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Ion Beam Sputter Deposited Diamondlike Films

Bruce A. Banks and Sharon K. Rutledge
Lewis Research Center
Cleveland, Ohio

NASA

ION BEAM SPUTTER DEPOSITED DIAMONDLIKE FILMS

by Bruce A. Banks and Sharon K. Rutledge

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

ABSTRACT

A single argon ion beam source was used to sputter deposit carbon films on fused silica, copper and tantalum substrates under conditions of sputter deposition alone and sputter deposition combined with simultaneous argon ion bombardment. Simultaneously deposited and ion bombarded carbon films were prepared under conditions of carbon atom removal to arrival ratios of 0, 0.36 and 0.71. Deposition and etch rates were measured for films on fused silica substrates. Resulting characteristics of the deposited films are: electrical resistivity of $>10^{11}$ Ω cm, densities of 2.1 g/cm^3 for sputter deposited films and 2.2 g/cm^3 for simultaneously sputter deposited and Ar ion bombarded films. For ~1700 Å thick films deposited by either process and at 5550 Å wavelength light the reflectance was 0.2, the absorptance was 0.7, the absorption coefficient was 6.7×10^4 cm^{-1}, and the transmittance was 0.1.

INTRODUCTION

Numerous researchers have reported the deposition of carbon films by vacuum processes that have exhibited some properties similar to diamond. Processes which have been reported to produce such diamondlike films include: vacuum evaporation (ref. 1), radio frequency sputtering using inert gas and carbon targets (ref. 2), RF plasma decomposition of hydrocarbon gases (refs. 3 to 6), DC glow discharge of predominantly hydrocarbon gases with a small fraction of argon (ref. 7), coaxial pulsed plasma acceleration using methane gas (ref. 8), vacuum arc using a graphite cathode (ref. 9), ion beam deposition with argon and hydrocarbon scission fragment ions (refs. 10 to 12), and deposition using pure carbon ion beams (ref. 13). Combinations of various techniques have also been utilized such as argon ion bombardment of evaporated carbon films (ref. 14), vacuum carbon arc plasma in conjunction with DC or RF potentials applied to substrates (ref. 9), and dual ion beam deposition and sputtering techniques (refs. 12, 15, and 16).

This paper will present and compare diamondlike films produced by ion beam sputter deposition with those produced by simultaneous ion bombardment during sputter deposition. The purpose of this comparison is to see what effect the energetic (1000 eV) ion bombardment has upon the rather low energy (1 to 20 eV) sputter deposited carbon. It is conceivable that the simultaneous ion bombardment of the carbon films could change the ratio of tetragonally to trigonally bonded carbon atoms through impact kinetics or selective sputter etching processes. Such changes in bond populations would probably manifest themselves in changes in optical, physical and electrical properties of the films.

Diamondlike films have the potential for numerous power electronics applications such as heat conductors for semiconductor devices (refs. 17 and

18) or as doped semiconductors (ref. 19). Successful realization of these applications will be contingent upon many considerations with respect to the cost-effective production and characteristics of these films. Some of these considerations include the ability to cheaply and reliably produce thick (10^1 to 10^2 μm) diamondlike films and a demonstration of acceptable film thermal conductivity, dielectric strength, semiconducting properties, mechanical integrity, intrinsic stress, and compatibility with integration into device fabrication. Film characteristics presented in this paper include electrical resistivity, density, optical reflectance, absorption, and transmittance.

APPARATUS AND PROCEDURE

Film Deposition

An 8 cm beam diameter argon ion source was utilized for all ion beam sputter etching and deposition of carbon reported in this paper. Figures 1(a) and (b) show a schematic drawing and a photograph, of the 8 cm argon ion beam source, respectively. The ion source was operated within a vacuum facility 4.5 m long by 1.5 m in diameter. The vacuum facility with the ion source in its operating position along with its power supplies is shown in Figure 2. The pressure within the vacuum facility was maintained between 1.8×10^{-5} and 4.0×10^{-5} torr during ion source operation. A detailed description of the vacuum facility is given in reference 20.

A top view drawn to scale showing the ion source in conjunction with the sputter target and the deposition substrate sample holder is shown in Figure 3. The ion source was operated to produce a 1000 eV argon ion beam. The ion source operating conditions are given in Table I. The ion beam was neutralized by a hot wire electron emitter to prevent breakdowns from occurring on nonconducting surfaces. The current density distribution at the location of the deposition substrates is also shown on Figure 3.

The technique used for the deposition of diamondlike films consisted of sputter etching carbon from a large (30.5 cm x 30.5 cm) pyrolytic graphite target to allow deposition on copper, tantalum, or fused silica substrates which were rotated at 5 revolutions per second by means of a motor attached to the sample holder. The slight divergence of the ion beam allowed deposition samples to be simultaneously sputter etched by the argon beam. To regulate the average current density that simultaneously performed sputter etching of the deposition substrates, a pair of pyroltic graphite combs (see figs. 4(a) and (b)) were used. Rotating the samples by means of the motor greatly reduces nonuniformity caused by sputter etching of ions passing through the open portions of the combs. The transparency of the combs could be varied from 0 to 50 percent by appropriate adjustment of their overlap. Thus by suitable adjustment of the ion beam transparency of the combs, samples could be deposited which had various relative rates of simultaneous sputter deposition of carbon and argon ion bombardment (and thus an associated sputter etching). The relative rate of ion bombardment could be set from no bombardment (0 percent comb transparency) to a rate of bombardment (at 50 percent transparency of the combs) where typically 0.7 atoms of carbon were removed, on the average, for each sputter deposited carbon atom. The combs and sample rotation motor were mounted on a rotatable and axially movable vacuum feed through shaft. To allow improved attachment of the sputter deposited films, substrates were ion beam sputter cleaned (for 5 to 300 sec depending upon specific test) by rotating the combs 180°, as shown in Figure 4(b), which caused a net sputter etching of the substrates.

