Phase Composition and Parameters in Crystal Structure of Ceramic Composites based on ZrO₂(MgO) – CaSiO₃ System



Chatchai Duangsupa, Kulkov S.N., Surat Wannasri, Suwatchai Chaiyaphan

Abstract: The synthesis and characterization of ceramic composites based on the ZrO₂(MgO)-CaSiO₃ system were investigated, focusing on their phase composition, crystal structure, and lattice parameters. The results demonstrate that sintering temperature and increasing wollastonite content significantly influence the structural formation, complex phase and phase transformations the composition. within $ZrO_2(MgO)$ -CaSiO₃ system. The phase composition of ZrO_2 and CaSiO₃ was analyzed across sintering temperatures ranging from 1000°C to 1650°C and wollastonite contents of 1-25 vol.%. The samples exhibited five distinct phases: tetragonal, cubic, and monoclinic phases of zirconium dioxide, along with monoclinic and triclinic phases of wollastonite. Notably, the interplanar spacings of the m-ZrO₂ and m-CaSiO₃ phases changed markedly within the 1200-1300°C sintering range. This is attributed to intensive interfacial interactions between zirconium dioxide and wollastonite, resulting in alterations to the structural state of the composites, phase composition, phase distribution, and lattice parameters of the zirconium dioxide and wollastonite phases.

Keywords: Phase Composition, Crystal Structure, Lattice Parameters, Ceramic Composite

I. INTRODUCTION

Recently, ceramics have been increasingly utilized as structural materials across various industries. This growing preference stems from their exceptional properties, including high heat resistance, corrosion and erosion resistance, wear resistance, and remarkable fracture toughness. Among all ceramic materials, zirconium dioxide stabilized with

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magnesium oxide holds a special position due to its unique combination of properties, such as high refractoriness, wear resistance, heat resistance, chemical resistance, radiation resistance, and biological compatibility. However, several questions regarding the ZrO₂–CaSiO₃ system remain unresolved. These include the formation of various phases during the sintering of the composite, as well as the structure, morphology, and grain size of both zirconium dioxide and wollastonite. These characteristics can vary significantly depending on the type and quantity of stabilizing additives introduced into zirconium dioxide.

II. LITERATURE REVIEW

Among ceramic materials, zirconium dioxide stabilized with magnesium oxide (ZrO₂-MgO) holds a special place due to its unique combination of properties, such as high refractoriness, wear resistance, heat resistance, chemical resistance, and radiation resistance, along with notable biocompatibility [1]. ZrO₂-MgO ceramics are particularly valuable in metallurgy for their excellent refractory and heat resistance properties, as well as their ability to withstand reactions with molten metals and slags. The strength properties of ceramics based on plasma-chemically synthesized ZrO₂-MgO powders remain insufficiently studied. There is no consensus regarding the influence of different reinforcements or secondary phases on the ultimate strength of ZrO₂-MgO ceramics. Wollastonite (CaSiO₃) has been identified as a potential reinforcing phase for the zirconium dioxide matrix [2].

Wollastonite is widely used in advanced industries, including the production of ceramic materials with ultra-high resistance, high-frequency ceramics, and bioceramics [3]. Its versatility makes it suitable for applications in dentistry, orthopedics, and other medical fields [4]. The addition of wollastonite to ceramic composites provides several advantages: it lowers the firing temperature [5], enhances mechanical strength, reduces shrinkage during drying and firing, and decreases the temperature required for glass phase formation [6]. Zirconium dioxide (ZrO₂) exists in three crystalline modifications: tetragonal [7], monoclinic, and cubic [8]. It is possible to modify the temperatures of phase transitions and adjust the temperature [9] range within which these transformations occur [10]. Studies have shown that the phase transition kinetics [11] and crystallite growth during the heat treatment of ceramic powders are significantly influenced by factors such as the amount of stabilizer used [12]. The stabilizer can notably alter the temperature range of

the tetragonal-to-monoclinic phase transition [13].

