE 1010, applying a unit pressure of 200,000 psi.

TABLE X

Lubricant	Load	Reduction
Emulsified lard oil containing active agent (70°C for 1 hour)	2360 lbs.	52.5%
Emulsified tall oil ester (70°C for 1 hour)	2440 lbs.	51.0%
Sodium stearate (dry)	2370 lbs.	53.0%
Calcium Stearate (dry)	2460 lbs.	47.0%
Die Gard 24 (pigmented)	2300 lbs.	39.4%
Die Gard 80 (unpigmented)	2220 lbs.	45.0%

As previously discussed under "Procedure" the determination of the coefficients of friction during reduction is inaccurate and considered impractical. However, it is interesting to note the coefficients of friction found using the above lubricants in combination with phosphate coatings at a unit pressure of 200,000 psi without reduction, given in Table XI.

TABLE XI

Lubricant	Load Req'd.	Apparent Coefficient of Friction Unit Pressure - 200,000 psi
Emulsified lard oil containing active agent (70°C for 1 hour)	2580 lbs.	0.052
Emulsified tall oil ester (70°C for 1 hour)	3180 lbs.	0.064
Sodium stearate (dry)	3200 lbs.	0.064
Calcium stearate (dry)	2240 lbs.	0.065
Die Gard 24	2260 lbs.	0.045
Die Gard 80	3100 lbs.	0.062

In the cases recorded in the foregoing table, the load is necessary only to overcome the external friction; in the former cases, shown in Table X, the load was required to overcome not only the external but also the internal friction offered by the material itself. A comparison of these loads clearly indicates that more favorable frictional conditions are present when reduction in cross section occurs.

This is explained by the fact that when the coating is crushed in the first cold-working of coated surfaces, the coating becomes flatter and smoother, in some cases becoming glass-like in appearance. Microscopic examination (microphotographs 1 and 2) confirms that the surface is much smoother, accounting for the difference in the friction conditions.

These tests approach, to a great extent, the practical conditions experienced in severe cold working operations. The tests, too, show that the coefficient of friction is not the sole deciding factor in proving the value of a lubricant and surface conditions. Properties, such as anti-welding, heat conductivity, or cooling effect, etc., of the lubricant or surface, are also important factors to be considered.

The dilute emulsion type lubricants proved to be the best in comparison with commercial lubricants in these tests. This is believed to be due to the greater cooling effect of the dilute type lubricant. The dry stearates evidence their effectiveness in these tests, the results of which are in agreement with the success experienced by the widespread use of such lubricants, as, for instance in the drawing of wire.

The results given in Table X for Die Gard 24 pigmentd, and Die Gard 80 unpigmented appear to indicate that a pigment may be detrimental to phosphate coatings, as has been experienced in industry.

e. Comparison of Coated and Uncoated Surfaces During Reduction (or Increase of Total Area) Under Compression (Cold Extrusion)

Three types of surface conditions were applied to the C1010 AISI rimmed steel test specimens, i.e., normal coating, thin coating, and uncoated. Emulsified sulphorated tallow was applied to all specimens for one hour at 70°C. Preparation and testing of the specimens are described under "Procedure". Three specimens for each surface condition were used for all reductions.

Table XII and Fig. 13 show the loads required for various reductions.

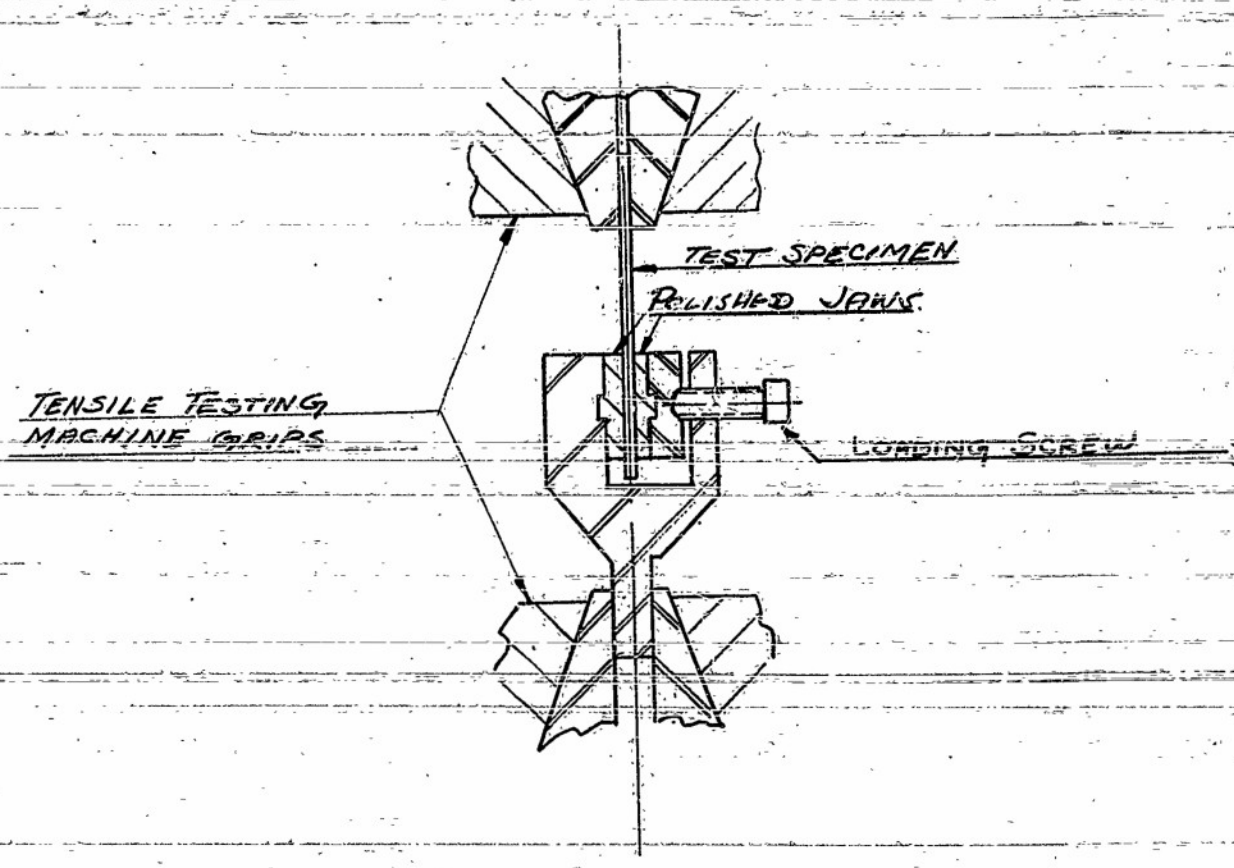
TABLE XII

Surface Condition	Reduct.	Load	Load psi	Remarks
Uncoated	20%	19,000	296,000	Heavy pickup - all specimens
Thin	20%	17,400	270,000	
Normal	20%	12,400	193,000	
Uncoated	40%	49,000	232,000	Heavy pickup - all specimens
Thin	40%	37,200	216,000	
Normal	40%	35,800	208,000	
Uncoated	60%	-	-	
Thin	60%	79,200	210,000	pickup on all specimens
Normal	60%	75,000	198,000	
Uncoated	85%	-	-	
Thin	85%	-	-	
Normal	85%	138,750	260,000	

The normal coatings, with which the lowest loads were obtained, proved successful for all reductions. Thin coatings resulted in comparatively higher loads with the first evidence of pickup appearing at 60% reduction. The highest loads were obtained with uncoated specimens, heavy pickup occurring even during the lowest reduction attempted (20%).

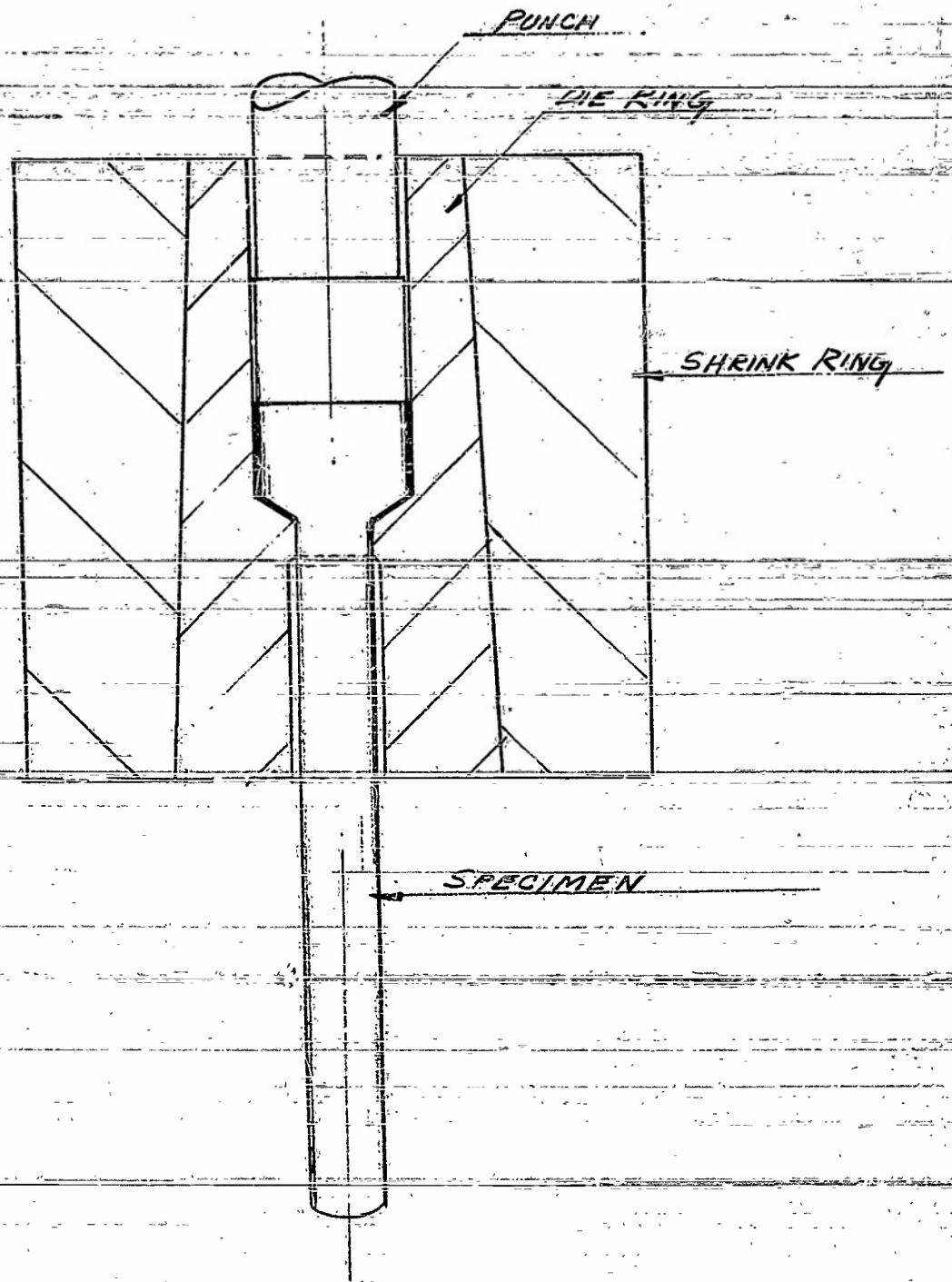
The foregoing evinces the desirability of the normal coating, the minimum weight of which is 1 gm/sq.ft.

