Med. & biol. Engng. Vol. 9, pp. 339–350. Pergamon Press, 1971. Printed in Great Britain

THIN FILMS OF GLASS AND THEIR APPLICATION TO BIOMEDICAL SENSORS*

K. D. WISE and R. H. WEISSMAN†
Department of Electrical Engineering, Stanford University, Stanford, California, U.S.A.

Abstract—Thin films of glass are attractive as a means for protecting integrated circuits for use in biological systems and are especially suitable for use with biomedical sensors. They offer advantages over polymer films since they can be deposited in thin, uniform layers and can be photoengraved using conventional techniques. This paper first reviews the requirements for film quality in using thin glassy films in these applications and then reviews the techniques available for film deposition. One technique which has been found especially suitable for biomedical work is described in detail, and the characterization of the resulting films is discussed.

Films of silicon dioxide and silicon nitride 0·2–0·3 μm thick have exhibited adequate adhesion and continuity for use with sensors fabricated using integrated circuit technology. Furthermore, they have provided excellent corrosion protection for unencapsulated transistors implanted in the brain for periods of 4 months. Such glassy films also provide good electrical insulation for applied voltages less than 1 V; however, when higher voltages are present, thicker films are required to avoid excessive leakage currents. Applications to specific sensors are considered.

1. INTRODUCTION

THE APPLICATION of integrated circuit technology to problems in biomedical instrumentation promises significant improvements in such systems and is being pursued by a rapidly increasing number of workers. Improvements in reliability and cost are already evident in a number of biomedical systems. However, from the standpoint of gaining a better understanding of biological systems, new technology is most important when it allows experiments to be performed which have been difficult or impossible in the past. Since biomedical sensors are the most frequent factor determining whether or not a given measurement is possible, some important advances can be expected as new technology is applied in this area. Integrated circuit technology is especially applicable to sensors because the precise dimensional control possible lends itself well to the small sizes required in many such transducer applications.

The ability to deposit thin films of glass is particularly important in biomedical applications both as a potential means for passivating devices intended for use in the body and, especially, as a means for insulating the sensor itself. It is the purpose of this paper to discuss the potential problems and requirements on film quality in using thin glassy films in these applications, and to review the techniques available for film deposition. One technique which has been found especially suitable for biomedical work will be described in detail, and the characterization of the resulting films will be discussed. The paper is intended for engineers who are interested in applying new technology to biomedical instrumentation and for physiologists interested in the techniques required for sensor fabrication.

2. APPLICATION TO BIOMEDICAL SENSORS

The study of insulating films continues to be an important area in biomaterials research. In the past, this research has dealt primarily with

* *Received* 29 *June* 1970.
† Present address: Hewlett-Packard Associates, Palo Alto, California, U.S.A.

materials for use as encapsulations for various implanted packages and for prosthetic devices. The function of these insulating films has been to shield the implanted object against the corrosive effects of body fluids and, sometimes, to provide electrical insulation as well. Corrosion problems have been especially severe, and many polymers such as polyurethane, polyethylene, polystyrene and others are extensively degraded (LEVINE, 1968). Nevertheless, materials such as polytetrafluoroethylene, polyvinyl chloride, and certain of the polysiloxanes (silicones) have been successfully employed in a wide variety of implant applications (HALPERN, 1968). The silicones have been especially important because of the inability of the body to attack these materials. The medical-grade silicones cause as little tissue reaction as any material known, and furthermore, their bulk electrical properties, including a volume resistivity of about 10^{14} ohm-cm, do not appear to degrade seriously with long term exposure to saline solutions (ROBB, 1968; BRALEY, 1968; WHITE, 1969).

When one looks at biomedical sensors, several new problems arise. The sensor must, by definition, contact the biological environment in some way in order to monitor the physiological event under investigation. Often only part of the total sensor area is active while the rest of the transducer must be insulated from the body fluids present. In the case of bio-electrodes, the recording or stimulating area must be exposed while the remainder of the electrode is electrically insulated from the extracellular fluid. In the case of strain or pressure sensors, the encapsulation must provide electrochemical integrity without significantly affecting the mechanical properties of the strain sensitive element. In the case of solid-state miniature temperature sensors, any encapsulation must present a very low thermal impedance between the active transducer area and the body.

The polymers mentioned above are difficult to apply in thin (less than 2 μm) coatings, and the areas coated cannot be accurately controlled. On the other hand, the materials silicon dioxide (fused quartz) and silicon nitride have excellent electrical and chemical properties, and in addition can be deposited in thin, uniform films which can then be etched using the planar photoengraving process (WARNER and FORDEMWALT, 1965) to achieve precise dimensional control. These insulating films are transparent in the visible and infra-red, possess good mechanical properties and a very low thermal impedance, and are inert in saline solutions. They are therefore attractive from the standpoint of sensor fabrication. Moreover, silicon dioxide and silicon nitride are utilized in the fabrication of integrated circuits and can be expected to play an important and necessary role in future transducers fabricated using integrated circuit technology.

3. FILM REQUIREMENTS

3.1 *Mechanical requirements*

Thin insulating films for use with biomedical sensors should meet a number of requirements, of which the mechanical requirements of continuity and adherence are two of the most important. The deposited film should form a continuous coating over the sensor surface, and should maintain its integrity over small steps in this surface. Since the film should be thin enough that the mechanical properties of the sensor are unaffected by its presence, the deposition technique should not be of the line-of-sight variety. In addition, the adhesion of the deposited film to the substrate must permit normal handling, implantation, and use *in vivo*.

3.2 *Chemical requirements*

Since the insulating film must contact the biological environment in these applications, one prime requirement is that the film be inert in biological fluids and produce a minimum of tissue reaction due to its long-term presence. Secondly, long-term exposure to biological fluids must not cause chemical changes in the film structure—the film must be stable over time. In addition, the corrosive environment of the body must be masked from vulnerable portions of the sensor by the film.

3.3 *Electrical requirements*

The deposited film should prevent stray electrical leakage from the sensor into the surrounding fluid or tissue. In addition, the film should preserve the electrical integrity of the sensor *in vivo*, preventing d.c. leakage along the transducer surface. The dielectric constant of the film should not be degraded in these applications so that a.c. isolation can be maintained where required.

