V. PROCEDURE

As noted in Section IV, two types of experiments were conducted in this study, deposition and vaporization. Also, in checking for contamination in the germanium film x-ray diffraction and electron microprobe analytical techniques were utilized. The procedure for deposition, vaporization, x-ray diffraction, and electron microprobe runs will be presented in that order.

A. Procedure for Deposition Runs

Deposition runs were necessary to produce germanium coated substrates. Vaporization rate data is a by-product of deposition runs.

The first step in a deposition run was to chemically clean the molybdenum substrate in a 50% HCl solution at 180°F for 1.5 minutes. Graphite substrates were not cleaned. Then, the substrate and the germanium sample (plus crucibles) were weighed. The equipment on the experimental stand (3-1) was arranged as shown in Figs. 8 and 12, except that the deposition shutter (8-3) was positioned between the germanium sample (4-6) and the substrate (5-2), not as shown in Fig. 12. The glass part of the inside mirror (8-4) was replaced. Port holes No. 4 and 5 of the experimental shield (11-1) were then closed off and the shield lowered over the experimental stand, as picture in Fig. 13.

The start up procedure was then begun. The bell jar (3-2) was lowered to the feedthrough ring (3-4) and the system evacuated to about 10^{-5} torr. The electron beam heater cooling water, the substrate preheater, and the electromagnet current were then turned on. At this point the electron beam heater was turned on and the electromagnet

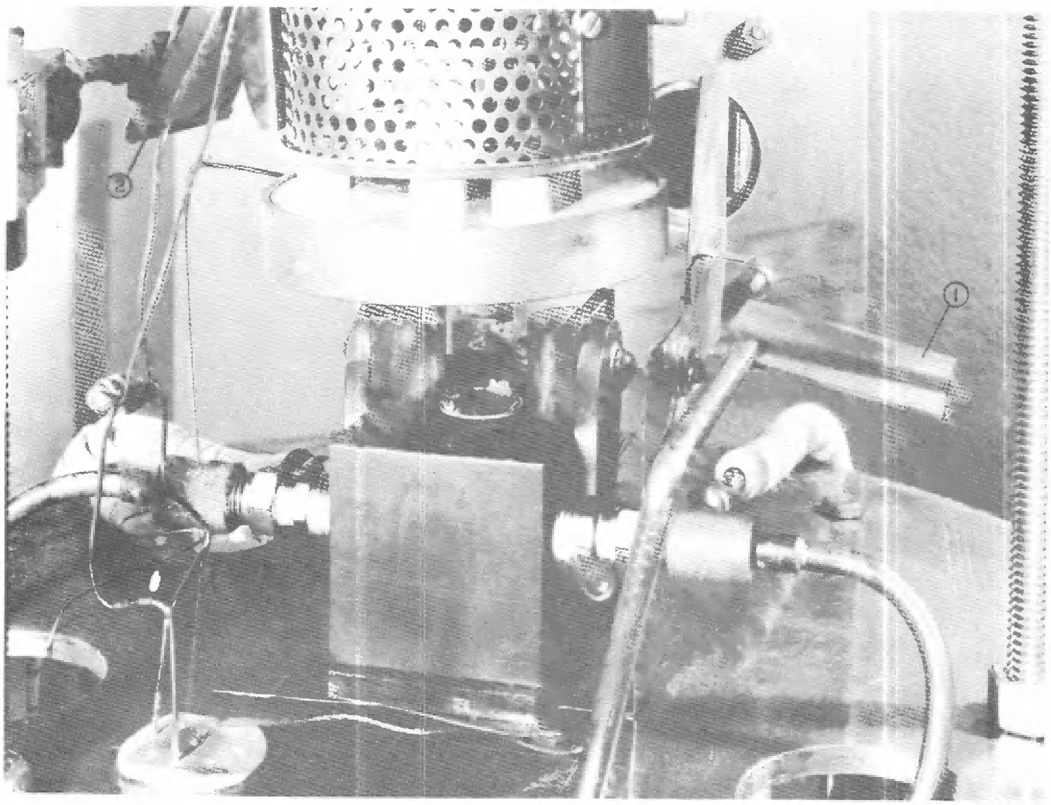

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Fig. 12. Experimental stand, arranged for a deposition run (close up).