As previously mentioned, zirconia and wollastonite are

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well-known for their excellent properties and wide range of applications across various industries and technologies [1]. However, the literature provides limited data on the fine crystalline structure and phase composition of materials based on ZrO₂-MgO when modified with the addition of wollastonite [14].

The objective of this study is to investigate the phase composition and crystal structure parameters of ZrO2-MgO composites at different sintering temperatures and with varying contents of CaSiO3. This research aims to provide phase comprehensive data on the structure and transformations of materials, these facilitating the development of new materials with tailored properties.

III. EXPERIMENTAL PROCEDURE

The materials used in this study were $ZrO_2(MgO)$ powders, obtained through the decomposition of liquid precursors in high-temperature plasma, and natural wollastonite (CaSiO₃) powders. The average particle size of the zirconia powders ranged from 1 to 10 µm, while the wollastonite powders had a particle size range of 10 to 60 µm. Powders with different CaSiO₃ contents (1, 5, 10, and 25 vol.%) were mixed homogeneously using a ball milling machine.

The mixed powders were then pressed into cylindrical shapes using cold isostatic pressing at a hydraulic pressure of 300 MPa. The samples were sintered at various temperatures (1000°C, 1100°C, 1200°C, 1300°C, 1400°C, 1500°C, and 1650°C) in a furnace. During sintering, a heating rate of 5°C per minute and a maximum cooling rate of 10°C per minute were applied.

The diffraction patterns of the samples were analyzed using the Rietveld method, to estimate the phase composition in these multiphase materials [15]. Quantitative phase analysis was conducted following the methodology described in [16]. This analysis allows for the determination of specific characteristics of individual phases, including precise measurements of crystal structure, crystallite size, and shape [17]. The phase composition, crystallite size, and lattice parameters of the samples were determined using X-ray diffraction (XRD) [18].

XRD analysis was performed with a CuK α radiation source ($\lambda = 1.54178$ nm) at a voltage of 40 kV and a current of 20 mA. Continuous scanning was carried out over a 2 θ range from 15° to 120°, with a step size of 0.05° and a step time of 1 second. The X-ray exposure at each point was sufficient to ensure a relative error in the background count rate of less than 3%. The relative coherence length (CL) was estimated by analyzing the broadening of the most intense X-ray reflections observed at small-angle scattering and diffraction.

The XRD profiles were refined using the Rietveld technique to extract detailed information about crystallite size, lattice parameters, and the phase composition of the mixtures. The data were corrected for Lorentz and polarization factors, as well as absorption effects. Phase identification was performed using an X-ray diffractometer (XRD).

The full-width at half-maximum (FWHM) of the diffraction peaks was compared with standard reference data from the International Centre for Diffraction Data (ICDD). The ICDD database, which includes the Powder Diffraction File (PDF), provides essential information such as d-spacings (related to the diffraction angles) and the relative intensities of observable diffraction peaks.

IV. RESULT AND DISCUSSION

Figure 1 shows the XRD patterns corresponding to the phase composition of bulk samples after sintering at 1650°C, with CaSiO₃ contents ranging from 1 to 25 vol.%. The results indicate that the phase composition of the ZrO₂(MgO)–CaSiO₃ ceramic composites changes after sintering, comprising five phases: a combination of tetragonal, monoclinic, and cubic zirconia phases, along with a mixture of monoclinic and triclinic wollastonite phases.

Quantitative phase analysis was performed using X-ray diffraction methods. X-ray analysis of the phase composition of the composites, based on $ZrO_2(MgO)$ –CaSiO₃, revealed the phase content of ceramics with different CaSiO₃ contents (1 and 10 vol.%) and sintering temperatures ranging from 1000°C to 1650°C, as shown in Figure 2.

