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DENSE ALUMINA CERAMICS OBTAINED BY GELCASTING AND COLD ISOSTATIC PRESSING WITH THE USE OF 2-CARBOXYETHYL ACRYLATE

In ceramic forming techniques high particles packing can provide better properties of the final ceramic products. The high quality of the material coupled with the shape complexity of the ceramic product is still challenging. The aim of this work was the optimization and preparation of the ceramic samples based on two alumina powders of different particle size (AA05: 0.5 μm and TM-DAR: 0.15 μm). Firstly, ceramic suspensions of 50vol.% solid loading and the volumetric ratio of AA05 to TM-DAR 1:1, 2:1, 3:1, 4:1, respectively have been prepared. The 2-carboxyethyl acrylate was applied as the new monomer limiting the negative effect of oxygen inhibition. Additionally, the cold isostatic pressing (CIP) was used in order to increase relative density of green bodies. The results of presented research have shown that samples with the ratio of AA05 to TM-DAR 2:1 were characterized by the highest green density (62%). Moreover, CIP process proved to be effective and increased the density of green bodies from 62% to 67%. The pore size distribution of the green bodies has been measured. Samples were sintered at different conditions (1400°C, 1450°C and 1500°C for 1h and 1300°C, 1400°C, 1450°C and 1500°C for 5h).

Keywords: gelcasting, cold isostatic pressing, alumina, mercury porosimetry

1. Introduction

In the last decades advanced ceramics have replaced metals in many applications and fields. Nevertheless, the requirements of both the properties and geometries of ceramic products are continuously increasing with technology development. The high quality of the material together with the shape complexity of the ceramic product is still challenging. A method which allows to obtain such ceramic materials is gelcasting [1-5]. Gelcasting is a process in which an organic monomer is added to a water-based ceramic suspension. The monomer is polymerized and thus the suspension is consolidated. Then the cast part is removed from the moulds, dried and sintered. The main challenge in the gelation process is to prepare homogeneous, highly concentrated and stable ceramic suspension which leads to high particle packing in the green state and full densification upon sintering [6-10]. Gelcasting allows to fabricate both dense and porous materials [11-15]. Gelation process has been also employed in the preparation of thin films, as a combination with tape casting technique [16] and also for the fabrication of multilayer and textured ceramics as a combination with magnetic field alignment [17-21].

The packing of particles has always been of interest to ceramists because it leads to high and uniform density of sintered bodies. Such increase in densities results from the reduc-

tion of diffusion distances and attainment of a larger number of particle-to-particle contacts. When the resulting fraction of pores in the sintered sample is small, the mechanical properties of the material are improved due to the presence of small amount of flaws.

In gelcasting high solid loading in ceramic suspension can provide high density of the sintered ceramic body. Generally, in colloidal processing the solid loading can be controlled at a desired level. The main challenge is therefore the preparation of ceramic slurry of low viscosity and as high as possible concentration of ceramic powder. Slurries having low viscosities can easily fill the moulds. Higher solid loading leads to the reduction in shrinkage of ceramic green bodies during drying and sintering. Lower shrinkage will decrease the problems with cracking and warping of the ceramic components. In all, high solid loading is desired in gelcasting. The higher densification can be obtained thanks to the combination of ceramic powders of different particle size.

The aim of the research was the preparation of the ceramic slurries based on two alumina powders of different particle size (0.5 μm and 0.15 μm) and different ratio of powder concentrations. The cold isostatic pressing (CIP) was used in order to increase the relative density of green bodies obtained by gelcasting. The main advantage of CIP is that the pressure used to compact the powder is applied equally in all directions, it leads

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to uniform density and shrinkage. Isostatic actions (hydrostatic pressure) can also form complicated shapes which cannot be compressed with an uniaxial press or machine press [22-25]. The combination of gelcasting and CIP processes may allow to obtain dense green parts of high mechanical strength.

2. Experimental procedure

2.1. Materials

Ceramic powders used in the research were a high purity (>99.99%) α -Al₂O₃ TM-DAR (*Taimei Chemicals, Japan*) of the average particle size $D_{50} = 0.15 \mu\text{m}$ and AA05 (*Sumitomo Chemical, Japan*) of the average particle size $D_{50} = 0.5 \mu\text{m}$. The density of alumina powders was 3.96 g/cm^3 .

