

Addition of yttria partially stabilized Zirconia for reinforcement of dental porcelain

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ARTICLE INFO

Article history:

Received: 28 May 2013;

Received in revised form:

20 August 2014;

Accepted: 28 August 2014;

Keywords

Porcelain,
Zirconia,
Reinforcement.

ABSTRACT

The aim of this study was to investigate the effects of addition of Yttria partially stabilized Zirconia powder to dental porcelain being incorporated in 3,5 and 7-percent by weight on some mechanical properties. The measured properties were flexural strength, modulus of elasticity, fracture toughness and hardness. Samples were prepared according to the manufacturers instructions. Zirconia powder was thoroughly mixed with the porcelain powder in the correct weight percent before use. Flexural strength, modulus of elasticity, and fracture toughness were measured using three-point bending test. The hardness was measured using a conventional microhardness tester. All data were collected and analyzed with one-way analysis of variance (ANOVA) and LSD test ($p=0.05$). The results indicated that the addition of 3 % and 5 % of Yttria partially stabilized Zirconia to the conventional dental porcelain powder resulted in increased flexural strength and fracture toughness of the porcelain. On the contrary, 3 % and 5 % zirconia decreased both the modulus of elasticity and hardness. In addition, 7 % zirconia decreased all tested properties. X-ray examination showed that zirconia addition increased the radiopacity of the material. It was concluded that zirconia-modified dental porcelain was stronger and tougher than the conventional unmodified ceramic.

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Introduction

Dental ceramics are the preferred materials for oral restoration due to some characteristics, such as adequate esthetics, high fracture strength and chemical stability. Currently, dental professionals have a large amount of ceramic systems to choose from, all of them having small differences regarding their chemistry, processing temperatures, mechanical strength and clinical applications. These differences lead to classification systems that are difficult to understand by professionals outside the dental field.^[1]

In the last few decades, there have been tremendous advances in the mechanical properties and methods of fabrication of ceramic materials Leucite-reinforced feldspar glass ceramics Glass-based systems are made from materials that contain mainly silicon dioxide (also known as silica or quartz), which contains various amounts of alumina.^[2] Dental porcelains have a high glass content, which provides the translucency necessary for esthetic restorations.^[3]

The principal ingredients of dental porcelains used for porcelain-fused to metal are essential potassium-sodium aluminosilicate glasses with TiO_2 , SnO_2 , and ZrO_2 added as opacifiers. ZrO_2 represents approximately from 0.16 to 5.16% in the different types of porcelains.^[4] The brittle behavior of ceramics and their low tensile strengths compared with those predicted from bonds between atoms can be understood by considering the stress concentration around surface flaws. Ceramics tend to have no mechanism for yielding to stress without fracture, as do metals, and thus, cracks may propagate through a ceramic material at low-average stress levels. As a

result, ceramics and glasses have tensile strengths that are much lower than their compressive strengths.^[5]

Because of its natural appearance and durability, porcelain is widely used for the restoration of individual teeth and for fixed bridgework. Porcelain has been used for jacket crowns since the early 1900s, and covers the entire coronal part of the tooth. The various components of the porcelain blended together by the manufacturer result in two principal phases. One is the vitreous (glass) phase, and the other is the crystalline (or the mineral phase). The glass phase formed during the firing process has properties typical of glass, such as brittleness, unidirectional fracture pattern, and flow under stress. The crystalline phase includes Lucite and certain metallic oxides added as a coloring agents or opacifiers.^[4]

A great effort has been expanded to overcome their principal deficiencies- brittleness, low fracture toughness, and low tensile strength. Methods used to overcome the deficiencies of ceramics. Fall into two general categories (1) methods of strengthening brittle materials and (2) methods of designing components to minimize stress concentrations and tensile stresses. One method of strengthening glasses and ceramics is to reinforce them with dispersed phase of a different material that is capable of hindering a crack from propagation through the material. A newer technique for strengthening glasses involves the incorporation of a crystalline material that is capable of undergoing a change in crystal structure when placed under stress. The crystalline material usually used is termed partially stabilized zirconia (PSZ). The energy required for the transformation of PSZ is taken from the energy that allows the crack to propagate.^[5,6] There are several fundamental

strengthening mechanisms of dental ceramics, such as crystalline reinforcement, chemical strengthening, and thermal tempering.^[6]