Film Characterization

Deposited film thicknesses were measured by means of a surface profiling instrument (Alpha-Step Profiler®, Tencor Instruments). Polyimide (Kapton®) tape, with a silicone adhesive, was used to protect a portion of fused silica slides from deposition or etching to obtain a surface profile trace with the stylus passing from the virgin fused silica surface to the deposited or etched surface.

The density of the deposited films was measured by a sink or float test in various density fluids. Deposited films on copper were found to easily spall from the smooth copper substrates. The spalled films were transferred to vials of known fluid mixtures of 1,1,2,2 tetrabromoethane (density = 2.967 g/cm^3) and 1,1,2 trichloroethane (density = 1.462 g/cm^3). The density of the fluid mixtures were calibrated and checked by means of volume and mass measurements, density calibration spheres, and actual samples of known density pyrolytic graphite. The immersed samples were allowed to remain undisturbed for 24 to 72 hours to determine if the film density was more or less than that of the specific fluid.

Resistivity measurements were made from deposited films of known thicknesses (using the fused silica samples to obtain thickness data) on tantalum. The tantalum was used as the negative contact to the film and a circular droplet (approximately 1.6 mm in diameter) of colloidal graphite conductive paint was applied to the film top surface as the positive contact. Currents were measured using a Hewlett-Packard® Model 4329A High Resistance Meter.

The optical transmittance, reflectance and absorptance of the deposited films on fused silica were documented by means of a Gier-Dunckle® integrating sphere in conjunction with a tungsten strip lamp light source and a monochromator. Figures 5(a) and (b) show apparatus and configurations used to measure transmittance, and reflectance plus transmittance, respectively. Absorptance was then calculated by substracting transmittance plus reflectance from one.

RESULTS AND DISCUSSION

Sputter Arrival and Removal Rates

Because all films were deposited upon ion beam sputter cleaned substrates, it is useful to know the amount of substrate removal caused by ion cleaning. Figure 6 shows a typical surface profile trace taken from a polyimide tape protected virgin fused silica surface to a region where net ion beam sputter etching had occurred. The configuration of the substrate during the ion beam cleaning does allow arrival of sputtered carbon atoms, however, their sputter removal rate is in excess of their arrival rate and net etching occurs. Figure 7 is a plot of surface profile data for various durations of ion beam sputter cleaning of fused silica substrates. The bars on the graph represent the uncertainty in readability of the surface profile traces and not any absolute precision index. As can be seen from Figure 7, the average rate of sputter etching of the fused silica during ion beam cleaning is 1.4 Å/second.

Deposition of carbon films, without ion bombardment, occurs as a very constant rate process. Measurements of the surface profiles of such films show traces which typically have an anomolously thick artifact at the junc-

tion of the film and virgin fused silica surfaces (see Fig. 8). This bump in the film may be caused by the presence of the polyimide type and its edge at this location during deposition. Removal of the tape causes the film to break in this transition region and, in addition, partial film substrate detachment may occur. Figure 9 is a plot of surface profile data for various sputter deposition durations all performed without ion bombardment (combs closed) except for 5 seconds of ion beam cleaning at the beginning of each test. The initial etching due to ion beam cleaning should represent an initial loss of less than 30 Å of fused silica thickness based on Figure 7. As can be measured from Figure 9, the average deposition rate is 0.275 Å/sec.

The arrival of carbon during sputter deposition is also accompanied by other species from the environment. These are predominantly Ar, N_2, O_2, H_2O, and small amounts of diffusion pump oil hydrocarbons. If one simplistically assumes, at a facility pressure of approximately 3×10^{-5} torr, argon gas is the sole contributor to this pressure, then one can obtain a comparison between the carbon deposition flux and the environmental gas flux. If one assumes an ideal gas with a Maxwell speed distribution, the number of gas molecules crossing a unit area in a given direction in one second is J_G where

$$J_G = \frac{n\bar{v}}{4}$$

in which n is the number of molecules per unit volume and v is the average molecular speed (ref. 21). From the ideal gas law

$$PV = NkT$$

P   gas pressure
V   gas volume
N   number of gas molecules
k   Boltzman's constant
T   absolute temperature

Thus

$$n = \frac{N}{V} = \frac{P}{kT}$$

and for an ideal gas (Ref. 22)

$$\bar{v} = \sqrt{\frac{8\,kT}{\pi m}}$$

Combining equations yields

$$J_G = \frac{P}{\sqrt{2\pi\,kTm}}$$

Substituting the below numbers for the following variables

P 3.0×10^{-5} torr = 4.0×10^{-3} Pa
k 1.38×10^{-23} J/K
T   300 K
M 6.63×10^{-26} kg for argon gas molecules

yields

$$J_G = 9.63 \times 10^{19} \text{ molecules/cm}^2\text{sec}$$

This should be compared with the carbon flux arriving at the deposition substrate surface. The number of atoms of carbon being deposited on the substrate per unit area in one second is J_C where

$$J_C = \frac{\rho x}{m}$$

and ρ density of carbon deposited = 2.1 g/cm^3
       (see density results later in paper)
      x   thickness of carbon film for 1 sec. of deposition = 0.275 Å
      m   mass of a carbon molecule = 1.99×10^{-26} kg

