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Ultra-Thin Overcoats for the Head/Disk Interface Tribology

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Raymond Hsiao, David B. Bogy, and Ian G. Brown

**Accelerator and Fusion
Research Division**

MASTER

May 1997

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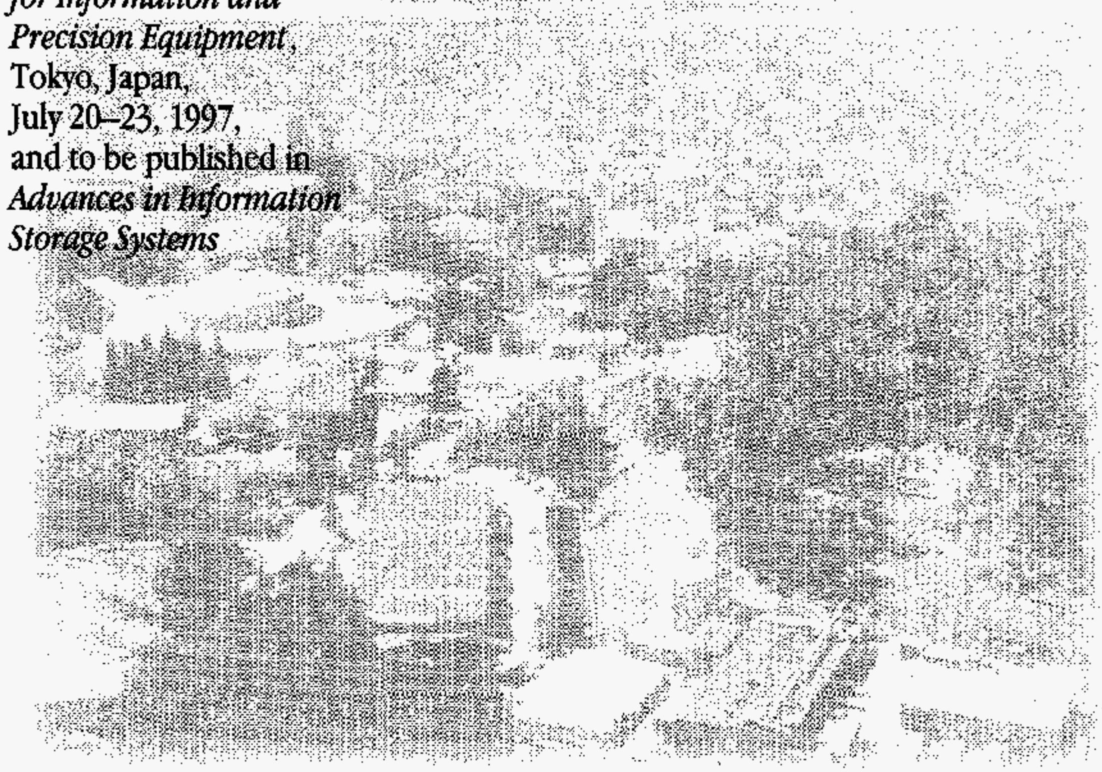

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C. Singh Bhatia,[1] Simone Anders,[2] Kamau Bobb,[3]
Raymond Hsiao,[3] David B. Bogy,[3] and Ian G. Brown[2]

[1]SSD/IBM
5600 Cottle Rd.
San Jose, CA 95193

[2]Advanced Light Source
Accelerator & Fusion Research Division
Ernest Orlando Lawrence Berkeley National Laboratory
University of California
Berkeley, CA 94720

[3]Department of Mechanical Engineering
University of California
Berkeley, CA 94720

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David B. BOGY[***], **and Ian G. BROWN**[**]

[*]*SSD/IBM, 5600 Cottle Road, San Jose, CA 95193*
[**]*Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, CA 94720*
[***]*Department of Mechanical Engineering, University of California, Berkeley, CA 94720*

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[*]*SSD/IBM, 5600 Cottle Road, San Jose, CA 95193*
[**]*Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, CA 94720*
[***]*Department of Mechanical Engineering, University of California, Berkeley, CA 94720*

ABSTRACT

Areal density in magnetic storage is increasing at a blistering pace of 60% annually. Recently IBM announced its mobile product with the industry highest areal density of 2.64 Gb/In^2. The areal density demonstrations have shown up to 5 Gb/In^2 possible. Reaching higher areal density targets dictate that magnetic spacing between heads and disks be reduced. For the example of a 10 Gb/In^2 areal density goal, the magnetic spacing should be ~25 nm [1]. In budgeting this magnetic spacing, it is required that disk and slider air bearing surface overcoats thickness be reduced to 5 nm range. Present choice of carbon overcoat in the magnetic storage hard disk drive industry is sputter deposited, hydrogenated carbon (CH_x) [2] with thickness in the range of 12-15 nm on heads and disks. Novel overcoats such as nitrogenated carbon (CN_x)

[3] and cathodic arc carbon films are being developed for future applications. Cathodic arc deposition forms ultra-thin amorphous hard carbon films of high sp^3 content, high hardness, and low coefficient of friction. These properties make it of great interest for head/disk interface application, in particular for contact recording. In many cases, the tribological properties of the head disk interface could be improved by factors up to ten by applying cathodic arc overcoats to the slider or disk surface. This paper reviews the results of cathodic arc ultra-thin (2-10 nm) carbon overcoats for head/disk interface tribological applications.

1. INTRODUCTION

Cathodic arc deposition has been used for decades to deposit hard coatings such as TiN in particular for tool applications. It was found also many years ago that amorphous hard carbon films deposited by this method exhibit properties reaching those of diamond [4]. The drawback of cathodic arc deposition is the production of macroparticles of the cathode material which have a typical size of microns and contaminate the films. Much effort has been put into the improvement of the deposition process by filtering the cathodic arc plasma from the macroparticles. Magnetic macroparticle filters have been used in most cases [5], and the film quality has improved drastically. Recently, a new filter system of very high filtering quality has been developed which allows the formation of films which comply with the requirements of magnetic storage devices [6]. In the present paper we describe briefly the filtered cathodic arc deposition process, summarize the properties of cathodic arc deposited amorphous hard carbon films, and outline some applications to head/disk interface tribology.

