Ceramics International **22** (1996) 1–5

0272-8842(95)00020-8
0272-8842/96/$15.00

Characterisation of a Saddle Field Source for Deposition of Diamond-like Carbon Films

A. A. Voevodin[a], J. M. Schneider, C. Caperaa,[b] P. Stevenson[c] & A. Matthews[c]*

[a]Laboratory of Electrochemical and Electrophysical Treatment, The Tula State Technical University, Tula, Russia
[b]Laboratoire de Physico-Chimie des Materiaux, Universite Blaise Pascal, Clermont-Ferrand, France
[c]Research Centre of Surface Engineering, University of Hull, Hull, HU6 7RX, UK

(Received 6 November 1994; accepted 3 January 1995)

Abstract: A saddle field cold cathode source was used to produce diamond-like carbon (DLC) coatings by dissociation and ionisation of acetylene in a low pressure vacuum. Langmuir probe investigations of the ion current density and plasma potentials showed the existence of a positive potential barrier at a distance 100 mm from the source with an ionisation rate up to 80% in the carbon flux in the near source region and less than 15% ionisation at larger distances. Film deposition rate, density and morphology were studied as a function of the geometrical arrangement of the samples. The feasibility of producing DLC films with good uniformity on insulating materials at distances more than 100 mm was shown.

1 INTRODUCTION

Ion sources are now widely used to produce diamond and diamond-like carbon (DLC) films. Ions accelerated by the electrostatic or magnetic field have an arrival energy sufficient to form sp^3 hybridised arrangements of carbon atoms. The main disadvantage in using ion sources is the accumulation of the positive charge on the film surface, when the film and/or substrate have high ohmic resistivity. This leads to a worsening in both film deposition rate and its properties after a certain thickness. Additional arrangements are needed to prevent charge build up on the film surface, i.e. by application of an r.f. field. In theory the deposition of DLC films from totally neutral carbon atoms of high kinetic energy is the best solution to this problem. One source supplying highly energetic carbon atoms is the saddle field source developed in the 1970s and 1980s. A short review of papers on the saddle field source will help in understanding its potential for DLC deposition.

The principle of the saddle field source is based on McIlraith's discovery of the electron electrostatic oscillator in 1965.[1] An oscillator consisting of two positive parallel anode rods symmetrically arranged around the main axis of the surrounding cathode cylinder was proposed. Electrons oscillating through a saddle point of the electrostatic field between two anodes have long trajectories and a high intensity cold cathode discharge is produced at low pressures. An aperture made in the cathode wall allows ions to escape from the source in the form of a highly energetic ion beam. This source was further studied and developed by Fitch and Rushton in 1972,[2] and ion beams of H, He, N and Ar produced by the source were studied by Ghander and Fitch in 1973–74.[3–5] Franks showed in 1973 that the saddle field configuration can also be achieved with a spherical cathode enclosing an annular anode.[6,7] After that, compact sources for thinning electron microscope specimens were produced by Ion Tech Ltd. Soon it was found that the thinning of insulator materials was also possible and, hence, the produced beams also incorporated neutrals, the content of which was initially estimated

*To whom correspondence should be addressed.

by Franks to be 30%. The efforts of Ion Tech Ltd in the development of sources with a high neutral component lead to the manufacture of a number of commercially available fast atom beam (FAB) sources with both rod (B110) and annular (B11) anodes.

The FAB sources were claimed by the manufacturer to be almost free of ions, which were thought to be neutralised by recombination with emitted secondary electrons produced by the collisions of ions with the cathode near the exit aperture.[8] However, Fitch *et al.*[9] showed that the symmetric resonance charge exchange between fast ions and slow neutrals can be the mechanism of neutralisation. The mechanism does not require the matching of momentum of high energetic ions and slow secondary electrons and was considered as the more probable one. A high concentration of the ions with low energies in the source output region can be predicted from this suggestion. The detailed studies of high energy fluxes produced by both cylindrical[9] and spherical[10] FAB sources revealed that the ion to neutral ratio and energy distribution of ejected particles is greatly determined by the voltage supplied to the anode, gas pressure in the source and type of gas. Nevertheless, saddle field sources produce ion and atom beams with a relative content of energetic neutrals higher then any other known sources.

