

Mechanical properties of zirconia toughened mullite composites fabricated by slip casting

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The main objective of this article is to obtain polyphasic ceramics belonging to the $\text{Al}_2\text{O}_3\text{-SiO}_2\text{-ZrO}_2$ system by reaction sintering of zircon, alumina, kaolinite and colemanite powder mixture was explored in this work. In addition, the study focused on low temperature synthesis and the mechanical properties of ZTM improved as expected following the addition of boron minerals such as colemanite ($\text{Ca}_2\text{B}_6\text{O}_{11}\cdot 5\text{H}_2\text{O}$). The effects of sintering temperature, solid concentration of the prepared suspensions and colemanite additive on the mechanical and other properties of mullite-zirconia composites were investigated. Green compacts were made by slip casting of stabilized aqueous suspensions with and without colemanite additive after which reaction-sintering was applied at temperatures ranging between 1450 to 1550 °C under different firing conditions. The resulting sintered materials were characterized in terms of bulk density, XRD and SEM and mechanical properties such as flexural strength, elastic modulus, hardness and fracture toughness. It was observed as a result of a comparison between the mullite-zirconia composites prepared via slip casting and studies in literature from a mechanical standpoint that composites in literature have yielded similar results in terms of mechanical properties. Mullite grains in composites formed a nearly continuous phase in which zirconia grains were distributed yielding better mechanical and fracture properties.

Key words: Slip casting, Reaction sintering process, Mechanical properties, Mullite-zirconia composites.

Introduction

Mullite is one of the most easily formed, commonly encountered and important compounds found in numerous industrial ceramics products [1]. The system of $\text{Al}_2\text{O}_3\text{-SiO}_2$ is one of the most important binary systems in ceramics and mullite is the only thermodynamically stable intermediate compound in this system. Although mullite is among the traditional refractory materials, it has started to gain importance as a candidate material for advanced ceramics applications in recent years [2].

However, mullite has a lower fracture toughness and mechanical strength. Zirconia, a metal oxide mineral, has different and superior properties in comparison with other oxide ceramics. It can be observed upon examining the mechanical properties of zirconia that it has high chemical and dimensional stability as well as mechanical strength and toughness. Various studies have been performed to improve the mechanical properties of mullite as a result of which it was determined that the incorporation of zirconia particles in mullite matrix enhances its mechanical properties either by transformation toughening mechanism or micro-cracking [3, 4]. After the transformation toughening property of zirconia was discovered in 1975, its development was established,

and studies on zirconia based ceramic composite materials began to increase rapidly. Recently, zirconia (ZrO_2) based ceramic composites have received special attention and are promising materials for structural applications, because they have excellent physical and mechanical properties such as high hardness, high elastic modulus and fracture toughness [2].

Various manufacturing methods have been developed to synthesize mullite-zirconia composites such as gel casting [5], spark plasma sintering [6], laser floating zone directional solidification method [7], sol-gel [8] and reaction sintering [9, 10]. Currently, composite slip casting has been developed rapidly over the past few decades. Low capital investment for the products to be produced is among the advantages of this technique. In addition, a wide variety of complex shapes can be produced by using this technique which could not be produced using other conventional methods. Slip casting has been employed for fabrication of these composites for obtaining fine and homogeneous microstructures.

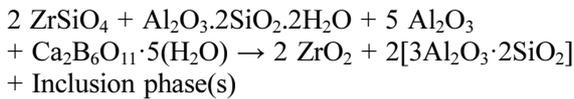
The main objective of the present work was to obtain zirconia toughened mullite composites that are a dense and have excellent mechanical properties by way of slip casting using natural starting raw materials such as zircon, alumina, kaolinite and colemanite. Another major objective of the study in addition to using colemanite ($\text{Ca}_2\text{B}_6\text{O}_{11}\cdot 5\text{H}_2\text{O}$) to improve the mechanical properties of the produced material was to enable the synthesis of mullite-zirconia composites at lower temperatures. In this context, this study particularly the principal slip

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casting conditions and colemanite addition were investigated. The structural and microstructural changes were evaluated together with the mechanical properties of the obtained composites. Due to the lower solid concentration of aqueous suspensions an incomplete reaction is expected, as a consequence multiphasic ceramics with different alumina, mullite, zircon and zirconia contents are expected.

Experimental Procedure

Alumina source was selected as alumina (Al_2O_3) and kaolinite ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) during the study on the production of zirconia toughened mullite composite and zircon (ZrSiO_4) was used as the source of silica and zirconia. Mullite- ZrO_2 composites were obtained by reaction-sintering using three different solid to liquid weight ratios (45/55, 50/50 and 55/45 wt.%). Mainly, two mixtures with different weight ratios (wt.%) were prepared with and without additive (colemanite) from the starting powders. The raw materials were weighed at specific stoichiometric ratios and mixed by assigning the codes of MZ0 and MZK. The following reaction was used as a base for the synthesis of mullite-zirconia composite.



