A Dry Powder Technique for the Preparation of Carbon Foils*

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ABSTRACT

One through eight mg/cm^2 carbon foils for use as accelerator targets have been prepared from an air suspension of graphite powder which was allowed to settle and was subsequently pressed. Keys to the process are the use of: 1. a DeVilbiss medicinal powder blower and 2. vacuum-evaporated carbon coated glass slides as the substrate for the settling and pressing operations. In all of several trials, the targets did not stick to the glass slides, could be handled with forceps, trimmed with a razor blade, and mounted over a 20 mm dia. hole. Uniformity was better than 10%.

INTRODUCTION

The method to be described is a successful effort to bridge a nebulous area between processes which produce pure carbon films by vacuum evaporation and those which produce films by straightforward pressing and/or machining. A principal attribute of the method is the short time required to produce a self-supporting film. Also, potential residues associated with liquid-medium settling techniques are not possible and no binder materials are used. While the use of amorphous carbon is precluded because it is hard and will not bind internally to produce a strong film, in contrast to graphite, additional properties of low thermal conductivity and high electrical resistivity[1] reveal it to be a poor choice for accelerator targets anyway.

A useful film size from 10 mm x 25 mm to 25 mm square can be routinely produced. All excess carbon remains in the column and is recoverable. The method is therefore economically feasible for isotopically enriched carbon provided it has been graphitized (recrystallized) at 2500°C or more, and enough material for an initial charge of 75 to 300 mg[2] is available.

Although the dimensional relationships of the settling column components were not found to be at all critical, a fully dimensioned figure has been provided. The DeVilbiss pocket-type powder blower no. 38, the heart of the setup, may be purchased at drugstores and medical supply houses.[3]

PROCEDURE

When the settling column has been assembled as shown in the figure, it is charged with 75 to 300 mg of 325 mesh graphite powder.[4] One then selects a 20 μg/cm^2 (not a critical thickness) vacuum-evaporated carbon coated glass slide (25 mm x 75 mm) to score and break into two 25 mm square pieces. After one of the pieces is weighed, it is placed on the top disk (carbon side up) of the removable support wire and lowered into the column. The appropriate height from the bottom of the column can be determined with a few settling trials. Seven centimeters from the bottom of the column (i.e. the top of the funnel) to the top support disk produced an even carbon powder layer with the apparatus described.

A 0.25 mm thick piece of surgical rubber sheet (or similar material) with an appropriate slit for the support rod is used as a top cover. With the cover resting in place and the supply gas or air[5)] adjusted to less than 30 psi, a foot operated free-venting air valve[6)] is tapped lightly and quickly one or more times.

Best results are obtained when the powder is allowed to settle somewhat between these pressure pulses. As each pulse of air in the column reaches the thin rubber cover, it bulges and collapses to provide a gentle brake and reverse pulse. This action discourages suspended powder from being carried out. Also, the free-venting feature of the air valve helps in controlling the carbon powder by providing a controllable pulse of shortened duration. By monitoring the height of the powder in the column with a flashlight and the room lights off, one can avoid blowing any powder out of the top.

Visual inspection is usually adequate to determine when the slide piece should be removed from the column to weigh the deposit. If the coating is too thick or is non-uniform in appearance, the deposit is dumped back into the column and the slide piece is reused for another trial. Of course, if the deposit is too thin, the slide is returned to the column for additional powder. Handling of the coated slide is not particularly difficult.

Having arrived at a satisfactory coating, one transfers the coated slide to a hardened platen and carefully places the mating 25 mm square slide piece (carbon side down) on top of it. This is the most delicate operation in the process. And if it is not done slowly and smoothly, carbon will blow out from the sides or shift on the slide. A mechanical lowering jig may be necessary if one encounters difficulty.

Another hardened platen, added after moving the assembly to a hydraulic press, completes the sandwich for pressing. Fourteen tons/in^2 (1.93×10^8 nt/m^2) is adequate to compress the graphite powder into a lustrous, somewhat flexible film. The pressure must be released as smoothly as possible.

Noteworthy at this point is the need for "reasonable" parallelism of the press platens. The lack of which, as one might expect, will cause the glass slide pieces to convert to powder with an impressive bang! Surrounding dust shields of cardboard, etc. are highly recommended.

Trimming, measuring and reweighing establish the final dimensions of the film. Any existing non-uniformities of 10% become very evident after pressing. Thicker areas are more reflective of light, thinner areas are dull, and shifted areas are revealed by boundary lines.

IMPLICATIONS

One should, hopefully, be able to apply modifications of the above technique in preparing some other desired thin films. Sulfur is one of these. To date, the column has produced good deposits with sifted sulfur powder but a satisfactory releasing substrate for the pressing operation has not been found. However, if one is willing to sacrifice the completely dry nature of the described process, it should be possible to coat the glass slide pieces (or other temporary substrate) with a substance to be dissolved in a suitable solvent after pressing.

