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Mechanical sensors

Glass-Based Biodegradable Pressure Sensor Toward Biomechanical Monitoring With a Controllable Lifetime

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Abstract—A new class of potentially implantable solid-state sensors is demonstrated utilizing biodegradable glass as the main structural material. The device behavior is manipulated via chemical decomposition, and then physically disintegrated in a controlled manner. It is based on the capacitive sensing mechanism, comprising an elastic insulator between two borate-rich glass substrates. This mesoscale pressure sensor is characterized by a range of pressure of up to 14 MPa in a phosphate buffer solution environment. The sensor exhibits good sensitivity and reversibility responding to compressive pressures and remains fully functional before a desired, sudden failure caused by dissolution. The operational lifetime can be modified by altering the chemical composition or thickness of the biodegradable glass component. The proposed device concept is a viable option toward various temporary implantable devices without needing an additional surgical procedure to remove them after their duty.

Index Terms—Mechanical sensors, biodegradable, glass, implantable, pressure, sensor.

I. INTRODUCTION

Various implantable devices, such as cardiac pacemakers and nerve stimulators, are widely used as permanent implants. In contrast, temporary implants are also necessary for short-term medical intervention and monitoring scenarios [1], [2]. However, if not surgically removed after their intended use, these devices are left inside the body and may cause future safety concerns. The concept of biodegradable device is an advanced and promising approach for this challenging condition. Ideally, biodegradable devices should be fully functional during their desired operational lifetime after which they lose the functionality by degradation and consequent absorption within the body without adverse effects. Recently, many electronic active devices have been proposed due to recent advancements in biodegradable materials, both organic [3], [4] and inorganic [5], [6].

Bioactive glass is one of inorganic biomaterials commonly used in tissue engineering, wherein the term "bioactive" means the ability to react with surrounding tissue to remain as part of the tissues [7], [8], [9]. Typical silicate-rich glass materials are not biodegradable to a practical degree, although pioneering work investigating the bioactivity of silicon had been reported almost three decades ago [10]. In contrast, the reaction rate of the bioactive glasses can be controlled from days to months by changing the composition, particularly with borate-rich and phosphate-rich glass compositions compared to those of common silicate-rich glasses. In addition, unlikely the organic counterparts, the bulk electrical, mechanical property of glass elements is completely retained until most portion of device is consumed since the reaction process happens at the glass surface. Therefore, the bioactive glass is an excellent candidate for the key structural material for biodegradable solid-state electronic devices with a controlled lifetime.

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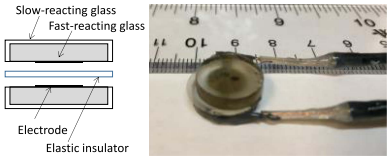

Fig. 1. (a) Exploded view of the biodegradable capacitive pressure sensor. (b) Photograph of an assembled sensor.

Our group demonstrated a glass-based *L-C* (inductor-capacitor) resonator as a proof-of-concept passive radio frequency identification to show an intended sudden signal loss [11]. Based on a similar mesoscale (millimeter range) glass disc platform, this work reports an active biodegradable, and potentially bioactive, sensor for biomechanical monitoring with a relatively high-pressure range. Many works on microsystem-based sensors have mostly focused on smaller ranges for intraorgan (an order of several-ten kPa) [12], [13], [14], [15]. Biodegradability of these microscale devices is preferable despite their tiny size and/or footprint. However, degradability is inevitable for mesoscale devices since the prolonged presence of objects with considerable sizes may give rise to biomechanical issues, electromagnetic interference, and latent complications. Therefore, as an exemplary application of the biodegradable glass material to mesoscale devices, we designed and characterized a pressure sensor with a wide dynamic range (0–14 MPa) toward potential orthopedic purposes such as spine monitoring [16].

II. METHODS

A. Operational Principle

The proposed device structure in Fig. 1 is based on a parallel plate capacitor where the application of an external compressive force

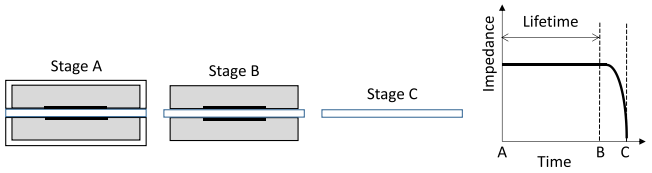

Fig. 2. Prospective behavior: After an intended operational lifetime (e.g., several months for A–B), the device loses its functionality within a short period (e.g., days to a week for B–C).

decreases the thickness of the elastic insulator between two electrodes within its compressive limit. Increase of the capacitance decreases the impedance of the capacitive device

$$C = \varepsilon_o \varepsilon_r A/d \quad (1)$$

$$Z = 1/wC \quad (2)$$

where C, ε_o, ε_r, A, d, Z, and w represent the capacitance, vacuum permittivity, relative dielectricity of insulator, electrode area, insulator thickness, capacitive impedance, and angular frequency, respectively. Additionally, this capacitive modality is compatible with wireless sensing after implantation in a later stage of development based on the *L-C* resonance. Capacitance change responding to the varying compressive pressure shifts the resonant frequency of the device, which can be translated to the pressure change.

