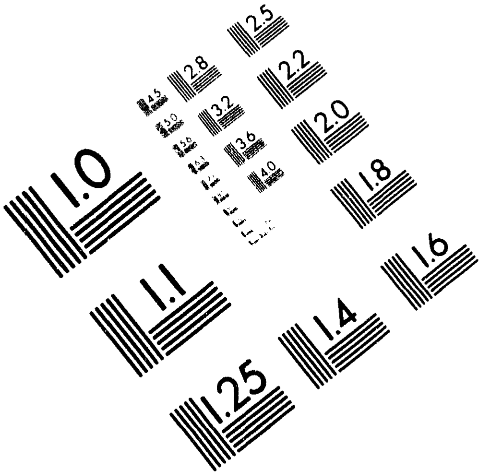

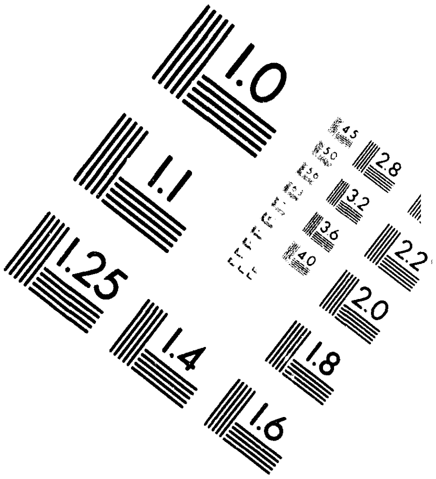

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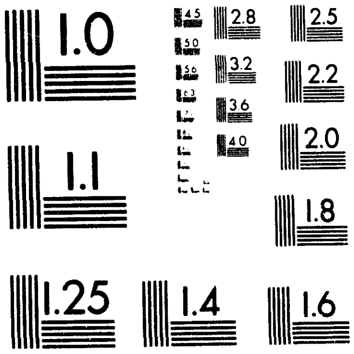

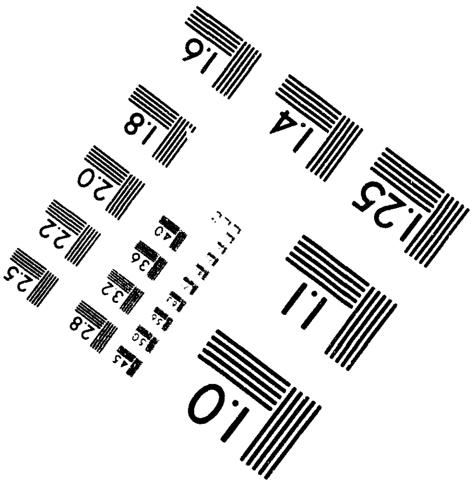

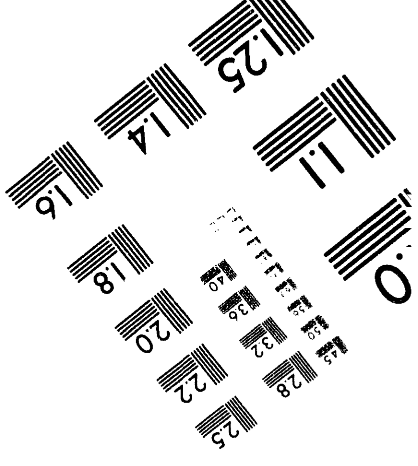

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Microfriction Studies of Model Self-Lubricating Surfaces

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ABSTRACT

Self-lubricating composites consist of at least one structural (matrix) phase and at least one phase to provide lubrication. Modeling the behavior of such composites involves ascertaining the frictional contributions of each constituent phase under varying conditions of lubricating film coverage. The ORNL friction microprobe (FMP), a specialized microcontact tribometer, was used to investigate the frictional behavior of both matrix and lubricant phases to support the development of self-lubricating, surfaces. Polished CVD-silicon carbide deposits and silicon wafers were used as substrates. The wafers were intended to simulate the thin silica films present on SiC surfaces at elevated temperatures. Molybdenum disulphide, in both sputtered and burnished forms, was used as the model lubricant. The effects of CVD-SiC substrate surface roughness and method of lubricant film deposition on the substrate were studied for single passes of a spherical silicon nitride slider (NBD 200 material). In contrast to the smooth sliding exhibited by burnished films, sputtered MoS_2 surfaces exhibited marked stick-slip behavior, indicating that the frictional behavior of solid lubricating coatings can be quite erratic on a microscale, especially when asperity contacts are elastically compliant.

INTRODUCTION

Self-lubricating composites consist of at least one structural (matrix) phase and at least one lubricous phase. There are numerous examples of such materials already in widespread use, particularly in applications in which external replenishment of the lubricant is difficult, or where a secondary, fail-safe lubrication system is needed in case the primary system fails. Self-lubricating materials range from traditional bushing materials like porous bronzes to structural ceramics or polymers impregnated with solid lubricants. Teflon(TM)-containing polymer composites [1,2], have found broad use as seals and light-duty bushings in machinery.

A common approach to modeling the macroscopic friction force (F) for sliding contact in a single-phase material pairing (in which there is no significant plowing) involves summing frictional resistances of individual contact spots.

$$F = \tau \sum_{i=1}^{n} A_i \qquad [1]$$

where τ is the effective shear strength of the material which determines the resistance to sliding and each A_i is the area of one of n contact spots which support the normal load in the interface at any given time. If several materials are present, then each contributes its own contact area and shear stress (τ_i) to the total force:

$$F = \sum_{i=1}^{n} \tau_i A_i \qquad [2]$$

For the simplest case of two dominant constituent phases, a linear rule of mixtures for friction has been shown to apply in certain situations [3]. It must be recognized, however, that the area fraction occupied by a given phase may change during sliding, and therefore the friction force will change accordingly. An effective solid lubricant should spread over the matrix phase during sliding, and in general, its area fraction in the load bearing area will be greater than its initial proportion of the bulk material. To complicate matters, as wear occurs, some of the debris may become mixed with the lubricant and its shear strength may be other than that of either matrix or lubricant phases.

The present paper reports the results of microscale friction experiments designed to understand the relative combinations of matrix and lubricating phases in self-lubricating materials. In the present case, two matrix phases and one lubricating species of interest were SiC, SiO_2, and MoS_2, respectively. The ORNL friction microprobe (FMP), a specialized micro-contact tribometer, was used in these studies in order to simulate the behavior of individual asperities as they slid over regions of matrix and lubricant phases.

In an earlier paper [4], the summation of contributions from frictional variations of individual asperities was described. It was demonstrated that as the number of asperities increases, their individual influences decrease, and the magnitude of momentary fluctuations in friction observed for the overall sliding system decreases. Therefore, the data from the current work should be viewed as typifying the behavior of single, relatively compliant asperity contacts rather than necessarily illustrating the behavior of heterogeneous interfaces with multiple contacts.

MATERIALS AND EXPERIMENTAL CONDITIONS

The current work has focussed on silicon carbide as one of the candidate matrix materials for elevated temperature, self-lubricating composites [5]. Coatings of SiC were produced in our laboratory by chemical vapor deposition (CVD) on a series of graphite disks within a cylindrical, hotwall CVD reactor. A mixture of methyl trichloro-silane (MTS) and hydrogen gases was introduced at 1000°C with a total chamber pressure of 50 Torr for 48 hours. By weight gain, the resultant coatings were at least 73 microns thick. The structure of those materials is typically beta-SiC, and the most common polytype is the cubic 3C (wurtzite) structure with stacked Si-C lattice planes [6]. SiC specimens were tested with two types of surface morphologies: (1) a highly porous structure, resulting from polishing down the as-formed, botyroidal SiC deposit to leave plateaus and open spaces between them (see Fig. 1), and (2) a non-porous, polished structure revealed by grinding down below the porous layer of the as-deposited surface.

