

***Effect of Sintering Time and Temperature on
Various Properties of Monolithic Translucent
Zirconium Oxide Ceramic***

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Dedication

This work is dedicated to

My dear parents,

Precious sister,

Beloved wife,

and lovely son

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Introduction

The increased popularity of all-ceramic materials as an alternative to metal-ceramic restorations is attributable to their excellent aesthetics, chemical stability and biocompatibility. However, the brittleness and low tensile strength of conventional glass-ceramics limits their long-term clinical application in restorations.^[1]

Several glass-ceramics have been introduced such as high alumina-content glass-infiltrated ceramic core material (In-Ceram Alumina) and disilicate glass-ceramic (Empress 2) which has been successfully used for crowns ^[2], anterior fixed partial dentures (FPDs) and three-unit FPDs replacing the first premolar. However, these materials do not have sufficient strength to allow reliable use for FPDs, especially in the molar region.^[3]

Recently, the development of advanced dental ceramics has led to the application of partially stabilized zirconia in restorative dentistry which can be produced from a computer assisted design/computer-aided manufacture (CAD/CAM) system. The use of zirconia-based ceramics for dental restorations has risen in popularity due to their superior fracture strength and toughness compared with other dental ceramic systems.^[4-6]

Zirconia-based restorations have become very popular due to their superior mechanical properties and excellent biocompatibility. Due to their high opacity zirconia core is usually veneered with veneering porcelain however, in clinical service, the most frequent failure is the chipping of the veneer, while the high-strength zirconia substructure is mostly not affected.^[7, 8] In specific clinical situations, such as when the occlusal or palatal space is limited or in cases where a patient's parafunctional activity (e.g., bruxism) may contraindicate this veneering application the use of unveneered zirconia ceramic seems to be an option for all-ceramic restorations.^[9]

Recently nano-zirconia was introduced which allows the use of full contour zirconia restorations without the need for any veneering ceramic. These restorations were able to attract increasing attention because of their unique combination of optical and mechanical properties.^[10]

Zirconia restorations are usually milled in a partially sintered state then this is followed by a sintering process to allow the restoration to reach its maximum density.^[11,12] Several manufacturers replaced their long sintering cycles with shorter ones by changing the heating and cooling rates and by modifying the sintering holding time. The effect of these changes on the final properties of the zirconia restoration remains in question.

Review of literature

Zirconia Restorations:

Zirconium dioxide ZO_2 , known as Zirconia is a white crystalline oxide of zirconium. Although pure zirconium oxide does not exist in nature, it is found in the mineral baddeleyite or zircon.^[7]

The research and development on zirconia as a biomaterial started in the late sixties. The first paper concerning biomedical application of zirconia was published in 1969 by Helmer and Driskell^[13], while the first paper concerning the use of zirconia to manufacture ball heads for Total Hip Replacements, which is the current main application of this ceramic biomaterial, was introduced by Christel et al. in 1988.^[14]

Zirconia is a polycrystalline ceramic without any glass component. Being polymorphic, three forms of zirconia exist: monoclinic, cubic and tetragonal. Pure zirconia assumes the monoclinic form at room temperature which is stable up to 1170°C. Beyond this temperature, a transformation to the tetragonal phase occurs, which is stable up to 2,370°C, after which the cubic phase transformation is seen. A transformation of tetragonal to monoclinic occurs while cooling to the temperature of 1170°C. This is associated with a volume expansion of 3% to 5%.^[15]

Addition of stabilizing oxides such as calcium oxide, magnesium oxide, cerium oxide and yttrium oxide stabilizes zirconia in its tetragonal phase at room temperature. Tensile stresses at a crack tip will cause the tetragonal phase to transform into the monoclinic phase with an associated 3-5% localized expansion. This volume increase creates compressive stresses at the crack tip that counteract the external tensile stresses and retards crack propagation. This phenomenon is known as transformation toughening.^[16]