The phase composition of the $ZrO_2(MgO)$ -CaSiO₃ ceramic composites was determined, and the obtained data were converted into percentage values using the following equation:

Phase composition (%) =
$$\frac{\sum_{i} A_{i}}{\sum_{ij} A_{i} + B_{i} + C_{i}} \times 100 \dots (1)$$

Where, \sum_{i} – an independent observation \sum_{ij} – the sum of independent observations A, B, C – the number of phases

According to the X-ray diffraction data, the content of the tetragonal zirconia phase in the samples decreases as the sintering temperature increases from 1100° C to 1300° C. It was observed that after sintering at temperatures higher than 1300° C, the tetragonal zirconia phase was no longer detected in any of the samples. The content of tetragonal zirconia (t-ZrO₂) in the ceramic composites ranged from 3% to 19%, depending on the CaSiO₃ content and the sintering temperature, as shown in Figure 3.

The cubic zirconia phase $(c-ZrO_2)$ appeared in samples with 1 and 5 vol.% CaSiO₃, sintered at temperatures ranging from 1300°C to 1650°C, with a content of 3% to 13%. Analysis of the results revealed that the proportion of

monoclinic zirconia increased with the sintering temperature.

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[Fig.1: X-ray Diffraction Patterns of ZrO₂(MgO) – CaSiO₃ Ceramic Composites with Varying Wollastonite Contents, Sintered at 1650°C]

Figure 4 shows that the monoclinic zirconia phase $(m-ZrO_2)$ is the dominant phase in the ceramic composites based on the $ZrO_2(MgO)$ –CaSiO₃ system. It was found that the content of the monoclinic zirconia phase reached its highest value of 94% in the samples containing 5 vol.% CaSiO₃.

The content of the monoclinic wollastonite phase (m-CaSiO₃) remained virtually unchanged across the system as the sintering temperature increased from 1000°C to 1650°C. However, when the CaSiO₃ content was increased to 25 vol.% and the sintering temperature rose from 1100°C to 1500°C, the content of the monoclinic wollastonite (m-CaSiO₃) phase increased to 3–8%, as shown in Figure 5.

It was established that the monoclinic zirconia phase and the mixture of monoclinic and triclinic modifications of wollastonite coexist in the ZrO₂(MgO)–CaSiO₃ composite system. Their content remained nearly constant as the sintering temperature increased.







[Fig.3: Dependence of Tetragonal Zirconia Phase Content (t-ZrO₂) on Sintering Temperature with Varying CaSiO₃ Contents]



[Fig.4: Dependence of Monoclinic Zirconia Phase Content (m-ZrO₂) on Sintering Temperature with Varying CaSiO₃ Contents]

Figure 6 presents the variation in the percentage of the triclinic wollastonite phase (t-CaSiO₃). The amount of triclinic wollastonite (t-CaSiO₃) ranged from 1% to 5%. It can be observed that both the sintering temperature and the CaSiO₃ content influence the content of the triclinic wollastonite phase (t-CaSiO₃). It was found that the sintering temperature and the increase in wollastonite content significantly affect the formation of the structure, the complex phase composition, and the phase transformations in the ZrO₂(MgO)-CaSiO₃ system. The results indicate that intensive interfacial interactions between zirconium dioxide and wollastonite during sintering at temperatures between 1100°C and 1300°C, lead to changes in the structure of the ceramic composites. These interactions are the primary factors driving changes in the phase composition and distribution within the samples.



[Fig.5: Content of the Monoclinic Modification of Wollastonite as a Function of Sintering Temperature and Initial Mixture Concentration]



[Fig.6: Content of the Triclinic Modification of Wollastonite as a Function of Sintering Temperature and Wollastonite Concentration in the Initial Mixture]

The lattice parameters were determined using the Rietveld method. The unit cell parameters were calculated using formulas specific to

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different crystal symmetries (syngonies), which relate these parameters to the interplanar distances (d_{hkl}) and the indices of the reflecting planes (h, k, l).