It is well established that in the presence of atmospheric oxygen, SiO_x phases can form on CVD beta-SiC at elevated temperatures [7]. The friction microprobe (FMP) used for the present work was not designed for elevated temperature, so testing was performed at room temperatures. Therefore additional experiments were performed on high-purity single crystal (001)-oriented Si wafers (which have typically 10 nm thick SiO_2 surface films) to simulate, at room temperature, the type of surface films on which sliding might occur at higher temperatures. Clearly, the shear strengths of substrates, oxide films, and candidate solid lubricants change as temperature is raised, so the present results should not be considered fully representative of elevated temperature behavior. However, many of the same type of interfacial phenomena, such as solid lubricant film spreading, transfer to the slider, and loss of films by wear, are often observed at elevated temperatures, so the mechanistic implications of the current experiments should be at least qualitatively applicable to higher-temperature behavior.

The FMP used in these tests is schematically shown in Fig. 2. It consists of a light-weight specimen holder suspended on elastic webs whose deflection under the action of a friction force is measured with a capacitance gap gauge. The deflection corresponds to a calibrated tangential force whose line of action passes through the sliding contact. The slider arm is brought down by remote computer control so that uncertainties of manually lowering the slider are removed. The stage is traversed by a motorized, encoded micrometer stage at 10 μm/s velocity. The normal force for these experiments was 10 gf (98.1 mN) and the stroke length for each pass was 440 μm. The FMP rests on a thick plate which stands on an air-suspension type, vibration-isolation system. Further details of the FMP have been published elsewhere [8-10].

The Hertzian elastic contact stresses for a 1.0 mm diameter silicon nitride ball at 0.981 N load on a SiC flat was calculated to be 1.36 GPa (contact diameter 11.8 μm) and that for the Si wafer flat was 0.890 GPa (contact diameter 15.0 μm). The mild sliding conditions were indicated by the absence of fracture of either the SiC or the Si wafer materials when they were observed by light microscopy after testing. On the silicon wafers, faint, light-colored wear tracks 15 μm wide were observed. Therefore, the elastic contact size calculations agreed well with measurements of sliding track width in the case of minimal wear.

Two forms of MoS_2 solid lubricant were used in these experiments: (1) a reagent grade powder of > 99% purity and (2) a thin film deposit produced by sputtering. The powdered material (see Fig. 3a) was applied by burnishing it gently onto the surface with short-knapped metallographic polishing cloth.

The sputtered film was applied as a 1.5 μm-thick layer by a commercial firm [11]. As was common practice, the vendor wiped the surface of the sputtered deposit with lint-free, cotton cloth after coating to improve its cosmetic appearance. Unfortunately, this procedure destroyed the as-sputtered surface morphology and left fine fissures in the coating. (see Fig. 3b).

Room-temperature tests were run in air with the flat specimen materials and conditions listed in Table 1. The slider material used for all experiments was Norton type NBD 200 silicon nitride in the form of 1.0 mm diameter Grade 3 bearing balls. Slider tips were carefully cleaned just prior to mounting in the FMP and examined on an optical microscope at 100 - 200X to assure a clean contact point. After testing, the tips and flat specimens were once more examined by optical microscopy.

Friction data were recorded using digital data acquisition software at a data rate of one sample/50 ms, so that about 1000 friction force readings were obtained for each stroke (one sample for each 0.5 μm sliding distance at constant sliding speed). Details of sliding friction could be obtained easily by expanding portions of the data on the computer monitor or on hardcopy printouts.

Results of the stroke-by-stroke friction tests are presented as plots of sliding time versus friction force or friction coefficient. It should be noted that the sliding velocity of the actual contact varied due to the fact that changes in friction necessitated a change in stage deflection. The average sliding velocity was that of the traversing stage (10 μm/s), but intermittent accelerations and decelerations of the slider tip relative to the flat specimen during unsteady friction also occurred, and velocities for these cases are discussed subsequently.

RESULTS

A typical friction record for sliding on the CVD SiC porous surface is shown in Fig. 4a. Rising linear portions of the test record represent merely the bending of the elastic supports on the specimen stage as tangential force rose to overcome momentary mechanical restraints of the slider tip in surface holes. The tremendous effects of mechanical asperity interlocking are evident from this experiment. It was impossible to ascribe a single value of friction coefficient to characterize that kind of widely-varying behavior. At about 32 seconds (see Fig. 4a), a short level portion of the plot suggests that sliding was occurring momentarily on a flat portion of the surface. The friction coefficient of that portion is about 0.23, which agrees well with the 0.24 obtained in an earlier study [8].

The sliding record for the polished SiC surface, without pores, is shown in Fig. 4b. Here, the nominal friction coefficient was about 0.29-0.31. This range of values is probably a better one to use for the matrix than those obtained on the porous specimen because the sliding surface was much more uniform. It should be noted that the friction coefficient for more macroscopic, pin-on-disk sliding tests of silicon nitride on polished, hot-pressed silicon carbide was about 0.54 [12], but that larger friction value involved significant mechanistic contributions from surface roughening and entrapped wear debris.

Sliding tests of the Si wafer specimen exhibited a reproducible variation in friction behavior from one stroke to the next (see Fig. 4c). The first stroke of each five-stroke experiment had a curved, decreasing trend in friction which leveled out near the end of the stroke to a friction coefficient of about 0.16. The second stroke had a more linear decrease along its length. The third stroke of each test was nearly level in friction. The fourth and last strokes began with a slight rise, drop, and rise behavior. The remarkable repeatability of these frictional variations from one test to another suggested that a well-defined sequence of sliding interface conditioning mechanisms was occurring with the SiO_2/Si_3N_4 sliding combination.

When a layer of fine MoS_2 powder was burnished onto the Si wafer surface, a nominal friction coefficient of about 0.14 was obtained (see Fig. 5a). This is only slightly lower than the value for the uncoated wafer. At the beginning of the stroke, variations may indicate the transfer of some of the lubricant to the ball tip. Fig. 6 shows the deposit on the silicon nitride ball after 5 strokes on the coated surface. The transfer layer deposit was quite heterogeneous. From these observations, we learn that simply because the friction coefficients for the coated and uncoated silicon specimens were similar, it should not be assumed that conditions are also identical in the interface.

Tests of MoS_2 sputter-coated onto Si wafers were also performed to determine whether the method of applying the lubricant would affect frictional behavior. A dramatic difference in the frictional behavior was in fact observed. The sputtered surfaces exhibited marked stick-slip behavior with tangential forces varying from about 3.0 to 7.0 gf in a very periodic manner (e.g., Fig. 5b). The stick-slip behavior was repeatable from one stroke to the next and from one test to the next. Enlargements of the trace showing the individual data (Fig. 7) indicated that the "slip" portion of the stroke took less than 0.05 s, the preset time between digital datum samples. Extremely linear behavior of the up-loading curves suggested that no significant slip was occurring during the elastic, stick portion of the trace. The sliding velocity during slip was therefore much higher than the nominal velocity of the FMP traversing stage. Based on the change in friction force and the known force-deflection calibration of the FMP stage, the actual sliding velocity during a slip was greater than 400 μm/s (19.96 μm relative motion occurring less than 0.05 s), about 40 times higher than the stage traverse velocity.