2. CATHODIC ARC DEPOSITION PROCESS

Cathodic arc deposition is based on the formation of plasma from the cathode material by a high current electrical discharge in vacuum or in a gas environment. The plasma is (in contrast to, e.g. sputter plasmas) fully ionized which allows for an easy way to control the ion energy during deposition - by biasing of the substrate. For deposition of high-quality amorphous carbon films the source needs to be equipped with an efficient macroparticle filter. Such a filter can be composed of two 90 degree magnetic macroparticle filters which are typically used for applications that do not require an extremely low macroparticle content of the films. Fig. 1 shows a schematic arrangement of the plasma source, the filter, and the substrate.

The plasma source used for the experiments described was operated in a pulsed mode with 100-300 A arc current, a pulse duration of 5 ms, and a pulse repetition rate of 1 Hz. It was found by a number of authors that an ion energy of around 100 eV is optimum for the production of films with the highest sp^3 content [7-9]. It seems to be the most important parameter for the formation of high sp^3 content hydrogen-free films (in the case of hydrogenated films the presence of hydrogen favors the formation of sp^3 bonds chemically). Pulsed biasing of the substrate is typically applied to the formation of high sp^3 amorphous carbon films to tune the ion energy to the optimum value.

3. PROPERTIES OF CATHODIC ARC DEPOSITED AMORPHOUS HARD CARBON FILMS

3.1. Content of tetrahedral bonds

Amorphous hard carbon contains carbon atoms in sp^2 and sp^3 bonding state. Electron Energy Loss Spectroscopy (EELS) [10] was performed to determine the sp^3 bond contents of films deposited a t substrate biases of 0, -100, -500, and - 2000 V. The valence plasmon and carbon K-edge losses were both collected under the same conditions. The K edge gives the fraction of π^* states in the conduction band and hence the fraction of sp^2 sites. The fraction of π^* states was determined by fitting a Gaussian to the first peak around 285 eV, including a small Gaussian around 287 eV, and a broad Gaussian to the 290 eV edge. The fraction of sp^2 sites (with an accuracy of ± 5%) was found by comparing the ratio of the area of the 285 eV peak to the 290 eV peak to that in polycrystalline graphite, which is 100% sp^2. The EELS results (as well as the other parameters which will be described in detail in the next sections) are summarized in Table I. The sp^3 content is greatest for the film deposited at -100 V and reaches up to 85% [11]. This is much higher than the sp^3 content CH_x films which are used typically for hard disks which.is of order 15-30%.

3.2. Mass density

The mass density of cathodic arc deposited hard carbon films was measured in three ways: by Rutherford Back-scattering Spectroscopy (RBS) and profilometry, by EELS, and by weighing. Films deposited at substrate biases of 0, -100, -500 , and -2000 V on Si were used. The number of carbon atoms per cm^2 determined by RBS and the film thickness determined by a stylus type profilometer resulted in data for the mass density given in table 1 [9]. In EELS the valence plasmon gives the valence electron density and hence the mass density [7, 10]. The results give the highest mass density of 3.0 g/cm^3 for a bias voltage of -100V [11]. The mass density of 3.0 g/cm^3 for films deposited at -100V bias voltage was confirmed by weighing a Si wafer before and after deposition of a 100 nm film and calculating the density from the film volume and mass increase of the wafer.

3.3. Hardness and elastic modulus

Hardness and elastic modulus of films deposited at various bias voltage were measured by nanoindentation. In most cases the film hardness and elastic modulus show a maximum not at the surface but about 40 nm below the surface, and the hardness and elastic modulus are the highest for films deposited at -100 V substrate bias. The hardness for this bias voltage is up to 75-80 GPa compared to typically 20 GPa for hydrogenated, sputtered diamond-like carbon films [11]. Measurement on 300 nm films are summarized in table 1 (the hardness measurements depend on the film thickness because the substrate influence cannot be excluded).

3.4. Thermal stability

The thermal stability of the hard carbon films is of importance for the application in head/disk interface tribology. It was found by a number of authors that deposition at elevated temperature leads to a deterioration of the film properties and a graphitization [8]. We have measured the thermal stability of films after deposition. The films were heat treated in air for a duration of 2h at the given temperature and studied by Raman spectroscopy and nanoindentation.

It was found that films containing a high sp^3 fraction sustain their hardness for temperatures at least up to 400°C and their structure for temperatures up to 500°C. They show a low thickness loss during heat treatment. CH_x films start to change their structure typically between 200-300°C during heat treatment depending on the heating duration and specific film properties.

3.5. Stress

Cathodic arc deposited hard carbon films show a considerable intrinsic growth stress. We have measured the stress as a function of the substrate bias by depositing films of 70 nm on thin Si wafers at different pulsed substrate bias. The wafer curvature was measured before and after deposition, and the film stress was determined using the Stoney equation [12]. We observed a maximum in the stress at -100V bias (table 1) [12]. This high stress in cathodic arc films can have detrimental effects on film adhesion and film compatibility with other materials for the formation of multilayered structures such as hard disks. In the next section we describe a carbon-carbon multilayer approach to overcome this problem.

4. MULTILAYERS OF AMORPHOUS HARD CARBON

The ability to vary the film properties over a wide range by changing the substrate bias allows the formation of carbon/carbon multilayers. We have formed multilayers of alternating hard and soft amorphous carbon films by varying the bias voltage during deposition between -100V for high sp^3 fraction and -2000V for low sp^3 fraction films. The multilayers have been investigated by transmission electron microscopy (TEM) and nanoindentation. Stress measurements have been performed also.

Three different multilayer structures have been deposited. All three structures consisted of 8 layers; the first layer at the substrate interface was a soft layer deposited at - 2 kV pulsed bias, and the top layer was a hard layer deposited at - 100 V pulsed bias. The ratio between the amount of carbon deposited at high and low bias was varied for the three structures. For the first structure the ratio was 50% soft phase/50% hard phase, for the second structure it was 10% soft phase/90% hard phase, and for the third structure it was 90% soft phase/10% hard phase. The total thickness of the multilayer structures was 250 nm. For comparison, films were deposited at high bias and low bias voltage only with the same total film thickness of 250 nm.

It was found by nanoindentation that the values for hardness and elastic modulus are almost a linear interpolation of the ratio of the hard and soft phases. Fig. 2 is a cross-section TEM image of the 50%/50% structure. In the upper left corner the silicon substrate is visible, on the lower right corner the glue for the sample preparation. The multilayer structure of four pairs of layers is clearly visible.