The smaller particles of the alumina correspond to the value of the theoretical pore diameter which was calculated from the relationship: $d_p = 0.315D_g$, where: d_p is the pore diameter (μm) and D_g is the grain diameter (μm) [26]. When comparing the calculated diameter of pores with the particle size of the larger alumina powder (AA05) the values are similar to the particle size of smaller one.

In former works the TM-DAR alumina powder was used in obtaining ceramic matrix composites [27,28] textured alumina-related compounds by colloidal processing [29] and in electrophoretic deposition of alumina suspensions in high magnetic field [30,31]. Commercially available 2-carboxyethyl acrylate (*Sigma-Aldrich*) was applied in the gelcasting of alumina as a new monomer which prevents the oxygen inhibition and allows to obtain defect-free green bodies. 2-carboxyethyl acrylate (CEA) was chosen because of its molecular structure. Authors expected that the presence of the carboxyl group in CEA molecule could have positive influence on the rheological behavior of ceramic suspensions and as a consequence the high quality of samples. High viscosity at low shear rates of CEA-based slurries prevents the diffusion of oxygen into the interior part of the ceramic green bodies in contrary to the slurries containing 2-hydroxyethyl acrylate which is commonly used in gelcasting. The limited diffusion of the oxygen prevents the oxygen inhibition during gelstion process. The phenomenon of the oxygen inhibition and the influence of 2-carboxyethyl acrylate on the rheological properties of Al₂O₃ slurries has been wider described elsewhere [8]. SYNTRAN 8250 was used as dispersing agent, L-ascorbic acid (*Sigma-Aldrich*) was an activator of the polymerization and ammonium persulphate (*Sigma-Aldrich*) was the initiator of the polymerization. According to the information given by the supplier (*Interpolymer Company*) SYNTRAN 8250 is a medium molecular weight aqueous solution based on polyacrylic acid homopolymer and was used in the form of 40% aqueous solution. The initiator ammonium persulphate was used in the form of 10% aqueous solution.

2.2. Preparation of slurries and samples

The first step was the preparation of the ceramic slurries. Dispersant was first dissolved in distilled water. Then, the appropriate amounts of ceramic powders and activator of polymerization were added and after prior homogenization the monomer was dropped into the slurry. The concentrations of all components are listed in Table 1. The composition of the ceramic suspensions was first chosen by set of trials. All the operations were conducted at room temperature. The suspensions were mixed by magnetic stirrer for 60 min at a mixing speed of 500 rpm. After mixing, the slurries were subjected to deaeration using THINKY ARE-310 Centrifugal Mixing and Degassing machine to remove air bubbles that may cause the formation of large pores in ceramic parts before and after sintering. In the second step, the initiator of the polymerization was added to the ceramic slurry. Just after mixing the slurries with the initiator, the slurries were casted into the PVC molds of diameter 20 mm and high 10 mm, gelation occurred at room temperature.

After gelation, the products were removed from the moulds and dried at room temperature for 48h. Half of the samples, in which the ratio of AA05 to TM-DAR was 2:1, were densified by Cold Isostatic Press (*Nikkiso, Japan*) at a maximum water pressure of 392 MPa for 10 and 20 minutes. The next step was the burnout of organic additives and sintering of ceramic samples. Samples were sintered at different conditions: from 1300°C to 1500°C with 1h and 5h dwell time.

TABLE 1

Compositions of the slurries used in the research

Slurry No.	1	2	3	4
Al ₂ O ₃ AA05 ^a	25.0	16.7	37.5	40.0
Al ₂ O ₃ TM-DAR ^a	25.0	33.3	12.5	10.0
water ^a	50.0	50.0	50.0	50.0
SYNTRAN 8250 ^b	1.4	1.4	1.4	1.4
2-carboxyethyl acrylate ^b	4.0	4.0	4.0	4.0
L-ascorbic acid ^c	1.0	1.0	1.0	1.0
ammonium persulfate ^c	0.8	0.8	0.8	0.8

^a vol%;^b wt% with respect to the amount of the ceramic powder;^c wt% with respect to the amount of the monomer.

