



High Temperature Lubrication via Hydrocarbon Vapors

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Industrial and Automotive Lubrication

Summary

The ability of directed streams of three representative hydrocarbon gases - acetylene C_2H_2 , ethylene C_2H_4 , and ethane C_2H_6 - to provide extended duration lubrication to high temperature sliding contacts via surface deposition of pyrolytic carbon has been demonstrated. One order- and two order-of-magnitude reductions of friction coefficient and wear rate of self-mated silicon nitride sliding contacts can be realized by this technique. The capacity of these gases to provide 'adequate' lubrication at high temperature is illustrated through a mapping of the normal load / temperature / precursor flowrate space over which reduced friction may be maintained. Acetylene was the most effective precursor for pyrolytic carbon deposition, providing adequate lubrication over the broadest range of normal load / temperature / flowrate combinations, while ethane was the least effective. The boundary of regions of adequate lubrication represent the locus of contact conditions with equal rates of lubricous carbon deposition and removal by wear. The shape of this boundary, as explored in the mapping study, supports a proposed model in which the removal rate is proportional to the product of normal load and sliding speed while the deposition rate is proportional to the product of precursor flowrate and an Arrhenius temperature dependence.

1. INTRODUCTION

At temperatures below 350°C conventional lubrication strategies have been demonstrated and employed both repeatedly and reliably. However, at more extreme temperatures the number of options to the design or lubrication engineer is significantly limited. Solid lubricants, lubricating powders, vapor phase lubrication, and gas phase lubrication greatly extend the operational range of tribological applications to well beyond the 350°C threshold otherwise posed by the thermal capabilities of commercially available fluid lubricant basestocks. The limited range of thermal stability of existing fluid lubricants, and the difficulty in replenishing predeposited solid lubricant films as they inevitably wear away during operation, leaves vapor phase lubrication as a promising methodology for numerous high temperature extended duration bearing applications. Vapor phase lubrication has been successfully applied over a wide range of materials and temperatures, and has been surveyed recently by Rao [1].

Vapor Phase Lubrication

Smith, Furey, and Kajdas [2] have successfully lubricated concentrated pin-on-disk sliding contacts of self-mated alumina through the tribopolymerization of monomers such as vinyl octadecyl ether, diallyl phthalate and lauryl methacrylate. The tribological contact experienced an initial maximum Hertzian contact pressure of 3.5 GPa, sliding speed of 25 cm/s, modest background temperature of 145°C, and a sliding distance of 500m. A continuous nitrogen gas stream with vaporized monomers directed into the contact region was initiated 10 to 15 seconds prior to the initiation of loading and sliding, presumably to generate a polymer film prior to the onset of wear. At a monomer delivery temperature of 165°C vinyl octadecyl ether was found to provide a 99% reduction in pin wear volume and a friction reduction of 38%, with coefficients of friction as low as $\mu=0.50$, as compared to tests performed in a nitrogen gas environment under an identical background temperature, normal load, and sliding speed.

Graham and Klaus had previously demonstrated the ability of tricresyl phosphate (TCP) and tributyl phosphate (TBP) vapors delivered to M50 bearing steel sliding surfaces to produce polymeric films capable of providing lubrication at higher temperatures of 370°C [3]. Hanyaloglu, Graham, et al. lubricated IN 800, IN 825, and MN 400 superalloys in reciprocating contacts with a vapor delivery of tri-aryl phosphate ester (TAP). The tribological contact conditions were an extremely modest average Hertzian contact pressure of 0.66 MPa, sliding speed of 12.7 cm/s, background temperature of 500°C, and a typical sliding distance of 450m. Friction coefficients of $\mu=0.02$ and $\mu=0.03$ were found for MN 400 and IN 800 respectively, and in both cases no wear of the base alloy was detected [4]. Profilometric analysis, however, revealed wear of the lubricous surface films formed by these vapors. It should be addressed that these solid lubricating films may be generated through reactions of vapor with the base alloy. Cyclic formation and removal of such surface films may gradually consume the contacting bodies as a chemical wear mechanism, especially if film formation is aggressive. Also, due to the modest contact pressures employed, it remains unknown whether the surface films formed are able to endure concentrated contacts more typical of bearing applications. Hanyaloglu and Graham [5] have since expanded this work to temperatures of 600°C for SiAlON/SiAlON and SiAlON/cast iron sliding interfaces.