The weight ratio of $\text{Al}_2\text{O}_3/\text{SiO}_2$ was kept approximately as 2.5 owing to the impurity content of the precursors (in addition to the molar ratio). The amount of colemanite consisting of natural sources was precisely controlled due to the limited CaO content that played a significant role in partially stabilizing zirconia. A $\text{Ca}_2\text{B}_6\text{O}_{11} \cdot 5(\text{H}_2\text{O})$ addition at a range of 7 wt.% range was used for studying the impact of colemanite addition on the properties of these composites. The weight ratio of colemanite during the preparation of the mixtures was calculated by considering the amount of CaO (~14 mol %) required for the zirconia to be partly stable.

The prepared mixtures were ground on a planetary mill (Retsch PM 200). Grinding media and grinding conditions were determined as given in Table 1. The obtained average particle size of the prepared MZ0 and MZK compositions were 1.8 and 1.9 μm and BET specific surface areas: 4.1 and 4.3 m^2/g respectively.

Table 1. Milling conditions.

Rotation speed	300 rpm/min.
Grinding ball	3 mm diameter ZrO_2 , 5 mm diameter ZrO_2
Grinder pot	ZrO_2
Media	Methanol
Duration	6 h.
Powder/alcohol/ball ratio	1 / 1.5 / 2.5

Aqueous suspensions at a pH ~ 9.02 containing 45-50-55% solids by weight were prepared in order to form the prepared mixtures by slip casting by using Darvan C [PMMA (polymethylmethacrylate)] as a dispersant at the rate of ~0.25 % by weight. The prepared concentrated suspensions were consolidated in a plaster mold to produce bars 7.5 mm \times 5 mm \times 55 mm in size; after which it was dried in an oven at a constant temperature of 100 °C following a day of natural drying.

Bars (MZ045, MZK45, MZ050, MZK50, MZ055 and MZK55) prepared from aqueous suspensions containing 45-50-55 % solids by weight were sintered at 1450 °C, 1500 °C and 1550 °C to synthesize the mullite-zirconia composite material by reaction sintering and to identify physical properties in the synthesized material. The sintering temperature was increased by 5 °C per minute; it was left at this temperature for 5 hours and was similarly allowed to cool down at 5 °C/min. Density of sintered composites was determined via the Archimedes' method. The crystalline phase formed was investigated via X-ray diffraction (XRD, Rigaku, MiniFlex) at 10-40° and a scanning speed of 2° (2θ) per minute for the composititons with and without colemanite. The flexural strength (σ , MPa) and elastic modulus (E , GPa) of the composites were measured using Instron 5581 device. A 2 kN load cell that moved at a rate of 0.5 mm min^{-1} was used during the tests. The bending strength and elastic modulus were determined using equations 1 and 2, respectively:

$$\sigma = \frac{3Pl}{2bh^2} \quad (1)$$

$$E = \frac{ml^3}{4bh^3} \quad (2)$$

Where P is the rupture load, l is the holder span, b is the width, h is the height and m is the slope of the strain - deformation ($\sigma - \epsilon$) curve. The hardness of the sintered composite samples was obtained using Vicker's Hardness experiments. The formula used for hardness measurement have been given below. According to Equation 3, if P is the applied load, D is the diagonal length of the indentation that the Vickers sinker tip created on the surface of the material. The composite samples were taken into a mold with epoxy resin, sanded with SiC paper, and polished with a diamond paste in order to correctly measure the indentation length that was created before the hardness test.

$$H_V = 1,844 \frac{P}{D^2} \quad (3)$$

The fracture toughness of the composite samples after sintering was calculated using the following Anstis equation. In the following equation (Equation

4); α is a constant (0.014), E is an elastic module, H_v is Vickers hardness value, P is the applied load and c is the crack length [11].

$$K_{IC} = \alpha \left(\frac{E}{H_v} \right)^{1/2} \left(\frac{P}{c^{3/2}} \right) \quad (4)$$

The microstructures were studied via scanning electron microscopy (FEI Nova NanoSEM 650) on the surfaces of composites.