2.3. Characterization techniques

The rheological properties of the slurries were measured using a Brookfield RV DV-II+ Pro Viscometer (Brookfield Engineering Laboratories Inc., USA). The flow curve of suspensions before the addition of the initiator was examined. In this case, the shear stress was measured as a function of the shear rate. The shear rate increased from 0.1 s^{-1} to 70 s^{-1} and back to 0.1 s^{-1} . All rheological measurements were carried out with the spindle S31.

The viscoelastic properties of the selected ceramic slurry was examined using a rotational rheometer KinexusPro (*Malvern*

Instruments, UK) with a plate-plate geometry. The gap between the plates was 0.5 mm. The linear viscoelastic region (LVER) was determined at a frequency of 1 Hz and then the elastic (G') and viscous (G'') modules at the strain corresponding to the LVER region were plotted as a function of the frequency.

The densities of the green and sintered specimens were measured by the Archimedes method in kerosene and water, respectively.

Porosity and pore volume were measured by using mercury porosimetry (*PoreMaster 60 porosimeter, USA*).

The tensile strength of green bodies was determined by the "Brazilian test". It was calculated as $\sigma_t = (2P/\pi TD)$, where P is the force causing the damage of the sample, T and D are the thickness and the diameter of the disc-shape sample. The diameter of the samples was 16.5 ± 0.1 mm and their thickness was 8.5 ± 0.2 mm.

Vickers indentations have been done on Digital Vickers Hardness Tester HVS-30T (*Huatac Group Corporation, China*) applying the load of 196 N. The photographs of the indentations and the measurements of the diagonals have been done on light microscope (*Nikon Eclipse LV150N, Japan*). Microscopic observations of green and sintered bodies were made with the use of scanning electron microscope, SEM (*Zeiss Ultra Plus, Germany*).

3. Results and discussion

3.1. Rheological behaviour of ceramic slurries

Fig. 1 shows the flow curves of the alumina slurries in which the ratio of Al_2O_3 AA05 to TM-DAR was 1:1, 2:1, 3:1 and 4:1. The measurements revealed that the slurries exhibited slight thixotropic behavior (hysteresis loop is visible) which increases with the increase of the amount of AA05 alumina. This indicates that the internal structure of slurries is destroyed during shear and rebuilds. The yield value of all slurries has been estimated as 0 Pa.

The values of the viscosity at a shear rate 10 s^{-1} were 0.16 Pas, 0.16 Pas, 0.23 Pas and 0.30 Pas for slurries in which

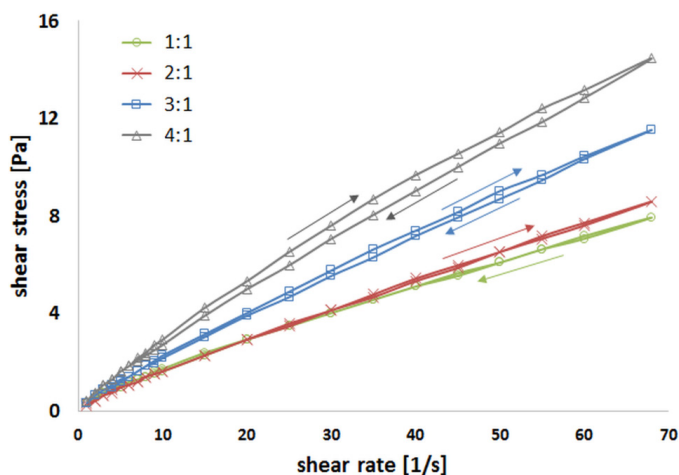


Fig. 1. Flow curves of the alumina slurries in which the ratio of Al_2O_3 AA05 to TM-DAR was 1:1, 2:1, 3:1 and 4:1

the ratio of AA05 to TM-DAR was 1:1, 2:1, 3:1 and 4:1, respectively. These values are relatively comparable to literature data, where the viscosity at a shear rate 10 s^{-1} was 0.8 Pas for slurry based only on alumina TM-DAR in which the concentration of solid phase was 50vol% [19]. The rheological characteristics of slurries in which the ratio of AA05 to TM-DAR was 1:1 and 2:1 was very similar, however lower viscosity values at shear rates below 10 s^{-1} were observed for the slurry of the AA05 to TM-DAR ratio 2:1. Taking into consideration that casting of slurries is performed at low shear rates, the slurry of the AA05 to TM-DAR ratio 2:1 has been chosen to further experiments.

