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SURFACE-TENSION MEASUREMENTS IN AIR OF LIQUID LUBRICANTS TO 200° C BY THE DIFFERENTIAL-MAXIMUM-BUBBLE-PRESSURE TECHNIQUE

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16. Abstract

A differential-maximum-bubble-pressure apparatus was used to determine the surface tension of liquid lubricants to 200° C in air. These lubricants included polyphenyl ethers, mineral oils, a synthetic paraffin, a silicone, and a polyether. A polyphenyl ether exhibited the highest surface tension (46.0×10^{-5} N/cm, 46.0 dynes/cm, at 23° C to 30.2×10^{-5} N/cm, 30.2 dynes/cm, at 200° C). A polyether exhibited the lowest surface tension (17.3×10^{-5} N/cm, 17.3 dynes/cm, at 23° C to 8.2×10^{-5} N/cm, 8.2 dynes/cm, at 200° C). The role of surface tension in heat transfer (cooling) and the effect of surface tension on lubricant starvation in elastohydrodynamic contacts are discussed.

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SUMMARY

A differential-maximum-bubble-pressure apparatus was used to determine the surface tension of liquid lubricants from 23° to 200° C in air. The temperature coefficients of surface tension ($-d\gamma_{LV}/dT$) and surface tension values at 23° C of the test lubricants are as follows:

Test lubricant	Temperature coefficient of surface tension, $-d\gamma_{LV}/dt$		Surface tension at 23° C	
	N/(cm/°C)	dynes/(cm/°C)	N/cm	dynes/cm
Five-ring polyphenyl ether	0.0883×10^{-5}	0.0883	46.0×10^{-5}	46.0
Four-ring polyphenyl ether	.0876	.0876	44.8	44.8
Modified polyphenyl ether (C-ether)	.0876	.0876	44.8	44.8
Glycol derivative	.0556	.0556	32.0	32.0
Synthetic paraffinic oil	.0640	.0640	30.3	30.3
Superrefined naphthenic mineral oil	.0640	.0640	30.3	30.3
Superrefined paraffinic mineral oil	.0770	.0770	29.8	29.8
Fluorosilicone	.0634	.0634	25.0	25.0
Fluorinated polyether	.0533	.0533	17.3	17.3

Provided a fluid wets the surface, its surface tension influences transport and distribution of lubricant and, hence, its effect on heat transfer and lubricant starvation can be important.

INTRODUCTION

Surface tension is one of the fundamental fluid properties. The unit of surface tension, 10^{-5} newton per centimeter (1 dyne/cm), is equivalent to 10^{-7} joule per square

centimeter (1 erg/cm^2). Therefore, surface tension is a direct measure of surface interfacial energy (ref. 1). The surface energy of a fluid relative to adjacent phases influences the transport and distribution of fluid. Thus, surface tension is found to be an important parameter in the behavior of seals, oil spreading rates, foam stability, capillarity, fluid flow through porous solids, two-phase flow, mist lubrication, wettability of low-energy solids, two-phase heat transfer, and lubricant starvation (refs. 2 to 8). In practice, these operations often take place over a range of temperatures. It is important, therefore, to know the surface tension of the fluids used and how it varies with temperature.

Numerous methods for the measurement of liquid surface tension are available. These include the sessile drop (ref. 2), capillary pull (ref. 2), capillary rise (ref. 9), drop weight (ref. 10), surface rupture (ref. 2), and maximum bubble pressure (refs. 11 to 13).

Each method has certain limitations, but for high-temperature liquid surface-tension measurements, the maximum-bubble-pressure technique appears to be the most advantageous. Some of these advantages are

(1) The bubble need not be observed under the liquid surface.

(2) All fluid is submerged in the constant-temperature bath, and thus temperature control is facilitated.

(3) The formation of fresh surfaces reduces the possibility of impurities collecting at the surface.

(4) The method is readily adapted for measurements over a broad temperature range.

(5) The bubble apparatus containing a given pair of capillary tubes can be calibrated with a standard fluid (e.g., water or benzene), so that it is not necessary to determine the exact tube radii.

Perhaps the most important advantage of the bubble-pressure technique lies in the fact that the method can often be assumed to be independent of the solid-liquid contact angle. Most other methods are quite dependent on the contact angle. The angle must be either assumed zero or measured, which is usually quite difficult. The assumption of complete wetting (0° contact angle) is the most common procedure, and this assumption is a good approximation for the great majority of fluids in contact with glass, provided the apparatus is properly cleaned.

