A RADIO-TRACER STUDY OF THE EFFECT OF SURFACE FINISH ON THE TRANSFER OF POLYCHLOROTRIFLUOROETHYLENE SLIDING ON MILD STEEL

by

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LIST OF SYMBOLS AND ABBREVIATIONS

PCTFE	Polychlorotrifluoroethylene
PTFE	Polytetrafluoroethylene
mg	Milligram
mm	Millimeter
Zn	Zinc
nvt	Neutrons x seconds/centimeters^2
nv	Neutrons/centimeters^2
t	Irradiation time in seconds
cm	Centimeter
N	Newtons (force)
lb	Pound (force)
in.	Inch
C	Degrees Centigrade
sec	Seconds
rpm	Revolutions per minute
Ge	Germanium
Li	Lithium
µm	Micrometer
rms	Root mean square
kg	Kilogram
hr	Hour
µg	Microgram
μ_k	Kinetic coefficient of friction

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I. INTRODUCTION

Interest in polymers as engineering materials, for use in tribological situations, has increased rapidly in the past two decades. Reasons for this include ease of fabrication, good corrosion resistance, and the typically low coefficients of friction associated with polymer parts.

Much research has been carried out in an effort to gain a better understanding of the basic physical and chemical mechanisms that determine both the frictional and the wear characteristics of typical engineering polymers. Most of this research has been directed toward mechanisms of polymer friction, and has led to an adhesion theory of friction which is generally accepted. Theories have also been formulated concerning polymer wear, but wear data are not at all plentiful. Research has shown that most polymer wear occurs by a mechanism involving transferred polymer films. Because of the minute amount of transfer which usually occurs (especially at low sliding velocities), quantitative wear data are very difficult to obtain.

Many scientific tools have been utilized during this research. Some were tried and proven techniques such as optical microscopy and profilometric techniques, which have been used for years by the metallurgist for friction and wear studies, while others were seemingly novel applications of newly developed techniques, such as auger emission spectroscopy, field ion, and scanning electron microscopy. The two most common sources of wear data have been gravimetric measurements and measurements of the width and thickness of the transferred films from which a mass is calculated.

Radio-tracer techniques which have been used extensively for metallic wear studies have seen almost no application in polymer wear studies. There are some obvious reasons for this exclusion. Engineering polymers such as nylons, polycarbonates, polyethylenes, acetal homopolymers, and the fluorocarbon polymers are composed primarily of carbon, hydrogen and oxygen. These are all relatively light atoms which when subjected to neutron bombardment do not become very radioactive. The isotopes that do result are too short lived to be useful in wear studies. Polymers are also much more prone to radiation damage than metals. Sufficient irradiation causes cross linking or degradation of the polymer and can result in changes of the bulk material properties. This would be unacceptable for fundamental friction and wear research. Studies involving radioactively tagged lubricants are not widespread because engineering polymers are usually employed in dry sliding situations.

In this study, the wear of polychlorotrifluorethylene (PCTFE) was measured by the radio-tracer technique. PCTFE contains chlorine as a side group within the basic repeat element of the polymer. Chlorine is readily activated using neutron bombardment and the isotope ^{38}Cl that results is relatively long lived, having a half-life of approximately thirty-seven minutes. Care was taken to limit the polymer's radiation doseage to levels below threshold damage levels reported for PCTFE.

Using neutron activation detection equipment along with a pin-on-disk apparatus, wear data were generated for PCTFE sliding on mild steel at a low sliding velocity, 0.20 cm/sec. A gravimetric method was used to obtain a mass calibration for the radio-tracer technique. Scanning electron microscopy was employed to examine the test specimens for an indication of the wear mechanisms that were involved.

The object of this work was to obtain wear data on the rate of transfer of a PCTFE pin to a mild steel disk as a function of surface roughness. A literature survey has shown that very little is known about wear rates which occur after an initial pass of polymer on a harder substrate.

II. REVIEW OF LITERATURE

In this study, a radio-tracer technique was used to measure the wear of an engineering polymer (PCTFE). To evaluate the efficacy of this technique, a review of existing polymer wear measurement techniques was performed. Special emphasis was placed in the area of radio-tracer techniques to ascertain that this study was indeed unique. Information concerning radiation effects on PCTFE was reviewed to determine the magnitude of the changes in the mechanical properties that would result from irradiation of the test specimens. Finally, the literature was surveyed for related friction and wear data for comparison to the results of this study.

A. Wear Measurement Techniques

Quantitative wear data for polymers has been obtained by gravimetric methods, linear measurements of wear tracks, and measurements of dimensional changes of test specimens. There are inherent uncertainties in all of these techniques that had to be dealt with in a variety of ways by different researchers in an effort to obtain accurate and repeatable wear data.

Dowson, Atkinson, and Brown (1) evaluated two of these techniques during a study of the wear of high molecular weight polyethylene. A pin-on-disk machine was employed, and one of the methods attempted was the measurement of dimensional changes of the pins. Both pin length and pin end diameter were measured before and after the wear tests. The volume of material lost was then computed as an indication of wear rate. Several disadvantages associated with this technique became apparent. Uncertainties in pin length measurements existed because:

1) Creep of the pins could occur during the test.
2) Thermal expansion was appreciable.
3) Absorption of water affected pin lengths.
4) Unevenness of wear introduced a "judgment" factor in the linear measurement after the test.
5) The measuring instrument exerted a small load on the pins and caused some deformation.
6) The collet that held the pins in the wear machine caused some permanent deformation.

Many of these uncertainties also applied to the measurement of pin end diameters. Additional uncertainties included:

1) Deformation of the pin during the test could cause some ovality.
2) A perfectly clear image could not be measured because of the limited depth of field of the optical microscope.

Weight loss measurements were also evaluated and the following disadvantages were identified:

1) Water absorption was significant; during a 1500-hour test the pins lost 2 mg of material by wear and gained 1 mg from water absorption.

2) Pick up of dust or wear debris during the test could cause gross errors in the data.

These researchers choose the weight loss technique for their study and monitored water absorption using a standard that was subjected to the same environment as the test pins.

Although most polymers do not absorb water as readily as polyethylene, this study still reflects the problems encountered with two commonly employed wear measurement techniques. The sensitivity of these two techniques, as reported by Dowson, Atkinson, and Brown ranged from 0.002 mm^3 to 0.006 mm^3 of wear. In terms of mass these minimum detection limits were approximately two to six micrograms.

The other popular method for wear rate measurements has been the measurement of wear tracks left on a harder substrate, or the measurement of a wear scar on the softer (usually polymer) specimen.

J. K. Lancaster (2) utilized the wear scar technique during a study of the wear of some 18 polymers as a function of temperature. During the course of his study, Lancaster observed that wear scars were subject to elastic recovery. It was decided that wear rates obtained in this manner had been overestimated by as much as 30 percent. To circumvent this problem, the wear scar measurements were replaced by a weight loss technique. The effect of weight gain due to water absorption was not mentioned in this work. The sensitivity of Lancaster's techniques were again on the order of micrograms.

In many metallic wear studies, wear rates have come from measuring the width and thickness of the wear track, from which a volume loss per unit sliding distance was obtained.

These measurements were usually taken from profilometric data, where profile traces were obtained across the wear track, perpendicular to the sliding direction. Bayer, et al. (3, 4) used this method to generate wear data for their "zero wear" model, which included work with engineering plastics.

Hollander and Lancaster (5) found that transferred polymer films were often too soft to resist penetration of a profilometer stylus, thus eliminating the usefulness of this technique for polymer wear studies.

Steijn (6) sliding steel spheres across sheets of polytetrafluoroethylene (PTFE), attempted to measure changes in the wear scars in the PTFE using profilometry. Steijn abandoned this technique because it lacked the necessary sensitivity. His study was completed using electron microscopy.

Electron microscopy, which includes the use of both transmission and scanning electron microscopes, has been used primarily to obtain qualitative wear data. The major advantage of electron microscopy over optical microscopy was the excellent depth of field obtainable at high magnifications.

Pooley and Tabor (7), in a comprehensive study of the friction and wear properties of some thermoplastics, used electron microscopy to detect transferred polymer films. They were also able to estimate the thickness of these films, but not with sufficient accuracy to attempt to quantify the wear data.

B. Radioactive Tracer Applications in Wear

Radioactive tracer techniques have been used extensively for metallic wear studies. Because of the problems discussed earlier, this technique has seen almost no application in polymer wear studies.

Because of the vast number of metallic wear studies that have employed radio-tracer techniques, and the fact that this study involved polymers, an exhaustive review of the literature concerning metallic wear data will not be presented here. The reader is referred instead to a comprehensive review of this subject authored by J. K. Lancaster (8).

Other than papers concerned with the wear of rubber automobile tires, there is only one reference in Lancaster's review to studies that involved the wear of engineering polymers. In this study, Ezran, studying the contact of phenolic resin gears, immersed the plastic in a sodium chloride solution and then activated the absorbed sodium. Resulting wear was then determined from the activity present in the lubricant. Lancaster commented:

> "This method is particularly valuable for plastic materials because the absorption of fluids and vapours by plastics introduces an appreciable uncertainty into wear determinations made from dimensional or weight changes."

No further polymer wear studies involving radio-tracer techniques were found in a search of the literature since Lancaster's review.

The many capabilities of the radio-tracer technique were demonstrated by Kerridge and Lancaster (9). While their study involved metallic wear, it is of interest here because it was directed at obtaining an understanding of the dynamics of the wear process. Kerridge and Lancaster irradiated brass pins and slid them across a much harder metallic ring. The activity of the brass which was transferred to the ring was measured with a Geiger-Müller counter that had been calibrated in terms of mass. Using this procedure, they determined that the wear rate reached a constant limiting value the same time that the amount of transferred material leveled off. By first running an irradiated pin until the transfer leveled off, and then running an unirradiated pin over the built up film, it was discovered that the transferred film was constantly being replenished and was therefore not static. Using slightly different experimental procedures, Kerridge and Lancaster were also able to determine the rate of loose wear particle production, and obtain measurements of back transfer.

The success of their technique was due largely to the long (250 day) half-life of the ^{65}Zn isotope that resulted from irradiating the brass. The wear rates obtained in this study were also about two orders of magnitude greater than those typically observed for engineering polymers. This was beneficial because accurate data were obtained without the need for extreme sensitivity.

C. The Effects of Radiation on PCTFE

One very important restriction that had to be observed during this study was the maximum radiation dose for the polymer that could be tolerated. Relatively small radiation doses can cause marked changes in the bulk material properties of engineering polymers through crosslinking or degradation of the polymer chains. A comprehensive review of the literature concerning radiation effects on polymers was performed by Charlesby (10).

As can be seen in Fig. 1, the elastic modulus of PCTFE has been found to remain essentially unchanged for reactor radiation exposures of up to 0.2×10^{18} nvt, while the elongation at break had already been reduced by a factor of 2 by an exposure of 0.06×10^{18} nvt.

Charlesby sites a conversion from radiation exposure to absorbed dose for PCTFE as:

$$1 \times 10^{18} \text{ nvt} \cong 500 \text{ megarads}$$

where "nvt" are units of radiation exposure derived from the neutron flux of the reactor (nv) multiplied by the irradiation time (t).

The neutron flux of the VPI & SU Research Reactor operating at 100 percent of its rated power, was 1.20×10^{12} neutrons/cm^2. The irradiation finally chosen for this study

