THE UNIVERSITY OF MICHIGAN

SOLID-STATE MATERIALS AND DEVICES

SEMIANNUAL PROGRESS REPORT NO. 3

Period Covering June 1, 1964 to December 1, 1964

Electron Physics Laboratory

Department of Electrical Engineering

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Tecumseh Products Company
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ABSTRACT

Effort during the current period has been directed toward establishing and operating facilities for the production of thin films by sputtering and by high vacuum evaporation. A number of sputtered copper films have been prepared to define the relation between sputtering parameters and film characteristics. In addition a good deal of work has been done to eliminate small but significant amounts of atmospheric contamination from the sputtering process. Most of the hardware required for the vacuum evaporated film studies was fabricated during the report period and a few films of silicon monoxide and of copper have been prepared.

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PERSONNEL

Scientific and Engineering Personnel		Time Worked in Man Months*
J. Rowe	Professor of Electrical Engineering	.45
M. Miller	Associate Professor of Electrical Engineering	1.82
V. Vis	Research Engineer	4.46
W. Rensel	Assistant Research Engineer	.88
M. Lubbers	Assistant in Research	2.55
Service Personnel		15.21

* Time Worked is based on 172 hours per month.

SEMIANNUAL PROGRESS REPORT NO. 3

FOR

SOLID-STATE MATERIALS AND DEVICES

1. Introduction (J. E. Rowe)

The purpose of this research program under the sponsorship of the Tecumseh Products Company is to assist that company in the establishment of a solid-state research facility in Ann Arbor and to carry out supporting solid-state research in the Electron Physics Laboratory at The University of Michigan. It has been decided to summarize the research findings at The University of Michigan in semiannual reports. This constitutes the third semiannual progress report on the research activity in the Electron Physics Laboratory.

In view of the ever increasing importance of microelectronics and integrated circuitry in the electronics industry and the prominent role of thin films and thin film techniques in the fabrication of devices it has been decided to develop a comprehensive research program on thin films to include metallic, semiconducting and insulating films. The following sections of this report outline the work done during the last six months on the subject program.

2. Work Accomplished to Date (V. A. Vis)

In accordance with the general program set forth in Semiannual Progress Report No. 1, effort during the current period has been directed toward establishing and operating facilities for the production of thin films by sputtering and by high vacuum evaporation. A number of sputtered copper films have been prepared to define the relation between sputtering

parameters and film characteristics. In addition a good deal of work has been done to eliminate small but significant amounts of atmospheric contamination from the sputtering process. Most of the hardware required for the vacuum evaporated film studies was fabricated during the report period and a few films of silicon monoxide and of copper have been prepared. This work has been delayed by the necessity of returning part of the vacuum equipment to the manufacturer to repair a leak which developed after a few months service. These matters are discussed more fully in the remainder of the report.

2.1 High-Vacuum Work. Several sequential evaporations of silicon monoxide and copper were carried out to verify the performance of the sources, shields, shutter and substrate heater. During each evaporation the vacuum was maintained at 4×10^{-8} torr is the titanium getter surface was cooled by liquid nitrogen. During October the nitrogen cooled shroud developed a leak which was so inaccessible that the entire base plate assembly was returned to the Ultek Corporation for repairs. During the report period design and fabrication of the mask changing gear was substantially completed though it has not yet been put into service. The mask changer and substrate heater are shown in Fig. 2.1. The substrate heater is a flat, resistance-heated molybdenum strip located between the substrate (a 3" x 1" glass slide) and an identical glass slide in which a thermocouple is embedded. Below the substrate is a paddle-shaped member which carries four spring-loaded mask supports and is rotatable about two perpendicular axes to bring any of the four masks into contact with the bottom of the substrate. Fabrication of masks from thin copper or stainless steel sheet by photoresist and etching techniques is under way.

