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Fiber-reinforced tough hydrogels

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ABSTRACT

Using strong fibers to reinforce a hydrogel is highly desirable but difficult. Such a composite would combine the attributes of a solid that provides strength and a liquid that transports matter. Most hydrogels, however, are brittle, allowing the fibers to cut through the hydrogel when the composite is loaded. Here we circumvent this problem by using a recently developed tough hydrogel. We fabricate a composite using an alginate–polyacrylamide hydrogel reinforced with a random network of stainless steel fibers. Because the hydrogel is tough, the composite does not fail by the fibers cutting the hydrogel; instead, it fails by the fibers pulling out of the hydrogel against friction. Both stiffness and strength can be increased significantly by adding fibers to the hydrogel. Before failure the composite dissipates a significant amount of energy, at a tunable level of stress, attaining large deformation. Potential applications of tough hydrogel composites include energy-absorbing helmets, tendon repair surgery, and stretchable biometric sensors.

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1. Introduction

Hydrogels are soft materials that consist of cross-linked networks of hydrophilic polymer chains dispersed in water. They have many applications including scaffolds in tissue engineering [1], carriers for drug delivery [2], and valves for microfluidic devices [3]. Many applications of hydrogels rely on the combined attributes of a solid that provides strength and a liquid that transports matter. Most existing hydrogels, however, are brittle, with fracture energies on the order of 10 J/m^2 [4,5]. This limits their use in structural applications where they are subject to mechanical loading. This problem could be circumvented by reinforcing the hydrogels with fibers to improve their mechanical behavior [6]. This effort is hampered, however, by the low toughness of hydrogels: as the composite is loaded, the fibers cut through the hydrogel matrix, destroying the synergy between fibers and matrix, and leading to rapid failure of the composite. A hydrogel-based composite is only effective if the hydrogel matrix is tough enough to resist the fibers cutting through the matrix. Cartilage is one example of such a composite taken from nature: cartilage consists of a collagen fiber-reinforced proteoglycan gel. It contains more than 70% water, but is remarkably stiff and tough [7,4]. It comes as no surprise, then, that hydrogel-based composites are being actively explored for use as synthetic tissues and in biocompatible products [8–15].

Many attempts have been made to improve the toughness of hydrogels [12,14–17]. Double-network hydrogels were the first class of hydrogels to exhibit significant fracture toughness, with fracture energies in the range of 100–1000 $J\,m^{-2}$ [14]. Recently, we developed a new group of hybrid hydrogels synthesized from polymers that form networks with covalent and ionic cross-links, and that have an extraordinary combination of toughness and stretchability [18]. These hybrid gels consist of a covalently cross-linked polyacrylamide (PAAm) network and an ionically

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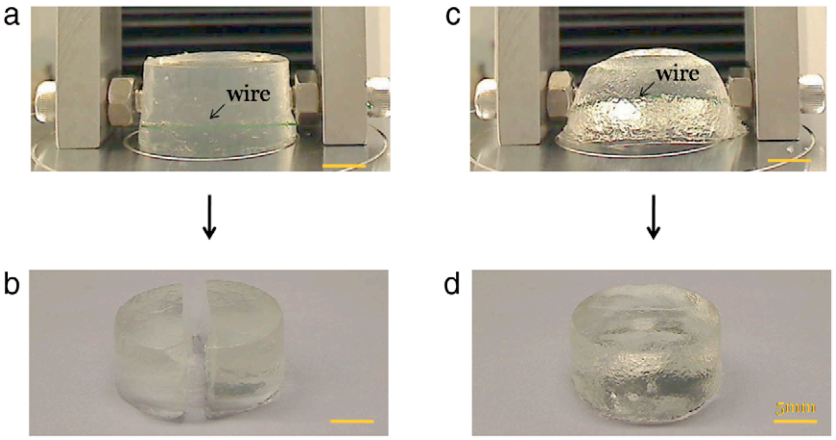

Fig. 1. Wire cutting tests for a brittle (a, b) and a tough hydrogel (c, d). (a) Alginate hydrogel being cut by a steel wire. (b) The cut sample is turned 90° and the two pieces are separated to show the cut. (c) Alginate–polyacrylamide hybrid hydrogel deforms, but resists cutting. (d) The sample recovers its original shape when the wire is removed.

