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NEW SHADOWING AND COATING APPARATUS (A-DECK) FOR ELECTRON MICROSCOPIC PREPARATION

by

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(with 3 Text-figures)

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The electron microscopic observation of geologic or paleontologic samples usually applies single or two stage replica technique. In either techniques, thin film of carbon and shadowing material is deposited by vacuum evaporation method.

The vacuum evaporation has been thought to be tedious by few reasons. First, high vacuum such as 10^{-5} to 10^{-7} torr. is not easy to obtain. Second, the evaporation of heavy metals for shadowing requires larger energy and the thickness or the rate of deposition of evaporating material is difficult to control. Few microscopists have been interested in finding the better method of the vacuum evaporation procedure for recent years.

The complex procedure to obtain high vacuum has been much improved by introducing over-sized diffusion pump system to save standby duration (HAY, W. W. 1965, personal communication), or to develop the full automatic control of an entire system such as reported by HONJO, MINOURA and KOYAMA (1968).

The recent development of solid state technology has encouraged the innovation of technique to evaporate the metals for the thin film study. Electron beam bombardment method has been successful to apply on shadowing of heavy metals, especially with high melting point or carbon coating technique either for two stage or single stage replica preparation.

The technique to control the thickness of deposited thin film of metals has been improved significantly. The electronic film thickness monitor or deposition rate monitor, has been used for the production of quality controlled thin film. The BRADLEY's formula (1961) of evaporation is also useful for routine works.

On the other hand, the mechanism to hold a specimen or plastic replica during the shadowing or carbon coating, has not been paid so much attention by microscopist. We have found the improvement of this mechanism promotes the efficiency of preparation.

In our "A deck" (the side view of the out-fit appears like character of A) system,

the entire mechanism is mounted on a stainless steel or brass frame of step-ladder shape. The sample to be treated is placed on the top facing to the bottom. A pair of carbon arc and a metal coating devices are mounted on the legs by sliding mechanism. The distance between the sample and sources of evaporation can be easily adjusted by sliding up and down the evaporation sources through the side frames. The specimen or the first stage replica to be shadowed and/or coated is mounted on a piece of slide glass or similar material and placed on the rotation and swiveling stage with a simple spring clipper.

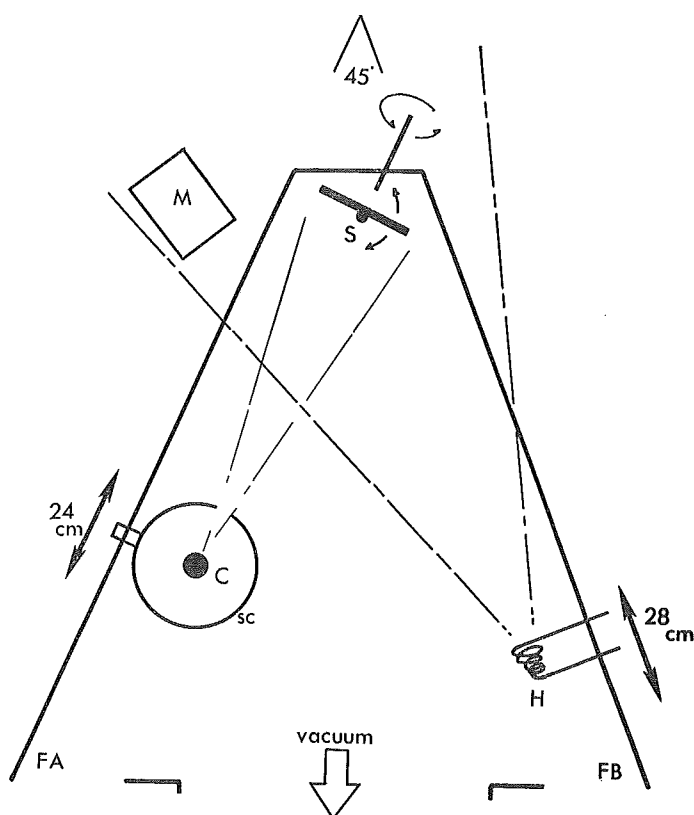


Fig. 1

The function of the A-deck system. The pair of carbon arc (C) in a shield case (sc) and the heater (H) for the evaporation of metal, can be moved up and downward along the frame FA and FB. The specimen stage (S) is swiveled as well as rotated to change coating and shadowing angle in two planes. The sensor-head (M) of the thickness monitor is placed facing to the heater, being protected from the radiation from the carbon arc by the shield case.

A pair of carbon arc is housed in a stainless steel shield case to avoid that the random radiation of carbon may coat unnecessary surface of the bell-jar. The ends of two spectroscopic grade carbon rods are shaped as are illustrated in Fig 2.

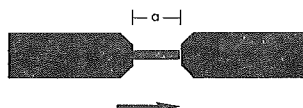


Fig. 2

When the thickness of the carbon rod at the portion *a* is 1.00 mm, the length is 4.5 mm, and the carbon throughout the portion A is completely evaporated, the following thickness of carbon film is obtained, Approximately 200 Å, when the distance between a sample and arc is 12 cm. Approximately 500 Å, when the distance is 7 cm.