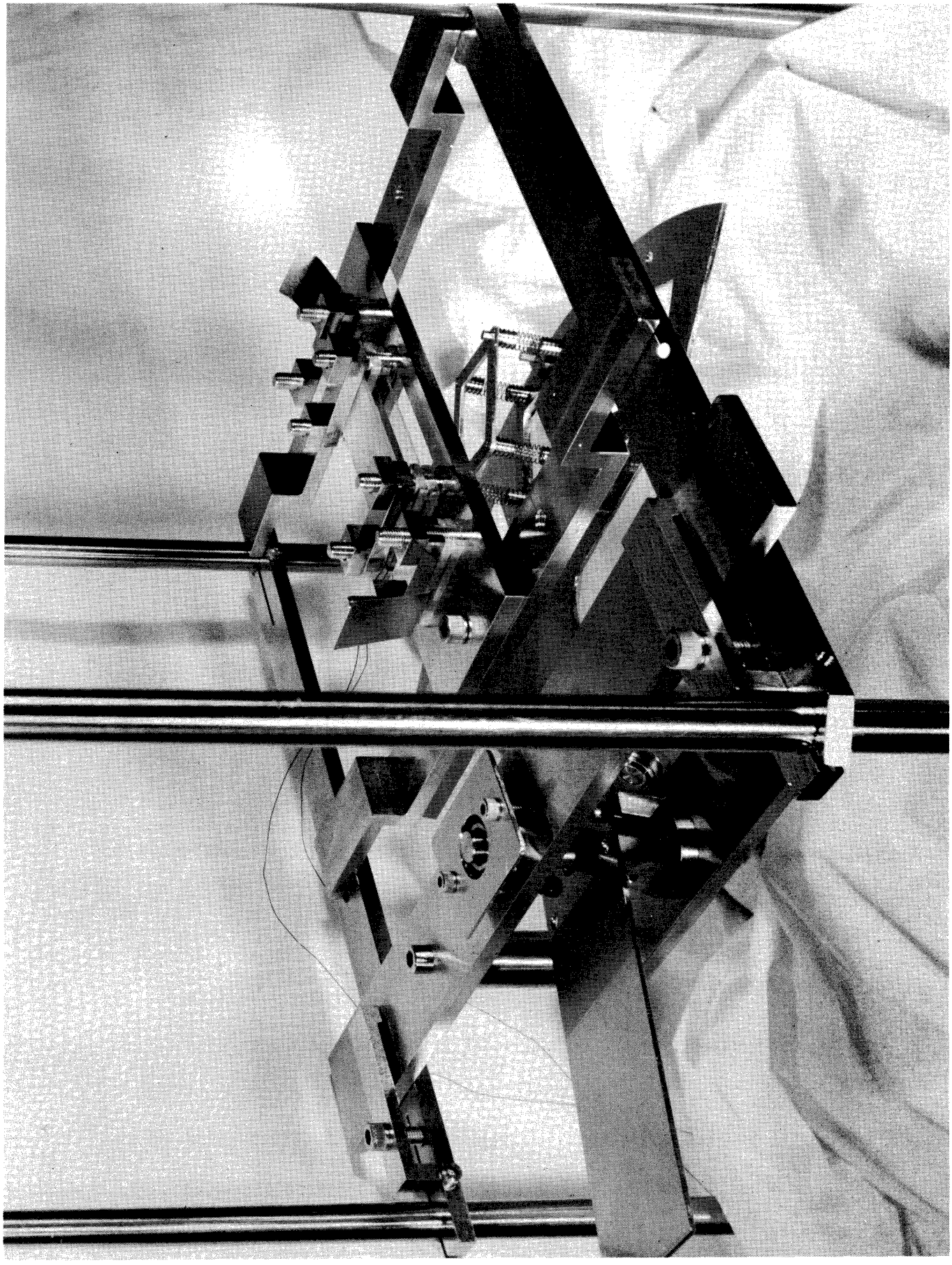


FIG. 2.1 SUBSTRATE HOLDER AND MASK CHANGER.

A Brush Model 280 two-channel recorder has been received and found to operate within the guaranteed 0.25 percent linearity of response after readjustment at the factory. The selection of a mass spectrometer (residual gas analyzer) has been delayed by the lack of opportunity to test various equipments on the Ultek machine.

2.2 Sputtered Film Work. The sputtering apparatus described in the preceding report has been used throughout the present report period to produce copper films on Pyrex substrates. A series of experiments was made to determine the dependence of film thickness (determined by chemical analysis) on sputtering current and voltage. The result is shown in Fig. 2.2. The relationship between the film thickness and transmission for three kinds of illumination is shown in Fig. 2.3. Transmission through copper films is a maximum at approximately 0.550 micron and transmission measurements at this wavelength can evidently be used as a nondestructive method of thickness determination up to 1000 Å. The slope of the logarithmic plot for 0.550 micron in Fig. 2.3 implies an extinction coefficient $k = 2.56$ (where k is the imaginary part of the index of refraction) which agrees fairly well with a published value of $k = 2.46$[1] obtained from evaporated films. (Parts of Figs. 2.2 and 2.3 appeared in the preceding report. They are repeated here for the sake of completeness.)

