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Research Article

The effect of mullite addition on wear properties of titania doped zirconia ceramics

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ARTICLE INFO

Article history:

Received xxx
Received in revised form xxx
Accepted xxx
Available online xxx

Keywords:

Wear, Zirconia, Titania, Mullite, Mechanical Alloying, Conventional Ceramic Production Route.

Doi: 10.24012/dumf.1053347

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ABSTRACT

In this study, whether there is a phase change in the zirconia-titania mixture at high sintering temperatures and the effect of mullite additive on the mechanical and especially wear properties of this mixture was investigated. We synthesized mullite and 8 mol % titania added zirconium dioxide (8 mol % titania - 92 mol % zirconia) powders by conventional ceramic production route. The mixtures were prepared by mechanical alloying method using zirconia ball mill in acetone environment. To synthesize mullite, Al_2O_3 and SiO_2 powders mixture was prepared with stoichiometric proportions and fired it in the air at 1600 ºC for 3 h. And the titania added zirconia composites were fired at 1300 ºC for 2 h. Thus, titania - zirconia and mullite composite phases were obtained and grinding and sieving processes were carried out. Then, mullite-free and 10% by weight mullite reinforced titanium oxide added zirconia mixtures were prepared by powder metallurgy method. The powders were pressed by uniaxial pressing after drying. The formed samples were sintered in a high temperature furnace in air conditions for 1 and 5 h at 1500 and 1600 ºC sintering temperatures. Finally, microstructure examinations of the composites with SEM, phase analysis with XRD, hardness, three-point bending and wear tests were performed. In addition, the results of water absorption, porosity and density from physical properties were investigated. It was founded that with titania and mullite adding to zirconia matrix, there was not define a new phase in the composite microstructure. But mullite additive increased the wear resistance, hardness and three-point bending strength of the titania-zirconia composites.

Introduction

Among ceramics, zirconia (ZrO_2) and its composites have become very popular for technological and many scientific studies because of their good mechanical properties, corrosion resistance, low thermal conductivities, higher temperature stabilities and higher chemical stabilities [1-3]. They are preferred as significant materials for refractory materials, high temperature furnaces, components that are resistant to wear, various cutting tools, dental studies and a lot of fields [1-4]. High-purity zirconia (ZrO_2) exhibits three polymorphs depending on temperature: monoclinic (m-ZrO_2) phase is stable at temperatures up to 1170 °C. After this temperature, the conversion from the monoclinic phase to the tetragonal phase begins and the tetragonal zirconia (t-ZrO_2) phase is stable up to 2370 °C temperatures. From this temperature to the melting temperature of 2680 °C, it is in the cubic zirconia phase (c-ZrO_2) [1,4]. Depending on the cooling processes, conversion from the t-ZrO_2 phase to the m-ZrO_2 phase takes place. Transformation is very important as it causes volumetric changes of around 3% to 5% and thus cracks. Due to preventing this transformation and stabilizing the zirconia, it is common to use stabilizers. Addition of stabilizers to zirconia, lowers temperature of the transformations, reduces volumetric growth or shrinking and blocks the polymorphic transformations. By using stabilizers, it is possible to make stable the high-temperature phases at low temperatures too [1-3]. Different stabilizers, such as, Al_2O_3 [4,5], CaO [6], CeO_2 [7,8], MgO [9], SiO_2 [10,11], TiO_2 [12,13], Y_2O_3 [14-17] and even a combination of them [18,19], stabilize and hold stable the zirconia in the t-ZrO_2 and/or c-ZrO_2 forms at low temperatures. It is possible to produce materials including only t-ZrO_2 or c-ZrO_2 or a mixture of these with m-ZrO_2 phases by adding different quantities of stabilizer. If less than sufficient stabilizing oxide is added, partially stabilized zirconia (PSZ) is obtained instead of fully stabilized zirconia. PSZ usually consists of two or more closely mixed phases. As a result of using stabilizers and obtaining fully or partially stabilized zirconia, could be achieved superb mechanical properties for example bending strength, hardness, fracture toughness [1,3,16].

