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# The Effect of Mullite Addition on Wear Properties of SiO2 Doped ZrO2 Ceramics

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**ABSTRACT**

Mullite ($3Al_2O_3.2SiO_2$) and 10 mol% silica added zirconia (10 mol% $SiO_2$ - 90 mol% $ZrO_2$) ceramic powders were synthesized by conventional ceramic processing 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 silica added zirconia composites were fired at 1300 ºC for 2 h. Thus, silica - zirconia and mullite composite phases were obtained and milling and sieving processes were carried out. Then, mullite-free and 10% by weight mullite reinforced silicon oxide added zirconia mixtures were prepared by powder metallurgy method. The powders were compacted by uniaxial pressing. 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 and the effect of mullite additive on the mechanical and especially wear properties of this mixture were investigated.

***Keywords:*** Wear, Zirconia, Mullite, Silica, Characterization.

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## 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]. 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. High-purity zirconia ($ZrO_2$) exhibits three polymorphs depending on temperature: monoclinic phase is stable up to about 1170 °C. After this temperature, the conversion from the monoclinic phase to the tetragonal phase begins and the tetragonal phase is stable up to 2370 °C. From this temperature to the melting temperature of 2680 °C, it is in the cubic zirconia phase [2]. 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 [3]. Different stabilizers, such as, $Al_2O_3$ [4], CaO [5], $CeO_2$ [6,7], MgO [8–10], $SiO_2$ [11-13], $TiO_2$ [14,15], $Y_2O_3$ [16,17] and even a combination of them [18,19], stabilize and hold stable the $ZrO_2$ in the tetragonal and/or cubic forms at room temperature. 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 [20,21]. 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 [22-25].

Powder metallurgy method is frequently used in the shaping and sintering processes of powder matrix alloy and reinforcement materials after mixing. In order to obtain a good microstructure, it is very important to distribute all the particles homogeneously within the structure. So, mechanical alloying method has an important place in powder metallurgy. Mechanical alloying is a solid-state powder production and synthesis method that enables the production of homogeneous materials. Mechanical alloying is the process of powder mixing and grinding in a dry or aqueous environment (acetone, alcohol, water, etc. is used as liquid) used to produce composite powders with small crystal grains and controlled microstructures. This method is one of the common techniques for producing ceramic powders by mechanical methods [26].

In the literature, mullite (3 $Al_2O_3$.2$SiO_2$) is described as the matchless stable middle crystalline phase for $Al_2O_3$-$SiO_2$ binary system, cost-friendly and exhibiting good refractory ability [27]. 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 [28]. 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 [22]. In addition, the temperature of sintering is also important, because of affecting some properties of ceramics through changing of the crystalline phases and microstructure [29].

Aguilar, D. H. et al., examined the crystallization properties by adding 2 - 80% $SiO_2$ into $ZrO_2$ matrix and stated that m-$ZrO_2$, t-$ZrO_2$ and $ZrSiO_4$ phases were formed in different proportions of compositions [11]. Vasanthavel, S. et al., mixing $SiO_2$ with $ZrO_2$ in varying proportions by sol-gel method, examined the effect of $SiO_2$ addition on phase behavior and stated that m-$ZrO_2$, t-$ZrO_2$ and c-$ZrO_2$ phases were formed in the structure [12]. Again, Vasanthavel, S. et al. prepared $ZrO_2$-$SiO_2$-Dysprosium mixtures in different proportions by sol-gel method and stated that m-$ZrO_2$, t-$ZrO_2$, c-$ZrO_2$, c-$SiO_2$, $ZrSiO_4$ phases were formed in the structure [13].

Also the wear properties of zirconia and mullite ceramics are investigated in many studies. In the study conducted by Akkuş and Boyraz [30], the wear properties of the samples prepared and produced with CaO, MgO and $ZrO_2$ oxide powders in different compositions and ratios were investigated. They reported that the wear values obtained by applying different wear loads and wear times to the samples increase in parallel with the increase in the applied load and wear time. Huang et al [31] stated that hardness, porosity, density and bending strength of the samples had important effects on the wear properties in their study to examine the wear properties of the composites produced by adding mullite additives at 0-10 mole ratios to zirconia (3Y-TZP). Especially adhesive and abrasive wear were observed in the samples.

In this study, mullite (3$Al_2O_3$.2$SiO_2$) and 10 mol % silica doped zirconia ($SiO_2$-$ZrO_2$) ceramic powders were synthesized by conventional ceramic production processing route. Then, the effect of mullite additive on the properties of this mixture was investigated.

