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Complexity of the relationships between the sintering-temperature-dependent grain size, airborne-particle abrasion, ageing and strength of 3Y-TZP ceramics

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ABSTRACT

Objectives. This study was designed to explore the complex relationships between the sintering-temperature-dependent grain size, airborne-particle abrasion, ageing and strength of 3Y-TZP ceramics.
Methods. Biomedical grade 3Y-TZP powder was used to fabricate 180 discs. Half of them were sintered at 1400 °C for 2 h and half at 1500 °C for 2 h. A total of 18 groups of 10 were formed and subjected to the fully crossed experimental protocol of airborne-particle abrasion with Al_2O_3 at 2.5 bar (no abrasion, 50 μm, 110 μm) and accelerated ageing at 134 °C (no ageing, 12 h, 48 h). The relative amount of monoclinic phase was determined with XRD. The biaxial flexural strength was measured and statistically analyzed using the three-way ANOVA followed by predetermined contrasts and Tukey's HSD test ($\alpha = 0.05$).
Results. The low-temperature-sintered, fine-grained ceramic exhibited an excellent ageing resistance, while the high-temperature-sintered, coarse-grained ceramic experienced a higher surface strengthening and a substantially improved ageing resistance with respect to the airborne-particle abrasion. The overall performance of this material was superior.
Significance. Our results show that the sintering temperature has a minor effect on the flexural strength, but it plays a crucial role in the surface strengthening and the ageing behaviour of 3Y-TZP dental ceramics.

1. Introduction

Yttria-partially-stabilized tetragonal zirconia polycrystalline ceramic (3Y-TZP) represents an important prosthodontic therapy option in aesthetically pleasing, all-ceramic dental restorations. Its main advantages are its chemical inertness, bio-compatibility and exceptionally high strength and fracture toughness. The last of these are attributable to its ability to undergo a martensitic phase transformation (*t-m*) of the

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thermodynamically metastable tetragonal grains into the stable monoclinic form as a result of mechanical stress, thereby developing transformation toughening [1].

The phase instability of 3Y-TZP ceramics, however, also has a disadvantageous side, known as low-temperature degradation (LTD), i.e., ageing. When the material is exposed to moisture at slightly elevated temperatures the tetragonal grains on the surface will start transforming spontaneously to the monoclinic phase [2]. Due to the volume expansion associated with the *t-m* transformation, the process is accompanied by surface roughening, grain pull-outs and extensive micro-cracking, which may ultimately lead to strength degradation [2–4] unless a small amount of alumina (0.25%) is added to decelerate the LTD [5], notably by strengthening the grain boundaries [6].

The moisture-induced *t-m* phase transformation progresses from the surface to the interior [7] by a mechanism similar to the corrosion of metals, forming a degraded surface layer with a distinct boundary to the underlying, unaffected material [3,8].

The strong reduction of the elastic modulus and the hardness of the degraded surface were confirmed with a nano-indentation test [9,10]. At higher indentation loads extensive micro-cracking appears along the impression edges, leading to chipping of the damaged area around the indentation [6].

LTD is temperature-dependent and proceeds most rapidly at temperatures between 200 and 300 °C [7]. However, it has also been shown to occur at human body temperature at a much lower rate [8]. The *t-m* phase transformation is also sensitive to the chemical and phase composition, microstructure, surface topography and testing conditions [3,11].

Nowadays, most forms of dental zirconia are of the same chemical composition (containing about 3 mol% of yttria in the solid solution), but they differ in their sintering temperature, which in turn has a direct influence on the grain size and thereby on the related stress-and/or moisture-induced transformability, ageing resistance and mechanical properties. In general, fine-grained 3Y-TZPs sintered at lower temperatures exhibit a superior ageing resistance and a higher strength, but a lower fracture toughness and damage tolerance, as compared to their coarser-grained counterparts, achieved by sintering at higher temperatures [12–14]. The sintering conditions, in particular the firing temperature of dental 3Y-TZP ceramics, are starting-powder-specific and are generally recommended by the producer. Depending on the specific surface area, crystallite and aggregate sizes, the presence of dopants and the compaction ability of the starting powder used, the final sintering temperatures are typically between 1350 and 1550 °C [15]. In restorative dentistry, finer grains are advocated not only for their higher initial strength and an enhanced ageing resistance, but also for the increased light transmittance achieved with grain sizes well below the wavelength of visible light [16], resulting in higher translucency and improved aesthetics. Conversely, in the case that superior damage tolerance is targeted, a higher-opacity, coarser-grained ceramic might be chosen.

After sintering, grinding and polishing are commonly required to adjust the occlusion, whereas airborne-particle abrasion is used to clean the cementation surface and to increase the surface area and improve the bonding with luting cements [17,18]. Because 3Y-TZP ceramic exhibits a stress-induced *t-m* transformation (i.e., toughening), the ground or abraded surfaces will be constrained and also damaged. While rough ground surfaces are commonly fine ground and/or polished to remove the most heavily damaged upper layer, the airborne-particle-abraded surfaces are only cleaned in terms of dust and adhered debris. This process does not alter the characteristic topography of the abraded surfaces, showing extensive plastic deformation, sharp scars, (sub)surface cracks and welded airborne-particle debris [19–21]. Regardless of whether it introduces surface flaws, airborne-particle abrasion generally increases the strength of 3Y-TZP ceramics, because, it appears that, the surface strengthening prevails over the mechanical damage [19,22–26]. Furthermore, residual compressive stress and grain refinement were found to suppress LTD [27], which in turn should have relevance for extending the lifetime of zirconia-based dental restorations that are exposed to humidity and cyclic stressing under clinical conditions.

It has recently been demonstrated that the initial monotonic strength of mechanically damaged and/or hydrothermally treated dental 3Y-TZP ceramics provides a very good basis for identifying whether failure would occur during subsequent cyclic stressing [28]. Since the initial strength is likely to be the major factor determining the survival of dental restorations, our study was designed to investigate the influence of airborne-particle abrasion and accelerated ageing on the strength of two sets of specimens of 3Y-TZP ceramics with the same nominal chemical composition, but differing in their grain size and transformability achieved by sintering at 1400 °C and 1500 °C. These two sintering temperatures were selected on the basis of the reported difference in the grain size, and the properties of the materials produced from the same powder grade [14,22].

2. Materials and methods

Biomedical grade 3Y-TZP powder (TZ-3YB-|E, Tosoh, Tokyo, Japan) was used to fabricate the materials used in this study. This powder grade is a commonly used raw material for the preparation of commercially available, pre-sintered zirconia blanks, containing 3 mol% of yttria in the solid solution and 0.25 wt.% of alumina addition intended to decelerate ageing. The powder is granulated and contains about 3 wt.% of an organic binder.

Disc-shaped specimens of 20 mm in diameter and 2 mm thick were uniaxially dry pressed at 150 MPa in a floating die and randomly divided into two groups of 90. One group was sintered at 1400 °C for 2 h and the other group was sintered at 1500 °C for 2 h. After sintering, the specimens were 15.80 ± 0.02 mm in diameter and 1.48 ± 0.04 mm thick. The grain-size evaluations were made on FE-SEM (Carl Zeiss, Supra 35LV, Oberkochen, Germany) micrographs using the linear-intercept method, based on the ASTM E112-13 standard without introducing any correction factors [29].

The relative density of both materials was determined with Archimedes' method using distilled water as the immersion liquid. Even though the specimens were sintered in the two-phase ($t+c$) region, the relative densities were