Fig. 2 shows oscillation tests for alumina slurry with the ratio of AA05 to TM-DAR 2:1. At first the LVER region was determined (Fig. 2a). The LVER was assigned at 0.8%. Fig. 2b shows viscoelastic properties of the alumina slurry as a function of frequency. The viscous modulus G'' slightly predominates over elastic modulus G' within the whole range of frequency. It means that in this slurry prevail viscous properties. The values of the elastic modulus G' and viscous modulus G'' at a frequency of 5 Hz are very similar and equaled 6.3 Pa and 7.9 Pa, respectively. Therefore the elastic and viscous modulus are in equilibrium in this slurry.

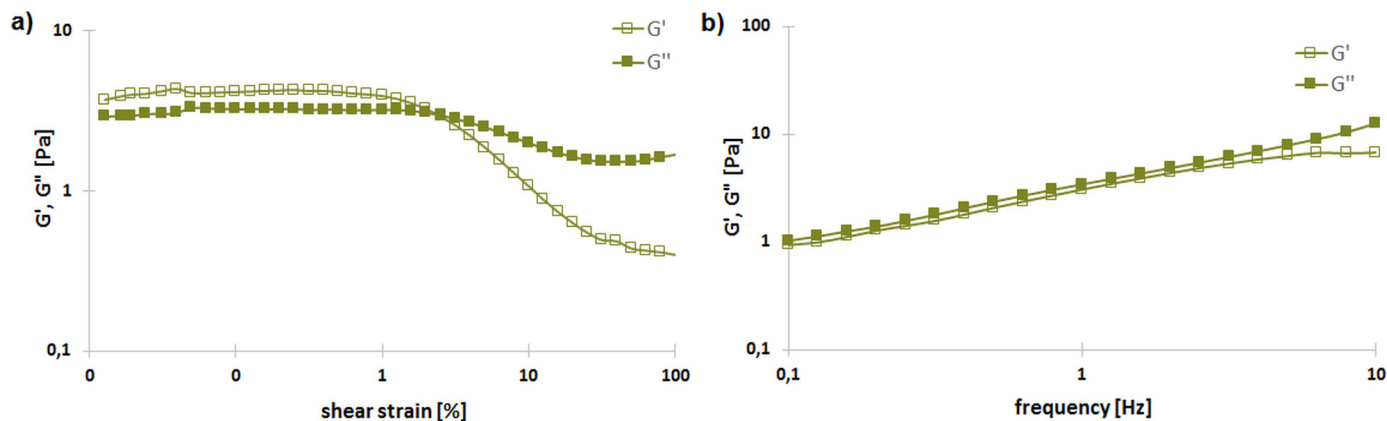


Fig. 2. Oscillation tests a) amplitude sweep analysis b) frequency sweep analysis of the slurry with the ratio of AA05 to TM-DAR 2:1

3.2. Characterization of the green bodies

All samples obtained by gelcasting were characterized by smooth surface, without any cracks or exfoliation. Fig. 3 lists the values of the relative density of the green bodies obtained by gelcasting in which the volumetric ratio of AA05 to TM-DAR equaled 1:1, 2:1, 3:1, 4:1, respectively. The measurements have been done for 15 samples of each composition. The highest density exhibited samples with the ratio of AA05 to TM-DAR 2:1 (62%). This value is higher comparing to the literature review where the value 58% for samples based on alumina TM-DAR was reported [17]. The lowest density was observed for samples in which the ratio of AA05 to TM-DAR was 3:1 and 4:1 (59%). Therefore, for the samples (2:1) the CIP was applied to additionally increase the relative density of the specimens. After application of CIP for 10 and 20 min, the relative density increased by 5% for both samples after CIP (from 62% to 67%) that is for each dwell time. It means that there is no reason to apply CIP more than 10 min.

