AC 2007-2581: SYNTHESIS AND CHARACTERIZATION OF BIOMIMICKED STRUCTURAL COMPOSITES

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Synthesis and Characterization of Biomimicked Structural Composites

Abstract

This paper presents the preliminary results of experiments conducted on the toughening of structural composites for shatter-proof applications. Layered composites inspired by tough biological structures such as nacre were fabricated and tested in tension and compression. The results are compared with monolithic structures fabricated and tested under the same conditions. Compared to monolithic samples, mechanical testing results show greater toughness associated with higher levels of ductility for the layered composites. The effect of materials selection for structural composites was investigated. It was concluded that biomimicked structures can be designed and fabricated with both higher levels of tensile strength as well as increased ductility (resulting in higher fracture toughness). The educational implications of this work in terms of the students participation and its usefulness in the outreach programs are discussed.

Introduction

Development of tough structural materials could potentially lead to a significant reduction of fatalities and property damage during earthquakes, tornados and hurricanes. Collapse of mortar and brick type structures, caused by the dynamic shear forces of earthquake can be mitigated or completely prevented if the mortar is chosen to be a thin polymer.

Robust biomechanical structures such as bone and nacre show great fracture toughness due to their intricate designs combining a soft phase polymer with a hard phase mineral. In nacre, parallel layers of aragonite tablets (Figure 1) are glued together by an inorganic polymer (proteins and polysaccharides). If the edge of the tablets are correlated into a tessellated arrangement (Figure 1) it resembles the structure of abalone. Alternative structure is the Noncorrelated edges of the tablets leading to the structures typical of pearl oyster. Superior mechanical properties of this structure include a high strength combined with high fracture toughness. Due to the lamellar nature of the structure, properties are anisotropic. Nominal strength ranges from 194-248 \textsuperscript{1} obtained from the 3-point bend tests on abalone and oyster with higher numbers belonging to pearl oyster.

An initial elastic response is observed during the tensile loading of nacre parallel to its plates. However, at a stress level of 110 MPa a steady state deformation (inelastic) commences that continues until fracture at 1\% strain. This inelastic secondary deformation is not seen during compression loading. Rather, an elastic deformation takes place that continues to fracture at very high stresses (exceeding 400 MPa) associated with a shorter fracture strain (0.5\%). When loading axis is at 45\° with respect to the plates, a uniform shear stress occurs across the plates. The steady state inelastic response that commences at a shear stress of 40 MPa continues until fracture at 8\% strain. The main role of inelasticity is its insensitivity to localized deformation sites that causes premature failure.
The robustness of nacre arises from four design principles as postulated by He et al.\textsuperscript{1}. These are: a) morphology which is optimized to maximize inelastic strain, b) nanoscale asperities that cause mechanical interlocking at the interface; sufficient adherence of ceramic layers by the bond layer and d) lubrication provided by the polymer\textsuperscript{1}. A newer study by the Katti et al.\textsuperscript{2} discounts the effect of nanoscale asperities to the overall mechanical strength including strain hardening. Instead, the continuity of the aragonite single crystal tablets through bridges over the polymer layers and the relative misorientation have been emphasized in the mechanical properties of nacre\textsuperscript{3,4}

![Figure 2: Schematic of the microstructure of Nacre with tablets misoriented with respect to each other in adjacent layers](image)

Biologically inspired layered structures made of construction materials have shown to be mechanically superior to their monolithic counterparts\textsuperscript{5}. Biologically inspired schemes used in developmental work include those used in the fabrication of micro-laminated ceramic-metal\textsuperscript{6-8}, ceramic-organic and organic-organic composites\textsuperscript{9}. Examples are B\textsubscript{4}C layered with Al\textsuperscript{10}, SiC layered with Al and B\textsubscript{4}C layered with polypropylene\textsuperscript{6,11,12}. Biomedical applications of nacre structure include hydroxyapatite scaffolds\textsuperscript{13}. The implementation of such biomechanical patterns to construction of tough structural composites can potentially lead to the buildings that are more resistant to dynamic shear forces of nature. This implementation will include the in-situ fabrication of composites made of concrete and polymer in alternate layers.