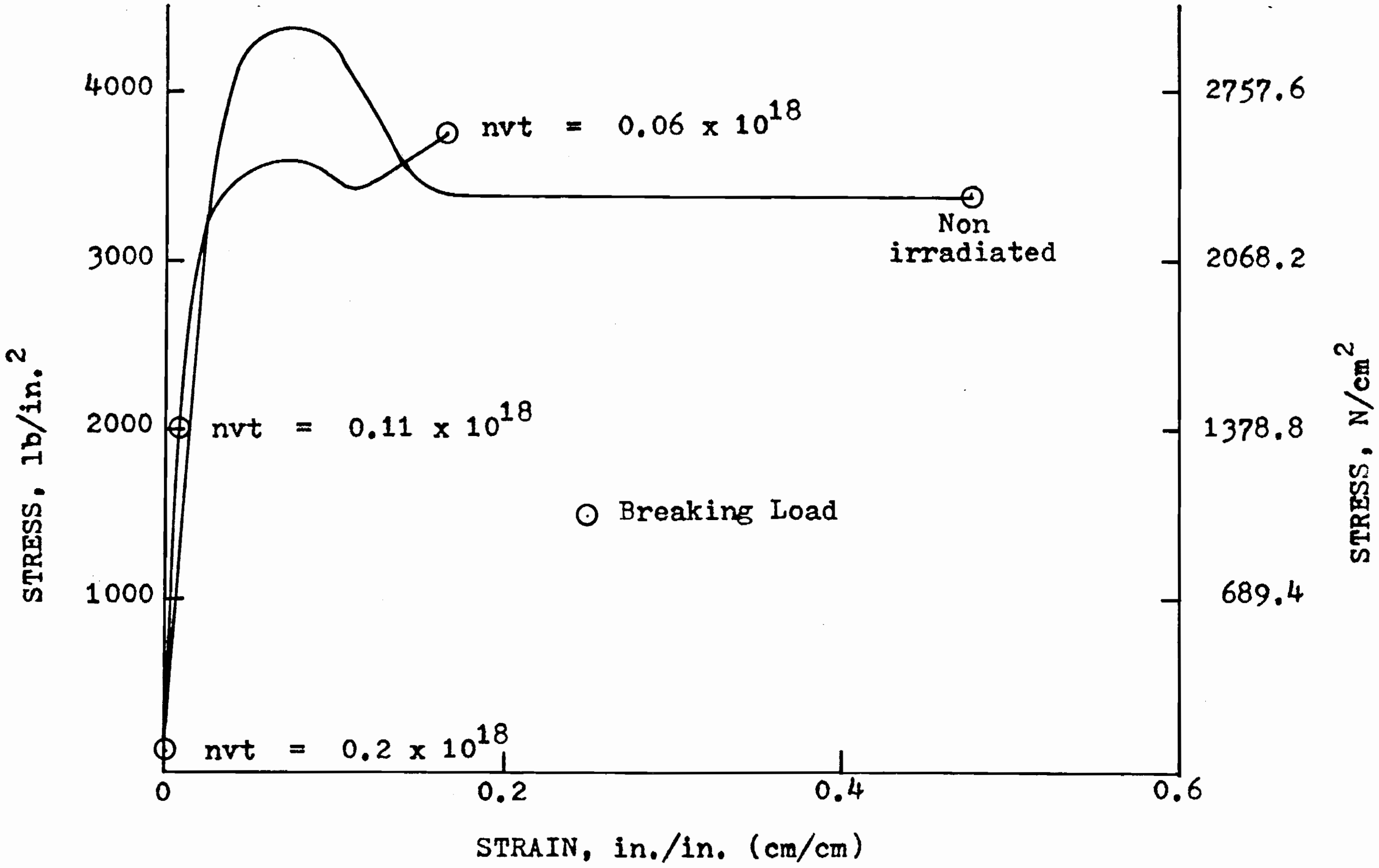


FIGURE 1. Radiation Effects on PCTFE

(From: Charlesby (10), ATOMIC RADIATION AND POLYMERS, Pg. 357) was 10 minutes. This resulted in a total exposure of 0.00072×10^{18} nvt (600 sec x 1.20×10^{12} nv). Referring to Fig. 1, this exposure was yet two orders of magnitude lower than the minimum exposure plotted in the figure. Using Charlesby's conversion, this exposure was equal to approximately 0.4 megarads.

Presented in Table I for comparison are the data supplied by the manufacturer of the PCTFE concerning radiation effects (11).

Based on this information it was decided that a 10-minute irradiation time would cause no significant change in the material properties of PCTFE.

D. Related Friction and Wear Studies Involving PCTFE and the Effects of Surface Finish

Friction and wear data for PCTFE have in the past been generated to study the effects of the addition of bulky side groups to the basic polytetrafluoroethylene (PTFE) molecule. Pooley and Tabor (7), sliding PCTFE on glass, found no appreciable transfer of polymer unless the ambient temperature was above the glass transition temperature (52 to 60 C) of PCTFE. They state that at room temperature, the interfacial adhesion between the polymer and glass must have been less than the bulk shear strength of the PCTFE; thus preventing transfer. Pooley and Tabor also slid PCTFE on abraded steel and reported:

> "When Teflon, FEP and PCTFE are slid on a rough surface, the friction and transfer do not

TABLE I. Radiation Effects on Crystalline PCTFE

MATERIAL PROPERTY (% Retained)	DOSE (megarads)	
	16	24
Tensile Strength	75	65
Elongation	20	3
Yield Strength	80	80
Modulus of Elasticity	110	105

> change because easy slip has already been inhibited by alterations to the molecular structure (referring to the structure of PTFE)."

Hollander and Lancaster (12) studied the effect of surface finish on the wear rate of a number of plastics and concluded that surface topography was the predominant factor influencing wear rate. They found that transferred polymer films could both decrease wear rate by smoothing an initially rough surface and increase wear rate by effectively roughening a polished surface.

It was evident that one aspect of surface topography which received very little attention was the effect of generating the same measured surface finish using different machining techniques. Only one brief reference to this topic was found in the literature. Richardson (13) stated that there was some evidence that ground surfaces resulted in lower wear rates than turned surfaces.

Since the coefficient of friction was also monitored in this study it was of interest to review the literature for typical friction coefficient data for PCTFE sliding on steel at low sliding velocities. Pooley and Tabor (7), and Bowers, Clinton, and Zisman (14) reported kinetic coefficients of friction for PCTFE of 0.28 to 0.30 for all surface topographies.

III. EXPERIMENTAL SYSTEM AND PROCEDURE

A. Experimental System

There were two basic components of the experimental apparatus:

1) Pin-on-disk apparatus.
2) Radiation counting equipment.

A friction and wear apparatus was developed to meet the following criteria:

1) Should be capable of very low sliding velocities (greater than or equal to 0.01 cm/sec).
2) Should be able to generate friction force data.
3) Should incorporate specimen sizes such that scanning electron microscopy and neutron activation analyses are possible.
4) Should be portable by two men.

Figure 2 illustrates the pin-on-disk apparatus and the necessary instrumentation. A pin-on-disk configuration was chosen because:

1) A short cylindrical pin could be used with existing reactor entry equipment.
2) A disk of maximum diameter = 7.6 cm and maximum thickness = 0.7 cm was the largest specimen which would fit conveniently into the chamber of the scanning electron microscope.

As can be seen in Fig. 3, the polymer pins were mounted in the end of a counterbalanced arm. Loading was accomplished

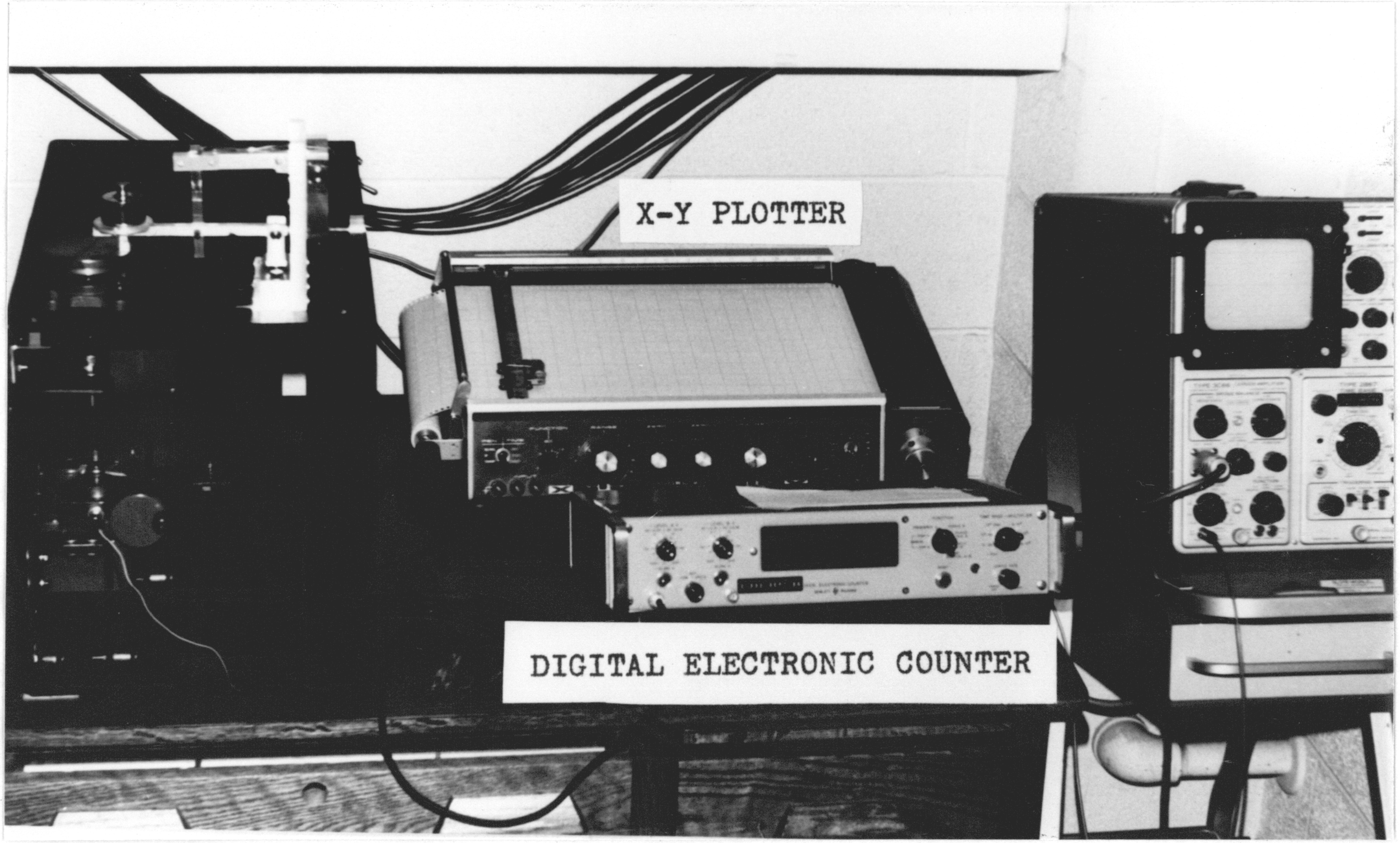


FIGURE 2. Experimental System

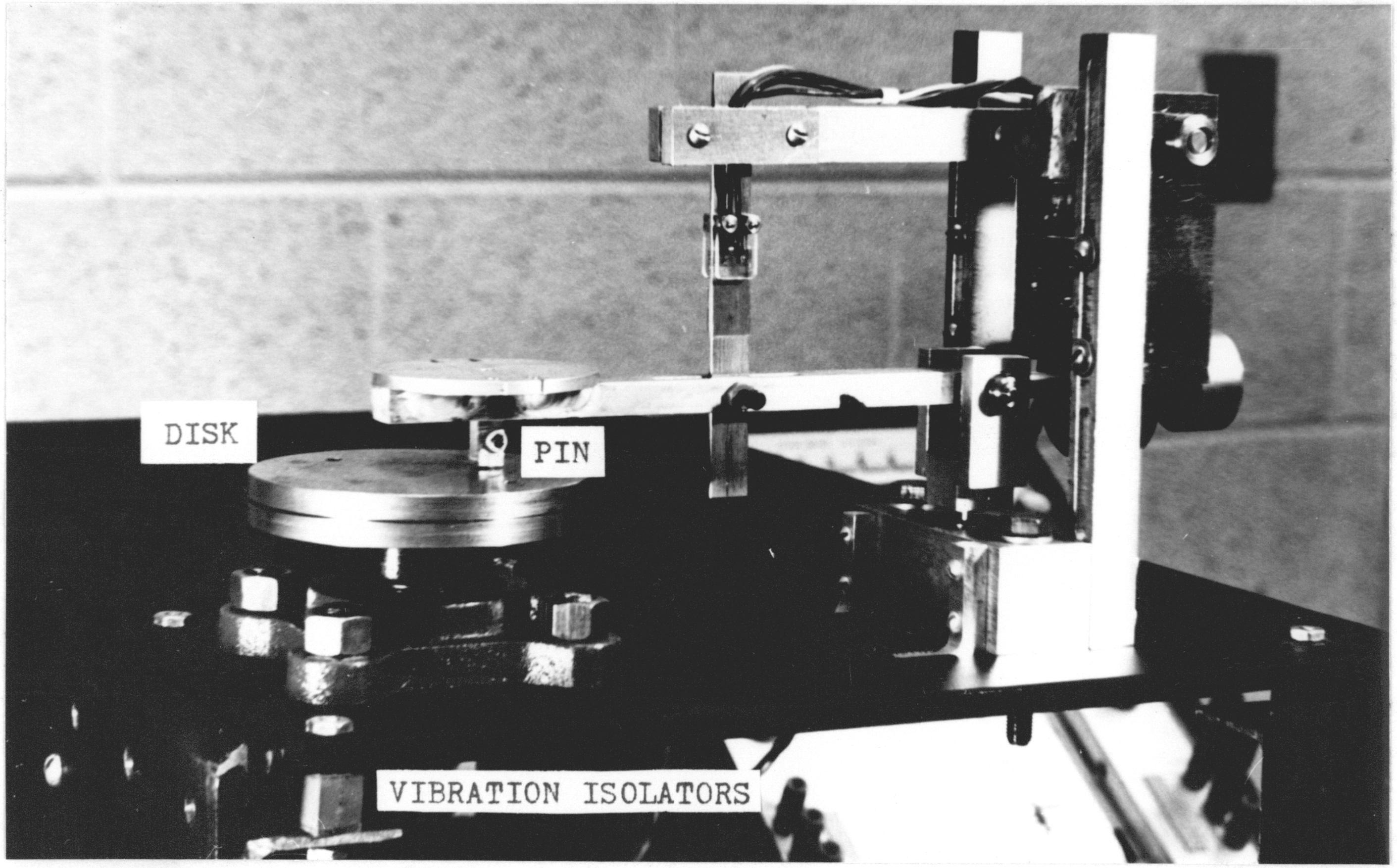


FIGURE 3. Pin-on-Disk Assembly

by placing a deadweight directly over the pin. The strain gages mounted on a cantilever beam which restrained the pin-support arm in the horizontal direction, produced an output voltage proportional to friction force. The strain gages were connected in a two arm bridge, one in tension and one in compression,for temperature compensation. Actual values for friction force were obtained after a direct deadweight calibration of the cantilever beam-pin-support arm assembly. The strain gage signal was amplified with a bridge amplifier and then used to drive an x-y plotter for the final output. As also can be seen in Fig. 3, the complete pin-on-disk assembly was isolated from drive train vibrations by mounting it on a plate supported by four vibration isolators.

