

Sintering densification, structural analysis, microstructure and mechanical properties of $\text{Al}_2\text{O}_3(\text{MgO})/\text{ZrO}_2(\text{Y}_2\text{O}_3)$ composite ceramics

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Composite ceramics between Al_2O_3 (6 mol% MgO); AM and ZrO_2 (4 mol% Y_2O_3); ZY with 40:60, 50:50, and 60:40 ratio were prepared. The samples were sintered under different sintering method between normal sintering and two-step sintering method. The effects of the different contents of AM and ZY and sintering condition on the structural analysis, densification, microstructure and mechanical behavior were studied. It was found that composites with higher proportions of ZrO_2 showed higher weight percentages of $t\text{-ZrO}_2$ and $c\text{-ZrO}_2$ phases which is obtained high density and good mechanics properties. The Fracture toughness mechanisms of composites are also discussed in detail around the sintering parameters. The AM:ZY composite ceramics that provides good mechanical properties were revealed in both sintering method in optimum conditions.

Keywords: Al_2O_3 , ZrO_2 , Composite, Ceramics, Sintering.

Introduction

Al_2O_3 has high elastic modulus and compressive strength but its low fracture toughness. Undoped Al_2O_3 ceramics, when heated in sintering process at high temperatures, often cause irregularities of grains in the microstructure due to abnormal grain growth. This will reduce the bulk density of Al_2O_3 ceramics and lead to low mechanical properties. Therefore, materials with inertia properties of MgO are added into Al_2O_3 to control the expansion of grains in the sintering process. MgO is a traditional additive to Al_2O_3 (AM) since it can reduce the sintering temperature and grain size. In order to improve the fracture toughness of Al_2O_3 ceramics, a second phase is usually introduced into them, so that the second phase is filled at the grain boundary of alumina, which is conducive to preventing the extension of fracture lines and improving the fracture toughness of alumina ceramics [1, 2]. Zirconia is interested as ceramic steel material in previous works [3-6]. ZrO_2 has high resistance to crack propagation, possesses very low thermal conductivity and high strength. The fracture strength of Al_2O_3 ceramics has been improved by adding ZrO_2 as the second phase and sintering to form intergranular or intragranular structure [7]. Zirconia is a polymorphic metastable material within the polycrystalline phase system, and pure zirconia reveals various crystalline phase structures under atmospheric

pressure at varying temperatures. ZrO_2 obtains in three crystalline forms, including high temperature cubic phase ($c\text{-ZrO}_2$), intermediate temperature tetragonal phase ($t\text{-ZrO}_2$) and low temperature monoclinic phase ($m\text{-ZrO}_2$) [8]. And there is a transition among the three phases with the ambient temperature. When the pure ZrO_2 ceramic are cooled back to room temperature, the tetragonal phase undergoes a reverse phase transition to monoclinic phase, resulting in an increase in volume that generates internal stresses, which in turn leads to cracking in ZrO_2 ceramics [9]. Zirconia in the tetragonal form at room temperature which has the best mechanical properties among all forms and provides toughening mechanisms associated with the phase transformation from $t\text{-ZrO}_2$ to $m\text{-ZrO}_2$ under stress [9, 10]. Many report suggested incorporating oxides as stabilizers into zirconia to prevent the reverse transformation [3, 11, 12]. Zirconia stabilized with Y_2O_3 (ZY) has the best properties for mechanical device applications. Stabilizing oxides (e.g. Y_2O_3) are used to stabilize cubic and tetragonal zirconia forms at room temperature. The stabilization of the tetragonal phase at room temperature is possible by the yttrium (Y^{3+}) substitution of zirconium (Zr^{4+}) and concomitant formation of charge neutralizing oxygen vacancies. The mechanical properties of ZrO_2 ceramics with 4 mol% Y_2O_3 addition depend strongly on the microstructure as well as composition as reported by Rittidech et al. [13]. The ceramic composites between Al_2O_3 and ZrO_2 are of the most widely used in many critical structural ceramics application because of their high strength and toughness [7, 14]. The mechanical properties of Al_2O_3 - ZrO_2 ceramic composites are

