TENSILE TESTING OF ABALONE NACRE MINIATURE SPECIMENS USING MICROSCOPY AND SPECKLE CORRELATION

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ABSTRACT

Nacre from seashells, also known as mother-of-pearl, has attracted much attention for its remarkable mechanical properties despite the structural weaknesses of its components [1],[2]. In this article, in situ-tensile tests of dry and wet nacre dogbone specimens are presented. Optical microscopy and digital image correlation techniques were used to determine deformation and strain fields. While dry nacre exhibits brittle behavior, hydrated nacre (as in its natural environment) exhibits ductile behavior and large deformations. This study is a first step to completely characterize nacre's mechanics in relation to its microstructure and components for the perspective of novel nano-composites designs.

INTRODUCTION

Composite materials achieve unique combinations of mechanical properties. Many structural materials found in nature use this concept for enhanced stiffness, strength or toughness. This is particularly true for biomaterials whose function is to protect soft tissues from mechanical aggressions from the outside environment: eggshells, insect exoskeleton, seashells.

For example, the abalone seashell is a bi-layered material: An external layer is composed of large prismatic aragonite crystals of fairly high hardness. Its function is to stop any impact from the outside environment. An inside nacreous layer is designed to absorb the kinetic energy from the impact, through ductility and enhanced toughness. This design is believed to be ideal for impact resistant armor [3]. Nacre is of interest here. This nano-composite achieves ductility and enhanced toughness using mainly aragonite, which is a brittle ceramic. Abalone nacre microstructure and its mechanics of deformation are examined, and an in-situ tensile test on a small specimen is presented. Deformations inside the gage region were monitored with a microscope, and optical methods (speckle correlation) were used to determine the strain fields.

NACRE MICROSRUCTURE

Nacre is mainly composed of aragonite polygonal tablets (95% in mass) and biological macromolecules between them (5% in mass). The aragonite tablets are between 6 and 8 μm in diameter and 0.5 μm in thickness. They are stacked in columns perpendicular to the layer, with some overlap between them. A cross section reveals their brick wall like structure, as showed on figure (1). The size and arrangement of the tablets is very regular throughout the nacreous layer.

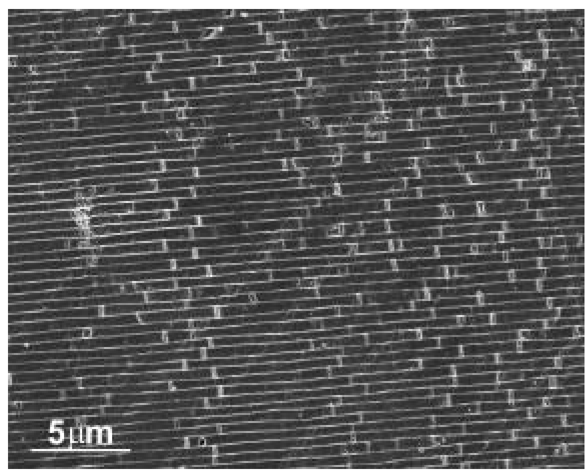

Figure 1: SEM image of a polished nacre specimen.

Electron microscopy on cleaved nacre reveals nanoscale aragonite asperities on the face of the tablets (figure 2,3). These asperities are grouped in islands, apparently independently from the tablet distribution. It has been shown that in some areas asperities from opposing tablets show a conformal geometry, i.e. they fit into each other [4]. Between the tablets lays an organic network composed of large biomolecules. This organic phase remains from the organic scaffold used for the biomineralization of aragonite, but has still a primordial mechanical role. Ligaments in the networks are firmly attached to the tablets, which hold them together (figure 3).

