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INFLUENCE OF ELEMENTAL DISTRIBUTION IN ZIRCONIUM SAMPLES ON MECHANICAL STRENGTH

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Abstract: *Zirconia has become an alternative to materials used in dentistry because it is characterized by high mechanical strength and a high aesthetic parameter of materials, which resulted in reduced strength and lack of material stability. Zirconia stabilized with yttrium oxide falls under the category of intelligent materials, it means that when applying the right force to the material geometry, the structure of the material – the arrangement of the crystals is transformed from tetragonal to monocyclic. Due to this phenomenon, this material may hinder the propagation of crack propagation. The purpose of this article is physicochemical analysis that allows determining the distribution of aluminum compounds in the cross-section of zirconia, which was subjected to mechanical strength tests for three-point bending. The authors presented a method of producing ceramic samples, static tests of three-point bending strength, testing using an XRF X-ray spectrometer. The XRF analysis was performed by comparing the compound peaks for the external surfaces and cross-section.*

Keywords: Zirconium dioxide, Mechanical strength, Sinterization, Aluminiu distribution, Cross section.

1. Introduction

Zirconia has become an alternative to materials used in dentistry because it is characterized by high mechanical strength and a high aesthetic parameter (Dapieve et al, 2018). In addition, this material, due to stabilization of yttrium oxide, is used in dentistry and appears as several point arches, crowns, bridges, veneers, thanks to the possibility of choosing the right color compatible with natural teeth, translucency and a structure similar to natural teeth. In addition, zirconium dioxide replaced the two-layer materials previously used, where the combination of two materials followed, which reduced the strength and instability of the material (Nakamura et al., 2015). Zirconia stabilized with yttrium oxide falls under the category of intelligent materials, it means that when applying the right force to the material geometry, the structure of the material - the arrangement of the crystals is transformed from tetragonal to monocyclic. Due to this phenomenon, this material may hinder the propagation of crack propagation (Badami et al., 2014). Another use of zirconium dioxide is to use it in the production of cutting tools, gas sensors, refractory materials, ceramic or hybrid bearings. Any dental procedure during the preparation of bridges, arches or crowns may result in additional machining operations such as milling or conditioning (Xible et al., 2006). The next process is burning this material in an induction furnace, where it is subjected to appropriate temperature cycles required for a given zirconia (Blatz et al., 2004). The impact of all these processes on mechanical and physicochemical properties has not yet been fully studied and understood (Ban et al., 2013). In order to know the properties of the material, the following X-ray diffraction, derivatography, spectral analysis and static three-point bending tests are performed (Wirwicki et al., 2016). To better illustrate the lifetime of construction materials, and in this case dental components will be characterized, they can be determined using equivalent load amplitude in fatigue life calculations (Ligaj et al., 2016). In the patient's mouth during eating a meal there are very high values of bending and shearing moments of the teeth system, therefore the structural elements that are subjected to such a high value of destructive moments must show adequate hardness of the surface (Musiał et al., 2017 and Andrzejewska et al., 2017). In dentistry, an additional porcelain application process is performed, which allows you to match the color, increase the aesthetics and hardness (Wirwicki et al., 2013 and Karolewska et al., 2020).

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Mechanical tests were also carried out for instruments used in biomedical engineering supporting the convalescence of patients (Andryszczyk et al., 2014). In mechanical engineering, zirconium dioxide is used in the production of rolling bearings, which are used in transport conveyors used in industry with high contamination (Borowski et al., 2016). The purpose of this article is physicochemical analysis that allows determining the distribution of aluminum compounds in the cross-section of zirconia, which was subjected to mechanical strength tests for three-point bending.

2. Materials and methods

Cercon zirconia from DeguDent was used for static tests. The above material is used, among others, for the manufacture of crowns and bridges in CAD/CAM technology. This agent is currently one of the most generatively advanced materials used for prosthetic and implantological restorations. The material was processed with a circular saw (IZOMET 5000) and a Mazak Vertical Center Smart 430A milling machine. Ceramic materials used in dentistry are characterized by significant shrinkage during the curing - sintering process. According to the material manufacturer, the technological shrinkage is 20 %. This process consisted of firing in a special furnace at 1410 °C for 8 hours. The snow-white material after sintering showed a clear increase in parameters related to mechanical properties. Fig. 1 shows an example of the geometry of the samples after the sintering process, whose average dimensions were about 2 mm x 2.5 mm x 25 mm, 30 samples were used for the tests.

