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Properties of filtered-ion-beam-deposited diamondlike carbon as a function of ion energy

P. J. Fallon*
Microstructural Physics Group, Cavendish Laboratory, Madingley Road, Cambridge CB3 0HE, United Kingdom

V. S. Veerasamy
Engineering Department, University of Cambridge, Trumpington Street, Cambridge CB2 1PZ, United Kingdom

C. A. Davis
School of Physics, University of Sydney, New South Wales 2006, Australia

J. Robertson
National Power Laboratories, Leatherhead, Surrey KT22 7SE, United Kingdom

G. A. J. Amaratunga and W. I. Milne
Engineering Department, University of Cambridge, Trumpington Street, Cambridge CB2 1PZ, United Kingdom

J. Koskinen
VTT Technical Research Centre of Finland, Metallurgy Laboratories, Espoo 02151, Finland

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A highly tetrahedrally bonded form on nonhydrogenated amorphous carbon (*a*-C) is produced by deposition from filtered medium-energy ion beams. A range of such films was grown and the sp^3-bonded fractions, plasmon energies, compressive stresses, and resistivities were measured as a function of ion energy. These properties are found to be strongly correlated and each to pass through a maximum at an ion energy of about 140 eV. The optimum ion energy is observed to depend on the type of carbon ions deposited and, possibly, on the deposition flux rate. The data are found to support deposition models in which the sp^3 bonding arises from the subplantation of incident ions, giving rise to a quenched increase in density and strain.

INTRODUCTION

Amorphous carbon (*a*-C) deposited from medium-energy ion beams contains significant fractions of tetrahedral bonding. The sp^3 bonding gives these *a*-C films valuable properties such as high hardness, and they are often referred to as "diamondlike carbon" (DLC).[1,2] The properties depend significantly on the fraction of sp^3 bonding, and there is therefore a need to understand the deposition processes which promote sp^3 bonding. There is particular interest in a form of *a*-C deposited from ion beams from which neutral and impurity species have been removed, as this material has the highest sp^3 fraction of any *a*-C and yet contains little hydrogen.[3–18] Their properties such as hardness, density, band gap, and resistivity have been studied as a function of ion energy by various groups and the degree of diamondlike character is found to pass through a maximum for deposition ion energies in the range 30–150 eV. However, there is presently some uncertainty over the optimum ion energy, being 30 eV according to McKenzie *et al.*[13,14] and about 150 eV according to Koskinen[8] and Ishikawa *et al.*[6]

The structure and bonding of *a*-C produced at the optimum ion energy have been studied by electron and neutron diffractions, electron microscopy, and electron-energy-loss spectroscopy (EELS) and are found to be reasonably well described by a tetrahedrally coordinated random network with a small fraction of sp^2 bonding.[14,15,17,18] However, we are not aware of any studies of the bonding as a function of ion energy. In this paper we report on the variation of sp^3 bonding with ion energy, together with the density, compressive stress, and resistivity. The measurements were performed on a range of films grown by the cathodic vacuum arc deposition process with a controllable bias voltage.

The results are compared with a recent theory for the deposition of diamondlike carbon by ion beams. Davis[19] and Robertson[20,21] have independently proposed that significant sp^3 bonding arises when medium-energy ions penetrate the surface atomic layer and produce a quenched-in increase in the density and compressive stress. The increased density and stress transform the local bonding from the stable sp^2 configuration toward the metastable sp^3 configuration. Additional ion energy is largely evolved as heat, which allows the bonding to relax back toward the more stable, lower-density, sp^2-bonded state.

