Effect of surface abrasion and temperature treatment on metastable tetragonal zirconium dioxide (X-ray diffraction phase analysis)

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Abstract

Background. Zirconium dioxide is widely used in prosthodontics due to its strength and biocompatibility. However, a major drawback is its susceptibility to breakage and the tendency for porcelain layers to delaminate or chip(1). The durability and mechanical properties of zirconium dioxide can vary significantly based on the fabrication process, specifically regarding abrasive blasting and regenerative firing(2)(3). There is considerable debate among manufacturers and technicians about the best practices for these processes(4).

Aim. The study aimed to determine the effects of sandblasting and diamond bur abrasive treatment on the hardness and phase state of yttrium-stabilized tetragonal zirconium dioxide (Y-TZP). Evaluate the outcomes and appropriateness of regenerative firing.

Methods. An experimental study was conducted using synthesized ZrO_2 stabilized with Y_2O_3 (Y-TZP). The samples were divided into five groups: Sandblasted without subsequent regeneration firing; Sandblasted followed by regeneration firing; Abrasive blasting with a diamond bur without additional firing. Treated with both diamond bur abrasive blasting and regeneration firing. Control group with no mechanical or thermal treatments. A total of 80 pre-sintered Y-TZP blocks were milled and fired. Fifty defect-free samples were selected and treated according to the group protocols. The samples underwent X-ray diffraction phase analysis to assess the phase composition and structural changes. Statistical analysis was performed using TIBCO Statistica v13 and ANOVA Origin statistical package.

Results. X-ray diffraction phase analysis revealed that: Sandblasting increased monoclinic phases to 8% compared to 0.1% in control samples. Regeneration firing post-sandblasting reduced monoclinic phases to 1.5%. Diamond bur treatment increased monoclinic phases to 3%. Regeneration firing after diamond bur treatment restored monoclinic phases to the original 0.1%.

Conclusions. The study demonstrated that sandblasting significantly increases monoclinic phases in Y-TZP, while regeneration firing effectively reduces these phases. Diamond bur treatment also increases monoclinic phases, but regeneration firing can fully restore the material's original phase state. These findings highlight the importance of selecting appropriate fabrication techniques to optimize the mechanical properties and durability of zirconium dioxide in prosthodontic applications. **(TCM-GMJ December 2024; 9 (2): P3-P7)**

Keywords: Metastable Tetragonal Zirconium Dioxide(Y-TZP), CAD/CAM technology, abrasion and temperature treatment, X-ray diffraction phase analysis.

Introduction

The widespread use of zirconium dioxide as a framework material for prosthodontic constructs in everyday dental practice has highlighted its main drawbacks: construct breakage and delamination or chipping of porcelain layers from the frame(5)(6). There is ongoing disagreement between manufacturers and technicians regarding the optimal implementation of the technological process. Sandblasting frames of prosthetic constructions is a common method used to increase surface roughness, thereby enhancing the adhesion of porcelain layers(7). Additionally, sandblasting the inner surface of the frame is frequently employed to improve the adhesion of the structure to the teeth.

To properly fit a fully fired zirconia framework, adjustments with a diamond bur of a suitable grade, performed under water cooling, are often necessary(6). After this stage, it is typically recommended to perform zirconia

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Received December 03, 2024; accepted December 21 , 2024.
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regeneration firing(8)(9). However, there is an opinion that abrasive blasting induces the formation of microcracks due to the tetragonal-to-monoclinic (T-M) phase transition, causing local expansion and creating areas that prevent further crack development(8).. Conversely, high-temperature annealing can reverse this phase transition, relieve material stress, and potentially lead to further crack development(9).

A review of specialized literature on the effects of abrasive and heat treatment on fully synthesized tetragonal zirconia reveals widely divergent opinions(8)(9)(4). Based on this, a study was conducted with the aims of: 1) Determining the type and extent of the effects of sandblasting and diamond bur abrasive treatment on the hardness and phase state of yttrium-stabilized tetragonal zirconium dioxide (Y-TZP), and 2) Evaluating the outcomes and appropriateness of so-called "regeneration" firing.