## Materials and Methods

Mullite (3$Al_2O_3$.2$SiO_2$) and silica doped zirconia ($SiO_2$-$ZrO_2$) ceramic powders were produced by conventional ceramic production processing route in this study. All precursory powder materials were obtained from Company Eczacıbası ($Al_2O_3$ and $SiO_2$ powders) 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 $^0$C before and after mixing. Mullite (3$Al_2O_3$.2$SiO_2$) and 10 mol% silica doped zirconia ($SiO_2$-$ZrO_2$) powders were synthetized by sintering from the powders prepared with stoichiometric ratios of Al2O3, SiO2 and ZrO2 powders after homogenized in ball mill. Mullite (3$Al_2O_3$.2$SiO_2$) was synthetized for 3h at 1600 $^o$C and 10 mol% silica doped zirconia ($SiO_2$-$ZrO_2$) composite powders were synthetized for 2 h at 1300 $^o$C. Thus, silica - zirconia and mullite composite phases were obtained and milling and sieving processes were carried out. Then, mullite-free and 10% by weight mullite reinforced silicon oxide added zirconium oxide composites were prepared by powder metallurgy technique (named SiZ00M and SiZ10M respectively). The sample was coded as SiZ10M16005 (SiZ: Silica doped zirconia; 10M: 10% by weight 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 furnace (Protherm™ Furnace) and in air conditions for 1-5 h sintering times and 1500-1600 $^o$C temperatures. The heating rate was 5 $^o$C/min. Then, microstructure investigations with SEM, phase analysis with XRD, the hardness, 3-point bending and wear tests and physical properties that are water absorption, porosity, shrinkage and density results were examined on the composites.

The three-point flexural strength tests were executed with crosshead speed of 0.5 mm/min 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):

$$\sigma = 3/2 * P * L / (b * h^2) \tag{1}$$

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

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 [32-35]. 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 forces. The samples were measured with a precision scale of $10^{-4}$ g. After the assigned wear times, the samples were scaled again and the wear amounts were calculated [30,36,37]. To determine the phases, XRD with Cu Kα radiation (Bruker AXS D8 Advance; 20kV-60kV, 6mA-80mA and θ = 10°-90°, 0.002°) 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.

Physical (shrinkage, water absorption, density and porosity tests) and mechanical (hardness, 3-point bending and wear) tests, SEM, EDS and XRD analysis results were included in this section. Calculations and measurements were repeated 5 times and arithmetic averages were taken. The shrinkage, porosity, water absorption, relative density and bulk density results are shown in Table 1, and also Fig. 1. In Fig. 1, the relative density values were taken at the rate of 1/3 of the actual values for the graph.

Table 1. Physical test results of SiZ00M and SiZ10M samples

| Samples | Bulk density ($g/cm^3$) | Relative density (%) | Water absorption (%) | Porosity (%) | Shrinkage (%) |
|---|---|---|---|---|---|
| SiZ00M15001 | 4,04 | 75,15 | 6,56 | 24,85 | 8,58 |
| SiZ00M15005 | 4,40 | 81,78 | 4,40 | 18,22 | 10,68 |
| SiZ00M16001 | 4,85 | 90,11 | 2,16 | 9,89 | 13,54 |
| SiZ00M16005 | 5,13 | 95,34 | 1,08 | 5,66 | 14,98 |
| SiZ10M15001 | 3,60 | 70,53 | 8,26 | 29,47 | 6,28 |
| SiZ10M15005 | 3,76 | 73,69 | 6,93 | 26,31 | 7,76 |
| SiZ10M16001 | 4,57 | 89,55 | 3,02 | 10,45 | 12,85 |
| SiZ10M16005 | 4,68 | 91,69 | 1,97 | 8,31 | 14,20 |

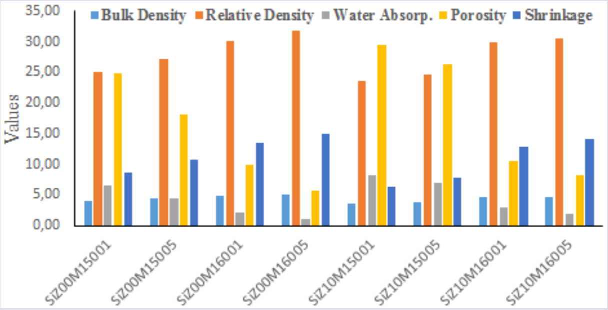


Figure 1. Physical test results graph of SiZ00M and SiZ10M samples.

When the results were examined, it was found that the shrinkage values increased with increasing sintering temperature and time in all samples, and accordingly the water absorption and porosity values decreased; it is seen that the experimental density and relative density values increase. In addition, it is seen that shrinkage, experimental density and relative density values are lower, water absorption and porosity values are higher in mullite added samples [36,37].

Table 2, Figure 2 and Figure 3 indicated the hardness and three-point flexural or bending strength results of SiZ00M and SiZ10M samples.

According to the results, it is seen that the hardness values for all samples increase with increasing sintering temperature and time. In addition, in the samples coded as 1500, the hardness values of the samples with mullite additives are lower than the samples without additives.