The drive train (see Figs. 4, 5) consisted of a one-half horsepower ac motor driving a variable speed transmission. The transmission was coupled to the disk through one or two precision gear reducers (one shown is 100:1 ratio). The arrangement shown could impart from 0.4 to 4.0 rpm to the disk. With the pin adjusted to a 1.25 cm radius, this corresponded to a range of sliding speeds from 0.04 cm/sec to about 0.40 cm/sec. Vibration transmitted to the disk from the output shaft of the last precision gear reducer was reduced by a flexible coupling.

Sliding velocity was monitored using a variable reluctance transducer and a 60 tooth steel spur gear mounted to the output shaft of the variable speed transmission (see

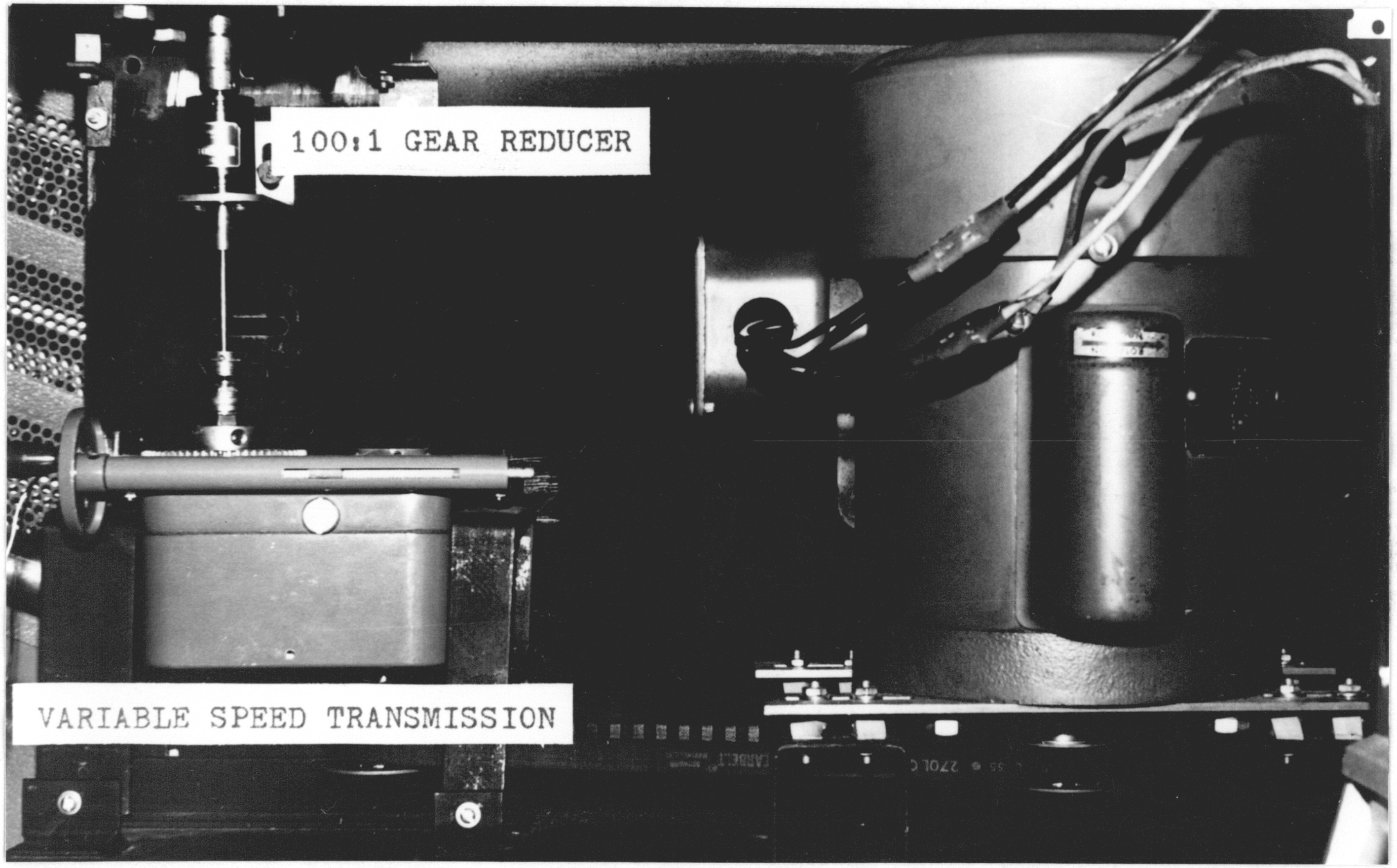


FIGURE 4. Apparatus Drive Train

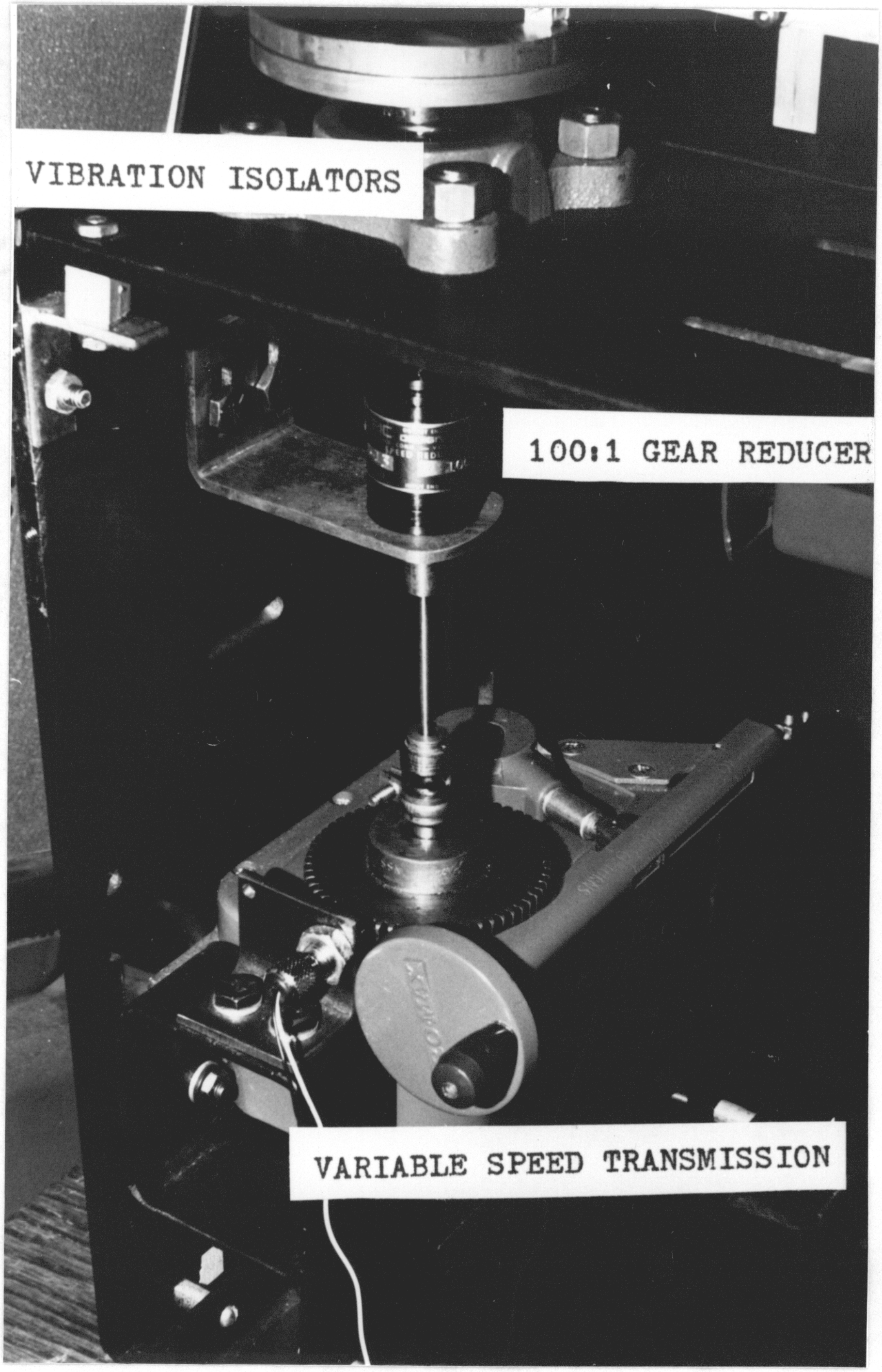


FIGURE 5. Speed Measurement System and Drive Train

Fig. 5). The signal from the transducer was counted and displayed as rpm by an electronic digital counter (see Fig. 2). Sliding velocities were then calculated knowing the final gear reduction and the wear track radius.

The entire friction and wear apparatus was isolated from room vibrations by three vibration isolators.

The radiation counting equipment is illustrated in Fig. 6 and schematically as a block diagram in Fig. 7. The detector was a Ge(Li) crystal (germanium semiconductor with dispersed lithium), stabilized using liquid nitrogen. A high voltage power supply imposed a 2500 volt potential across the crystal. Signals from the detector were immediately amplified with a preamplifier to overcome the line attenuation between the detector and the linear amplifier. The fully amplified signals were passed through a single channel analyzer adjusted to allow only signals corresponding to higher energy radiation to reach the multichannel analyzer. This effectively reduced the background counts that could actually become output. The multichannel analyzer stored the data which was subsequently fed through a digital converter for a digital output of the data recorded by an electric typewriter. Fluctuations in alternating current powering all of the counting equipment were eliminated using a constant voltage transformer. A list of the equipment appears in Appendix A.