3.4 *Other requirements*

Two other requirements should be mentioned. First, the deposition temperature should be as low as possible consistent with obtaining a high quality film. The deposition process should not degrade the sensor in any way, and therefore, the material deposited and the associated deposition process must be compatible with all of the sensor materials used. For example, with integrated circuit sensors having gold metalization a low deposition temperature is especially important due to the low silicon–gold eutectic (375°C).

A second requirement is cost. There are significant differences in the costs of various deposition techniques and one need not always sacrifice quality for an inexpensive system. These considerations will be discussed in the next section.

4. DEPOSITION TECHNIQUES

There are several techniques by which thin glassy films may be deposited, and these techniques may be generally classed as pyrolytic (vapour deposition), vacuum evaporation, and sputtering. Pyrolytic methods are based on the thermal decomposition of various silicon compounds. Differences among the different techniques arise in how the decomposition energy is applied and what compound is decomposed. The simplest of these methods is the thermal oxidation of silane to obtain silicon dioxide (KERN and HEIM, 1970; BURGER and DONOVAN, 1967). Film growth rate is a function of both the silane and oxygen flow rates and the substrate temperature. Although some deposition occurs at room temperature, substrate temperatures of 300°C or higher are necessary to obtain high quality films. Since the deposition does not rely on line-of-sight techniques, the continuity of the films produced is good. Deposition temperatures are low, and the system is the least expensive of any of the techniques known. Its prime disadvantage is a lack of flexibility in that it is restricted to the silicon–oxygen glasses and that exact film composition may be difficult to reproduce. Other pyrolytic techniques (BURGER and DONOVAN, 1967) which operate on the decomposition of certain alkoxysilanes require minimum substrate temperatures above 600°C and will not be considered further for the present biomedical applications.

Vacuum evaporation of silicon monoxide (YORK, 1963) to deposit silicon dioxide-like films has been employed in some laboratories; however, when substrate temperatures less than 600°C are used, the resulting films contain large amounts of elemental silicon. The porosity and poor electrical characteristics of such films limit their usefulness for biomedical sensors.

Radio-frequency sputtering (BURGER and DONOVAN, 1967; FRANK and MOBERG, 1970) utilizes an electrical discharge set up between parallel electrodes in a bell jar containing a low pressure of inert gas such as argon. The ionized gas atoms bombard the target material, and the target atoms, in turn, are knocked out to be deposited on the substrate. A wide variety of materials can be sputtered, and, hence, these systems allow great versatility. Substrate temperatures are low and film quality, under the proper deposition conditions, is high. However, the deposition process can damage integrated devices (especially MOS), and sputtering is a relatively sophisticated process requiring a great deal of empirical effort to define the proper deposition conditions. The apparatus is also relatively expensive.

One further technique which we have found especially well adapted to meeting the film requirements for biomedical sensors is the radio-frequency glow-discharge technique (SWANN *et al.*, 1967; JOYCE *et al.*, 1967). In the glow discharge method, film deposition is achieved by

the decomposition of the appropriate gases in a radio-frequency excited glow-discharge plasma. The resulting films are of high quality and the film composition is easily controlled. Using the glow discharge technique a variety of insulating materials can be deposited. Furthermore, the system is relatively inexpensive to build and is easy to operate. Because we feel this technique is well suited for biomedical applications and because we have had extensive experience with it, this technique will be described in detail in the next section. The characteristics of the resulting films are then discussed in Section 6.

5. GLOW-DISCHARGE DEPOSITION OF INSULATING FILMS

A number of different types of insulating films can be formed using the glow-discharge method by suitable selection of the reactant gases. As illustrative examples, we consider the deposition of silicon dioxide and silicon nitride films. Both of these glassy materials have been successfully utilized for protection of biomedical sensors in our laboratory. To form silicon dioxide, two gases are used—silane* (SiH_4) and nitrous oxide (N_2O). Silicon nitride is obtained by using ammonia (NH_3) in place of nitrous oxide. The reactant gases are fed into a continuously pumped bell jar. The sample to be coated (substrate) is placed inside the bell jar. Surrounding the bell jar is a coil that is used to produce the radio-frequency field which decomposes the gases. The decomposed gases react in the glow-discharge plasma and are deposited onto the substrate. As will be shown below, the film properties are dependent on (1) the relative amounts of silane and nitrous oxide (or ammonia) in the bell jar, (2) the substrate temperature during the deposition, and (3) the applied r.f. power. The manner in which these deposition parameters are controlled is discussed in detail in the next section.

5.1 *System description*

A diagram and photograph of the system used for the glow discharge deposition of thin films are shown in Fig. 1. The 2-in. (5-cm) dia. bell jar is made of high purity quartz. The base plate, valves and gas connections are of stainless steel. The substrate is supported on a graphite pedestal which also acts as a substrate heater. The nichrome wire heater is potted inside the pedestal and is powered by a variable a.c. supply. The a.c. field of the heater has negligible effect on the characteristics of the plasma.

The substrate temperature is monitored by a chromel-alumel thermocouple imbedded in the pedestal at a point just below the top surface. Any temperature difference between the thermocouple reading and the actual substrate temperature was neglected. After stabilization the thermal drift was less than 2°C per hour.

The bell jar is evacuated by means of a rotary vacuum pump and the pressure in the bell is monitored with an NRC type 501 thermocouple vacuum gauge.† A Lepel‡ Model T5N, 5 kW radio-frequency oscillator supplies current to a four turn copper coil which surrounds the bell jar. The amount of r.f. power applied to the plasma is adjusted by varying the oscillator current. The position of the coil is varied to localize the plasma in the region above the substrate.

