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13. ABSTRACT *(Maximum 200 words)*

A multidisciplinary program of basic research was conducted with emphasis on potential ways of reducing friction and wear under conditions of boundary lubrication. A special aim was the development of lubricating systems for applications at temperatures above 300°C. Both experimental and theoretical (computer modeling) approaches were used in parallel.

Among the most important results were:

- Stable carbonaceous gases or vapors (ethylene, benzene propane, propanol) form a lubricating carbon deposit on nickel-containing steels at temperatures above 350°C. Under wear fresh metal surfaces are exposed providing continuous catalytic regeneration of the solid lubricant as long as the gas is supplied.
- The same concept also works on ceramic surfaces such as silicon nitride, Sialon, silicon carbide or zirconia. However, the nature of the carbon (possibly cracking-type coke) is spectroscopically different. Friction coefficients as low as 0.02 and negligible wear could be achieved.
- A parallel ultrahigh vacuum SEM study showed catalytic carbon deposition on nickel from ethylene in the same temperature range where the friction coefficient was low.
- New computer models of polymer friction in the temperature-controlled region and of EHD by fracture mechanics were devised.

14. SUBJECT TERMS
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1. STATEMENT OF PROBLEMS STUDIED

The purpose of this multifaceted program of interdisciplinary basic research was to find ways of reducing friction and wear under conditions of boundary lubrication. Special emphasis was to be given to lubrication at temperatures of 300°C and above, and at high loads, which requires special additives to liquid lubricants, replenishable solid lubricants, or tribosurfaces of materials requiring no lubricants. Both experimental and theoretical (computer modeling) approaches were to be used in parallel.

1.1. Approach and Comments

Surface science, mechanical engineering, chemistry and chemical and materials engineering, and computer science were among the disciplines used. Faculty and graduate students from the Departments of Mechanical Engineering, Chemical Engineering and Materials Engineering participated.

The program experienced severe funding cuts every year during its four-year lifetime. Therefore only those areas of research were continued in successive years, that appeared to be the most promising. The last (ninth) half-year was a no-cost extension with only the P.I. working on the project.

The research yielded 39 publications in archival journals or books. One paper won a Best Paper Award (Paper #3 on the List of Publications). Every participating professor was *invited* by a national engineering or scientific society at least once during the life of the project to speak on work sponsored by this contact.

2. SUMMARY OF THE MOST IMPORTANT RESULTS

2.1 Lubricating Carbon Films on Nickel and Palladium

2.1.1 Concept

The formation of carbon ("coke") on transition metal surfaces during the catalytic dehydrogenation of the hydrocarbons has been well-known in petroleum refining for a long time. If the carbon is also lubricating--as was found to be the case in this research program--than it could be exploited for high-temperature lubrication with continuous feed. The concept is this:

- Feed a thermally stable gas from a supply at ambient temperature to hot (350-650°C) friction surfaces.
- Catalytically decompose this gas at the friction surfaces to form a lubricating carbon deposit. Water (steam) is the only other product.
- Preferred metallic surfaces are alloys containing nickel and palladium because of their known catalytic activity.

2.1.2 Approach

Fig. 1 shows the pin-on-disc apparatus used. Its core is the inner shell, a prolate ellipsoid of revolution containing the friction contact at one focus and a quartz halogen lamp, the heater, at the other. Treatment gases (ethylene) are injected only into the conjunction region. The surrounding environment can be air or an inert gas. The outer chamber is filled with an inert gas; it provides a safety shield. Friction is measured continuously, wear periodically by determining wear track volume and pin volume change. Contact pressures ranged between 200 and 800 MPa, linear velocities between 3 and 30 cm/sec.

Fig. 2 shows friction vs. time traces obtained at different temperatures with a sapphire pin on a silicon nitride disc overcoated with nickel. Note the sharp drop of friction on ethylene introduction especially at 500°C. The change is more sluggish at the other temperatures. Wear changes paralleled the friction changes.

Fig. 3 gives the steady state surface carbon coverage, as determined by Auger electron spectroscopy (AES) at various substrate temperatures. This experiment was carried out independently on a pure Ni(110) film in a different RPI laboratory. It is clear that this result is in good agreement with the temperature dependence of film formation in the wear experiment.

During the wear experiment fresh metal surfaces were continuously produced and graphitic carbon deposits formed. These deposits adhered strongly to the substrate, being partly dissolved in it.

