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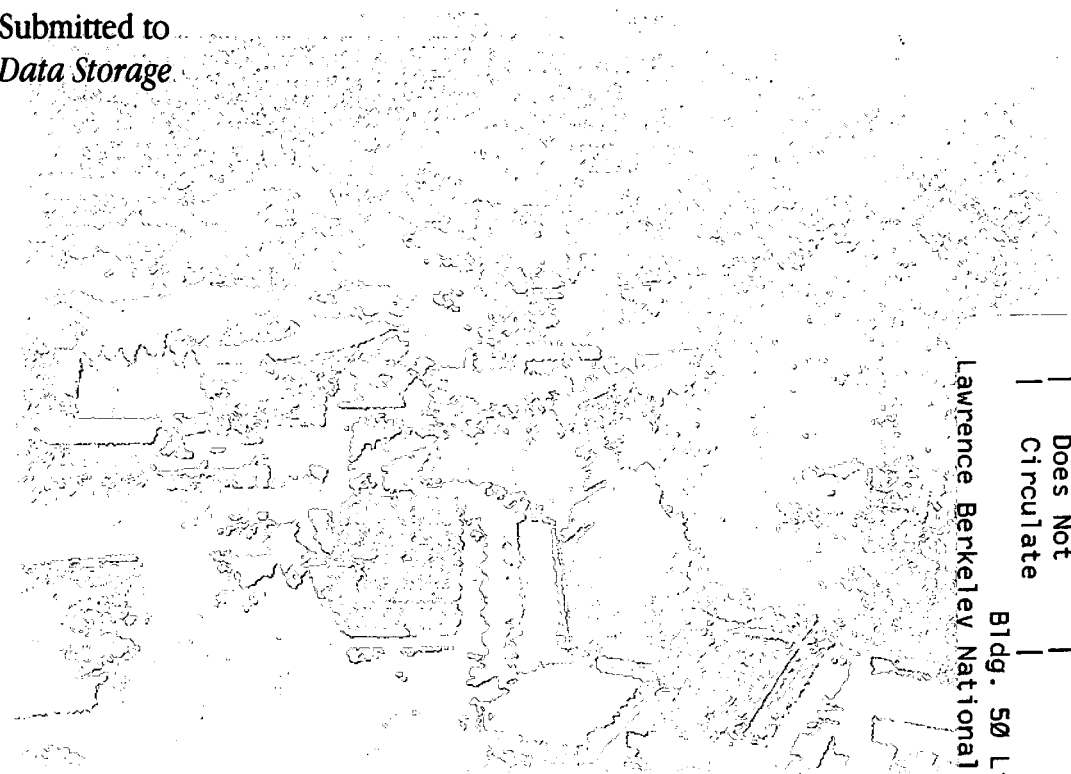
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Simone Anders, Ian G. Brown,
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Accelerator and Fusion
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CATHODIC ARC DEPOSITED DIAMOND-LIKE CARBON FILMS FOR HEAD-DISK TRIBOLOGY APPLICATIONS

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Abstract

Hydrogen-free, amorphous hard carbon films formed by filtered cathodic arc deposition are the hardest amorphous carbon films produced by any present deposition method, with a hardness approaching that of diamond. This paper describes the cathodic arc deposition equipment including new, improved filter and plasma homogenizer arrangements. The film properties of cathodic arc deposited diamond-like carbon depend strongly on the ion energy which in turn can easily be controlled by substrate biasing. Cathodic arc carbon films have been deposited onto computer hard disks and sliders, leading to remarkably improved tribological performance, which is reported on in this paper. Film properties as a function of the ion energy are summarized as well.

1. Introduction

A cathodic arc discharge is a high current, low voltage discharge which transforms the material of the cathode into the plasma state. The plasma production is a non-equilibrium process, and the cathodic arc plasma is fully ionized, often containing multiply charged ions. This plasma is used in cathodic arc deposition for the formation of thin films of the cathode material (metals, carbon) when the plasma source operates in vacuum, or compounds of the cathode material and the ambient gas (such as metal oxides and nitrides) if the plasma source operates in a reactive gas. Cathodic arc deposition is an established technique for the formation of metallurgical and hard protective coatings such as TiN.

It was observed 20 years ago that vacuum arc deposited amorphous diamond-like carbon is extremely hard, and has a very high elastic modulus and mass density [1,2]. A major obstacle to successful application of cathodic arc deposited amorphous carbon films to high-tech applications has been the contamination of the films by macroscopic carbon particles (typical diameter of the order a micron). Much effort has been invested in the improvement of the deposition equipment by implementing various types of macroparticle filters, and today these films can be deposited using the best filters and most advanced sources with a quality and cleanliness that matches to the needs of the disk drive industry [3].

In the present paper we give an overview of cathodic arc deposition sources and filters, and describe the properties of cathodic arc deposited amorphous hard carbon films. The potential benefits of the application of these films to improvement of the head-disk interface are demonstrated by a number of experiments with cathodic arc coated sliders and disks.

2. Cathodic arc deposition equipment

Cathodic arc plasma sources typically consist of a cathode surrounded by a cylindrical anode, and some mechanism to trigger the discharge. The cathodes are usually cylindrical or conical, and for sources operating dc or at high duty cycle the cathode needs to be water-cooled. The cylindrical anode serves as an electron collector for the discharge, and it needs to be cooled also for dc or high duty cycle operation. There are two major trigger principles - mechanical triggering and triggering by a high voltage spark. For plasma deposition sources mechanical triggering is preferred because it is very robust and well suited for triggering in the 1 Hz and below range. High voltage spark triggering is typically applied in vacuum arc ion sources with an arc trigger rate in the 10-100 Hz range. Typical arc currents are in the 50-200 A range, and the arc voltage is about 20V. Fig. 1 shows a cathodic arc plasma source (developed at Lawrence Berkeley National Laboratory, LBNL) with water-cooling of the cathode and anode and with an electro-mechanical trigger. The cathode has a 5 cm diameter, and the source can operate for hours in the dc mode. Much smaller source have also been developed at LBNL, operating in a pulsed mode with low duty cycle of 1-5% and dedicated mainly to research applications.

To obtain high quality films the source needs to be equipped with a macroparticle filter. Most laboratories use 90° bent magnetic ducts [4-7], but 20° [8] and 45° [9] magnetic filters have also been applied. The filtering is based on the effect that electrons in the vacuum arc plasma are magnetized in the magnetic field of the filter (typically in the 10-100 mT range), gyrating with very small radius (microns) around the magnetic field lines of the filter field. Due to the strong electrostatic interaction between electrons and ions, the electrons draw the ions along with them through the filter by the ambipolar field established. The macroparticles, which have charge to mass ratio that is many orders of magnitude smaller than the ions, are not ducted and are deposited on the filter

walls. Because the carbon macroparticles are solid there is a certain chance of reflection, and additional measures such as the insertion of baffles in the filter have to be taken to prevent multiple reflection. Fig. 2 shows a free-standing filter in operation. This kind of filter was used for research to study the plasma transport properties. Deposition filters normally have walls and baffles inside. This also allows biasing the wall, which can further improve the filter efficiency.

The plasma ion current generated a vacuum arc is about 10% of the arc current, and most ions can be extracted from a well designed source. An optimized 90° filter has a typical ion transmission of about 25% [10], so the total output of a filtered source of this kind is about 2.5% of the arc current. If this ion flux is deposited over an area of about 100 cm², the deposition rate is typically 50-200 nm/s. This rate can be increased or reduced by varying the arc current and source-sample distance.