Photographs 1 through 4 show the extruded specimens. Galling and pickup are to be seen on all uncoated specimens and on the thin coated specimen reduced 60%. It is obvious that special surface treatment is necessary for the successful cold extrusion of steel. The use of organic lubricants, unaided, for this purpose is not possible since they lack the necessary properties found in an inorganic coating or layer, such as anti-welding, heat-resistance, and adherence to the metal surface.



SCHEMATIC DIAGRAM OF DEVICE USED TO DETERMINE THE COEFFICIENT OF FRICTION

No. 1



SCHEMATIC DIAGRAM SHOWING METHOD OF EXTRUSION

No. 2

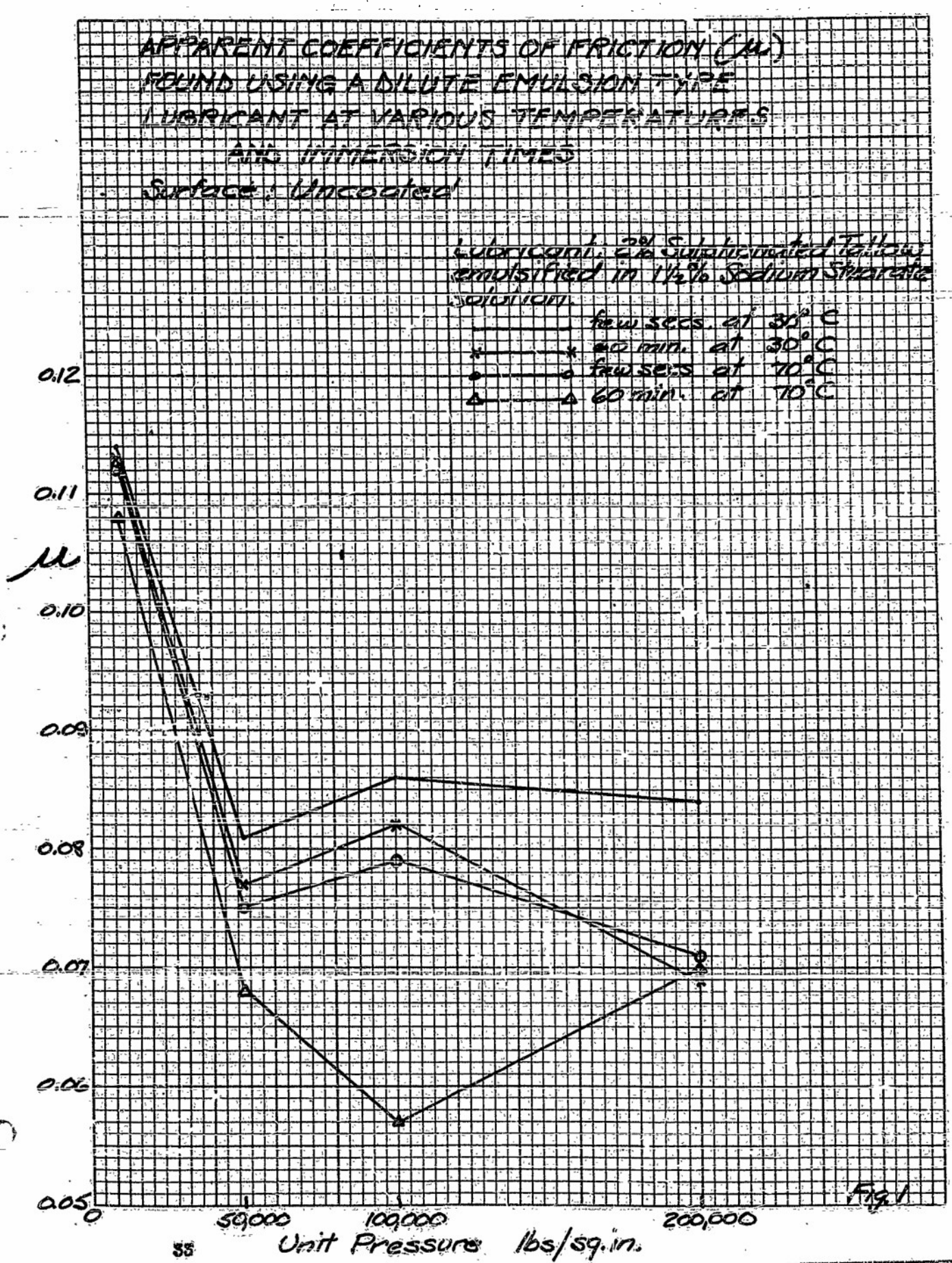

APPARENT COEFFICIENTS OF FRICTION (μ)
FOUND USING A DILUTE EMULSION TYPE
LUBRICANT AT VARIOUS TEMPERATURES
AND IMMERSION TIMES
Surface: Uncoated
Lubricant: 50% Sulphonated Tallow
emulsified in 1½% Sodium Stearate
solution.
few secs. at 30° C
60 min. at 30° C
few secs. at 70° C
60 min. at 70° C
0.12
0.11
μ
0.10
0.09
0.08
0.07
0.06
0.05
0
50,000
100,000
200,000
Unit Pressure lbs/sq.in.

Fig. 1

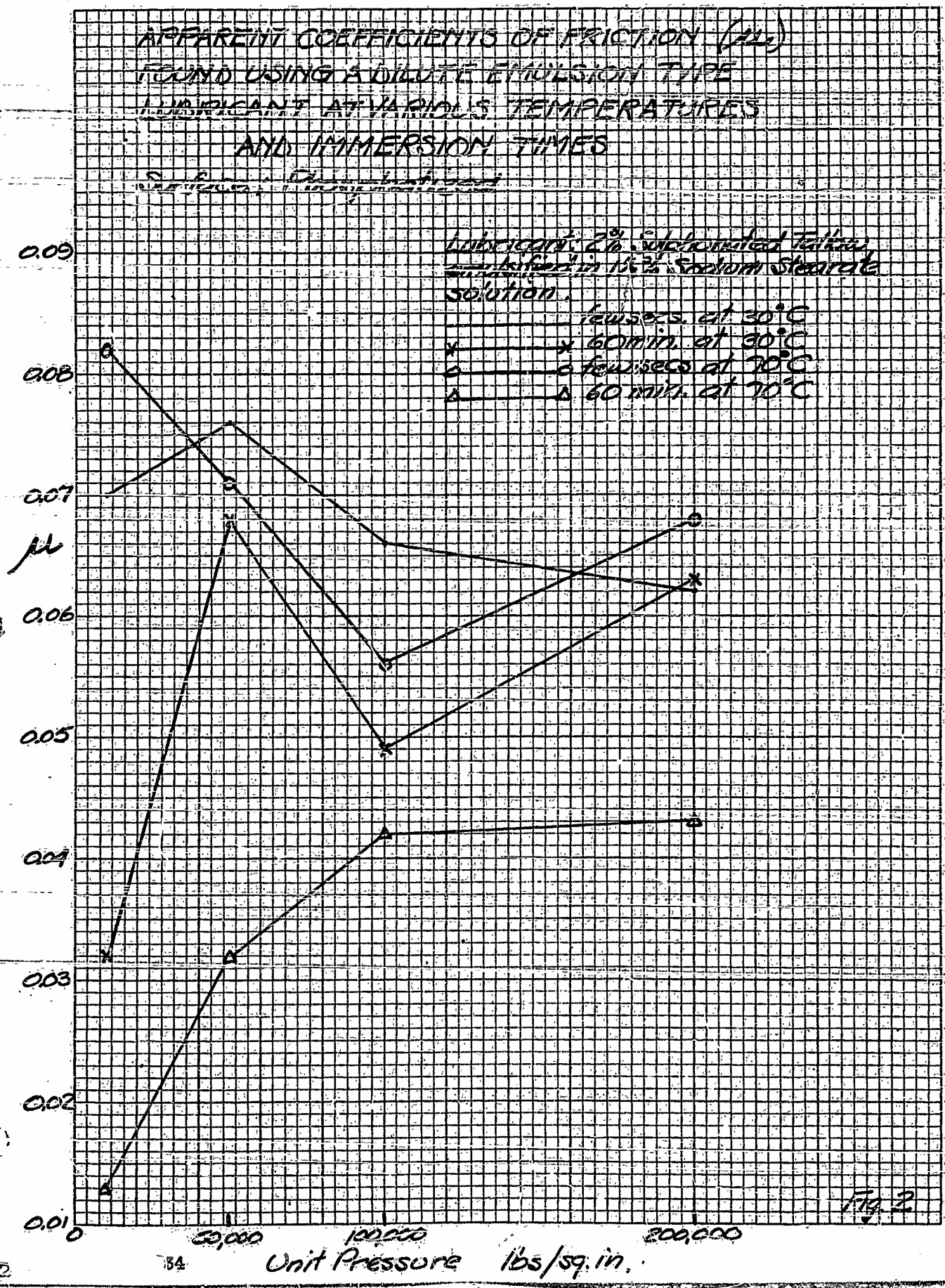
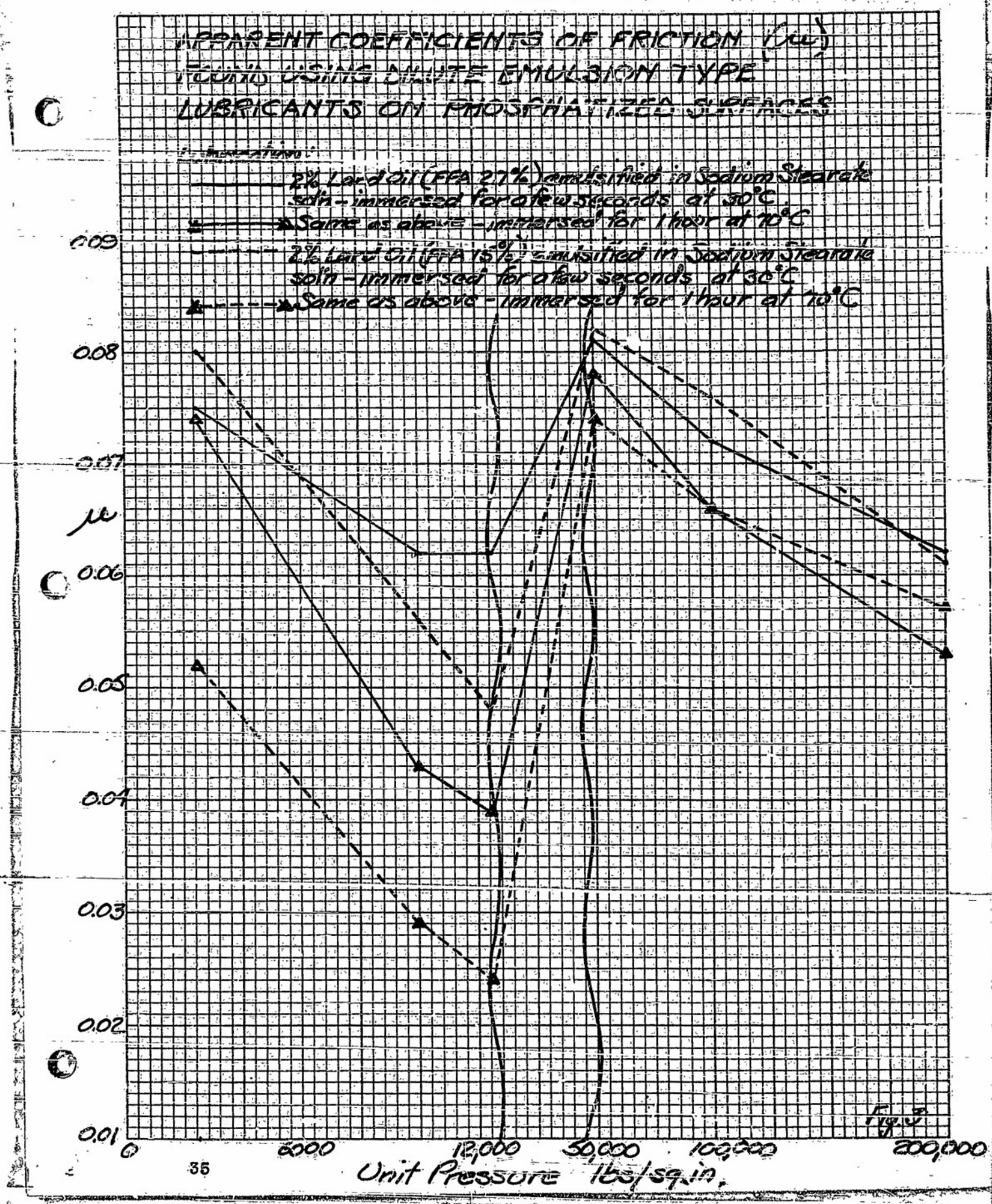

