❖ Nanoindentation of microstructural elements in bone cement

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Introduction: Poly(methyl methacrylate), or PMMA bone cement has been a critical material in total joint replacement for over four decades. Modifications used to improve the standard polymer powder/methacrylate monomer system have included the addition of fiber reinforcing phases, bioactive ceramics, antibiotic agents, etc. The microstructural phases in PMMA bone cement can affect its fracture and fatigue behavior [1]. Bone cements originally designed and used for artificial joints are now being used in, for example, vertebroplasty, with increased amounts of radiopacifier [2]. Understanding the microstructural contributions to mechanical performance may be an effective approach to designing or improving bone cements for different applications. In this study we report on using nanoindentation to investigate local properties of microstructural elements of bone cement.

Methods: Commercially available PMMA bone cement (Simplex P, Stryker, Inc.) was mixed following standard clinical procedures, including vacuum mixing. Beam bending specimens were sectioned into approximately 5mm thick wafers (cross sectional dimensions = 10mm x 10mm). Each wafer was polished with sequentially smaller alumina or diamond grit polishing powder, with a final polishing treatment using 0.5 µm powder. The specimens were indented using a nanoindentor (Hysitron Inc., TriboIndenter). Load controlled indentations were conducted to approximately 600 µN. Indents were attempted on both the polymer bead and inter-bead matrix phases of the bone cement. This method was applied to cements maintained in four different environments: dry at 37ºC, dry at room temperature, in saline at 37ºC, and in saline at room temperature. Indentations were made at different time intervals after curing: 1 to 5 hours, 1 week, 2 weeks, and 1 month. Using the indentor's microscope, the surface was examined to identify possible beads within the microstructure (*Figure 1*). A total of four indents were made inside and outside of each bead identified.

Results: We were able to place distinct indentations on the bead phase and in the interbead matrix of the bone cement specimens (*Figure 1*). The nanoindentor, with careful operation, was able to distinguishing between, and hence determine the local properties of different microstructural phases based on load-depth data analysis.

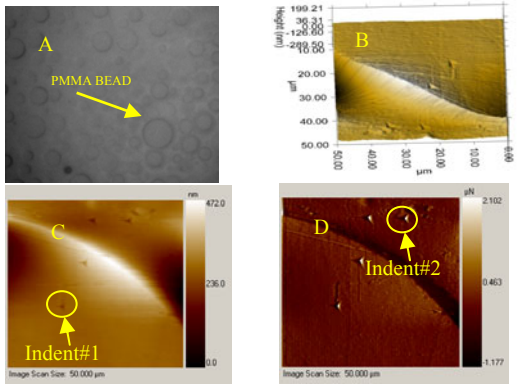

Figure 1: PMMA in 37ºC-dry 2weeks. A) 10X objective lens image, B)3D graph of the bead height(nm) vs. depth(µm), C)Topography showing Indent#1, in bea,d D)Gradient, Showing Indent #2, in matrix.

In each of the specimens tested, the Hardness (MPA) and Young's Modulus (GPA) were determined both inside and outside the beads. *Figure 2* shows the average Hardness inside the bead for each of the four conditions. The Young's Modulus was also graphed and compared.

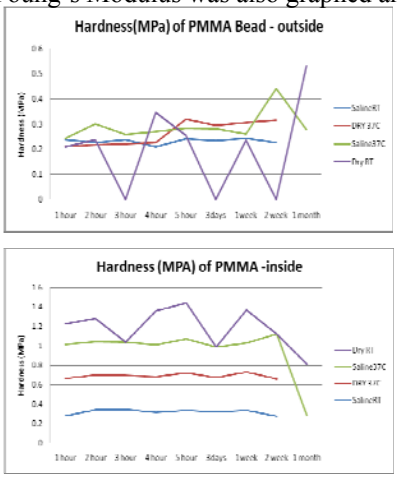

Figure 2: Hardness (MPA) graph for outside (matrix) and inside the bead.

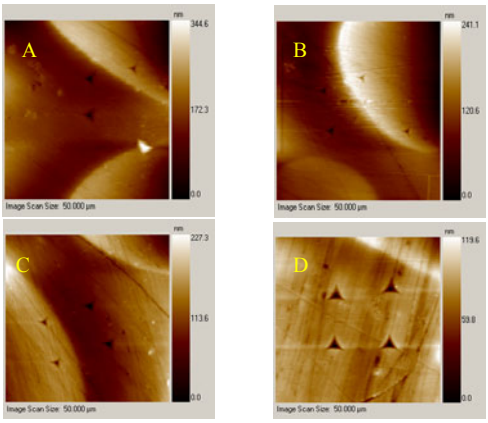

Figure 3:A, B,C and D show the topography indentations at 1 week under different conditions: A) 37ºC-dry, B) Room temp-saline, C) 37 ºC-saline, and D) dry- 1month.

Figure 3 shows the topography of the bone cement in different environmental conditions; it is clear that environmental factors affect the material.

Both hardness and Young's modulus were different inside and outside the bead, under all external conditions. For example, in indents #1 and #2 *(figure 1),* the Young's Modulus and Hardness were 6.85 GPa; 0.42 MPa and 5.79 GPa; 0.32 MPa respectively. After one month there was 52% increase in hardness, and a 76% increase in Young's Modulus in the dry-room temperature values over the saline in 37ºC.

Conclusions: This work demonstrates that we are able to determine properties in bone cements from local microstructural elements using nanoindentation.

References: [1] Molino, LN, Topoleski, LDT, *J Biomed Mater Res,* **31(1):** 131-137 1996.
[2] Kurtz, SM, Villarraga, ML, Zhao, K, Edidin, AA, *Biomaterials* 26 (17):3699-712, 2005