PHASE TRANSFORMATION, ROUGHNESS AND MICROHARDNESS OF ARTIFICIALLY AGED MAGNESIA- AND YTTRIUM-STABILIZED ZIRCONIA FEMORAL HEADS

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Introduction: Zirconia oxide (ZrO_2) ceramics, used in orthopedic implant materials, are usually stabilized with magnesia oxide (Mg-PSZ; ASTM F2393) or yttrium (Y-TZP; ASTM F1873). Y-TZP is slightly stronger due to its near 100% tetragonal microstructure, but is also well-known to undergo tetragonal-to-monoclinic phase transformation at low temperatures [1]. The resulting net increase in surface roughness [2-4] may make Y-TZP less suitable as a bearing surface [4]. The purpose of this study was to characterize and compare the effects of artificial aging on Mg-PSZ and Y-TZP femoral heads.

Methods: Twenty never-implanted ZrO_2 femoral heads (Whiteside Biomechanics, Inc.) were used in this study, including ten Mg-PSZ (Xylon) and ten Y-TZP heads (CeramTec). Five heads from each group were artificially aged by autoclaving at 134 °C and 207 kPa [1] in stages up to a total of 49 hr. After each aging treatment, roughness (Ra) at the pole region of each head was evaluated by linear profilometry (Dektak ^{3}ST, ~2.5 μm stylus tip radius), and the roughness of each non-aged specimen was also measured. Monoclinic phase transformation was determined by X-ray diffraction [5] (PANalytical X'Pert Pro, Energy Conversion Devices, Inc.), and Vickers microhardness tests (ASTM C1327; Instron Tukon 2100) were employed to measure changes in surface hardness. Digital images of all specimens were obtained via light microscopy (Zeiss Axiotech) at 100x. Differences between groups were evaluated by a two-sided unpaired t-test, with $p < 0.05$ for significance.

Results: Figure 1 illustrates the significant increases in roughness of Y-TZP heads after each artificial aging treatment ($p \leq 0.02$). Mg-PSZ heads were much more stable, and did not exhibit any significant increase in roughness. The effects of 49 hr of artificial aging on the roughness, phase transformation, and microhardness of Y-TZP and Mg-PSZ are summarized in Table 1. For each parameter measured, Mg-PSZ specimens exhibited no significant difference between aged and non-aged groups. In contrast, aged Y-TZP specimens had significantly higher monoclinic phase content and surface roughness and significantly lower microhardness compared to non-aged heads. Figure 2 shows representative microscopy images from each group, with small surface scratches on both aged and non-aged Mg-PSZ. The surface of non-aged Y-TZP specimens was relatively featureless, but exhibited a rough "orange peel"-like appearance after 49 hr of artificial aging.

Discussion: The tetragonal-to-monoclinic phase transformation associated with aged Y-TZP heads has previously been correlated to increased roughness [2-4] and decreased hardness [6], as in the current study. However, to the best of our knowledge only one previous study [7] had directly compared changes in Mg-PSZ and Y-TZP due to artificial aging. As in the current study, artificial aging increased % monoclinic phase content only in Y-TZP specimens. Each hour of the aging protocol used was said to be equal to 4 years *in vivo* [1,8], but the absence of mechanical stresses and subsequent observations of retrieved heads [9] suggest that the conversion factor may be up to an order of magnitude too high. While Mg-PSZ is not quite as strong as Y-TZP, its surface appears to be much more stable over the long term and thus may be better suited as a bearing material. Previous studies reported the strength of Mg-PSZ to decrease with artificial aging [10,11], but the microhardness of aged Mg-PSZ was unchanged in the current study. The increased roughness of the aged Y-TZP specimens was manifested in an "orange peel"-like surface, but Mg-PSZ specimens only exhibited small scratches regardless of whether they were aged. Similar surface morphologies were found for retrieved Mg-PSZ and Y-TZP femoral heads [9]. Finally, although the roughness of Y-TZP significantly increased with aging, a roughness of ~12 nm is still very smooth. Although the software filter required to subtract the curvature of the heads obscured waviness data, preliminary 3D non-contact profilometry scans (Figure 3; ZoomSurf, Micro Photonics) indicate the "orange peel" has a period of 20-25 μm with peaks an average of ~28 nm high. Future research will investigate the clinical significance of the rougher surface of aged Y-TZP vs. aged Mg-PSZ via a hip wear simulator.

