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COMPOSITES THEORY AND PRACTICE

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EFFECT OF ALUMINA NANOPOWDER ADMIXTURE ON CONSOLIDATION OF $3Y_2O_3$-ZrO_2 MICROPOWDER IN PROSTHODONTIC APPLICATION CONTEXT

Zirconia micropowder stabilized with 3 mol% yttrium oxide is used to produce bisques, being the pre-sintered blanks intended for the manufacturing of all-ceramic dental restorations. The bisques should be characterized by mechanical strength and fracture toughness suitable for the precise milling of thin-walled items afterwards sintered to full density, ensuring good performance. The aim of this research was to study the effect of a 0.2 mass% nano-Al_2O_3 addition to the TZ-3Y Tosoh powder on its behaviour during low and high-temperature consolidation, and on the properties of green compacts, bisque-sintered and fully-sintered TZP materials. The bisque-sintered materials were manufactured at 500÷900°C, and characterized in terms of their densification, mechanical strength, hardness and fracture toughness, and compared to the Cercon and ICE Zirconia green materials. The materials sintered at 1200÷1550°C were analysed to show the influence of the alumina addition and pre-sintering temperature on the densification and microstructure. Comparison was also made to sintered bodies derived from the Cercon and ICE Zirconia materials. Both unmodified and Al_2O_3 modified TZ-3Y powder can be used for the fabrication of bisques for dental applications. An increase in strength and hardness of the bisques with a pre-treatment temperature was observed, and it significantly affected their behaviour during milling; the bisques pre-sintered at 700 and 900°C showed the best machining properties. The addition of 0.2 mass% nano-Al_2O_3 to the TZ-3Y powder contributed to lowering the final sintering temperature, and to obtaining a microstructure analogous to the Cercon derived one.

Keywords: **3Y-TZP, Al_2O_3, bisque, all-ceramic dental restorations**

WPŁYW DOMIESZKI NANOPROSZKU Al_2O_3 NA KONSOLIDACJĘ MIKROPROSZKU $3Y_2O_3$-ZrO_2 W KONTEKŚCIE ZASTOSOWAŃ PROTETYCZNYCH

Proszek dwutlenku cyrkonu, stabilizowanego dodatkiem 3% mol. tlenku itru, znalazł zastosowanie do produkcji biskwitów przeznaczonych do wytwarzania podbudów pełnoceramicznych uzupełnień protetycznych. Biskwity poddawane są obróbce mechanicznej, dlatego powinny cechować się odpowiednią wytrzymałością mechaniczną i odpornością na kruche pękanie, aby możliwe było frezowanie precyzyjnych cienkościennych elementów. Etapem końcowym jest spiekanie wyfrezowanych podbudów do pełnego zagęszczenia w celu nadania odpowiednich właściwości użytkowych. W pracy przedstawiono wpływ dodatku 0.2% mas. nanometrycznego Al_2O_3 na konsolidację do postaci biskwitów i gęstych spieków komercyjnego mikroproszku cyrkoniowego TZ-3Y (Tosoh), a otrzymane wyniki porównano z prezentowanymi przez polikryształy tetragonalnego dwutlenku cyrkonu (TZP) pochodzące z proszku niemodyfikowanego dodatkiem tlenku glinu oraz przez materiały handlowe - Cercon i ICE Zirconia, stosowane w technice dentystycznej. Biskwity materiałów TZP modyfikowanych i niemodyfikowanych dodatkiem nano-Al_2O_3 otrzymywano na drodze prasowania i wstępnego spiekania w temperaturach 500, 700 i 900°C przy czasie wytrzymania 0,5 h. Aby ocenić przydatność do obróbki mechanicznej, biskwity charakteryzowano pod względem stopnia zagęszczenia oraz wytrzymałości mechanicznej, twardości i odporności na kruche pękanie. Spiekanie do pełnego zagęszczenia prowadzono w temperaturach 1200÷1550°C, stosując dwugodzinny czas wytrzymania. Charakterystyka spieczonych tworzyw obejmowała badania nad wpływem dodatku Al_2O_3 i temperatury wstępnego spiekania biskwitów na ostateczne zagęszczenie spieków. W pracy wykazano, że komercyjny proszek TZ-3Y Tosoh, nie modyfikowany i modyfikowany dodatkiem Al_2O_3, może być zastosowany do produkcji biskwitów przeznaczonych do wytwarzania pełnoceramicznych uzupełnień protetycznych. Wzrost wytrzymałości i twardości biskwitów w wyniku podnoszenia temperatury obróbki wstępnej wpływa znacząco na obróbkę mechaniczną, dlatego najlepsze warunki obróbki mechanicznej obserwowano w przypadku biskwitów wytwarzanych w temperaturach 700 i 900°C. Zastosowany dodatek 0,2% mas. Al_2O_3 do tworzyw 3Y-TZP nie wpłynął znacząco na obróbkę mechaniczną biskwitów, ale przyczynił się do obniżenia temperatury spiekania końcowego. Przyczynił się też do uzyskania mikrostruktury gęstych tworzyw TZP analogicznej do prezentowanej przez spieki uzyskane z tworzywa Cercon.