The silane, ammonia and nitrous oxide gases were purchased from the Matheson Company§ with purities of 99·9, 99·99 and 98·5 per cent, respectively. The reactant gases enter the bell jar at the top and flow downward over the substrate. Precision needle valves are used to control the flow rates of the gases, and the flows are measured using manometer-type flow meters. These meters are made of glass and the manometer legs are filled with Dow–Corning‖ silicone oil (type 705)—a liquid that has a very low vapour pressure and which does not react

* Silane is spontaneously combustible in air and care should be taken to evacuate all of the gas lines prior to the deposition.
† National Research Corp., Newton, Mass.
‡ Lepel High Frequency Labs, New York, N.Y.
§ Matheson Gas Products Co., Newark, California.
‖ Dow–Corning Company, Midland, Michigan.

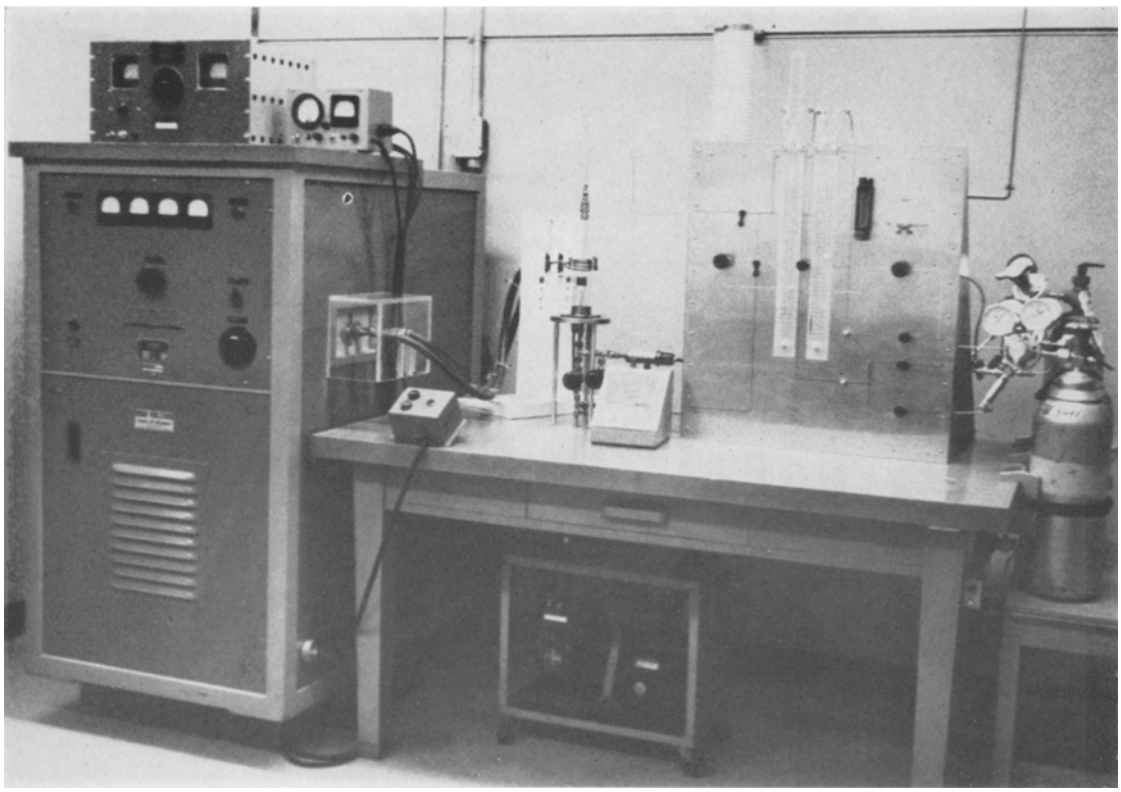

FIG. 1(b). The radio-frequency glow discharge system for thin film deposition.
(a) System diagram.
(b) Photograph of the system used.

(*facing p.* 342)

