A SIMPLE TECHNIQUE FOR THE PREPARATION OF ULTRA-
THIN CARBON FILMS FOR ELECTRON MICROSCOPY*

by

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ABSTRACT

Ultra-thin supporting membranes of carbon are valuable in electron microscopy of biological samples and for some applications as examination of fine particles or thin metallic films obtained by vacuum evaporation which require high resolution.

A simple method has been developed for production of support films thinner than 20 Å. These specimen supports show a good mechanical stability and a total open area of a 400-mesh grid is usable.
INTRODUCTION

In the technique described here, a thin layer of carbon is condensed in a vacuum upon the surface of Parlodion film (pyroxylin, cellulose nitrate 1% solution in amyl acetate) already deposited on the copper grid of the microscope and simultaneously on a glass slide "indicator" with a thin wire (4 mils) across, touching the surface (see Figure 2). This is useful for making an accurate measure of the thickness of the carbon films (the actual thickness is measured by the optical interference method of Tolanski within 30 Å, using a Thallium radiation of 5370 Å).

After the evaporation of carbon, the grids are disposed on a large grid and placed above amyl acetate vapor in order to dissolve the Parlodion film for 2 hours at room temperature. (Longer exposure of the copper grid under isoamyl acetate vapor can produce copper carbonate and contaminate the support film.) A grid control with a film of 2% parlodion without carbon is placed at the same time and observed later in the TEM to be sure that the parlodion film has been dissolved.

PROCEDURE

A typical evaporation is carried out with the glass slide indicator placed at 3.5 cm from the source and the target grids at 13.5 cm which are placed on top of a rotating disc. The suitable arrangement is shown in Figure 1.

"E" is the large pyrex tubing of 12.00 cm OD placed under the carbon rods where we settled the grids on the center (bottom).
"F" is the smaller pyrex tubing of 3 cm OD placed above the carbon rod and where we settled the system holding the clean glass slide with its wire.

Both glass tubings are lightly smeared with silicon vacuum grease to partially reflect the carbon atoms scattered from this surface onto the substrate and the glass slide. The diameter of both pyrex tubing and the distance between grids, carbon rods and glass slide are determined so as to ensure that the evaporation of carbon obeys the inverse square law.

A fine control (micro-contact) was incorporated in the electrical circuit of the evaporator in order to be able to do the evaporation by short flashing. In general two or three flashes were convenient to obtain an amount of carbon of about 150 Å on the glass slide.

CONCLUSION

The calculated amount of carbon deposited is 10% of that deposited on the indicator glass slide surface placed above the carbon rod. Thicknesses ranging from 150 Å to 200 Å were thus measured.

Ultra-thin films of 15 ± 3Å thus produced, have proven to be extremely strong and stable under the electron beam and covered the total of the open area of the copper mesh grid.

*Films of the order of <100 Å (<10 Å on the grid), can be easily evaporated but it is not possible to measure them by interference methods.
REFERENCES

3. R.C. Williams, and all, Tweney-Ninth Annual EMSA Meeting, page 482.