Comparison of Mechanical and Structural Properties of Zirconia Femoral Head Implants *in vitro* and *in vivo*

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Statement of Purpose: Tetragonal Zirconia Polycrystal (TZP) in biomedical applications was reported in 1969 [1] and introduced in 1985 as an alternative to alumina, because of zirconia's superior hardness and resistance to fracture [2]. Mean polyethylene were rate is 0.08 mm/ year in associated with zirconia heads and 0.17 mm/ year in associated with the cobalt-chromium heads (p =0.004) [3]. Thus, zircinia is the material of choice currently for ball heads and over 500,000 TZP ball heads have been implanted since 1985. Recently, several episodes of failure of zirconia ceramics femoral heads of total hip prostheses have alarmed the medical and scientific community regarding aging (phase transformation) problem in zirconia prostheses. The purpose of our study is to examine the surface and bulk mechanical properties of explanted TZP femoral heads compared to factory sealed controls. The hypothesis is that degradation of surface and mechanical properties occurs with increasing time of implantation because of the surface material phase transformation from tetragonal to monoclinic phase. The aim is to compare the control specimens to explanted samples that have been subjected to the rigors of implantation and determine the extent of degradation, if any, which occurs over time in the human body. We also correlate these properties on the controlled laboratory aged zirconia implants.

Methods: Zirconia femoral heads made by various manufactures were collected after revision total hip arthroplasty in University of Alabama at Birmingham (UAB) retrieval center, separated from their femoral stems and washed in an acetone bath to remove organic residue. Thermally aged zirconia femoral head samples without HIP treatment (first generation) supplied by Japan Medical Materials Corporation (JMMC) with monoclinic fraction (mol%) 0 (control), 10, 50 and 78 were also selected for analysis. Tetragonal and monoclinic phase composition of the retrieval samples was examined using glancing angle XRD with glancing angle of 3.5-degree. Peaks from the XRD output were compared to library controls and the tragonal/monoclinic zirconia ratio was determined using the integrated intensity on the tetragonal (101) and two monoclinic (-111) and (111) peaks as describe by Garvie *et* Nicholson [4]. The propagation of tetragonal to monoclinic phase transformation into the bulk was observed by SEM. In order to measure the surface hardness and Young's modulus of zirconia femoral head samples, nanoindentation measurements were carried out using a Nanoindenter XP (MTS Systems, Oak Ridge TN) system. Surface morphology were examines by Atomic Force Microscope (AFM)

Results / Discussion: Transformed monoclinic layer was clearly observed in SEM images and the penetration depth of the transformation layer is approx. 8 micron for 78 mole% of aged zirconia sample. Moreover, some nano-cracks were observed at grain boundary in the transformed monoclinic layer. XRD results reflect that TZP explanted implants shows varying degrees of monoclinic phase transformation with time of implantation. Again it was observed from the XRD analysis that the monoclinic to tetragonal phase ratio in zirconia implants increases with time of implants life *in vivo*. The variation of surface hardness and Young's modulus of different zirconia femoral head surfaces compared with time of implants *in vivo*. It has been also found that surface hardness and modulus decreases with increase of time of the implants *in vivo*. We also tried to correlate surface hardness and modulus with monoclinic to tetragonal phase ratio of different sample and nanoindentation testing revealed good correlation between increasing surface monoclinic phase and decreasing surface hardness and modulus. Mechanical properties of aged zirconia femoral heads samples show that the hardness and Young's modulus were decreased with increasing of aging. Control sample (zirconia with tetragonal phase) has hardness (H) and Young's modulus (E) of 16.6 GPa and 254.3 GPa respectively and it decrease to H = 9.7 GPa and E = 185.3 GPa with increase of monoclinic phase in zirconia (78 mol%). Atomic force microscope (AFM) and optical micrographs confirm the scratch marks and metallic transfer on the retrieved zirconia femoral head surfaces.

Conclusions: We have investigated the mechanical and structural properties of different explanted zirconia implants and try to correlate the properties on the laboratory aged implants. We have found a clear trend of monoclinic to tetragonal phase transformation in zirconia prostheses over time *in vivo* and also the amount of aging undertaken artificially in the laboratory. Mechanical properties mainly hardness (H) and Young's modulus (E) values were measured by nanoindentation technique on the surface of these implants. The results show that both H and E values decreases with increase of monoclinic phase in zirconia and confirm the phase transformation overt time *in vivo* and also *in vitro*. This work is supported by NIH (NIBIB) under Grant No. IROI-EB001715-01A2

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