Because of the relatively low tensile strength and brittleness of the porcelain, it has been generally fused to a metal substrate to increase resistance to fracture. More recently, newer types of all-ceramic restorations have been developed that may prove to have a lower incidence of clinical fracture. These materials need veneering with conventional feldspathic porcelain to reproduce the contour and shade of a natural tooth.^[7]

The lower the fracture toughness the lower is the clinical reliability of the ceramic restoration because the K_{IC} value defines the critical stress intensity level at which catastrophic failure occurs due to a critical micro defect. Therefore every new dental ceramic material should be tested not only with respects to its flexural strength but also with respect to its fracture toughness before introducing into the market. The fracture toughness can be measured either by bending or indentation method. The indentation method is not an adequate tool to exactly determine the fracture toughness of unknown ceramic material. This method can be only used for a first rough K_{IC} estimation.^[8]

The load bearing capacity of the ceramic itself may be enhanced using a second, reinforcing phase embedded in the glass matrix. However, in common with metal substructures, reinforced ceramic cores may have less than ideal esthetic properties so require to be overlaid with conventional porcelains.^[9] The aim of this work was to test the hypothesis that the addition of Yttria partially stabilized zirconia to the conventional dental porcelain will significantly increase its mechanical properties. The properties will be studied are flexural strength, modulus of elasticity, fracture toughness, and hardness. In addition to the radiographic examination.

Materials and methods

The materials used in this study were Yttria partially stabilized Zirconia powder [Promochem GmbH, Germany) and Vita VMK 95 dental porcelain (Vita Zahnfabrik H. Rauter GmbH & Co. Säckingen, Germany).

Flexural properties

A total of 40 porcelain samples were prepared, 10 samples for each group. Yttria partially stabilized Zirconium powder was thoroughly mixed with the porcelain powder in a 3, 5, and 7 % by weight. The normal dimensions of the porcelain samples were chosen as 35 mm length, 3 mm width and 4 mm height.^[10] The control group was made without any additions. 10 samples were made for each concentration of stabilized ZrO_2 added. Samples were manufactured according to company's recommendations. Dough consisting of the porcelain powder and solvent was prepared and pressed into a split stainless steel mould fixed on a fiber fleece to prevent sticking of the samples to the firing tray. The mould was overfilled with porcelain and the excess liquid brought to the surface and removed using a tissue. The surface of the porcelain was leveled using a straight blade to produce samples of uniform thickness, the metal mould was opened and the sample placed on the fiber fleece on the firing tray.

Porcelain bars were dried in front of an opened furnace door (Vita Vacumat 40, Vita Zahnfabrik . Bad Säckingen, Germany) at 600°C for 10 minutes, then placed inside the furnace and gradually vacuum fired up to 930°C. On reaching this temperature, the furnace was switched off and the samples were allowed to cool to room temperature. Any samples with visible surface imperfections were discarded. Minor adjustment can be

done using smooth silicone carbide paper. The porcelain bars were ground with carbide disks of grit size 400 and 600. The glaze was applied to the entire sample surface; excess glaze was removed with a dry brush until only a film of glaze remains. All samples were glaze fired without vacuum on plane honeycomb firing trays. The samples were placed at the entrance of the furnace at a temperature of 600°C for 10 minutes and then elevated into the furnace. The temperature was raised to 900°C gradually without vacuum. On reaching this temperature, the samples were cooled slowly in front of the furnace. This glazing temperature was well above the glass transition temperature of the porcelain ($T_g = 581.7^\circ\text{C}$) so that healing of most surface micro cracks in the samples would be expected. The samples were stored at room temperature in a desiccator until required for testing since under ambient conditions dental porcelains undergo static fatigue failure due to hydrolysis of silicate bonds.^[11-13]

The flexural strength was determined in a three-point bending testing apparatus (Lloyd instruments LS 500 LTD; England) at a crosshead speed of 0.5mm/minute. Before testing, each sample was thoroughly measured with a digital micrometer. The flexural strength value finally determined from the following equation:^[10]

$$\sigma = 3FL / 2bh^2$$

Where σ is the flexural strength in Mpa.

