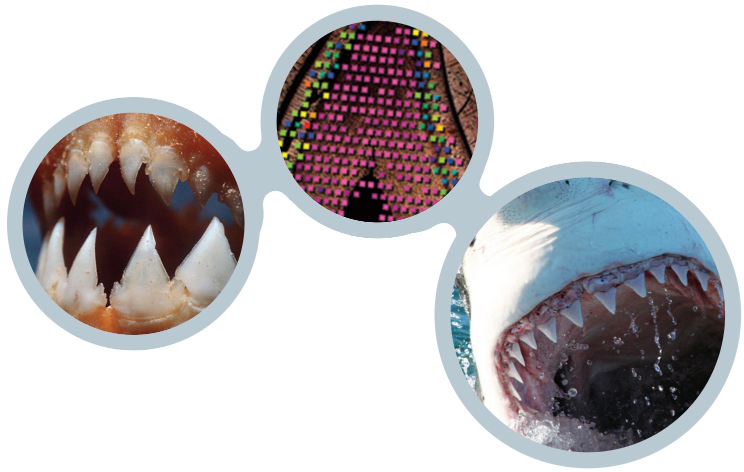


Application Note #1525

Nanoindentation on Marine Teeth: Studying Dentin and Enameloid in Dry and Hydrated Conditions

Biomimetic or bio-inspired materials are based on biological substances that have naturally developed over time to optimally and simultaneously serve multiple purposes in nature. Many of these complex systems are made up of organic and inorganic components structured in such a way that the overall mechanical properties of the material are vastly superior to those of the individual material constituents.[1,2] Such biological material systems not only possess exceptional mechanical properties; the aqueous environment processing conditions by which such systems are formed is also extraordinary. This is especially interesting considering some of the extreme processing conditions (temperature, pressure, etc.) required to produce high-performance man-made materials with comparable properties. These biological structures are hierarchically organized from the macro (structural) down to the nano (molecular) levels, and thus each of these structural levels are important to study in order to understand and ultimately replicate the mechanisms by which these materials are designed to function.

Methods

A Hysitron® TriboIndenter® was used to perform nanoindentation tests on one Great White Shark (GWS) and one Piranha tooth sample. The samples were embedded in epoxy and polished into a cross-section prior to testing. A diamond fluid cell Berkovich probe was used to perform nanoindentation tests on the samples. All tests were performed in load-controlled feedback mode to a peak force of 1000 µN. A load function consisting of a 5-second loading to peak force segment, followed by a 5-second hold segment, and 1-second unloading segment was used. First, maps of indents were performed along the entire surface of the samples. The indents were spaced approximately 50 to 100 µm apart and performed using automation. Next, the samples were hydrated in D-PBS solution and allowed to sit for approximately 2 hours prior to testing. An additional 20 indents were performed with the same load function in the dentin and enameloid regions of each sample to compare the dry and hydrated mechanical properties of the teeth.

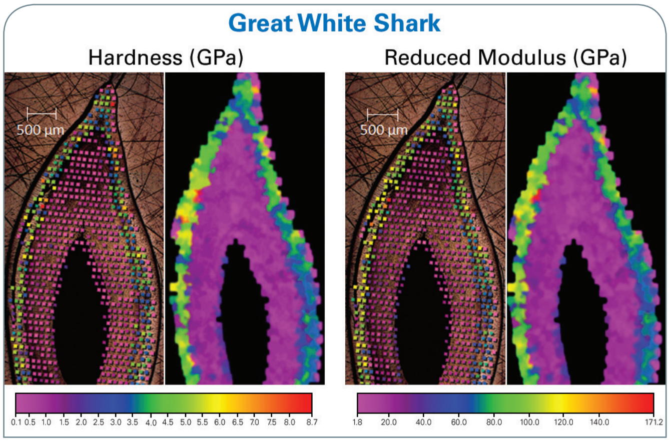


Figure 1. Hardness and reduced modulus surface plots from the series of 753 indents on the dry cross-sectioned GWS tooth sample.

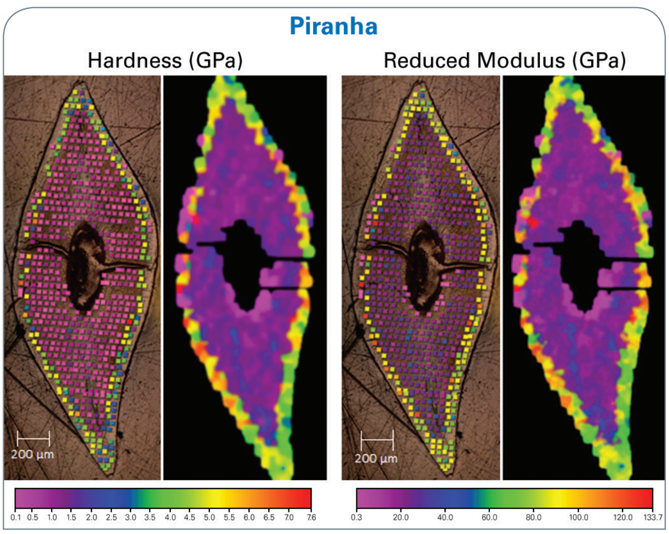


Figure 2. Hardness and reduced modulus surface plots from the series of 699 indents on the dry cross-sectioned Piranha tooth sample.

