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**DEVELOPMENT OF PASSIVE ELECTRONIC COMPONENTS
FOR INSTRUMENTATION OF IMPROVED GEOTHERMAL
LOGGING TOOLS AND COMPONENTS**

Semiannual Progress Report. Report No. 1

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Arizona University
Tucson, Arizona

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
Division of Geothermal Energy

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LOGGING TOOLS AND COMPONENTS

ERDA Contract No. E(11-1)-4081

Semi-Annual Progress Report

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ABSTRACT

The logging of geothermal wells requires instrumentation that can survive at temperatures up to 500°C. The instrumentation can be cooled using an external source for a limited time, or the instrumentation can be designed using active and passive electronic components that will operate at these temperatures without degradation.

The active components are receiving attention from several research groups; however, passive components such as resistors and capacitors, with 500°C temperature specifications, are currently unavailable and must be developed.

The primary goal of this investigation is to produce passive components, resistors, capacitors, metallization, and passivation, that will withstand temperatures to 500°C and have extremely low temperature coefficients. The lower the temperature coefficients, the more exacting the instrumentation designs can be without elaborate compensation techniques.

The method of fabricating the thin film components is that of chemical vapor deposition (CVD) which is a major deviation from the standard approach. The films are grown in a CVD reactor by passing various reactant gases over a heated substrate.

The reactor was modified in order to accommodate the gases needed in the deposition of metals. The reactor can be operated over a temperature range of 400°C to >1200°C, and at any pressure between atmospheric and 1×10^{-3} torr.

Tungsten, tungsten-silicon, and silicon nitride have been successfully deposited on oxidized silicon wafers. The tungsten is used for interconnects and capacitor plates, tungsten-silicon is used as a high resistivity material for resistors, and silicon nitride is used as a dielectric for the capacitors and as a passivation layer. The materials are currently being studied in terms of their deposition parameters and electrical characteristics.

INTRODUCTION

The design of instrumentation for geothermal well logging requires high temperature active and passive components. The components will be operated at temperatures up to 500°C without external cooling. The active and passive components must also be mutually compatible.

The active components currently under investigation are integrated thermionic devices and solid state devices. Both types of active devices utilize microelectronic fabrication processes. Designing the passive components to fully utilize available microelectronic techniques will insure maximum compatibility between active and passive components, and it will also provide for high density electronic instrumentation.

The primary goal of this investigation is to produce passive components, resistors, capacitors, metallization, and passivation, that will withstand temperatures up to 500°C and have extremely low temperature coefficients. The lower the temperature coefficients are, the more exacting the instrumentation designs can be without elaborate compensation techniques.

The initial phase of the program covers the development and characterization of high temperature resistors, capacitors, interconnects, and passivation. The second phase will be spent optimizing the processing and integrating the passive components into simple networks to demonstrate the compatibility of all of the components. The method of fabricating the thin film passive components is that of chemical vapor deposition (CVD) which is a major deviation from the standard methods, sputtering and thermal evaporation. The desired materials are grown on a substrate by passing various reactant gases over the heated substrates. The passive components require the depositions of metals, dielectrics, and composite materials.

The method of depositing the material by CVD provides distinct processing advantages that are very necessary for the realization of high stability, high temperature components, and at the same time provide a large degree of design flexibility.

PROGRAM TASKS AND RESULTS

The program development and milestone chart is shown in Table 1. The various major tasks are listed along with the time schedule for their completion.

The first three months of the program were completely occupied with the reactor modifications and process preparations. During the second three month period, the following tasks were started:

1. Materials Development and Analysis
2. Component Development
3. Process Optimization

Reactor Modifications

The modified chemical vapor deposition reactor is shown in Fig. 1. The modifications were completed in December, 1976. Figure 2 is an identification of the various control panels and sections of the reactor.

The reactor was designed to be extremely flexible in terms of the process parameters and the materials that can be deposited.

The gas control panel has a total of ten rotometer type flowmeters for use with the carrier gases as well as the reactant gases. The controls for the tungsten hexafluoride (WF_6) and silane (SiH_4) are separated from the main controls for safety reasons. The flowrates of both WF_6 and SiH_4 are measured with electronic mass flowmeters. The flowrates are displayed on the front panel.

