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STUDY OF PHYSICOCHEMICAL PROPERTIES OF ZIRCONIUM DIOXIDE ZRO2 3Y - TZP USED IN DENTISTRY

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Abstract: *The article presents a chemical analysis and static mechanical strength tests on samples, with a geometry suggested by the authors. The market of dental materials is one of the most dynamically developing markets in the world. The materials used in the dental prosthetics must feature high biocompatibility, aesthetics and durability. In particular, the tests for materials used for dental crowns are widely developed. The test results related to the analysis of physical and chemical properties are used to improve the quality of services provided by the dental practices. The authors presented a newly developed method to prepare the mini-samples. 120 samples in 4 test groups were prepared: X-ray diffraction, derivatography, spectral analysis, 3-point bending static test. A chemical analysis showed that the tested material - zirconium dioxide is not susceptible to changes in temperature, and also showed numerous inclusions of other elements in the material.*

Keywords: physicochemical properties, zirconium dioxide, dentistry, 3 point bending tests

1. Introduction

The market of dental materials is one of the most dynamically developing markets in the world. The materials used in the dental prosthetics must feature high biocompatibility, aesthetics and durability. The development of dental materials requires improvement of the following features: chemical composition - biocompatibility, aging resistance of materials, which is reduced with the cyclic load (Munck et al., 2004). The main issue related to the development of dental materials is a lack of reliable analysis of physical and chemical properties of dental crowns and bridges. Zirconium dioxide (ZrO_2) and feldspar are the most commonly used dental prosthetic materials (Denry et al., 2008). Many laboratories around the world test the mechanical properties of dental materials. A review of the literature shows diversity of test methods related to the analysis of zirconium dioxide strength and stability. In particular, the tests for materials used for dental crowns are widely developed (Hisbergues et al., 2009). The test results related to the analysis of physical and chemical properties are used to improve the quality of services provided by the dental practices (Egilmez et al., 2014).

The article presents a method to prepare samples of zirconium dioxide, chemical analysis and static mechanical strength tests.

2. Materials and methods

The following material was used in the physicochemical analysis of zirconium dioxide: Cyrkon Lava by 3M ESPE. The material provided by the manufacturer is available as 60 mm x 25 mm x 16 mm blocks. The sample preparation technique includes three stages: the first involves cutting the material using Buehler ISOMET 5000 precision saw. The next involves polishing the surface with wet abrasive paper. The last stage involves laser cutting using Alfalas WS workstation. The processed samples were sent to the laboratory certified by the manufacturer for sintering (compacting). The process consists in firing the samples in a special furnace at 1410°C for 8 hours, during

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which the material shrinks by about 20% in volume. The tests were carried out on 120 samples (1.5 mm x 1.5 mm x 12 mm), divided into 4 groups depending on the physicochemical tests: X-ray diffraction, derivatography, spectral analysis and 3-point bending static test.

The phase composition of tested samples was determined based on the radiographic tests using x-ray diffractometer with URD 6 goniometer by Seifert with CuKα radiation and X-ray diffraction nickel filter. The analysis of data consisted in the comparison of X-ray patterns of tested samples with cards from the JCPDS database.

The thermal analysis was carried out in air atmosphere using Q 1500 D (MOM Budapest) 5°/min derivatograph, which measures change of the sample mass in time. A temperature curve (T), differential thermal analysis curve (DTA), thermogravimetric curve (TG) and thermogravimetric derivative curve (DTG) were plotted on a single graph. The temperature curve (T) allows to determine the transition temperatures.

The x-ray analysis was carried out to determine the chemical composition of the sample surface layer. The tests were carried out using Bruker S1 TITAN handheld X-ray fluorescent (XRF) analyser for a quick elemental analysis. The elements in the tested areas of the samples were quantified.

Static 3-point bending tests using Instron 8874 at ± 25 kN, ± 100 Nm were carried out to determine the mechanical strength of the samples.

3. Results

The X-ray diffractometry allowed to determine the crystallographic phases in the sample. Fig. 1 shows the X-ray pattern of the tested samples. The X-ray patterns were compared with the X-ray patterns for standard substances. A solid line in the graph is the X-ray pattern of the sample, whereas the points indicate peaks in the standard X-ray patterns.