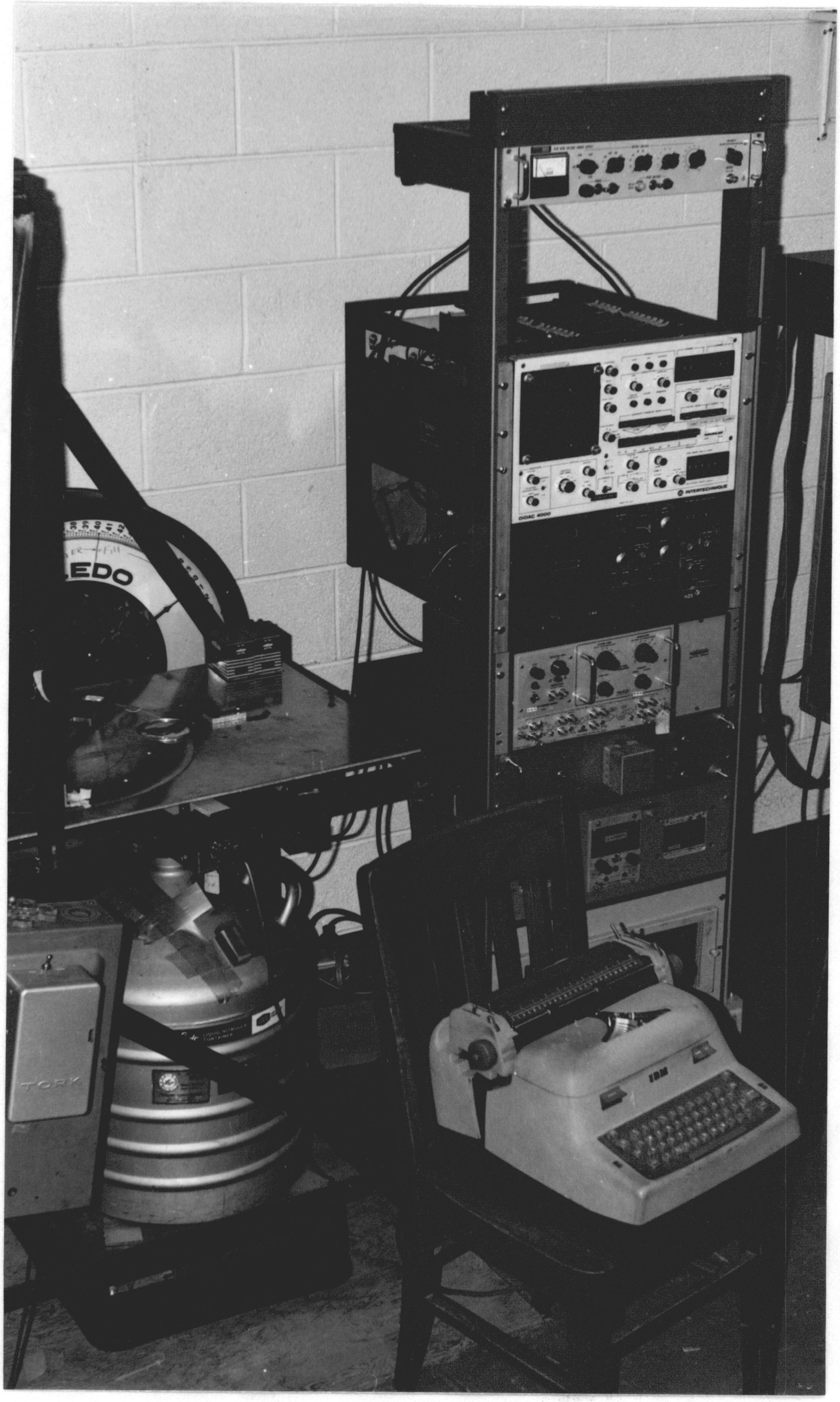


FIGURE 6. Radiation Counting Equipment

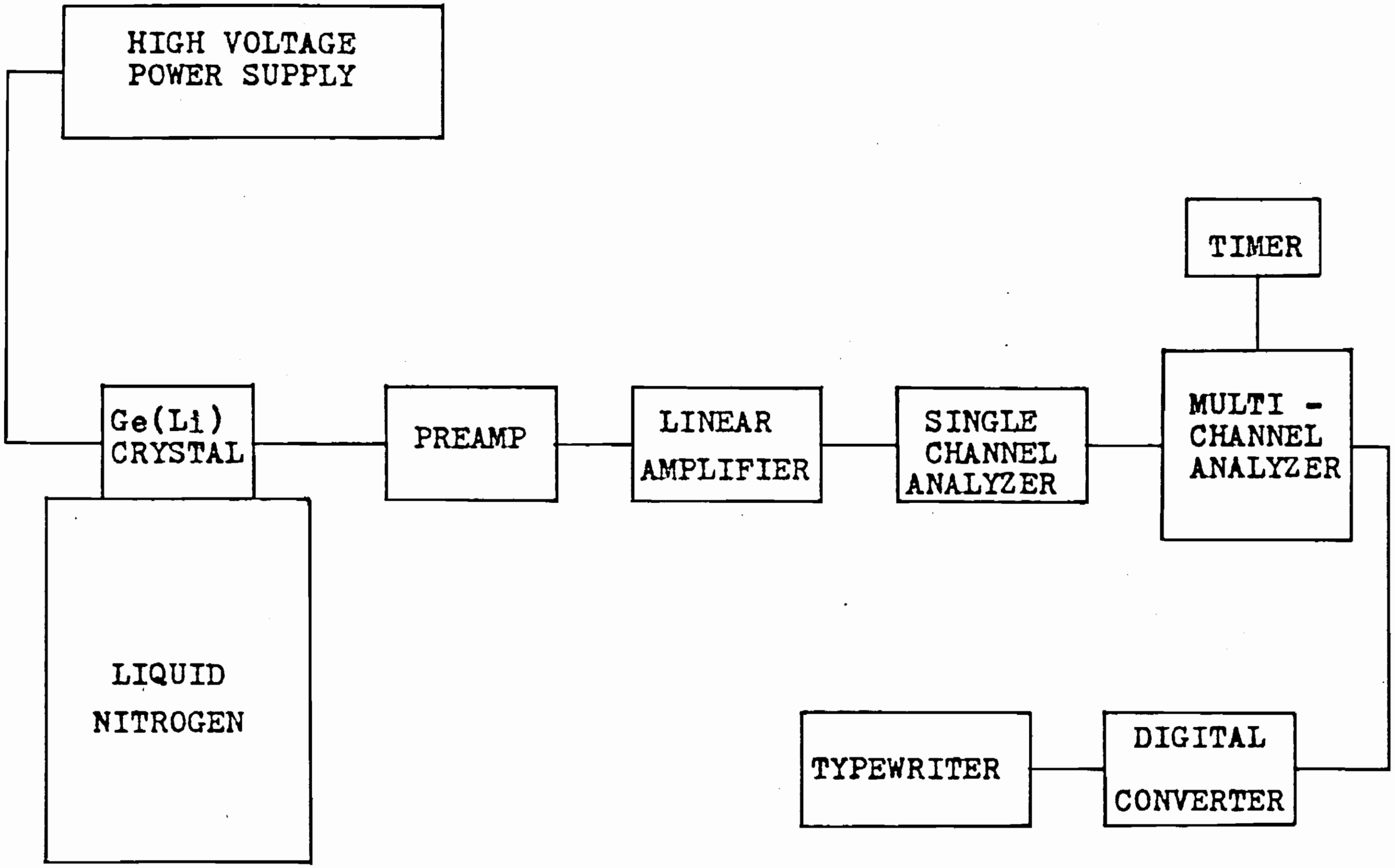


FIGURE 7. Block Diagram of Radiation Counting Equipment

B. Experimental Procedure

1) Steel Disk Preparation.

All of the steel disks were machined from a bar of 1018 cold rolled steel. The disks were ground on a surface grinder to a finish of approximately 0.38 µm - rms. Three different surface finishes were then produced by various methods. A finish of 0.10 µm - rms was produced by hand polishing with lapping compound. Polishing was performed so that the final abrasion marks would be unidirectional. Another surface finish studied was produced by unidirectional abrasion with emery paper (3M-fine) to a final finish of 0.81 µm - rms. The third topography was a random finish produced by air blasting with 0.0074 to 0.0147 cm glass beads to a final finish of 1.73 µm - rms.

All of the disk specimens were cleaned ultrasonically (plus occasional scrubbing with a cotton swab) for 30 minutes in methanol at room temperature. They were finally air dried and stored in a desiccator.

2) PCTFE Pin Preparation.

The polychlorotrifluoroethylene used for the study was manufactured by the 3M Company and fabricated by Chemplast Inc. The polymer pins were cut from an extruded rod (3.175 mm diameter) of PCTFE. The resulting geometry was a cylinder, 3.175 mm diameter by 3.175 mm long. The pin specimens were cleaned in pentane and then attached to a polymethylmethacrylate pinholder using Loctite quick-set adhesive (404). The pins were stored in polyethylene bags until used.

3) Preparation of the Apparatus for the Irradiated Pins.

Wear measurements were to be made after 1, 10, 20, and 40 revolutions of the disk. To eliminate variability between disks, each of these four experiments was performed on one disk. This was accomplished using a fresh pin positioned at a different radius on the disk for each experiment.

Before each experiment, a disk was mounted on the apparatus and the speed was adjusted to provide a sliding velocity of 0.20 cm/sec at the pin position. This velocity was chosen to stay below reported thresholds for frictional heating effects.

The normal load chosen for all of the experiments in this study was 1 kg. This load did not cause gross deformation of the PCTFE pins and resulted in measurable transfer.

Immediately preceeding the experiment the room temperature and relative humidity were recorded. The recorded temperatures were always 24 C ± 1 C, and the relative humidity was always 70 ± 1 percent.

4) Irradiation of PCTFE Pins.

The PCTFE pins were irradiated in the VPI & SU Research Reactor using the entry-exit system shown in Fig. 8. This was a pneumatic system using compressed nitrogen controlled by a series of electronic relays.

To use this system, the pin and pinholder assembly first had to be sealed in a small polyethylene vial. The small, sealed vials were then placed in larger polyethylene

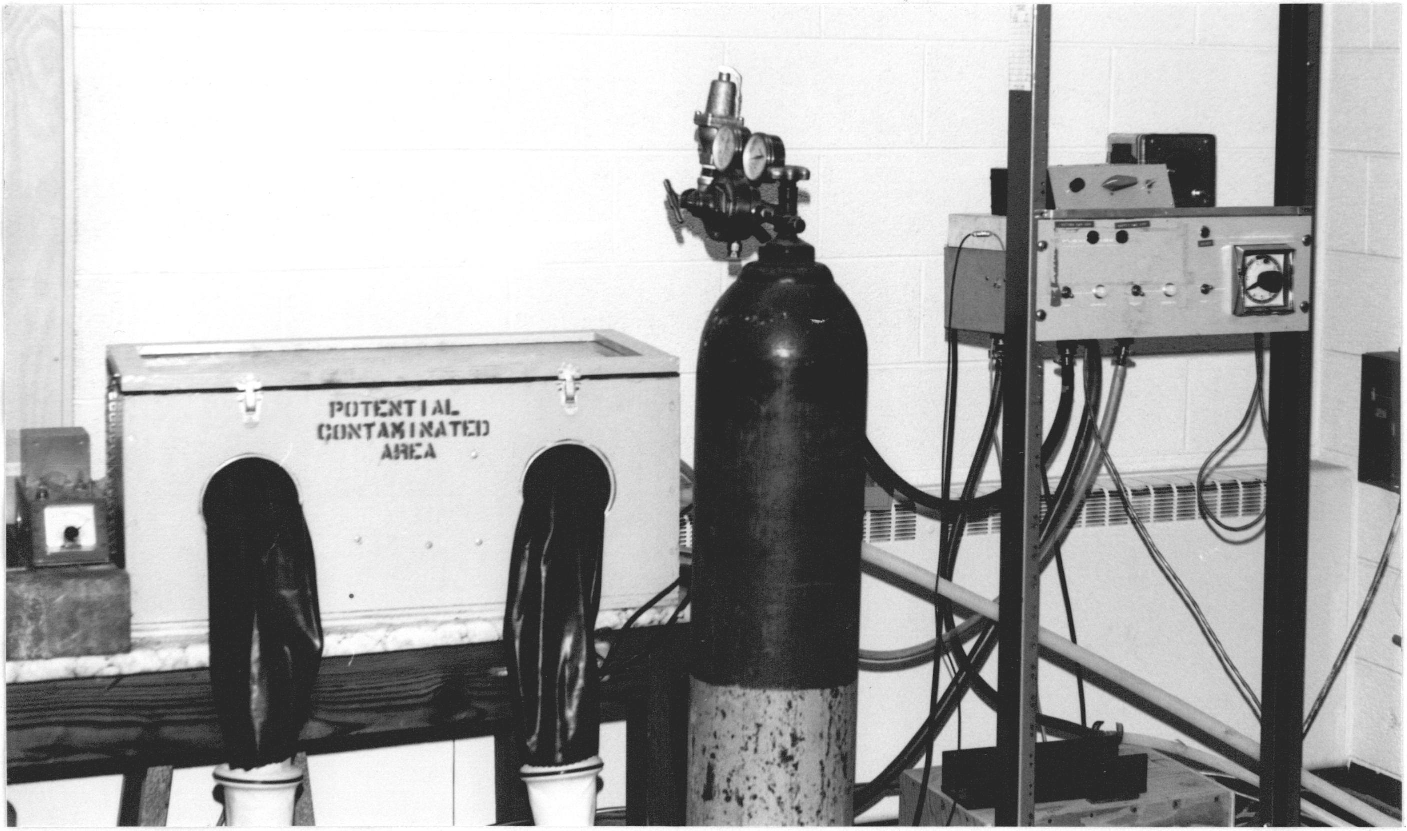


FIGURE 8. Reactor Entry-Exit System and Automatic Timer

capsules used to transport samples into and out of the reactor.

The pins were shot into the reactor and the entry time was recorded to the nearest second. The automatic timer system shown in Fig. 8 was used to return the specimens after exactly 10 minutes had elapsed. The 10-minute irradiation time was decided upon as a compromise considering sensitivity, the level of activity that could be safely worked with, and the problem of radiation damage of the polymer as discussed in the Review of Literature.

Better sensitivity could have been obtained by using longer irradiation times. However, the length of irradiation time was limited by the level of activity which could be safely handled. This level of activity was below threshold levels for radiation damage of the PCTFE.

The decay of ^{38}Cl results in both beta and gamma radiation but handling problems are encountered primarily because of the energetic gamma. The energy levels associated with the disintegrations and the frequency of occurrence expressed in (percent) of the disintegrations are as follows (from Stehn (14)):

BETA; 4.8 Mev (53)
1.1 Mev (31)
2.8 Mev (16)
and GAMMA; 2.1 Mev (47)
1.6 Mev (31).

The activity of the irradiated pin was measured after a 2-minute delay to allow the fluorine activity (11 second half-life) to decay. The activity of a typical pin, measured at approximately 10 cm from the monitor, was 250 milliroentgens/hr total (beta plus gamma) and 170 milliroentgens/hr gamma radiation.

5) Transfer of Polymer and Coefficient of Friction Measurement.

The pin and holder were removed from the small polyethylene vial and mounted in the friction and wear apparatus. The pin was positioned such that the axis of the cylindrical pin was parallel to the direction of sliding.

The normal load was then applied and the apparatus and x-y plotter were started simultaneously. Friction traces were obtained during passes (one pass equals one revolution of the disk) 1, 5, 10, 20, 30, and 40 of the 40 pass experiments; 1, 5, 10, and 20 of the 20 pass experiments; 1, 5, and 10 of the 10 pass experiments; and during the entire single pass experiments.

6) Counting Activity of the Transferred Polymer.

Immediately following the transfer of the polymer, the disks were removed from the apparatus and placed (transfer side down) on top of the Ge(Li) detector. The count was started and the time again recorded to the nearest second. The timer and multichannel analyzer were adjusted such that an integral count was taken for 1.8 seconds and then stored in one channel of the analyzer. This happened 999 times (filling 999 channels of the analyzer), resulting in an integral count (after summation) over a 30-minute time interval. This 30-minute integral count was typed out as a final output.

A 30-minute background count was taken once a day in a similar manner, except that a blank disk was placed on top of the detector. The procedures involved in reducing the counting data to quanitative wear data are presented in Appendix B and Appendix C.

7) Scanning Electron Microscopy.

Photographs of the resulting transfer and of scars on the PCTFE pins were obtained from a scanning electron microscope. An x-ray spectrometer was used to ascertain that deposits appearing in the photographs were actually PCTFE.

IV. DATA AND RESULTS

A. Wear Data

One measurement was obtained for each experiment discussed earlier. Presented in Table II are confidence intervals for each of these observations. A description of the calculation of these confidence limits is presented in Appendix B and Appendix C.

It should be noted that these confidence limits reflect uncertainty in the radiation counting and mass calibration procedures for each observation and do not represent expected limits for future, individual transfer measurements. The tabulated values are measurements of the mass of PCTFE transferred to the disk, after the indicated number of passes. The accuracy of the mass measurements was ± 0.3 μg. This was established from the largest standard error (due to counting and calibration uncertainty) associated with any mass measurement. This is also discussed in Appendix C.