F is the failure load in Newton.

L is the distance between the supports in mm.

b is the width of the samples in mm.

h is the height of the sample in mm.

The modulus of elasticity was determined from the following equation:^[10]

$$E = P_1 I^3 / 4bd^3\delta$$

Where: E is the modulus of elasticity in Gpa.

P_1 is the load at a selected point of the elastic region of the stress-strain plot.

I is the distance between the supports.

b is the width of the sample.

d is the thickness of the sample.

δ is the deformation of the sample at P_1 .

Fracture toughness testing

A total of 40 samples were made according to the manufacturer's instructions. The bar-shaped samples were made of 30mm length, 6mm width and 3mm thickness using split stainless steel moulds as mentioned before. The surface of the samples was ground on a rotation ground machine using silicone nitride ground papers grit 800 and 2400. The ground bar samples were notched using single-edge notched-bend samples (American Society for Testing and Materials - ASTM C1421, "Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics, it is a high-quality, technically rigorous, full-consensus standard that may have finally gives real fracture toughness of ceramics). The ratio between notch depth and sample thickness was 0.5. The notches were sharpened by the razor blade method so that the tip of the notch had a width of less than 30 μm . The samples were loaded in a four-point-bending test arrangement. The crosshead speed of the testing machine was 1mm/minute. The fracture toughness was calculated from the following equation.^[8]

$$K_{IC} = \frac{L_{max}}{t\sqrt{h}} \times \frac{l_o - l_i}{h} \times \frac{3\Gamma M\sqrt{d/h}}{2(1-d/h)^{3/2}}$$

Where L_{max} = is the maximum load.

l_o and l_i = are the outer and inner roll spans, respectively.

t and h = are thickness and height of the samples.

d = is the depth of the notch.

$$\Gamma M = \frac{1.9887 - 1.326d/h - [3.49 - 0.68 d/h + 1.35(d/h)^2] d/h(1-d/h)}{(1 + d/h)^2}$$

Hardness testing

A total of 20 porcelain discs 8mm in diameter and 0.6mm in thickness were prepared, 5 samples for each group. To fabricate the samples, molds were fabricated by embedding wax discs into refractory investment material. Wax discs were removed when the investment was dry. Dough consisting of the porcelain powder and solvent was placed into the mold, vibrated, and the excess moisture was blotted dry. Porcelain discs in the investment molds were processed as mentioned before. Investment material was removed by sandblasting. The porcelain discs were ground with carbide disks of grit size 400 and 600. The glaze was applied to the entire sample surface; excess glaze was removed with a dry brush until only a film of glaze remains. The samples were fired as mentioned before. The hardness of the samples was determined using a micro hardness tester (FM, Future tech.Corp. Tokyo, Japan). The Vickers hardness test was performed using a diamond indenter with 100 g load and a dwell time of 15 seconds. The mean of 5 measurements was used for each sample. The VHN was calculated for each group.

X-ray examination

Broken samples of the conventional and modified porcelain were examined using periapical X-ray films to test the radiopacity of the samples.

Statistical analysis

The data obtained were analyzed using one-way analysis of variance (ANOVA). LSD test was conducted when there was a significant difference was found.

Results

The mean flexural strength (Table 1), modulus of elasticity (Table 2), fracture toughness (Table 3) and hardness (Table 4) were obtained. The statistical analysis (ANOVA) of the data showed that there were highly significant differences between all groups ($p \leq 0.001$ & $p \leq 0.01$). The results of this study indicated that the addition of 3 % and 5 % of Yatria partially stabilized Zirconia resulted in increased flexural strength and fracture toughness of the porcelain. On the contrary, 3 % and 5 % zirconia decreased both the modulus of elasticity and hardness. In addition, 7 % Zirconia decreased all tested properties.

