



Low-temperature degradation in zirconia with a porous surface

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ABSTRACT

Today there is growing interest in zirconia in the dental field, but its use is still recent. Dental zirconia is mainly found in the form of yttria-stabilized zirconia crowns, bridges and abutments, and several companies are developing zirconia implants as an alternative to the standard biomedical grade titanium. In order to favor bone in-growth and osseointegration of zirconia implants, several strategies are now being explored to process rough and/or porous surfaces. The aim of this paper was to evaluate the resistance to environmental degradation of yttria-stabilized zirconia coated with a porous layer. We show that specific conditions of processing to generate the porous layer at the surface can lead to an accelerated tetragonal–monoclinic transformation of the porous layer in the presence of water. The impact of the transformation was evaluated in terms of structural integrity. Bending strength was not affected but the cohesion of the porous coating and its adhesion with the dense part deteriorated. We show that other processing conditions insure much better stability. Low-temperature degradation resistance of such porous surfaces should therefore be carefully followed and controlled in order to avoid critical problems in the future.

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1. Introduction

In the 1990s, yttria-doped tetragonal zirconia polycrystal (Y-TZP) became a popular alternative to alumina as a structural bioceramic because of its substantially higher fracture toughness and strength [1,2]. The use of Y-TZP, first in orthopedics, opened the door towards new implant designs that were not possible with the more brittle alumina. Early examples are small Y-TZP femoral heads and Y-TZP knee joints, and more recently there has been a large range of dental devices [3] (crowns, bridges, abutments and more recently implants). Biomedical grade Y-TZP exhibits the best mechanical properties of single-phase oxide ceramics: it is the consequence of phase transformation toughening, which increases its crack propagation resistance. Stress-induced phase transformation involves the transformation of metastable tetragonal grains to the monoclinic phase at the crack tip. It is accompanied by a volume expansion and induces compressive stresses, which hinder crack propagation [4]. Y-TZP ceramics can exhibit toughness higher than 6 MPa \sqrt{m} and strength larger than 1000 MPa. On the other hand, due to this metastability, Y-TZP is prone to low-temperature degradation (LTD, also referred as aging) in the presence of water [5]. Aging is a progressive tetragonal to monoclinic transformation at the surface triggered by water molecules, which results in sur-

face modification (roughening) and microcracking. This inevitably influences the performance and reliability of zirconia devices and reduces their lifetime [6]. In the case of orthopedic devices, the sequence of degradation and its impact on implants has been described well over the last 10 years [6–9]:

- The wear performance of hip joint heads is reduced, as roughening increases the wear rate of the antagonist part of the prosthesis (generally polyethylene), while the coupled effects of microcracking and wear also generate pull-out of zirconia grains.
- The extension of the microcracked, transformed zone also generates large cracks that may grow with the transformed zone and lead to delayed failure.

Y-TZP manufacturers considered this problem as a minor issue until 2001, when hundreds of failures of Y-TZP femoral heads were reported within a very short period [6,8,9]. Even if limited in time and number, and clearly identified to be process controlled, these events have had a negative impact on the use of zirconia in orthopedics. More importantly, clinical reports show that Y-TZP can exhibit a progressive aging degradation even under well-controlled process conditions, which limits its long-term stability [6]. Y-TZP is therefore no longer used in orthopedics and major companies in this field have switched to alumina–zirconia composites. In contrast, monolithic 3Y-TZP is increasingly used in dental applications [3]. This is due to its excellent strength and toughness, but also undoubtedly to its aesthetic properties (translucency and

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possibility of being colored), the availability of new powders with a superior aging resistance (i.e. TZ3Y-E, E grade being relative to small alumina doping, as proposed by today by Tosoh, Japan) and its ability to be manufactured by computer-aided design and manufacturing procedures. Even if few general papers devoted to dental zirconia underline the need to “keep in mind that some forms of zirconia are susceptible to aging and that processing conditions can play a critical role on the low-temperature degradation (LTD) of zirconia” [3], the problem of LTD in dental zirconia seems to be underemphasized so far. However, a recent paper from Lughii and Sergo [10] showed that LTD might be relevant also for dental ceramics.

Most of the research works on zirconia dental ceramics today focus on mechanical properties of the devices [11], their fatigue resistance [12] and surface modifications [13–15] that could enhance bone in-growth. This is particularly the case for dental implants, where a direct contact with bone is present. The search for better implant–bone integration has led researchers and companies to develop methods to increase surface roughness and/or to create microporosity. Among them are sandblasting [13], chemical etching [14], spray drying of a bioactive phase and coating by a porous zirconia layer [15]. All these surface modifications may have a positive effect on bone apposition and bone in-growth, but also could lead to a modification of the stability of the tetragonal phase under a humid atmosphere.

Therefore the aim of the present paper was to evaluate the resistance to environmental degradation of biomedical grade yttria-stabilized zirconia samples coated with a porous zirconia layer, processed via two slightly different strategies. The global objective was to predict their *in vivo* behavior and the impact of aging on structural integrity. The role of process conditions on LTD was also investigated.