In contrast to hardness and elastic modulus it was found that the stress is not a linear interpolation between single layer properties but is considerably lower (Fig. 3). It can be expected that the stress can be further reduced by reducing the layer thickness and increasing the number of layers in the structure. This gives the opportunity of changing to a certain degree independently the hardness and stress in the structure.

5. APPLICATIONS TO HEAD/DISK TRIBOLOGY

5.1. Coefficient of friction

Cathodic arc deposition combined with a high voltage pulsed biasing of -2 kV of the substrate was used to modify the surfaces of two-rail sliders consisting of 70% Al_2O_3 and 30% TiC. The pulsed biasing leads to a superposition of deposition (between bias pulses) and ion implantation (during bias pulses). A total dose of 2×10^{16} ions/cm^2 carbon ions with 30% implantation phase and 70% deposition phase was applied to the sliders. The surface of the slider was modified to a depth of 20 nm with a fraction of the implanted ion species decreasing from 90% at the slider surface to 20% at a depth between 5-15 nm [13].

Continuous sliding against unlubricated carbon-coated hard disks was performed with a low sliding speed of 3 cm/s with a normal load of 0.16 N in air of 40% humidity and 27°C. Fig. 4 shows the coefficient of friction of unmodified heads in comparison to carbon modified heads. At the beginning the coefficient of friction is about 0.2 and comparable for unmodified and modified sliders. For the unmodified slider the coefficient of friction increases drastically during the first 1000 revolutions and then remains very high (about 1.3) for the rest of the testing. The modified slider shows a very stable and low coefficient of friction over the entire testing [13].

5.2. Nano-wear tests

Cathodic arc deposited amorphous hard carbon films of 10 nm thickness were deposited on a Si wafer at -100V pulsed bias voltage. A nano-wear test was performed on this film using a point contact microscope. An area of 2μmx2μm was scanned with a light load to obtain an image of the surface topography. The scan size was reduced to 1μmx1μm at the same location and the load was increased to a preset value. After two wear cycles the loading force and scan size were reset to their original values and the surface topography was measured again. Wear depth versus wear cycles is plotted in Fig. 5. A load of 100μN was hardly enough to modify the film surface. The wear depth of 1 nm for 30 cycles indicates the superior scratch resistance of this film. Typical CH_x films show a wear rate of 7.5-25 nm (depending on the hydrogen content) after only 12 cycles and a much lower load of 28μN. It is interesting to note that the nano-wear test on cathodic arc carbon using 200μN load shows three distict zones which can be identified as the carbon film, the SiC interface and the Si substrate.

5.3. Coating of hard disks

Supersmooth, 65mm disks were coated with cathodic arc amorphous hard carbon of 10 nm thickness. The disks were lubricated with 1.5 nm Z-dol 2000. Wear test using sliders in contact with the disk at a speed of 13 m/s were performed. The normal load on the sliders was 40 mg. The worn volume at the face of the slider was measured using an Atomic Force Microscope (AFM). Fig. 6 shows the worn volume as a function of time comparing the cathodic arc carbon coated disk with a disk coated with a 10 nm CH_x film. The worn volume of the slider in contact with the cathodic arc coated disk is a factor of almost 20 lower than the worn volume of the slider in contact with the sputter coated disk. This suggests a superior performance of cathodic arc carbon for contact recording applications. The coatings were also tested in various environments (air with relative humidity of 50%, vacuum, nitrogen and argon) using a normal load of 40 mg and a disk velocity of 0.42 m/s. The coefficient of friction was between 0.09 and 0.11 for all conditions on a cathodic arc coated disk whereas the coefficient of friction was about a factor of 2 larger in all test environments using a disk coated with a CH_x film. The wear volume of the contact pad was found to be very low also in different environments (air with a relative humidity of 65% and 10%, nitrogen, and air) [14].

5.4. Coating of sliders

50% sliders of Al_2O_3-TiC with 6g normal load were coated with an amorphous hard carbon film of 2 nm thickness using cathodic arc deposition. For the first 10% of the deposition time a high pulsed bias of -2 kV was applied to improve the adhesion of the film whereas a low bias of -100V was applied for the rest of the coating to form a film with a high sp^3 content. The thickness of the carbon coatings measured by Auger depth profiling was 2 nm.

Contact Start Stop (CSS) tests were performed on 95mm, mechanically textured disks coated with 15 nm CH_x and lubricated with 1 nm Z-dol 2000. The wear durability of the sliders with and without cathodic arc carbon coatings was compared. The data recorded were the average friction per revolution, touch down velocity (TDV) and stiction values. A cycle profile consisted of acceleration to 3600 rpm, constant velocity at 3600 rpm for 10 seconds and then deceleration to zero. At the beginning of the test a single revolution at low rpm was run and the average value of friction was calculated. This measurement was taken every 1000 cycles throughout the duration of the test and plotted versus cycles. The TDV was measured every 2000 cycles and plotted versus cycles. CSS tests of 100,000 cycles were run. Figures 7 and 8 show the average friction and the touch down velocity as functions of number of cycles. The uncoated sliders failed after 7500 cycles. Failure was indicated by an increase in the TDV and a simultaneous decrease in friction. Debris was visible on the rails of the slider when examined under a microscope, and there was a visible wear track on the surface of the disk. The tests consisting of sliders coated with cathodic arc carbon completed 100,000 CSS cycles without failure. The slider rails contained minimal debris on the leading edge of both the inner and outer rails and there was no visible wear track on the surface of the disk. The stiction increased from 1.7 g initially to 3.2 g after 100,000 cycles. Four tests were run in each group and the results are repeatable. Al_2O_3-TiC sliders coated with carbon by cathodic arc deposition exhibit greater wear durability than those that are uncoated.

6. CONCLUSIONS

a) Cathodic arc deposition has been developed into a technique suited for magnetic storage applications due to improved macroparticle filtering.

b) Amorphous hard carbon films deposited by this method exhibit a very high hardness and elastic modulus, very low roughness, low coefficient of friction, high sp^3 content, and high mass density. The properties of the films can be varied over a great range by controlling the ion energy which is one of the key parameters which determines the film properties. The film properties can be modified further by operating the plasma source in a gaseous environment and thus incorporating dopants such as nitrogen, hydrogen, etc. It is expected that cathodic arc films will also show a superior corrosion protection due to their high mass density and smoothness.

c) Several applications to head/disk interface tribology demonstrate the extraordinary properties of these films and make it a promising candidate for future magnetic storage devices including contact recording.

