

Influence of temperature during sintering of biomaterial - zirconium dioxide on mechanical strength

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Abstract. Zirconium dioxide is a material that has been known since the 1960s. Its first application took place in devices used by NASA. Zirconium dioxide is used in the production of all-ceramic and hybrid bearings. Since the 1990s, it has been the most popular application in dentistry, where it is used for crowns, arches and all-ceramic bridges. It is characterized by biocompatibility, translucency and high mechanical strength. Zirconium dioxide is supplied in round blocks with dimensions of 180 x 30 mm, its properties are similar to gypsum. Full mechanical properties are obtained during sintering - baking in the oven at the temperature of 1400 degrees Celsius for 8 hours. During this process, a shrinkage occurs, which is approximately 20%. The article presents three materials from zirconium dioxide from different manufacturers, and the original treatment of zirconium dioxide was presented. Each of the processed materials has been divided into three groups. Each group for a given material was sintered in a different way. The 3-point bending strength tests were carried out using the proprietary holder on the Instron 8874 servo-hydraulic testing machine. The results of the tests were presented on the charts and summarized by the discussion.

1. Intruduction

Zirconium dioxide is a construction material used in the construction of machines and in dental prosthetics. In 1963, first bearings with rolling elements made of ceramics were designed and used. This innovative component was used by NASA in one of the space programs. Since then, you can see a lot of interest in the use of these bearings [1]. Table 1 presents a comparison of selected properties of a typical bearing steel and ceramic materials used for bearing production - Si_3N_4 , ZrO_2 , Al_2O_3 . They pay attention to higher values of hardness and temperatures of ceramic materials in relation to typical steel used in bearings. Ceramics densities are lower than steel [3-4]. Hence, ceramic bearings are used in extreme conditions such as: elevated temperature and the risk of corrosion as well as a reduction in the weight of the entire system. Ceramic bearings are made of three different materials depending on the application and costs: [4]. Silicon nitride Si_3N_4 is used to work in an environment where the shaft is accelerated to significant rotational speeds and the bearing works under heavy loads, eg jet engines. Al_2O_3 aluminum oxide is used to work in an environment where there is a likelihood of corrosion, eg water turbines.

While analysing the selection of materials, machine parts are also conducted durability and strength tests, which allow a better diagnosis of survival of a given part [5-6].

ZrO_2 zirconium shows the greatest fatigue durability, and the production itself is very profitable. Hence, it is the most commonly used material for the production of ceramic bearings.



Table 1. Comparison of ceramic materials and typical steel used in bearing production [2].

Properties	Unit	Typical bearing steel	Si ₃ N ₄	ZrO ₂ (Yttria)	Al ₂ O ₃ (99,7%)
Density	g/ cm ³	7,6	3,20-3,30	6,00	3,95
Hardness	HV	700	1500-1800	1200	1800
	HRC	62	75-80	70	80
Young's modulus	GPa	208	300-320	210	380
Max. temperature of use	°C	120	800	500	1850
Poisson's ratio	-	0,30	0,26	0,30	0,27
Compressive strength (800°C)	MPa	400	1400	2100	1500

The second branch of applications is prosthetic and aesthetic dentistry, which is relatively young because the zirconium dioxide has been used since the 90s of the twentieth century. These materials had a different biocompatibility. Dentists are increasingly turning to zirconium dioxide because it has one more important feature in aesthetic dentistry - very high translucency (light penetration through the material). Nowadays, very complex refills, eg 6-point all-ceramic bridges, are possible thanks to very high mechanical strength of up to 1200 MPa. CAD / CAM technology gives the possibility to design and make a crown or bridge. Today, it is a process that can be performed in a dentist's office at the patient [7]. The very high biocompatibility of the material and chemical stability made this material optimal for applications in a non-hostile environment due to the presence of corrosive fouling [8-9]. What's more, this material has a very low wear and aging rate, which makes it one of the best dental materials on the market today [10]. Zirconium is a material that is characterized by an additional feature that distinguishes it from other materials. During the propagation of the cracking of the zirconium dioxide structure, they change their phase from the tetragonal to the monocycle phase, which causes the molecules to transform, thus strengthening the structure. This phenomenon can be called a strengthening transformation. During this process, the volume of zirconium dioxide particles increases by 3-5%, which reduces the destructive energy and thus decreases the propagation of the crack. This process reduces the spread of microcracks in the structure [11-12]. Another feature of ceramic materials, which is worth paying attention to is porosity. During firing, the mass is reduced by about 14-20%. This shrinkage depends on the amount of water used in the preparatory process of the ceramic mass for firing, the content of organic components, the shape and size of the grains of the powder. By adding additives such as fluxes and feldspar, and at the same time reducing the kaolin content, the porosity can be reduced. The grain strength of ceramic powder before its firing also affects the strength of the ceramic and its degree of smoothness [13].