Słowa kluczowe: **3Y-TZP, Al_2O_3, biskwit, pełnoceramiczne uzupełnienia protetyczne**

INTRODUCTION

Since the discovery of transformation toughening by Garvie et al. in 1975 [1], zirconia ceramics have become an important oxide material with attractive properties not only for high temperature structural and functional applications but also for low temperature, advanced and high performance structural applications. High strength, fracture toughness, ionic conductivity, and low thermal conductivity are available in different quantitative combinations in zirconia ceramics, and are the reasons for the wide use of specialized zirconia materials in energy, wear, bearing, thermal barrier coating and biomedical applications [2]. Over the past several years, the manufacturing of all-ceramic dental restorations is the most interesting biomedical application of zirconia in the form of tetragonal zirconia polycrystals (TZP) because of their outstanding low temperature mechanical properties, biocompatibility as well as aesthetic benefits.

The preparation of all-ceramic dental restorations from TZP material, typically stabilized with 3 mol% yttria (3Y-TZP), consisted in milling a bisque-sintered material and subsequent full-sintering. The bisques are formed by uniaxial and/or cold isostatic pressing, followed by pre-sintering to receive a state of consolidation giving properties suitable for machining. Homogeneous density, appropriate mechanical strength and fracture toughness are the most important parameters for the milling of thin-walled TZP products. According to the literature [3-6], a small amount of Al_2O_3 added to the 3 mol% Y_2O_3-ZrO_2 powder modifies its densification behaviour during consolidation and therefore the microstructure of the resultant TZP material. The results given by Matsui et al. [3, 4] show that the rate of densification of 3Y-TZP with 0-1 mass% Al_2O_3 increased with an increasing alumina addition. These levels of Al_2O_3 addition also cause an increase in grain size, a decrease in density at higher sintering temperatures and a resultant change in the mechanical properties [6, 7].

The aim of this research was to investigate the effect of a 0.2 mass% nano-Al_2O_3 addition to the commercial TZ-3Y (Tosoh) powder on its behaviour during low temperature consolidation, pre-sintering and pressureless full-sintering. Special focus is on a collection of data on the temperature dependent evolution of porosity in pre-sintered bisque samples, their mechanical properties and machinability, followed by comparison to relevant data for selected commercial materials used in modern dental technology. A second focus is to study the sinterability and microstructure of the alumina modified TZ-3Y bisque samples with comparison to undoped and selected commercial pre-sintered samples.

EXPERIMENTAL PROCEDURE

Commercial zirconia powder containing 3 mol% Y_2O_3 (TZ-3Y grade, Tosoh, Tokyo, Japan) and nanometric, high purity Al_2O_3 powder (TM-DAR, Taimei Chemicals Co. Ltd., Tokyo, Japan) were used as the starting components. Zirconia TZ-3Y powder with a 0.2 mass% Al_2O_3 addition, termed TZ-3Y-A, was prepared by wet-milling with a gravitational ball mill for 3 h under peptizing conditions created by distilled water of pH $\cong$ 2. The resultant homogeneous slurry was flocked at a pH of ~7 just before drying at 105°C to a constant weight. Both the TZ-3Y and TZ-3Y-A powders were shaped into disks by using first uniaxial pressing under 73 MPa and then cold isostatic pressing under 300 MPa. A part of the green compacts was pre-sintered at 500, 700 or 900°C for 0.5 h in air, using a heating rate of 5°C/min to 350°C, and 10°C/min above 350°C. Afterwards, the bisque-sintered specimens and the rest of the green compacts were fully sintered for 2 h at temperatures ranging from 1200 to 1550°C in air under atmospheric pressure, using the heating schedule indicated above. The specimens fabricated from the TZ-3Y and TZ-3Y-A powders are called 3Y-TZP and 3Y-TZP-A, respectively. The samples of Cercon (Degudent) and ICE Zirconia (Zirkonzahn) materials were included in the procedure of full sintering.