APPARENT COEFFICIENTS OF FRICTION (μ)
FOUND USING A DILUTE EMULSION TYPE
LUBRICANT AT VARIOUS TEMPERATURES
AND IMMERSION TIMES
Lubricant: 2% saturated Tallow
Lubricant in 1½% Sodium Stearate
solution.
few secs. at 30°C
60 min. at 30°C
few secs at 70°C
60 min. at 70°C
0.09
0.08
0.07
μ
0.06
0.05
0.04
0.03
0.02
0.01
0
50,000
100,000
200,000
Unit Pressure lbs/sq. in.

Fig. 2


APPARENT COEFFICIENTS OF FRICTION (μ)
FOUND USING DILUTE EMULSION TYPE
LUBRICANTS ON PHOSPHATIZED SURFACES
2% Lard oil (FFA 2.7%) emulsified in Sodium Stearate soln - immersed for a few seconds at 30°C
Same as above - immersed for 1 hour at 70°C
2% Lard oil (FFA 15%) emulsified in Sodium Stearate soln - immersed for a few seconds at 30°C
Same as above - immersed for 1 hour at 70°C
0.09
0.08
0.07
μ
0.06
0.05
0.04
0.03
0.02
0.01
0
2000
12,000
50,000
100,000
200,000
Unit Pressure lbs/sq.in.

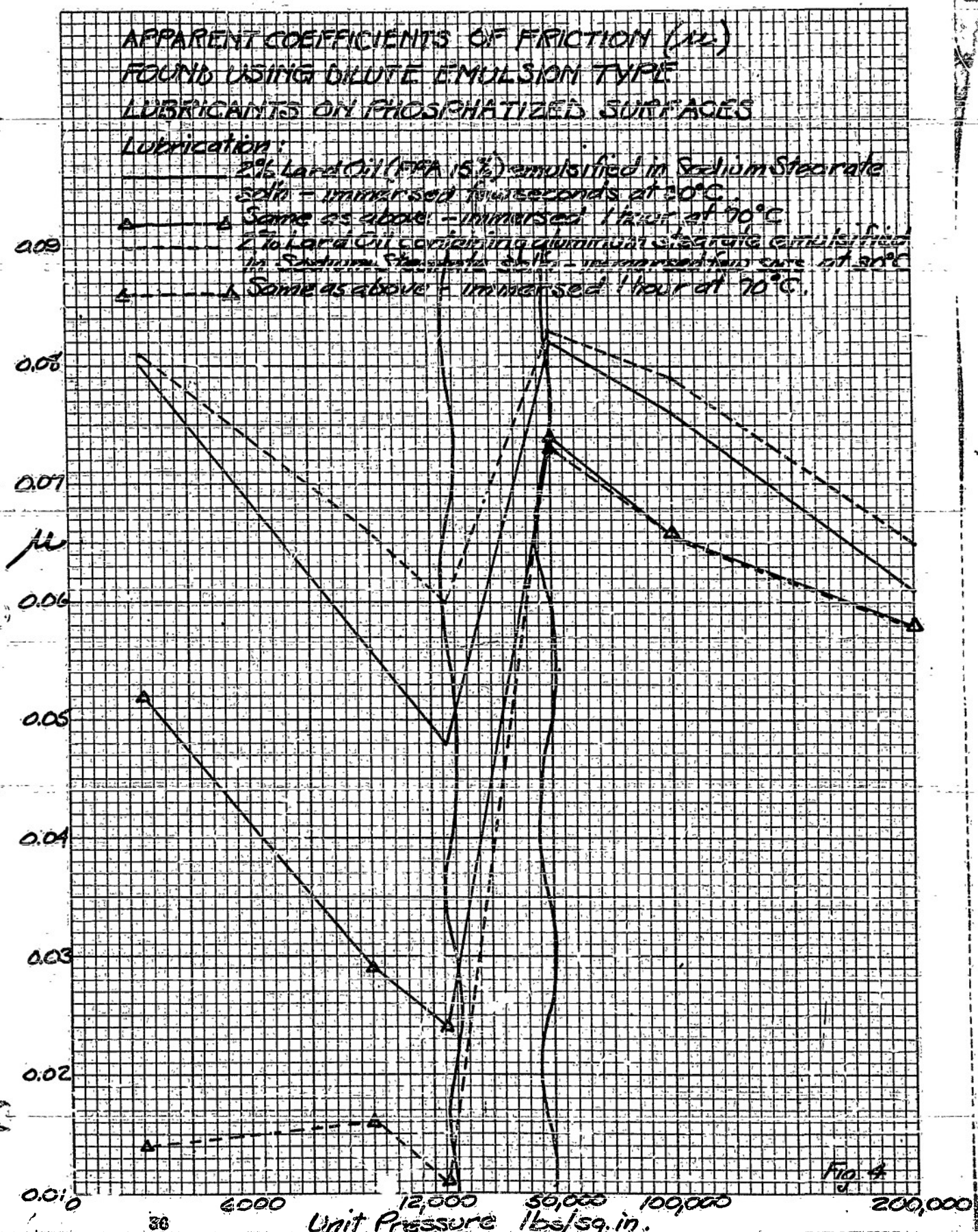

APPARENT COEFFICIENTS OF FRICTION (μ)
FOUND USING DILUTE EMULSION TYPE
LUBRICANTS ON PHOSPHATIZED SURFACES
Lubrication:
2% Lard Oil (FFA 15%) emulsified in Sodium Stearate soap - immersed fifteen seconds at 20°C.
Same as above - immersed 1 hour at 70°C.
2% Lard Oil containing aluminum stearate emulsified in Sodium Stearate soap - immersed one at 20°C
Same as above - immersed 1 hour at 70°C.
μ
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
6000
12,000
50,000
100,000
200,000
Unit Pressure lbs/sq. in.