Results and Discussion

Densification and phase development

The results obtained as a result of the XRD analyses on additive-free mixtures (MZ045, MZ050 and MZ055) and mixtures containing colemanite (MZK45, MZK50 and MZK55) sintered at 1550 °C can be seen in Fig. 1 and Fig. 2, respectively. Accordingly; mullite has been detected as the main phase in addition to t-ZrO₂ and m-ZrO₂ phases of zirconia along with some tridymite and

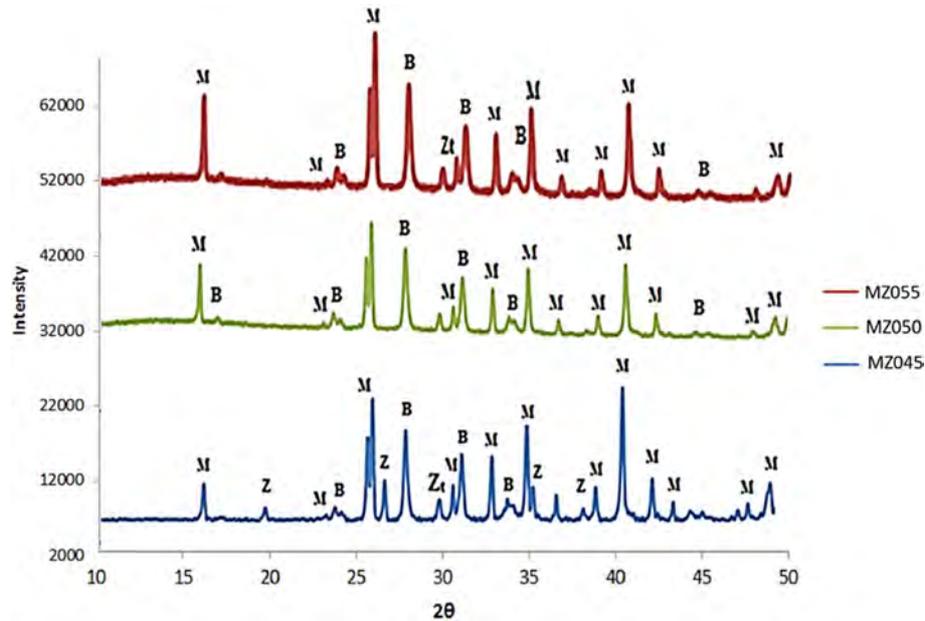


Fig. 1. XRD patterns of sintered specimens at 1550 °C for 5 h for the additive-free compositions (MZ045, MZ050 and MZ055). M: mullite; B: monoclinic-ZrO₂; Z_t: tetragonal-ZrO₂; Z: ZrSiO₄.

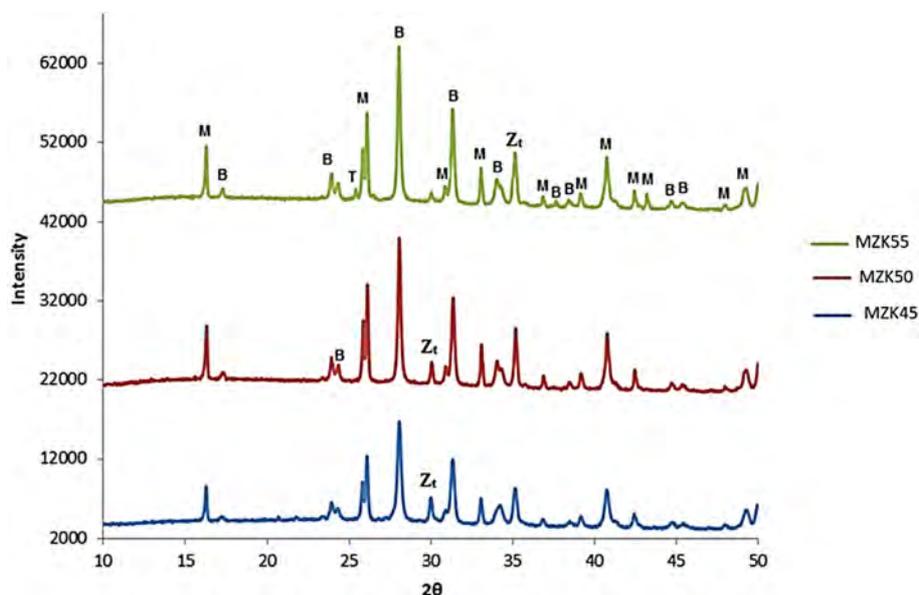


Fig. 2. XRD patterns of sintered specimens at 1550 °C for 5 h for the compositions containing colemanite (MZK45, MZK50 and MZK55). M: mullite; B: monoclinic-ZrO₂; Z_t: tetragonal-ZrO₂; Z: ZrSiO₄, T: tridymite.

zircon phases A small peak of tridymite (SiO_2) at 1550 °C was observed on the ZMK55 composition XRD pattern. The tridymite peak was probably nucleated from the liquid phase with increased temperature and incorporated into the mullite formation with a progressive reaction. It could be concluded from phase analyses of additive-free mixtures (MZ045, MZ050, and MZ055) that the decomposition of zircon increases with increasing solids concentration.

