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16. Abstract

The thinning characteristics of five fluids were studied by measuring film thickness as a function of time. The mercury squeeze film capacitance technique was used. All tests were performed at room temperature. The synthetic hydrocarbon plus a nematic liquid crystal, N-(p-methoxybenzylidene)-p-butylaniline, thinned according to a Newtonian model and retained its bulk viscosity. The synthetic hydrocarbon plus a phosphonate antiwear additive and the synthetic hydrocarbon plus n-hexadecanol produced residual thick films. The synthetic hydrocarbon base fluid and the synthetic hydrocarbon plus a paraffinic resin displayed viscosity increases during thinning, but no residual films were formed.

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FILM THICKNESS MEASUREMENTS ON FIVE FLUID FORMULATIONS BY THE MERCURY SQUEEZE FILM CAPACITANCE TECHNIQUE

by William R. Jones, Jr., Robert L. Johnson, I. Hyslop,* and R. Day*

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SUMMARY

The thinning characteristics of a synthetic hydrocarbon base fluid and four additive formulations were studied by measuring film thickness as a function of time. The mercury squeeze film capacitance technique was used. The formulations included the synthetic hydrocarbon base fluid plus a proprietary organic phosphonate antiwear additive (formulation I), plus 5.0 weight percent superrefined paraffinic resin (formulation II), plus 0.5 weight percent n-hexadecanol (formulation III), and plus 0.1 weight percent N-(p-methoxybenzylidene)-p-butylaniline (formulation IV). All measurements were performed at room temperature (22^{o} to 25^{o} C).

A fluid containing a nematic liquid crystal (formulation IV) thinned according to a Newtonian theoretical model. This formulation retained its bulk viscosity down to a film thickness of 2×10^{-9} meter (200 Å). All other fluids either produced residual thick films (formulations I and III) or showed increases in thin film viscosity during thinning (synthetic hydrocarbon base fluid and formulation II).

The synthetic hydrocarbon plus a phosphonate antiwear additive (formulation I) and the synthetic hydrocarbon plus 0.5 weight percent n-hexadecanol (formulation III) produced residual thick films of about 30×10^{-8} and 9×10^{-8} meter (300 and 900 Å), respectively. The synthetic hydrocarbon base fluid and the synthetic hydrocarbon plus 5.0 weight percent superrefined paraffinic resin (formulation II) displayed viscosity increases during thinning but did not form residual thick films.

A qualitative correlation with literature results was observed when thinning results for the synthetic hydrocarbon base fluid were compared with those for a chemically similar but more viscous synthetic paraffin. No correlation was observed when the results for the synthetic hydrocarbon plus a phosphonate antiwear additive were compared with those for the same additive in the more viscous synthetic paraffin.

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INTRODUCTION

Boundary lubrication refers to the condition where contacting solid surfaces are lubricated by a liquid. The friction and wear characteristics are determined by the interactions between the solids and between the solids and the liquid. The bulk viscosity of the liquid lubricant is generally not considered to play an important role in boundary lubrication (ref. 1).

Boundary lubrication is extremely important because many machine elements operate in this regime. Nevertheless, very little is known about the properties of the boundary lubricating fluids near solid surfaces. There are predominantly two schools of thought about this phenomenon. A number of investigators believe that fluids exhibit abnormally high viscosity or a degree of rigidity near solid surfaces. These investigators include Drauglis et al. (refs. 2 and 3); Allen et al. (ref. 4); Askwith, Cameron, and Crouch (ref. 5); and Fuks (ref. 6). Further, earlier work by Deryaguin et al. (ref. 7) and Needs (ref. 8) supports this view. Others, such as Hayward and Isdale (ref. 9), refute this view and are supported by the earlier work of Bulkley (ref. 10) and Bastow and Bowden (refs. 11 and 12). Allen and Drauglis (ref. 13) have published a comprehensive review of the literature (to 1969) concerning this controversial topic.