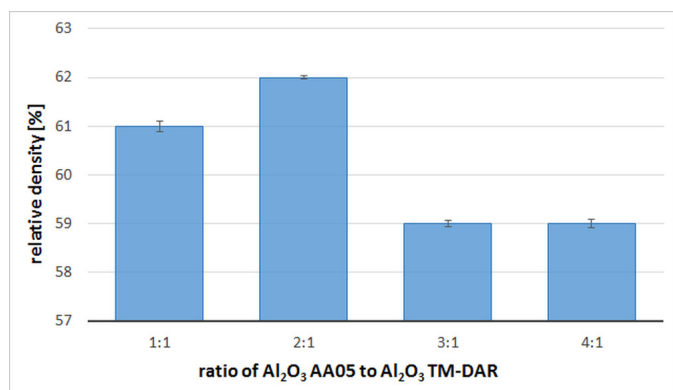


Fig. 3. The relative density of green bodies with different ratio of AA05 to TM-DAR

The pore size distribution after measurements on mercury porosimetry of the green bodies is shown in Fig. 4. All the samples exhibited a bimodal distribution of pores. In sample with the ratio of AA05 to TM-DAR 3:1 there exist a large number of pores of diameter 0.65 μm and lower amount of pores of diameter 0.1 μm . This sample is characterized by the highest porosity (40.61%) and the largest pores (TABLE 2) what corresponds to the lower relative density of these samples (Fig. 3). Similar porosity exhibits sample with the ratio of AA05 to TM-DAR 4:1 (39.45%), there is a large number of pores of diameter 0.2 μm and lower amount of pores of diameter 0.075 μm . Samples with the ratio of AA05 to TM-DAR 1:1 and 2:1 are characterized by lower porosity (37.45% and 35.13%, respectively). In sample with the ratio of AA05 to TM-DAR 1:1 there exist a large number of pores of diameter 0.2 μm and similar amount of pores of diameter 0.09 μm . In sample with the ratio of AA05 to TM-DAR 2:1 pores of diameter both 0.3 μm and 0.1 μm are visible. The lowest porosity has sample with the ratio of AA05 to TM-DAR 2:1 after CIP process (30.10%), what corresponds to the high value of relative density of this sample (Fig. 3). In the sample

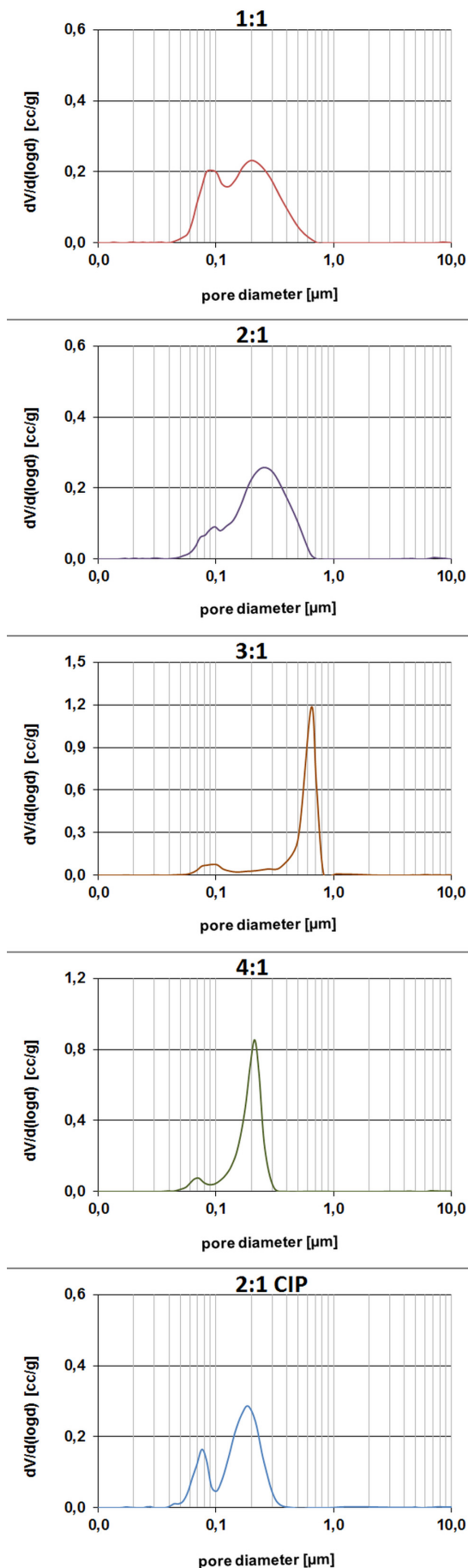


Fig. 4. Pore size distribution of green bodies with the ratio of AA05 to TM-DAR 1:1, 2:1, 3:1, 4:1 and 2:1 after CIP

there exist a large number of pores of diameter 0.18 μm and lower amount of pores of diameter 0.08 μm . Comparing to the sample with ratio of AA05 to TM-DAR 2:1 without applying CIP, the porosity decreased about 5%. Both the pores diameter (from 0.3 μm to 0.13 μm) as well as number of larger pores have decreased.