EXPERIMENTAL DETAILS

The *a*-C films were grown from a filtered beam of C^+ ions produced by a cathodic carbon arc.[12,14,22] The cathodic arc produces a relatively high flux of ions from the graphite source disk, allowing a reasonable deposition rate—it is thus a viable technique for the deposition of DLC films. The resulting plasma is filtered by a curved

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magnetic solenoid to remove neutral species and macroscopic particles, leaving a beam of carbon ions. A relatively high magnetic field (approximately 60 mT with a 7-A coil current) was used for the magnetic filter causing deposition by mainly C^+ ions. Mass spectrometry in the deposition chamber found a C^+ to C^{2+} ion ratio of about 99 to 1, and spectroscopic UV emission line measurements in the filter region indicate a C^{2+}:C^+:C ratio of approximately 5:25:1. The base pressure of the present deposition system is 10^{-7} torr, but rises to 10^{-5} torr during deposition. The films were deposited on *n*-type silicon substrates held against a copper block for cooling. The substrate temperature was observed during deposition to rise to a maximum 75 °C and not to increase above this value. The incident ion energy is varied by applying a negative-bias voltage to the substrate, and since the ions are estimated to leave the cathode with an energy of 20 eV,[10] the ion energy is approximately 20 V higher than the bias voltage. The system allows the consecutive deposition of films at five different bias voltages in a single run. The measurements presented here were taken from films grown in two separate runs.

The compressive stress of each film was determined from the curvature of its substrate, measured both before and after deposition using a Dektak 3030 stylus profilometer. Stoney's equation was used to calculate the stress,

$$\sigma=\frac{Y}{6(1-\nu)}\frac{t_s^2}{t}\left[\frac{1}{R}-\frac{1}{R_0}\right], \tag{1}$$

where Y is Young's modulus, ν Poisson's ratio, t_s and t are the substrate and film thickness, respectively, the film thickness being determined by ellipsometery, and R_0 and R are substrate radii before and after deposition, respectively.[23] The uncertainty in the stress arises mainly from the nonuniformity of the substrate curvature.

The resistivity was determined by depositing 1-mm-diam gold contacts onto the *a*-C film. The resulting *n*-type Si/*a*-C/Au sandwich structure shows rectifying I/V characteristics due to the presence of the Si/*a*-C heterojunction.[22] The resistivity of the *a*-C is deduced from the slope of the forward current, which was resistance limited above about 8 V for films ranging in thickness from 1000 to 3000 Å. Errors in the resistivity values arise mainly from variations in the quality of the gold contacts on the *a*-C.

The fraction of sp^2 and sp^3 bonding was determined from the 285–310-eV region of carbon *K*-edge EELS spectra.[11,18,24] The EELS analysis was performed on a Vacuum Generators HB501 scanning transmission electron microscope with a dedicated parallel EELS spectrometer.[18,25] The films were prepared for microscopy by removing the silicon substrate with a HF and HNO_3 acid solution. The remaining film segments were collected and placed on electron-microscopy support grids. Each set of films was measured during a single session using identical microscope and EELS settings. EELS spectra were taken at two different parts of each film to check for variations in plasmon energy and sp^3 fraction. The analysis indicated film thickness to be in the range 15–25 nm.

Each carbon *K*-edge EELS spectrum consists of a peak at 285.5 eV due to excitations from the 1*s* level to empty π^* states of sp^2 sites, followed by a step at around 289 eV due to transitions from the 1*s* level to empty σ^* states of both sp^2 and sp^3 sites. The fraction of sp^2 sites in any sample can be extracted by normalizing the area of its 285.5-eV peak with the area of a large section of the spectrum and comparing this ratio to the value for graphitized carbon which contains only sp^2 sites. This method is independent of the hydrogen concentration of the material.[18,24]

The plasmon energy of each film was measured from the low-energy EELS spectrum in order to obtain an indirect measurement of the material's density. The plasmon energy E_p is related to the local valence-electron density by

$$E_p=\hbar\{ne^2/(\varepsilon_0 m)\}^{1/2}, \tag{2}$$

where n is the number of valence electrons per unit volume, m is the effective electron mass, and the symbols $\hbar$, e, and ε_0 have their usual meanings.

The densities of the *a*-C films were also measured by Rutherford backscattering (RBS) and Dektak 2A surface profilometer thickness measurements. The RBS parameters were a 2.0-MeV He^+ beam of 15 nA, a spot size of 0.5×0.5 mm^2, and a scattering angle of 170°. The area density of carbon, in atoms per cm^2, on the silicon surface was obtained from both the shift of the silicon edge and the width of the carbon signal in the RBS spectrum. The spectra were simulated using the RUMP program[26] and the stopping cross sections were obtained from Ref. 27. Using conventional liftoff lithography, the carbon films were patterned to an array of 1-mm-wide stripes each 1 mm apart. The thickness measurements were then performed by Dektak profiles of the *a*-C film stripes on the silicon substrate. The thickness measurements were performed around the RBS beam spot to prevent errors that might be caused by density changes induced by the He^+ ion beam. The film's density was obtained by dividing the area density by the thickness. The dominant sources of error are the film thickness variations and the film's stress-induced surface curvature, which caused variance in the profilometer results. The possibility of sputtering the silicon substrate prior to deposition is the principle cause of any overestimation of the density.

