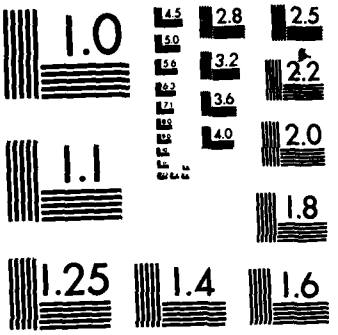
AD-A160 350 DEVELOPMENT OF A SYSTEM TO PRODUCE SEMICONDUCTOR INSULATOR MULTI-LAYER STRUCTURES(U) CLEMSON UNIV SC DEPT OF ELECTRICAL AND COMPUTER ENGINEERING 1/1

UNCLASSIFIED D J DUMIN 26 SEP 85 N00014-84-K-2015 F/G 7/1 NL



DEVELOPMENT OF A SYSTEM TO PRODUCE SEMICONDUCTOR INSULATOR MULTI-LAYER STRUCTURES

D. J. Dumin

Performed on Contract
N00014-84-k-2015
Naval Research Labs

(12)

DEVELOPMENT OF A SYSTEM TO PRODUCE SEMICONDUCTOR INSULATOR MULTI-LAYER STRUCTURES

D. J. Dumin

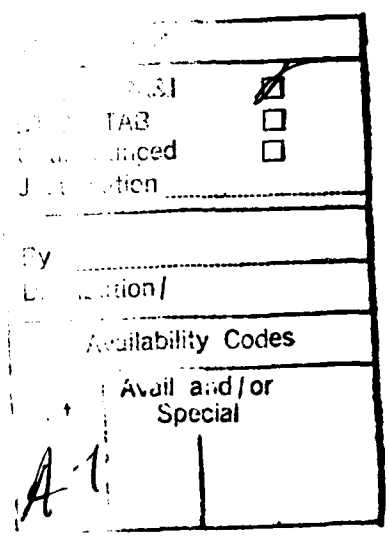
Performed on Contract
N00014-84-k-2015
Naval Research Labs

TABLE OF CONTENTS

Preface

This final report prepared by Clemson University under Contract N00014-84-K-2015, describes research performed in the Department of Electrical and Computer Engineering, Dr. A. Wayne Bennett, Department Head.

The principal investigator was Dr. David J. Dumin. The graduate student working on the project was Mr. Perry J. Robertson. The rf generator used in this work was provided by Dr. Charles C. Fain of the Department of Ceramic Engineering. The Department of Ceramic Engineering provided the space to house the epitaxial reactor. The gas cooled reactor was provided by Gracie Davis and Marty Peckerar of the Naval Research Labs. This assistance was greatly appreciated.



LIST OF FIGURES

LIST OF TABLES

SECTION I

ABSTRACT

The development of Silicon-on-Sapphire (SOS) and Silicon-on-Insulator (SOI) technologies has lead to applications of these technologies in high speed circuits, high density circuits, circuits with freedom from 4-layer latch-up, and radiation hard circuits. There has been significant effort aimed at commercialization of these processes. One of the major problems limiting wide spread use of SOS is the high cost of the sapphire substrate. Many attempts to produce lower cost SOI substrates using ion implantation of insulating materials and various annealing techniques to improve the crystallinity of poly crystal silicon layers have been attempted. An alternate technique to produce SOI materials has been to grow multiple heteroepitaxial silicon-insulator-silicon layers on silicon or sapphire substrates and use these layers for the fabrication of integrated circuits. Several insulators have been used in these structures. There is a continuing search for an optimum insulator to use in multiple silicon-insulator structures.

One insulator that has been used for these multiple heteroepitaxial layers is boron phosphide. The purpose of the present work was to design and build an epitaxial reactor capable of growing silicon and boron phosphide layers on silicon and sapphire substrates and to use this reactor to grow layers of silicon and boron phosphide on silicon substrates.

We have designed and built an epitaxial reactor, and have used this reactor to grow silicon on silicon, silicon on sapphire, boron phosphide on silicon, and silicon on boron phosphide on silicon. Several examples of silicon on sapphire films grown at different

growth rates from 1 um/min to 5 um/min have been provided as part of this contract. Several wafers of silicon on boron phosphide on silicon have been provided. These Si-BP-Si wafers had different thicknesses of silicon and boron phosphide.