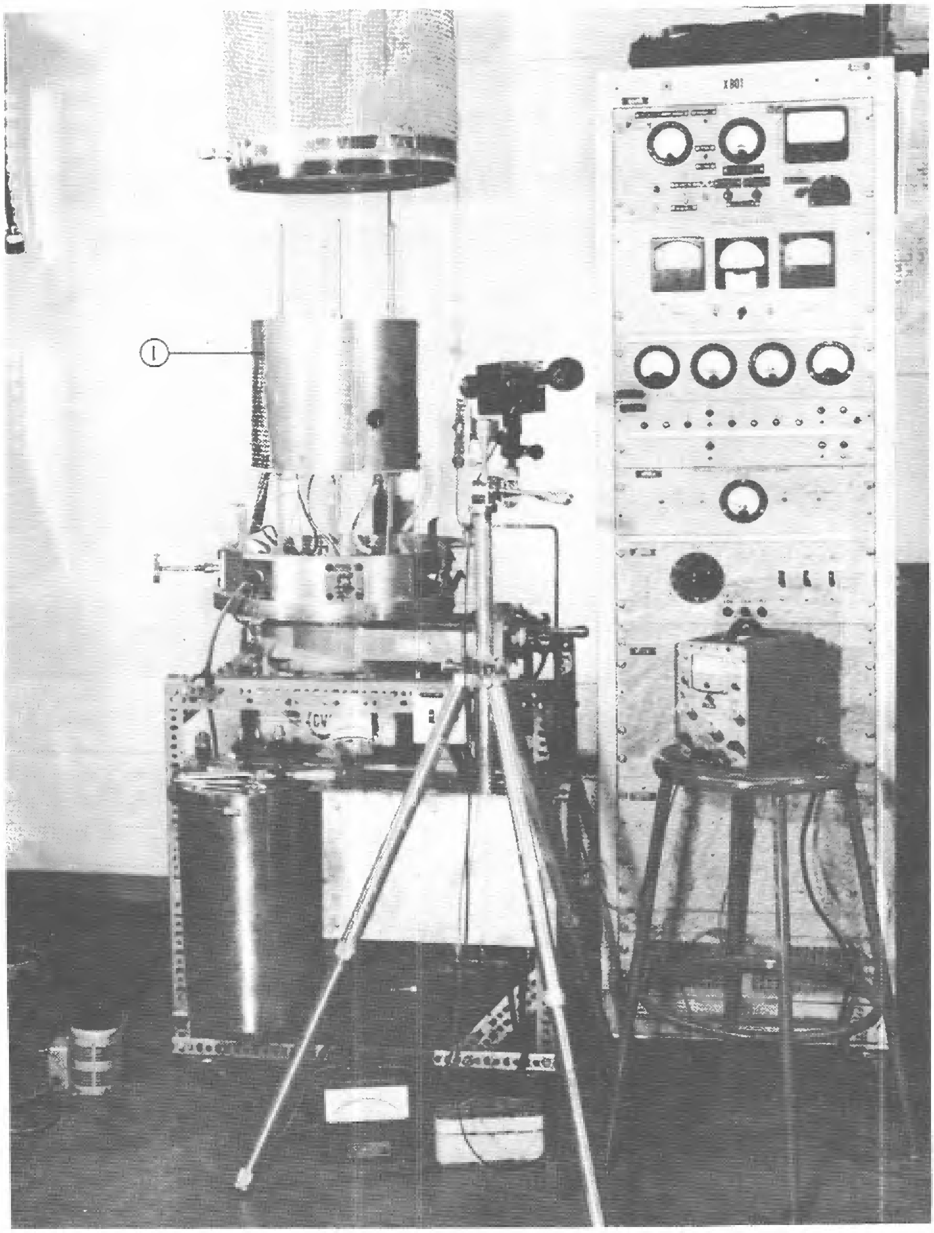

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Fig. 13. Experimental system, ready for deposition run.

current adjusted to guide the beam into the germanium sample. When the germanium sample and the substrate reached the desired temperatures the deposition shutter (12-1) was moved to the position shown in Fig. 12 to allow vaporizing germanium to plate the surface of the preheated substrate.

During the run the germanium sample could be viewed with the aid of the inside mirror through viewing port No. 2 (11-2) and the pyrometer was positioned, as in Fig. 13, to determine the temperature of the germanium sample.

When it was felt that the film was thick enough, the deposition shutter was closed and the system shut down. When shut down was completed, the coated substrate and the germanium sample plus crucibles were removed and weighed.

B. Procedure for Vaporization Runs

In vaporization runs, a coated substrate was heated to about 1000°C. The vaporization rate of germanium was measured and the surface integrity of the liquid germanium film was noted.

The first step in a vaporization run was to weigh the coated substrate. Figure 9 shows the experimental stand arranged for a vaporization run. Once the equipment was arranged in this manner and the glass part of the inside mirror replaced, viewing ports 4 (11-4) and 5 (11-5) of the experimental shield were opened and the shield lowered over the experimental stand. The bell jar was lowered and the vacuum chamber evacuated.

Then the cooling water, the electromagnet, and the electron beam heater were turned on. The electromagnetic current was adjusted until the electrons deflected by the magnetic field struck the back of the coated substrate.

By use of the pyrometer, positioned as shown in Fig. 3, the apparent film temperature was recorded through viewing port 5. The temperature of the black body hole on the back of the substrate was measured through viewing port 4, with the aid of the outside mirror (9-4). The observed black body temperature in combination with the apparent temperature of the liquid germanium film, was used to determine the emmissivity of liquid germanium.