Smith and Cameron (refs. 14 and 15) contend that most of the rigid films reported in the literature were due to chemical reactions and that clean pure fluids will not produce thick films. Similar results are reported by Stejskal and Cameron (ref. 16). However, upon examining the thinning behavior of a number of pure fluids, they found strong evidence for viscosity changes near solid surfaces even though a rigid film was not formed.

The objective of this investigation was to study the thinning characteristics of a synthetic hydrocarbon base fluid and four additive formulations by measuring film thickness as a function of time through the mercury squeeze film capacitance technique. Experiments were performed at room temperature (22° to 25° C).

MERCURY SQUEEZE FILM APPARATUS

The mercury squeeze film apparatus which determined film thickness by capacitance measurement is shown in figure 1. It consists of a glass slide onto which a rectangular area of chromium has been vacuum deposited. The chromium area acts as one plate of a capacitor. The contact strip and the edges of the chromium capacitor plate are covered with a 1- to 2-micrometer layer of vacuum-deposited glass. The additional capacitance of the glass covered chromium areas is negligible. The contact lead is attached to the slide by being soldered to a copper layer sputtered onto the end of the chromium contact strip. The movable glass slide is positioned in a glass test tube which contains triply distilled mercury and the test fluid.

ELECTRICAL SYSTEM

The electrical system utilized for these experiments is shown schematically in figure 1. The capacitance of the mercury - oil film - chromium surface is measured in order to calculate the thickness of the test fluid film, which acts as a dielectric of the capacitor. A schematic of the mercury - oil film and oil film - chromium interfaces appears in figure 2. An autobalance bridge is used in making these measurements. This bridge provides a peak output voltage of 40 millivolts, which is sufficient to cause dielectric breakdown of very thin oil films; thus the capability of the instrument to measure thin films is limited. Therefore, the voltage applied to the oil film is reduced by inserting an attenuator between the output of the bridge and the capacitance cell and an amplifier between the cell and the input terminals of the bridge. This system is calibrated to give capacitance measurements with an accuracy of ±2 percent. A more detailed description of this system appears in reference 16.

CLEANING PROCEDURE

The glass test tube is cleaned in chromic acid. It is then thoroughly rinsed, first with distilled water and second with acetone. The glass slide is cleaned by ultrasonic agitation for 3 minutes in a solution of a phosphate-free surface active agent and deionized distilled water. It is then rinsed thoroughly in tap water and distilled water. The slide is then placed in an emulsifier solution containing tetrachlorotetrafluoroethane, agitated for 10 minutes, and rinsed by the procedure just described. After 5 minutes of agitation in distilled water, the slide is rinsed several times in acetone and finally submitted to a further 10 minutes of agitation in more acetone. The slide is then dried and placed in the cell for immediate use.

The fluids were tested in the as-received condition since the normal cleaning procedure of percolation through silica gel would have removed the additives.

EXPERIMENTAL PROCEDURE

The glass test tube is partially filled with triply distilled mercury. The slide is lowered into the tube until the chromium surface is completely covered with mercury.

The bridge circuit is connected, and the capacitance and conductance are measured. This step is a test for faults in the electrical connections, as the capacitance should be zero, and indicates the maximum conductance possible in the circuit for comparison with later readings during the experiment.

The slide is raised until the chromium surface is above the mercury. Then the test fluid is introduced in sufficient quantity to cover the chromium surface. The slide remains in this position for a "growing period," which is 30 minutes for all fluids. The slide, with its adherent oil film, is again lowered into the mercury. Simultaneously a clock is started, and the bridge circuit is switched on. The capacitance of the oil film is then recorded as a function of time.

CALCULATION OF OIL FILM THICKNESS

The thickness of the oil film on the chromium surface is calculated from the formula

$$h = \frac{A_c K \epsilon_0}{C} \tag{1}$$

where h is the film thickness, A_c is the area of capacitor surface, K is the dielectric constant of the oil film, ϵ_0 is the permittivity of free space, and C is the capacitance registered on the bridge. (All symbols are defined in the appendix.) The value of ϵ_0 is constant, A_c can be measured, and K is determined by using a cylindrical capacitor of known separation. A bulk dielectric constant of 2.1 was used for all fluids.