Carbonaceous Gas Phase Lubrication

The ability to provide and continuously replenish lubricous carbon deposits through pyrolysis of directed hydrocarbon feed streams, which drastically reduce both sliding friction and wear, has been successfully demonstrated by Lauer et al. in concentrated ($p > 1$ GPa) contacts at temperatures of up to 600°C [6-9]. An example of this lubrication performance is shown in figure 1, where a one order-of-magnitude reduction in friction and a two order-of-magnitude reduction in wear volume are realized in a self-mated sliding contact of silicon nitride at 520°C. Barnick, Blanchet and Sawyer [10] subsequently demonstrated the applicability of this lubrication technique over a broad range of materials including ceramics, nickel superalloys, and steels. Pin-on-disk sliding tests of self-mated couples were performed at 520°C in an atmosphere

purged with 3 liters/minute of nitrogen, with a directed acetylene admixture supplied to the contact at 0.2 liters/minute. Friction coefficients of less than $\mu = 0.08$ were recorded for all metal alloys tested - AISI M50, 52100, 440C and 1018 steel, as well as K-Monel (500) and Hastalloy C276. Friction coefficients of less than $\mu = 0.10$ were found for all engineering ceramics tested - alumina, silicon nitride, tungsten carbide, and zirconia. As the contacting bodies are not reactants in the pyrolytic generation of surface films, this lubrication technique does not pose the a corresponding chemical wear mechanism. In addition to acetylene, the utility of ethylene, carbon monoxide/hydrogen mixtures, and even combustion exhaust gas (though to a lesser extent) in providing lubrication to high temperature sliding and rolling contacts has similarly been demonstrated [8,9,11,12].

Previous works [11,12] have shown that 'adequate' lubrication, as indicated during experiment by low friction, requires a favorable balance of the deposition rate of solid carbon compared to the removal rate of this carbon through wear. Mapping the locus of combinations of normal load, temperature, and gas flow rate at which transitions from 'adequate' lubrication ($\mu < 0.1$) to 'inadequate' lubrication ($\mu > 0.5$ typically) occurred enabled the dependencies of rates of carbon formation and removal on contact conditions to be examined. For example, it was shown that deposition rate increased linearly with admixture flowrate and had an Arrhenius dependence on temperature, while the removal rate increased linearly with normal load. Though not investigated experimentally in these works [11,12], it was hypothesized that removal rate (per unit time) also increases linearly with sliding speed. The ability for increasing sliding speed to induce transitions from adequate to inadequate lubrication was verified by Sawyer and Blanchet [13] in an investigation of the high-temperature traction behavior of such replenishable pyrolytic carbon deposits in concentrated combined roll/slide contacts using a Wedeven Associates WAM-1 test machine.

In addition to parameters such as normal load, sliding speed, temperature, and precursor gas flow rate, the type of precursor gas employed certainly affects the potential for adequate lubrication. A study was therefore undertaken in which three similar hydrocarbon gases with various carbon bond saturations were investigated. Acetylene (C_2H_2), ethylene (C_2H_4), and ethane (C_2H_6) have triple, double and single carbon-carbon bonding, respectively. Existing methodologies, described above, were applied to map the fraction of the normal load/temperature/flow rate space over which each of the three hydrocarbon feed gases possesses the capacity for adequate lubrication.