The final experiments in this study were designed to see whether sliding of silicon nitride on a MoS_2 surface would pick up enough transferred lubricant to produce residual friction effects when it slid on uncoated matrix material. A five stroke test on NBD 200 on sputtered MoS_2 was conducted and the surface of the tip was examined. Only a very small amount of lubricant transferred (Fig. 6). A fresh slider was prepared and slid three times on MoS_2. Then the flat specimen was replaced with a clean specimen of Si to observe whether there would be any effect of the transferred material on the ball tip. The resultant friction trace did not differ from the others which started with a clean ball tip.

DISCUSSION

Friction data from friction microprobe studies should not be compared directly with more macroscopic, traditional friction test results because the contact conditions are more sensitive to highly-localized sliding conditions on the asperity level and because its force measurement sensitivity requires an elastically-compliant stage. Data represent frictional behavior in the absence of significant amounts of sliding energy dissipation due to the sliding of rough surfaces. In addition, the complicating effects of wear debris accumulation on friction can usually be neglected since little or no wear occurs in FMP tests.

The fact that the friction of Si_3N_4 on SiO_2 was lower than that of silicon carbide suggests that at higher sliding temperatures, where the formation of SiO_2 on SiC is promoted, frictional benefits should occur. However, Yust's recent work [5] indicated that when sliding NBD 200 on CVD-SiC in air at temperatures up to 750 °C, the friction at the higher temperature was increased about 25% over that at room temperature. This contrasts with a 53 % reduction of friction when sliding NBD 200 spheres on the silicon wafer instead of polished CVD-SiC in the current study. This observation suggests that other factors, such as wear surface roughness and the change in film shear strength with rising temperature, can counteract the frictional influences of elevated-temperature SiO_2 formation.

If the surface coverage by lubricant is not complete, then a simple mixtures rule, similar to that discussed in Ref. [3], may be applied, viz.

$$F = X_m F_m + X_l F_l \qquad [4]$$

where X_m and X_l are the area fraction of the matrix and lubricant, respectively. Assuming that the normal force is distributed proportionately among the contact areas,

$$\mu = (1-X_l)\,\mu_m + X_l\,\mu_l \qquad [5]$$

where, $\mu_{m,l}$ are friction coefficients for the limiting cases of complete surface contact by either phase.

Graphically, the representation of Eqn. [5] is simple: a linear plot of X_l, the area fraction of lubricant coating the contact area, versus friction coefficient with the left intercept representing the case of sliding only on matrix material and the right intercept value equivalent to sliding on a continuous lubricant film. This type of diagram has been called a Friction Process Diagram and is described more fully elsewhere [13]. Using the lowest measured friction values for MoS_2 (on the burnished surface), diagrams for Si/MoS_2 and SiC/MoS_2 are given in Fig. 8. The plot represents the average level of friction to be expected for various amounts of surface coverage by MoS_2 on these two substrate materials if the composite were tested under similar conditions to that used in this investigation.

Perhaps the most remarkable result of the current study is the finding that sputtered MoS_2 coatings can exhibit significant stick-slip behavior on the asperity scale. The physical reason for this behavior is not the periodic failure of the coating, as might have been expected in the absence of other evidence, but rather the development of periodic scallops (depressions) in the coating itself with no break-through to the substrate. Through the use of differential interference contrast optical microscopy, the undulating appearance of the wear groove in the MoS_2 sputtered surface was revealed (Fig. 9). The number of undulations along the track is consistent with the number of stick-slip events recorded in the friction record for each stroke. Higher magnification examination along the slider groove revealed no evidence of Si substrate exposure. Therefore, the stick-slip behavior was probably caused by plastic indentation of the hard ceramic slider tip into the soft surface, followed by its sudden release only to become lodged in the next impression. It is suggested that the reason why this behavior is not typically observed in more macroscopic friction tests of sputtered MoS_2 is that the asperities on the surface of a larger, stiffer body have insufficient individual elastic compliance to develop the stick-slip behavior, and further that, averaged over many asperities, this kind of localized behavior would not normally be detected.

It is interesting to note that the stick-slip behavior of the sputtered film produced magnitudes of friction variations similar to those of the porous SiC surface, although the mechanistic reasons for this behavior clearly were not the same. In the porous SiC, the tip of the slider was caught in pre-existing holes in the surface, whereas with the MoS_2 coating, plastic indentation of the coating and tip adhesion to the material within the impression occurred as a direct result of the sliding process.

The lack of transfer by the sputtered MoS_2 onto the silicon nitride surface implies that pockets of MoS_2 in a composite microstructure must be very closely-spaced if there is to be any chance to make it self-lubricating against silicon nitride. A self-lubricating coating should be smooth enough to eliminate asperity interlocking effects while still having enough volume percent lubricant to form lubricant bridges from one lubricant inclusion to another along the sliding path.

CONCLUSIONS

Friction microprobe experiments on an asperity-scale showed that for the sliding of small, silicon nitride sliders:

1. The friction of silicon nitride on natural silica layers (on single crystal silicon) was lower than that for sliding on polished silicon carbide surfaces. This effect ran counter to the observed increase in friction for silicon nitride sliding on silicon carbide at elevated temperatures, as found in other experiments. Thus, silica film formation has less effect on friction at high temperatures than did surface roughening due to increased wear.

2. Mechanical interlocking was seen to cause significant and erratic variations in tangential force on the asperity scale.

3. The method of applying MoS_2 to surfaces can change the frictional behavior of model asperities from being low and having relatively smooth sliding (burnished powders) to having a marked stick-slip response (sputtered coating).

4. MoS_2 was not prone to transfer to silicon nitride in large enough measure to assure that friction reduction would be maintained after the silicon nitride slider left an area of the surface which was covered by MoS_2. Therefore, it is not expected to be a good lubricant for silicon nitride unless there is full and continuous surface coverage.

ACKNOWLEDGEMENTS

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Table 1.

MATERIALS USED FOR FLAT SPECIMENS

(NBD 200 slider, 98.1 mN load, 10 μm/s speed, in air)

Material	Form
SiC	CVD deposit polished to produce flat plateaus
SiC	CVD deposit polished flat with no pores
Si	high-purity, single crystal wafer with coating of ambient SiO_2
Si/MoS_2	burnished deposit on a Si wafer
Si/MoS_2	sputtered deposit (1.5 microns) on a Si wafer

Figure Captions:

1. Optical photomicrograph of silicon carbide coating showing polished plateaus widely-separated by regions of as-deposited coating morphology.

2. Schematic diagram of the friction microprobe. Elastic suspension webs at each end of the "boat" extend perpendicularly to the plane of the diagram.

3. Scanning electron micrographs of (a) high-purity MoS_2 powder showing thin platelets of various sizes. (b) Sputtered deposits of MoS_2 after wiping with link-free cotton. There are numerous fine fissures in the soft surface surrounding a harder crystallite in the film.

4. Typical friction force versus time data for silicon nitride sliding on (a) a quite porous SiC surface, (b) a flat, polished SiC surface, and (c) a silicon wafer.

5. Friction force versus time data for silicon nitride on (a) burnished MoS_2 and (b) sputtered MoS_2.

6. Deposit of MoS_2 on the tip of the slider after 5 strokes.

7. High-resolution detail showing individual data during the "slip" portion of a stroke on sputtered MoS_2.

8. A Friction Process Diagram for Si and CVD SiC, each assumed to operate by a linear rule of frictional mixtures with MoS_2.

9. Differential interference contrast image of the sliding track in the sputtered MoS_2 surface showing periodic undulations which correspond in number to stick-slip events in the recorded friction trace.