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The authors wish to acknowledge, with thanks, the National Storage Industry Consortium (NSIC) for its support of this work. A portion of this work was sponsored by the Defense Advanced Research Projects Agency (DARPA) under Grant No. MDA972-93-1-0009 with NSIC. The authors wish to express their thanks to George M. Pharr, Daniel L. Callahan, Shaun D. McAdams, Ting Y. Tsui, John Robertson, Ravi Silva, Joel W. Ager, André Anders, Bo Wei, Kyriakos Komvopoulos, Zhi Wang, and Kin Man Yu for their help in characterizing the cathodic arc deposited carbon films. Another portion of this work was supported by the U.S. Department of Energy, Division of Advanced Energy Projects, under contract No. DE-AC03-76SF00098.

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TABLES

Table 1: Summary of the properties of cathodic arc deposited amorphous hard carbon films.

Pulsed bias voltage (V)	sp^3 fraction (%)	Mass density (g/cm^3) EELS	Mass density (g/cm^3) RBS and profilometry	Hardness (GPa)	Elastic modulus (GPa)	Compressive stress (GPa)	Coefficient of friction
0	81	2.91	2.6	23	200	7.5	0.148
-100	85	3.00	2.75	59	400	10.5	0.099
-500	47	2.18	2.5	27	235	6.9	0.114
-2000	39	2.14	2.2	20	190	3.1	0.158
Reference	[11]	[11]	[9]	[9, 11]	[11]	[12]	[9]

CAPTIONS FOR FIGURES

Fig. 1: Schematic arrangement of plasma source, improved macroparticle filter, and substrate.

Fig. 2: TEM cross-section image of hard phase/soft phase amorphous carbon multilayer on silicon. Top left - silicon substrate, bottom right - glue for sample preparation.

Fig. 3: Stress of single layers of soft and hard carbon in comparison to multilayer structures containing soft and hard carbon layers in different thickness ratios.

Fig. 4: Coefficient of friction as a function of the number of revolutions during continuous sliding test for unmodified and modified heads.

Fig. 5: Wear depth versus wear cycles for two different loads.

Fig. 6: Worn volume at the longitudinal facet as a function of time for a cathodic arc coated and sputter coated disks.

Fig. 7: Average friction as a function of number of cycles for the uncoated and coated slider.

Fig. 8: Touch down velocity as a function of number of cycles for the uncoated and coated slider.