For very high quality coatings an improved filter has been developed [3] which consist of two 90° bent magnetic filters in series. This filter allows the deposition of films with very low macroparticle content and has been used for all the applications to head-disk tribology described in section 4. For further improvement of the deposition quality a magnetic multicusp (magnetic bucket) can be applied at the filter exit [11]. The magnetic multicusp leads to a homogenization and flattening of the plasma density distribution and therefore the deposition profile. Application of such a "plasma homogenizer" results in a uniformity of the film thickness that varies less than 5% over a substrate of 10 cm diameter. This bucket can be scaled up or down depending on the required sample size and film homogeneity. Fig. 3 shows schematically the plasma source, improved macroparticle filter and magnetic multicusp. The improved filter has a transmission similar to the product of two 90° filter, i.e., about 6%. This leads to a deposition rate of the overall system, with the source operated dc, of about 10-50 nm/s.

3. Diamond-like carbon films produced by cathodic arc deposition

Cathodic arc deposited amorphous hard carbon (diamond-like carbon, or dlc) films exhibit properties which are of great interest for tribological applications. The film properties can be controlled by varying the ion energy, and this is easily achieved by substrate biasing. But even films deposited without bias are close in properties to the films deposited at optimum bias, since the ion energy which leads to the highest hardness and sp^3 content is about 100 eV [12-14]; this is valid not only for cathodic arc deposition but has been found also for other deposition techniques such as laser ablation, ion beam deposition, and plasma beam deposition.

The ion energy was controlled during the deposition of the diamond-like carbon films by applying a repetitively pulsed bias to the substrates. Typically, the bias pulse length was 2 μ s and the pulse off-time was 6 μ s. The arc source was operated in a pulsed mode with 5 ms pulse duration and 1 Hz repetition rate; the substrate bias pulsing was gated on during the plasma pulse. A small cathodic arc plasma source with a 6 mm diameter cathode was used for our studies. Pulsed biasing has two advantages in comparison to dc biasing: Possible charge build-up at the sample surface during the bias pulse is removed between the pulses, and it is possible to apply much higher voltages without breakdown between sample and plasma. A number of film parameters have been measured as a function of the pulsed bias voltage.

sp³ fraction

The sp^3 fraction was measured by Electron Energy Loss Spectroscopy (EELS) by scaling the peak of the π^* states at 285 eV to the value of polycrystalline graphite (Fig. 4) [15].

Mass density

The mass density was measured in three different ways: by EELS scaling the valence plasmon to the plasmon of graphite [15]; by determining the number of carbon atoms per area by Rutherford Backscattering spectrometry (RBS) and the film thickness using a

profilometer [14]; and by depositing a film of known thickness on a pre-weighted wafer and weighing the wafer after deposition. All methods gave very similar results (Fig. 4).

Hardness and Elastic Modulus

The hardness and elastic modulus were measured by nanoindentation (Fig. 5) [15]. The numbers in Fig. 5 are comparative values rather than absolute hardness and elastic moduli, since the measurements were performed on 300 nm thick films which still show a substrate influence. For films of thickness 500 nm, a hardness of up to 88 GPa was measured. Remarkable is the very high elastic modulus indicating that these films show a very low brittleness.

Stress

The high hardness is correlated to a high stress in the films. This was measured by depositing on thin wafers and measuring the curvature before and after deposition (Fig. 6) [16]. An approach to reduce the stress while maintaining high hardness is described later in this paragraph.

Coefficient of Friction

The coefficient of friction was measured for sliding against diamond in room atmosphere (Fig. 6) [14].

All these measurements show that the most "diamond-like" character is obtained at a negative substrate bias of 100 V. The values for zero bias are not too far from the 100 eV bias films because the ions in the carbon vacuum arc have a "natural" energy of 20 eV. At higher bias the films are softer and more graphitic. This strong influence of the substrate bias on the film parameters led us to the idea of forming carbon-carbon multilayers of alternating "hard" and "soft" carbon (which is still very hard, at 20 GPa) [17, 18]. Fig. 7 shows a TEM image of such a multilayer. It was possible to reduce the stress below 1 GPa with this approach without losing hardness or elastic modulus.

Another successful approach to reducing the stress without losing the hardness is the incorporation of other elements such as silicon or nitrogen [19, 20].

The thermal stability of cathodic arc amorphous hard carbon films deposited at 100V bias was tested by annealing in air and in vacuum [21,22]. 450 nm thick films annealed in air were investigated by Raman spectroscopy before and after annealing. The Raman spectra showed no change after heating in air for 2 h up to 525 °C except reduction of the total signal due to films thickness loss by oxidation. The films started to oxidize around 450 °C and were completely oxidized at 550 °C. During annealing in vacuum we recorded the Near Edge X-ray Absorption Fine Structure (NEXAFS) spectrum at the carbon K edge. No change in the spectrum was found for annealing up to 700 °C, and for higher temperatures up to 850 °C a small change in the spectrum was recorded. A peak appeared at the wavelength of the graphite plasmon indicating the graphitization of the sample surface. The Raman spectra after 850 °C annealing in vacuum showed a very small modification (appearance of a small shoulder at 1330 cm^{-1} indicating slight graphitization) whereas nanoindentation measurements show no change in hardness and elastic modulus after annealing up to 850 °C in vacuum. The differences in the results of NEXAFS, Raman spectroscopy, and nanoindentation can be explained by the different surface sensitivity of the methods. The graphitization seems to start at the surface where it is detected by NEXAFS and Raman spectroscopy whereas the bulk properties measured by nanoindentation remain stable. These experiments demonstrate the high thermal stability of these films.

Atomic Force Microscopy (AFM) was used to study the roughness of cathodic arc deposited films [23]. AFM studies of the surface of a 30 nm thick diamond-like carbon film prepared at an ion energy of 100 eV showed an rms surface roughness of 0.07 ± 0.01 nm, which when corrected for the inherent roughness of the substrate reduces to 0.05 ± 0.02 nm. Thicker dlc films (200 nm) showed the same surface roughness, and 30 nm films prepared using a higher ion energy of 2 keV showed a slightly less smooth surface

with roughness 0.08 ± 0.02 nm (corrected). This difference in the smoothness of films prepared at 100 eV and at 2 keV is only marginally significant, but agrees with the fact that the most diamond-like character of the films is achieved at 100 eV ion energy.

Cathodic arc deposited films have been well characterized themselves. For head/disk interface applications it will be of great importance how the films interact with the magnetic layer and the lubricant, and how their properties influence the tribochemistry occurring during wear.

4. Application to head-disk interface tribology

Increased magnetic data storage density requires smaller spacing between disk and slider, and thinner overcoats to reduce the magnetic spacing. This in turn requires better protective overcoats with lower wear ("no-wear" head-disk interface being the ultimate goal). Hydrogenated or nitrogenated sputtered amorphous carbon films are the choice for protective overcoat in the presently used technology, but for thicknesses below about 7 nm the durability of the films is drastically decreased. Intense research is being pursued for novel overcoat materials, and cathodic arc amorphous hard carbon is one of the new candidates [24]. We have applied cathodic arc films to disks and sliders, and some of the results are described in this section.

Two-rail sliders consisting of 70% Al_2O_3 and 30% TiC were exposed to a cathodic arc carbon plasma and pulse-biased to -2000V for a total carbon dose of $2 \times 10^{16} \text{cm}^{-2}$. This surface treatment leads to a depth profile of the carbon as shown in Fig. 8. Wear testing for sliding against unlubricated carbon-coated disks was performed at a sliding speed of 3 cm/s and a normal load of 16g in air of 40% humidity and 27°C. As shown in Fig. 9, the coefficient of friction is much lower and much more stable for the modified slider in comparison to the unmodified slider.