TABLE I PROGRAM DEVELOPMENT AND MILESTONES

TASKS	Oct. 1	Nov. 1	Dec. 1	Jan. 1	Feb. 1	Mar. 1	Apr. 1	May 1	Jun. 1	Jul. 1	Aug. 1	Sep. 1	Oct. 1	Nov. 1	Dec. 1	Jan. 1	Feb. 1	Mar. 1	Apr. 1	May 1	Jun. 1	Jul. 1	Aug. 1	Sep. 1				
	(Completed)																											
Reactor Modification	Δ																											
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Process Preparation	Δ																											
I. Material Development and Analysis																									Δ			
A. Tungsten																												
B. Tungsten-Silicon													Δ															
C. Dielectric													Δ															
D. Alternate Dielectric																									Δ			
E. Substrates															Δ													
II. Component Development																									Δ			
A. Resistors													Δ															
B. Capacitors													Δ															
C. Interconnects																									Δ			
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D. Cycling																									Δ			
V. Technology Transfer																									Δ

Fig. 1 Front View of the Modified Chemical Vapor Deposition Reactor

Fig. 2 Outline of the Controls on the Modified CVD Reactor

The main reaction tube is nominally four inches in diameter and the graphite susceptor is 2-1/2" x 6" which allows us to process two 2" diameter substrates at a time. The temperature range of the reactor is 400°C to >1200°C.

Optical monitoring equipment was installed in the reactor for making in-situ optical measurements on the substrates during the deposition cycles; however, the optical carriage has been removed until an improved means of support can be installed. This does not limit the current development work in the reactor.

The majority of the reactor modifications in one way or another resulted from the use of WF_6 in the system. The reactions using WF_6 are carried out at pressures less than 200 torr. The reaction chamber and all associated lines must operate at pressures of 1×10^{-4} torr without leaking. Tungsten hexafluoride gas is extremely corrosive and toxic. The source and gas controls for the system are designed with a minimum of internal volume and only acceptable materials come into intimate contact with the gas for any extended period of time.

The major components of the system are shown in Fig. 3. The carrier along with the various reactant gases are injected into the main reaction chamber where they pass over the R.F. heated graphite susceptor. The carrier gas, unspent reactant gases, and by-product gases are pumped out of the main reaction chamber. The gas then enters a secondary reaction chamber which is designed with a large high temperature surface area to insure that all of the WF_6 has been reacted. The gas is cooled and passed through a large column of potassium hydroxide to remove hydrofluoric gas, which is a by-product of the reaction of WF_6 and H_2 . The final stage is a

Fig. 3 Major Internal System Components in the Modified Reactor

vacuum pump that provides system vacuum and exhausts the remaining gases out of the building.

Initial deposition runs using the modified reactor were made during the first part of January 1977. The reactor performed exceedingly well and will provide us with a very versatile system for growing the materials needed during this program.

Process Development

The passive components are fabricated from thin film materials. Thicknesses range from 0.01 μm to $>1.0 \mu\text{m}$. The initial materials used in fabricating the resistors, capacitors, interconnects, and passivation are:

1. Tungsten
2. Tungsten-Silicon
3. Silicon Nitride

These materials are formed by the following reactions:

Tungsten

Carrier Gas: H_2

Tungsten-Silicon

Carrier Gas: H_2 or H_2 and He

Silicon Nitride

Carrier Gas: H_2 or He

The tungsten is used as the interconnect metallization between components as well as for the capacitor plates. The tungsten-silicon material is used for resistive components. Care is taken not to refer to the resistive material as tungsten silicide since this would imply a given composition, and in reality we have a very large range of material compositions which does not fall into exact stoichiometric ratios.

The silicon nitride is used for the capacitor dielectric and for device passivation. In a later stage of the program a number of alternate dielectric materials will be deposited and characterized for use with the high temperature components.

The materials for the passive components are physically grown on an insulating substrate, such as sapphire or an oxidized silicon wafer, which serves as the mechanical support for the components. Figure 4 illustrates a resistor and a capacitor as viewed from the top side and also in cross-section. The various layers of materials will be grown by chemical vapor deposition (CVD).

The general deposition process (CVD) is shown in Fig. 5 where the substrates are placed on a graphite susceptor. The susceptor is then loaded into a quartz reaction chamber and heated inductively by the RF coil that is placed around the outside of the quartz tube. A carrier gas such as hydrogen or helium is introduced into the reaction tube where it replaces all of the atmospheric gases. The susceptor is then heated until the correct deposition temperature is reached, typically 400°C to over 1000°C. One or more reactant gases are then added to the flow stream of the carrier gas. Upon reaching the heated substrate surface the gases react on the surface depositing the desired thin film of material and rejecting the by-products of the reaction back into the gas stream as illustrated in Fig. 6 for the

Fig. 4 Cross-sectional View of a Thin Film Resistor, Capacitor, Interconnects and Passivation

Fig. 5 General Deposition System for Chemical Vapor Deposition

The reactant gases, along with a carrier gas, are introduced into a quartz reaction chamber where they react on the heated substrate surfaces to form the desired materials.

Fig. 6 Deposition of Tungsten from Tungsten Hexafluoride and Hydrogen

CVD reaction that is used for depositing tungsten for the interconnecting metallization and capacitor plates. Note that in this reaction the carrier gas (hydrogen) is also a reactant.