However, the polyphenyl ether class of lubricants has exhibited wettability problems in some instances (refs. 14 and 15). These fluids exhibit finite contact angles on many clean metals and nonmetals because of their autophobic properties (ref. 4). An assumption of a 0° contact angle with these fluids could lead to serious errors in surface-tension measurements with most methods.

Adam (ref. 16) states that if the contact angle is not greater than 90°, the assumption of contact-angle independence is valid for the bubble-pressure technique. If

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the contact angle is greater than 90°, the liquid recedes to the outer edge of the capil-
lary tube and invalidates the measurements. Contact angles of many autophobic liquids
were measured by Fox, Hare, and Zisman (ref. 4) and found to be much less than 90°.
Therefore, it is assumed that surface tensions of totally and partially wetting fluids may
be measured by the bubble-pressure technique with comparative accuracy.

Several methods are available for predicting surface tensions at elevated tempera-
tures (ref. 17), but they generally require a knowledge of other fluid properties (critical
temperature, refractive index, and vapor density) and are most applicable for pure com-
pounds.

The objective of this study was to determine the surface tension of liquid lubricants
to 200° C in air by using the differential-maximum-bubble-pressure technique. The
test lubricants were (1) a five-ring polyphenyl ether, (2) a four-ring polyphenyl ether,
(3) a modified polyphenyl ether (C-ether), (4) a glycol derivative, (5) a synthetic paraf-
finic oil, (6) a superrefined naphthenic mineral oil, (7) a superrefined paraffinic mineral
oil, (8) a modified fluorosilicone, and (9) a fluorinated polyether.

BACKGROUND

Effect of Surface Tension on Heat Transfer

Turbine engines of advanced high Mach number aircraft will be required to operate
at ever increasing temperatures. Therefore, lubricating systems will be subjected to
higher heat inputs, and the cooling capacity of the lubricant will become increasingly
important.

Lubricant properties (thermal conductivity, specific heat, specific gravity, and
kinematic viscosity) and lubricant flow rate are important in determining heat-transfer
(cooling) rates. Surface tension, by its effect on spreadability, may also influence the
cooling capacity.

Poor wettability has been recognized as a factor in reducing heat transfer involving
phase changes for many years (ref. 6). In boiling water experiments Jakob and Linke
(ref. 18) and Insinger and Bliss (ref. 19) reported increases in heat-transfer coefficients
(at constant heat flux) of 23 and 20 percent, respectively, upon the addition of wetting
additives.

Two-phase heat transfer which occurs in nucleate boiling is probably also present
in the lubricating system of a turbine engine. However, in lubrication, temperatures
would be too low to produce significant vapor formation by lubricant boiling. Bubble
formation could be caused by dissolution and coalescence of entrained or dissolved gases

under the combined effects of heating and agitation occurring in the bearings, seals, and gear components.

Figure 1 illustrates the effect on bubble shape of differences in liquid surface wettability. Clearly, the totally wetted condition of figure 1(a) would be more conducive to efficient heat transfer than the nonwetting condition of figure 1(b). The completely wetted surface would have a much greater percentage of area covered by liquid than the surface in the nonwetting condition. Heat transfer through the vapor phase would be only a fraction of that obtained through the liquid. In actual practice, the intermediate condition of partial wetting (fig. 1(c)) would probably occur. Nevertheless, the previous argument would still be valid.

Lubricant surface tension should affect not only bubble formation and size but also the rate of liquid return to the void by bubble collapse or detachment.

Surface tension can also be an important factor in determining the wettability characteristics of a lubricant. In autophobic liquids or in systems with organic contamination, low-energy surface films are formed. Fluids having surface tensions greater than the surface energy of the low-energy films will not spread on these layers (ref. 4).

Examples of surface contamination which produced nonwetting situations in ball bearings which led to premature failure are reported by Freeman, Allen, and Singer (ref. 20).

Role of Surface Tension in Elastohydrodynamics

The life of highly loaded bearings is intimately related to the ability of an elastohydrodynamic (EHD) film to separate the bearing surfaces sufficiently so that asperity contact can be reduced or eliminated. A fundamental feature of the EHD problem that has been quite well established from theory and experiment (refs. 8, 21, and 22) is that film thickness is determined by the EHD pressure generated in the convergent inlet region. If there is an insufficient supply of lubricant in the inlet region, this EHD pressure generation will be inhibited; and the resulting film thickness will be diminished.