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due to the various proportions of Al_2O_3 and ZrO_2 . Composites with a high content of Al_2O_3 are used in applications requiring high Young's modulus and high hardness, while composites with a high ZrO_2 content can be used where higher compliance is required [15]. The alumina–zirconia system is interesting to study [16, 17]. The ceramic composite between AM and ZY at a ratio of 40:60, 50:50, and 60:40 was prepared. The process of heat treatment is important in preparation of ceramics. The aim of the sintering process of ceramic composite is to obtain a samples with a high densification and homogeneous microstructure consistent of well packed. The microstructure of Al_2O_3 - ZrO_2 composite ceramics can be controlled by two ways i.e., either by using additives to prohibit the grain growth, used for obtaining highly dense ceramics, or it can be controlled by using novel processing techniques to modify the microstructure. The process of heat treatment in optimal sintering conditions is important factors. The normal grain growth can be processed by pressureless sintering with appropriate thermal treatment conditions. However, the different proportions composites between Al_2O_3 and ZrO_2 are therefor suitable for the heating process sintering using different conditions. Sintering is defined as thermal treatment of fine-grained material at elevated temperature, but below the melting point of the main constituent, for the purpose of increasing its grain size and strength by bonding together the particles [18]. Two-stage sintering process is one of the ways of eliminating grain growth in the final stage of sintering reported by Chen and Wang [19]. Two stage sintering methodologies involve sintering with thermal pretreatment at a high sintering temperature for short holding times, followed by a second stage at lower temperature for long holding times.

In the present work, the effect of different ZrO_2 content on the toughness strength of Al_2O_3 ZrO_2 composites was investigated in characteristic behavior under possible sintering heat treatment. Composite ceramics between Al_2O_3 using addition of 6 mol%MgO; AM and ZrO_2 addition of 4 mol% Y_2O_3 ; ZY with 40:60, 50:50 and 60:40 ratio were prepared under heating in sintering at different conditions, both conventional and two stage sintering. The influence of firing conditions on bulk densification, percentage of shrinkage, phase formation, crystal structure calculated by Rietveld refinement, microstructure and microhardness is also investigated.

Experimental

Al_2O_3 - ZrO_2 composite ceramic were used as matrix powders to which Al_2O_3 was added MgO 6.0 mol% to form AM and ZrO_2 was added Y_2O_3 , 4.0 mol% to form ZY. The powder of AM :ZY at 60:40, 50:50 and 60:40 ratios were mixed together in different content and then wet ball milled for 24 h. After ball-milling, drying in electronic furnaces and sieving with 120 mesh, the

resulting powders were annealed at 1200 °C, with dwell times of 120 min and heating/cooling rates of 10 °C/min. The (100- x)AM- x ZY composite powders ($x=40, 50, 60$) were then pressed at 3MPa to form pellets having 1.5 cm diameter using a hydraulic press and sintered in an alumina crucible under two stage sintering (TS) with three different conditions and normal sintering (NS) with two conditions according to our the previous research based on alumina zirconia materials [13, 20, 21]. The two stage sintering conditions were (TS-1) T_1 temperature at 1600 °C for 30 min and T_2 temperature at 1450 °C for 5 h, (TS-2) T_1 temperature at 1600 °C for 30 min and T_2 temperature at 1450 °C for 10 h, (TS-3) T_1 temperature at 1650 °C for 30 min and T_2 temperature at 1450 °C for 5 h. The normal sintering (NS-4) temperature at 1600 °C for 5 h, and (NS-5) temperature at 1650 °C for 5 h. The sintering experiments were carried out in an electrical furnace (Nabertherm, Germany). The bulk densities of sintered sample were calculated using Archimedes' method. The phase composition was investigated by an X-ray powder diffraction using an X-ray diffractometer (Bruker model D8 advance). Quantitative phase analyses were performed by fitting the measured data using the Rietveld refinement method in TOPAS software. Microstructural analysis was performed by using Scanning Electron Microscopy (SEM) (JEOL JSM-840A) of sintered samples on a polished surface. The microhardness of the bulk ceramics was measured using a micro scan from Vickers and Knoop (FM- 700e type D, Future Tech., Japan).

Results and Discussion

Figures 1(a-c) were obtained XRD patterns of (100- x) AM- x ZY ($x = 40, 50, 60$) composites ceramic with different sintering conditions. All three XRD patterns consist of two main phases of alumina and zirconia. Apart from the alpha (α)- Al_2O_3 and monoclinic (m), tetragonal (t), cubic (c) zirconia was found. The main reflections in the pattern form around 2θ as 25, 35, 43, 57 and 67° that closely match the characteristic reflections of alumina phase and 2θ as 28, 30, 31, 35, 50, 60 confirmed zirconia phase consistent with the characteristic reflections of Al_2O_3 - ZrO_2 [21-24]. X-ray diffraction patterns of AM-ZY composites ceramic with more content of ZY (in Fig. 1(a)) was clearly revealed the high intensity peak of ZrO_2 phase. The conditions of heating number 1, 2 and 3 are two stage sintering. The number of 1 and 2 of two stage sintering condition are different at the length of holding times in the second step. It was found that for the length of holding times is revealed a sharp peak of XRD. The conditions of heating number 4 and 5 are normal sintering where there is a difference sintering temperature between 1600 °C and 1650 °C. It can be found that the normal sintering method were showed a high intensity and sharp XRD peaks. Densities of sintered samples were determined