In another experiment Al₂O₃-TiC sliders were coated with cathodic arc amorphous carbon to a thickness of 2 nm using a negative pulse bias of 100V to form a film with the highest sp³ content. Contact Start Stop (CSS) testing was performed on 95 mm textured disks lubricated with 1 nm Z-dol 2000. Fig. 10 shows that the average friction for the coated slider is very stable and the slider lasts for at least 100,000 cycles (when the test was terminated) whereas the uncoated slider fails after less than 10,000 cycles.

Super-smooth 65 mm hard disks were coated with 10 nm cathodic arc deposited amorphous hard carbon films. The negative pulse bias during deposition was 100V. The disks were lubricated after deposition with 1.5 nm Z-dol 2000. Wear tests 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. 11 shows the worn volume as a function of time, comparing the cathodic arc carbon coated disk with a disk coated with a 10 nm hydrogenated, sputtered amorphous carbon 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.

5. Summary

Cathodic arc deposition has been developed into a technique suited for magnetic storage applications due to improved macroparticle filtering. A number of companies are looking into the commercialization of cathodic arc plasma sources with high quality macroparticle filters, and it can be expected that they will be available on the market in the near future.

Amorphous hard carbon films deposited by cathodic arc deposition 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 very wide 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 superior corrosion protection due to their high mass density and smoothness. Several applications to head/disk interface tribology demonstrate the extraordinary properties of these films and make cathodic arc deposited amorphous hard carbon films a promising candidate for future magnetic storage devices including contact recording.

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Biographies

Simone Anders studied physics at Humboldt University, Berlin, and plasma physics at Moscow State University, Russia, and holds the MS degree ('84) and Ph.D. degree ('88) in physics from Humboldt University. She is Staff Scientist at Lawrence Berkeley National Laboratory (LBNL) where she was working in the field of vacuum arc plasma and ion sources, vacuum arc deposition, ion implantation and plasma immersion ion implantation. In 1996 she joined the Advanced Light Source (synchrotron) at LBNL where she is heading the Photoemission Electron Microscopy project. Her research interests include materials analysis and microscopy using X-rays.

Ian Brown is a Senior Physicist and Leader of the Plasma Applications Group at the Lawrence Berkeley National Laboratory of the University of California, Berkeley. His research involves the development of plasma and ion beam sources and their application for materials synthesis and modification. He received his Ph.D. in plasma physics from the University of Sydney and has held research and teaching positions at Princeton University, the University of California at Berkeley, and the Max-Planck Institute for Plasma Physics at Garching, Germany. He is author / co-author of over 200 research papers and his book, "The Physics and Technology of Ion Sources" (Wiley, 1989), has become a standard in the field. His work on vacuum arc ion sources and materials surface modification has led to two R&D100 awards and has resulted in a number of patents. He is a Fellow of the APS, the IEEE, the Institute of Physics UK, and the Australian Institute of Physics, and a member of the AVS, MRS, and the Society for Biomaterials.

Dr. C. Singh Bhatia is a Senior Technical Staff Member (STSM) in the Advanced Magnetic Recording Lab.(AMRL), Storage Systems Division of IBM at San Jose. Singh has worked extensively in the area of Head/Disk Interface (HDI) tribology. He has

published broadly in the field of HDI tribology. Singh is committee chairman of the Tribology of Magnetic Storage Systems, American Society of Mechanical Engineers, ASME. He is also a National Storage Industry Consortium (NSIC) team leader for the 10 Gb/In² & Beyond Tribology project.

David B. Bogy holds the William S. Floyd, Jr. Distinguished Professorship in Engineering at the University of California, Berkeley. He has been on the faculty of the Department of Mechanical Engineering since 1967, and has served as chair of ME since 1991. He is the founding Director of the Computer Mechanics Laboratory. He is author / co-author of over 200 archival journal papers. His work is in the area of solid mechanics, fluid mechanics, dynamics and tribology, especially as they relate to data storage systems, and in particular the head/disk interface of hard disk drives. He is a member of the National Academy of Engineers.

Captions for Figures

- Fig. 1: Cathodic arc plasma source (dismantled) with a titanium cathode and a mechanical trigger.
- Fig. 2: Macroparticle filter in operation.
- Fig. 3: Plasma source, improved macroparticle filter and magnetic multicusp (schematically).
- Fig. 4: sp^3 fraction and mass density of cathodic arc deposited amorphous hard carbon films as a function of the substrate bias.
- Fig. 5: Hardness and elastic modulus of cathodic arc deposited amorphous hard carbon films as a function of the substrate bias.
- Fig. 6: Stress and coefficient of friction of cathodic arc deposited amorphous hard carbon films as a function of the substrate bias.
- Fig. 7: Multilayer structure of hard and soft cathodic arc deposited amorphous carbon films.
- Fig. 8: Depth profile for carbon modification of sliders.
- Fig. 9: Coefficient of friction for continuous sliding against unlubricated disk in air for the modified and unmodified slider.

Fig. 10: Average friction during CSS testing of coated and uncoated slider.

Fig. 11: Worn volume at the longitudinal facet as a function of time for a cathodic arc coated and sputter coated disks.