The specific surface area of the TZ-3Y and TZ-3Y-A powders was measured by nitrogen adsorption (Nova 1200e, Quantachrome Inc.) and calculated from the BET isotherm. The particle size distributions were measured by the laser diffraction method (Mastersizer 2000, Malvern Instruments) using the powders dispersed in distilled water with an ultrasonic wave generator of a 100 W capacity. The compaction behaviour of the TZ-3Y and TZ-3Y-A powders was characterized by determining the densification curves using a Z 150 Zwick/Roell testing machine with a loading rate of 5 mm/min and a maximum load of 5.69 kN. Densification of the green compacts and bisques was characterised by the Archimedes' method. The pore size distributions of the green compacts together with the Cercon and ICE Zirconia samples were measured by mercury porosimetry (PoreMaster 60, Quantachrome Inc.). The bending strength was measured in a bi-axial bending test with a loading rate of 1 mm/min (Z 2.5 Zwick/Roell). The hardness and critical stress intensity factor, K_{Ic}, were measured by Vickers indentation under 4.9 N for 10 s (Microhardness Tester FM-700, Futur-Tech). Calculations of the K_{Ic} value were made according to the Palmqvist crack model [7]. The microstructure of the full-sintered specimens was observed by scanning electron microscope (Nova Nanosem 200, FEI Company) of surfaces that were polished and thermally etched for 2 h at 1350°C. The grain size was obtained by multiplying by 1.56 the average linear intercept length of at least 200 grains. The relative density of the bisque- and full-sintered samples was determined by the Archimedes' method. The geometrical density was determined for the green compacts.

RESULTS AND DISCUSSION

Characteristics of powders, green compacts and bisques

The specific surface area of the original TZ-3Y and modified with Al_2O_3 TZ-3Y-A powders were 15.0 ± 0.1 m^2/g and 14.7 ± 0.5 m^2/g, respectively. These results are comparable within the limits of measurement error. There is reason to suggest that the preparation of the TZ-3Y-A powder did not cause the development of the specific surface area by breaking-up the zirconia and alumina particles.

The TZ-3Y powder originally consisted of granules as a result of the production by Tosoh. In the case of the TZ-3Y-A powder, the agglomerates were formed during flocking, drying and rubbing on a sieve. The procedure of powder preparation for primary particle size analysis leads to the destruction of the granules and agglomerates. Details of the size analysis of the Al_2O_3, TZ-3Y and TZ-3Y-A powders were presented in [9]. Summarizing, it can be mentioned that the cumulative curve of particle size distribution of the Al_2O_3 powder was very narrow and monomodal with a grain size in the range of 46 to 316 nm, and a modal value located at 125 nm. The particle size distribution of TZ-3Y showed three-modal characteristics in the range of 0.035÷÷79.4 μm. The modal values of the major and minor population were 1.73 and 0.15 μm, respectively. A small population of particles with a modal value of ~15 μm probably consisted of remnants of undamaged granules. The TZ-3Y-A powder showed a narrow, monomodal particle size distribution with a grain size in the range of 0.316 to 6.607 μm. The modal value of 2.05 μm was determined. It was larger than the mode of major population in the TZ-3Y powder. An increase in the modal value of about 18% and narrowing the particle size distribution were observed due to alumina incorporation and the related homogenization process which included both peptization and flocculation of the dispersion. The original granules of the TZ-3Y powder were destroyed during that process and a new state of granulation was rebuilt by subsequently rubbing the dried powder on the sieve. However, the new granules (agglomerates) of TZ-3Y-A powder attained lower strength as confirmed by the densification curves shown in Figure 1. The breaking points in the curves indicate pressures causing the deformation of granules and primary agglomerates that formed each granule. Comparing to TZ-3Y, the granules and agglomerates of TZ--3Y-A deform under lower pressures, which proves their lower mechanical strength.