SECTION II

INTRODUCTION

Heteroepitaxial growth of multiple layers of silicon and boron phosphide on silicon substrates has been demonstrated. (1) Up to 4 layers of silicon-boron phosphide have been grown on silicon and up to 2 layers have been grown on SOS wafers. (2) The quality of the top silicon layer was sufficient to allow the fabrication of MOS integrated circuits on the top layer of each of these multi-level structures. (2) Since this multiple heteroepitaxial work had been done while the author was in Japan, it was decided to attempt to grow similar structures in a chemical reactor in the U.S.

An epitaxial reactor was designed and built. The reactor had provisions for the use of high purity hydrogen as the reactor gas and for the incorporation of three reaction gases. This reactor has been used to grow silicon on sapphire and layers of boron phosphide on silicon and silicon on boron phosphide on silicon. The reactor has an additional input port for the addition of another reaction gas.

SECTION III

REACTOR DESIGN AND OPERATION

A schematic diagram of the gas flow system of the chemical reactor is shown in Figure 1. The reactor chamber was either a gas cooled or water cooled vertical reactor. The reactor used a motor-generator to provide rf power to inductively heat the susceptor. The susceptor was a silicon carbide coated graphite block that supported substrates up to 2" diameter. The hydrogen carrier gas was purified by passage through a palladium purifier. The nitrogen flush gas was purified by passage through a catalytic purifier. All of the reaction gases were diluted with ultra high purity hydrogen. The reactor chamber was either a gas cooled reactor provided by the Naval Research Labs or a water cooled reactor purchased for this project. The faster cycle time and lower exhaust gas temperatures associated with the water cooled reactor made use of this reactor preferable. The quality of the films produced by the water cooled reactor appeared to be higher than that of the films produced in the gas cooled reactor. The smaller diameter of the gas cooled reactor lead to higher vertical gas flow rates and higher film growth rates. The substrate temperature was measured using an optical pyrometer and was monitored with an infrared pyrometer. The infrared pyrometer had its peak sensitivity at 1.0 um and was used to measure the in-situ growth rate. (3) Rotameters were used to measure the flow rates of the gases.

A typical growth cycle for an SOS wafer is described in Table 1 and for a Si-BP-Si wafer in Table 2. The wafers were chemically cleaned and loaded into the reactor. The wafers were either (100) silicon or $(1\bar{1}02)$ sapphire. The reactor was flushed for 2 min. with