As ever in nuclear target-making, the desire to economically produce thin films from expensive materials precipitates an involving work in an evolving playground.

Reference and Footnotes

*Work supported in part by the National Science Foundation.

1. Coordination and editing by J. F. Hogerton and R. C. Graff, Reactor Handbook - Materials, Tech. Information Service U.S.A.E.C., p. 133, declassified 1955.

2. Larger quantities are easier to work with.

3. Also available from The DeVilbiss Co., P.O. Box 552, Somerset, PA 15501.

4. 99.5% graphite powder is available from Alpha Division of Ventron Corp., 152 Andover St., Danvers, MA 01923.

5. According to the Reactor Handbook - Materials, p. 141, "Graphite will absorb about 0.09 weight-percent of water vapor in a saturated atmosphere at 20°C. Absorption at this temp from an atmosphere of 60-percent relative humidity is negligible."

6. A free-venting air valve is a normally-closed valve of 3-way design that exhausts the down stream line to atmosphere when the valve is unenergized.

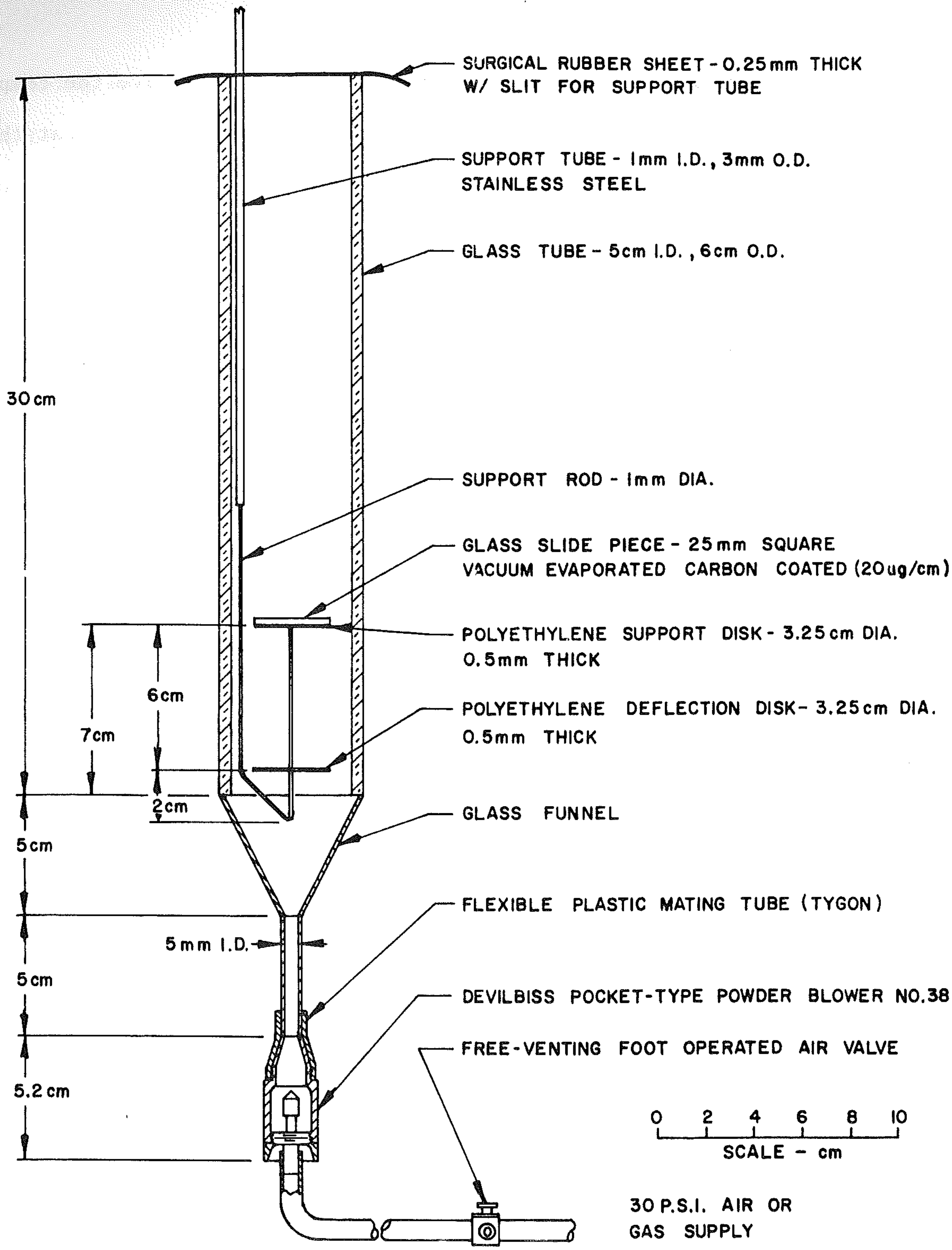

Figure 1. Air suspension/settling column for graphite powder.