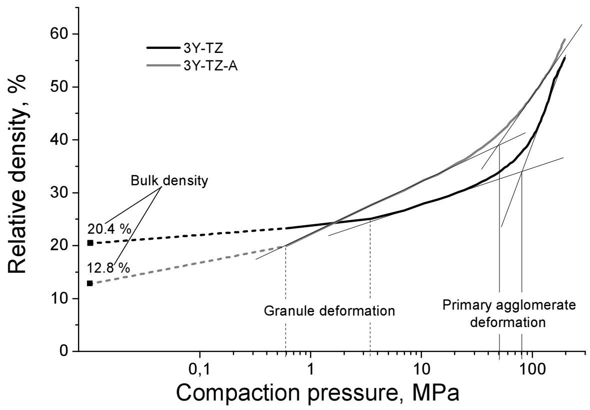


Fig. 1. Compaction curves of TZ-3Y and TZ-3Y-A powders

Rys. 1. Krzywe zagęszczania proszków TZ-3Y i TZ-3Y-A

Soft agglomerates of low mechanical strength show a stronger tendency to better packing during pressing than hard agglomerates. This is proved by the results of relative density measurements for the green compacts. After pressing under 300 MPa, the 3Y-TZP and 3Y-TZP-A green compacts reached a density of 49.9±0.2% and 51.4±0.3%, respectively (Fig. 2). A state of greater densification of the TZ-3Y-A powder derived compacts was also present after pre-sintering, as shown in Figure 2.

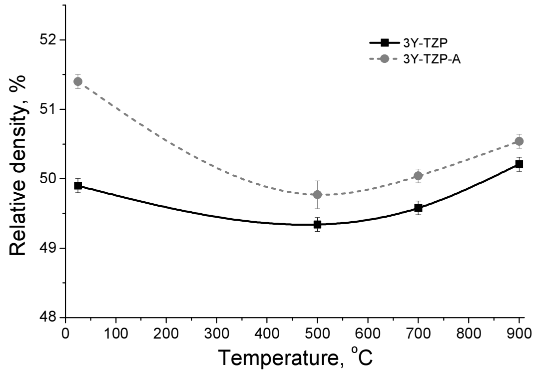


Fig. 2. Relative density of green compacts and bisques of 3Y-TZP and 3Y-TZP-A as a function of temperature

Rys. 2. Gęstość względna wyprasek i biskwitów tworzyw 3Y-TZP i 3Y-TZP-A w funkcji temperatury

Mercury porosimetry measurements revealed monomodal pore size distributions in both the green compacts and bisques of the powders studied (Fig. 3).

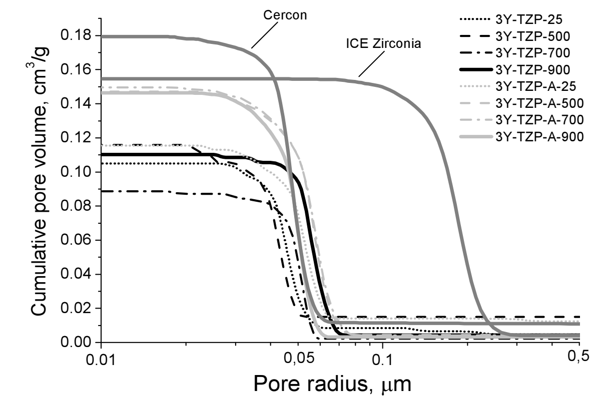


Fig. 3. Pore size distributions of 3Y-TZP and 3Y-TZP-A compacts: as-received (25°) and pre-sintered at 500, 700 or 900°C. For comparison, data for commercial ICE Zirconia and Cercon materials are also included

Rys. 3. Krzywe sumacyjne rozkładu wielkości porów w próbkach materiału 3Y-TZP i 3Y-TZP-A: surowej (25°C) i wstępnie spiekanych w 500, 700 lub 900°C. Dla porównania włączono również dane dotyczące komercyjnych materiałów ICE Zirconia i Cercon

The measurements also documented a transient pore size increase, accompanying the temperature increase, which was influenced by the Al_2O_3 addition. In the case of the 3Y-TZP material, an increase in the modal pore size, from 0.04 μm for the green compacts to 0.07 μm for the bisques pre-sintered at 900°C, was observed. However, in the case of the alumina doped 3Y-TZP-A bisque materials, the modal pore size increased from 0.05 µm for the green compacts to 0.07 µm for the bisques pre-sintered at 700°C, and then in the case of bisques pre-sintered at 900°C it decreased to 0.05 µm. This proves the influence of the Al_2O_3 addition on powder sinterability. Moreover, the pore size distributions in the 3Y-TZP and 3Y-TZP-A compacts pre-sintered at 900°C were comparable to the commercial Cercon material and were significantly lower than in the ICE Zirconia with a modal pore size of 0.2 µm (Fig. 3).