Zirconia exhibits better mechanical properties than other ceramics. However, like all other ceramics, it is fragile and cannot be formed at room temperature. Therefore, it is desirable to increase the toughness of these materials. So, some energy absorbing mechanisms such as transformation toughening and fiber reinforcement are used in ceramic matrices [17]. In the ceramic - ceramic mixed structure

formation process, which is a method of increasing the fracture toughness, the strength and toughness are increased by adding ceramic whiskers, fibers or particles to the main phase. This method is based on creating a physical barrier to the progressive crack. With higher tensile strength than polycrystalline material, whiskers are a good barrier to propagation cracking. So, the fracture toughness of zirconium dioxide can be increased more by adding some secondary phases for example nano particles, nano sized fibers or nano sheets into zirconia matrix [20]. For the last decades, improvement of nanomaterials offers new alternatives to reinforce ceramic composites. Carbon nanotubes have attracted important caution as reinforcement materials because of their superior properties. However, at high temperatures, they are prone to react with oxide matrixes leading to reduction in some mechanical properties of carbon nanotubes and limited the reinforcing effect on the composites [21]. So, it is stated that incorporation of mullite which is another type of ceramic toughening method with its high temperature oxidation and corrosion resistance and the other superior properties, might be preferred [17]. In the literature, mullite ($3\ Al_2O_3.2SiO_2$) is described as the matchless stable middle crystalline phase for Al_2O_3-SiO_2 binary system, cost-friendly and exhibiting good refractory ability [22-25]. Mullite has received significant attention for technological applications because of its well properties like low coefficient of thermal expansion, high melting temperature, good resistance to creep, good chemical stability and satisfactory hardness [22-25]. In short, fracture toughness of zirconia can be advanced with mullite reinforcement as the secondary phase into the ZrO_2 matrix and so, the other mechanical properties can be improved too [17]. In addition, the temperature of sintering is also important, because of affecting some properties of ceramics through changing of the crystalline phases and microstructure [14,15,26]. Studies continue on the effect of different sintering temperatures on the microstructure and mechanical properties of mullite-zirconia ceramics [17].

Various studies have been carried out on the wear properties of ceramics. For example, Boyraz and Akkuş [27] investigated the wear properties of the samples they produced by sintering the mixtures they prepared with porcelain, aluminum titanate and mullite powders in different compositions and ratios at various temperatures and times. They reported that the wear rate of the samples increased too with increasing the wear time and load for all of the samples. Huang et al [28] stated that the hardness, porosity, density and bending strength of the samples have significant effects on the wear properties in their study to examine the wear properties of the composites they produced by adding mullite additives at 0-10 mole ratios to zirconia. They indicated that especially adhesive and abrasive wear were observed in the samples.

In this study, we synthesized mullite ($3Al_2O_3.2SiO_2$) and 8 mol % titanium dioxide doped zirconium dioxide powders by conventional ceramic production method. We examined whether there are phase differences in the TiO_2-ZrO_2 mixture depend on sintering temperatures and times and the effect of mullite on the physical, mechanical and microstructural properties of these mixtures.

Materials and Methods

Mullite and titania doped zirconia powders were produced by conventional ceramic production method in this study. All precursory powder materials were obtained from Company Eczacıbası (Al_2O_3 and SiO_2 powders), Refsan (TiO_2) and Chemicals of Handan Yaxiang Trading Co. (ZrO_2). The powders were mixed in acetone environment by mechanical alloying method. The powders were heated for 24 hours in oven at 110 ºC before and after mixing. Mullite ($3Al_2O_3.2SiO_2$) and 8 mol% titanium dioxide doped zirconium dioxide (TiO_2-ZrO_2) powders were synthetized by sintering from the prepared powders with stoichiometric ratios of Al_2O_3, SiO_2, TiO_2 and ZrO_2 powders after homogenized in ball mill. Mullite ($3Al_2O_3.2SiO_2$) was synthetized for 3h at 1600 ºC and 8 mol% titania doped zirconia (TiO_2-ZrO_2) composite powders were synthetized for 2 h at 1300 ºC. Thus, titania - zirconia and mullite composite phases were obtained and grinding and sieving processes were carried out. Then, mullite-free and 10% by weight mullite reinforced titanium dioxide added zirconium oxide composites were prepared with powder metallurgy technique (these mixtures were coded as TiZ00M and TiZ10M). The sample was coded as TiZ10M16005 (TiZ: Titania doped zirconia; 10M: 10% Mullite addition and 16005: 1600 °C sintering temperature and 5 hours sintering time). After the composite powders were milled for 24 h in acetone environment with zirconia ball mill, sieved and dried. Then, the composite mixtures were pressed to 56x12 mm sizes mold gap by uniaxial pressing machine at 200 MPa load. The pressed samples were sintered in a high temperature owen (Protherm) and in air conditions for 1-5 h sintering times and 1500-1600 ºC temperatures. The heating rate was 5 ºC/min. Then, with SEM, microstructure investigations, phase analysis with XRD, 3-point bending, hardness and wear tests, absorption of water, porosity, shrinkage and density results were examined on the composites.