Converting to consistent units and substituting results in

$$J_C = 2.9 \times 10^{14} \text{ molecules/cm}^2 \text{ sec}$$

Thus

$$\frac{J_G}{J_C} = 3.3 \times 10^5$$

indicating that the gas arrival rate is five orders of magnitude over the carbon deposition rate. Obviously this high background gas arrival rate has potential for significant impact upon the resulting film characteristics. It should be noted that the environmental gas species are not anywhere near 100 percent argon as simplistically chosen for purposes of flux calculation. The vacuum facility pressure with the ion source and its argon gas feed shut off was typically 90 percent of the pressure experienced during ion source operation. Sputter deposition of tantalum within this vacuum facility resulted in very nonconducting clear deposits (probably tantalum oxide) which supports the premise that most of the environmental gases were normal atmospheric air species.

Deposition of carbon films, with the pyrolytic graphite combs open to allow 25 percent transparency to the ion beam for simultaneous grazing incidence ion bombardment of the depositing films, produced much more scattered results than pure sputter deposition. Figure 10 is a plot of surface profile data for various durations of deposition with bombardment. The plot shows 3-6 profile measurements for each film. The fused silica substrates were ion beam sputter cleaned for 5 seconds immediately prior to deposition. As can be seen from Figure 10 in addition to a much wider scatter in data there may be a segment in time (between 0 and 100 sec) when there is net etching. The initial

sputter removal is quite possible if, during this interval, the fused silica is only slightly covered with sporadic sites of sputter deposited carbon and the ion bombardment of the silica areas causes sputter etching at a rate exceeding the carbon deposition. Once the surface is populated with carbon atoms, their lower sputter yield allows a uniform rate of net deposition.

The net sputter deposition rate, N, of carbon deposited with simultaneous ion beam bombardment through partly open pyrolytic graphite combs is given by

$$N = A - R$$

where

A   carbon arrival rate
R   carbon removal rate

More specifically

$$A = T + C$$

where

T   carbon arrival rate from the pyrolytic graphite target
C   carbon arrival rate from forward sputtering of the pyrolytic graphite comb edges

and

$$R = tr$$

where

t   fractional ion beam transparency of the combs
r   sputter removal rate caused by ion bombardment of carbon films
      if the combs were 100 percent transparent

Values for the above variables could be calculated based on tests with 0, 25 percent and 50 percent transparency combs and are given in Table II. The net deposition rate of carbon upon a carbon film for partially open combs was found to be

$$N = 0.33 - 0.47t \text{ Å/sec}$$

It should be noted that this rate equation is valid only if the combs are partially open (such that C is constant) and that there is an initial carbon film already on the fused silica substrate. The higher sputter yield of SiO_2 in fused silica prevents an immediate carbon film buildup probably leading to much of the scatter in the Figure 10 data. As can be seen the carbon surfaces were being quite heavily bombarded at 25 percent and 50 percent transparency of the combs, where about 1/3 and 2/3, respectively, of the carbon atoms are being sputter removed as they deposit.

When the pyrolytic graphite combs were opened to allow 50 percent transparency to the incident bombarding ion beam, carbon films would not deposit on virgin fused silica substrates and a net sputter etch was always observed. However, if an initial thin film of carbon was deposited on the fused silica substrate (with the combs closed) then when the combs were opened to 50 percent transparency, a net increase in carbon deposition would occur. This was most clearly demonstrated in a test in which a portion of a silica

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substrate was sputter deposited with 850 Å of carbon as shown in Figure 11. After opening the combs to 50 percent transparency and exposing the sample to simultaneous sputter deposition and ion bombardment, a net carbon additional deposit occurred on the carbon coated surface while, at the same time, the fused silica witnessed a net etch.

The respective deposition and etch rates observed were such that the fused silica etched at four times the rate of carbon deposition. Potential applications of this phenomena may exist in ion beam sputter etching of microelectronics, where long sputter mask life and deep etching is required. Since a carbon mask actually becomes thicker, sputter durability should not be a problem. Organic photo or electron beam lithography mask compounds may potentially be used as a start mask if ion bombardment and scission of the polymers causes an adequate free carbon surface population to develop.

The carbon films deposited (from a starting carbon film) under the ion bombardment of 50 percent transparent combs is a very ion worked surface; approximately 70 percent of the deposited carbon atoms are sputter removed by the ions passing through the openings in the combs. The resulting low net deposition rates (0.095 Å/sec) precluded the practical deposition of films thick enough for further characterization.

Film Characteristics

Electrical resistivity measurements of films both sputter deposited only and sputter deposited with simultaneous ion bombardment (at 25 percent comb transparency) resulted in resistivities of $>10^{11}$ Ω cm. Surface conductivity of the measurement fixturing limited detection of higher resistivities. Attempts to obtain lower resistance test areas by using larger than 2 mm diameter contacts resulted in a high fraction of direct shorts, probably due to inclusion of film imperfections from particulate debris on the film surface. Figure 12 is a scanning electron photograph showing a 1670 Å carbon film with a typical imperfection.