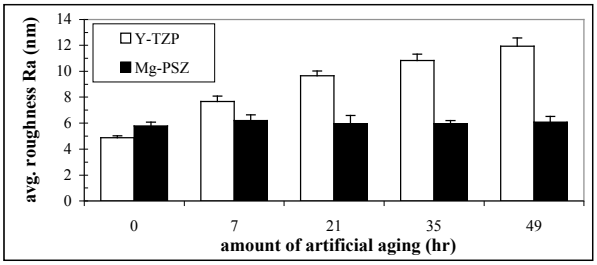


Figure 1. Average roughness (Ra) values of Y-TZP and Mg-PSZ heads as a function of artificial aging time. Error bars are standard deviation.

Table 1. Properties (avg. ± S.D.) of aged and non-aged femoral heads (n = 5). **A**. Y-TZP heads; **B**. Mg-PSZ heads. N.S. = not significant.

A. Y-TZP heads	Non-aged	Aged 49 hr	*p* value
Roughness Ra (nm)	4.89 ± 0.13	11.9 ± 0.63	< 0.0001
% monoclinic phase	5.58 ± 0.54	34.7 ± 1.7	< 0.00001
Microhardness (GPa)	12.2 ± 0.082	12.0 ± 0.11	< 0.01
B. Mg-PSZ heads	Non-aged	Aged 49 hr	*p* value
Roughness Ra (nm)	5.78 ± 0.28	6.08 ± 0.43	N.S.
% monoclinic phase	6.72 ± 1.4	7.76 ± 1.9	N.S.
Microhardness (GPa)	10.3 ± 0.14	10.4 ± 0.079	N.S.

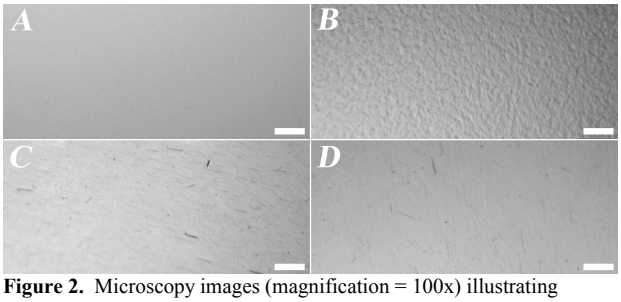


Figure 2. Microscopy images (magnification = 100x) illustrating topography of ZrO_2 heads: **A**. Y-TZP, not aged; **B**. Y-TZP, aged 49 hr; **C**. Mg-PSZ, not aged; **D**. Mg-PSZ, aged 49 hr. Bars represent 50 μm.

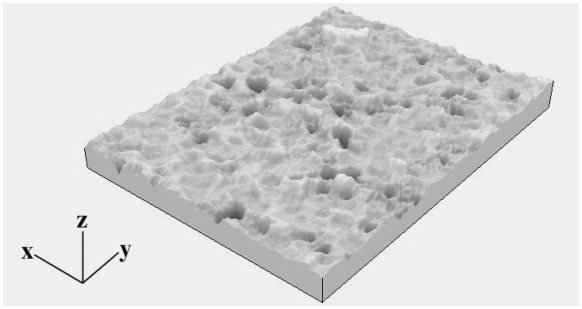


Figure 3. 3D non-contact profilometry scan of aged Y-TZP specimen. Lateral axes = 148 μm x 198 μm; z-axis scale = 182 nm, to show detail.

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