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TEMPERATURE EFFECT ON THERMAL CHANGES AND PHASE ANALYSIS OF 3Y-TZP ZIRCONIA RESTORATIONS

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Abstract

This study aimed to investigate the effect of final sintering on the thermal properties and phase composition of yttrium-stabilized zirconium dioxide.

Totally 15 yttrium-stabilized zirconium oxide standard ovoid samples were studied (3Y-TZP, IPS e.max ZirCAD LT, Ivoclar Vivadent, Schaan, Liechtenstein). This material contains the following components: ZrO2 - 87.0 - 95.0%, Y2O3 - 4.0 - 6.0%, HfO2 - 1.0 - 5.0%, Al2O3 - 0.0 - 1, 0%. Initially, X-ray diffraction analysis was performed. After that, differential scanning calorimetry was carried out up to a temperature of 900°C. X-ray diffraction analysis was performed to assess the effect of the firing process on the phase analysis of zirconia samples (Selected samples were tested on an X-ray diffraction instrument (EMPYREAN, PANalytical, Lelyweg, The Netherlands) The thermal changes study in zirconia samples resulting from repetitive firing processes was performed then. Samples were placed in a differential scanning calorimeter (NETZSCH STA 409 PC Luxx, Bavaria, Germany) and heated at a rate of 10°C/min from 50°C to 900°C.) Then, differential scanning calorimetry data (the mass and structural changes of zirconia samples depending on temperature) was recorded.

The percentage of the monoclinic phase increases on average from 3.6% to 7.5%, but this difference is not statistically significant (p=0.1). Two exothermic peaks were observed when the samples were heated from 50°C to 900°C.

The final firing of 3Y-TZP zirconium crown up to 900°C slightly increases the content of the monoclinic phase and causes exothermal changes.

Keywords: *zirconium oxide, X-ray diffraction, calorimetry, dental crowns.*

Introduction

The excellent mechanical properties and biocompatibility of zirconia, as well as advances in computer-aided design & computer-aided manufacturing technology (CAD/CAM), have led to the growing popularity of this material in prosthetic dentistry [*Piconi C, Maccauro G, 1999; Denry I, Kelly J, 2008*]. Zirconium dioxide was introduced into dental practice in the 1990s and is now widely used in dentistry as a framework for ceramic restorations [*Ban S et al., 2007*], as a full anatomy restoration [*Carrabba M et al., 2017*] and as a material for abutments in implant dentistry [*Nakamura K et al., 2010; Ekfeldt A et al., 2011; Volberg R et al., 2019*].

Pure zirconium has three different crystallographic phases: monoclinic at room temperature

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up to 1170 C, tetragonal at temperatures from 1170 to 2370°C and cubic at temperatures above 2370°C [*Chevalier J et al., 2006; Baldissara P et al., 2018; Camposilvan E et al., 2018; Edelhoff D et al., 2019*]. The tetragonal structure of zirconium dioxide can be retained in the metastable phase at room temperature by adding oxides such as CaO, MgO, Y2O3, or CeO2 [*Denry I, Kelly J*, 2008]. Yttrium oxide turned out to be an excellent option in this context [*Sailer I et al., 2018*], however, the phase transformation of tetragonal to monoclinic can be caused by various stimuli, such as stress or aging at low temperatures in the presence of moisture, which leads to deterioration of the properties of zirconium [*Souza R et al., 2013*].

Despite advances in manufacturing CAD/CAM systems and pre-sintered ceramic blocks, dental technicians or clinicians still manipulate already sintered zirconia surfaces [*Aboushelib M et al., 2009*], in some cases to better fit the restoration. These procedures can initiate a t → m phase transformation, creating a layer of compressive stress, due to an increase in the volume of the material (zirconia) by about 3%, in addition to some surface defects in the material. When the depth of these defects exceeds the thickness of the compressive layer, the defects can act as zones of stress concentration, which can also deteriorate the mechanical properties [*Kosmač T et al., 1999; Guazzato M et al., 2005; Quinn G et al., 2005; Orekhova L et al., 2010*].