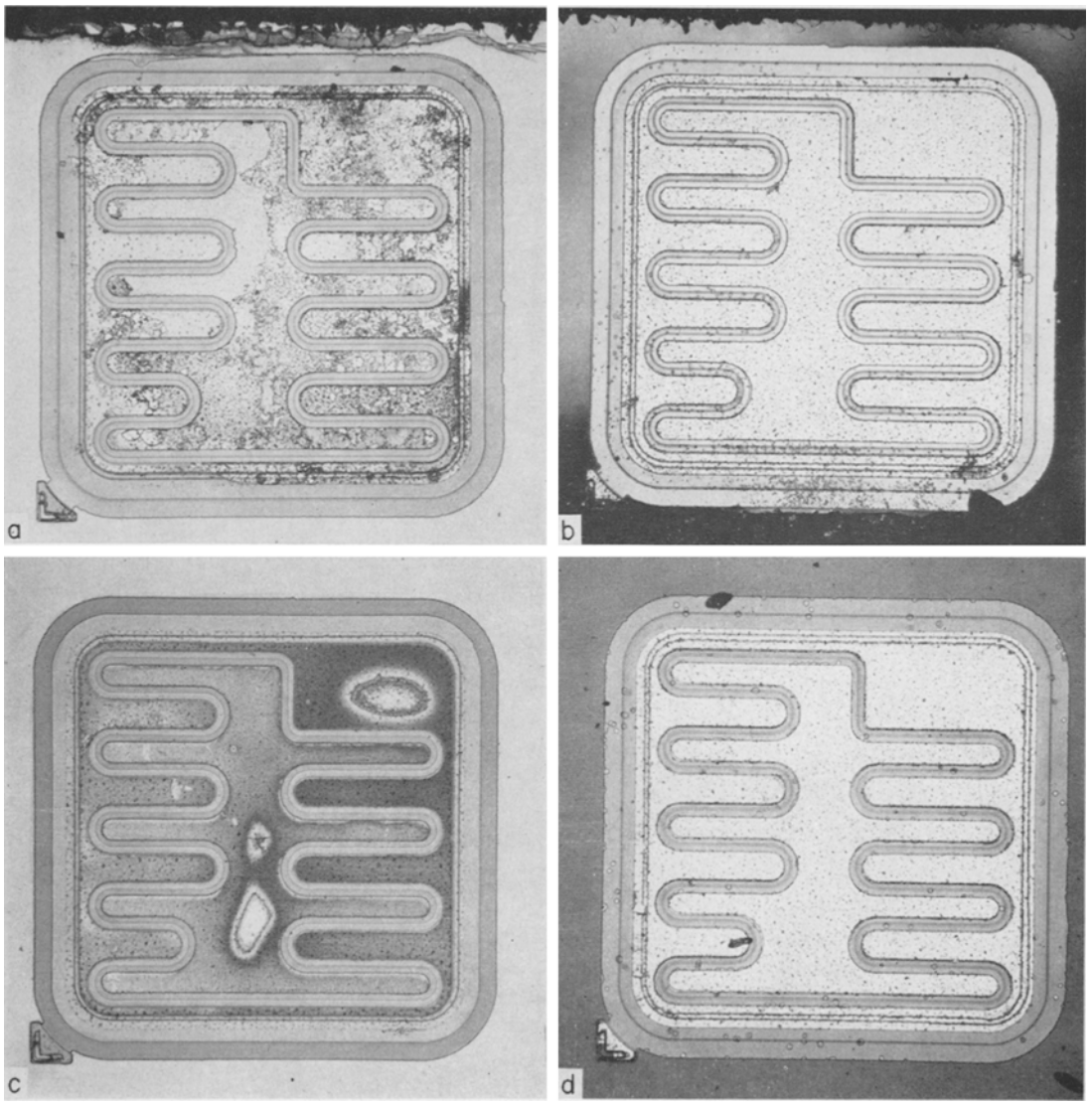

FIG. 4. Corrosion protection offered by thin films of glass deposited over conventional bipolar transistors.

(a) Unprotected transistor after 3 month subdural implantation.
(b) Transistor protected by 0·3 μm of deposited silicon dioxide after 4 month subdural implantation.
(c) Unprotected device after 15 h in physiological saline at 57°C.
(d) Transistor protected with 0·2 μm of deposited silicon nitride after 100 h in physiological saline at 57°C.

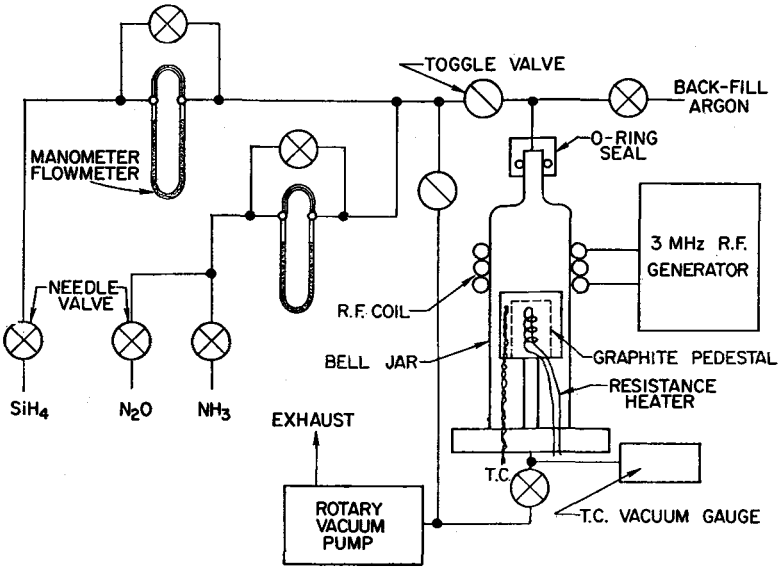

FIG. 1(a).

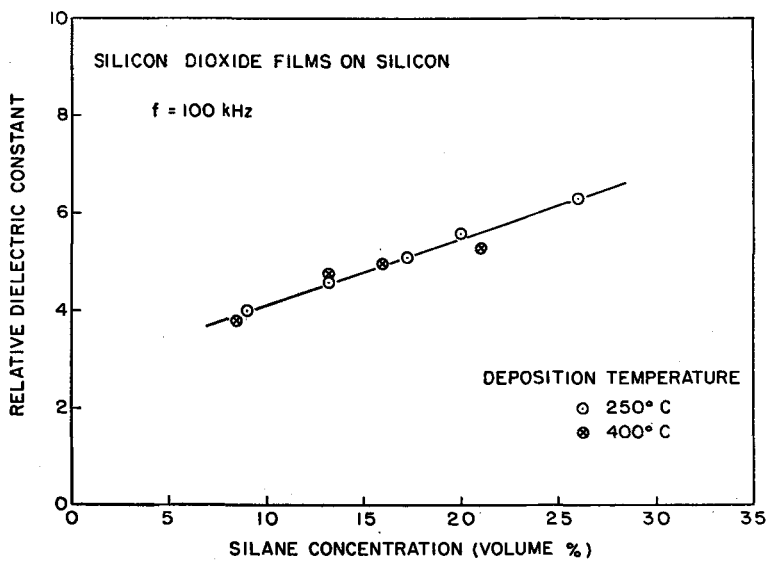

FIG. 2. Relative dielectric constant of silicon dioxide as a function of plasma silane concentration. Total gas flow rate was 100 cm^3/min.

chemically with the gases used. Due to the attendant difficulties in handling pure silane and ammonia, the flow rates of these gases could not be calibrated directly. Therefore, the flowmeters were calibrated using argon, nitrogen, oxygen, and nitrous oxide. Using appropriate correction factors for differences in viscosity, calibration curves for silane and ammonia were obtained. In the discussion that follows, the plasma gas composition is expressed in terms of percentage volumetric flow rates of the gases. The total flow rate was 100 cm^3/min in all cases.

5.2 *Effect of deposition parameters*

The effects of substrate temperature and gas composition (per cent silane) on film composition are clearly observed in the dielectric constant and infrared absorption spectra for these deposited films. The dielectric constants of the silicon dioxide films, prepared using various silane flow rates and substrate temperatures, were obtained by measuring the capacitance of metal–insulator–semiconductor structures having a known insulator thickness. The dielectric films were deposited on 0·01 ohm-cm, *n*-type silicon and aluminum dots (area = $7{\cdot}3\times 10^{-4}$ cm^2) were used as the metal electrodes. Approximately 15 capacitors were measured for each dielectric sample at a frequency of 100 kHz. Selected capacitors were measured between 10 Hz and 1 MHz to check for any variation in dielectric constant with frequency. None was observed.