**Material**

To fabricate the layered structures the following materials were used:

- **Cement:** Quikrete\textsuperscript{TM} Quick Setting Cement # 1240 with a compression strength of 20-44 MPa
- **Polymer:**
  1. Liquid Nai\textsuperscript{TM} Glue #LN-275, a Benzene-based synthetic rubber with a shear strength of 0.8 – 2.0 MPa
  2. Gorilla Glue\textsuperscript{TM}: Polymer with 70% urethane with 30% polymer MDI
3. Quikrete™ Concrete Bonding Adhesive #9908 with a shear strength of 0.7 – 1.0 MPa
- Reinforcement: Interwoven Fiberglas™ placed 45º with respect to the length of the samples.

Experimental Procedures

Dog-bone shape samples of layered composites were made using the aluminum mold shown in Figure 1. Deposition take place by spreading the mixed cement paste in the mold cavities leveling off the top to create a smooth topical surface. The fabrication scheme was inspired by the layered microstructure of nacre. The mold shown in Figure 1 contains four dog-bone shape cavities used for fabrication of 4 different types of composites. The mold was mounted on the upper crosshead of an Instron mechanical load frame which provided incremental elevation of the mold in desired intervals. The deposition of the concrete layer was followed by the application of the polymer layer using a brush after a time laps of about 5 minutes. The quick setting paste was stiff enough by then to withstand the application of the bond layer. The deposition of the pair of the concrete and polymer layers was repeated until a total thickness of 25 mm was achieved. A time laps of about 30 minutes between layers allowed stiffening of the glue enough to facilitate the deposition of the next concrete layer.

Samples were aged for about 48 h to gain significant strength. All samples were made at the same time and within the same mold to reduce fabrication-related variation of properties. A layered monolithic sample was made along with the layered composites to establish a baseline for comparison of mechanical properties. The description of the samples fabricated is presented in Table 1. All samples had the same size, shape and geometry with a gage length of 100 mm, thickness of 25mm and a width of 25 mm. The ends were triangular with sides sloping at 30º. Samples are illustrated in Figure 2.
Tensile Testing

Mechanical testing of the samples was achieved by tensile loading of the dog-bone samples using an Instron™ load frame. The cross-heads of the load frame were equipped with special grips shaped to house the triangular end of the dog-bone shape samples. The top grip was mounted on a 2000 lb load cell connected in series with a factory installed 30,000 lb load cell. The loadcells were calibrated with hanging a known weight to the smaller loadcell. Epoxy and resin was used to properly align the sample thus minimizing any bending moment imposed by misalignment. A strain rate of $2.5 \times 10^{-4}$ was used for all samples tested. All samples were tested one after the other within the same session to minimize variation due to aging. LabView™ (National Instrument Corp., Houston, TX) data acquisition system connected to a 2000 lb load cell was used to monitor load.

Table 1- Designation and description of the layered samples fabricated and tested

<table>
<thead>
<tr>
<th>Designation</th>
<th>Type of Samples</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Q0</td>
<td>Monolithic</td>
<td>Concrete with no glue</td>
</tr>
<tr>
<td>QL</td>
<td>Layered composite</td>
<td>Concrete + Liquid Nail Polymer</td>
</tr>
<tr>
<td>QG</td>
<td>Layered Composite</td>
<td>Concrete + Gorilla Glue</td>
</tr>
<tr>
<td>QCA</td>
<td>Layered Composite</td>
<td>Concrete + Concrete Bonding Adhesive</td>
</tr>
<tr>
<td>QGF</td>
<td>Reinforced Composite</td>
<td>Concrete + Gorilla Glue + fiberglass</td>
</tr>
</tbody>
</table>

Figure 2 - Samples made from various combination of concrete, polymer and fiberglass

Results and Discussion

The results of the tensile tests performed on the monolithic and composite structures are presented in Table 1. Monolithic samples show the smallest amount of strain. All composite structures exhibited higher levels of ductility. The strength of the composites were not, however, higher than monolithic structure for all composites. While QGF and QG showed ultimate tensile strengths greater than that of monolithic sample, the other two composites, namely QBA and QL
had relatively lower strengths. The main difference between the mechanical properties of the composites and the monolithic was the increased level of toughness in these layered composites.

The addition of fiberglass to concrete significantly increased the tensile strength of the reinforced sample as expected. Although the fibers run at 45° with respect to the tensile axis and were of roughly 40 mm length (less than half of the gage length), nevertheless, they increased the tensile strength of the reinforced composite by more than one order of magnitude. Since the fibers did not run along the length of the sample, the observed strength can not be directly related to the strength of virgin fibers (~ 3.5 GPa).