Fig. 2 shows the prospective behavior of the proposed sensor. The major portion of the two identical substrates is made of a fast-reacting glass material coated with a thin film of slow-reacting glass with a negligible volume. The two metal electrodes are also biodegradable. The slow-reacting glass portion is first consumed in the body fluid within the time frame between stages A and B. This duration is the intended operational lifetime during which the device stays fully functional. The lifetime can be manipulated by changing the composition of the encapsulating thin slow-reacting glass layer. Altering the thickness of this layer is an alternative option to control the lifetime.

The interval between B and C indicates an unstable device operation due to rapid structural deformation, during which the fast-reacting glass layer occupying most of the device volume is reacting. The rapid failure before the stage C represents the steep decrease of impedance caused by short-circuiting of two electrodes through a conductive body fluid. This sudden failure plays the role of preventing the continued use of unstable medical devices beyond the prescribed lifetime. After the complete consumption of all glass and metal portions, the thin layer of biodegradable elastomer is the only component that is also to be eventually degraded.

B. Materials and Device Preparation

The device dimensions were decided to obtain a sensitivity over an order of 10 MPa. This range is to cover the pressure applied to the lumbar of an adult during various movements [16]. In this proof-of-concept stage, there is no encapsulating slow-reacting film deposited over the borate glass substrate. Therefore, this work was focused on demonstrating the device performance for a short amount of time depicted between stages B and C in Fig. 2. As described in our previous study [11], a borate glass was prepared by melting $Na_2B_4O_7$ (anhydrous sodium tetraborate, 99.5%, Alfa Aesar) to make a simple binary composition of 69.2:30.8 (B_2O_3:Na_2O wt%) without other elements. As a comparison, the composition of 45S5, one of the most common silicate-rich glasses with a slow rate, is 45% SiO_2, 24.5% CaO, 24.5% Na_2O, and 6% P_2O_5 (wt%), without B_2O_3. The fast-reacting borate glass substrates were prepared manually in form of discs as reported before [11].

The elastic insulator (polydimethyl siloxane or PDMS, Sylgard 184, Dow Corning) and metal (gold) materials used in this study are not biodegradable, which can be easily replaced with their biodegradable counterparts in a later phase of development [5], [6], [17], [18]. The PDMS was prepared using two-part mixture of base and curing agents. The mixture was spin-coated with a coater (WS-400, Laurell) followed by curing at room temperature. The resulting thickness was approximately 250 μm. It was reported that PDMS thickness above 200 μm exhibited bulk elastic property with repeated compressive force application [19]. The elastomer layer hence obtained was cut into circular pieces. The electrodes (8 mm diameter) were prepared with gold (120 nm thick) deposited by sputtering using a flash coater (E5400, Biorad). The electrode was patterned on the glass disc using a shadow mask during deposition.

As the final assembly, two glass substrates and the elastomer with patterned electrodes were bonded by a plasma treatment using a reactive ion etcher (PE-200, Plasma Etch) where oxygen gas was used as the reactant. Finally, coaxial cables were connected to the two electrode traces formed over the sidewall of the glass substrate with a silver paste and sealed using an RTV silicone to complete a capacitive sensor as shown in Fig. 1(b).

C. Characterization

The device was connected to an electrochemical potentiostat (Femtostat, Gamry) for ac impedance monitoring. Dissolution test performed in this experiment was conducted in 2 L phosphate buffer saline (PBS) solution (pH 7.4, Sigma-Aldrich) stirred by a magnetic bar rotated at 60 r/min. The solution temperature was kept at 37 °C using an incubator. This PBS is used to simulate the body environment.

A precision vise (EVSD-S60, MSC Interstate) was used to manually apply force on the device to mimic the biomechanical pressure (e.g., spinal loading). To determine the compressive modulus (i.e., stress–strain relationship) of the PDMS elastic layer, the precision vise along with a commercial load cell (LC302, Omega) and a digital microscope (KH-8700, Hirox) were used. The load cell and a prepared sample were placed between the two jaws of the vise for measurements. Optical images were recorded to measure the change of PDMS thickness (i.e., strain) with the microscope with the applied compressive pressure (i.e., stress) read by the load cell.