Data related to flexural strength showed that there were significant differences between the control group and 3-percent and 7-percent zirconia-reinforced groups. There was no significant difference between the control group and 5-percent reinforced group (Table 1).

Data related to modulus of elasticity showed that there were significant differences between the control group and the other reinforced groups. There were no significant difference between 3-percent and 5-percent reinforced groups or between 5-percent and 7-percent zirconia reinforced groups (Table 2).

Data related to the fracture toughness showed that there were significant differences between the control group and 3-percent and 5-percent zirconia-reinforced groups. There was no significant difference between the control group and 7-percent reinforced group (Table 3).

Data related to the hardness showed that there were significant differences between the control group and the other reinforced groups. There was no significant difference between 3-percent and 5-percent reinforced groups (Table 4).

X-ray examination

The radiopacity of the reinforced samples were increased

Discussion

Dental porcelain is universally acknowledged for superior esthetics. Unfortunately, its brittleness and low tensile and shear strengths render the porcelain restoration liable to fracture during mastication. A well-established explanation for porcelain failure is crack propagation. A fracture commonly originates at a surface flaw and propagates through others in the material.^[7] All dental ceramics display low fracture toughness when compared with other dental materials, such as metals.^[14,15]

Zirconium oxide is present in the components of the porcelain powder about 5.16 wt %.^[16] Yatria partially stabilized added in three mole percent for stabilization of zirconium oxide crystals. Zirconia tetragonal polycrystals are less than 0.6 μm in size. Polycrystalline solid zirconia exhibits stress induced phase transformations in the vicinity of a crack tip to reduce the local stress.^[17]

Because glasses are brittle, they fail under tension or bending by the propagation of preexisting flaws (e.g., scratches, porosities). Several approaches that are based on impeding the propagation of flaws have been used to strengthen dental porcelains, including bonding to metals, adding microcrystalline phases, and surface treatments (i.e., polishing, ion exchange, and hydration). Through these methods, porcelain systems are used routinely for all-ceramic anterior restorations; however, porcelain-fused-to-metal restorations remain the most reliable for posterior applications.^[3]

Previous studies on ion exchange of dental ceramics have shown that the biaxial flexural strength can be improved by exchanging potassium for sodium ions at temperatures below the strain point. The effect of surface ion exchange is better than that of auto-glazing or polishing. The friction and wear behavior of dental ceramics depend on the material itself especially after the surface-strengthening layer disappeared. Abrasive wear, adhesion and micro cracking characterize the wear mechanism of dental feldspathic ceramics after the surface-strengthening treatments.^[18] The strengthening mechanism whilst dependent on surface texture was independent of defect severity.^[19]

Zirconia (ZrO_2) is a white crystalline oxide of zirconium that is used as a refractory, in insulation, abrasives, enamels, and glazes. Pure zirconia has a monoclinic crystal structure at room temperature and transitions to tetragonal and cubic at increasing temperature. The volume expansion caused by the cubic to tetragonal to monoclinic transformation induces very large stresses, and will cause pure ZrO_2 to crack upon cooling from high temperatures. These stresses together with the high tensile stresses about the crack tip can nucleate micro cracks are believed to increase the fracture toughness of the material. Several oxides are added to zirconia to stabilize the tetragonal and/ or cubic phases. Magnesia (MgO), Yatria (Y_2O_3), Calcia (CaO), and Ceria (CeO), amongst others.^[20]

Zirconium dioxide dispersion-strengthened cores and crowns and zirconia-based enamels for metal substrates may be developed to take advantages of the high fracture strength of zirconia. Transformation toughening is possible with zirconia. This involves phase of transformations that create compressive stresses across the plane of propagating cracks (crack shielding), thereby reducing the tensile stresses acting at the crack tip.^[11]

The microstructural differences between the ceramic materials resulted in different behaviors in terms of fracture strength, structural reliability and slow crack growth.