Mechanical strength, hardness and fracture toughness are the most important parameters for the milling of bisque-sintered materials. An increase in these parameters with the temperature of heat treatment was observed for both the studied materials. The corresponding results of the three-point bending test and Vickers indentation, conducted to determine the hardness and K_{Ic}, are shown in Table 1 for the green compacts and bisques. Pre-sintering at 900°C resulted in a bending strength of 38.0±0.8 MPa and 36.5±0.9 MPa for the 3Y-TZP and 3Y-TZP-A compacts, respectively. The higher strength of the 3Y-TZP materials is a consequence of the smaller pore size (Fig. 3). A similar relationship is present in the case of hardness; the highest values of 525±5 MPa and 494±6 MPa were measured for the 3Y-TZP-900 and 3Y-TZP-A-900, respectively. The largest values of K_{Ic} were obtained for the same bisques; they were 0.31±0.02 $MPa{\cdot}m^{0.5}$ and 0.28±0.02 $MPa{\cdot}m^{0.5}$, respectively. For comparison, the hardness and fracture toughness of commercial ICE Zirconia material were 490±5 MPa and 0.50±0.03 $MPa{\cdot}m^{0.5}$, respectively.

TABLE 1. **Bending strength, hardness and fracture toughness of 3Y-TZP and 3Y-TZP-A green compacts and bisques**

TABELA 1. **Wytrzymałość na zginanie, twardość i odporność na kruche pękanie wyprasek i biskwitów tworzyw 3Y-TZP i 3Y-TZP-A**

Material / Temperature, [°C]	Bending strength [MPa]	Hardness [MPa]	K_{Ic} [$MPa{\cdot}m^{0.5}$]	
3Y-TZP	25	7.5 ± 0.3	203 ± 1	0.12 ± 0.01
	500	10.5 ± 0.5	201 ± 2	0.12 ± 0.01
	700	21.5 ± 0.4	325 ± 1	0.15 ± 0.01
	900	38.0 ± 0.8	525 ± 5	0.31 ± 0.02
3Y-TZP-A	25	6.8 ± 0.2	183 ± 1	0.11 ± 0.01
	500	9.8 ± 1.3	210 ± 1	0.11 ± 0.01
	700	19.5 ± 1.4	319 ± 1	0.17 ± 0.01
	900	36.5 ± 0.9	494 ± 6	0.28 ± 0.02

Characteristics of sintered samples

The effects of the Al_2O_3 addition on the densification of the sintered materials are shown in Figures 4 and 5. The TZ-3Y powder with the addition of Al_2O_3 indicated lower starting and ending temperatures of sintering, confirming that the increase of sintering activity results in easier densification of the bisques at temperatures lower than 1400°C, but limits the maximum density obtained at higher temperatures. In the case of the 3Y-TZP-A material, the highest density was possible to achieve even at 1300°C, but these values were lower than for the 3Y-TZP. The highest densities of 99.89±0.5 and 99.44±0.2 were achieved for the 3Y-TZP-25 sintered at 1450°C and 3Y-TZP-A-500 sintered at 1400°C, respectively.

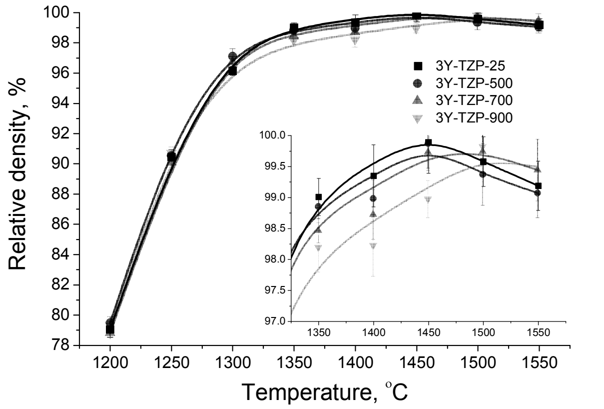


Fig. 4. Relative density of 3Y-TZP sinters as a function of temperature; details of densification data at highest sintering temperatures applied are shown in inset

Rys. 4. Gęstość względna spieków 3Y-TZP w funkcji temperatury; szczegóły wyników zagęszczenia w najwyższych temperaturach spiekania pokazano we wkładce