The means of Table II (experimentally measured points) are plotted in Fig. 9. This illustrates the effects of both surface finish and the number of passes on the buildup of transferred PCTFE on the steel disks. These effects appear significant but any statistical significance cannot be substantiated without repeated experiments.

TABLE II. Confidence Intervals for Measured Transfer

PASSES	95% Confidence Interval (μg)		
Glass Bead Blasted (1.73 μm - rms)			
	Lower Limit	Mean	Upper Limit
1	7.5	8.0	8.3
5	15.5	16.1	16.5
10	17.7	18.4	18.9
20	19.8	20.5	21.0
40	24.0	24.8	25.4
Emery Abraded (0.81 μm - rms)			
	Lower Limit	Mean	Upper Limit
1	3.5	3.9	4.1
10	7.7	8.2	8.5
20	11.4	11.9	12.3
40	13.3	13.9	14.4
Polished (0.10 μm - rms)			
	Lower Limit	Mean	Upper Limit
1	- 0.1	0.3	0.5
10	0.0	0.4	0.6
20	0.6	1.0	1.2
40	1.7	2.1	2.3

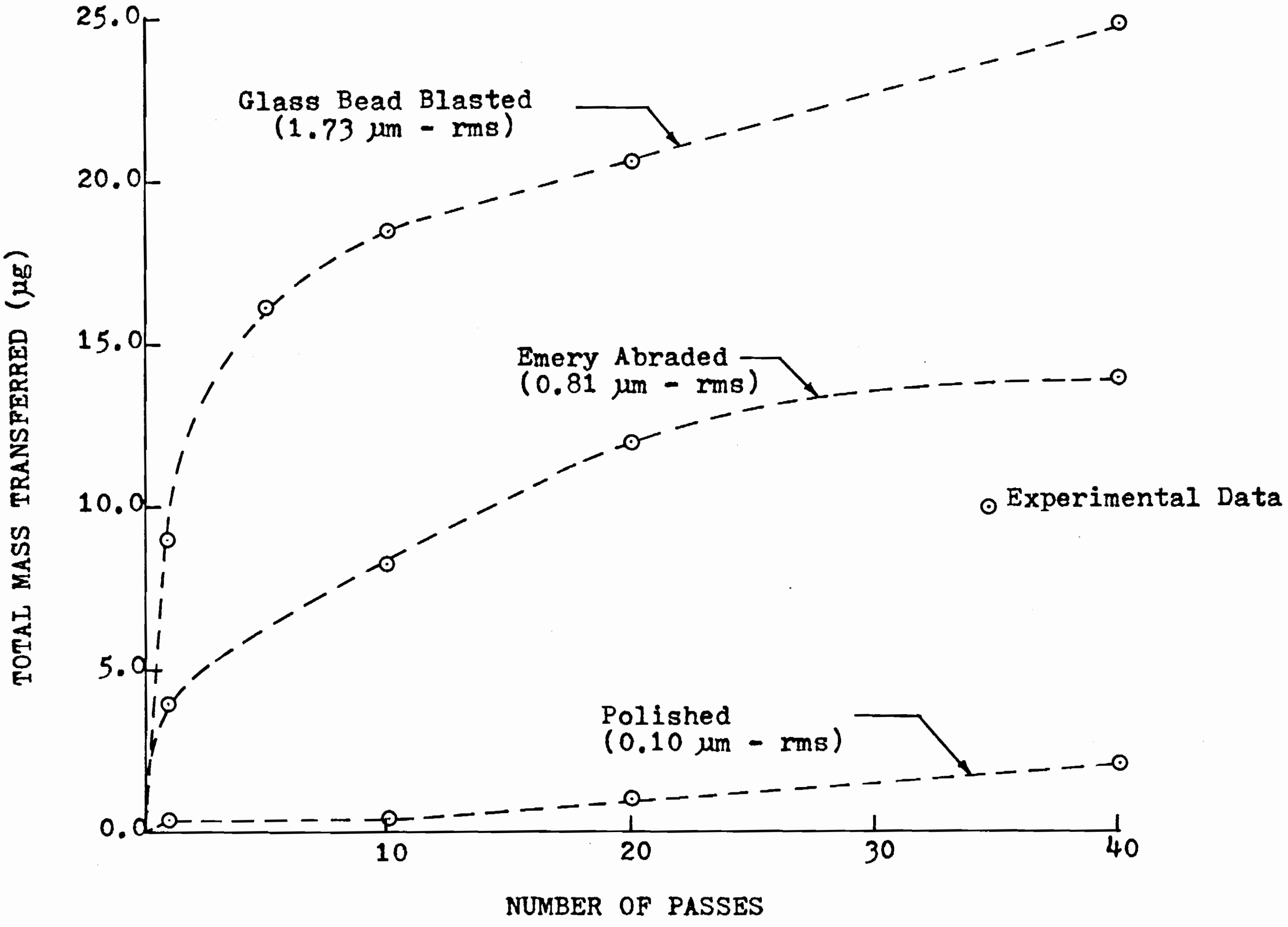


FIGURE 9. Effects of Surface Finish on Beginning Transfer

B. Friction Data

In general, observed kinetic coefficients of friction ranged from about 0.14 to 0.38, depending upon surface finish and the number of passes. This variation in coefficients existed only during the first pass of the pin. After 40 passes, all of the surfaces tended toward a kinetic coefficient of friction of 0.30.

Shown in Fig. 10 is a typical friction trace. This trace was obtained during the 20 pass experiment, run on a 0.10 µm - rms surface finish. As can be seen, the friction started out relatively low ($0.14 \leq \mu_k \leq 0.19$) and after repeated passes increased considerably (during the twentieth pass: $0.29 \leq \mu_k \leq 0.33$). This trace also shows that stick-slip did not occur until the fifteenth pass, and then became more pronounced (greater amplitude) as more PCTFE was transferred.

This gradual increase in friction was also observed with the 0.81 µm - rms surface finish. The only differences were that the friction started out higher, and that some stick-slip existed even during the first pass. With both of these surfaces, the friction was higher while the pin was traveling perpendicular to the abrasion scratches than during sliding parallel to the scratches. This accounts for the sine-wave appearance of the trace in Fig. 10.

The frictional characteristics observed during experiments with the glass bead blasted 1.73 µm - rms finish were

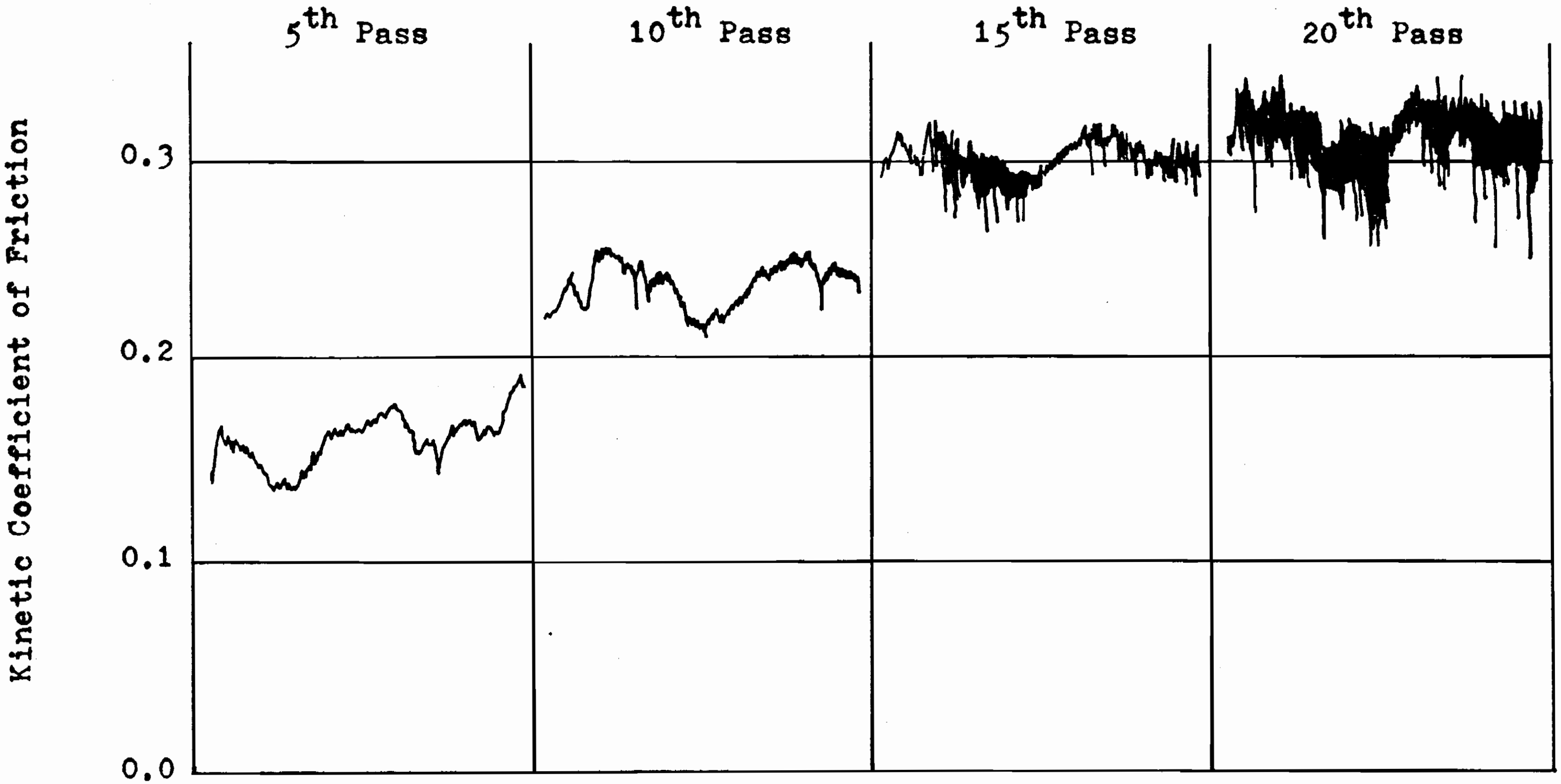


FIGURE 10. Actual Friction Trace of Repeated Traversals on a 0.10 μm - rms Surface

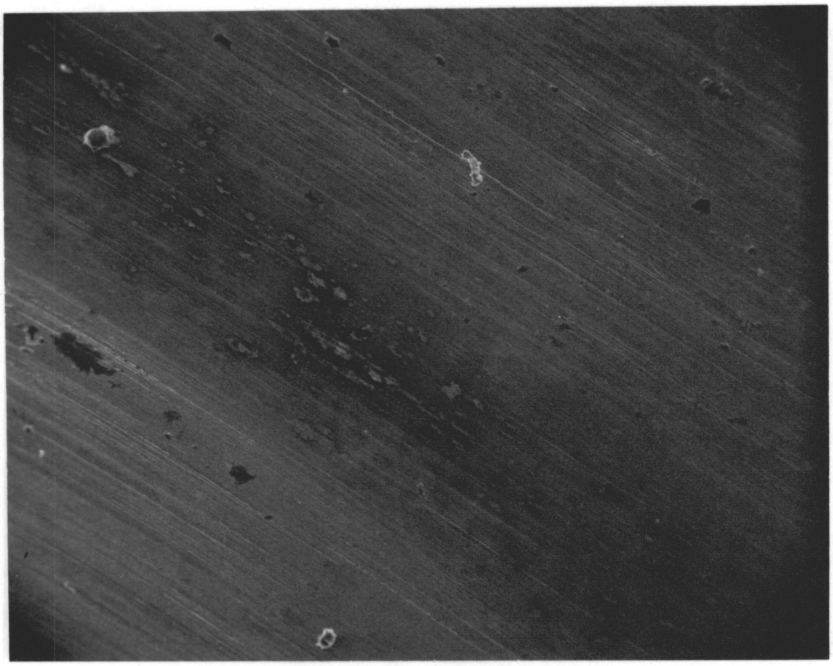
somewhat different than those described above. In this case, the beginning friction was the highest observed ($\mu_k = 0.38$ during the first pass). The most pronounced difference with this finish was that with repeated traversals the kinetic coefficient of friction decreased slightly (from 0.38 to 0.30 after the fourtieth pass). Another obvious difference was that the sine-wave appearance was not obtained due to the random, glass bead blasted surface.

C. Scanning Electron Microscopy

The photographs obtained from the scanning electron microscope provided much information which was very useful in gaining an understanding of the friction and transfer characteristics of the PCTFE steel combination.

Figure 11 shows the difference between the transfer obtained, after 40 passes, sliding parallel and then perpendicular to the abrasion scratches. This shows that the high friction observed during sliding perpendicular to the lay of the surface was accompanied by higher transfer also. Both of these photographs were obtained at 140x magnification.

