THE UNIVERSITY OF MICHIGAN

SOLID-STATE MATERIALS AND DEVICES

SEMIANNUAL PROGRESS REPORT NO. 1

Period Covering June 1, 1963 to December 1, 1963

Electron Physics Laboratory

Department of Electrical Engineering

By: J. E. Rowe

V. A. Vis

Approved by: J. E. Rowe

J. E. Rowe, Director
Electron Physics Laboratory

Project 05733

Tecumseh Products Company
Tecumseh, Michigan

February, 1964

ABSTRACT

A basic and applied research program on thin films has been initiated with the objective of developing the fundamental technology of film production. Simultaneously measurement schemes are being evaluated for the determination of both physical and electrical properties. A longer range objective involves the use of these films to perform device functions in electronic systems.

TABLE OF CONTENTS

PERSONNEL

Scientific and Engineering Personnel		Time Worked in Man Months*
J. Rowe	Professor of Electrical Engineering	1.16
C. Yeh	Associate Professor of Electrical Engineering	.20
V. Vis	Research Engineer	2.55
W. Rensel	Assistant in Research	.83
J. King	Research Assistant	2.74
Service Personnel		10.79

* Time Worked is Based on 172 hours per month.

SEMIANNUAL PROGRESS REPORT NO. 1

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 first such report.

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 to date on this program.

2. Program and Objectives (V. A. Vis)

During the report period a thin film program was set up and investigation was begun. The objectives of the work are:

a. To develop the technology of making thin films,

b. To measure relevant properties of thin films,

c. To utilize thin film properties to perform device functions.

The variety of methods and materials for preparing thin films is enormous and the general philosophy will be to seek a thorough understanding of a small part of the range rather than to attempt a broad coverage which would necessarily be more superficial. If the initial choice is made wisely the results will be readily applicable to other circumstances.

It is planned to confine attention chiefly to films formed by vacuum evaporation-condensation or by sputtering in a gaseous atmosphere. One or two examples of each of the main categories (metal, semiconductor, or insulator) will be selected; initially copper, germanium, and silicon monoxide. In addition to the nature of the film-forming material the other main variables are:

a. The nature of the substrate upon which the film is formed. We will use soft glass, Pyrex, rock salt or any of these covered by silicon monoxide.

b. The pressure and composition of the gaseous ambient.

c. The rate of deposition.

d. The substrate temperature.

e. The presence of a "foreign" nucleating agent on the substrate. Small amounts of a material which is more tightly bound to the substrate than is the film material have a profound effect on the early stages of film formation.

f. The degree of ionization of the impinging material.

The various measures of film properties may be divided into two categories, those which are applicable to the early stages when the substrate is less than 100 percent covered and those which are applicable to a continuous film. In the first category are weight per unit

area, electrical (sheet) resistance, reflection and transmission of visible light, x-ray diffraction, magnetic properties and electron microscopy of replicas and of the stripped film. Similar measurements may be made on continuous films and in addition such properties as tensile strength, magnetostriction, and "strain gage" behavior. It is expected that some films will be single crystal but attention will not be confined to this variety. We plan to investigate multilayer films formed by the alternate deposition of two or more materials. Such a sandwich could be built up to a thickness which would permit easy manipulation of the unsupported film. The sandwich arrangement also has the advantage that the inner layers are prevented from reacting with the atmosphere.

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

Effort to date has consisted of searching the literature, carrying out some preliminary experiments, and purchasing or building equipment needed for further work. All relevant articles have been read and abstracted in The Journal of Applied Physics from 1960 to the present and in The Transactions of the American Vacuum Society from 1961 to the present. Less extensive reading of other journals, including The Review of Scientific Instruments, has been done. In addition Walter B. Rensel and Vincent A. Vis attended the Tenth National Symposium of The American Vacuum Society in Boston, October 16-18, 1963, where they heard accounts of current work on thin films and saw a wide variety of related equipment on display. One may safely conclude that although there is a considerable body of current and past work in this field only a small part of the area has been adequately investigated. This is

particularly true since much of the reported work was done in atmospheres of uncontrolled composition and pressures in the neighborhood of 10^{-5} Torr. It is now well known that such atmospheres have an important bearing of the characteristics of the resulting film in most cases.

