



Hydrothermal degradation and wear behaviour of 3Y-TZP zirconia ceramic with ternary and quaternary oxide additions

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Yttria stabilised tetragonal zirconia polycrystal (Y-TZP) ceramics have attracted particular attention due to their outstanding mechanical properties amongst advanced engineering ceramics due to martensitic phase transformation mechanism. Y-TZP can exhibit bending strength of more than 1000 MPa, with a toughness of approximately 5–10 MPa \sqrt{m} which is remarkable properties withing oxide ceramics. It has been opened the way to new structural devices and designs not available with standard, more brittle ceramics like alumina. Excellent tribological characteristics, paired with bioinertness are important for products in knee and hip endoprosthesis. This combination is the prerequisite for long-term durability of the product, since there are up to five million load cycles per year and therefore approximately 125 million load cycles for an average implant durability of 25 years with this application.

Y-TZP is the main reinforcement agent in the latest generation and word leading hip joint prosthesis of CeramTec.

Drawback of such a transformation is hydrothermal degradation .The attempt is to slow down the aging kinetic in this type of material.

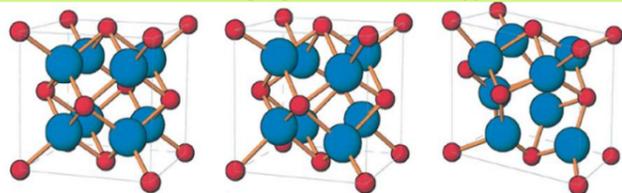


Figure 1: polycrystal ZrO₂ lattice. Left to right: cubic, tetragonal, monoclinic. Blue spheres: Zr, Red spheres: O

The applications where zirconia has been successful in the bio medical area are:

- Load bearing implants
- endodontic posts and brackets
- crowns
- bridges for restorative dentistry

In the current work, the hydrothermal degradation and wear behaviour of a 3Y-TZP with and without small additions of Al₂O₃ and La₂O₃ was investigated. Degradation was done at 134°C in superheated steam. This temperature was chosen as it is believed that one hour in the superheated steam represents one year in vivo. Ceramics disks were prepared by **co-milling** the oxide powders in methanol, **uniaxial, isostatic pressing** followed by sintering. Sintering conditions were chosen to ensure that the same grain size and fracture toughness were achieved in all materials which were close to theoretical density.

Table 1: material property after sintering

	Grain size (nm)	Density (gr/cm ³)	Vickers hardness(GPa)	Fracture toghness (MPa.m ^{1/2})
Z	285±8	6.09	12.3	5.62
AZ	302±4	6.1	12.4	5.70
LAZ	265±2	6.09	12.7	5.63

Z: 3Y-TZ, AZ: 0.1 wt% Al₂O₃ doped 3Y-TZP, LAZ: 0.1 % wt La₂O₃ 0.1% Al₂O₃ doped 3Y-TZP

Prior to degradation test in the conventional degradation bomb fig (2) and wear test in the UMT tribometer fig (3), the surfaces were prepared by careful polishing to produce an extremely flat surface R_a<3 nm. Thermal etching eliminated residual stress produce in polishing step and also in this way the starting surface was fully tetragonal zirconia.



Figure 2: sealed Teflon lined general-purpose bomb



Figure 3: UMT tribometer

To characterise the aged surface early stages of degradation were followed by **atomic force microscopy** while the overall kinetics was quantified through **X-ray diffraction** and measurement of the transformation depth using surface cross-sections in SEM imaging.

Materials with ternary oxide additions exhibited superior degradation resistance to the base zirconia material fig (4). In addition, the mechanism of degradation appeared to be fundamentally different. **Raman spectroscopy, scanning electron microscopy, contour Gt, focused ion beam microscopy** was applied to study of the aged and worn behaviour. Wear test was done for 24 hours in water and bovine lubricants. Wear rate of the doped material was less than undoped materials.

SURFACE

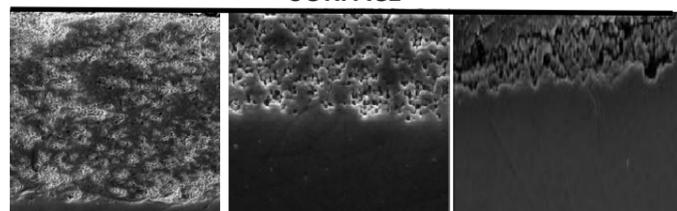


Figure 4: SEM secondary images of cross section of degraded layer of each material. 24h exposure time, 134 °C. Left to right Z, AZ, LAZ. LAZ sample shows the minimum degraded depth while Z base material with no dopant addition shows maximum depth of the degraded layer.

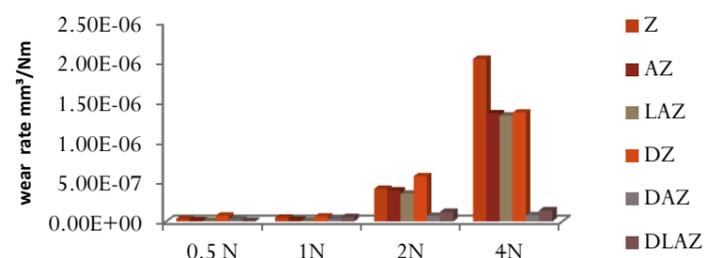


Figure 5: wear rates for the reciprocating wear tests of all materials against high purity alumina ball lubricated with ultra pure water. (D: degraded material)



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