The Potential of Al_2O_3 – ZrO_2 -Based Composites, Formed via CSC Method, in Linear Infrastructure Applications Based on Their Mechanical, Thermal and Environmental performance

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Ceramic-ceramic composites have been prepared using the centrifugal slip casting method (CSC). The method has so far been mainly utilized in making ceramic-metal composites. Al_2O_3 – ZrO_2 composites have been obtained with different shares of ZrO_2 , *i.e.*, 15, 50 and 75 vol pct, respectively. Prior to sintering the composite samples, the rheological properties of the casting slips were investigated and thermogravimetric tests were performed. Upon sintering, all three series of the ceramic composites showed a density close to 100 pct and no microcracks or delamination. Phase, microstructural and mechanical investigations were carried out to determine what effect the share of ceramics has on the composites produced. An increase in the share of ZrO_2 in the Al_2O_3 – ZrO_2 composite leads to a reduction in the growth of Al_2O_3 grains and a decrease in the average hardness. Cross-sectional hardness testing of the composites showed no evident gradient in any of the samples. Life cycle analysis (LCA) results indicate that the further optimization of the composite formation process, including up-scaling, allows to obtain Al_2O_3 – ZrO_2 -based pipes possessing more favorable environmental characteristics compared with materials conventionally used for linear infrastructure, which is important in light of the global transformation toward sustainable construction and the circular economy.

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I. INTRODUCTION

IN our age of sustainable development, which involves teaming economic and industrial activity with the preservation of natural balance, great emphasis is placed on the acquisition of new materials or on the improvement of those already available. Moreover, the concept of sustainability in materials engineering fits well with current global changes focused on reducing energy consumption and the greenhouse gas emissions associated with the production of new materials. Consequently, in recent years, intensive work has been carried out on the development of technological concepts enabling the formulation of materials that meet well-defined properties. One example of such materials is ceramic-ceramic composites. Such materials include ZTA (zirconia-toughened alumina),^[1] *i.e.*, composites where Al₂O₃ is the matrix and ZrO₂ is the dispersed phase, or ATZ (alumina-toughened zirconia),^[2] *i.e.*, materials where ZrO₂ is the matrix and Al₂O₃ is the dispersed phase. These composites, compared to classical ceramic materials, are characterized by high abrasion resistance, low specific gravity, very good corrosion resistance in both acids and alkalis, and an elevated hardness.^[3–5] Application of the above-mentioned composites may result in longer service life and, thus, economic benefits. Therefore, developing such materials

fits well into the current trend of sustainable development.

The present article presents a study on the application of a centrifugal slip casting technology developed for obtaining pipes containing different Al₂O₃ to ZrO₂ ratios. So far, the CSC method has been mainly used to fabricate pipes from the ceramic-metal system.^[6,7] In this study, the method will be adapted to the fabrication of Al₂O₃ and ZrO₂-based shapes. Al₂O₃ and ZrO₂-based materials play a key role in high-temperature applications.^[8–10] The CSC method proposed in this work enables the fabrication of shapes that could be used as thermocouple protection tubes. The high melting points of 2327 K for Al₂O₃^[11] and 2988 K for ZrO₂^[12] make it possible for materials made from these powders to operate at high temperatures. In addition, manufacturing products using the CSC method helps in reducing process-related costs, which ultimately translates into lower costs associated with the operation of such elements. The proposed method of tube formation is associated with a reduction in energy consumption, water consumption and greenhouse gas emissions, as well as the amount of waste generated, compared to the conventional centrifugal casting method, which is mainly used to produce pipes from liquid metal.

The aim of this study was to produce and characterize selected properties of Al₂O₃ and ZrO₂-based

Table I. Parameters of the Starting Powders Given by the Manufacturers

	Manufacturer	Average Size μm	Particle Size μm	Granule Size μm	Crystallite Size Å	Specific Surface Area m ² /g	Density g/cm ³
Al ₂ O ₃ (TM_DAR)	Taimei Chemicals Co.	0.05 to 0.2	—	—	—	14.5	3.95
ZrO ₂ (TZ-3YS-E)	Tosoh Corporation	0.09	60	60	380	7 ± 2	6.05

composites. Three types of composites differing in the volume content of ZrO₂ were characterized in this work. Samples containing 15, 50 and 75 vol pct of ZrO₂, respectively, were fabricated. The first stage of the study focused on determining the rheological properties of the ceramic casting slips. Afterwards, thermal analysis was performed to determine the phenomena occurring in relation to temperature at the established temperature process. In the next step, the sintered shapes were characterized in terms of phase composition, microstructure, hardness and resistance to brittle fracture. The influence of ZrO₂ content on the growth of Al₂O₃ grains and their shape was determined based on stereological analysis. In the final stage, life cycle analysis (LCA) was carried out to assess environmental impacts related to the acquisition of the raw materials required for the formation of Al₂O₃ and ZrO₂-based composites and the operations performed during the manufacturing process.

The article presents the results of interdisciplinary research combining the fields of materials engineering, colloid chemistry and environmental engineering. The research presented in this article concerns the formation and characteristics of a finished composite part in the form of a tube. The described relations between the structure and functional properties of the produced Al₂O₃ and ZrO₂-based composites make it possible to design a finished product that fulfills specific functions for applications in many priority developing areas of the world economy. The obtained research results make a significant contribution to the development of fundamental knowledge in the field of ceramic-ceramic composites.

II. EXPERIMENTAL PROCEDURE

A. Materials

The present study utilized α-Al₂O₃ powder by TM_DAR and commercially available ZrO₂ powder (TZ-3YS-E). The Al₂O₃ powder was manufactured by Taimei Chemicals Co., Ltd. (Japan) while the ZrO₂ powder by Tosoh Corp. (Japan). These powders have a high purity of ≥ 99.99 pct. The zirconium oxide powder used in this study was stabilized with 3 mol pct of Y₂O₃. Table I contains a summary of the powders' parameters as given by the manufacturers.