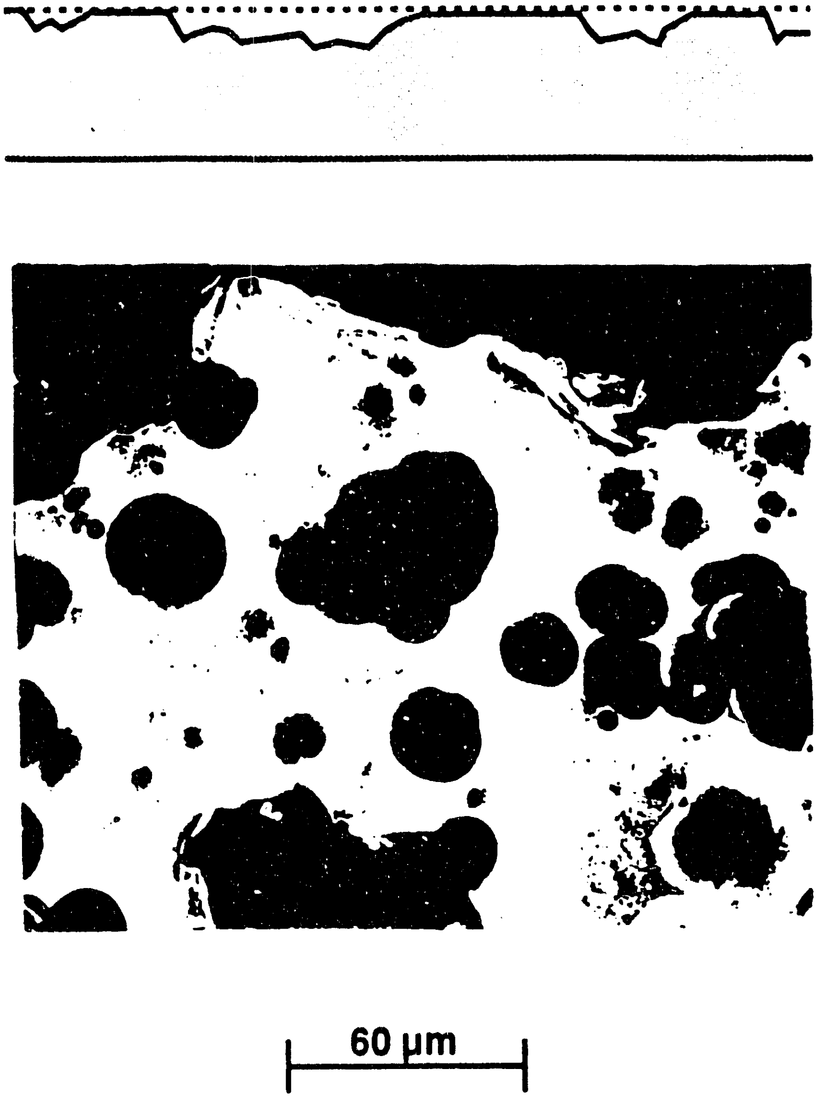

FIG 1

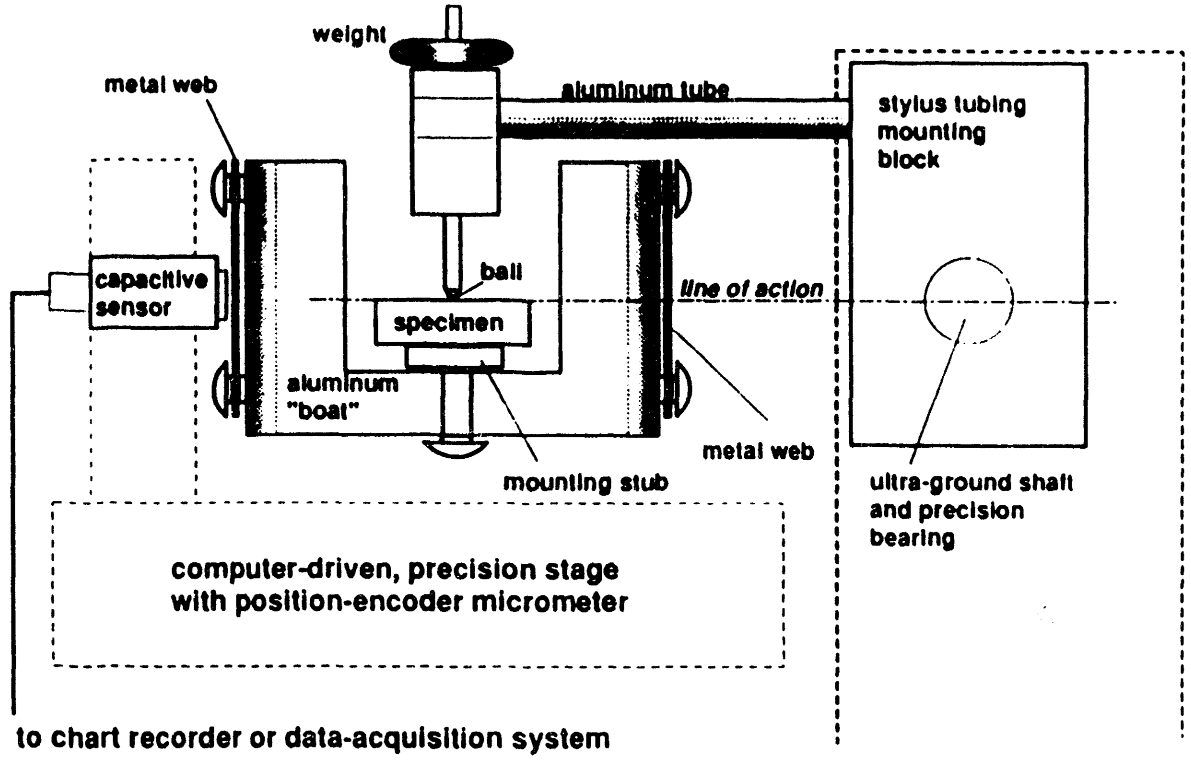
weight
metal web
aluminum tube
stylus tubing mounting block
capacitive sensor
ball
line of action
specimen
aluminum "boat"
metal web
mounting stub
ultra-ground shaft and precision bearing
computer-driven, precision stage with position-encoder micrometer
to chart recorder or data-acquisition system