Various heat treatment protocols have been proposed to induce surface recovery by promoting reverse conversion of the monoclinic phase to the tetragonal phase. The protocols relieve the stress present in the formed compression layer, as well as the residual stresses arising on the surface of zirconium dioxide [*Deville S et al., 2006*]. It is also known that the final stage of sintering in zirconium restorations with ceramic application is glazing, which takes place at an average temperature of 900°C [*Sailer I et al., 2013*]. Ideally, restorations should maintain an intact glaze surface and the final glaze coat has been shown to be the most acceptable surface [*Zarone F et al. 2019*]. However, there may be cases where ceramic restorations require corrections, after which a re-glazing is required [*Matzinger M et al., 2019*].

The aim of this study was to study the effect of the final firing on the thermal properties and phase composition of 3Y-TZP zirconium dioxide. The null hypothesis is that the final firing up to 900°C does not change the phases of the zirconium dioxide [*Tholey MJ et al.,2011*].

Materials and methods

In this study, 15 yttrium-stabilized zirconium oxide standard ovoid samples were studied (3Y-TZP, IPS e.max ZirCAD LT, Ivoclar Vivadent, Schaan, Liechtenstein). This material contains the following components: ZrO2 - 87.0 - 95.0%, Y2O3 - 4.0 - 6.0%, HfO2 - 1.0 - 5.0%, Al2O3 - 0.0 - 1, 0%. Initially, X-ray diffraction analysis was performed. After that, differential scanning calorimetry (DSC) was carried out up to a temperature of 900°C. Then X-ray diffraction analysis was carried out again.

X-ray diffraction analysis was performed to assess the effect of the firing process on the phase analysis of zirconia samples (identification of the phase content of cubic-ZrO2, tetrogonal-ZrO2 and monoclinic-ZrO2). Selected samples were tested on an X-ray diffraction instrument (EMPYREAN, PANalytical, Lelyweg, The Netherlands) using a wavelength of 1.5419 (Kα), a scan range of 20° to 100°, a step size of 0.005° and a scan rate of 0.067°/s.

The thermal changes study in zirconia samples resulting from repetitive firing processes was performed then. Samples were placed in a differential scanning calorimeter (NETZSCH STA 409 PC Luxx, Bavaria, Germany) and heated at a rate of 10°C/min from 50°C to 900°C.) Then, DSC data (the mass and structural changes of zirconia samples depending on temperature) was recorded.

To overcome it is possible, due to the uniting the knowledge and will of all doctors in the world

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One-way ANOVA test was used with a significance level of $p<0.05$ in StatPlus software (AnalystSoft Inc. WALNUT, CA, USA) to assess the results obtained and carry out comparative characteristics.