The amount of lubricant in the inlet region is determined by the location of the inlet lubricant boundary as shown in figure 2. The inlet distance S ahead of the contact region is the distance over which the EHD pressure can build up. (Symbols are defined in the appendix.) By definition

$$S = x_b - a \tag{1}$$

where x_b is the distance from the lubricant boundary to the center of the Hertzian region and a is the calculated Hertzian radius of dry contact. For point contacts (ref. 8)

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the required inlet distance to obtain a flooded condition S_f is approximated by

$$S_f = \frac{3.52 \ R h_o^{2/3}}{a^{1/3}} \tag{2}$$

Thus, as bearing speed increases (i.e., h_o becomes larger), a greater inlet distance S_f is required to obtain a flooded condition.

The inlet distance is a function of several variables, including the quantity of available lubricant, contact geometry, speed of bearing surfaces, lubricant viscosity, and surface tension. The role of surface tension can be illustrated by considering two lubricated surfaces (e.g., a ball loaded on a flat) in static contact as shown in figure 3. Figure 3(a) shows the shape of the lubricant-air interface for a hypothetical case of a zero-surface-tension fluid. A real fluid with a finite surface tension will cause a pressure differential across an interface wherever the surface is curved. The pressure differential Δp is given by

$$\Delta p = \frac{\gamma_{LV}}{r_b} \tag{3}$$

where γ_{LV} is the surface tension, and r_b is the effective radius of curvature at the lubricant boundary. To establish equilibrium, the interface will tend to approach an infinite radius of curvature, and thus form a fluid meniscus around surfaces of close proximity (see fig. 3(b)). In this way surface tension should aid the filling of an EHD inlet region. Under dynamic conditions the shape of the fluid meniscus will be affected by the distribution of lubricant on the bearing surfaces and by the pressure and flow fields around the contact region. The effectiveness of surface tension to recapture and maintain lubricant in the inlet region will, according to equation (3), depend on the curvature of the lubricant boundary and the surface tension of the lubricant.

APPARATUS

The bubble-pressure apparatus (fig. 4) consists of two parts. The lower part is a heat-resistant glass tube which acts as a reservoir for the test fluid. The upper part contains two capillary tubes and one open tube. It is sealed to the lower part by a ground-glass joint. The two capillary tubes are of the same length but different diameters, and both may be opened or closed with stopcocks. The capillary diameters are

approximately 1 and 0.02 millimeter. The open tube is connected to a 2000-cubic-centimeter flask which acts as a pressure damping device. The flask is connected to a vacuum line and a differential strain-gage pressure transducer. This transducer measures the differential pressure between the test chamber and the ambient room pressure. The bubble apparatus is placed in a constant-temperature circulator containing glycerol as the heat-transfer fluid. Temperature control in the circulator is specified to be ±0.01° C from 40° to 200° C. However, because of the high viscosity of the glycerol, temperature control is poorer from 40° to 80° C.

PROCEDURE

Experimental Procedure

The bubble apparatus is cleaned in hot chromic acid solution and rinsed with tap water and then with distilled water. Approximately 100 cubic centimeters of test fluid (1×10^{-4} m^3) are placed in the reservoir. The test fluid itself acts as a lubricant for the ground-glass joint. The apparatus is placed in the circulator, and the desired temperature is set. Then 10 to 15 minutes are allowed for temperature equilibrium to be established.

The large capillary tube is opened, and a slight vacuum is pulled in the test chamber. The pressure differential across the chamber causes air to be drawn through the capillary and results in the formation of a stream of bubbles. The pressure is regulated so that one or two bubbles a second are formed. The differential pressure rises to a maximum as the bubble is being formed and drops sharply as the bubble breaks away from the capillary tip and travels to the surface. Then the pressure differential increases again and the process is repeated. These pressure changes are recorded on an x, y-recorder. A schematic of a typical recorder trace appears in figure 5.

After several pressure spikes have been recorded for the large capillary, the small tube is opened and the large tube is closed. The procedure is repeated. The smaller capillary, of course, requires a greater pressure difference for bubble formation. The manner of bubble formation is also different. The pressure difference rises to a maximum, and then a stream of bubbles appears. This process decreases the pressure difference, and bubble formation stops. The pressure difference increases again, and the process is repeated. A recorder trace for this behavior also appears in figure 5.