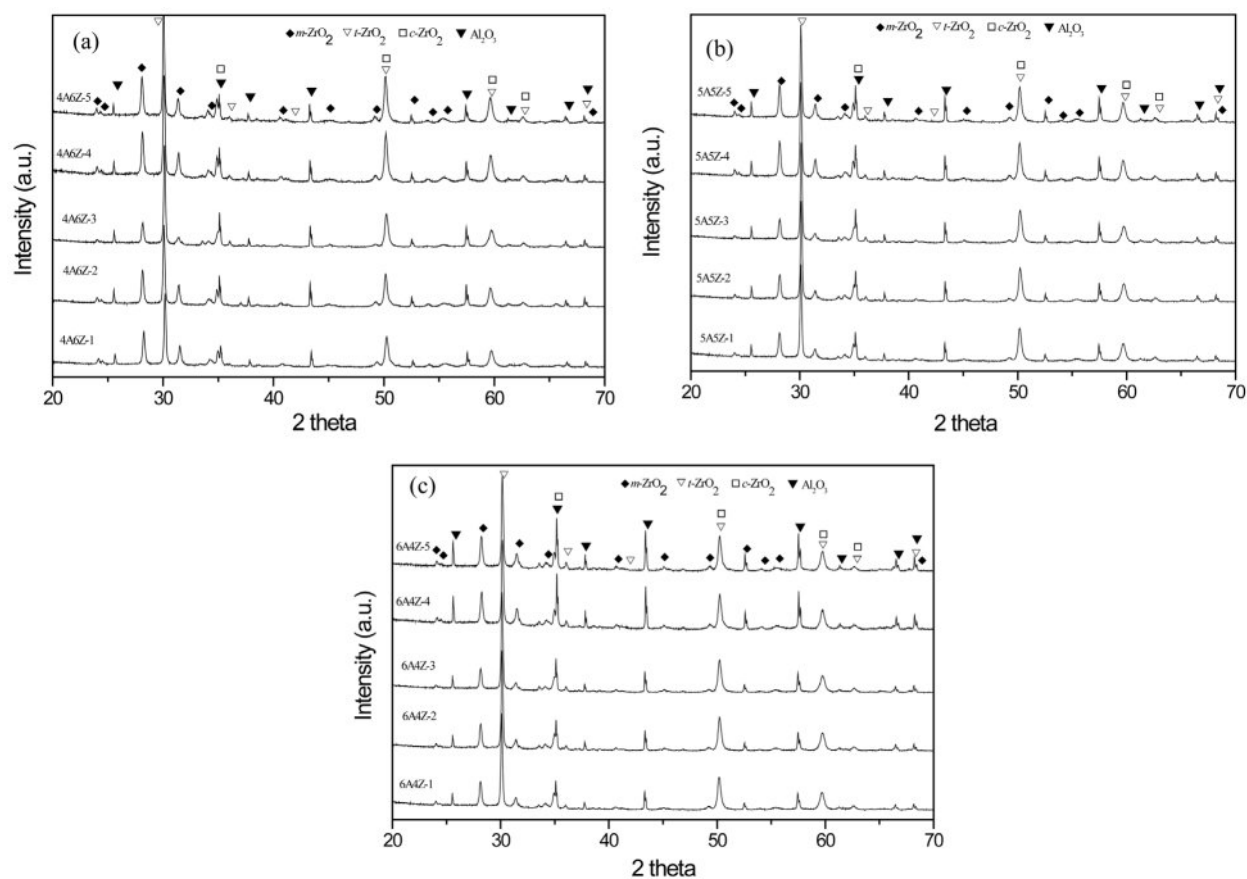


Fig. 1. XRD patterns of $(100-x)\text{AM}-x\text{ZY}$ ($x = 40, 50, 60$) composites ceramic with different sintering conditions (a) 40AM-60ZY (b) 50AM-50ZY (c) 60AM-40ZY.

Table 1. The densities, percentage of shrinkage, crystallize size and average gain size of $(100-x)\text{AM}-x\text{ZY}$ ($x = 40, 50, 60$) composites ceramic with different sintering condition.

Sintering conditions	Samples	Density (g/cm^3)	Shrinkage (%)	Crystallize size of AM (nm)	Crystallize size of ZY (nm)	Average grain size of AM (μm)	Average grain size of ZY (μm)
1	4A6Z	4.45	13.06	55.492	32.581	0.867	0.587
	5A5Z	4.27	13.11	54.680	32.068	0.914	0.612
	6A4Z	4.04	13.45	59.291	32.853	0.922	0.601
2	4A6Z	4.52	14.04	57.308	32.872	0.943	0.627
	5A5Z	4.33	13.73	57.854	32.520	1.011	0.645
	6A4Z	4.15	13.27	59.292	33.034	1.105	0.689
3	4A6Z	4.50	14.09	57.984	33.141	1.022	0.688
	5A5Z	4.36	13.41	56.551	32.450	1.054	0.712
	6A4Z	4.13	13.29	58.435	33.173	1.201	0.707
4	4A6Z	4.56	13.02	59.212	31.871	1.159	0.744
	5A5Z	4.37	10.78	58.691	31.938	1.210	0.812
	6A4Z	4.17	10.10	60.173	32.480	1.257	0.810
5	4A6Z	4.52	12.89	60.833	36.862	1.198	0.825
	5A5Z	4.38	11.43	60.192	33.105	1.248	0.866
	6A4Z	4.19	12.10	61.005	32.765	1.277	0.954