cross-linked alginate network, and can attain fracture energies as large as 9000 J m^{-2}. The need for a tough composite matrix is illustrated graphically in Fig. 1, where we use a metal wire to cut through two different types of hydrogels, a standard technique for measuring the fracture toughness of food and soft gels [19,20]. Two examples are shown: (1) a brittle alginate gel and (2) a tough alginate–polyacrylamide hybrid hydrogel. The difference between the gels is obvious. The alginate gel is readily cut by the wire; the hybrid gel deforms elastically, but resists being cut. Thus an alginate matrix would result in a composite with poor mechanical properties. The hybrid gel, on the other hand, would make a very good matrix in a fiber-reinforced composite. Such a composite would have significantly better stiffness and strength than the hybrid hydrogel, and could be used in structural applications. Lin et al. [12] have investigated the toughening mechanism of alginate–polyacrylamide hydrogels reinforced by a stretchy fiber mesh fabricated from thermoplastic polymers. When a notched sample of such a composite is deformed, the hydrogel matrix maintains the integrity of the sample, while fracture of the fibers in the bridging zone dissipates mechanical energy.

In this study we investigate the mechanical behavior of composites that consist of a tough alginate–polyacrylamide hydrogel matrix reinforced with a random network of stiff fibers, for which we conveniently use stainless steel wool. Steel wool fibers are strong and can easily create a random continuous fiber network—they have been used for this purpose in a number of applications [21,22]. We evaluate the stress–strain curves of the fiber-reinforced gels and evaluate the mechanism by which they fail.

2. Experimental details

2.1. Synthesis of fiber reinforced hydrogels

Powders of alginate (FMC Biopolymer, LF 20/40) and acrylamide (Sigma, A8887) were dissolved in deionized water. After the powders were fully dissolved, the solution was held at 35 °C for 1 h. Weights of alginate and acrylamide were fixed at 1:6, and the water content was fixed at 86 wt%. Ammonium persulfate (AP; Sigma, A9164), 0.0017 times the weight of acrylamide, was added as a photo initiator for the acrylamide polymerization process. N,N-methylenebisacrylamide (MBAA; Sigma, M7279), 0.0006 times the weight of the acrylamide, was added as the cross-linker for the acrylamide. N,N,N′,N′-tetramethylethylenediamine (TEMED; Sigma, T7024), 0.0025 times the weight of acrylamide, was added as the cross-linking accelerator for the polyacrylamide. Calcium sulfate slurry ($CaSO_4 \bullet 2H_2O$; Sigma, 31 221), 0.1328 times the weight of alginate, was added as the ionic cross-linker for alginate. Stainless steel wool (Type 316, fine, McMaster-Carr 7364T74) with an average cross-sectional area of the fibers of $\sim$80 μm $\times$ 70 μm was chosen to provide the reinforcing fibers. The fibers in this type of steel wool are arbitrarily oriented and have lengths that exceed the sample dimensions. After the wool was distributed evenly inside dumbbell-shaped molds, the gel solution was poured into the molds and covered with a glass plate. Before casting the gel solution, the steel wool was thoroughly wetted with the solution to reduce the formation of bubbles inside the samples. The composite samples were then cured at room temperature by exposing them for eight minutes to ultraviolet light with a wavelength of 350 nm (OAI LS 30 UV flood exposure system, 1.92 W/cm^2 power density). The samples were kept in a sealed container at room temperature for one day to ensure complete reaction. The weight fraction of steel wool in the composite hybrid gels was varied from 0 to 12 wt%.

2.2. Wire cutting test

Hydrogel samples with a diameter of 35 mm and a thickness of 17 mm were fabricated from an unreinforced alginate–polyacrylamide hybrid hydrogel and an alginate hydrogel (Fig. 1). Wire cutting tests were performed in air, at room temperature, using a tensile tester (Instron model 3342) with a 1000 N load cell. A 580 μm diameter steel wire was pulled through the specimen at a displacement rate 10 mm/min.

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