The relative dielectric constant of the deposited silicon dioxide film is shown in Fig. 2 as a function of the silane concentration (expressed as a percentage of the total volumetric flow rate of 100 cm^3/min). The relative dielectric constant, ϵ_r, changes from 3·8 to 6·0 as the silane flow rate is varied between 8·5 and 26 per cent. This increase in ϵ_r is attributed to an increase in the amount of elemental silicon in the deposited film at the higher silane flow rates. At 8·5 per cent silane, the deposited silicon dioxide film has a relative dielectric constant equal to 3·8 which is similar to the value of 3·4–3·85 reported for thermally-grown silicon dioxide (BURGER, 1967).

Experimental data is given in Fig. 2 for substrate temperatures of 250°C and 400°C. Within the limits of experimental error (15–20 per cent), the relative dielectric constant is independent of the deposition temperature. Since most of the film properties are functions of

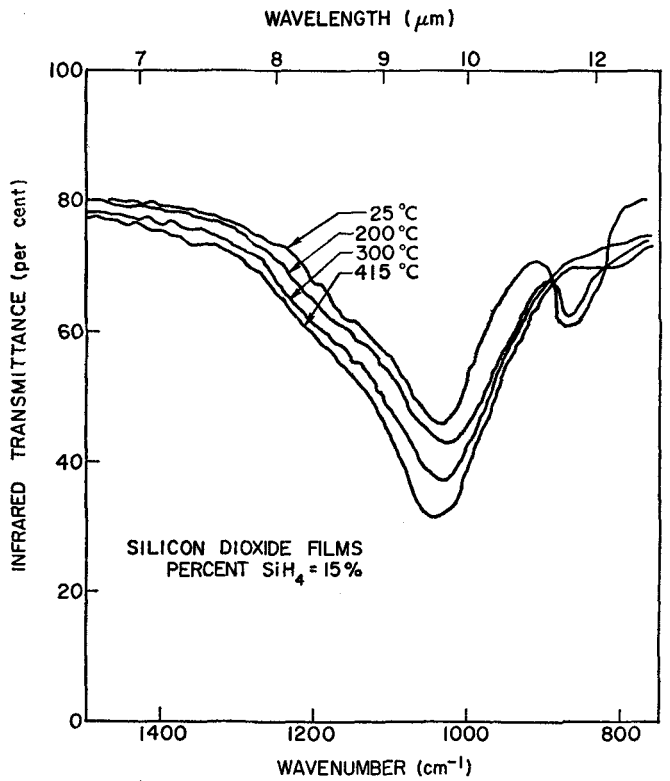

FIG. 3. Infrared transmittance of thin silicon dioxide films deposited at various substrate temperatures by the radio-frequency glow discharge technique.

temperature, the lack of any change in ϵ_r with temperature was an unexpected result. A possible explanation for this temperature independence may be that there are two compensating effects due to a simultaneous change in the film density and silicon content. Evidence for the latter change is seen in the infrared spectra shown in Fig. 3. As the substrate temperature increases from 200 to 400°C, the peak at 11·3 μm disappears, indicating a decrease in the amount of excess silicon in the film (PLISKIN and LEHMEN, 1965). From the slope of ϵ_r vs. per cent silane (Fig. 2), it is seen that the lower the silicon content of the film, the smaller the value of ϵ_r. However, the dielectric constant increases with an increase in film density as shown by the Clausius–Mosotti equation (KITTEL, 1956), and thus, at a constant silane flow rate, these two effects can compensate to yield a dielectric constant that is independent of substrate temperature. The third variable, applied r.f. power was constant for all experiments reported here.

Similar effects are observed for silicon nitride films. The variations of the dielectric constant and infra-red absorption with silane flow rate are given in the literature (SWANN *et al.*, 1967).

In general, a substrate temperature of at least 275°C is recommended when high quality glass films are desired. Furthermore, to avoid the partial conversion of the silicon nitride films to silicon dioxide a deposition temperature of 275°–300°C should be used.

6. FILM CHARACTERIZATION

6.1 *Mechanical properties*

The requirements placed on deposited dielectric films for use in biomedical sensors were given in Section 3. The first requirement, continuity, is satisfied by all glow-discharge deposited films. Film deposition occurs on all exposed surfaces, and film continuity over surface irregularities has been repeatedly demonstrated by corrosion studies both *in vitro* and *in vivo*. These studies are discussed below.

The adherence of glow discharge deposited films of silicon dioxide and silicon nitride to silicon is excellent. The films are hard and could not be scratched with the sharp tip of a pair of metal tweezers. Films up to 0·9 μm thick were deposited without cracking. Thermal cycling in hydrogen or either wet or dry nitrogen has in no way altered the adherence or film appearance for temperatures up to 700°C.

The adherence of glassy films of the type described here to metals is generally proportional to the strength of the metal–oxygen bonds formed (CUNNINGHAM, 1965), and this bond strength is related to the free energy of formation of the metal oxide. Thin glassy films do not adhere well to the more noble metals, which do not form oxides readily. However, we have observed in connection with the insulation of gold microelectrodes (WISE *et al.*, 1970) for neurological studies that as long as the width of the metalization is less than about 30 μm, adherence is adequate. The use of more extensive areas of noble metals should be avoided where possible.

In order to test the continuity, adherence, and corrosion protection of these thin films of glass *in vivo*, films of silicon dioxide and silicon nitride were glow-discharge deposited on silicon substrates containing double-diffused *n–p–n* transistors. The substrate dimensions were typically 3 × 1·5 × 0·2 mm. The transistors had aluminum contacts, and unprotected aluminum is known to be rapidly corroded by physiological saline. The substrates were carefully slipped beneath the dura of adult rabbits and were left chronically implanted for periods of from 3 to 4 months. They were then removed and cleaned. In all cases, film adhesion and continuity were adequate. Furthermore, none of the protected transistors showed any significant change in any electrical characteristic as a result of the implantation period. The glass deposition process did not alter the electrical characteristics of any of the transistors used.