In general the flowrate of the carrier gas is 100 to 10,000 times greater than the flowrates of the other gases. The carrier gas serves to provide correct gas environment for the reaction and also to keep the gas velocity at a reasonable rate through the reactor.

Materials Development

The initial depositions in the modified reactor demonstrated that tungsten, tungsten-silicon, and silicon nitride could be successfully deposited in the reactor.

The three tasks, (1) materials development, (2) component development, and (3) process optimization, are carried on as parallel tasks since each deposition is evaluated with respect to all three areas.

Tungsten

Thin films of tungsten were grown on oxidized silicon wafers. They were delineated with the resistivity test pattern shown in Fig. 7, and the resistance was measured using the configuration illustrated in Fig. 8. The thicknesses were measured by using a Nomarski Interferometer on a Reichert "Zetopan" research microscope at two different wavelengths, 5000A and 6000A. Polaroid pictures of the devices and the interference fringes were taken. The fringe separation on the pictures was subsequently measured and the thickness determined.

The resistance of the CVD tungsten was measured on a 1250A tungsten film, and the resistivity was calculated to be 7.8×10^{-6} ohm-cm. The bulk value given for tungsten was 5.5×10^{-6} ohm-cm. The measured value was about

Fig. 7 Resistivity Test Pattern Used to Determine the Resistivity of CVD Deposited Tungsten

Fig. 8 Test Arrangement for Measuring the Conductivity of Tungsten

40% higher than the bulk value; however, for a film thickness of only 1250A this was a very reasonable result. Additional attention during the next few months will be directed toward minimizing the resistivity of the tungsten, but the current value is very acceptable for use with the high temperature components.

In developing the tungsten a series of runs were made using various pressures, flowrates, and total flow velocities. Uniformity in the deposited material was difficult to achieve. The tungsten thickness often varied by a factor of 100 across the two substrates. The uniformity was improved until the thickness varied by less than a factor of 2. Improvement in the uniformity is needed, but the current uniformity presents no significant problems to the fabrication of devices.

Tungsten-Silicon

Thin films of tungsten-silicon were deposited on oxidized silicon wafers. The films were of varying resistivity and thickness. The initial depositions were very non-uniform with the sheet resistance varying from $65 \Omega/\square$ to $>60,000 \Omega/\square$. The non-uniformity was the result of deposition conditions which left a composition gradient as well as a thicknesses gradient across the two wafers on the susceptor. The leading edge of the first wafer had the highest concentration of tungsten and was also the thickest region. The concentration of tungsten decreased along with the thickness across the two wafers. By using deposition conditions that were near the same conditions used for the improved tungsten uniformity, the uniformity of the tungsten-silicon films was improved to the point where the sheet resistance varied less than $\pm 10\%$ of the nominal center value on each wafer.

The films were tested using a standard four-point probe at room temperature before they were delineated. The resistive films were delineated with the resistor pattern shown in Fig. 9. Following delineation, the resistors were measured using the test arrangement shown in Fig. 10. The probes were tungsten wire positioned and held on the resistor pads with micromanipulators. The contacts between the resistor pads and the probes were extremely noisy, preventing the making of highly accurate measurements on the resistors. The high noise level was an expected phenomenon.

The contact noise can be reduced by adding low resistance metal contacts to the resistors. The contact problem will be discussed in greater detail in the section of this report on resistor development.

The most significant result from this work is the verification that thin film resistive material can be grown by means of CVD.

Dielectrics

Silicon nitride, Si_3N_4 , was deposited at 750°C from the reaction of ammonia and silane in a carrier of helium. A total of 23 Si_3N_4 depositions were made in order to improve the deposition uniformity. The deposition parameters were varied in a systematic manner in order to produce uniform Si_3N_4 .

The uniformity was improved to the point of $\pm 5\%$ thickness variation across the front wafer in a reproducible manner.

The test pattern that will be used for making dielectric measurements is shown in Fig. 11. The center dot has an area of 1.00 mm^2 , and it is surrounded by a guard ring. For test purposes the dielectric will be grown over a continuous layer of CVD tungsten which forms one of the capacitor plates. A second layer of tungsten will be deposited by CVD and

Fig. 9 Resistor Test Pattern Used to Delineate Test Resistors in Tungsten Silicon Thin Films

Fig. 10 Test Configuration for the Plotting of Resistance Versus Temperature Curves

Fig. 11 Dielectric Test Pattern

Area of center dot is 1.0 mm^2 .

delineated into the test pattern. Contact will be made to the top center dot and to the bottom metallization. The test pattern forms a parallel plate capacitor from which the characteristics of the dielectric material can be derived. At the same time, the test pattern provides the data needed to characterize the operation of the capacitors.