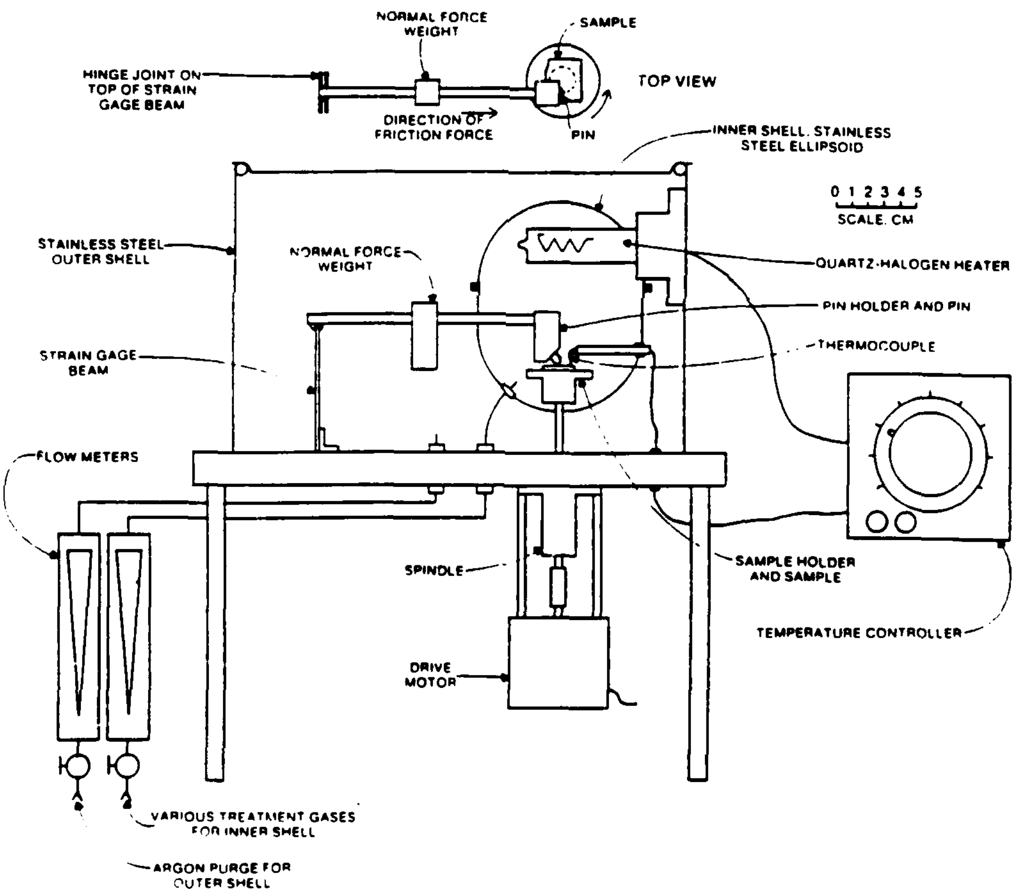

Fig. 1 Pin- on-Disc Apparatus

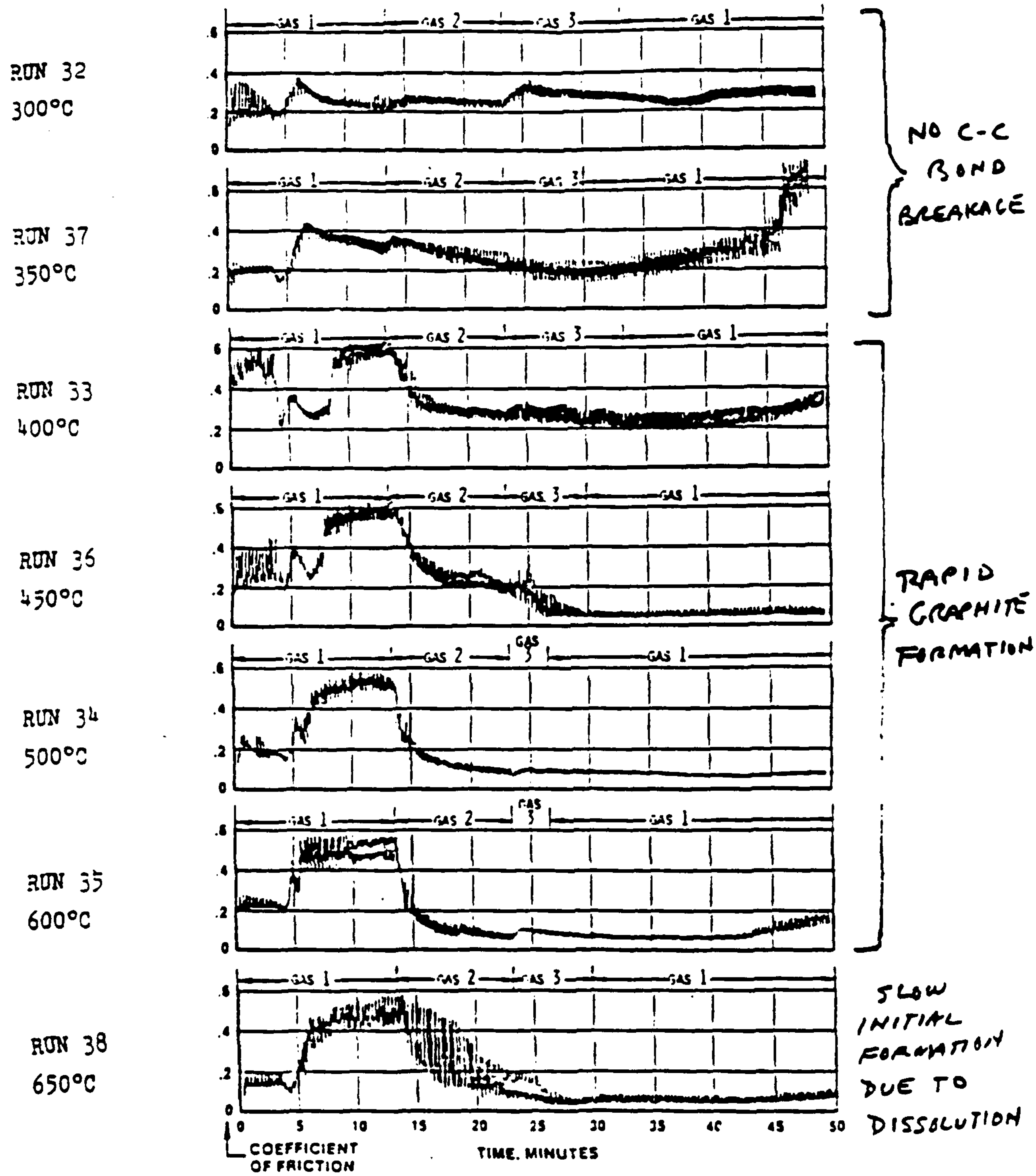

Fig. 2 Changes of FrictionCoefficient with Temperatures in the Pin-on-Disc Tribometer. (Nickel-coated Sialon