The paper presents the results of three-point bending strength tests for three ceramic materials used in dentistry and machine building. The material was prepared using the original method of processing. The method of sintering for particular groups of materials and the geometry of the tested samples were analyzed.

2. Materials and methods

Three producers named: Zirconium Lava from 3M ESPE, Prettau Zirkon from ZirkonZahn and Cercon from DeguDent were used for analysis. The materials were delivered by the manufacturer in various forms: for the Cirkon Lava material, these were 60 mm x 25 mm x 16 mm blocks, Prettau's discs with a diameter of 95 mm x 16 mm and Cercon discs with a diameter of 105 mm x 20 mm. Thirty samples of each material were taken for the tests, the geometry of the samples used was selected in accordance with the PN-EN 843-1 standard. In order to obtain the intended cross-section, the authorial processing process of the obtained zirconium dioxide, consisting of: cutting the material, milling, was used. The cutting process was carried out using a Buehler ISOMET 5000 circular saw. This tool has a special fastening, thanks to which the machined surface is parallel to the cutting blade. The device also has an implemented program that adjusts the amount of coolant, feed and rotational speed depending on the material being processed. The device allows you to adapt the program to the type of material.

Parameters that selected the machine were as follows: the speed of rotation of the cutting blade was 4950 rpm and the feed was 19.0 mm / min. The next machining process was milling the shape of the sample using the Mazak Vertical Center Smart 430A milling machine. Machine settings did not allow over-heating of zirconium dioxide. Then the material was sent to a dedicated laboratory of each producer, in which in a specialized furnace at 1410 ° C for 8 hours it was sintered - hardened. The sintering process is characterized by approx. 20% zirconia contraction. The material after sintering obtains a snow-white color, and the mechanical properties clearly increase.

Prior to testing, the samples were measured with a Mitutoyo 150mm AOS Absolute calliper with a resolution of 0.01 mm and an accuracy of ± 0.02 mm. 90 samples were used for the assessment, with a cross-section of 2 mm x 2.5 mm x 25 mm. The obtained material was divided into 3 groups of 30 samples (A - Lava Cyrkon, B - Prettau Zirkon, C - Cercon).

Table 2. Analysis of the geometry of the tested zirconia samples.

Cross section [mm]	Average value of width and height [mm]	Standard deviation [mm]	Relative deviation standard [%]
2 x 2,5	2,03	0,09	4,42
	2,43	0,07	3,01

The results presented in Table 2 were analyzed, which showed that for the 2 mm x 2.5 mm cross section the standard deviation for X and Y was 0.09 mm and 0.07 mm, respectively, and the relative standard deviation was 4.42% and 3.01%. Repeatability in the area of the cross-section is caused by: low variability of obtained geometrical parameters (in the X and Y range) and reduction of the influence of external factors during material processing. The distribution of the results obtained is shown in Figure 1.

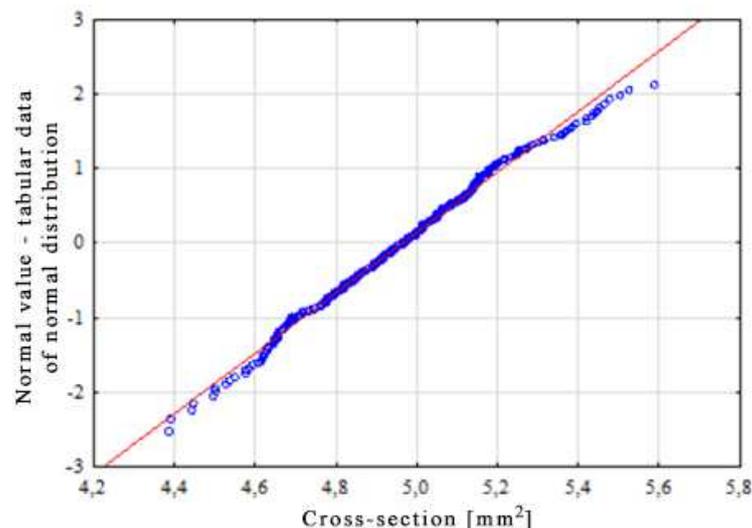


Figure 1. Graph of distribution of results distribution relative to normal for samples.

Figure 2 presents the spread of the results of the absolute dimension of the X axis of the processed samples for three groups of sintered materials provided by the manufacturer. Figure 3 shows the dispersion of the results of the absolute Y-axis dimension of the processed samples for three groups of sintered materials provided by the manufacturer. Figure 4 presents the scattering of the results of the absolute cross-section for three groups of samples tested.