The most important and obvious difference between the mixtures, MZ045 and MZK45 mixtures obtained via slip casting was that the XRD patterns of the MZK45 mixture did not contain zircon-phase peaks. The colemanite additive enabled zircon to decompose at 1550 °C. Colemanite contains more than 20 % by weight of CaO. The presence of basic oxides in silica fluids, and especially of coarse cations such as CaO, increases the alkalinity ratio of the silica rich liquid. The fluidity and erosive properties of the liquids increase with the basicity ratio. The glassy phase, which decreases its viscosity due to both the increasing temperature and the basic oxide content, achieves the high erosive effect and the ability to decompose, thereby increasing the amount and kinetics of the zircon phase [12].

Another reason for this situation is the solid concentration of prepared mixture. The decomposition of zircon in the lack of any additive in the prepared mixture (MZ045); however, it was still not completed at 1550 °C. It is thought that this causes the reactions not to be completed especially during sintering at 1550 °C since packing and intergranular interactions between grains in a composition containing 45 % solids by weight are low. One possible mechanism is segregation of particles of different sizes, which is increased when solid loading is low and, thus, hindered settling is reduced [13, 14].

Density values of additive-free (MZ045, MZ050 and MZ055) and composite specimens with colemanite additive (MZK45, MZK50 and MZK55) sintered at 1450, 1500 and 1550 °C have been given in Table 3. As can be seen from the table, bulk density values increased in all specimens obtained via slip casting with increasing solid concentration and sintering temperature. The highest density value was achieved with (MZK55) the composition containing colemanite sintered at 1550 °C.

Microstructure characterization

Microstructures of the fracture surface and phase distribution of sintered samples sintered at different temperatures were observed via scanning electron microscope (SEM, FEI Nova NanoSEM 650) with energy dispersive X-ray spectroscopy (EDX). Figs. 3-4 show the SEM photomicrographs of the sintered specimens. SEM photomicrograph and corresponding EDX spectra of the sample MZK50 sintered at 1550 °C

Table 2. Bulk density values (g cm^{-3}) of mullite-zirconia composites as function of colemanite content, solid concentration and sintering temperature.

Temperature (°C)	MZ045	MZK45	MZ050	MZK50	MZ055	MZK55
1450	1.69	2.61	1.82	2.91	1.87	3.30
1500	2.29	3.18	2.60	3.28	2.70	3.37
1550	2.47	3.25	2.60	3.37	3.01	3.49

Table 3. The bending strength values of additive-free mixtures (MZ045, MZ050, MZ055) and mixtures containing colemanite (MZK45, MZK50 and MZK55) obtained by slip casting.

	MZ045 σ (MPa)	MZ050 σ (MPa)	MZ055 σ (MPa)
Temp. (°C)	1450	1450	1450
	52	56	65
	1500	1500	1500
	62	71	79
	1550	1550	1550
	69	93	94
	MZK45 σ (MPa)	MZK50 σ (MPa)	MZK55 σ (MPa)
Temp. (°C)	1450	1450	1450
	106	133	142
	1500	1500	1500
	152	161	186
	1550	1550	1550
	180	170	187

has been shown in Fig. 5. The mullite phase (grey spot in Fig. 5) is well surrounded by grain boundary regions of zirconia (white spot in Fig. 5).

It can be stated when the microstructures of the samples with and without colemanite sintered at 1550 °C for 5 hours are compared that the colemanite containing mullite-zirconia composites are denser despite the presence of closed pores. This case was also compatible with bulk density values of mullite-zirconia composites in Table 3.

Mullite grains are equiaxed in nature, but also some elongated grains have developed. Zirconia grains have occupied the intergranular as well as intragranular positions in mullite matrix. Two types of zirconia grains are present which, one is inter-granular zirconia located between the mullite grains and the other one is intra-granular zirconia present within mullite matrix. Many researchers are shown that intra-granular particles as spherical shape are tetragonal ZrO_2 , while the inter-granular ZrO_2 is mainly [15-17] monoclinic. Rounded morphology of the ZrO_2 is due to the transient liquid phase.

Especially, for the mixtures containing colemanite sintered at 1550 °C, the microstructure was a well-defined mullite (dark grains) ceramic matrix with a homogeneous distribution imbibed zirconia grains (bright grains), this microstructure was comparable to the one obtained by different manufacturing routes [5, 17].