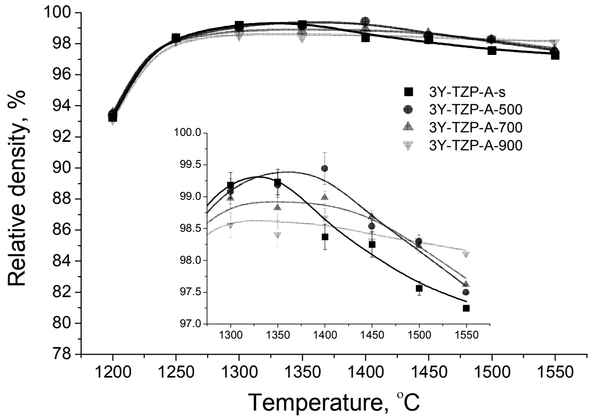


Fig. 5. Relative density of 3Y-TZP-A sinters as a function of temperature; details of densification data at highest sintering temperatures applied are shown in inset

Rys. 5. Gęstość względna spieków 3Y-TZP-A w funkcji temperatury; szczegóły wyników zagęszczenia w najwyższych temperaturach spiekania pokazano we wkładce

The comparison of bisques sintered at 900°C with the commercial materials (Fig. 6.) showed that the course of the densification curve for 3Y-TZP-A-900 is similar to Cercon while 3Y-TZP-900 to ICE Zirconia.

The microstructures of the TZ-3Y materials undoped and doped with 0.2 mass% Al_2O_3 are shown in Figure 7, and compared to the Cercon and ICE Zirconia full-sintered materials. The increase in grain size is observed due to the alumina addition. The 3Y-TZP-900 and 3Y-TZP-A-900 bulk materials show a grain size of

0.50±0.07 μm and 0.55±0.08 μm, respectively. The Cercon and ICE Zirconia microstructures are composed of grains of 0.56±0.07 μm and 0.59±0.09 μm, and some alumina segregation was detected by EDS analysis (Figs. 7c and 7d). This feature was not observed in the case of the 3Y-TZP-A-900 material, which is very similar to the Cercon one microstructurally. The results concerning the impact of the studied alumina addition on the densification and microstructure obtained in this study are consistent with Matsui's reports on the behaviour of 2.9 mol% Y_2O_3-doped ZrO_2 powders with 0÷1 mass% Al_2O_3 during the initial sintering stage [3, 4]. In those reports, the change of the diffusion mechanism from grain-boundary diffusion to volume diffusion by Al_2O_3 was stated and an enhanced sintering mechanism was assigned to the segregated dissolution of Al_2O_3 at the ZrO_2 grain boundaries.