The sink or float density measurements of numerous flakes indicated that the carbon films deposited by sputter deposition alone had a density of 2.1 g/cm^3. The films deposited by simultaneous sputter deposition and ion bombardment with the combs set at 25 percent transparency (thus about 1 out of every 3 arriving carbon atoms was sputter removed) resulted in a density of 2.2 g/cm^3. Because pure single crystal graphite has a density of 2.26 g/cm the observed densities are extremely high, unless there is a reasonable fraction of tetragonal bonded carbon. Because the films are typically amorphous (as determined by electron beam diffraction) and there is a significant opportunity to have voids from entrapped environmental gases during deposition, one would expect to observe densities much below 2.26 g/cm^3. However, a significant population of more dense tetragonal bonding may allow the observed densities to be realized. Ion bombardment of the depositing surface may reduce the amount of trapped gases or cause an increase in the amount of tetragonally bonded carbon by either ion bombardment kinetics or a slighty selective removal of trigonally bonded carbon.

Highly dense contaminant materials could also give rise to anomalously high densities. However energy dispersive X-ray analysis of the films indicated the only contaminant being traces of silicon. The densities of amorphous silicon, amorphous silica, and silicon carbide are typically between 2.2 and 3.2 g/cm^2. Thus, in trace amounts, such low density contaminants would not be expected to cause significant contributions to density. The observed

high densities of these hard and scratch resistant carbon films is probably one of the most straight forward indicators of the degree to which they are diamondlike.

The optical properties of reflectance, absorptance, and transmittance for both sputter deposited and simultaneously sputter deposited and ion bombarded (at 25 percent comb transparency) carbon films on fused silica substrates is shown in Figures 13(a), (b), and (c), respectively. For comparison purposes of optical properties, an uncoated fused silica substrate (0.762 cm thick) is also shown on the graphs. An effort was made to optically characterize carbon films of approximately the same thickness. As can be seen, the films are very similar in optical character. One must remember however that based on the density measurements the simultaneously sputter deposited and ion bombarded film may have about 5 percent more carbon atoms in it. Thus on a per carbon atom basis, the simultaneously deposited and ion bombarded films indicate a slight merit toward diamondlike optical properties. The optical absorption coefficient for films deposited by both techniques is approximately 6.7×10^4 cm^{-1} at the peak of the spectral sensitivity of the eye (5550 Å ref. 23).

SUMMARY

A single argon ion beam source was used to sputter deposit carbon films on fused silica, copper, and tantalum substrates under conditions of sputter deposition alone and simultaneous sputter deposition with 1000 eV argon ion bombardment. Deposited carbon films were prepared under conditions of carbon removal to arrival ratios of 0, 0.36 and 0.71. At carbon removal to arrival ratios of 0 and 0.36, films developed on fused silica substrates. However at the 0.36 ratio wide scatter existed in the rate of deposition. At the 0.71 ratio, carbon films would only build up on substrates having a previously deposited carbon film and net etching would result on the virgin fused silica substrates.

Electrical resistivities of all films were at least 10^{11} Ω cm. The density of the simultaneously ion bombarded films was 2.2 g/cm^3 in comparison to 2.1 for the sputter deposited only films. The optical reflectance, absorptance, and transmittance of both methods of deposition were very similar, however, on a per carbon atom basis the simultaneous ion bombardment results in more diamondlike optical characteristics. For approximately 1700 Å thick films deposited by either process and at 5550 Å wavelength light, the reflectance was 0.2, the absorptance was 0.7, the absorption coefficient was 6.7×10^4 cm^{-1}, and the transmittance was 0.1.

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TABLE I - ARGON ION SOURCE OPERATING CONDITIONS

Ion beam exit diameter, cm 8

Net ion energy, eV 1000

Accelerator grid potential, V . . . . -500

Ion beam current, mA 55

Cathode emission current, A . . . . 0.5 to 0.8

Discharge voltage, V . . . . 44 to 50

Accelerator drain current, mA . . . . 0.8 to 1.0

Argon gas flow rate, standard cm^3/min . . . . . . 3.5 to 5

TABLE II - ARRIVAL, REMOVAL, AND NET RATES OF CARBON DEPOSITION ON CARBON COATED SUBSTRATES FOR SIMULTANEOUS SPUTTER DEPOSITION AND ION BOMBARDMENT

[T = 0.275 A/sec = carbon arrival rate from pyrolytic target

C = 0.0556 A/sec = carbon arrival rate from forward sputtering of the pyrolytic graphite comb edges

t = Fractional ion beam transparency of the combs]

Comb transparency, t	Carbon arrival rate, A = T + C, A/sec	Carbon removal rate, R = tr, A/sec	Ratio of removal to arrival rate, R/A	Net deposition rate, N = A ± R, A/sec
0	0.275	0	0	0.275
.25	.331	.118	.36	.213
.50	.331	.235	.71	.0954

C-82-1676

Figure 2. - Vacuum facility with ion source in position along with its power supplies.