The morphology of the ceramic powders used in the experiment is shown in Figure 1. Based on SEM observations, it was observed that the actual size of the powders was close to the manufacturer's stated size.

It was found that the powders used tend to form agglomerates. The observations also confirmed that the zirconium oxide powder comes in the form of granules consisting of nano-particles. Based on the observations, it can also be stated that the shapes of Al₂O₃ and ZrO₂ particles are slightly heterogeneous.

The specific surface area of the powders used in the experiment was determined by the nitrogen sorption method using an ASAP 2020 device at liquid nitrogen temperature. The obtained multipoint measurements were approximated by the Brunauer–Emmett–Teller (BET) adsorption isotherm, which was used to determine the total specific surface area (SBET [m²/g]). The measurement results show that Al₂O₃ powder has a specific surface area of 11.3 [m²/g], while ZrO₂ has a specific surface area of 6.48 [m²/g]. The determined values of the specific surface area are close to those given by the manufacturer.

The choice of the above powders was based on the fact that they constitute primary structural ceramic materials. Powders such as Al₂O₃ and ZrO₂ are widely used in the aerospace, automotive and petrochemical industries. These materials are used as components of process line equipment, machine elements or cutting tools. What is more, the good biocompatibility of both powders enables their use as elements of endoprostheses, e.g., hip joints. The combination of ZrO₂ with Al₂O₃ makes it possible to obtain ZTA (zirconia-toughened alumina)^[13] or ATZ (alumina-toughened zirconia).^[14] The literature shows that, at present, a wide range of research is being carried out to develop effective technologies for the preparation of ZTA and ATZ composites.^[15–17] The present study is also part of this trend. Additionally, by using the same powders as those utilized by many researchers around the world, it will be possible to make easy references to global research results.

Other substances used in this work included liquefying agents in the form of diammonium hydrogen citrate (DAC; POCH, analytical grade) and citric acid (CA; Sigma-Aldrich, anhydrous, ≥ 99.5 pct). A 10 pct solution of polyvinyl alcohol (PVA; POCH, analytical grade) was used as a binder to obtain the samples. The type, as well as the amounts, of liquefiers and binder added was determined experimentally based on previous experiments.^[18–20] Distilled water was used to prepare the suspensions.

B. Preparation of Slurries

The following slurries were prepared: 15 vol pct ZrO₂ + 75 vol pct Al₂O₃, 50 vol pct ZrO₂ + 50 vol pct Al₂O₃

Fig. 1—The morphology of the starting powders: (a) Al_2O_3 , (b) ZrO_2 .

and 75 vol pct ZrO_2 + 15 vol pct Al_2O_3 . All tested suspensions contained 50 pct solid phase in the casting slip. In addition, the slips also contained distilled water, binder and liquefiers.

Fluxing agents and the binder were added to the water to prepare the slurry. Upon dissolving and mixing the additives, Al_2O_3 and ZrO_2 powders were added to the mixture. The mixture thus prepared was stirred in a Retsch PM400 planetary mill at 300 rpm for 1 h. After the mixing process, the slurry was degassed in a THINKY ARE250 device. The degassing process was carried out at 2200 rpm for 11 minutes.

The casting slips prepared in this way were used to form composites and sourced for rheological studies.

C. Determination of the Casting Slips' Rheological Properties

Rheological properties were examined using a Kinexus Pro rheometer by Malver Instruments (UK). The tests were carried out in a plate-to-plate configuration. The lower plate had a diameter of 65 mm, the upper plate 20 mm, while the gap between the plates was 0.5 mm wide. Measurements were carried out at 22 °C and the temperature was stabilized by a Peltier system. All the tested casting slips were characterized by a solid phase concentration of 50 vol pct.

The experiment was designed to determine the nature of the casting slips being tested by analyzing changes in viscosity and shear stress as a function of shear rate. The

experiments were performed for increasing and then decreasing values of shear rate in the range between 0.1 and 100 s^{-1} and between 100 and 0.1 s^{-1} . The choice of such a shear rate range made it possible to observe the thixotropic effect and make references to previous work carried out by the team.

D. Thermogravimetric Tests

To investigate the phenomena occurring in the formed ceramic shapes in relation to temperature at the established temperature process, a thermogravimetric analysis was performed. The scope of the tests included measurement of mass loss and thermal effects as a function of temperature using a Netzsch TG 449 F1 Jupiter thermogravimetric analyzer. The measurements were carried out in alundum crucibles. The samples for analysis were constituted shapes that had been crushed in an agate mortar. The analytical samples weighed 30 mg each. Such small amounts ensured that good signals were obtained. The analysis was carried out in a flow of synthetic air (80/20 mixture of nitrogen and oxygen). The measurement was carried out in a temperature range between 24 °C and 1400 °C with a heating rate of 10 °C/min. The heating rate was much higher than the heating rate during the sintering process because the signals in the DTA curve are more pronounced under such measurement conditions. Conversely, the small size of the sample meant that even at this rate of temperature

Table II. Parameters of Gypsum Given by the Manufacturer

Type of Gypsum	Setting Time	Mixing Ratio Gypsum/Water	Time of Mixing	Abrasion Resistance	Linear Expansion (2 h)
Stodent II White	14 to 18 min	100 g/50 g	90 s	11 ± 2 MPa	Max. 0.25 pct

Fig. 2—A microphotograph SEM of a plaster mold used for the production of samples.

increase, the temperature distribution in the sample was uniform. During the measurement, automatic recording of sample mass loss and thermal effects was carried out.

Prior to measuring the samples, a calibration measurement was carried out on empty crucibles, which were later used for testing. The calibration measurement was conducted under the same conditions and according to the same temperature program as the actual measurements to compensate for signals related to device effects. Thermogravimetric measurements as a function of temperature were then performed for the test samples.