2. EXPERIMENTAL

The pin-on-disk tribometer is comprised of two nested chambers. The inner chamber contains the contacting pin and disk specimens, self-mated silicon nitride in this investigation, as well as a precursor admixture line, a nitrogen gas line, and an electric resistance heater. The flat disk is mounted on top of a drive spindle, while the hemispherically-tipped ($r=1.6\text{mm}$) pin is contained within a holder located at the end of a cantilevered arm. The purged outer chamber contains and blankets the inner chamber from laboratory air. This tribometer is capable of continuous operation to ambient temperatures of 520°C or higher. The pin holder arm is hinged, which allows for a dead weight load to dictate the contact normal force. An AC motor controller monitors and maintains a constant disk spindle speed. The temperature of the test environment is monitored using a thermocouple located at the center of the disk surface. This thermocouple also provides feedback to the temperature controller which drives the resistance heaters located in the inner chamber.

Four strain gauges are mounted to the base of the pin holder arm. They are used to measure the tangential loads on the cantilever resulting from friction forces in the contact. Connected to this tribometer and strain gauge bridge is a computer controlled data acquisition system which monitors the strain gauge bridge voltage at an acquisition rate of 10 kHz. These values are then converted into friction coefficients, and a time-averaged value representing each consecutive ten-second period is recorded to a datafile. Sliding motion produces a circular wear track on the disk surface, as well as a wear scar on the pin. The diameter of the circular wear scar formed on the hemispherically-tipped pin can be measured post-test by optical microscopy, and used to calculate pin wear volume.

The mapping of boundaries separating regions of adequate lubrication ($\mu < 0.1$) and inadequate lubrication ($\mu > 0.5$, typically) for each gas required the detection of transitions in friction coefficient induced during excursions in the studied test variables: normal load, sliding speed, and temperature. Sliding speed was held constant in these studies at 4.4cm/s . Excursions were made slowly, so that friction coefficients recorded instantaneously approximate steady-state behavior. Tests were initiated under conditions known a priori to provide adequate lubrication, with low friction and wear rate, and excursions in contact conditions were made until transitions to high friction were noted. By inducing transitions in the direction from adequate to inadequate lubrication, wear-induced damage and alteration of the surfaces during the slow excursions is minimized. A typical transition is shown below in figure 2. Mapping was performed in three modes: constant temperature; constant load; and constant flow rate. In each mode, numerous excursion tests were run to determine the locus of transition combinations of the non-constant parameters defining the boundary between adequate and inadequate lubrication.

3. RESULTS

The test variables under consideration in this study are: temperature; normal load; admixture gas flowrate; and hydrocarbon admixture gas species. The experimental program was run in three different series: constant temperature; constant normal load; and constant flowrate. In figures 3-5 data points plotted represent combinations of the test variables causing transition from adequate to inadequate lubrication, found using techniques described in the experimental section.

Tests run to investigate the dependencies of adequate lubrication on load and flowrate involved holding the temperature constant at 520°C and finding transitions from adequate lubrication to inadequate lubrication over a range of normal loads from 2N to 8N. For each load, a test was started at a sufficiently high flowrate to ensure initially adequate lubrication and low friction. Subsequently, flowrate was gradually reduced until a sharp increase in friction signified transition to inadequate lubrication. The results of this constant temperature test series are shown with both acetylene and ethylene plotted on the same scatter plot in figure 3. On this plot the regions of adequate lubrication lie above the least square regression lines. It is clear that acetylene gas, which has a lower carbon bond saturation, can provide adequate lubrication over a larger range of normal loads than ethylene for any given admixture flowrate.

Tests run to investigate the dependencies of adequate lubrication on temperature and flowrate involved holding the normal load constant at 3.4N. Each test was started at a sufficient combination of flowrate and temperature to ensure initially adequate lubrication and low friction. Subsequently, temperature was gradually reduced until a sharp increase in friction signified transition to inadequate lubrication. This procedure was repeated for various flowrates. The results of these constant load test series are shown with both acetylene and ethylene plotted on the same scatter plot in figure 4. On this plot the regions of adequate lubrication lie above the regression curves. It is clear that acetylene gas, which has a lower carbon bond saturation, provides adequate lubrication over a broader range of combinations of flowrate and temperature than ethylene.