ACCELERATOR
GRID
NEUTRALIZER
SUPPLY
HOT WIRE
NEUTRALIZER
LOOP
SCREEN
SUPPLY
ACCELERATOR
SUPPLY
BEAM
CURRENT
METER

J_B

MAGNETS
POLE PIECES
SCREEN GRID
CATHODE
FILAMENT
ANODE
ENGINE
BODY
DISCHARGE
SUPPLY

PLENUM
ARGON
INLET
APERTURE

FILAMENT
HEATER
SUPPLY
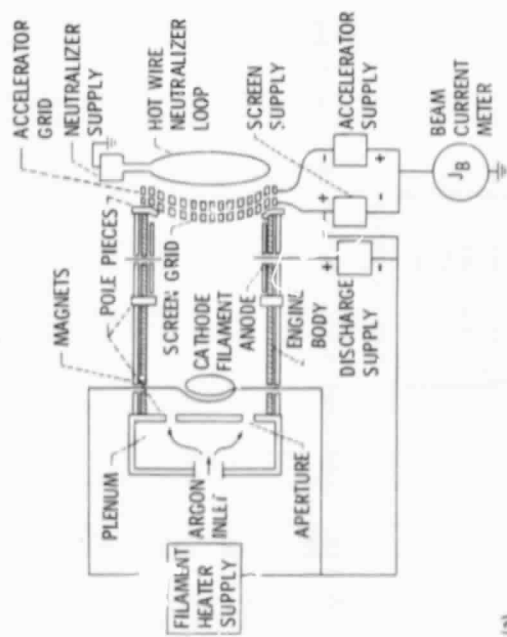
(a)
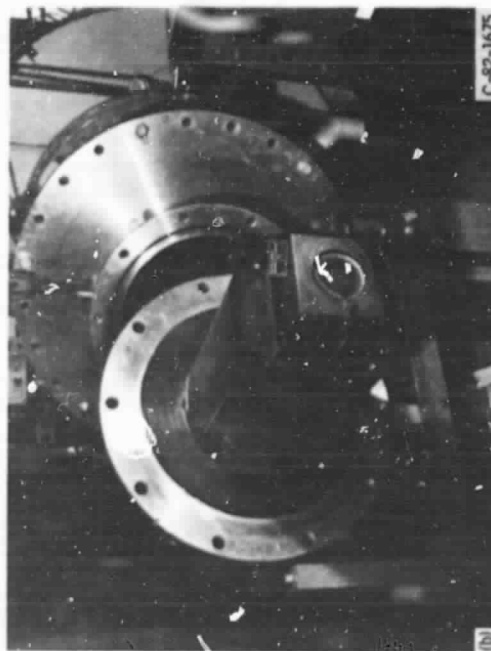
(a) Schematic of ion source.

(b) Photograph of 8 cm argon ion beam source.
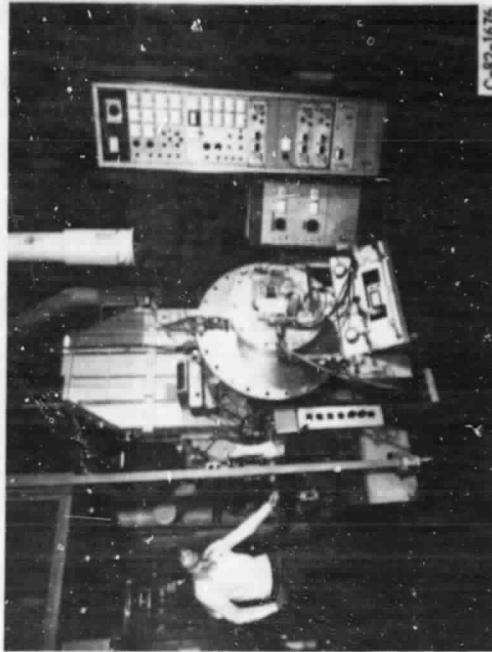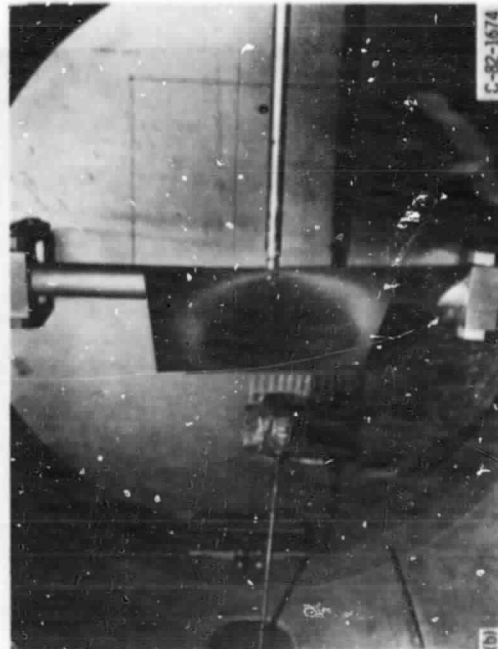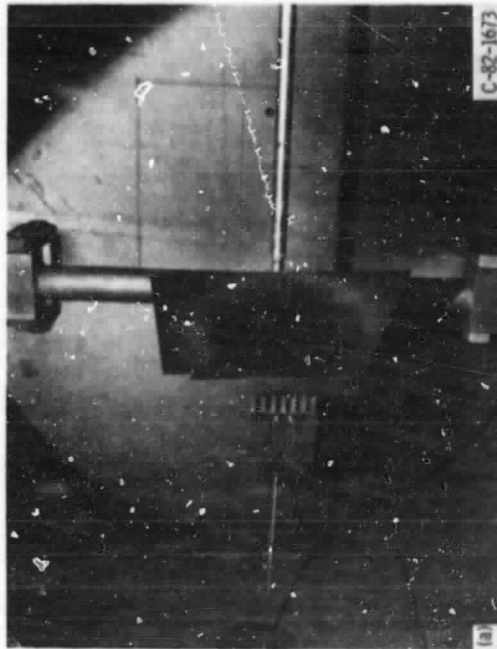
Figure 1. - 8 cm argon ion beam source.