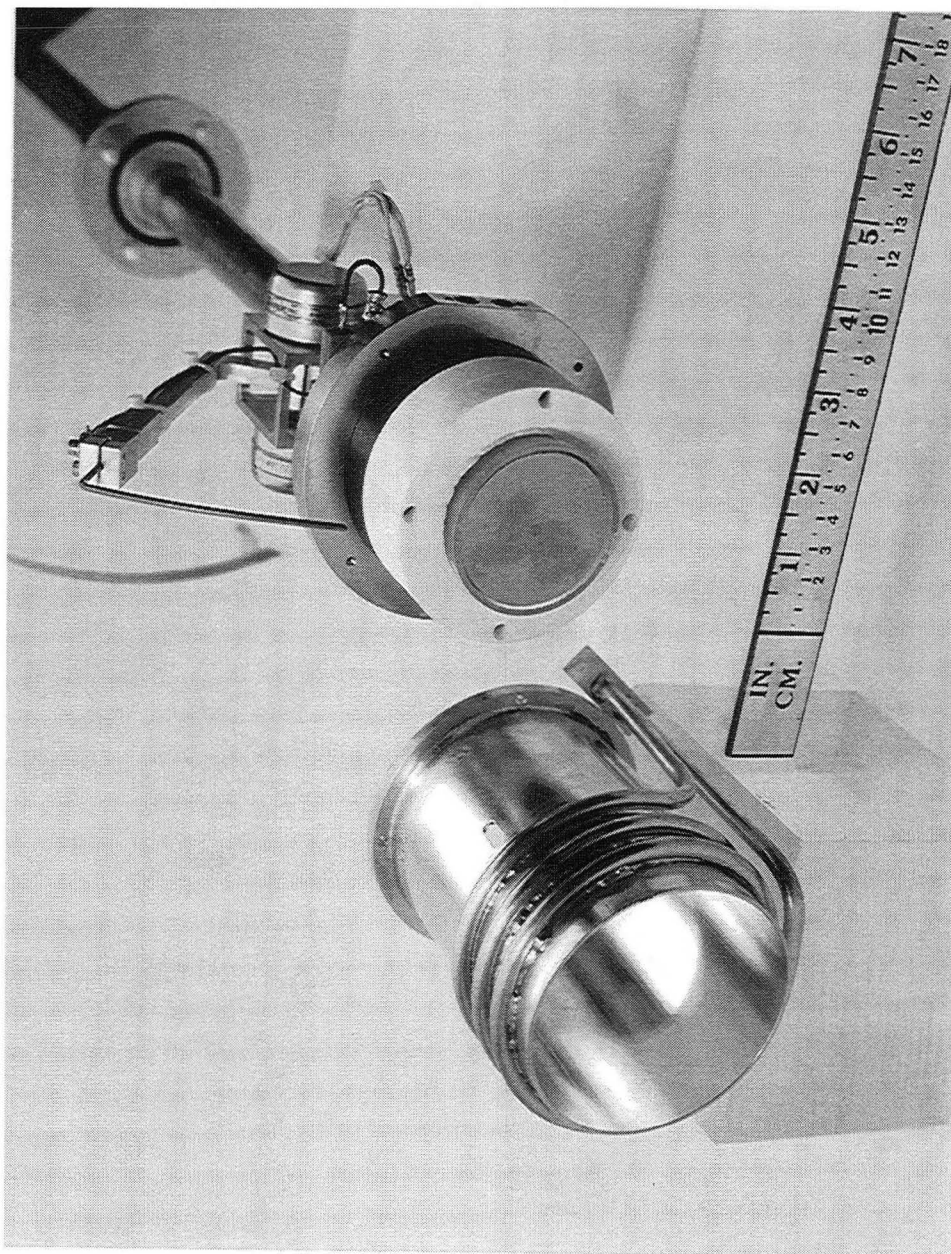


Fig. 1

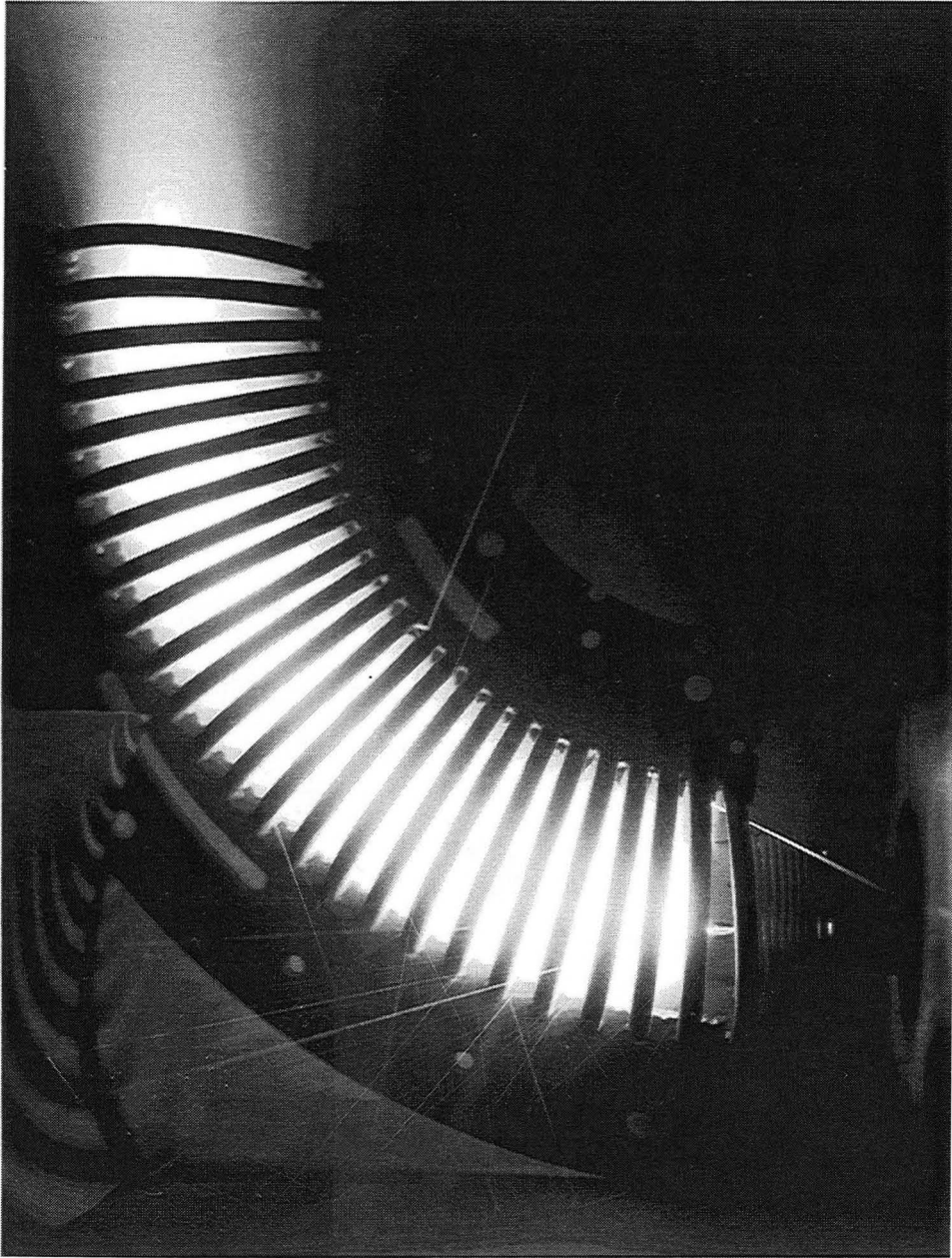
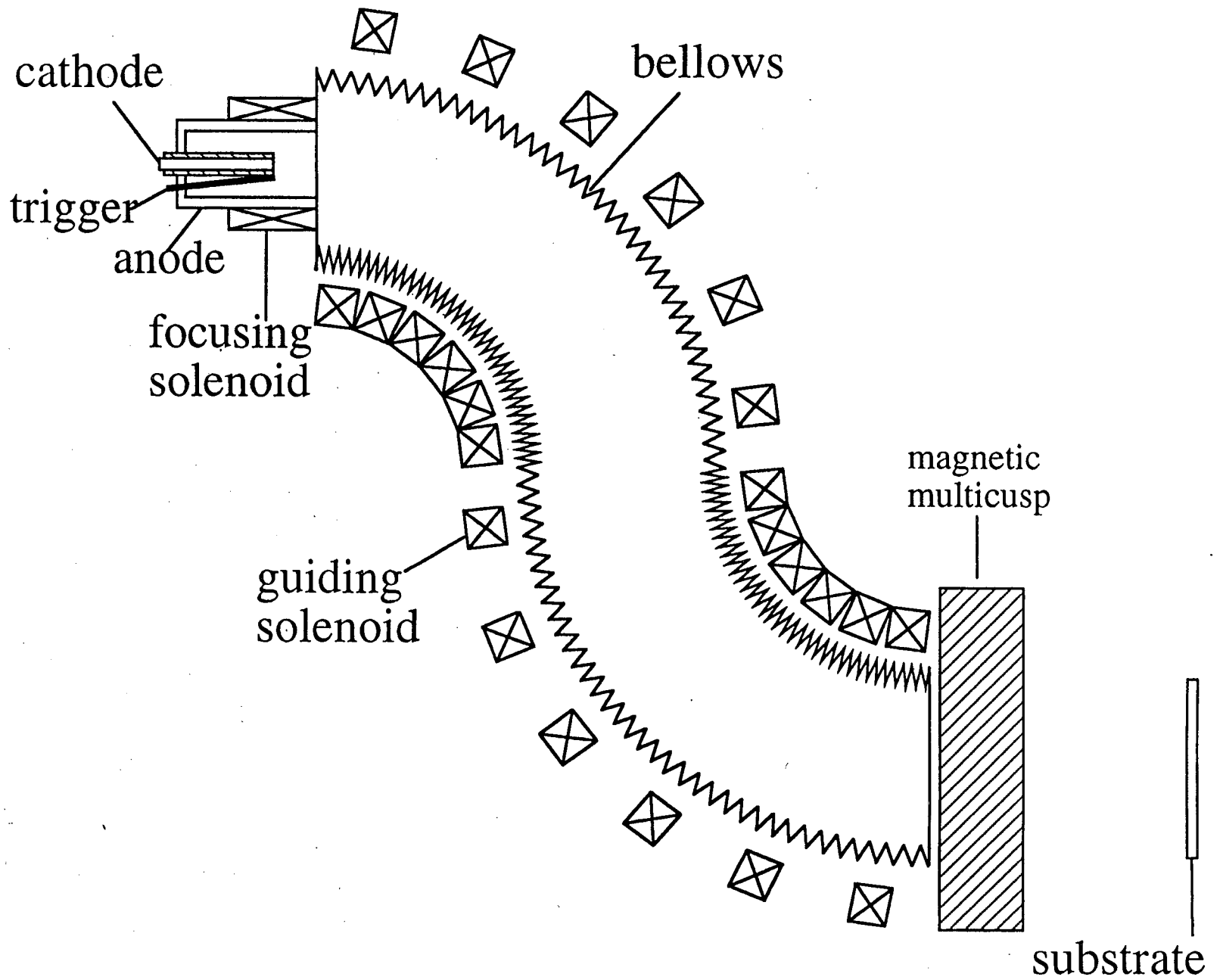