Fig. 4

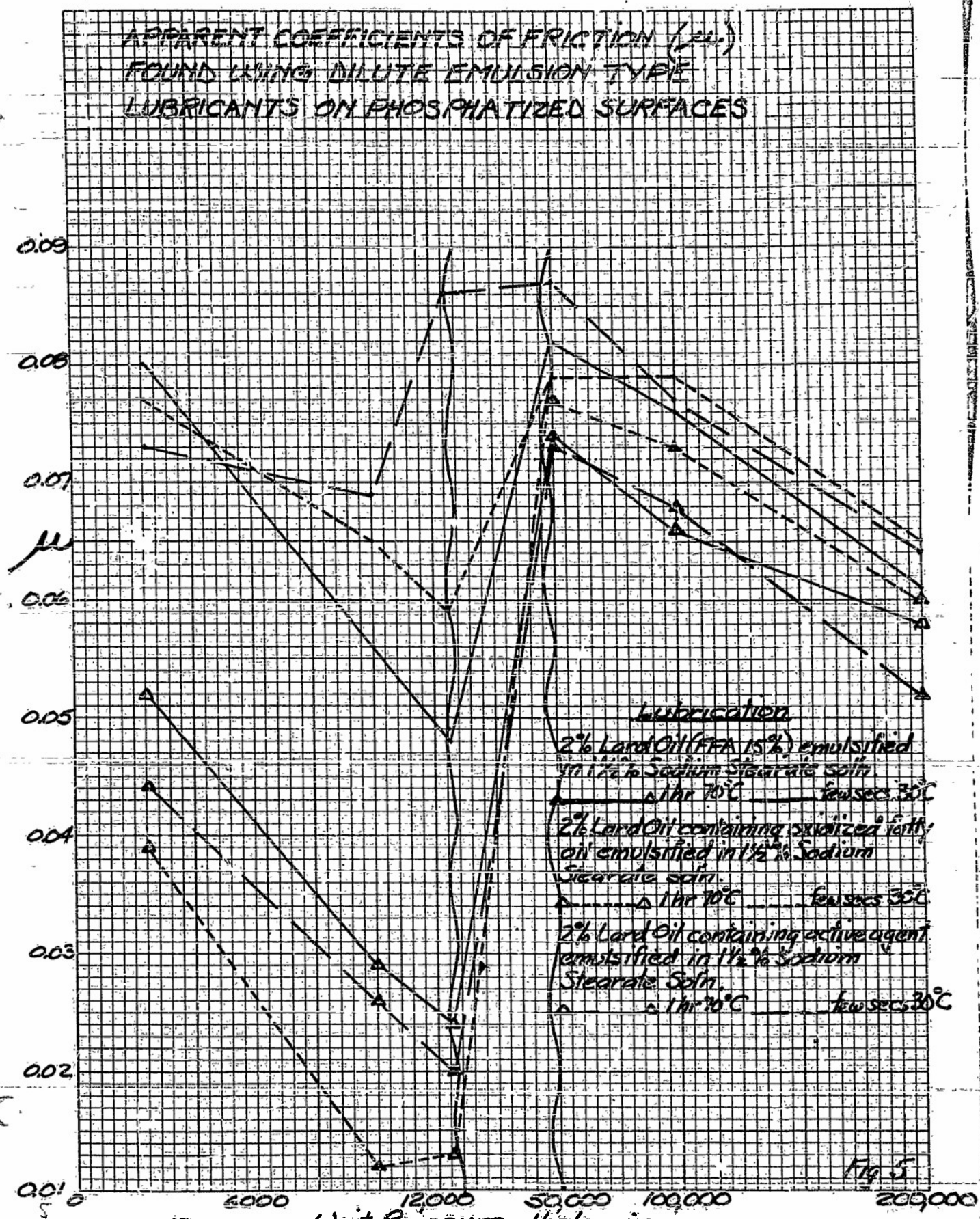

APPARENT COEFFICIENTS OF FRICTION (μ)
FOUND USING DILUTE EMULSION TYPE
LUBRICANTS ON PHOSPHATIZED SURFACES
0.09
0.08
0.07
μ
0.06
0.05
0.04
0.03
0.02
0.01
0
6000
12,000
50,000
100,000
200,000
Unit Pressure lbs/sq. in.
Lubrication
2% Lard Oil (FFA 15%) emulsified in 1½% Sodium Stearate soln.
1 hr 70°C
few secs 30°C
2% Lard Oil containing oxidized fatty oil emulsified in 1½% Sodium Stearate soln.
1 hr 70°C
few secs 30°C
2% Lard Oil containing active agent emulsified in 1½% Sodium Stearate Soln.
1 hr 70°C
few secs 30°C

Fig 5

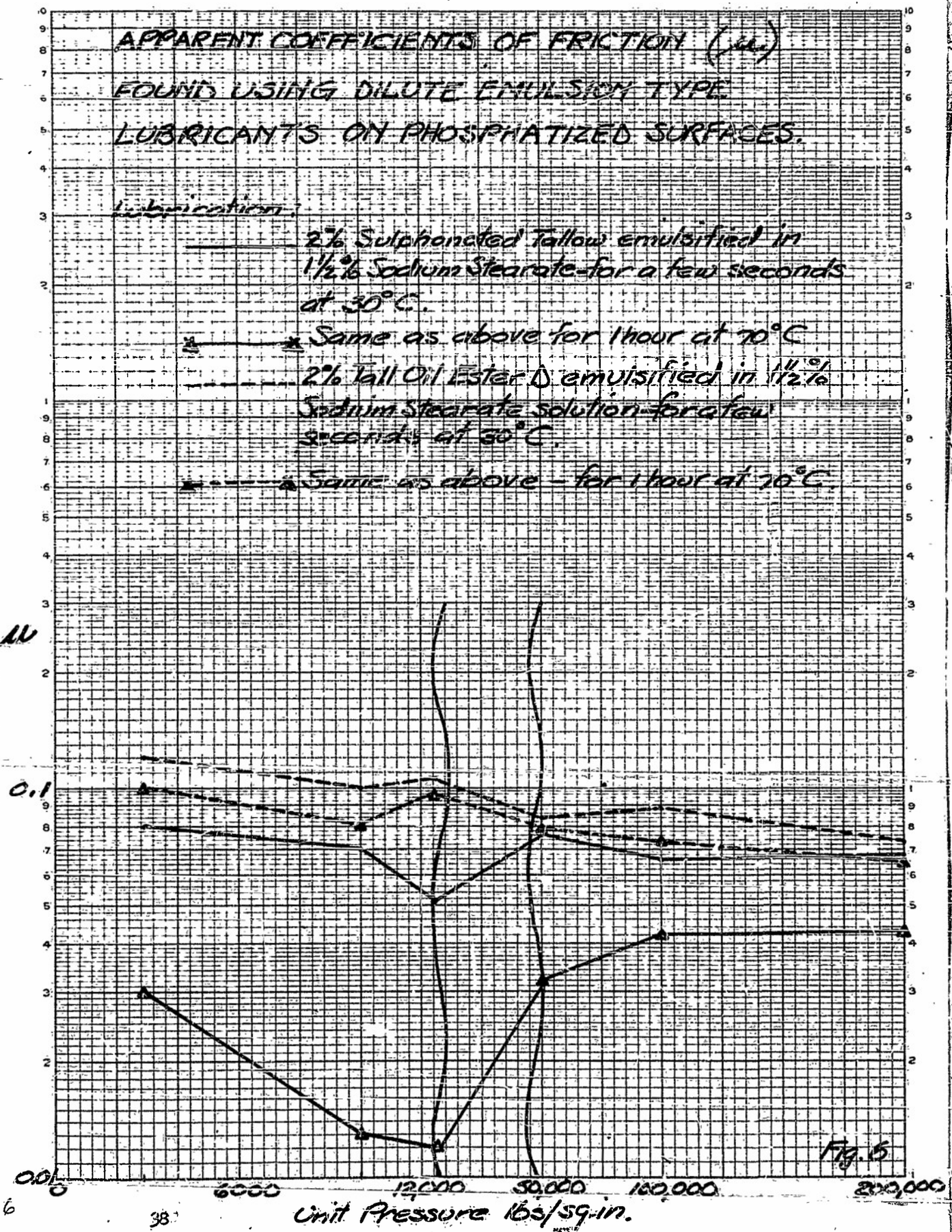

APPARENT COEFFICIENTS OF FRICTION (μ)
FOUND USING DILUTE EMULSION TYPE
LUBRICANTS ON PHOSPHATIZED SURFACES.
Lubrication:
2% Sulphonated Tallow emulsified in 1½% Sodium Stearate-for a few seconds at 30°C.
Same as above for 1 hour at 70°C
2% Tall Oil Ester D emulsified in 1½% Sodium Stearate solution-for a few seconds at 30°C.
Same as above - for 1 hour at 70°C.
μ
0.1
0.01
0
6000
12,000
50,000
100,000
200,000
Unit Pressure lbs/sq-in.
NO. 340-L310 EDCO EFFICIENCY-SEMI LOGARITHMIC—3 CYCLES X 5 DIVISIONS