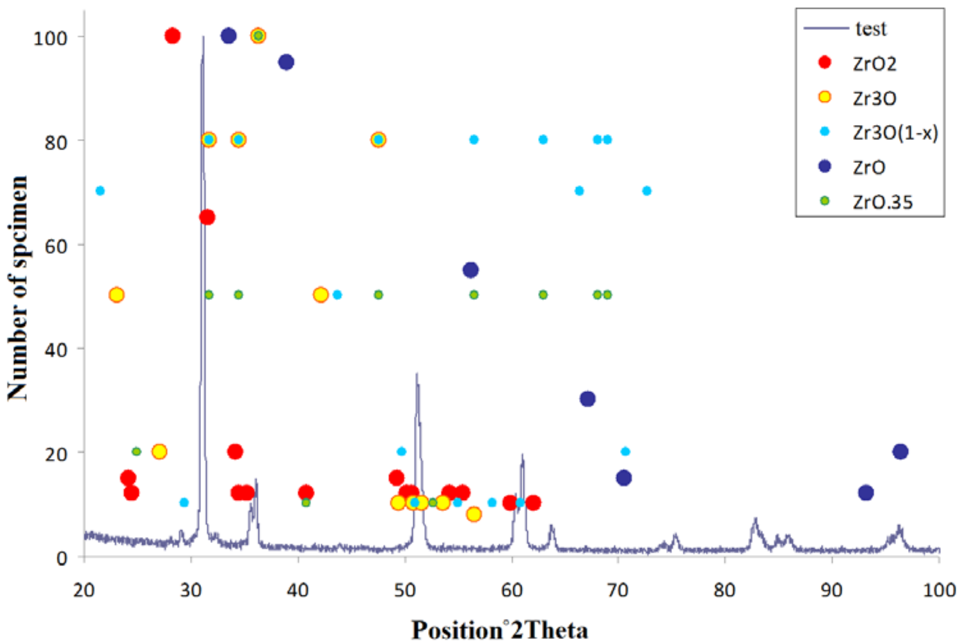

Fig. 1: Interpretation of the X-ray patterns for tested samples

The X-ray pattern allowed to determine the dominant phases in the analysed sample. An oxide phase, with the largest fraction is designated as phase I, whereas the fraction of phase II, which is also dominant is significantly lower. The result shows that the sample contains mostly ZrO and ZrO_2. It is possible, that other phases may be present, but were not identified due to the lack of suitable standards.

The thermogravimetric curves show the transitions temperatures, based on which a change in sample mass in mass percentage at initial, end and maximum temperature can be determined. The graph shows that the sample mass in the analysed temperature range does not change, which verifies the tested system resistance at this temperature range. Fig. 2 shows the thermogravimetric curves and record the temperature curve (T) curve Differential Thermal Analysis (DTA) , thermogravimetric curve (TG) , a derivative of a thermogravimetric (DTG).

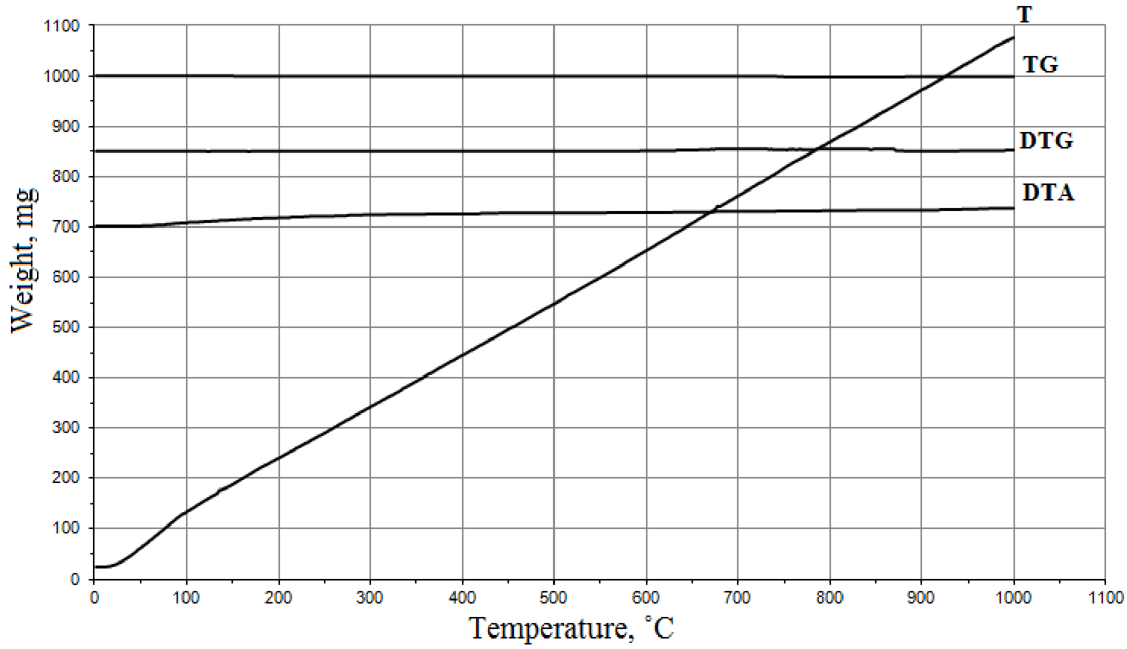

Fig. 2: Thermogravimetric curves of the sample, as change in the sample mass vs temperature.

