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Water-Soluble Glass Substrate as a Platform for Biodegradable Solid-State Devices

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ABSTRACT A biodegradable glass material is utilized as a novel functional element of solid-state devices. A water-soluble borate glass substrate serves as the structural platform on which thin film device is built. The glass substrate completely dissolves in a saline solution in approximately 40 h. Intentional failure of the spiral device (RLC resonator circuit) as a result of rapid structural disintegration by dissolution is demonstrated in DC, AC, and RF ranges that agrees well with simulation. Adopting water-soluble glass elements is expected to be a viable approach to develop reliable all-inorganic biodegradable devices that are fully functional during an intended operational lifetime followed by rapid degradation.

INDEX TERMS Biodegradation, bioactive glass, wafer, impedance, resonance.

I. INTRODUCTION

Biodegradable devices are in the emerging concept stage, involving temporary implanted devices with limited lifetime. The devices are to be fully functional during an intended lifetime after which they lose functionality via dissolution. A wide range of biomedical applications that can potentially benefit from short-term implants was reviewed [1]. An increasing need for biodegradable devices has been addressed in the literature, focusing on passive devices including metal or polymer stents [2], [3].

Accordingly, the concept of active biodegradable devices including electronics and sensors have been demonstrated [5]–[11]. These devices are composed of organic materials (e.g., biodegradable polymers) and biodegradable metals/semi-conductors. Typically, the organic elements serve as the structural substrates and insulation layers for biodegradation, except in one case that used metal foils [12]. Organic materials are degraded by bulk hydration that continuously changes the original bulk properties during degradation. Retaining the structural integrity of substrates and the insulating property of insulation layers is critically important to maintain the device reliability during intended use [12]–[15]. Inorganic materials are superior in this respect since the hydrolysis occurs only at the surface, allowing the retention of bulk mechanical, thermal and chemical properties until those elements are largely consumed.

We report a new approach that uses biodegradable glasses as the substrate. This is to aim devices prepared compatibly with traditional solid-state processes to function reliably during lifetime followed by a rapid failure. Simple thin film devices were prepared on the glass substrates to demonstrate the device behavior in various frequency ranges during the course of structural degradation in solution.

II. DEVICE OPERATION CONCEPT

The biodegradable glass materials, commonly known as bioactive glasses, are a class of biomaterials commonly used in tissue engineering that react with surrounding tissue to become a part of the tissue itself [16], [17]. Their reaction rates can be controlled by their compositions ranging from

slow (e.g., ∼ 10 % weight loss per day with silicate-rich glass) to fast rate (e.g., ∼ 50 % weight loss per day with borate-rich glass) [18]. Traditional silicate-based biodegradable glasses are mainly composed of the oxides of silicon, sodium and calcium. The reaction involves the initial dissolution of sodium and subsequent formation of silicate-rich gel layers. Calcium ions then react with phosphates from the body fluid, which leads to the formation of a porous hydroxyapatite scaffold which promotes the in-growth of tissue. In borate-based glasses, where silicate is replaced with borate, the highly soluble borate glass disintegrates much faster compared to silicate [18]–[20]. In the absence of calcium, the entire material eventually dissolves and is absorbed without forming any residual structure of hydroxyapatite.

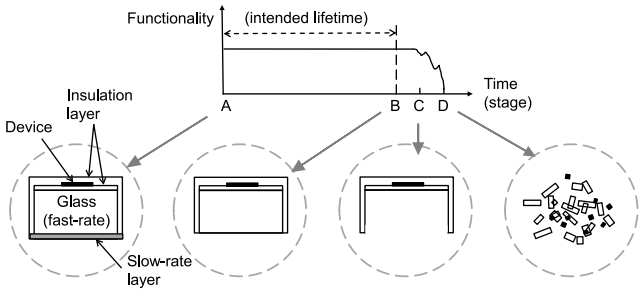


FIGURE 1. Conceptual diagram of device functionality versus time (i.e., "rapid failure" after the intended operational lifetime): initial structure (stage A), dissolution of slow-rate glass film (e.g., several months) (stage B), dissolution of fast-rate glass substrate (e.g., 1-2 days) (stage C) and physical disintegration of crust insulation and device layers (stage D).