III. RESULTS AND DISCUSSION

A. Dissolution Behavior of Glass

It takes approximately 25 h for each glass substrate weighing approximately 1.0 g to be completely dissolved in a 2 L phosphate buffer solution. Since these samples do not have the slow-reacting layer, this time duration represents the stages B–C in Fig. 2. The reduction rates of thickness and diameter of glass discs are presented in Fig. 3. As expected, the reduction rate of thickness is higher than that of diameter due to the larger circular area of the top and bottom surfaces exposed to solution than that of the side wall. This implies that the sudden structural failure, after the intended lifetime, can be triggered by a compressive force causing the fracture of disc with decreasing thickness.

B. Dielectricity and Compressive Modulus of Elastomer

A voltage signal sweep from 1 to 100 kHz with a 500 mV RMS (root mean square) amplitude was given and the corresponding impedance

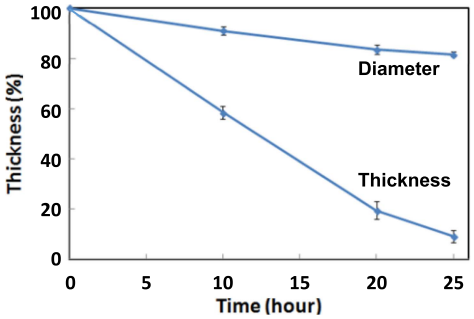

Fig. 3. Dissolution behavior of fast-reacting borate glass substrates (2.7 mm thick, 14 mm diameter) without slow-reacting encapsulation film in phosphate buffer solution (pH 7.4 at 37 °C).

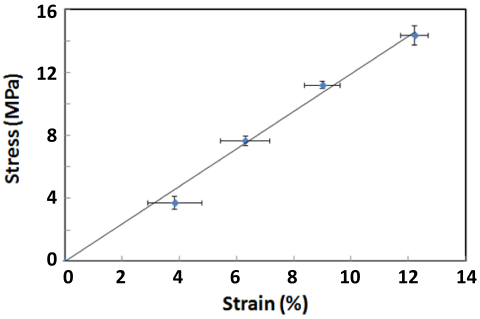

Fig. 4. Mechanical properties of PDMS elastic elements (250 μm thickness, 14 mm diameter) used as pressure-sensitive elastomer.

values were measured. Calculated dielectricity (ε_r) of the PDMS from the measured impedance according to (1) and (2) was 2.57 that is close to the reported value [20].

The stress versus strain plot of Fig. 4 enables us to determine the dynamic range and the repeatability of the pressure sensor within which the elastic shape retention capability can be maintained. The stress and strain represent the compressive pressure (= F / A) and the ratio between compressed and original thicknesses (= Δd / d). Here, F, A, Δd, and d are defined as compressive force, force-receiving area, compressed thickness, and original thickness, respectively. It shows a linear relationship with a slope of approximately 119 MPa (i.e., compressive modulus) within the test range of 14 MPa. This observed modulus of the prepared samples was in the range of that obtained from PDMS samples prepared in accordance with American Society for Testing of Materials (ASTM) D575-91 standards [21].

C. Device Time Response and Sensitivity

Various compressive pressures up to 14.35 MPa were applied by the precision vise to obtain the temporal change of impedance (based on the measured currents of 21–24 nA RMS range with a 1 kHz, 500 mV RMS signal applied). The lower impedance regions in Fig. 5(a) indicate the pressure is applied during that time interval, leading to an increase in capacitance. The reversible recovery of impedance to the original baseline upon removal of pressure implies that the elastic behavior of PDMS insulator serving as a pressure-sensitive element with reversibility.

The pressure sensitivity was presented in Fig. 5(b) where the percentage increase in capacitance was plotted for incremental pressure. As shown, there is a nearly linear relationship between them as expected from (1) and (2) and the constant compressive modulus