FIG. 2

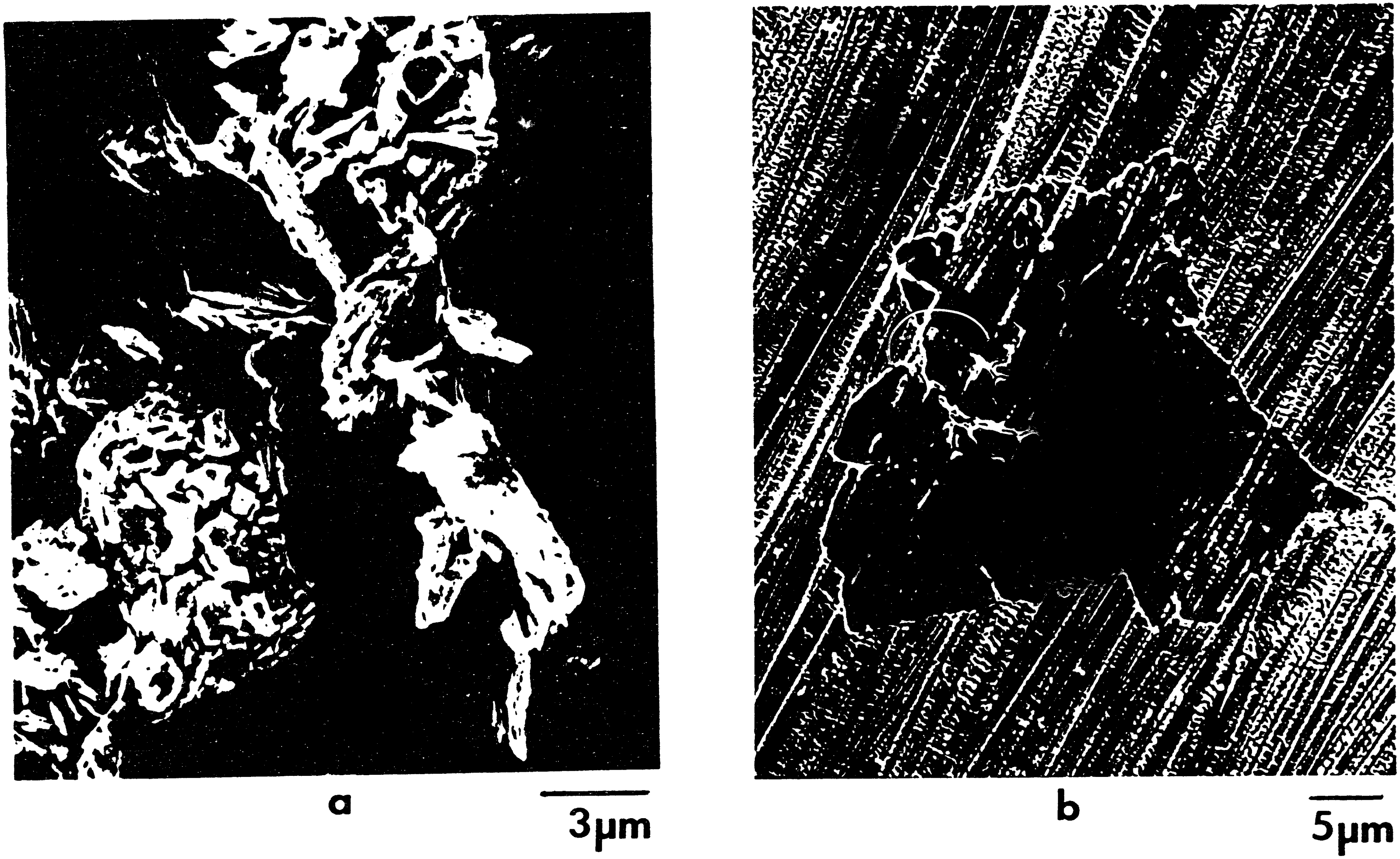

Fig 3

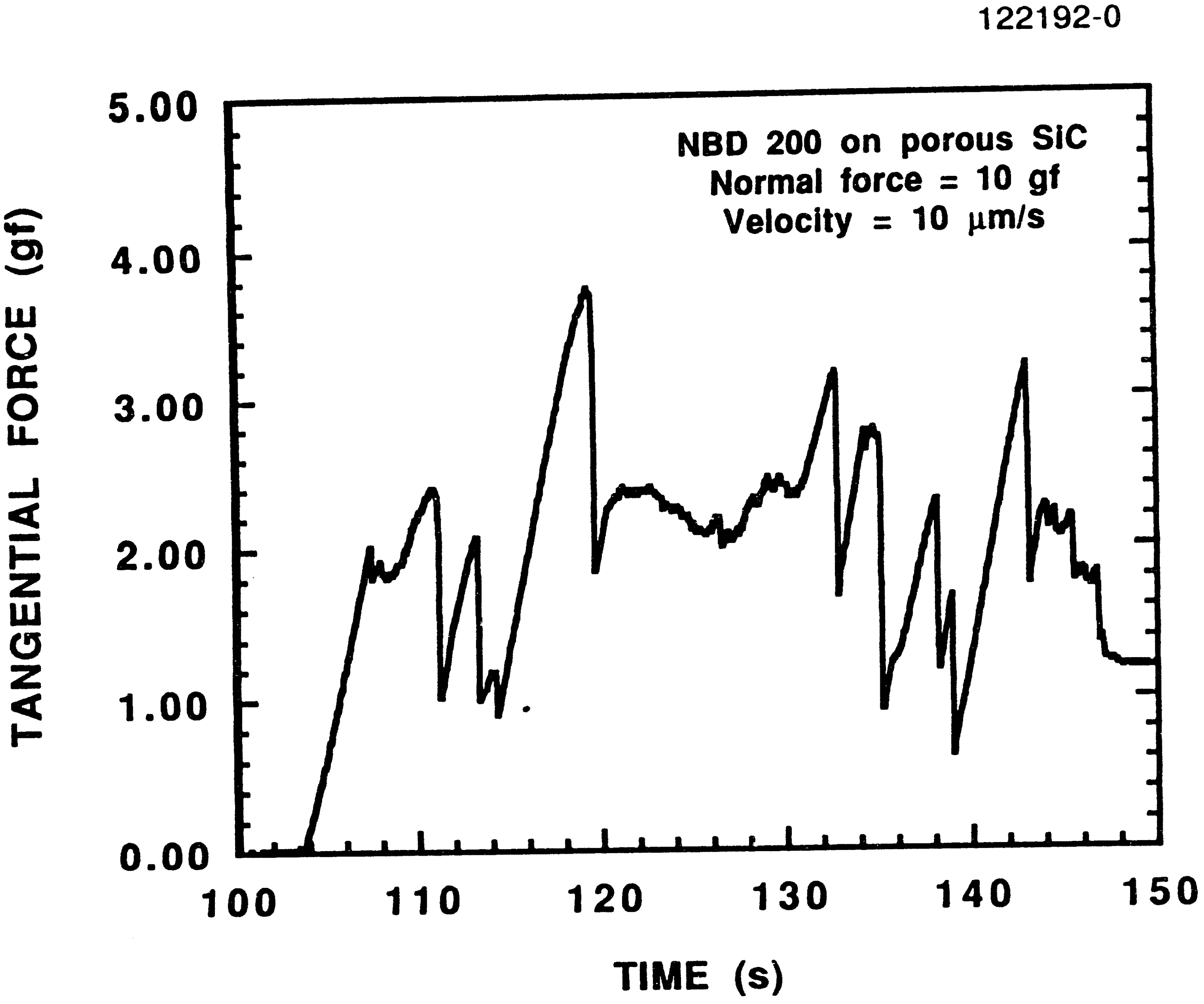
122192-0
NBD 200 on porous SiC
Normal force = 10 gf
Velocity = 10 µm/s
TANGENTIAL FORCE (gf)
5.00
4.00
3.00
2.00
1.00
0.00
100
110
120
130
140
150
TIME (s)

Fig. 4(a)