Tests run to investigate the dependencies of adequate lubrication on temperature and normal load involved holding the admixture flowrate constant, while finding combinations of temperature and normal load that induce transition from adequate to inadequate lubrication. Due to the effectiveness of acetylene (relative to ethylene) in depositing pyrolytic carbon, constant flowrates of 0.2 l/min for acetylene and 1.3 l/min for ethylene were used so that transitions could be mapped over the same range of normal load and temperature. Each test was started at a sufficient combination of normal load and temperature to ensure initially adequate lubrication and low friction. Subsequently, temperature was gradually reduced until a sharp increase in friction signified transition to inadequate lubrication. This procedure was repeated for various normal loads. The results of this test series are shown with both acetylene and ethylene plotted on the same scatter plot in figure 5. On this plot the regions of adequate lubrication lie below the regression curves. The loci of transition combinations of normal load and temperature for these two gases overlap under the given test conditions, indicating that acetylene has the capacity to provide adequate lubrication over the same range of normal load and temperature as ethylene with a flowrate of only 15% that required for ethylene.

Though also capable of providing adequate lubrication, experiments with ethane as a feed gas for pyrolytic carbon deposition and high temperature lubrication proved difficult. As shown in figure 6 at a temperature of 520°C and a low load of 2.4N, a flow rate in excess of 2.5 l/min is required for adequate lubrication and low friction ($\mu < 0.1$). This represents, respectively, a one and two order-of-magnitude increase, respectively, in required flowrates for adequate lubrication when compared to ethylene and acetylene admixtures at the same temperature and load. Normal loads higher than 2.4N and temperatures lower than 520°C, as used in mapping the performance of acetylene and ethylene, would require even higher ethane flow rates. As such high hydrocarbon flow rates posed potential experimental difficulties, the mapping of ethane performance over a similar temperature / normal load / flow rate space was not pursued.

4. DISCUSSION

This study demonstrated the deposition lubricous graphitic carbon from three different hydrocarbon feed gases on silicon nitride sliding surfaces at high temperature, capable of maintaining low friction coefficients ($\mu < 0.1$) and greatly reduced wear rate (in some cases over two order-of-magnitude reduction) for extended durations. Pyrolytic carbon deposition was confirmed by Raman spectroscopy, with spectra demonstrating the characteristic disorder (sp^3) and graphitic (sp^2) bonding peaks, centered at relative wavenumbers of approximately 1350 and 1580 cm^{-1} respectively, as shown in the example spectrum in figure 7. The range of contact conditions over which each of these hydrocarbon gases can provide adequate lubrication varied greatly, with acetylene demonstrating the most broad effectiveness and ethane the least.

The adequate lubrication of sliding contacts via in situ pyrolytic decomposition of directed hydrocarbon admixtures is believed to exist when the deposition rate of lubricous carbon exceeds the removal rate of this carbon. A proposed model [11,12] representing the net rate of change of carbon on the surface, $\partial C/\partial t$, is

$$\partial C/\partial t = a f \exp(-E_a/RT) - b V F_n \quad (\text{eq. 1})$$

which includes the admixture flowrate (f), the ambient temperature (T), the sliding velocity (V), and the normal load (F_n). The right hand side of this equation subtracts the removal rate of carbon (second term) from the deposition rate (first term). This difference is the net rate (volume per unit time) of carbon accumulation on the surface of the specimen. The constants, a and b , are coefficients for the deposition and removal terms respectively. The deposition rate is assumed to have an Arrhenius temperature dependence described by activation energy E_a , with universal gas constant represented by R . Each gas precursor type may be characterized by a deposition coefficient a and activation energy for pyrolysis E_a . It can be shown that the removal coefficient b is the specific wear rate of pyrolytic carbon from the sliding surface, which may not vary among the three different hydrocarbon precursors.