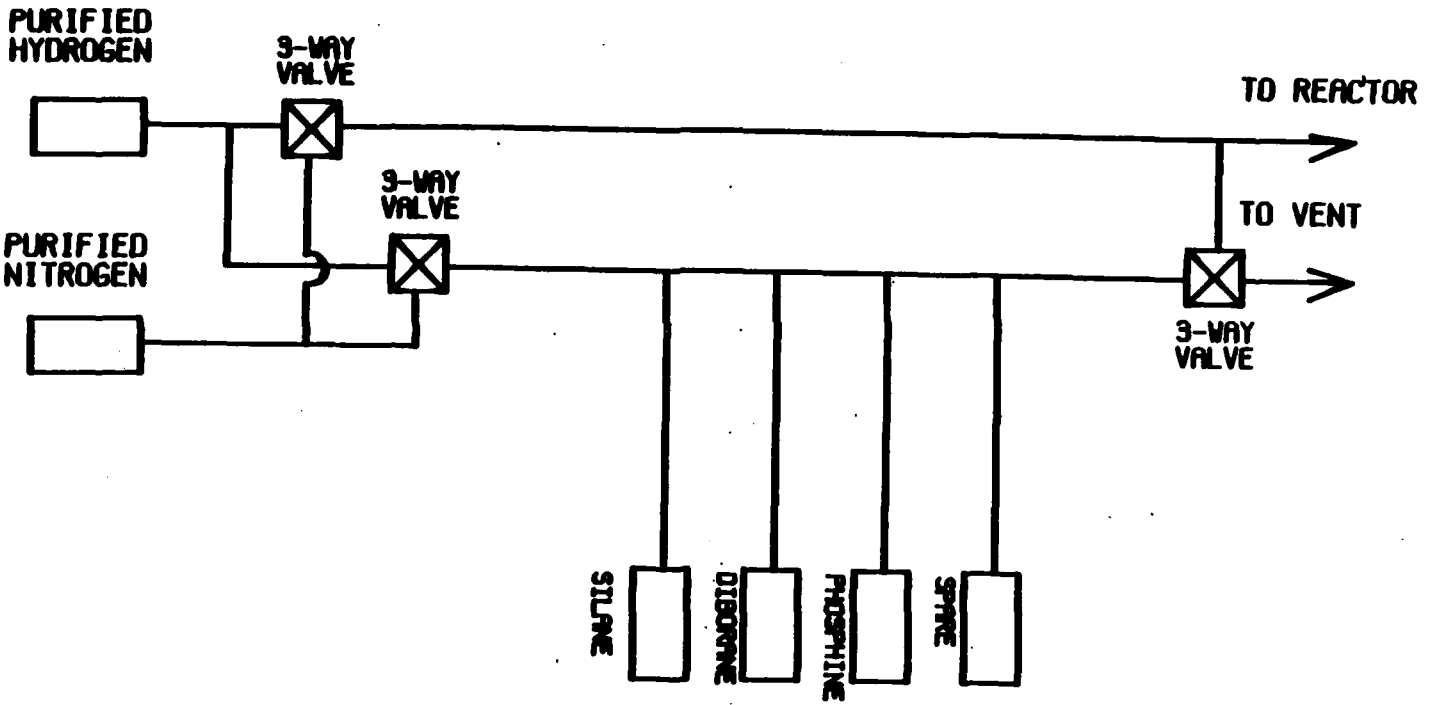


FIGURE 1

SCHEMATIC DRAWING OF EPITAXIAL REACTOR FLOW SYSTEM

TABLE 1

GROWTH CYCLE USED TO GROW SILICON ON SAPPHIRE FILMS

1. Clean wafer: hydrogen peroxide/sulfuric acid, water rinse, dry wafer.

2. Load into reactor, nitrogen flowing, switch to hydrogen after 2 min.

3. Heat substrate to growth temperature in hydrogen (typically about 1000C)

4. Grow silicon in hydrogen/silane mixture. Concentration of silane controlled the growth rate. Monitor growth with infrared pyrometer. Stop run when desired thickness had been reached.

5. After growth, flush reactor with hydrogen, cool in hydrogen, switch to nitrogen.

6. Remove wafer from reactor. Characterize the wafer.

SECTION VII

CONCLUSIONS

A vertical epitaxial reactor has been constructed and tested. The reactor has been used to grow silicon on sapphire films at growth rates up to 5 um/min and, with the addition of 100% silane, could be used at higher growth rates. The reactor has been used to grow single crystal boron phosphide films on silicon and silicon films on boron phosphide on silicon. Growth rates of 0.05 to 0.2 um/min have been used to grow the boron phosphide. All films have been grown at growth temperatures of 950C to 1050C. The reactor is now capable of growing specific films for specific needs or applications. Several wafers of silicon on sapphire films and silicon on boron phosphide on silicon have been provided as part of this contract.

needed.

One of the problems associated with the use of BP as an insulator is the autodoping of the silicon by boron and/or phosphorus from the BP. A more benign insulator is desireable. This epitaxial reactor could be used for a study of the growth of silicon carbide, boron nitride, or silicon oxide on silicon. All of these materials could be synthesized using hydride sources and the reaction gases could be added to the reactor with minimal effort.

SECTION VI

SUGGESTIONS FOR FUTURE WORK

There are several possible uses for the reactor constructed under this contract. It is desireable to use SOS films in the range of 0.2 to 0.4 um in thickness for submicron geometry SOS circuits. In the past it has proved impossible to directly grow high mobility SOS films in this thickness range. Various regrowth techniques have been reasonably successful, but have been difficult to implement using silicon films as thin as 0.3 um. It will be possible to use our reactor to explore the possibility of growing high mobility films by utilizing very high growth rates. If 100% silane is used as the reaction gas rather than the 5% mixture used in the initial film growths, it should be possible to obtain growth rates in excess of 20 um/min. Measurement of transistor characteristics fabricated on high growth rate, very thin SOS films would establish the feasibility of using high growth rates for production of device quality SOS films in the thickness range of 0.2 to 0.4 um.

