Technical Brief

CARBON AND METAL VACUUM EVAPORATORS INCORPORATING EMITECH K250, K400 & K450 CARBON 'FLASH' UNIT and K950, K975 & K975S CARBON ROD AND METAL EVAPORATORS

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INTRODUCTION

Carbon Films, because of their mechanical stability, good electrical conductivity, and low background signal are commonly used in sample preparation techniques for Electron Microscopy (EM).

Thin films of the order of up to 5 nm (50A) are used in Transmission Electron Microscopy (TEM) as particle support and as isolating layers in autoradiography. Thick films somewhat more than 5 nm (50A) are used in Scanning Electron Microscopy (SEM) also for support, in addition to coating for X-Ray Microanalysis. It may also be a requirement to subsequently treat support films by means of Glow Discharge.
In general there is a need for all these films to be fine grain, even coating, with uniform and reproducible film thickness.

The advent of Carbon fibre has resulted in a new principle for Evaporation of Carbon in EM applications. The basis is a carbon filament which at high temperature burns very quickly,somewhat in the order of 1 second or less, from which has evolved the terminology Carbon‘Flash’ Evaporation. This, together with the low total power input, due to short coating times,distinguishing it from the somewhat longer process of carbon rod evaporation. It is further distinguished in total cycle time, which is approx. 4 minutes including pumping and outgassing, by the fact that it can be carried out at Rotary Vacuum Pump pressures. These pressures (relative Vacuum) of the range 0.05 to 0.01 torr giving mean free paths of the order of 1 to 5 mm respectively.

Evaporation under these conditions is not unidirectional as the particles engage in a number of collisions before they arrive at the surface, enhancing the possibility of a uniform coating, even on sculptured surfaces.

PHYSICAL CONSIDERATIONS.

The carbon particle evaporation is dependent on several factors. The surface area, temperature, and the length of the filament. The filament itself can take several forms including multistranding and “made up form” carbon fibre. Although generally the “made up form” is more convenient. The Fig. 1. shows a range of samples alternatives for use as filaments.

Table (a) Fibre
(b) String
(c) Cord.

Figure 1.
Note: the carbon fibre provided by Emitech, catalogue number C5461 is very much recommended to give the best performance (string and cord are available if required)

Differing arrangements may be used to cover a range of applications. It is more convenient to have a fixed length filament for both mechanical simplicity, and in determining the requirementsof the Power Supply, this also ensures for particular arrangement that high repeatability of Carbon Evaporation can be achieved.It is considered that a filament length of 10 mm (Exposed length for evaporation, the actual length being somewhat longer to accommodate clamping) is satisfactory. The power supply requirements can now be determined which are twofold, it being necessary to flash the carbon filament rapidly with full applied power to ensure a good repeatability and also prior to this, to outgas the filament at the higher end of the red heat temperature band, for a time period and with variable current facilities to allow for the differing arrangements of filament. In the vacuum range we are considering of 0.05 to 0.01 Torr where the mean free path lengths are relatively short, of the order of 1-5 mm, then if the specimen is several times this spacing from the filament, the coating will not be as sensitive to vacuum. In general the outgassing would be 0.1 Torr with flash evaporation, at 0.05 Torr or better and it is good practice to repeat parameters to ensure consistent results. The filament itself tends to act as a point source and in the flash process ‘breaks’ as part of the process without fully evaporating along its complete length. The geometric arrangement is indicated in Fig. 2. which has a shutter used during the outgassing process to avoid heat input to the specimen. The filament length (10 mm) can normally be considered as a point source and theoretically the thickness (t) of coating will vary inversely proportional to the height of the specimen to the filament squared. (t proportional to $1/h^2$). In practice due to the short mean free path this relationship is somewhat less than that indicated.