C-82-1675

(b)

(a) Deposition configuration.

(b) Ion beam sputter cleaning configuration.

Figure 4. - Pyrolytic graphite target, combs and rotating sample holder.
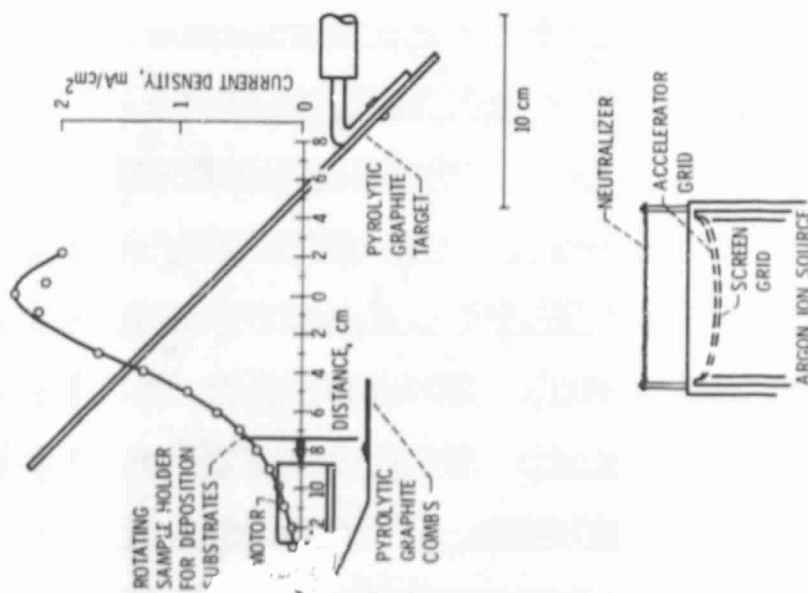
Figure 3. - Top view layout drawing of experiment with current density superimposed.

SPUTTER ETCHED SURFACE

VIRGIN SURFACE (PROTECTED BY POLYIMIDE TAPE)

RELATIVE SURFACE HEIGHT, IN HUNDREDS OF ANGSTROMS

TRANSVERSE SURFACE DISTANCE, mm
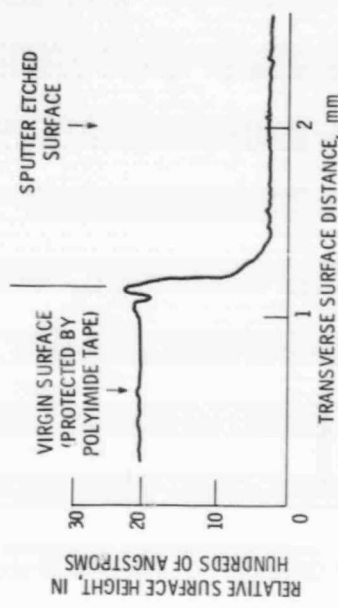
Figure 6. - Typical surface profile trace showing 1750 Å removal of fused silica by ion beam sputtering.

THICKNESS, Å

TIME, sec
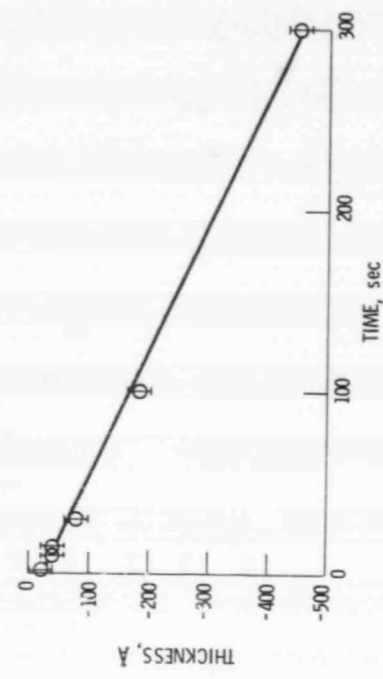
Figure 7. - Ion beam cleaning of fused silica.

MgO COATED SPHERE

DETECTOR (RADIOMETER)

SAMPLE IN BEAM

TRANSFER OPTICS

MIRROR

SAMPLE OUT OF BEAM, REFERENCE POSITION

ROTATING FLAT MIRROR (THE REST ARE SPHERICAL)