6.2 *Chemical properties*

The materials silicon dioxide (fused quartz) and silicon nitride are generally accepted as inert in the body, and no evidence of any reaction with

body fluids has been observed. The films are chemically stable, and have provided excellent corrosion protection for solid state devices both in chronic implant situations and under simulated conditions in physiological saline. Figure 4a shows an unprotected bipolar transistor after a 3 month chronic implantation beneath the dura as described above. The aluminum metalization is severely corroded while the remaining device materials, silicon and silicon dioxide, are unaffected. Figure 4b shows a similar device, protected by a 0·3 μm-thick film of deposited silicon dioxide, after an implantation period of four months. The aluminum was not attacked; however, numerous small circles (1–5 μm dia.) appear on the surface where the deposited film has pulled free of the substrate. This is believed to have occurred primarily during removal and cleaning of the sample. No corrosion *in vivo* is observed at such sites, indicating film continuity. Figure 4c shows the results of accelerated aging at 57°C in physiological saline for an unprotected device. The sample was aged for 15 h and the aluminum metalization has almost entirely disappeared. Figure 4d shows a device aged 100 h at 57°C in the same solution. This device was protected with a 0·2 μm thick film of silicon nitride glass and there is no evidence of any corrosion. Similar results have been achieved with silicon dioxide films.

The silicon dioxide films are easily photoengraved using photoresist as an etch mask and hydrofluoric acid buffered with ammonium bifluoride as the etchant. Dimensions are commonly held to ± 1 μm. Silicon nitride films are more difficult to etch but photoengraving is still possible using phosphoric acid as the etchant (SCHNEER *et al.*, 1968).

6.3 *Electrical properties*

Organic polymers, including the silicones, are not noted for their resistance to moisture penetration. Based on measured vapour transmission rates, even polyvinylidene chloride, one of the least permeable organic materials, can transmit enough moisture to result in serious leakage currents along the surface of a coated device within minutes of exposure to an aqueous environment (WHITE, 1969). Since such surface leakage has not been observed in practice, White has proposed that the primary function of these organic coatings in preventing surface leakage is to prevent moisture from adsorbing or condensing on the surface rather than to block all permeation. Furthermore, it is noted (WHITE, 1969) that silicone coatings must be at least 10 μm thick to prevent serious leakage.

To test the electrical properties of glow discharge deposited films of silicon dioxide and silicon nitride, d.c. leakage measurements have been conducted under both dry and wet environments. The metal–insulator–silicon capacitors used for the dielectric constant measurements were utilized for dry d.c. conduction tests. Current flow through the film was measured with a Keithley Model 610A electrometer, while d.c. bias was applied to the capacitor with a variable supply. The d.c. conduction was found to be dependent on the deposition conditions. For either silicon dioxide or silicon nitride deposited at 400°C using a silane flow less than 23 per cent, typical conduction with ± 50 V applied is 10^{-8} A/cm^2. This leakage is sufficiently small that the operation of a typical integrated circuit transistor with an exposed area of approximately 10^{-4} cm^2 would not be affected. At silane flow rates greater than 23 per cent, the deposited dielectric films became excessively leaky. These films are such that the current varies as the square root of the applied voltage, and this dependence indicates that these films contain a high density of electron traps (SZE, 1967).

To more closely simulate conditions found in the body, two additional conduction tests were made. First, the leakage through films of silicon dioxide and silicon nitride deposited on silicon was measured using the experimental apparatus shown in Fig. 5a. The saline electrolyte forms one electrode, while the silicon substrate acts as the other contact. Measured leakage on the samples varied appreciably from sample to sample and increased with time. The data presented in Fig. 5b summarize the results for two typical samples.

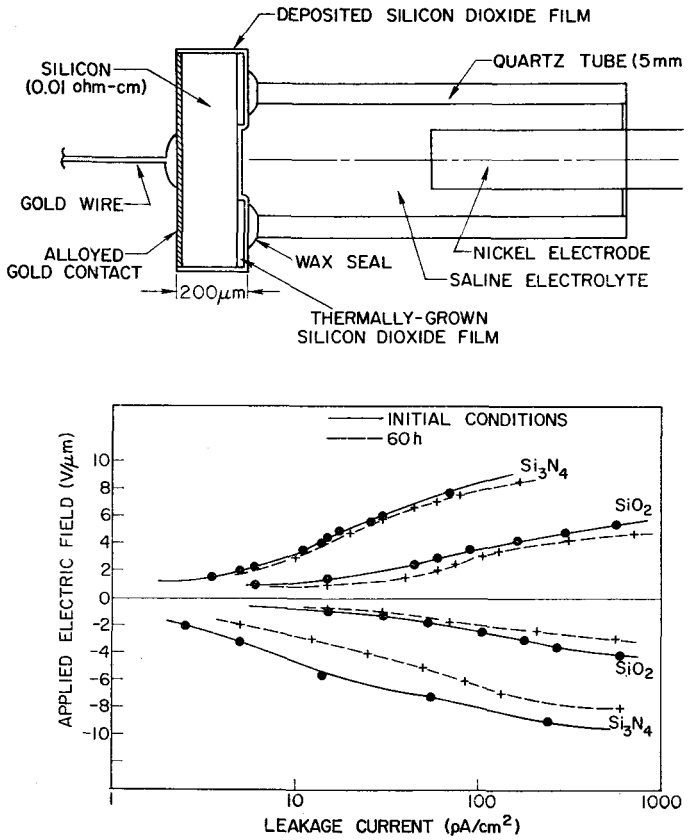

FIG. 5. Electrical properties of deposited films of silicon dioxide and silicon nitride. All measurements were performed at 24°C.
(a) Experimental apparatus used.
(b) Leakage current through the film as a function of the applied electric field.

The silicon dioxide and silicon nitride film thicknesses were 0·33 μm and 0·2 μm respectively. The deposition temperature was 300°C, and the silane percentages used were 11 and 20 per cent, respectively. Film growth rates under these conditions are 7·5 nm and 3·3 nm per minute respectively.