Table 2. Hardness and 3-point bending strength values of SiZ00M and SiZ10M samples

| Samples | Hardness (HV) | 3-Point bending strength (MPa) |
|---|---|---|
| SiZ00M15001 | 166,80 | 50,374 |
| SiZ00M15005 | 197,70 | 52,565 |
| SiZ00M16001 | 297,10 | 55,527 |
| SiZ00M16005 | 375,53 | 62,268 |
| SiZ10M15001 | 140,73 | 42,375 |
| SiZ10M15005 | 172,43 | 44,842 |
| SiZ10M16001 | 395,25 | 48,394 |
| SiZ10M16005 | 425,56 | 50,056 |

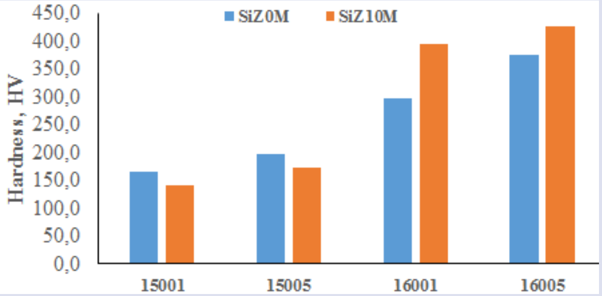


Figure 2. Hardness graph of samples.

In the samples coded as 1600, it is seen that the hardness values of the samples with mullite additives are higher than the samples without additives. We think that this is a result of possible phase changes in the microstructure.

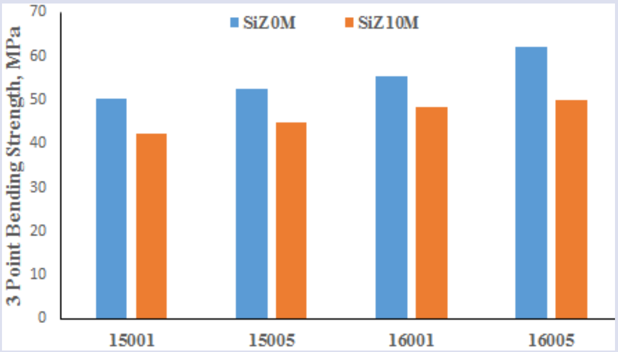


Figure 3. 3-Point bending strength graph of samples.

When we look for the flexural strength results, we see that the three-point flexural strength values increase with increasing sintering temperature and time in all samples, and the three-point flexural strength values of the mullite-added samples are lower than the un-doped samples.

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 forces. The samples were measured 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 shown in Table 3, 4, 5 and Figure 4, 5, 6. In addition, graphs showing the load-dependent wear volume of the samples coded as 16005 are also presented in Figure 7.

Table 3. Wear results of SiZ00M and SiZ10M samples at 50 N load

| Samples | Wear Volume (mm$^3$), 50 N | | |
|---|---|---|---|
| | **5 min.** | **10 min.** | **15 min.** |
| SiZ00M15001 | 3,99 | 8,68 | 16,77 |
| SiZ00M15005 | 3,53 | 8,27 | 15,88 |
| SiZ00M16001 | 2,40 | 5,36 | 9,33 |
| SiZ00M16005 | 1,72 | 4,15 | 8,23 |
| SiZ10M15001 | 5,35 | 10,86 | 18,70 |
| SiZ10M15005 | 4,99 | 9,22 | 16,55 |
| SiZ10M16001 | 1,51 | 6,09 | 9,86 |
| SiZ10M16005 | 1,28 | 2,89 | 4,65 |

Table 4. Wear results of SiZ00M and SiZ10M samples at 100 N load

| Samples | Wear Volume (mm$^3$), 100 N | | |
|---|---|---|---|
| | **5 min.** | **10 min.** | **15 min.** |
| SiZ00M15001 | 5,24 | 12,82 | 18,69 |
| SiZ00M15005 | 3,79 | 11,15 | 17,25 |
| SiZ00M16001 | 2,76 | 6,21 | 10,21 |
| SiZ00M16005 | 1,99 | 4,87 | 8,79 |
| SiZ10M15001 | 8,57 | 15,25 | 21,58 |
| SiZ10M15005 | 7,96 | 13,75 | 19,57 |
| SiZ10M16001 | 1,93 | 3,57 | 5,58 |
| SiZ10M16005 | 1,56 | 3,18 | 5,12 |

Table 5. Wear results of SiZ00M and SiZ10M samples at 150 N load

| Samples | Wear Volume (mm$^3$), 150 N | | |
|---|---|---|---|
| | ***5 min.*** | ***10 min.*** | ***15 min.*** |
| SiZ00M15001 | 12,56 | 29,58 | 43,92 |
| SiZ00M15005 | 7,68 | 11,45 | 28,56 |
| SiZ00M16001 | 3,27 | 7,01 | 11,96 |
| SiZ00M16005 | 2,25 | 5,50 | 9,42 |
| SiZ10M15001 | 15,74 | 31,26 | 45,21 |
| SiZ10M15005 | 13,78 | 29,16 | 38,76 |
| SiZ10M16001 | 2,62 | 3,70 | 6,00 |
| SiZ10M16005 | 2,13 | 3,57 | 5,75 |