E. Preparation of Gypsum Molds

The centrifugal casting method utilizes porous gypsum molds. The gypsum, through the action of capillary forces, draws the solvent (water) from the casting slip, allowing solid samples to be obtained. The gypsum molds were made of Stodent II white dental gypsum. The basic parameters of the gypsum used, based on the data provided by the manufacturer, are presented below in Table II.

The process of preparing gypsum molds begins with weighing water and gypsum in separate containers in proper, predetermined proportions. Then, gypsum is mixed with water; after thorough mixing, the obtained mixture is filtered into a separate container to remove any agglomerates. The hydrated gypsum is then poured into a previously prepared die, which is responsible for reproducing the appropriate shape of the mold. Dies made of epoxy resin were used in the process. The gypsum thus

poured into the mold is then placed on a shaker for about 1 minute to remove any air bubbles trapped inside the slip. After the gypsum binder has cured, the produced gypsum mold is separated from the master mold and placed in a laboratory dryer until completely dry (48 hours, 30 °C). In addition, cylindrically shaped molds for testing physical properties were made using an identical manufacturing process. The test was carried out using the Archimedes method, where the gypsum molds were subjected to soaking in water for 10 minutes.

Figure 2 presents selected microstructural micrographs of the gypsum molds used in the experiment. The molds were characterized by an apparent density of 1.10 ± 0.02 g/cm³, open porosity of 36.66 ± 1.67 pct and absorbability of 33.41 ± 2.06 pct. They featured higher values of porosity and absorbability compared to the molds used in previous studies, which utilized gypsum molds with a porosity ranging from 16.69 to 29.55 pct, absorbability from 8.9 to 16.58 pct and open porosity values depending on the gypsum employed.^[21] The use of molds with higher porosity and absorbability is expected to draw the liquid medium away from the casting slip more quickly during the molding of the specimens and thus allow for better compaction of the material.

F. Molding of Shapes by Centrifugal Slip Casting

Three composite series were produced in the study: Series I—15 vol pct ZrO₂; Series II—50 vol pct ZrO₂; Series III—75 vol pct ZrO₂. The samples were produced by centrifugal slip casting. The resulting slurry was

poured into a gypsum mold, which was placed in the metal housing of the device in the centrifuge chamber. The centrifuge was driven by a motor. The centrifugal casting process was conducted at 3000 rpm for 110 minutes at 25 °C. After casting, the shape was removed from the gypsum mold and placed in a laboratory dryer to remove the remaining water. The obtained samples were dried for 48 hours at 30 °C. This method allowed to produce a finished tube-shaped product. The sintering process of the raw shapes was carried out in a type HTF 17/5 Carbolite furnace. The sintering process parameters were: heating 5 °C/min to 150 °C, then 2 °C/min to 750 °C, then 2 °C/min to 1450 °C, 2 hours holding at 1450 °C and cooling 5 °C/min to 50 °C.

G. Microscopic Observations

The morphology of the starting powders and the microstructure of the shapes after the sintering process were determined by microscopic observation using a JEOL JSM-6610 scanning electron microscope. The microstructure was analyzed by observing the fracture surface of the sample after sputtering with a conductive carbon layer.

H. Phase Composition Analysis

Phase composition analysis was performed using a Rigaku Miniflex II X-ray diffractometer with a CuK α anode. The measurements were carried out at 30 kV and 15 mA. The θ angle was varied over a range of 20 to 100 deg, with a rotation of 0.01 deg and a counting time of 1 second. The test was carried out on flat parallel raw sample surfaces after sintering. MDI JADE 7 software (Materials Data, Inc.) was used for phase composition analysis. The ICDD PDF-4+ 2020 X-ray standard database was used to interpret the results.

I. Hardness and Resistance to Brittle Fracture

The hardness of the samples after sintering was determined by the Vickers method using a Struers DURA SCAN 70 hardness tester. The hardness measurement was conducted from the inner to the outer edge. Approximately seven impressions were made on each test specimen at a load force of 10 kg (98.1 N). After the force was reached, the load was maintained for 10 seconds. After the impression was made and the indenter was removed, the diagonals of said impression were measured on the surface of the test material. The specimens to be measured were ground and polished with diamond pastes of 6, 3 and 1 μ m grit.

The K_{IC} coefficient was then determined. The Niihara equation^[22] was used to determine fracture toughness:

$$K_{IC} = 0.067 \cdot \left(\frac{E}{HV} \right)^{0.4} \cdot \left(\frac{c}{a} \right)^{-1.5} \cdot (HV \cdot a^{0.5})$$

For comparison purposes, the Anstis equation in the form presented in Reference 23 was also used to determine fracture toughness:

$$K_{IC} = 0.016 \cdot \left(\frac{E}{HV} \right)^{0.5} \cdot \frac{P}{c^{1.5}}$$

where E is the Young's modulus [GPa], HV is the Vickers hardness [GPa], P is the indenter load during measurement [N], c is the distance from the center of the impression to the end of the fracture and a half of the diagonal of the impression.

Young's modulus values for each series were determined using the rule of mixtures and respectively amounted to: 356 GPa (Series I), 300 GPa (Series II) and 260 GPa (Series III).

