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THE PRODUCTION OF THIN Be FOILS

by

Hugh Bradner

March 18, 1948

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ABSTRACT

A procedure for making Be foils between 10^{-5} cm. and 10^{-3} cm. thick, and with diameters up to an inch and a half is described, and methods of mounting these foils are indicated.

THE PRODUCTION OF THIN Be FOILS

By Hugh Bradner

March 18, 1948
Contract No. W-7405-Eng.-48

Radiation Laboratory
University of California
Berkeley, California

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THE PRODUCTION OF THIN Be FOILS

ABSTRACT: A procedure for making Be foils between 10^{-5} cm. and 10^{-3} cm. thick, and with diameters up to an inch and a half is described, and methods of mounting these foils are indicated.

Introduction

The problem of making thin Be foils was presented in connection with focussing the proton beam in the Berkeley Linear Accelerator, and although the foils are not now used in the accelerator, they have proved to be quite useful as thin targets, target supports, thin windows, etc.

The Be was evaporated in vacuum and deposited on a metal collector plate. The problems encountered were: (1) obtaining reproducible quantities of evaporated Be; (2) stripping of Be from the collector plate; (3) preventing curl of the foils when they were stripped; and (4) mounting the foils.

Apparatus

Figure (1) shows an exploded view of the vacuum chamber and apparatus used in evaporation. The chamber itself, approximately 22" in diameter, and 14" deep, was mounted on a 14" oil diffusion pump which made it possible to evacuate the chamber to a working pressure of 10^{-5} mm. in a few minutes. Liquid air traps were not needed. The Be was evaporated from a BeO crucible (1) held in a 40 mil thick, 200 mil wide tantalum ribbon filament (2). The filament, supported on its two electrodes is in the proper position in the figure. The crucible extended to within 5 mm. of the top of the helix. The radiation shield (3) normally rested on the bottom of the vacuum tank and extended around the filament. The metal sheet (5) upon which the Be (4) was evaporated was held approximately 8" above the filament. A plate (6) with one or more punched holes was directly underneath the collector plate and served to limit the size of the foil. The collector plate could be held in place by a lavite block (7) with nichrome wire for preheating the collector. A thermocouple (8) could be attached to the corner of the collector plate. The progress of the evaporation could be watched through a glass port at (9). Not appearing in the picture is a shutter mounted on the front plate of the vacuum system. The shutter extended between the crucible and the collector and could be operated through a Wilson Seal.

The BeO crucible was made by dampening BeO powder with approximately five drops of water and one drop of nitric acid per gram of powder. These proportions were not at all critical. The moistened powder was tamped into a mold of the correct length and outside diameter, but having a small core. The consistency of the paste was so chosen that the tamped crucible could be gently removed from the mold and placed in an oven to be fired at 1200°C. Then it was drilled out to a wall thickness of about 2 mm., using an ordinary drill turned by hand. After this, the crucible was fired to 1800°C or 2000°C. BeO is a very satisfactory crucible material because it does not react with Be and because it has a high thermal conductivity at high temperatures.

Be used was "pure extruded" machinable material.

The Production of Thin Be Foils (cont)

Procedure

Be which was broken into fragments approximately 3 mm. on a side was washed in clear acetone and placed in the BeO crucible. The collector plate was also washed with clean acetone, ordinarily followed by a drop of silicone oil which was rubbed off with tissue paper as thoroughly as possible. After this preparation, the collector plate was laid in place between the defining plate and the backing, and the vacuum system was pumped down to 5×10^{-5} mm. of Hg. or better. The crucible was heated for a few minutes with the shutter in place above it so as to shield the collector plate. Normal operating conditions were 85 amperes at 6 volts, which raised the Be to a temperature of approximately 1400°C. After the shutter was swung aside, the progress of evaporation was observed through the port. After one to five minutes of evaporation, a very noticeable change in the color of the collector plate accompanied the "first deposit" of the Be, and it was found that quite reproducible thicknesses could be obtained if the time of the evaporation after the first coat was kept the same. After approximately fifteen minutes of evaporation, the crucible heater was turned off, and when the crucible had cooled, the collector plate was removed from the vacuum.