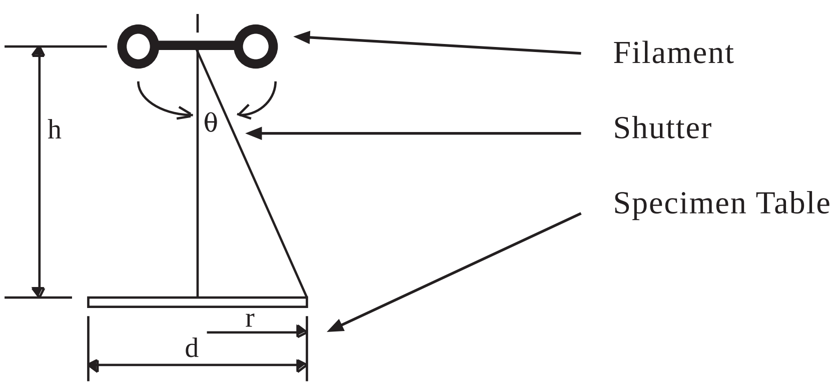

Fig. 2.

The apparent point source of the filament also suggests another consideration in coating a flat substrate, which is related to the angle for differing heights. If the angle is large the variation of coating of a central area to that on an outer edge will be somewhat different. This is given in the table showing the percentage coating for an angle of c which can also be related to the height h to radius r, although again due to collision with the short path length it is not as sensitive as may be anticipated from the table. However it serves as a good basis for considering the height range requirement (usually variable) and specimen table diameter (usually fixed). It might be reasonable to consider a figure of 85% as the acceptable limit, with a ration of 0.4. If we use a specimen table of diameter d of 50 mm (r=25 mm). Then h would need to be some 65 mm. In practice the system has a 50 mm. Diameter specimen table with variable height facility centring of 65 mm. with a + - 20 mm. range giving 45 mm to 85 mm. As has already been mentioned however, in practice the coatings tend to be much more evenly deposited over this range. The Fig. 3 gives examples of slides which have “blanked off” areas for visual effect, indicating varying grey tones from uncoated to a reasonably heavy coated slide. This is covering a range of thickness, and indicates the versatility of the technique. The accompanying table giving the range of parameters used. NOTE: Print reproduction may not repeat readily the changes in grey tones apparent on the original slides.

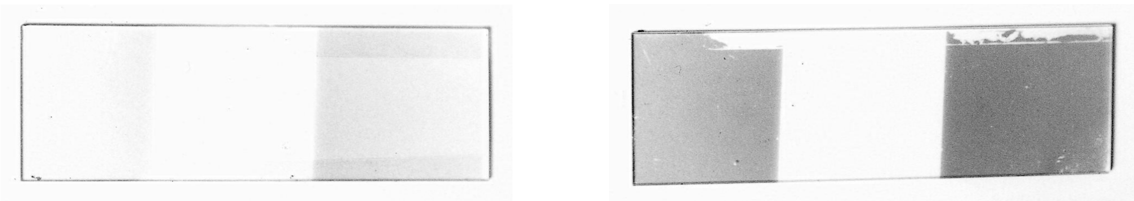

Fig. 3.
Note: the carbon fibre provided by Emitech, catalogue number C5461 if very much recommended to give the best Note: the carbon fibre provided by Emitech, catalogue number C5461 if very much recommended to give the best performance (string and cord are available if required)
Although the thicknesses are estimated, what is usually significant is the high repeatability. The total heat Input of the system and the final Heat Input to the specimen are also low compared to conventional carbon rod evaporation.