EXIT SLIT

ENTRANCE SLIT

MONOCHROMATOR

TUNGSTEN STRIP FILAMENT LAMP

SOURCE HOUSING

SOURCE FOCUSING MIRROR

(a)
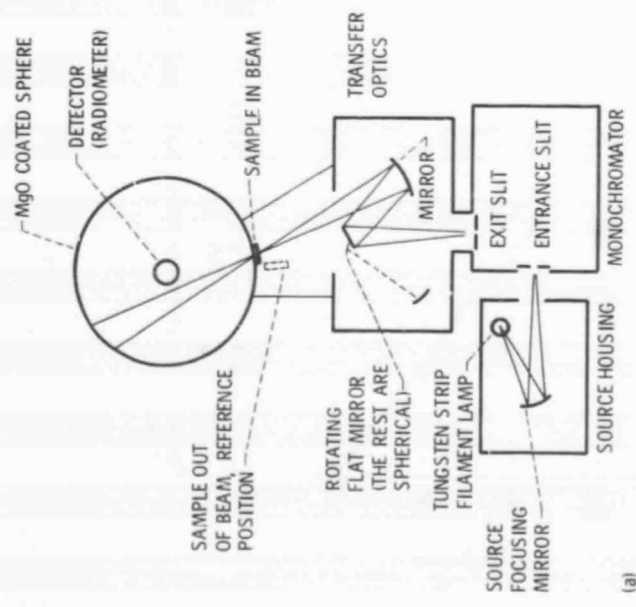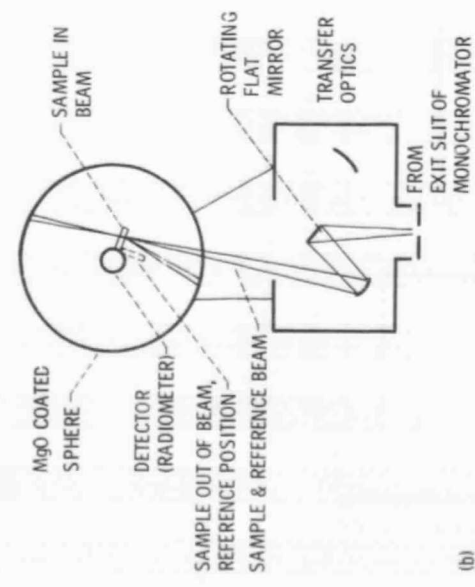
(a) Arrangement for transmittance measurements.

MgO COATED SPHERE

SAMPLE IN BEAM

ROTATING FLAT MIRROR

TRANSFER OPTICS

DETECTOR (RADIOMETER)

SAMPLE OUT OF BEAM, REFERENCE POSITION

SAMPLE & REFERENCE BEAM

FROM EXIT SLIT OF MONOCHROMATOR

(b)

(b) Arrangement for transmittance plus reflectance measurements.

Figure 5. - Apparatus and configuration used to document optical properties of diamond like films on fused silica.
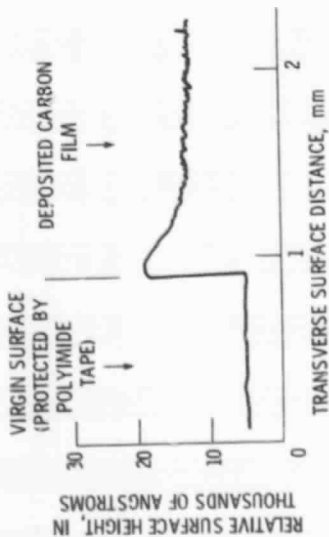
Figure 10. – Simultaneously sputter deposited and ion bombarded carbon film thickness versus time (25% comb transparency).

Figure 8. – Typical surface profile trace showing 750 Å of sputter deposited carbon film on fused silica.
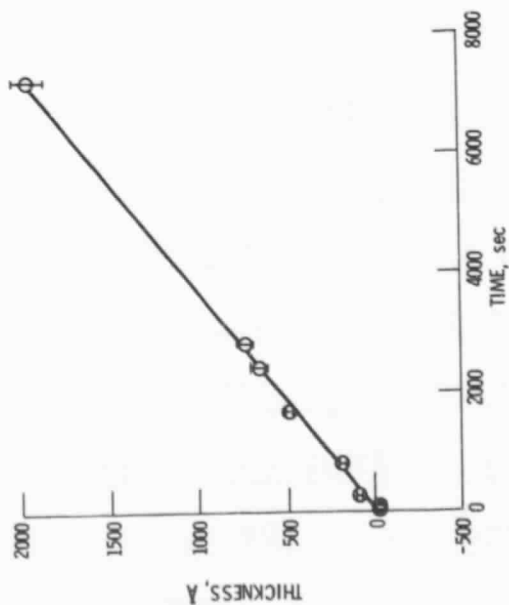
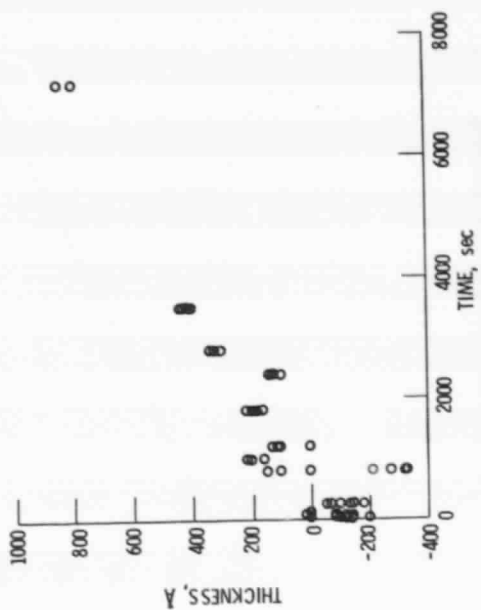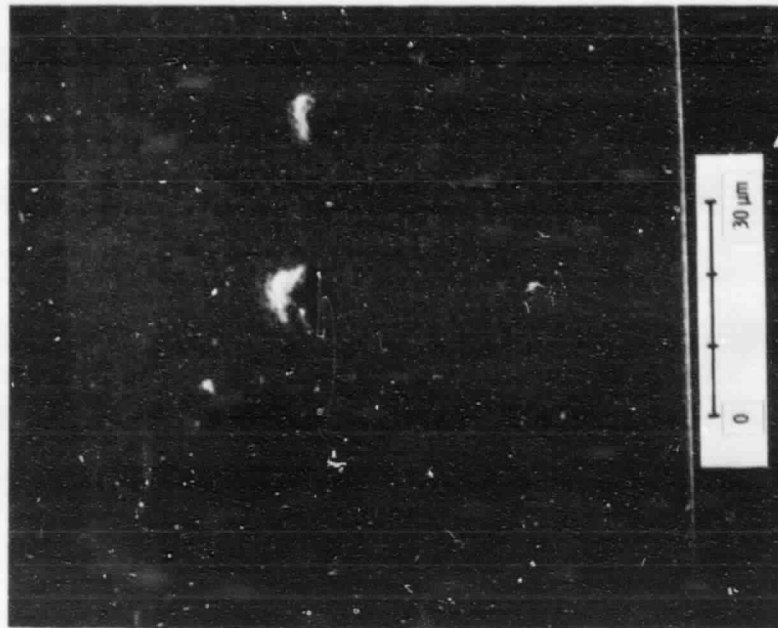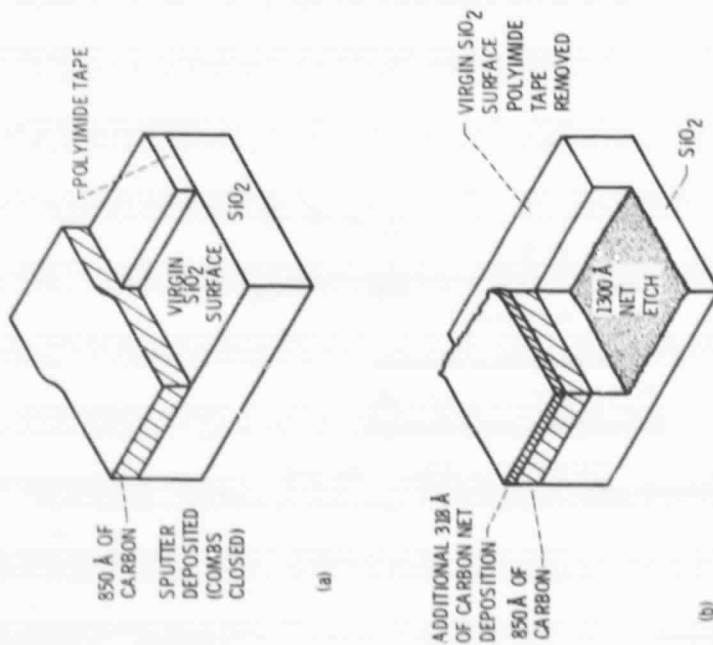Figure 9. – Sputter deposited carbon film thickness versus time,