J. Stereological Analysis

Stereological analysis was performed using Micro-Meter v.086b software.^[24,25] The analysis makes it possible to perform a quantitative description of three-dimensional structures on the basis of a two-dimensional image. In addition, it helps to obtain information about the shape of the analyzed grains. In this study, image analysis was used to determine the effect of ZrO₂ content on the growth of Al₂O₃ grains during the sintering process. Moreover, the basic parameters of Al₂O₃ grains, *i.e.*, elongation ($\alpha = d_{\max}/d_2$), curvature of the grain boundary ($R = p/(\pi - d_2)$) and convexity ($W = p/p_c$) (where $d_{\max}$ = maximum diameter of grain projection [μ m]; d_2 = diameter of a circle of the same surface as the surface of the analyzed grain [μ m]; p = perimeter of grain [μ m]; p_c = Cauchy perimeter [μ m]).^[24,25]

K. Environmental Analysis

The environmental impact associated with the formation of Al₂O₃ and ZrO₂ composites has been assessed using the life cycle assessment (LCA) method according to the requirements of the ISO 14044^[26] and EN 15804^[27] standards. The scope of the LCA covers the acquisition and processing of the raw materials (module A1) and operations carried out during the composite fabrication process (module A3). The allocation is based on basic mass. The environmental impacts were calculated per declared unit (DU), which was one sinter with the weight resulting from the formulation. All impacts related to the production of raw materials and additives such as ZrO₂, Al₂O₃, DAC, CA, PVA and distilled water were considered in module A1. The impacts related to the generation and consumption of electric energy supplying the equipment used in the formation of sinters in laboratory conditions were included in module A3. The electric energy consumption values were determined based on the operation times and manufacturer's data on the maximum equipment power. Table III presents the electricity consumption related to each operation of the composites formation process. The waste generated was estimated as 1.5 pct loss of the initial weight of the slurries after venting. The inventory data (LCIA) and environmental indicators used for LCA calculations come from the Ecoinvent v. 3.7

Table III. Life Cycle Inventory of Al₂O₃–ZrO₂ Composites Formed With the CSC Method

Operation	Electric Energy Consumption Per One Al ₂ O ₃ –ZrO ₂ Composite Sinter
Homogenization in a Planetary Mill	2.10 kWh
Venting in a High-Speed Homogenizer	0.16 kWh
Centrifugal Slip Casting	0.68 kWh
Drying	0.34 kWh
Sintering	
Heating to 150 °C	0.18 kWh
Heating to 1450 °C	3.99 kWh
Dwelling	0.74 kWh
Cooling to 50 °C	0.06 kWh
Total	kWh

Fig. 3—Viscosity curves of casting slips.

database, Type III Environmental Product Declaration (EPD) and emission factors issued by the National Centre for Emissions Management (KOBiZE).^[28]

III. RESULTS AND DISCUSSION

A. Analysis of the Casting Slips' Rheological Properties

The results of rheological measurements, which were aimed at determining the viscosity curves of the prepared ceramic slurries, are presented in Figure 3. Based on the results, it can be concluded that the slurries are characterized by a non-Newtonian flow character. In the studied shear rate range, *i.e.*, from 10^{-2} to 10^2 s⁻¹, the viscosity of the studied systems decreased, which therefore defines them as shear-diluted systems. The results obtained correspond well in this respect with literature reports.^[29,30] It is worth noting, however, that in the case of suspensions in which 50 and 75 vol pct by volume of the solid phase constituted zirconium oxide, small areas of shear thickening were observed. The shear thickening effect for the 50 vol pct ZrO₂ sample was 1.7 Pa s and occurred in the shear rate range between 0.32 and 0.51 s⁻¹. For the 75 vol pct ZrO₂ system, a viscosity increase of 0.4 Pa·s was observed between 0.40 and 0.50 s⁻¹. These observations confirm our previous reports that

the addition of Al₂O₃ to an aqueous suspension of ZrO₂ can result in shear thickening of low value and at relatively low shear rates.^[31] It is for these reasons, and considering the applied casting method (CSC), that the phenomenon should not adversely affect the final product.

Upon analyzing the data presented in Figure 3, it is also worth pointing out that the viscosity of the prepared slurries increases with the share of zirconium oxide in relation to alumina. For example, the initial viscosity of the system in which ZrO₂ constituted 75 vol pct of the solid phase was 211 Pa s, while for the samples of 50 vol pct ZrO₂ and 15 vol pct ZrO₂ it was only 133 and 48 Pa s. Clear differences in the viscosity values recorded were also observed with increasing applied shear forces (see Table IV). The obtained results correlate well with previously published studies of our own^[31] as well as with those of other researchers.^[32,33]

Based on the obtained flow curves, presented in Figure 4, it can be additionally observed that all the prepared ceramic slurries display thixotropic properties. This is evidenced by the occurrence of small hysteresis loops. Considering previous literature reports,^[31,34,35] such a result was to be expected.

B. Thermogravimetric Analysis

Figure 5 shows mass changes for a Series I sample: 15 vol pct ZrO₂ along with TG (thermogravimetric curve) and DTA (differential thermal analysis) curves as a function of temperature. In the sample containing 15 vol pct ZrO₂ (Figure 5), a total mass loss of 0.65 pct occurred. In the first stage, a mass loss of 0.01 pct was recorded in the temperature range RT to 129 °C with an associated endothermic effect achieving a minimum at 79.0 °C. The endothermic effect and temperature range likely indicate that removal of physically bound water took place. This was followed by a mass loss of 0.64 pct in the temperature range 129 °C to 479 °C. This process occurred in three stages with a maximum rate of mass loss at approximately 169 °C, 229 °C and 384 °C. The DTA curve shows a fuzzy exothermic peak with a maximum at about 279 °C. Above 479 °C, mass loss is negligible, indicating that all organic additives present in the sample have been removed. At 839 °C, an exothermic signal appears on the DTA curve, which is not accompanied by a mass change. The peak may be the

Table IV. Summary of Viscosity and Compressive Strength Values of the Tested Casting Slips

Suspension	Viscosity at 0.1 s ⁻¹ Shear rate (Pa s)	Viscosity at 1 s ⁻¹ Shear Rate (Pa s)	Viscosity at 10 s ⁻¹ Shear Rate (Pa s)	Viscosity at 100 s ⁻¹ Shear Rate (Pa s)
15 vol pct ZrO ₂	21.3	9.4	2.4	0.4
50 vol pct ZrO ₂	30.7	19.6	3.7	0.7
75 vol pct ZrO ₂	38.4	18.1	4.0	0.8

Fig. 4—Flow curves of casting slips.

result of polymorphic changes occurring in the zirconium oxide, which was a component of the sample.