TABLE 2

Open porosity and total pores volume of the prepared samples in a green state

Al_2O_3 AA05 : TM-DAR ratio	P_o [%]	V_{por} [cm^3/g]
1:1	37.45	0.1607
2:1	35.13	0.1495
3:1	39.45	0.1782
4:1	40.61	0.1780
2:1 CIP	30.10	0.1176

The tensile strength was measured by “Brazilian test” for samples with and without CIP in which the ratio of AA05 to TM-DAR was 2:1. Samples after CIP process were characterized by almost 25% higher tensile strength (3.15MPa) than samples without CIP (2.35MPa). It is due to the higher densification of the samples after CIP process. Additionally, it confirms that 2-carboxyethyl acrylate can form strong polymeric network without any external cross-linking agents. According to the literature data concerning dense alumina bodies prepared by gelcasting process, the tensile strength of CEA-based samples is higher than in case of other water-based monomers such as 3-O-acrylic-d-glucose (1.0 MPa) [32], glycerol monoacrylate (1.23 MPa) [33], methylolurea (0.35 MPa) [34].

3.3. Characterization of sintered bodies

Samples in which the ratio of Al_2O_3 AA05 to TM-DAR was 2:1 were sintered at different conditions: at 1400°C, 1450°C and 1500°C with dwell time of 1h and at 1300°C, 1400°C, 1450°C and 1500°C with dwell time of 5h. Table 3 lists the values of the apparent density of the sintered bodies.

TABLE 3

The apparent density of sintered parts

Temperature [°C]	Dwell time [h]	Apparent density [g/cm^3]
1500	1	3.68 \pm 0.01
1450		3.41 \pm 0.02
1400		3.09 \pm 0.02
1500	5	3.88 \pm 0.00
1450		3.68 \pm 0.02
1400		3.52 \pm 0.02
1300		3.05 \pm 0.14

The highest apparent density exhibited samples sintered at 1500°C with 5h dwell time. Samples sintered at lower tempera-

tures and shorter sintering time have been not fully densified.

Ceramic samples with applied CIP process were sintered at 1300°C, 1400°C, 1450°C and 1500°C for 5h. Table 4 lists the values of the relative density of the sintered bodies.

TABLE 4

The relative densities of sintered bodies without and with CIP process

Temperature [°C]	Dwell time [h]	Relative density without CIP [%]	Relative density with CIP [%]	Linear shrinkage without CIP [%]	linear shrinkage with CIP [%]
1500	5h	98	~100	16.3 \pm 0.31	14.5 \pm 0.28
1450		93	99	15.6 \pm 0.47	13.8 \pm 0.45
1400		89	97	14.3 \pm 0.53	12.8 \pm 0.44
1300		77	84	8.9 \pm 1.12	7.2 \pm 0.71

The highest relative density exhibited samples sintered at 1500°C after CIP process. Samples sintered at 1450°C were also almost fully densified. Samples sintered at 1300°C even with 5 h dwell time have not been well densified. In all cases the application of CIP process resulted in the increase of the relative density of samples, as expected. It seems that the sintering temperature equaling 1450°C is high enough to densify the samples in which CIP was performed. Linear shrinkage for samples pressed by CIP is smaller comparing to the shrinkage of the samples without CIP what is related to the density of green bodies. It is due to