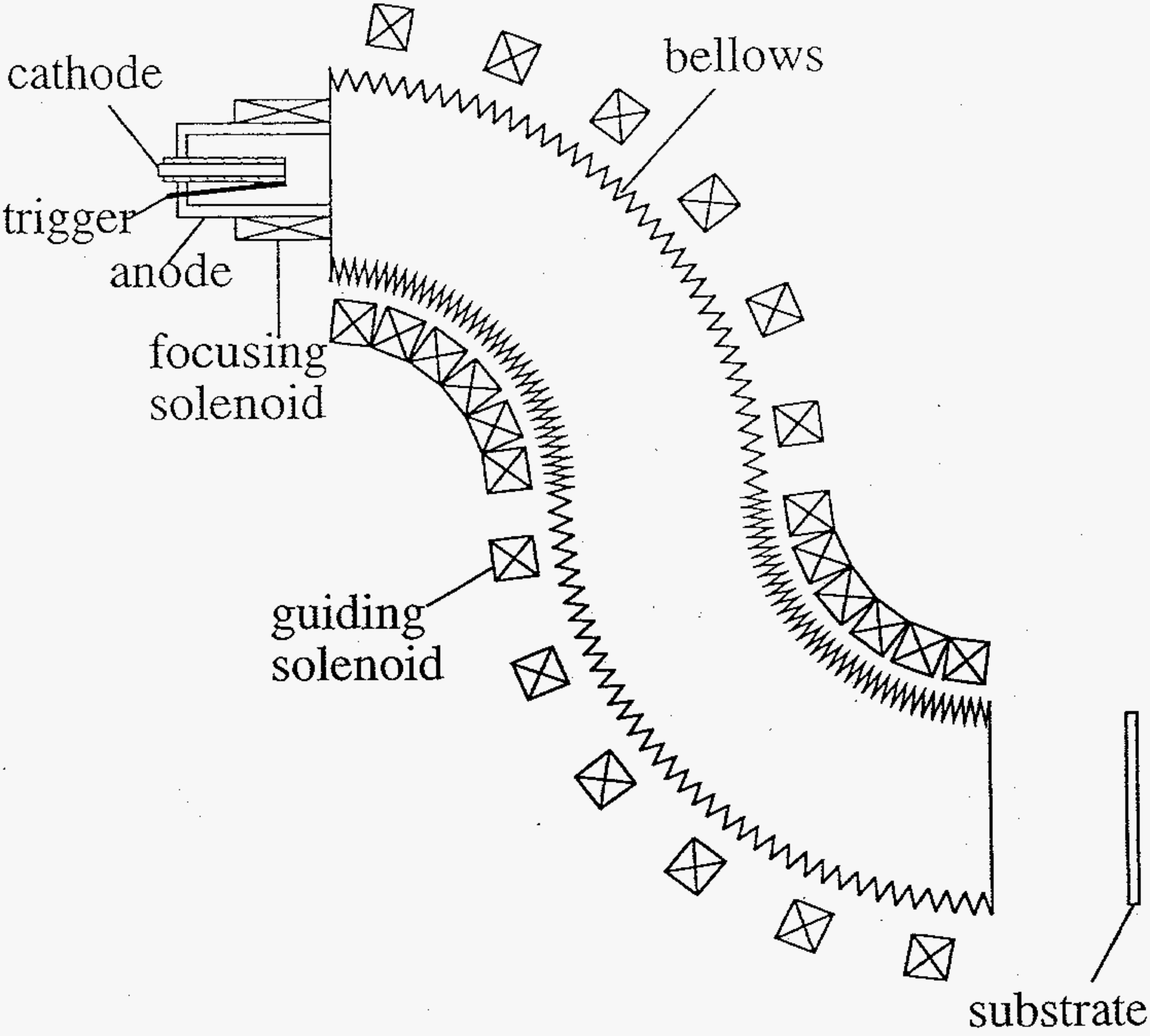

cathode
trigger
anode
focusing solenoid
bellows
guiding solenoid
substrate

Fig. 1

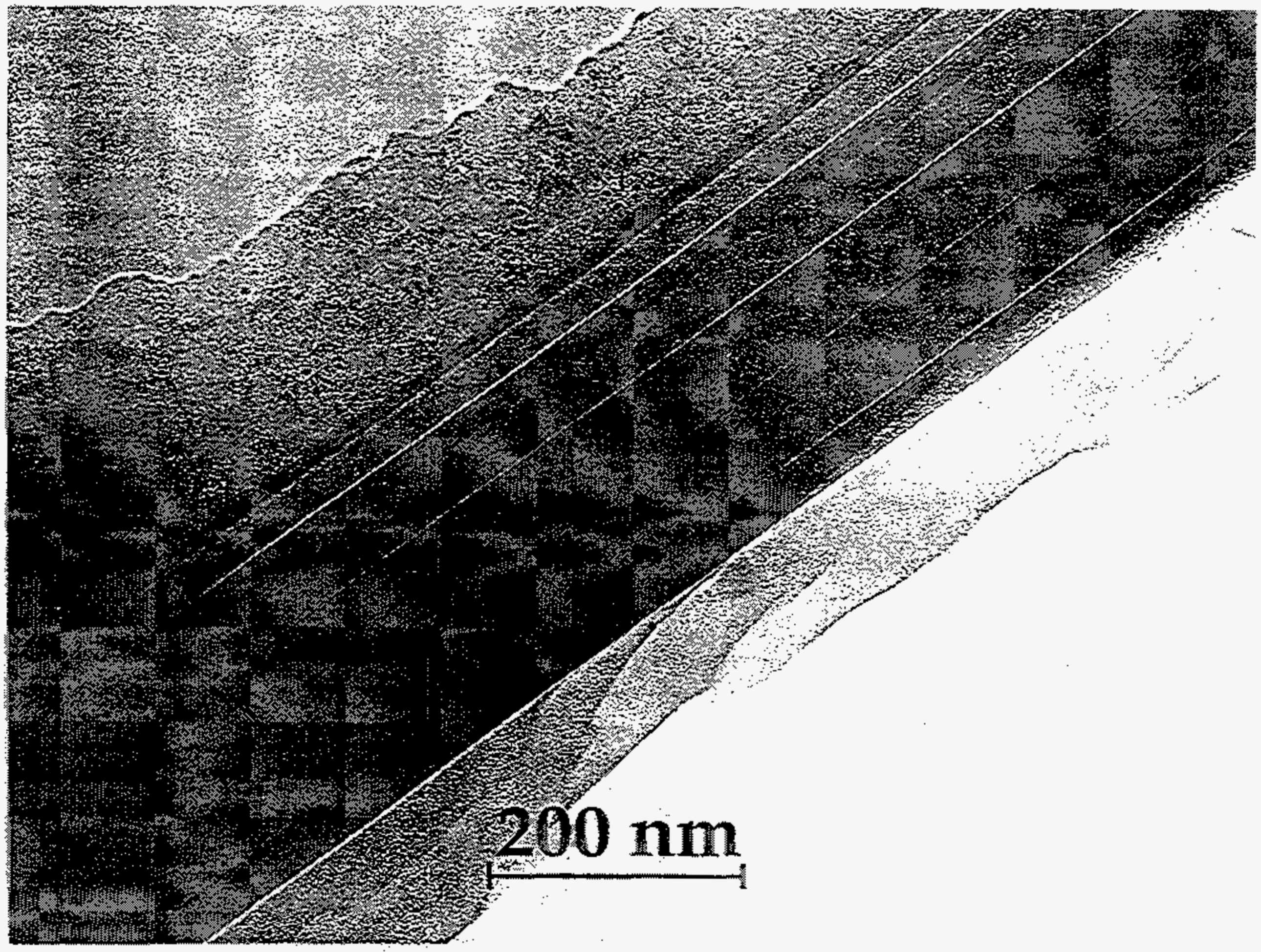


