

Investigation of wear properties of CaO, MgO added stabilized zirconia ceramics produced by different pressing methods

Ahmet Akkus^a and Tahsin Boyraz^b

^aDepartment of Mechanical Engineering, Cumhuriyet University, Sivas, Turkey

^bDepartment of Metallurgical and Materials Engineering, Cumhuriyet University, Turkey

Stabilized zirconia has superior high temperature properties such as high tolerance for thermal shock, low thermal conductivity, mechanical properties, elevated melting point, good phase stability and excellent oxidation resistance. The present paper describes the fabrication and wear behaviour of 8 mole% CaO, 4 mole % CaO-4 mole% MgO and 8 mole% MgO - stabilized zirconia (ZrO₂) based composites fabricated by the conventional ceramic production process. CaO/MgO added stabilized zirconia ceramics were fabricated by using a combined method of ball milling, cold press-cold isostatic pressing and sintering. The powder blends containing different amounts of % 8 mole CaO/MgO were wet-milled for 1 hr in a vibratory ball-mill using zirconia vial and balls. After drying, powders were compacted to cylindrical preforms with a diameter of 12.7 mm by uniaxial pressing at 300 MPa and by cold isostatic pressing at 300 MPa. The green compacts were sintered at 1600 °C for 3 hrs under air. Phase and microstructural characterizations of the sintered materials were carried out by using X-ray diffraction technique (XRD) and scanning electron microscope (SEM). The Micro Vickers hardness testing was performed using the Shimadzu HMV-MIII hardness tester. Plint brand abrasion tester was used for the abrasion tests of ceramics. As a result, it has been observed that the hardness is increased by increasing the amount of MgO and the ceramics produced by cold isostatic pressing are harder.

Key words: Stabilized zirconia, Characterization, Hardness, Wear.

Introduction

Zirconia plays an important role in the development of high-performance ceramics subjected to mechanical/thermal stress, such as in the adiabatic diesel engine, cutting tools, wear-resistant structural components, high-temperature components for nozzles, extrusion dies [1]. Pure zirconia in equilibrium state exists in three polymorphic form: the monoclinic phase is stable up to 1170 °C where it transforms into the tetragonal phase, which is stable up to 2370 °C, and the cubic phase exists up to the melting point at 2680 °C [2,3]. Obtaining stable sintered zirconia ceramic products is difficult because of the large volume change accompanying the transition from tetragonal to monoclinic (about 9%). Stabilization of the tetragonal polymorph of zirconia over wider range of temperatures is accomplished by substitution of some of the Zr⁴⁺ ions in the crystal lattice with slightly larger ions, e.g., those of Ca²⁺ and Mg²⁺ (ionic radius of 1,0 and 0,72 Å). The resulting doped zirconia materials are termed stabilized zirconia. The superior mechanical such as hardness, strength and fracture toughness, thermal such as high tolerance for thermal shock, low thermal conductivity, electrical and

chemical properties of calcia and magnesia stabilized zirconia have recently become the focus of growing scientific and technological interest [4, 5].

Recently, considerable attention has been applied to stabilized zirconia ceramics, because of their special mechanical, chemical and thermal properties. For the application of high capacity attrition mills for fine grinding and homogenization millings, beads with a large surface and high energy input are required. High density, good sinterability and improved fracture toughness make stabilized zirconia very attractive as wear resistant materials. However, their potential applications are still limited by the lack of reliable data concerning the wear behaviour and mechanism. Although a number of studies have been carried out to investigate the wear characteristics in a variety of conditions, most of them deal with metal/ceramic contacts and ceramic/ceramic friction is not well known as yet. The wear behaviour of zirconia ceramics seems to be very sensitive to the structure of the material, and to the test parameters, such as temperature, environment and sliding speed [6]. Scott^{*} studied the wear behaviour of MgPSZ and suggested that their particular wear resistance depended on the amount of transformable phase in the sample. Calcium zirconate (CaZrO₃) is a ceramic oxide of the perovskite family that presents several properties of interest as structural as well as functional material [7-11].