Zirconia core materials are usually fabricated by milling technology.^[17] The restorations are processed either by soft machining of partially sintered blocks, followed by final sintering at high temperature or by hard machining of fully sintered blocks with a density of approximately 99.5% of the theoretical density.^[11,12] Partially sintered frameworks are

milled from porous blocks with incomplete sintered grains and open boundaries to larger dimensions and require further sintering for the ceramic to gain its full density.^[17, 18] This sintering procedure is accompanied by a sintering shrinkage of about 20% to 30%.^[19]

Soft machining is much easier and prevents the stress induced transformation from tetragonal to monoclinic phase; however, the shrinkage during the additional sintering results in frameworks with less accurate marginal fit.^[20] Hard machining is technically more difficult, wears the machining hardware at a much higher rate, induces a significant amount of monoclinic zirconia, and may introduce micro cracks in the material that result in higher susceptibility to lower temperature degradation and lower reliability.^[12,21] However, it offers higher precision since only 1-step sintering is sufficient.^[20]

Sailer et al in 2007 ^[22], published the results of a prospective clinical cohort study to determine the success rate of three to five units zirconia frameworks for posterior FPDs after 5 years of clinical observation. They evaluated forty five patients with fifty seven FPDs made up of zirconia frameworks. They found that the success rate of the zirconia frameworks was 97.8% and concluded that zirconia offers sufficient stability as a framework material for three to five units posterior FPDs.

Molin and Karlsson in 2008 ^[23], evaluated the clinical performance of fully sintered hot isostatic pressed yttria-partially-stabilized zirconia (Denzir) 3-unit fixed partial dentures. After five year examination all FPDs were intact and they concluded that yttria-partially-stabilized zirconia 3-unit FPDs with anatomically designed frameworks are promising prosthetic alternatives, even in the premolar and molar regions.

Roediger et al in 2010 ^[24], published the results of a four year prospective study where they evaluated seventy five three to four units FPDs. Among all of the patients thirteen exhibited ceramic veneer fracture or chipping. They concluded that within a mean observation period of 4 years, sufficient survival rates for zirconia-based posterior FPDs could be verified. The main complication was fracture of the ceramic veneering material.

Rinke et al in 2013 ^[25], in a seven years clinical evaluation of seventy five patients treated with ninety nine posterior FPDs concluded that the main cause of failure was fracture of the veneering ceramic which was shown in thirty two events.

Monolithic Zirconia:

A new innovative possibility for dental restorations is the construction of monolithic restorations without veneering. These restorative solutions have no porcelain overlay material to risk shear or fracture, nor do they require specialized pressing techniques and equipment.^[26] Fabricating mono-block restorations from pure zirconia could increase the mechanical stability and expand the range of indications however, zirconia is known as a whitish, opaque core material. Optical appearance of opaque zirconia might be improved by modifications in the fabrication and sintering process, which were shown to increase translucency.^[27] The clinical advantage of these restorations is defined by significantly reduced material thickness in comparison to veneered zirconia restoration or other monolithic restorations. These restorations can be preshaded or colored prior to sintering, followed by characterization by staining thus good esthetic results in the posterior region can be achieved, even in cases with substantially reduced space.^[28, 29] **Beuer et al in 2012** ^[30], reported that glazed full-contour zirconia crowns showed similar translucency, contact wear of the restoration and contact wear at antagonist as veneered zirconia crowns. However, glazed full-contour zirconia crowns showed higher fracture loads than veneered zirconia crowns.