Fig. 5

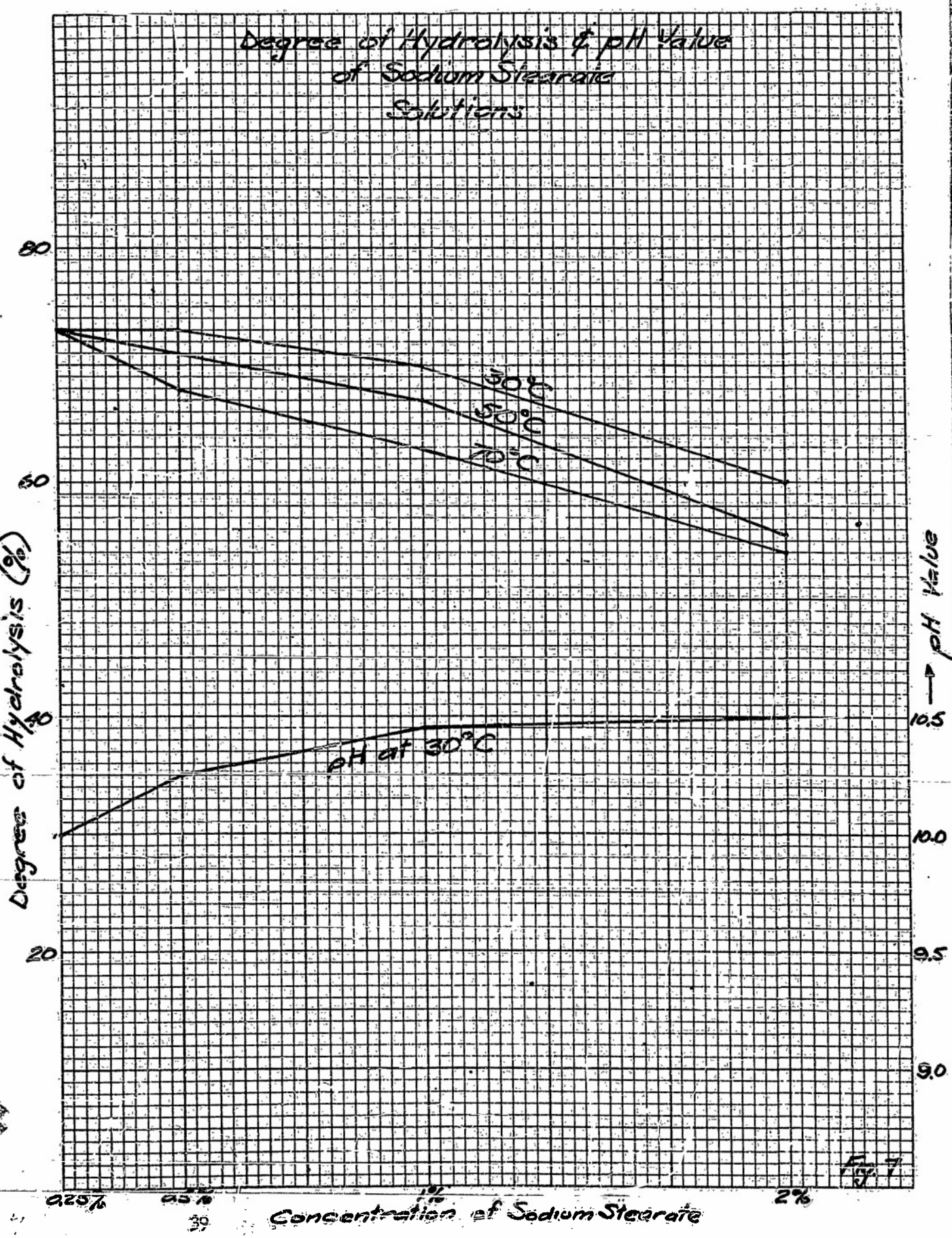
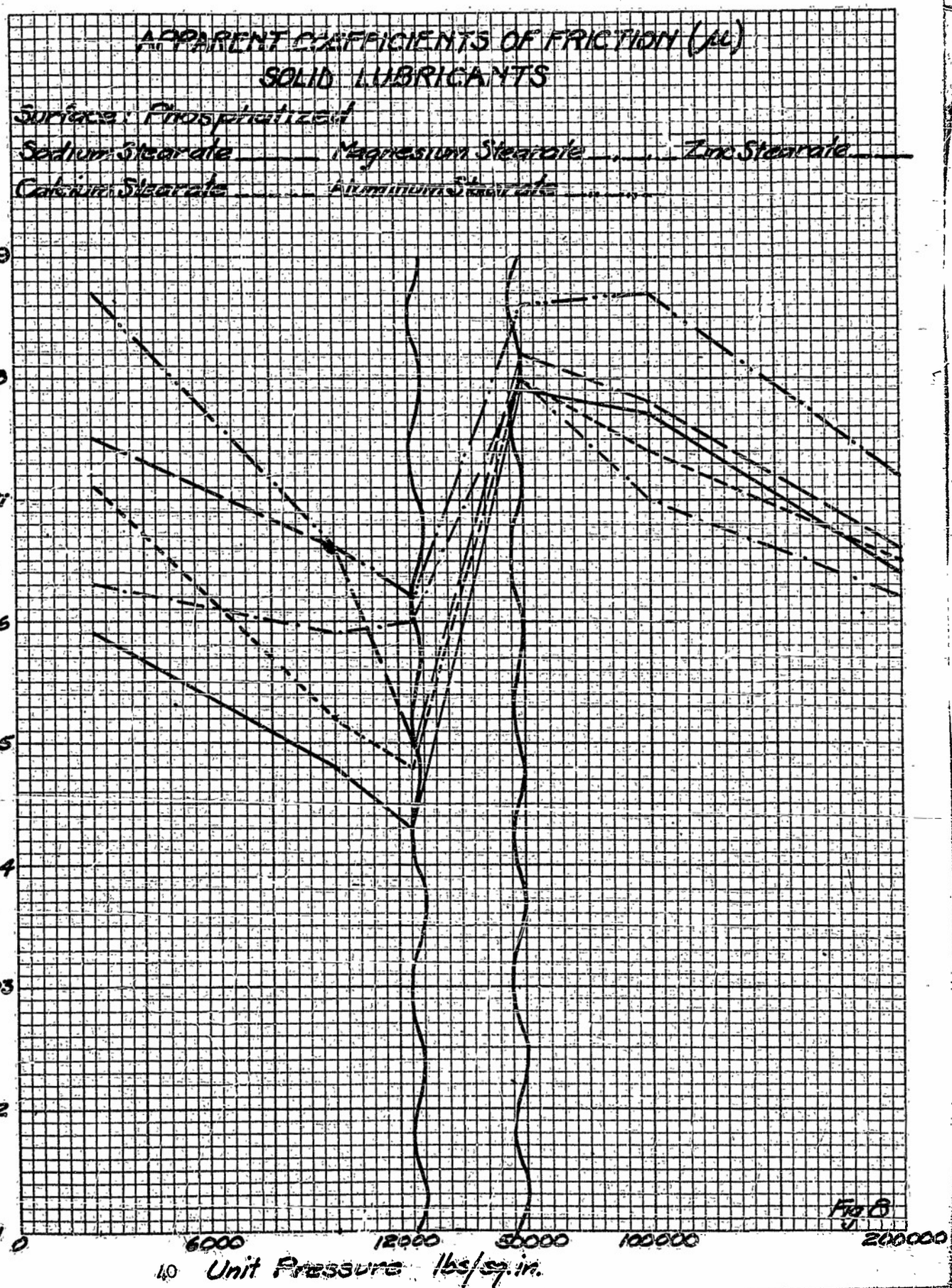

Degree of Hydrolysis & pH Value of Sodium Stearate Solutions
80
60
40
20
Degree of Hydrolysis (%)
50°C
30°C
10°C
pH at 30°C
10.5
10.0
9.5
9.0
pH Value
0.25%
0.5%
1%
2%
Concentration of Sodium Stearate

Fig. 7


APPARENT COEFFICIENTS OF FRICTION (μ)
SOLID LUBRICANTS
Surface: Phosphatized
Sodium Stearate
Magnesium Stearate
Zinc Stearate
Calcium Stearate
Aluminum Stearate
0.09
0.08
0.07
μ
0.06
0.05
0.04
0.03
0.02
0.01
0
6000
12000
50000
100000
200000
Unit Pressure lbs/sq.in.
Fig 8

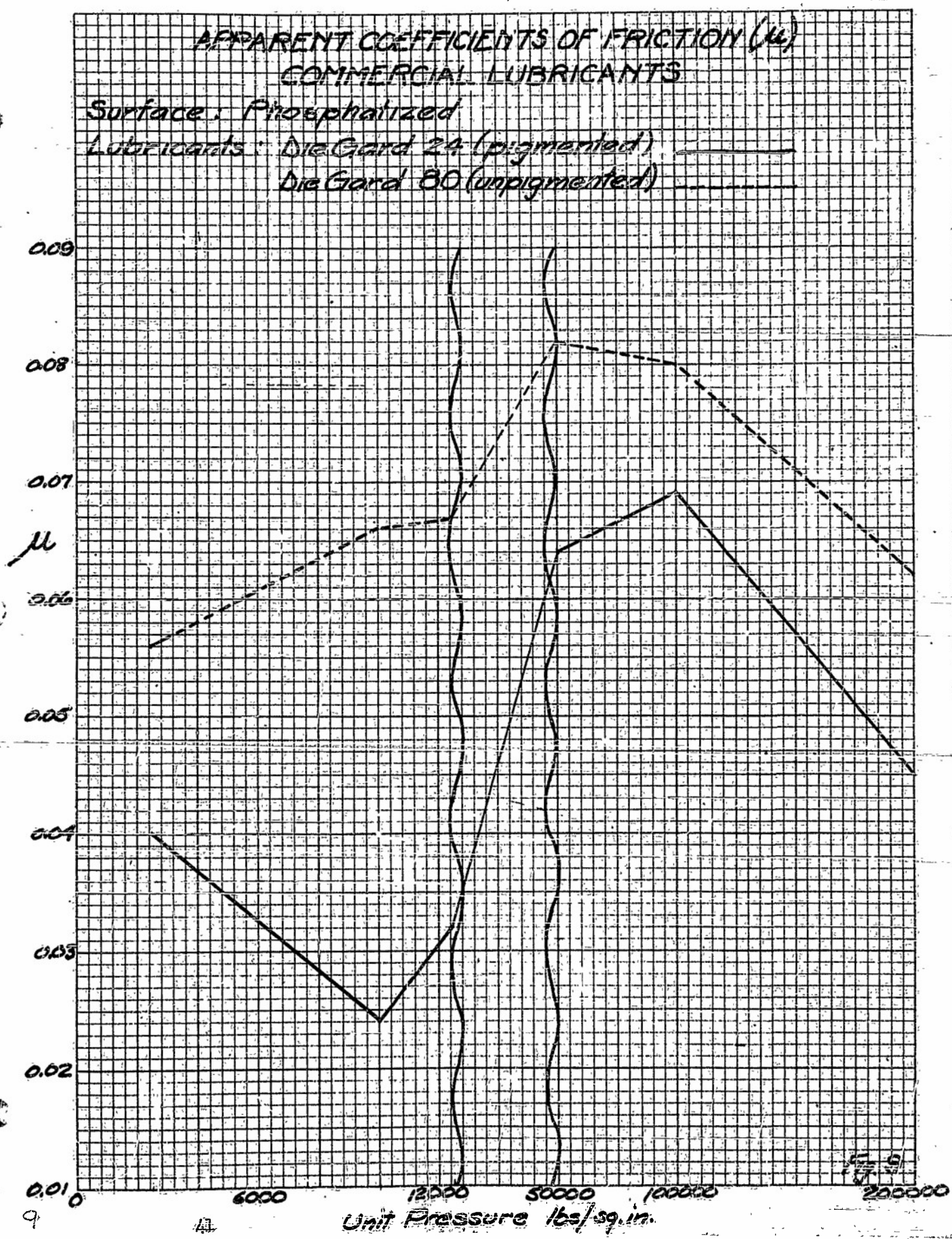

APPARENT COEFFICIENTS OF FRICTION (μ)
COMMERCIAL LUBRICANTS
Surface: Phosphatized
Lubricants: Die Gard 24 (pigmented)
Die Gard 80 (unpigmented)
0.09
0.08
0.07
μ
0.06
0.05
0.04
0.03
0.02
0.01
0
6000
12000
50000
100000
200000
Unit Pressure lbs/sq.in.