In addition to transmission measurements, the electrical (sheet) resistivity of the films is routinely measured as soon as the film is removed from the sputtering chamber. The four-point probe apparatus used is shown in Fig. 2.4. The four spring-loaded probes are tipped with platinum spheres which make contact with the film. The resistance of very thin, slowly deposited films is particularly sensitive to the concentration

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1. Hass, G., AIP Handbook, Second Edition, McGraw-Hill Book Co., New York, N. Y., p. 6-109; 1963.

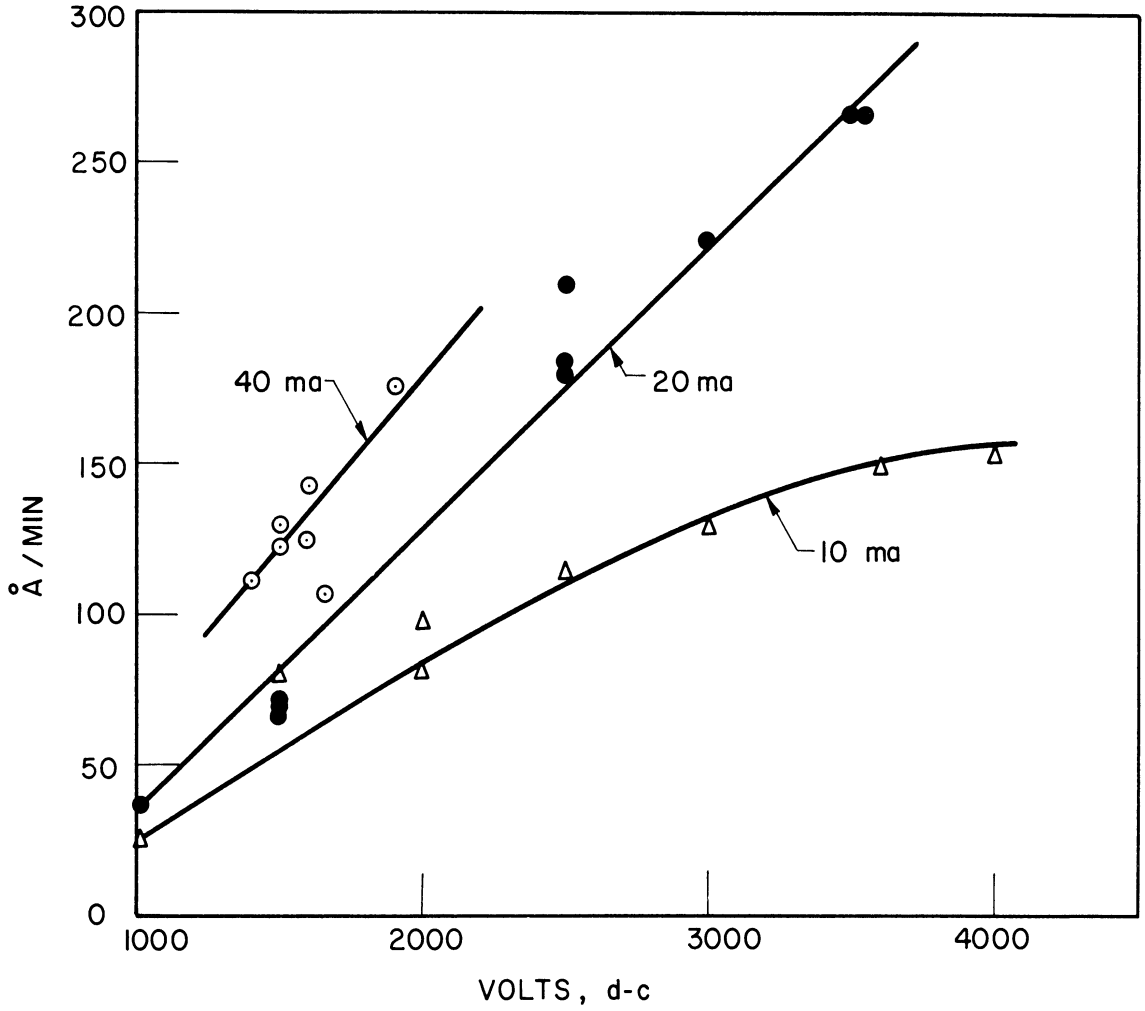


FIG. 2.2 DEPOSITION RATE VS. CURRENT AND VOLTAGE FOR SPUTTERED COPPER.