Figure 12. - Scanning electron photograph of typical film imperfection in a 1670 Å thick carbon film on fused silica.

(a) Before simultaneous carbon deposition and ion bombardment.

(b) After 3333 seconds of simultaneous deposition and ion bombardment with polyimide tape removed to reveal virgin surface reference.

Figure 11. - Fused silica sample before and after sputter deposition with ion bombardment having combs set at 50% transparency.
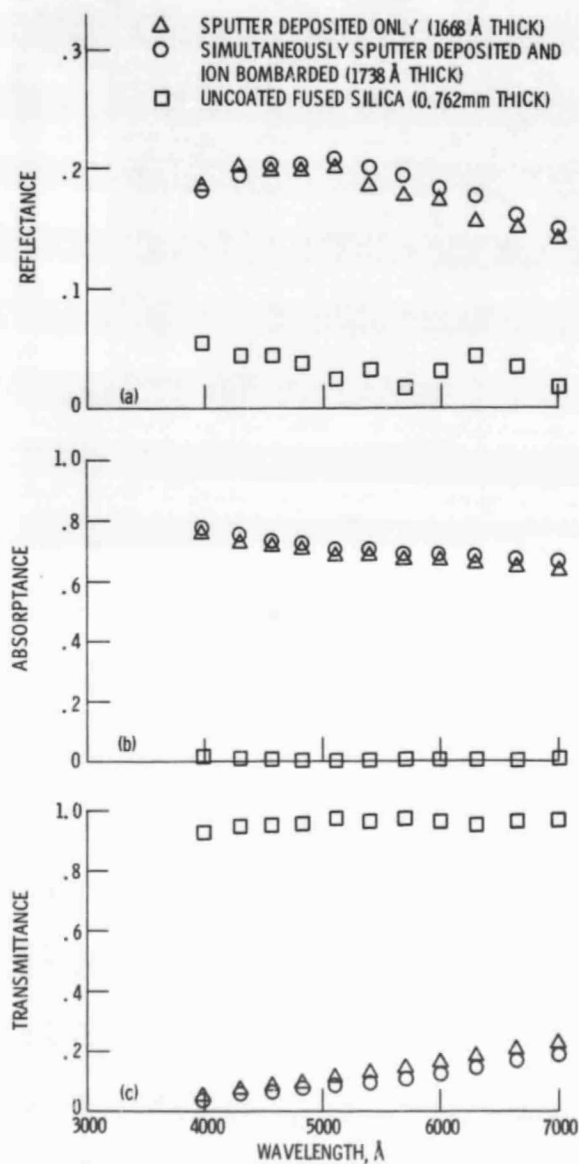
(a) Reflectance versus wavelength.

(b) Absorptance versus wavelength.

(c) Transmittance versus wavelength.

Figure 13. - Optical properties of carbon films on fused
silica substrates.