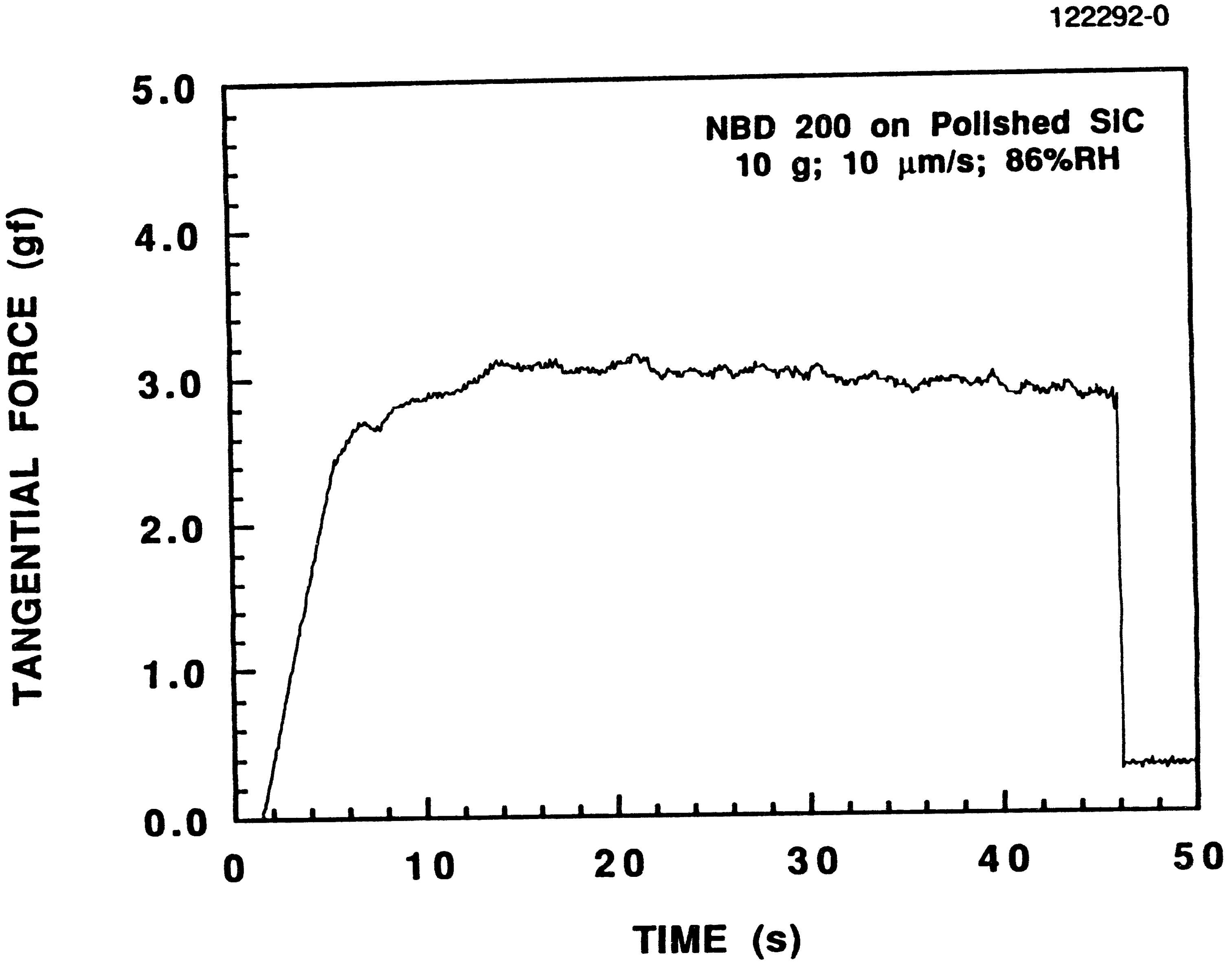
122292-0
NBD 200 on Polished SIC
10 g; 10 μm/s; 86%RH
TANGENTIAL FORCE (gf)
5.0
4.0
3.0
2.0
1.0
0.0
0
10
20
30
40
50
TIME (s)

Fig 4 (b)

FMP FILE NUMBER 121592-1
NBD 200 ON SILICON WAFER
10 gf; 10 µm/s; 440 µm stroke

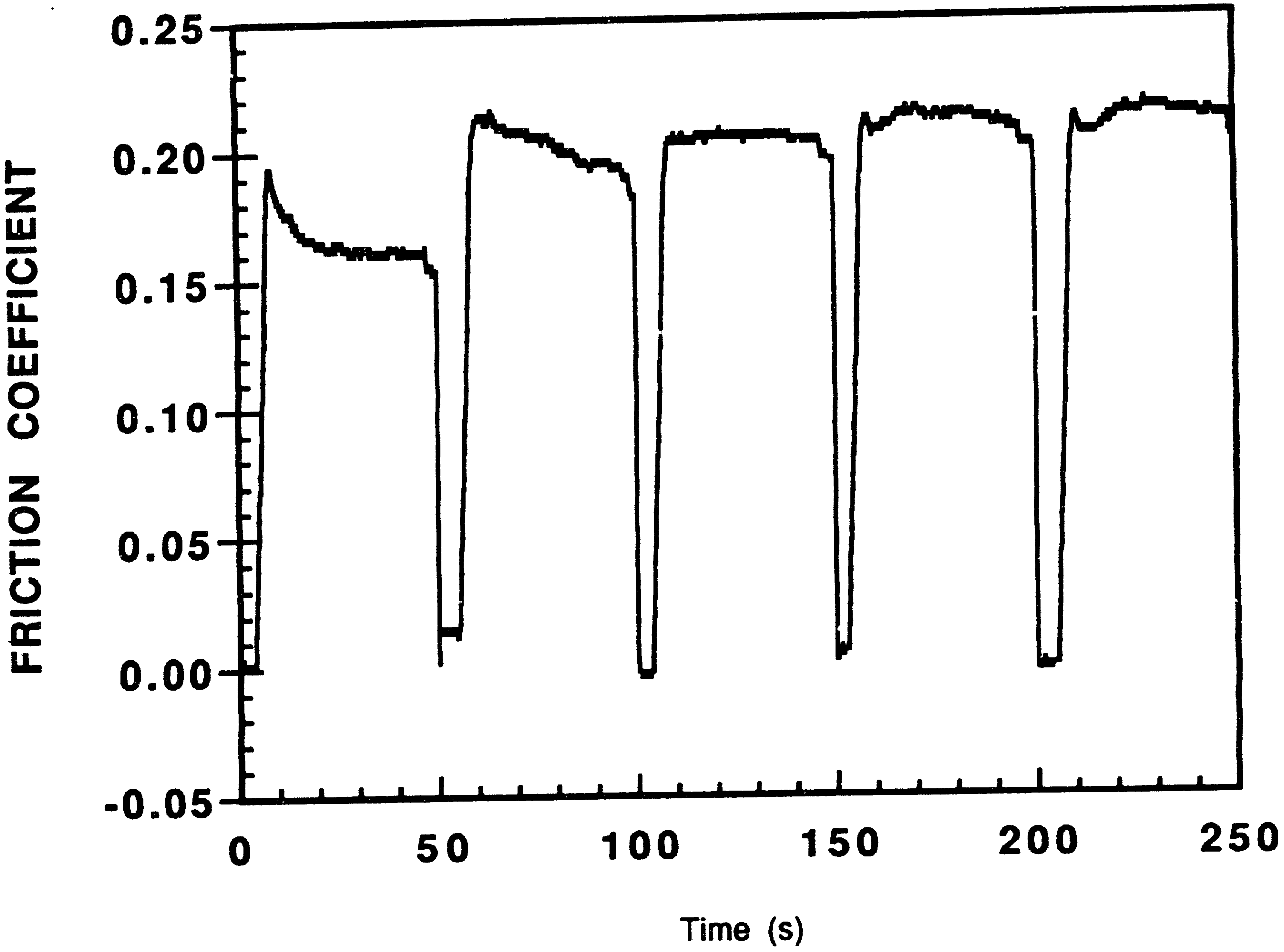

Fig. 4(c)