Mechanical characterization

Elastic module and strength

The bending strengths and elastic modulus of all samples produced via slip casting and sintering at

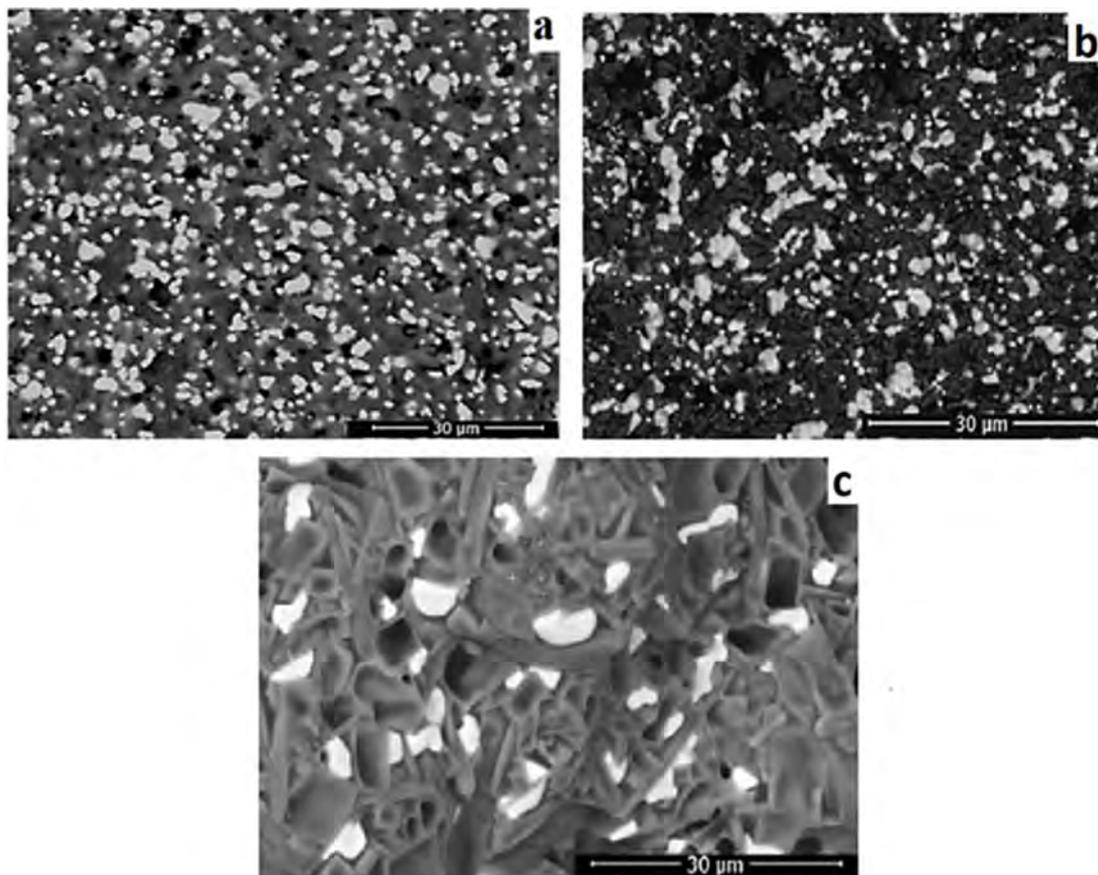


Fig. 3. Microstructure of MZ045, MZ050 and MZ055 specimens after sintering at 1550 °C for 5 h. (a) MZ045, (b) MZ050 and (c) MZ055.