Transitions from adequate lubrication ($\partial C/\partial t > 0$) to inadequate lubrication ($\partial C/\partial t < 0$) are signified, as previously described, by sharp increases in friction coefficient. At transition ($\partial C/\partial t = 0$) the the deposition and removal rates balance, and can be equated.

$$a f \exp(-E_a/RT) = b V F_n \quad (\text{eq. 2})$$

The coefficients a and b can be grouped into a transition constant $C_{\text{trans}} (= b/a)$ which, as a , should be specific for each precursor gas.

$$C_{\text{trans}} = (f/VF_n) \exp(-E_a/RT) \quad (\text{eq. 3})$$

Any combination of contact conditions (f, F_n, T, V) which yields a value of the right hand side of eq. 3 equal to C_{trans} should represent a transition condition, falling on the boundary between adequate and inadequate lubrication. This formulation agrees with the linear relationship between transition admixture flowrate and normal load observed at constant temperature (figure 3). The Arrhenius description of temperature dependence by the proposed model can be checked by taking the natural logarithm of eq. 3 and rearranging it into a slope-intercept form where $Y = \ln(V F_n / f)$, $X = (1/RT)$.

$$\ln(V F_n / f) = -E_a (1/RT) - \ln(C_{\text{trans}}) \quad (\text{eq. 4})$$

For either gas (acetylene or ethylene) the constant flowrate and the constant normal load transition data, when plotted in such an X-Y fashion (figure 8), fall along straight lines of equal slope indicating that the Arrhenius temperature dependence proposed by the model is applicable. The constant flowrate and constant normal load data furthermore superimpose along the same line, with the constant temperature data clustering about a point on that line, indicating that the normal load and flowrate dependencies proposed in the model also hold. The adequate lubrication regime lies below these regression lines and it is again clear that the acetylene can provide adequate lubrication over a wider range of contact conditions.

The relative difficulty previously mentioned in using ethane as a feed gas for pyrolytic carbon deposition at 520°C is, of course, not beyond thermodynamic explanation. In the proposed model, it is assumed that the Gibbs free energy change ΔG for the pyrolysis reaction is negative. Activation of the forward reaction must overcome barrier height E_a , while activation of the reverse reaction faces a greater barrier height ($E_a - \Delta G$). In the proposed model it is also assumed that the magnitude of ΔG is sufficiently large, and activation barrier for the reverse reaction therefore sufficiently large, that the reverse reaction occurs at a negligible rate relative to the pyrolysis reaction. In such a case, the temperature dependence of the lubrication mechanism is sufficiently described by the Arrhenius representation. Over the range of temperatures tested, this appears to be the case for acetylene and ethylene. The activation energies for acetylene and ethylene, as expressed in these experiments, can be estimated from the slopes of linear data sets in figure 8 as $E_a = 67 \pm 5$ and 43 ± 3 kJ/mol respectively. Using tabulated thermochemical data, the free energy changes for the pyrolysis of acetylene and ethylene at 520°C can be approximated as roughly $\Delta G = -160$ and -100 kJ/mol, respectively [14]. The magnitude of these Gibbs free energy changes are large relative to the activation energy, and should indeed cause the reverse reaction to be of negligible concern.

For ethane, however, the free energy change during pyrolysis at 520°C is only roughly $\Delta G = -20$ kJ/mol, small compared to the activation energy barrier expected for this pyrolysis reaction. Furthermore, ΔG is a function of temperature, and in the case of ethane pyrolysis becomes positive at temperatures not far below 500°C. The difficulties in lubricating contacts by pyrolytic carbon from ethane feed gases can be understood in these terms. Also, it should be noted that the magnitude of Gibbs free energy decrease for the pyrolysis of acetylene decreases with increasing temperature, with ΔG eventually becoming positive. The Arrhenius temperature dependence within the proposed model is not expected to hold when the magnitude of Gibbs free energy decrease for pyrolysis becomes small relative to the activation energy. Furthermore, over certain temperature ranges the various hydrocarbon feed gases candidates for continuously replenished lubricous pyrolytic carbon supply to bearing contacts may face thermodynamic limitations.