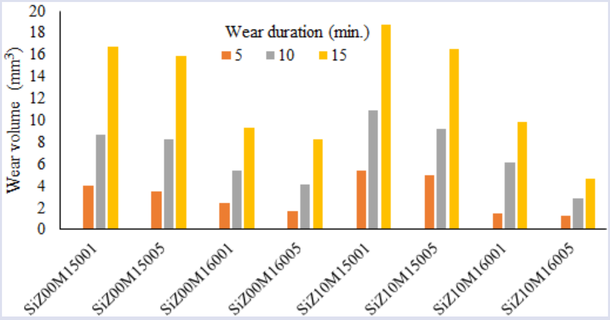


Figure 4. Wear test results graph for 50 N load.

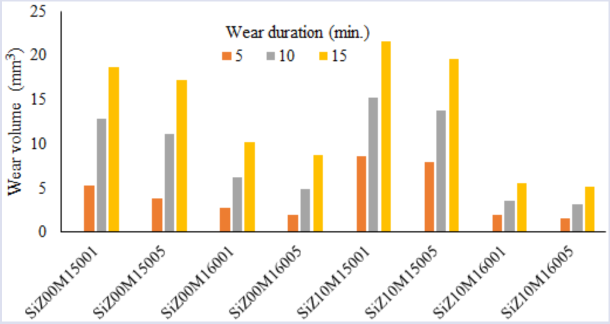


Figure 5. Wear test results graph for 100 N load.

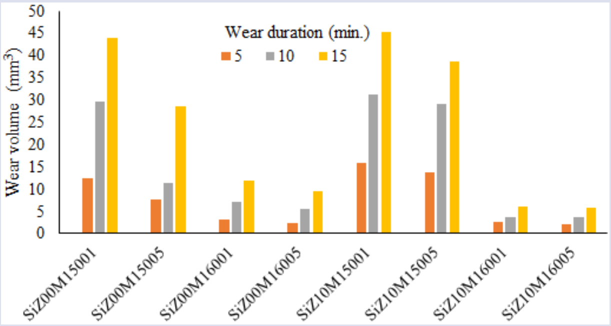


Figure 6. Wear test results graph for 150 N load.

When the results given in the tables are examined, it is seen that the wear resistances increase and the wear volume values decrease with increasing sintering temperature and time in all samples.

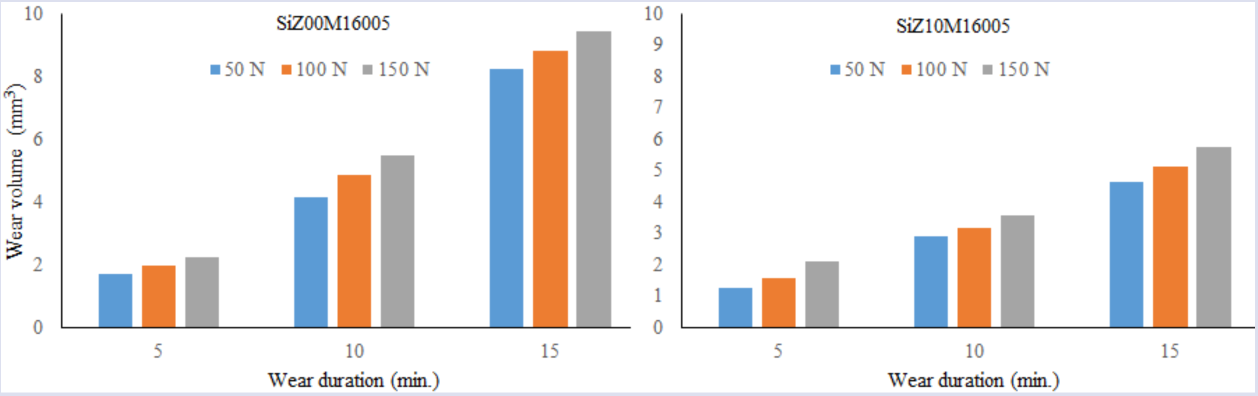


Figure 7. Wear results graphs of SiZ00M16005 and SiZ10M16005 samples.

In addition, while the wear resistance of the samples with mullite additive coded as 1500 is lower than the samples without additives; In the samples coded as 1600, it is seen that the wear resistance of the samples with mullite additive is higher than the samples without additives. We think that this is related to hardness values. Although adhesive type wear is observed in the samples in general, it has been observed that with the increase in 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 SiZ00M and SiZ10M samples were analyzed and the basic phases that emerged in the structure were shown in Figure 8 and Figure 9.