123092-0

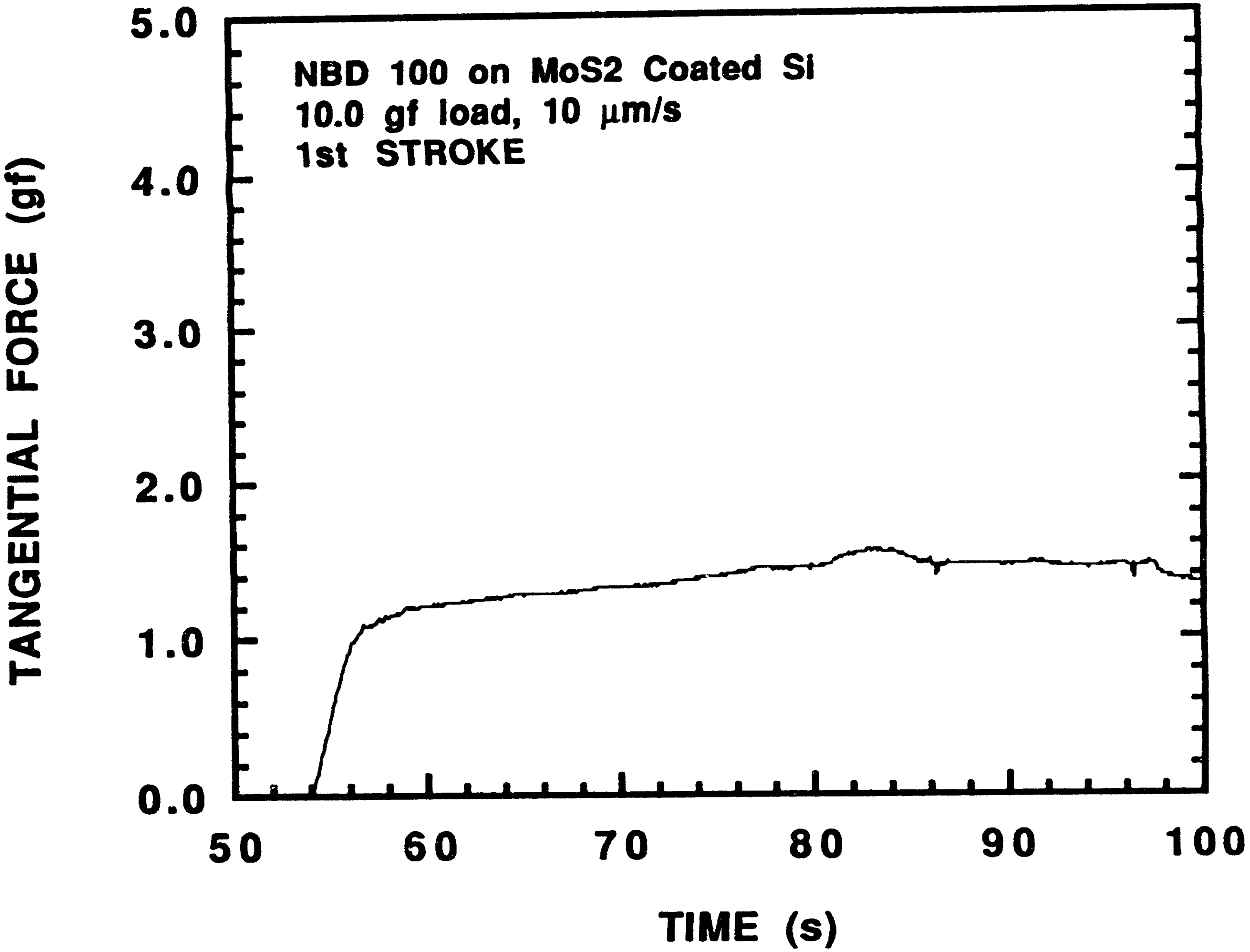

Fig 5(a)

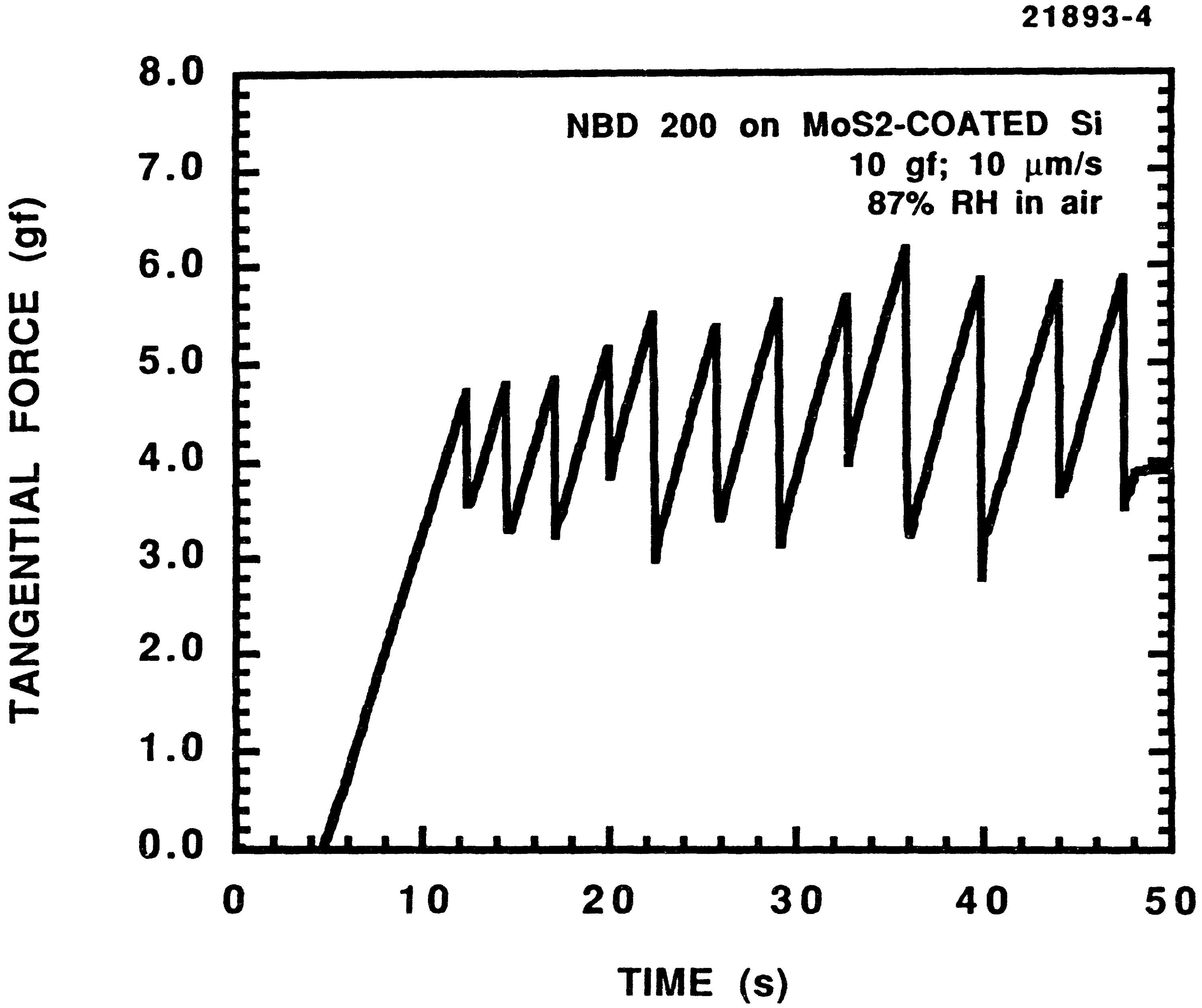
21893-4
NBD 200 on MoS2-COATED Si
10 gf; 10 µm/s
87% RH in air
TANGENTIAL FORCE (gf)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
10
20
30
40
50
TIME (s)

Fig 5(b)