5. CLOSING REMARKS

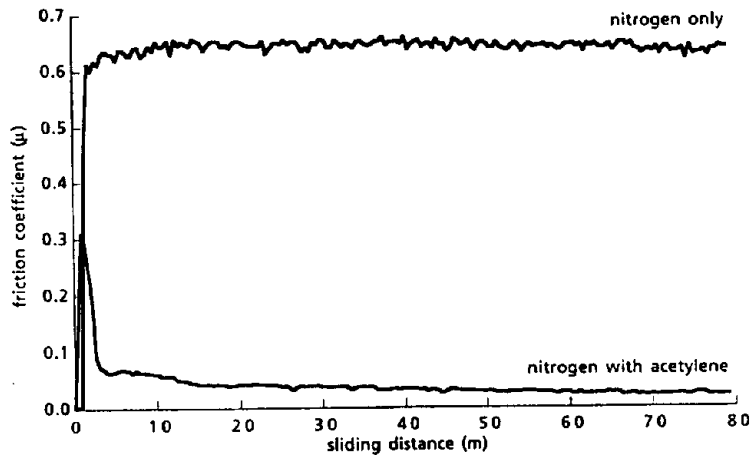
The ability of directed streams of three representative hydrocarbon gases to provide extended duration lubrication to high temperature sliding contacts via surface deposition of pyrolytic carbon has been demonstrated. Acetylene, with its carbon-carbon triple bond, was the most effective precursor for pyrolytic carbon deposition, providing adequate lubrication ($\mu < 0.1$) over the broadest range of normal load / temperature / flowrate combinations. Ethane, having single-bonded carbon-carbon, proved least effective. In addition to this new information concerning the effect of hydrocarbon precursor unsaturation, the mapping study also provided further support to a proposed model for the dependence of the lubrication mechanism on normal load, temperature, and precursor gas flowrate. Studies of the additional effects of sliding and rolling speed and contact geometry on the lubrication mechanism have since been initiated.

6. ACKNOWLEDGMENTS

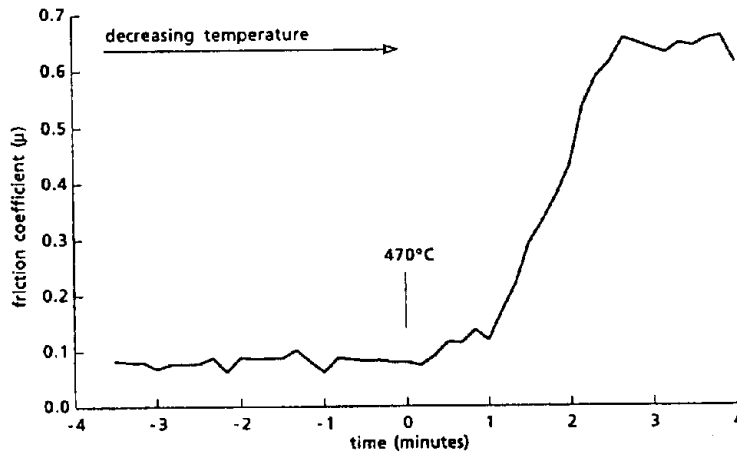
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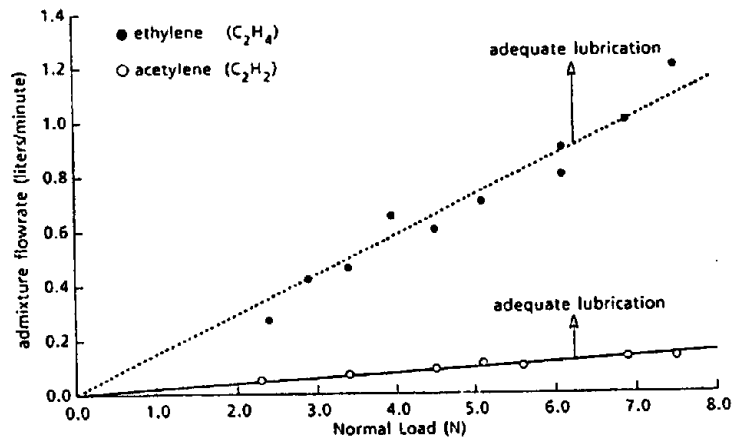
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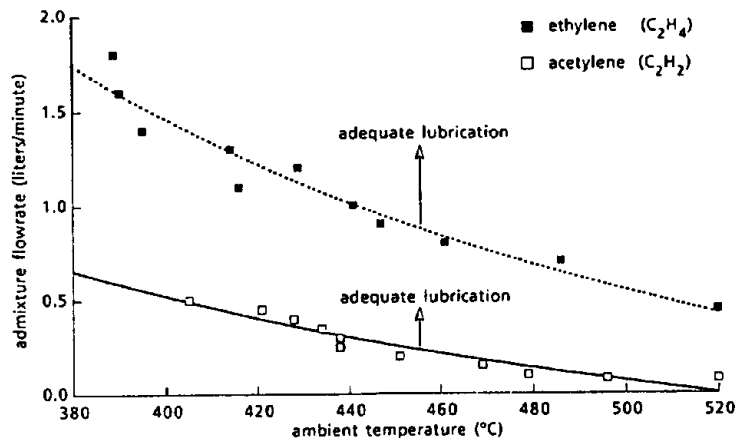
1. Friction coefficient of a self-mated silicon nitride sliding contact at 520°C in a nitrogen environment with and without a directed acetylene admixture for pyrolytic carbon deposition.