Fractography

Fracture of monolithic sample is consistent with brittle materials. Details of the fracture surface of the monolithic concrete sample is shown in Figure 4(a). No effect of layering is observed in the fracture surface of the monolithic sample. Crack path is nearly perpendicular to the tensile axis as expected in brittle fracture. Fracture of QG sample is shown in Figure 4(b). Crack is seen to change its plane going from one layer to another. This is better seen in the fracture surface of the

Table 1: Fracture strength and strain for monolithic and biomimicked composites

<table>
<thead>
<tr>
<th>Type of Composite</th>
<th>Stress</th>
<th>Strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monolithic</td>
<td>1.1</td>
<td>0.008</td>
</tr>
<tr>
<td>Fiber Glass/G. Glue Composite</td>
<td>14.9</td>
<td>0.03</td>
</tr>
<tr>
<td>Concrete Bonding Adhesive</td>
<td>0.58</td>
<td>0.012</td>
</tr>
<tr>
<td>Gorilla Glue Composite</td>
<td>1.82</td>
<td>0.06</td>
</tr>
<tr>
<td>Liquid Nail Composite</td>
<td>0.84</td>
<td>0.025</td>
</tr>
</tbody>
</table>

QL sample shown in Figures 5(a-b). Crack is seen to travel across the layers as well as in the interface between the layers. Almost in all samples crack started at the vicinity of the transition region at the end of the gage length. Crack was also observed to initiate at one side and
propagate to the other side (Figure 5-a). Crack branching, as well as multiple cracks were observed. One instance of crack bridging is seen in Figure 5(b).

![Figure 4](image)

**Figure 4 - Fracture of the monolithic and composite structures: (a) Monolithic, (b) QG: concrete with Gorilla Glue**

**Educational Implications**

The Mechanical and Manufacturing Engineering Technology (MMET) courses taught at Northern Kentucky University include EGT-116: Introduction to Materials and Manufacturing Processes, EGT-261: Engineering Materials, EGT-317: Senior Research in Technology and EGT-417: Senior Design Project. All deal with materials and manufacturing. Other related courses are in the field of robotics, construction management, and nanotechnology. Most of these courses require students to complete a project. While undergraduate research is emphasized to replace or complement capstone project course such as EGT-417, course projects serve well in exposing students to innovative, developmental work that can benefit them in future practical applications. The outreach programs that target young high school students as well as adult learners can also benefit from hands-on projects that combine innovation, creativity and analytical skills. It is desirable to incorporate new ideas, innovative schemes and application-based learning into manageable projects that can be carried out by students on different levels.

![Figure 5](image)

**Figure 5 – Crack propagation in the QL sample (a) Top surface (b) Side surface revealing details of crack path across the layers, (c) Crack bridging and Multiple cracking shown by arrows**

Robotic construction of biomimicked structures, as the main research area of the first author has engaged several students in various courses mentioned above at different levels. Small class
projects were developed and assigned to students of robotics, manufacturing and engineering materials and nanotechnology. All students completed their assigned projects successfully, contributing to the overall progress of the work in the field of biomimicked robotic construction. As an example, the second author, a student of the MMET program helped machine the mold and fabricate the samples. An engineering summer camp arranged for the junior and senior level high school students benefited from this work. Students participated in the fabrication of layered structures using RV-M1 robots in geometries they designed and developed. The outcome was a good rating of this part of their learning indicated in their final survey. Aspects of this work are planned to be used for three workshops and two summer camps that will take place this summer at NKU. Currently three students are working on the fabrication of an Autonomous Ground Vehicle, AGV for the deposition of layered biomimicked structures on a real life scale. Students learn about automation, LabView™ data acquisition and controls, programming, fabrication, mechanical testing and microstructural characterization.

Conclusions

1. Biomimicked structures made from cement and polymer were fabricated and tested in tensile loading
   a. Layered structures were inspired by nacre which is known to have high fracture toughness
   b. Compared to monolithic structure layered composites exhibited either a larger tensile strength, a larger fracture toughness or both.
   c. Reinforced composites exhibited a very high tensile strength associated with a large level of fracture toughness.

References
