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# Effect of heat treatment and in vitro aging on the microstructure and mechanical properties of cold isostatic-pressed zirconia ceramics for dental restorations

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ARTICLE INFO

*Article history:*
Received 14 April 2013
Received in revised form 8 July 2013
Accepted 21 May 2014

*Keywords:*
Y-TZP zirconia ceramics
Nano-hardness
Elastic constant
Heat-treatment
Aging
Monoclinic zirconia
XRD
FTIR
SEM

ABSTRACT

*Objectives.* The temperature variations during the veneering firing cycles of a zirconia dental ceramic can negatively affect its mechanical properties. A possible synergistic effect of both heat-treatment and aging while exposed to the oral environment could result to catastrophic failure. The aim of the present study was to investigate the effect of heat treatment followed during veneering and in vitro aging on the mechanical and microstructural properties of zirconia dental ceramics.
*Methods.* Three specimens from each of two zirconia blocks (Ivoclar IPS e.max ZirCAD (IV) and Wieland ZENO Zr (WI)) were cut by CAD/CAM technology, fully sintered and polished. Each one was cut in four equal parts. One part was used as control (C), one was heat-treated (H), one was aged (A) (134 °C, 2 bar, 10 h) and one was heat-treated and subsequently aged (HA). The mechanical properties (nano-hardness ($H$) and elastic modulus ($E^*$)) were investigated by nano-indentation tests while the surface characterization was carried out with XRD, FTIR and SEM.
*Results.* Different treatments on IV and WI samples resulted in a reduction of both $H$ and $E^*$ values, however the differences were not statistically significant ($p > 0.05$). The combination of treatments imposes an overall effect ($p < 0.001$), enhancing the influence on both $H$ and $E^*$ values. This reduction in mechanical properties was followed by an increase of monoclinic content. Greater variations in both $H$ and $E^*$ values were recorded for WI samples.
*Significance.* The clinical performance of zirconia dental ceramics may be affected during firing and aging resulting in increased probability of failure.
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http://dx.doi.org/10.1016/j.dental.2014.05.017
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## 1. Introduction

Y-TZP zirconia ceramics have attracted the interest of dental technology in manufacturing core materials for fixed all-ceramic restorations due to their excellent mechanical properties (i.e. bending strength and toughness) [1,2]. The high strength of Y-TZP zirconia ceramics is attributed to a toughening mechanism related to the transformation of the tetragonal phase (*t*), to the monoclinic (*m*) phase (natural equilibrium) [3]. This $t \rightarrow m$ transformation may be triggered by an applied stress and is associated with a net 4 vol% expansion due to the larger volume occupied by the monoclinic phase compared to the tetragonal one [4]. In case of a pre-existing crack the volume expansion associated to the $t \rightarrow m$ transformation, enables the closure of the crack and diminishes its propagation. This crack propagation prevention minimizes the risk of catastrophic failure of the material due to fracture and is called the "transformation toughening mechanism" [5].

The main drawback of Y-TZP zirconia ceramics is their sensitivity to low temperature degradation (LTD) – aging that leads to exaggerated $t \rightarrow m$ transformation and a degradation that starts on the surface and propagates into the depth of the material, diminishing its mechanical properties [6–8]. This is of particular interest for dental zirconia ceramics, as their degradation in the oral environment due to the exposure to oral fluids and mechanical stress over prolonged periods of time cannot be overlooked. Although minimally exposed to the oral environment, the margins of zirconia restorations allow a continuous contact of zirconia core with saliva or other fluids that may start and progressively lead to the degradation of the material. Furthermore, there is growing popularity of monolithic zirconia restorations where much larger areas of zirconia are in contact with the oral environment.The majority of the models proposed to explain the spontaneous $t \rightarrow m$ transformation taking place during LTD-aging, are either based on the formation of zirconium hydroxides (Zr-OH) [9,10] or yttrium hydroxides ($Y(OH)_3$) or Y(O)OH) [11] due to the diffusion of water through the material, promoting phase transition with local stress concentration or variation of the yttrium/zirconium ratio. According to the most recent proposed model [12] oxygen anions are responsible for the transformation nucleation and therefore for the LTD. Due to volume changes the transformed grains cause microcracks and the material becomes degraded.