2. Materials and methods

2.1. Experimental samples

Nobel Biocare provided the experimental samples, which consisted of cylinders of length 15 mm and diameter 5 mm, with a small groove of 0.5 mm depth at 5 mm of the edge (the “ideal shape” of a dental implant; see Fig. 1). They were processed from a biomedical grade 3 mol.% yttria-stabilized zirconia spray-dried powder (Tosoh TZ-3Y SB-E, Tokyo, Japan). Table 1 summarizes the two different process steps used for the manufacture of the samples that might be used for real implants. For both processes, a porous surface was achieved by coating 12 mm of the cylinder (including the groove) with a slurry containing zirconia powder and a pore former (patent application SE03022539-2). Further

Table 1

Process steps in manufacturing P1 and P2 3Y-TZP samples (differences are in bold).

Step	Process 1 (P1)	Process 2 (P2)
1	Cold isostatic press of 3Y-TZP powder	Cold isostatic press of 3Y-TZP powder
2	Mill into cylindrical shape	Mill into cylindrical shape
3	Pre-sinter	Pre-sinter
4	Mill into final shape	Mill into final shape
5	Sintering to full density	Spray of the porous coating
6	Hot isostatic press	Sintering of both bulk and coating
7	Reoxidation	Hot isostatic press
8	Spray of the porous coating	Reoxidation
9	Sintering of the porous coating	Packaging
10	Packaging	

sintering of the sample yielded to the burn-off of the pore former and to a porous surface. The two processes (hereafter called P1 and P2) varied mainly by the fact that the porous surface was coated on a pre-sintered (P2) or a sintered (P1) ceramic piece. For P2 samples, sintering of both the core and the porous surface therefore occurred at the same time. In contrast, sintering of the porous surface was performed on an already dense core for P1. In both cases, the presence of the coating gave rise to a rough and porous surface 15 μm thickness with 45 vol.% interconnected porosity. Typical microstructure of the porous surface is given in Fig. 1.

2.2. Accelerated aging tests and aging kinetics

Aging of the samples was evaluated by performing accelerated aging tests in water steam at 140, 134 or 120 $^{\circ}\text{C}$, or in water at 90 $^{\circ}\text{C}$, for different durations, depending on the treatment temperature. Aging kinetics were determined with two methods:

- Follow up of tetragonal–monoclinic (*t*–*m*) transformation of the porous coating by X-ray diffraction (XRD).
- Determination of the depth of the transformation zone on cross-sections of the cylinders after a given period of aging.

2.2.1. Follow-up of *t*–*m* transformation of the porous coating by XRD

The monoclinic content was measured by an XRD technique (Cu K_{α} radiation) in a θ – 2θ mode ($2\theta \in [27^{\circ} - 33^{\circ}]$) on a Bruker D8 Advance instrument (Bruker, Karlsruhe, Germany; scan speed of $0.2^{\circ} \text{min}^{-1}$ and step size of 0.02°). Monoclinic content was then calculated using the equation given by Garvie and Nicholson [16]:

$$X_m = \frac{I_{m(\bar{1}11)} + I_{m(111)}}{I_{m(\bar{1}11)} + I_{m(111)} + I_{t(101)}} \quad (1)$$

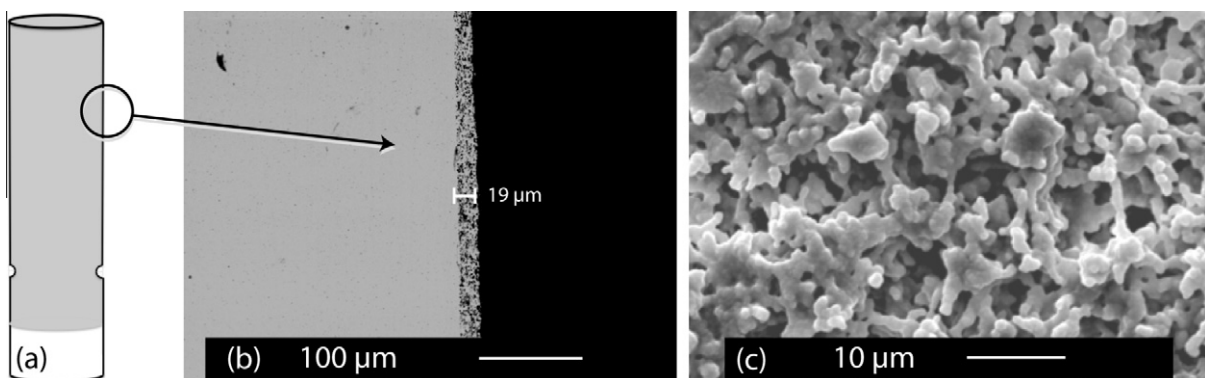


Fig. 1. Geometry of the cylindrical samples, with emphasis on the zone coated with the porous layer in gray (a), cross-section of a sample in a zone coated with the porous layer before aging (b) and microstructure of the porous coating (c).

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