For heteroepitaxial SOI films to be useful in MOS integrated circuit fabrication, it will be necessary to produce material with low surface state density at every silicon-insulator interface. It will be necessary to initiate a study to determine the film properties as a function of the growth conditions and, particularly, the techniques needed to produce low surface state density films as a function of the growth conditions. The characteristics of boron phosphide grown on silicon and of silicon grown on boron phosphide are not well understood. The dependence of film properties on the growth conditions is not well known. A detailed study of the properties of these thin film combinations as a function of growth conditions is

All of the films were N-type with relatively low resistivities. The cause of these low resistivities is not yet understood, and further experiments will be necessary to determine the cause of the low resistivities. All of the films were shiny and free from haze. Selected films were tested in the SEM and all films were determined to be (100) single crystals..

FIGURE 4

CHARACTERISTICS OF SI ON BP ON SI FILMS PROVIDED UNDER THIS CONTRACT

Wafer #	Layer Thickness (um)			Type	Resistivity
	Bottom Si	BP	Top Si		(ohm-cm)
133	0.5	0.1	0.5	N	0.0047
134	0.43	0.224	0.43	N	0.0086
135	0.43	0.34	0.43	N	0.0037
136	0.43	0.44	0.43	N	0.0039
137	0.5	0.2	1.0	N	0.008

All films described above had growth rates of:
Si 1um/min
BP 0.05um/min

132 0.05 um of BP grown on top of 1 um of silicon
N-type, 0.0024 ohm cm.
Same growth rates as shown above

TABLE 3

CHARACTERISTICS OF SOS FILMS PROVIDED UNDER THIS CONTRACT

Wafer #	Thickness (um)	Growth Rate (um/min)	Type	Resistivity (Ohm-cm)
126	0.3	2.0	N	0.046
127	0.25	1.0	N	0.075
128	0.3	2.7	N	0.57
129	0.42	4.8	-	high
130	0.3	4.4	-	high
131	0.86 um of silicon grown on 0.1 um of BP grown on sapphire BP growth rate of 0.1 um/min Si growth rate of 1.0 um/min			

SECTION V

FILMS PROVIDED BY THIS CONTRACT

A summary of the characteristics of films provided by this contract appears in Table 3 and Table 4. The silicon on sapphire films are described in Table 3. The silicon on sapphire films were all grown with a thickness target of 0.3 um. Films of this thickness should provide a good test to determine the carrier mobility in thin SOS films. The highest growth rate that could be achieved in the water cooled reactor using 5% silane in hydrogen was 4.8 um/min. In order to achieve higher growth rates 100% silane would have to be added to the reactor. The gas cooled reactor provided higher growth rates, but the films were cloudy indicating an air leak. The quartz was beginning to devitrify and may have become porous.

The silicon on BP on silicon films provided under this contract are described in Table 4. These films were grown with about 0.4 to 0.5 um of silicon on the silicon followed by the BP and then the top layer of silicon. For films 133 to 136 the top layer of silicon was 0.4 to 0.5 um. The thickness of the BP layer was varied from 0.1 um to 0.44 um to determine if the thickness of the BP layer affected the crystal quality of the silicon. There was no observed change in top layer silicon quality as the BP layer thickness was changed. It will be necessary to make more detailed measurements of the films to determine the details of the relationship between BP growth conditions and silicon quality. Of particular interest would be the surface state density at the Si-BP interfaces. One film with BP as the top layer was provided. This film, 132, consisted of 1 um of silicon grown on silicon followed by 0.05 um of BP. This film can be examined to determine the quality of the BP layer.

the interference pattern is a good indication of the film quality and, thus, film quality can be estimated while the film is still in the reactor.