*Corresponding author:
Tel : +90-346-219-11-64
Fax: +90-346-219-11-65
E-mail: aakkus@cumhuriyet.edu.tr

This study describes the fabrication and wear behaviour of 8 mole% calcia (CaO), 4 mole % CaO-4 mole% MgO and 8 mole% magnesia (MgO)-stabilized zirconia (ZrO_2) based composites fabricated by the conventional ceramic production process.

Experimental

In this study, zirconia based ceramic powders were used. Mixtures of zirconia (ZrO_2 Serp, France), magnesia (MgO Merck, Germany) and calcia (CaO Merck, Germany) of different compositions were prepared. 8 mole% calcia (CaO), 4 mole % CaO-4 mole% MgO and 8 mole% magnesia (MgO)-stabilized zirconia (ZrO_2) based composites fabricated by the conventional ceramic production process. CaO/MgO added stabilized zirconia ceramics were fabricated by using a combined method of ball milling, cold pressing (CP)-cold isostatic pressing (CIP) and sintering. These mixtures were prepared by mechanical alloying method and used zirconia ball mill to homogenize the blend with acetone as medium. The powders were dried in oven at 110 °C for 24 hrs before mixing. After drying, powders were compacted to cylindrical preforms with a diameter of 12.7 mm by uniaxial pressing at 300 MPa and by cold isostatic pressing at 300 MPa in the silicone mould. Silicone moulds are used to shape the samples. It has been working in the water environment with boron oil. Ceramics produced by cold isostatic pressing method were kept under pressure of 200 MPa for 5 minutes. The green compacts were sintered (Protherm) at 1600 °C for 3 hrs under air using a heating rate of 5 °C min^{-1} .

The morphological parameters of the various phases were characterized by using a semiautomatic image analyser, EDX and the formed phases were analysed by X-ray powder diffractometer (Rigaku, Dmax, IIC) using Cu K α radiation. After sample sections were mechanically polished and then thermally etched at 1420 °C for 4 hrs in air, the microstructural characterization of the sintered samples was carried out using scanning electron microscopy (Jeol 840). Micro hardness (Shimadzu, HMV-MIII) was measured on the polished surface of the samples at room temperature. At least six individual tests with a peak load of 2000 g and a loading time of 20 s were performed for each set of composites. Plint brand abrasion tester was used for the abrasion tests of ceramics. Silicon carbide discs is used as wear disc. Wear tests were performed on each sample at 3, 6, 9 min wear duration and 57, 67, 77 N force. First, the specimen was measured with a precision scale of 0.0001 g, and the amount of wear was determined by measuring again after the specified wear time.

Results and Discussion

Table 1 shows Compositions of ZrO_2 -CaO-MgO

Table 1. Compositions of ZrO_2 -CaO-MgO powders prepared.

Composition code	Chemical Composition (% mole)		
	ZrO_2	CaO	MgO
C0	90,5	–	9,5
C50	90,5	4,75	4,75
C100	90,5	9,5	–