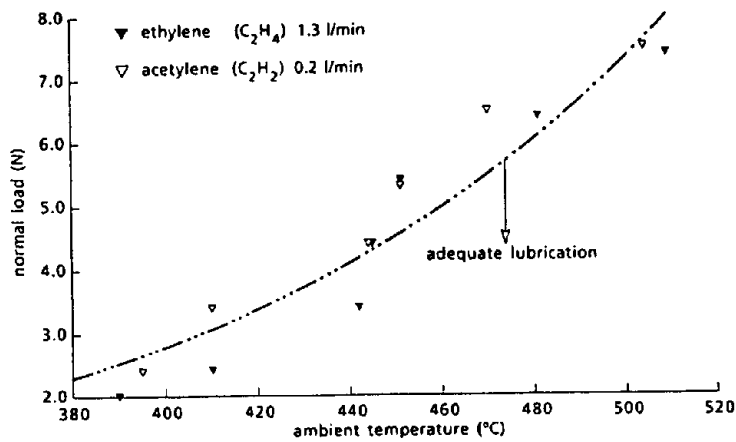
2. Friction coefficient of a self-mated silicon nitride sliding contact in the presence of a directed stream of acetylene at 0.15 l/min (normal load 3.4N, sliding speed 4.4cm/s) as temperature is gradually reduced with increasing time. After being reduced to 470°C, transition to high friction is observed.



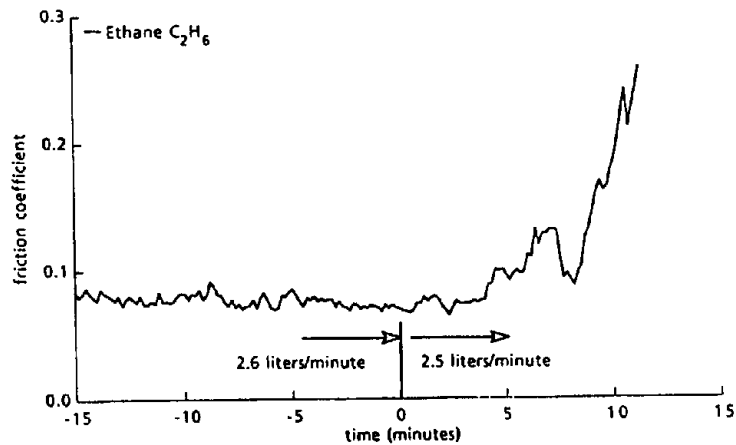
3. Combinations of admixture flowrate and normal load at which transition from adequate to inadequate lubrication is noted for sliding contacts provided with acetylene and ethylene at a constant temperature of 520°C and sliding speed of 4.4cm/s.



