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Citation: Review of Scientific Instruments **31**, 197 (1960); doi: 10.1063/1.1716924
View online: http://dx.doi.org/10.1063/1.1716924
View Table of Contents: http://scitation.aip.org/content/aip/journal/rsi/31/2?ver=pdfcov
Published by the AIP Publishing

Preparation of Thin Self-Supporting Carbon Films

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(Received November 16, 1959)

A method is described for the preparation of self-supporting carbon films of thickness between 4 $\mu g/cm^2$ and 1 mg/cm^2 and areas up to 1.5 cm^2. These have many applications as targets and backings, and have been used as beam strippers in the Harwell Tandem Generator.

INTRODUCTION

A RECENT paper[1] has described the preparation of thin carbon films by the method of cracking methyl iodide onto thin nickel foils, and subsequently dissolving the nickel in acid. During this process, there are frequent losses due to the strains induced by the chemical reaction, and there is the risk of some contamination unless the washing is thorough. Furthermore, the method does not lend itself easily to the preparation of a number of foils simultaneously.

We have used a different process for the past six years and found it very satisfactory. The technique is based on the method of evaporation of carbon from an arc *in vacuo* onto a prepared plate as first described for the production of replicas in electron microscopy by D. E. Bradley.[2] The procedure, as applied to the preparation of targets and backings, hitherto has not been described in detail, but was outlined briefly in an account of elastic scattering experiments.[3]

As mentioned by Kashy *et al.*,[1] the thinness and low atomic number of the carbon foils make them especially valuable as backings for evaporated targets in elastic scattering investigations; they are also useful in the study of other nuclear reactions owing to the high purity attainable, and the fact that carbon is likely to be present on the target after bombardment, in any case, as a result of the charring of grease films by the beam. The foils will withstand a considerable beam current for appreciable periods of time, for example 5 μamp of alpha particles at 2 Mev for several hours without damage. Foils of about 5 $\mu g/cm^2$ have been used successfully as beam strippers in the Harwell Tandem Electrostatic Generator over the past eight months. The stripper foils are $\frac{3}{8}$ in. diam and appear to withstand around 5 μa of protons at several Mev almost indefinitely, the losses being attributable to films which were faulty in the first instance or to accidental surges of pressure in the machine. Indeed, there is evidence that the films become stronger after 50-hr bombardment. At these low thicknesses, and with the low atomic number of carbon, the scattering of the beam is not serious, while the larger area of the foil stripper places less stringency on focusing and alignment of the beam than does a gas-stripping tube.

[1] Kashy, Perry, and Risser, Nuclear Instr. and Methods 4, 167 (1959).
[2] D. E. Bradley, Brit. J. Appl. Phys. 5, 65 (1954).
[3] G. Dearnaley, Phil. Mag. 45, 1213 (1954); 1, 821 (1956).

EXPERIMENTAL PROCEDURE

A carbon-arc apparatus is constructed with dimensions to allow mounting in a vacuum evaporator. Rods of carbon $\frac{1}{4}$ in. diam are suitable and can be obtained spectroscopically pure. The pure carbon yields stronger films, probably owing to the reduced outgassing during the evaporation. As shown in Fig. 1, these are spring loaded in copper tubes recessed into the brass blocks to avoid the possibility of copper being sputtered from the ends onto the plate carrier. This may consist of two bars which can support several 3×1-in. microscope slides at a distance of 10–15 cm from the arc. The carbon rods are best turned so that one has a point and the other a truncated conical end. The springs maintain the pressure lightly between the rods as the carbon is evaporated away. A supply of at least 100 amp at 12 v is required, but only need be rated for short-period use. An appreciable pumping speed of at least 50 liters/sec is advantageous to deal with the outgassing from the hot carbon rods.

The procedure is to coat the slides thinly on one side with a liquid detergent such as Shell "Teepol" (alkyl sulfate). The surface is polished as it dries so that only a thin uniform layer remains. The slides are placed with coated side downwards above the carbon-arc assembly, and the apparatus pumped to about 10^{-4} mm Hg. The 12-v supply then is switched on for a period of 1 to 3 sec, after which the chamber is allowed to pump for 10 sec or so. Three to 10 such flashes give a grey-brown deposit on the slides corresponding to a thickness of 5 to 20 $\mu g/cm^2$; the carbon layer is opaque when greater than about 80 $\mu g/cm^2$. The films then may be floated off on water by lowering the plate slowly into the water at an angle of about 30° to the surface. The detergent dissolves and the film is supported by surface tension. Before floating the films off, it is advisable to rub the edge of the slide with a wooden pencil in order to loosen the film at its edges, where it tends to adhere to the glass. The film is cut conveniently into pieces of the required size with a pointed instrument before being floated off; a 1×3-in. slide will provide up to 8 target backings, and several slides can be prepared by a single