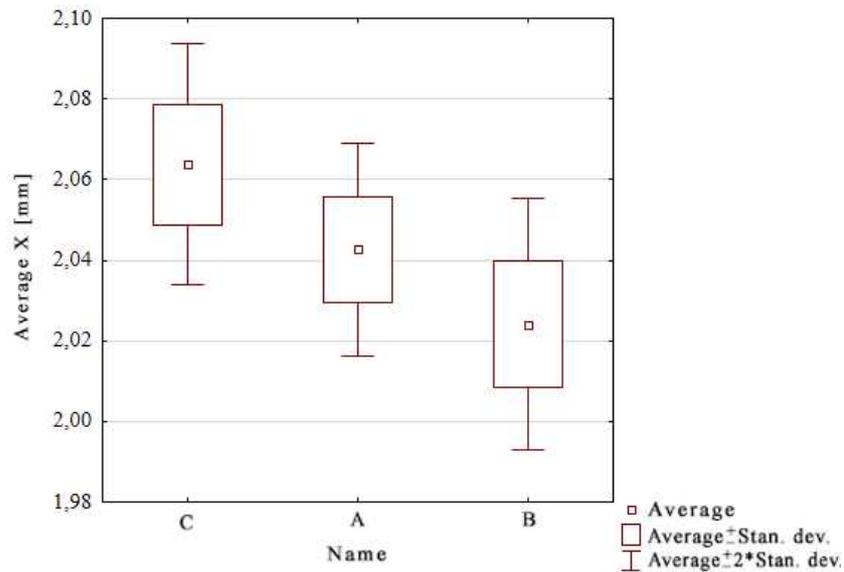


Figure 2. Dispersion of absolute results for the X-axis dimension and three groups of samples.

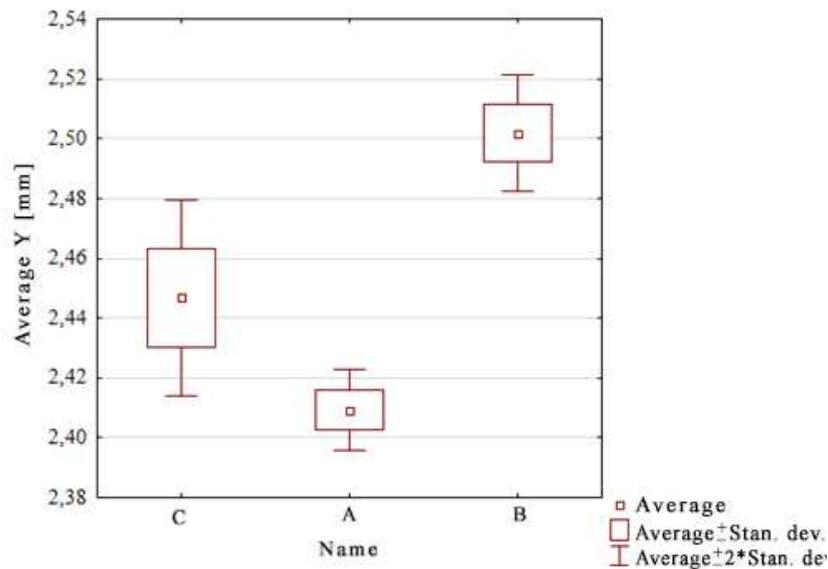


Figure 3. Dispersion of absolute results for the dimension of the Y-axis and three groups of samples.

During the tests, an Instron 8874 servo-hydraulic testing machine was used with strength and moment, respectively: ± 25 kN, ± 100 Nm. The device for three-point bending has been designed and made in accordance with the recommendations of the PN-EN 843-1 standard: Technical advanced ceramics: Mechanical properties of monolithic ceramics at room temperature. Part 1: Determination of bending strength. Adjustable supports on the handle allow for testing samples from 5 mm to 45 mm long.