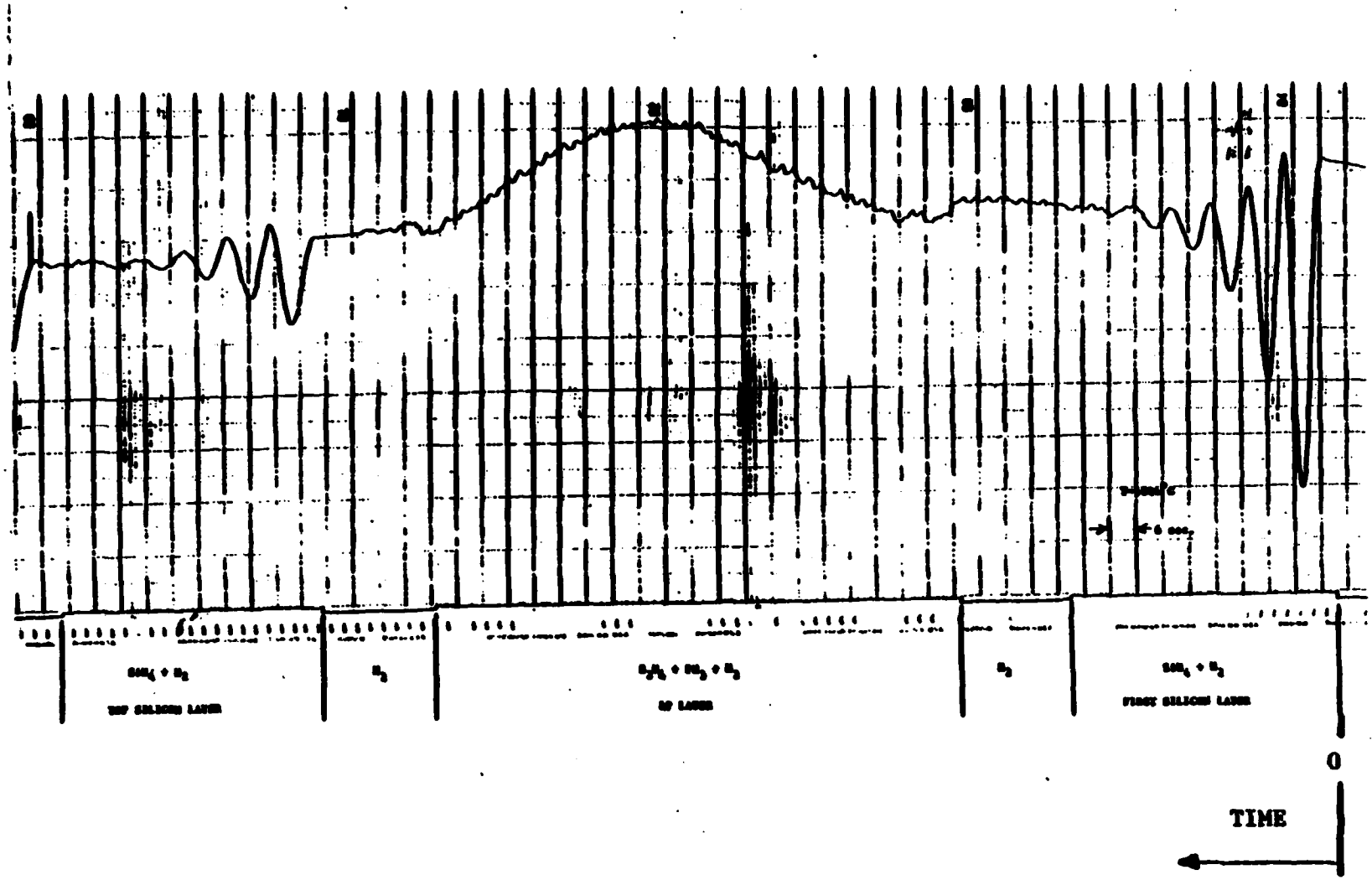


FIGURE 3

OUTPUT FROM THE INFRARED PYROMETER DURING FILM GROWTH

gas cooled reactor were of variable quality with the better films being comparable to those grown in the water cooled reactor, but with the average quality being lower. Several films grown in the water cooled reactor with film thicknesses near 0.3 um grown at growth rates from 1 um/min to 5 um/min were provided as part of this contract. The properties of the SOS films provided by this contract are described in Section V. The growth rate of the silicon films grown on the boron phosphide films was 1 to 4 um/min.