Fig. 9

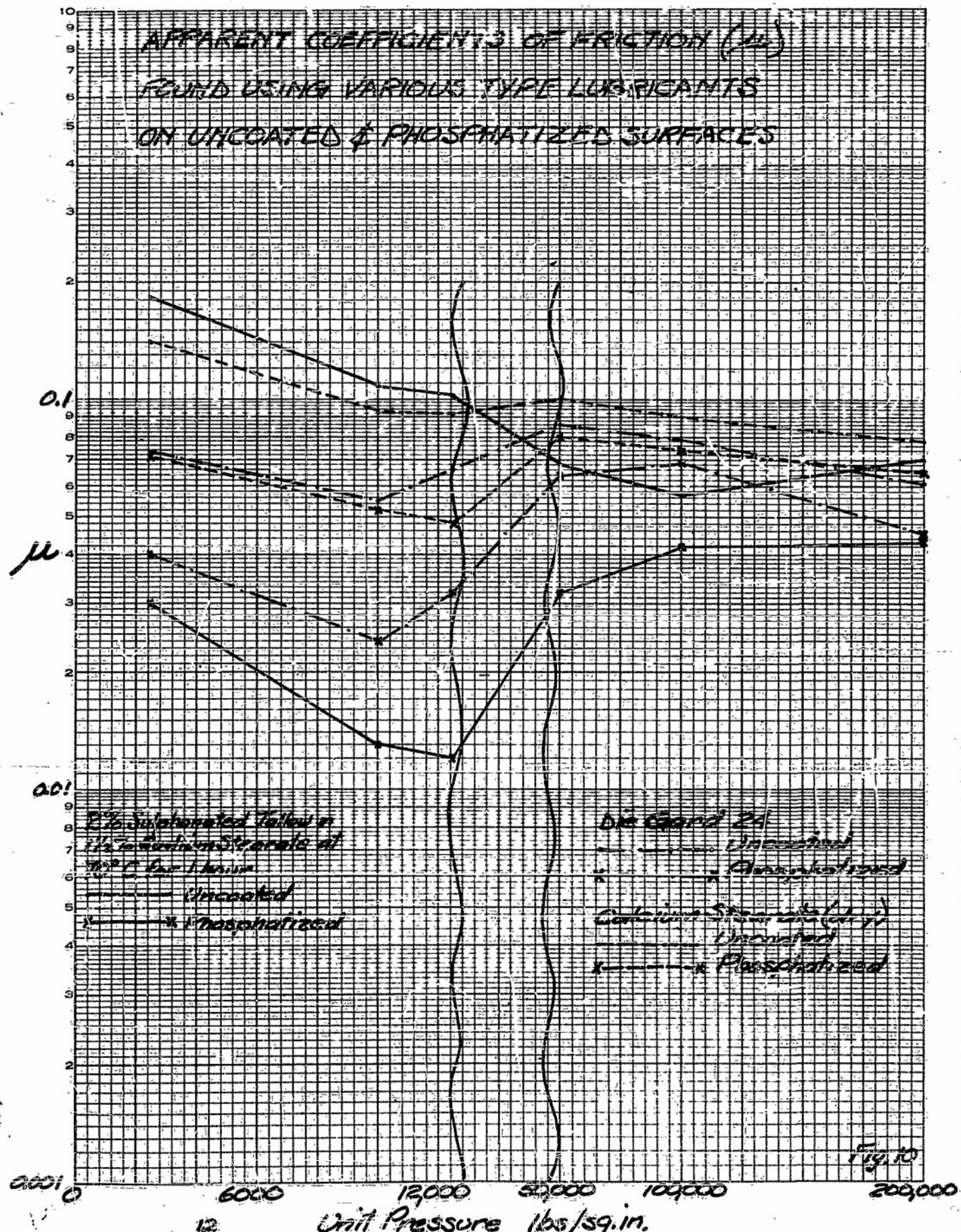

APPARENT COEFFICIENTS OF FRICTION (μ)
FOUND USING VARIOUS TYPE LUBRICANTS
ON UNCOATED & PHOSPHATIZED SURFACES
μ
0.1
0.01
0.001
0
6000
12,000
100,000
200,000
Unit Pressure lbs/sq.in.
2% Sulphonated Tallow in
Uncoated
Phosphatized
Die Grand 24
Uncoated
Phosphatized
Uncoated
Phosphatized

Fig. 10

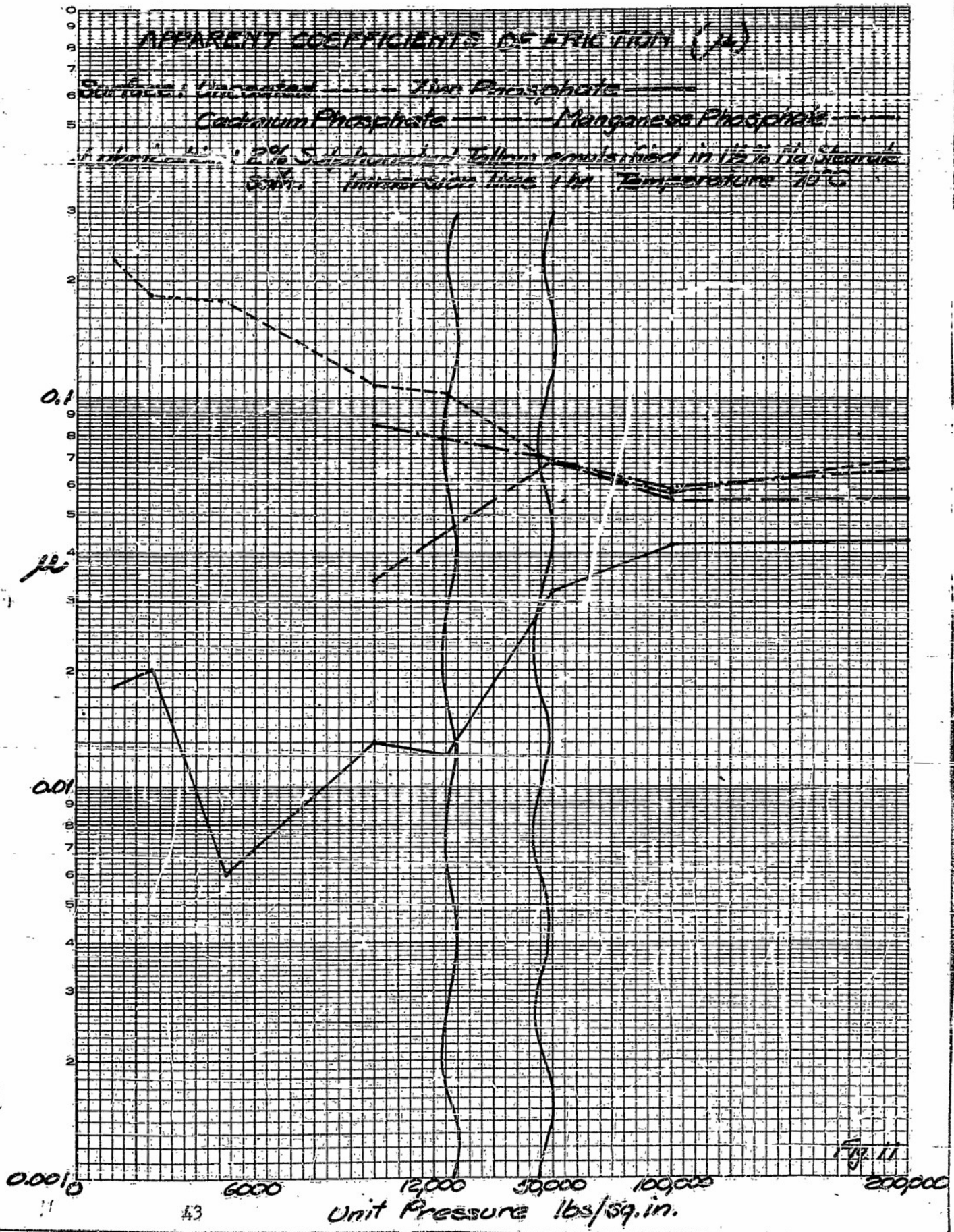

APPARENT COEFFICIENTS OF FRICTION (μ)
Surfaces: Lubricated ------ Zinc Phosphate ------
Cadmium Phosphate ------ Manganese Phosphate ------
Lubricant: 1% of Sulphurized Tallow emulsified in 12% Naphtha Soap.
Time of Test 1 hr. Temperature 70°C.
0.1
μ
0.01
0.001
0
6000
12,000
50,000
100,000
200,000
Unit Pressure lbs/sq.in.

Fig. 11

LOAD REQUIRED & REDUCTION OBTAINED IN DRAWING SAE 1010 USING VARIOUS TYPE LUBRICANTS

Surface: Phosphatized

Unit Pressure: 200,000 psi

Lubricants:

A - Emulsified Lard Oil containing active agent

B - Emulsified Tall Oil Ester

C - Calcium Stearate

D - Sodium Stearate

E - Die Gard 2A

F - Die Gard 80

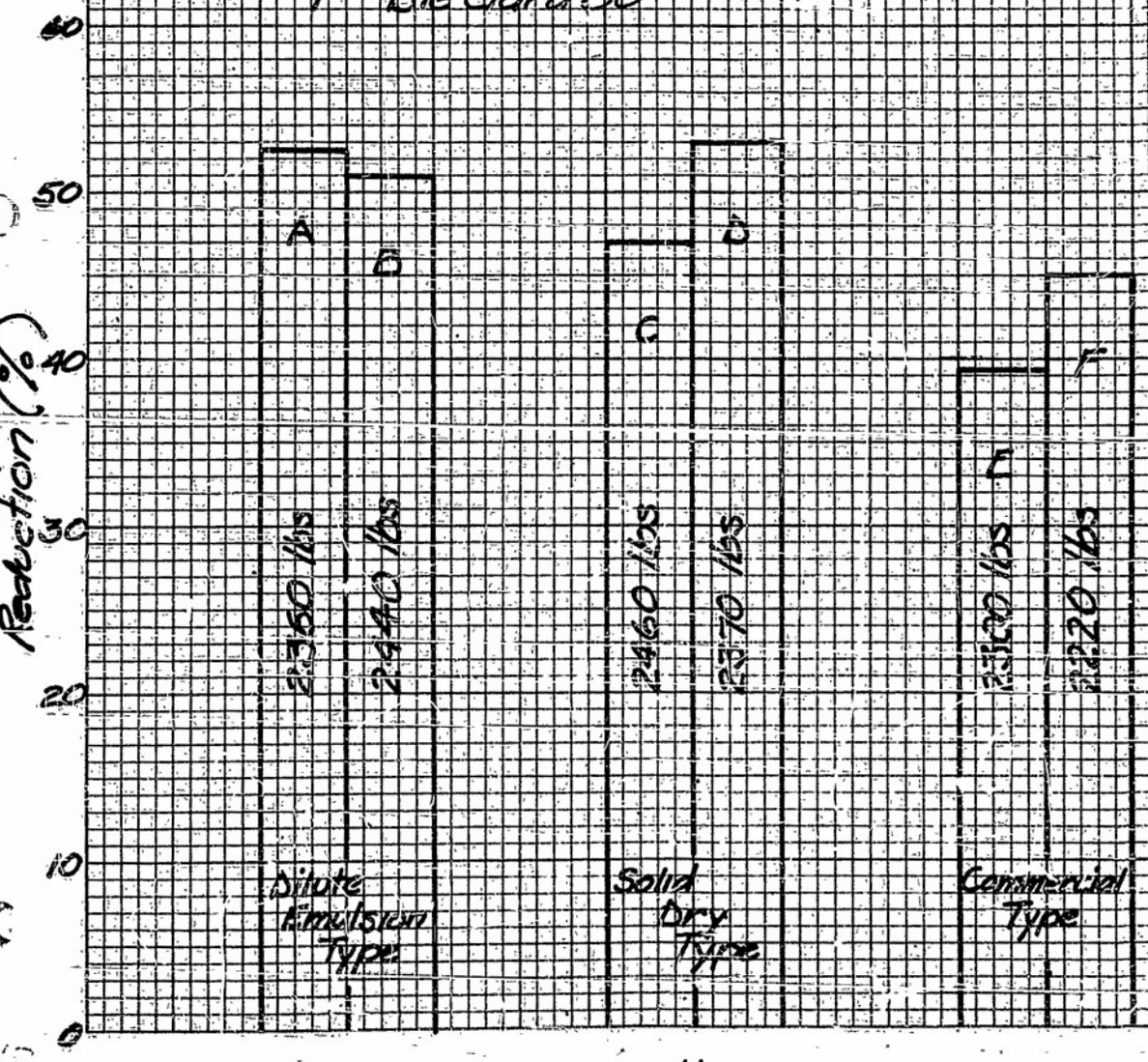


Fig 18

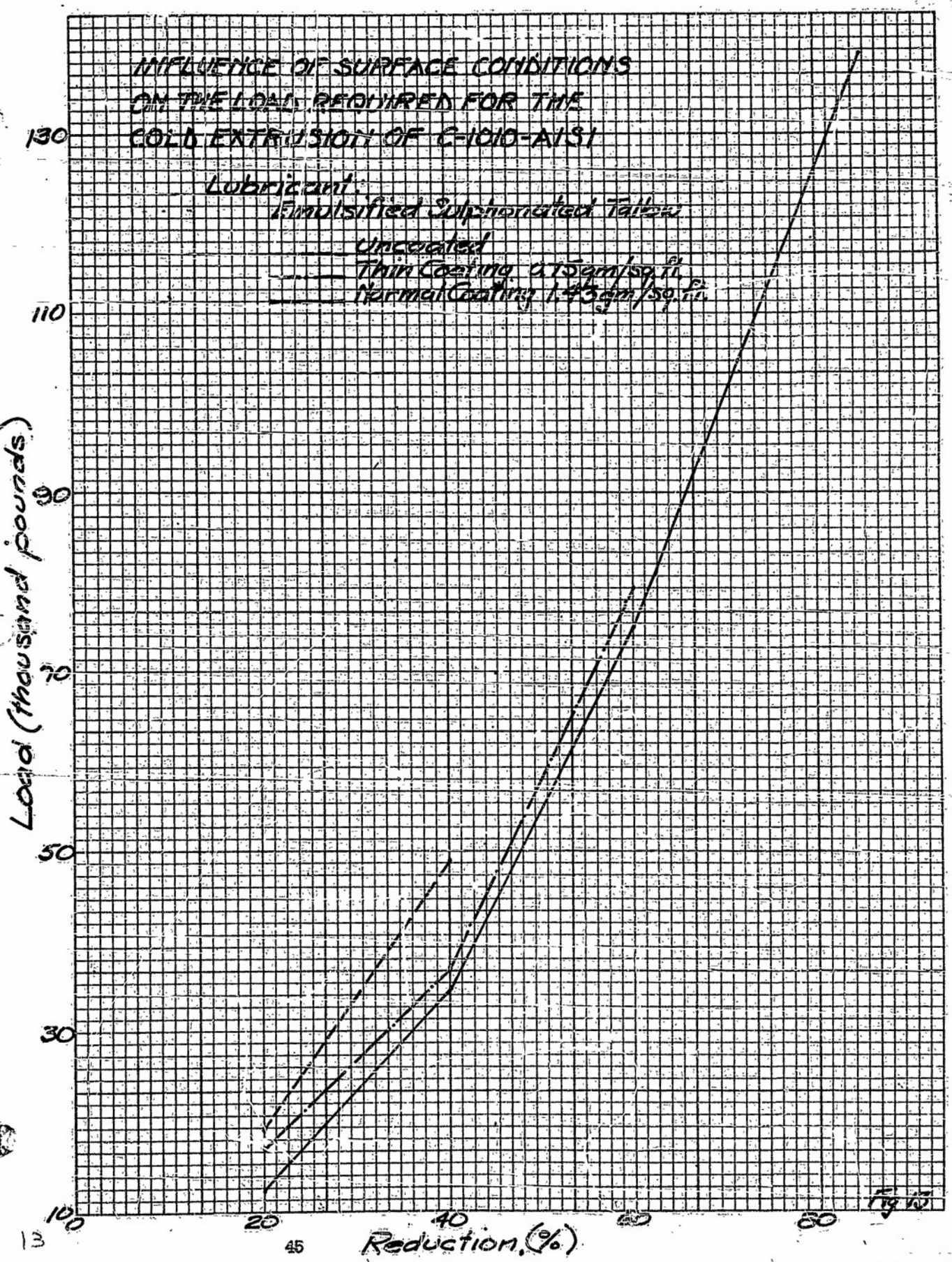
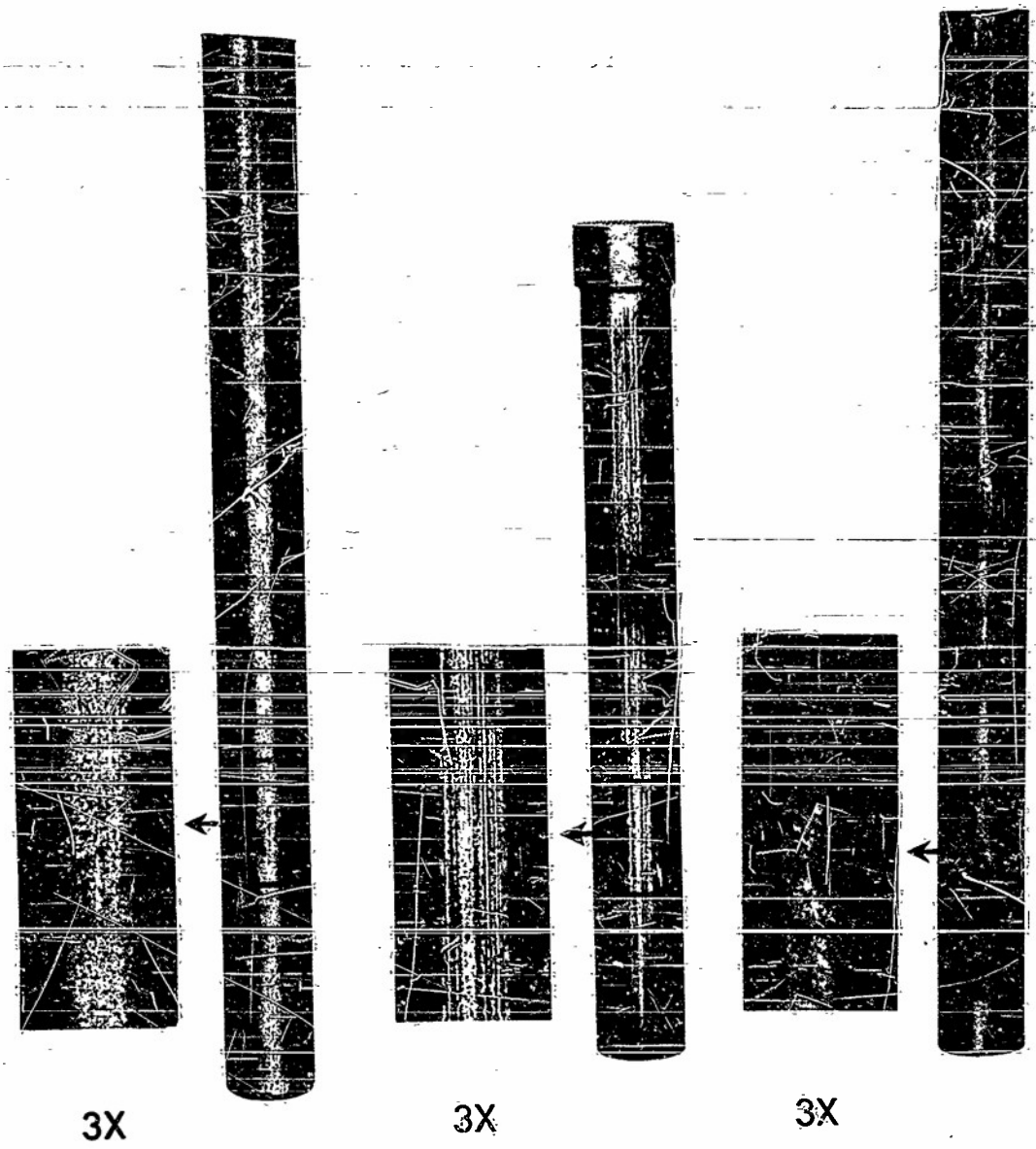

INFLUENCE OF SURFACE CONDITIONS
ON THE LOAD REQUIRED FOR THE
COLD EXTRUSION OF C-1010-AISI
Lubricant:
Emulsified Sulphonated Tallow
Uncoated
Thin Coating 0.15 gm/sq.ft.
Normal Coating 1.43 gm/sq.ft.
Load (thousand pounds)
130
110
90
70
50
30
10
0
20
40
60
80
Reduction (%)