RESULTS

X-ray diffraction analysis showed typical pat-

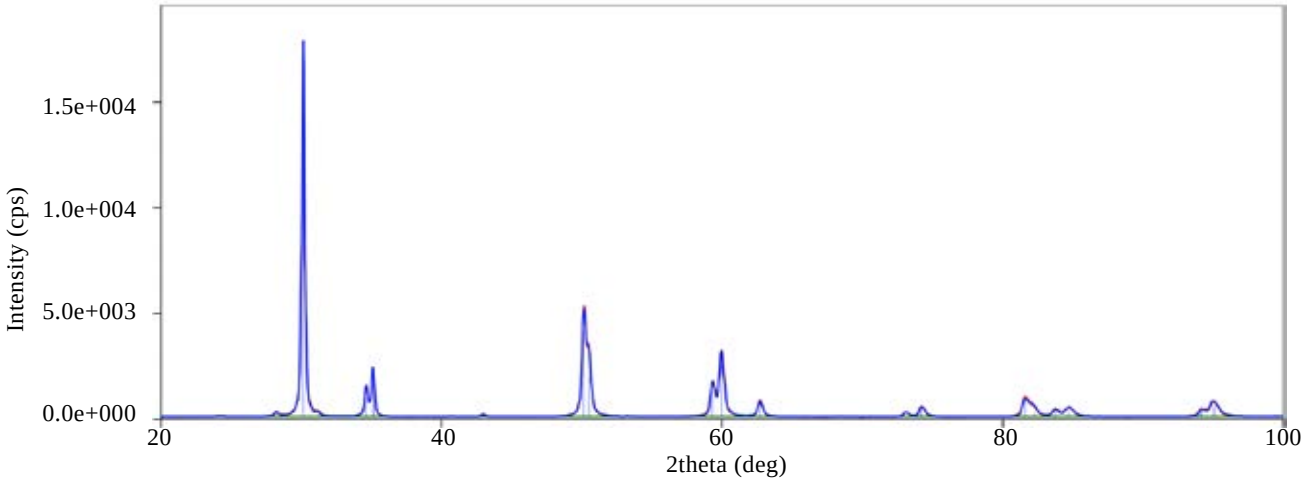

FIGURE 1. *X-ray diffraction of zirconia samples before sintering.*

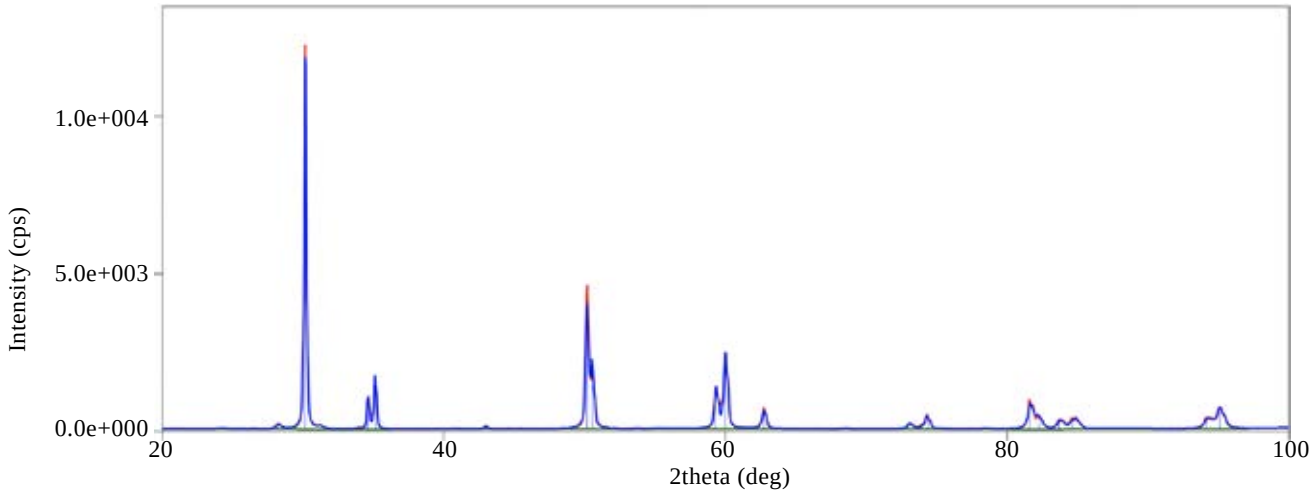

FIGURE 2. *X-ray diffraction of zirconia samples after 900°C sintering.*

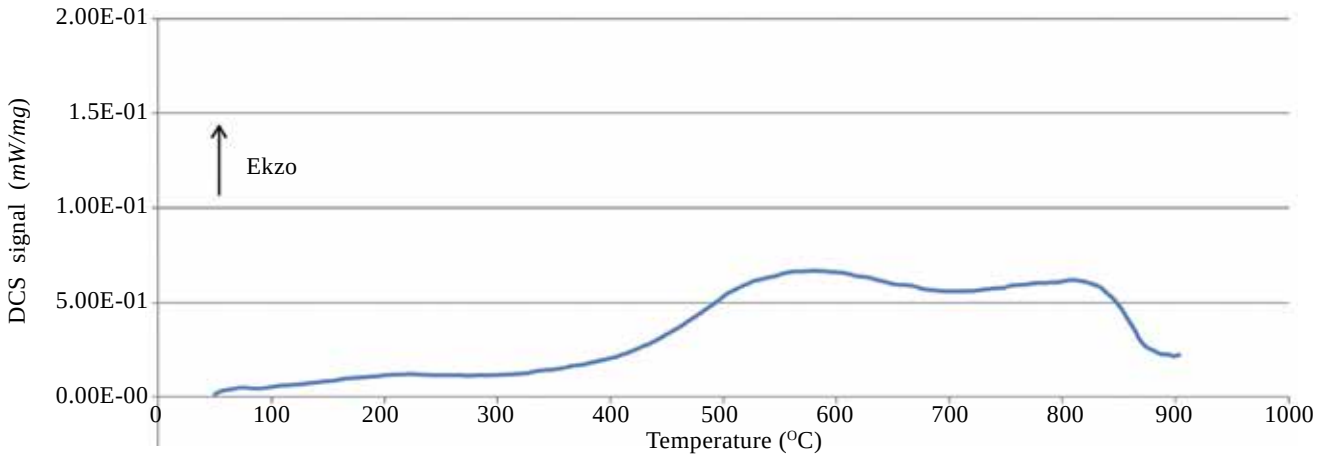

FIGURE 3. *DSC analysis of samples during the sintering process*

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terns of stabilized zirconia before and after heating (Figures 1 and 2). The peaks of the monoclinic phase were found in the region of 28.2°, and the percentage of the monoclinic phase increased on average from 3.6% to 7.5%, but this difference is not statistically significant (p=0.1). There is also a slight decrease in the peak at 30.2° (p=0.7), which represents the tetragonal phase.

The results of the DSC analysis of zirconia samples are presented in figure 3. When the samples were heated from 50°C to 900°C, exothermic peaks were observed in the region of 582°C and 809°C.

Discussion

One of the objectives of this study was to study the phase analysis of zirconium dioxide before and after heating at 900°C. According to the phase analysis data, the samples before and after firing differ insignificantly and are mixtures of tetragonal and monoclinic modifications of zirconium oxide. The amount of the monoclinic phase increases after firing, but insignificantly, which does not negate the null hypothesis.

Zirconium dioxide firing in the temperature range of 50-900°C is in the range of the monoclinic phase [*Chevalier J et al., 2006; Camposilvan E et al., 2018; Baldissara P et al., 2018; Edelhoff D et al., 2019*], thus, from this point of view, zirconium falls into the range of existence of the monoclinic phase, and this can lead to an increase in its fraction. The behavior of the phase transformation is directly related to the sintering temperature of zirconium dioxide [*Keuper M et al., 2014*]. However, when the restoration is sintered at temperatures above 1450°C for 1 hour, a higher susceptibility to degradation at low temperatures has been reported [*Keuper M et al., 2014*].