Figure 6 shows the mass change of a Series II sample—50 vol pct ZrO₂—together with the TG and DTA curves as a function of temperature. In the sample containing 50 vol pct ZrO₂ (Figure 6) there was a total mass loss of 0.88 pct. A mass loss of 0.19 pct occurred in the first stage in the temperature range RT to 148 °C. This was followed by a mass loss of 0.69 pct in the temperature range 148 to 493 °C. The maximum rate of mass loss was recorded at 218 °C and 323 °C accompanied by a fuzzy exothermic effect with a maximum at 293.0 °C. This stage is attributed to the oxidation of organic additives. At 833.0 °C, a fuzzy peak is visible on the DTA, which is not accompanied by mass changes.

Figure 7 shows mass changes of a Series III sample—75 vol pct ZrO₂ along with TG and DTA curves as a function of temperature. In the sample containing 75 vol pct ZrO₂ (Figure 7), there was a total mass loss of 0.54 pct. In the temperature range RT to 134 °C, a mass loss of 0.07 pct occurred, which was due to the evaporation of physically bound water, as indicated by the endothermic effect on the DTA curve at about 74 °C. This was followed by a mass loss of 0.47 pct in the temperature range 134 °C to 509 °C, which occurred in several stages with maximum mass loss rates at 179 °C, 254 °C and 334 °C, accompanied by a fuzzy exothermic

effect with a maximum at 334 °C. The mass loss was due to the oxidation of organic additives. At 839 °C, small exothermic signals on the DTA are visible, which are not accompanied by mass changes and are due to polymorphic transformations occurring in the sample.

A summary of the analysis is given in Table V. From the analysis, it can be concluded that the first mass loss in all samples occurs in the temperature range 129 °C to 148 °C and is related to water removal. The water evaporation temperature is higher in this case because of water being physically bound to the components of the samples. Water molecules can be adsorbed on the surface of the powders and in the structure of the binder, which comprised PVA.^[36]

The mass loss recorded during the subsequent temperature increase is related to the thermal decomposition of the organic substances present in the sample (DAC, CA and PVA). Based on temperature data, the different stages of mass loss can be attributed to the thermal decomposition of the organic additives. DAC decomposes in the temperature range of about 180 °C to 600 °C and occurs in two stages at ca. 180 °C to 250 °C and ca. 400 °C to 600 °C. Citric acid decomposes at a slightly lower temperature of 180 °C to 500 °C, and this process also occurs in two stages (180 °C to 250 °C and 250 °C to 500 °C).^[37] PVA decomposes in the temperature range 200 °C to 450 °C.

C. Characteristics of Specimens Formed by Centrifugal Slip Casting

The method used to form the specimens made it possible to obtain tube-shaped pieces. The sintering process, carried out in several stages, did not cause deformation of the produced elements. No micro-cracks or delamination was observed in any of the samples produced (15, 50, 75 vol pct). The produced shapes were characterized by a post-sintering density close to 100 pct.

To identify individual phases in the produced composites, a phase composition analysis was performed before and after sintering. Figure 8 shows the diffractograms obtained. Based on the X-ray analysis, it was observed that before sintering, all the produced composites consisted of three phases: Al₂O₃, t-ZrO₂ and m-ZrO₂. After sintering at 1450 °C, however, regardless of the ZrO₂ content, two phases were found in the composites: Al₂O₃ and t-ZrO₂. The absence of the m-ZrO₂ phase in the sintered samples is likely the result

Fig. 5—Series I 15 vol pct of ZrO_2 : the change of sample mass with TG and DTA curves as a function of temperature.

Fig. 6—Series II 50 vol pct of ZrO_2 : the change of sample mass with TG and DTA curves as a function of temperature.

of the starting ZrO_2 powder having been stabilized by the addition of 3 mol pct Y_2O_3 . It is worth noting that the stabilization of ZrO_2 is an important process. Zirconium oxide at room temperature has a monoclinic system structure.^[38] When sintering at high temperatures, however, it transitions to a tetragonal structure.^[39] This transition occurs at temperatures above 1170 °C.^[38–40] Then, as the temperature increases above 2377 °C, the tetragonal phase changes to a regular phase.^[38–40] Interestingly, each of these structures differs in density, which consequently causes volume changes and can result in defects in the sinters. Therefore, ZrO_2 powder is doped with oxides of lower values, *i.e.*, MgO , CaO or Y_2O_3 , which make it possible for ZrO_2 to appear in several phases (t- ZrO_2 , m- ZrO_2 , j- ZrO_2) rather than forming a monoclinic structure at room temperature.^[38–40] The addition of the oxide causes a significant decrease in volume during sintering. In the present study, ZrO_2 powder (TZ-3YS-E) was stabilized with 3

Fig. 7—Series III 75 vol pct of ZrO_2 : the change of sample mass with TG and DTA curves as a function of temperature.

mol pct Y_2O_3 . This made it possible to obtain a partially stabilized t- ZrO_2 structure in a raw state, as confirmed by XRD results. In addition, the use of stabilized ZrO_2 enabled the complete transition of the monoclinic phase into a tetragonal phase during sintering at 1450 °C.

Figure 9 shows micrographs taken using secondary electron beam analysis (BSE) of the fractures of composites containing different ZrO_2 contents after sintering. The white areas in the micrographs correspond to ZrO_2 and the gray areas to Al_2O_3 . From the observations, it can be concluded that the samples are homogeneously compacted. No pores or delamination was observed in the composites, which was confirmed by the previously determined relative density of nearly 100 pct. The composites produced, regardless of ZrO_2 content, are characterized by a uniform distribution of zirconium oxide. The use of a casting slip with a solid phase content of 50 vol pct prevented a zirconium oxide gradient from forming at the cross-section.