Figure 1 shows the expected behavior of the device exposed to body fluids. The major portion of substrate is made of a fast reacting glass material. The bottom layer is a slowly reacting glass film. It is consumed during a presumed lifetime (i.e., A to B stage) with the active device being functional due to its top insulation layer. The lifetime can be manipulated by adjusting the compositions and thickness of the bottom layer. The dissolution of the entire glass substrate occurs rapidly once exposed to solution, leaving only a top layer. This crust layer is expected to structurally disintegrate without mechanical support. It is critically important to design the duration of failure (i.e., B to D stage) as short as possible to prevent false results and safety concerns caused by continued use.

Common silicon-based films (e.g., SiO_2 and Si_3N_4) can serve as the top insulation layer since these have been used extensively for insulating liquid sensors including ion-sensitive field-effect transistors [21]. Silicon-based biodegradable devices was first suggested by Canham *et al.* [22], [23]. Although the dissolution rates are slow, the fragments of silicon-based crust layer eventually decompose to silicic acid to become a metabolite or excreted from body. Therefore, it is highly feasible to implement silicon-based biodegradable devices having proper form factors (i.e., large surface-to-volume ratio) as demonstrated by porous silicon drug delivery vehicles [24] and silicon-based biodegradable electronics [8], [12], [14], [25].

III. EXPERIMENTAL DETAILS

A. GLASS SUBSTRATE PREPARATION

The glass substrates were manually prepared. Borate glass was prepared by melting sodium tetraborate (12305, Alfa Aesar) in a platinum crucible at 1,000 °C for 30 minutes. The melt was poured into a stainless steel cylindrical mold and annealed at 450 ° C for 30 minutes to form a glass rod (14 mm diameter, 30 mm). Sliced circular substrates were mounted on aluminum holders and ground using silicon carbide foils (180 to 1200 grit sizes, Struers) followed by polishing with a diamond media (DP spray P, Struers). The final surface roughness was approximately 0.25 um measured by atomic force microscopy (Nanoscope IIIa, Digital Instrument).

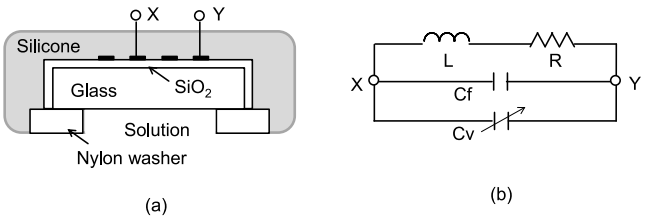


FIGURE 2. (a) Cross-sectional view of the spiral thin film device after encapsulation to demonstrate the stage B to D in Fig. 1. (b) Simplified model of the device (L: inductance of spiral film, R: series resistance along spiral film, C_F: fixed capacitance through SiO_2 and silicone; C_V: variable capacitance through glass and solution). Au film can be replaced with biodegradable film (e.g., Ag, Mg) for actual applications.

B. DEVICE FABRICATION

The prototype device in Fig. 2(a) without the slow-rate layer is intended to demonstrate the B to D stage in Fig. 1. The top and side surfaces of the glass substrates were coated with a silicon dioxide (SiO_2) blanket insulation layer (1 um thick) by sputtering (Discover 18, Denton Vacuum). A spiral coil device (2.4 mm diameter, 0.2 mm line width, 0.15 mm line spacing) was fabricated with a gold layer (120 nm thick) deposited by sputtering (Bio-Rad E5400 flash coater) and patterned using a stainless steel shadow mask (4 mil thick). An SMA RF connector (0731000115, Molex) was connected to the device with silver paste (Z04969, SPI). The entire SMA connector was encapsulated in silicone (3140, Corning). A nylon washer (11 mm inner diameter, 0.5 mm thick) was glued at the bottom surface with silicone to ensure a fixed exposed area.

C. MEASUREMENT SETUP

All dissolution tests were conducted in a simulated body fluid (SBF) solution (pH 7.4) [26]. The device was submerged in an 1 L solution placed in an incubator (Heratherm, Thermo Scientific) at 37 °C and tested for dissolution behavior. For monitoring the change in DC

and AC impedance levels during dissolution, the device was connected to an electrochemical potentiostat (Femtostat, Gamry). The scattering parameter (S11) was monitored in an RF frequency range with a vector network analyzer (VNA) (E8753, Agilent). A 50 ohm cable was used to connect the device to the VNA. Since the VNA was calibrated only at the port while the device was present after the cable, the 1-port 3-term error model [27] was used to obtain the actual response of the device.