Fig 2



Fi. 3

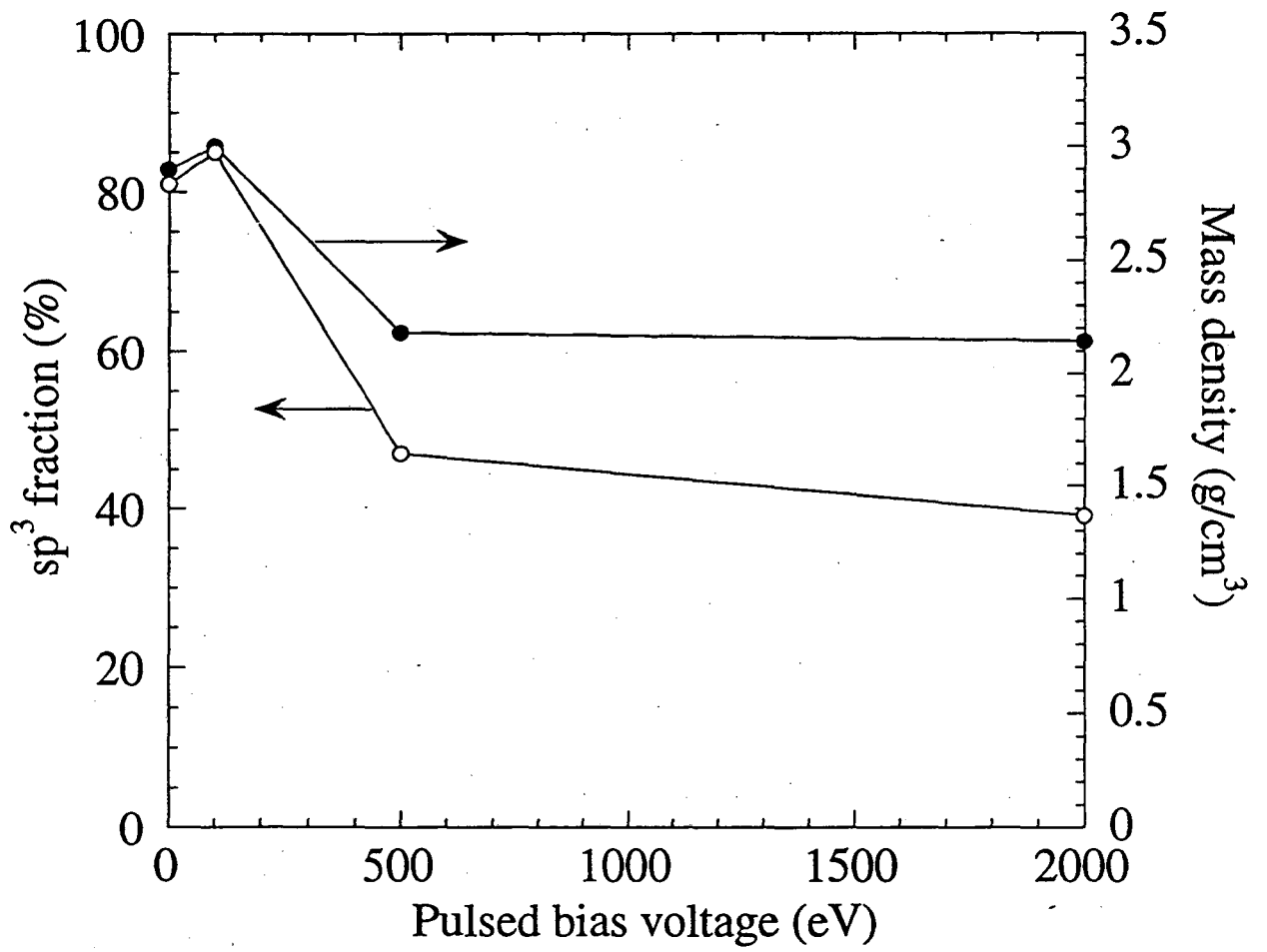


Fig. 4

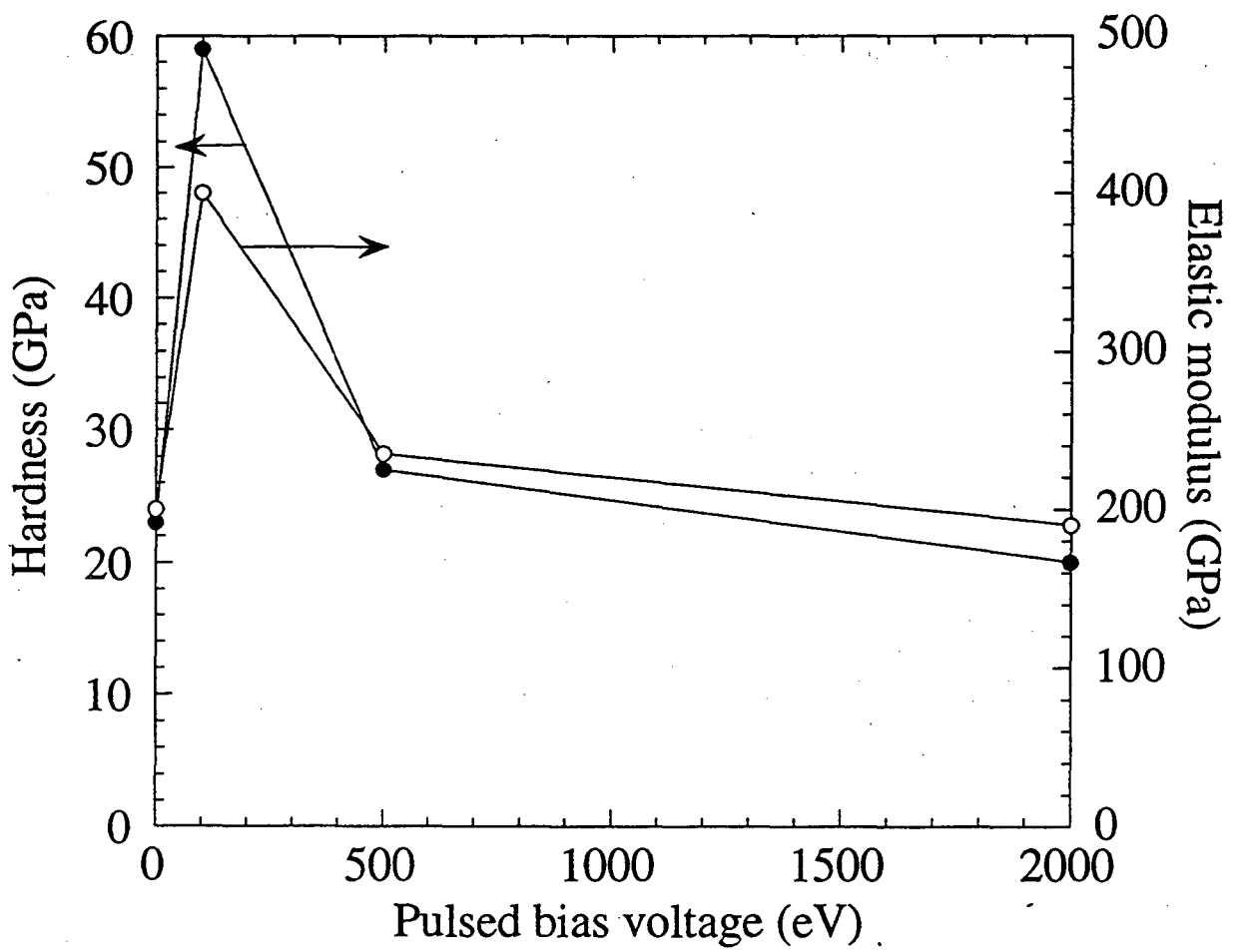


Fig. 5