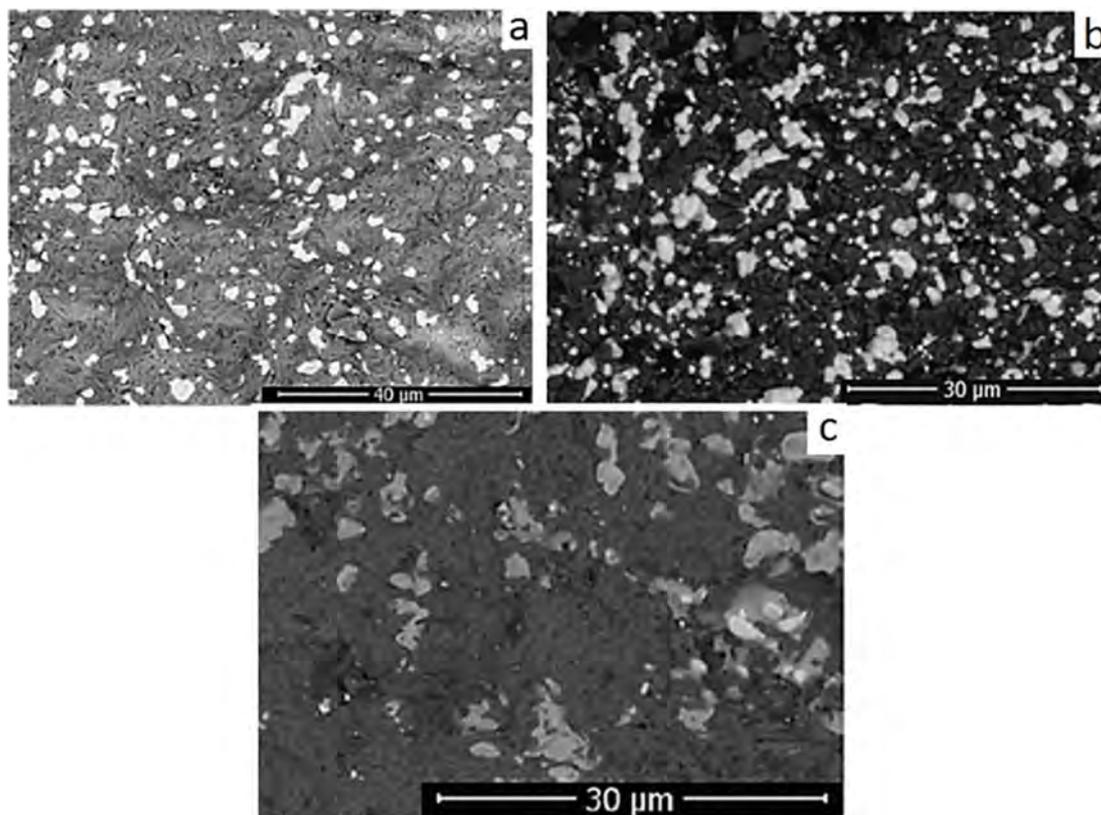


Fig. 4. Microstructure of MZK45, MZK50 and MZK55 specimens after sintering at 1550 °C for 5 h. (a) MZK45, (b) MZK50 and (c) MZK55.

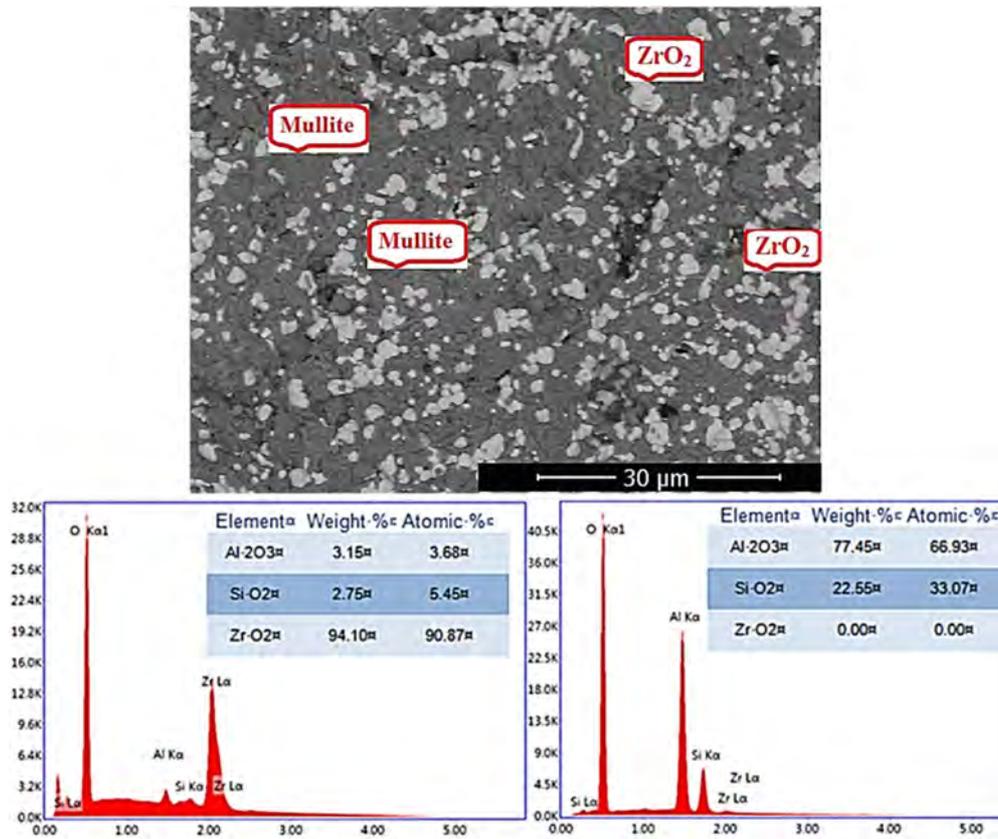


Fig. 5. SEM photomicrograph and corresponding EDX spectra of the specimen MZK50 sintered at 1550 °C for 5 h.

Table 4. The elastic modulus values of the additive-free mixtures (MZ045, MZ050 and MZ055) and mixtures containing colemanite (MZK45, MZK50 and MZK55) obtained by slip casting.