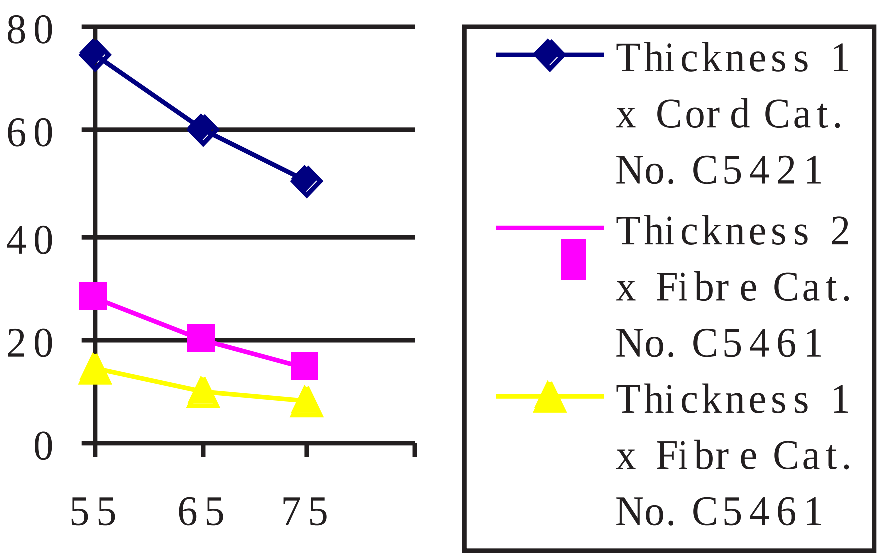

Fig 4. Emitech K450 Carbon Coater which is suitable for use with a range of Carbon Fibre Filaments now available. The K450 is a fully automatic unit outgassing the fibre and subsequently flash coating. Note: This unit is also available as the semi-automatic K400, which also allows flexibility of control by the customer.

Fig. 5. Emitech K250 Carbon Coating Attachment which is manually operated

This is usually demonstrated with freshly broken polystyrene, however a typical specimen would, of course, only be subject to a relatively small percentage of the total energy input. It can be considered that if we satisfy the Physical considerations discussed then we should be able to produce Carbon Films which are Fine Grain, very repeatable, with a flexible range in Carbon "Flash" Evaporation systems.

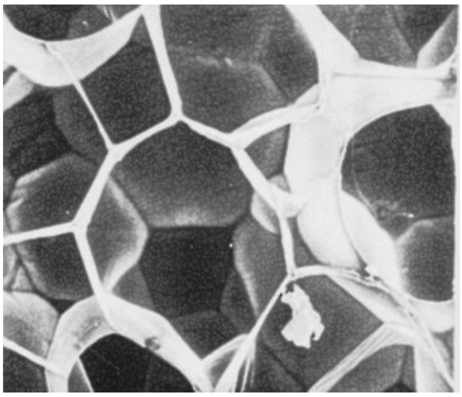

Fig. 6. Scanning Electron Micrograph of expanded polystyrene coated with carbon. Magnification x 1500. this is a very sensitive sample which is free of heat damage.

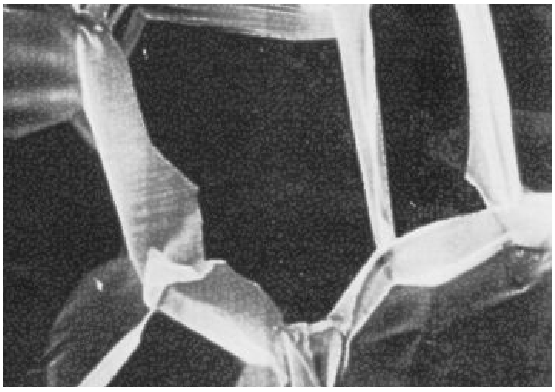

Fig 7. Similar specimen to 6, cool sputtered with Gold included as an undamaged standard.

TURBO PUMPED, HIGH VACUUM SYSTEMS

K950 Turbo Pumped system, with multi-change head system for Carbon rod, Carbon fibre and metal evaporation. The unit also allows for Glow Discharge treatment of samples using +/-HT dc supply option.