Fig. 2

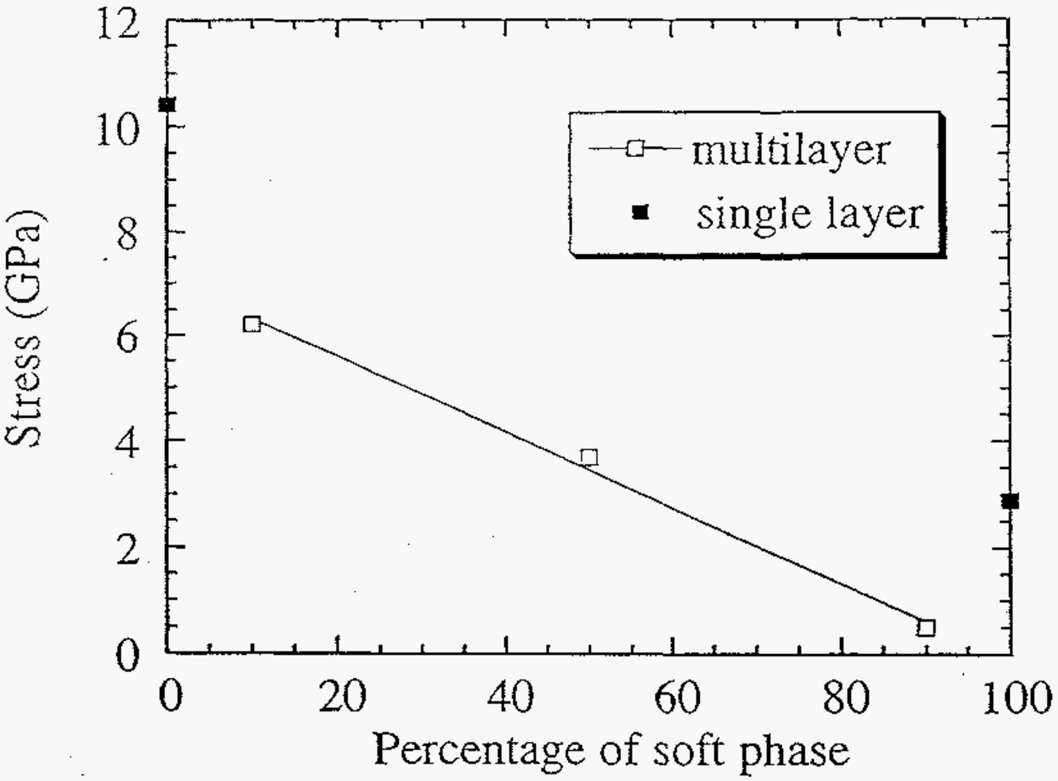

12
10
8
6
4
2
0
Stress (GPa)
0
20
40
60
80
100
Percentage of soft phase
multilayer
single layer