EXPERIMENTAL FLUIDS

The base fluid for these studies is a synthetic hydrocarbon whose fully formulated version meets the hydraulic fluid specification MIL-H-83282. Four additive formulations were prepared from this base fluid (which contains no additives itself):

(1) Formulation I, the base fluid plus a proprietary organic phosphonate antiwear additive. This additive has been studied extensively by a number of investigators in a more viscous version of the synthetic hydrocarbon base fluid. These investigations have included boundary lubrication studies (ref. 17), elastohydrodynamic film thickness measurements (ref. 18), traction measurements (refs. 19 and 20), studies of pressure and viscosity characteristics (ref. 21), bearing studies (refs. 22 to 25), and thin film rheology measurements (ref. 2).

(2) Formulation II, the base fluid plus 5.0 weight percent of a superrefined paraffinic resin. This additive has also been previously studied. These investigations have included boundary lubrication studies (refs. 26 and 27), studies of pressure and viscosity characteristics (ref. 21), bearing studies (refs. 24, 25, and 28), and wettability studies (refs. 29 and 30).

(3) Formulation III, the base fluid plus 0.5 weight percent of a nonreactive polar compound, n-hexadecanol. The n-hexadecanol was of reagent grade purity.

(4) Formulation IV, the base fluid plus 0.1 weight percent of a nematic liquid crystal, N-(p-methoxybenzylidene)-p-butylaniline. This compound was also of reagent grade purity.

Some physical properties of the test fluids appear in table I. The additives in formulations I and IV did not appreciably alter the bulk properties of the base fluid and their properties are, therefore, reported together. A summary of the additive contents of the test fluids appears in table II.

The variations in additive concentrations are the result of two factors. In formulations I and II the values reflect those in common usage, whereas the concentrations in formulations III and IV were dictated solely by solubility considerations.

RESULTS AND DISCUSSION

A theoretical model of the thinning behavior of a Newtonian fluid under squeeze film conditions has previously been developed (ref. 16). This model allows one to compare the experimental and theoretical thinning curves and determine if any unusual rheological phenomena are taking place. Simplified theory predicts the following:

$$h \propto \left(\frac{\eta_0}{t}\right)^{1/2} \tag{2}$$

where h is the fluid film thickness, η_0 is the actual fluid kinematic viscosity, and t is the time since thinning started. As can be seen, a plot of $\log h$ as a function of $\log t$ will yield a straight line with a slope of -1/2. Therefore, all experimental data will be presented in this manner so that they may be compared with the theoretical relation. A computed iterative technique was used to solve a more rigorous equation in order to calculate the theoretical thinning curves. The computed solution bears out the relation among film thickness, viscosity, and time predicted by the simplified theory (eq. (2)).

If the experimental data show a decrease in the rate of thinning compared with the theoretical curve, an increase in the thin film viscosity is indicated. If the experimental curve eventually forms a plateau, a "thick" residual film has been formed.

The determination of the film thickness from equation (1) requires a knowledge of the thin film dielectric constant during thinning. Since this value as a function of film thickness and time is difficult to measure, one normally assumes a constant K based on the bulk oil value. This appears to be a good assumption when dealing with pure clean fluids (ref. 14). However, in this study, where surface active additives and impurities were present, variations in the dielectric constant of the thin films could be expected. Therefore, the possibility exists that deviations from the ideal thinning curves may have been the result of unknown changes in the dielectric constant. However, pure fluids percolated through silica gel have shown enhanced viscosity during thinning. In addition, the formation of thick films has been observed in similar experiments, and their presence has been confirmed by electron microscopy (ref. 15). Therefore, it is believed that the thinning phenomena observed in this study were real rather than artificial effects. Obviously, there is need for further work, wherein the in situ film thickness is measured by an independent method such as interferometry.