RESULTS

The variation of the *a*-C EELS spectra with ion energy is shown in Fig. 1. The area of the 285.5-eV peak is seen to pass through a minimum at a bias of −120 eV. Here the sp^3 fraction reaches a maximum of about 80%. This is consistent with earlier EELS measurements by Berger, McKenzie, and Martin[11] and neutron-diffraction studies by McKenzie *et al.*[13] and Gaskell *et al.*[15]

The results in Fig. 2 show the variation in sp^3 content, plasmon energy, compressive stress, and resistance with bias voltage. These properties are seen to be well correlated with each other and to pass through a broad peak at

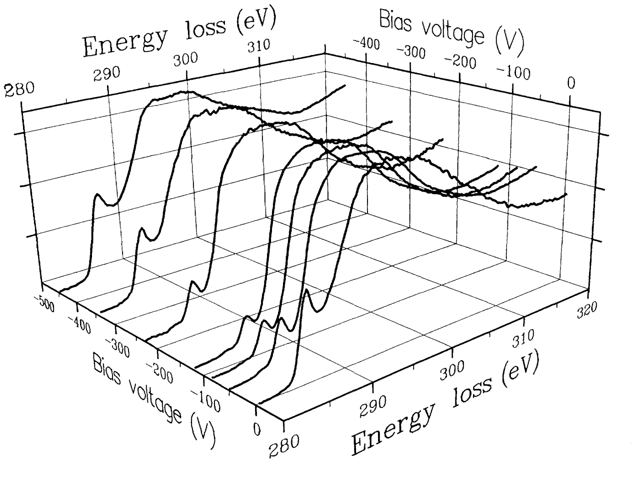

FIG. 1. EELS carbon K edge for a-C films grown at various bias voltages. The 1s to π^* peak centered at 285.5 eV indicates the amount of sp^2 bonding and is observed to be small for bias voltages in the region of -120 V.

a bias of about -120 V. The bias dependence is seen to be similar to that found by other workers but with less spread in the data. The peak energy is closer to that found by Ishikawa *et al.*[6] and Koskinen[8] than to that found by McKenzie *et al.*[13] and Veerasamy *et al.*[22]

The variation in density, as deduced from the valence plasmon energy and RBS measurements, as a function of the sp^3 fraction, is shown in Fig. 3. The figure shows a general linear relationship between the sp^3 fraction and the density. This is as expected if the density depends primarily on short-range order. A similar linear relationship was observed by Cuomo *et al.*[28] for laser-ablated a-C and by Fallon and Brown[18,24] for various different forms of nonhydrogenated a-C. The density determined by RBS in Fig. 3 is seen to be always slightly higher than that determined by the plasmon energy, and it is significantly overestimated at the high sp^3 fractions. The sputtering of the silicon substrate prior to deposition is a possible cause of any overestimation of RBS density.

Figure 4 displays the correlation of the sp^3 fraction to the compressive stress. The data clearly indicate that the sp^3 fraction increases with compressive stresses, as predicted by McKenzie *et al.*[13] However, the data also show more scatter than that in Fig. 3, and so it is unclear whether there exists a linear relationship between the sp^3 fraction and stress or whether the sp^3 fraction only becomes significant above a threshold stress of the order 5 GPa. The open circle points, taken from the work of McKenzie *et al.*,[13] tend to favor a linear relationship. On the other hand, a transition has been observed for sp^2-bonded hexagonal BN at low stresses and sp^3-bonded cubic BN for compressive stresses greater than 4 GPa.[29] The BN does, however, crystalize subsequent to deposition, whereas the a-C studied here remains amorphous.