The fracture of ceramic material can result from a crack initiated by a single microstructural defect or the coalescence of multiple defects(1)(10). In practical applications, the resistance of the framework materials to elasticity and crack formation is of primary interest(11)..

X-ray diffraction phase analysis provides information about the constituent elements and phase state of crystalline materials. An important advantage of these studies is obtaining objective research results without causing significant physical damage to the material.

Methods

To conduct the research, samples of synthesized ZrO_2 stabilized with Y_2O_3 (hereinafter referred to as Y-TZP) were prepared and divided into five groups:

The first group samples were sandblasted without subsequent regeneration firing.

The second group samples were subjected to sandblasting followed by regeneration firing.

The third group samples underwent abrasive blasting with a diamond bur without additional firing.

The fourth group samples were treated with both diamond bur abrasive blasting and regeneration firing.

The fifth group samples were not subjected to any mechanical or thermal treatments, serving as the control group.

Statistical analysis of the results was performed using TIBCO Statistica v13 2022 software and the ANOVA Origin statistical package for analysis of variance. A total of 80 pre-sintered Y-TZP blocks (VITA In-Ceram YZ Cubes for inLab, Vita Zahnfabrik) were used as the research material. After milling, the final firing was conducted in a VITA ZYRCOMAT® 6100 MS furnace (Vita Zahnfabrik, Germany). Fifty defect-free samples were selected from the 80 using a light microscope and then divided into the following groups:

SB Group: Sandblasted with Al_2O_3 of 110 μm dispersity for 10 seconds, at a distance of 1 cm from the frame, at a pressure of 3 bar.

SBS Group: Treated as the SB group and additionally subjected to "regeneration" firing at 1000°C for 15 minutes (regeneration mode: 25 minutes, starting at 550°C, with a heating rate of 100°C/min).

D Group: Abrasively treated with a specialized diamond bur for zirconium dioxide "ZR6850.FG.016 Crown and Bridge" (Komet, Germany).

DS Group: Treated as the D group, followed by the same firing procedure as the SBS group.

C Group (Control): Not subjected to any mechanical or thermal treatments.

All sample surfaces were cleaned and dried, and final dimensions were measured with an accuracy of 0.01 mm.

Quantitative and qualitative X-ray phase analysis of the surface of Y-TZP test samples

X-ray diffraction phase analysis is an important method for obtaining information about the elemental and phase composition of materials with a crystalline structure. The essence of this analysis is that any crystalline body is characterized by its specific crystal lattice, a certain chemical composition, and a specific arrangement of atoms. The geometry of the crystal lattice takes into account changes in interplanar distances, and accordingly, Bragg angles (θ) are determined during diffraction by a given radiation.

X-ray diffraction phase analysis of polycrystalline bodies allows for the following:

Investigation of the phase composition of the material.

Determination of the parameters of the elementary component of a unit of material.

Determination of the exact composition of a solid.

Structural analysis to determine the coordinates of the constituent atoms.

Investigation of phase transitions of the material.

One of the important advantages of X-ray phase analysis is that when examining a polycrystalline material, it is not subject to the physical effects and separations that we encounter, for example, when analyzing powders. In our case, this advantage plays an important role and ensures the objectivity and high degree of accuracy of the research results.

Diffraction analysis is a method for objectively assessing the qualitative and quantitative phase composition of a material with a crystalline structure. X-rays incident on the crystal at different angles make it possible to determine individual modifications characteristic of the specified body. Thus, quantitative and qualitative X-ray phase analysis is based on the phenomenon of X-ray diffraction by a crystal lattice.

The method of qualitative phase analysis is based on determining the position and relative intensities of diffraction maxima. This phenomenon occurs due to the constituent atoms of the crystal lattice when X-rays are reflected.

Quantitative analysis is based on determining the intensity of diffraction reflection from the corresponding phase component in the object under study. Using this method, you can determine the quantity of different types of phases in a crystalline body. In our case, this method is optimal for studying possible phase transformations resulting from the mechanical and thermal treatment of zirconium dioxide.

In quantitative and qualitative X-ray phase analysis, radiation with a wavelength λ equal to the interatomic distance of the crystal is used. An X-ray beam incident on an object at an angle θ will freely pass through a monatomic layer, although it will be partially reflected at the same angle θ. Waves reflected from different planes interact with each other, resulting in interference. The interference of coherent waves is determined by their phases and amplitudes.