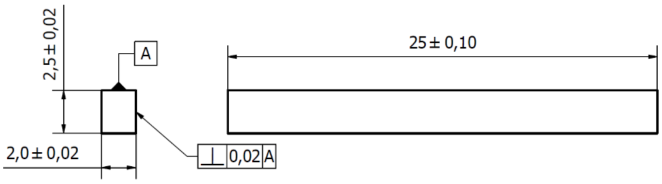

Fig. 1: Geometry of test samples.

During the tests, an Instron 8874 servo-hydraulic testing machine was used with strength and moment, respectively: ± 25 kN, ± 100 Nm. Sample height max. 100 mm and a rotation angle of 130 °. The device for three-point bending has been designed and made in accordance with the recommendations of the PN-EN 843-1 standard. After mechanical testing, a Bruker XRF portable X-ray spectrometer (S1 TITAN model) was used. The chemical composition of the surface layer of the samples was determined and subjected to X-ray analysis. The elements included in the samples in the studied areas were quantified. To better illustrate the destruction of the samples, a two-parameter analysis of the Weibull distribution was performed, which, as can be seen from the literature review for this type of research, is a standard. This analysis is described in the standard related to statistical analysis of advanced ceramics.

3. Results

The Instron 8874 testing machine performed static three-point bending strength tests according to PN-EN 843-1. The test was carried out at the battery speed of 0.5 mm / min to destroy the sample, and the frequency of recording the test results was 5 Hz. Tab. 1 presents the results of tests for three-point bending. Tab. 2 presents the results of the Weibull distribution for three-point bending tests.

Tab. 1: Results of monotonic three-point bending.

Material	Medium bending strength [MPa]	Standard deviation [MPa]	Relative standard deviation [%]
Cercon	802.18	186.33	23.22

Tab. 2: Results of Weibull distribution analysis.

Material	N	σ_0 [MPa]	R^2	m
Cercon	30	888.68	0.9754	7.3

Scanning microscopy, which was used to study zirconia samples, showed the relationship between the elemental composition of the surface layer and the interior of the sample. Fig. 2a shows a photo from the Scanning Electron Microscope (SEM) using an X-ray fluorescent attachment (XRF), showing a view of the sample surface. Fig. 2b shows a cross-section of a sample obtained as a result of three-point bending. Tab. 3 presents the elemental composition of the tested sample surfaces. On each of them several measurements were made, of which 2 characteristics were selected and compared. The composition of the outer surface of the sample showed a much richer element content as opposed to the interior of the ceramic sample. In both studied cases, the elements included zirconium, carbon and aluminum in the form of oxides.

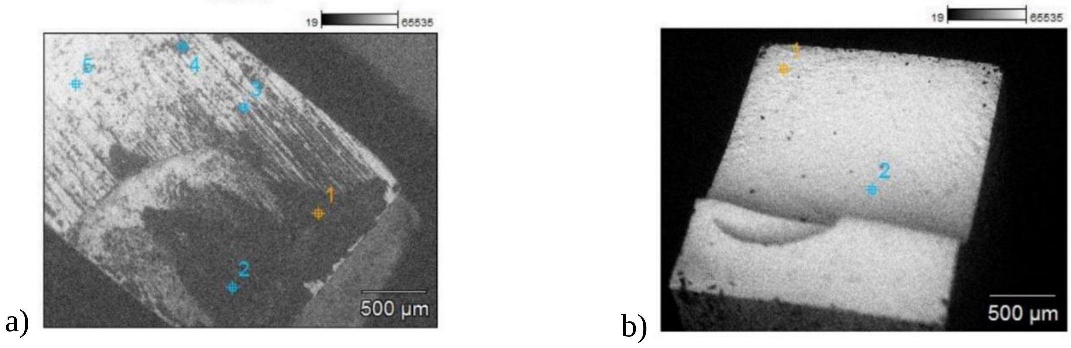

Fig. 2: Figures from an XRF X-ray spectrometer: a) external surface of the sample;, b) cross-section of the sample.