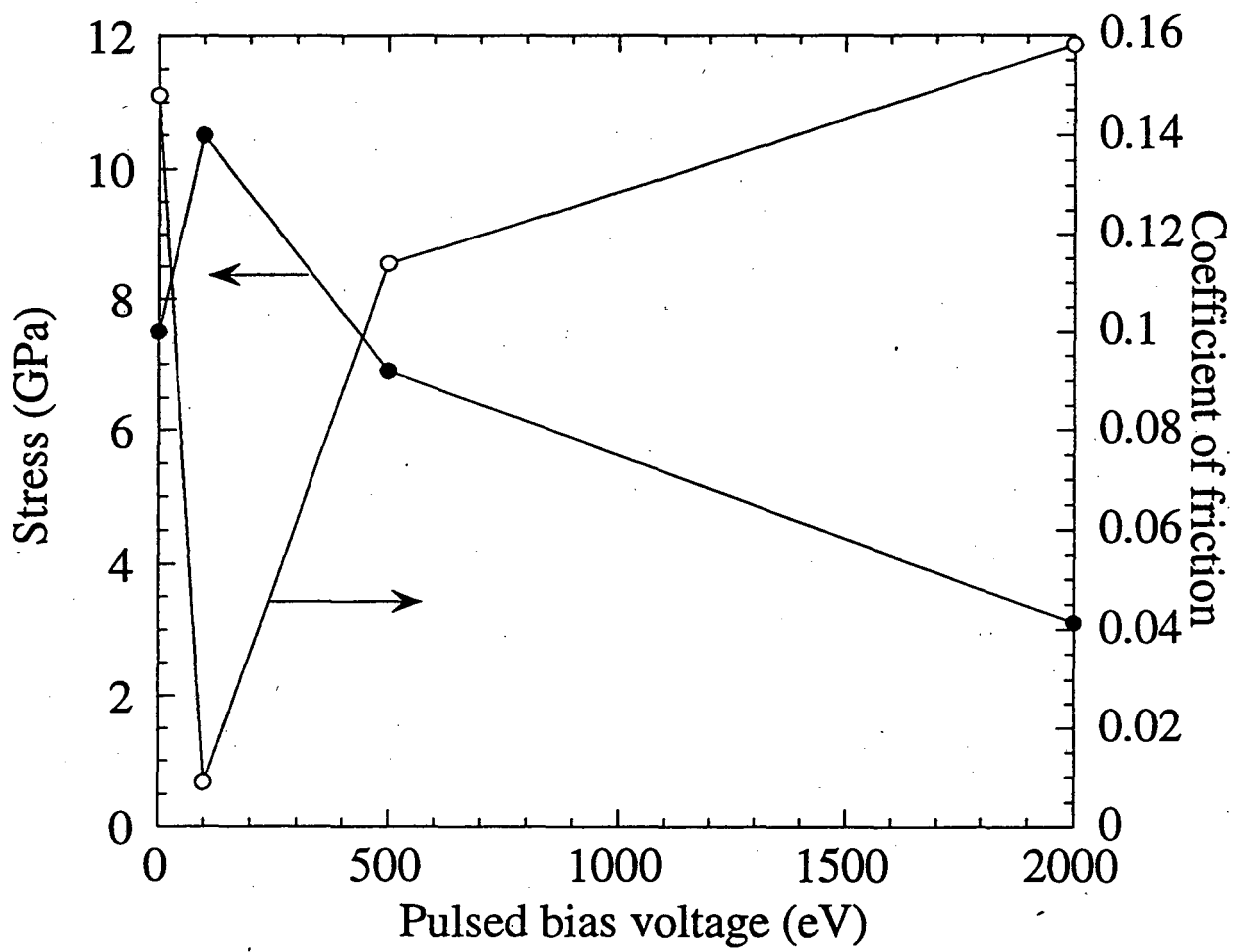


Fig. 6

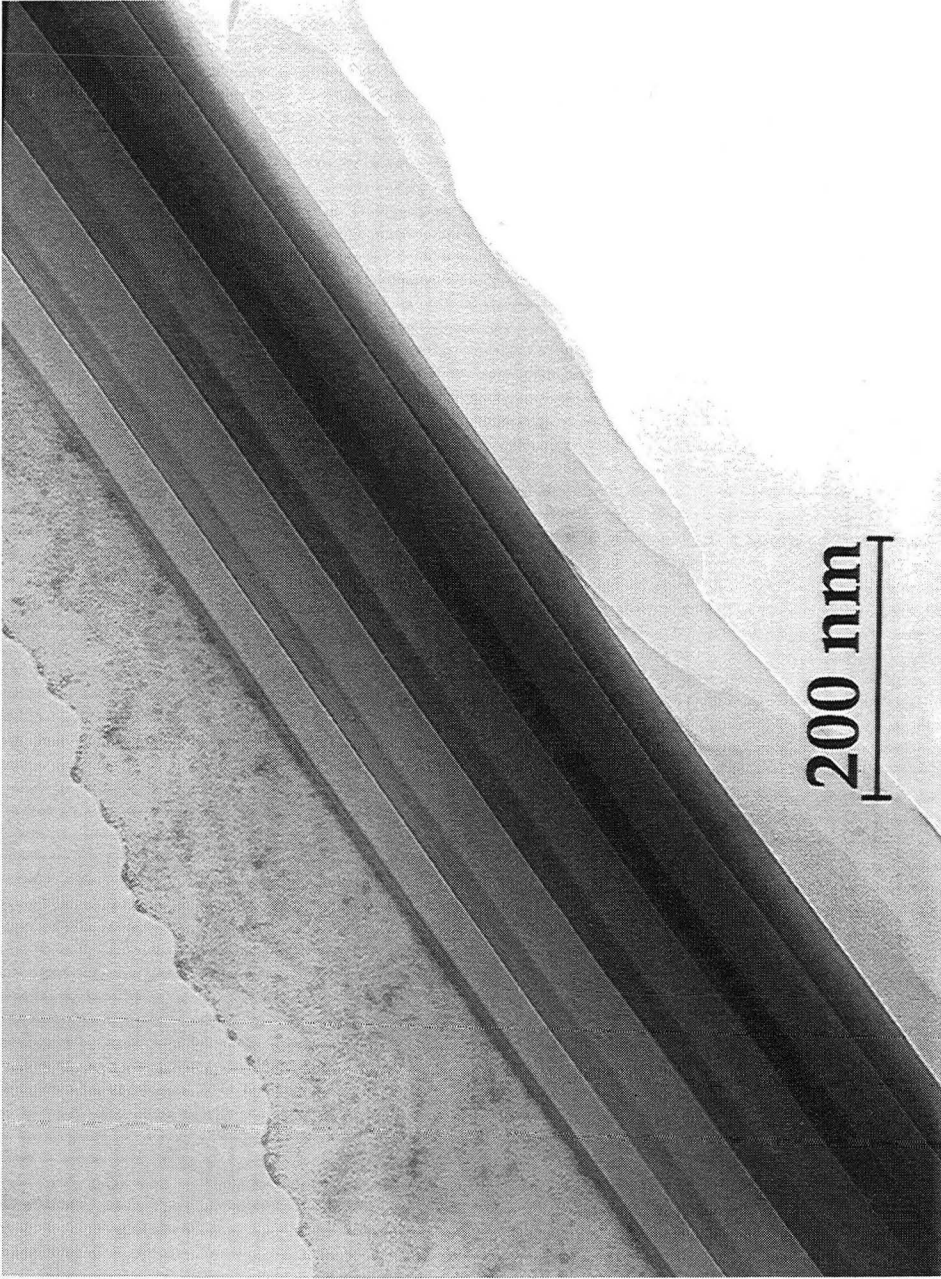


Fig. 7