Currently, accelerated tests at intermediate temperatures (100–300 °C) are the only basis for the estimation of the transformation rate and, hence, of the product lifetime [13], although a lot of controversy exists about the validity of the extrapolated predictions [14]. According to Chevalier [13] aging of zirconia with 1 h of autoclave treatment at 134 °C and 2 bar pressure results in a significant $t \rightarrow m$ transformation that has theoretically the same effect as 3–4 years in vivo, while the ISO standard imposes a maximum of 25 wt% of monoclinic zirconia to be present after an accelerated aging test conducted for 5 h at 134 °C and 2 bar [15]. However Lughi and Sergo [14] state that these accelerating in vitro tests provide only a rough estimate and any extrapolation could lead to a significant error in estimating body temperature lifetimes.

To achieve highly aesthetic zirconia restorations, zirconia core is veneered with a feldspathic porcelain coating and is subjected to firing at high temperatures (750–900 °C) followed by subsequent cooling. This process takes place at least once, but usually it takes two to five firing cycles in order to obtain an aesthetically acceptable restoration [16,17]. The fracture strength [16], microhardness [17] and flexural strength [17] are significantly reduced after veneering firing cycles. According to Oilo et al. [17] this effect is predominantly observed after the first firing cycle while subsequent cycles do not cause a further deterioration of properties. The reduced mechanical properties of zirconia ceramics after heat-treatment have been attributed to residual compressive stresses due to milling and various processing steps – such as grinding and sandblasting – that are released during the heat treatment, as well as to the $t \rightarrow m$ transformation and the alteration of the grain size that take place during the firing cycles of the veneering process [17]. This degradation of mechanical properties can further reduce the strength of the material after exposure to the oral environment, so a possible synergistic effect of both heat-treatment and aging could result to a probably irreversible premature failure.

The aim of the present work was to investigate the effect of heat treatment followed during veneering and the in vitro aging on critical mechanical and microstructural properties at the nano-scale of cold isostatic-pressed zirconia ceramics for dental restorations. The research hypotheses investigated were:

1. the heat treatment does not affect the nano-hardness (*H*) and the elastic ($E^*$) constant of the zirconia ceramic cores;
2. the in vitro aging does not affect the nano-hardness (*H*) and the elastic ($E^*$) constant of the zirconia ceramic cores;
3. the combination of heat-treatment and subsequent in vitro aging does not affect the nano-hardness (*H*) and the elastic ($E^*$) constant of the zirconia ceramic cores.

## 2. Materials and methods

Three bar-shaped specimens (28 mm × 4 mm × 2 mm) milled from two zirconia blocks [Ivoclar IPS e.max ZirCAD (LOT: PX0075), and Wieland ZENO Zr (LOT:20090728-07)] by the CAD/CAM technology were sintered to full density, polished with diamond pastes of 3 and 1 μm under running water until a mirror-like surface was achieved, and cut with a diamond bur under water cooling into four equal parts each (7 mm × 4 mm × 2 mm). One part was used as control (designated as C), one was heat treated (designated as H), one was aged (designated as A) [steam 121 °C/2 bar/10 h, Kavo autoclave sterilizer 2100 (KavoDental, Biberach/Riss, Germany)] and the last was heat-treated and subsequently aged (designated as HA). The applied heat-treatment corresponded to four firing cycles of the veneering porcelain as shown in Table 1 according to manufacturers' instructions.

### 2.1. *Nano-mechanical properties evaluation*

The *H* and $E^*$ values were assessed by means of a Hysitron Ubi-1 TriboLab modular nano-indentation instrument (Tribolab,

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