Shown in Fig. 12 are photographs of multiple pass experiments, run first on the 0.81 μm - rms surface and then, for comparison, on the 0.10 μm - rms surface. In the top photograph, the visible tracks are, from left to right, the 10, 20, and 40 pass experiments run on the 0.81 μm - rms surface. The bottom picture shows a 40 pass experiment on the left and

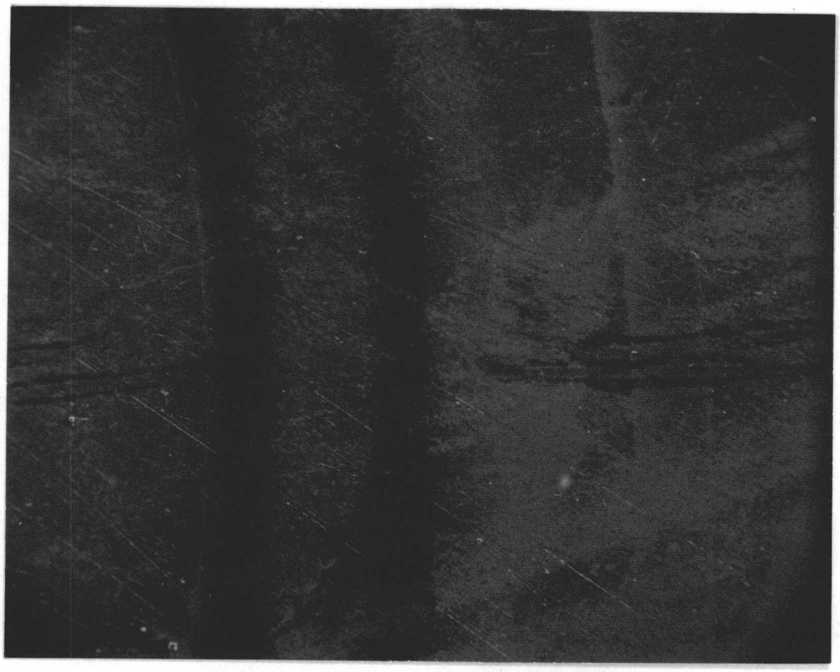


PARALLEL SLIDING

PERPENDICULAR SLIDING

FIGURE 11. Effects of Sliding Perpendicular and Parallel to the Machining Marks (0.81 μm - rms Surface, 140x Magnification)

0.81 µm - rms SURFACE



0.10 µm - rms SURFACE

FIGURE 12. Effects of Surface Finish on Transfer (35x magnification)

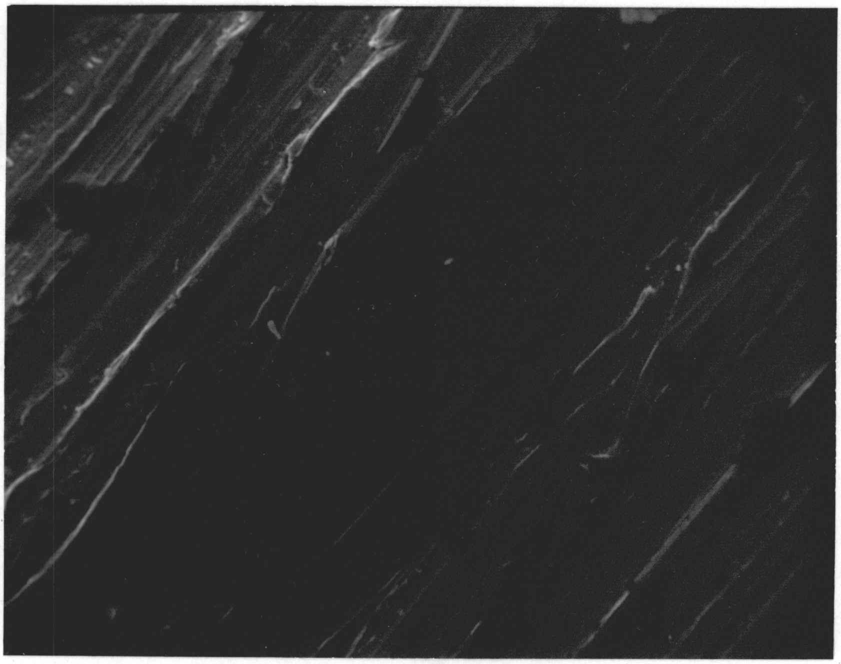
a 20 pass experiment on the right; both on a 0.10 µm - rms surface finish. Both of these photographs were taken at a 35x magnification. In this comparison it can be seen that bulk transfer rather than film transfer has occurred for both surfaces, and that polymer is much more prevalent on the rougher surface.

A similar comparison is presented in Fig. 13. These pictures were both obtained at 700x magnification after 20 pass experiments. The areas appearing in both photographs were within regions where sliding was perpendicular to the abrasion scratches. The top photograph shows a very large PCTFE deposit present on the 0.81 µm - rms surface. The bottom photograph illustrates that the transfer to the 0.10 µm - rms surface occurred in much smaller deposits.

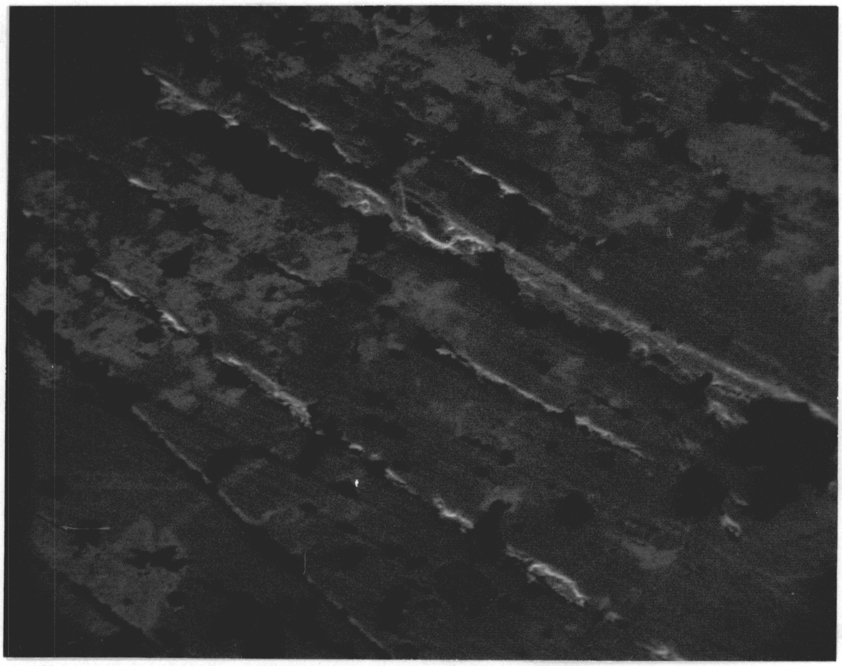
X-ray spectrographs, taken in areas of the two photographs in Fig. 13 where polymer deposits are apparent, are shown in Fig. 14. The two peaks present in each spectrograph correspond to chlorine (small; on the left) and iron (large; on the right). The presence of PCTFE is substantiated by the chlorine peak which appears in both x-ray spectrographs.

Shown in Fig. 15 are pictures of the wear scar on the PCTFE pin resulting from the 20 pass experiment on the 0.81 µm - rms surface. The amount of transfer measured after this experiment was 11.9 µg.

For comparison, Fig. 16 shows the results of 20 passes over the 0.10 µm - rms surface finish. The transfer measured after this experiment was 1.0 µg.

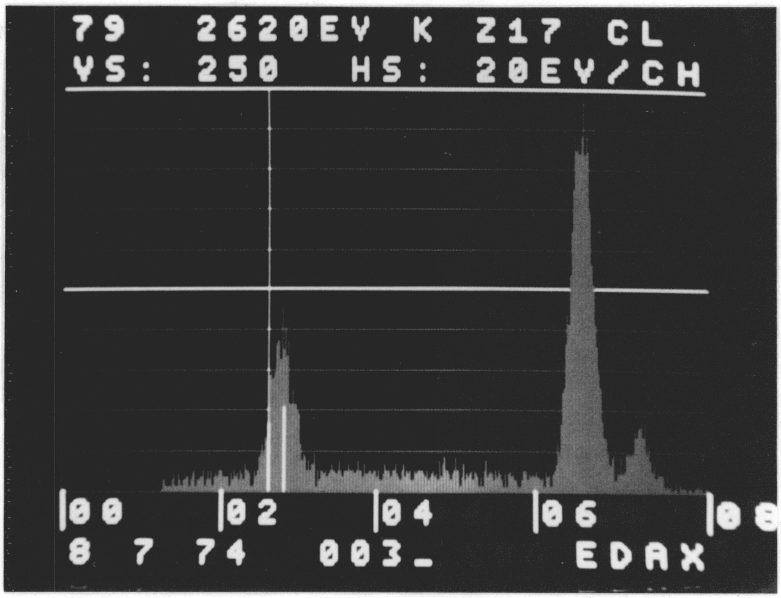


0.81 µm - rms SURFACE

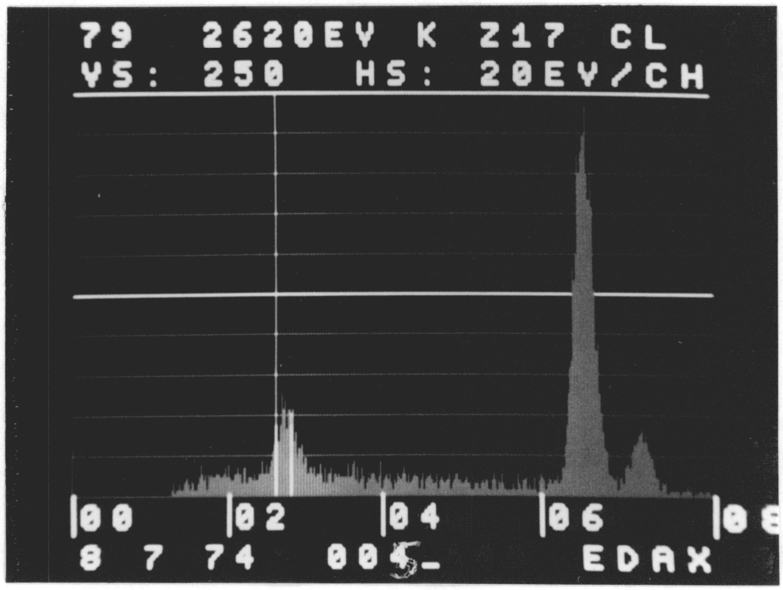


0.10 µm - rms SURFACE

FIGURE 13. Effects of Surface Finish on Transfer (700x magnification)



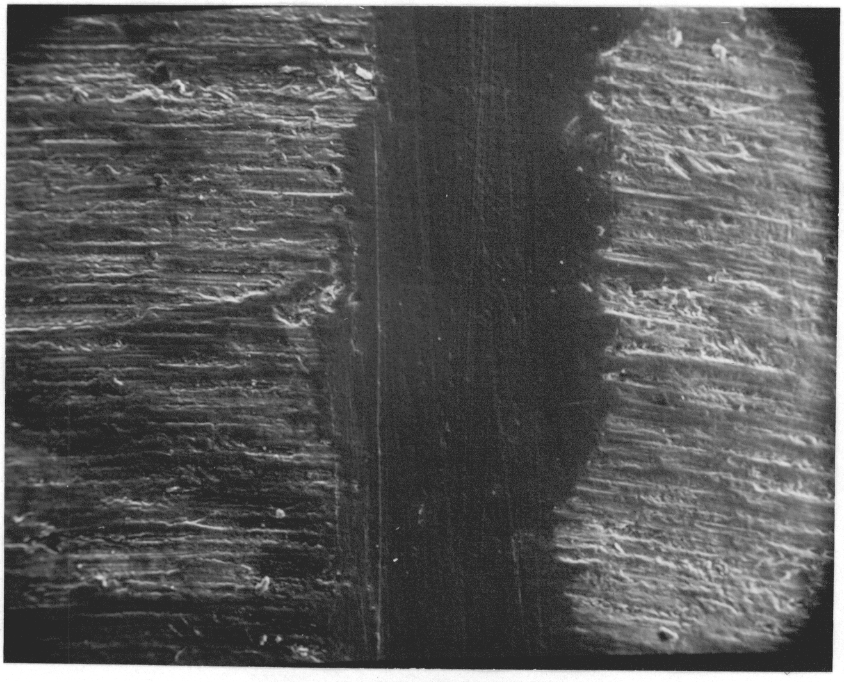
0.81 µm - rms SURFACE



0.10 µm - rms SURFACE

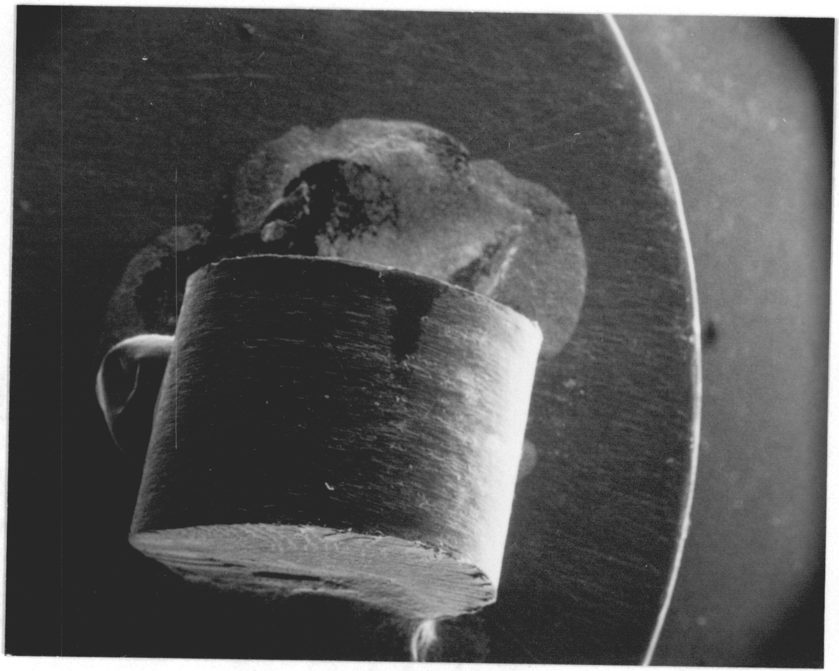
FIGURE 14. X-Ray Spectrographs of Transferred Material

19x MAGNIFICATION



95x MAGNIFICATION

FIGURE 15. PCTFE Pin After 20 Passes on 0.81 μm - rms Surface



19x MAGNIFICATION

95x MAGNIFICATION

FIGURE 16. PCTFE Pin After 20 Passes on 0.10 μm - rms Surface

V. DISCUSSION OF RESULTS

All of the data obtained indicate that surface topography cannot be ignored when considering the friction and transfer characteristics of the PCTFE-mild steel interface. One aspect of the data that tends to obscure the effect of measured surface finish alone, is the fact that the 1.73 µm - rms finish was a random surface and the 0.81 µm - rms was a unidirectionally abraded surface. As evidenced by Fig. 11, the amount of transfer to the 0.81 µm - rms surface is a function of the relationship between sliding direction and the machining marks. For this reason, one should not try to compare results between surface finishes, such as abraded and glass bead blasted, without considering the effect of the lay or randomness of the surface.