Synthetic Hydrocarbon Base Fluid

The thinning behavior as well as the theoretical curve for the base fluid appear in figure 3. For the first 20 minutes of thinning, the experimental and theoretical curves are almost identical. After 20 minutes the rate of thinning decreases, indicating an increase in film viscosity below 80×10^{-8} meter (8000 Å). After 100 minutes, the film suddenly becomes conducting with a conductance almost as high as that of the dry mercury-chromium contact (dielectric breakdown). Several repeat tests confirmed this behavior.

Thinning behavior for n-hexadecane (a chemically similar but less viscous hydrocarbon) has been previously reported (ref. 16). In these tests n-hexadecane which had been cleaned by percolation through silica gel thinned ideally and became conducting after about 100 minutes. Therefore, the small viscosity change observed with the synthetic hydrocarbon base fluid was apparently due to impurities. These impurities could have been fluid oxidation or decomposition products.

Formulation I

The thinning behavior for formulation I (synthetic hydrocarbon plus phosphonate antiwear additive) also appears in figure 3. Since the additive did not appreciably affect the bulk viscosity characteristics, the theoretical curve for this formulation is identical to that for the base fluid.

The inclusion of the antiwear additive in the base fluid prevented dielectric breakdown and also caused an apparent decrease in film thickness. However, since the additive did not affect the bulk viscosity, this apparent change must have been due to a large increase in the dielectric constant of the thin film. One may make a crude estimate of the thickness of this residual film by superimposing the experimental curve on the theoretical curve as shown in figure 4. This indicates that a thick residual film of approximately 30×10^{-8} meter (3000 Å) was formed. This film had a relatively high conductivity, which showed that it was highly ordered. These results indicated that the antiwear additive was preferentially adsorbed onto the chromium surface and reacted with it to produce the residual film.

Drauglis et al. (ref. 2) have published mercury squeeze film data on two similar fluids. They reported data on a synthetic paraffin which is a more viscous version of the synthetic hydrocarbon used in these studies. They also studied this fluid formulated with the same antiwear additive as in our formulation I. Their results are replotted in the $\log h$ against $\log t$ format in figure 5. Both base fluids displayed viscosity increases during thinning, and both exhibited dielectric breakdown in the film thickness range 50×10^{-8} to 200×10^{-8} meter (5000 to 20 000 Å). However, the synthetic paraffin plus the antiwear additive did not cause a large change in the dielectric constant, and no residual thick film was formed with this formulation. The authors concluded that the antiwear additive was not very surface active. The reason for this lack of correlation was not known.

It is interesting to note that the phosphonate antiwear additive used in formulation I has produced anomalous results in elastohydrodynamic (EHD) studies. Parker and Kannel (ref. 18) found that this additive caused an increase in the EHD film thickness when compared with the nonadditive base fluid. They attributed this effect to surface film formations which either directly or indirectly influenced the rheological character of the lubricant within the contact region. In studies by Trachman and Cheng (refs. 19 and 20) this antiwear additive altered the traction characteristics of the formulated fluid compared with the base fluid.

Formulation II

The thinning behavior for formulation II (synthetic hydrocarbon plus 5.0 wt. % paraffinic resin) appears in figure 6. Since this additive altered the bulk viscosity characteristics of the base fluid, a slightly different theoretical curve resulted.

The paraffinic resin preferentially physically adsorbed at the surface and prevented electrical breakdown. As thinning progressed, the surface concentration of the resin increased and therefore the film viscosity also increased.

This paraffinic resin has exhibited antiwear activity in four ball tests (ref. 27) and in vane pump tests (ref. 26). The resin has also provided improved bearing operation in high-temperature tests (refs. 24 and 28) and improved wetting characteristics in mist lubrication studies (refs. 29 and 30).