Fig. 3

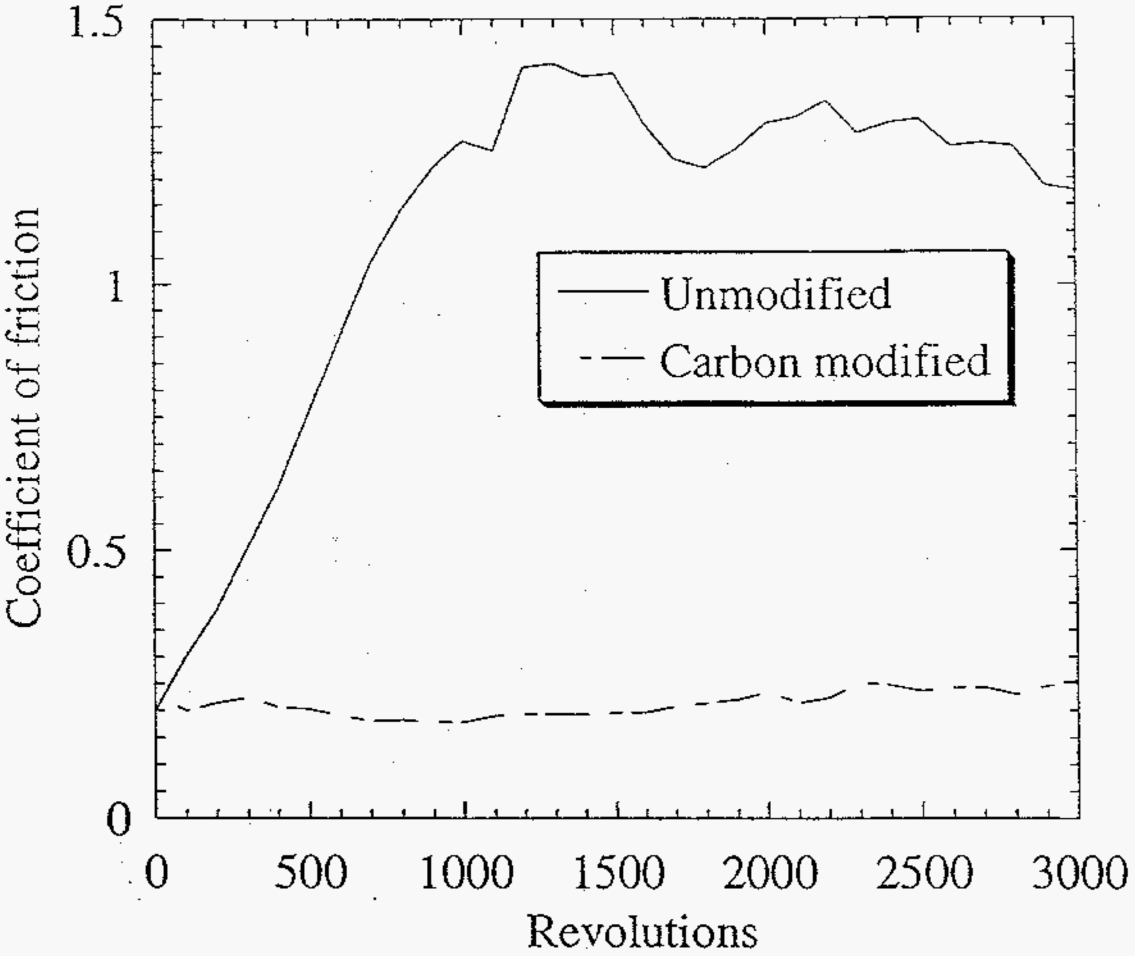
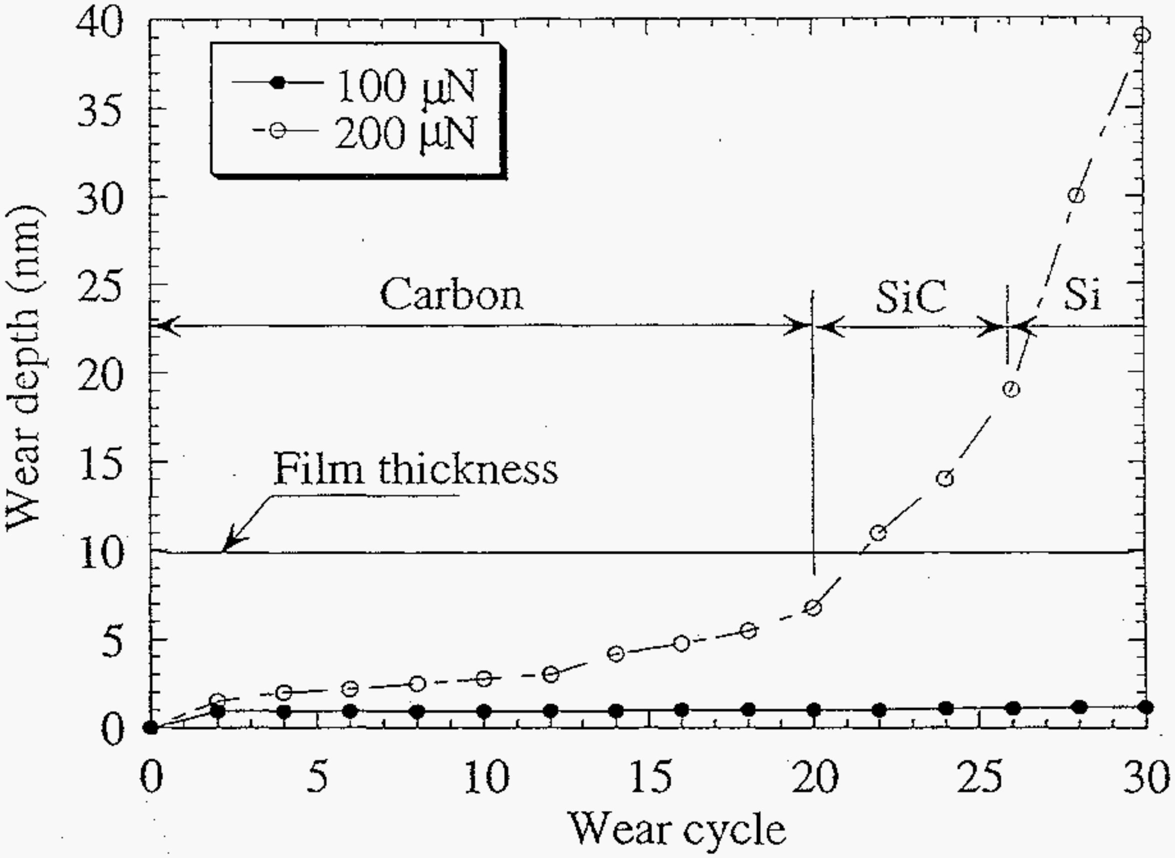
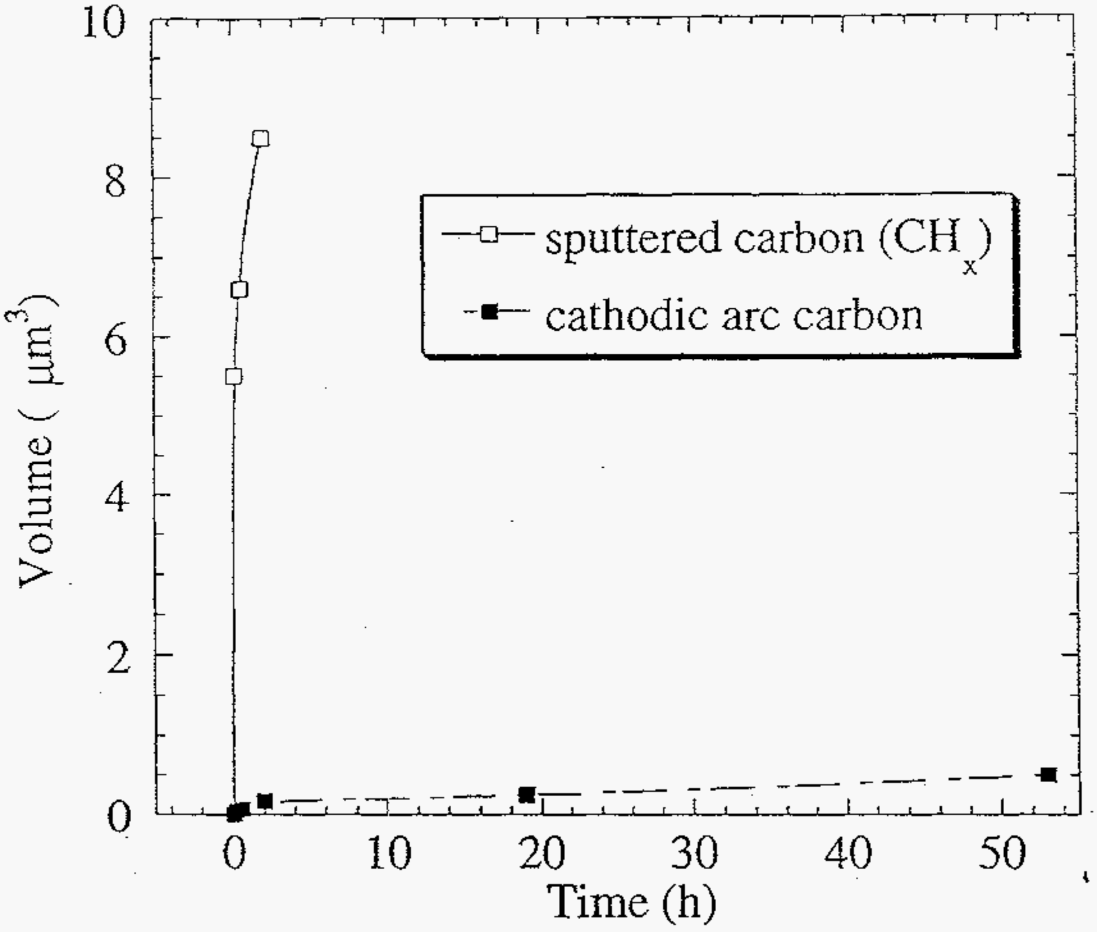