disc, sapphire pin. Gas 1 = He+Ar, Gas 2 = C_2H_4 + Ar, Gas 3 = C_2H_4 + H_2+Ar)

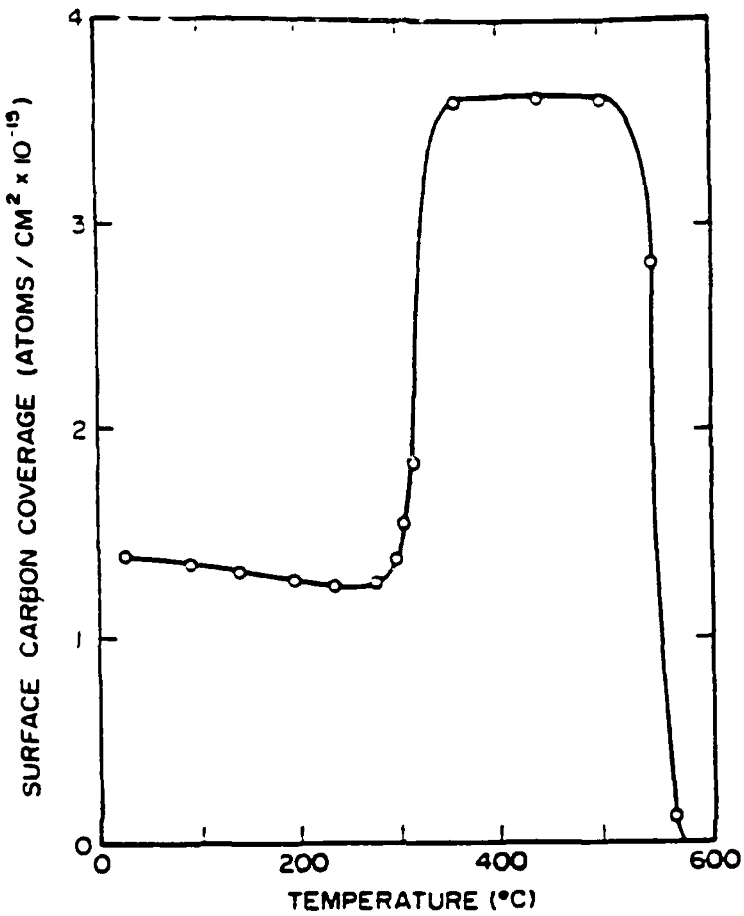

Fig. 3 Steady state surface carbon coverage, as determined by AES, at various substrate temperatures.