Formulation III

The thinning behavior for formulation III (synthetic hydrocarbon plus 0.5 wt. % n-hexadecanol) appears in figure 7. The initial thinning rate for this fluid was close to the theoretical prediction. After about 50 minutes, the increases were indicated by a decreased thinning rate. Finally, a residual thick film of about 9×10^{-8} meter (900 Å) was formed. This additive prevented electrical breakdown. No dielectric or conductivity increases were observed during the thinning process. One would not expect n-hexadecanol, which is a nonreactive polar molecule, to form a residual thick film. Therefore, the residual thick film formed with this formulation was probably due, in part, to acidic impurities in the base fluid.

Formulation IV

The thinning behavior for formulation IV (synthetic hydrocarbon plus 0.1 wt. % N-(p-methoxybenzylidene)-p-butylaniline) appears in figure 8. This additive functions as a nematic liquid crystal in the temperature range 10° to 245° C. A schematic representation of the nematic structure is shown in figure 9.

The additive apparently formed a strongly adsorbed monolayer and prevented dielectric breakdown. However, this formulation thinned in a Newtonian fashion and did not produce a residual thick film. It retained its bulk viscosity down to a thickness of 2×10^{-8} meter (200 Å). These results were somewhat surprising since Allen and Drauglis (ref. 13) have proposed that many boundary films are probably due to an ordered structure that behaves like a liquid crystal.

CONCLUDING REMARKS

In conclusion, the mercury squeeze film capacitance technique appears to be a sensitive tool for studying the adsorption characteristics of boundary lubricating films. It may be able to provide information which could help elucidate mechanisms in boundary lubrication and thus lead to improved boundary additives.

SUMMARY OF RESULTS

The thinning characteristics of a synthetic hydrocarbon base fluid and four additive formulations were studied by measuring film thickness as a function of time. The mercury squeeze film capacitance technique was used. All tests were performed at room temperature (22° to 25° C). The major results were the following:

1. The synthetic hydrocarbon plus a nematic liquid crystal, N-(p-methoxybenzylidene)-p-butylaniline, thinned according to a Newtonian theoretical model and retained its bulk viscosity down to a film thickness of 2×10^{-8} meter (200 Å).

2. The synthetic hydrocarbon plus a phosphonate antiwear additive and the synthetic hydrocarbon plus n-hexadecanol produced residual thick films of about 30×10^{-8} and 9×10^{-8} meter (3000 and 900 Å), respectively.

3. The synthetic hydrocarbon base fluid and the synthetic hydrocarbon plus a paraffinic resin displayed viscosity increases during thinning but did not produce residual thick films.

4. A qualitative correlation with literature results was observed when thinning results for the synthetic hydrocarbon base fluid were compared with those for a chemically similar but more viscous synthetic paraffin. No correlation was observed when the results for the synthetic hydrocarbon plus a phosphonate antiwear additive were compared with those for the same additive in the more viscous synthetic paraffin.

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National Aeronautics and Space Administration,
Cleveland, Ohio, November 21, 1975,
505-04.

APPENDIX - SYMBOLS

A_c	area of capacitor surface, m^2
C	capacitance across oil film, F
h	oil film thickness, m
K	dielectric constant of oil film, dimensionless
t	time, min
ϵ_0	permittivity of free space, 8.87×10^{-12} $C^2/(N)(m^2)$
η_0	kinematic viscosity of oil, $(m^2)(sec^{-1})$

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TABLE I. - PROPERTIES OF EXPERIMENTAL FLUIDS

Fluid	Temperature, °C						Pour point, °C	Flash point, °C	Fire point, °C	Autogenous ignition temperature, °C	Temperature, °C	
	23		38		99						38	149
	Kinetic viscosity										Density, kg/m^3 (g/ml)	
	m^2/sec	cS	m^2/sec	cS	m^2/sec	cS						
Base fluid and formulations I and IV	2.87×10^{-5}	28.7	1.66×10^{-5}	16.6	3.8×10^{-6}	3.8	-54	221	249	371	0.806	0.734
Formulation II	3.65×10^{-5}	36.5	2.1×10^{-5}	21.0	--------	---	-54	221	249	371	.806	.734
Formulation III	2.81×10^{-5}	28.1	1.63×10^{-5}	16.3	--------	---	-54	221	249	371	.806	.734