The silicon films grown on boron phosphide were grown using the growth sequence shown in Table 2. The films were all grown on freshly cleaned (100) silicon substrates. The characteristics of the silicon on boron phosphide on silicon films provided by this contract are described in Section V. It was observed that the growth rate and crystallinity of the boron phosphide films was a function of the growth temperature, the phosphine and diborane flow rate, and the mole ratios of the phosphine and diborane. A more detailed study will be necessary to determine the influence of the growth conditions on the growth rate and crystallinity of the boron phosphide films and on the influence of the boron phosphide growth conditions on the quality of the silicon films. As was the case with the SOS films, higher quality films were grown using the water cooled reactor chamber.

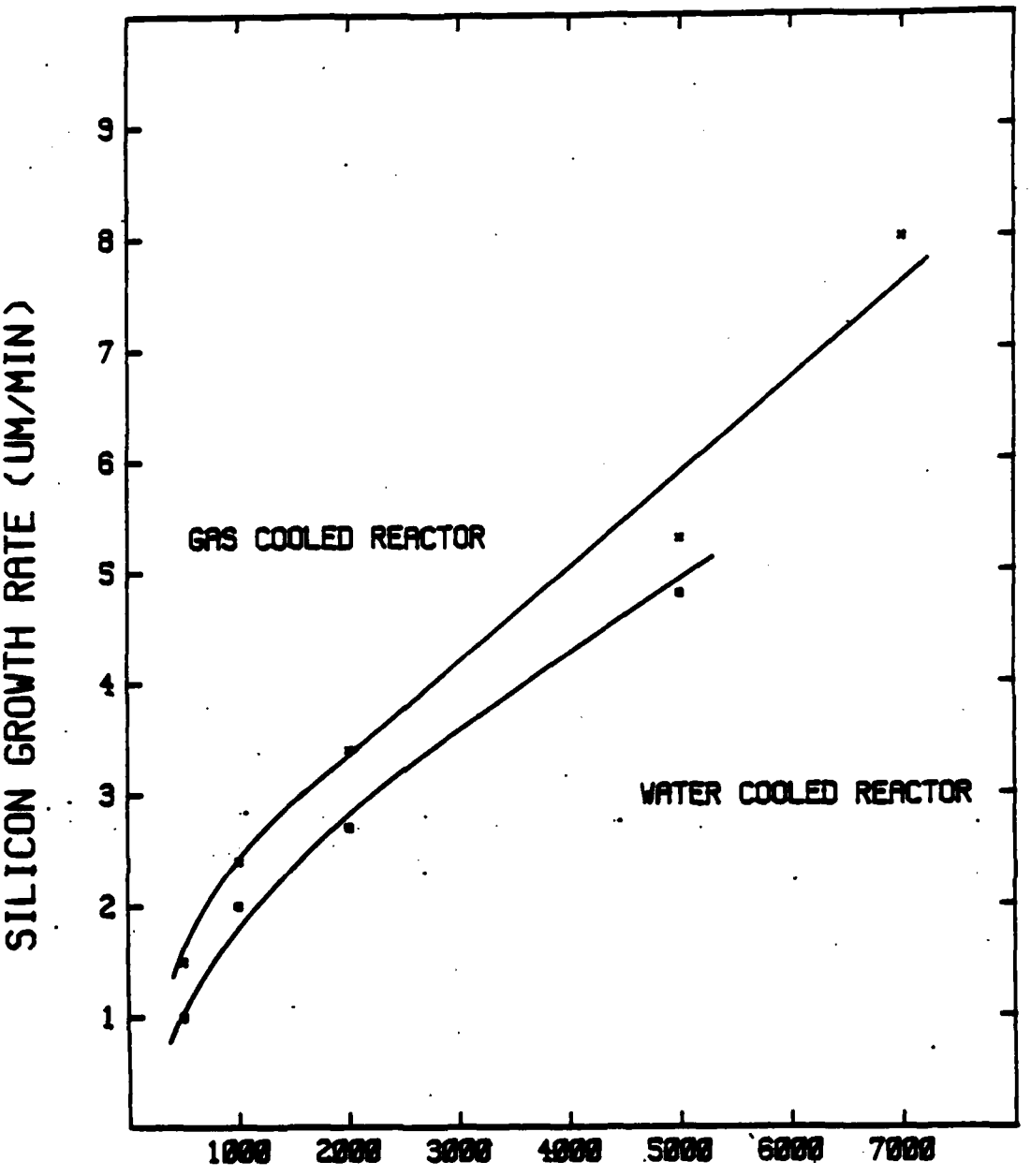
The output from the infrared pyrometer during the growth of a multiple Si-BP-Si layer is shown in Figure 3. The growth temperature was 1006 C. Initially a 1.0 um thick silicon layer was grown for 1 min. The reactor was flushed for 30 sec while the silane was turned off and the diborane and phosphine flows were established. The BP was grown for 2 min followed by a 30 sec flush and the growth of the top silicon layer. The delay between the switching of the three-way valve and the growth of the layer is evident in Figure 3. The sharpness of