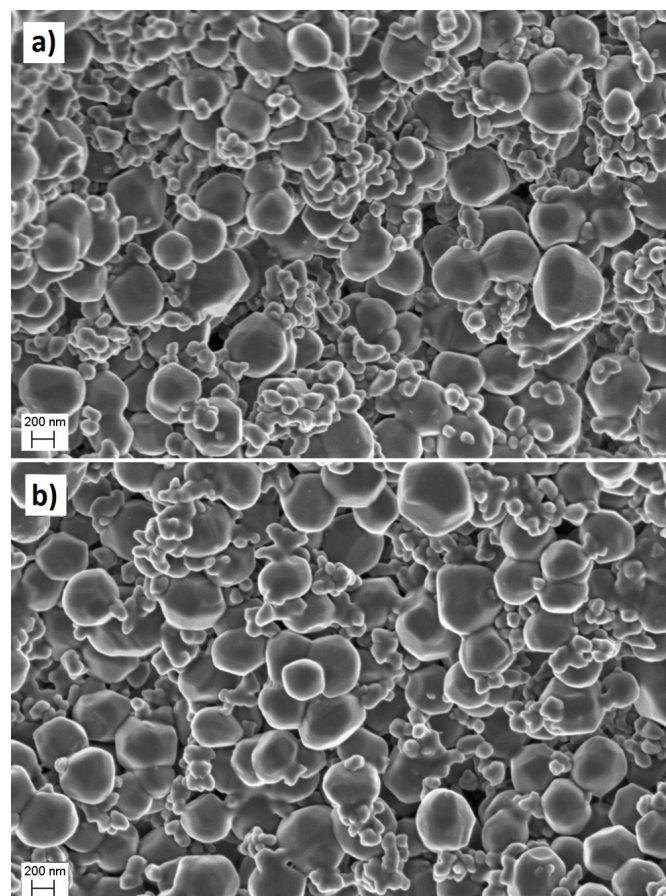


Fig. 5. SEM images of green bodies without CIP (a) and with CIP (b)

the higher densification of the samples after CIP process. The measurements have been done for 15 samples with and without CIP for each sintering temperature.

The microstructure of green (Fig. 5) and sintered bodies (Fig. 6) have been then examined.

Fig. 5 presents the SEM images of green bodies without CIP (a) and with CIP (b) in which the ratio of Al_2O_3 of AA05 to TM-DAR was 2:1. The micrographs indicate on the high homogeneity of the specimens. The microstructure of both samples looks similar, there are clearly visible larger particles of Al_2O_3 AA05 and smaller particles of Al_2O_3 TM-DAR.

Fig. 6 presents the SEM images of samples sintered at 1500°C with 1 h dwell time (Fig. 6a,b) and 5 h dwell time (Fig. 6c,d) without and with prior CIP (Fig. 6e,f).

In all SEM images showing the polished and thermally etched surface of samples there are visible wholes which appeared during polishing process and are the result of pulling grains out of the material. It is related to the using of high purity alumina powders (99.99%). Lack of impurities in the powders provided that sintering occurred without liquid phase which usually strengthens grains boundaries. The similar wrenches of small grains are visible in SEM images of fractures. All samples