The research team of Glidewell Laboratories' led by **Ken Knapp in 2011**, developed a method for producing non agglomerated 3nm nanocrystalline zirconia powder using revolutionary bottom-up nanotechnology technique known as "Gas-phase condensation". This method consists of colliding high-energy yttrium, zirconium and oxygen ions together in an energetic gaseous phase and condensing yttria zirconia nanocrystal particles resulting from atomic collisions during flight in the gas phase. The condensed yttria zirconia nanocrystal particles are separated from the gas phase and collected in the form of nanocrystalline powder. According to Knapp, "The key to making transparent polycrystalline zirconia material is

starting with non-agglomerated yttria zirconia primary crystal size less than 5nm”.^[31]

Effect of sintering times and temperatures:

Partially sintered zirconia requires a sintering stage to reach its maximum density. In this stage heat is transmitted to the surface of the material and reaches its core by thermal conduction. The sintering cycle is divided into a heating stage, a holding stage at the final sintering temperature and a cooling stage.^[32]

Hjerppe et al in 2009 ^[33], conducted a study to evaluate the effect of sintering time on flexural strength of zirconia restorations. Fifty six zirconia discs (Zircon Zahn) divided into two groups were used in his study; the first group was sintered with a rising time of 3 hours and a holding time of 2 hours, while the second group was sintered with a rising time of 1 hour 40 minutes and a holding time of 1 hour. Both groups were sintered at a temperature of 1500°C. Half of the samples in each group were thermocycled. They concluded that there was no statistically significant difference in the biaxial flexural strength between the two groups.

Jiang et al in 2011 ^[27], evaluated the effect of sintering temperatures on the translucency of zirconia discs. They used the sintering temperatures 1350°C, 1400°C, 1450°C, and 1500°C with a heating rate of 200°C/h and a holding time of 2 hours. Their results concluded that there was an increase in the relative amount of light transmitted thus translucency through the specimens as the sintering temperature increased.

Stawarczyk et al in 2013 ^[34], studied the effect of sintering temperature on the biaxial flexural strength and contrast ratio of zirconia discs. Zirconia specimens (Ceramill ZI, Amann Girbach) were divided into nine groups according to the following sintering temperatures: 1300°C, 1350°C, 1400°C, 1450°C, 1500°C, 1550°C, 1600°C, 1650°C, and 1700°C. They were sintered at a heating rate of 8°C/min and a holding time of 2 hours. Results showed that the highest flexural strength was between temperatures 1400°C and 1550°C. They also concluded that as the sintering temperature increases the contrast ratio of the zirconia specimens decreases.

Kim et al in 2013^[35], evaluated the effect of sintering time on the translucency of two commercial brands of zirconia specimens (Kavo and Lava). They sintered the specimens at the following sintering times: 20 minutes, 2 hours, 10 hours, and 40 hours. Both types of zirconia showed the highest translucency when sintered for 2 hours and the lowest translucency when sintered for 40 hours. Also the Kavo specimens showed more translucency than the Lava specimens at all sintering times.

Color and Translucency:

An objective method of evaluating color differences requires an ordered system for the classification of color, as well as equipment capable of quantifying color differences. The use of colorimetric measurements provides interpretation of subjective evaluations related to the perception of color as physical values.^[36]

The widely recognized CIE L*a*b* color order system, developed in 1978 by the Commission Internationale de l'Eclairage (International Commission on Illumination), is commonly used in dental research.^[37,38] This system defines color in terms of 3 coordinate values (L*, a*, and b*), which locate the color of an object within a 3-dimensional color space. The L* coordinate represents the brightness of an object represented on the y-axis, the a* value represents the red (positive x-axis) or green (negative x-axis) chroma, and the b* value represents the yellow (positive z-axis) or blue (negative z-axis) chroma. The color difference (ΔE) between 2 objects, or in the same object before and after it is subjected to particular conditions, can be determined by comparing the differences between the respective coordinate values for each object or situation.^[38]

Translucency is defined as the relative amount of light transmission or diffuse reflectance from a substrate surface through a turbid medium. The translucency of dental ceramic has a close relationship with its chemical composite and microstructure.^[39] The chemical nature, the amount of crystals, the size of particles, the pores and the sintered density determine the amount of light that is absorbed, reflected, and transmitted. All of the above influence the optical property of core ceramics.^[40]