The effect of ZrO_2 content variation on the growth of Al_2O_3 particles in the composites was investigated using scanning microscope image analysis (Figure 10). The stereological analysis provided information not only on the size of the alumina particles, but also on their shape. The figure presents histograms showing grain distribution in the individual composites. The grain distribution histogram of ZrO_2 in composites containing 75 vol pct of ZrO_2 is not included in the figure (Figure 10), as it was not possible to contour the ZrO_2 grains.

From the results presented, it can be concluded that the growth of Al_2O_3 grains decreases with increasing ZrO_2 content. The study showed that for tubes containing 15 vol pct ZrO_2 , the average grain size of Al_2O_3 was $0.57 \pm 0.27 \mu\text{m}$, whereas for samples containing 50 vol pct ZrO_2 , the average grain size of Al_2O_3 was $0.29 \pm 0.11 \mu\text{m}$. For samples containing the most ZrO_2 (75 vol pct), the average grain size of Al_2O_3 was equal to $0.28 \pm 0.11 \mu\text{m}$. The obtained Al_2O_3 grain size values are significantly smaller than in a previous study, where composites formed using the same powders but different sample fabrication process parameters were

Table V. Summary of Thermogravimetric Analysis

Sample Designation	Composition (Volume Fraction)		Temperature Range Including Weight Loss	Weight Loss Recorded	Temperature of Maximum Rate of Mass Loss	Temperature and Nature of the Heat Effect
	Al ₂ O ₃	ZrO ₂				
ZrO ₂ _15	85 pct	15 pct	RT to 129 °C	0.01 pct	79 °C	69 °C (endo)
			129 to 194 °C	0.07 pct	169 °C	278 °C (exo)
			194 to 273 °C	0.12 pct	229 °C	
			273 to 479 °C	0.36	384 °C	
			—	—	—	840 °C (exo)
ZrO ₂ _50	50 pct	50 pct	RT to 1400 °C	0.65 pct	—	—
			RT to 148 °C	0.19 pct	—	—
			148 to 238 °C	0.15 pct	218 °C	293 °C (exo)
			238 to 493 °C	0.54 pct	323 °C	
			—	—	—	833 °C (exo)
ZrO ₂ _75	25 pct	75 pct	RT to 1400 °C	0.88 pct	—	—
			RT to 134 °C	0.07 pct	74 °C	74 °C (endo)
			134 to 199 °C	0.05 pct	179 °C	334 °C (exo)
			199 to 274 °C	0.11 pct	254 °C	
			274 to 509 °C	0.31 pct	334 °C	
			—	—	—	839 °C (exo)
			RT to 1400 °C	0.54 pct	—	—

Fig. 8—Diffraction patterns of composite samples before and after sintering.

Fig. 9—Sample fractographic observations of composites which contained: (a) 15 vol pct, (b) 50 vol pct, (c) 75 vol pct of ZrO_2 .

characterized.^[20] In the earlier study, composites containing 2.5, 10 and 25 vol pct were found to have average Al_2O_3 grain sizes of 1.7 μm , 1.08 μm and 1.12 μm , respectively.^[20] The differences in the obtained grain sizes were most probably influenced by several factors. In the present study, pre-homogenization in a planetary mill and a different type of gypsum mold were used. The use of pre-homogenization made it possible to obtain a homogeneous casting slip, which was then placed in molds with higher porosity, which may have resulted in better compaction of the material during the molding process. Moreover, in the experiment presented here, the sintering process was carried out in several stages, which certainly influenced grain growth. In addition, based on the analysis of the results, it was found that as the ZrO_2 content in the composites increased, so did the grain size of ZrO_2 .

The application of centrifugal force in the process of sample casting, together with differences in the particle size of powders or their density, may contribute to the formation of a gradient distribution of components in the material structure. The presence or absence of a gradient in centrifugally cast ZTA composites can be largely influenced by the solid content of the casting slip. This relationship was confirmed in a study by Wang *et al.*, who observed a decreasing gradient distribution in the structure with increasing solid phase content in the slip. High solid phase content may affect the viscosity of the produced casting slips and hinder the segregation of component particles during the casting process.^[41]

Hardness testing in the produced composites was carried out in a linear manner along the cross-section of the obtained and properly prepared samples. An analysis of the obtained results shows that there are subtle

Fig. 10—Histograms of Al_2O_3 and ZrO_2 grain size distribution depending on the ZrO_2 phase content in the composites.

differences in hardness along the cross-section, which diminish with increasing ZrO_2 content in the structure, but make it impossible to distinguish the gradient clearly. Therefore, further analysis utilized average values of hardness and fracture toughness.

Figure 11 shows the hardness results for the individual composites. The obtained Vickers hardness results for the samples in each series clearly show that an increase in ZrO_2 content in the structure contributes to a decrease in the hardness of the ZTA composite material. The series with the lowest ZrO_2 content in the structure, equal to 15 vol pct, demonstrated the highest hardness, with an average value of 17.7 ± 0.56 GPa. The lowest hardness was obtained for the series with the highest ZrO_2 content (75 vol pct). The average hardness for this series was 13.9 ± 0.11 GPa.