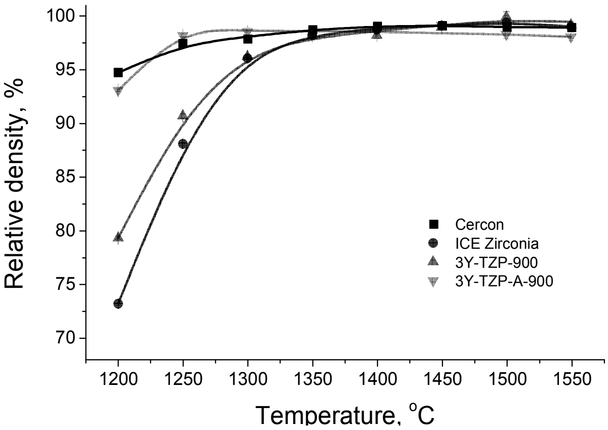
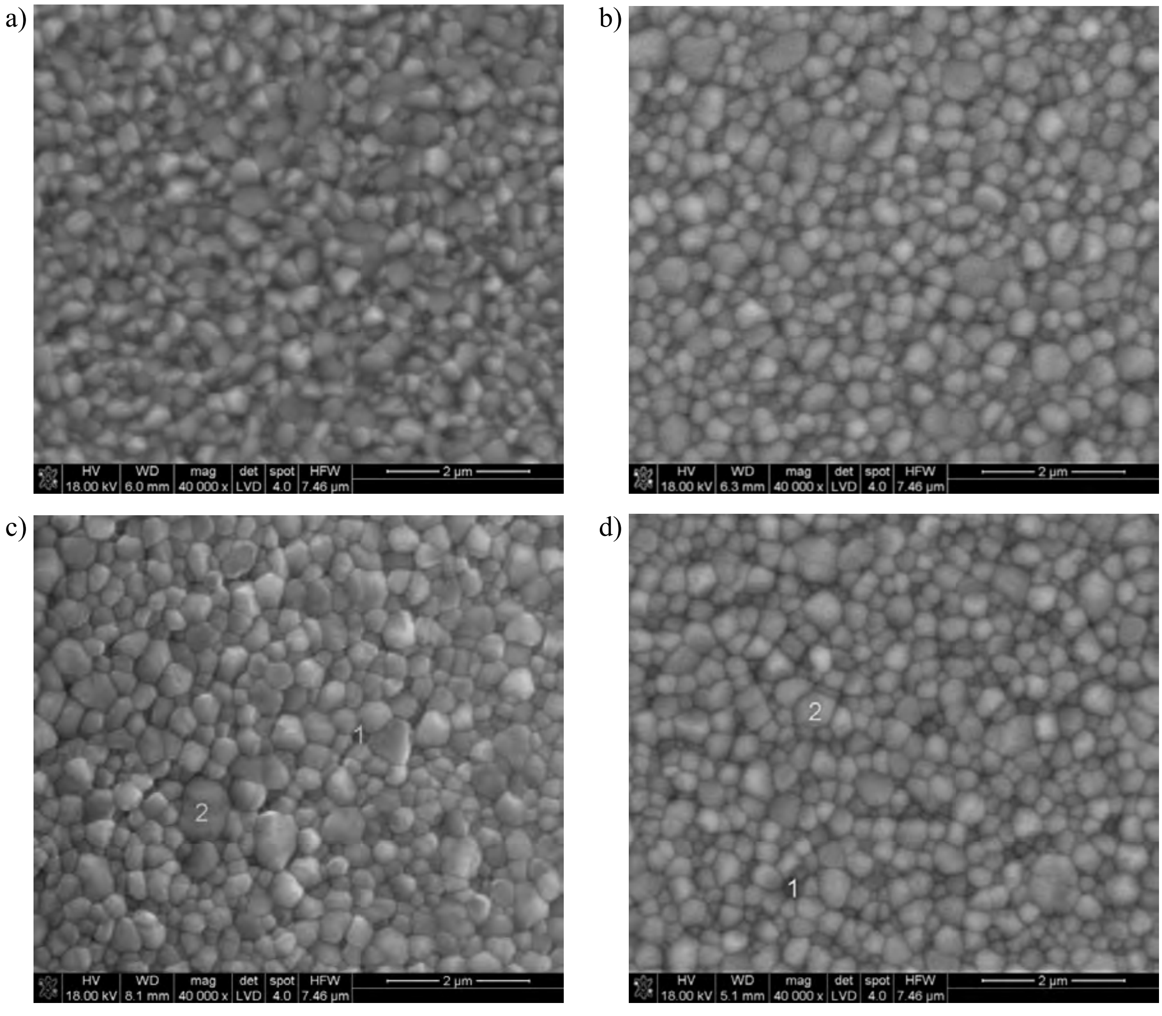


Fig. 6. Comparison of temperature dependencies on density of commercial materials, and bisques pre-sintered at 900°C

Rys. 6. Porównanie gęstości względnej materiałów komercyjnych z biskwitami 3Y-TZP-900 i 3Y-TZP-A-900



Element	Concentration [mass%]			
	O	Al	Y	Zr
Point 1	31.61	8.69	4.97	74.73
Point 2	28.43	0.20	5.27	66.10

Element	Concentration [mass%]			
	O	Al	Y	Zr
Point 1	24.04	3.34	4.32	68.30
Point 2	25.13	0.38	4.95	69.54

Fig. 7. SEM images of bulk materials sintered for 2 h at 1400°C: a) 3Y-TZP-900, b) 3Y-TZP-A-900, c) ICE-Zirconia, d) Cercon

Rys. 7. Obrazy SEM materiałów spieczonych przez 2 h w 1400°C: a) 3Y-TZP-900, b) 3Y-TZP-A-900, c) ICE-Zirconia, d) Cercon

Application research

The application research included the preparation of bisques, and then milling and sintering them to obtain dental crown substructures (Fig. 8). The 3Y-TZP and 3Y-TZP-A bisques were prepared in accordance with the earlier mentioned parameters of pressing and pre-sintering. The milling was performed by using a Zirkograph milling machine according to the manufacturer Zirkonzahn system. The milling machine has been designed to copy with a corresponding size increase to compensate the shrinkage during sintering. More details were presented in [9].