	MZ045	E (GPa)	MZ050	E (GPa)	MZ055	E (GPa)
Temp. (°C)	1450	28	1450	34	1450	36
	1500	53	1500	40	1500	57
	1550	37	1550	78	1550	78
	MZK45	E (GPa)	MZK50	E (GPa)	MZK55	E (GPa)
Temp. (°C)	1450	71	1450	82	1450	105
	1500	122	1500	105	1500	136
	1550	126	1550	127	1550	141

1450, 1500 and 1550 °C were obtained during three-point bending tests. It was observed upon examining the results that the bending strength and elastic modulus values of sintered samples varied with the increase in sintering temperature. The most important causes for the difference in bending strength values were observed as the density and accordingly the amount of porosity in the material in addition to the phase composition in the composite material [18].

Bending strength and elastic modulus values were in parallel with the density acquisition behavior (Table 3) when considered in relation to additive-free mixtures (MZ045, MZ050 and MZ055) and mixtures containing

Table 5. The Vickers hardness values (in GPa) of additive-free mixtures (MZ045, MZ050 and MZ055) and mixtures containing colemanite (MZK45, MZK50 and MZK55) obtained via slip casting.

Temperature (°C)	1450 °C	1500 °C	1550 °C
MZK45	3.26	8.39	8.57
MZ045	1.98	2.52	3.02
MZK50	4.62	8.56	8.69
MZ050	2.03	2.56	3.92
MZK55	5.17	9.21	9.60
MZ055	2.21	3.71	3.93

Table 6. The fracture toughness values of additive-free (MZ045, MZ050 and MZ055) and mixtures containing colemanite (MZK45, MZK50 and MZK55) obtained via slip casting in MPa.m^{1/2}.

Temperature (°C)	1450 °C	1500 °C	1550 °C
MZK45	2.43	2.98	3.00
MZ045	1.94	1.99	2.65
MZK50	3.30	3.92	4.27
MZ050	2.91	3.09	3.49
MZK55	3.52	4.28	5.00
MZ055	3.00	3.53	4.12

colemanite (MZK45, MZK50 and MZK55) depending on the solids concentration of the prepared slip and the

Table 7. Mechanical properties of various mullite/zirconia composites.

References	Sintering temperature (°C)	K _{1C} (MPam ^{1/2})	H _v (GPa)	σ (MPa)	E (GPa)
Aksel, (2003) [25]	1500	1.2	-	165	15.2
L.B. Garrido, (2006) [19]	1600	3.5	9-12	-	-
Hamidouche, (2007) [26]	1560	2	11	197	-
Liang, (2008) [23]	1150	5.13	10-13	520	-
Hemra, (2014) [27]	1600	-	-	190	-
Carvalho, (2014) [7]	1400	3.5	11,3-21,2	534	-
Ashrafi, (2015) [28]	1400	-	11.1	-	-
Kumar, (2015) [3]	1550	6	-	225	-
Rendtorff, (2014) [24]	1500	4.3	9	-	-

sintering temperature.

Hardness and fracture toughness

The measured hardness and calculated fracture toughness values of all samples at various temperatures have been given respectively in Table 6 and Table 7. The hardness values varied between 1.98 and 9.60 GPa (under a load of 9.8 N) according to the measurements carried out after sintering. There were significant differences between the hardness and fracture toughness values of the mixtures with and without additives at solid concentrations of 45 %, 50 % and 55 %. The lowest hardness and fracture toughness values were determined for the additive-free composition containing 45 % solids sintered at 1450 °C, due to high porosity. A linear increase in the hardness and fracture toughness values for all composite products was observed depending on the increasing solids concentration and consequently the density and temperature.

Conclusions

XRD conclusions and theoretical approaches

It can be put forth as a result of XRD phase analyses of MZ0 and MZK compositions that three major phases have been identified, viz., mullite, tetragonal zirconia and monoclinic zirconia at 1550 °C except for MZ045. Trace amount peaks appeared at this temperature in MZK55 indicating that complete reactions have taken place between the starting materials and that mullite-zirconia composites have been obtained. Colemanite reduced the decomposition temperature of zircon thereby enabling mullite-zirconia composite synthesis in comparison with mixtures without colemanite (MZ0).

The zircon phase was not detected in the mixture of MZK sintered at 1550 °C, while the mixture of MZ045 zircon peaks appeared at 1550 °C. These findings can be explained by our current XRD results. Both colemanite additive and solid concentration of mixtures have reduced the decomposition temperature of zircon and complete reactions have taken place between the starting materials thereby forming mullite-zirconia composites.

It is believed that with the increase in the solid

concentration of the mixture, the granules are packed tighter in slip casting and that the reactions proceed faster by the subsequent sintering process.