The experimental work done during the report period has consisted chiefly in preparing films of several types in order to develop the detailed technology. Substrates of soft glass, Pyrex, and fused silica have been used and various cleaning methods evaluated including chemical, detergent, vapor degreasing, fire polishing, and glow discharge. Sputtered films of platinum, Inconel, and copper and evaporated films of NIORO, nickel, Nickrome, and Cu have been prepared. Electron micrographs of replicas of substrate and film surfaces have been made. The electrical conductance of some of the copper films was monitored during deposition. A successful ohmic contact to single crystal cadmium sulfide was made by an evaporated indium coating. A vapor source of general utility which will permit reproducible control of evaporation rate is being developed and a colorimetric chemical determination of the mass of deposited metal is being set up for use in cases where the weight is too small to be accurately measured by an analytical balance.

Film preparation to date has been done in a diffusion pumped system capable of maintaining pressures in the 10^{-7} Torr. range during deposition. It is known that gases such as oxygen and water at this pressure react with many metals and alter the properties of thin films. In order to eliminate this effect lower pressures, on the order of 10^{-9} Torr., are required and equipment to produce such a vacuum has been ordered. After investigation of the currently available types

a sputter-ionization system with supplementary gettering, manufactured by The Ultek Corporation, Palo Alto, was chosen. Delivery is expected by the end of 1963.

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

After the new vacuum machine is put in operation various accessories will be developed such as sources, shutters, masks, and temperature-controlled substrate support. Once these items are operating properly the program of investigation outlined above can begin.

Biographical Data

Rowe, Joseph E. Professor of Electrical Engineering

Education: The University of Michigan: B.S.E. (EE), 1951; B.S.E. (Math.), 1951; M.S.E. (EE), 1952; Ph.D. (EE), 1955.

Employment:

Academic

The University of Michigan: Teaching Fellow, 1949-51; Lecturer, 1952-55; Assistant Professor, 1955-57; Associate Professor, 1957-60; Professor, 1960-.

Other Professional

The University of Michigan, Office of Research Administration: Research Assistant, 1951-53; Research Associate, 1953-55; Project Director, Electron Physics Laboratory, 1955-58; Director, Electron Physics Laboratory, 1958-.

Experience: Microwave circuitry and measurements, microwave electron devices, radar circuitry, high-vacuum tubes, masers and plasmas, countermeasures, low-noise theory.

Professional and Honorary Societies: American Physical Society; Institute of Electrical and Electronics Engineers; Society for Industrial and Applied Mathematics; American Mathematical Association; Eta Kappa Nu; Phi Kappa Phi; Tau Beta Pi; Sigma Xi; Science Research Club, The University of Michigan.

Listed in: American Men of Science; Who's Who in the Midwest.

Biographical Data

Vis, Vincent A. — Research Engineer

Education: The University of Michigan: B.S. (CE), 1941; B.S. (Math.), 1941; California Institute of Technology: M.S. (EE), 1948; The University of Michigan: Ph.D. (EE), 1963.

Employment:

Other Professional

DuPont Laboratories: Chemical Engineer, 1941-46; The University of Michigan, Office of Research Administration: Electronic Technician, 1950-54; Research Assistant, 1954-55; Research Associate, 1955-57; Associate Research Engineer, 1957-63; Research Engineer, 1963-.

Experience: Five years as chemical engineer with DuPont engaged in research and development of pilot plant and full scale high-pressure, organic synthesis. Two years as technician engaged in the construction of a large-scale analog computer and the design of a small digital computer. Four years research in electro-physiology on nerve transmission and allied topics both in life and in vitro. Eight years in infrared technology including four years investigation of non-scanning, image-forming systems utilizing temperature sensitive materials and four years experimental and theoretical study of the steady state and transient photoconductive behavior of single-crystal tellurium and the random fluctuations of its electrial conductivity. Thin film theory and technology.

Professional and Honorary Societies: American Physical Society; Optical Society of America; American Vacuum Society, Thin Film Division; Phi Eta Sigma; Sigma Xi; Science Research Club, The University of Michigan.