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Calculation of Surface Tension

The surface tension is calculated by using the equation developed by Sudgen (ref. 12):

$$\gamma_{LV} = AP\varphi \tag{4}$$

where A is the apparatus constant, P is the pressure difference between the two capillaries ($H_1 - H_2$, from fig. 5) in newtons per square centimeter, and φ is a correction factor, which is given by the following equation:

$$\varphi = 1 + \frac{0.69\ r_2 gD}{P} \tag{5}$$

The apparatus constant is determined by using distilled water as the calibrating fluid. The constant A for the apparatus was determined by this method to be 3.32×10^{-3} centimeter. To check the accuracy of this constant, the surface tension as a function of temperature was determined for two other pure compounds, benzene and bromobenzene. Both were of reagent grade purity. The benzene and bromobenzene surface tensions were compared to literature values, and a maximum error of 1.9 percent and an average error of less than 1.0 percent were obtained.

RESULTS AND DISCUSSION

Surface-Tension Results

Table I lists the lubricants studied in this report and some of their typical properties. Surface tensions for all fluids as a function of temperature appear in figure 6. The general rule of a linearly decreasing surface tension with increasing temperature is evident. At any one temperature the order of surface tensions is as follows: five-ring polyphenyl ether > four-ring polyphenyl ether = modified polyphenyl ether (C-ether) > glycol derivative > synthetic paraffinic oil = superrefined naphthenic mineral oil > superrefined paraffinic mineral oil > fluorosilicone > fluorinated polyether. The temperature coefficient of surface tension (the slope of the curves in fig. 6) and surface tensions at five specific temperatures are summarized in table II.

Surface tensions were measured to 200° C for most fluids. Above 200° C, pressure fluctuations, caused by thermal gradients in the vapor space prevented accurate meas-

urements. Also, in previous work (ref. 15) wettability problems were encountered with a polyphenyl ether from approximately 150° to 200° C.

Surface Tension and Chemical Structure

Zisman (ref. 3) lists surface tensions at 20° C for various liquids of different chemical structure. The class with the highest surface-tension range (42×10^{-5} to 46×10^{-5} N/cm; 42 to 46 dynes/cm) is the polyaromatics. Aliphatic hydrocarbons ranged from 27×10^{-5} to 31×10^{-5} newton per centimeter (27 to 31 dynes/cm) and methyl silicones from 14×10^{-5} to 20×10^{-5} newton per centimeter (14 to 20 dynes/cm). Fluorinated compounds exhibited surface tensions less than 20×10^{-5} newton per centimeter (20 dynes/cm) and a glycol had a surface tension of 33×10^{-5} newton per centimeter (33 dynes/cm). These literature values correlate favorably with the experimental values of this report. Only the fluorosilicone appears to have a higher surface tension than one might predict.

No high-temperature surface-tension data are available to compare with the results of this report. However, a few values at 25° are available from the manufacturer or from reference 23. These values appear in the 23° C column in table II. Of those available, only the surface-tension value for the five-ring polyphenyl ether does not correlate within 2 percent of the experimental values measured in this study. The variation for the five-ring ether is 8.5 percent.

SUMMARY OF RESULTS

A differential-maximum-bubble-pressure apparatus was used to determine the surface tension of liquid lubricants to 200° C in air. Since surface tension influences the transport and distribution of lubricant, its effect on heat transfer and lubricant starvation can be important. The following results were obtained:

1. The order of measured surface tensions over the entire temperature range was five-ring polyphenyl ether > four-ring polyphenyl ether = modified polyphenyl ether (C-ether) > glycol derivative > synthetic paraffinic oil = naphthenic mineral oil > paraffinic mineral oil > fluorosilicone > fluorinated polyether.

2. The 23° C surface tensions ranged from a maximum of 46.0×10^{-5} newton per centimeter (46.0 dynes/cm) for the five-ring polyphenyl ether to a minimum of 17.3×10^{-5} newton per centimeter (17.3 dynes/cm) for the fluorinated polyether.

Lewis Research Center,
  National Aeronautics and Space Administration,
    Cleveland, Ohio, May 4, 1971,
      126-15.