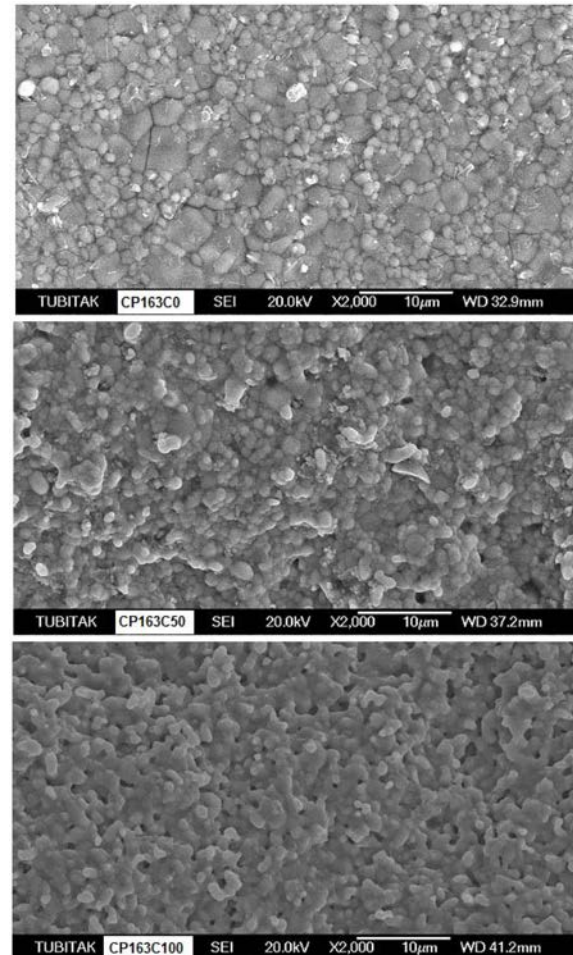


Fig. 1. SEM micrographs of CPC0, CPC50 and CPC100 samples.

powders for the different mixtures prepared. In ceramics formed by cold isostatic method, denser material was obtained after syncing. A relative density of 98% was achieved in ceramics shaped with cold isostatic press (CIP) obtained by sintering at 1600 °C for 3 hrs.

Fig. 1 is representative SEM micrographs taken from the surface of the sintered CPC0, CPC50 and CPC100 samples. It can be seen that CaO and MgO is uniform dispersed in the matrix.

In the microstructure studies, when the composition of C0 was examined according to the EDS results, the initial Mg content was around 2.00 wt%, while the overall EDS analysis resulted in a net content of 1.64 wt%, Mg content 5.65 wt% based on the grain boundary, and Mg found to be 1.39 wt%. There is an increase in the amount of Mg in the grain boundaries.

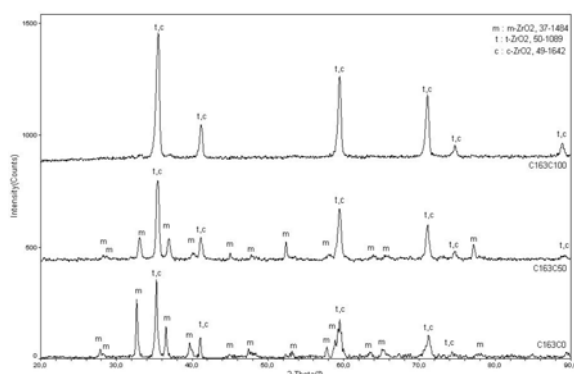


Fig. 2. Diffraction patterns of samples of CIP163 (Sintered at 1600 °C for 3 hrs shaped with cold isostatic press).

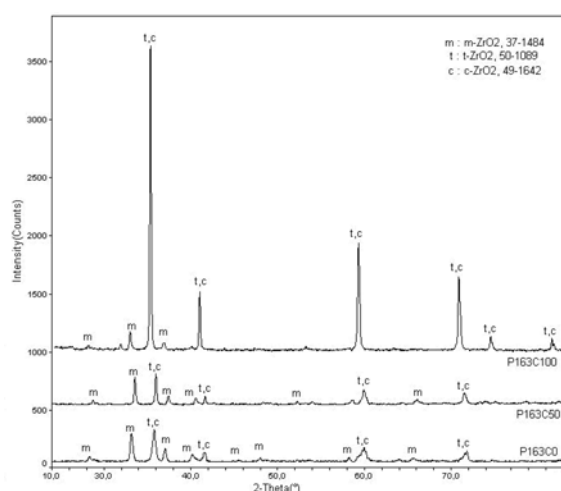


Fig. 3. Diffraction patterns of samples of CP163 (Sintered at 1600 °C for 3 hrs shaped with cold press).