The three-point flexural strength tests were executed with 0.5 mm/min (crosshead speed) in a Shimadzu brand tensile-compression device. For each sample, measurements were taken five times and their average were taken as the bending strength results of the samples. The strength calculations were made with the formula (1):

$$\sigma = \frac{3}{2} PL \,/\, (bh^2) \qquad (1)$$

(In (1) the letters mean that, P: maximum force, L: the distance of between supports, b: width of samples, h: height of samples).

After 180, 320, 600, 1200 and 2500 grit sanding process, polishing is done for each sample. With Vickers hardness tester that was Mitutoyo brand, the measurements of hardness were executed by 1 kg load for 10 seconds. For each sample, measurements were taken five times and their average were taken as the hardness results of the samples

[29,30]. The wear tests of samples were executed with Plint brand wear tester. For wear tests, steel discs were used. For each sample, wear tests were executed at 400 rpm rate, 5, 10 and 15 min wear durations and 50 N, 100 N and 150 N forces. The samples were weighted with a precision scale of 10^{-4} g. After the assigned wear times, the samples were scaled again and the wear amounts were calculated [31-33]. To determine the phases, XRD with Cu Kα radiation (Bruker AXS D8 Advance; 20kV-60kV, 6mA-80mA and θ = 10º-90º, 0.002º) was used. The phases of the samples seen in XRD patterns were defined with the Panalytical X'Pert program. The microstructural characterization of the samples was done with the Mira3XMU FE-SEM (Tescan, Czech Republic) brand scanning electron microscope machine and energy dispersion spectrum. The results were presented in various graphics and tables and some comments on these results were made.

Results and Discussion

Physical (shrinkage, water absorption, density and porosity tests) and mechanical (wear, 3-point bending and hardness) tests, SEM, EDS and XRD analysis results were included in this section. Calculations and measurements were repeated 5 times and arithmetic averages were taken.

Table 1. Physical test results of TiZ00M and TiZ10M samples.

Samples	Bulk Den.	R.D (%)	WA (%)	Por. (%)	Shr. (%)
TiZ00M15001	5.24	93.65	1.52	6.35	12.72
TiZ00M15005	5.43	97.08	0.85	2.92	13.10
TiZ00M16001	5.48	97.86	0.78	2.14	13.54
TiZ00M16005	5.39	96.36	1.01	3.64	13.51
TiZ10M15001	4.51	85.54	3.06	14.46	9.93
TiZ10M15005	4.79	90.85	1.48	9.15	11.78
TiZ10M16001	4.90	92.88	0.91	7.12	12.42
TiZ10M16005	4.92	93.38	0.86	6.62	12.60

The shrinkage, porosity, water absorption, relative density and bulk density results are shown in Table 1, and also Figure 1. In Figure 1, the relative density values were taken at the rate of 1/3 of the actual values for the graph.

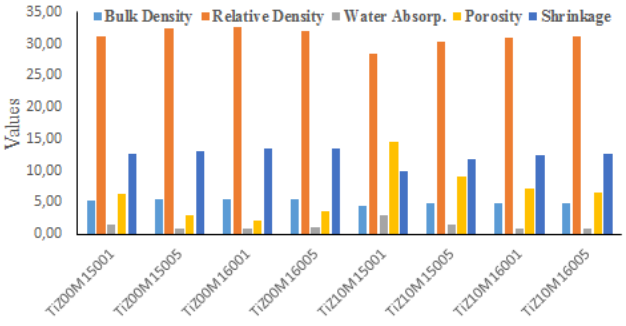


Figure 1. Physical test results graph of TiZ00M and TiZ10M samples.

When the results were examined, it was found that with increasing sintering temperature and time, the shrinkage values increased in general, and accordingly the water absorption and porosity values decreased; It is seen that experimental density and relative density values increase. It is thought that the decrease in the density value of the 16005 sample without mullite additives is caused by defects such as excessive grain growth and formation of large pores in the microstructure depending on the sintering time.