The applications of the saddle field source in vacuum technologies were, until recently, limited to sample etching and thus the beams of the noble gases He, Ne, Ar and Xe were studied mostly. Recently, their usage in the production of DLC films from hydrocarbon gases was reported by Franks,[11] and the films' optical and mechanical properties were evaluated.[12–15] However, there were no detailed studies of the ion to neutral ratio of the beams produced from hydrocarbon gases. Taking into account the controversial data on earlier applications of FAB sources, these studies are needed to create a better understanding of processes occurring in DLC deposition using FAB saddle field sources. Here, studies of ion component and ionisation rate of highly energetic fluxes produced with this kind of source are reported.

2 EXPERIMENTAL DETAILS

2.1 FAB source parameters

A FAB saddle field source (type BP3 from Ion Tech Ltd) with rod anodes and a gridded aperture of 25 mm in diameter was used to produce highly energetic carbon fluxes (Fig. 1). The source was operated with acetylene gas at a background pressure

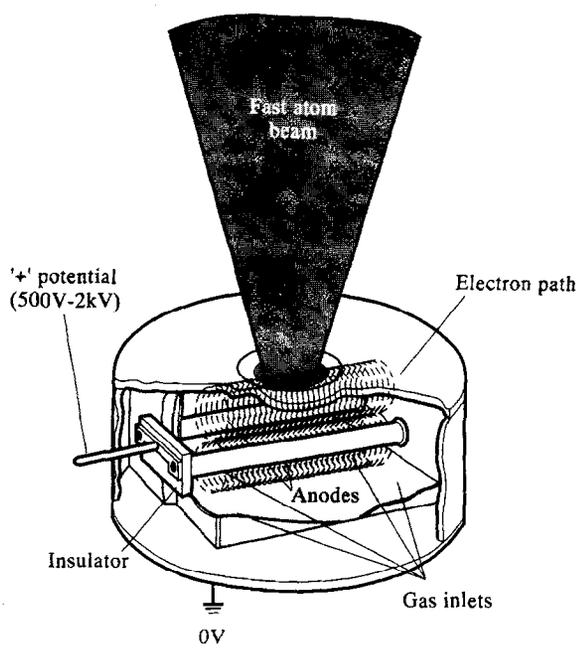

Fig. 1. Schematic of the fast atom beam (FAB) source.

in the chamber of 10^{-3} Torr, the current on the anode was 0·1 A and the anode voltage was kept constant at 1000V. The substrates or the plasma probe were placed at different distances z from the source outlet and at different radii R from the main axis of the source.

2.2 Flux characterisation

The ion component of the fluxes produced was investigated with a Langmuir probe comprising a plane disk. The probe was shielded at the back and sides and had a fixed aperture 10 mm in diameter in front of the collecting disk. Measurements of both the plasma floating potential U_f and the current to the probe at earth potential were carried out. Since no substrate bias was used, readings of the probe current can be said to represent the ion current density j to substrates placed between the source and the grounded chamber walls. The $U_f(z,R)$ and $j(z,R)$ functions were found by performing measurements for different cylindrical co-ordinates z and R in the chamber geometry.

To estimate the total flux density in the direction of the substrate, Langmuir probe investigations were combined with thickness and mass measurements of the films produced, followed by calculations of the film density. Then the ionisation rate i of the fluxes was estimated by the comparison of the ion current and total flux densities, using the following equation:

$$i = \frac{Z^{-1}e^{-1}j}{N_a \rho M^{-1} d} \tag{1}$$

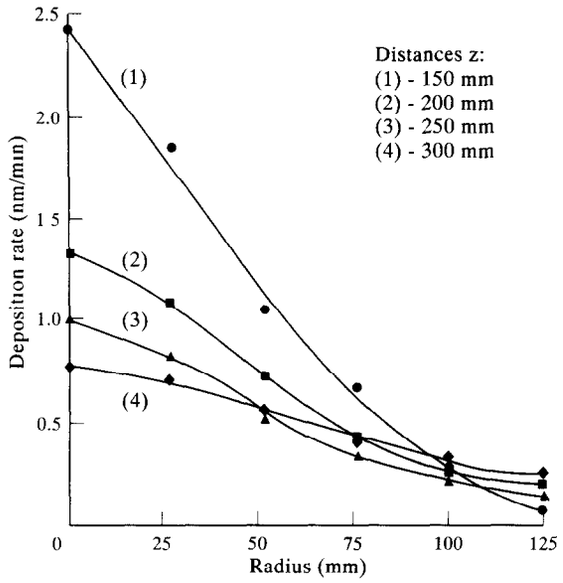

Fig. 2. Variation of the deposition rate with the radius R for different distances z.