TABLE II. - ADDITIVE CONTENTS OF EXPERIMENTAL FLUIDS

Fluid	Additive
Synthetic hydrocarbon base fluid	None
Formulation I	Proprietary organic phosphonate antiwear additive
Formulation II	Superrefined paraffinic resin (5.0 wt. %)
Formulation III	n-hexadecanol (0.5 wt. %)
Formulation IV	N-(p-methoxybenzylidene)-p-butylaniline (0.1 wt. %)

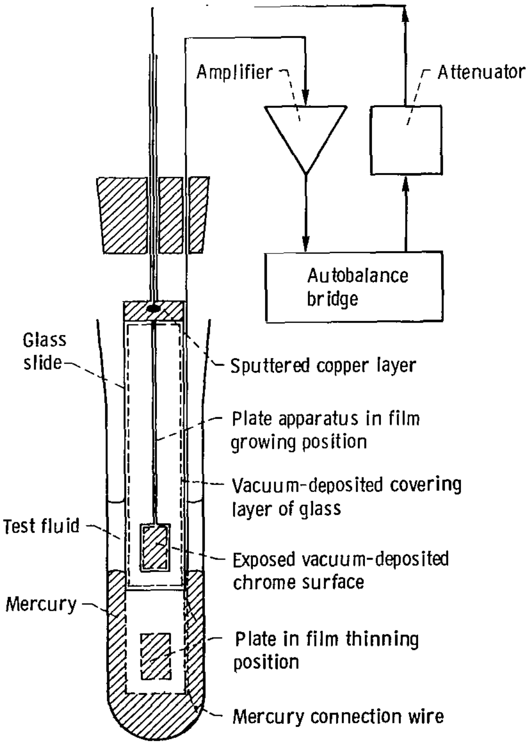

Figure 1. - Mercury squeeze film apparatus.

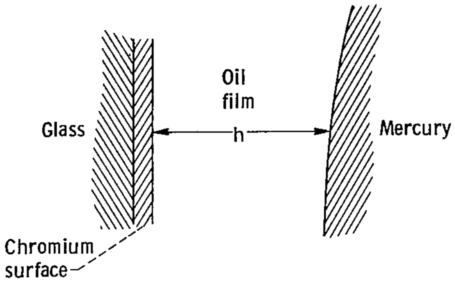

Figure 2. - Schematic of mercury - oil film and oil film - chromium interfaces.

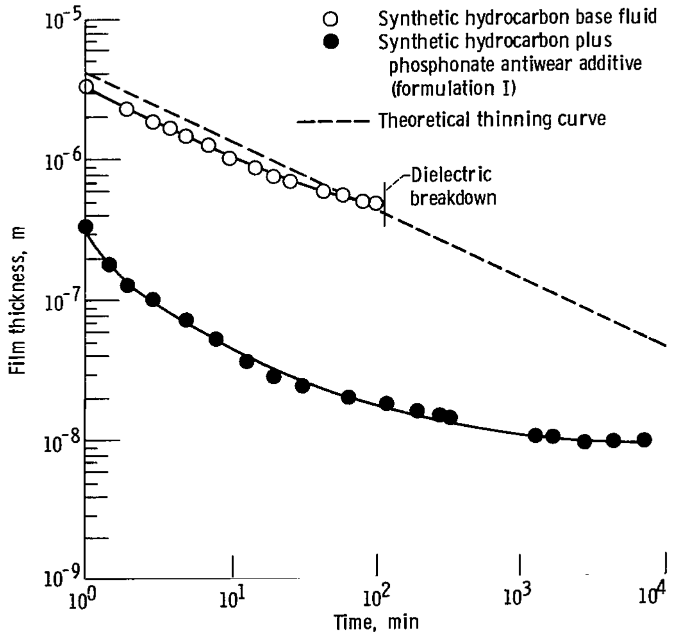

Figure 3. - Film thickness as function of time for synthetic hydrocarbon without phosphonate antiwear additive and with additive (formulation I).