2.2 Lubricating Carbon Films on Silicon Nitride (Sialon) and Nitride (Sialon) and Silicon Carbide

A major breakthrough in this study was the finding that lubricating carbon was produced on Sialon and other plates in the above-mentioned experiment even in the absence of a nickel overcoat. Friction coefficients as low as 0.02 could be achieved for loads similar to those used in the nickel experiments. The drop of friction on introduction of ethylene was two step; a wear-in period and a final period (Fig. 4). Raman spectra obtained from the carbon coating, which was formed only on the wear track, showed significant differences between the carbon now produced and the (presumably) graphite produced on nickel. Furthermore the Raman spectra also indicated that a silicon carbide (SiC) was produced in the wear-in step. SiC could provide nuclei for further carbon deposition. Raman spectroscopy proved to be an invaluable tool in the characterization of the lubricating carbon.

• TRIBOCHEMICAL WEAR-IN

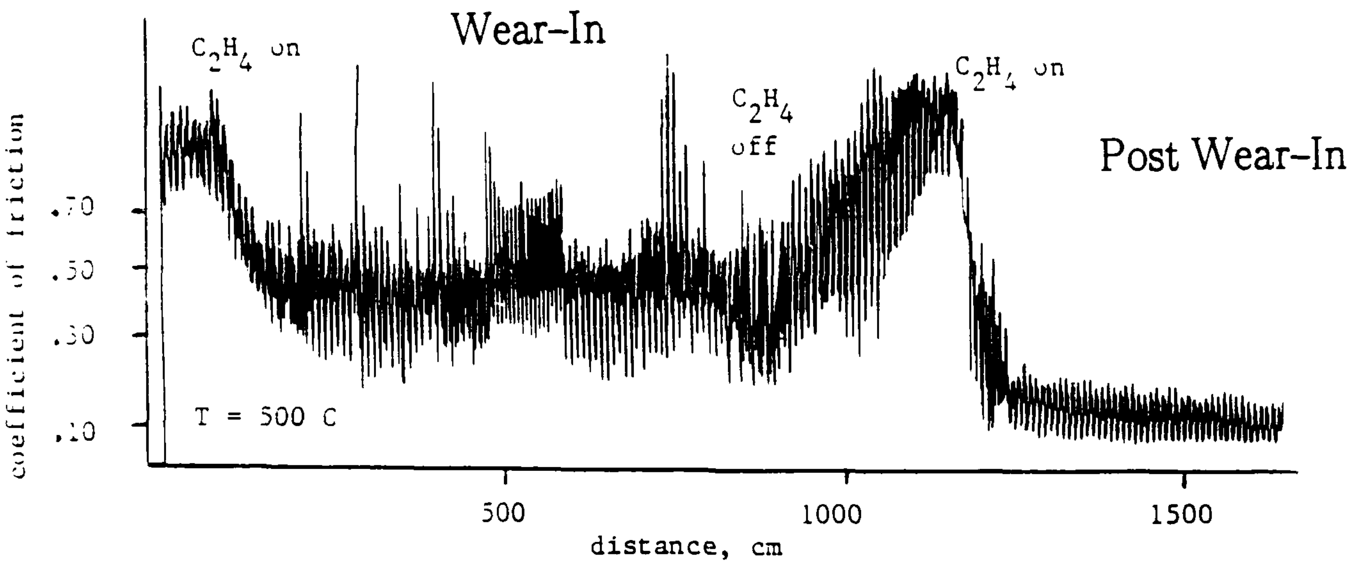

Fig. 4 Coefficient of friction vs. sliding distance for sapphire on bare Sialon. The ethylene reaction produces a much lower coefficient of friction after the wear-in period

This work is continuing under partial industrial sponsorship. The concept might be applicable to the uncooled diesel engine projected, inter alia, by an Army group.