using the Archimedes principle. Table 1 contains data about the densities, percentage of shrinkage, crystalline size and average grain size of (100- x)AM- x ZY ($x = 40, 50, 60$) composites ceramic with different sintering condition. It was observed that the density was between 4.04-4.56 g/cm³. The AM-ZY composites ceramic with more content of ZY were showed a highly dense of composites ceramic. The optimal density was obtained in the sample with the two stage sintering condition of TS-3 (T_1 temperature at 1650 °C for 30 min and T_2 temperature at 1450 °C for 5 h and normal sintering using sintering temperature at 1650 °C for 5 h (NS-5). The promotion of densification by the addition of yttria in zirconia and magnesia in alumina under optimal sintering condition is revealed a high final density than

when using undoped [21, 25]. The crystalline sizes of AM and ZY phases calculated from the XRD patterns using Scherrer equation [26] are summarized in Table 1. The crystalline sizes of AM were in the range 54.680-61.005 nm and for ZY they were in the range 31.871-36.862 nm. Fig. 2(a)-(e) shows the SEM micrographs of as-received 50AM-50ZY composite ceramics with different sintering condition. The microstructure of the sintered 50AM-50ZY composite ceramics is showed a smaller spherical grain shape of the ZY phase, while the AM phase is larger and darker grains supported by the other research [21, 27, 28]. Microstructural characteristics in Fig. 2(a)-(e) were observed, i.e., uniform sized grains with well-packed and continuous grain structure. By applying the linear intercept method [29] the average