Table 1. Mean flexural strength (Mpa) and SD of control and modified dental porcelain.

ZrO ₂ concentration	Mean \pm SD	F-value	P-value
Control	123.95 ^(B) \pm 7.86	68.262	0.0000*
3 % modified Porcelain	142.55 ^(A) \pm 6.57		
5 % modified porcelain	131.06 ^(B) \pm 7.19		
7 % modified porcelain	81.06 ^(C) \pm 6.49		

LSD value = 9.741 at alpha = 0.050

Means with different superscripts are significantly different.

* P \leq 0.001 very highly significant.**Table 2. Mean modulus of elasticity (Gpa) and SD of control and modified dental porcelain**

ZrO ₂ concentration	Mean \pm SD	F-value	P-value
Control	7.95 ^(A) \pm 0.34	7.318	0.0026*
3 % modified Porcelain	6.091 ^(C) \pm 0.186		
5 % modified porcelain	6.783 ^(BC) \pm 1.81		
7 % modified porcelain	6.96 ^(B) \pm 0.235		

LSD value = 0.8501 at alpha = 0.050

Means with different superscripts are significantly different.

* P \leq 0.001 very highly significant.**Table 3. Mean fracture toughness (K_{IC}, Mpa.m^{1/2}) and SD of control and modified dental porcelain**

ZrO ₂ concentration	Mean \pm SD	F-value	P-value
Control	0.538 ^(C) \pm 0.049	164.65	0.0000*
3 % modified Porcelain	1.424 ^(A) \pm 0.064		
5 % modified porcelain	0.782 ^(B) \pm 0.106		
7 % modified porcelain	0.489 ^(C) \pm 0.069		

LSD value = 0.1039 at alpha = 0.050

Means with different superscripts are significantly different.

* P \leq 0.001 very highly significant.**Table 4. Mean hardness (VHN) and SD of control and modified dental porcelain.**

ZrO ₂ concentration	Mean \pm SD	F-value	P-value
Control	470.9 ^(A) \pm 14.93	176.044	0.0000*
3 % modified Porcelain	440.4 ^(B) \pm 4.26		
5 % modified porcelain	430.48 ^(B) \pm 4.06		
7 % modified porcelain	355.88 ^(C) \pm 3.63		

LSD value = 11.03 at alpha = 0.050

Means with different superscripts are significantly different.

* P \leq 0.001 very highly significant.

The lithium disilicate glass-ceramic was more susceptible to slow crack propagation than the other materials tested, whereas the leucite-based glass-ceramic and the alumina-glass composite obtained the highest values, and thus have the least susceptibility to slow crack growth. The clinical longevity of ceramic restorations is often limited by lifetimes that are controlled by a slow crack growth process. Moreover, knowing these parameters can assist in understanding how the microstructure of ceramic materials can be modified to increase their lifetime in service. [21]

It has been demonstrated that ceramic materials show considerable variation in strength, primarily due to their extreme sensitivity to the presence of cracks of different sizes. The unstable fracture of ceramics starts from critical flaws, and this phenomenon may be explained by the "weakest link" theory, which determines that fracture always propagates from the largest flaw favorably oriented to the tensile stress. For a given ceramic material, the distribution of crack size, shape, and orientation differs from sample to sample and its strength is statistically distributed according to the flaw size distribution. It

is recognized that the strength of ceramics needs to be analyzed using different statistical approaches, especially because the probability of failure depends on the presence of a potentially dangerous crack of size greater than a characteristic critical crack size. Also, fracture depends on the stressed area or the volume of the material, since larger areas or volumes increase the probability of critical flaw content. [22]

Fracture toughness (K_{IC}) is an important material characterization value. Its value characterizes the resistance of a material against a propagating crack. The higher the K_{IC} value is the better is the mechanical behavior of the material. The bending method was used in this study to evaluate the fracture toughness because it is the most accurate and reliable method, while the indentation method is not an adequate tool to exactly determine the fracture toughness of unknown ceramic material. The fracture of ceramics in service occurs with little or no plastic deformation when cracks propagate in an unstable manner under applied tensile stresses. Fracture occurs when the stress intensity factor at the crack tip (KI) reaches a critical level (K_{IC}). [8]