Table 2, Figure 2 and Figure 3 indicated hardness and 3-point bending strength values of TiZ00M and TiZ10M samples.

Table 2. Hardness and 3-point bending strength values of TiZ00M and TiZ10M samples.

Samples	Hardness (HV)	3-Point bending strength (MPa)
TiZ00M15001	159.72	15.87
TiZ00M15005	202.38	11.91
TiZ00M16001	275.13	11.12
TiZ00M16005	260.33	8.78
TiZ10M15001	171.53	17.37
TiZ10M15005	215.60	24.25
TiZ10M16001	280.21	37.26
TiZ10M16005	293.62	46.71

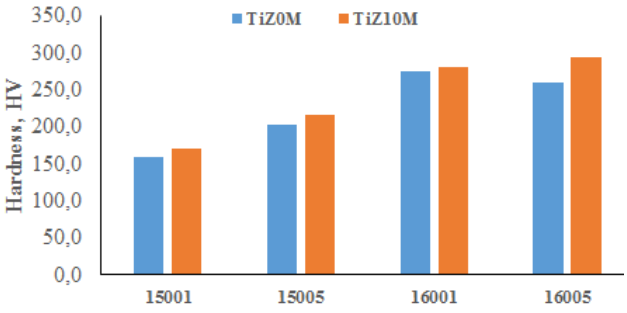


Figure 2. Micro hardness graph of samples.

When the results are examined, it is seen that the hardness values generally increase with increasing sintering temperature and time, and there is a decrease in the hardness value of the 16005 sample without mullite additives. It is thought that this situation is related to the decrease in density and therefore the defects that occur in the microstructure. In addition, it is seen that the hardness values of the mullite added samples are higher.

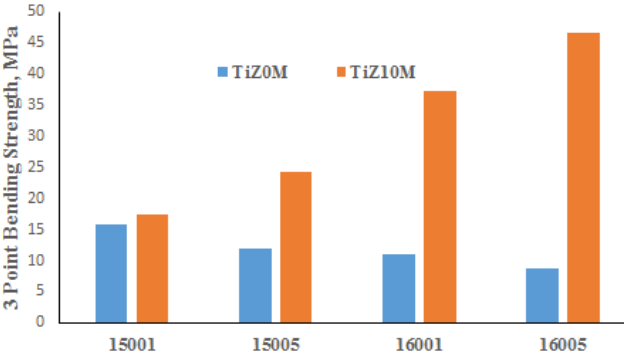


Figure 3. 3-Point bending strength graph of samples.

When the three-point bending results were examined, it was found that the three-point bending strength values decreased with increasing sintering temperature and time in mullite-free samples; on the other hand, it is seen that it increases in mullite added samples. In addition, it is seen that the three-point bending strength values of the mullite added samples are higher. This indicates that the mullite additive improves the microstructure and functions as a toughening mechanism.

The wear tests of samples were executed with Plint brand wear tester. For wear tests, steel discs were used. For each sample, wear tests were executed at 400 rpm rate, 5, 10 and 15 min wear durations and 50 N, 100 N, 150 N loads. The samples were weighed with a precision scale of 10^{-4} g. After the assigned wear times, the samples were scaled again and the wear amounts were calculated. Wear results are seen in Table 3, 4, 5 and Figure 4, 5, 6. In addition, graphs showing the load-dependent wear volume of 16005 samples are also presented in Figure 7.

Table 3. Wear results of TiZ00M and TiZ10M samples at 50 N load.

Samples	Wear Volume (mm^3), 50 N		
	5 min.	10 min.	15 min.
TiZ00M15001	7.54	16.13	25.82
TiZ00M15005	6.80	13.57	19.86
TiZ00M16001	3.63	5.33	9.71
TiZ00M16005	5.51	8.44	13.10
TiZ10M15001	7.12	15.41	20.21
TiZ10M15005	6.45	11.39	15.12
TiZ10M16001	3.21	5.14	9.17
TiZ10M16005	2.88	4.24	7.03

Table 4. Wear results of TiZ00M and TiZ10M samples at 100 N load.