The dielectrics will be used both in the capacitors and as a passivation layer.

Alternate Dielectrics and Substrates

No work has been started on alternate dielectrics at this time, and only oxidized silicon wafers have been used as substrates.

In a previous investigation it was demonstrated that the characteristics of sputtered W-Si resistors were not dependent on either oxidized silicon wafers or polished sapphire wafers. The low cost, ease of processing, and large area make using oxidized silicon wafers very attractive for development substrates. All of the processes will be interchangeable between the substrates.

Component Development

As was mentioned earlier all of the major tasks are carried out in parallel, and there is no definite line of division between tasks; however, as the various materials are developed to the point where they can be used in component fabrication, they will be discussed from the viewpoint of component fabrication.

Resistors

The resistors are formed by delineating a resistor pattern in a thin film of CVD deposited tungsten-silicon (W-Si).

The sheet resistance of the thin film is determined by the resistivity of the material and its thickness.

$$R_s = \frac{\rho}{d} \Omega/\square$$

where

R_s = sheet resistance

ρ = resistivity

d = thickness

$\Omega/\square$ = ohms per square

The number of squares in a simple dog-bone type of resistor is the ratio of its length to width.

$$\text{Number of Squares} = \frac{\text{Length}}{\text{Width}}$$

Then the total resistance is equal to the number of squares times the sheet resistance

$$R = (\text{No. of Squares})(R_s) \text{ ohms}$$

A delineated set of resistors is shown in Fig. 12.

Making accurate measurements on the delineated resistors was impossible due to the noise generated between the resistor material and the tungsten probes. Both the probes and the resistor were made from extremely hard materials so that the area of contact was small, and any slight movement of the probe would change the indicated resistance value. Room temperature measurements could be made, but heat cycling the resistors for resistance versus temperature measurements was impossible due to the motion of the probes.

Fig. 12 Delineated Tungsten-Silicon Resistors

To circumvent the contact problem an intermediate process is being developed which will provide low noise contacts. The resistors are delineated by the standard process, and then coated with approximately 2000Å of silicon dioxide, SiO_2 . Contact windows are etched in the SiO_2 over the resistor pads, and aluminum is thermally evaporated over the entire wafer. Following the aluminum deposition, the aluminum contact pads are delineated.

The process is a standard approach to the problem, and it was used successfully for the high temperature measurements made on the Siemens Corporation resistors. The Siemens' resistors were tested to 550°C without contact problems. It should be noted that this is only a temporary solution, and it will be replaced at a later date with tungsten. A major advantage to this approach is that the resistor will be characterized without additional heat cycling that would be required for the deposition of tungsten. The processing sequence is illustrated in Fig. 13. As soon as the contact process has been completed, high temperature characterization measurements will be made.

Capacitors

Capacitor development is not scheduled to begin until the seventh month of the program. The preliminary work has been completed. Several runs have also been made in which silicon nitride was deposited over CVD tungsten with no apparent problems.

Interconnects

Tungsten has been successfully deposited and delineated. The resistivity of the material was $7.8 \times 10^{-6} \Omega\text{-cm}$ which is 40% greater than the bulk value.

Fig. 13 Processing Sequence for the Delineation of Aluminum Contacts on Tungsten-Silicon

The remaining work to be completed on the interconnects is the actual interconnecting of several components into a network, and measuring the contact resistance between the resistor material and the tungsten.

Passivation

The passivation of the components will initially be done with silicon nitride; the results of material development and analysis will be directly related to the use of silicon nitride as a passivation material.

Process Optimization

Process monitoring and analysis is being carried on concurrently with the materials and component development. The one exception is the optical monitoring which has temporarily been removed from the reactor.

Program Management

The program development and milestone chart is given in Table I. At the end of our sixth month, March 1977, we had completed the reactor modifications and all process preparations. The tasks of material development, component development, and process optimization have been initiated.

The equipment and processes are performing very well, and no problems that would delay the progress of the current development schedule are anticipated.

Resources

The total manpower expended for the first six months is 4176 manhours. The number of manhours expended as a function of time is plotted in Fig. 14.

The money expenditures for the first six months is plotted in Fig. 15.

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TASKS	Oct. 1	Nov. 1	Dec. 1	Jan. 1	Feb. 1	Mar. 1	Apr. 1	May 1	Jun. 1	Jul. 1	Aug. 1	Sep. 1	Oct. 1	Nov. 1	Dec. 1	Jan. 1	Feb. 1	Mar. 1	Apr. 1	May 1	Jun. 1	Jul. 1	Aug. 1	Sep. 1
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Fig. 14 Manhours Versus Project Time

Fig. 15 Money Versus Time