To check the surface integrity of the film, the germanium surface was viewed through viewing port 3, with the aid of the inside mirror.

Following shut down the coated substrate was removed from the system and weighed to determine the weight loss.

C. Procedures for X-ray Diffraction and Electron Microprobe Techniques

To determine the degree of contamination of the germanium, X-ray diffraction and the electron microprobe techniques were employed. Both of these processes were carried out at the University of California, Lawrence Radiation Laboratory in Berkeley.

In the x-ray diffraction measurements, x-rays were directed upon the surface of the germanium film. By measuring the angle and intensity at which the x-rays were diffracted, the chemical compounds in the film were identified.

After x-ray diffraction the coated substrate was prepared for the electron microprobe. First, the substrate was cut in half, from front to back, with a spark cutter. One half was mounted in a bakelite holder, as shown in Fig. 14, and then polished. The holder was placed into the electron microprobe and the elementary composition (amount of various elements) determined as a function of position along lines A, B, C, and D of Fig. 14. See Section III or Heinrich[9] for a discussion of the operation of the electron microprobe.

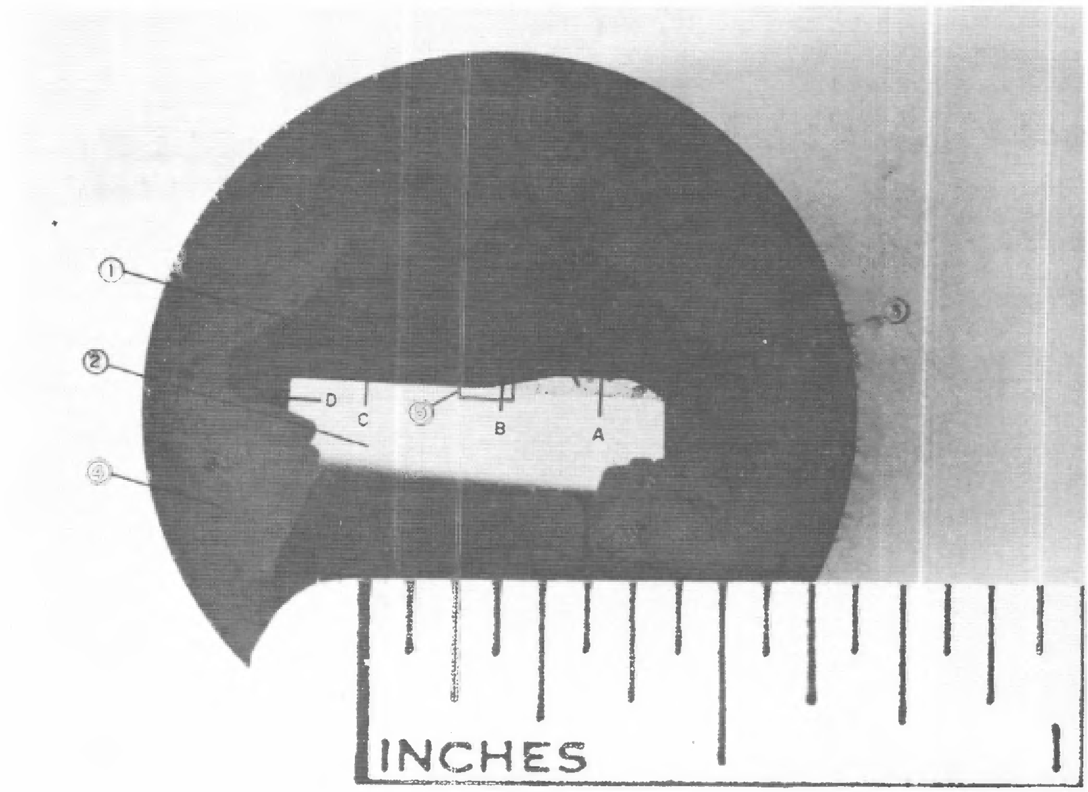

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Fig. 14. Cross section of coated substrate, mounted for electron microprobe.

DESCRIPTION OF FIGURE 14

Object Number	Name of Piece of Equipment	Function and Specifications of Piece of Equipment
(14-1)	Bakelite mount	To hold the spark cut piece of coated substrate for electron microprobe analysis
(14-2)	Substrate cross-section (spark cut in half from front to back). (The front of the substrate is facing up and the back facing down).	
(14-3)	Germanium film cross-section (after germanium droplet was formed during vaporization run)	
(14-4)	Carbon film	Was spread over surfaces of bakelite and part of substrate cross-section to conduct electrons away from the coated substrate during electron microprobe analysis.
(14-5)	Photomicrograph area	This area is pictured in the photomicrograph, Fig. 18.
(14-A, B, C, D)	Probe lines	Lines along which electron microprobe measurements were taken.