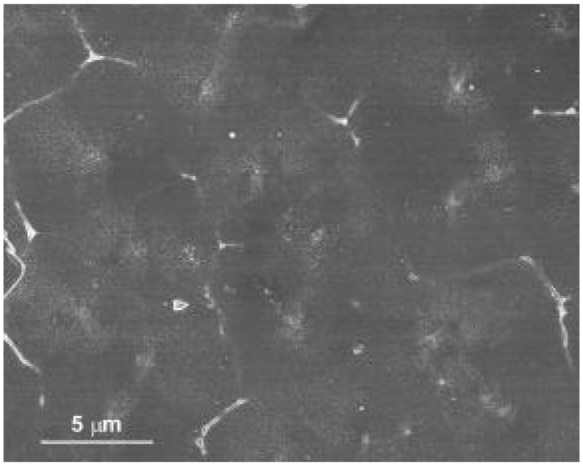

Figure 2: SEM image of a nacre specimen cleaved along the tablets and thermally etched: islands of asperities are visible

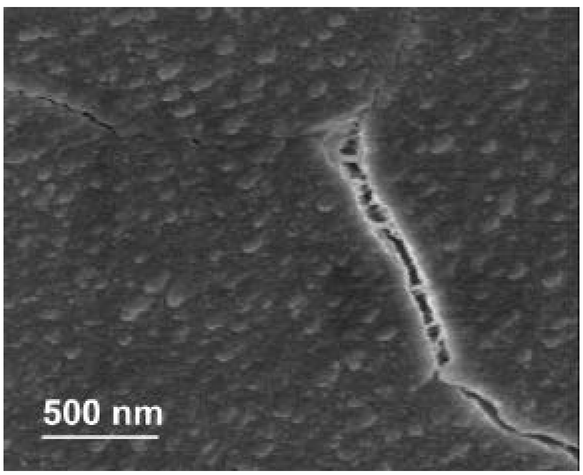

Figure 3: SEM image of a nacre specimen which was tested in tension then cleaved along the tablets and thermally etched: asperities are visible, as well as organic ligaments bridging 2 adjacent tablets.

NACRE MECHANICS

Because of its microstructure, nacre is transversely isotropic and has different behaviors in tension and compression. We will focus our attention on tensile behavior along the tablets. Tensile loading is usually more critical for ceramics, and tension parallel to the tablets corresponds to what the nacreous layer experiences in the case of impact normal to the shell. Under these conditions, the nacreous layer exhibits ductile behavior and large deformations. At a sufficient load the tablets begin sliding over each other. Normally we would view the sliding region as the weakest point in the gage and would expect the specimen to fail at this location. However all potential sliding areas are activated, which leads to large deformations before failure (figure 4).

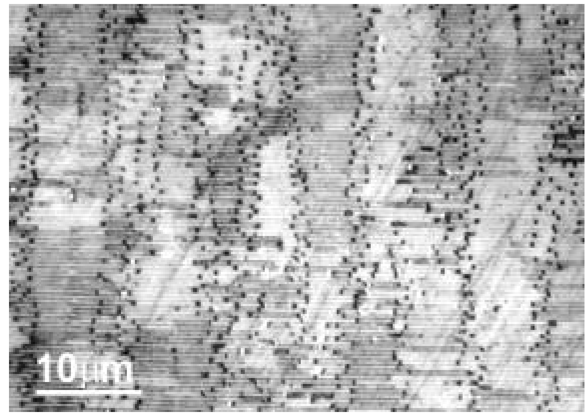

Figure 4: Optical micrograph of a polished nacre specimen tested in tension along the tablets. The darks stains are due to the fluoresœnt dye used to enhance the contrast of the interfaces

This suggests a hardening effect during sliding. It has been suggested that the sliding of the tablets involves climbing of opposing asperities, which creates in turn a transverse compressive stress responsible for the observed hardening [5]. However this transverse compression build up should cease when all asperities have been climbed, which occur early in the sliding process. A second hardening mechanism should be present, and the possibility of hyperelastic ligament stretching during sliding will be investigated in the future. The respective contribution of these two mechanisms is currently unknown.