Fig 10



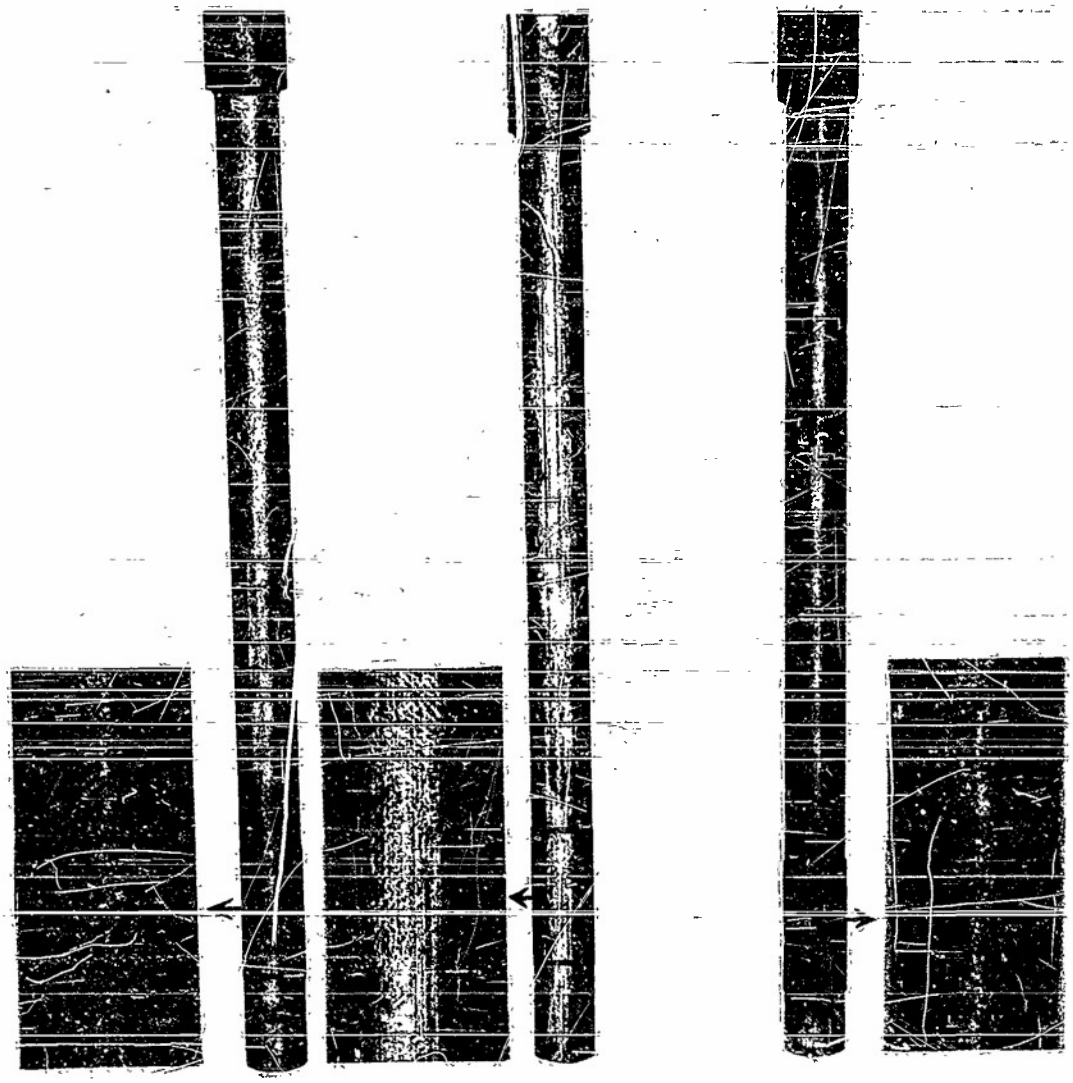
20% REDUCTION

THIN COATING

UNCOATED

NORMAL COATING

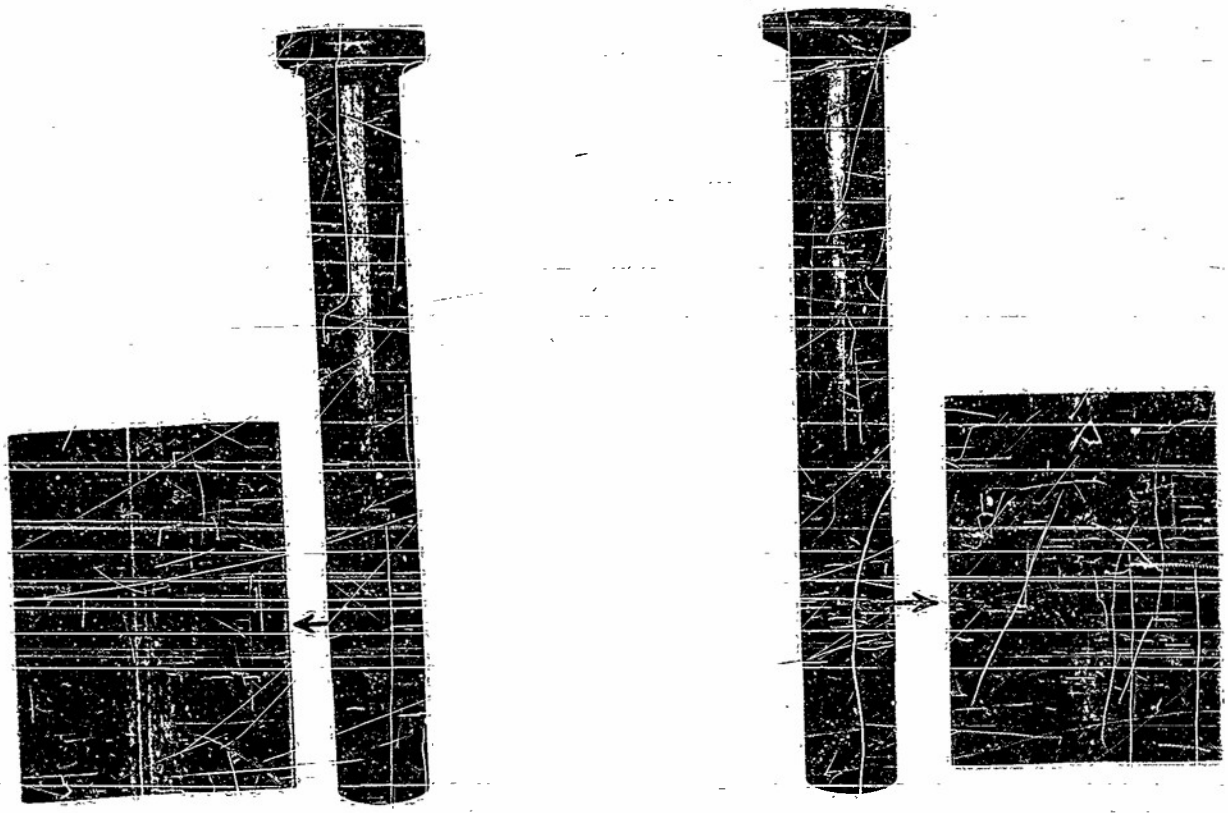
1



3X 40% REDUCTION 3X

THIN COATING UNCOATED NORMAL COATING

2



3X

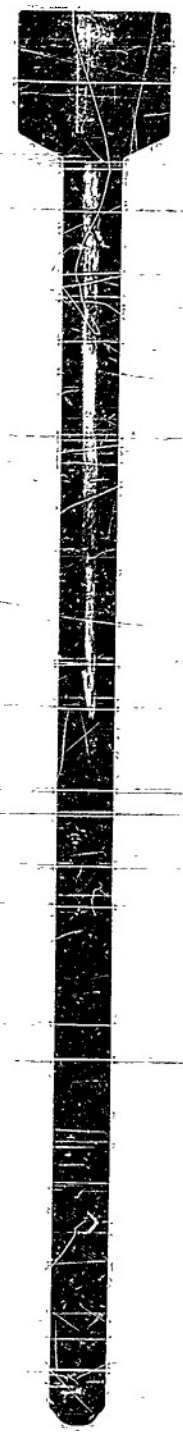
3X

60% REDUCTION

THIN COATING

NORMAL COATING

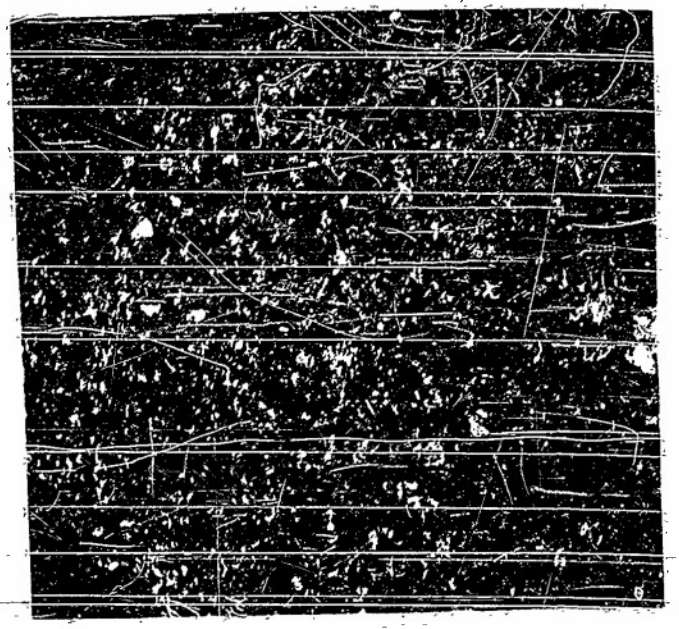
3



85% REDUCTION
NORMAL
COATING

UNDEFORMED PHOSPHATE
COATING

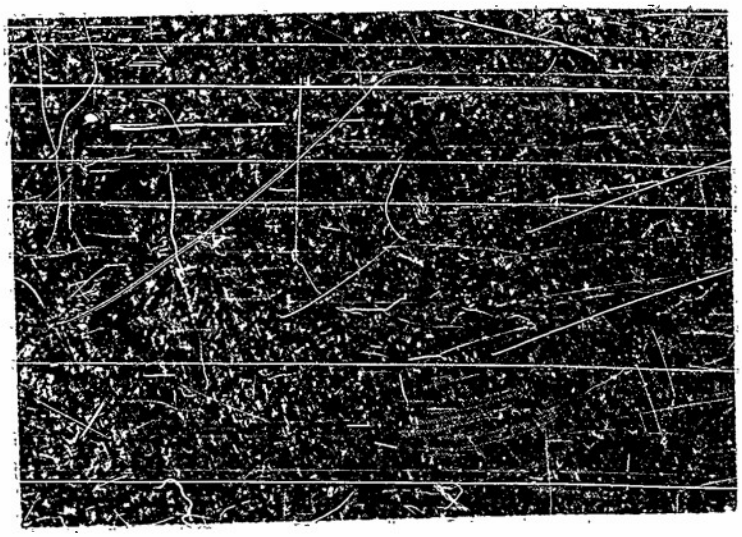
100 X



MICROPHOTOGRAH 1

DEFORMED PHOSPHATE
COATING

100 X



MICROPHOTOGRAPH 2

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