It should be noted in Figs. 3 and 4 that the points for low- (-20 V) and high- (-450 V) bias voltages lie on the same curve in both cases, indicating that the density and compressive stress are each single-valued functions of the

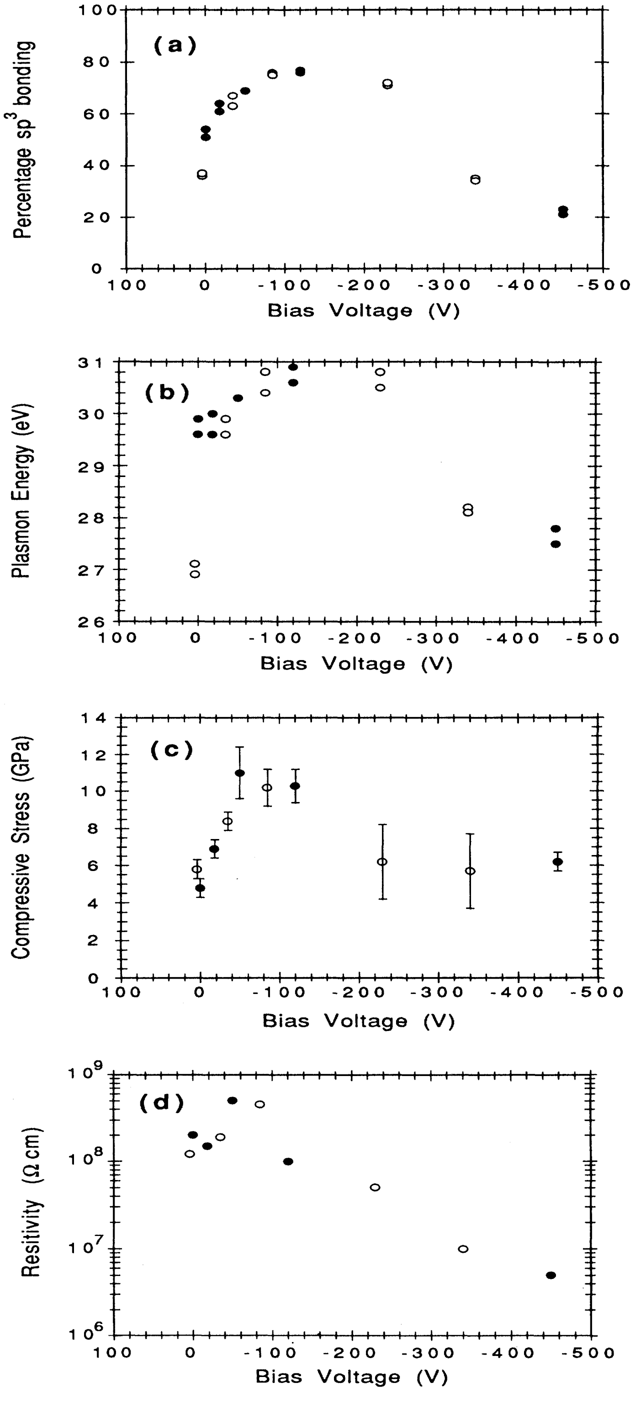

FIG. 2. Graphs of (a) sp^3 fraction, (b) plasmon energy, (c) compressive stress, and (d) the resistivity as a function of bias voltage. The solid circles represent a series of films produced in one deposition run and examined in a single EELS session. The open circles represent a second series of films again produced in a single run and examined in a single EELS session. For each film examined by EELS, two separate pieces of the film were measured and the variation of the results gives an indication of the error for graphs (a) and (b). The ion energy of the deposited carbon ions is the absolute value of the bias voltage plus 20 eV (see text for further details).