1.5
1
0.5
0
0
500
1000
1500
2000
2500
3000
Revolutions
Coefficient of friction
Unmodified
Carbon modified

Fig. 4



100 µN
200 µN
Carbon
SiC
Si
Film thickness
Wear depth (nm)
Wear cycle
0
5
10
15
20
25
30
35
40

Fig. 5



10
8
6
4
2
0
Volume (μm³)
0
10
20
30
40
50
Time (h)
sputtered carbon (CHx)
cathodic arc carbon

Fig. 6

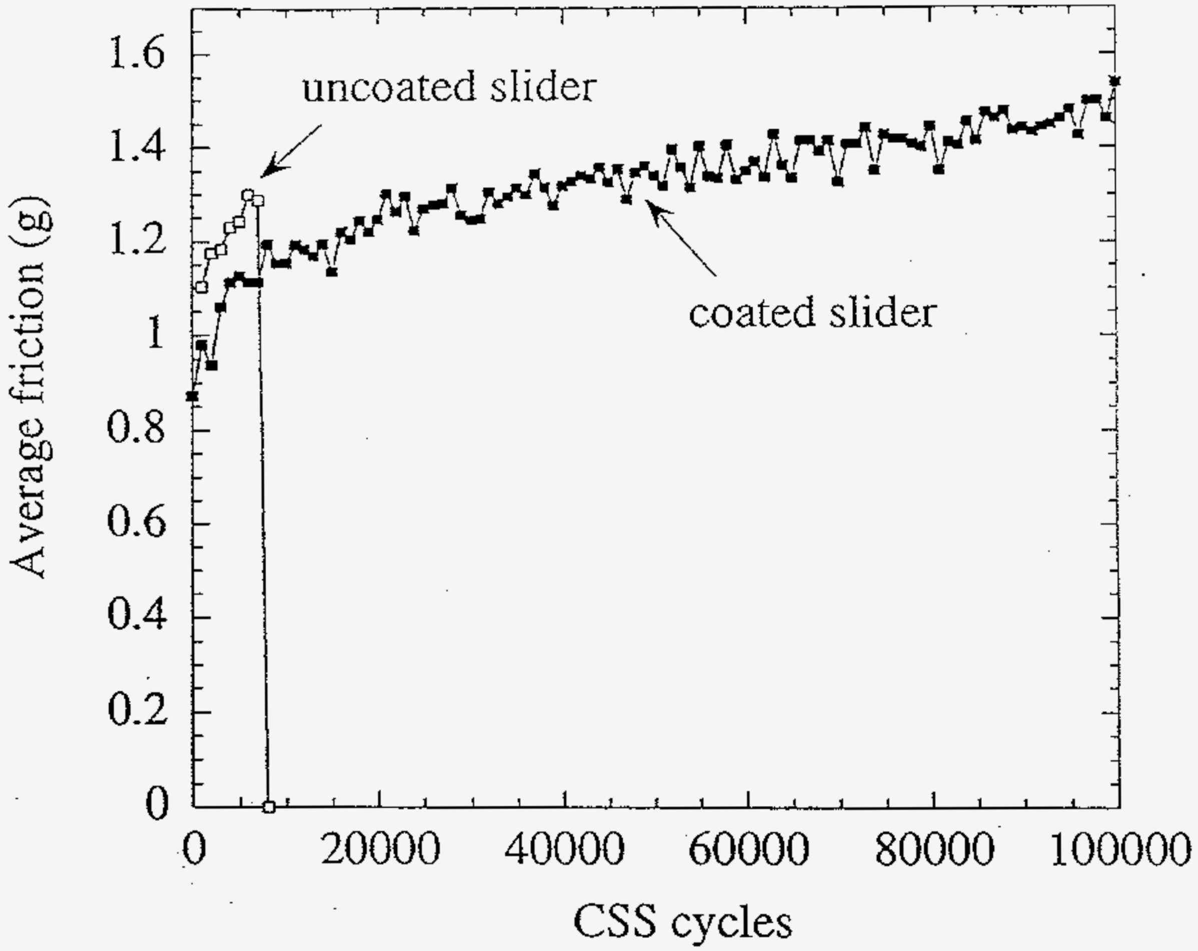

uncoated slider
coated slider
Average friction (g)
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
CSS cycles
0
20000
40000
60000
80000
100000

Fig. 7

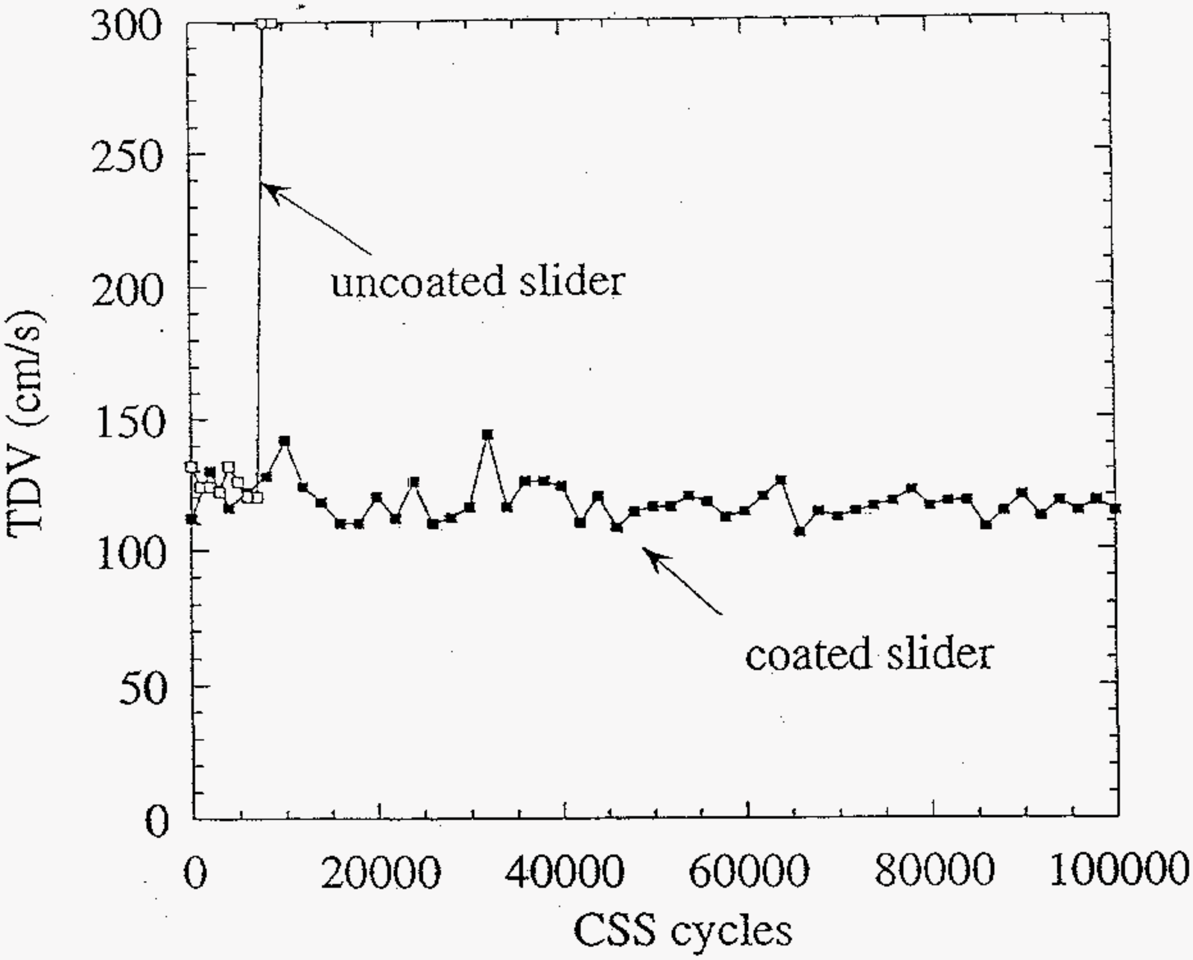

300
250
200
150
100
50
0
TDV (cm/s)
uncoated slider
coated slider
0
20000
40000
60000
80000
100000
CSS cycles

Fig. 8