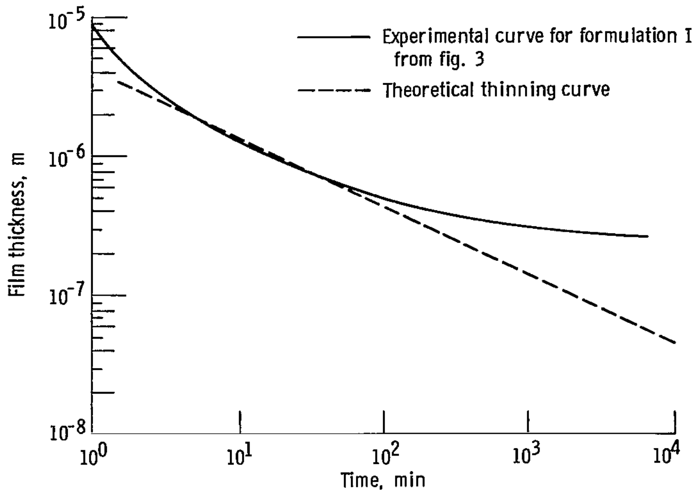

Figure 4. - Estimate of residual film thickness made by superimposing experimental curve for formulation I from figure 3 on theoretical curve.

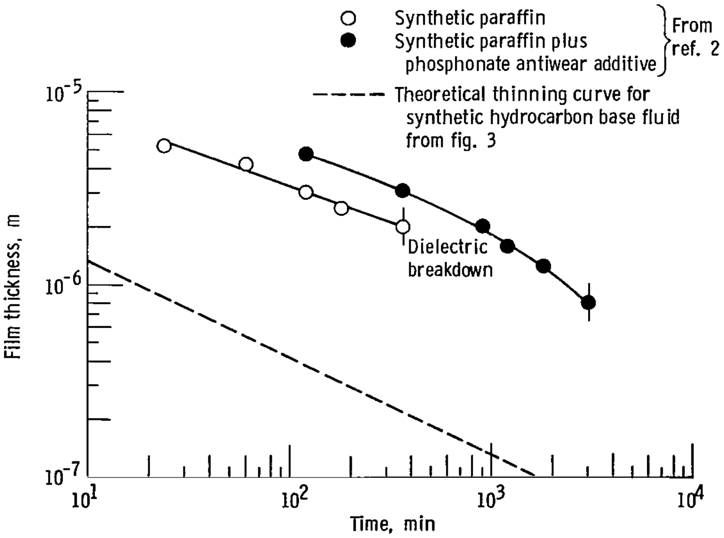

Figure 5. - Film thickness as function of time for synthetic paraffin with and without phosphonate antiwear additive (reploted from ref. 2). Test temperature, 38° C.

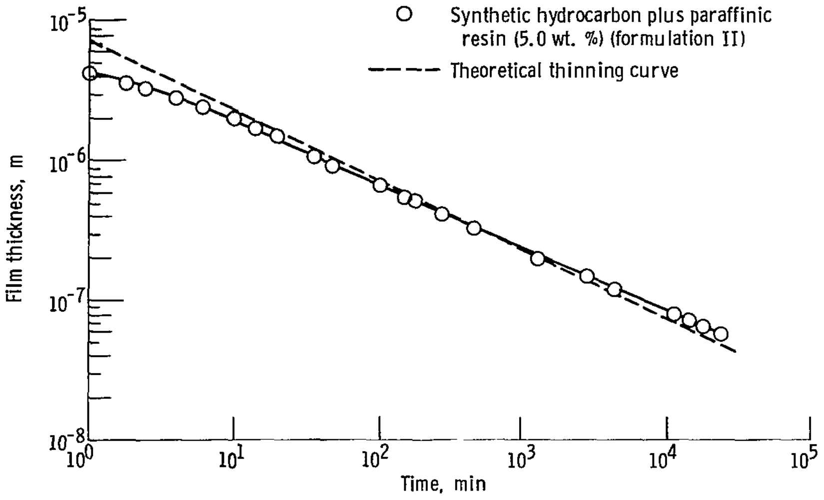

Figure 6. - Film thickness as function of time for synthetic hydrocarbon plus 5.0 weight percent paraffinic resin (formulation II).