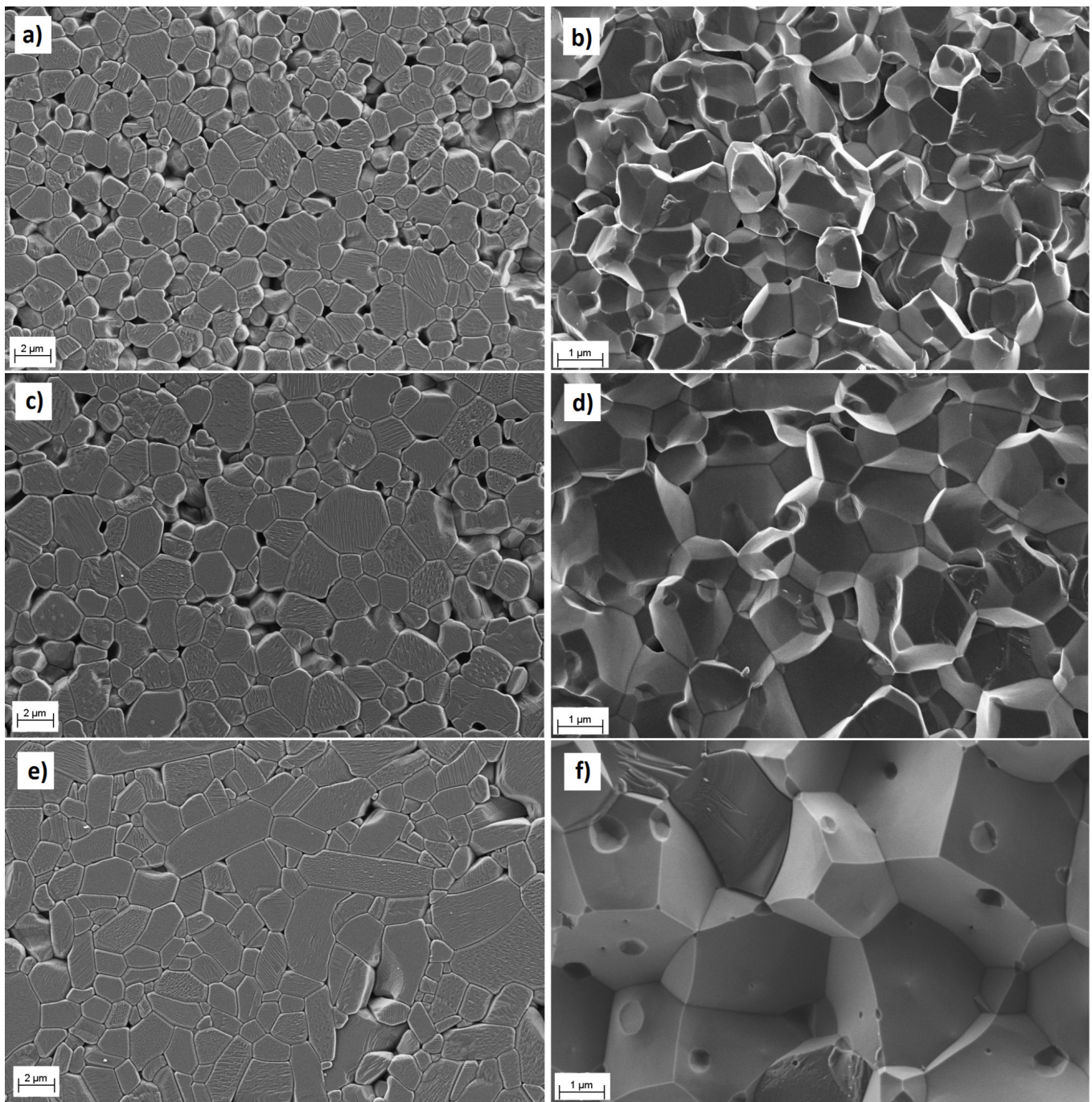


Fig. 6. SEM images of sintered bodies: a),b) without CIP, sintered at 1500°C with dwell time 1h, surface and fracture respectively; c),d) without CIP, sintered at 1500°C with dwell time 5h, surface and fracture respectively; e),f) with CIP, sintered at 1500°C with dwell time 5h, surface and fracture respectively

have been well densified, however, in case of sample sintered with 1 h dwell time and without CIP process some porosity is observed (Fig. 6b). The increase of dwell time from 1 h to 5 h increased the relative density but also the grain growth, as expected. Much larger grain growth was observed for sample being additionally isostatically pressed (Fig. 6e,f). This effect can be caused by the difference in particles size which additionally have been highly packed. Thus during sintering larger grains can easily consume smaller ones. The Vickers hardness of Al_2O_3 without CIP equaled 16.2 GPa, while of the Al_2O_3 with CIP equaled 18.3 GPa. The values are relatively comparable to the literature data described by Auerkari [35], Casellas [36] and Pedzich [37], where the Vickers hardness of pure alumina equaled 17-17.5 GPa.

4. Conclusions

Green and sintered bodies made of two alumina powders of different particles size were successfully produced by a gelcasting process. Commercially available monomer 2-carboxyethyl acrylate allowed to obtain defect-free samples. The ceramic slurry in which the ratio of Al_2O_3 of AA05 to TM-DAR was 2:1 exhibited shear thinning and slight thixotropic behavior. The results of research have shown that samples with the ratio of Al_2O_3 AA05 to TM-DAR 2:1 were characterized by the highest green density (62%). Measurements of porosity and density have confirmed that CIP process proved to be effective and increased the density of green bodies from 62% to 67%. The Vickers hardness of Al_2O_3 samples without previous CIP equaled 16.2 GPa, the hardness of the Al_2O_3 with applied CIP equaled 18.3 GPa. Sintering at 1500°C for 5 h turned out to be effective and provided fully densification of the samples. Much larger grain growth was observed for sample being additionally isostatically pressed than for samples without CIP process.

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