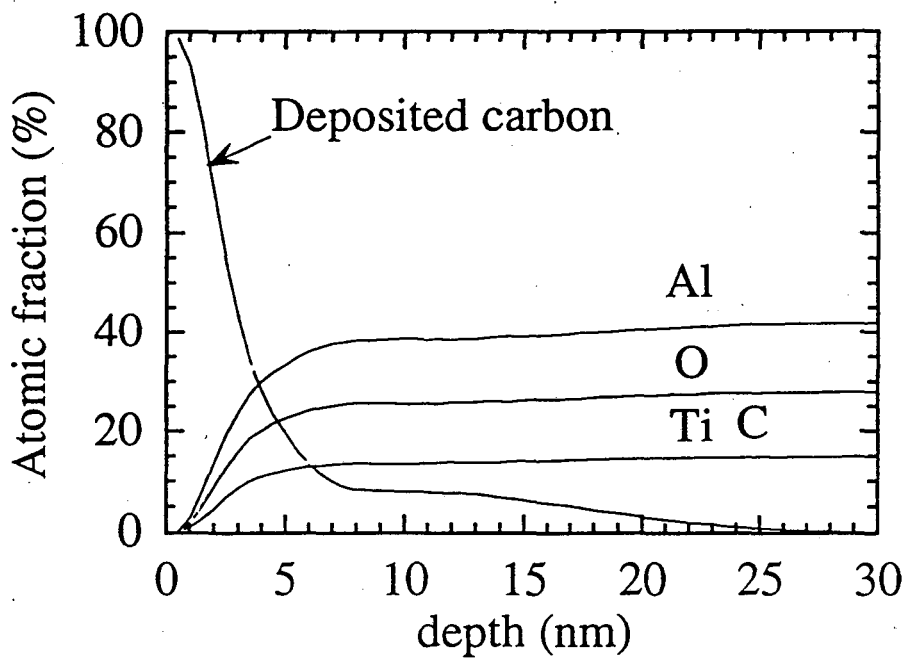


Fig. 8

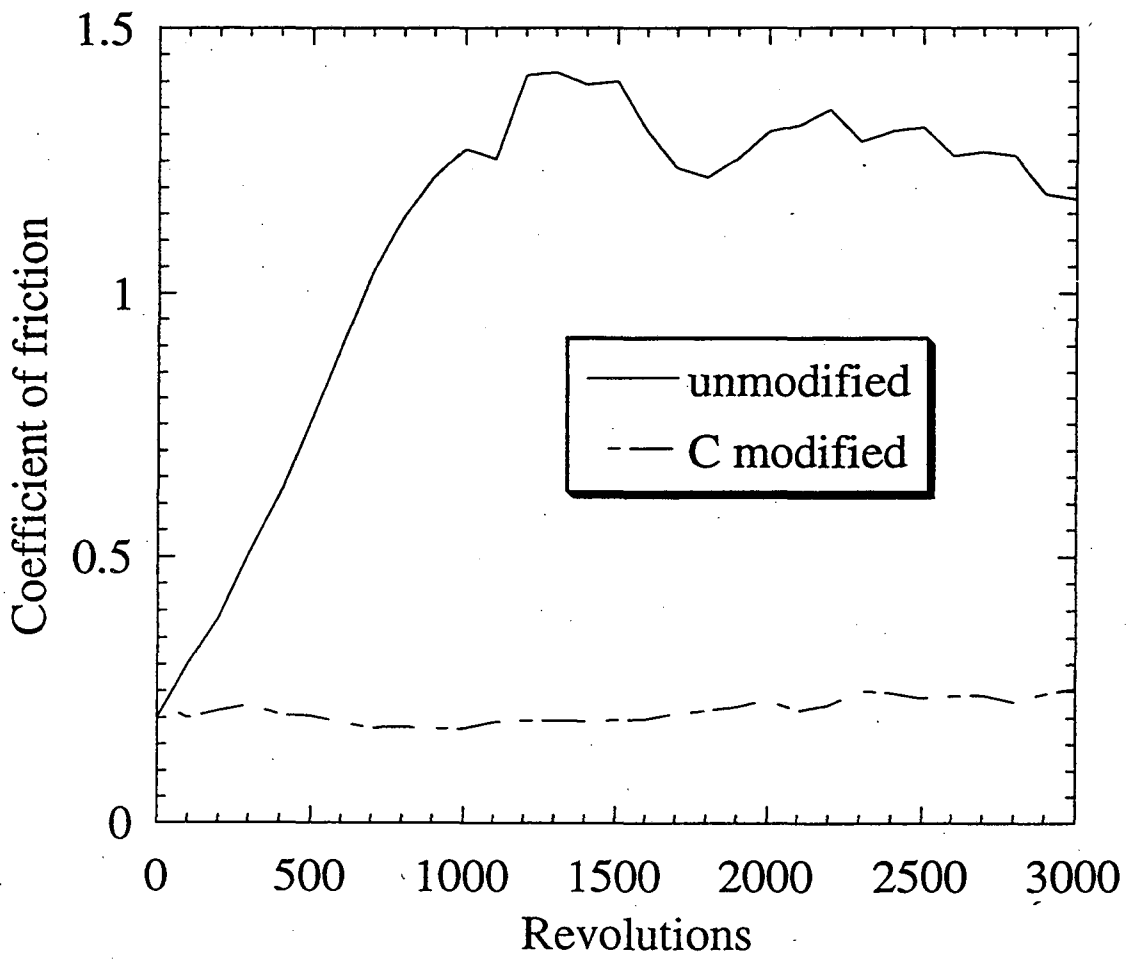
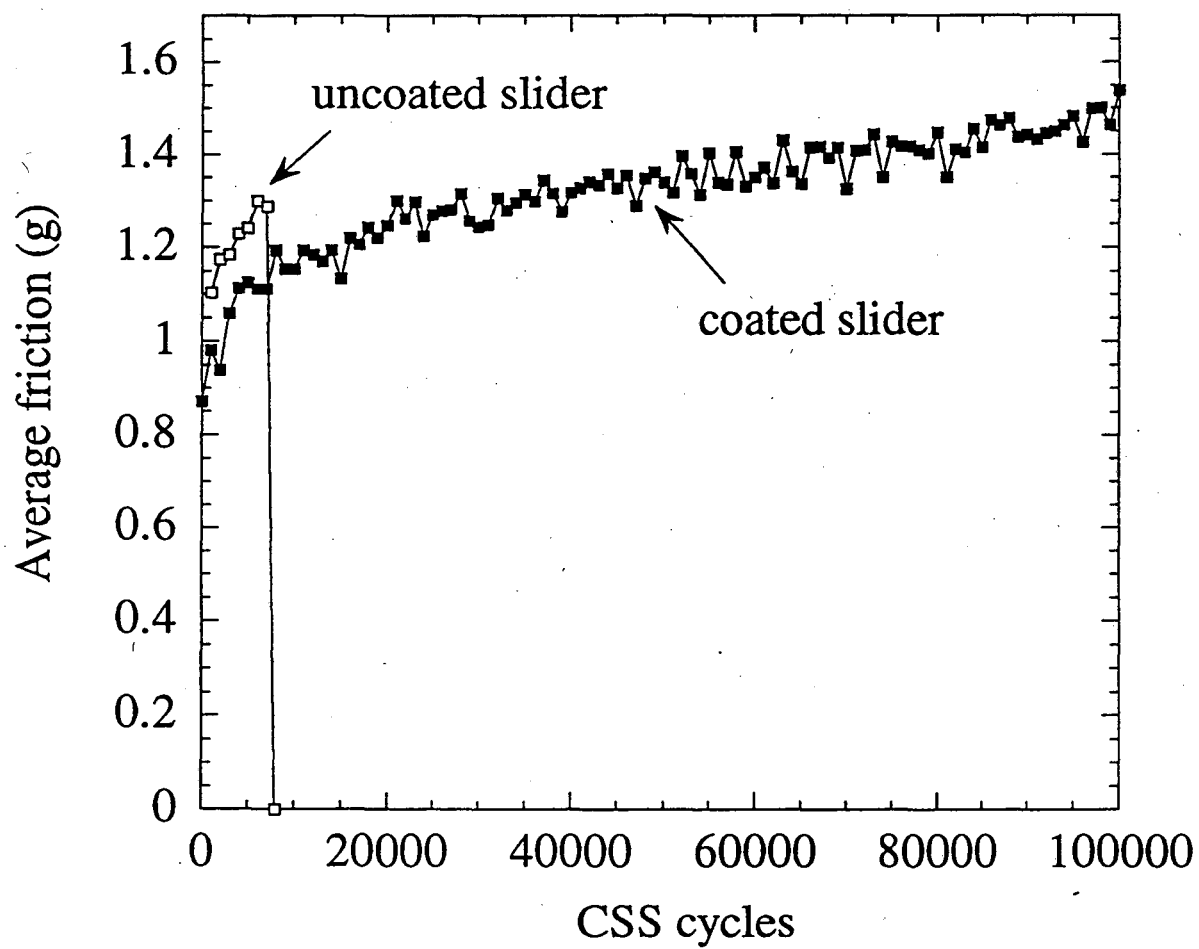