The equation for calculating the unit cell parameters for various crystal symmetries (syngonies) is as follows:

Cubic =
$$\frac{1}{d^2} = \frac{H^2 + K^2 + L^2}{a^2}$$
 ... (2)
Tetragonal = $\frac{1}{d^2} = \frac{H^2 + K^2}{a^2} + \frac{L^2}{c^2}$... (3)

Monoclinic =

$$\frac{1}{d^2} = \frac{H^2}{a^2 \sin^2 \beta} + \frac{K^2}{b^2} + \frac{L^2}{c^2 \sin^2 \beta} - \frac{2HL\cos\beta}{ac\sin^2 \beta} \quad \dots \quad (4)$$

Triclinic =

$$\frac{1}{d^2} = \frac{1}{V^2} [s_{11}H^2 + s_{22}K^2 + s_{33}L^2 + s_{12}HK + s_{23}KL + s_{13}HL] \quad \dots \quad (5)$$

Where d – interplanar Å distances, HKL – indexes reflecting planes (where H = nh, K = nk, L = nl), a, b and c - the vectors, β – adjusted full width at half height, V – the volume of the unit cell.

The formula for calculating the volume (V) is as follows:

$$V = abc\sqrt{1 - \cos^2 \alpha - \cos^2 \beta - \cos^2 \gamma + 2\cos \alpha \cos \beta \cos \gamma} \quad \dots \quad (6)$$

Where $S11 = b2c2 \sin 2\alpha$, $S22 = a2c2 \sin 2\beta$, $S33 = a2b2 \sin 2\beta$ γ , S12 = abc2(cos α cos β - cos γ), S23 = a2bc (cos β cos γ - $\cos \alpha$) μ S13 = ab2c ($\cos \gamma \cos \alpha - \cos \beta$).

Table I presents the variation in the lattice parameters of monoclinic zirconia in samples containing 10 vol.% CaSiO₃, sintered at temperatures ranging from 1000°C to 1650°C. The lattice parameters a and b decreased, while c increased with increasing sintering temperature. Additionally, the angle β (in degrees) of monoclinic zirconia decreased as the sintering temperature increased from 1100°C to 1650°C, as shown in Figure 7.

Table 1: Lattice Parameters of Monoclinic Zirconia

Temperature	Lattice Parameters						
(°C)	a [Å]	b [Å]	c [Å]	β[]	V [Å ³]		
1000	5.348	5.218	5.186	96.92	143.7		
1100	5.294	5.203	5.167	99.31	140.4		
1200	5.263	5.192	5.205	98.83	140.6		
1300	5.137	5.188	5.297	99.33	139.3		
1400	5.153	5.184	5.308	98.69	140.1		
1500	5.289	5.173	5.274	96.76	143.2		
1650	5.239	5.175	5.272	95.97	142.2		

Table 2: Lattice Parameters of Monoclinic Wollastonite

Temperature	Lattice Parameters						
(°C)	a [Å]	b [Å]	c [Å]	β[]	V [ų]		
1000	15.205	7.041	7.045	95.221	751.1		
1100	15.334	6.962	7.071	95.193	751.8		
1200	15.411	6.971	6.815	99.499	722.1		
1300	15.208	7.478	7.434	91.048	845.3		
1400	15.143	7.213	7.047	95.964	765.4		
1500	15.110	7.292	7.031	95.286	771.4		
1650	14.829	7.478	6.995	95.953	771.4		

<u>Table II</u> shows the changes in the lattice parameters of monoclinic wollastonite (m-CaSiO₃) in samples containing 25 vol.% CaSiO₃, sintered at temperatures ranging from 1000°C to 1650°C. It was observed that the lattice parameters a and b decreased, while c increased with increasing sintering temperature.

Figure 8 illustrates the dependence of the angle β (in degrees) of monoclinic wollastonite on the sintering temperature for different CaSiO3 contents. It was found that during sintering at 1300°C, the angle β of the monoclinic phase of wollastonite in samples containing 10 vol.% CaSiO₃ increased significantly, due to changes in the lattice parameter a, with an anomalous increase in β . In samples containing 25 vol.% CaSiO₃, the angle β of the monoclinic phase of wollastonite decreased, likely due to the anomalous increase in the lattice parameter c, as shown in Figure 8.