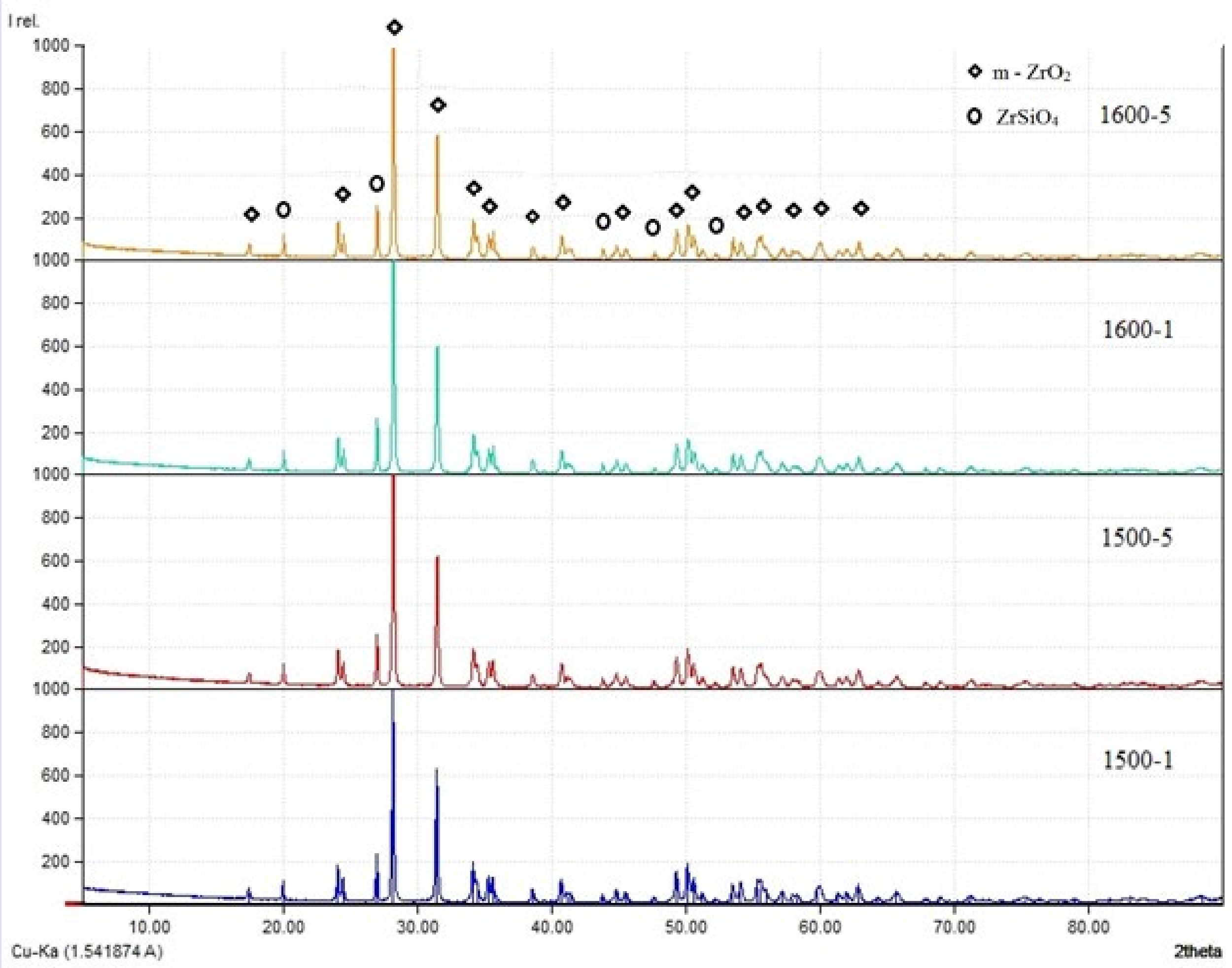


Figure 8. XRD patterns of SiZ00M samples.