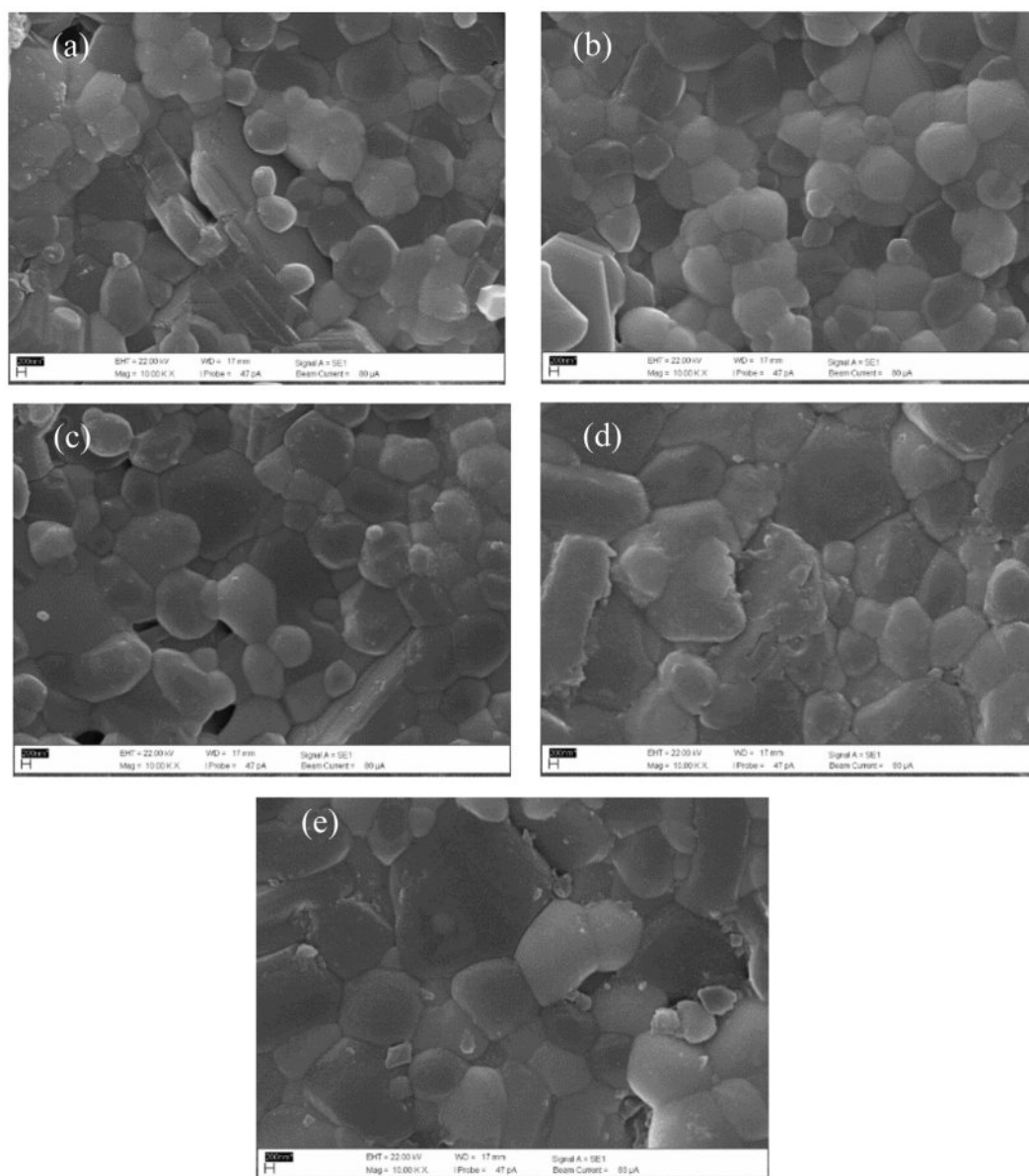


Fig. 2. SEM micrographs of as-received 50AM-50ZY composite ceramics with different sintering condition (a) TS-1 (b) TS-2 (c) TS-3 (d) NS-4 (e) NS-5.

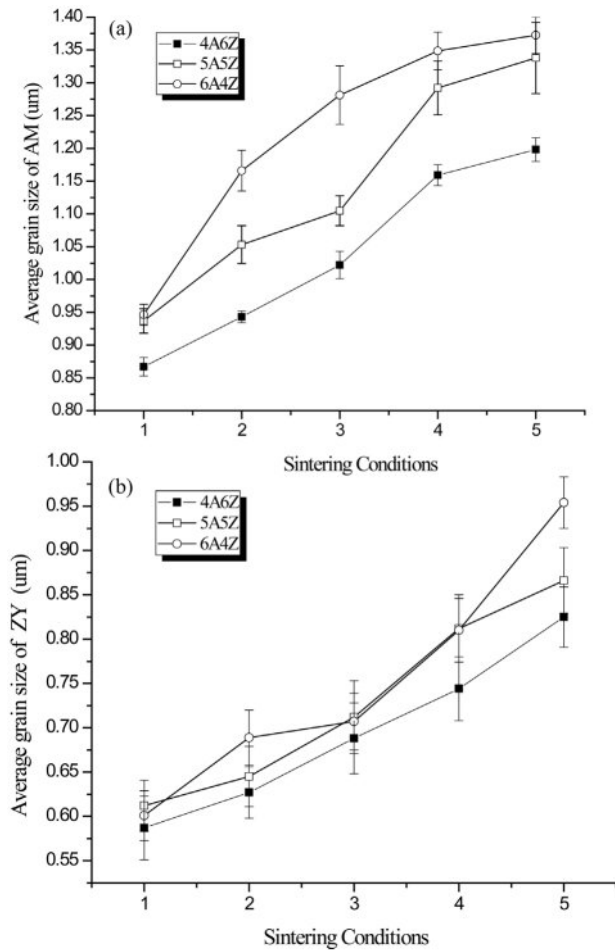


Fig. 3. The relationship between average grain sizes values and sintering conditions (a) AM (b) ZY.

grain sizes in SEM micrographs were measured for these samples as shown in Table 1. It can be seen that (100- x) AM- x ZY ($x = 40, 50, 60$) composite ceramics with different sintering condition showed average grain sizes for AM in the range 0.867-1.257 μm (in Fig. 3(a)), while the average grain sizes of ZY was 0.587-0.812 μm (in Fig. 3(b)). When a comparison of the grain sizes of (100- x)AM- x ZY ($x = 40, 50, 60$) composite ceramics with different sintering condition between two stage sintering (TS-1, TS-2, TS-3) and normal sintering (NS-3, NS-4), it was found that the sizes of grains (100- x)AM- x ZY ($x = 40, 50, 60$) in composite ceramics with two stage sintering condition was smaller than in ceramics from normal sintering. Moreover, it can be found the average grain size value tend to increase with the length of holding times in the second step of two stage sintering and the high temperature with normal sintering. Almost no abnormal grain growth appeared. This supported previous research, in which the composite ceramics between alumina and zirconia restrained abnormal grain growth in alumina-based materials [30, 31]. In addition to the appropriate sintering condition MgO doped in alumina is also an important factor in controlling grain size as reported in the other works [32]. Full pattern matching refinement of XRD patterns was performed using the TOPAS program based on the rietveld method. This obtained more detailed information on crystallographic spectra of (100- x)AM- x ZY ($x = 40, 50, 60$) composite ceramics with different sintering condition and selected samples of 50AM-50ZY composite ceramics with the two stage sintering of TS-3 as shown in the XRD refinement pattern in Fig. 4. The fitted patterns were in