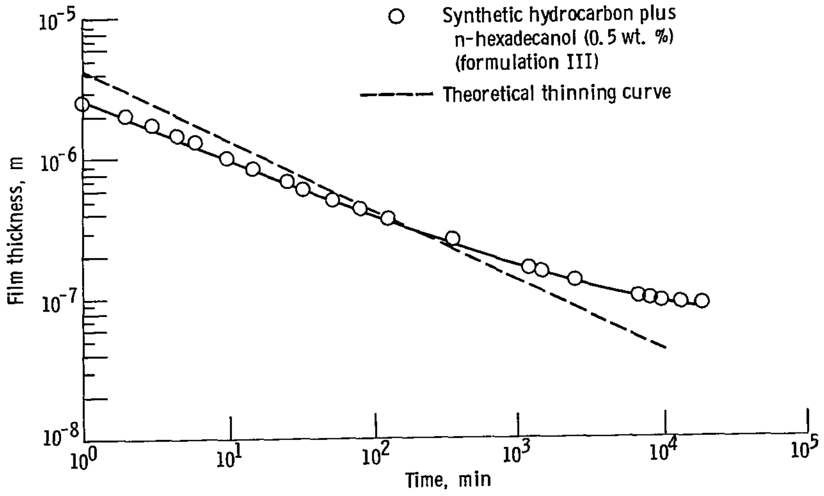

Figure 7. - Film thickness as function of time for synthetic hydrocarbon plus 0.5 weight percent n-hexadecanol (formulation III).

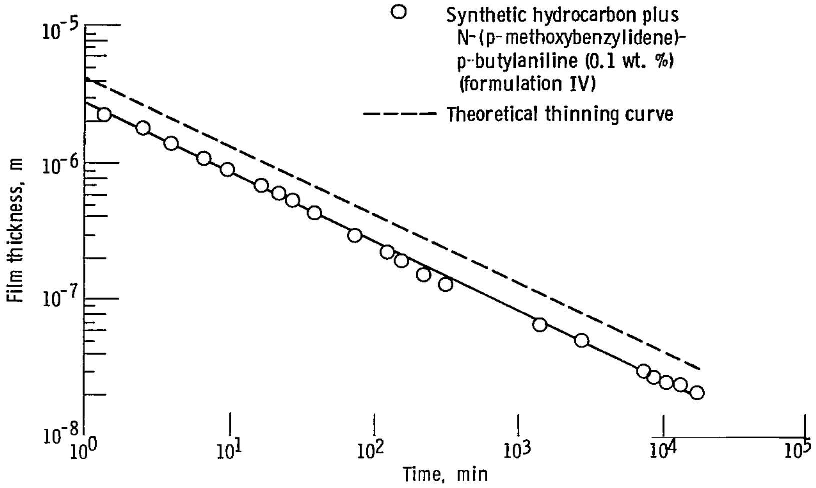

Figure 8. - Film thickness as function of time for synthetic hydrocarbon plus 0.1 weight percent N-(p-methoxybenzylidene)-p-butylaniline (formulation IV).

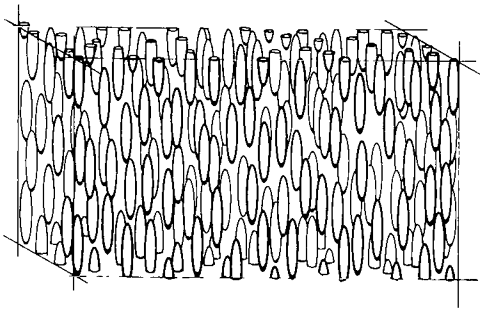

Figure 9. - Schematic representation of nematic liquid crystal.

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