APPENDIX - SYMBOLS

A	bubble-pressure apparatus constant, cm
a	Hertzian radius, cm
D	liquid density, g/cm^3
g	acceleration due to gravity, 981 cm/sec^2
H	$H_1 - H_2$, N/cm^2; in. of water
H_1	differential pressure for bubble evolution from small capillary, N/cm^2; in. of water
H_2	differential pressure for bubble evolution from large capillary, N/cm^2; in. of water
P	pressure, N/cm^2; $dynes/cm^2$
Δp	pressure differential across fluid-air interface
R	reduced radius of contact, $1/R = 1/R_1 + 1/R_2$, cm
R_1, R_2	radius of contacting bodies, cm
r_b	effective radius of curvature of lubricant boundary, cm
r_1	small capillary radius, mm
r_2	large capillary radius, mm
S	inlet distance, $x_b - a$, μm
S_f	inlet distance to obtain flooded condition, μm
u_1, u_2	velocity of bearing surfaces, cm/sec
x, y, z	coordinates
x_b	distance from inlet lubricant boundary to center of Hertzian region, μm
γ_{LV}	surface tension, N/cm; dynes/cm
φ	correction factor, $1 + 0.69 \, r_2 g D/P$, dimensionless

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TABLE I. - PROPERTIES OF TEST LUBRICANTS

[Manufacturers' data.]

Lubricant	Additive	Temperature, $^{\circ}$C		Pour point, $^{\circ}$C	Density at 25° C g/cm^3	Thermal conductivity at 38° C, J/(m)(sec)(K)	Specific heat at 38° C, J/(kg)($^{\circ}$C)
		38	99				
		Kinematic viscosity, m^2/sec (cS$\times 10^{-6}$)					
Five-ring polyphenyl ether	None	3.6×10^{-4}	1.3×10^{-5}	5	1.20	0.13	1540
Four-ring polyphenyl ether	None	6.3×10^{-5}	6.0×10^{-6}	-12	1.18	.12	1500
Modified polyphenyl ether (C-ether)	None	2.5×10^{-5}	4.1×10^{-6}	-25	1.19	.12	1450
Glycol derivative	None	1.2×10^{-4}	1.4×10^{-5}	-43	1.0	.16	1900
Synthetic paraffinic oil	None	4.47×10^{-4}	4.0×10^{-5}	-51	.84	.13	2000
Superrefined naphthenic mineral oil	Antiwear, antioxidant	7.8×10^{-5}	8.2×10^{-6}	-34	.89	.12	1900
Superrefined paraffinic mineral oil	Antiwear, antioxidant	1.5×10^{-5}	3.3×10^{-6}	-57	.85	.12	2000
Fluorosilicone	Antiwear	2.9×10^{-5}	6.2×10^{-6}	-65	1.15	(a)	(a)
Fluorinated polyether	None	8.5×10^{-5}	1.0×10^{-5}	-43	1.89	.09	1000

[a]Not available.

TABLE II. - SUMMARY OF SURFACE-TENSION RESULTS

Lubricant	Temperature coefficient of surface tension, $d\gamma_{LV}/dt$, N/(cm)(^{o}C) (dynes$\times10^{-5}$/(cm)(^{o}C))	Temperature, ^{o}C				
		23	50	100	150	200
		Surface tension, N/cm (dynes$\times10^{-5}$/cm)				
Five-ring polyphenyl ether	0.0883×10^{-5}	46.0×10^{-5} [a]49.9	43.7×10^{-5}	39.0×10^{-5}	34.7×10^{-5}	30.2×10^{-5}
Four-ring polyphenyl ether	.0876	44.8 [a]44.9	42.3	38.0	33.6	29.3
Modified polyphenyl ether (C-ether)	.0876	44.8 [b]45.3	42.3	38.0	33.6	29.3
Gylcol derivative	.0556	32.0	30.5	27.8	25.1	22.5
Synthetic paraffinic oil	.0640	30.3 [b]30.8	28.7	25.6	22.2	19.3
Superrefined naphthenic mineral oil	.0640	30.3 [b]30.9	28.7	25.6	22.2	(c)
Superrefined paraffinic mineral oil	.0770	29.8	27.2	23.4	20.0	(c)
Fluorosilicone	.0634	25.0	23.2	20.0	16.9	14.0
Fluorinated polyether	.0533	17.3 [a]17.6	15.9	13.3	10.7	8.2

[a]Manufacturer's data (25° C).
[b]Ref. 18 (25° C).
[c]Not determined.
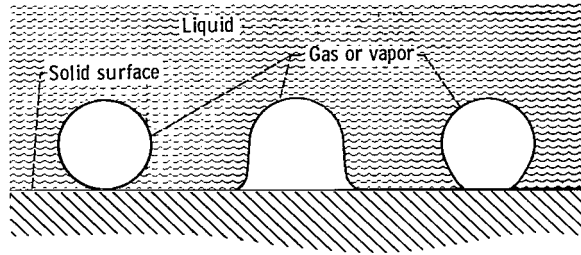
(a) Zero contact angle,   (b) High contact   (c) Low contact angle,
complete wetting.   angle, no wetting.   partial wetting.