From the measured data, it is evident that these thin films develop appreciable d.c. leakage when potentials of a few volts are applied across them. Circuit tolerance of such leakage is obviously a function of the configuration and devices used, but leakage greater than 10^{-5} A/cm^2 may be considered harmful to transistor performance. Thus, although satisfactory at biases less than 1 V, potentials in excess of a few volts would result in excessive leakage currents which would prove harmful to both the circuit and, perhaps, to the physiological system. Leakage between parallel gold electrodes spaced 50 μm apart on an insulating substrate and protected from a saline environment by 0·3 μm of silicon dioxide has not indicated any significant leakage along the substrate surface but rather conduction through the electrolyte. The a.c. properties of these films are not degraded appreciably by the saline environment.

7. SPECIFIC APPLICATIONS

7.1 *Microelectrode fabrication*

Thin layers of glass have been used for many years as a means of insulating the metal micro-

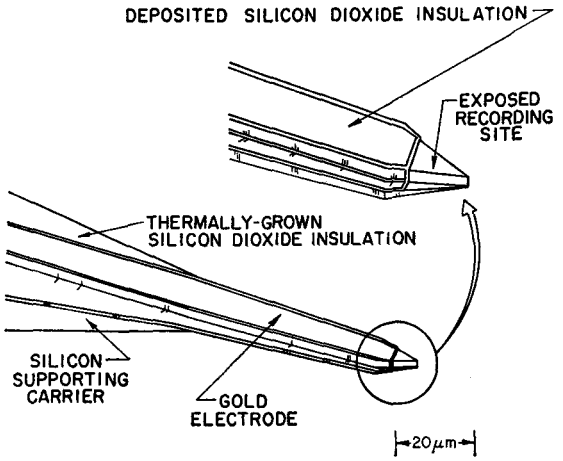

FIG. 6. An extracellular microelectrode for use in neurophysiology insulated with a deposited film of silicon dioxide.

electrodes and micropipettes in common use for recording biopotentials in neurophysiology (FRANK and BECKER, 1964). A disadvantage of the techniques used to fabricate these electrodes has been the inability to accurately control the size of the exposed recording sites. However, a recent approach (WISE, 1970) to electrode fabrication based on integrated circuit technology promises to overcome this disadvantage and illustrates one important application of deposited insulating films. The structure is shown in Fig. 6. The gold electrode is supported on a silicon carrier and is insulated from the carrier by a film of thermally-grown silicon dioxide 1 μm thick. The electrode is insulated from the extracellular electrolyte by a layer of glow-discharge deposited silicon dioxide, which is selectively removed from the electrode tips to precisely define the recording areas. Recording sites as small as 15 μm^2 have been achieved, and these electrodes have successfully isolated single units in cat auditory cortex. The use of deposited silicon dioxide films together with photoengraving technology allows the mechanical and electrical characteristics of the electrodes to be designed for any given application. As with other integrated biomedical sensors under development, film deposition is an essential part of the fabrication sequence.

7.2 *Other sensors*

It has been known for some time that certain doped glasses exhibit membrane potentials which are selective to specific ions in a surrounding electrolyte. These membranes have been studied extensively (EISENMAN, 1962; MOORE, 1968) and have been used to measure ion concentrations *in vivo*. An example is boron doped glass, which is specific to sodium. Boron is a commonly used dopant in integrated circuit fabrication, and the development of small ion selective concentration monitors in close proximity with metal microprobe arrays is a possibility.

Sensors designed specifically for the measurement of arterial and venous pressure and consisting of diffused silicon resistors in a thin silicon diaphragm (TUFTE *et al.*, 1962) promise to combined small size with stability and reliability *in vivo*. The resolution of 1 mm Hg pressure variations with diaphragms no larger than 1mm in diameter requires a diaphragm thickness of about 4 μm. A thin film of deposited silicon dioxide or silicon nitride over the resistors and other circuit elements present should protect and minimize any electrical surface leakage on these sensors without affecting their mechanical properties. Such diaphragm transducers are now being developed in our laboratory.

8. CONCLUSIONS

Thin films of silicon dioxide and silicon nitride are an important part of integrated circuit technology and should find wide application as this technology is applied to the fabrication of biomedical sensors. Such films are especially suitable for sensors since they can be deposited in thin, uniform layers and can be controllably etched using photoengraving techniques. The requirements on film quality for use in biomedical sensors have been discussed and the available deposition techniques have been described. One method—the r.f. glow-discharge technique—has been described in detail. The mechanical and chemical properties of thin (0·3 μm) deposited silicon dioxide and silicon nitride films have been found to be acceptable for sensor use. The d.c. electrical leakage of such thin deposited films can be excessive when applied potentials exceed 1 V, however. In such applications thicker films and an additional silicone coating should be used where possible to minimize leakage and protect against abrasion *in vivo*.

Acknowledgement—The authors express their sincere appreciation to Dr. WILLIAM H. DOBELLE of the Division of Artificial Organs, University of Utah, for his help in implanting the solid-state devices used in connection with the *in-vivo* tests reported in this paper.

REFERENCES

BRALEY, S. (1968) The silicones as subdermal engineering materials. *Ann. N.Y. Acad. Sci.* **146,** 148–157.

BURGER, R. M. and DONOVAN, R. P. (1967) In: *Fundamentals of Silicon Integrated Device Technology*, Vol. 1. Prentice-Hall, Englewood Cliffs, New Jersey.

CUNNINGHAM, J. A. (1965) Expanded contacts and interconnections to monolithic silicon integrated circuits. *Solid-St. Electron.* **8,** 735–745.

EISENMAN, G. (1962) Cation selective glass electrodes and their mode of operation. *Biophys. J.* **2,** 259–311.

FRANK, K. and BECKER, M. C. (1964) Microelectrodes for recording and stimulation. In: *Physical Techniques in Biological Research*, Vol. 5. (Ed. W. L. NASTUK) Chapter 2. Academic Press, New York.