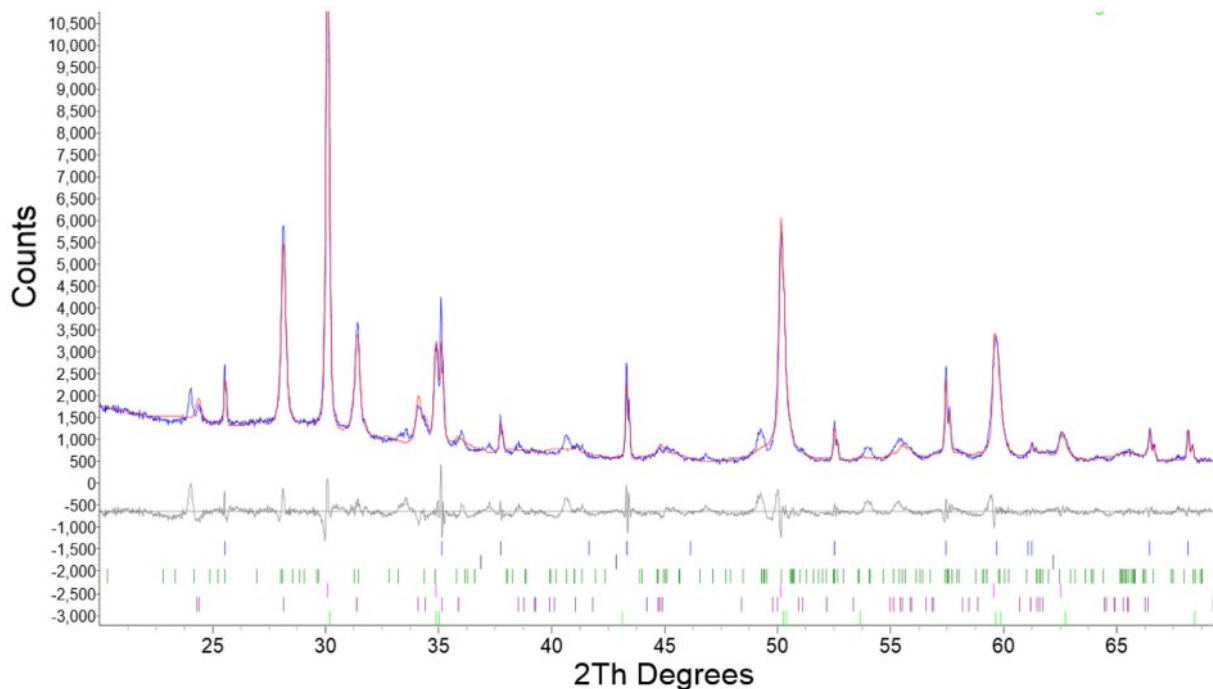


Fig. 4. Rietveld refinement of 50AM-50ZY composite ceramics with two stage sintering of TS-3 condition.

Table 2. Parameters obtained from Rietveld analysis (100-*x*)AM-*x*ZY (*x* = 40, 50, 60) composite ceramics with sintering condition of TS-3 and NS-5.

Samples	Phase present	Lattice parameter				Phase content (%)	GOF
		<i>a</i> (nm)	<i>b</i> (nm)	<i>c</i> (nm)	β (°)		
6A4Z-3	Al ₂ O ₃	0.4760	0.4760	1.2999	99.36	49.56	3.17
	m-ZrO ₂	0.5281	0.5311	0.5325		17.90	
	t-ZrO ₂	0.3610	0.3610	0.5110		7.12	
	c-ZrO ₂	0.5138	0.5138	0.5138		19.43	
5A5Z-3	Al ₂ O ₃	0.4761	0.4761	1.3002	99.10	48.64	2.84
	m-ZrO ₂	0.5160	0.5253	0.5284		19.11	
	t-ZrO ₂	0.3638	0.3638	0.5128		7.69	
	c-ZrO ₂	0.5139	0.5139	0.5139		17.23	
4A6Z-3	Al ₂ O ₃	0.4763	0.4763	1.3006	99.35	48.72	3.09
	m-ZrO ₂	0.5326	0.5108	0.5273		20.55	
	t-ZrO ₂	0.3641	0.3641	0.5131		12.31	
	c-ZrO ₂	0.5140	0.5140	0.5140		19.48	
6A4Z-5	Al ₂ O ₃	0.4681	0.4681	1.3018	99.05	49.10	2.84
	m-ZrO ₂	0.5732	0.5462	0.4415		13.87	
	t-ZrO ₂	0.3637	0.3637	0.5129		8.01	
	c-ZrO ₂	0.5140	0.5140	0.5140		14.54	
5A5Z-5	Al ₂ O ₃	0.4675	0.4675	1.3016	99.02	48.45	2.92
	m-ZrO ₂	0.5749	0.5443	0.4367		15.33	
	t-ZrO ₂	0.3629	0.3629	0.5108		7.90	
	c-ZrO ₂	0.5142	0.5142	0.5142		16.56	
4A6Z-5	Al ₂ O ₃	0.4680	0.4680	1.3010	99.01	48.22	3.41
	m-ZrO ₂	0.5814	0.5521	0.4988		19.41	
	t-ZrO ₂	0.3610	0.3610	0.5009		13.05	
	c-ZrO ₂	0.5144	0.5144	0.5144		15.39	