The observed relationship is consistent with the available literature data. While the hardness of pure Al_2O_3 ceramics reaches values exceeding 20 GPa, the hardness of ZrO_2 ceramics is almost half that value (15 to 16 GPa).^[30] Therefore, increasing the share of ZrO_2 , *i.e.*, the component with lower hardness, leads to a decrease in the hardness of the composite. An analogous

relationship was observed in previous studies conducted by the team, where in ZTA composites produced by the same method, the hardness decreased with an increase in the share of ZrO_2 in the microstructure.^[30] The hardness results obtained for the different series are within the range of hardness obtained in various studies available in the literature. In the work of Maroran *et al.*, in which ZTA composites were produced by linear pressing combined with CIP, the hardness of a composite with 16 wt pct ZrO_2 in the structure was in the range of 21.3 GPa, while at 80 wt pct ZrO_2 it was 15.3 GPa, making these values higher than those obtained in the present study.^[42] Conversely, in the study by Rejab *et al.*, in which the effect of additives on the microstructure and mechanical properties of ZTA composites produced by uniaxial pressing was investigated, the ZTA composite with 20 wt pct ZrO_2 content showed a hardness close to 15.3 GPa, which is lower than that of the corresponding composite presented in this study (Series I).^[43] Similar values for a composite of the same composition (20 wt pct ZrO_2), also produced by pressing, were obtained in the work of Manshor *et al.*, where the measured hardness of the sample was 14.9 GPa.^[44]

Fig. 11—Hardness values for the produced composites with different ZrO₂ content.

Fig. 12—Fracture toughness values for the produced samples.

The next step involved the determination of the fracture toughness of the produced shapes. The results are presented in Figure 12. Among the tested composite samples, Series I, *i.e.*, the series with the lowest ZrO₂ content, featured the highest fracture toughness, for which the K_{IC} value was $4.9 \pm 0.31 \text{ MPa m}^{0.5}$. A decrease in fracture toughness was observed with an increase in the ZrO₂ content in the material. The lowest K_{IC} value was, therefore, characteristic of Series III, with the highest ZrO₂ content, for which the K_{IC} value = $4.31 \pm 0.21 \text{ MPa m}^{0.5}$. It is worth noting, however, that the differences between the maximum and minimum values are minimal. A similar relationship was observed for the Anstis equation. Here, also, the highest fracture toughness was found for Series I with the lowest ZrO₂ content in the structure (25 vol pct), in which $K_{IC} = 10.65 \pm 0.45 \text{ MPa m}^{0.5}$, while the lowest was found for Series III with 75 vol pct ZrO₂, in which K_{IC} was equal to $9.31 \pm 0.22 \text{ MPa m}^{0.5}$.

Despite the large discrepancies in the obtained values of the K_{IC} fracture toughness coefficient, a certain recurring trend can be identified; the increase in fracture toughness of ZTA composites is directly correlated to an increase in ZrO₂ content in the material structure.^[42] This is due to phase transformation (transformation toughening) occurring in ZrO₂ under the influence of stress at the end of the propagating crack.^[45] It should be noted that this dependence is observed for small ZrO₂ contents in the composite structure. As shown by the

results of Maoshan *et al.*, when increasing the ZrO₂ content in the composite, the K_{IC} value increases up to a certain limiting ZrO₂ content and then begins to decrease.^[46] To some extent, this explains the decreasing K_{IC} value trend as a function of ZrO₂ content in the material, as observed in the present study. The fracture toughness coefficient values observed in the literature are characterized by a large discrepancy. The K_{IC} values obtained in this study, determined by the Anstis equation, are much higher than those found in the available literature, where, for samples with 20 wt pct ZrO₂ content (Series I), the K_{IC} value was $6.5 \text{ MPa m}^{0.5}$ in the study by Maoshan *et al.*^[46] or $5.93 \text{ MPa m}^{0.5}$ in the results published by Manshor *et al.*^[47] Conversely, the K_{IC} values determined using the same equation for the composite with 85 wt pct ZrO₂ content in the study by Xia *et al.* reached only $3.3 \text{ MPa m}^{0.5}$.^[48] The K_{IC} values determined from the Niihara equations described in the literature are also characterized by a large discrepancy.

In the study by Chai *et al.* for a composite with 24 vol pct Al₂O₃ content in the structure, the K_{IC} value was $5.81 \text{ MPa m}^{0.5}$, which is higher than that of the series produced in the present study with similar ZrO₂ content (Series III).^[49]

It is noted that it is not possible to compare fully the obtained results for either hardness or fracture toughness. The results are influenced by a number of factors, such as the starting powders used, the method of obtaining the raw samples, the course of the sintering process, including the sintering temperature, and the microstructure of the material after sintering. In the case of fracture toughness, additional factors making it difficult to compare results include the method of measurement and, in the case of the Vickers indentation method, the dependence of the results obtained on the equation used in the calculation and on the load applied during indentation.^[50]

Table VI summarizes the results of the life cycle analysis (LCA) of the composite sinters formed via the CSC method with 15, 50 and 75 vol pct of ZrO₂. The environmental impacts associated with the acquisition and processing of the Al₂O₃, ZrO₂, distilled water and additives are presented in modules A1. Modules A3 present burdens resulting from the consumption of electricity for operations performed during the manufacturing process. When analyzing the results, one can observe that the increase in ZrO₂ phase content in the composite results in higher value of the sinter's environmental footprint, which can be noted in the value of the A1 module indicators. Thus, for 1 kg of the formed sinters, the greenhouse gas (GHG) emissions expressed as equivalent of CO₂ were determined at the level of 2.41, 2.65 and 2.85 kg eq. CO₂ for the composites with 15, 50 and 75 vol pct of ZrO₂ respectively. Adding the burden associated with the manufacturing stage (A3), the obtained values are slightly higher than the burdens associated with the production of high-density polyethylene granulate (HDPE) ($2.25 \text{ kgCO}_2/\text{kg}$), polypropylene granulate ($2.21 \text{ kgCO}_2/\text{kg}$)^[51] and steel pipes ($2.28 \text{ kgCO}_2/\text{kg}$)^[52] which are commonly used in the construction sector for linear infrastructure applications.