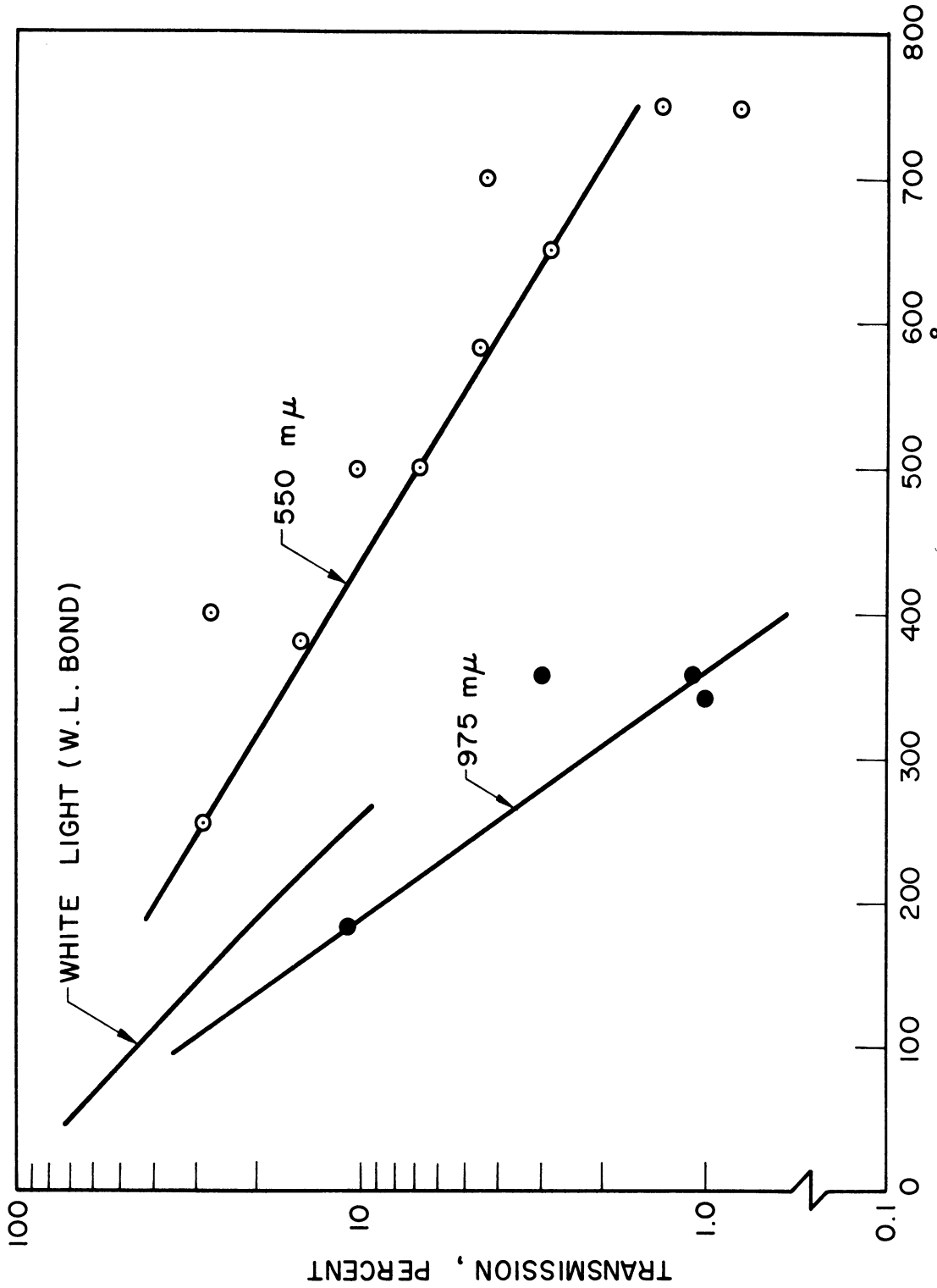


FIG. 2.3 TRANSMISSION VS. THICKNESS FOR SPUTTERED COPPER.