EXPERIMENTAL PROCEDURE

Abalone shells were purchased from a seashell store in California. Small dogbones specimens were fashioned from the nacreous layer so that the tablets were aligned with the axis of the gage. The gage was 1 mm in length, and 1 mm x 1mm in cross section. All cutting and machining operations were performed at low speed and lubricated with water. The ends of the dogbone specimens were attached between soft PVC holders, which in turn were clamped into a miniature loading device (Ernest F. Fullam, Albany NY). The holder insured that not parasitic load was transmitted from the clamping area to the gage. A picture of the setup is showed on figure 5. For the tests in wet conditions, the specimens were left in tap water for five hours prior to testing. The loading device was then placed under a microscope. Shallow scratches were created on the surface of the gage with fine sandpaper to create the dark and light features needed for digital image correlation. The contrast of the features was further increased by application of a fluorescent dye. A reference picture of the gage was taken at low magnification (50x, see figure 6). The specimen was loaded in tension at very low strain rate (1.5×10^{-5} s^{-1}).

While continuously loading the specimen, pictures of the gage were taken at regular intervals, and the load was simultaneously recorded.

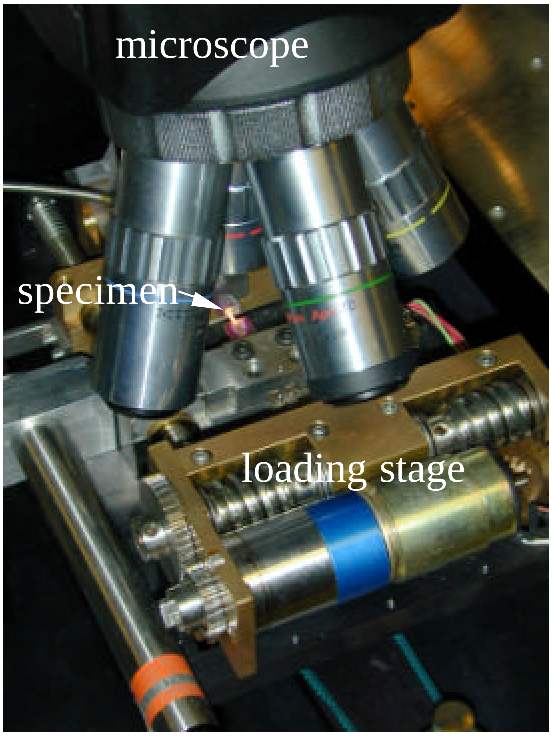

Figure 5: Experimental setup

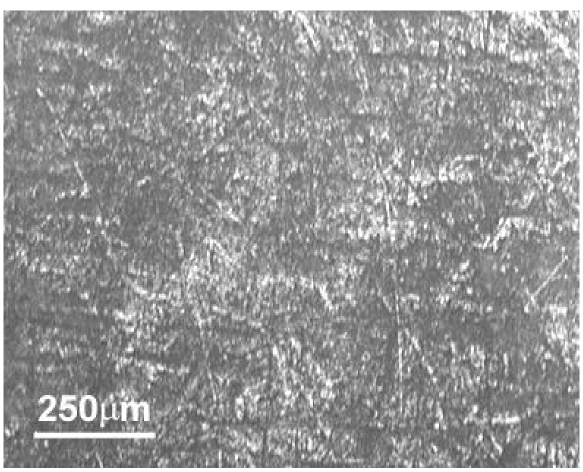

Figure 6: Optical micrograph of the dogbone gage taken during the tensile test

Each of the deformed images was correlated to the reference picture, as illustrated on figure 7. A subset was picked in the reference picture and its intensities were mapped with a spline surface. An optimization scheme was then used to find the displacement of the subset in the deformed image that satisfies the best matching between intensities. In addition, the subset was allowed to linearly deform during the search. Several correlations of subsets at regular intervals yielded the full displacement field, with a sub-pixel resolution. All correlations were performed using the VIC 2D software (Video Image Correlation Package for 2-dimensional problem. © Correlated Solutions, 1998). More details about the speckle correlation technique can be found elsewhere [6],[7]. The displacement field was fitted with a plane surface using a least square fitting method, which yielded the longitudinal strains in the gage. The stress in the gage was simply given by dividing the load recorded by the load cell by the cross section area of the gage.