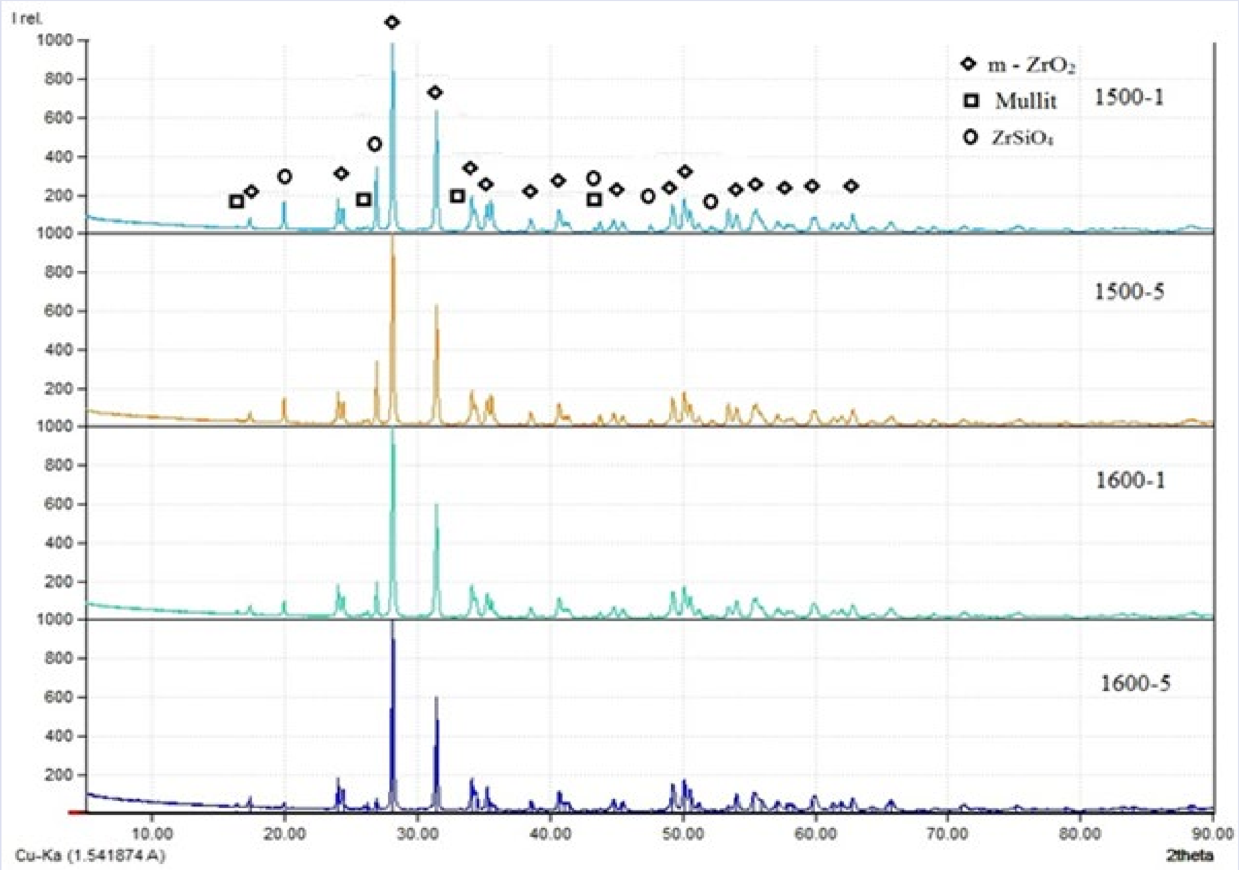


Figure 9. XRD patterns of SiZ10M samples.

As can be seen from Figure 8 and Figure 9, while m-$ZrO_2$ and $ZrSiO_4$ phases are detected in the samples without mullite additives, it is seen that there is a mullite phase in addition to these phases in the samples with mullite additives. When the XRD patterns given in Figure 8 and Figure 9 are compared, it is understood that the higher hardness value of the samples coded as 1600 mullite-added samples compared to the un-doped samples coded as 1600, depends on the zircon ($ZrSiO_4$) phase in the structure. The fact that the zircon phase of samples coded as 1600 with mullite additives was less than the samples without additives increased the hardness values of these samples.

The microstructure image of SiZ00M16005 and SiZ10M16005 samples is given in Figure 10.

As can be seen from the SEM image given in Figure 10, the amount and size of the pores on the surface decreased with the addition of mullite and a more homogeneous surface was obtained. It can be predicted that this will have a positive effect on the hardness values of the samples and may increase the hardness value of the mullite added sample.