Samples	Wear Volume (mm^3), 100 N		
	5 min.	10 min.	15 min.
TiZ00M15001	13.25	28.32	45.95
TiZ00M15005	10.86	18.36	32.25
TiZ00M16001	7.25	12.55	16.53
TiZ00M16005	8.41	14.41	19.14
TiZ10M15001	14.04	24.26	33.42
TiZ10M15005	10.12	17.81	26.56
TiZ10M16001	5.15	11.49	14.74
TiZ10M16005	4.18	6.75	10.63

Table 5. Wear results of TiZ00M and TiZ10M samples at 150 N load.

Samples	Wear Volume (mm^3), 150 N		
	5 min.	10 min.	15 min.
TiZ00M15001	19.85	38.54	65.33
TiZ00M15005	16.12	28.17	52.32
TiZ00M16001	10.64	19.58	30.53
TiZ00M16005	12.47	22.13	36.51
TiZ10M15001	18.31	27.46	43.21
TiZ10M15005	14.44	21.98	35.66
TiZ10M16001	9.19	15.71	21.38
TiZ10M16005	6.21	10.57	16.18

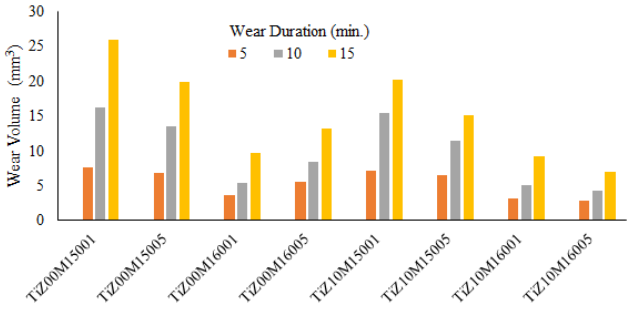


Figure 4. Wear test results graph for 50 N load.

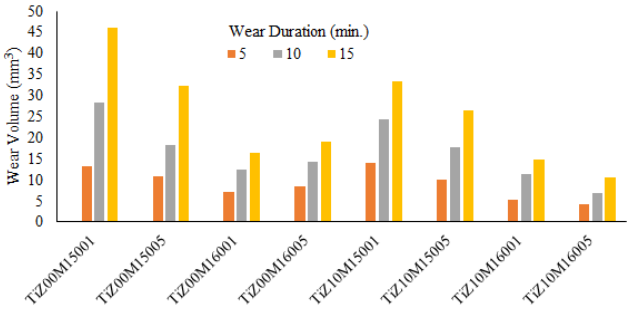


Figure 5. Wear test results graph for 100 N load.

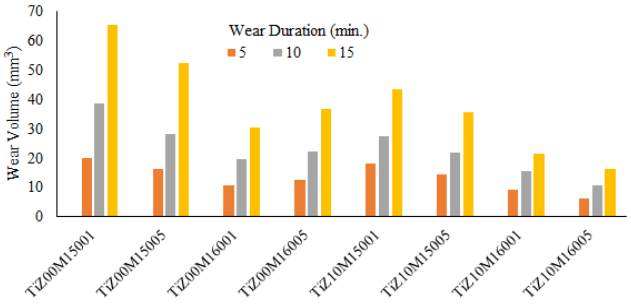


Figure 6. Wear test results graph for 150 N load.

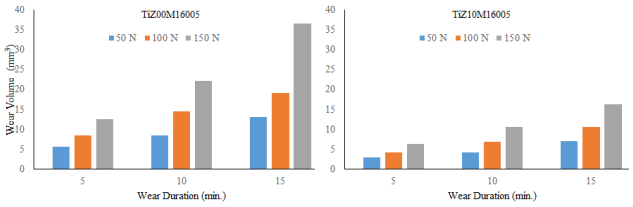


Figure 7. Wear results graphs of TiZ00M16005 and TiZ10M16005 samples.

When the wear test results are examined, it is seen that the wear volume values vary depending on the hardness, bending strength and wear time of the samples. In addition, it is seen that the wear volume values of the mullite-added samples are lower and therefore their wear resistance is higher. In general, it is understood that the wear amount of the samples increases as the wear time increases, the wear resistance of the samples with high hardness and bending strength is higher, and the mullite additive improves the wear resistance of these samples. Although adhesive type wear is observed in the samples in general, it has been observed that with the increase of the wear time in some samples, cracks occur on the worn surface and very small pieces break off and cause abrasive wear.

The phase changes in the sample structure depending on the sintering temperature and time of TiZ00M and TiZ10M samples were analyzed and the basic phases that emerged in the structure were shown in Figure 8 and Figure 9.