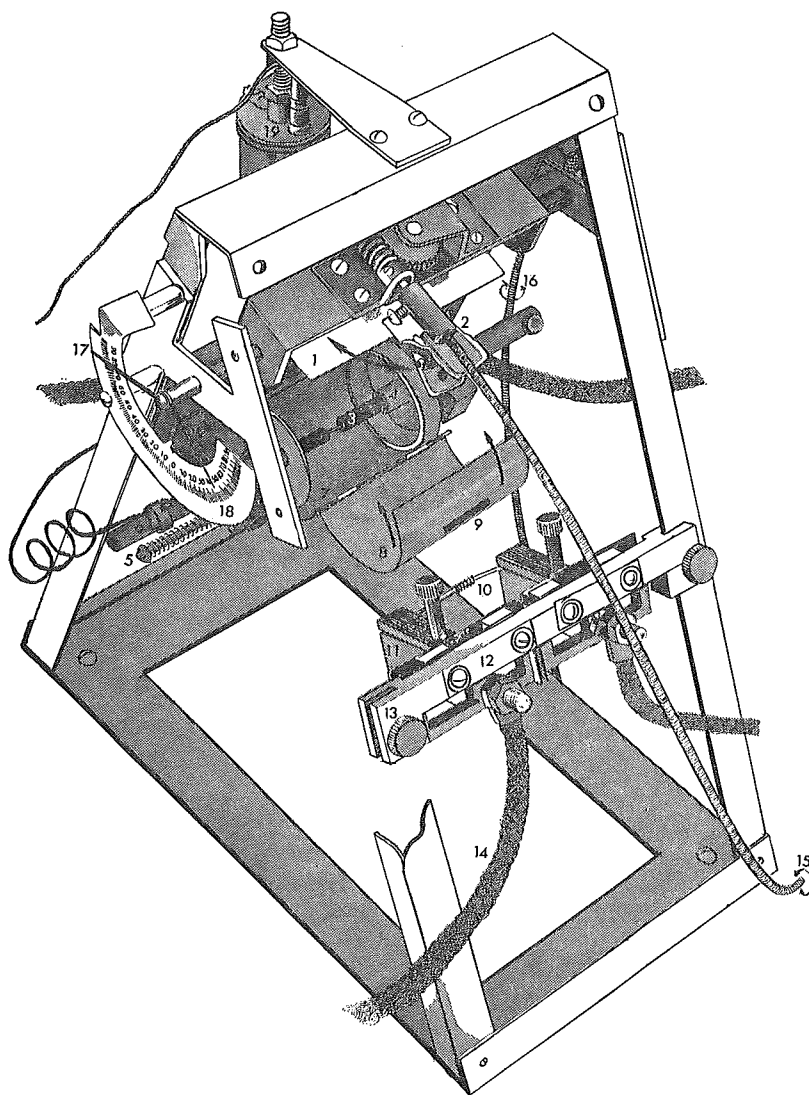
A table-top type lathe is used to shape carbon rods. The feeder mechanism of arc, especially the rod pushing spring is shield from the heat which is generated by the radiation. This practice encourages the reliability of operation.

Any common type of heater for metal evaporation can be accommodated on an A-deck. We use a tungsten wire basket for chromium shadowing. The platinum-paradium wire is evaporated from a tungsten rod (0.7 mm thick). A molybdenum or tungsten boat can be used if desired. The holder is insulated for the currency of 200 A at 30 V AC at the maximum. A small electron bombardment furnace is utilized for the evaporation of metals with very high melting point such as tantalum (Bachmann in Bradley, 1962).

The sample can be rotated around two axis. The sample holder is manipulated by flexible wire through vacuum feed-through mechanism (feed-thru's). Almost any combination of the shadowing angle and the angle of carbon coating can be produced. Especially the mechanism is convenient for the "portrait shadowing technique", by shifting the angle from one to the other on an plane during the shadowing practice. The film thickness monitor system (i.e. HONJO and OKADA, 1967) is useful to find the best contrast in the portrait shadowing technique. The "zimple coating" (coating from even direction) of heavy metal or carbon coating on a spheric sample can be practiced by rotating as well as swiveling the sample holder by rotating two axis manipulating from the outside of vacuum through the vacuum feed-thru's.

The dimension of the base of our A-deck is 26 cm by 15 cm, the height is 32 cm. It can be used in regular glass bell-jar with more than 14 inches of diameter. The bell-jar collar requires that at least two rotation feed-thru's are equipped.

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**Fig. 3**

1: A sample holder, a piece of slide glass is used for routine works. 2: A paper-clipper to hold the sample holder. 3: A pair of carbon for carbon film coating. 4: A carbon rod holder with clamps. 5: A pushing spring for arc feeding. 6: An auxiliary leading wire for the pushing-rod. 7: A shield case. 8: The lid for the shield case. 9: A slit to let carbon vapor out. 10: A heater for metal evaporation. 11: Heater clamps. 12: Insulators, 13: Clamp holders for the heater mechanism. 14: Leading wire for the heater. 16,17: Flexible wire for the tilting and rotation of sample holder. 17: A needle indicating the angle of carbon radiation against the sample. 18: A needle indicating the shadowing angle. 19: A sensor head of film thickness monitor.

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