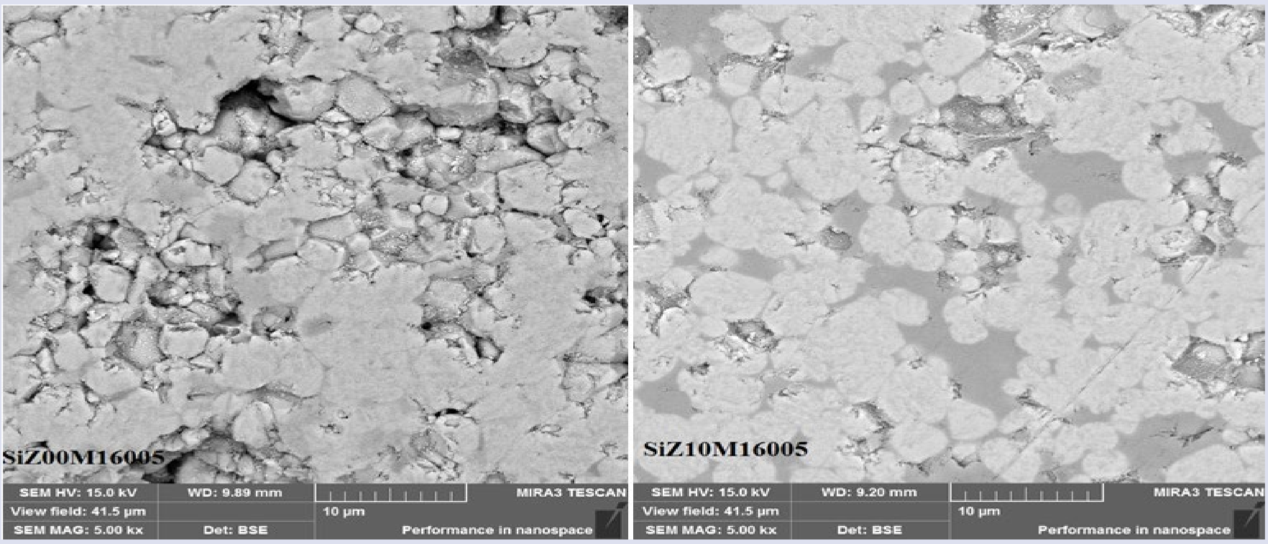
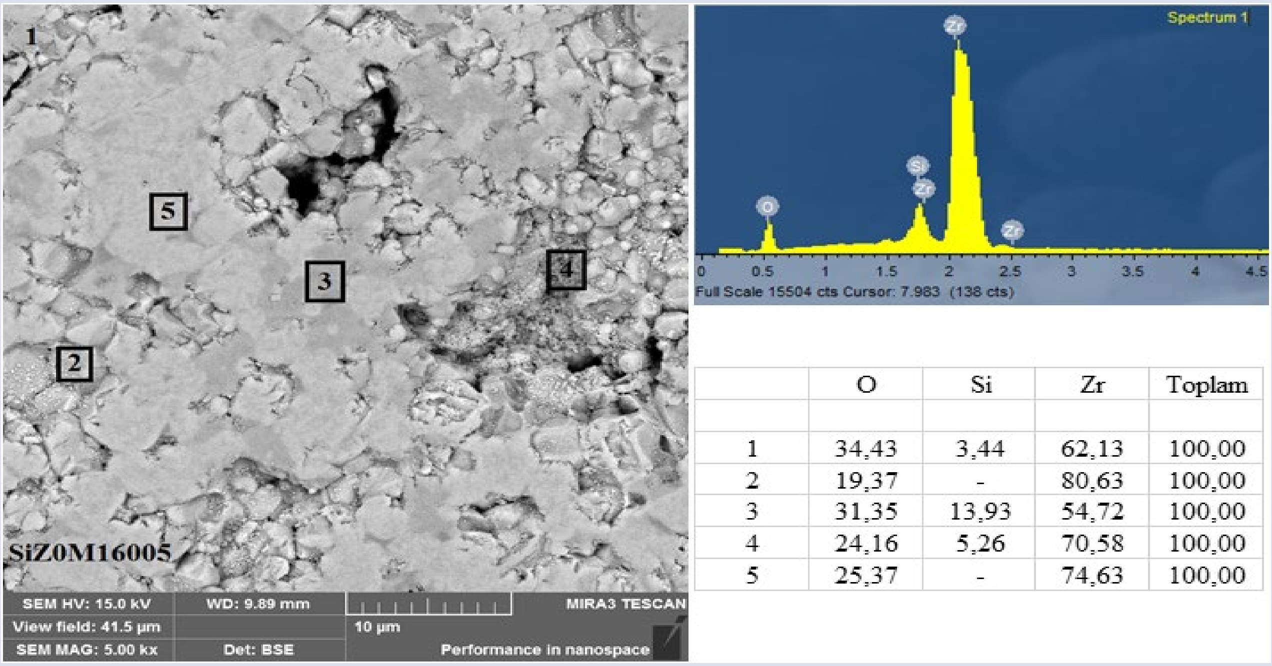


Figure 10. SEM images of SiZ00M16005 and SiZ10M16005 samples.