Fig. 8. K950

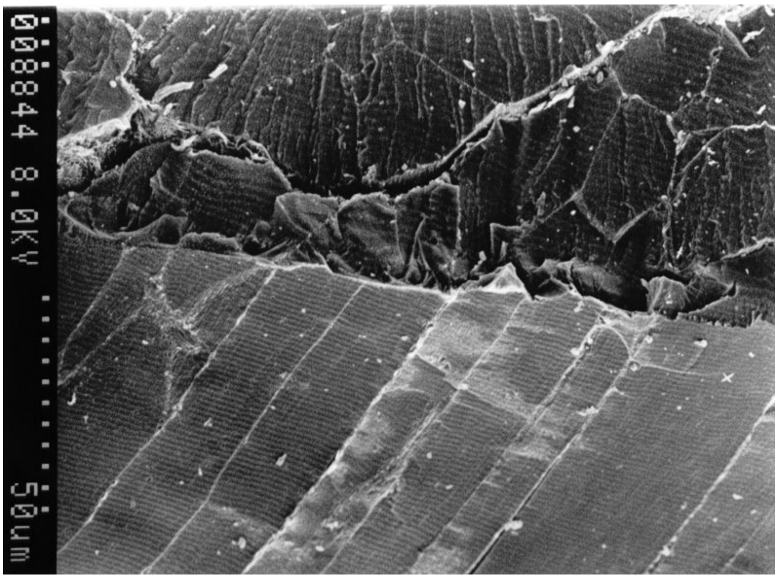

Fig. 9. Micrograph of leg muscle of a mouse, dehydrated in alcohol, frozen fractured, thawed in alcohol, then critical point dried in Emitech K850, resin embedded, Rotary Carbon coated, the coated with Platinum.

K975 Turbo pumped high vacuum system, with large 10 inch (250mm) Chamber with multi-source supply, and rack out drawer loading system. Rotating and tilting stage and planatary stages adjustable under vacuum with accurate external tilt control. Unique anti-stick, constant pressure carbon gun for controlled evaporation, sequential coatings without breaking vacuum.