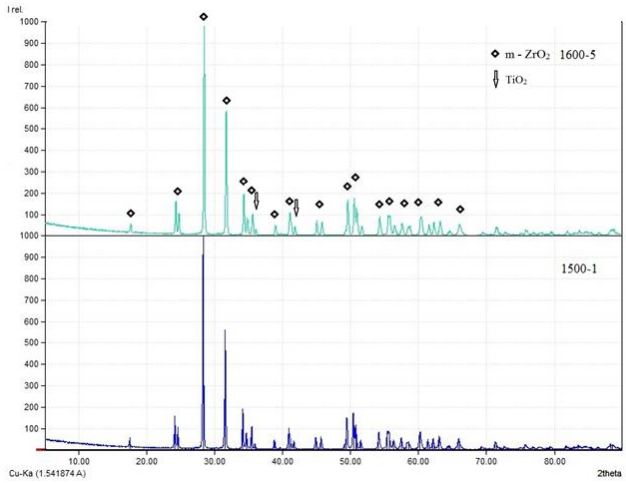


Figure 8. XRD patterns of TiZ00M samples.

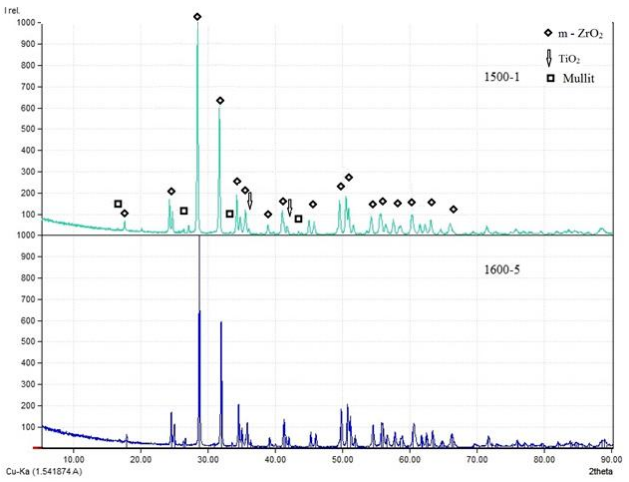


Figure 9. XRD patterns of TiZ10M samples.

As can be seen from Figure 8 and Figure 9, while m-ZrO_2 and TiO_2 phases are detected in samples without mullite additives, it is seen that there is a mullite phase in addition to these phases in samples with mullite additives. It is understood that different phases or t-ZrO_2 or c-ZrO_2 are not formed in the structure and the mullite additive improves the bending strength and other properties of the samples with the second phase toughening mechanism.

The microstructure image of TiZ00M16005 and TiZ10M16005 samples is given in Figure 10.

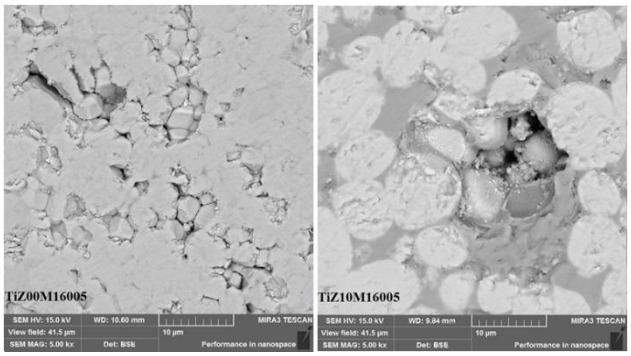
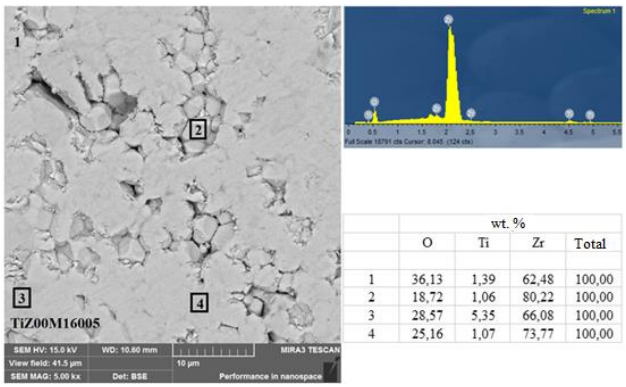


Figure 10. SEM images of TiZ00M16005 and TiZ10M16005 samples.