Figure 1. – Effect of surface wettability on bubble shape.
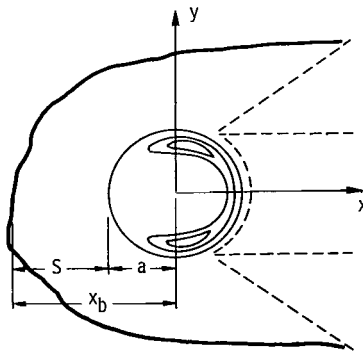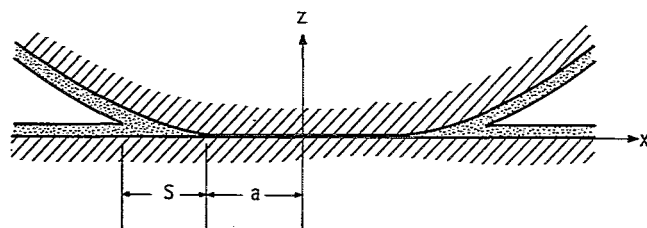

(a) Plan view showing requirements for starvation.
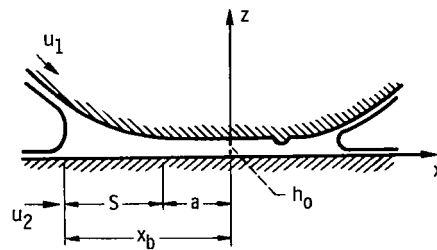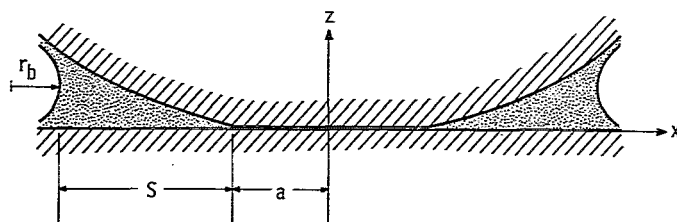

(b) Cross-sectional view along centerline.

Figure 2. – Contact geometry.

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(a) Lubricant with zero surface tension.