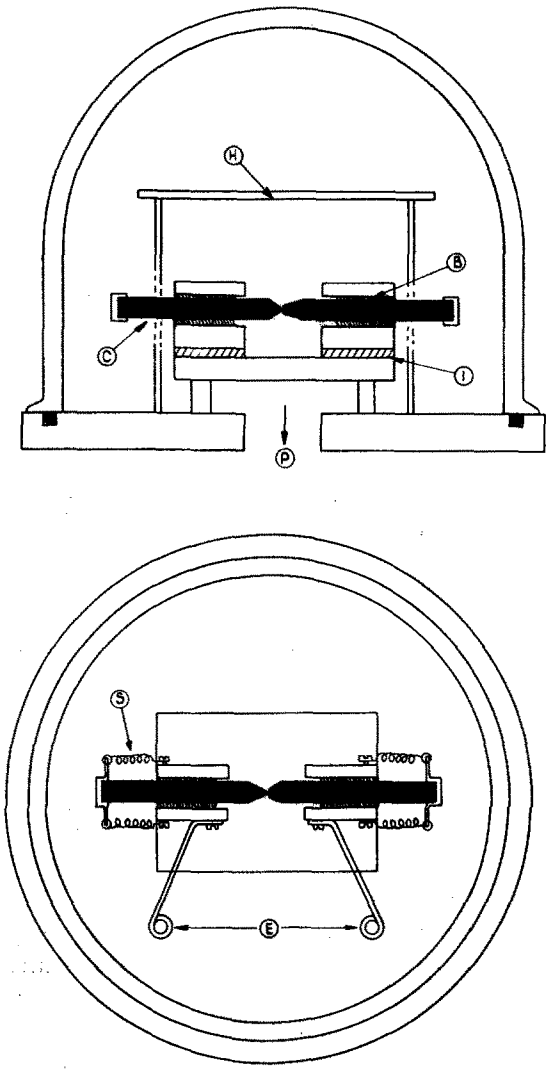

FIG. 1. Apparatus for carbon-arc evaporation. B, copper bush recessed into brass block; C, carbon rod; E, electrode; H, slide holder; I, insulator; P, to diffusion pump; S, spring.

evaporation. It proves necessary to clean the apparatus thoroughly between successive evaporations, as otherwise the films produced are less strong and tend to shred to pieces on the water surface. Possibly this is due to the poorer vacuum during evaporation. The films may be picked up on flat metal frames by dipping these into the water and raising them slowly at a steep angle. Either a single or a double film can be raised in this manner, and it is best to allow the film to drain for a few seconds with its lower edge in the surface before removal. Breakages are reduced, at the risk of very slight contamination, by the addition of a drop of detergent to the bath before lifting the films, in order to lower the surface tension. A momentum spectrum of protons scattered from such a target, using magnetic analysis, does not reveal any detectable contamination due to this procedure. It seems easier to float the foils off within a day or two of the evaporation; if left for a longer period, the adhesion is greater.

It has not been possible to prepare films of weight less than 4 $\mu g/cm^2$, as below this thickness the films break up, when one attempts to float them off. It is best to use very thin layers of detergent for these thin foils, and to float them off on warm water which dissolves the detergent more easily and has a lower surface tension, and so is less likely to tear the film apart. At weights above about 50 $\mu g/cm^2$, the film shows a tendency to curl up into filaments on the water surface. Thicker films of up to 1 mg/cm^2 have been prepared by picking up double films on frames made tacky with water-insoluble adhesive. When dry, more adhesive can be applied and another film picked up.

Once dry, the films become very robust and will keep indefinitely, without deterioration, in air. The momentum spectrum of protons scattered from the films shows them to be of very high purity and uniformity. It is possible to evaporate a wide range of materials onto the carbon, to thicknesses considerably greater than that of the backing film. We have evaporated beryllium onto the foils at about a 1300°C temperature without fracturing them. Some materials (e.g., LiF) decrepitate and puncture the film, while the alkali metals produce a curious crinkling and weakening of the foil. Alternatively, it is possible to deposit the target material onto the carbon while still on the glass slide. A soluble material, such as LiF, will not dissolve through the film into the water.

I am indebted to Mr. J. G. Robins and Mr. M. Nobes of the Atomic Energy Research Establishment for accounts of their experience in the preparation of carbon foils.