Although more data was needed before statistically valid conclusions could be drawn, the data collected in this study did illustrate the efficacy of the radio-tracer technique. Two major advantages became apparent while using this technique. First, the only part of the procedure where extreme care had to be taken to avoid errors due to moisture absorption and dust pickup was during the calibration. This is in sharp contrast to the techniques discussed in the Review of Literature where the above factors are a constant source of uncertainty. The other major advantage was the sensitivity of the radio-tracer technique. The minimum detection limit with this method was 0.3 µg. This is significantly lower than the 2.0 µg limit reported by Dowson, Atkinson, and Brown (1) using gravimetric methods.

The results of the friction measurements were also interesting. As reported earlier, all of the coefficients of friction approached 0.30 after repeated traversals. This is in good agreement with results reported by Pooley and Tabor (7), and Bowers, Clinton, and Zisman (14). The friction results obtained in the transition region (from 0 to 40 passes) with the 1.73 µm - rms surface seem to support the work of Hollander and Lancaster (12) who concluded that the transfer of polymer could decrease friction and transfer by effectively smoothing a very rough surface.

Results obtained with the polished surface indicate that transfer actually increased subsequent friction and transfer. It is felt that initial transfer to the polished disk was predominantly abrasive and that as transfer built up, the wear mode shifted to adhesion (polymer to polymer). The increase in friction and the aggravation of stick-slip, which was most pronounced with the 0.10 µm - rms surface (see Fig. 10), support this idea. The increase in transfer mentioned above is illustrated in Fig. 9.

Results from the scanning electron microscope and the measurements of transfer to the rougher (0.81 µm - rms) surface indicate that abrasive transfer remains predominant for more passes than was observed with the polished surface. The results shown in Fig. 11 illustrate that abrasion was still the major mode of wear after 20 passes on the 0.81 µm - rms surface. Transfer also aggravated stick-slip with this surface. This is a possible indication that a shift in the wear mode might be observed after many passes on the rougher surfaces.

VI. CONCLUSIONS AND RECOMMENDATIONS

This study has shown that radio-tracer methods are very effective tools for the study of the wear properties of PCTFE. The sensitivity and ease with which this technique can be used are the major advantages of this wear measurement technique.

The data obtained indicate that bulk transfer does occur when PCTFE slides on mild steel, even at room temperature (approximately 24 C), somewhat contrary to the reported results of Pooley and Tabor (6).

The quantitative transfer data, along with the photographs from the scanning electron microscope, illustrate that the surface topography can control the mode of wear (either abrasive or adhesive) during the early stages of transfer. It was concluded that beginning transfer occurred due to abrasion, regardless of the surface finish (for the three studied), and that subsequent wear could be attributed to adhesion. The rougher surfaces prolonged the transition from abrasive to adhesive wear.

From a design standpoint, observations made in this study indicate that for very smooth surfaces, the transfer of PCTFE to mild steel is not beneficial, due to an accompanying increase in friction and transfer.

It is recommended that further quantitative data be taken, using the procedures established in this study, on the transfer of PCTFE as a function of surface topography. This could result in conclusive evidence of the effect of surface finish on the transfer of PCTFE sliding on mild steel.

As an improvement in the technique, it is recommended that more calibration samples be used to establish the calibration curve. This would reduce the uncertainty in the calibration procedure considerably and therefore result in much more accurate data with little added effort. The resulting increase in sensitivity could also be used to expand the capabilities of the technique. In this same area, added shielding around the Ge(Li) crystal would reduce uncertainty in the counting procedure (by reduction in background) and therefore improve the results.

It should also be noted that this radio-tracer technique could be applied to the study of polymers other than PCTFE. This would be possible by doping the polymers with some readily activatable element. Suitable tracer elements might be elements such as samarium or dysprosium which become very radioactive with short exposures. The resulting activity is of much lower energy than the chlorine gamma and therefore easier to handle.

Studies such as those recommended above could lead to a better understanding of the transfer properties of many polymers.

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VIII. ACKNOWLEDGMENTS

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In the actual preparation of this thesis, Miss Janet Mabie is to be thanked for her patience while typing this thesis in its final form.

In addition to those that aided in the actual research and preparation of this thesis, the author would like to thank the Army Research Office - Durham for the grant that made this study possible.

The author is also deeply indebted to his parents and fiancé for their encouragement and support during both his undergraduate and graduate studies.

IX. APPENDIX A

List of Equipment

1) High Voltage Power Supply, Model 415B, Ser. No. 1722, Fluke Company.
2) Multichannel Analyzer, Model DIDAC 4000, Ser. No. 1209, Intertechnique Inst. Inc.
3) Timer, Model H23, Ser. No. 1253, Intertechnique Inst. Inc.
4) Single Channel Analyzer, Model C44, Ser. No. 109, Intertechnique Inst. Inc.
5) Linear Amplifier, Model TC 200, Ser. No. 630, Tennelec.
6) Digital Converter, Model 70, Ser. No. 180, Packard.
7) Regulated Power Supply, Model LN-130, Ser. No. A 1199, Lambda.
8) Regulated Power Supply, Model LM 260, Ser. No. C7672 and C7608, Lambda.
9) Constant Voltage Transformer, Model CVH, Ser. No. A 72, Sola.
10) Preamplifier, Model 120-3F, Ser. No. 520, Ortec.
11) Ge(Li) Crystal, Harshaw Chemical Company.
12) Electronic Counter, Model 5223L, Ser. No. 539-00682, Hewlett-Packard Company.
13) Oscilloscope, Type 564, Ser. No. 004993, Tektronix, Inc.
14) Time Base, Type 2B67, Ser. No. 014168, Tektronix, Inc.
15) Carrier-Bridge Amplifier, Type 3C66, Ser. No. 001557, Tektronix, Inc.

16) X-Y Plotter, Model 2D, Ser. No. 1217, F. L. Moseley Company.

17) Variable Speed Transmission, Model E2, Zero-Max Company.

18) Speed Reducer (100:1), Cat. No. U2-13, Pic Design Corp.

19) $\frac{1}{2}$ HP Motor, Type CS, Ser. No. 406871-LQ, Eastern Industries.

X. APPENDIX B

Counting Data Reduction

The experimental observations required to obtain the mass transferred during any one experiment were:

1) Beginning irradiation time (T_o).
2) A 30-minute integral count of activity transferred to the disk (N_2).
3) Time that the above count was started (T_2).
4) A 30-minute integral background count (N_B).
5) The integral count of the previous experiment (activity existing on the disk = N_1).
6) The time that the count for the previous experiment was started (T_1).
7) The radii that the pins were positioned on the disk for both experiments.

A decay constant, λ, for the activity of the ^{38}Cl was also needed in the data reduction. It was determined from repeated countings of a single source (at different delay times out of the reactor) that zero-time equivalent counts were most repeatable when $\lambda = 0.018347 \text{ min}^{-1}$. This corresponds to a calculated half-life of 37.78 min rather than the 37.2 minutes expected for the chlorine decay. The cause of this discrepancy was never discovered, but repeated countings showed that the decay constant chosen was necessary for good repeatable time transformations of the data.

The first analytical step in the reduction of the data was to calculate confidence limits for the experimentally observed number of counts which were obtained in each experiment. The observed number of counts per channel (integral count/999 channels) was assumed to be the true mean of the Poisson distribution formed by the 999 recorded samples of the counts per channel. The fact that there were 999 observations of this occurence made this a good assumption. With this many observations, the Poisson distribution can be approximated by the normal distribution. From Brownlee (15), the basic form of the confidence interval for the mean of this distribution is:

$$M_E - \sqrt{\frac{Var}{n}} \cdot U_{1-\frac{\alpha}{2}} \leq M \leq M_E + \sqrt{\frac{Var}{n}} \cdot U_{1-\frac{\alpha}{2}}$$

where M = Expected mean

M_E = Estimate of the expected mean

n = Number of observations (999)

Var = Variance (of the Poisson distribution in this case)

α = Chosen level of significance

$U_{1-\frac{\alpha}{2}}$ = Corresponding percentage point of the normal distribution.

The estimated expected mean is of the form:

$$M_E = \left(\frac{N_2 - N_B}{999}\right) - \left(\frac{N_1 - N_B}{999}\right)e^{-\lambda T}$$

where N_2, N_1, N_B, and λ are as defined in terms of the experiment, and T represents the amount of time that the previously transferred film had been decaying ($T = T_2 - T_1$). This equation includes two necessary corrections to the experimentally observed integral count N_2. First, the background counts have been subtracted, and second, the existing activity (from the previous experiment) has been brought forward in time ($e^{-\lambda T}$), and then subtracted.

Now, letting:

$$\frac{N_2}{999} = \dot{N}_2$$

$$\frac{N_1}{999} = \dot{N}_1$$

and $$\frac{N_B}{999} = \dot{N}_B, \text{ and}$$

rearranging, the expression above becomes:

$$\dot{N}_2 - \dot{N}_1 e^{-\lambda T} - (1 - e^{-\lambda T})\dot{N}_B = M_E$$

Again, this is the estimate for the expected mean (average counts per channel). An expression for the variance corresponding to this mean was then found. For a Poisson distribution, the variance also equals the expected mean. The only changes in the expression above involve the coefficients. Noting that $e^{-\lambda T}$ is a constant and not a random variable, the variance becomes:

$$\mathrm{Var} = \dot{N}_2 + (e^{-\lambda T})^2 \dot{N}_1 + (1 - e^{-\lambda T})^2 \dot{N}_B$$

Using these expressions for M_E and Var and the basic form of the confidence limit given earlier, 95 percent ($\alpha = .05$) confidence limits were constructed for all of the data. The lower limit, mean, and upper limit of each interval were then multiplied by 999 to obtain a confidence interval corresponding to a 30-minute integral count.

These limits were then all transformed to values at zero-time (T_o) so that all data, regardless of differences in delay time, could be compared. This was accomplished with the exponential decay function (inverted):

$$N_o = Ne^{+\lambda(T_2 - T_o)}$$

where N_o = Number of counts at zero-time (T_o)

N = Number of counts at time (T_2)

$\lambda = 0.018347 \text{ min}^{-1}$

The next step in the reduction of data was necessary because the Ge(Li) crystal used in the counting apparatus was sensitive to the radial position of the radioactive material on the crystal surface. This geometry factor caused less counts to be recorded the further that the radioactive source was from the center of the top of the counter. The data had to be corrected for this geometry effect because the four wear tracks on each disk had different radii.