SECTION IV

RESULTS OF FILM GROWTH

The calibration of the epitaxial reactor involved determining the growth rate, the crystal structure, the thickness, the conductivity type, the resistivity, and the smoothness of the films as a function of the growth parameters. The growth parameters were growth temperature, hydrogen flow rate, reaction gas flow rates, mole percents of the reaction gases, and growth time. In order to suppress gas phase decomposition of the reaction gases, it was desireable to keep the hydrogen carrier at its maximum flow rate to provide the maximum amount of reactor cooling. Thus, the hydrogen flow rate was kept at 9 l/min in the main line and 1 l/min in the diluent line during all of the growths described below.

The first calibration runs involved growth of silicon on sapphire. The growth rate was determined by recording the output from the infrared pyrometer as a function of time after the silane had been introduced into the reactor. There was about a 2 sec. delay between the switching of the reaction gas with the 3-way valve and initiation of film growth in the reactor. In Figure 2 the growth rate of silicon vs. flow rate of silane in the water cooled reactor is plotted. The reaction gas was silane, diluted to 5% in hydrogen in the tank further diluted with 1 l/min of diluent gas. The growth rate of silicon appeared to be independent of temperature in the temperature range from 950C to 1050C. The films were (100) single crystal as determined by examination in the SEM. Since the films were all less than 1um thick, the measured values of resistivity were often very high. The films were shiny and free of haze and were considered to be of device quality when grown in the water cooled reactor. Films grown in the



GROWTH RATE VS. FLOW RATE

FIGURE 2

nitrogen and flushed for 1 min. with hydrogen before the rf power was applied. After the rf power had been applied it took about 5 min. for the substrate to reach growth temperature. While the substrate was heating to the growth temperature the reaction gases were set to the conditions desired for that particular growth run. After the substrate had reached the growth temperature and the growth temperature had stabilized, the reaction gases were introduced into the growth chamber using the 3-way valve in the reaction gas line. The film growth was terminated by switching the reactor gases to the vent. After film growth the reactor was flushed for 30 sec. with hydrogen, and the rf power removed. The reaction gases were turned off while the sample returned to room temperature. The carrier gas was switched to nitrogen when the substrate temperature had been reduced below 600C. After the substrate had cooled to near room temperature the sample was removed from the reactor, a new substrate was loaded into the reactor and a new growth run was started.

At the start of every day's growth runs, the susceptor was coated with a 0.5 um thick film of silicon. This layer was used to help insure that the reactor plumbing was clean and that the susceptor was clean.

After the wafer had been removed from the reactor the films were examined for cleanliness, color, and appearance. The resistivity and conductivity type were measured.