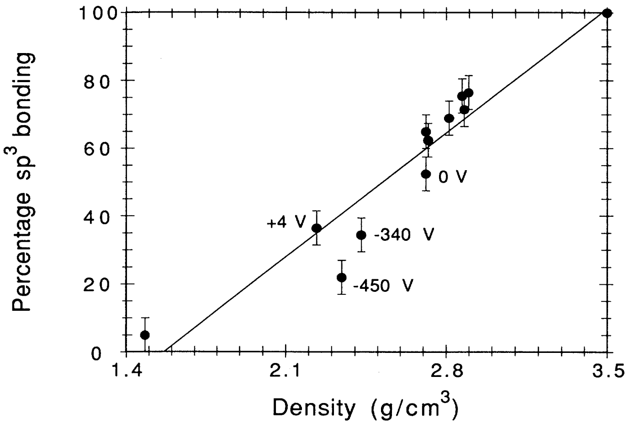

FIG. 3. Graph of the atomic density calculated from both the valence plasmon energy and the RBS measurements as a function of the percentage of tetrahedral bonding. The solid line represents a linear fit to the data.

sp^3 fraction, rather than each having an additional explicit dependence on the ion energy. This suggests that the disorder of *a*-C is qualitatively similar at the two ion energies.

STRESS AND DENSITY VARIATION WITH ION ENERGY

The deposition mechanism of highly tetrahedrally bonded *a*-C has been somewhat contentious. Early suggestions that sp^3 bonding arose from either preferential sputtering or preferential displacement of sp^2 sites have been found to be wrong.[9,21] Davis[19] and Robertson[21] have recently developed a model of the deposition process based on the subplantation ideas of Lifshitz *et al.*[9] and Windischmann.[30] They proposed that the incident ion flux becomes implanted at subsurface positions when the ion energy exceeds a threshold value needed to penetrate the surface layer. This gives rise to a local quenched-in increase in the density of strain. The penetration probability increases rapidly with ion energy above this threshold, but any excess ion energy is evolved as heat. This local heating then allows a relaxation of the density increment according to the thermal spike model of Seitz and Koehler.[31] The thermal spike model suggests that strong scattering events produce a large uniform local temperature rise which then decays by thermal conductivity. Although the subsequent molecular-dynamics simulations showed that the thermal spike model is an oversimplification, it is used here to allow a tractable, analytic description of the overall deposition process to be given. Thus Davis and Robertson deduce broadly similar equations for the density increment $(\Delta\rho/\rho)$ or strain (ε) as

$$\varepsilon \sim \frac{\Delta\rho}{\rho} \sim \frac{f}{b + 0.016p(E/E_0)^{5/3}} , \tag{3}$$

where E is the ion energy, E_0 is the activation energy of the relaxation process, b is the ratio of the depositing flux to the bombarding flux (taken as 1 by Robertson[21]), and p is a material diffusion parameter of order unity from the thermal spike model.[31] f was taken as the forward-sputtering fraction,

$$f = c\sqrt{E} , \tag{4}$$

by Davis[19] and as the penetration fraction by Robertson.[21]

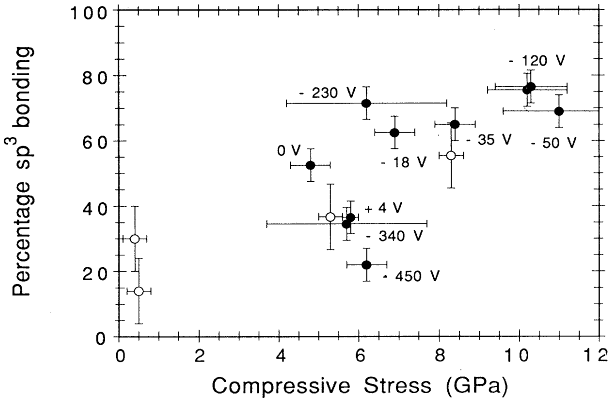

FIG. 4. Graph of the percentage of tetrahedral bonding as a function of the compressive stress. The solid circles represent the present work and are labeled by their bias voltages. The open circles represent plasmon energy versus stress points, taken from McKenzie *et al.* (Ref. 13) and converted to percentage sp^3 values using the relationship in Fig. 3. The points indicate that a high compressive stress is present in films with a high fraction of tetrahedral bonding.