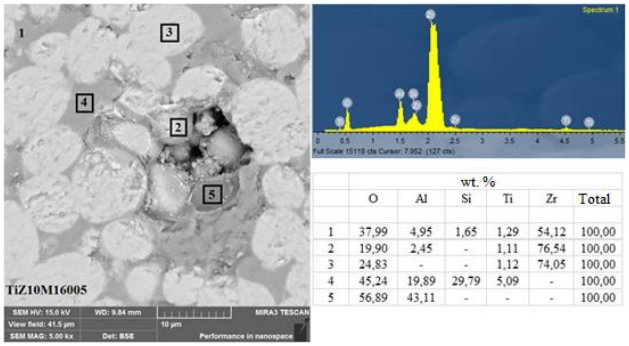
As can be seen from the SEM image given in Figure 10, the grain boundaries of the mullite-free sample appear to be separated from each other and the deep slit appearances show that there are cracks on the surface and internal structure of these samples, which will adversely affect the microstructure and mechanical properties. On the other hand, since a more homogeneous structure is obtained in the mullite-added sample despite the significantly larger grain size, it can be predicted that the mullite additive can improve the mechanical properties. As a matter of fact, in the experimental results, the hardness and bending strength values of the TiZ10M16005 sample were found to be higher than the hardness and bending strength values of the TiZ00M16005 sample.

Elemental analyzes of TiZ00M16005 and TiZ10M16005 samples with EDS are given in Figure 11 and Figure 12.



	wt. %			
	O	Ti	Zr	Total
1	36,13	1,39	62,48	100,00
2	18,72	1,06	80,22	100,00
3	28,57	5,35	66,08	100,00
4	25,16	1,07	73,77	100,00

Figure 11. EDS analyses of TiZ00M16005 sample.



	wt. %					
	O	Al	Si	Ti	Zr	Total
1	37,99	4,95	1,65	1,29	54,12	100,00
2	19,90	2,45	-	1,11	76,54	100,00
3	24,83	-	-	1,12	74,05	100,00
4	45,24	19,89	29,79	5,09	-	100,00
5	56,89	43,11	-	-	-	100,00

Figure 12. EDS analyses of TiZ10M16005 sample.

According to the EDS analyzes given in Figure 11 and Figure 12, the evaluation of the EDS analysis results on the TiZ00M16005 and TiZ10M16005 samples was made from general (1) and parts 2, 3, 4, 5. It has been observed that the results of the EDS elemental analysis made from the general field survey (1) and other parts are compatible with the contribution rates and XRD results made to the samples.

Conclusion

In this study, utilization of mullite in the manufacturing of titania doped zirconia and the effect of mullite addition on wear and other some properties of these ceramics were investigated.

With increasing sintering temperature and time in TiZ00M coded samples, generally shrinkage, experimental and relative density, hardness values and wear resistance increased; It was observed that water absorption, porosity and three-point flexural strength values decreased. Shrinkage, experimental and relative density, hardness values and wear resistance values of the 16005 sample without mullite additives were less than the 16001 sample. It is thought that this is due to defects such as excessive grain growth and formation of large pores in the microstructure depending on the sintering time.

In TiZ10M coded samples, with increasing sintering temperature and time, generally shrinkage, experimental and relative density, hardness, three-point bending strength values and wear resistance increased; It was observed that the water absorption and porosity values decreased. Whereas the mullite additive decreased the shrinkage, experimental density and relative density values of the samples; It increased the water absorption, porosity, hardness and three-point bending strength values and wear resistance.

While m-ZrO_2 and TiO_2 phases were detected in the samples without mullite additives, it was determined that there was mullite phase too in addition to these phases in the samples with mullite additives. The absence of t-ZrO_2, c-ZrO_2 or other phases in the structure shows that the mullite additive improves the microstructure and improves the bending strength and other properties of the samples with the secondary phase toughening mechanism.

Ethics committee approval and conflict of interest statement

There is no need to obtain permission from the ethics committee for this article prepared.

There is no conflict of interest with any person in this article prepared. All authors declare that they have no conflict of interest.

Authors' Contributions

All authors contributed equally.

Acknowledgement

This work is supported by the Scientific Research Project Fund of Sivas Cumhuriyet University under the project number M-767. Authors would like to acknowledge Scientific Research Project Fund of Sivas Cumhuriyet University.

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