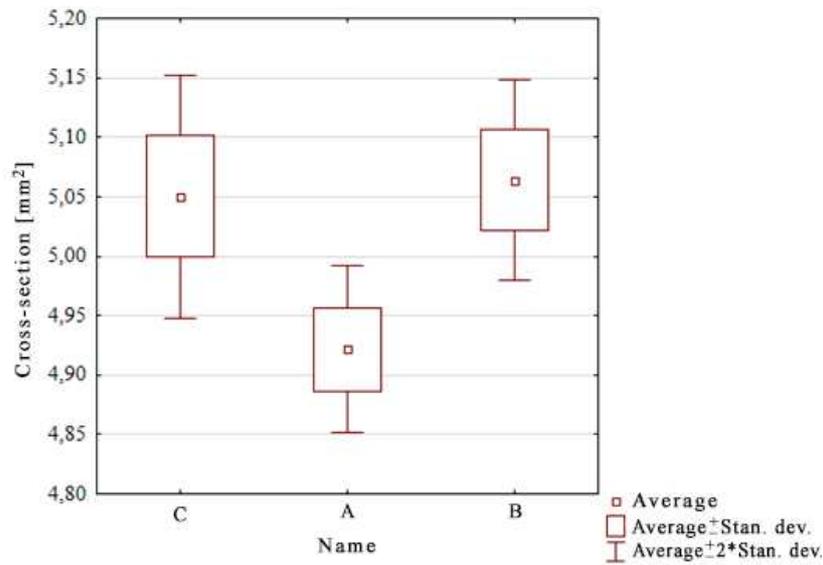


Figure 4. The distribution of absolute cross-section results for three groups of samples tested.

3. Results

Table 3 presents the results of descriptive statistics and the normal distribution for the materials tested. The results obtained for materials synthesized in standard parameters show the presence of statistically significant differences. Average values of breaking stress for material A - 985 MPa, B - 900 MPa, C - 802 MPa. Figure 5 presents a scatterplot of results for groups of samples taken for three-point bending tests with respect to the cross-sectional area.

Table 3. Results of descriptive statistics and normal distribution of the materials tested.

Name	N	x	Me	Min	Maks	Q1	Q3	SD	V	Normality tests P value
A	30	985,6	1003,0	640,5	1305,7	840,4	1155,5	182,9	19%	0,56
B	30	900,4	899,6	601,6	1293,3	797,9	984,1	151,8	17%	0,96
C	30	802,2	764,8	401,4	1215,8	684,0	948,1	186,3	23%	0,87

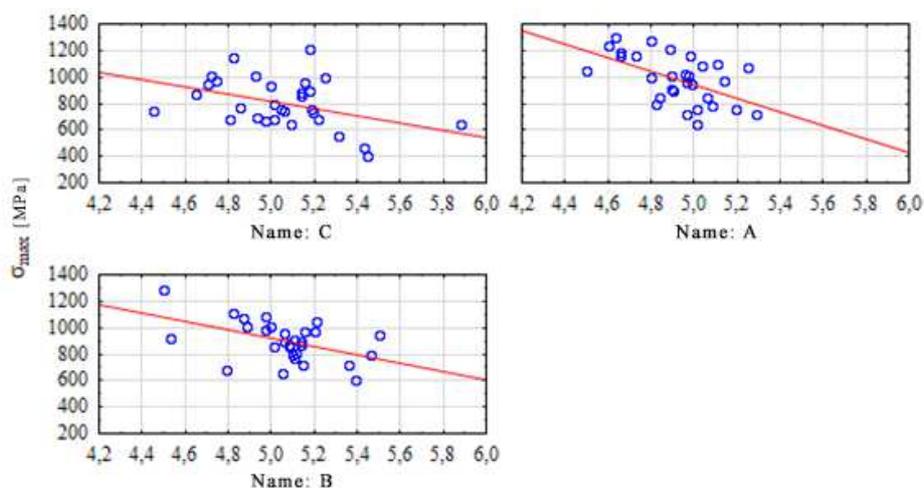


Figure 5. Scatter plot relative to the surface area of the sample for three groups of materials.

4. Conclusion

The analysis of the sintering results (geometry) for material B has a significant higher sintering index, which in this case must be understood as an increase in the Y-dimension in relation to the other materials A and C. If we analyze the values of the X-axis, which was presented on drawing 2 of all samples taken for testing, it can be seen that B has material that has reduced its size to the least extent. The remaining groups of samples have this higher ratio, which may indicate that the change in X and Y dimensions is much more propagated in one direction for samples B, and the dimensional values for A and C are similar to each other. In the Y-axis it can be seen that the samples sintered for material A show much less spread than for material B and C. This may be due to different oven temperatures and chemical composition. Analyzing the surface area of all samples, it can be seen that A has the lowest cross-section in relation to B and C, which may be the best machining properties. The tests carried out show that the material which is sintering has a shrinkage scatter, causes the lack of control over the process of burning samples (sintering). An additional problem is the scattering of research results, this may be due to the phenomenon of amplification. Randomly scattered air bubbles, which take a permanent form only after the sintering process, can cause such a scatter.

Zirconium dioxide is a "young" material, therefore further research related to the recognition of its mechanical properties must be carried out. The conducted research is preliminary research, showing that there is a large research potential for a given group of materials used in the construction of machines and dentistry. The next step is to study the sintering of the selected material under different temperature and time conditions. In addition, it is necessary to analyze the chemical composition and structure of the material in order to achieve full compliance, which takes place in the material during loading.

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