Figure 5(a) shows a fit of Eqs. (3) and (4) to the compressive stress data. The fitted curve is seen to follow the data well. The parameters are found to take the values $pE_0^{-5/3} = 0.012$ and $c = 1.4$ when b is taken as 1. The thermal stability of DLC suggests that E_0 lies between 2.5 and 3 eV, which gives a value for $p \approx 0.1$. Though this value is less than unity, it is still not unreasonable for the thermal spike model. The fitting procedure gives a value of 16 eV for the ion energy at zero bias, a value close to that measured by Martin *et al.*[10]

Figure 5(b) shows the fit to the sp^3 fraction using Eq. (3) and the penetration fraction f calculated by TRIM simulations[21] and, to simplify the overall fitting procedure, approximated by

$$f = 1 - \exp\left[-\frac{E - E_1}{E_2} \right] , \tag{5}$$

with $E \approx 60$ eV. With E_1, the penetration threshold given by the displacement threshold minus the surface binding energy,[21] constrained to lie above 20 eV and $b = 1$, a best fit was found with $E_1 = 20$ eV and $pE_0^{-5/3} = 0.012$, the latter similar to that found above.

Equation (3) is seen to give a reasonably good description of overall shapes of the compressive stress and the sp^3 fraction in Figs. 5(a) and 5(b). The fitted parameter values are physically reasonable, which suggests that the subplantation theory of Davis[19] and Robertson[21] describes the major features of the deposition process.

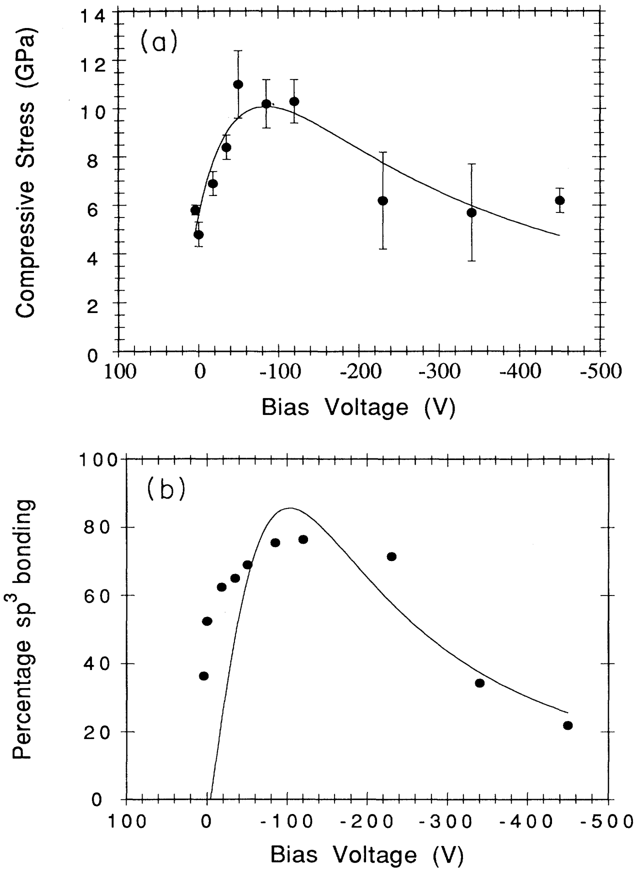

FIG. 5. (a) Theoretical description for the compressive stress, described by Davis (Ref. 19), fitted to the experimental data from Fig. 1(c). The function is described by Eqs. (3) and (4) and discussed in the text. (b) The equation proposed by Robertson (Ref. 21) fitted to the fraction sp^3 data from Fig. 1(b).

DISCUSSION

It is of interest that the present experiments find an optimum in ion energy of the order 140 eV, similar to Ishikawa *et al.*[6] and Koskinen,[8] but higher than that found in closely related work by McKenzie *et al.*[12] and Veerasamy *et al.*[22] This difference is believed to be significant and not due to statistical error. It is notable that the present data have less scatter than generally found previously. The cause is likely to be differences in deposition systems. The present apparatus and that of McKenzie *et al.*[13] and Veerasamy *et al.*[22] only filter the ion beam of neutral and particulate matter—they do not provide a complete mass selection of single species. The present films were produced with a higher magnetic field than in the previous study by Veerasamy *et al.*,[22] causing the present plasma to be dominated by C^+ ions. The plasma is thus similar to the mass selection apparatus of Ishikawa *et al.*[6] and Koskinen[8] who deposited *a*-C using pure C^+ beams. Since the transport efficiency of carbon ions is expected to increase with their charge state,[32] the average charge state of measurements by Veerasamy *et al.*, and possibly by McKenzie *et al.*, may have been nearer to 2. Although this would explain the lower values of the optimum bias found in these works, other factors are needed to account for it being so much lower. Additional factors may be the ion flux, which was 50% higher in the present work than in the earlier work by Veerasamy *et al.*,[22] and changes in the magnetic field which may also effect the plasma potential and therefore the ion energy—these possibilities are currently under investigation.