To date, the discussion about the transformation of the tetragonal phase into the monoclinic phase during firing remains open. Ozdogan A. (2020), Ebeid K. (2014) and their co-authors in their studies, as well, demonstrated that changes in temperature and time at various sintering parameters did not cause tetragonal-monoclinic phase transformations. The authors reported a complete transformation of the monoclinic phase into tetragonal in zirconium dioxide samples that were exposed to a temperature of 500-1000°C for 15 minutes [*Song J et al., 2013*]. Alkurt M. and colleagues (2016) reported that additional roasting processes, carried out by 2, 5, and 10 times, do not cause the transformation of the tetragonal phase into the monoclinic phase in the zirconium dioxide samples. However, Hjerppe J. and co-authors (2009) in their study showed that the content of the monoclinic phase in zirconium dioxide, which was subjected to heat treatment at 500-1200°C for 5 minutes, remained on the surface.

It is interesting to note that the phase transformations of highly transparent zirconia differ from the phase composition of traditional yttrium-stabilized zirconia [*Pereira G et al., 2016*]. The authors demonstrated the monoclinic phase absence in highly transparent zirconia before and after glazing [*Nam M et al., 2018*]; however, in our study, it was present before and after the final firing.

The transformation rate can be related to the grain size in zirconia [*Hjerppe J et al., 2009*]. According to the DSC analysis, there are exothermic effects that can be associated, firstly, with the transition of a small fraction from the tetragonal to the monoclinic modification [*Ebeid K et al., 2014*], and, secondly, with the oxide grain growth, and the bifurcation of the DSC peaks may indicate the superposition of these processes.

Conclusion

In this study it was demonstrated that the final firing of 3Y-TZP zirconium oxide up to 900°C slightly increases the percentage of the monoclinic phase and causes exothermal changes. We also believe that the inconsistencies in the literature are related to the different types and compositions of the zirconia used, as well as the different protocols for changing temperature.

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