To account for this effect, a curve describing the geometry effect was experimentally determined (see Fig. 17). The percentage change, either plus or minus, caused by running

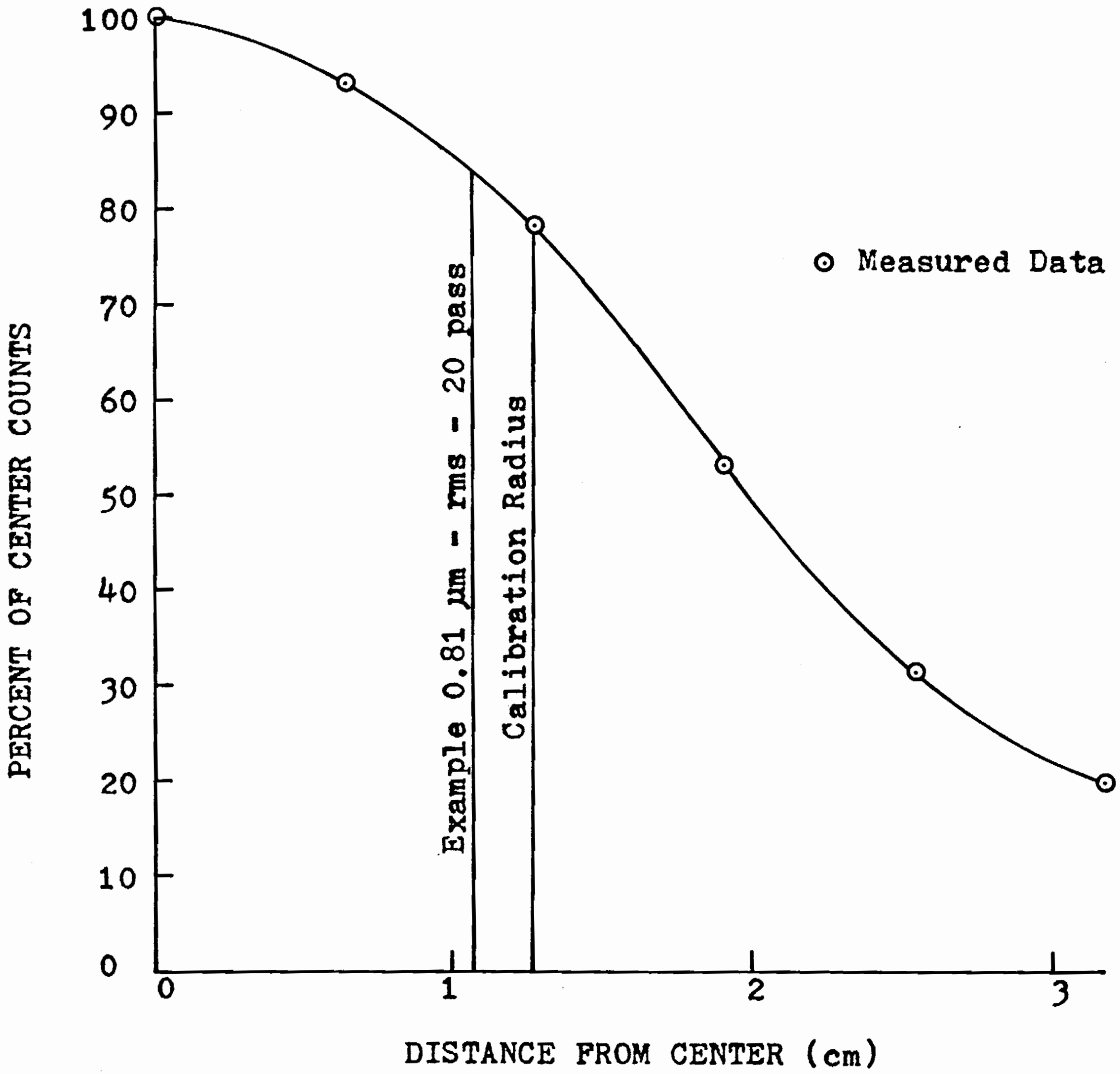

FIGURE 17. Geometry Effects on Ge(Li) Crystal

an experiment away from a "normal" radius (at which calibration was performed) was then obtained from this plot. Figure 17 shows the positions of the normal radius and the radius of the wear track resulting from the 20 pass experiment on the 0.81 µm - rms surface. The mean and upper and lower limits of the "zero-time" confidence interval were then multiplied by the appropriate factor to normalize the data with respect to the counting radius. These confidence limits are presented in Table III.

At this point in the data reduction, the number of counts were related to equivalent mass using the mass calibration described in Appendix C.

TABLE III. Confidence Limits for Time Corrected, Normalized Data

PASSES	95% Confidence Interval (counts - 30 min)		
Glass Bead Blasted (1.73 µm - rms)			
	Lower Limit	Mean	Upper Limit
1	86,528	87,269	88,010
5	175,143	176,242	177,340
10	200,465	201,617	202,769
20	223,218	224,514	225,812
40	270,209	271,835	273,458
Emery Abraded (0.81 µm - rms)			
	Lower Limit	Mean	Upper Limit
1	41,896	42,425	42,953
10	88,777	89,543	90,309
20	129,548	130,655	131,761
40	151,334	152,597	153,861
Polished (0.10 µm - rms)			
	Lower Limit	Mean	Upper Limit
1	2,554	2,745	2,934
10	3,972	4,213	4,454
20	10,164	10,522	10,880
40	22,167	22,793	23,420

XI. APPENDIX C

Calibration for Wear Data

A direct calibration, using known masses of PCTFE, was obtained to relate the number of counts to an equivalent mass. Seven particles of PCTFE (obtained from the same stock as the pins) were first cleaned in pentane and then weighed to the nearest 0.1 µg with a microbalance. Care was taken in the handling of these standard specimens to protect against chlorine contamination.

These samples were then irradiated for 10 minutes with the appropriate times being recorded just as in the experiments. The irradiated samples were then taped to a disk at the "normal" radius to simulate a transferred film of PCTFE. Each sample was counted for 30 minutes and the resulting integral counts were returned to zero-time using the exponential relationship given in Appendix B. The results of this procedure are presented in Table IV.

A linear regression, forced through the point (mass = 0, counts = 0) was then performed on the data. The slope of the regression line was calculated from the expression:

$$b = \frac{\sum_i X_i Y_i}{\sum_i X_i^2}$$

where X_i = The i^{th} mass (µg)

Y_i = The "zero-time" integral count (the random variable) corresponding to the i^{th} mass.

TABLE IV. Mass Calibration Data

Mass (µg)	"Zero-time" Integral Counts
16.2	200,699
27.6	302,548
68.0	742,374
120.0	1,372,312
164.6	1,751,564
212.5	2,388,720
248.7	2,671,224

The inverse slope obtained in this manner was $b' = 9.137 \times 10^{-5}$ µg/count. The experimental data from Table IV and the regression line are shown in Fig. 18. Confidence limits were then calculated for an indication of the uncertainty involved in the process of using the regression line in reverse (as is commonly done with calibration schemes). Brownlee (15) gives the following expression for the lower limit of the mass obtained by using the regression line in reverse:

$$LL = \frac{b\,Y'}{A} - \frac{t_{1-\frac{\alpha}{2}}(S)}{A} \cdot \left[\frac{A}{m} + \frac{(Y')^2}{\sum_i X_i^2}\right]^{\frac{1}{2}},$$

where LL = Lower confidence limit.

$$A = b^2 - \frac{\left[t_{1-\frac{\alpha}{2}}\right]^2 \cdot S^2}{\sum_i X_i^2}$$

b = Slope obtained for the regression

$t_{1-\frac{\alpha}{2}}$ = $1 - \alpha/2$ percentage point of the t distribution

m = Number of times that the new observation was measured. ($m = 999$)

Y' = Newly observed number of counts (results from data reduction of Appendix B)

S^2 = Error mean square from the regression.

Also, $$S^2 = \frac{\sum_i Y_i^2 - \left(\sum_i X_i\, Y_i\right)^2 / \sum_i X_i^2}{k - 1}$$

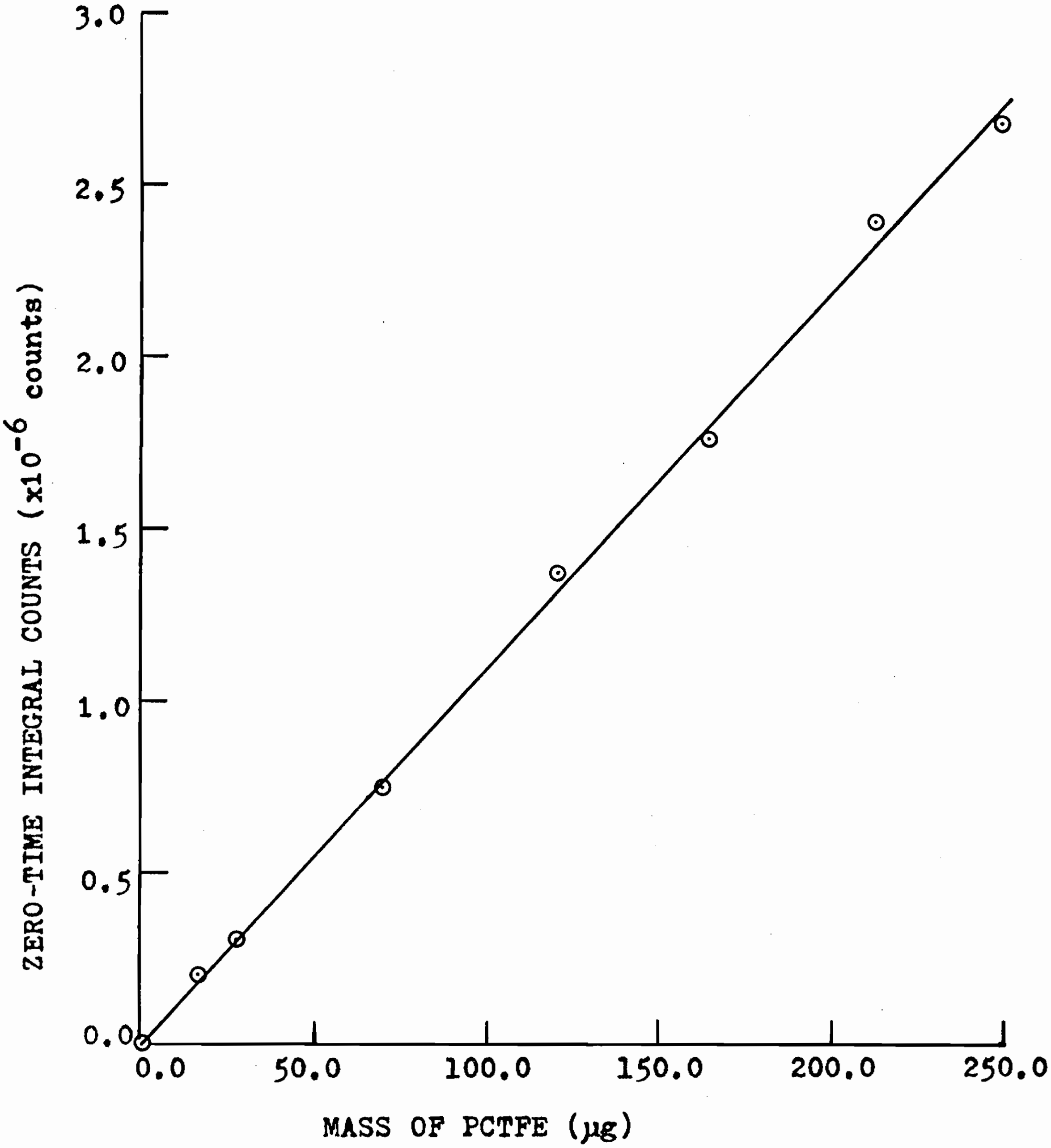

FIGURE 18. Regression Line For Mass Calibration

where k = number of points included in the regression, and X_i and Y_i are as originally defined for the regression. The upper confidence limit can then be obtained by substituting the $t_{\frac{\alpha}{2}}$ percentage point for the $t_{1-\frac{\alpha}{2}}$ percentage point in every place that it appears.

This procedure was used to calculate a 95 percent confidence limit for each of the limits presented in Table IV. The final data, reported in Table II, consisted of means of each measurement, and the extremes of each observation. These extremes reflected the uncertainty involved in the counting and the calibration procedure.

The accuracy, or the minimum mass which could be measured with the same level of significance as all of the data, was calculated with an iterative procedure. The accuracy is limited by the uncertainty in both the counting and the calibration procedures.

It was decided that the lower limit would be reached when the measured mass was small enough to make the standard error (after both confidence limit calculation procedures had been applied) equal to the observed mass. This estimate of the accuracy was made conservative by using the largest recorded level of background activity and therefore the largest error contribution due to background. It was found that with a 30-minute background count equal to 3000 and a measured mass of 0.3 μg, the standard error of the measurement was also 0.3 μg. Therefore 0.3 μg is the smallest mass which could be resolved with rigid statistical confidence.

XII. VITA

The author was born on May 9, 1950 in Baltimore, Maryland, son of Mr. and Mrs. Richard D. Doolittle. He attended and was graduated from Baltimore County public schools.

In September of 1968 he enrolled in Essex Community College also of Baltimore County, Maryland. In September 1970 he transferred to Virginia Polytechnic Institute and State University. While an undergraduate he became a member of the American Society of Mechanical Engineers and Pi Tau Sigma. He was graduated with the Bachelor of Science Degree in Mechanical Engineering in June of 1973.

He began graduate study in Mechanical Engineering at Virginia Polytechnic Institute and State University in September of 1973. This thesis is the final requirement of that study to obtain the Master of Science Degree.

Stephen D. Doolittle

A RADIO-TRACER STUDY OF THE EFFECT OF SURFACE FINISH ON THE TRANSFER OF POLYCHLOROTRIFLUOROETHYLENE SLIDING ON MILD STEEL

by

Stephen Dale Doolittle

(ABSTRACT)

The effect of surface finish on the transfer of polychlorotrifluoroethylene (PCTFE) to mild steel was studied using a radio-tracer technique.

A pin-on-disk apparatus was used in conjunction with neutron activation of PCTFE to study the initial stages of transfer. Quantitative transfer and friction data were obtained as functions of surface finish and the number of repeated passes of polymer over mild steel. Qualitative supporting evidence for postulated transfer mechanisms was obtained using scanning electron microscopy.

The results showed that bulk transfer of PCTFE to mild steel does occur at room temperature. Initial transfer to polished (0.10 µm - rms) surfaces, abraded (0.81 µm - rms) surfaces, and glass bead blasted (1.73 µm - rms) surfaces was attributed to an abrasive wear mechanism. An apparent transistion to adhesive wear was observed during later stages of transfer. This transition occurred much sooner with the polished surface than with the rough surfaces.

This work also demonstrated that a radio-tracer technique could be effectively applied to the study of polymer wear. The minimum mass detection limit with this technique was 0.3 µg. This sensitivity is at least one order of magnitude better than that obtained with reported polymer wear measurement methods.