The Relationship Between Nanoindentation Testing Conditions and the Mechanical Properties of Bone

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Statement of Purpose: Measurement of the mechanical properties of bone is important for estimation of the local mechanical response of bone cells to loading experienced on a larger scale. An increasing number of measurements of the hardness and Young's modulus of bone tissue have been taken using nanoindentation (Turner CH. J Biomech. 1999; 32: 437-441. Rho JY. J Mat Sci; Mat Med. 1999; 10: 485-488). However, testing conditions have not been uniform. The interactions that can occur between testing condition parameters were considered in this study, and average hardness and Young's modulus were obtained as a function of nanoindentation creep testing conditions (maximum load, loading/unloading rate (both equal in magnitude), load-holding time, and indenter shape).

Methods: Two dehydrated monkey vertebra bone specimens, coded 4760 and 4814, and one dehydrated bovine cortical bone specimen were used in this work. Taguchi methods (Singer G. J Bone Joint Surg. 1998; 80: 1498-1506) were used to design experiments. Taguchi proposed techniques for performing fractional factorial experiments in the form of orthogonal arrays. The balance between parameter levels in the orthogonal array allows averages to be computed that isolate the effect of each parameter by averaging over the levels of the remaining parameters. Experiments were performed with a Hysitron Triboindenter™ in the load-control mode using Berkovich and 1 μm (radius) conospherical indenters. The Oliver-Pharr method [Oliver WC. J Mat Res. 1992; 7: 1564-1583] was used to determine Young's modulus *E* and hardness *H* for each indentation.

Results/Discussion: Figure 1 shows that both Young's modulus and hardness display a sharp initial increase with increasing maximum load, before decreasing above 1000 μN. The unloading rate shows less effect on Young's modulus and hardness during testing. In addition, modulus and hardness do not change significantly when the holding time is extending from 2 s to 15 s. In addition, tests were performed on bovine cortical bone using both 1 μm (radius) conospherical and Berkovich indenters, as shown in Table 1. The Berkovich indenter, which has a radius of curvature in the 100 to 200 nm range, yielded significantly higher modulus values accompanied by a significantly smaller contact area. This suggests that the anisotropic nature of bone and/or the nature of its plasticity have a significant effect on the unloading behavior during nanoindentation.

Conclusions: The maximum load had the largest effect on the measured modulus and hardness values compared to unloading rate and hold time. Combined with the results which essentially produced significantly different contact-affected zones (i.e., Berkovich vs. 1 μm tips), these results suggest that the anisotropy, scale of surface features (lamellae, osteons, etc.), and/or the plastic properties of bone (e.g., compaction, densification, etc.) will affect mechanical property measurements via indentation methods and should be accounted for. Future work includes investigation of enhanced load/loading rate ranges to track the mechanical response transition from nano to micro-scale, and to apply numerical analyses to simulate such transitions.

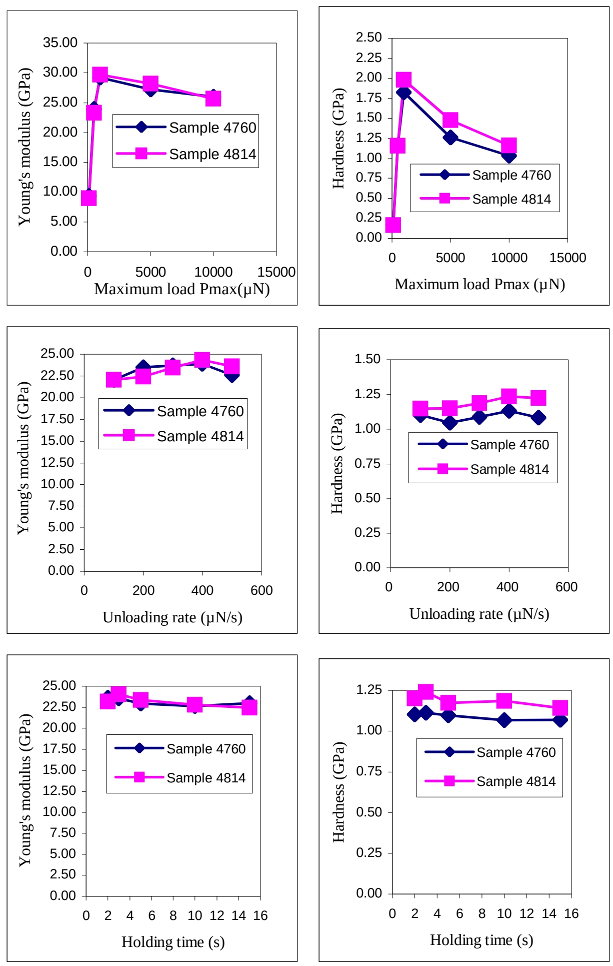

Figure 1. Relationships between Young's modulus, hardness, and testing conditions.

Indenter	E (GPa)	Area (μm^2)	Depth (nm)
Berkovich	39.9 (4.8)	3.4 (0.2)	1030 (75.6)
1 μm	12.0 (3.0)	33.2 (11.3)	1646 (121)

Table 1. Average modulus, contact area, and indentation depth for Berkovich and 1 μm conospherical tips (standard deviation in parentheses) on bovine cortical bone. Maximum load = 10 mN, load/unload rate = 375 μN/s, 5 s hold time.

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