To generate the research results, a "X'Pert MRD Pro" (Malvern Panalytical GmbH, Germany) diffractometer was used. Experimental research was conducted in accordance with the ISO 6872:2008 (Dentistry-Ceramic materials) standard in the laboratories of specialized centers in Germany (Fraunhofer-Institut für Schicht- und Oberflächentechnik IST).

The components of the diffractometer are a goniometer, an X-ray tube, an X-ray sensor, and a measuring device. The beams, focused by the X-ray tube, are directed towards S1 and S2, after which they are diffracted at the surface of the sample and recorded by the detector S (**Figure 1)**.

It should be noted that on the goniometer, the surface of the sample coincides with the plane of focus. All parts of the measuring device are fixed on the goniometer. In the diffractometer, the sample under study was focused using the Bragg-Brentano method. The position in the thickness of the sample surface at angle θ is zero. To improve the formation of the diffraction pattern, the sample under study was rotated. When the detector position changed, the sample was rotated through an angle θ. The detector and sample holder are moved by a synchronous electric motor. The intensity of X-rays entering the detector is calculated by counting the pulse frequency $n = N/t$, where N is the number of pulses detected during time t.

The content of crystalline phases on the surface of the test sample was estimated from integral maxima in the range of 2θ values from 2° to 40°. Quantitative determination of crystalline phases in the sample was carried out by relative integral intensity. The most intense peaks of integral intensity were selected for analysis.

Results

On the surfaces of the samples under study, when calculating the quantitative phase composition, the following was revealed:

1. An increase in monoclinic phases during sandblasting up to 8 percent (compared to control samples, where it was 0.1 percent).

1.1. Subsequent regeneration firing reduced the content of monoclinic phases to 1.5%.

2. Treatment of samples with a diamond bur increased the content of monoclinic phases on the surface to 3 percent.

2.1. With further regenerative firing, it completely returned to its original state, where the content of monoclinic phases was 0.1%.

Conclusion

A detailed analysis of the phase composition of Y-TZP samples using X-ray diffraction revealed the effectiveness of regenerative firing. It was also found that sandblasting zirconium dioxide leads to a significant increase in monoclinic phases compared to diamond-bur processing. These findings highlight the importance of selecting appropriate fabrication techniques to optimize the mechanical properties and durability of zirconium dioxide in prosthodontic applications.