Table VI. Results of Life Cycle Analysis (LCA) of Composites Obtained via the CSC Method With 15 Vol Pct, 50 Vol Pct and 75 Vol Pct of ZrO₂

Indicator	Unit	15 Vol Pct ZrO ₂		50 Vol Pct ZrO ₂		75 Vol Pct ZrO ₂	
		DU: 1 Sinter (g)		DU: 1 Sinter (g)		DU: 1 Sinter (g)	
		A1	A3	A1	A3	A1	A3
Global Warming Potential	kg CO ₂ eq.	1.27E-01	5.94E+00	1.57E-01	5.94E+00	1.87E-01	5.94E+00
Depletion Potential of the Stratospheric Ozone Layer	kg CFC 11 eq.	1.56E-08	0.00E+00	3.84E-08	0.00E+00	3.84E-08	0.00E+00
Acidification Potential of Soil and Water	kg SO ₂ eq.	8.59E-04	7.54E-03	9.23E-04	7.54E-03	9.23E-04	7.54E-03
Formation Potential of Tropospheric Ozone	kg Ethene eq.	4.78E-05	0.00E+00	4.95E-05	0.00E+00	4.95E-05	0.00E+00
Eutrophication Potential	kg (PO ₄) ³⁻ eq.	2.16E-04	6.18E-04	2.94E-04	6.18E-04	2.94E-04	6.18E-04
Abiotic Depletion Potential for Non-fossil Resources	kg Sb eq.	1.67E-06	2.20E-05	4.30E-06	2.20E-05	4.30E-06	2.20E-05
Abiotic Depletion Potential for Fossil Resources	MJ	1.58E+00	5.74E+01	2.20E+00	5.74E+01	2.20E+00	5.74E+01
Total Use of Renewable Primary Energy Resources	MJ	1.19E-01	7.96E+00	3.06E-01	7.96E+00	3.06E-01	7.96E+00
Total Use of Non-renewable Primary Energy Resources	MJ	1.28E+00	6.02E+01	1.74E+00	6.02E+01	1.74E+00	6.02E+01
Use of Secondary Materials	kg	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Use of Renewable Secondary Fuels	MJ	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Use of Non-renewable Secondary Fuels	MJ	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Net Use of Fresh Water	m ³	3.16E-02	1.00E-02	7.85E-02	1.00E-02	7.85E-02	1.00E-02
Hazardous Waste Disposed	kg	1.07E-06	0.00E+00	2.05E-06	0.00E+00	2.05E-06	0.00E+00
Non-hazardous Waste Disposed	kg	4.98E-02	7.90E-04	4.23E-02	8.89E-04	4.23E-02	9.87E-04
Radioactive Waste Disposed	kg	3.00E-06	0.00E+00	5.21E-06	0.00E+00	5.21E-06	0.00E+00
Components for Re-use	kg	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Materials for Recycling	kg	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Materials for Energy Recover	kg	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00	0.00E+00

One should remember that the Al₂O₃-ZrO₂ composite sinters were obtained under non-optimized laboratory conditions. Therefore, based on the obtained results, we can conclude that the further optimization of the Al₂O₃-ZrO₂ composite formation process via the CSC method, including up-scaling, can provide Al₂O₃-ZrO₂-based products exhibiting more favorable mechanical, strength and thermal parameters, as well as lower environmental footprint in comparison with conventional materials commonly used for linear infrastructure applications.^[53,54] Nowadays, such an approach becomes important in light of the global transformation toward sustainable construction and the circular economy.^[55,56] An analysis of the obtained LCA results, in light of the authors' previous papers, proves that the proper selection of laboratory is a key factor determining the environmental characteristics of the process. For Al₂O₃ and ZrO₂ composites produced with the CSC method, the power demand to make one sinter amounts to ca. 8.25 kWh, while making a sinter with the slip casting (CS) method that eliminates the centrifugal casting stage consumed ca. 14.5 kWh.^[57] Such high power demand per single product is typical for processes executed in non-optimized laboratory conditions. The size of the furnace chambers turned out to be a factor responsible for such a significant difference in the power characteristics of the processes in laboratory conditions.

IV. CONCLUSION

The results have demonstrated that the CSC method can be successfully used to produce ceramic-ceramic composite materials. The obtained composite shapes with different proportions of Al₂O₃ to ZrO₂ were characterized by lack of deformation after sintering. The resulting ceramic-ceramic composites are hermetic, as evidenced by the absence of delamination and pores and a density close to 100 pct. The gradient effect is negligible on the cross-section of the samples. This is due to a casting slip with a 50 pct solid phase content by volume that was used to produce the shapes. The sintered samples are characterized by the presence of only two phases, namely Al₂O₃ and t-ZrO₂. Based on the results, it is concluded that the composites produced are suitable for potential use as thermocouple covers.

Moreover, it is also concluded that an increase in the share of ZrO₂ in the Al₂O₃-ZrO₂ composite results in a decrease in the average Vickers hardness and a decrease in the average grain size of Al₂O₃. In contrast, an increase in the average grain size of ZrO₂ is inversely proportional to the above relationship. Despite such marked differences in the samples, the fracture toughness varies little, with a slight decrease observed as the ratio of ZrO₂ to Al₂O₃ in the composite increased.

When analyzing the environmental aspect, it has been found that formation of the $\text{Al}_2\text{O}_3\text{--ZrO}_2$ sinters causes greenhouse gas (GHG) emissions at a level slightly higher, but still comparable to the production of HDPE and PP granulates and steel pipes. Based on the obtained results, it can be argued that the further optimization of the composite formation process via the CSC method, including up-scaling, can provide $\text{Al}_2\text{O}_3\text{--ZrO}_2$ -based products exhibiting more favorable mechanical, strength and thermal parameters, as well as the lower environmental footprint compared with conventional materials commonly used for linear infrastructure applications, which is important in light of the global transformation toward sustainable construction and the circular economy.

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COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence those works reported in this paper.

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