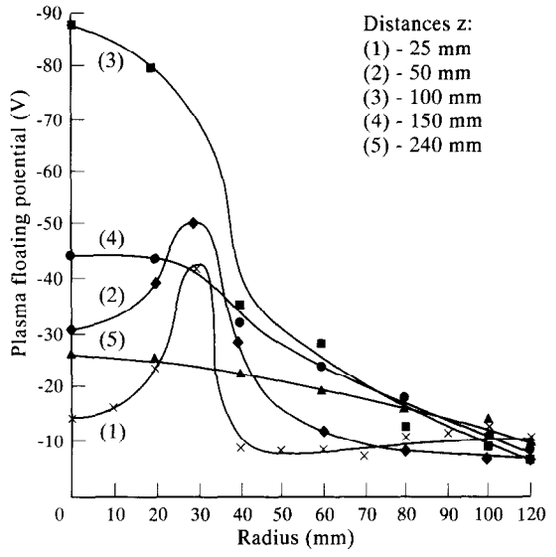

Fig. 4. Variation of the plasma floating potential via radius R for different distances z.

where e is the elementary charge, Z is the average charge of ions, d is the deposition rate, ρ is the film density, N_a is the Avogadro constant and M is the atomic mass of carbon. Charge Z was assumed to be equal to +1 for carbon, since the difference in ionisation energies between states +1 and +2 is a factor of two.[16] Film thickness and mass gain measurements were undertaken to find both deposition rate and density of the films produced.

The film thickness was measured by means of a surface profilometer. The film mass gain was measured with an error of 0·1 mg with a balance. Additionally the film surface morphology was investigated by scanning electron microscopy (SEM).

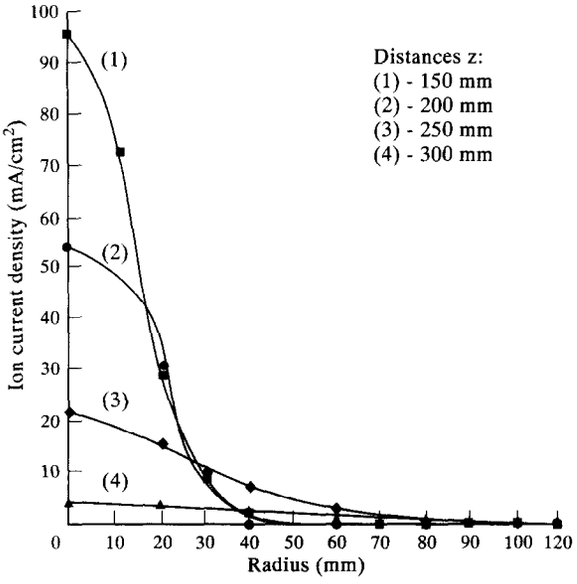

Fig. 3. Variation of the ion current with the radius R for different distances z.

3 RESULTS AND DISCUSSION

The variations of the deposition rate $d(z,R)$ (Fig. 2), the ion current density $j(z,R)$ (Fig. 3) at different points in the chamber were found to be as expected for strongly oriented beam sources — with a maximum at $R = 0$ close to the source and decreasing gradient of the functions $d(R)$ and $j(R)$ for larger z values due to the beam scattering in collisions with gas molecules.

The function $U_f(z,R)$ has different characteristics with a drop in the floating potential near the source outlet (Fig. 4). Only positive plasma potentials were fixed in all positions within the chamber. The near source drop can be attributed to the increased electron density in this region due to the secondary emissions of electrons from collisions of ions and the cathode grid. The drop of the plasma potential creates a potential barrier with maximum height at 90 V at a distance of 100 mm from the source (Fig. 5, curve 1). This barrier is non-transparent for low energetic ions created near the source outlet as a result of charge exchanges between colliding highly energetic ions and gas molecules, and thus a high concentration of ions exists constantly in the region $0 < z < 100$ mm (Fig. 5, curves 2 and 3). On the other hand this barrier is totally transparent for high energetic ions in the beam accelerated to 1 kV by the anode potential, which lose only 10–20 eV on passing through it due to the additional acceleration on the right slope of the barrier. The ion current to the probe for distances more than 150 mm (Fig. 5, curves 2 and 3) decreases by a factor of 10 and this represents the ratio of the low to high energetic