TABLE 2

GROWTH CYCLE USED TO GROW SILICON ON BORON PHOSPHIDE ON SILICON

1. Clean wafer in sulfuric acid/hydrogen peroxide, water rinse, hydrofloric acid, water rinse, dry wafer.

2. Load wafer into reactor, nitrogen flowing, switch to hydrogen after 2 min.

3. Heat wafer to growth temperature in hydrogen.

4. Grow 0.3 um of silicon at 1 to 4 um/min.

5. After film growth, flush with hydrogen for 30 sec.

6. Grow boron phosphide on silicon at the same growth temperature used to grow the silicon. Growth rate and growth time determined by diborane and phosphine flow rates and thickness of BP desired.

7. After growth of BP, flush reactor with hydrogen for 30 sec.

8. Grow top layer of silicon. Thichness and growth rate determined by silane flow rate, growth time and desired thickness.

9. After film growth, flush reactor with hydrogen, cool in hydrogen, switch to nitrogen when temperature dropped below 600C.

10. Remove wafer from system and characterize the films.

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: Final	2. GOVT ACCESSION NO. AD-A160 106
3. RECIPIENT'S CATALOG NUMBER: -	
4. TITLE (and Subtitle): Development of a System to Produce Semi-conductor Insulator Multi-layer Structures	5. TYPE OF REPORT & PERIOD COVERED: Final 27 Aug. 84 to 26 Aug. 85
	6. PERFORMING ORG. REPORT NUMBER: -
7. AUTHOR(s): David J. Dumin	8. CONTRACT OR GRANT NUMBER(s): N00014-84-K-2015
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Dept. of Electrical and Computer Engineering Clemson University Clemson, SC 29631	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: -
11. CONTROLLING OFFICE NAME AND ADDRESS: Department of the Navy Naval Research Labs. Washington, DC 20375	12. REPORT DATE: 26 Sept. 85 13. NUMBER OF PAGES: 21 + vi
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office): Office of Naval Research Georgia Inst. of Tech. Rm. 206, O'Keefe Bldg. Atlanta, Ga. 30332	15. SECURITY CLASS. (of this report): Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE: -

16. DISTRIBUTION STATEMENT (of this Report)
unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)
-

18. SUPPLEMENTARY NOTES
-

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)
Silicon on Insulator, Epitaxy,
Silicon on Boron Phosphide
SOS

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The development of Silicon-on-Sapphire (SOS) and Silicon-on-Insulator (SOI) technologies has lead to applications of these technologies in high speed circuits, high density circuits, circuits with freedom from 4-layer latch-up, and radiation hard circuits. There has been significant effort aimed at commercialization of these processes. One of the major problems limiting wide spread use of SOS is the high cost of the sapphire substrate.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE
S/N 0102-LF-014-6601

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

20. ABSTRACT (cont.)

Many attempts to produce lower cost SOI substrates using ion implantation of insulating materials and various annealing techniques to improve the crystallinity of poly crystal silicon layers have been attempted. An alternate technique to produce SOI materials has been to grow multiple heteroepitaxial silicon-insulator-silicon layers on silicon or sapphire substrates and use these layers for the fabrication of integrated circuits. Several insulators have been used in these structures. There is a continuing search for an optimum insulator to use in multiple silicon-insulator structures.

One insulator that has been used for these multiple heteroepitaxial layers is boron phosphide. The purpose of the present work was to design and build an epitaxial reactor capable of growing silicon and boron phosphide layers on silicon and sapphire substrates and to use this reactor to grow layers of silicon and boron phosphide on silicon substrates.

We have designed and built an epitaxial reactor, and have used this reactor to grow silicon on silicon, silicon on sapphire, boron phosphide on silicon, and silicon on boron phosphide on silicon. Several examples of silicon on sapphire films grown at different growth rates from 1 um/min to 5 um/min have been provided as part of this contract. Several wafers of silicon on boron phosphide on silicon have been provided. These Si-BP-Si wafers had different thicknesses of silicon and boron phosphide.

REFERENCES

1. K. Nomaka, C. K. Kim, and K. Shono, J. Crystal Growth, vol.50, p 549, 1980.

2. S. Sugiura, T. Yoshida, Y. Kaneko, K. Shono, and D. J. Dumin, To be published in IEEE Trans. on Electron Devices, Nov. 1985.

3. D. J. Dumin, Review of Sci. Instr., Vol. 38, p 1107, 1967.

END

FILMED

11-85

DTIC