The foils were loosened by flexing the collector concave toward the beryllium. Normally, flexing alone, plus a little persuasion by thumbnail flexing just outside the edges of the beryllium was sufficient to remove the foil. Sometimes a very thin stainless steel "pusher" was used.

At the working distance of 8", the Be beam was substantially uniform over a 4" diameter area and therefore seven one-inch diameter foils could be deposited at a time. In order still further to speed the foil production, a cube was made with collectors on four faces so that four times as many foils could be made with a single evacuation.

The physics of stripping foils and of their curl is not understood in detail. We operated on the theory that a differential expansion of the Be and the collector plate loosened the Be, providing this expansion was not large. If it were too large, the foils cracked. The curl was affected both by the average temperature of the collector and by the change of temperature of the collector during the process of deposition. Thus if the collector plate started out cold and ended up warm, the early deposit of Be was more dense than the later layers, causing the Be to curl from the collector when it cooled. Using the above ideas, it was in general possible to correct for curl of the foils by controlling the temperature of the collector. By adjustment of the collector distance and crucible temperature, it was possible to find a position in which preheating was not necessary.

Smooth "mill run" surfaces were as satisfactory as highly polished ones. Sheets with pronounced scratches or roll marks could not be used, for the foils tended to crack along such marks.

Data on representative runs without the preheater are given below:

The Production of Thin Be Foils (cont)

Distance to collector	8"	8"	8"	8"	8"
Material of collector	Stainless Steel	Cold Rolled Steel	Cold Rolled Steel	Monel	Monel
Pressure during run - (mm. of Hg.) x 10^{-5}	1.4	1.8	4	2.6	2.6
Voltage	6	6	6	6	6
Amps	85	85	85	85	85
Thermocouple temperature at start (C^o)	25	28	25	30	35
Thermocouple temperature at end (C^o)	92	107	92	112	102
Time until shutter was opened (minutes)	1-1/2	1-1/2	1-1/2	5	4
Time first deposit	6-1/4	6	6-1/2	8-1/4	7
Total running time after first deposit	8	8	3-1/2	21	17
Foil thickness (microinches)	24	21	9	60	50

Foils have been evaporated on monel, cold rolled steel, and stainless steel with good success. Nickel, copper, aluminum, and tin have been used for short runs, but their expansion coefficients are too large to permit their use for thick foils. Be was used as a collector for producing very thick foils from a few tenths of a mil to a few mils. Stripping is, however, a difficult problem with foils thicker than 200 microinches, since the Be cannot be flexed readily without cracking. Be was also used as a collector with a different type of crucible which radiated large amounts of heat and raised collector temperatures several hundred degrees.

The thinnest foils made by this evaporation process were 5.9×10^{-6} inches or approximately .02 mg. per square centimeter. All foils show small pinholes. Microscope and electron diffraction studies indicate that there is no preferred crystal orientation and that grain is very fine. Foils have not been annealed and consequently are very brittle.

A very few 3/4" diameter self-supporting foils approximately one per cent transmitting to visible light were made by evaporating Be onto thin "formvar" films and subsequently removing the formvar in a vapor phase degreaser. Plastic films can also be burned off, but it is not known how much deposit remains.

Mounting

A satisfactory technique of mounting thin foils is to moisten the surface on which they are to be held with a small amount of thinned collodion, and then to lower the surface onto the foil. Surface tension will pull the foil flat and smooth. One-inch diameter foils, which have curled up into a 1/8" diameter cylinder, have been caused to unroll and mount satisfactorily in this way.

The author wishes to express gratitude to Mr. Frank Grobelch and Mr. Richard Crawford who helped work out the methods for producing foils, and to Mr. Nelson Garden, who cooperated in the early phases of the problem. This work was done under the auspices of the Atomic Energy Commission under Contract No. W-7405-eng-48.