Microstructure characterization conclusions

The microstructure of mullite-zirconia composites sintered at different sintering temperatures (1450-1550 °C), BSE-SEM analysis has been performed associated with density and porosity. As can be seen from Fig. 4, the grains grow up and the porosity decreases as the sintering temperature increases from 1450 °C to 1550 °C. It can be observed from Fig. 3 that obvious pores and loose structures are observed in addition to the interconnected growth of grains, indicating low density. It can be clearly seen in Fig. 5 that crystalline phases are mullite and zirconia since the color is directly related to the atomic number. The different phases observed have been labeled (zirconia, mullite) among which the dark grain phase is mullite and the bright grain is zirconia. The fact that density and grain size increase with decreasing porosity is more obvious in Fig. 4, indicating that the increase of linear shrinkage is due increasingly to the intensive interconnection between grains.

Densification behaviour of composites

MZK55 sample has the highest density of 3.49 g cm⁻³ when the temperature increases to 1550 °C. Bulk densities of all samples increased at the same temperature with increasing solid concentration. It was observed from the results for the MZ0 samples that density increases with increasing sintering temperature. As was the case in our previous study [15] high-density values in mixtures containing colemanite result from temporary liquid phase formations that develop during sintering at mullite-zirconia composites obtained via slip casting. The sintering process can remove the inter capillary forces. Solid state sintering process requires the transport of atoms from one place to another (diffusion of material) through diffusing (diffusion). As the temperature increases, the particles are sintered together and pores on grain boundaries are reduced by solid-state diffusion [5]. These findings can be explained by our current

research results, which shows that mullite-zirconia composites initiated effective densification at 1550 °C.

Mechanical characterization conclusions and discussions

It was observed that satisfactory results were obtained when bending strength and elastic module values (Table 4 and Table 5) of additive-free (MZ045, MZ050 and MZ055) and containing colemanite (MZK45, MZK50 and MZK55) composite products produced via slip-casting specimens sintered at 1450, 1500 and 1550 °C were compared with those of the specimens from similar studies in literature (Table 7). Other mechanical properties are close to those of similar composite materials studied in literature except for the theoretically calculated fracture toughness.

The micro-hardness value of mullite-zirconia composites produced from alumina-zircon mixture, depending on the raw material contained in the specimens sintered at 1550 °C was an average of 11 GPa during the study carried out by L.B. Garrido et al.; it was ~14 GPa in the alumina reinforced mullite/zirconia composite at 1500 °C; in the study K.Das et al. carried out on the zirconia mullite composite, it was measured as 10.1 GPa in the 5% yttrium oxide (Y_2O_3) containing mullite zirconia composites at 1550 °C [19-21]. The best results in our study were measured as about 9.60 GPa at 1550 °C for the MZK55 specimen containing colemanite with 55% solids concentration (Table 6).

Hardness values vary between 1.98 and 9.60 GPa (under a load of 9.8 N) according to the results of the measurements carried out after sintering. The main reasons why many different results are observed in literature are, besides the applied forming and sintering methods, the starting raw materials and the phases formed in the material after sintering [22].

As a result of fracture toughness calculations; the highest fracture toughness value was obtained as 5 $MPa\sqrt{m}$ at 1550 °C for MZK55 specimen containing colemanite with 55% solids concentration obtained via slip casting (Table 4.4). In the study carried out by N.M.Rendtorff et al., the highest fracture toughness value of mullite-zirconia composites at 1500 °C was 4.3 $MPa\sqrt{m}$; the best fracture toughness value in mullite-zirconia composites produced from alumina-zircon mixture by L.B. Garrido et al, was 3.5 $MPa\sqrt{m}$ in specimens first sintered at 1450 °C for 2 hr. and then heat-treated at 1600 °C for 2 hr.; in the study M.M.S. Wahsh et al. carried out, it was approximately 4.5 $MPa\sqrt{m}$ for the alumina reinforced mullite-zirconia composite at 1500 °C; also, it was found to be 5.13 $MPa\sqrt{m}$ in the study carried out at between 1620-1700 °C by L. Shuquan et al. [19, 20, 23, 24].

CaO in colemanite ($CaB_3O_4(OH)_3 \cdot H_2O$) has a significant effect on the stabilization of tetragonal zirconia ($t-ZrO_2$) in the mullite matrix. Changes such as a volumetric growth of ~ 4 % and shear stress of 0.16

(~ 7 %) take place during polymorphic conversion from $t-ZrO_2$ to monoclinic zirconia ($m-ZrO_2$). Such reversible polymorphic transformations absorb the diffusion energy of the crack formed on the material. Therefore, zirconia ceramics being doped with low amounts of additives makes the material, the tetragonal phase of which is the subject of discussion, important in terms of toughness [3, 18].

Consequently; parallel to the study carried out by Aydin in 2013, when the strength, elastic module, hardness and fracture toughness values are examined, it can be said that colemanite is an effective additive for mullite zirconia composites produced with different solids concentrations of slip cast zircon, kaolin and alumina mixture [18].

Compliance with ethical standards

Conflict of interest

The authors declared that they have no conflict of interests to this work.

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