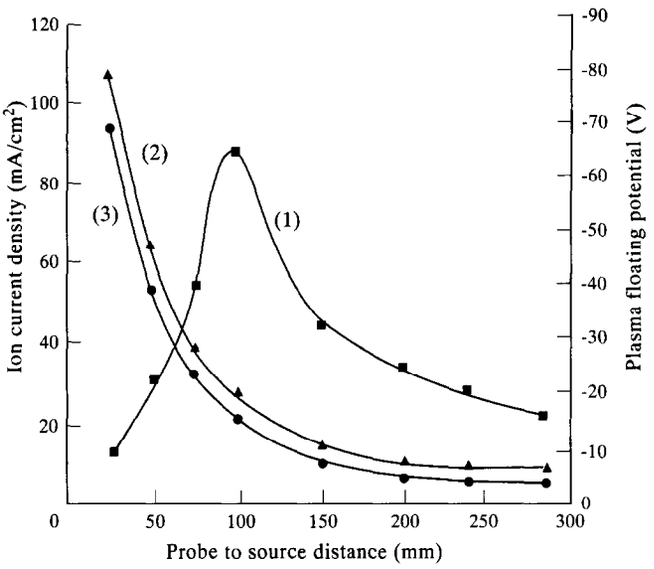

Fig. 5. Variation of the floating potential (1) and the probe currents at earth (2) and at –50F (3) with the radius R.

ions in the near source region. The beam investigations with the probe under –50V potential (Fig. 5, curve 3) showed a slight increase in the ion current in comparison to the probe measurements at earth potential (Fig. 5, curve 2). This confirms that the results obtained correspond to the positive current to the probe created by the arriving ions, rather than to the negative current from the probe by emitting secondary electrons from collisions of the high energetic neutrals with the probe area. We did not observe any radiation in the near probe region and presume that the secondary electron emission from the probe can be neglected in the treatment of the results.

Films produced in the near source region are non uniform in morphology (Fig. 6(a)), while

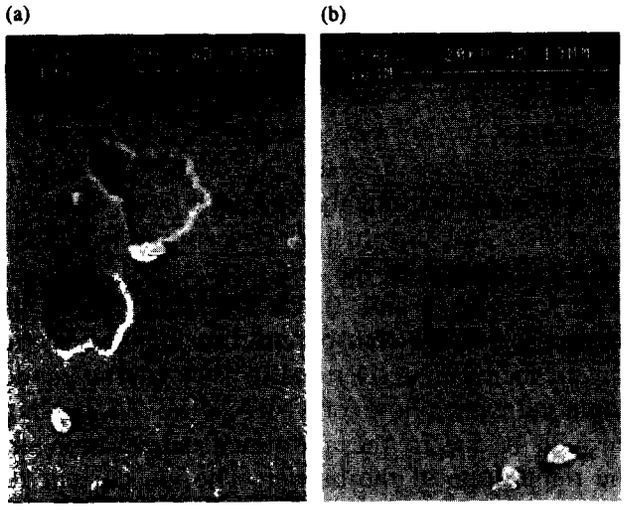

Fig. 6. Secondary electron image of the film morphology produced at (a) 50 mm and (b) 250 mm distance z to the FAB source.

Table 1. Film densities for different substrate to source distances

Distance z (mm)	Deposition rate at R = 0 (nm/min)	Density of films (g/cm³)
150	2·38	1·82 ± 0·32
200	1·32	1·87 ± 0·12
300	0·77	1·94 ± 0·18

films produced at distances of more then 150 mm from the source are more dense and homogeneous (Fig. 6(b)), their densities are given in Table 1. The film densities are approximately the same for all distances and have a value of 1·87 g/cm³ which was then used to calculate the ionisation rate of the fluxes. The results are presented in Fig. 7. The shaded area shows the range of uncertainty due to the error in the film density calculations. Nevertheless, it confirms that for $z > 150$ mm the level of ionisation of the fluxes is a constant in the range 10–20%. This value corresponds to highly energetic ions in the beam and can hardly be neglected in considering film growth, as was previously done by researchers.[11,12] The level of ionisation in the near source region was calculated to be as high as 80%, and rapidly decreases with increasing z to 100 mm.