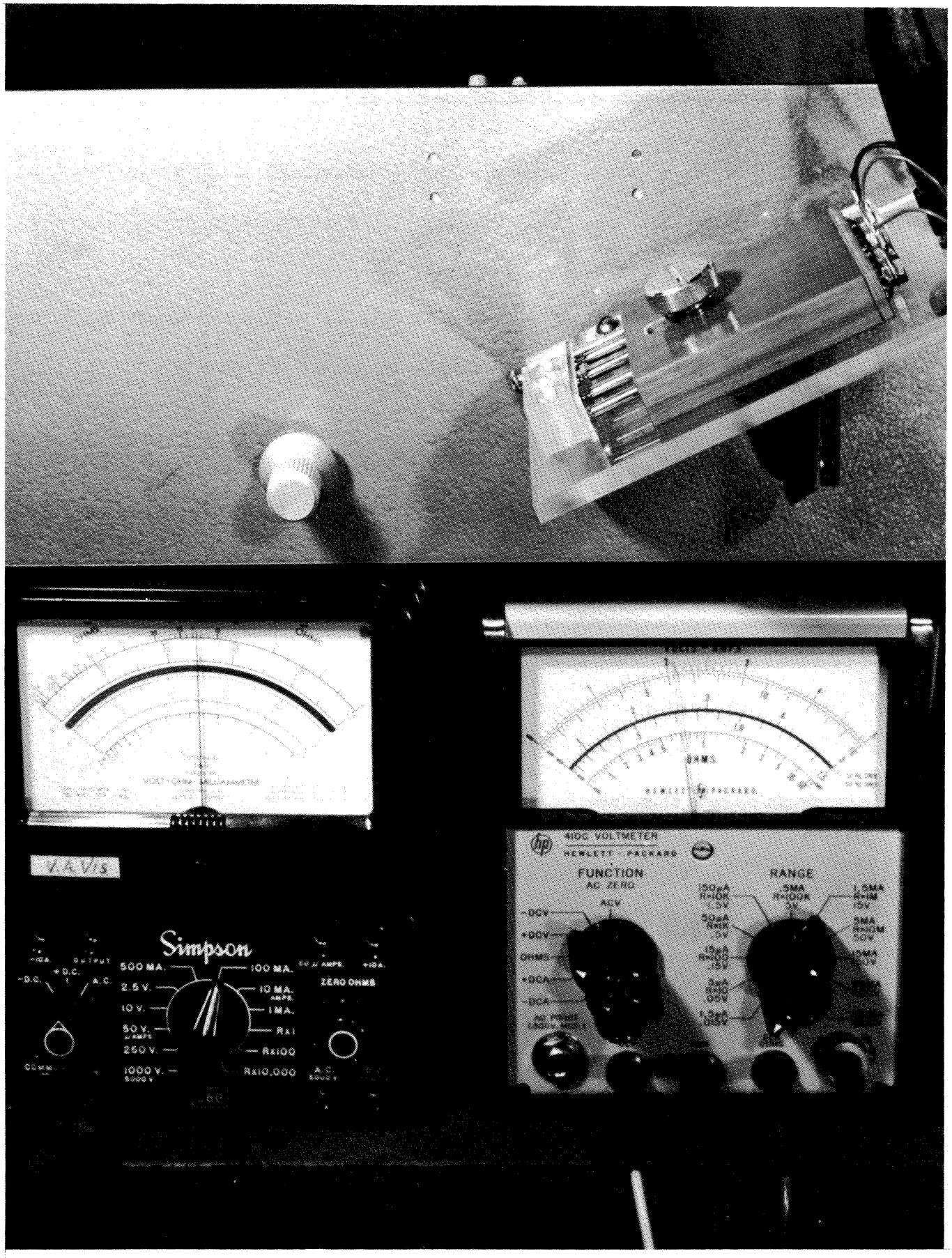


FIG. 2.4 FOUR-POINT PROBE APPARATUS.

of oxygen in the sputtering gas. For example, films made by sputtering at 1000 v and 10 ma for five minutes are 125 Å thick according to Fig. 2.2. Some films prepared under these conditions had a blue color and sheet resistivity greater than 10^9 ohms/square, while others had a "copper" color and resistivities as low as 5.5 ohms/square. A number of such films were prepared to improve operating procedures.

The lowest resistivity measured to date is four times larger than the bulk value and 1.5 times larger than the value corresponding to the Sondheimer-Fuchs relation[2] which takes into account the scattering of electrons by the surface. It is not known whether this difference is due to factors such as roughness or to the presence of significant amounts of oxygen or other contaminant.

The symposia held by the American Vacuum Society and by the Thin Film Division of the AVS September 19 to October 2, 1964, in Chicago were attended by V. A. Vis. A report of these meetings is on file in the Electron Physics Laboratory.

3. Plans for Future Work (V. A. Vis)

The mask changer will be completed and put into service. Films of copper will be deposited on glass and on silicon monoxide surfaces. The sheet resistivity of the films will be monitored during and after deposition.

The sputtering system will be operated with an additional gettering chamber to purify the argon before it enters the sputtering chamber. When it appears that no further significant improvement in the purity of the films is possible attention will be directed to materials other than copper.

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2. Kittel, C., Introduction to Solid State Physics, Second Edition, John Wiley and Sons, New York, N. Y., p. 239; 1957.

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