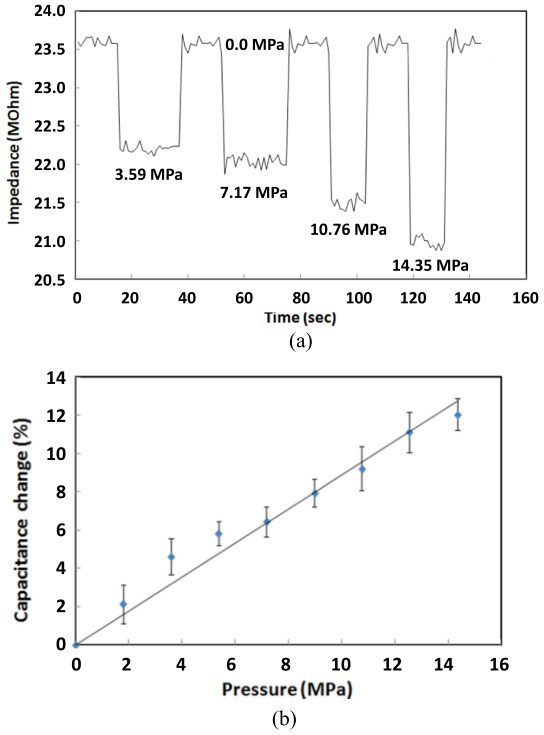

Fig. 5. (a) Typical time response of the sensor impedance (1 kHz, 500 mV RMS sinusoidal signal). (b) Pressure sensitivity of sensors with a wide biomechanical pressure range.

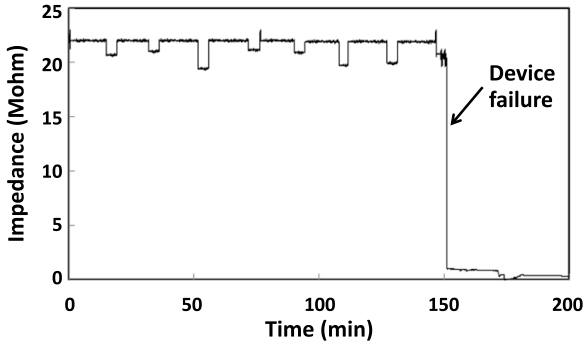

Fig. 6. Exemplary dissolution behavior of a device in phosphate buffer solution (pH 7.4, 37 °C) to demonstrate the sudden failure (stages B–C in Fig. 2).

from Fig. 3. The proposed sensor covers a wide dynamic range for monitoring biomechanical pressure when an orthopedic patient performs light physical movements. For example, the intervertebral disc pressure range was reported to be 0.1 MPa when lying supine, 0.5 MPa with relaxed standing, and 1.1 MPa when holding 20 kg close to the body, respectively [16]. These values are equivalent to approximately 1.1, 5.7, and 12.5 MPa, respectively, in Fig. 5 due to the different force-receiving areas of sensor and typical adult male vertebral body.

D. Device Degradation Behavior

Fig. 6 represents the later part of interval B–C depicted in Fig. 2. After approximately 15 h of immersion in a solution, the pressures were repeatedly applied to the device for approximately 5 s each time. The low impedance region shows that pressure (between 7 and 14 MPa) was applied to the device. After the 2.5-h operation with a sporadic pressure

application, the device impedance steeply decreased, indicating the physical collapse of the entire structure. This dissolution behavior of the device is the proof-of-concept of the intended rapid disintegration to prevent the continued use of device after the expiration of functional lifetime, as described earlier.

The fast-reacting glass used in this study is a borate-rich material without containing calcium elements. As such, the demonstrated devices leave little residue after degradation. Calcium-containing glasses, however, convert to carbonated hydroxyapatite layers with a chemical structure similar to that found in human bone by exchanging the elements with surrounding tissue and forming a firm bond with neighboring hard tissues. This hydroxyapatite formation is important in the hard tissue regeneration process, known as osteogenesis or ossification, since a large portion of human bone is a modified form of hydroxyapatite, known as bone mineral [7], [8], [9].

The device reported herein is intended to demonstrate only a sensing performance without leaving a trace after biodegradation. However, remaining part of sensors prepared with glass materials that can be converted to hydroxyapatite is expected to provide additional therapeutic functions such as stimulating osteosynthetic fusion or monitoring fixation. In addition, due to the ability of biodegradable glasses to incorporate and deliver drugs, the device can promote wound healing and/or vascularization of soft tissues. Therefore, a wide range of potential applications are anticipated, depending on the composition of sensor structural material.

IV. CONCLUSION

Borate-based biodegradable glass material was successfully used as a novel functional platform to fabricate solid-state sensors toward temporary medical implantation. This demonstrates that the use of biodegradable glass substrate is a viable approach to develop temporary active electronic devices for interventional monitoring and therapeutic scenarios with desired operational lifetime. Manipulating the glass composition tailored for specific medical treatment allows enabling of biodegradable implanted sensors that become part of regenerated bones for orthopedic healing. The function of such devices is not limited to sensing, but also serving as an actuator that triggers the treatment process of bone fusion and/or regrowth at the implanted site. In addition to medical applications, it is anticipated that this new class of devices can serve as field-deployable devices to minimize the pollution in agricultural and environmental monitoring that leaves no trace after degradation.

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