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EXPERIMENTAL DESIGN TO PROBE AUTONOMIC RESTORATION OF ELECTRICAL CONDUCTIVITY

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ABSTRACT

A new protocol is introduced for assessing the ability of an electrical circuit to self-heal. We have developed several microcapsule-based strategies for autonomic restoration of electrical conductance to mechanically damaged circuits, necessitating the development of an experimental method to (1) reliably induce mechanical damage to electrical pathways in a model circuit and (2) quantify the loss and restoration of electrical conductance in the circuit. Our self-healing systems utilize microencapsulated healing agents such as conductive liquid metal and suspensions of conductive particles. We introduce a model multilayer circuit design to incorporate microcapsules and enable rupture of the microcapsules when the electrical circuit is damaged.

We have adopted a four-point bend fracture protocol to reliably damage and cause loss of conductivity in our circuits. The multilayer specimens, shown schematically in Figure 1, consist of patterned conductive lines on a dielectric substrate, followed by a thin epoxy dielectric layer, with or without microcapsules, bonded to a glass substrate containing a notch. The entire stack is then bonded to a ductile acrylic layer that serves as a crack stop. When the specimen is loaded in four-point bending, a crack initiates at the notch and propagates through the top glass layer, the epoxy-only or epoxy/microcapsule layer, and the conductive lines. The specimen is designed such that the crack reliably arrests at the second glass/epoxy or epoxy/acrylic interface, causing one of these interfaces to delaminate and preventing complete through-thickness failure.

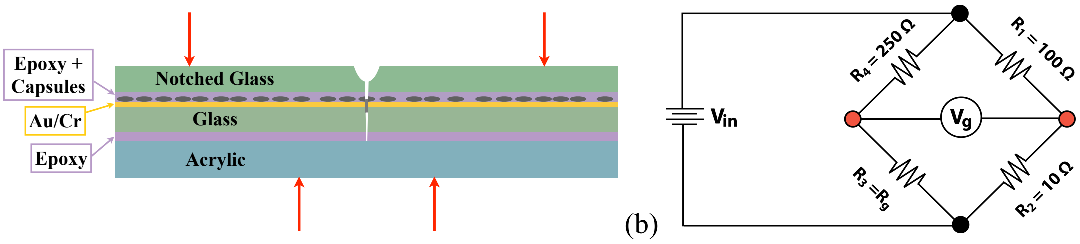
We use one constant voltage Wheatstone bridge circuit per conductive line for monitoring electrical conductance, as shown in Figure 1b. The values R_1, R_2, and R_4, correspond to physical resistors with 10-W power ratings nominally fixed resistances of 100 Ω, 10 Ω, and 250 Ω, respectively. R_3 is the gauge resistor, R_g, corresponding to the test specimen. The bridge voltage of the Wheatstone bridge circuit, V_g defined in Eq. 1, is sensitive to slight changes in resistance in the conductive line and also has a finite range of values, spanning the virgin and fully fractured conductive line cases. The voltage V_∞ in Eq. 2 is defined as the bridge voltage, V_g, as the resistance of R_3 approaches infinity. The normalized bridge voltage, V_{norm}, in Eq. 3 is defined such that the value of V_{norm} ranges from 1 for a fully conductive specimen to 0 for a specimen with no electrical conductance.

$$V_g = V_{in}\left[\frac{R_1}{(R_1 + R_2)} - \frac{R_4}{(R_g + R_4)}\right] \tag{1}$$

$$V_\infty = \frac{V_{in} \cdot R_2}{R_1 + R_2} \tag{2}$$

$$V_{norm} = \frac{V_h - V_\infty}{V_{go} - V_\infty} = \frac{R_{go} + R_4}{R_h + R_4} \tag{3}$$

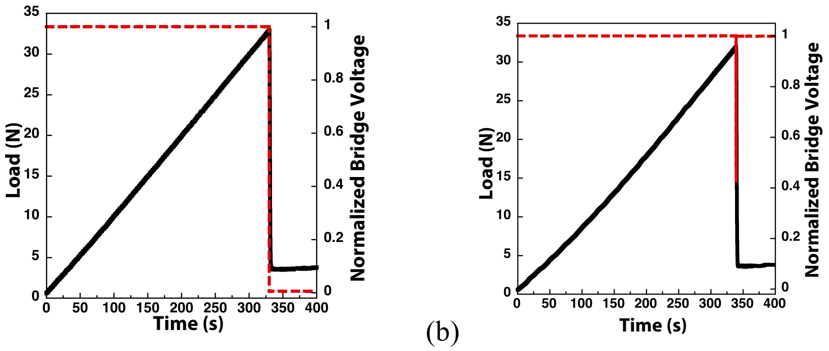
We monitor the Wheatstone bridge voltage and the load-displacement data from the four-point bend test simultaneously to correlate the fracture event, the loss of conductance, and, if applicable, the autonomic restoration of conductance, allowing us to determine the time-scale of autonomic restoration.



(a) (b)

Figure 1: (a) Schematic view of the mechanical specimen under four-point bending, showing the sandwich structure and location of each sample element including the Au film, the microcapsules, the epoxy dielectric layers, the acrylic layer, and the glass layers; (b) Constant voltage Wheatstone bridge circuit diagram, where the conductive line acts as R_3, and the bridge voltage is monitored as shown.

Representative test results are shown in Fig. 2 for a specific self-healing system under development that utilizes microencapsulated conductive liquid metal Gallium-Indium (Ga-In). The multilayer specimen is approximately 12.0 mm wide, 75.0 mm long, and 5.0 mm thick, with layer thicknesses of 1.5 mm acrylic, 180-250 µm epoxy (Epon 828-DETA), 1.0 mm glass substrate, 10 nm Cr, 100 nm Au, 180–250 µm epoxy and Ga-In microcapsules that rest at the Au-epoxy interface, and 1.0 mm of glass treated with (3-trimethoxysilylpropyl)diethylenetriamine with a central rounded notch around 500-µm deep. Five 1.0 mm wide conductive Cr-Au lines with 1.5 mm spacing are deposited on the central glass substrate with a photolithography mask using electron-beam deposition. The notched top glass layer and epoxy/microcapsule layer are 60-mm long to accommodate electrical contacts on either end of the Au lines. The custom four-point bend experimental setup includes a base with adjustable pin spacing (nominally 55-mm), top fixture with 16-mm pin spacing, 10-lb load cell with amplifier, linear actuator for displacement of the top fixture, and LabVIEW 2009 for actuator control and load data acquisition. The load-displacement response of the specimen is linear until fracture, followed by a plateau in the load with continued displacement corresponding to delamination of the final glass/epoxy or epoxy/acrylic interface. The normalized bridge voltage is 1.0 in the linear loading region, and then drops to 0.0 upon fracture of the conductive lines. In the case of control specimens (Fig. 2a), no healing occurs and the bridge voltage remains 0.0. In great contrast, the bridge voltage for the self-healing samples (Fig. 2b) rapidly increases to a value close to 1.0 due to release of the conductive healing system. We define the efficiency of the electrical conductance restoration as the normalized bridge voltage recovered after restoration. The microencapsulated Ga-In system has an average healing efficiency of 99%.



(a) (b)

Figure 2: Representative data of load (black) and normalized bridge voltage (red) as a function of time for (a) a plain epoxy dielectric control specimen and (b) a self-healing specimen with Ga-In capsules