Fig 9.



F_J-10

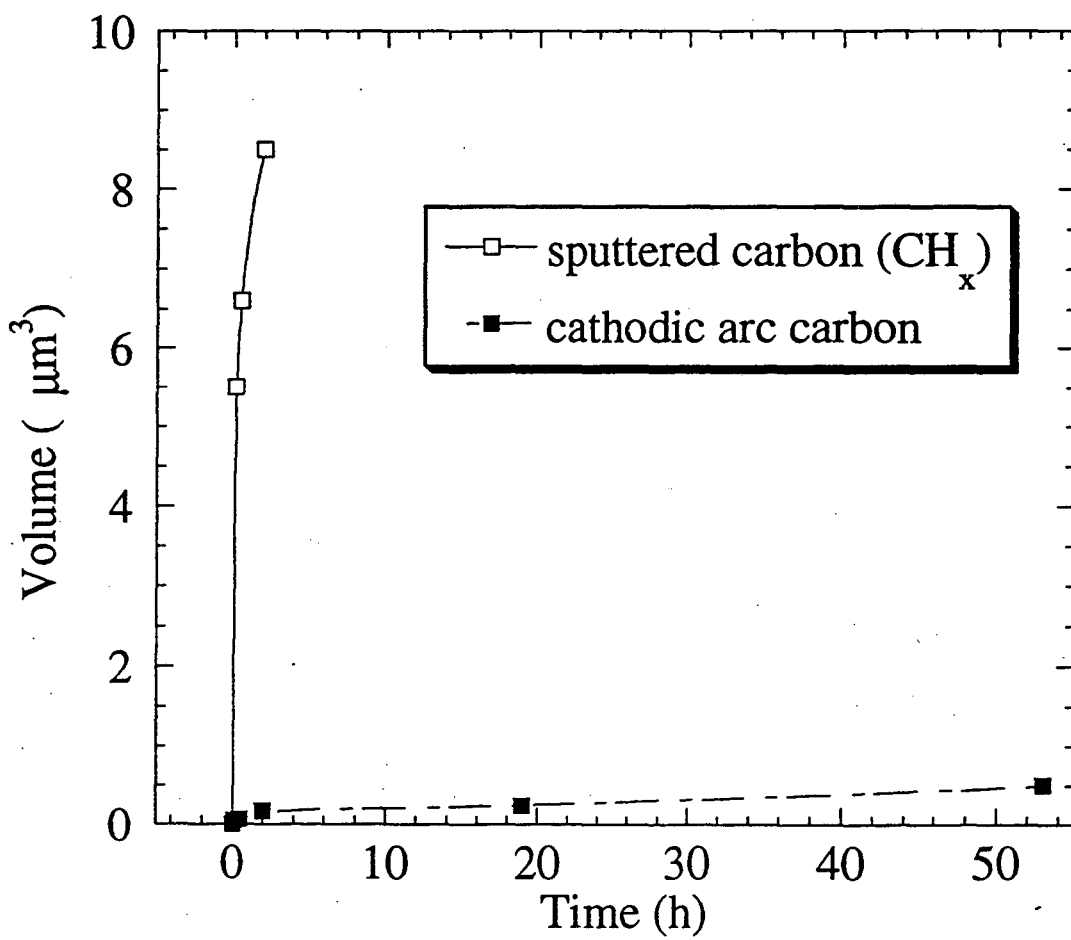


Fig 11

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