(b) Lubricant with finite surface tension.
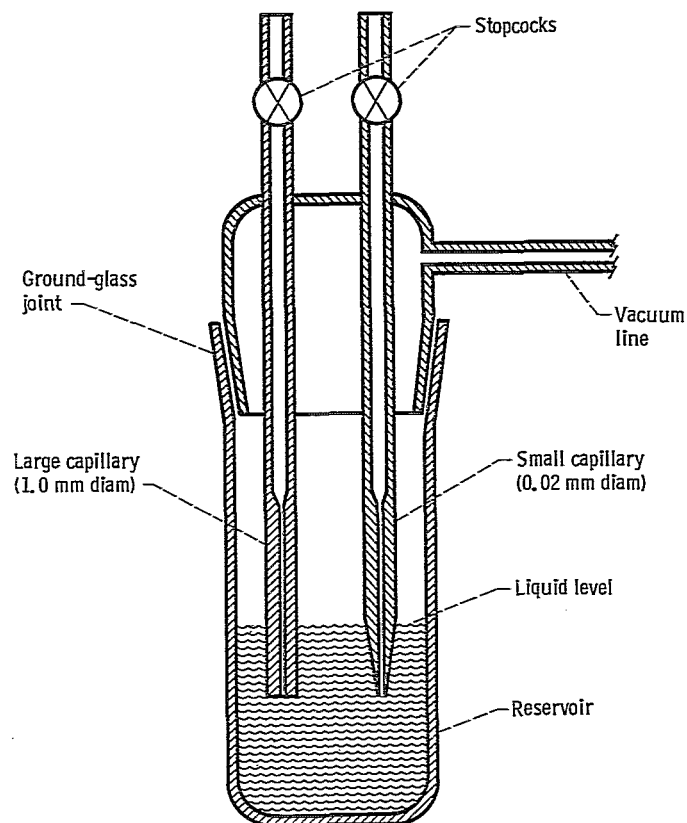
Figure 3. – Contact geometry under static conditions.
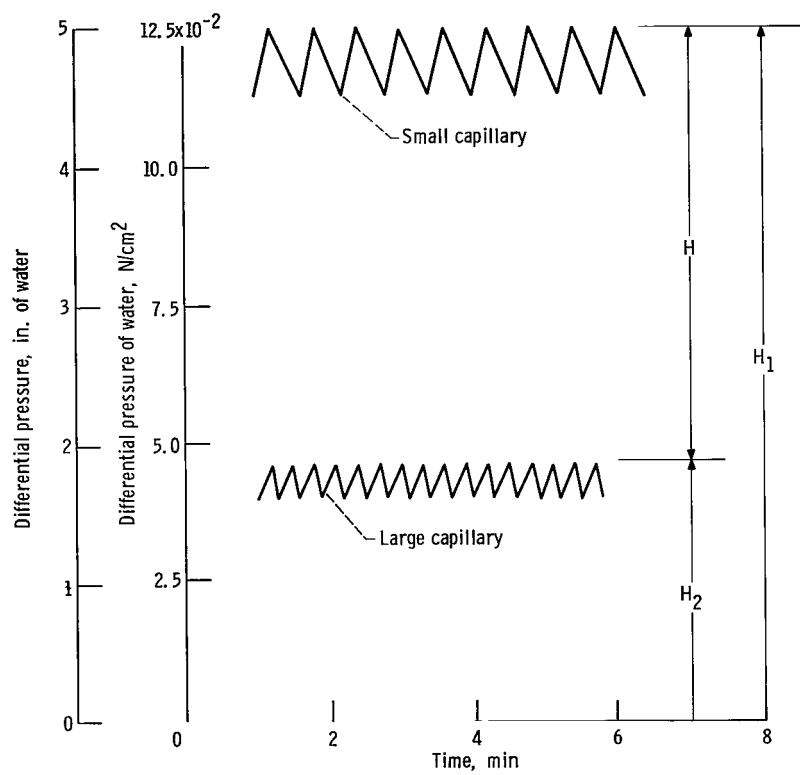

Figure 4. – Bubble-pressure apparatus.

Figure 5. – Typical differential pressure curves for large and small capillary tubes.
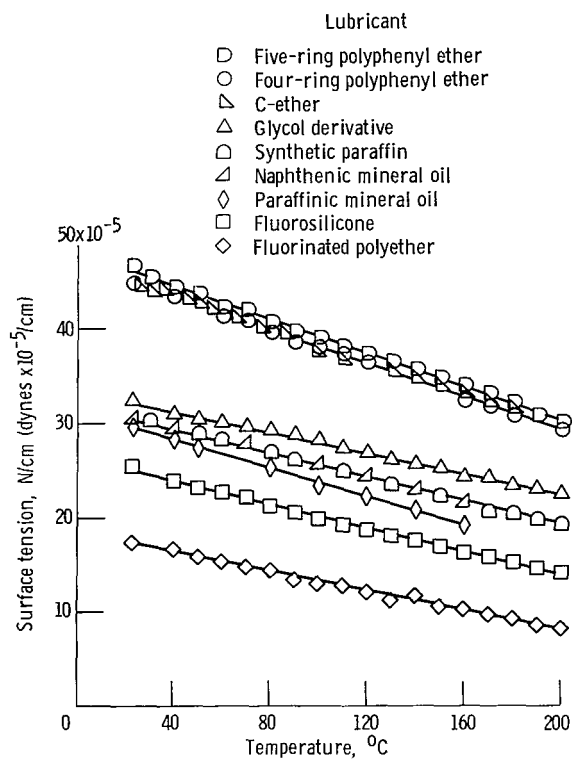
Lubricant

⬭ Five-ring polyphenyl ether
○ Four-ring polyphenyl ether
◁ C-ether
△ Glycol derivative
⌂ Synthetic paraffin
◿ Naphthenic mineral oil
◇ Paraffinic mineral oil
▢ Fluorosilicone
◇ Fluorinated polyether

Figure 6. – Surface tension as function of temperature for lubricants.

The y-axis is labeled "Surface tension, N/cm (dynes x10^{-5}/cm)" with values 50×10^{-5}, 40, 30, 20, 10. The x-axis is labeled "Temperature, $^{\circ}$C" with values 0, 40, 80, 120, 160, 200.

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