D. SIMULATION

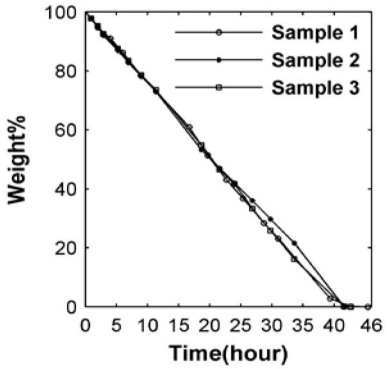
Microwave studio (Computer Simulation Technology) was used to simulate the resonance behavior of the RLC model shown in Fig. 2(b). Inductance L was calculated by a data fitted monomial expression [28], whereas series resistance R was measured with a multi-meter. Complex dielectric constants of the glass and solution were calculated from S11 parameter using waveguide method with the same VNA [29]. Dielectric properties of SiO_2 and silicone were taken from [30] and [31]. Utilizing these values, two parasitic capacitive elements, C_F and C_V, were calculated by a known method [32]. The calculated resonance frequency was 2.1 GHz similar to the range of other biodegradable resonance devices [5], [8], [10].

IV. RESULTS AND DISCUSSION

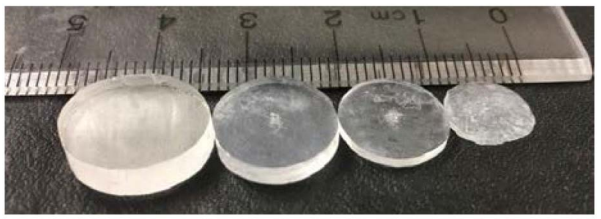
A. SUBSTRATE DISSOLUTION

Fig. 3(a) shows the rapid weight loss during the course of dissolution of bare borate glass substrates without any coated layer. The linear loss rate is approximately 60 % weight loss in a single day (or 5.5 mg/hour with an initial surface area of 4.35 cm^2). As expected, this rate is higher than those of the borate-rich glasses containing silicate [18]. Particularly, since this substrate does not contain any calcium compound, the entire substrate is dissolved without residual structure (i.e., 100% weight loss) in approximately 40 hours. Fig. 3(a) represents the rapid failure after a presumed lifetime (i.e., t = 0 hour being the onset of stage B in Fig. 1) since the bare substrates has no other coated layer.

Although each substrate prepared manually was used as a single "dice" at this phase, it is expected that large diameter "wafers" can be mass-produced by a refined manufacturing procedure. Therefore, the biodegradable glass substrate is considered as an excellent candidate that maintains the structural integrity to support the devices during use followed by a rapid disintegration.



(a)



(b)

FIGURE 3. (a) Dissolution behavior of bare glass substrates (2.7 mm thick, 14 mm diameter, no coating/encapsulation) in a simulated body fluid (SBF) solution (pH 7.4 at 37 °C). (b) Photographs of glass substrates taken at various stages of dissolution (from the left: original, 12-hour, 24-hour and 36-hour).

B. DC RESISTANCE AND AC IMPEDANCE

Changes in DC resistance and AC impedance of the devices were measured during substrate dissolution. Fig. 4 shows that both DC and AC values stay at a certain range followed by a steep decrease after one day. This change clearly demonstrated the expected behavior of rapid failure. Once the significant glass portion was consumed, the device collapsed and eventually short-circuited through the conductive solution. As expected, the device is largely resistive at this frequency range evidenced by the negligible contribution of reactive elements by comparing the DC and AC levels.

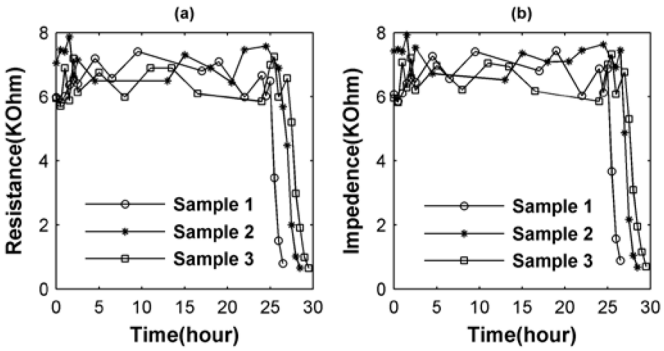


FIGURE 4. Temporal change of (a) DC resistance (with 0.1 V) and (b) AC impedance (with 0.1 V_{RMS}, 1 kHz) of spiral thin film devices during the dissolution of glass substrate (2.2 mm thick, 14 mm diameter). This demonstrates the device performance during the stage B - D in Fig. 1.

