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# Long-term stability of an injection-molded zirconia bone-level implant: A testing protocol considering aging kinetics and dynamic fatigue

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## ABSTRACT

**Objective.** Separately addressing the fatigue resistance (ISO 14801, evaluation of final product) and aging behavior (ISO 13356, standardized sample) of oral implants made from yttria-stabilized zirconia proved to be insufficient in verifying their long-term stability, since (1) implant processing is known to significantly influence transformation kinetics and (2) aging, up from a certain level, is liable to decrease fatigue resistance. Therefore, the aim of this investigation was to apply a new testing protocol considering environmental conditions adequately inducing aging during dynamic fatigue.

**Methods.** Zirconia implants were dynamically loaded ( $10^7$  cycles), hydrothermally aged ( $85^\circ$ , 60 days) or subjected to both treatments simultaneously. Subsequent, monoclinic intensity ratios ( $X_m$ ) were obtained by locally resolved X-ray microdiffraction ( $\mu$ -XRD<sup>2</sup>). Transformation propagation was monitored at cross-sections by  $\mu$ -Raman spectroscopy and scanning electron microscopy (SEM). Finally, implants were statically loaded to fracture. Linear regression models (fracture load) and mixed models ( $X_m$ ) were used for statistical analyses.

**Results.** All treatments resulted in increased fracture load ( $p \leq 0.005$ ), indicating the formation of transformation induced compressive stresses around surface defects during all treatment modalities. However, only hydrothermal and combinational treatment were found to increase  $X_m$  ( $p < 0.001$ ). No change in  $X_m$  was observed for solely dynamically loaded samples ( $p \geq 0.524$ ). Depending on the variable observed, a monoclinic layer thickness of 1–2  $\mu$ m (SEM) or 6–8  $\mu$ m (Raman spectroscopy) was measured at surfaces exposed to water during treatments.

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*Significance.* Hydrothermal aging was successfully induced during dynamic fatigue. Therefore, the presented setup might serve as reference protocol for ensuring pre-clinically long-term reliability of zirconia oral implants.

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## 1. Introduction

Based on well-documented clinical outcome, oral implants made from titanium are a valid treatment option in patients care when replacing missing or deteriorated teeth [1]. For patients opposing metals, oral implants made from polycrystalline zirconium dioxide represent the only nowadays available alternative [2]. Zirconium dioxide ( $ZrO_2$ , zirconia) presents the phenomenon of allotropy, characterized by temperature-dependent crystal structures [3]. When stabilizing the tetragonal phase at room temperature, the polymorphous character of zirconia allows for stress-induced phase transformation toughening (PTT) [4]. This involves the transformation of metastable tetragonal crystallites to the monoclinic phase in the crack tip stress field.  $T$ - $m$  transformation is accompanied by volume expansion and induces compressive stress in the zirconia matrix, acting to resist crack propagation. Yttria-stabilized tetragonal zirconia (Y-TZP) is therefore known as ceramic material with comparatively high fracture toughness and strength [5]. On the other hand, a potentially serious limitation of stabilized zirconia for its application in the oral environment was discovered: Y-TZP is prone to low temperature degradation (LTD; “aging”) in presence of water vapor [6]. In contrast to stress-triggered PTT, LTD is defined as spontaneous and progressive  $t$ - $m$  transformation at low temperatures in the presence of water molecules. Once started, phase transformation continuously progresses from the surface into the volume in a layer-by-layer manner, following a linear time law [7]. Aging can result in intergranular micro-cracking, surface-roughening and, up from a certain level, in reduced strength [8]. Finally, PTT and LTD are based upon the same phenomenon and using Y-TZP for the fabrication of biomedical implants, one might not exploit the advantage of the former without being exposed to the risk of the latter. To date, it is unclear whether LTD is of clinical relevance when using Y-TZP as dental implant material. Given the importance of aging kinetics and fatigue resistance of Y-TZP implants, a testing protocol should account for the specific brittle nature of ceramics and the specific behavior of zirconia in terms of phase transformation [9]. To date, two separate international ISO standards are available: ISO 13356 imposes that a maximum of 25 wt% of monoclinic phase is present in test specimens after an accelerated aging test (5 h at 134 °C in a humid atmosphere with an air pressure of 0.2 MPa) [10], whereas ISO 14801 requires a dynamic loading procedure subjecting the implants to different loads, to finally obtain a fatigue resistance curve [11]. Regrettably, only the latter standard evaluates the “market-ready” product but it misses to provide any environmental condition that induces aging. Several hundred failures of hip prosthesis reported in a short time period might have shown that this can be

considered irresponsible [12]. ISO 13356 prescribes the evaluation of test specimens with a simplified geometry (bending bars) and a polished surface. However, complex geometries as well as post-processing steps like micro-roughening to enhance osseointegration are known to significantly compromise the mechanical properties and, even more important, accelerate the aging kinetics [13]. In conclusion, ISO 13356 does not account for the real transformation rate of samples with roughened surface and a non-porous bulk [9]. Even if experimental methods to predict and simulate LTD in a given zirconia ceramic are deemed to be highly accurate [7,14], the impact on reliability of final implant designs in combination with stress-inducing dynamic fatigue has not yet been evaluated. There is no standardized protocol available, allowing to assess the effect of aging and simultaneous dynamic fatigue on the mechanical properties of a given type of implant. For instance, an escalating effect of spontaneous (LTD) and stress-triggered (PTT)  $t$ - $m$  transformation (e.g. when implants are subjected to dynamic load in the humid environment of the oral cavity) might be possible.

Therefore, the purpose of the present investigation was to measure the amount of phase transformation, either induced by applied mechanical stress or spontaneously occurring during a hydrothermal treatment, and evaluate its impact on the fracture strength of a screw-retained zirconia prototype implant produced by ceramic injection-molding. The issue of a potential escalating effect of both phenomena should be addressed.

## 2. Materials and methods

### 2.1. Sequence of experimental procedures

A total of 32 implants were used for the experiment and divided into four groups of eight samples each. Three groups (hereinafter referred to as “test groups”) were subjected to treatments, intended to induce phase transformation: two groups were either mechanically loaded in a chewing-simulator (ZM) or aged by means of a hydrothermal treatment (ZH), whereas the third group underwent both mentioned treatments simultaneously (ZMH). The remaining zirconia group received no treatment (ZN) and served as reference for comparison. Subsequently, phase transformation of all zirconia groups was evaluated by locally resolved X-ray microdiffraction ( $\mu$ -XRD<sup>2</sup>) at 4 different surface positions per sample ( $n=32$ ). Furthermore, a cross-section of one sample per test group was evaluated by Raman spectroscopy and scanning electron microscopy ( $n=3$ ). Except for the three implants used for cross-sections, all implants were finally loaded to fracture in a static loading test and statistically analyzed.

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