Results and Discussion

In both the GWS sample and the Piranha sample, the protective outer enameloid region of the sample showed about 5 times greater hardness value, around 4.1 GPa, than the inner dentin region, which had hardness values around 0.7 GPa. This trend was also seen in terms of reduced modulus with the enameloid of both samples being around 85 GPa compared to a dentin with a modulus around 21 GPa. Surface plots showing the indentation results on each sample can be seen in Figure 2. The analysis of the indents performed on the GWS and Piranha teeth in hydrated conditions provides valuable insight into the mechanical properties of marine teeth. The hardness and modulus of the dentin reduces significantly in hydrated conditions while there is no significant change in enameloid, which is highly mineralized and contains less than 5% organic components and water. Figure 3 shows representative force versus displacement curves from indents performed on the GWS sample that clearly illustrate mechanical property differences between regions in dry and hydrated states. The dry versus hydrated indentation results are compared in Figure 4.

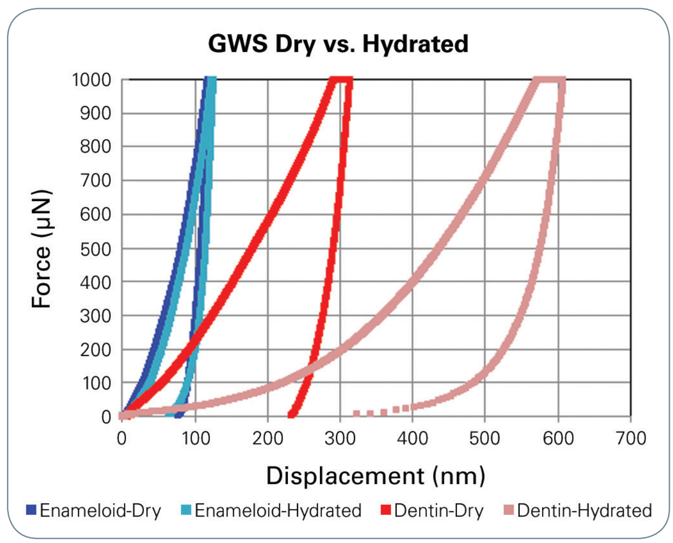
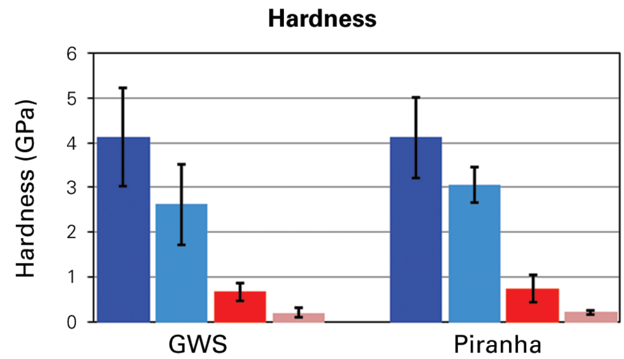
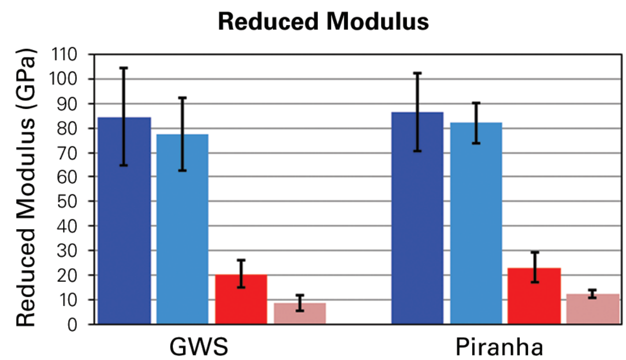


Figure 3. Force versus displacement curves from one representative indent on the enameloid and dentin regions of the GWS sample in dry and hydrated conditions.



	Enameloid-Dry	Enameloid-Hydrated	Dentin-Dry	Dentin-Hydrated
GWS	4.13±1.1	2.62±0.9	0.68±0.2	0.21±0.1
Piranha	4.12±0.9	3.05±0.4	0.75±0.3	0.22±0.04



	Enameloid-Dry	Enameloid-Hydrated	Dentin-Dry	Dentin-Hydrated
GWS	84.35±19.9	77.20±14.8	20.38±5.6	8.66±3.1
Piranha	86.49±15.9	81.92±8.4	22.98±6.0	12.38±1.6

Figure 4. Average hardness and reduced modulus results from the series of nanoindentation tests on the GWS and Piranha samples in dry and hydrated conditions.

Conclusion

The nanoindentation results show similar trends as found in previous microhardness measurements on similar sample sets.[1,2] Microhardness of enameloid and dentin regions of a GWS sample were found to be 1.56 and 0.31 GPa, respectively, while the enameloid and dentin regions of a piranha sample were found to be 1.36 and 0.30 GPa, respectively (both tested in dry conditions). The size of microhardness indents were ~60 μm and the distance between indents was ~200 μm, compared to ~2 μm sized indents spaced ~75 μm for nanoindentation testing. The increase in hardness from nanoindentation to microindentation is likely due to indentation size effect with respect to the small volume of material tested. A limited amount of data points can be obtained using microindentation on small biological materials such as piranha teeth, making nanoindentation a suitable and necessary test method when testing certain types of small biological samples.

References

1. Chen, P.-Y., et al., "Structure and mechanical properties of selected biological materials." *Journal of the Mechanical Behavior of Biomedical Materials*, **1** (2008) 208-226.

2. Chen, P.-Y., et al., "Predation versus protection: Fish teeth and scales evaluated by nanoindentation." *Journal of Materials Research*, **25** (1) (2012) 100-112.

Acknowledgements

Po-Yu Chen
University of California San Diego

Bruker Nano Surfaces Division
Minneapolis, MN · USA
+1.952.835.6366
productinfo@bruker.com

www.bruker.com/nanomechanical-testing