FIG. 6

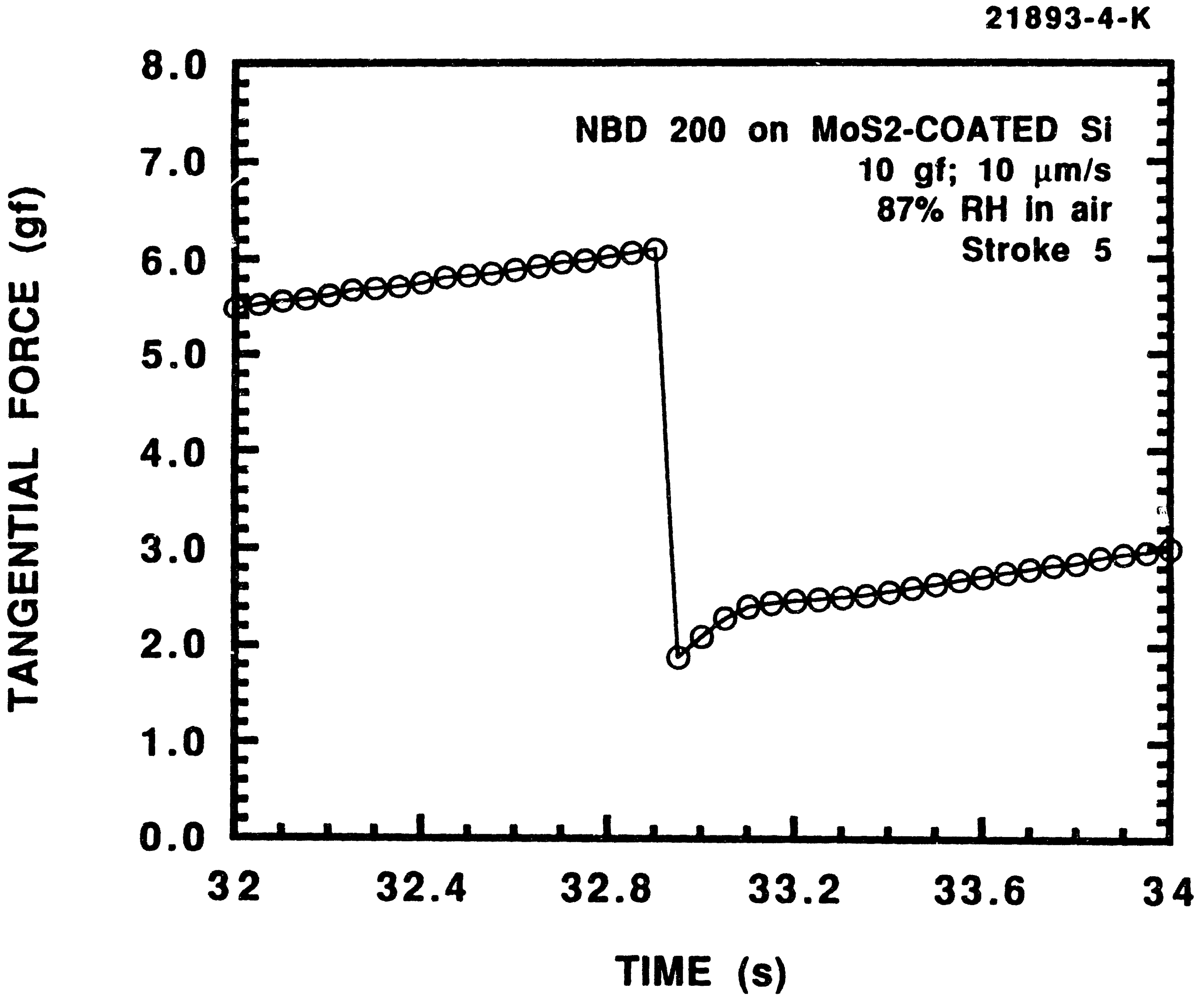
21893-4-K
NBD 200 on MoS2-COATED Si
10 gf; 10 µm/s
87% RH in air
Stroke 5
TANGENTIAL FORCE (gf)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
32
32.4
32.8
33.2
33.6
34
TIME (s)

Fig. 7.

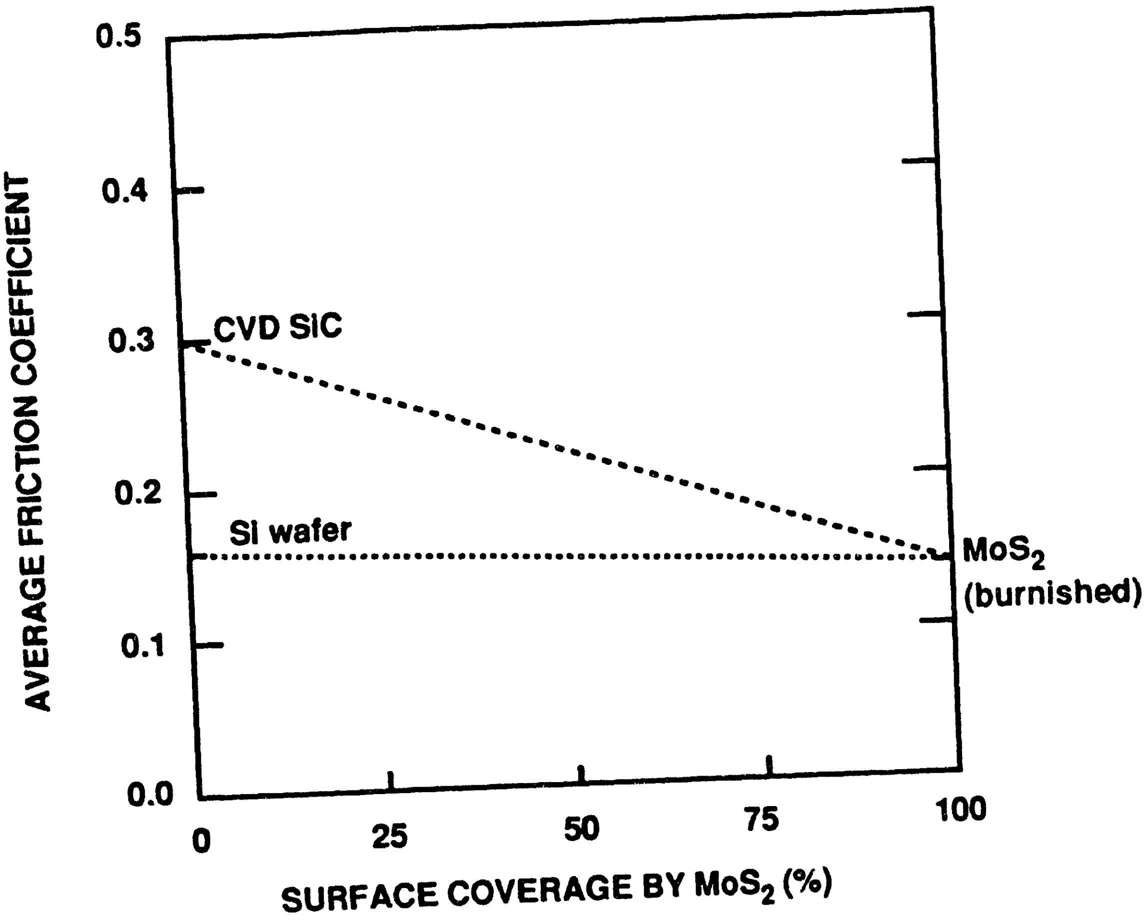
0.5
0.4
0.3
0.2
0.1
0.0
AVERAGE FRICTION COEFFICIENT
CVD SIC
SI wafer
MoS$_2$
(burnished)
0
25
50
75
100
SURFACE COVERAGE BY MoS$_2$ (%)

FIG. 8

Fig 9
.65x

DATE
FILMED
10/17/94

END