Hugh Bradner
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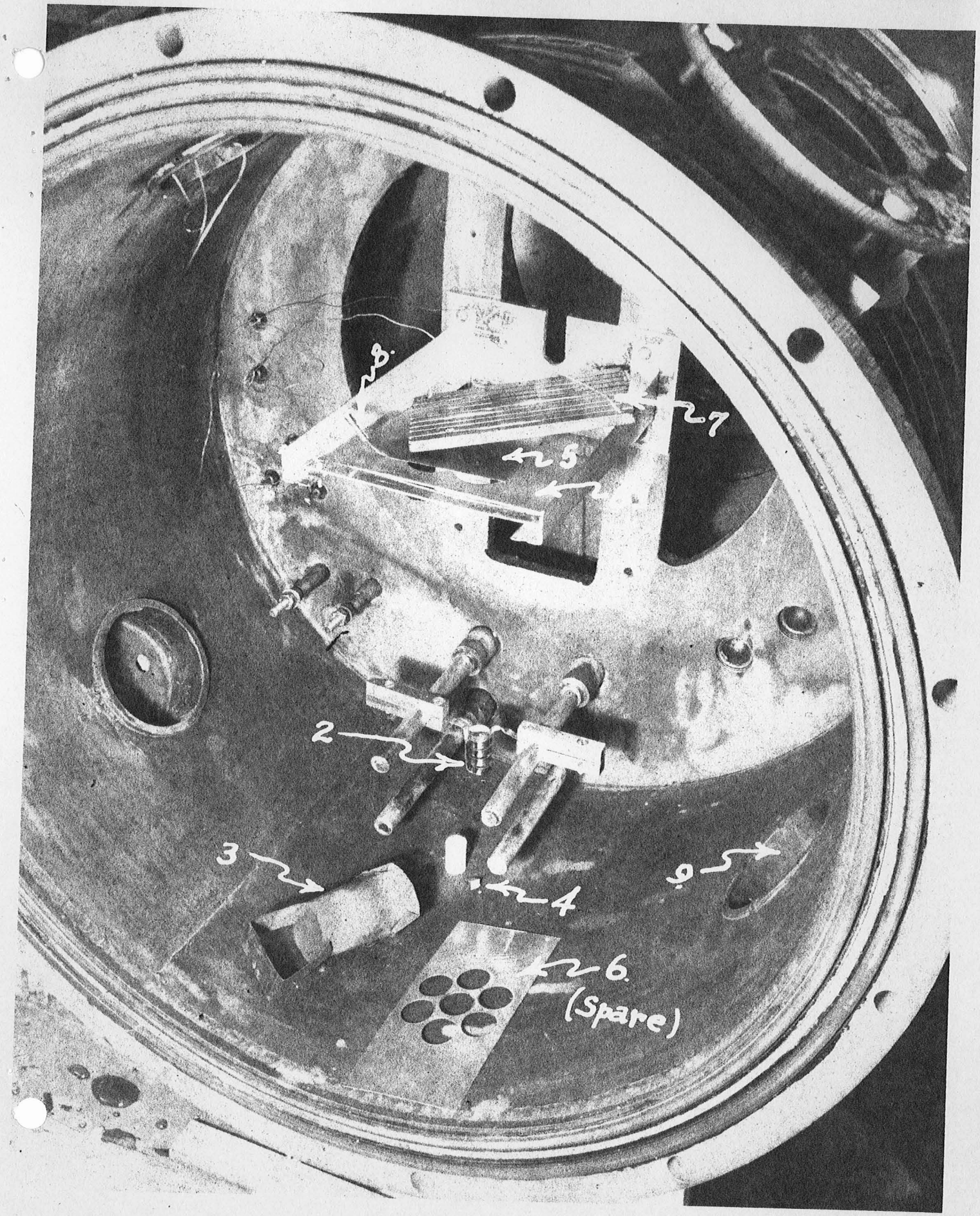
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