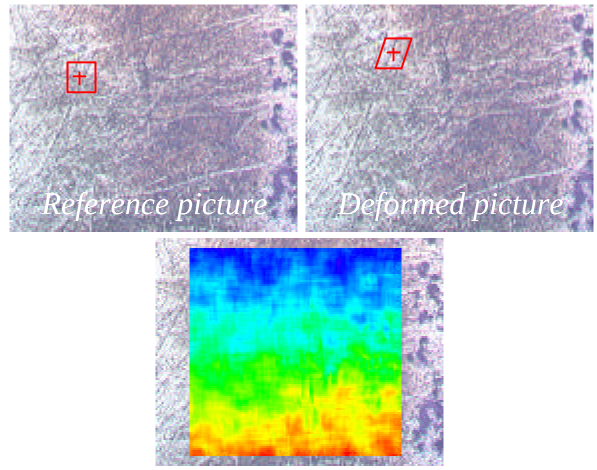

Figure 7: Top left: subset in the reference picture, top right: corresponding subset in the deformed configuration, bottom: full field displacement.

RESULTS

Both dry and hydrated (wet) nacre specimens were tested in tension along the tablets. The resulting stress-strain curves are shown in figure 8.
Dry nacre exhibited brittle behavior while wet nacre showed ductile like behavior.

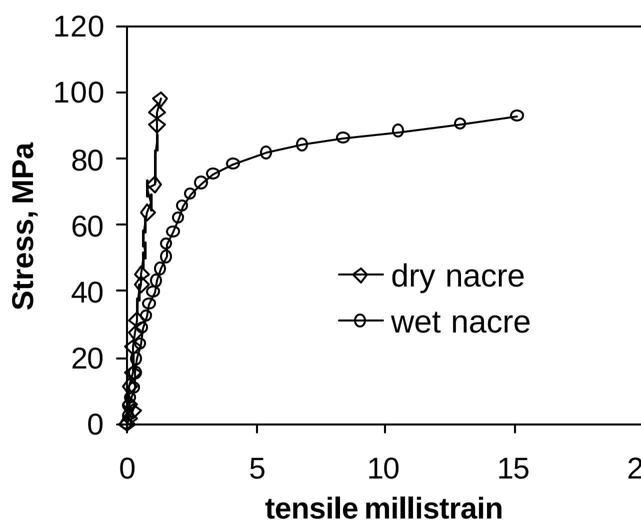

Figure 8: Stress-strain curve of nacre in tension along the tablets

This emphasizes the importance of the organic interface, which requires hydration to allow tablet sliding and large deformations. While hydrated, the organic network can withstand large stretching and might be acting as a lubricant.

The modulus was found to be 90 GPa for the dry condition and 50 GPa for the hydrated (wet) condition. This difference indicates that again the hydrated organic network promotes sliding of the tablets at very early deformation stages. This is consistent with observations of strain recovery and viscoelasticity made in the past [8]. Time dependant behaviors at small deformations indicate that in this regime the organic phase contributes to the mechanical behavior. At 70 MPa nacre softens: A hypothesis is that at this stage all potential sliding sites in the gage have been activated. Further SEM/AFM in-situ experiment will investigate relative tablet motion at micro- and nanoscale.

After this point there is some hardening effect until failure at 15 millistrains, which is a remarkable deformation level for a material composed of 95% of brittle ceramic.

CONCLUSIONS AND FUTURE WORK

The importance of the nacre microstructure with respect to its mechanical properties was discussed. The key role of the organic network was demonstrated, and future tests will further investigate its mechanical behavior. The high ductility achieved by tablet sliding probably dissipates a large amount of energy through friction and redistribution of the deformations, which could be the main factor of the toughness and impact resistance of nacre. The tests presented here are a first step to experimental investigations of deformations and fracture mechanisms at multiple scales and strain rates, with the objective to fully characterize the mechanisms involved in nacre deformation and fracture.

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