When we examined the composition of C100 according to the results of EDS, the initial Ca content was around 3.57% by weight, while the overall EDS analysis showed a net weight of 3.43%, a Ca network of 3.49% and a Ca network. Found to be 3.83%. It is seen here that Ca is distributed evenly and homogeneously in the structure.

Fig. 2 shows the variation of the ceramics sintered at 1600 °C for 3 hrs in the diffraction patterns according to the composition, which is formed by cold isostatic pressing method.

When we evaluate the composition according to the composition, a clear result is obtained in the ZrO₂ phase conversion as the C0 composition is changed to the C100 composition. Here, the m-ZrO₂ phase is seen in the CIP163C0 composition and the m-ZrO₂ is seen in the CIP163C50. When the composition is CIP163C100, the m-ZrO₂ phase is completely transformed into the t-ZrO₂ and c-ZrO₂ phases. It is understood from this that CaO addition affects phase transformations in ZrO₂ ceramics. When the other diffraction peaks are examined from the other side, it is seen that the shaping method is very effective. In other words, ceramics shaped by cold isostatic pressing (CIP) are more intense and phase

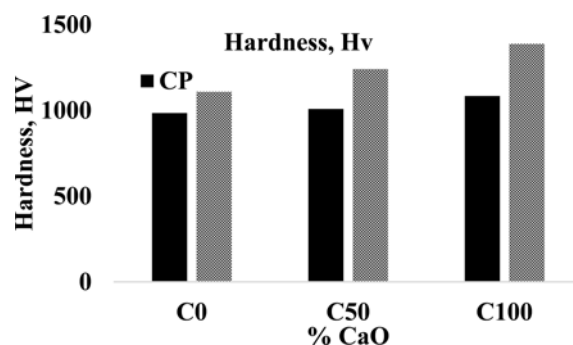


Fig. 4. Micro Vickers hardness.



Fig. 5. PLINT brand abrasion tester.

transformations are almost complete.

Fig. 3 shows the change in the diffraction patterns according to the composition in samples sintered for 3 hrs at 1600 °C moulded with a dry press. Here, the m-ZrO₂ phase is seen in the composition of CP163C0, CP163C50 and CP163C100. However, although the m-ZrO₂ phase does not completely convert to the t-ZrO₂ and c-ZrO₂ phases by the increase of CaO addition, there is a decrease in the m-ZrO₂ phase and an increase in the phases t-ZrO₂ and c-ZrO₂.

In the CIP163C0 sample, the crystal grain size was 41.1 nm while it reached 45.5 nm on the CIP163C100 specimen. That is, the addition of CaO at about 10.7% enlarged the crystal size. The crystal grain size of CP163C0 was 40.0 nm and 58.8 nm on CP163C100. That is, about 47.0% of the CaO addition increased the crystal size.

Micro Vickers hardness measurements of the obtained test specimens are presented in the following graph (Fig. 4). For hardness tests, Shimadzu HMV-MIII hardness tester is used.

According to hardness test results, it is observed that cold isostatic press specimens have higher hardness than cold pressing obtained specimens. Also, the highest (1389 HV) value is occurred in the CIP163C100 materials. As the amount of CaO increases, the hardness of the material also increases. In the cold pressed specimens, the CaO amount has not a visible effect on hardness

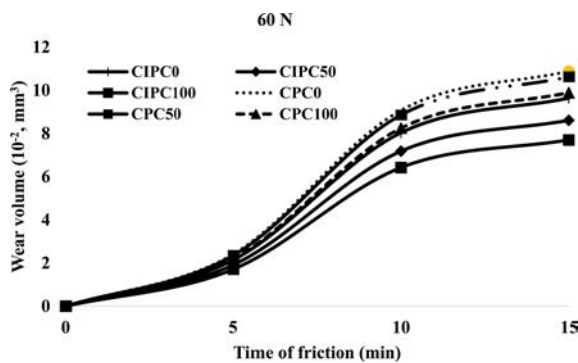


Fig. 6. Wear rates in 60 N Wear force.