The spectral analysis was carried out on a sample surface in point mode. Fig. 3 shows the example measurement point. The samples showed high and stable zirconium content at the surface. Due to the location of the measuring point, the statistical analysis showed no significant difference between the zirconium content at the sample surface. It indicates uniform distribution of this element in the tested system.

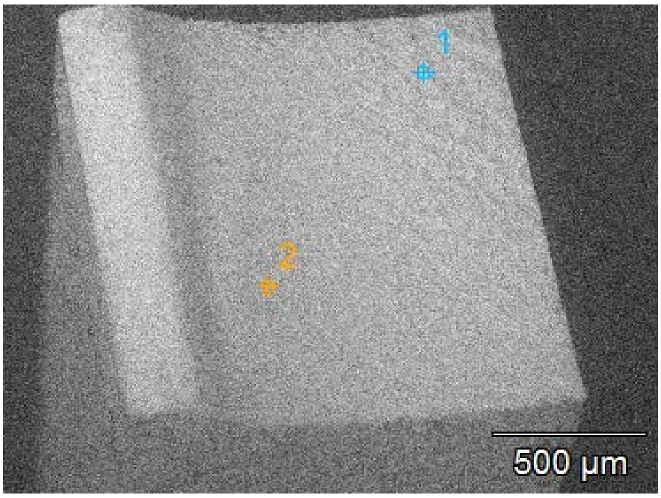

Fig. 3: Measuring points.

The results are represented as a spectrum. Each element emits a specific energy, and depending on its percentage content, its reflection on the graph differs in shape or height. Fig. 4 shows a spectrum for the analysis of a dental material with a non-diversified chemical composition. The main component of the tested sample is zirconium (Zr) with the highest energy, other peaks indicate Ca, C and O presence.

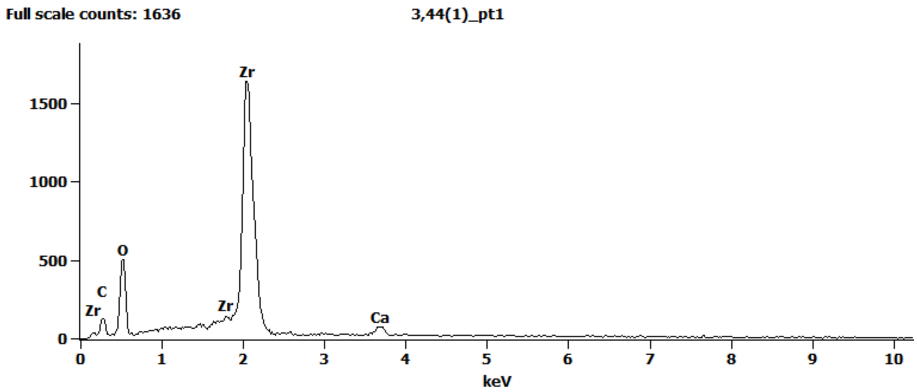

Fig. 4: Example XRF spectrum for Zr, C, O and Ca.

The results were exported to determine the percentage of major elements. The elemental composition and element ratio are similar for all samples. 50-60% zirconium content was determined in randomly selected points. The characteristics of zirconium content in relation to other elements in all measuring points is similar. The test results and statistical analysis show that the zirconium content is in fact the highest.

The last test was a static 3-point bending test to PN–EN 843–1. The standard specifies all test parameters, e.g. testing machine actuator speed, which is 0.5 mm/min. Table 1 shows the results of monotonic 3-point bending for a selected geometry from group 4: 1.5 mm x 1.5 mm x 12 mm. The tests were carried out on 30 samples.

Table 1. Monotonic 3-point bending test results for Cyrkon Lava and group 4 sample geometry: 1.5 mm x 1.5 mm x 12 mm

Material	**Average bending strength [MPa]**	**Standard deviation [MPa]**	**Relative standard deviation [%]**
Lava	978.17	78.32	8.11

4. Conclusions

The tests and analyses of physical and chemical properties of zirconium dioxide used in the dentistry for dental crowns and bridges were carried out. The analysis showed that the prepared zirconium dioxide based structure is characterized by good aesthetic, physical and chemical properties. The tests showed characteristic features of the material. The thermogravimetric analysis showed high thermal stability in a selected temperature range. The results were also verified by the analysis of the derivatographic curves. An interpretation of the X-ray patterns showed ZrO and ZrO_2 as the predominant oxides. The spectral analysis verified a significant fraction of zirconium in the tested samples. The physicochemical analysis (microscopic fractography, chemical analysis, roughness analysis and X-ray diffractometry) of the sample fracture shows changes in chemical composition of zirconium due to the loss of oxygen, increase in roughness and change in ZrO/ZrO_2 ratio. All the observed features of the sample fracture may indicate relation between the crack initiation and nature. A monotonic 3-point bending test and analysis of the results did not show any significant differences in mechanical strength. Comparison of the results with the results available in the scientific literature for similar materials did not show any significant differences in bending strength and standard deviation (Adams et al., 1997).

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