good agreement with the respective experiment data, denoted by GOF factors and selected from highly dense samples with sintering condition of the TS-3 and NS-5 listed in Table 2. The lattice parameter of the AM is showed to be in the range of 0.4675–1.3018 nm, while the ZY phase consists of three different crystallographic system; *m*-ZrO₂, *t*-ZrO₂, *c*-ZrO₂. It was found that two stage sintering condition by the length of holding times at *T*₂ temperature and normal sintering with high temperature were obtained the high percentage of phases content of *t*-ZrO₂, *c*-ZrO₂. The mechanical properties of (100-*x*)AM-*x*ZY (*x* = 40, 50, 60) composite ceramics with different sintering conditions were investigated by measuring microhardness by Knoop and Vickers techniques and then calculating the fracture toughness values. The selected 50AM-50ZY samples with two sintering conditions of TS-3 are shown as indentation of Vickers and Knoop hardness micrographs in Fig. 5(a-b). Fig. 6 shows the relationship between fracture toughness values of (100-*x*)AM-*x*ZY (*x* = 40, 50, 60) composite ceramic and the different sintering conditions. The fracture toughness values of (100-*x*)AM-*x*ZY (*x* = 40, 50, 60) composite ceramic is obtained to be in the range of 0.287–0.423

GPa.m^{1/2}. Two stage sintering conditions (TS-1, TS-2, TS-3), it was found that the fracture toughness values increased with *T*₂ temperature during length holding times as TS-2 condition and at high temperature of *T*₁ with short holding times as TS-3 condition. While, normal sintering conditions (NS-4 and NS-5), the optimal sintering temperature were obtained from NS-5, which is used sintering temperature at 1650 °C with holding time for 5 h. The sample of 40AM-60ZY composite ceramics with sintering conditions of TS-3 and NS-5 had a high fraction of *t*-ZrO₂, *c*-ZrO₂ phases. These results confirm that increases in hardness were due to the well-packed and continuous grain structure, corresponding to densities and phase analysis. The fracture toughness values of all compositions was higher than that of the Al₂O₃-ZrO₂ with only doped Y₂O₃ in previous reported [21]. The highest value for fracture toughness using two sintering conditions was demonstrated by 40AM-60ZY composite ceramics using TS-3 condition as *T*₁ temperature at 1650 °C for 30 min and *T*₂ temperature at 1450 °C for 5 h and obtained from 40AM-60ZY composite ceramics using normal sintering from NS-5 condition with sintering temperature at 1650 °C for 5 h, which

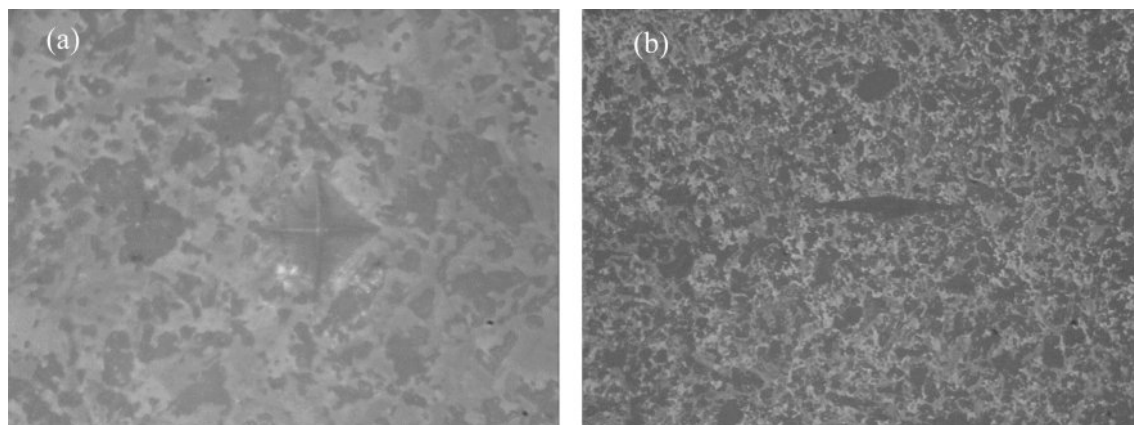


Fig. 5. Indentation of microhardness of 50AM-50ZY composite ceramics with two stage sintering of TS-3 condition (a) Vickers (b) Knoop.