4 CONCLUSIONS

It has been shown that the fluxes produced by the saddle field source contain a significant percentage of ions and, hence, cannot be considered as fully neutral fast atom beams. The ion composition of fluxes greatly depends on the distance to the source. In the near source region a level of ionisation of

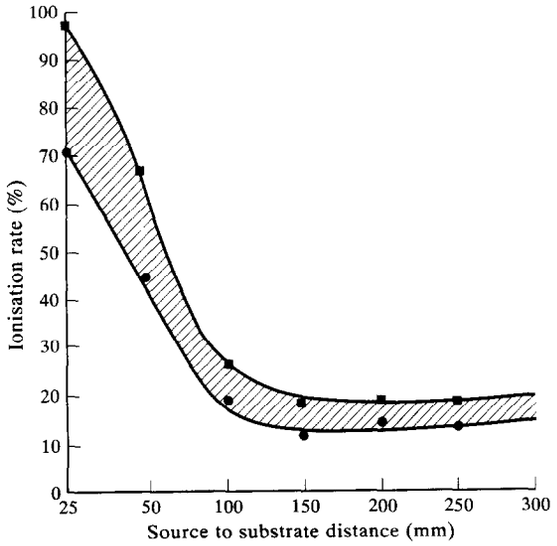

Fig. 7. Ionisation rate of the beam for different distances to the source in the axial direction.

80% was observed, the main contribution to it being from the ions with energies less then 90 eV trapped by the near source plasma potential drop. For distances more then 150 mm the ionisation rate is constant in the range 10–20% and corresponds to the highly energetic ions produced by the source. The ion component of fluxes influences the film morphology and has to be taken into account in the development of deposition processes of DLC films with such sources. Additional methods for film growth control can also be provided by using substrate biasing or placing additional electrodes between the source and substrate.

ACKNOWLEDGEMENTS

The authors are indebted to Dr J. Franks for helpful discussions on the FAB source. The equipment was provided by Ion Coat Ltd.

REFERENCES

1. MCILRAITH , A. H., 1965 British Patent No. 20592/65.
2. FITCH, R. K. & RUSHTON, G. J., *J. Vac. Sci. Technol.*, **9** (1972) 379–82.
3. GHANDER, A. M. & FITCH, R. K., *Vacuum*, **23** (1973) 269–70.
4. GHANDER, A. M. & FITCH, R. K., *Vacuum*, **24** (1974) 483.
5. CLARK, R. B., FITCH, R. K., GIANDER, A. M. & SMITH. A. G., *J. Phys. E: Scientific Instruments*, **7** (1974) 566–8.
6. FRANKS, J., 1973 British Patent No. 44718/73.
7. FRANKS, J. & GHANDER, A M., *Vacuum*, **24** (1974) 489–91.
8. FRANKS, J., *Vacuum*, **34** (1984) 259–61.
9. FITCH, R. K., KHORASSANY, M. & MAWLOOD, T. N., *Proc 7th Int. Vac. Conf. Solid Surfaces*. Vienna, 1977, pp. 285–8.
10. WOODFIN, V. & LIGON, J., *J. Mass Spectrometry and Ion Physics*, **41** (1982) 205–8.
11. FRANKS, J., *J. Vacuum Science and Technology*, **7** (1989) 2307–10.
12. DEHBI-ALAOUI, A., MATTHEWS, A. & FRANKS, J., *Surf. Coat. Technol.*, **47** (1991) 722–9.
13. HOLIDAY, P. DEHBI-ALAOUI, A. & MATTHEWS, A., *Proc. 1ST ASM Heat Treatment and Surface Engineering Conference*. Amsterdam, 1991, pp. 643–54.
14. SMITH, J., HOLIDAY, P., DEHBI-ALAOUI, A. & MATTHEWS, A., *Proc 2nd Conf. on Diamond, Diamond-like and Related Materials*. Nice, 1992, pp. 355.
15. DEHBI-ALAOUI, A. OLLIVIER, B. & MATTHEWS, A , *Proc. 11th Int Conf. Vacuum Metallurgy*. Paris, 1992, pp. 221–6.
16. LIDE, D. R., *Handbook of Chemistry and Physics*, CRC Press, 1991–2.