[Fig.7: Dependence of the Angle β of Monoclinic Zirconia on Sintering Temperature with Varying Contents of CaSiO₃]



[Fig.8: Dependence of the Angle β of Monoclinic Wollastonite on Sintering Temperature with Varying Contents of CaSiO₃]

The obtained results show that the content of monoclinic, cubic, and tetragonal phases of zirconia, as well as the monoclinic and triclinic phases of wollastonite, is consistent with the lattice parameter measurements determined by the Rietveld method, and aligns with the known standards of the International Centre for Diffraction Data (ICDD).

Figures 9 and 10 show the changes in interplanar distances of the samples sintered at different temperatures ranging from 1000°C to 1650°C. The

interplanar distance of the {111} plane of monoclinic zirconia (ZrO_2) varied

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between 2.8257 Å and 2.8459 Å, while the interplanar distance of the {-431} plane of monoclinic wollastonite (CaSiO₃) ranged from 1.9826 Å to 1.994 Å.

Figure 9 shows a sharp change in interplanar distances at sintering temperatures between 1200°C and 1300°C, marking the phase transition from tetragonal to monoclinic zirconia. This sharp change, observed in the diffraction patterns, corresponds to intense interaction between the components of the composite material during this temperature range.

It was observed that an increase in temperature from 1200°C to 1300°C leads to intense interfacial interactions between zirconium dioxide and wollastonite, resulting in changes to the lattice parameters in the tetragonal, cubic, and monoclinic phases of zirconium dioxide, as well as the triclinic and monoclinic phases of wollastonite.



[Fig.9: Change in the Interplanar Spacing d₁₁₁ of Monoclinic Zirconia Sintered at Different Temperatures]



[Fig.10: Change in the Interplanar Distance d-431 of **Monoclinic Wollastonite Sintered at Different Temperatures**]



[Fig.11: Change in the Interplanar Distance d₁₁₁ of Monoclinic Zirconia and Its Density]



[Fig.12: Change in the Interplanar Distance d-431 of the Monoclinic Modification of Wollastonite and Its Density]

Figures 11 and 12 show the dependence of interplanar distances on the density. The interplanar distances in the phases of zirconia and wollastonite exhibit abrupt changes between 1200°C and 1300°C, which corresponds to the intensity of the interaction between the components of the composite material. This change is consistent with the observed density range. An analytical study of the relationship between interplanar distances and material density reveals that the sharp change in interplanar distances, observed from diffraction patterns, occurs within the temperature range of 1200°C to 1300°C. At this temperature, compression due to interfacial interactions is observed, which results in changes to the structure of the composites. The density increases as the average grain size of the sintered materials decreases, leading to modifications in the crystal structure parameters.

V. CONCLUSION

The X-ray diffraction analysis revealed changes in the phase composition during the sintering process, with five detected in the ceramic phases composites of ZrO₂(MgO)-CaSiO₃: tetragonal, monoclinic, cubic ZrO₂, and mixtures of monoclinic and triclinic CaSiO₃. At low sintering temperatures, the phase composition consists of a mixture of monoclinic zirconia and a combination of monoclinic and triclinic wollastonite. The content of the m-ZrO₂ phase increases with higher sintering temperatures.

These results demonstrate that both increased sintering temperatures and higher wollastonite content influence the formation of the structure, the complex phase composition, and the crystal structure parameters in ceramic composites based on the ZrO₂(MgO)-CaSiO₃ system. It was observed that the interplanar spacings of both ZrO2 and CaSiO3 phases undergo sharp changes at temperatures between 1200°C and 1300°C. This interfacial interaction, which affects the structural state of zirconium dioxide, results in modifications to the lattice parameters of the monoclinic, cubic, and tetragonal phases of zirconium dioxide, as well as the monoclinic phase of wollastonite in

the ZrO₂(MgO)–CaSiO₃ system.

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