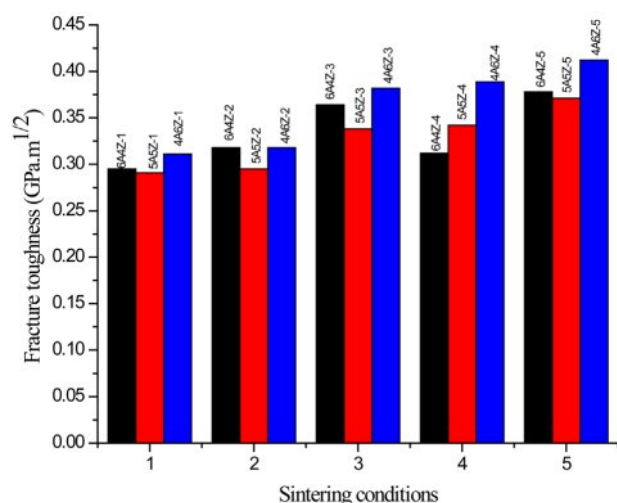


Fig. 6. The relationship between fracture toughness values of (100-x)AM-xZY ($x = 40, 50, 60$) composite ceramics.

corresponds to the highest percentage fraction in the tetragonal phase and cubic phase by XRD. It had high densification and optimal microstructure revealed by SEM. The addition of ZrO_2 promoted the sintering of composites, resulting in a higher density and higher hardness in agreement with the studies of Yildiz et al. [7], Vakhshouri et al. [31]. When consideration the use of different heat treatment between two stage sintering and normal sintering in (100-x)AM-xZY ($x = 40, 50, 60$) composite ceramics, it was found that more content of AM that using two stage sintering were revealed composite ceramics with a high density, an appropriate microstructure and good mechanical properties. These results support a two stage sintering mechanism that inhibits abnormal grain growth in alumina [21]. While (100-x)AM-xZY ($x = 40, 50, 60$) composite ceramics with high content of ZY that using normal sintering at high temperature was obtained a highly dense composite ceramics, which is the result of addition of ZY phase in AM phase that reduces abnormal grain growth at high

sintering temperature. However, from the past research related to alumina zirconia composite materials, it was revealed that the different proportion between Al_2O_3 and ZrO_2 influenced the heat treatment with different sintering conditions. The previous work by Suthapintu et al. [20] have studied the preparation of alumina zirconia composites at proportion of 50:50 using two stage sintering routes with T_1 at 1600 °C for 30 min and T_2 at 1450 °C for 5 h and showed the fracture toughness of 0.298 $\text{GPa.m}^{1/2}$. A. Rittidech et al. [33] prepared alumina zirconia composites at proportion of 50:50 under two stage sintering routes with T_1 at 1600 °C for 60 min and T_2 at 1450 °C for 10 h. It was found that the alumina zirconia composites were obtained an increased the fracture toughness of 0.320 $\text{GPa.m}^{1/2}$. In this research, when compared to Al_2O_3 - ZrO_2 composite materials in the same proportion (50:50) with modified thermal condition in both normal and two stage sintering. The alumina zirconia composites using normal sintering with temperature at 1650 °C for 5 h (NS-5) was showed a fracture toughness of 0.371 $\text{GPa.m}^{1/2}$ while using a two stage sintering with T_1 at 1650 °C for 60 min and T_2 at 1450 °C for 5 h (TS-3) was obtained a fracture toughness of 0.338 $\text{GPa.m}^{1/2}$.

Conclusions

The (100-x)AM-xZY ($x = 40, 50, 60$) composite ceramics with five different sintering conditions between normal sintering and two-step sintering method were successfully synthesized. The effect of heat treatment and various content of AM-ZY was studied by characterization of densification, phase analysis, morphology and mechanical properties. The optimal mechanical properties using two sintering conditions was demonstrated by 40AM-60ZY composite ceramics using TS-3 condition as T_1 temperature at 1650 °C for 30 min and T_2 temperature at 1450 °C for 5 h and obtained from 40AM-60ZY composite ceramics using normal sintering from NS-5 condition with sintering temperature at 1650 °C for 5 h.

These samples had a high percentage fraction of *t*-ZrO, *c*-ZrO₂ phase composition, and they showed high bulk densities of ceramic with the well-packed and continuous uniformed grain structure. The (100-*x*)AM-*x*ZY (*x* = 40, 50, 60) composite ceramics with a high content of AM are suitable for using two stage sintering to suppress abnormal grain growth when using high sintering temperature for a long times.

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