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Acta Biomaterialia 3 (2007) 377–382
Acta BIOMATERIALIA
www.elsevier.com/locate/actabiomat

Effects of loading rate on the mechanical behavior of a natural rigid composite ☆

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Received 22 May 2006; received in revised form 10 August 2006; accepted 26 September 2006

Abstract

The effects of loading rate variations on the stress–strain behavior, failure mechanisms, fracture modes, and energy-dissipating capability of the spicules of the sponge *Euplectella aspergillum* have been investigated. Comparisons were made with similar measurements on a silicate glass. It was concluded that the very thin (5–10 nm) organic layers that are interspersed with thicker layers of hydrated silica in the concentric ring structure of the spicules strongly influence all aspects of the mechanical behavior.

Keywords: Sponge spicules; Mechanical behavior; Loading rates; Energy dissipation

1. Introduction

Interest in sponge spicules stems from their interesting combinations of stiffness, strength and energy-absorbing capabilities. Important lessons may be learned from the structure–property relations found in sponge spicules for the practical purpose of designing new synthetic composite materials. Earlier work has concentrated on the strength and stiffness of spicules, with only passing note of their flexibilities and damping capacities. The evidence for flexibility and viscoelasticity in organic and hybrid organic systems stems from observations described by Levi et al. [1], Sarikaya et al. [2], Mayer et al. [3], and from the bodies of evidence in scientific papers and in a comprehensive collected volume of papers [4–6]. In the first instance, observations were made of a long cylindrical spicule of a siliceous sponge comprising hydrated silica ($SiO_2 \cdot nH_2O$ where n is 2–5) sandwiched with thin layers of proteinaceous material (in a concentric ring structure form in cross section, as in Fig. 1). When the spicule was bent into a circular form, it did not fail, and, when the load was released, the original shape recovered, although nothing was stated about whether this happened instantaneously or over a period of time. Such behavior was observed in long, thin, cylindrical spicules of *Hexactinellid* sponges. Subsequent studies confirmed these observations. Experiments on spicules of a sponge that were performed at Oak Ridge National Laboratory, and cited in Ref. [3], included several tests that were carried out at different strain rates. Those results were not studied in any detail at the time, but there was enough information to indicate that a notable rate sensitivity existed in the spicule fibers of *Euplectella aspergillum* (Fig. 2).

The ability of the unusual composite structure of the spicule to dissipate energy during deformation, before catastrophic failure, is the major reason for interest by designers of composite structures. Energy dissipation is a very important factor that can be beneficially put to use in advanced synthetic composite materials.

It was decided to conduct a much more detailed investigation into the rate sensitivity of this hybrid composite material, with emphasis on the effects of loading rate and the thin organic layers on energy dissipation. The organic layers that exist in the spicules of the sponge that were studied are of the order of 5–10 nm in thickness, and it

☆ Research presented at the TMS 2006 Biological Materials Science Symposium.

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1742-7061/$ - see front matter
doi:10.1016/j.actbio.2006.09.008

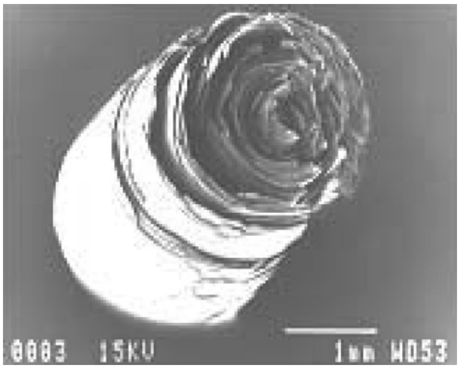

Fig. 1. Cross section of *Monorhaphis* spicule (Courtesy of P.Lehuede).