FRANK, R. K. and MOBERG, W. L. (1970) Reactively sputtered silicon oxynitride as a dielectric material for metal–insulator–metal capacitors. *J. Electrochem. Soc.* **117,** 524–529.

HALPERN, B. D. (1968) Polymers in medicine and surgery—a survey. *Ann. N.Y. Acad. Sci.* **146,** 193–201.

JOYCE, R. J., STERLING, H. F. and ALEXANDER, J. H. (1967–8) Silicon oxide and nitride films deposited by an r.f. glow-discharge. *Thin Solid Films* **1,** 481–494.

KERN, W. and HEIM, R. C. (1970) Chemical vapor deposition of silicate glasses for use with silicon devices. *J. electrochem Soc.* **117,** 562–573.

KITTEL, C. (1956) *Introduction to Solid-State Physics*, 2nd Edn, p. 163. Wiley, New York.

LEVINE, S. N. (1968) Survey of biomedical materials and some relevant problems. *Ann. N.Y. Acad. Sci.* **146,** 3–10.

MOORE, E. W. (1968) Cation measurements in biological materials. *Ann. N.Y. Acad. Sci.* **148,** 93–109.

PLISKIN, W. A. and LEHMEN, H. S (1965) Structural evaluation of silicon oxide films. *J. electrochem. Soc.* **112,** 1013–1019.

ROBB, W. L. (1968) Thin silicone membranes—their permeation properties and some applications. *Ann. N.Y. Acad. Sci.* **146,** 119–137.

SCHNEER, G. H., VAN GELDER, W., HAUSER, V. E. and SCHMIDT, P. F. (1968) A metal–insulator–silicon junction seal. *IEEE Trans. Electron. Dev.* **ED-15,** 290–293.

SWANN, R. C. G., MEHTA, R. R. and CAUGE, T. P. (1967) The preparation and properties of thin film silicon–nitrogen compounds produced by a radio frequency glow discharge reaction. *J. electrochem. Soc.* **114,** 713–717.

SZE, S. M. (1967) Current transport and maximum dielectric strength of silicon nitride films. *J. appl. Phys.* **38,** 2951.

TUFTE, O. N., CHAPMAN, P. W. and LONG, D. (1962) Silicon diffused-element piezoresistive diaphragms. *J. appl. Phys.* **33,** 3322–3327.

WARNER, R. M. and FORDEMWALT, Eds. (1965) *Integrated Circuits—Design Principles and Fabrication.* McGraw-Hill, New York.

WHITE, M. L. (1969) Encapsulation of integrated circuits. *Proc. IEEE* **57,** 1610–1615.

WISE, K. D., ANGELL, J. B. and STARR, A. (1970) An integrated-circuit approach to extracellular microelectrodes. *IEEE Trans. Biomed. Engng* **BME-17,** 238–247.

YORK, D. B. (1963) Properties of evaporated thin films of SiO. *J. electrochem. Soc.* **110,** 271–275.

PELLICULES DE VERRE MINCES ET LEUR APPLICATION À DES SENSEURS BIOMÉDICAUX

Sommaire—Les pellicules de verre minces sont intéressantes comme moyens de protection de cricuits intégrés employés dans les systèmes biologiques et sont particulièrement appropriés à l'emploi avec les senseurs biomédicaux. Ils offrent des avantages par rapport aux pellicules polymériques car ils peuvent être déposés sous forme de couches minces, uniformes et peuvent

être photogravées de façon classique. Cet article passe d'abord en revue les qualités de pellicule nécessaires à des pellicules de verre dans ces applications, puis les techniques disponibles pour la déposition de pellicule. On décrit en détail une technique qui a été trouvée être particulièrement appropriée au travail biomédical et l'on discute la caractérisation des pellicules qui en résultent.

Des pellicules de bioxyde de silicium et de nitrure de silicium de 0,2 à 0,3 μm d'épaisseur ont montré une adhésion et une continuité adéquates pour l'emploi avec des senseurs fabriqués par la technologie de circuits intégrés. De plus ils ont offert une excellente protection contre la corrosion de transistors non-encapsulées implantés dans le cerveau pendant des périodes de 4 mois. De telles pellicules de verre offrent également une bonne isolation électrique pour des tensions appliquées inférieures à 1 V; cependant pour des tensions plus élevées, des pellicules plus épaisses sont nécessaires pour éviter des courants de fuite excessifs. On considère des applications à des senseurs spécifiques.

DÜNNE GLASSFILME UND IHRE VERWENDUNG FÜR BIOMEDIZINISCHE MESSFÜHLER

Zusammenfassung—Dünne Glassfilme besitzen als Mittel zum Schutz integrierter Schaltungen in biologischen Systemen eine Anziehungskraft und sind für Gebrauch mit biomedizinischen Messfühlern besonders geeignet. Sie bieten gegenüber Polymerfilmen Vorteile, da sie in dünnen, gleichmässigen Schichten abgelegt und in üblichen Verfahren im Klischee hergestellt werden können. Dieser Bericht behandelt zunächst die Bedingungen der Filmeigenschaft im Gebrauch von dünnen Glassfilmen für diese Anwendungszwecke und behandelt dann die erhältlichen Verfahren für Filmlagerung. Eine Technik, welche für biomedizinische Arbeit besonders geignet gefunden wurde, wird in Einzelheiten beschrieben und die Charakterisierung der sich ergebenden Filme wird besprochen. Filme aus Siliziumdioxyd und 0,2–0,3 μm dicken Siliziumnitrid haben ausreichende Adhäsion gezeigt und Beständigkeit für Gebrauch mit Messfühlern, welche mit integrierter Schaltungstechnologie hergestellt waren.

Darüberhinaus haben sie ausgezeichneten Korrosionsschutz für ungekapselte, ins Gehirn gepflanzte Transistoren für Dauern von 4 Monaten geboten. Diese Glassfilme geben auch gute elektrische Isolation für verwendete Spannungen von weniger als 1 V. Wenn allerdings hönere Spannungen vorhanden sind, werden dickere Filme benötigt, um übermässige Streuströme zu vermeiden. Es werden Verwendungszwecke für spezifische Messfühler in Betracht gezogen.