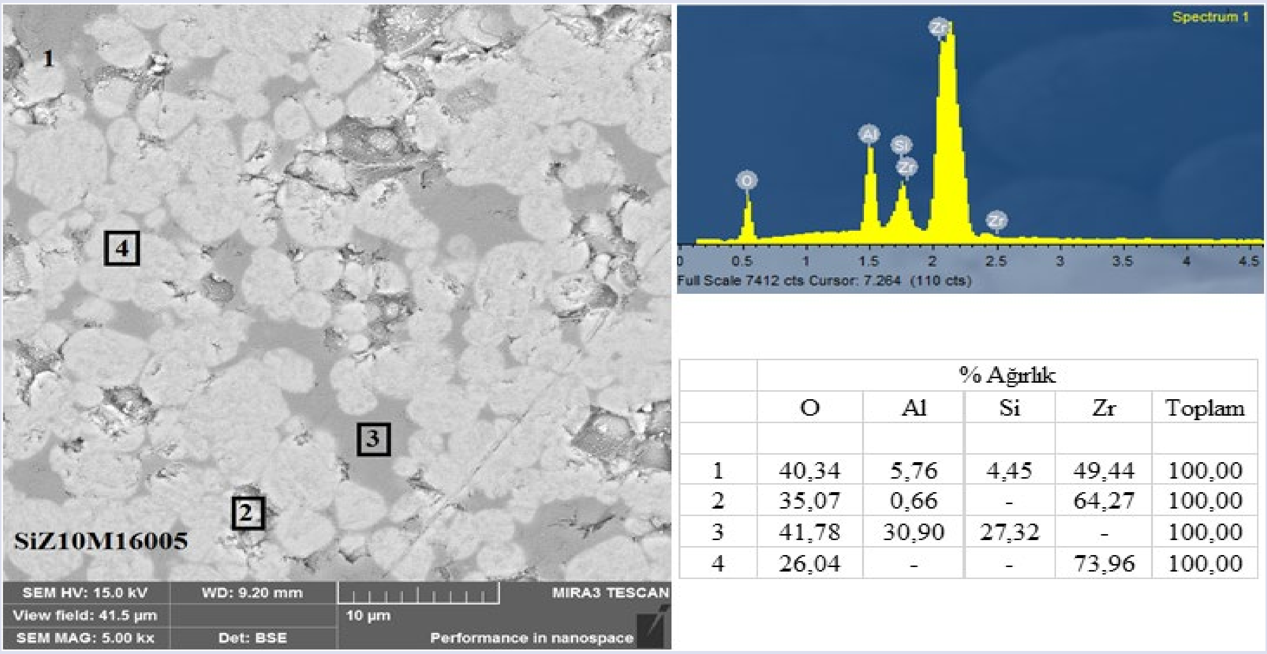
As a matter of fact, in the experimental results, the hardness value of the SiZ10M16005 sample was found to be higher than the hardness value of the SiZ00M16005 sample.

Elemental analyzes of SiZ00M16005 and SiZ10M16005 samples with EDS are given in Figure 11 and Figure 12.



| | O | Si | Zr | Toplam |
|---|---|---|---|---|
| | | | | |
| 1 | 34,43 | 3,44 | 62,13 | 100,00 |
| 2 | 19,37 | - | 80,63 | 100,00 |
| 3 | 31,35 | 13,93 | 54,72 | 100,00 |
| 4 | 24,16 | 5,26 | 70,58 | 100,00 |
| 5 | 25,37 | - | 74,63 | 100,00 |

Figure 11. EDS analyses of SiZ00M16005 samples.



| | % Ağırlık | | | | |
|---|---|---|---|---|---|
| | O | Al | Si | Zr | Toplam |
| | | | | | |
| 1 | 40,34 | 5,76 | 4,45 | 49,44 | 100,00 |
| 2 | 35,07 | 0,66 | - | 64,27 | 100,00 |
| 3 | 41,78 | 30,90 | 27,32 | - | 100,00 |
| 4 | 26,04 | - | - | 73,96 | 100,00 |

Figure 12. EDS analyses of SiZ10M16005 samples.

According to the EDS analyzes given in Figure 11 and Figure 12, the evaluation of the EDS analysis results on the SiZ00M16005 and SiZ10M16005 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 our study, utilization of mullite in the production of silica doped zirconia was explored. The effect of $SiO_2$ and mullite contribution to $ZrO_2$ main matrix was evaluated.

In all SiZ00M and SiZ10M coded samples, shrinkage, experimental density, relative density, hardness values, three-point bending strength values and wear resistance increase with increasing sintering temperature and time.

It is observed that the water absorption and porosity values decrease.

While the mullite additive reduces the shrinkage, experimental density, relative density and three-point bending strength values of the samples; It is observed that the water absorption and porosity values increase. The reason for this is that the density value of the mullite additive is lower than the $ZrO_2$ - $SiO_2$ composite and it is more stable in terms of shrinkage value at high temperatures compared to this composite. Therefore, in mullite added samples, shrinkage, experimental density and relative density are lower, whereas water absorption and porosity values are higher.

The hardness values and wear resistance of the mullite added samples in 1500 samples are lower than the mullite-free samples. But in the samples coded as 1600, it is seen that the hardness values and wear resistance of the samples with mullite additives are higher than the samples without additives. When the XRD patterns are compared, it is understood that the higher hardness values of the samples coded as 1600 mullite-added samples compared to the samples coded as 1600 undoped samples, depend on the $ZrSiO_4$ (zircon) phase in the structure. The fact that the amount of zircon phase in samples coded as 1600 with mullite additive is less compared to the samples without additive, increases the hardness values of these samples.

While m-$ZrO_2$ and $ZrSiO_4$ phases are detected in the samples without mullite additives, it is determined that there was a mullite phase in addition to these phases in the samples with mullite additives.

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## Conflicts of interest

All authors declare that they have no conflict of interest.

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