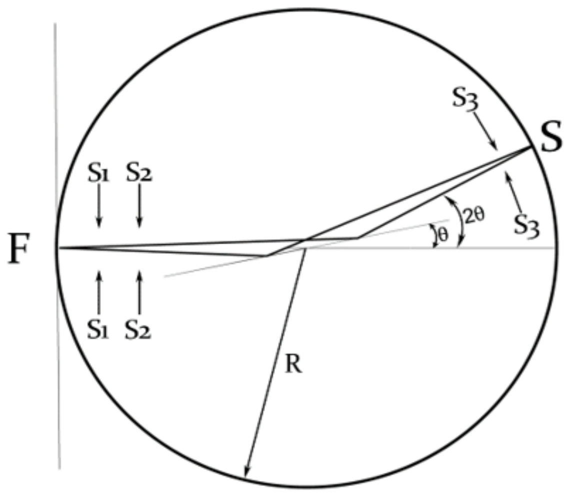

Figure 1. X-ray Diffractometer Operation Diagram

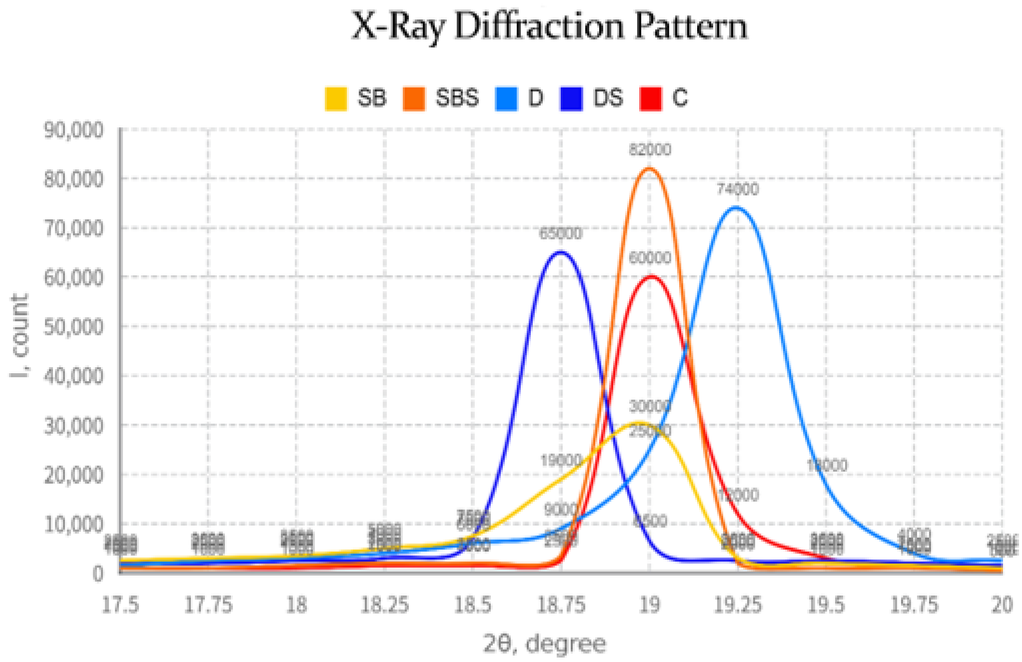

Figure 2. The diffractogram was analyzed, and the maximum intensity peaks corresponding to the monoclinic and tetragonal phases of zirconium dioxide were identified.

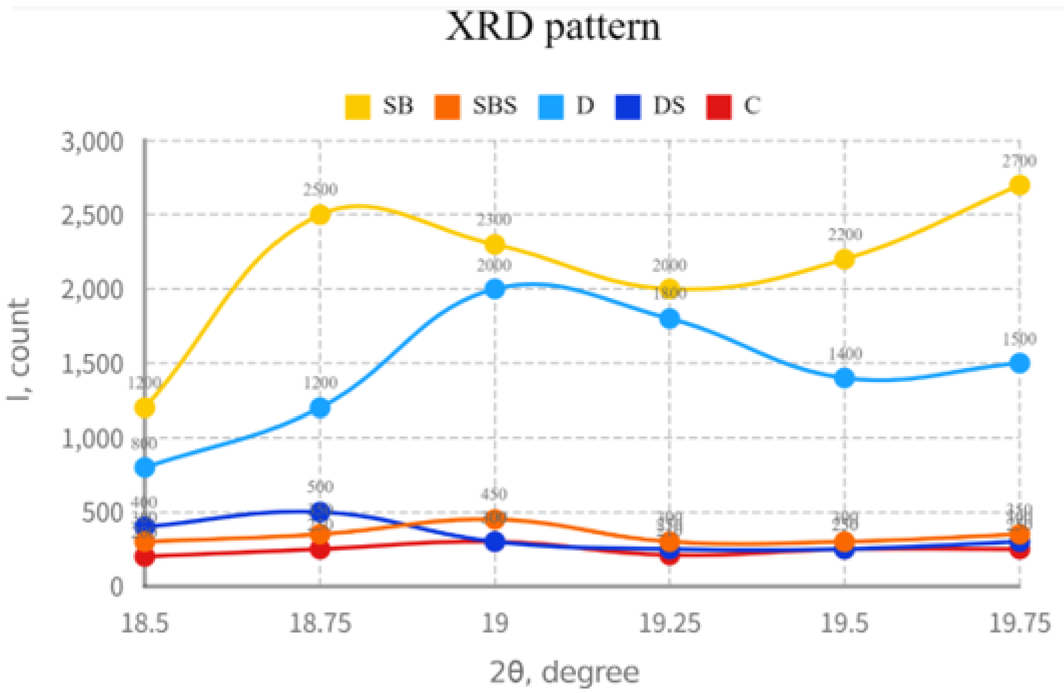

Figure 3. The diffractogram was analyzed, with the maximum intensity peaks of the monoclinic phase distinctly highlighted in a separate section.

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