2.3 Influence of Friction Heating on the Sliding Friction of Elastomers and Polymers

Both theoretical and experimental research led the thermal control model, which is based on the concept that surface temperatures in a sliding contact cannot increase indefinitely as conditions are made more severe. Eventually an upper-bound, limiting temperature T_f is attained. This is called the Friction Defined Temperature, which remains at a constant value as the severity of the sliding is increased.

Only an extremely thin layer of material at the surface need be at T_f. Once thermal control has been initiated, the friction coefficient is given by

$$\mu = \frac{1.45(T_f - T_a)}{KP}\left(\frac{k\rho c}{VB}\right)^{1/2}$$

where

T_a ambient temperature
K proportion of generated heat to moving surface
P interface pressure
k thermal conductivity
ρ density
c specific heat
V sliding speed
B length of contact in direction of sliding

The effect of thermal control for three different loads, $W_1 < W_2 < W_3$ is shown in Fig. 5. The decrease of friction coefficient with increasing speed is characteristic of this operation. This result can be especially important to the friction of tires on pavement: The braking distance at high speeds is increased further by a lower coefficient of friction.

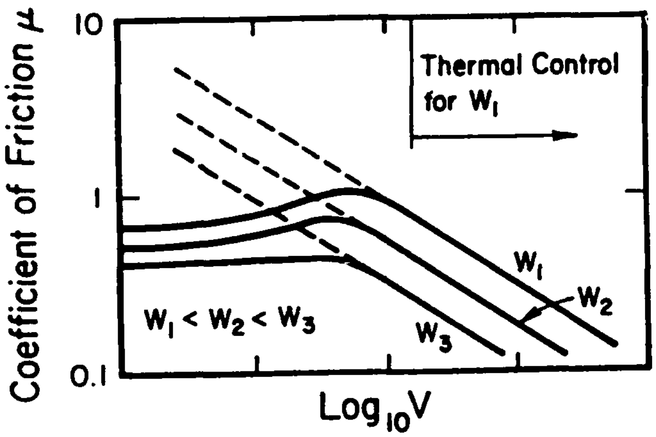

Fig. 5 The effect of thermal control on the level of friction.

2.4 Sliding Experiments in the Scanning Electron Microscope (SEM)

An SEM was modified to allow the direct IN SITU observation of friction and wear on a microscopic scale. A pin was made to plow into the mantle of a rotating cylinder. The process was recorded on videotape. The nature of the wear debris was shown to depend on the absence or presence of a lubricant. Plastic deformation can play an important part in wear. A plastically deformed layer can cover grooves and scratches of the original surface and thereby reduce their function as lubricant reservoirs. Plastically extruded layers were observed on either side of plowing marks when wear became severe. These layers became a source of wear debris in the form of platelets.

The videotapes documenting this work, which encompassed different materials and experimental parameters, have been very popular at lectures and seminars on wear. The modified SEM head is now commercially available.

2.5 Fractal Geometry Applied to Tribology

Fractal geometry is clearly a natural model for describing such self-similar phenomena as elastohydrodynamic lubrication (EHD) and micro-EHD. In other words there are asperities on asperities and all them can be elastically deformed. A new exponential scaling law was discovered, making it possible to join continuum mechanics to surface physics or chemistry.

2.6 Facilitation of Metal Forming and Shaping by Sulfur Compounds

When such compounds as disulfides, sulfides or thiophene were present as vapors in the environmental air in a steel pin-on-disc fixture designed to simulate a cutting operation, friction could be greatly reduced. This finding was not new; however, the location of the sulfur on the steel disc was surprising. It was found by Auger electron spectroscopy (AES) only in the wear track or the wear debris and than that of only some 40 nm below the surface. The top layer was an iron oxide of different crystal shape then a normal oxide would be. Later it was learnt that a group at Case Institute of Technology found the same effect when iron was first exposed to sulfurous vapors and subsequently heated in air at 700°C.

The implication of this finding is that the material reducing friction could be an iron oxide produced by iron sulfide conversion rather than iron sulfide. If this concept can be further extended, numerous applications are likely.

2.7 Steady-State Moving Loads on Elastic-Plastic Solids

Local stress and deformation fields for contact loads moving steadily on an elastic-plastic, strain-hardening solid under plane strain and small-scale yielding conditions were analyzed numerically on a Super-Computer by a steady-state finite element interative procedure. The numerical results revealed the development of elastic unloading zones trailing the moving contact load. The influence of strain hardening and friction condition on the local stress and deformation states became apparent.

2.8 Other Results

As is clear from the titles of the 39 publications on work partially funded by this project, many other research results were obtained. Much of the work seeded by this contract is continuing.

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