There are three modes of crack surface displacement (a) mode I, tensile mode; (b) mode II, sliding mode, and (c) mode III, tearing mode. The I subscript for K_{IC} stands for mode I, or tensile mode, crack displacement. In general, K_{IC} is low for brittle materials and high for ductile materials. Additionally, K_{IC} typically increases with decreasing grain size as composition and other micro structural variables are held constant.^[23]

The results of this study were within the range reported by Craig.^[4] The authors reported that the transverse strength of porcelain is between 62 and 90 Mpa, the elastic modulus is 69 Gpa and knoop hardness is 460 Kg/mm². One important aspect of the critical cracks in ceramic materials is the fact that they may exhibit a slow and stable crack growth when subjected to stresses below the critical value, especially in the presence of water or water vapor, as observed in the oral environment. The oral environment has many elements that favor stable crack growth in ceramic restorations, such as water from saliva and from the dentin tubules, masticatory stresses, temperature and pH variations.^[24]

Yttria partially stabilized zirconia (ZrO_2) caused increased flexural strength and toughness of ceramics. This improvement in the mechanical properties was due to zirconia is affected by the presence of other phases and by the metastability of the tetragonal to monoclinic transformation. Microscopic investigation and X-ray diffraction revealed the important role played by the tetragonal to monoclinic phase transformation and by the relationship between the glassy matrix and the crystalline phase in the strengthening and toughening mechanisms of these ceramics. So, zirconia-based dental ceramics are stronger and tougher materials than the conventional glass-ceramics.^[25]

The results of this study indicated that the hardness and flexural modulus were not improved by addition of partially stabilized zirconia to dental porcelain. This was in a consistent with a previous study concluded that addition of Yttria stabilized zirconia particles to glass ionomer not improve the hardness and compressive strength.^[26] The reduction in the hardness noticed in this test may be advantageous to decrease wearing of the natural teeth by the opposing porcelain restorations. The friction and wear behavior of dental ceramics depend on the material itself especially after the surface-strengthening layer disappeared. Abrasive wear, adhesion and micro cracking characterize the wear mechanism of dental feldspathic ceramics after the surface-strengthening treatments.^[27]

Dispersion strengthening is a process by which the dispersed phase of a different material (such as alumina, leucite, zirconia, etc.) is used to stop crack propagation as these crystalline phases are more difficult to penetrate by cracks.^[28] Other researchers added wollastonite into porcelain powder. They concluded that incorporation of wollastonite increased the flexural strength of dental aluminous core porcelain. The highest increase (30%) in the flexural strength was achieved by the addition of 2% wollastonite.^[29] The reinforcement of a porcelain with continuous $GdAlO_3/Al_2O_3$ fibers resulted in increased flexural strength and elastic modulus. Such improvement on the mechanical properties is related to several toughening mechanisms associated to the use of long fibers, such as crack bridging at the crack wake, microcrack toughening, and the absorption of energy by fracture of the fibers.^[30]

The improvement in both strength and toughness has made it possible to expand the range of indications to long-span fixed partial prostheses, implant abutments and implants. Due to the unsurpassed mechanical properties of partially stabilized

zirconia, its introduction to the dental market, almost a decade ago, considerably expanded the range of applications of ceramics in dentistry, a field where they are classically in demand due to their chemical inertness and a wide combination of optical properties, allowing excellent esthetics.^[31]

Conclusions

Within the limitations of this study the following conclusions were drawn:

1. Addition of Yttria partially stabilized zirconia in 3 and 5 percent by weight resulted in increased flexural strength and fracture toughness of the conventional dental porcelain, while the modulus of elasticity and hardness were decreased. On the contrary, 7 wt % decreased all studied properties.
2. Radiographic examination revealed that Zirconia render the porcelain more radiopaque and can be detect easily by X-ray.

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