Although a simple metal thin film device is demonstrated in this proof-of-concept stage, silicon-based devices is expected to be biodegradable based on this approach. Two possible approaches include the amorphous thin film devices and the silicon-on-insulator (SOI) structure utilizing biodegradable glass wafers. Traditional substrates (e.g., single crystal silicon, Pyrex glass) are not appropriate for the intended sudden failure due to their extremely low dissolution rate. Therefore, biodegradable electronics

with a higher electron mobility are anticipated that can cover a wider bandwidth than that of biodegradable polymer devices.

C. SCATTERING PARAMETER (S11)

Fig. 5(a) shows the temporal change of the S11 parameter. Upon reaching the presumable lifetime (i.e., t = 0 hour in Fig. 5 or stage B in Fig. 1), the resonant peak started to shift toward the lower frequency side and become negligible (i.e., less than -5 dB) after a day. The shifting of the resonance was a result of increasing parasitic capacitance through the glass substrate (C_v) that was being replaced with the capacitance through the solution with a higher real dielectric constant. In addition, this rich electrolytic solution was a lossy medium that lowered the resonance peak magnitude.

The variation in resonance frequency and peak magnitude is plotted in Figs. 5(b) and (c) with respect to time. A simulation was conducted based on the varying volume of glass estimated from the weight loss observation. Both experimental and simulation data show a similar trend and agree well taking into account discrepancies originated from the simplicity of model and the variations in the device preparation.

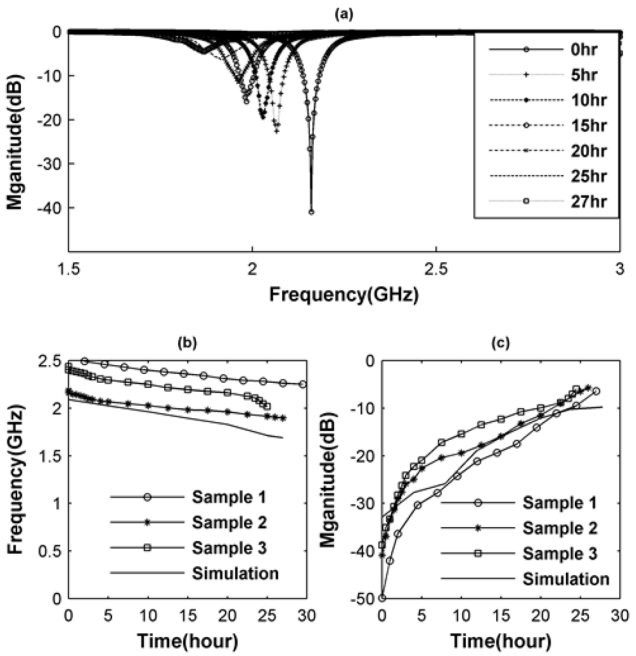


FIGURE 5. Temporal change of resonance behavior of spiral thin film devices over the course of substrate dissolution (2.2 mm thick, 14 mm diameter): (a) Scattering parameter (S11) spectrum, (b) Resonant frequency and (c) Peak magnitude. This represents the stage of B - D in Fig. 1. After 30 hours, noticeable resonances (less than 5 dB) were not observed.

The resonance frequency of the reported device can be easily manipulated with varying spiral designs and the size of the substrate depending on future needs. Operating frequencies of implantable devices can range from kHz to several GHz range, depending on various factors including transmission distances, implanted depths, transmitter/receiver sizes and applications (e.g., signal transmission, power delivery, and thermal therapy). In this report, tests were done in a broad frequency range to explore the feasibility of various application scenarios including simple DC (e.g., device bias), low frequency (e.g., sensing) and high frequency (e.g., RF transmission).

V. CONCLUSION

The rapid failure of device functionality (i.e., in one day after presumed lifetime) built on biodegradable glass substrates was demonstrated over a wide frequency range, which is in good agreement with simulation based on a simple model. The inorganic biodegradable substrates are more compatible with traditional device processes than other types of biodegradable substrates. It is expected to sustain the structural integrity without compromise in device reliability during operation followed by a rapid disintegration. Therefore, the use of water-soluble glasses is expected to be a viable approach to develop reliable biodegradable devices and systems.

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