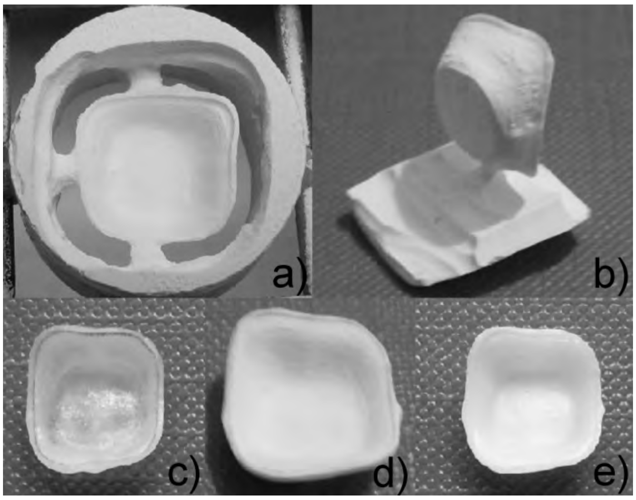


Fig. 8. Dental crown substructure in successive stages of 3Y-TZP-A-900 bisque processing: a) bisque in milling fixture, b) bisque removed from fixture, c) resin model of substructure, d) bisque before sintering, d) substructure after sintering for 2 h at 1400°C; parts in c), d) and e) are presented on same scale for comparison purposes among sizes of resin model and substructures before and after sintering. Substructures made of 3Y-TZP-900 bisque were qualitatively similar to 3Y-TZP-A-900 counterparts. In case of ICE Zirconia, smoother edges of detail both in green and sintered state were observed as sole differences

Rys. 8. Podbudowa pod koronę protetyczną na różnych etapach przetworzenia biskwitu 3Y-TZP-A-900: a) biskwit w uchwycie frezarki, b) biskwit po wyjęciu z uchwytu, c) model podbudowy z żywicy, d) biskwit przed spiekaniem, e) podbudowa cyrkoniowa po spiekaniu w 1400°C przez 2 h; elementy c), d) i e) przedstawiono w tej samej skali dla porównania wymiarów modelu oraz wyfrezowanego elementu przed i po spiekaniu. Podbudowy wykonane z biskwitu 3Y-TZP-900 nie różniły się jakościowo od swoich odpowiedników 3Y-TZP-A-900. W przypadku ICE Zirconia, jako jedyne różnice zaobserwowano bardziej gładkie krawędzie detali, zarówno w stanie surowym, jak i spieczonym

The machining performance of the bisques was qualitatively assessed by visual observations of the sample behaviour when removing the material and the edges of the machined details. The milling finished successfully for the 3Y-TZP and 3Y-TZP-A bisques sintered at 700 and 900°C. Precise milling of the bisques sintered at 500°C was not possible as a result of the low mechanical strength of the samples and their dramatic cracking when removing the material. The Al_2O_3 addition did not cause significant changes in the machining performance of the bisques. Better machining and smoother edges of the details were observed in the case of the ICE Zirconia material, when compared to the 3Y-TZP-900 and 3Y-TZP-A-900. This can be attributed to the higher fracture toughness of the ICE Zirconia comparing to the studied bisques. The Al_2O_3 addition did not cause significant changes in the machining performance of the bisques. The higher fracture toughness of the ICE Zirconia resulted in better machining of the details and smoother edges, when compared to the 3Y-TZP-900 and 3Y-TZP-A-900.

CONCLUSIONS

The effect of the 0.2 mass% nano-Al_2O_3 addition on the properties of the TZ-3Y Tosoh powder and the resultant bisque-sintered and fully-sintered 3Y-TZP materials was studied, and compared to Cercon and ICE Zirconia derived bodies. The findings of this work have confirmed that the Al_2O_3 admixture of 0.2 mass% does produce slight effects on the properties of the green compacts and bisque-sintered bodies, but significant improvements are present with respect to the sinterability of the alumina modified TZ-3Y powder. The alumina addition did not cause noticeable changes in the milling behaviour of the TZ-3Y bisques. They obtain the best properties for the machining of all-ceramic crown substructures at 700 and 900°C. The 0.2 mass% alumina addition to TZ-3Y leads to a material microstructure analogous to the Cercon derived one.

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