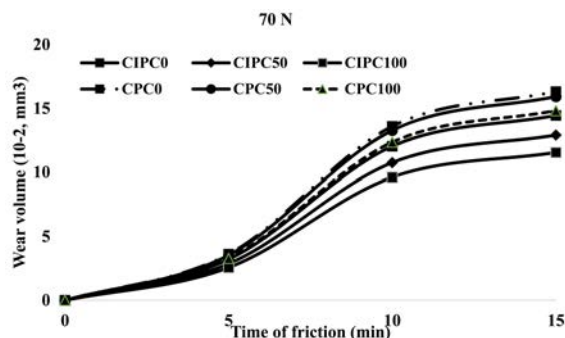


Fig. 7. Wear rates in 70 N Wear force.

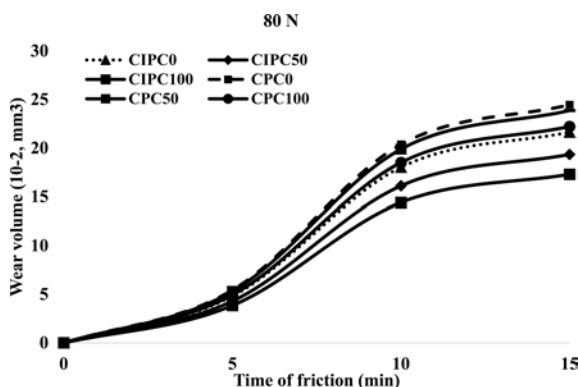


Fig. 8. Wear rates in 80 N Wear force.

values.

Plint brand abrasion tester (Fig. 5) was used for the abrasion tests of ceramics. Silicon carbide is used as wear disc. Wear tests were performed on each sample at 5, 10, 15 min wear duration and 60, 70, 80 N force. First, the specimen was measured with a precision scale of 0.0001 g, and the amount of wear was determined by measuring again after the specified wear time. The wear results are shown in Fig. 6,7 and 8.

The specimens produced by cold pressing exhibited more wear on specimens produced with cold isostatic pressing in wear tests [12]. Similarly, as the load and time increased, the amount of wear increased. The wear volume in the CIPC0 specimen under 60 N load for 5 minutes was $2,14 \times 10^{-2} \text{ mm}^3$ while it reached

$21,64 \times 10^{-2} \text{ mm}^3$ at 15 minutes under 80 N load [13, 14]. The wear volume in the CPC0 specimen under 60 N load for 5 minutes was $2,41 \times 10^{-2} \text{ mm}^3$ while it reached $24,45 \times 10^{-2} \text{ mm}^3$ at 15 minutes under 80 N load. The C0 (MgO-ZrO_2) sample was more abraded than the C100 (CaO-ZrO_2) sample. This value was measured as $2,1441 \times 10^{-2} \text{ mm}^3$ for CIPC0 and $5,7141 \times 10^{-2} \text{ mm}^3$ for CIPC100 in 5 minutes under 60 N load.

Conclusions

In this study, 8 mole% CaO, 4 mole % CaO-4 mole% MgO and 8 mole% MgO-stabilized zirconia (ZrO_2) based composites were fabricated by the conventional ceramic production process. It was intended to study hardness and the rate of wear for different ceramics under the influence of different time and loads. According to hardness test results, it is observed that cold isostatic press specimens have higher hardness than cold pressing obtained specimens. As the amount of CaO increases, the hardness of the material also increases. In the cold pressed specimens, the CaO amount has not a visible effect on hardness values. The specimens produced by cold pressing exhibited more wear on specimens produced with cold isostatic pressing in wear tests. The wear load and wear time increased, the amount of wear increased in all test specimens. The C0 (MgO-ZrO_2) sample was more abraded than the C100 (CaO-ZrO_2) sample. This result indicates that CO is more effective than MgO on the wear resistance of ZrO_2 based ceramic materials.

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