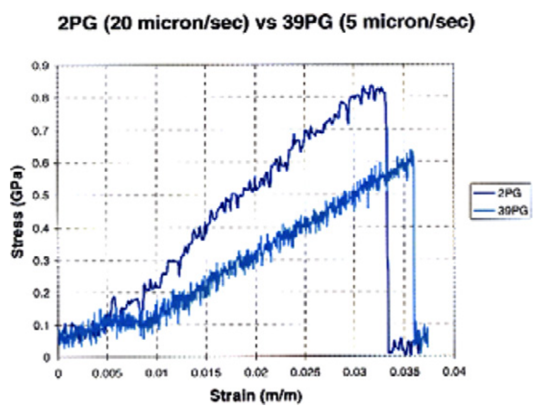

Fig. 2. Stress–strain curves for spicules under two different strain rates [3].

has not been possible to interrogate them directly using atomic force microscopy methods, as was done in the case of nacre by Smith et al. [7]. Tests at different loading rates were conducted in order to shed more light on the elastic and viscoelastic characteristics of the thin organic layers. Such layers have been identified in other, different, sponge species by Sumerel and Morse as several different silicateins [8], i.e. classes of silicon proteins. Although it was not within the scope of this study, the authors believe that, while the organic constituents of the thin layers of the sponge that was studied in this work are probably different from the silicateins that have been identified, they will still be proteins that are based on silica.

2. Materials and methods

E. aspergillum sponges were selected for this study, because earlier specimens of *Hexactinellids*, of which this system is a representative, had shown interesting combinations of resilience and time-dependent mechanical behavior, along with substantial strength and stiffness. Also, a number of the skeletons of this sponge were readily available to us for the research. On the other hand, handling, gripping and testing of the very fine fibers, on the order of 50 μm, was a challenging task. All mechanical testing was carried out on specimens from two sponge skeletons

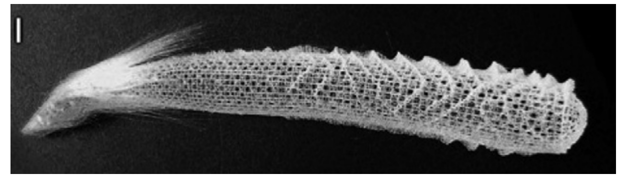

Fig. 3. Skeleton of *E. aspergillum*, showing spicule fibers that were tested (taken from the separate fibers toward the left, basal side.) Scale bar = 1 cm.

(Fig. 3 from an unpublished example), working with only smooth, straight sections of spicule fibers, from the approximate regions noted on Fig. 3. Fiber sections at the very basal end of the skeleton tend to have short branches, or an anchor-like end section [9]. The latter fiber sections were discarded from consideration for mechanical testing, since their stress states would have been too complex for analysis. The diameters of fibers included in the mechanical tests ranged from 40 μm to 70 μm. It should be noted that the cross section of the fibers is different from what is seen in Fig. 1, in that a larger inner core of hydrated silica is present, as shown schematically in Fig. 4. That inner core has been related to the optical transmission characteristics of *E. aspergillum* [9]. At the very center of the solid inner core is a small square cylindrical core (of probably a) protein.

As a basis for comparison, fibers of a silicate glass, identified as an electronic-grade glass, EMGO 360, and containing BaO and Na_2O in a silicate glass, with diameters 40–70 μm (that had been drawn from the melt by NIST) were included for testing and analysis. The sponge skeletons were generously donated by Nature's Creations® of Sammamish, WA. Those skeletons had been pre-treated with a diluted bleach solution and subsequently rinsed with distilled water to remove both the outer softer "spongy" layers and any debris that might have been present. Fiber diameters were carefully examined for flaws with optical microscopy and diameters were also measured for the smooth fibers that were selected for mechanical testing.

Tests at different loading rates were conducted in three-point bending, using a dynamic mechanical analysis (DMA) system (Perkin–Elmer Model 7e), in both the static and dynamic modes, in order to shed more light on the elastic and viscoelastic characteristics of the thin organic layers. Both the spicule fibers with the concentric ring structure and the silica fibers used as reference material were tested under similar conditions. Thirty samples of each type of fiber were tested at the high loading rate in order to establish a reasonable database. Ten samples of each fiber were tested at low and moderate loading rates. Loading rates of 1 mN/min, 10 mN/min, and 100 mN/min were employed. After fracture, the fiber segments were carefully collected for examination by scanning electron microscopy (SEM).

Frequency scans were also conducted on the spicules and glass fibers in order to examine damping characteristics. The tests scanned from 1 Hz to 51 Hz at a load of