It is seen in Fig. 2 that the resistivity declines more rapidly with ion energy than the sp^3 fraction. On the other hand, the sp^3 fraction was found to be strongly correlated to the plasmon energy at both high and low ion energies (Fig. 3). This suggests that the short-range order and density are well correlated at both low and high ion energies; the higher ion energies do, however, produce additional disorder in the films which increases the density of gap states which in turn decreases their resistivity. This could explain the higher than expected plasmon energies for the −450- and −340-V-bias films seen when comparing Figs. 1(a) and 1(b). It is possible that the high plasmon energies are due to graphitization or partial graphitization of microscopic regions in the amorphous matrix.[18] Nanometer-sized graphitic particles have been observed in *a*-C films produced by the vacuum arc process.[17]

We finally discuss the carbon *K*-edge EELS spectra in more detail. The minima in the 285.5-eV peak at bias voltages around −120 V is accompanied by a slight shift in the peak positions to higher energies (Fig. 6). This is attributed to the larger band gaps of these materials. A small peak centered at 283.5 eV is also seen in these spectra and is similar to one observed by Pappas *et al.*[33] in 80% sp^3-bonded *a*-C produced by laser ablation. This peak may be present in the other spectra but be obscured by the larger 1*s* to π^* peak. The nature of the 283.5-eV peak is uncertain. We find that it does not change in films which have been grown in a hydrogenated plasma and thus is unrelated to hydrogen concentration. It is unlikely that this feature is due to empty π-valence-band states caused by impurities in the film lowering the Fermi

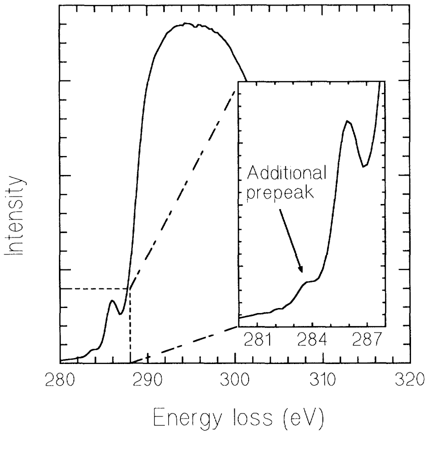

FIG. 6. Carbon *K*-edge EELS spectrum for the film produced at −120 V bias. In addition to the 285.5-eV 1*s* to π^* peak, a small additional prepeak is visible at 283.5±0.5 eV.

energy level,[34] as these films have large band gaps and low impurity concentrations. The possibility of this feature being caused by a core exciton is also thought to be unlikely. The feature may be due to dangling bonds causing band gap states. The feature would then increase with increasing sp^3 fractions, as presently observed. There are problems, however, with this interpretation as band-gap states would cause a large conductivity which is not observed.

CONCLUSION

The sp^3 fraction, plasmon energy, compressive stress, and resistivity of amorphous carbon deposited from a filtered ion beam of C^+ ions have been studied as a function of ion energy. The properties are found to be strongly correlated and to attain their most diamondlike values at an ion energy of about 140 eV, at which the maximum sp^3 fraction of about 80% is obtained. It was found that films with similar sp^3 fractions, produced at bias voltages both above and below the optimum value, exhibit similar properties and are thus deduced to be similar in structure. The dependence of the sp^3 fraction on ion energy was found to be well described by recent theory which attributes the high sp^3 fraction to a subplantation of incident ions below the surface atomic layer where they give rise to a quenched-in strain and density increment.

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[*] Present address: De Beers Diamond Research Laboratory, P.O. Box 916, Johannesburg 2000, South Africa.

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