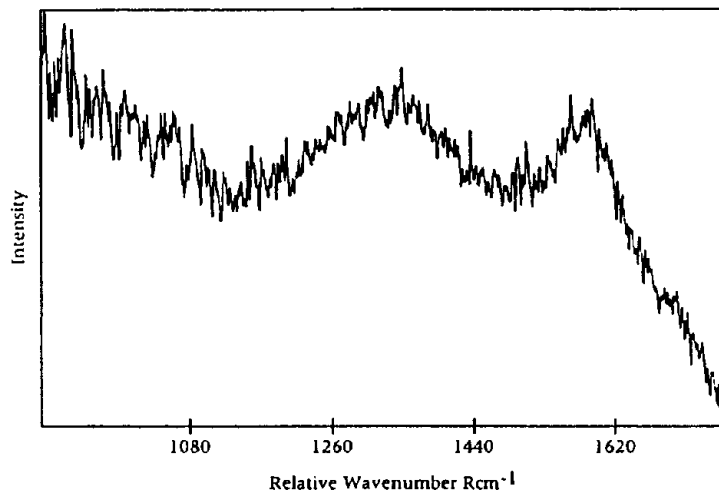
4. Combinations of admixture flowrate and temperature at which transition from adequate to inadequate lubrication is noted for sliding contacts provided with acetylene and ethylene at a constant normal load of 2.4N and sliding speed of 4.4cm/s.



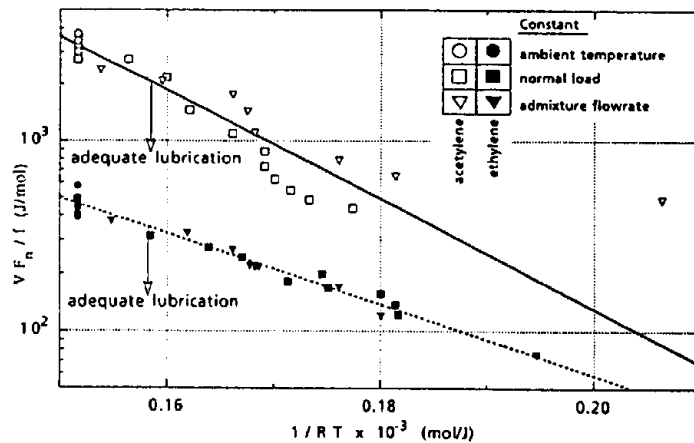
5. Combinations of normal load and temperature at which transition from adequate to inadequate lubrication is noted for sliding contacts provided with acetylene and ethylene at a constant admixture flowrate. A constant flow rate of 0.2 l/min is used for acetylene, and 1.3 l/min for ethylene. Sliding speed 4.4cm/s.



6. Friction coefficient of a self-mated silicon nitride sliding contact (temperature 520°C, normal load 2.4N, sliding speed 4.4cm/s) in the presence of a directed stream of ethane as precursor flowrate is gradually reduced with increasing time. After being reduced to 2.5 l/min, transition to high friction is observed.



7. Microfocused Raman spectrum taken from the pin wear scar, following lubrication by a directed stream of acetylene, displaying disorder and graphitic peaks at roughly 1350 and 1580 Rcm^{-1} characteristic of lubricious pyrolytic carbon.



8. Summary plot of combinations of normal load, temperature, and admixture flow rate at which transition from adequate to inadequate lubrication is noted for sliding contacts provided with acetylene and ethylene at a sliding speed of 4.4cm/s. Choice of axes to facilitate evaluation of proposed model. Note, admixture flowrates have been converted from volume per time to moles per time.