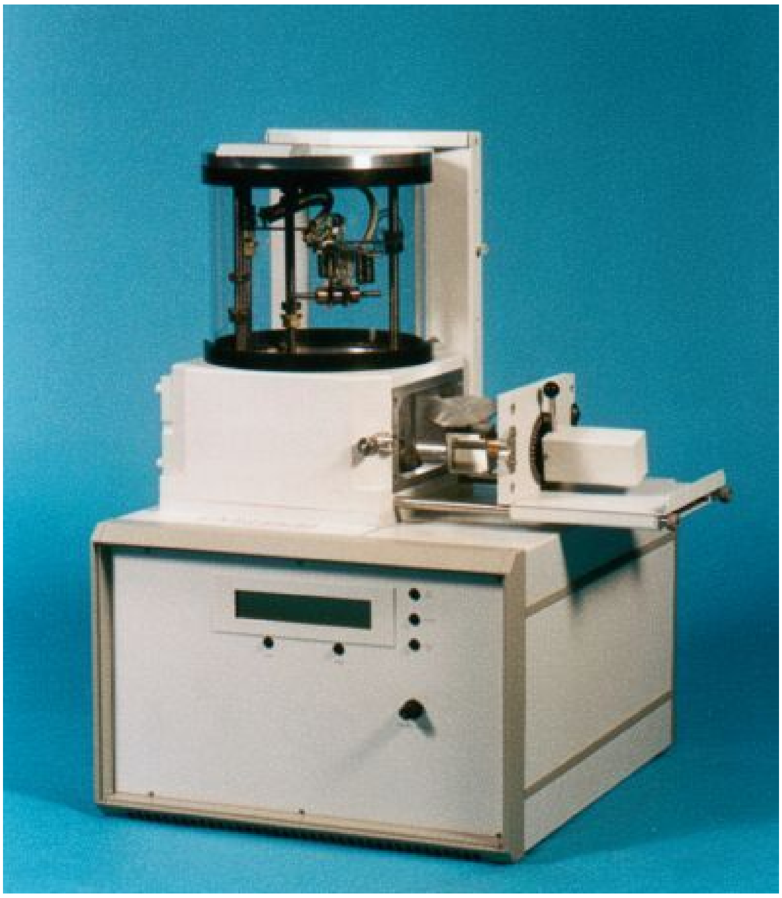

Fig. 10. K975

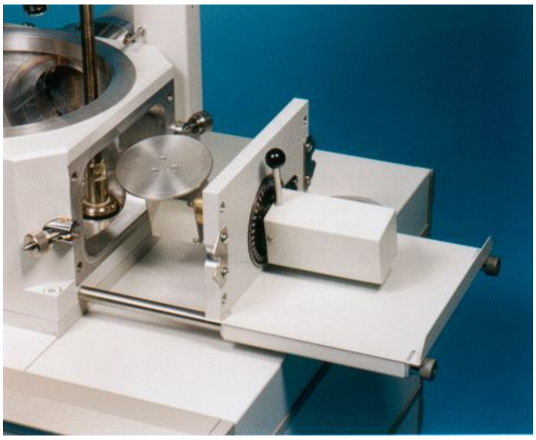

Fig. 11. Rack out drawer with rotate/tilt stage.

Fig. 12. Multi-source chamber and jigging.

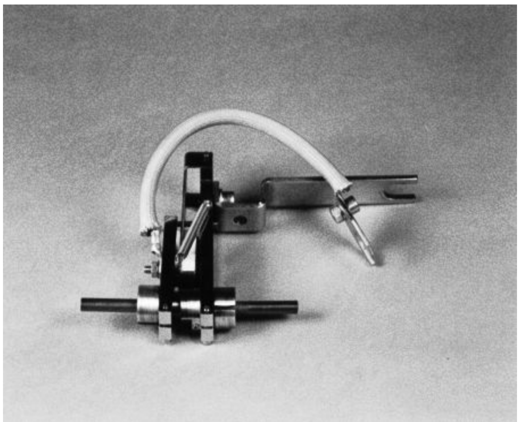

Fig. 13. Anti-stick carbon gun.

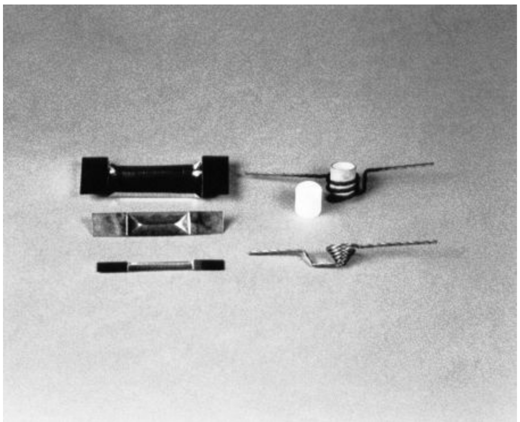

Fig. 14. Range of evaporation sources for use in K975, including Boron Nitride Crucibles.

K975S. This is the same unit as the K975, however, a special loading stage is fitted to allow for 8 inch (200mm) wafers with carbon coating or other samples upto 5 1/2 inch (140mm) square.

SURFACE TREATMENT

Freshly made carbon support films tend to be hydrophobic surface which inhibits the spreading of suspensions of particles in negative staining solutions. However, after glow discharge treatment with air, the carbon film is made hydrophilic and negatively charged, thus allowing easy spreading of aqueous suspensions. With subsequent magnesium acetate treatment, the surface is made hydrophilic and positively charged.

Atmosphere	Surface	Charge
Air	Hydrophilic	Negative
Air	Hydrophilic	Positive (With Subsequent Magnesium acetate treatment)
Alkylamine	Hydrophobic	Positive
Methanol	Hydrophobic	Negative

In addition to glow discharge treatment using air, other process gases may be used to modify surface properties. For example, if Alkylamine is used as a process gas the carbon film surface will become hydrophobic and positively charged. On the other hand using methanol as a process gas results in the surface becoming hydrophobic and negatively charged. Such treatments can facilitate the optional absorption of selected biomolecules.

Note: The Emitech GD350, glow discharge attachment is available for subsequent surface treatment of carbon films produced with the Emitech carbon coating range, e.g. K450, K950. In addition separate free standing glow discharge unit, the K100 is available.