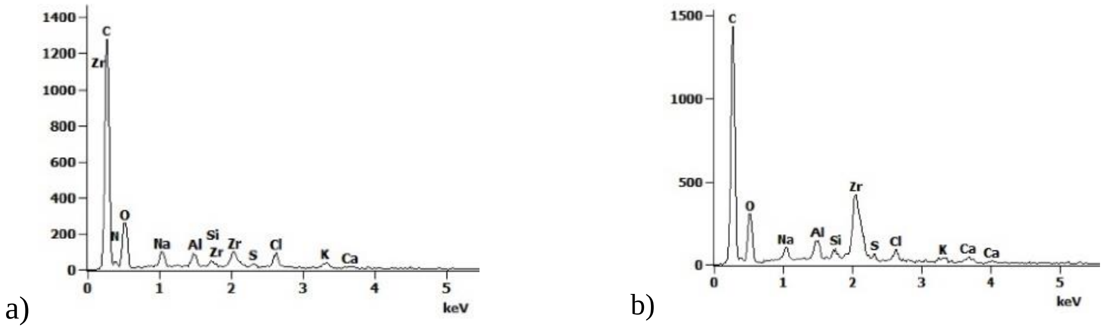

Fig. 3: XRF spectroscopy spectra for sample a (outer surface).

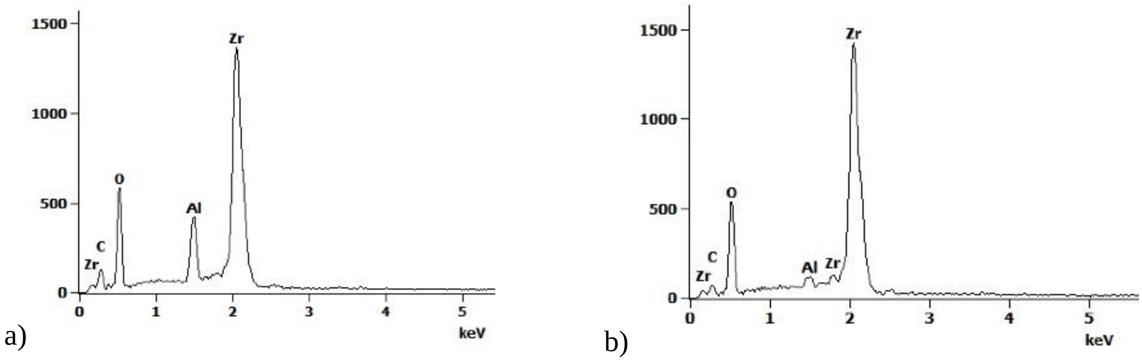

Fig. 4: XRF spectroscopy for sample b (cross section).

Tab. 3: Results of quantitative analysis for XRF spectroscopy.

	C-K	O-K	Zr-L	Ca-K	Al-K	Cl-K	K-K	Na-K	N-K	Si-K	S-K
a_1	43.0	30.6	5.0	0.5	1.5	2.6	1.8	2.3	11.5	0.7	0.4
a_2	45.9	24.0	20.8	1.5	1.8	1.8	1.0	1.7		0.6	0.8
b_1	3.8	25.6	65.4		5.1						
b_2	2.1	26.6	70.5		0.8						

3. Conclusions

Zirconia is a material with many applications for everyday and dental equipment. Contemporary material testing of new biomaterials, including dental ceramics, in many cases includes not only direct material characteristics such as compressive strength, bending, stretching, hardness or brittleness, but also a forecast of maintaining these characteristics in operating conditions. The operation process is closely related to the quality of zirconium products. Their quality determines the elemental composition of the material, especially the oxidation processes of the elements in the sintering process and their decomposition after sintering. The obtained results indicate a large impact of aluminum content on the mechanical strength of the material. Oxides of other metals depositing on the surface cause weakening of the material towards the external surface with an increase in strength inside the tested sample due to the presence of aluminum and zirconium. From the obtained mechanical and physical-chemical results it can be concluded that the mechanical strength of zirconium dioxide is strictly dependent on its composition and composition of inclusions in mass. This indicates the possibility of developing the topic in further scientific work.

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